THE NATIONAL CARBON CAPTURE CENTER
AT THE POWER SYSTEMS DEVELOPMENT FACILITY

TOPICAL REPORT

BUDGET PERIOD THREE
JANUARY 1, 2011 – DECEMBER 31, 2011

DOE COOPERATIVE AGREEMENT
DE-NT0000749

Prepared by:
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ABSTRACT

The Power Systems Development Facility (PSDF) is a state-of-the-art test center sponsored by the U.S. Department of Energy and dedicated to the advancement of clean coal technology. In addition to the development of advanced coal gasification processes, the PSDF features the National Carbon Capture Center (NCCC) to study CO_2 capture from coal-derived syngas and flue gas.

The NCCC includes multiple, adaptable test skids that allow technology development of CO_2 capture concepts using coal-derived syngas and 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.

During the Budget Period Three reporting period, efforts at the NCCC/PSDF focused on testing of pre-combustion CO_2 capture and related processes; commissioning and initial testing at the post-combustion CO_2 capture facilities; and operating the gasification process to develop gasification related technologies and for syngas generation to test syngas conditioning technologies.

ACKNOWLEDGEMENT

The authors wish to acknowledge the contributions and support provided by various project managers, including Morgan “Mike” Mosser of the Department of Energy. The project is sponsored by the U.S. Department of Energy’s National Energy Technology Laboratory under Cooperative Agreement DE-NT0000749.

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LIST OF ABBREVIATIONS AND ACRONYMS

ACC—Aker Clean Carbon
B&W—Babcock & Wilcox
BOP—Balance of Plant
BP1 through BP3—Budget Periods One through Three
CCAD—Continuous Coarse Ash Depressurization
CEMS—Continuous Emissions Monitoring System
CFAD—Continuous Fine Ash Depressurization
CMS—Carbon Molecular Sieve
DCC—Direct Contact Cooler
DEPG—Dimethyl Ether of Polyethylene Glycol
DOE—Department of Energy
EPA—Environmental Protections Agency
EPRI—Electric Power Research Institute
FGD—Flue Gas Desulfurization
FTIR—Fourier Transform Infrared
GC—Gas Chromatograph
GTA—Glycerol Triacetate
HSS—Heat Stable Salts
ICP-MS—Inductively Coupled Plasma Mass Spectrometry
IGCC—Integrated Gasification Combined Cycle
MEA—Monoethanol Amine
MM5—Modified Method 5
MMD—Mass Median Diameter
MPT—Media & Process Technology
MS—Mass Spectrometry
MTR—Membrane Technology & Research
MTU—Mobile Test Unit
NCCC—National Carbon Capture Center
NDIR—Non-Dispersive Infrared
NETL—National Energy Technology Laboratory
NIOSH—National Institute of Occupational Safety & Health
OSHA—Occupational Safety and Health Administration
P&ID—Piping and Instrumentation Diagram
PC4—Post-Combustion Carbon Capture Center
PCD—Particulate Control Device
PDAC—Pressure Decoupled Advanced Coal
PDMS—Polydimethyl Siloxane
PRB—Powder River Basin
PSDF—Power Systems Development Facility
PSTU—Pilot Solvent Test Unit
R&D—Research and Development
R01 through R07—Tests Runs 1 through 7
RSAT™—Regenerable Solvent Absorption Technology
SCS—Southern Company Services
SCU—Syngas Conditioning Unit
SRD—Specific Reboiler Duty
SRI—Southern Research Institute
TCA—Total CO_2 Analysis
TROC™—Transport Oxy-Combustion
WGS—Water-Gas Shift
WPI—Worcester Polytechnic Institute

1.0 EXECUTIVE SUMMARY

The Power Systems Development Facility (PSDF) is a key national asset for ensuring continued, cost-effective, environmentally acceptable energy production from coal. Sponsored by the U.S. Department of Energy's (DOE's) National Energy Technology Laboratory (NETL), the PSDF is an engineering scale test center located in Wilsonville, Alabama. The PSDF staff has effectively developed advanced power systems to meet the national need for cleaner, more efficient power production from coal. Building on its previous success, PSDF now houses the National Carbon Capture Center (NCCC), established in 2009 to address the nation's need for cost-effective, commercially viable CO_2 capture options for flue gas from pulverized coal power plants and syngas from coal gasification power plants.

The NCCC is leading the way in the development of lower-cost CO_2 capture technologies that will enable coal-based power generation to remain a key contributor to providing affordable, reliable, and clean power generation. The facilities accommodate a range of equipment sizes and provide commercially representative test conditions that allow results to be scaled confidently to commercial application, a crucial element in shortening development times.

Project Partnership with DOE. The DOE conceived the PSDF as the premier advanced coal power generation research and development (R&D) facility of the world, to "serve as the proving ground for many new advanced power systems." Since operations began in 1996, the PSDF has been a center for national efforts to develop high efficiency, coal-based power generation technologies that are reliable, environmentally acceptable, and cost effective. Two significant achievements–in addition to many secondary goals that were met–were the development of hot gas filtration to improve energy efficiency and the development of a gasifier suitable for use with low-rank coals, which comprise over half of the total coal reserves in the U.S. and the world. These two technologies have progressed to commercialization with an integrated gasification combined cycle (IGCC) power plant under construction in Kemper County, Mississippi.

Project Mission and Approach. Offering a world-class neutral test facility and a highly specialized staff, the National Carbon Capture Center accelerates the commercialization of advanced technologies to enable coal-based power plants to achieve near-zero emissions. Work at the NCCC supports the next stages of coal-fired power technologies and the continued operation of conventional coal-fired power plants under CO_2 emission constraints.

In undertaking its mission, the NCCC is involved in a range of activities to develop the most promising technologies for future commercial deployment, thereby maximizing the impact of project funds. The test facilities, shown in Figure 1, include the original PSDF site, which houses the gasification process and pre-combustion CO_2 capture test site, and the Post-Combustion Carbon Capture Center (PC4), located at a major power plant on the same Alabama Power (Southern Company) property.

Figure 1. NCCC/PSDF Facilities.

Pre-combustion CO_2 capture is vital for the advancement of efficient gasification processes needed for state-of-the-art coal-fired power plants. The NCCC pre-combustion CO_2 capture facilities are designed to test an array of solvents, membranes, sorbents, and water-gas shift processes. Slipstreams are available with a range of gas flow rates and process conditions using coal-derived syngas.

The NCCC is also exploring post-combustion capture technology development. For both new and existing power plants, post-combustion CO_2 capture must be made more efficient and cost-effective by developing alternative technologies, such as solvents with lower heat of regeneration and lower cost equipment compared with the current technically viable but economically challenged options. The PC4 provides a wide range of process conditions for testing with coal-derived flue gas.

Further, the NCCC staff is evaluating oxy-combustion as a way to significantly reduce CO_2 emissions from coal-fired power generation. Researchers at the NCCC are building on their previous operational experience with the Transport Reactor to investigate the economic feasibility of operating it as an oxygen-blown, pressurized, circulating fluid bed combustor.

Work also continues in developing technologies specifically related to coal gasification processes. While the previous successful testing of the PSDF gasification process has led to commercialization of several processes (e.g., the Transport Gasifier and continuous fine ash removal systems), important work remains to make IGCC processes more reliable, efficient, and commercially viable. Including CO_2 capture in advanced coal power plants will increase the plant cost of electricity, so opportunities to reduce cost in every part of the gasification process are pursued. The considerable on-site expertise in gasification processes, as well as the unique testing opportunities afforded by the existing PSDF gasification process, make continued gasification process development a logical and cost-effective undertaking.

In partnership with the DOE, the NCCC established and met the milestones listed in Table 1 during Budget Period Three (BP3).

Table 1. Major Project Milestones for Budget Period Three.

Research Area	Milestone	Planned Completion Date	Actual Completion Date	Report Reference Section
Pre-Combustion	Conduct off-line test of additional CO_2 capture solvents in batch system	Mar 2011	Mar 2011	2.2
Post-Combustion	Commission Pilot Solvent Test Unit (PSTU)	Apr 2011	Apr 2011	3.2
Post-Combustion	Operate PSTU with baseline solvent	May 2011	May 2011	3.2
Gasification	Conduct gasification test run R06 and continue automatic temperature control improvements	Jun 2011	Sep 2011	5.3
Pre-Combustion	Develop the infrastructure to provide up to 50 lb/hr of clean syngas in support of CO_2 and hydrogen membrane testing through the expansion of the pre-combustion advanced membrane separation testing capability	Jun 2011	May 2011	2.0
Pre- and Post-Combustion	Complete annual technology screening to aid in setting BP4 priorities	Jun 2011	Jun 2011	--
Pre-Combustion	Conduct H_2 membrane testing for third party developers	Sep 2011	Sep 2011	2.0
Gasification	Conduct gasification test run R07, continue long-term filter element exposure testing, and support development of new filter elements and failsafes	Nov—Dec 2011	Dec 2011	5.5
Post-Combustion	Conduct testing of Babcock & Wilcox solvent in PSTU	Sep—Dec 2011	Dec 2011	3.4
Post-Combustion	Conduct testing of Aker Clean Carbon test module	Sep—Dec 2011	Dec 2011	3.3
Pre-Combustion	Complete SCU upgrade for syngas at 300 to 600ºC to new hydrogen membrane	Nov—Dec 2011	Dec 2011	2.0
Post-Combustion	Commission and test MTR CO_2 membrane	Nov—Dec 2011	Dec 2011	3.5
Pre-Combustion	Planning for upgrading buildings and electrical system	Dec 2011	Dec 2011	2.0
Gasification	Begin replacement of standpipe	Dec 2011	Dec 2011	5.1
Pre-Combustion	Support testing of CO_2 capture sorbent	Nov – Dec 2011	Dec 2011	2.7

Project Partners. Southern Company Services manages and operates the facility, and other project participants include the Electric Power Research Institute (EPRI) and leaders in the power and coal industries, including American Electric Power, Luminant, NRG Energy, Arch Coal, Peabody Energy, and Rio Tinto.

2.0 PRE-COMBUSTION CO_2 CAPTURE

To advance pre-combustion CO_2 capture technologies, the NCCC has the capacity to investigate key processes including gas/liquid contacting systems, solvents for CO_2 capture/separation, water-gas shift processes, and emerging syngas processes. The infrastructure for pre-combustion CO_2 capture testing provides for a wide range of test conditions, and includes the Syngas Conditioning Unit (SCU). During BP3, the SCU was modified, and various CO_2 capture and water-gas shift (WGS) tests commenced, as did support of gas separation membrane testing by outside researchers. Several technology developers also tested metallic hydrogen membrane coupons of advanced materials for syngas exposure in the SCU vessels.

2.1 SYNGAS CONDITIONING UNIT MODIFICATIONS

The SCU is a flexible slipstream facility that can accommodate multiple, small-scale tests, such as water-gas shift, COS hydrolysis, desulfurization, and CO_2 capture. The SCU, shown in Figure 2, consists of small reactor vessels, arranged to allow operation in series or in parallel, which accommodate a range of flow rates, temperatures, and pressures. The unit is also used to support outside technology developers by providing a test facility with various syngas conditions, and it has supported development of technologies such as CO_2 capture solvents, gas separation membranes, CO_2 capture solid sorbents, fuel cells, and heavy metal removal processes.

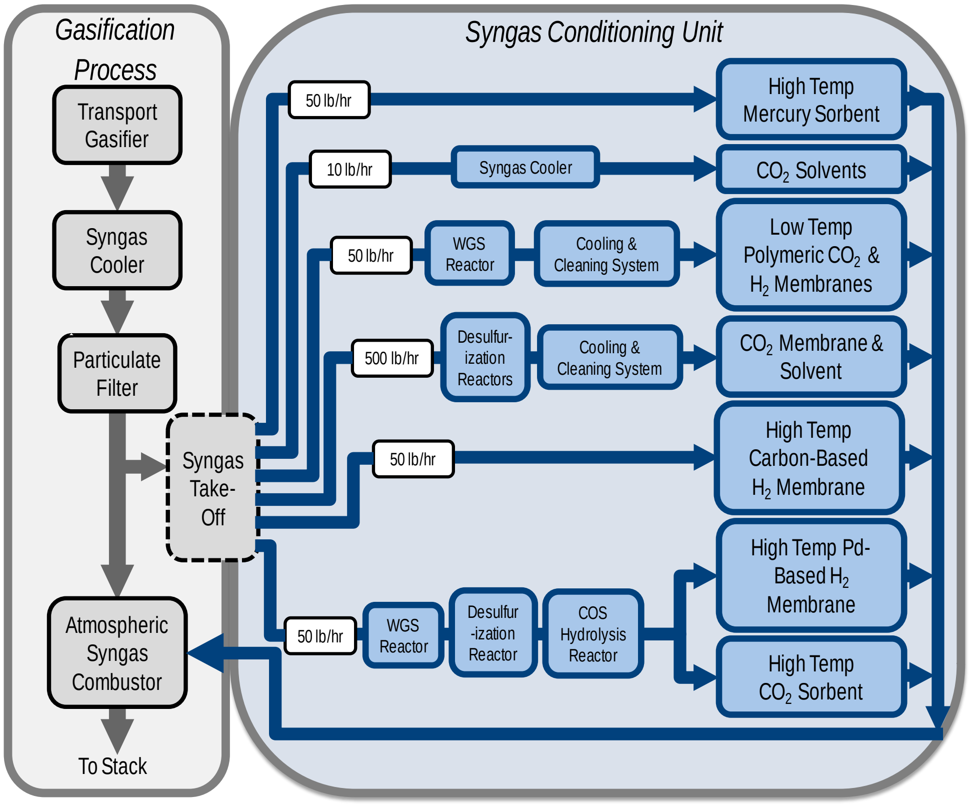


Figure 2. Flow Diagram of the Syngas Conditioning Unit.

SCU Modifications. Upgrading the SCU continued during BP3 to accommodate the ever-expanding range of technology development. The most significant of these modifications included:

- Commissioning and utilizing the new syngas treatment system upstream of the Membrane Technology & Research (MTR) membrane, and identifying improvements to be made prior to test run R07. The new system consists of a syngas cooling and hydrocarbon-scrubbing vessel, circulating pump, heat exchanger, chiller, and associated piping, instrumentation and electrical power.
- Identifying modifications for supplying shifted and desulfurized syngas to the Worcester Polytechnic Institute (WPI) and TDA Research skids off a shared sub-header and designing and installing the needed flow control, flow measurement, and pressure control capabilities
- Preparing for Media & Process Technology's (MPT's) new 50 lb/hr test skid, which involved reviewing design documentation prior to the equipment arrival to the site, adding a structure and concrete pad, and installing syngas supply/return, power, input/output capability, and utility connections needed for run R07
- Incorporating additions to the water-gas shift reactor process to improve data quality, which included a cooling coil was to condense water from the syngas to determine moisture content, as well as a heating jacket and thermocouples to better regulate reactor temperature

2.2 BATCH REACTOR CO_2 CAPTURE TESTING

During the reporting period, NCCC researchers investigated CO_2 capture characteristics of the CO_2 physical solvent glycerol triacetate (GTA), first using bottled gases, and then with syngas during R06. The R06 testing was completed to confirm the initial CO_2 capture results and to evaluate the absorption and regeneration characteristics with H_2S. The GTA solvent was compared to the well-documented solvent dimethyl ether of polyethylene glycol (DEPG) and previously tested polydimethyl siloxane (PDMS).

Absorption tests with syngas at 160 psig and 104°F were run until the GTA was saturated with both CO_2 and H_2S. Reducing the pressure to atmospheric (flash regeneration) freed almost all the CO_2 and about half of the H_2S. The solution was then heated to temperatures varying from 104 to 237°F to determine the thermal regeneration characteristics of H_2S. The effectiveness of regeneration was determined from the absorption capacity of the GTA during the subsequent runs to saturation. Heating to 212°F freed almost all the H_2S remaining following flash regeneration.

Figure 3 presents some of the CO_2 absorption results collected with the three physical solvents tested at 68°F. In the absence of water, CO_2 capture efficiency with GTA was similar to that with DEPG, and both were superior to capture with PDMS. When 10 percent water was added to the solvents, the performances of GTA and PDMS was unaffected, but that of DEPG deteriorated. Water is immiscible with GTA and PDMS but is soluble in DEPG, and this may account for the difference in performance. System analysis is required to determine if this difference offers any economic advantage.

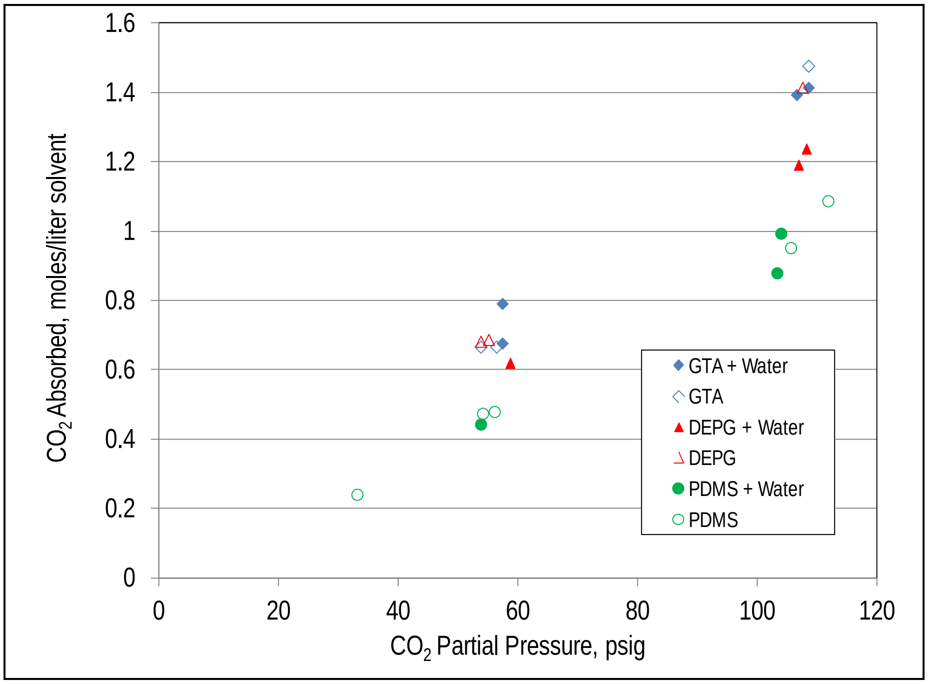


Figure 3. Absorption of CO_2 with and without Water Addition for Three Solvents.

2.3 WATER-GAS SHIFT CATALYST TESTING

For R06, a previously untested catalyst was installed in the WGS reactor. This catalyst was tested for a total of 480 hours. Parametric testing indicated that variations in the syngas inlet temperature and flow rate did not significantly effect the conversion of CO. The modified moisture measurement procedures were successful in improving heat and mass balance closures for the data. No degradation of the catalyst was found during the post-run inspection.

During R07, another vendor's previously untested WGS catalyst was tested for a combined 850 hours. A series of parametric tests were performed to evaluate the effects of inlet steam-to-CO ratio, temperature, and pressure. Following the parametric testing, the catalyst was tested another 730 hours for long-term material assessment at constant conditions, as defined by the catalyst vendor. Analysis indicated that good mass balance closures were achieved, giving confidence in the data collected. The catalyst demonstrated stable CO conversion of 70 percent at a steam-to-CO ratio of 1.0, which agreed well with the vendor's laboratory test data. Compared to commercially available WGS catalysts, the catalyst showed higher activity at lower temperatures and lower steam-to-CO ratios. The effect of pressure was not significant over the small range of pressure tested (from 128 to 182 psig). Post-test inspections showed no deterioration of the catalyst.

Figure 4 presents data for the four WGS catalysts tested to date and shows how the measured CO conversion varies with steam-to-CO ratio. The trend for all data sets is asymptotic, with catalysts B and D having higher conversions than A or C. However, the latter two catalysts may have lower costs or better resistance to contamination. At a steam-to-CO ratio of 1.6, all four catalysts convert sufficient CO to support CO_2 capture of around 65 percent and achieve emissions equivalent to those from a natural gas combined cycle process.

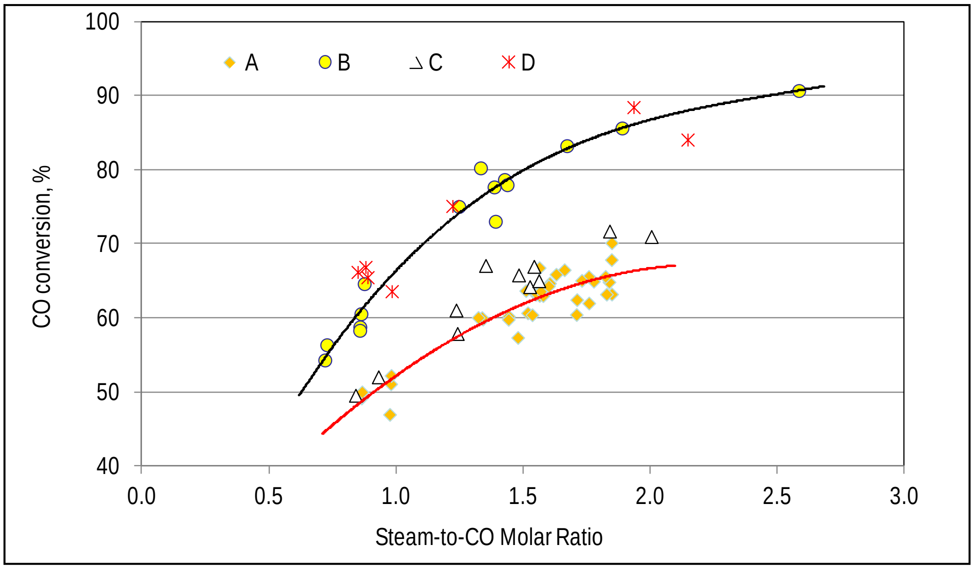
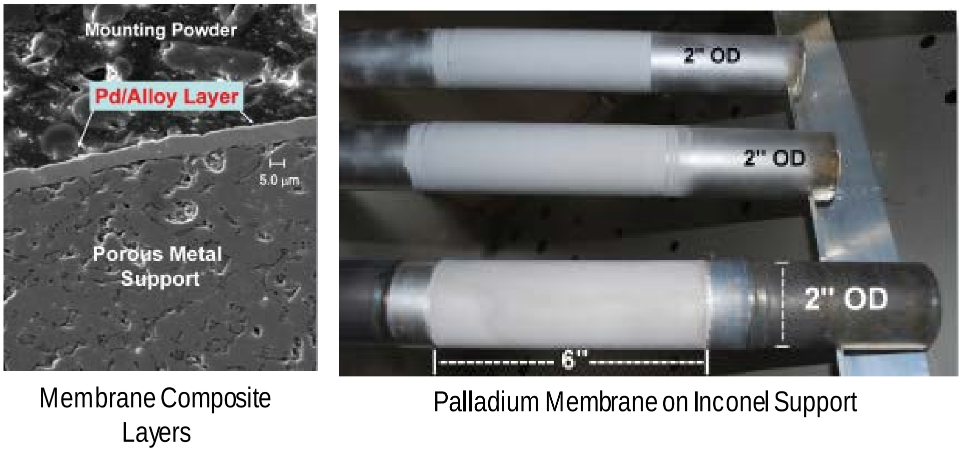


Figure 4. Performance of Four Water Gas Shift Catalysts.

2.4 WORCHESTER POLYTECHNIC INSTITUTE HYDROGEN MEMBRANE

The Center for Inorganic Membrane Studies at WPI is developing a palladium-based hydrogen membrane to separate hydrogen from syngas for power generation. The palladium causes hydrogen molecules to dissociate into atoms that diffuse across the membrane to produce pure hydrogen. Rather than the more common ceramic supports, WPI places the palladium on a porous metal support. The greater strength of the metal allows the palladium membranes to be thinner and less expensive to manufacture, and easier to integrate into reactors and other process equipment. Figure 5 shows the membrane components.



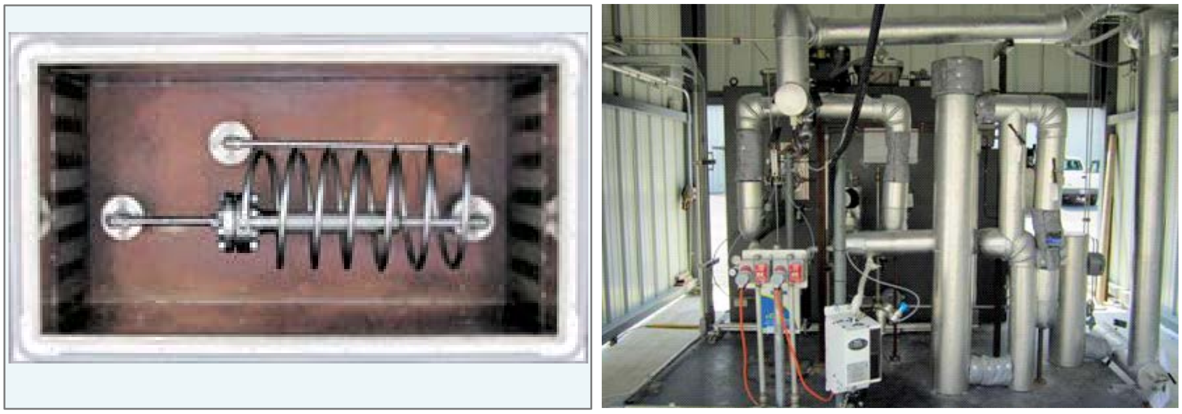
Membrane Composite Layers

Palladium Membrane on Inconel Support

Figure 5. WPI Membrane Components.

In July 2010, WPI received a DOE award to test its palladium and palladium alloy membrane technology to separate hydrogen and carbon dioxide from coal-derived syngas. Testing was initiated at the NCCC. The project team also includes Membrane Technology and Research (providing the skid design and construction), Siemens and T3 Scientific (providing a sulfur resistant coating).

WPI's membrane skid was designed to produce at least 2 lb/day of hydrogen from coal-derived syngas. The skid, consisting of an oven, piping spool, sulfur guard bed, chiller, and buffering vessel, was delivered in April 2011. NCCC staff completed installation of various items including seal-welds on threaded joints, heat tracing, piping insulation, nitrogen cylinders for heat-up and cooling, and a zinc oxide catalyst in an upstream pressure vessel for sulfur polishing. Membrane coupons were also installed in the zinc oxide vessel in the space above the catalyst for syngas exposure testing. Figure 6 shows the membrane module assembly inside the oven (which is designed to maintain an operating temperature of 842°F, or 450°C) and the skid piping and vessels behind the oven. The skid processes 10 lb/hr syngas with 0.2 to 0.3 lb/hr hydrogen enrichment to achieve a hydrogen concentration of about 33 vol%.



Membrane Installed in Oven

Skid Piping and Equipment

Figure 6. WPI Membrane Installed in Oven.

Test run R06 was the first time WPI membranes operated on coal-derived syngas. Three different palladium-based hydrogen membranes, M-01, M-03, and M-04, were tested. The principal characteristics of these membranes are shown in Table 2, and a view of the pure palladium and gold-coated palladium membrane tubes is provided in Figure 7.

Table 2. Characteristics of the WPI Hydrogen Membranes Tested in R06.

Membrane	Palladium Thickness, microns	Gold Thickness, microns
M-01	8.2	0.16
M-03	10	0.20
M-04	13.1	None

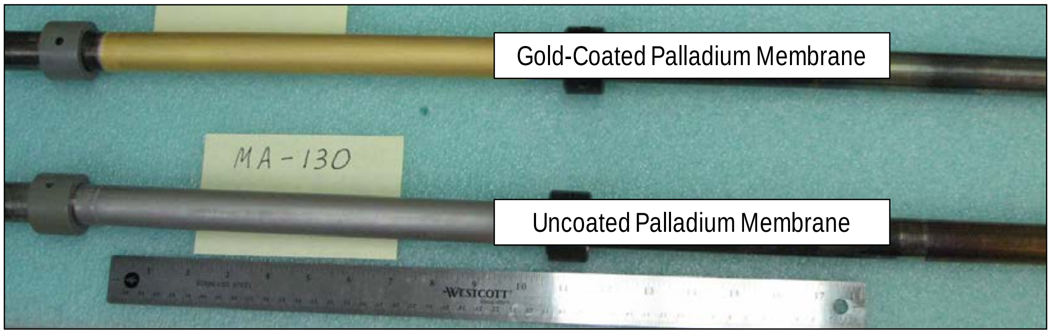


Figure 7. Photograph of Coated and Uncoated WPI Membranes.

All three membranes achieved high purity hydrogen production of 99.2 to 99.9 percent during R06. However, two out three membranes showed signs of leaks toward the end of the test as evidenced from the decline of hydrogen purity shown in Figure 8. Nevertheless, this initial test of palladium and gold-coated palladium membranes in coal-derived syngas was a success and demonstrated the potential for further development.

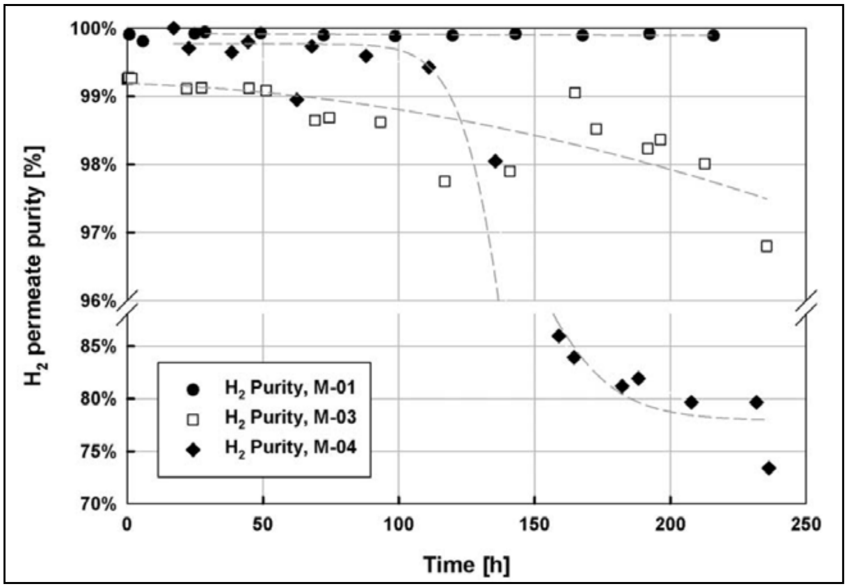


Figure 8. WPI Hydrogen Membrane Performance during R06.

In R07, a water gas shift reactor was incorporated to minimize the CO concentration in the syngas, which could cause coking on membrane surfaces. Two membranes were tested in R07 for a combined 646 hours, 582 hours in hydrogen-enriched syngas, with the balance in a nitrogen/hydrogen mixture. (A third membrane was prepared but not tested.) This length of time is a milestone in palladium membrane syngas operation. The principal characteristics of these membranes are listed in Table 3. Unlike observations in R06, no initial hydrogen flux decline was observed during R07. The flux stayed much more stable throughout the test. High purity hydrogen product—greater than 99.5 percent—was obtained as demonstrated by Figure 9. No leaks developed in either of these membranes for the duration of the test run.

Table 3. Characteristics of WPI Membranes Prepared for R07.

Membrane	Palladium Thickness, microns	Gold Thickness, microns
M-05	8.7	0.50
M-06	9.6	None
M-07 (not tested)	13	0.90

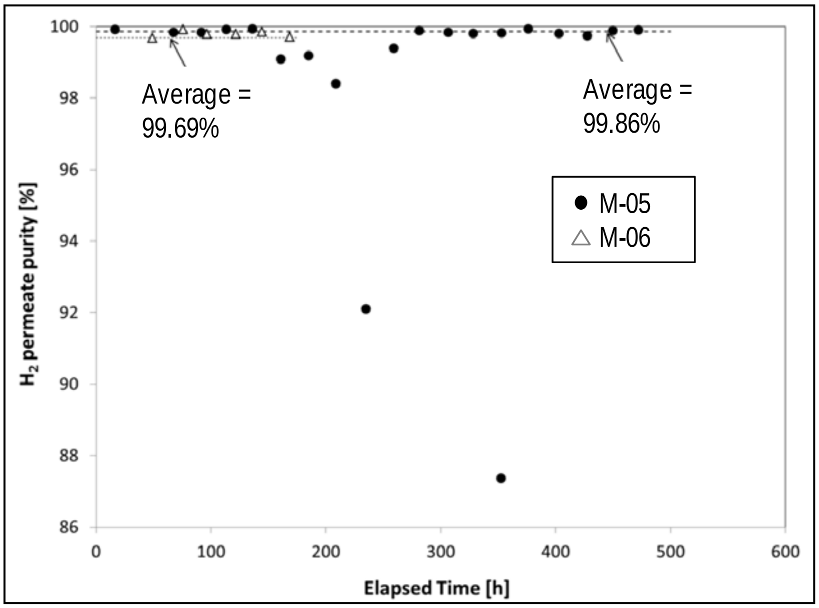


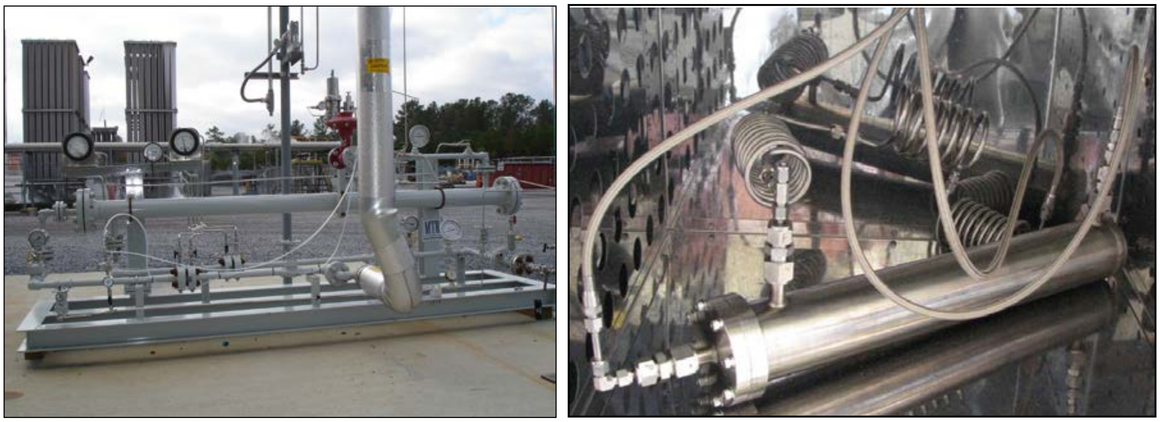
Figure 9. WPI Membrane Performance during R07.

Key observations from these two test runs are:

- WPI’s palladium membrane demonstrated an initial hydrogen flux high enough to produce 2 lb/day, reaching the DOE target of Phase I. However, the flux decreased to 1 to 1.4 lb/day for different membranes over the test duration.
- High purity hydrogen product, as high as 99.9 percent, and more than 470 hours of continuous operation were achieved at a pressure differential of about 11 bar with a syngas containing 35 vol% hydrogen. This represents a breakthrough for palladium-based membranes.
- WPI’s lab measurements of the hydrogen permeance before and after field testing show that 35 to 60 percent of initial permeance was lost. Most of the loss was not recoverable after exposure of the membrane to pure hydrogen at high temperatures. X-ray photoelectron spectroscopy analysis revealed the presence of a significant amount of carbon and lesser amounts of sulfur and mercury on a palladium layer. Carbon also penetrated into bulk palladium and could be the major reason for the flux decline. Continued testing and research efforts will focus on understanding the impact of these contaminants on membrane performance.

2.5 MEMBRANE TECHNOLOGY & RESEARCH CO_2 AND HYDROGEN MEMBRANES

In 2009, MTR began testing two types of polymeric membranes at the NCCC using coal-derived syngas: a CO_2-selective Polaris™ membrane and a hydrogen-selective Proteus™ membrane. MTR envisions using both of these membranes in a process to remove CO_2 from syngas. Membranes were tested during both runs R06 and R07 of BP3. Prior to R06, the hydrogen membrane was scaled up from a 1-lb/hr stamp cell to a 10 lb/hr module. For the CO_2 membrane, a conditioning system for the inlet syngas equipped with a chiller was added prior to R06 for improved hydrocarbon removal and temperature control. The membrane test units are shown in Figure 10.



CO_2 Membrane Skid Hydrogen Membrane Module

Figure 10. MTR Syngas Membrane Test Units.

Table 4 summarizes the representative syngas conditions for the two types of membranes.

Table 4. MTR Membrane Operating Conditions.

	CO_2 Membrane	Hydrogen Membrane
Syngas Flow Rate, lb/hr	50	10
Inlet Temperature, °F	90	250
Inlet Pressure, psia	170	170

Test results from the Polaris CO_2 membrane demonstrated:

- CO_2 enrichment as much as fivefold (see Figure 11), consistent with expected performance based on small-scale laboratory tests
- Stable operation in the presence of 300 to 500 ppm H_2S and mixed-gas H_2S/CO_2 selectivity of about 3.0
- Stable separation performance during the whole period of normal operation for the four modules, indicating that there was no aging or performance deterioration of the membrane modules after exposure to the raw syngas

- Higher selectivity for an improved membrane structure that was tested during R07 (modules 6478 and 6479 in Figure 11)

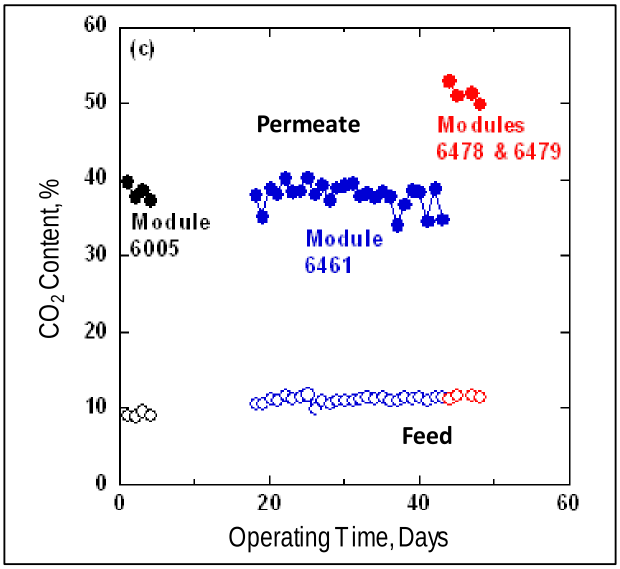


Figure 11. Typical Test Results for the MTR Polaris CO_2 Membrane.

Testing of the Proteus hydrogen membrane showed:

- Hydrogen enrichment up to 65 vol% in the permeate (see Figure 12), in agreement with expected performance
- Stable operation at temperatures up to 300°F for extended periods and in the presence of sulfur and other contaminants

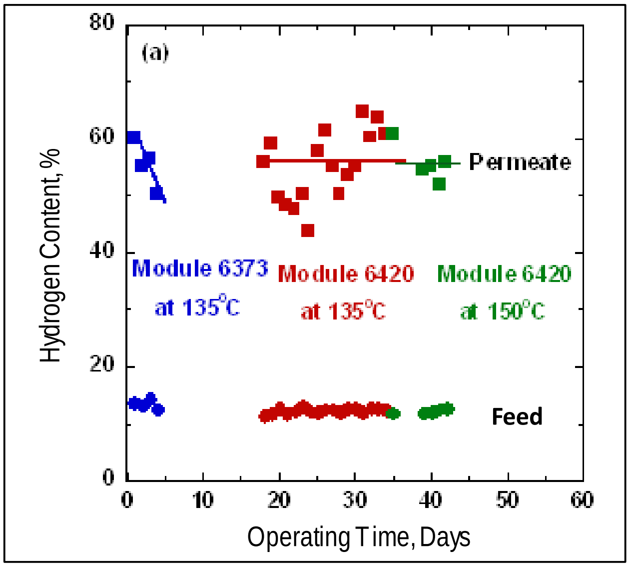


Figure 12. Typical Operating Results for the MTR Proteus Hydrogen Membrane.

In summary, both the CO_2-selective Polaris membrane and H_2-selective Proteus membrane have performed well using coal-derived syngas. The results are consistent with those from MTR's bench-scale test using simulated syngas. Stable membrane performance under industrial conditions is encouraging for further development of polymeric membrane technology.

2.6 MEDIA & PROCESS TECHNOLOGY HYDROGEN MEMBRANE

MPT first tested its carbon molecular sieve (CMS) membrane at the site in 2008, which marked the first time a hydrogen-selective membrane was successfully operated on untreated coal-derived syngas. The initial testing was conducted with a single tube configuration, and over time, the design has been scaled up to 10 and later 14 tubes, and, for R07, MPT tested an 86-tube module.

The 86-tube bundle was installed in the field test unit in mid-October 2011, and testing was conducted with this membrane for approximately 215 hours using raw syngas without any pretreatment. In general, the operating temperature and pressure were maintained at about 480°F and 200 psig. The permeate pressure was maintained at 3 to 5 psig. The feed rate of raw syngas was varied at times to assess the influence of gas flow rate on membrane performance, but in general, it was maintained at about 25 to 40 lb/hr. Figure 13 shows the hydrogen and CO_2 composition of the feed and permeate gas streams. Water content in the feed syngas was about 6 to 9 vol%, and the balance was primarily nitrogen with small amounts of CO. In addition, H_2S content of the feed was about 350 ppm. Under these conditions, the permeate hydrogen content was about 30 vol%.

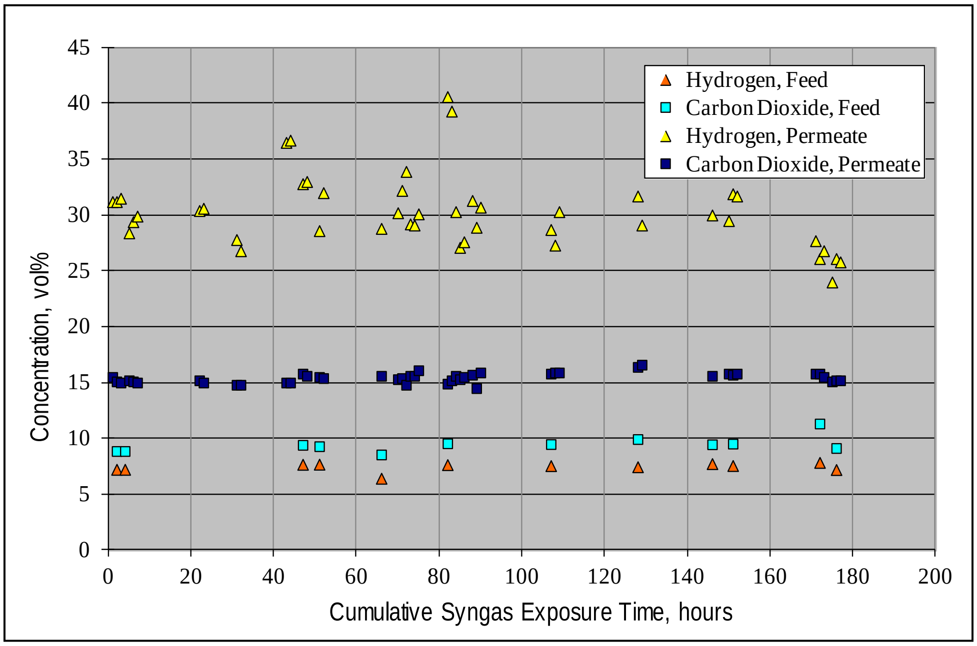


Figure 13. Operating Results with the MPT Carbon Molecular Sieve.

The primary goal of this phase of the CMS testing was to demonstrate the performance stability of the membrane and bundle in the presence of raw syngas with no pretreatment. In previous work at the NCCC, the single tube membranes were highly stable, particularly if operated at temperatures above 430°F to avoid condensation of tar-like species on the membrane surface. Periodic checks using He/N_2 mixture, presented in Figure 14, showed no deterioration in membrane permeability.

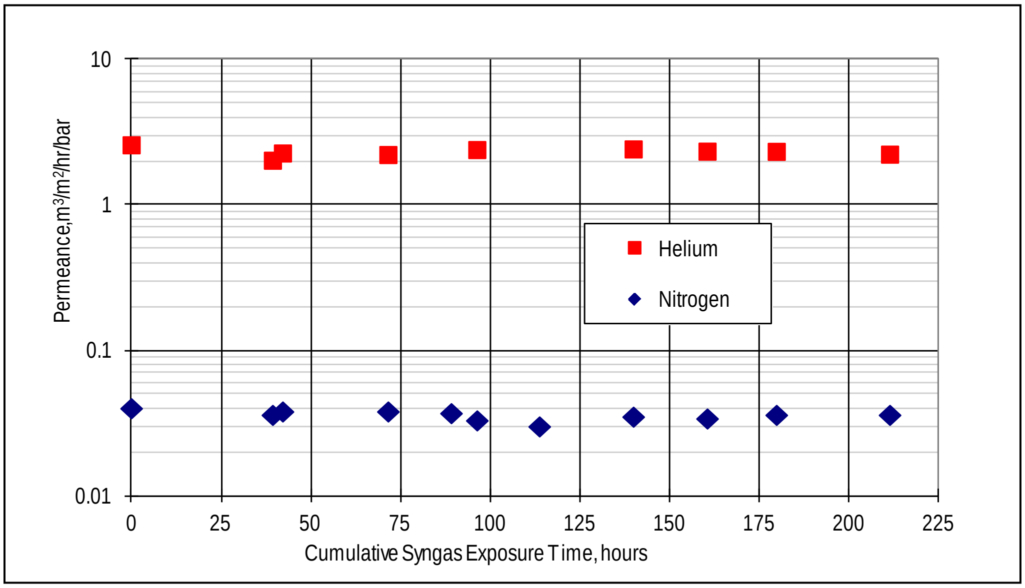


Figure 14. Result of Stability Tests with the Carbon Molecular Sieve.

Actual hydrogen permeability with syngas was approximately half the expected value, which may have been the result of poor syngas distribution in the bundle. MPT plans to test baffle arrangements to improve the distribution and improve effective permeability. Hydrogen purity leaving the CMS was around 80 percent with some H_2S present. This stream was passed over a sulfur sorbent and then through a palladium membrane, yielding hydrogen purity greater than 99 percent.

2.7 TDA RESEARCH SOLID CO_2 SORBENT

TDA Research is developing a new pre-combustion CO_2 capture technology under DOE funding. The objective of this project is to develop a low cost, high capacity CO_2 sorbent and to demonstrate its technical and economic viability for pre-combustion CO_2 capture. In addition to TDA Research, the project team includes MeadWestvaco (which will perform an estimate of the sorbent cost as a function of production volume) and University of California at Irvine (which will carry out a detailed engineering and economic analysis).

The sorbent is a proprietary carbon-based material modified with surface functional groups to physically adsorb CO_2 from shifted syngas. The CO_2 capture system operates at relatively high temperature (greater than 400°F) and uses four beds to operate based on pressure-swing adsorption and regeneration. Because the sorbent and the CO_2 do not form a true covalent bond, the energy needed to regenerate the sorbent (5.0 kcal/mol of CO_2) is much lower than that observed for either chemical absorbents (e.g., 29.9 kcal/mol CO_2 for sodium carbonate) or amine-based solvents (e.g., 14.2 kcal/mol CO_2 for monoethanolamine, MEA), and comparable to that of Selexol. If desired, the sorbent can be regenerated isothermally and CO_2 can be recovered at pressure (about 150 psia). Thus, the total energy needed to regenerate the sorbent and to compress the CO_2 for sequestration is expected to be significantly lower than that for any technology reported to date.

TDA Research's sorbent, which was tested in run R07, was the first CO_2 capture solid sorbent technology tested at the NCCC. The sorbent skid is designed to capture CO_2 from 10 lb/hr syngas. The CO_2 sorbent adsorbs CO_2 at 464^{o}F and syngas inlet pressure (about 190 psig) and releases CO_2 with heated N_2 purging at the same temperature and a pressure greater than 54 psig.

The raw syngas is treated in a water-gas shift reactor, a zinc oxide reactor for sulfur removal, and a COS hydrolysis reactor for sulfur polishing upstream of the skid. The skid, shown in Figure 15, consists of two cabinets. Cabinet #1 houses a syngas conditioning system including a zinc oxide sorbent guard bed in two reactors operating in parallel and a reactor used for additional syngas or nitrogen heating. Cabinet #1 also accommodates two gas analyzers. Syngas passes through Cabinet #1 and then Cabinet #2, which includes four CO_2 sorbent reactors and one accumulator. A chiller for sample syngas cooling is also installed in this cabinet.

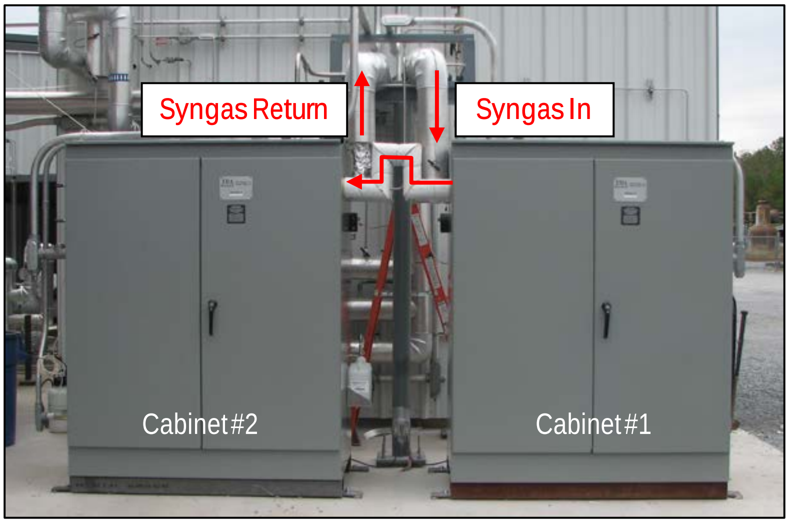


Figure 15. TDA Research Solid CO_2 Sorbent Test Skid.

The TDA sorbent skid operated during R07 for more than 1,200 cycles and over 300 hours on syngas. The four-bed CO_2 sorbent reactors operated as designed with multiple steps for each cycle, (adsorption, equalization, blow-down/regeneration, and re-pressurization). During these cycle tests, TDA's system successfully removed more than 98 percent CO_2 from the syngas. The sorbent regeneration cycles were carried out at pressures greater than 54 psig, resulting in recovery of CO_2 at pressure. Figure 16 plots representative field test data showing the CO_2 concentration in the feed and the product syngas. The feed CO_2 concentration was taken from a single measurement since a continuous measurement was not available.

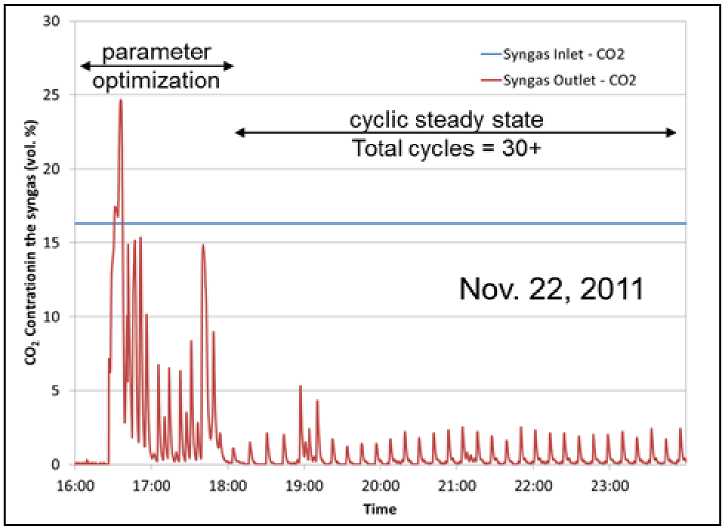


Figure 16. Representative Test Data for the TDA Research CO_2 Sorbent.

In order to optimize the sorbent performance, TDA varied the operating parameters such as bed temperature, cycle time, and the purge flow rate during the testing period. Figure 17 shows an example of the results from varying the bed temperature and regeneration pressure. The CO_2 capture increased from 98.3 to 98.7 percent.

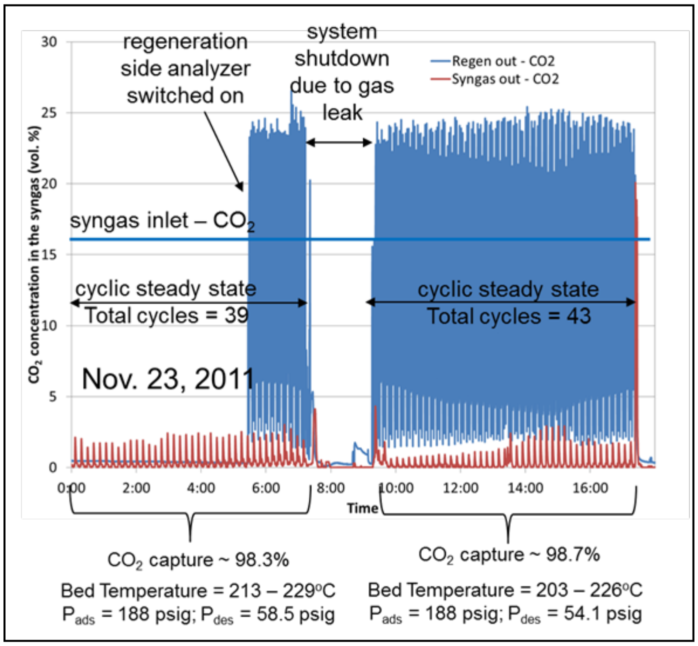


Figure 17. Parametric Test Data for the TDA Research CO_2 Sorbent.

Following test run R07, the skid was decommissioned and returned to TDA Research for modifications based on lessons learned during the run. TDA Research plans to bring the skid back to NCCC in 2012 with improved sorbents capable of WGS functionality.

3.0 POST-COMBUSTION CO_2 CAPTURE

The Post-Combustion Carbon Capture Center was constructed to advance flue gas CO_2 capture technologies at a range of operating conditions and technology development stages. A major area of the PC4 is the Pilot Solvent Test Unit (PSTU), which is designed to test absorption and regeneration of CO_2 solvents at flue gas rates up to 5,000 lb/hr (equivalent to 0.5 MWe). The PC4 also features test bays for larger units (up to 1.0 MWe) and for bench-scale units (up to 0.1 MWe each). See Figure 18. The site also includes a control room/administration building, electrical infrastructure, and a balance of plant (BOP) area containing utilities and chemical storage/handling facilities.

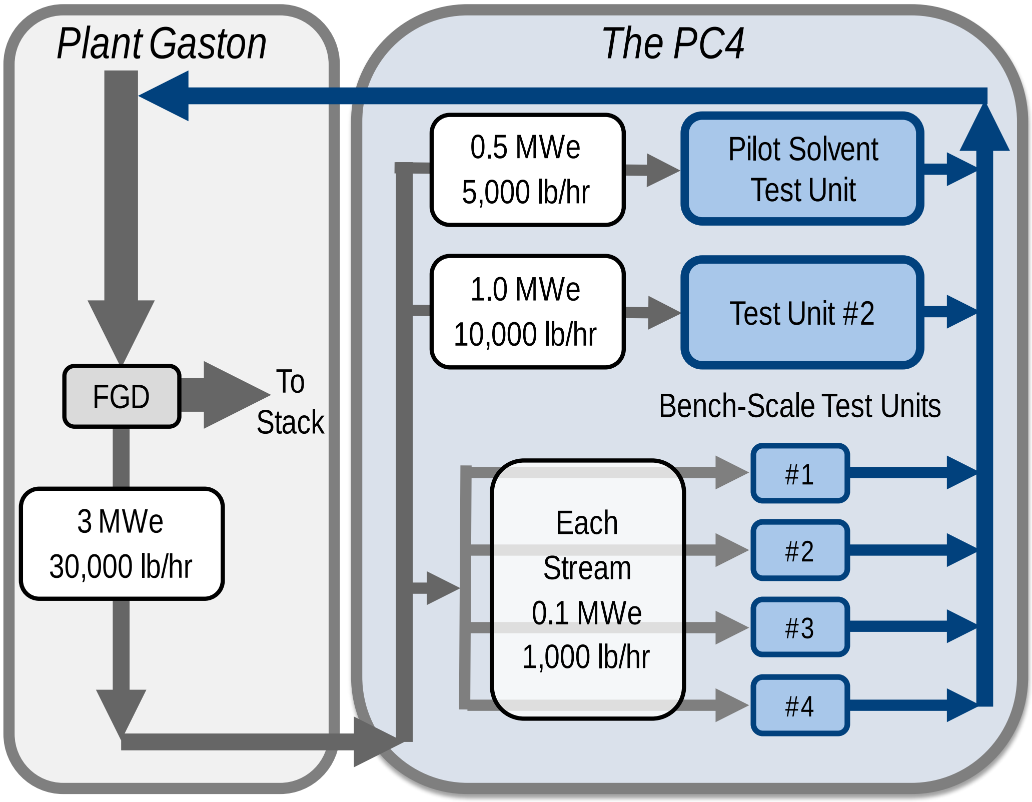


Figure 18. PC4 Test Facilities.

After completing final design, construction, and commissioning activities for the PC4 in early 2011, testing at the PC4 began in earnest. Commissioning of the PSTU was completed in May. Technology developers Aker Clean Carbon, Babcock & Wilcox, and MTR performed testing at the PC4 during the year.

3.1 DESIGN AND CONSTRUCTION

BOP Construction. Much of the final construction in the BOP area included electrical work, such as installing instrumentation, heat tracing, and wiring, and completing instrument loop checks. Some of the major systems completed in the BOP were:

- Steam Supply—Installation of the superheated steam piping from the extraction point on the Plant Gaston unit to the utility bridge was completed, as well as low and medium pressure steam header piping within the PC4 battery limits. All steam piping was tested and prepared for operation, and installation of related equipment, such as insulation, steam traps, and pumps were finalized.
- Chemical Storage Area—Work to finalize construction of the chemical storage area consisted of construction of a truck unloading station slab and piping for all chemical storage area systems, including caustic and amine storage, condensate collection and return systems, demineralized water, additive storage, and waste water systems. Electrical and instrumentation work was completed, which included installation of a continuous emissions monitoring system (CEMS).
- Fire Alarm System—Installation of the PC4 fire alarm system to allow monitoring from the PSDF site control room was completed. The initial design called for the more commonly used multi-mode fiber optic cable. However, the long distance from the PC4 control room to the PSDF control room resulted in signal loss. An improved design was implemented which utilized single-mode fiber optic cable to eliminate signal degradation, allowing the project to be completed.
- Instrument Air Compressor—Work consisted of the installation and commissioning of a second instrument air compressor and dryer to provide capacity for additional technologies and act as spare to the initial system.

3.2 PSTU COMMISSIONING

The PSTU was designed in 2009 and fabricated and installed in 2010. Construction of the balance of plant and system checkouts were completed in March 2011. Commissioning of the PSTU with a 20-wt% solution of MEA solvent began on March 22. MEA was used as the reference solvent against which other solvents are to be compared.

As operational and data collection issues were progressively resolved, the first balance period was completed on April 11, and testing was concluded on May 17, 2011, with nominal MEA concentrations of 20, 30, and 40 percent being evaluated. A total of 23 balance periods were completed, and the process data collected were carefully scrutinized to identify sources of error. No serious flaws were discovered, but a few corrections were made to calculation procedures.

For the major components, the mass balance closures achieved were all close to 100 percent, which is exceptional for such a large pilot plant and validates the accuracy of the flow and composition measurements. Heat balance closures were in the range of 95 to 100 percent, and were influenced by the quality of solvent specific heat data available in the literature. Test equipment for sampling MEA degradation products was established, and analytical procedures were identified for determining their concentrations in the CO_2-depleted flue gas stream leaving the absorber. Preliminary results indicated that any degradation products were present at very low levels. The system proved easily capable of greater than 90 percent CO_2 capture with the MEA solvent.

3.2.1 PSTU DESCRIPTION

The PSTU was designed to achieve 90 percent CO_2 capture using a 30 wt% aqueous MEA solution. This is the reference solvent to determine baseline performance against which other solvents tested will be compared. These solvents may include hindered amines, amino acid salts, and ionic liquids. To accommodate the range of solvent properties, the PSTU design is very flexible operationally. A schematic of the PSTU is presented in Figure 19.

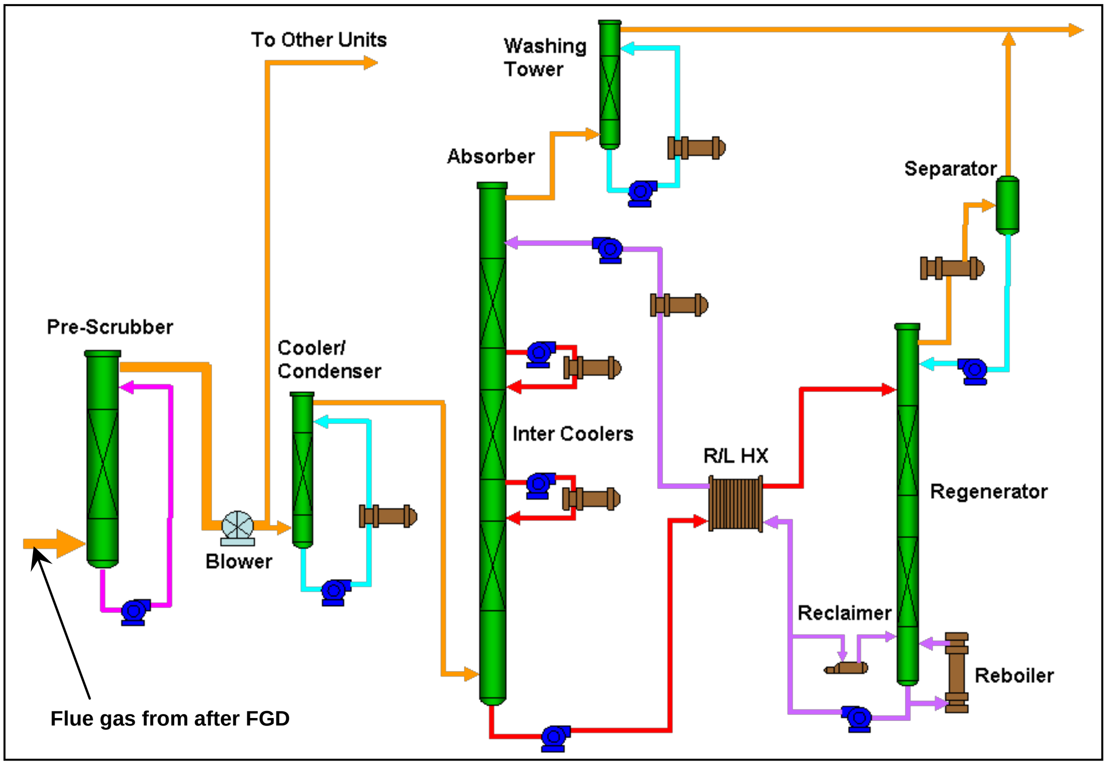


Figure 19. PSTU Schematic.

The PSTU was built off-site by McAbee Construction in Tuscaloosa, Alabama, in twelve modules. These were assembled prior to delivery to ensure that pipes were aligned and that there were no mechanical interferences. Overall, the quality of construction was excellent. One of the few mechanical issues experienced was that pump drives aligned at the fabrication facility required realignment once assembled at PC4, most likely as a result of movement during transportation.

3.2.1.1 SUBSYSTEMS

Up to 35,000 lb/hr of flue gas is extracted from downstream of the Plant E.C. Gaston Unit 5 flue gas desulfurization (FGD) system and is utilized for PC4 testing activities. Of this amount, 5,000 lb/hr of flue gas flows to the PSTU. There are five major columns, or subsystems, which include:

- The pre-scrubber which removes the small amount of SO_2 remaining in the flue gas
- A cooler/condenser unit that cools the flue gas to appropriate reaction temperatures and removes flue gas moisture
- An absorber to promote efficient gas-liquid contacting to remove CO_2 from the flue gas
- A washing tower that cools the CO_2-depleted flue gas, removing trace amounts of entrained solvent
- A regenerator that provides heat to release the CO_2 from the solvent

Further description of the PSTU subsystems has been published previously (see NCCC/PSDF Topical Report for Budget Period 2, which can be found at the web address www.NationalCarbonCaptureCenter.com).

3.2.1.2 INSTRUMENTATION

The PSTU is heavily instrumented to enable comprehensive process data collection and thorough characterization of equipment and solvent performance. The major control instrumentation and gas and liquid analysis methods are discussed briefly.

Flow Measurement. V-cone differential pressure flow meters are used to measure the untreated flue gas entering the pre-scrubber, the treated flue gas entering and the CO_2-depleted flue gas exiting the absorber, and the CO_2 stream exiting the regenerator. Coriolis meters are used to measure the flow of cool-lean solvent entering the absorber and cool-rich solvent exiting. These meters can also determine the liquid density, and so also serve to monitor solvent composition.

Gas Analysis. The gas analyzers used and their locations are listed in Table 5.

Table 5. Gas Analysis Techniques.

Stream	Species	Technique
Absorber Inlet	Oxygen	Zirconia sensor
	CO_2	NDIR
	Moisture	Capacitance
	SO_2	Ultra violet
Absorber Outlet	Oxygen	Paramagnetic
	CO_2	NDIR/FTIR
	Moisture	FTIR
	NH_3	FTIR
	NO_x	FTIR
	NO	FTIR
	NO_2	FTIR
	Amines	FTIR/condensation
Regenerator Outlet	Moisture	Capacitance
	CO_2	By difference

Liquid Analysis. An auto titration system is used to determine the solvent concentration and the CO_2 loading. The water concentration is determined by difference, although it can be determined by the Karl Fischer method if required. The liquid samples are extracted from these four locations:

- Hot-lean solution leaving the regenerator, typically 230°F
- Cool-lean solution entering absorber, typically 110°F, and having the same composition as the hot-lean solution
- Cool-rich solution leaving the absorber, typically 130°F
- Hot-rich solution entering the regenerator, typically 215°F with the same composition as the cool-rich solution

The auto titrator takes a sample automatically every 30 minutes, so each location is sampled once every two hours. To determine the CO_2 loading, the samples are titrated with potassium hydroxide and with sulfuric acid to determine the solvent concentration. The hot samples are cooled to around 100°F to prevent CO_2 flashing from the solution, thereby altering its composition.

The auto-titration values are crossed checked with laboratory analysis of samples taken manually from the cooled sample streams close to the time the auto-titration samples are taken. The total CO_2 analysis (TCA) procedure adapted at the NCCC is used to check the CO_2 content. This technique adds sulfuric acid to the solution and measures the volume of CO_2 released. The solvent content is cross checked using the laboratory titration procedure. Standard quality control checks, such as spiking the solvent concentration, are used to confirm the accuracy of the laboratory procedures.

3.2.2 OPERATIONAL EXPERIENCE AND LESSONS LEARNED

The following sections cover the major lessons learned during the commissioning activities.

3.2.2.1 OPERATION

Start-Up Team. As the design for PC4 was being finalized in January 2010, the team responsible for the PSTU commissioning operation began preparatory work. The team, consisting of process engineers and power plant operators, developed commissioning plans, operating procedures, clearance procedures, and training material in preparation for startup and operation. This preparatory work served to familiarize the team with the equipment, particularly during verification of process flow diagrams and process and instrumentation diagrams (P&IDs), which expedited start-up. The team also participated in training on amine-based plant operations taught by a third-party consultant.

Hydraulic Testing. In December 2010, water was circulated in the unit to check out the pumps, tune liquid circuit control loops, flush out debris, and check for leaks. Installation of heat tracing required more time and resources than was anticipated. To start the hydraulic test when planned and minimize delays to the startup schedule, commissioning and operation of the water piping were completed before heat tracing and insulation work were completed. Despite maintaining water circulation at all times, there was limited freeze damage to some valves and short pipe sections primarily at safety showers and piping headers blinded for future use due to the lower than expected ambient temperatures at the time.

Freeze Protection. System surveys performed to ensure freeze protection revealed that the rich/lean, plate-and-frame heat exchanger did not have a low-point drain. This design oversight was rectified following completion of the commissioning.

Caustic Solution Freeze Concerns. It was originally planned to store caustic at 50 wt% for use in the pre-scrubber and reclaimer. However, this solution freezes at 58°F and would have required extensive trace heating. To avoid this need, the specifications were revised for 25 wt% caustic, which freezes at 0°F.

Pump Filters. At the end of the hydraulic test, 3 wt% MEA was circulated to clean the absorber and regenerator of scale, particulate, and oils. The cleaning solvent flow was circulated through particulate filters and a carbon bed. The particulate filters were changed daily, and the cleaning process took approximately five days. Two of the circulating pumps had filters on their external seal flush systems that plugged frequently and slowed the cleanup process considerably. Discussions with the pump vendor revealed that the seal filters were finer than the main particulate filters. It was agreed to increase the seal filter pore size such that they did not blind as frequently but still provided adequate protection to the pump seals.

Seal Flush Connections. The external seal flush systems for the two circulating pumps included threaded connections that leaked when circulating the hot MEA solution. The threaded sections were replaced with Swagelok fittings that successfully eliminated the leaks.

Steam Blow. The reboiler heat exchanger, the steam supply lines, and the condensate return lines were all steam blown to remove scale. The blow took ten days, and the supply of steam had to be carefully coordinated with the power plant. Besides removing significant amounts of scale, a number of pin-hole leaks were identified and repaired.

Power Plant Output Fluctuations. Even with automatic control systems in service, disturbances occurred from perturbations in power plant operation. These can arise from system load changes resulting in variations in flue gas composition, unplanned de-ratings or shutdowns, and fluctuations in services such as filtered water supply. There is regular communication with the power plant operators to prepare for such perturbations, and the PSTU operating procedures include appropriate ways to respond to such events.

Equipment Alignment. The vessel internals were inspected by McAbee prior to shipment, and all items were declared to be correctly installed. Post-run inspection revealed that the liquid distributor in the absorber was misaligned and may have shifted during transportation and installation. Retrospectively, the internals should have been inspected once erected at the PC4 to detect and rectify such occurrences.

Solenoid Valves. The solenoids operating the block valves in the liquid lines proved unreliable. Attempts to improve performance were not fruitful, and all the solenoids were replaced with a different design.

Flue Gas Condensate Draining. Condensation occurring in the main flue gas header drawing saturated flue gas from the power plant required draining. The design includes automatic float valves which require considerable time to set up correctly. In hindsight, conventional traps would have worked equally as well under the slight vacuum of the header.

Removal of CO_2 in the Pre-Scrubber. A concern raised during process design was that the caustic used to remove residual SO_2 in the pre-scrubber, might also remove CO_2. Initially CO_2 was removed, but once a small amount of sodium carbonate was formed, no additional CO_2 capture was observed.

Process Controls. There are a number of process interactions and automatic control systems that are necessary to line out the system and to maintain steady conditions. Achieving this status was given high priority during the initial commissioning phase. Two important examples are given.

- Achieving a water balance and maintaining the required MEA concentration in the circulating solvent is a key process parameter that must be closely controlled. This is achieved by controlling the temperatures, and hence the humidity, of the two exiting gas steams (CO_2-depleted flue gas from the absorber and the CO_2 stream from the regenerator) at the same temperature as the flue gas entering the absorber. The on-line titration system monitors the solvent composition and allows operators to respond promptly to any changes in concentration.
- The cool-rich solvent and hot-lean solvent are pumped from the bottom of the absorber and regenerator, respectively. In both cases, the suction head is provided by maintaining a level of solvent at the foot of the column. The sump volumes are very small, and well-tuned automatic controls are essential to maintaining the level and so the suction head.

3.2.2.2 INSTRUMENTATION

Activities to validate process instrumentation and data collection during the commissioning and testing period are discussed briefly.

Liquid Flow Meters. During the water circulation tests, the absorber inlet and outlet liquid flow meters were checked volumetrically. A known flow for a given period was discharged into a tank and the change in level measured. Both flow meters were determined to be accurate. The flow meters during these tests indicated that the absorber exit flow was around one percent higher than that of the inlet. This is within the tolerance of the instruments and as it was not possible to determine which flow was inaccurate, no corrections were made.

Gas Flow Meters. Initially gas flow rates from the three V-cone meters were suspect. Specialists used duct traverses with pitot tubes to check the flow measurements of the following streams:

- Flue gas entering the absorber downstream of the condenser/cooler (S-type pitot)
- CO_2-depleted flue gas leaving the absorber (S-type pitot)
- CO_2 flow leaving the regenerator (straight pitot)

The traverses identified errors in the measured flow rates that were corrected. The supplier incorporated more appropriate gas densities in the calculation procedure. Also, low points in the impulse lines from the meter to the transmitter were removed that were collecting condensate and biasing the differential pressure, resulting in inaccurate flow determinations.

The flow meter at the inlet to the absorber had been specified for the maximum design flow, so during normal operation the differential pressure is less than half of full scale. This results in the meter being sensitive to minor perturbations at part-load flow rates.

Liquid Analysis. Based on the manual TCA and MEA analysis results and from preliminary mass balances, adjustments were made to the automatic titrator calibration curves. These adjustments are considered to be validated by the quality of the final mass balance closures achieved.

Gas Analysis. The CO_2 and oxygen analyzers are concluded to have operated with good accuracy as evidenced by the quality of the mass balance closures.

The only gas analyzer that gave problems was the Fourier Transform Infrared (FTIR) system used for specialized analysis of the CO_2-depleted flue gas stream leaving the absorber. The heating of the flexible pipe delivering the sample to the analyzer was insufficient to evaporate the water droplets in the stream, which were more numerous than expected. The excessive condensation stripped species such as ammonia from the gas, causing the analyzer to register low values, and of course, the moisture reading was also low. Fortunately, gas moisture content can be calculated with good accuracy from process data, so the ability to complete mass balances was not compromised.

To reduce the carryover of water droplets from the duct, a new demister was added, and the extraction probe was set at an angle to allow condensate to fall back into the duct. The effectiveness of this modification will be assessed during subsequent testing.

Other Instrumentation. Differential pressure measurements across the packed beds in the absorber and regenerator were unreliable. The impulse lines from the sensor to the transmitters are long, and temperature variations along the lines affect the pressure and bias the measured differential. This issue was alleviated partially by insulating the impulse lines, and work continues to reach final resolution.

3.2.3 MEA BASELINE TESTING AND DATA VALIDATION

The first mass balance period was completed on April 11 and the final one on May 17, 2011. During this 36-day period, Gaston Unit 5 took a five-day outage from April 23 to 27. The range of conditions covered is presented in Table 6. Although the intercoolers were tested, no balance periods were completed with the intercoolers in service. The reclaimer system also was not tested.

Table 6. Summary of Test Conditions.

Test Condition	Value
Total operating hours	1140
Number of balance periods	23
Balance period durations, hours	3 to 8
Absorber CO_2 mass balance closure, %	96.6 to 104.6*
CO_2 removal rates, %	58.5 to 98.9
Total CO_2 captured, tons	400
MEA concentration, wt. %	17.5 to 39.3 (nominally 20, 30 and 40)
Absorber inlet flue gas flow rates (G), lb/hr (kg/hr)	3,120 to 5,040 (1,410 to 2,290)
Absorber inlet liquid flow rates (L), lb/hr (kg/hr)	12,000 to 27,500 (5,440 to 12,500)
L/G mass ratio (liquid/gas)	2.78 to 5.69
Reboiler steam flow rates (S), lb/hr (kg/hr)	680 to 2,460 (310 to 1,120)
S/L mass ratio (steam/liquid)	0.044 to 0.121
CO_2 loading of absorber inlet MEA, mol/mol	0.123 to 0.343
CO_2 loading of absorber outlet MEA, mol/mol	0.409 to 0.551
Absorber inlet flue gas composition, vol% wet	
CO_2	11.4 to 12.9 **
Oxygen	4.6 to 7.1
Moisture	6.3 to 7.3
SO_2	Less than 1 ppmv
*(CO_2 in inlet flue gas)/(CO_2 in CO_2-depleted flue gas + CO_2 removed by solvent)	
**No CO_2 removed by caustic in pre-scrubber	

In completing the mass balances, some adjustments were made to the process data where appropriate. These are discussed below.

__Gas Mass Flow Rate Measurements.__ The flow from the differential pressure V-cone meters are determined by the following simplified equation:

$$M_g = k \text{ x } (dP \text{ x } \rho_g)^{0.5} \text{ x } Y$$

where:

M_g = mass flow rate of gas in lb/s
k = a constant incorporating g_c, meter dimensional parameters, and meter discharge coefficient
dP = differential pressure, inches of water
ρ_g = gas density at conditions upstream of meter, lb/ft^3
Y = gas expansion factor

The flow from the transmitter was pressure- and temperature-compensated, but the density was a fixed value regardless of gas composition. To gain a more accurate flow measurement, the variation in gas compensation and its effect on density must be accounted for. This is especially true for the CO_2-depleted flue gas stream leaving the absorber, where the CO_2 content varies inversely with capture efficiency. As part of the data analysis, the density was adjusted for gas composition. This is not required for operational control, where a representative fixed value for density will suffice. Also, linking the flow rate to the gas composition could be problematic during gas analyzer malfunctions.

Gas composition also affects the gas expansion factor, but the effect is too small to warrant correction.

The absorber inlet and outlet gas flows are considered reliable, but the CO_2 flow from the regenerator did not always match the CO_2 removed in the absorber. For the first ten runs, the CO_2 regenerator flow showed reasonable agreement, although the impulse lines had been connected the wrong way and the signal was reversed to compensate. During the five-day outage, the impulse lines were installed correctly, but thereafter the CO_2 regenerator flow rates were lower than those projected from the absorber data. This was likely due to a low spot in one of the impulse lines biasing the pressure differential over the meter. Having validated the meter, this fault was not detected until close to the end of the run. As there was good agreement between the CO_2 removed from the absorber gas and liquid flow streams, the average of these two values was used as the regenerator outlet flow.

Gas Analysis. For two of the periods, the absorber inlet oxygen analyzer was reading low, resulting in a poor oxygen balance closure. These errors occurred following calibration, so the values were changed to those immediately prior to calibration. The cause of the problem was rectified.

Liquid Analysis. For the majority of the runs, the values for MEA and CO_2 determined by automatic titration gave satisfactory mass balance closures. Some of the values for the 40 wt% MEA runs resulted in poor MEA and CO_2 closures and were replaced by values from analysis of the manual samples.

3.2.4 MASS BALANCE CLOSURES ACHIEVED FOR ABSORBER

The mass and heat balance envelope was originally drawn around the absorber and wash tower. This produced acceptable mass balance closures but resulted in some errors in the heat balances. The wash tower includes a cooling circuit and is instrumented to allow the heat removed from the CO_2-depleted flue gas to be estimated. For freeze protection, the cooling circuit is heat traced and insulated, and some of the heat removed by the cooler was introduced by the heat tracing. This was noted during a period when the flue gas flow was off but the cooling water temperature was still increasing. To avoid this error, the envelope was drawn around only the absorber. The flue gas composition was unchanged, and there was sufficient information to determine the moisture content at the wash tower inlet. The MEA content at the inlet was not estimated, but this flow is considered to be sufficiently low as to not introduce significant error.

Following a change in test conditions, process parameters were closely monitored to identify when steady conditions were re-established and the next test condition could proceed. In addition to temperatures and pressures throughout the absorber and regenerator, parameters monitored included:

- Liquid levels in vessels
- Flow rate of flue gas entering and CO_2-depleted flue gas exiting the absorber
- CO_2 content of flue gas entering and CO_2-depleted flue gas exiting the absorber
- Flow rate of CO_2 leaving the regenerator
- Flow rate of steam to the reboiler
- Flow rate and CO_2 content of lean amine entering the absorber and rich amine leaving (The CO_2 contents can be determined by chemical analysis or inferred from the density of the amine determined by the Coriolis mass flow meter.)

Once these parameters are steady, the solvent contained within the absorber and regenerator is allowed to be displaced one more time (for about 2 hours), after which the next test period can proceed. Figure 20 is a plot of key data trends in selecting the balance periods. The liquid solvent flow was increased to increase the liquid-to-gas (L/G) ratio and the steam flow to increase the steam-to-liquid (S/L) ratio, with the flue gas flow remaining constant. Once the solvent CO_2 loadings had stabilized, taking longer than the other parameters, the balance period was selected.

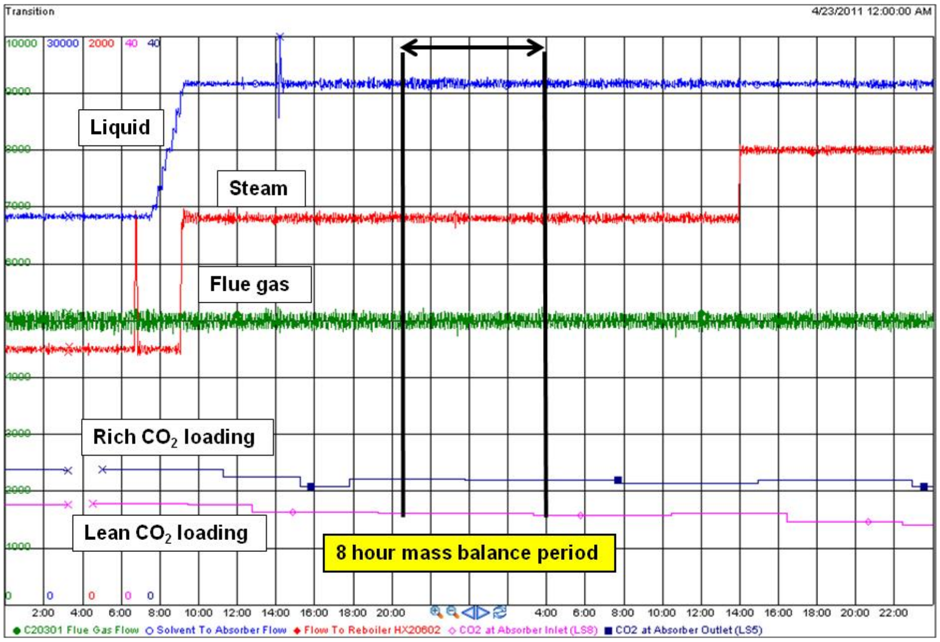


Figure 20. Typical Approach to Mass Balance Period.

The quality of the mass balance closures is demonstrated in a series of figures, as detailed below.

Figure 21 compares the total flows (gas plus liquid) entering and leaving the absorber. This is primarily a comparison of the liquid flows, as the liquid represents 75 to 85 percent of the total. There is a slight bias in the data, with the exit flow being on average 0.8 percent higher than the inlet flow.

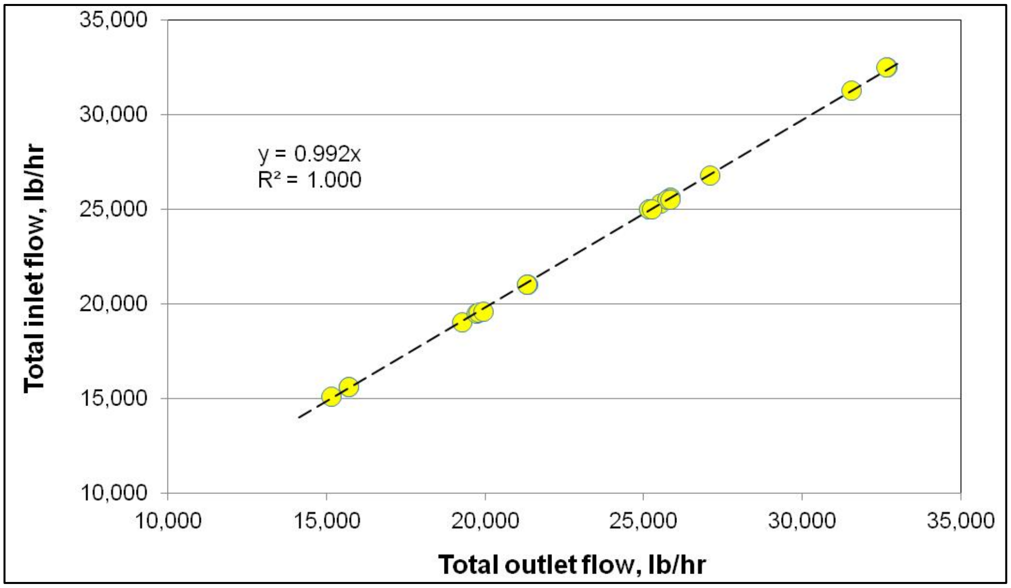


Figure 21. Comparison of Total Flows Entering and Leaving the Absorber.

Figure 22 compares the water flow (gas plus liquid) entering and exiting the absorber. Water represents 60 to 80 percent of the solvent flow. There is almost perfect agreement between the inlet and outlet flows.

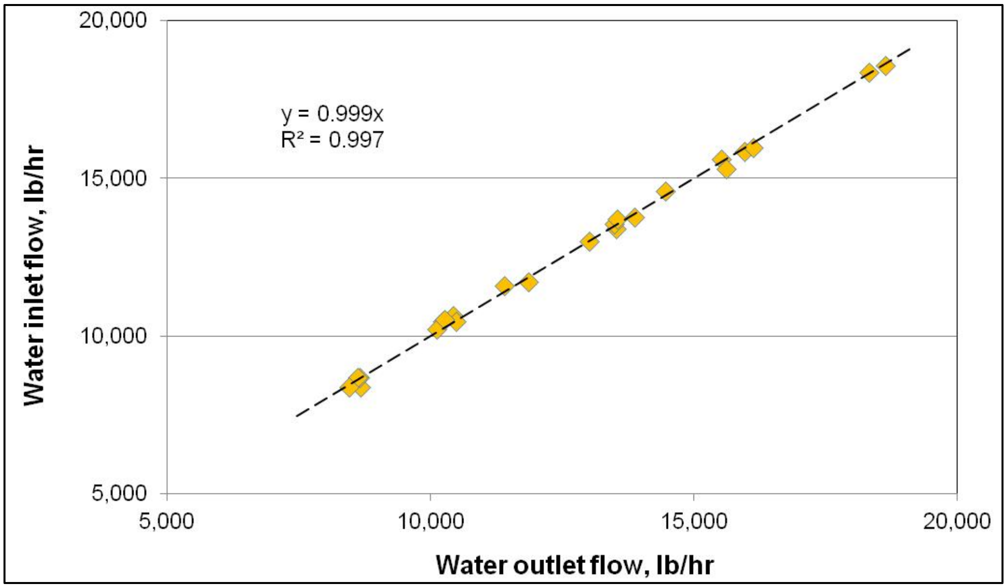


Figure 22. Comparison of Water Flows Entering and Leaving the Absorber.

Figure 23 compares the MEA flow entering and exiting the absorber. MEA represents 20 to 40 percent of the solvent flow. There is a slight bias in the data, with the exit flow being on average 0.7 percent higher than the inlet flow.

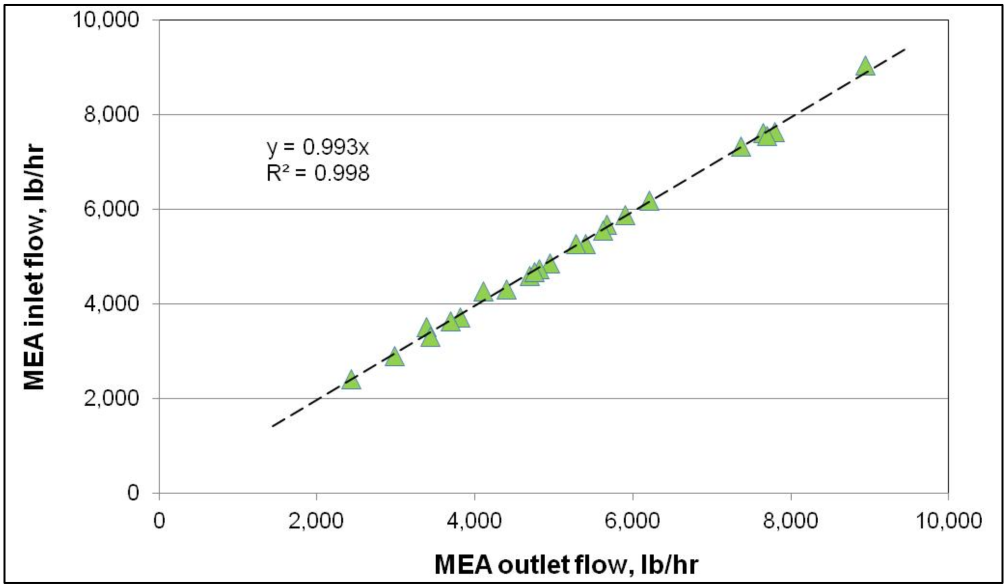


Figure 23. Comparison of MEA Flows Entering and Leaving the Absorber.

Figure 24 compares the total CO_2 flow (in gas and liquid) entering and leaving the absorber. CO_2 represents 5.2 to 9.1 percent of the total flow. There is a slight bias in the data, with the exit flow being on average 0.2 percent higher than the inlet flow.

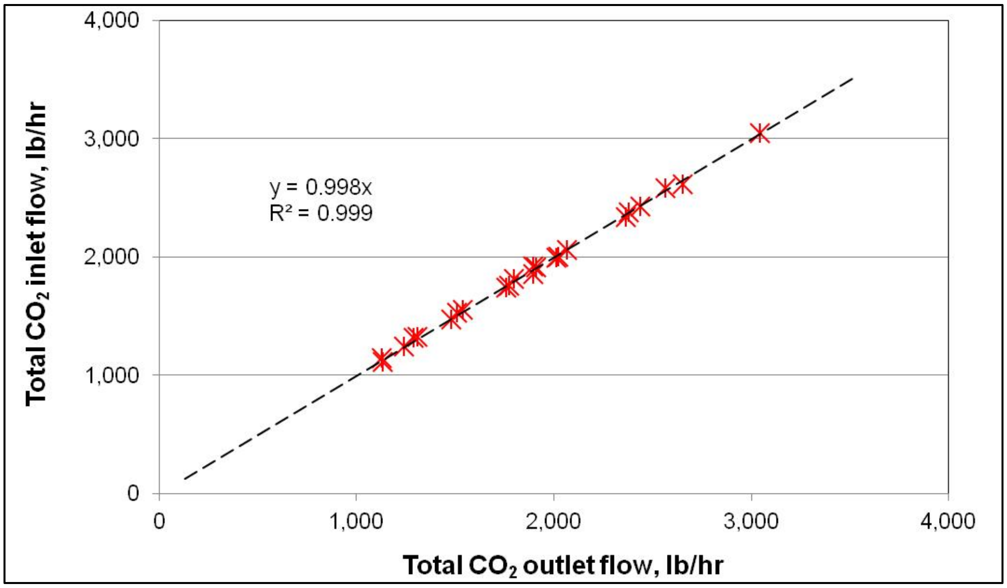


Figure 24. Comparison of CO_2 Flows Entering and Leaving the Absorber.

Figure 25 compares the CO_2 removed from the flue gas in the absorber to the CO_2 absorbed by the solvent. The two flows agree well.

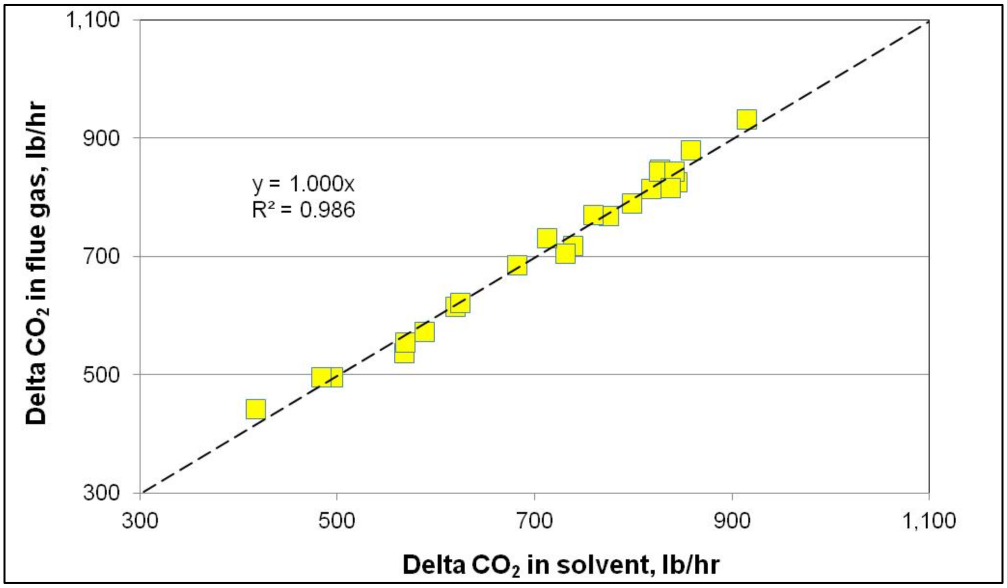


Figure 25. Comparison of Changes in CO_2 Content of Solvent and Flue Gas in Absorber.

Figure 26 compares the oxygen in the flue gas entering and exiting the absorber. There is a slight bias in the data with the exit flow being on average 0.3 percent higher than the inlet flow.

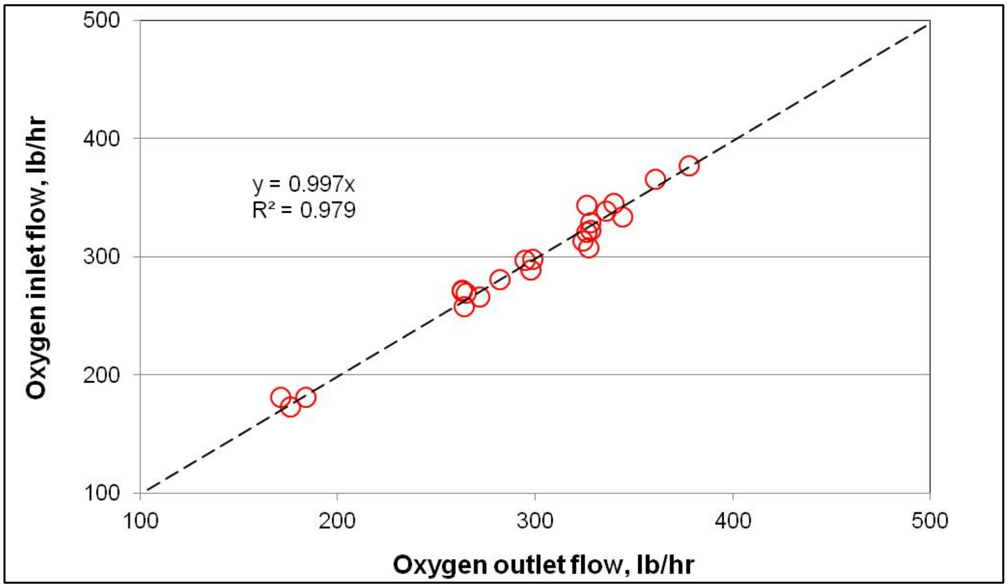


Figure 26. Comparison of Oxygen Flows in Flue Gas Entering and Leaving the Absorber.

Figure 27 compares the nitrogen in the flue gas entering and exiting the absorber. This is determined by difference, and the fit emphasizes the accuracy of the measured values, moisture, oxygen, and CO_2. There is a slight bias in the data with the exit flow being on average 0.1 percent lower than the inlet flow.

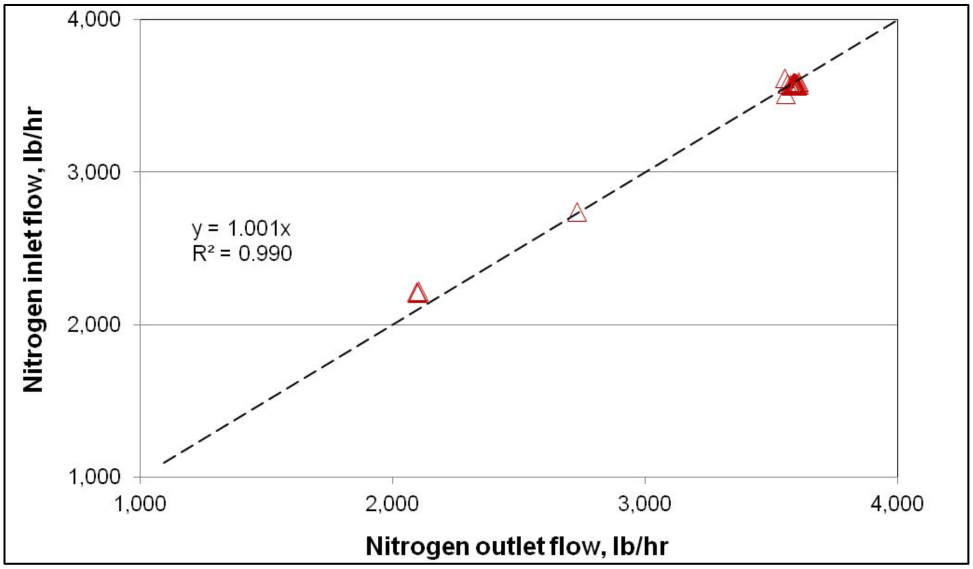


Figure 27. Comparison of Nitrogen Flows in Flue Gas Entering and Leaving the Absorber.

The data have an underlying bias with the outlet flows on average being slightly higher than the inlet flows. This slight bias is considered to arise from the small difference in liquid flow rate measurements discussed earlier. Nevertheless, the closures achieved on all species are excellent for such a large pilot plant and validate the accuracy of the flow and composition measurements. The only measurement found to be invalid was the CO_2 exit flow from the regenerator. (As discussed earlier, this most likely arose from a blocked impulse line, which has subsequently been rerouted.) The data are plotted in Figure 28 for the two periods, prior to April 28 and after April 28, (before and after the Gaston Unit 5 outage) as discussed earlier. The initial data show good agreement, but the later data are in less agreement.

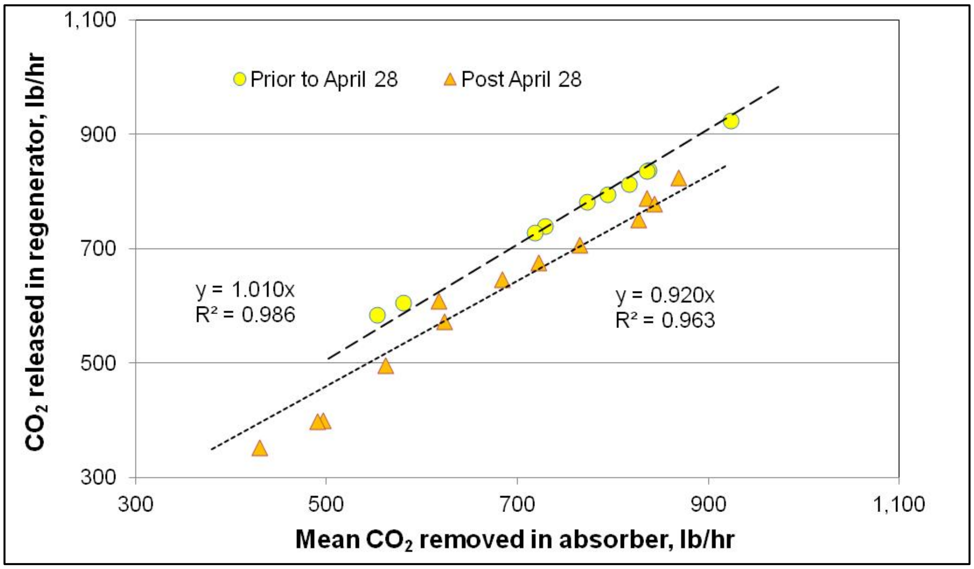


Figure 28. Comparison of CO_2 Released in Regenerator with that Removed in Absorber.

3.2.5 HEAT BALANCE CLOSURES ACHIEVED FOR ABSORBER

The liquid streams account for a large proportion of the absorber heat balance. For example, about 80 percent of the heat entering the absorber is in the liquid stream. Hence, the heat balance is very sensitive to the specific heat of the liquid, which varies with temperature, MEA concentration, and CO_2 loading. Values calculated from a commercial modeling software routine are presented in Figure 29 and Figure 30. The figures show, respectively, how specific heat varies with temperature and CO_2 loading for 30 percent MEA and how it varies with temperature and MEA concentration for 0.2 molar CO_2 loading. At higher temperatures, the values fall as vapor forms and contributes to the specific heat.

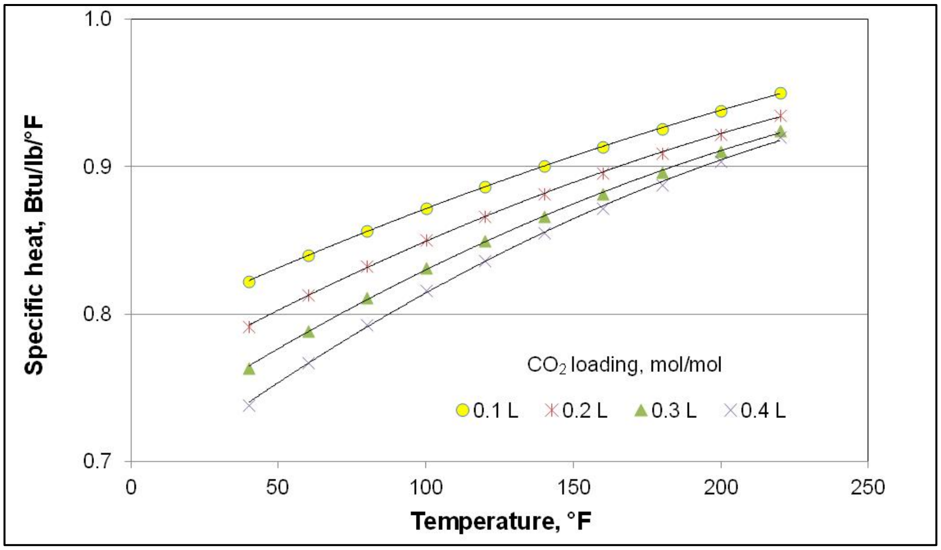


Figure 29. Variation of Solvent Specific Heat with CO_2 Loading and Temperature for 30 wt% MEA Solution.

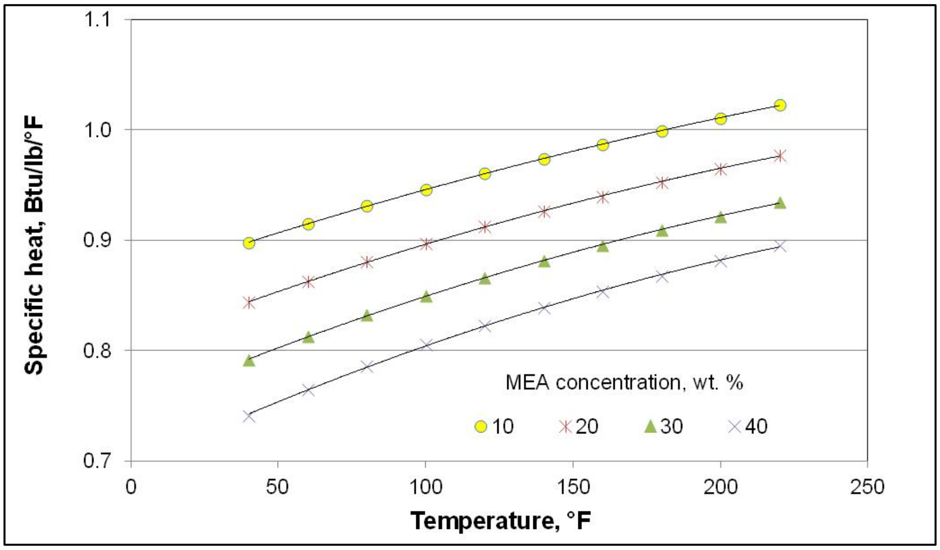


Figure 30. Variation of Solvent Specific Heat with MEA Concentration and Temperature for 0.2 Molar CO_2 Loading.

Values from the software were used in the formulation of the heat balances. Closures in the range 95 to 100 percent where achieved, which are acceptable but not as good as those for the mass balances. Given that the mass balance closures were so good, it was concluded that the predicted values for specific heat were inaccurate. At present, values predicted by other modeling software are being processed to improve the heat balance closures achieved. Additional solvent baseline tests would further improve the balances.

3.2.6 MEASUREMENT OF AMINE DEGRADATION PRODUCTS

The flue gas leaving the absorber of a commercial post-combustion CO_2 capture process discharges to atmosphere, so it is important to determine the amine and amine byproducts carried over in this stream. Southern Research Institute (SRI) developed the sampling and analytical procedures required to characterize the flue gas stream leaving the PSTU absorber.

To determine whether the vapor of the MEA-based materials could be collected by condensation, vapor pressure data were determined for some of the critical species expected to be present. Figure 31 presents the temperature dependence of vapor pressure for various species.

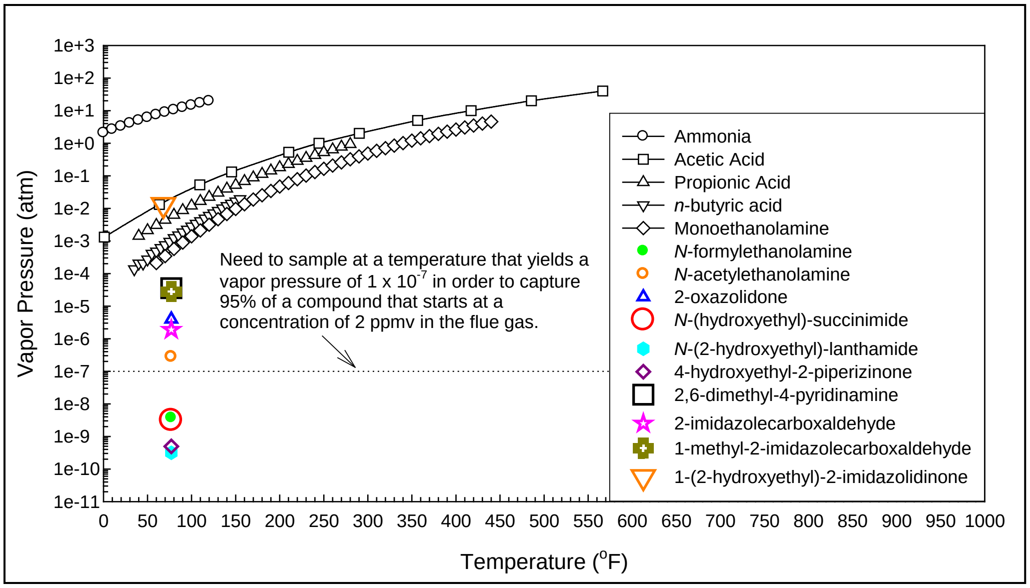


Figure 31. Vapor Pressure as a Function of Temperature for Various Amine Degradation Products.

Based on thermodynamic modeling and the assumption that the concentration of the species of interest in the flue gas exiting the wash tower is around 2 ppmv, to capture 95 percent of the species the vapor pressure would have to be below10^{-7} atmospheres. From Figure 31, very few of the degradation products have vapor pressures below this vapor pressure at 32ºF, indicating that condensation in an ice bath alone would not be effective at collecting all the degradation products. Therefore, the sampling system would need to include provisions for capturing the portion of the species that remains in the vapor phase after the ice bath. Because of heat transfer limitations, the ice bath typically only cools the sampled flue gas sample to 40°F, increasing the vapor-phase concentrations of the species present.

Based on the above discussion points, it was decided to use an Environmental Protection Agency (EPA) Modified Method 5 (MM5) extractive sampling train coupled with the use of sorbent tubes to capture the species that remain as vapor. The CO_2-depleted flue gas stream exits the wash tower at around 125°F and 15.1 psia and is saturated with water vapor with some droplet carryover. As the degradation products could be present in the flue gas as either vapor or droplets, the sampling is completed isokinetically to ensure that representative amounts of the various components are collected. To yield reliable data, saturating the resins in the sorbent tubes with moisture must be avoided. As so much moisture is present (saturated vapor plus droplets) in the gas stream, it is essential to collect all water droplets and reduce the dew point as much as possible. This approach collects almost all the moisture present in the sample, droplets and vapor, and the condensable portion of the degradation products present. The vast majority of the condensed material is water, resulting in very dilute solutions of the degradation products. Fortunately, there are analytical techniques that can measure the expected degradation products in the microgram-per-liter range. The vapors present in the stream leaving the cooler are collected in the sorbent tubes.

The MM5 sampling system used is illustrated in Figure 32. The flue gas sample passes directly through a condenser coil chilled with circulating ice water and then through three vessels sitting in an ice bath—a knockout trap, an open-tube impinger, and an impinger with an impact plate. The majority of the water (condensed vapor and droplets) is removed in the knock out trap, with very little collecting in the impingers. Particulate was not seen in the condensate, but tests with a heated filter will be carried out in the future to determine how much may be present.

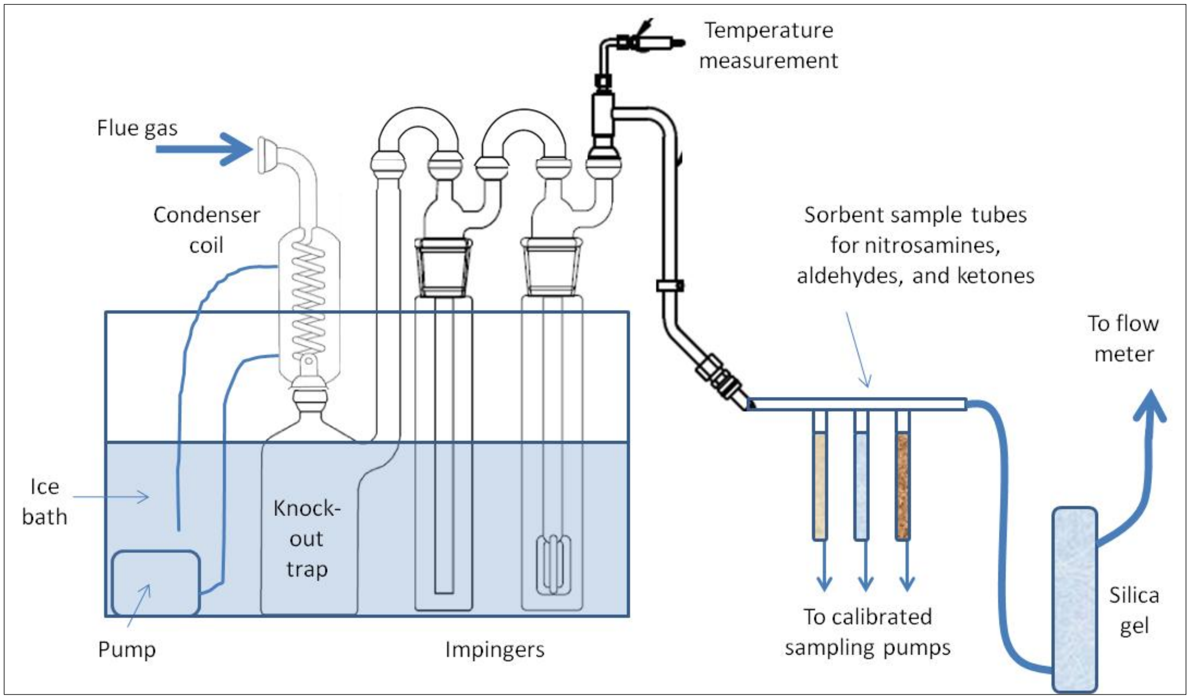


Figure 32. Sampling System Used to Measure Carryover of Degradation Products.

The exiting gas flow at around 40°F is drawn through separate sorbent tubes arranged in parallel that contain specially treated resins to capture the degradation products present as vapor. These tubes were developed for industrial hygiene applications to monitor exposure to contaminants in the work environment. The techniques for using and analyzing these tubes have been developed by the National Institute of Occupational Safety and Health (NIOSH) and the Occupational Safety and Health Administration (OSHA). Both organizations have conducted extensive testing of the tubes and the associated analytical procedures to demonstrate that they are accurate for the specified components.

The liquid samples and the sorbent tubes were analyzed for degradation products at outside laboratories in accordance with the appropriate NIOSH and OSHA analytical protocols. Table 7 provides the analytical methods used. For one run, the gas stream was bubbled through 0.02-N sulfuric acid to determine if this treatment stage reduced the amount of the degradation products present.

Table 7. Analytical Methods Used for Condensates and Sorbent Tubes.

Chemical Species in Condensate	Analytical Procedure	Technique
Volatile organic compounds including ketones	EPA 8260C	Gas Chromatograph (GC)/Mass Spectroscopy (MS)
Non-halogenated organic compounds including amines	EPA 8015B	Direct Injection GC
Carbonyl compounds including aldehydes	EPA 8315A	High-Performance Liquid Chromatography
Nitrosamines	RJ Lee Internal Method	Thermal Energy Analyzer
Chemical Species in Vapor	**Analytical Procedure**	**Sorbent Tube Description**
Ketones	NIOSH 1550	Coconut Shell (CS) Carbon
	OHSA 31	CS Charcoal for Total Hydrocarbons
Amines	OSHA 60	Naphthylisothiocyanate on XAD-2 Resin
Aldehydes	NIOSH 2532M	2,4-Dinitrophenylhydrazine on Silica Gel
Formaldehyde	NIOSH 2539 & OHSA 52	Hydroxymethyl Piperadine on XAD-2 Resin
Nitrosamines	NIOSH 2522	Thermosorb/N
	OHSA 31	Glass Fiber for Nitrosodiethanolamine

Preliminary results for the vapor measurements are summarized in Table 8.

Table 8. Summary of Degradation Products Identified in Flue Gas Exiting Absorber Wash Tower.

Species	Phase	Concentration, ppmv		OSHA Permissible Exposure Limit, ppmv
		Without Acid Wash	With Acid Wash	
Formaldehyde	Liquid * Vapor **	0.057-0.112 0.0047	<0.0023	0.75
Acetaldehyde	Liquid * Vapor **	0.015-0.179 0.562	0.093	200
Butylaldehyde	Vapor **	<0.0045	0.0049	None
Ethylamine	Vapor **	0.128	<0.0279	10
Dimethylamine	Vapor **	0.043	<0.0279	10
Nitrosomorpholine	Liquid * Vapor **	0.0287 0.000025	<0.000014	None
Nitrosodiethanolamine	Vapor **	0.00213	ND	TBD

* Compound present in water removed by knock-out trap expressed as the equivalent vapor concentration in flue gas
** Compound present in flue gas as vapor after knock-out trap measured by sorbent tubes
***ND = Not detected
****TBD = To be determined

The following observations are made from the available data with respect to the vapor phase:

- Only two nitrosamine compounds were detected above the lower detection limit of the analytical techniques employed; nitrosomorpholine and nitrosodiethanolamine were both detected well below the parts-per-million level in the vapor phase.
- Compounds detected at parts-per-billion level were formaldehyde, acetaldehyde, butylaldehyde, ethylamine, and dimethylamine.
- All compounds detected were well below OSHA Permissible Exposure Limits. These limits represent the safe weighted-average concentration for continuous exposure during an eight-hour work shift.
- Bubbling the flue gas sample through dilute sulfuric acid was effective in reducing emission levels, in some cases to below the lower detection limit. The reduction may also have arisen by bubbling through water; more testing is needed to understand this result.

3.2.7 SUMMARY OF MEA BASELINE TESTING

Construction and pre-commissioning of the PSTU was completed in early March, with commissioning proceeding as follows:

- March 10—Hot water circulation began in the absorber/regenerator circuit.
- March 22—Operation with 20 wt% MEA solution began.
- April 11 through May 17—Balance period testing was conducted.

Process issues were steadily resolved and control circuits tuned, allowing the plant to be operated in a controlled and safe manner at steady conditions. The process operated as designed, and flow rates and analysis procedures for the gas and liquid streams were all validated. This operational success demonstrated that the plant is ready to support testing of developers' solvents.

A total of 23 balance periods were completed, and the process data collected were carefully scrutinized to identify sources of error. No serious flaws were discovered, but a few corrections were made to calculation procedures, resulting in mass balance closures very close to 100 percent for the absorber. This successful outcome has two benefits:

- The developed understanding and the resolution of data collection issues raises confidence in the ability to provide developers with reliable information when testing their solvents. This will allow them to make technology development decisions with confidence.
- The data collected can be used with confidence by modelers within the Carbon Capture Simulation Initiative and contribute to accelerating the development of cost-effective CO_2 capture technologies.

Achieving good heat balances is complicated by the absence of reliable data for solvent specific heat, which is a function of temperature, MEA concentration, and CO_2 loading. Other data bases are being investigated to find suitable data.

MEA degradation products were measured in the CO_2-depleted flue gas stream exiting the wash tower. The degradation products are all at very low levels and, where they have been defined, well below OHSA Permissible Exposure Limits. Only two nitrosamines were detected, and these were present below the parts-per-million level. It is emphasized that these results are preliminary and that the sampling and analytical procedures are still under development.

3.2.8 ADDITIONAL MEA TESTING

A second MEA run would be beneficial in confirming the current process results and supporting the completion of higher heat balance closures. Other benefits include:

- The viscometers installed to support testing of Babcock &Wilcox's (B&W's) OptiCap™ solvent can also be used with MEA to increase the database on solvent properties. The Coriolis flow meters already determine solvent density.
- The sampling and analytical procedures for solvent degradation products will be refined through supporting the B&W testing and tests could be repeated to confirm the accuracy of the current data. Also testing could be carried out to determine the presence of degradation products in the CO_2 flow stream leaving the regenerator.
- A major MEA degradation product is ammonia, and because of the FTIR problems, this species was not monitored during operation. Such data can now be collected.
- Liquid samples could be analyzed using gas chromatography-atmospheric pressure chemical ionization time-of-flight mass spectrometry to identify the formation of species with time, establishing whether degradation products formed initially are a precursor to the formation of addition products.
- The reclaimer could also be operated to determine if the process to remove heat stable salts also removes degradation products and other solvent contaminants.

3.3 AKER CLEAN CARBON MOBILE TEST UNIT

Aker Clean Carbon (ACC) is a member of SOLVit – a major scientific research and development program established to produce cost-effective, improved amines for CO_2 capture from flue gases. This eight-year €40 million ($54million) program, funded primarily by Gassnova, was launched in 2008 together with Sintef, an independent research organization, and the Norwegian University of Science and Technology. In 2009, the energy companies E.ON, Scottish Power, and Stadtkraft joined the program.

A new laboratory was built at Trondheim and includes a 30-m tall, 0.2-m diameter processing column (98-ft x 8-inches). The height is similar to that of a commercial unit. The test objectives are to develop and test improved solvents with:

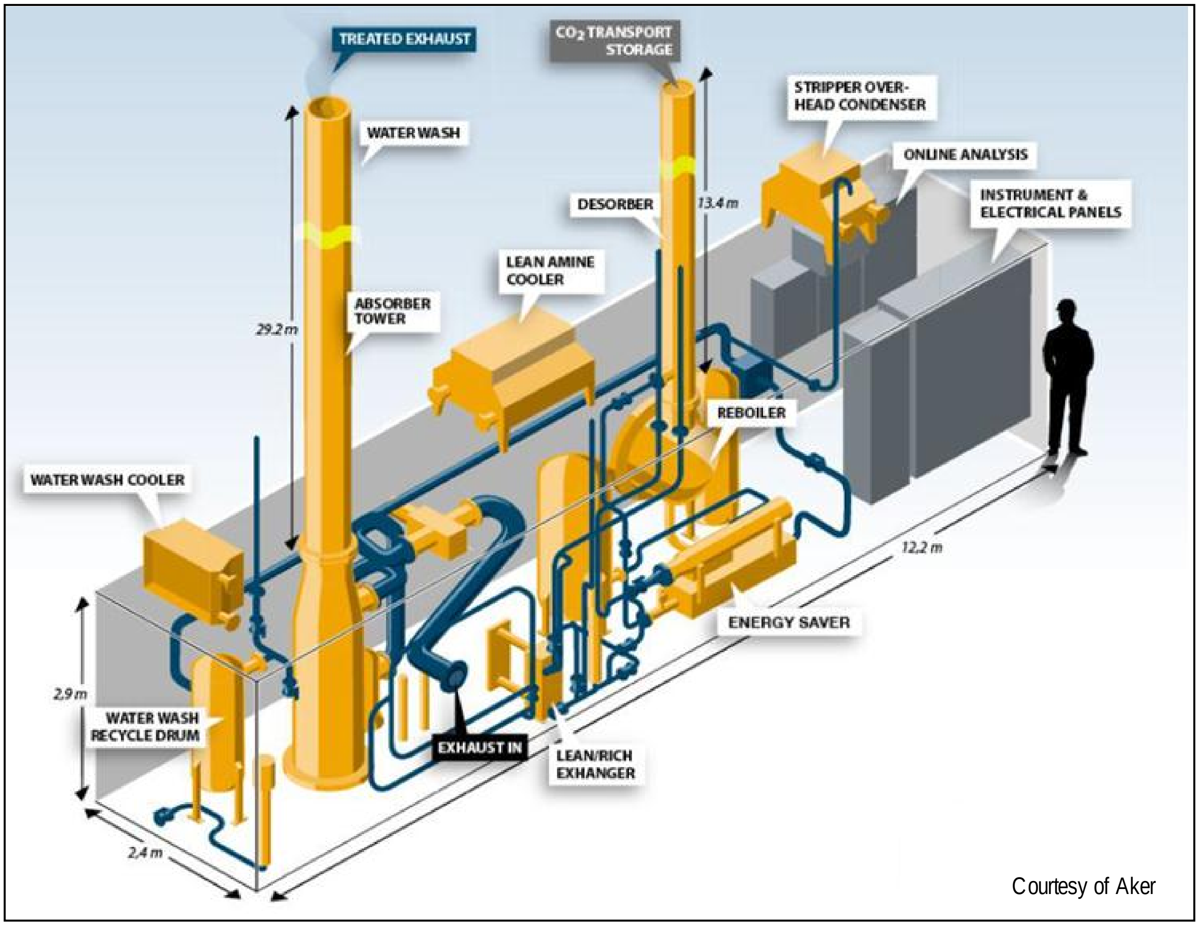
- Low regeneration energy
- Low corrosion rates
- Low degradation rates
- Low environmental impact

An advanced simulation model has been developed, and one of the design objectives is to develop uncomplicated simple, standard design to reduce capital and maintenance costs.

A second test facility funded by SOLVit, the Mobile Test Unit (MTU), was built by ACC to provide reliable operational data from field tests. The MTU is designed for easy transport to different sites allowing for testing with different flue gases. Construction was completed in Verdal, Norway in September, 2008, and it was transported to the gas research center at Risavika, near Stavanger, for a 137-day test program on a simple-cycle gas turbine plant. From the beginning of 2009 until the end of 2010, the MTU was stationed at Scottish Power's Longannet coal-fired power station near Edinburgh, Scotland. It was then moved to the NCCC for extended testing with ACC's CCAmine solvent.

The MTU is designed to process 1,000 actual m^3/hr (equivalent to around 1,100 kg/hr, 35,300 actual ft^3/hr and 2,400 lb/hr), with a CO_2 capture rate of up to 400 lb/hr. It weighs 20 tonnes (22 tons) and is split into four standard-sized containers (one large and three small) for easy handling by ship and truck. Much of the apparatus remains assembled in the larger container and the others include the absorber and regenerator columns, and items such as the blower, direct contact cooler (DCC), reclaimer, and tools.

A diagram of the arrangement is presented in Figure 33. The MTU modules were delivered to the PC4 site at the end of April and set on the prepared foundation for assembly. All installation work was completed by mid-June, and ACC commenced commissioning shortly afterwards. Figure 34 shows the MTU as it was being installed. A motor/generator required to convert standard U.S. power (voltage and frequency) to meet European standards (240 V and 50 Hz) was designed and installed. As the generator delivery was in August, a temporary diesel generator was used initially to enable shakedown and testing.



Courtesy of Aker

Figure 33. Schematic of MTU Equipment.

Figure 34. Installation of the MTU at PC4.

Some observations on the operational experience are as follows:

- Round-the-clock operation was achieved at controlled conditions. This was not possible at Longannet where the plant started up in the morning and shut down at night as determined by its position in the dispatch merit order.
- The extended continuous operation increased understanding of plant and equipment performance and improved the ability to respond to upsets in power plant operation.
- The MTU was designed for northern European operation and was supplied with air-cooled heat exchangers. Summer temperatures in Alabama required the addition of water cooling to some circuits.

During this time, a series of parametric tests were completed, although in the main the data are still being evaluated. A more complete report will be issued following complete data analysis.

- The regeneration energy was determined for different operating conditions.
- A 30 wt% MEA test was completed to compare performance with ACC's solvent. To account for any rig effect, this was carried out in the MTU rather than using the PSTU MEA baseline data.
- Degradation products and solvent carry-over in the absorber and regenerator gas exit streams were measured. As there is no superstructure, access to the sampling points was by bucket truck. See Figure 35.
- Results from ACC's degradation measurement technique were compared with samples taken by the NCCC sampling.
- Emission levels were reduced by adjusting demister operating conditions.
- The reclaimer operated and reduced the concentration of impurities by 80 percent with only minimal loss of solvent.

Figure 35. Manual Gas Sampling from the MTU Absorber Discharge.

3.3.1 TEST PLAN

The main test objectives for the campaign were to evaluate the following:

- Emissions and emission control
- Solvent degradation
- Energy requirements

In addition, the campaign was used to develop and improve understanding of the following:

- Solvent performance
- Reclaiming
- Equipment performance
- Methods for analysis of degradation products
- Plant operation

The total energy demand per unit mass of CO_2 removed and emission are perhaps the most important parameters when assessing the performance of a CO_2 capture process, affecting environment and overall process economics and operating costs. The various test objectives have had different priorities, with the highest priority on emissions, amine degradation, and energy requirement.

3.3.2 MTU OPERATION

Campaign Overview. The CCAmine campaign commenced on June 27, and long-term and parametric tests were performed until November 18, 2011. From November 19 through December 13, 2011, a short MEA campaign was performed to establish a reference solvent. The hours and total CO_2 captured for these two campaigns are provided in Table 9.

Table 9. Summary of ACC Test Campaign Hours and Total CO_2 Captured.

Campaign	Hours with Solvent Circulation	Hours with CO_2 Capture	Total CO_2 Captured, kg
CCAmine	3,092	2,182	228,928
MEA	542	410	40,396

3.3.3 ENVIRONMENTAL

3.3.3.1 ACC EMISSION REDUCTION TECHNOLOGY

An amine based CO_2 absorber is normally equipped with one or more water wash sections above the CO_2 absorption section. The main objectives of the water wash is to absorb amine vapor in order to minimize emission of amine to air and to cool the gas and condense water in order to fulfill the requirement for water balance across the entire capture plant. The condensation of water from the flue gas is a source for wash water make-up. Excess water in the water wash system is bled off and routed to the amine section below, and the effect of these make-up and bleed streams is reduced amine concentration in the water wash liquid.

One assumes close approach to equilibrium in the top of the water wash packed section with liquid recycling, meaning the gas will contain an amine concentration equivalent to the amine vapor pressure from the solution, which again is dictated by the wash liquid temperature, amine concentration, CO_2 loading and pH. Hence, a water wash operating at low temperature with high liquid replacement rate is preferred in order to minimize the amine slip to atmosphere, since lower temperature and lower amine concentration decrease corresponding vapor pressures.

Ammonia and alkyl amines produced by degradation of solvent amines in the process tend to escape the absorber via the emitted flue gas in the same rate as it is produced, since the solubility of these compounds in the solvent and water wash sections is very limited and equilibrium concentration will be quickly obtained.

The MTU contains two water wash sections, where the upper section can be utilized for additional emission control (pH controlled polishing section) in dedicated campaigns. Both sections consist of packed beds with liquid recycling. By controlling the pH in the upper wash section, the vapor pressure of ammonia and amines are eliminated. In addition to the two wash sections, the upper amine section was sometimes used as a third wash section during the MTU campaign in Alabama. In this case, only the lower amine bed of 11.3 meter depth was applied for CO_2 absorption.

ACC performed three emission measurements campaigns on the MTU in Alabama, where off-line gas sampling from the top of the absorber was conducted. The MTU is also equipped with a Gasmet FTIR for on-line monitoring of ammonia and amine emission down to ppm levels.

3.3.3.2 EXPERIMENTAL

Gas sampling was performed from the top section of the absorber, above the demister at the absorber top. A 4-mm inner diameter Teflon probe was applied, the length of the sampling line from the extraction point in the centre of the absorber top to the first impinger bottle was 0.5 m. The sampling was non-isokinetic, and the sampling probe was perpendicular to the gas direction. The sampling train was located on a bracket connected to the top of the tower. A mobile lift was used for access.

The sampling train consisted of three impinger bottles in series. The first two bottles were filled with 20 ml 0.1 M sulfamic acid. The objective was to collect all relevant compounds (amines, nitrosamines, and ammonia) but at the same time avoid artifact formation of nitrosamines. Sulfamic acid is a nitrite scavenger, and previous lab tests have shown that it is less likely to form nitrosamines than is 0.1 N sulfuric acid.

The third bottle was an empty liquid trap, in case of carry over from the upstream bottles. The bottles were weighed before and after sampling for determination of water content in the gas. A gas tight pump and a gas meter was located downstream of the three impingers. The impingers were stored in an ice bath during sampling, and the gas meter measured the actual accumulated gas flow. Each sampling was done over a period of approximately 1 hour in order to collect sufficient amounts of analytes for subsequent liquid analysis.

Solvent (lean amine) and water wash samples were taken at the same time as the gas samples. The collected samples (impinger content, solvent, and water wash samples) were sent to Sintef for analysis. The samples were analyzed for amines including alkyl amines, ammonia, nitrosamines, and nitramines. The main analyses were done by mass spectrometry. By knowing the total impinger content and amount of gas sampled, the gas concentrations of the various elements in the treated flue gas are calculated.

The analyses of ammonia and alkyl amines was originally performed by GC-MS after derivatization with benzene sulfonyl chloride. This method has a very high sensitivity of about 1 ug/L for alkyl amines and 1 mg/L for ammonia. However, it was discovered that sulfamic acid was a poor choice for sampling of ammonia. After the third test campaign, it was found in the literature that sulfamic acid in water solution may hydrolyze to ammonium bisulfate. GC-MS analysis of a blank 0.1M sulfamic acid sample prepared on site showed indeed an ammonia concentration of 14.5 mg/L, which was comparable to the impinger analysis in this work and would correspond to an ammonia emission of a few ppmv. Hence, sulfamic acid is not applicable for sampling of ammonia; reagents like hydrochloric or sulfuric acid should be used.

3.3.3.3 RESULTS

After starting up the plant in June, high amine emission was seen on the FTIR, even with operation of the pH controlled wash that eliminates emission of ammonia and volatile alkyl amines. Similar emission behavior was previously seen on Longannet under certain circumstances, where the amine emission could not be explained by the water wash vapor-liquid equilibrium assumption. This clearly indicated that the emission of solvent amine was mist controlled. Hydrophilic compounds such as solvent amines tend to be accumulated in the mist. Because of the tiny size of the mist particles (around 1 µm), they tend to penetrate wash sections and conventional knitted mesh demisters. For the same reason, isokinetic gas sampling is not required for mist sampling. The FTIR gas analyzer samples and evaporates the mist in the heated sampling line (working at 180°C), and hence measures the total amine content as vapor. It has been discovered that the mist precursors are ultrafine particles entering the absorber. The origin of these particles is not clear, but potential sources are sulfuric acid mist, ammonium sulfate formed in the power plant's selective catalytic reduction process, Trona powder (for SO_3 control), or combustion generated fly ash.

The fine particles are initially acting as nuclides for heterogeneous condensation of water in regions inside the absorber where water-saturated gas is rapidly cooled. When formed, the mist droplets absorb amine from the surrounding gas phase. Earlier emission measurements have proven that, in the absorber system with two proper water wash sections, the mist borne emission is the main contributor to the overall amine emission. In particular, amines that show high reaction rates with CO_2 are more likely to follow the mist out of the absorber, since the amine gets tied up as carbamate in the mist droplets, which reduces the amine vapor pressure and hence the rate of amine desorption from the mist droplets in regions where the partial pressure of amine is lower (e.g., second water wash or pH controlled wash section).

On the other hand, the observed emission of ammonia without the emission control in operation is not related to the mist emission. This is ascribed to the very limited solubility of ammonia and hence very low accumulation in the mist droplets. Any significant ammonia emission will be in vapor form, and the average ammonia emission over time will equal the ammonia production rate.

ACC has developed a mist abatement technology (Anti-Mist), which was incorporated in the MTU and was tested out at the NCCC in Alabama. Figure 36 shows the emission of solvent amine for periods where the MTU was operated with conventional as well as with anti-mist design, clearly demonstrating the benefits of the anti-mist design.

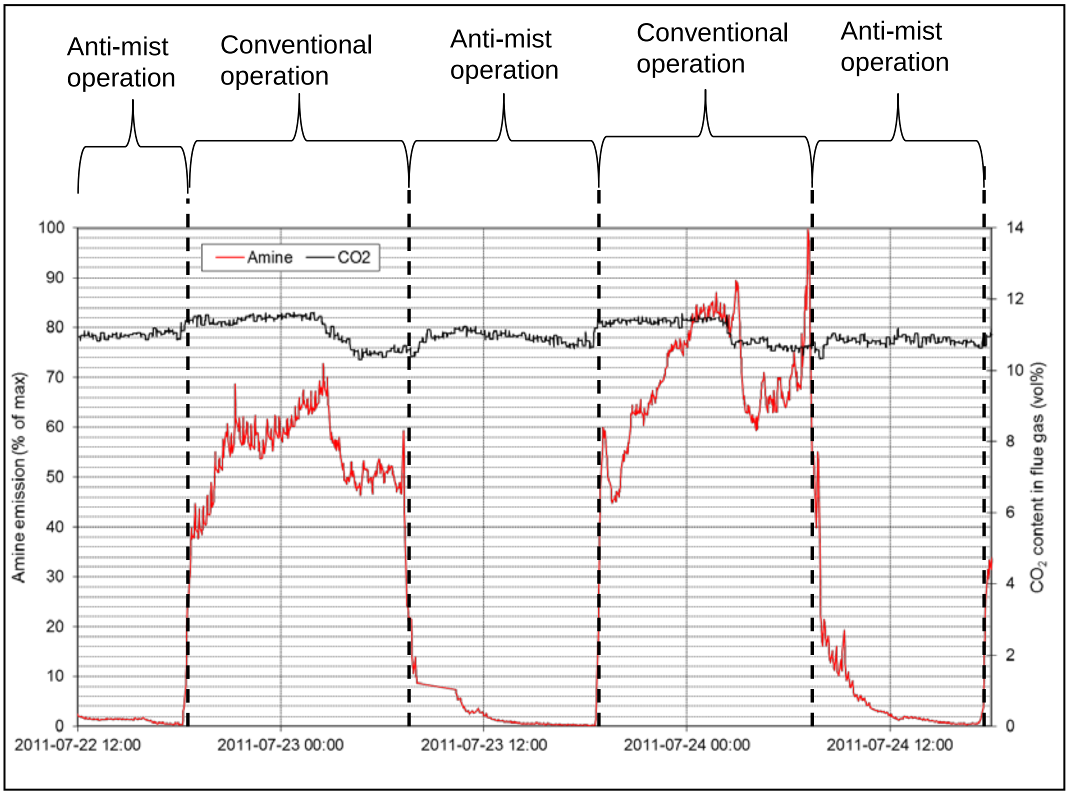


Figure 36. Solvent Emissions with Conventional Operation and with Operation of Anti-Mist Technology.

Due to low concentrations, the FTIR is not able to detect alkyl amines. With the anti-mist system in operation, the solvent amines were also below detection limit of the FTIR. Manual sampling according to the procedure outlined previously were performed. Table 10 and Table 11 show results from the last emission campaign (Emission Test Campaign 3) carried out in November 2011. The emission control system was in service during Tests 4 and 5.

Table 10. Results from Gas Emission Measurement for ACC Test Campaign 3.

Test	Amine 1, ppm dry	Amine 2, ppm dry	Nitros-amines, ppm dry	Dimethyl-amine, ppm dry	Methyl-amine, ppm dry	Ethyl-amine, ppm dry	Di-ethyl-amine, ppm dry
1: Base Case, 45°C Absorber Top	0.06	1.15	0.0028	0.0013	0.002	0.0027	0.0009
2: Base Case, 42°C Absorber Top	0.07	3.10	0.0057	0.0009	0.002	0.0025	0.0009
3: Base case, 50°C Absorber Top	0.11	4.57	0.0050	0.0012	0.004	0.0058	0.0018
4: Acid Wash	0.06	1.60	0.0021	0.0007	0.002	0.0002	0.0001
5: Acid Wash	0.04	1.48	0.0025	0.0006	0.001	0.0002	0.0001
Note: Red font indicates samples below detection limits							

As explained previously, the manual measurements of ammonia failed due to artifact formation of ammonia in the sulfamic acid solution used as impinger absorbent. Reduction in ammonia emission during pH controlled wash tests was, however, seen on the FTIR readings. Table 11 compares average on-line emissions recorded by the FTIR with values from manually taken samples.

Table 11. Comparison of Gas Emission Measurement and FTIR for ACC Test Campaign 3.

Test	Manual Sampling		FTIR		
	Amine 1, ppmv dry	Amine 2, ppmv dry	Amine 1, ppmv wet	Amine 2, ppmv wet	NH3, ppmv wet
1	0.06	1.15	0.3	0.4	3
2	0.07	3.10	0	1.5	2.5
3	0.11	4.57	0	2.7	4.8
4	0.06	1.60	0	0.9	<1
5	0.04	1.48	0	0.6	<1

From the test results above it can be concluded that the pH controlled wash system has been proven effective at capturing gaseous phase alkaline compounds, i.e. ammonia and alkyl amines. The test results also show the low amine emissions achieved with the anti-mist design; however, additional reduction may be achieved through further optimization. The MTU is currently being modified to further improve the anti-mist performance.

The emission control (pH controlled wash) system is operated adiabatically to avoid condensation of water from the flue gas. The emission control removes only contaminants which are already at ppm or ppb levels. The circulating liquid will mainly contain ammonium salt, which is highly soluble at the relevant temperatures. A bleed maintains a steady state salt concentration. It is known that slightly acidic conditions favor nitrosamine formation if secondary amines and nitrite are present. pH controlled wash tests performed at the MTU at the PC4 where an acidic solution containing nitrite scavenger was used for pH control showed no formation of nitrosamine in the liquid wash solution.

Some dedicated samples from Test Campaign 3 were analyzed for Nitramine 1 and Nitramine 2. No nitramines were in the solvent, water wash, wash water from emission control, or in impinger solutions used for flue gas emission sampling. The detection limits in lean solvent were 10 µg/L and 100 µg/L for Nitramine 1 and Nitramine 2, respectively. The detection limits in wash samples and impinger solution were 0.1 µg/L and 1 µg/L for Nitramine 1 and Nitramine 2, respectively. The impinger solution detection limits correspond to gas phase emissions detection limits of 0.04 µg/Nm^3 for Nitramine 1 and 0.4 µg/Nm^3 for Nitramine 2.

3.3.3.4 MEA CAMPAIGN

A 30 wt% MEA campaign was performed from November 11 through December 13, 2011. The purpose of these runs were to collect baseline data for the MTU and to facilitate comparison of ACC's improved amine to a reference solvent.

The anti-mist mode of operation was well demonstrated during the MEA campaign. Figure 37 shows the FTIR readings of MEA concentration in the absorber outlet. From the beginning, the absorber was operated in "conventional mode", causing high MEA emissions. Then on November 28, the operation was changed to partial anti-mist mode. The emission of MEA improved, but was still around 20 ppm. The full anti-mist mode was applied on November 29, and the FTIR reading of MEA dropped to less than 1 ppm. The CO_2 capture was maintained at 90 percent throughout the period, and the plant was running with neutral water balance. The MTU remained in stable anti-mist operation until December 6, when conventional operation was resumed, and then the MEA emissions returned to previous levels.

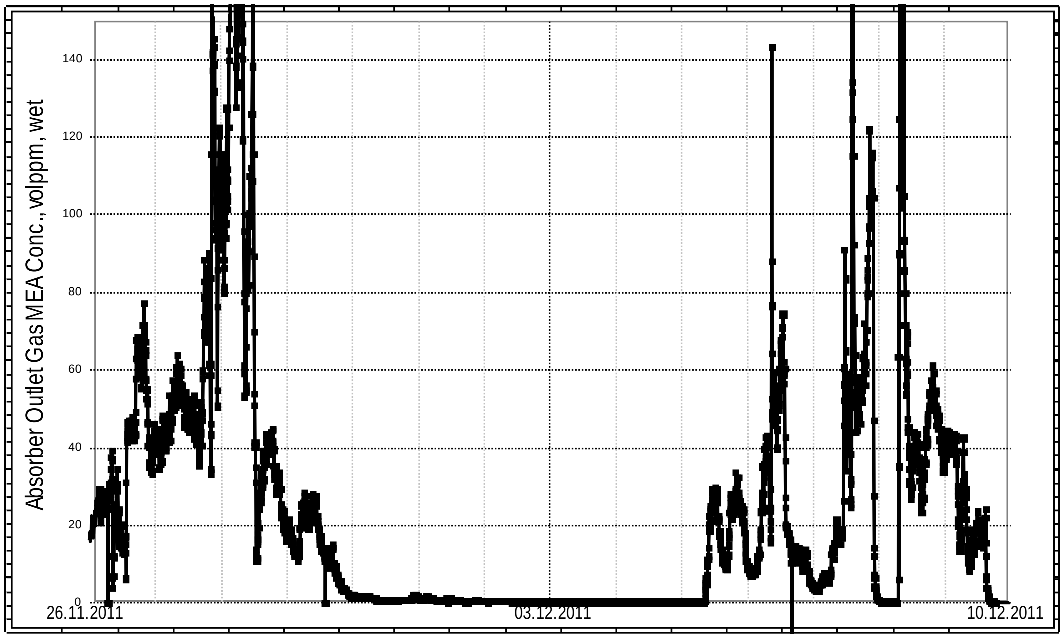


Figure 37. Trends of Emissions during the MEA Campaign.

The generating unit was in stable high load operation for the entire period except for a short upset on November 26, causing low CO_2 concentration in the flue gas. The MTU was down for several hours on December 8 before the campaign was stopped on December 10.

No manual emission sampling was performed during the MEA campaign, but the recorded FTIR data strongly indicates that the MEA emission is mist-borne, and that MEA behaves similarly to the Amine 2 compound in CCAmine. There was no pH control of the emission control section during this campaign.

Overall, the tests have indicated that the combination of anti-mist design and pH controlled polishing can virtually eliminate emissions of alkaline constituents from amine based post combustion capture plants. Both the anti-mist system and the emission control system are patent-pending.

3.3.4 PROCESS PERFORMANCE

3.3.4.1 CO_2 MASS BALANCE AND CO_2 CAPTURE

CO_2 mass balances are calculated based on three methods: a) CO_2 mass flow in CO_2 product stream, b) from CO_2 flow in and out of absorber, and c) from CO_2 lean and rich liquid loadings. Results are plotted in Figure 38. For all calculations of derived parameters based on CO_2 capture (for instance, specific reboiler duty, or SRD) the average is based on CO_2 stream and gas side only. This is due to erratic solvent flow readings on both the rich and lean sides.

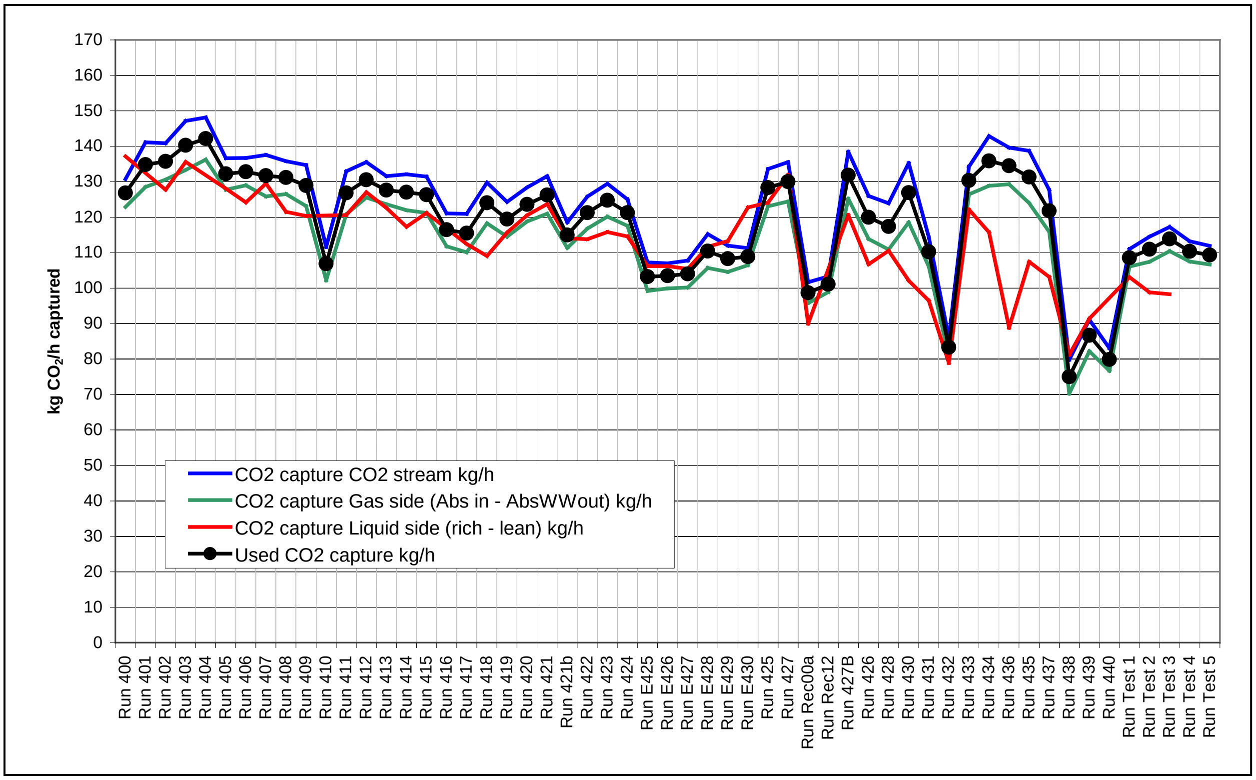


Figure 38. CO_2 Mass Balances for ACC Test Campaigns.

3.3.4.2 REGENERATION ENERGY CONSUMPTION

SRD is calculated from the reboiler duty and CO_2 mass balances. Representative optimum SRD levels were found by optimization of operating conditions for both CCAmine and MEA. SRD for the CCAmine solvent was shown to be approximately 18 to 20 percent lower than MEA. Values were obtained without the use of Energy Saver, both for MEA and CCAmine. The improved performance of CCAmine compared to MEA is due to differences in solvent characteristics. It was found that CCAmine gives lower stripper overhead temperature, which results in less steam loss to overhead. The working capacity of the solvent is also higher in CCAmine compared to MEA, resulting in less sensible heat loss.

Parameters affecting SRD were tested. Solvent total amine concentration and solvent amines blend ratio were factors found to have large effect. By varying total amine concentration by 30 percent and blend ratio by 30 percent, SRD numbers varied by 20 percent. The effect of rich-lean heat exchanger cold side temperature approach is also large; SRD varied by more than 30 percent when comparing approach temperatures below 10 K and above 25 K, explained by large sensible heat loss at high approach temperatures. The effect of DCC outlet temperature on SRD was insignificant in the range of 45 and 49^{o}C. Reboiler temperature is important for optimization of SRD.

3.3.4.3 DEGRADATION AND CORROSION INTENSITY

The heat stable salt (HSS) level was analyzed by ion exchange and titration, and results are shown in Figure 39. The data is for the period from June through November 2011.

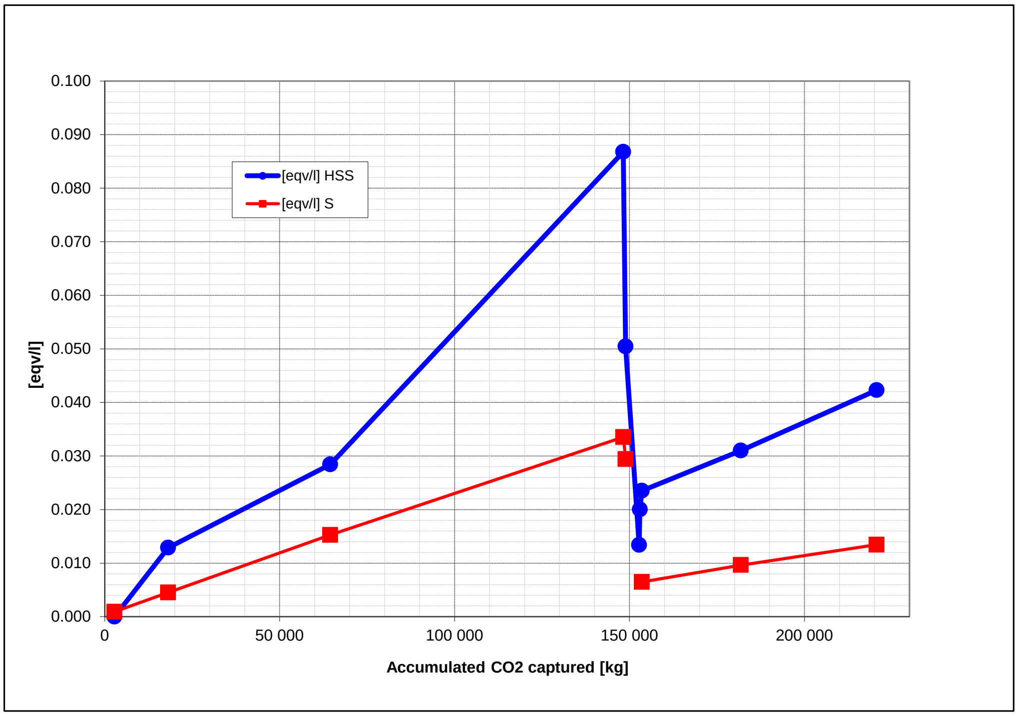


Figure 39. HSS Analysis Data from the CCAmine Campaign.

The drop in HSS concentrations after capture of approximately 150 tons of CO_2 is due to solvent reclaiming, which removed approximately 80 percent of non-volatile compounds.

Metals and sulfur concentrations in the liquid solvent were analyzed by inductively coupled mass spectrometry (ICP-MS), and are plotted against the cumulative amount of captured CO_2 in Figure 40. The data covers the period from June through November 2011. The low metal ion concentrations indicate a low corrosion potential of the CCAmine solvent. Amine loss due to degradation was estimated based on inventory control, emission losses, waste logging, and sample extraction.

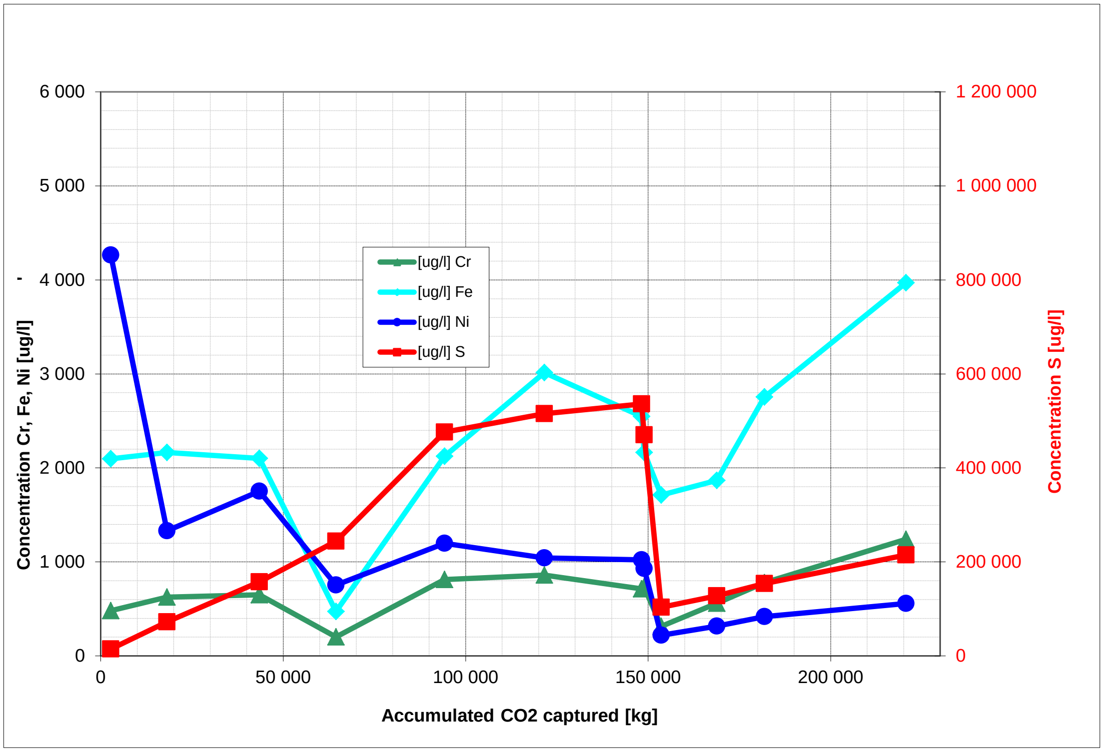


Figure 40. Solvent Analysis Data from the CCAmine Campaign.

3.3.4.4 RECLAIMER CAMPAIGN OVERVIEW

A reclaimer campaign was carried out during week 39, from September 26 through October 1, 2011. The actual reclamation took place over 47 hours, with a total of 1,721 kg amine treated at a constant feed rate of 40 kg/hr while injecting sodium hydroxide for neutralization.

Conclusions from the reclaimer campaign were:

- Approximately 84 percent of the HSS in the lean amine before beginning reclaimer operation was removed during the solvent feed period of the reclaiming process.
- There was no net loss of free amine during the reclaiming period, as the free amine concentration in the lean solvent was the same before and after reclaiming. Also, single amine levels were practically unchanged after reclaiming.

- The viscosity was decreased in the lean solvent by reclaiming, while the viscosity in the reclaimer liquid increased significantly during the reclaiming process.
- There is no significant increase in total heat duty during reclaiming.
- SRD values before and after reclaiming are equal.

3.3.4.5 WORKING CAPACITY

Net CO_2 loading defined as the difference between CO_2 rich and lean solvent loadings given in mol CO_2/L solvent was calculated for the executed runs with CCAmine. Maximum net loading obtained was 2.3 mol CO_2/L solvent, corresponding to a working capacity for the solvent of 10 wt%. The majority of results were, however, in the range of 1.6 to 2.0 mol CO_2/L solvent, corresponding to a working capacity for the solvent between 7 and 9 percent. MEA has a working capacity lower than CCAmine, typically between 5 and 7 wt%.

3.3.4.6 TOTAL SOLVENT REGENERATION ENERGY

No heat integration was included in the design of ACC's MTU pilot tests in Alabama. The only major contribution to heat supply was by means of an electrical heater in the stripper reboiler. Regeneration energy consumption as described previously hence becomes the total solvent regeneration energy.

3.3.4.7 KINETICS

Kinetic characteristics for the solvent were assessed based on obtained CO_2 loading in the CO_2 rich solvent. Maximum CO_2 loading in rich solvent obtained in the MTU CCAmine campaign can be seen from Figure 41, which shows rich flow CO_2 loading versus CO_2 capture for experiments performed at full absorber packing height. It can be seen that CO_2 loading drops when approaching and exceeding 90 percent CO_2 capture.

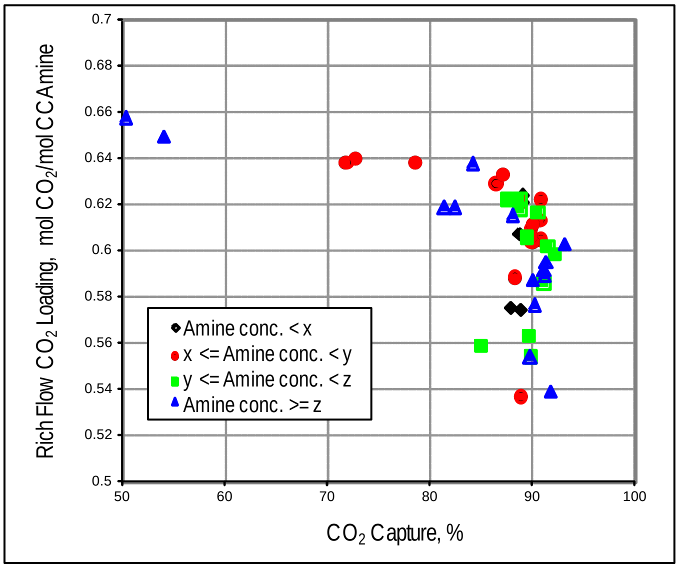


Figure 41. Rich Solvent CO_2 Loading Obtained in MTU CCAmine Campaign.

The effect of changing the absorber feed point was investigated, and comparable runs with lower/upper feed points indicate that rich loading decreases by 12 percent when reducing packing height by 39 percent. SRD was not significantly increased.

3.4 BABCOCK & WILCOX SOLVENT

In September 2011, Babcock & Wilcox Power Generation Group began a three-month test program of its OptiCap^{TM} solvent in the PSTU. This section will provide a summary of preliminary results in quantitative areas such as regeneration energy, solvent degradation, and corrosion, as well as non-quantitative results such as lessons learned and operating experience gained for a post-combustion carbon capture pilot plant in a coal-fired utility power plant. Quantitative results will be provided with comparisons made to benchmark solvents such as MEA.

Prior to the campaign, B&W worked with Southern Company Services (SCS) to create a robust test plan which included (1) corrosion and solvent degradation programs, (2) procedures for solvent sampling and analysis and instrument calibration, (3) a test matrix of fifty-eight different test conditions to gather model validation data, and (4) important solvent safety and handling information.

In addition, a technical assessment was performed by B&W, and a document describing the appropriate procedures for a wide range of risk scenarios was created for the operators and engineers at the PSTU. During the three-month campaign, a number of events occurred which required implementation of these contingency plans, and following the recommended course of action resulted in an expedient return to normal and safe operation.

3.4.1 B&W'S INITIAL DEVELOPMENTAL WORK

Bench-Scale Unit. The bench-scale, Regenerable Solvent Absorption Technology (RSAT^{TM}) simulator is a fully functional process test facility. The unit contains most of the equipment which would be included in a large-scale facility, including an absorber column, a regenerator column, and an electrically heated reboiler. The bench-scale unit is designed to capture approximately one kilogram of CO_2 per hour. The columns are of modular design, and the process can be operated in a variety of modes, which provide excellent flexibility for process analysis and development work. The unit provides an initial indication of the performance of a new solvent in an integrated system. This fully integrated bench-scale process also facilitates parametric studies of independent process variables and provides data for validating computer simulation models.

RSAT Pilot Plant. Following laboratory and bench-scale evaluations, the most promising solvents are tested at large scale in B&W's RSAT pilot plant. Relative to the data provided by the CO_2 control lab, the RSAT pilot plant provides high quality, quantitative data which is representative of full-scale systems. Different process flow schemes which can affect CO_2 absorption rates and regeneration energy for a given solvent are tested and evaluated in the pilot unit, with a focus on minimizing the overall energy consumption of the CO_2 capture process. The RSAT pilot plant can process approximately 3,100 lb/hr of flue gas and capture approximately 7 tons/day of CO_2.

The pilot plant can also be operated in recirculation mode, wherein the captured CO_2 is mixed with nitrogen and other gases to simulate actual flue gas from a coal-fired power plant before being recycled to the inlet of the absorber.

The most promising solvents identified in the laboratory by bench-scale testing and computer simulation modeling are then run through a series of test campaigns in the RSAT pilot plant. As a result of its extensive research test program, B&W selected the most promising solvent candidate for more in-depth development and testing. The result was the OptiCap™ solvent, which has been tested extensively at lab- and pilot-scale.

Pilot testing of the OptiCap solvent has shown favorable performance characteristics. Under similar test conditions, a lower reboiler heat duty was attained for the OptiCap solvent, as compared to 30 wt% MEA solvent. The minimum reboiler heat duty attained was comparable to the heat duties claimed by other solvent and process providers of 1,200 to 1,275 BTU/lb CO_2 (122 – 130 kJ/mol CO_2). Additional properties of the OptiCap solvent which are expected to provide additional savings in capital and operating costs (i.e., resistance to degradation, high mass transfer rate, high CO_2 carrying capacity, etc.), have been verified in these campaigns, and will be further quantified in future test campaigns.

The results described above are considered to be only an early indication of the potential of the OptiCap solvent. B&W has simulated several process design cases which could further reduce the energy penalty of solvents by using heat integration with the power plant. Some of these design cases are specifically related to the unique properties of the OptiCap solvent (for example, the ability to regenerate at higher temperatures and pressures). Computer models have indicated that these process improvements are feasible, and projected energy requirements in the range of 1,000 to 1,100 Btu/lb CO_2 are possible for the OptiCap solvent.

Below are some of the characteristics of the OptiCap solvent which are expected to be confirmed and further quantified as a result of testing at the PC4:

- Resistance to Oxidative Degradation—Most solvents degrade in the presence of high concentrations of oxygen, which can occur in coal combustion flue gas. Preliminary testing of the OptiCap solvent indicates a relatively high level of resistance to this phenomenon, which offers the potential for lower solvent make-up rates as well as lower solid waste generation rates.
- Resistance to Thermal Degradation—Testing thus far has shown the OptiCap solvent to be stable at operating temperatures up to 300^{o}F. This attribute offers the potential for regeneration at higher operating temperatures and pressures, which could lead to significant energy savings in terms of CO_2 compression.
- Ease of Reclaiming—Results thus far indicate that thermal reclaiming is likely the primary technology for removing degradation species formed using the OptiCap solvent. Thermal reclaiming is a well-known technology which has been used successfully for decades for solvent regeneration. Other potential technologies for solvent regeneration include systems such as carbon beds and ion exchange systems.

- Lower Volatility—Compared to 30 wt% MEA, the OptiCap solvent shows decreased volatility. Lower volatility reduces solvent losses to the exhaust stack, and decreases energy requirements for heat exchanger cooling in the solvent wash section of the absorber.
- Increased Mass Transfer Rate—The rate of absorption of CO_2 for the OptiCap solvent is approximately twice that of 30 wt% MEA. This kinetic advantage allows the absorber towers to be designed with less packing than towers designed for 30 wt% MEA. This characteristic offers capital cost savings with reduced absorber tower height, quantity of packing, structural steel, foundations and installation cost. Also, reduced tower height results in auxiliary power consumption savings, due to decreased pressure drop through the absorber and decreased pump power required for solvent recirculation due to decreased head pressure. Approximately 75 percent of the electrical power required to operate the RSAT system is consumed by the fan or blower to move the flue gas through the flue gas cooler and absorber, so cost savings generated by decreased pressure drop through the absorber towers can be substantial.
- Increased CO_2 Carrying Capacity—Because the OptiCap solvent can be loaded with approximately twice the amount of CO_2 per unit of solvent, the solvent recirculation rate is decreased, saving not only the energy required to pump the solvent within the system, but also the energy required to heat and cool the solvent in the various process stages.

Testing under actual power plant flue gas conditions (for example, at NCCC) is required in order to confirm the research performed in both the B&W lab and pilot plant regarding the characteristics of the OptiCap solvent. In addition, phenomena such as solvent degradation, system corrosion, and waste stream formation must be studied over time periods which exceed the duration of most lab- or bench-scale test campaigns. Thus, long-term testing is critical to gaining a complete understanding of both the technical and financial risks associated with any PCC capture technology.

In addition, the ability to accurately predict cost and operating performance is essential, not only for process optimization, but also to quantify the risks and understand the remedies associated with offering performance guarantees. The ability to offer process performance guarantees will play an important role in commercial plant deployment. Thus, field testing on coal flue gas at a facility such as the PSTU at NCCC is a critical step on the path to completing the required analysis.

3.4.2 TEST OBJECTIVES AND PRE-CAMPAIGN PLANNING

Along with confirming and quantifying the solvent benefits described above, a host of other objectives existed prior to the start of the test campaign. These objectives can be summarized as follows:

- To gather data for process simulation model validation
- To characterize effluent streams, including stack emissions as well as liquid and solid wastes
- To gather data on the oxidative, thermal, and chemical aspects of solvent degradation and reclamation

- To perform corrosion studies in order to determine appropriate materials of construction for a commercial plant.

In order to accomplish the test objectives indicated above, as well as to provide a written guide to the PC4 operators and engineers, a comprehensive test plan was created. This plan included complete corrosion and solvent degradation programs; checklists and procedures for solvent sampling and analysis as well as instrument calibration; a test matrix of fifty-eight different test conditions for purposes of gathering model validation data; and important solvent safety and handling information.

In addition to the test plan, several other initiatives were completed throughout 2011 in preparation for the test campaign. For example, in February, B&W personnel traveled to NCCC to meet with engineers and operators to discuss solvent handling and preparation (a process that ultimately led to modifications being made to the PSTU). In April, B&W personnel traveled to NCCC to witness and provide feedback on the MEA baseline tests which were being performed at that time by NCCC personnel. In August, B&W personnel provided installation supervision and operator training for the corrosion probes and coupons which were installed as part of the corrosion testing portion of the test campaign. In addition, B&W participated in several other conference calls, meetings, and discussions regarding hazard operation planning, equipment commissioning and other related topics.

In March, B&W decided to perform a detailed evaluation of the technical risks involved with testing the OptiCap solvent in a coal-fired utility power plant. A series of brainstorming sessions were held, and several tools typically used in Lean Six Sigma methodology were used in order to create a matrix containing a list of various failure modes and effects, ranked according to composite scores which were calculated based on probability and impact. This matrix was then used to create a list of corrective actions which could be used to significantly decrease the technical risk for the project.

The operating scenarios with the highest composite scores from this analysis included:

- Extended planned shutdown
- Contaminants
- Low flue gas temperature
- Piping leaks and other equipment failures
- Emergency evacuation due to an anhydrous ammonia alarm or inclement weather
- Black plant
- Unplanned shutdown

The list of corrective actions which was generated as a result of this effort included several different categories of actions, including areas for further research, additional operating procedures, and potential equipment modifications. Accordingly, the start of the test campaign was postponed for approximately three months while further research was performed and procedures written. In addition, collaboration of B&W and NCCC personnel resulted in modifications made to the PSTU both to address items arising from the risk assessment as well as items which were requested in order to increase the amount and quality of data which could be obtained from the pilot test campaign.

3.4.3 MODIFICATIONS TO THE PSTU

Several modifications were made to the PSTU in preparation for testing the OptiCap solvent. These modifications are summarized below.

One of the most prominent scenarios in the risk assessment exercise was a black plant scenario with the OptiCap solvent. Although the probability of a black plant was very low, the magnitude of the impact could be significant. During a black plant scenario, all steam, flue gas, and compressed air are lost. With the original PSTU design, this would leave the OptiCap solvent distributed throughout the entire process (vessels and piping). If precipitation occurred due to cooling, the entire process could require cleaning. The existing process did not include a means to address this condition. As a result of this analysis, piping with manual drain valves was added to the piping upstream of the pumps following the absorber, regenerator, and flash tank sumps, which terminated at a solvent storage tank. This allowed for the sumps of these vessels to drain to a single location via gravity, as the vessel sumps were elevated, and the solvent tank was located at grade.

Experience from the pilot testing of the OptiCap solvent and lab work indicated that precipitation could be dissolved by adding heat. At B&W's recommendation, a portable hot water heater and water nozzle connections at each pump were added. This allowed for the injection of hot water directly to any area where precipitation might occur.

The only PSTU piping which could not be drained via the recommendations was the piping directly surrounding the cross-flow heat exchanger. B&W requested that drains be added to the heat exchanger for emergency situations.

Along with controlling temperature, another mechanism that is available to prevent precipitation is to control the CO_2 loading of the solvent. If the bulk CO_2 loading is on the lean side, which was always true in the PSTU because the bulk of the lean-solvent inventory is stored in a tank, then CO_2 can be injected to increase the bulk solvent loading. To add CO_2 when required, B&W recommended installing a Dewar vessel with an induction nozzle to promote mixing.

In addition, B&W also requested that some instrumentation and nozzles be added to the system in order to facilitate the gathering of information of data. For example, local gauges for temperature indication were added to the cooling water outlets of several heat exchangers. Also, nozzles were added to several vessels for installation of electrical resistance probes and weight loss coupons so that corrosion information could be gathered throughout the campaign.

3.4.4 OPTICAP SOLVENT TESTING

On September 15, 2011, B&W began the test campaign for the OptiCap solvent. After start-up, 24-hour, 7 day per week operation began, with B&W personnel present at the PSTU from approximately 6:00 AM until 1:00 AM each day.

By mid-October, many test runs on B&W's original test matrix had been completed. However, a number of events had also occurred such that the PSTU had been shut down several times, and some operating time had been lost, to the extent that making the original schedule seemed unlikely. In addition, typical fluctuations in boiler load for Plant Gaston Unit 5 had occurred such that steady state operation could not be sustained for the minimum time required per the test specifications. In late October, B&W increased coverage at the PSTU such that round-the-clock operation would be possible, in part to accelerate the test schedule, and in part to take advantage of the fact that Unit 5 boiler load was typically more steady during the overnight hours. Within approximately two weeks, significant progress had been made in accelerating the test schedule, and B&W coverage once again returned to the original two-shift paradigm.

The original matrix of approximately 58t test conditions was completed by November 9, 2011. A few test conditions were repeated in an effort to increase data integrity for a few of the test runs which had previously been run. Testing of the thermal reclaimer was performed during the week of November 14. The PSTU was then prepared for temporary shutdown for the Thanksgiving holiday, with B&W providing assistance to the plant operators. The plant continued to operate in a so-called "warm recycle" mode during the holiday weekend. The PSTU was re-started on November 29, with B&W personnel again providing assistance to the plant operators. No issues were encountered during the re-starting of the system. After the system was brought to the required operating conditions, B&W engineers proceeded to test under selected operating conditions where parameters such as regeneration energy had been measured at their lowest values during the campaign. During the following week, additional tests were run in these areas of interest. All testing was completed on December 7, 2011.

During the test campaign, a total of approximately 115 test conditions were run. Information regarding the variation of certain operating parameters is listed in Table 12. Throughout the campaign, mass balances were closed to within 5 percent or less (often much lower), and a significant quantity of data was gathered to compare with B&W's process simulation models.

Table 12. Range of Operating Parameters for OptiCap Solvent Test Campaign.

Parameter	Range
Solvent Recirculation Rate, lb/hr	Varying
Lean Solvent Temperature, °F	Varying
Wash Temperature, °F	As Required
CO_2 Removal Efficiency, %	80 to 95
Flue Gas Flow Rate, lb/hr	3,500 to 5,000
Flue Gas Temperature, °F	104 to 130
Intercooler Usage	On / Off
Intercooler Temperature, °F	Varying
Regenerator Pressure, psig	Varying
Reflux Route	Regenerator / Absorber
Reboiler Steam Flow Rate, lb/hr	As Required
Thermal Reclaimer Usage	On / Off

In addition to the operating data gathered for process model validation and verification, the test campaign also yielded a great deal of data in other areas of interest, such as corrosion, solvent degradation, and effluent stream characterization. In addition to automatic samples which were continuously withdrawn from the system for purposes of auto-titration to check solvent concentration and CO_2 loading, manual samples were withdrawn at regular intervals both to serve as a check to the automatic samples as well as to further characterize the chemical composition of various process streams.

In addition to the previously described liquid samples, technicians from SRI worked with PSTU staff and B&W personnel in order to perform gas-phase sampling at various locations in the system. Sampling locations are shown in Figure 42. The purpose of these samples was to analyze for airborne compounds such as the OptiCap solvent, ammonia, aldehydes and nitrosamines which could be present in either the gaseous state, or possibly entrained in the liquid phase. Chemical analysis of these samples, and interpretation of the results, is pending as of the publication of this report.

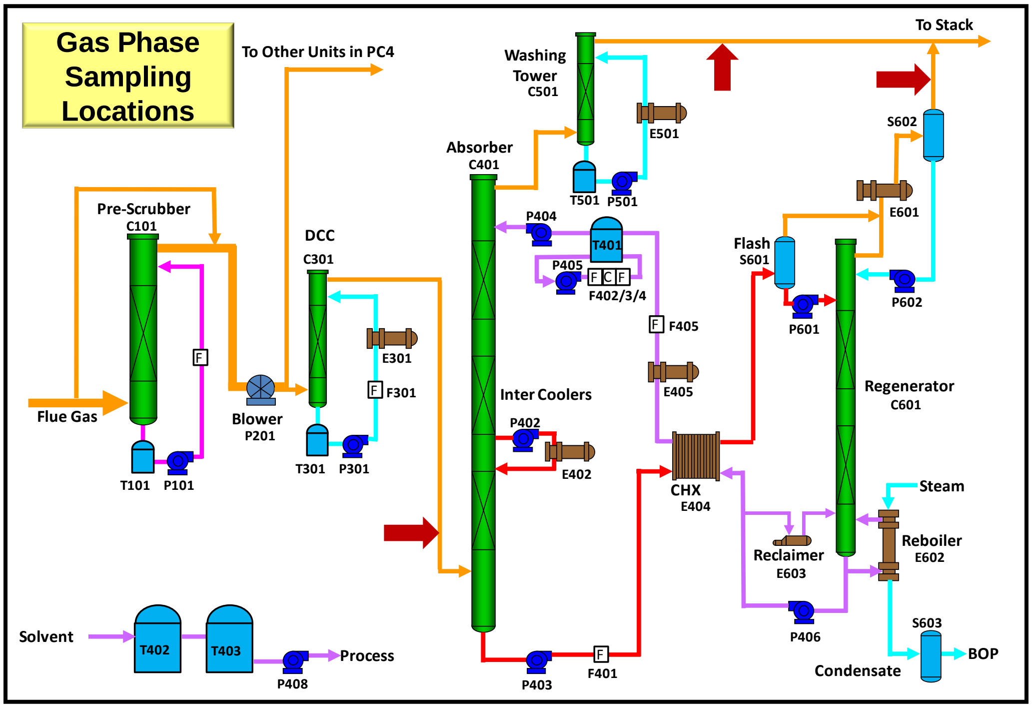


Figure 42. PSTU Gas-Phase Sampling Locations.

In addition to the quantitative data described previously, a significant amount of non-quantitative information was also generated during the campaign. For example, many of the scenarios from the previously described risk assessment did in fact occur during the course of the test campaign. Flue gas supply was lost on more than one occasion, unplanned shutdowns did occur (both with prior notice and without), and other events transpired which are typical of the normal operation of a coal-fired power plant. In addition, boiler load cycling continuously affected the operation of the PSTU, sometimes making steady state operation a challenge.

However, throughout the range of scenarios which occurred, no major interruptions in testing were encountered. In fact, the procedures which were provided by B&W prior to the start of the campaign, when applied, often resulted in relatively smooth start-up and shutdown of the PSTU, even in circumstances where little to no advance notice was available. Partially by design and partially due to unplanned events, B&W was afforded the opportunity to explore the so-called “solubility window” of the OptiCap solvent, on both the rich and lean sides of the operating curves. The target CO_2 removal (which was typically 90 percent), was routinely achieved with relative ease. Finally, no solvent precipitation occurred in any of the solvent circulation piping in the PSTU during the campaign.

3.4.5 PRELIMINARY RESULTS

As described previously, B&W requested that additional nozzles be installed in various vessels and piping throughout the PSTU before the start of the OptiCap test campaign so that critical corrosion data could be gathered. A total of 19 nozzles were installed at 12 different locations. Retractable weight-loss coupon holders were placed at each location, with two C1010 carbon steel and two 316L stainless steel coupons attached to each holder. In addition, electrical resistance probes were also installed at seven of these locations. Six of the probe elements were constructed of C1010 carbon steel, and one was constructed of 316L stainless steel.

At the end of the campaign, the coupons were removed from the system and sent to the B&W research center for inspection and analysis. The probes and coupon holders were left in place at NCCC’s request so that they could be used for future test campaigns. Solvent samples were withdrawn from the system at weekly intervals so that chemical analysis could be performed specifically for corrosion species. Analysis of the weight loss coupons and corrosion samples is pending at this time.

Overall results to date, which are primarily based on visual observations and on-line measurements from the electrical resistance probes, indicate that corrosion rates for the OptiCap solvent are relatively low, as compared to other benchmark solvents such as MEA. The most significant corrosion has generally occurred in areas of the plant where operating conditions are considered to be the least favorable, as expected. Many of the on-line corrosion rate measurements to date are less than 1 mil per year (mpy). However, these preliminary results must be confirmed via weight loss coupon analysis before definitive recommendations regarding suggested materials of construction can be made.

Figure 43 shows electrical resistance probes which were temporarily removed from two different locations in the plant during routine inspections. The probe from the regenerator appears to have more significant corrosion than the probe from the absorber. This is expected due to the presence of CO_2 in the regenerator, coupled with high operating temperatures and pressures relative to the absorber.

Resistance Probe from Absorber

Resistance Probe from Regenerator

Figure 43. Electrical Resistance Probes Inspected during OptiCap Solvent Testing.

Figure 44 shows coupons removed from the system during a routine inspection. Note that the two carbon steel coupons on the left exhibit more corrosion than the two stainless steel coupons on the right.

Figure 44. Weight Loss Coupons on Retractable Holder Inspected during OptiCap Solvent Testing.

Another significant area of interest for solvents used for carbon capture is degradation, which can occur through a variety of different mechanisms, including thermal degradation from prolonged exposure to high temperature, oxidative degradation from reaction with oxygen in the flue gas, and other types of degradation such as reaction with acid gases and other flue gas contaminants to form heat stable salts. Solvent samples were withdrawn from the system at regular intervals so that chemical analysis could be performed specifically for solvent degradation species. Analysis of these samples is pending at this time.

Based primarily on visual observations, the chemical composition of the solvent appears to have changed, as indicated by a change in color as shown in Figure 45. Although changes in solvent color are not necessarily indicative of the presence of heat stable salts or other degradation species, changes in solvent composition due to degradation are often accompanied by color changes similar to those observed for the OptiCap solvent during the subject campaign.

Chemical analysis will confirm and quantify the extent of degradation species present in the solvent as a function of time.

Figure 45. Solvent Color Changes during the OptiCap Solvent Testing.

A third significant area of interest for carbon capture solvents is regeneration energy. As described previously, measurements performed by B&W and others at lab- and pilot-scale facilities indicate that significant savings in regeneration energy can be achieved using the OptiCap solvent, as compared to benchmark solvents such as MEA. Overall, the results achieved tend to support this claim.

During benchmark MEA testing at NCCC, the lowest measured value for 30 wt% aqueous MEA was 1,507 BTU/lb CO_2, which was achieved at 86 percent CO_2 removal. During the same testing, the lowest measured value for 40 wt% aqueous MEA was 1,245 BTU/lb CO_2, which was achieved at 82 percent CO_2 removal. The lowest value achieved at 90 percent CO_2 removal (which was the typical value during OptiCap testing) was 1,300 BTU/lb CO_2, which is generally consistent with published values.

During the OptiCap test campaign, values in the range of 1,100 to 1,125 BTU/lb CO_2 were achieved on multiple occasions, at a CO_2 removal efficiency of 90 percent. These results are consistent with lab- and pilot-scale measurements, as well as B&W process simulations, which indicate that further energy savings are possible when using a cross-flow heat exchanger optimized for the OptiCap solvent (which was not the case with the equipment in the PSTU). Table 13 summarizes these results.

Table 13. Comparison of Performance of OptiCap and MEA Solvents.

Solvent	CO_2 Removal, %	Energy Consumption, BTU/lb CO_2
30 wt% MEA	86	1,507
40 wt% MEA	90	1,300
OptiCap	90	1,110

3.4.6 SUMMARY AND CONCLUSIONS

In September 2011, B&W began a three-month test program of its OptiCap solvent in the PSTU at the NCCC. The goals of the test campaign were (1) to gather data for process simulation model validation, (2) to characterize effluent streams (including stack emissions as well as liquid and solid wastes), (3) to gather data on solvent degradation and reclamation, and (4) to perform corrosion studies in order to determine appropriate materials of construction for a commercial plant.

Prior to the campaign, B&W worked with SCS to create a robust test plan which included (1) corrosion and solvent degradation programs, (2) procedures for solvent sampling and analysis and instrument calibration, (3) a test matrix of over 58 different test conditions for purposes of gathering model validation data, and (4) important solvent safety and handling information.

A technical assessment was performed by B&W, and a document describing the appropriate procedures for a range of risk scenarios was created for the operators and engineers at the PSTU. During the campaign, a number of events occurred which required implementation of these contingency plans, and following the recommended course of action resulted in an expedient return to normal and safe operation.

During the test campaign, a total of approximately 115 test conditions were run. A significant quantity of data was gathered to compare with B&W's process simulation models. In addition to the operating data gathered for process model validation and verification, the test campaign also yielded a great deal of data in other areas of interest, such as corrosion, solvent degradation, and effluent stream characterization.

Liquid samples were routinely withdrawn using the automated system for the purpose of checking solvent concentration and CO_2 loading. In addition, manual samples were withdrawn at regular intervals to serve as a check to the automatic samples and to further characterize the chemical composition of various process streams. The samples have been sent for chemical analysis to identify and quantify corrosion and solvent degradation species. Also, NCCC staff worked with B&W personnel to perform gas-phase sampling at various locations in the system. The purpose of these samples was to analyze for airborne compounds such as the OptiCap solvent, ammonia, aldehydes, and nitrosamines which could be present in either the gaseous state or possibly entrained in the liquid phase. Analysis of samples is pending at this time.

Overall results indicate that corrosion rates for the OptiCap solvent are relatively low, as compared to other benchmark solvents such as MEA. The most significant corrosion has generally occurred in areas of the plant where expected. Many of the on-line corrosion rate measurements to date are less than 1 mpy. These preliminary results must be confirmed via weight loss coupon analysis before definitive recommendations regarding suggested materials of construction can be made.

The chemical composition of the solvent appears to have changed, as indicated by a change in color. Although changes in solvent color are not necessarily indicative of the presence of degradation species, changes in solvent composition due to degradation are often accompanied by color changes similar to those observed for the OptiCap solvent during the subject campaign. Chemical analysis will confirm and quantify the extent of degradation species present in the solvent as a function of time.

During the OptiCap test campaign, values in the range of 1,100 to 1,125 BTU/lb CO_2 were achieved at a CO_2 removal efficiency of 90 percent. These results are consistent with both lab- and pilot-scale measurements as well as process simulations, which indicate that further energy savings are possible when using a cross-flow heat exchanger optimized for the OptiCap solvent.

A more detailed report will be published at a later date, when all samples taken during the campaign have been analyzed and data reduction is complete.

3.5 MEMBRANE TECHNOLOGY & RESEARCH POST-COMBUSTION MEMBRANE

Since 2007, MTR has explored membrane technology for CO_2 capture for power generation in both pre- and post-combustion areas. A new polymeric membrane, Polaris™, has been developed which permeates CO_2 approximately ten times faster than a commercial cellulose acetate membrane. In addition, MTR has also developed and patented a novel process using incoming combustion air as sweep gas to generate the driving force for CO_2 permeation across the second stage membranes. This results in reduction of energy penalty for CO_2 separation.

Previously, MTR has demonstrated a 1 ton/day (TPD) or the equivalent of a 50 kW membrane system, under DOE funding, using flue gas from Arizona Public Service's Cholla coal-fired power plant. In 2010, MTR received a $15 million award from DOE (total project cost $18.75 million) to scale up the CO_2 membrane from 1 to 20 TPD or the equivalent of 1 MW and demonstrate its performances at a coal fired power plant. It is a five-year project from 2010 to 2015. In addition to the membrane performance evaluation, a study will be carried out to understand the impact of CO_2 recycle on boiler performance and plant integration (with B&W) and flue gas water recovery and management (with EPRI). The Cholla site was originally chosen to be the host site for the 1-MW system demonstration. However, Arizona Public Service withdrew from the project, and NCCC was eventually selected as the host site.

Based on lessons learned at the Cholla plant, MTR modified the 1-TPD skid, and it was installed at the PC4 by mid-October 2011. The skid consists of a flue gas compressor, sweep air compressor, chiller, vacuum pump, flue gas pretreatment skid, and a membrane skid. Figure 46 shows part of the system installed at the PC4, as well as the membrane modules. Flue gas is cooled with chilled water and its moisture removed before it flows to the flue gas compressor, which compresses the flue gas to about 30 psig upstream of the membrane skid. The first stage membrane extracts most of the CO_2 by creating a vacuum on the permeate side (low pressure side). The second stage membrane further extracts CO_2 from residual flue gas exiting the first stage membrane to achieve a higher CO_2 capture rate using sweep air to sweep CO_2 from the permeate side.

Chiller (Blue Cabinet) and Sweep Air Compressor (Yellow Cabinet)

CO_2 Membrane Modules

Figure 46. MTR 1-TPD CO_2 Membrane Components.

A design hazard review of the MTR unit resulted in additions and modifications to the unit, including the addition of pressure relief valves to some of the skids, adding temperature measurement at the MTR flue gas compressor discharge, personnel protection on compressor discharge piping, and modifications to the vacuum pump level switch. Engineers from MTR and NCCC then worked together to perform commissioning of electrical and controls, leak testing, and shakedown operation.

During shakedown of the 1-TPD system, the following issues surfaced:

- Pressure and temperature set points on the chiller were not appropriate for NCCC operating conditions and required manufacturer adjustments.
- The vent line on the feed compressor needed to be connected to the flue gas return line to the stack to prevent leakage of flue gas into the atmosphere during system operation.
- The vacuum pump discharge line was under a vacuum of 18 inches H_2O. The unexpectedly high vacuum caused the water level in the sealing water tank to rise significantly, resulting in constant loss of sealing water and pump shutdown.

The issues were mainly due to the fact that the system was originally designed and built for testing at the Cholla power plant. The different operating conditions at NCCC required adjustment of the engineering design. With the site service provided by the equipment vendors, MTR and NCCC engineers resolved all the system issues and completed the system shakedown activities.

After insulation was installed on the membrane skid by NCCC personnel, the system was started up in mid-November and ran smoothly for two days before the final chiller adjustment for long-term operation. The system was then re-started on November 30 and operated until an outage occurred at the PC4 site on December 1, and the system was shut down and remained off-line during the rest of the month due to the holiday season. The system was re-started in mid-January and ran for two days before flue gas was shut down. During this run time, the system exhibited similar performance to that obtained during operation in early December.

Figure 47 is a flow diagram of the 1-TPD system with performance results obtained during initial operation. The system has two separation steps: the first step uses cross-flow modules to remove approximately 60 percent of the CO_2 from the flue gas, and enrich the CO_2 by a factor of six in the permeate stream; the second step uses countercurrent/sweep modules to remove about half of the remaining CO_2 in the residue stream from the first step. Overall, the system removed 78 percent (0.89 metric ton/day) of the CO_2 from post-FGD flue gas that contained 1.15 metric tons/day CO_2. The CO_2 content of the flue gas was reduced from 12.1 percent in the feed to 3.3 percent in the treated gas. Process simulations confirmed that the overall CO_2 removal rate and high CO_2 enrichment factor were consistent with expected performance. MTR plans to slightly reduce the flue gas flow rate in the future test to the design value (1 metric ton/day), to verify that the membrane process can reach the target CO_2 capture rate of 90 percent.

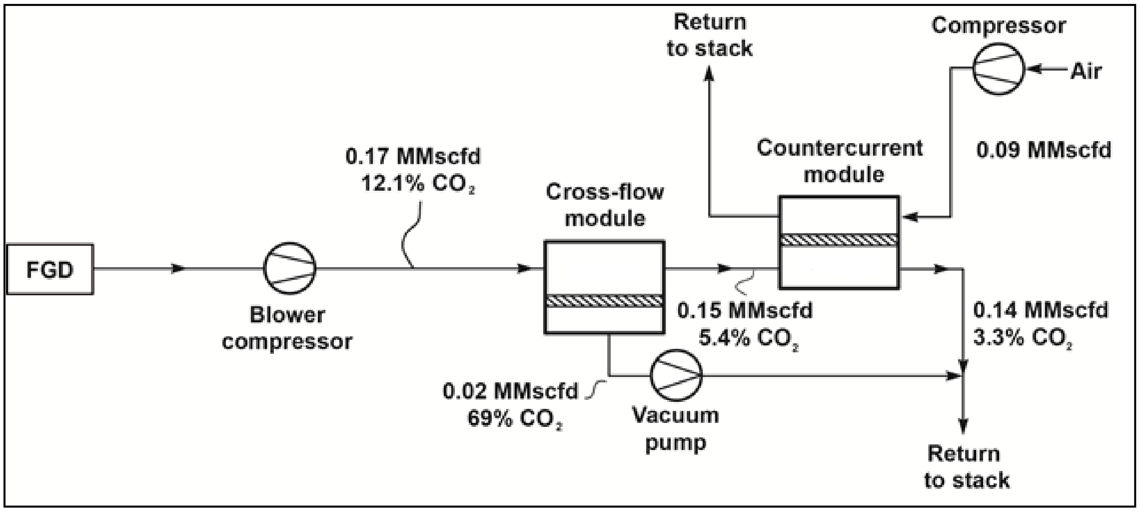


Figure 47. Flow Diagram of the MTR 1-TPD System with Initial Performance Results.

Future work will be focused on operating the system to quantify membrane lifetime and to serve as a test vehicle for membrane and module improvements developed in the lab. The lessons learned on the 1-TPD skid will also be useful for the design and future operation of the 20-TPD system, which is scheduled for installation at the NCCC in 2013.

4.0 OXY-COMBUSTION

For the past several years, a team of researchers at the NCCC, the Engineering & Economic Evaluations group, has been investigating the feasibility of coal-fired power production using the Transport Reactor as a pressurized, oxygen-blown fluid bed combustor. The technology is known as Transport Oxy-Combustion, or TROC^{TM}. Commissioning of the PSDF Transport Reactor in combustion mode in the mid to late 1990s showed that the reactor works very well as a coal combustor, the extremely high mass flux rates in the reactor providing nearly complete carbon conversion. With the drive to develop power systems with very low-CO_2 emissions, the TROC technology appears to be a viable option based on NCCC's research.

Other currently supported oxy-combustion concepts for both pulverized coal boilers and circulating fluidized bed combustors use oxygen mixed with recycled CO_2 as the oxidant for coal combustion. In both concepts, operations are at near atmospheric pressure, and CO_2 from downstream of the combustion system is used to moderate the temperature of the highly exothermic oxy-combustion process. The TROC process, by contrast, uses recycle solids to moderate the combustion temperature and a fluidized-bed solids cooler to maintain the recycle solids temperature. Also, it operates at pressure, which confers several benefits, and has inherently low NOx and SOx emissions without downstream controls.

Following initial screening and detailed studies completed in BP1 and BP2, two further assessments of the TROC technology were completed in BP3. One study was an update for a previously conducted screening study in 2009 screening for a 500 MW greenfield TROC as a technology for capturing CO_2 from coal based power plants. This current study, "Planning Study of a Greenfield Transport Oxy-Combustion System," updated the greenfield TROC evaluation with knowledge gained in 2010 during a subsequent repowering study. This updated study also compared the economics of the revised TROC greenfield technology to other coal based options for CO_2 capture. The study results indicated that the TROC greenfield case had the lowest levelized cost of electricity of the options considered.

Through past efforts, the NCCC has evaluated TROC technology for both greenfield and repowering applications, comparing TROC with other coal-based technologies for CO_2 capture. The second evaluation, "Economic Comparison of Transport Oxy-Combustion and Natural Gas Combined Cycle with Post Combustion CO_2 Capture," extended these past efforts by comparing the economics of TROC to a natural gas combined cycle with CO_2 capture. The results show that TROC offers a viable economic alternative at capacity factors above 40 percent and an economic benefit at capacity factors above 70 percent.

5.0 GASIFICATION

The PSDF gasification process, represented in Figure 48, features key components of an IGCC plant. These include high pressure solids feed systems; a Transport Gasifier; syngas coolers; a hot gas filter vessel, the particulate control device (PCD); and continuous ash depressurization systems developed at the PSDF for ash cooling and removal. Operation of the gasification process occurred over two test runs during BP3—test runs R06, and R07—for a total of 2,204 hours. Test run R06 began on May 18, 2011, but the run was interrupted by the failure of a nitrogen tank in the on-site vendor-operated nitrogen plant (see details below). Operation resumed on July 7, and the gasification process ran continuously until August 21, 2011. R07 was performed between October 20 and December 12, 2011. Both these runs utilized Powder River Basin (PRB) coal as the gasifier fuel. At the conclusion of R07, the gasification process had operated for over 16,800 hours on coal. The test runs provided the opportunity to test processes specific to gasification, as well as to provide syngas for SCU testing.

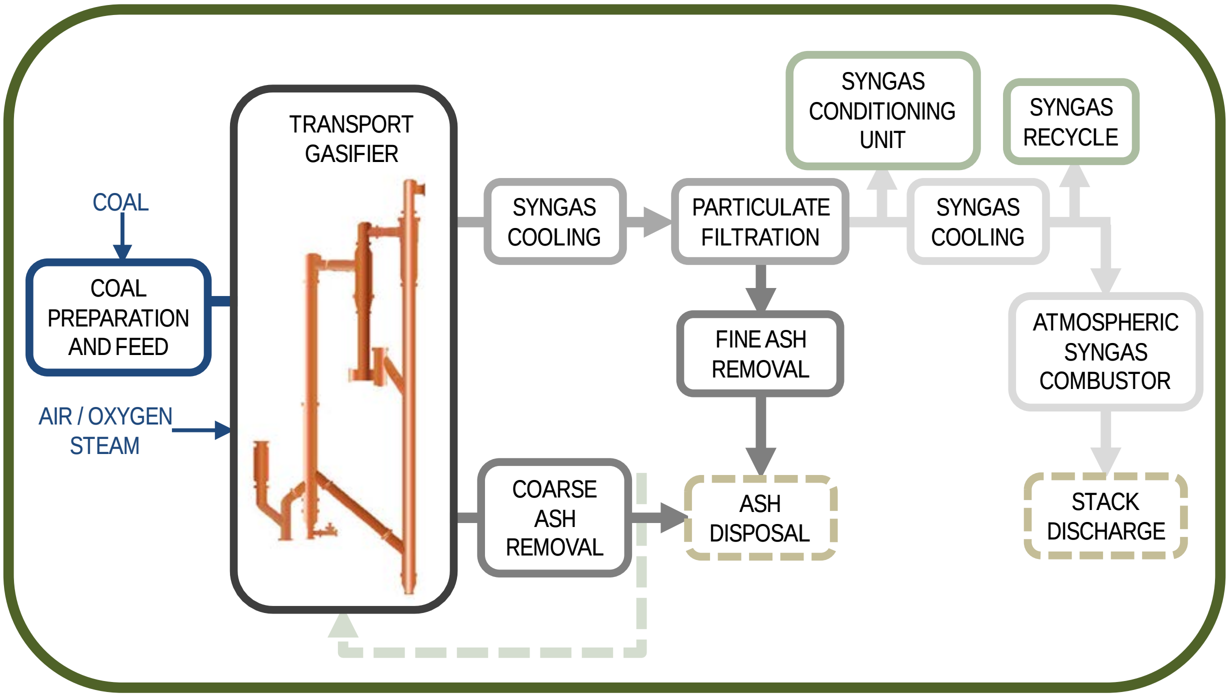


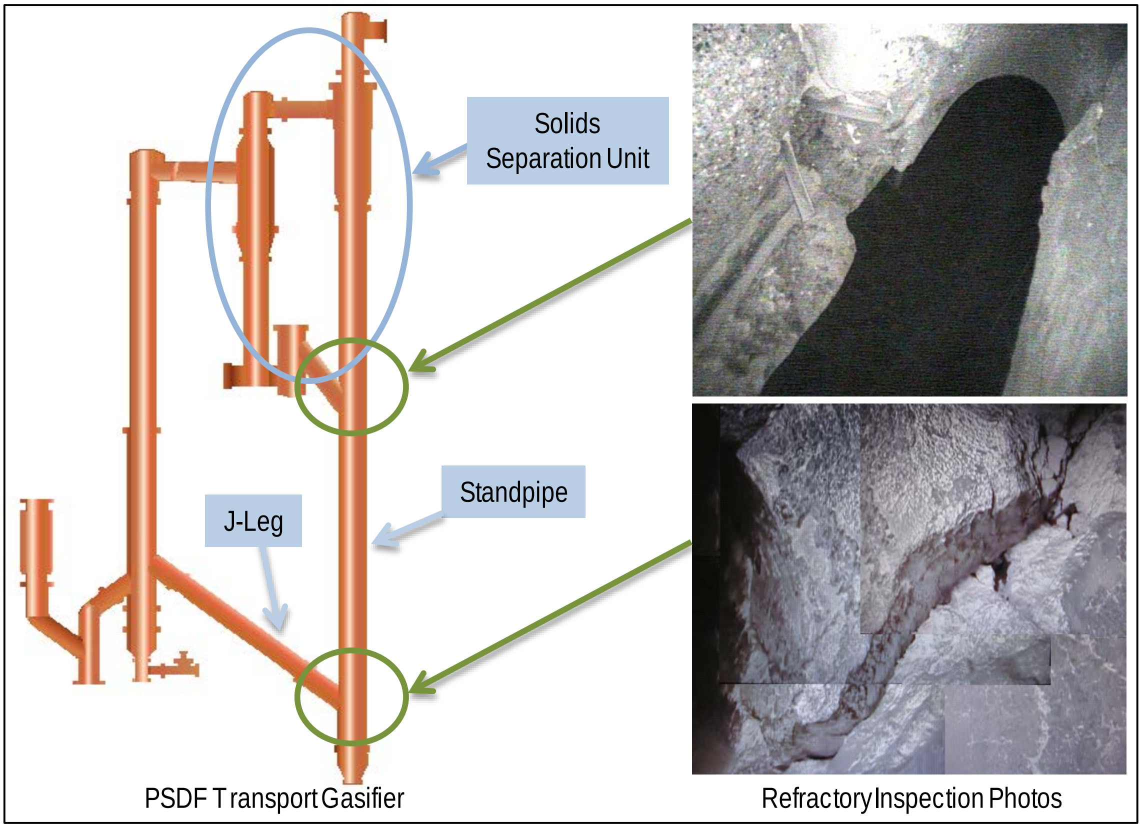
Figure 48. Flow Diagram of PSDF Gasification Process.

<u>R06 Nitrogen Tank Failure.</u> R06 gasification operations were suspended due to a brittle fracture of a carbon steel accumulator tank inside the nitrogen generating plant operated by the Linde Group on May 24. Southern Company and Linde personnel jointly concluded a root cause analysis of the event and a safety review of the nitrogen plant process equipment during June. The analysis revealed that the root cause of the failure was an electronic failure of two safety control features for the low pressure nitrogen liquid vaporization system, likely the result of a power surge or over-current. The consequences of these failures allowed liquid nitrogen to flow to a carbon steel accumulator tank, causing a brittle fracture failure due to a loss of vessel ductility.

The damaged tank was inspected by Linde personnel with oversight from Southern Company personnel. The inspection showed that the damage was confined to the areas near the saddle supports on either end of the vessel. The area immediately adjacent to each saddle support was removed and new plate and saddle supports were fabricated and welded into place. The vessel was repaired in compliance with applicable industry codes and standards and was re-installed. In addition to the vessel repair, a process safety review of the liquid vaporization system and associated control logic was conducted. Several steps were implemented as a result of this evaluation which included the following: installing an independent pneumatic low temperature protection system; restoring the existing vaporizer switching system with a new electronic temperature switch downstream of the vaporizers; modifying control logic program to provide greatest amount of reserve capacity for emergency short term operation; installing additional thermocouples to permit real time trending and additional alarm generation; and developing additional procedures to enhance the ability to respond to generated alarms on the nitrogen supply.

5.1 STANDPIPE REPLACEMENT

A major 2012 project deliverable for DOE is the replacement of the gasifier standpipe. During routine inspections of the gasifier in 2009, refractory damage was identified in the standpipe and entailed hot-face refractory loss. Two places of concern were the intersection of the solids separation unit and standpipe and the intersection of the J-leg and standpipe, as presented in Figure 49. The refractory in the standpipe and J-leg was the refractory originally installed in the gasifier, and the damage was caused by the numerous thermal cycles the gasifier has undergone over the past fifteen years.



PSDF Transport Gasifier

Refractory Inspection Photos

Figure 49. Refractory Damage in the Transport Gasifier.

Since 1996, the system had undergone the following: 5,000 hours of operation as a combustor with six commissioning runs and five test campaigns; about 17,000 hours of operation as a gasifier, with four commissioning runs and 27 test campaigns; and a number of trips and associated start-ups and shutdowns as well. A commercial unit is expected to see fewer start-ups and trips. Also, as the pipe diameters will be larger, the stress levels experienced by the refractory will be lower. Hence, the refractory life for a commercial unit is projected to significantly exceed the life of refractory achieved in the pilot plant.

Figure 50 presents images of the region about the J-leg and standpipe intersection with the damaged refractory. The thermal image shows higher temperatures in the upper region where the refractory damage was incurred. The second view shows some of the surface thermocouples and the fittings making up the water deluge system that were emplaced as precautionary measures to allow continued gasifier operation while the replacement equipment was being fabricated.


Thermal Imaging during R06
Thermocouples and Water Deluge System

Figure 50. Intersection of J-Leg and Standpipe with Damaged Refractory.

In BP3, the replacement standpipe was fabricated off-site and delivered to the NCCC (see Figure 51). During the gasifier operation with the existing standpipe in place, surface temperatures were monitored with thermocouples and thermal imaging to ensure that the carbon steel gasifier shell was not damaged due to high temperatures from refractory failure. As planned, upon completion of test run R07 in mid-December, demolition of small bore piping and dismantling of related instrumentation/equipment began. The standpipe replacement is scheduled for completion in the second quarter of 2012.


Standpipe Fabrication
Finished Standpipe in Storage

Figure 51. Replacement Gasifier Standpipe.

5.2 DEVELOPMENTAL COAL FEEDER

Over test runs R06 and R07, the developmental Pressure Decoupled Advanced Coal (PDAC) feeder operated for about 1,750 hours, with 34 hours of operation as the sole coal feeder. The coal feed rate varied from 500 to 4,800 lb/hr. Improvements to the feeder were ongoing throughout the reporting period. These improvements are detailed below.

Modifying Nitrogen Flow Distribution. The distribution of nitrogen gas addition to the bottom of the feeder was modified to decrease nitrogen flow rate variability and improve overall nitrogen flow control. Since nitrogen gas addition to the feeder is used to control the coal feed rate to the gasifier, this improvement in nitrogen flow control also resulted in decreased coal feed rate variability. Figure 52 illustrates the decreased variability in nitrogen flow from run R04 to run R06. The internal configuration of the feeder bottom was also changed, reducing coal particle segregation and providing more consistent coal feed to the gasifier.

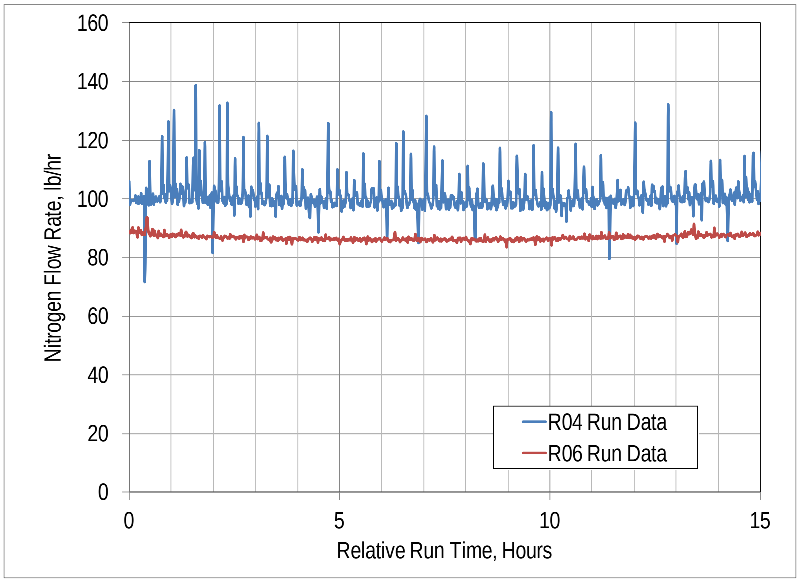


Figure 52. PDAC Feeder Nitrogen Flow Rate during R04 and R06 Operation.

Modifying Feed Rate Trim Controller. Improvements to the control logic continued with the modification of the coal feed rate trim controller. This controller was designed to detect instantaneous decreases in coal feed rate and respond by automatically increasing nitrogen flow to the feeder to increase coal feed. The modification made to the controller included adding a new control point utilizing the feeder conveying line differential pressure transmitter, which has been shown to provide a good instantaneous indication of coal feed rate. This change was successfully tested during R06 and R07 and served to maintain a more constant feed rate to the gasifier. Figure 53 demonstrates the nitrogen flow valve response to the modified controller. The controller can be modified in the future to use other coal flow measurement devices.

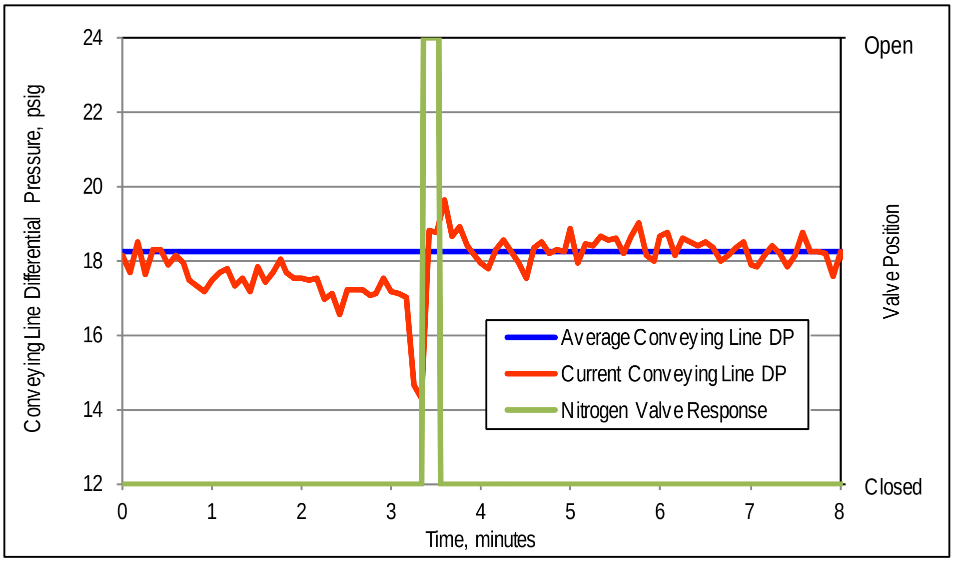


Figure 53. PDAC Feeder Trim Controller Response to Change in Coal Feed Rate.

5.3 GASIFIER OPERATION

Gasifier operation was stable during runs R06 and R07, with steady-state carbon conversions comparable to previous test runs, as demonstrated by Table 14. The historical PRB coal carbon conversion represents average values from PRB test runs since the 2006 gasifier modifications were completed.

Table 14. Steady State Carbon Conversions for R06 and R07.

	Maximum	Minimum	Average
Historical PRB Coal Carbon Conversion, %	99.6	95.3	98.6
R06 PRB Coal Carbon Conversion, %	99.6	96.6	98.7
R07 PRB Coal Carbon Conversion, %	99.2	97.3	98.8

Gasifier temperature control enhancements utilizing upper and lower mixing zone air flow adjustments were ongoing during R06 and R07. Since gasifier temperatures respond rapidly to changes in air feed rate, further tuning of the individual and total air flow controllers to prevent temperature fluctuations was required to maintain steady-state operation during previous runs. Beginning in R06, a more aggressive algorithm was created that accounted for sudden increases and decreases in gasifier temperature resulting from non-steady-state operation, such as variations in coal feed rate. Using a rate of change analysis for gasifier temperature, the controller can scale its response by varying the total air flow rate set point more aggressively. After steady-state operation has been reached again, the slower tuned controllers take control again. Figure 54 illustrates the improvement in gasifier exit temperature control achieved by these controllers. Gasifier temperature variation has been reduced to less than 5°F.

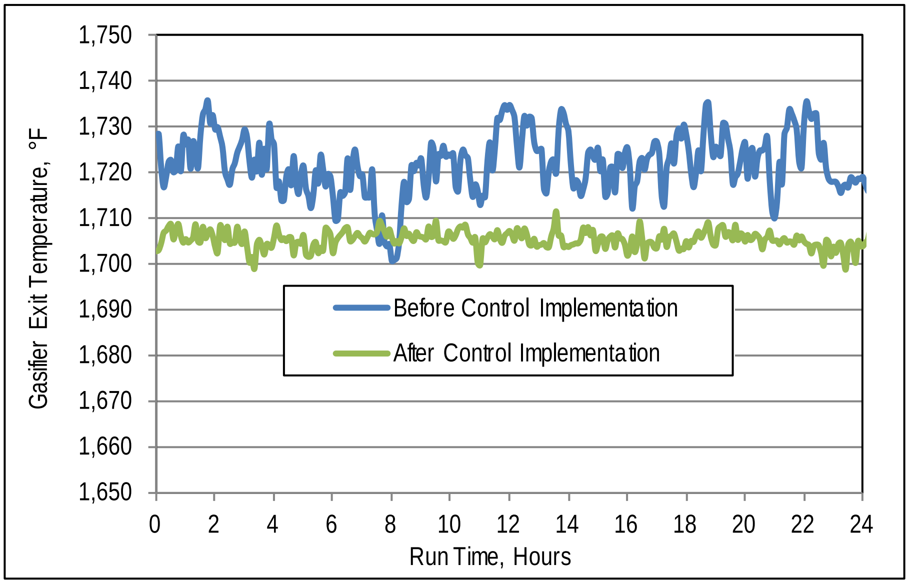


Figure 54. Gasifier Temperature Control during Non-Steady-State Operation.

5.4 SENSOR DEVELOPMENT

Because of the research nature of the NCCC/PSDF and the unique process conditions, significant effort has gone into the development of instrumentation. During BP3, sensor development included a sapphire thermowell for gasifier service, a coal flow measurement device, and coal feeder level probes.

5.4.1 SAPPHIRE THERMOWELL

A sapphire thermowell was installed in the gasifier riser prior to run R04 to test the durability of this material in a high-velocity, erosive environment. The performance of the sapphire thermowell was compared with an existing HR-160 thermowell installed in the same plane of the gasifier. Both thermowells contained Type-N thermocouples, which have an accuracy of +/- 0.75 percent. The percent difference between the average steady state temperature indications of the thermocouples averaged about one percent during R04 and averaged about 3.5 percent during R05.

During R06, the sapphire thermowell and thermocouple assembly continued to show good responsiveness during gasifier temperature changes, but the temperature indication continued to drift relative to the reference thermocouple. Following R06, the thermowell was taken out of service due to this discrepancy in temperature readings, which is demonstrated by Figure 55. The sapphire thermowell and assembly was in operation for over 2,700 hours of gasification operation.

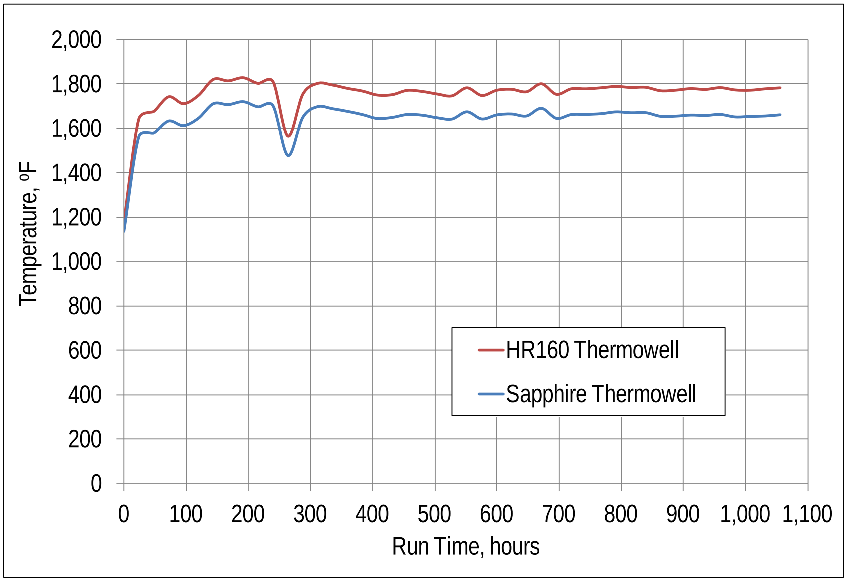
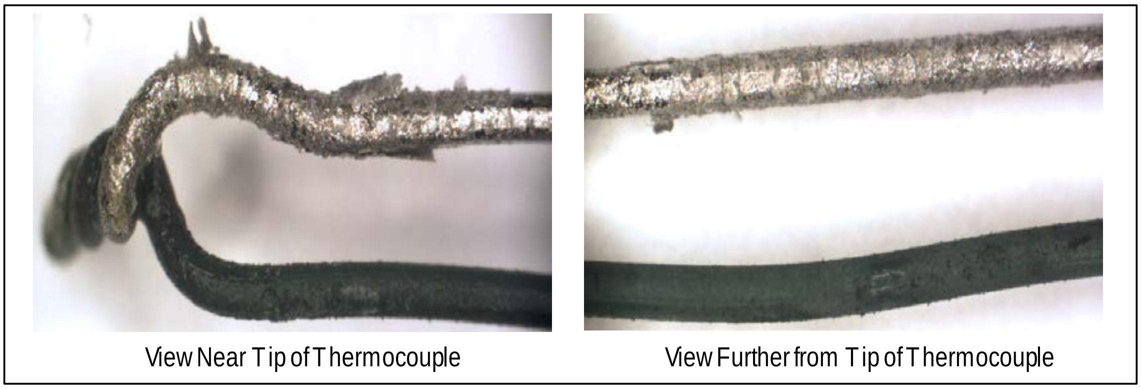


Figure 55. Comparison of Temperature Readings for Sapphire and HR-160 Thermowells.

Following the R06 run, the thermowell assembly was removed from the gasifier for visual inspection. The thermowell revealed virtually no sign of erosion or ash deposition. To determine the root cause for the temperature drift, the thermowell assembly was sent off-site further analysis. However, upon arrival at the analytical laboratory, the ceramic outer sheath, insulation rod, and sapphire tube were found badly damaged even though proper precautions had been taken in packaging the unit. The damage prevented a definitive root cause analysis but analysis did show corrosive attack and micro-cracking of the thermocouple, as illustrated by Figure 56, indicating that syngas contact had occurred.


View Near Tip of Thermocouple

View Further from Tip of Thermocouple

Figure 56. Corrosion Attack and Micro-Cracking of Thermocouple.

It is likely that the thermowell had a crack or weak point prior to the beginning of gasifier operation, leading to the syngas exposure and slow decay in thermocouple reading. Emerson personnel are currently fabricating a new thermowell assembly to re-install in the same location prior to run R08. They will be on-site for assistance during the initial inspection and installation of the assembly. Discussions are also underway to look at potential tests of alternate sapphire thermowell designs in other gasifier locations.

5.4.2 DENSFLOW COAL FLOW METER

A new coal flow measuring device, the DensFlow meter from SWR Engineering, was installed in the PDAC feeder discharge line to the gasifier prior to R04. This device is a non-intrusive technique of measuring solids flow using a patented alternating electromagnetic field. The solids flowing through the device absorb this field energy, and a measurement of density of the material is inferred. Simultaneously, conveying velocity is also calculated by the same sensors. The combination of the two measurements with the cross sectional area of the device yields a mass flow rate of material through the feeder discharge line.

Operation of the flow meter during R04 with Mississippi lignite showed good agreement when compared to the existing weigh cell loss of weight calculation. The measurement was also sensitive to instantaneous changes in coal feed rate when compared to existing conveying line differential pressure readings. During R05, the flow meter again displayed good agreement with the weigh cell calculation at higher flow rates, but at lower rates, the agreement was poor.

Calibration of the coal flow measurement device from SWR Engineering was performed during the outage prior to the R06 run. However, after re-installing the flow meter, it was discovered that the ceramic insert inside the meter had cracked, likely when tightening the meter flanges. Therefore, the flow meter was sent off-site for repairs and was unavailable during R06. Testing of the DensFlow coal flow meter from SWR Engineering resumed during the R07 run after repair of the ceramic insert that had been previously damaged prior to R06. New installation procedures were implemented to ensure proper alignment of the unit and tightening of the flanges. The device was also calibrated to PRB coal per the manufacturer’s instructions prior to installation.

Performance of the device during R07 revealed a lower coal feed rate indication when compared to the feed rate calculated from the feeder weigh cells. As a result of this finding and the limited range of operation during the run, PDAC feeder off-line testing was planned for the post-R07 outage to establish flow meter operation over a range of coal feed rates and conveying line velocities. Collaboration with SWR Engineering personnel regarding the outcome of the data generated will determine the path forward for future testing.

5.4.3 COAL FEEDER LEVEL PROBES

Dynatrol Level Probes. Long-term evaluation of Dynatrol vibration level probes continued during runs R06 and R07. The level probes were installed in the PDAC feeder dispense vessel and the lock vessel prior to the R02 test run and have operated for more than 4,700 hours. The probes, which were installed to replace older, less reliable capacitance probes, continued to demonstrate consistent indication of solids levels inside the vessels for both lignite and subbituminous coals.

Drexelbrook Level Probe. Evaluation of the Drexelbrook point sensitive level probe also continued during runs R06 and R07. The probe was vertically installed in the top of the PDAC feeder dispense vessel prior to the R04 test run to permit higher level control of the coal inventory in the vessel. Since installation, the probe has continually provided steady solids level control, mitigating the impact of solids level variation on coal feed rate. A total of 3,500 hours of reliable operation has been achieved to date.

5.5 HOT GAS FILTER ELEMENTS

The PCD operated during runs R06 and R07 with high collection efficiency (greater than 99.999 percent) and stable pressure drop. Research efforts continued to focus on long-term evaluation of material performance. Several types of filter elements and failsafe devices were tested during the runs. The failsafes installed included Pall Dynalloy devices constructed of forward (supporting screen on the inner surface of the failsafe) and reverse structures (supporting screen on the outer surface), as well as PSDF-designed devices constructed of HR-160 material. The filter elements installed consisted of sintered fiber elements—some elements manufactured by Porvair and HR-160 elements from Pall—and sintered powder filter elements—iron aluminide elements from Pall and elements with a high alloy material from Mott. The elements with the greatest exposure time, which are iron aluminide elements, have accumulated over 15,700 hours of syngas and fine ash particulate exposure to date.

Materials testing efforts were also continued during R07 including a series of test coupons of metal alloys being investigated for hydrogen separation membranes. The coupons were placed downstream of the filter elements where they were exposed to particulate free raw syngas in order to assess their resistance to degradation arising from syngas stream impurities, such as hydrogen sulfide. To date, approximately 50 different alloy compositions have been exposed over the course of three gasifier runs with a total exposure time of 2,123 hours. Duplicates were also tested during R07 to ensure repeatability of the results. Preliminary analysis shows that the varied nature of syngas impurities produced significant differences compared to controlled laboratory conditions.

5.6 JOHNSON MATTHEY MERCURY SORBENT

In collaboration with NETL, Johnson Matthey has been developing a solid, sorbent-based process to remove mercury and other trace contaminants, such as arsenic and selenium, at high temperature in coal gasification processes. Compared to low-temperature capture by activated carbon, high-temperature capture of these trace elements retains the high thermal efficiency of the coal gasification process in IGCC power plants.

The high-temperature, palladium-based (2 wt%), mercury adsorbent supplied by Johnson Matthey was tested in run R06 at the SCU. Around 10 pounds of sorbent (which had been reduced on site to its metallic form) were placed in a pressure vessel in a fixed-bed arrangement. A catalyst bed height of 14 inches was selected to achieve the required space velocity of 2,800/hr. Table 15 lists the test conditions for this run. These operating conditions were identical to those from test run R05 except that the palladium content in the sorbent was 5 wt% in R05. The primary objective of the testing in R05 and R06 was to compare the efficiency of the sorbent with different palladium contents.

Table 15. Operating Conditions for Mercury Sorbent Testing in R06.

	Value
Syngas Flow Rate, lb/hr	25
Operating Temperature, °F	500
Operating Pressure, psia	195
Hours of Operation	1,011
Palladium Content, wt%	2

During the run, twelve gas samples, six each from the inlet and outlet of the sorbent bed, were collected using a modified EPA Method 29 for trace metal analysis. Analyses were carried out primarily for mercury, arsenic, and selenium. After the run, the sorbent was removed from the reactor in 7 layers and returned to Johnson Matthey for elemental analyses to determine contaminant penetration levels. Results suggested no breakthrough of these species, indicating that all of the trace metals were captured by the sorbent. Figure 57 shows the syngas mercury concentration in the sorbent reactor inlet and outlet during R06. The outlet concentrations are below detection limits, except data at hour 173 and hour 508. The reason for the high outlet mercury concentration at hours 173 and 508 is still being investigated. Johnson Matthey's sorbent analysis supports near-complete absorption of mercury, as the penetration of mercury and other trace metals was limited to the first two to three layers in the sorbent bed.

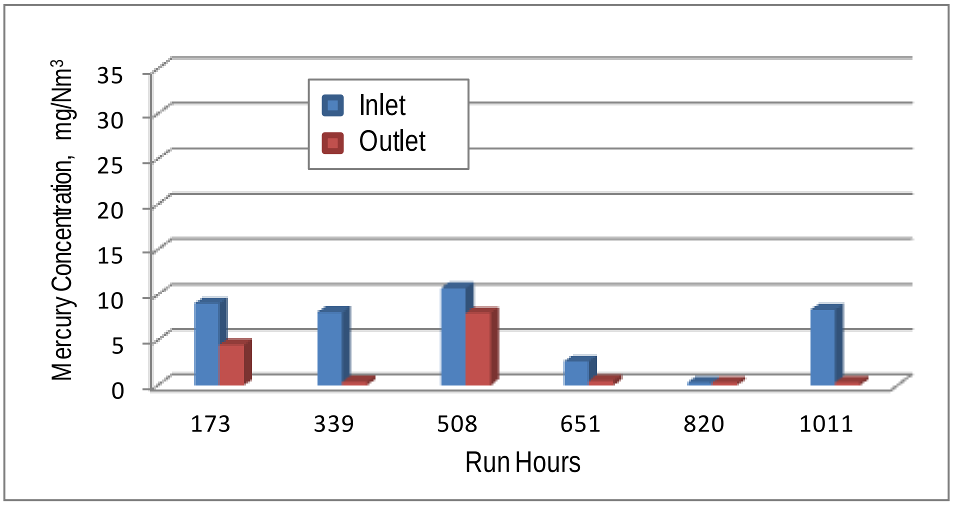


Figure 57. Results of Mercury Sorbent Testing.

6.0 CONCLUSIONS AND LESSONS LEARNED

Conclusions and lessons learned from studies and from experience gained during BP3 are discussed in the following sections.

6.1 PRE-COMBUSTION CO_2 CAPTURE

- In the absence of water, capture efficiency with CO_2 capture solvent GTA was similar to that with DEPG, and both were superior to capture with PDMS. When 10 percent water was added to the solvents, the performances of GTA and PDMS was unaffected, but that of DEPG deteriorated.
- At a steam-to-CO ratio of 1.6, all four water-gas shift catalysts tested convert sufficient CO to support CO_2 capture of around 65 percent and achieve emissions equivalent to those from a natural gas combined cycle process.
- WPI's palladium membrane demonstrated an initial hydrogen flux high enough to produce 2 lb/day, reaching the DOE target of Phase I. However, the flux decreased to 1 to 1.4 lb/day for different membranes over the test duration. High purity hydrogen product, as high as 99.9 percent and more than 470 hours of continuous operation was achieved at a pressure differential of about 11 bar with a syngas containing 35 vol% hydrogen. This represents a milestone for development of palladium-based membranes.
- Lab measurements of the WPI membrane hydrogen permeance before and after field testing showed that 35 to 60 percent of initial permeance was lost. Most of the loss was not recoverable after exposure of the membrane to pure hydrogen at high temperatures. X-ray photoelectron spectroscopy analysis revealed the presence of a significant amount of carbon and lesser amounts of sulfur and mercury on a palladium layer. Carbon also penetrated into bulk palladium and could be the major reason for the flux decline. Continued testing and research efforts will focus on understanding the impact of these contaminants on membrane performance.
- Testing of MTR's CO_2 membrane demonstrated: CO_2 enrichment as much as fivefold; CO_2/H_2 selectivity of 3 to 6; stable operation in the presence of 300 to 500 ppm H_2S; mixed-gas H_2S/CO_2 selectivity of about 3.0; stable separation performance, indicating that there was no aging or performance deterioration of the membrane modules after exposure to the raw syngas; and higher selectivity for an improved membrane structure that was tested during R07.
- Testing of MTR's hydrogen membrane demonstrated: hydrogen enrichment up to 65 vol% in the permeate, a H_2/CO_2 selectivity of about 14, and stable operation at temperatures up to 300°F for extended periods and in the presence of sulfur and other contaminants.
- The results of the tests with MPT's Carbon Molecular Sieve indicated that the performance of the membrane was quite stable when there was no pretreatment of the raw syngas. Treating the CMS permeate stream with a palladium membrane yielded hydrogen purity greater than 99 percent.
- The TDA Research solid CO_2 sorbent system operated as designed with multiple steps for each cycle, (adsorption, equalization, blow-down/regeneration, and re-pressurization). During these cycle tests, TDA Research's system successfully removed more than 98 percent of CO_2 from the syngas.

6.2 POST-COMBUSTION CO_2 CAPTURE

The following observations and conclusions were drawn from PSTU commissioning:

- A team of engineers and plant operators developed plans and procedures in preparation for start-up, commissioning, and operation of the PSTU. This preparatory work served to familiarize the team with the equipment, which expedited start-up.
- Even with automatic control systems in service, disturbances in operation occurred from perturbations in power plant operation. There is regular communication with the power plant operators to prepare for such perturbations, and the PSTU operating procedures include appropriate ways to respond to such events.
- During commissioning, the CO_2 and oxygen analyzers operated with good accuracy, as evidenced by the quality of the mass balance closures. The only gas analyzer that gave problems was the FTIR system used for specialized analysis of the CO_2-depleted flue gas stream leaving the absorber. The heating of the flexible pipe delivering the sample to the analyzer was insufficient to evaporate the water droplets in the stream, which were more numerous than expected. Excessive condensation in the piping stripped species such as ammonia from the gas, causing the analyzer to register low values. To reduce the carryover of water droplets from the duct, a new demister was added, and the extraction probe was set at an angle to allow condensate to fall back into the duct. The effectiveness of this modification will be assessed during subsequent testing.
- Differential pressure measurements across the packed beds in the absorber and regenerator were unreliable. The impulse lines from the sensor to the transmitters are long, and temperature variations along the lines affect the pressure and bias the measured differential. This issue was alleviated partially by insulating the impulse lines, and work continues to reach final resolution.
- In analyzing data, the mass and heat balance envelope was originally drawn around the absorber and wash tower, which produced acceptable mass balance closures but resulted in some errors in the heat balance due to a cooling circuit in the wash tower. To avoid this error, the envelope was drawn around only the absorber. The flue gas composition was unchanged, and there was sufficient information to determine the moisture content at the wash tower inlet. The MEA content at the inlet was not estimated, but this flow is considered to be sufficiently low as to not introduce significant error.
- Process issues were steadily resolved and control circuits tuned, allowing the plant to be operated in a controlled and safe manner at steady conditions. The process operated as designed, and flow rates and analysis procedures for the gas and liquid streams were all validated. This operational success demonstrated that the plant is ready to support testing of developers' solvents.
- A total of 23 balance periods were completed, and the process data collected were carefully scrutinized to identify sources of error. No serious flaws were discovered, but a few corrections were made to calculation procedures, resulting in mass balance closures very close to 100 percent for the absorber. This successful outcome has two benefits. First, the understanding developed and the resolution of data collection issues raises confidence in the ability to provide developers with reliable information when testing their solvents. This will allow them to make technology development decisions with confidence. Second, the data

collected can be used with confidence by modelers within the Carbon Capture Simulation Initiative and contribute to accelerating the development of cost-effective CO_2 capture technologies.

- Achieving good heat balances was complicated by the absence of reliable data for solvent specific heat, which is a function of temperature, MEA concentration, and CO_2 loading. Other data bases are being investigated to find suitable data.

Conclusions from the ACC Mobile Test Unit are provided below.

- Round-the-clock operation was achieved at controlled conditions. The extended continuous operation increased understanding of plant and equipment performance and improved the ability to respond to upsets in power plant operation.
- The MTU was designed for northern European operation and was supplied with air-cooled heat exchangers. Summer temperatures in Alabama required the addition of water cooling to some circuits.
- The reclaimer was operated and reduced the concentration of impurities by 80 percent with only minimal loss of solvent.
- The analyses of ammonia and alkyl amines were originally performed by GC-MS after derivatization with benzene sulfonyl chloride. This method has a very high sensitivity of about 1 ug/L for alkyl amines and 1 mg/L for ammonia. However, it was found that sulfamic acid was a poor choice for sampling of ammonia, as sulfamic acid in water solution may hydrolyze to ammonium bisulfate. GC-MS analysis of a blank 0.1M sulfamic acid sample prepared on site showed indeed an ammonia concentration of 14.5 mg/L, which in fact was comparable to the impinger analysis in this work and would correspond to an ammonia emission of a few ppmv. Hence, sulfamic acid is not applicable for sampling of ammonia. Absorbents like hydrochloric or sulfuric acid should be used in the future.
- With the full anti-mist mode applied, the FTIR reading of MEA concentration in the absorber outlet was less than 1 ppm.
- Net CO_2 loading, defined as the difference between CO_2 rich and lean solvent loadings, was calculated for the executed runs with CCAmine, and a maximum net loading was 2.3 mol CO_2/L solvent, corresponding to a working capacity for the solvent of 10 wt%. The majority of results were, however, in the range between 1.6 and 2.0 mol CO_2/L solvent, corresponding to a working capacity for the solvent between 7 and 9 percent. MEA has a working capacity typically between 5 and 7 wt%.
- Kinetic characteristics for the solvent were assessed based on obtained CO_2 loading in the CO_2 rich solvent. CO_2 loading dropped when approaching and exceeding 90 percent CO_2 capture.
- The pH controlled wash system proved effective at capturing gaseous phase alkaline compounds, i.e. ammonia and alkyl amines. The test results also show low amine emissions achieved with the anti-mist design, and additional reductions may be achieved through further optimization.
- The effect of changing the absorber feed point was investigated, and comparable runs with lower/upper feed points indicate that rich loading decreases by 12 percent when reducing packing height by 39 percent. Specific reboiler duty was not significantly increased.

- Approximately 84 percent of the heat stable salts in the lean amine in the MTU before start of reclaimer operation was removed during the solvent feed period of the reclaiming process.
- There was no net loss of free amine during the reclaiming period, as the free amine concentration in the lean solvent was the same before and after reclaiming. Also, single amine levels were practically unchanged after reclaiming.
- The viscosity was decreased in the lean solvent by reclaiming, while the viscosity in the reclaimer liquid increased significantly during the reclaiming process.
- There is no significant increase in total heat duty during reclaiming. The specific reboiler duty values before and after reclaiming are equal.

Conclusions from the B&W solvent testing at the PC4 include:

- A technical assessment was performed by B&W, and a document describing the appropriate procedures for a range of risk scenarios was created for the operators and engineers at the PSTU. During the campaign, a number of events occurred which required implementation of these contingency plans, and following the recommended course of action resulted in an expedient return to normal and safe operation.
- During the test campaign, a total of approximately 115 test conditions were run. A significant quantity of data was gathered to compare with B&W's process simulation models. In addition to the operating data gathered for process model validation and verification, the test campaign also yielded a great deal of data in other areas of interest, such as corrosion, solvent degradation, and effluent stream characterization.
- Liquid samples were routinely withdrawn using the automated system for the purpose of checking solvent concentration and CO_2 loading. In addition, manual samples were withdrawn at regular intervals to serve as a check to the automatic samples and to further characterize the chemical composition of various process streams. The samples have been sent for chemical analysis to identify and quantify corrosion and solvent degradation species. Also, NCCC staff worked with B&W personnel to perform gas-phase sampling at various locations in the system. The purpose of these samples was to analyze for airborne compounds such as the OptiCap solvent, ammonia, aldehydes, and nitrosamines, which could be present in either the gaseous state, or possibly entrained in the liquid phase. Analysis of samples is pending at the time of this report publication.
- Overall results indicate that corrosion rates for the OptiCap solvent are relatively low, as compared to other benchmark solvents such as MEA. The most significant corrosion has generally occurred in areas of the plant where expected. Many of the on-line corrosion rate measurements to date are less than 1 mpy. These preliminary results must be confirmed via weight loss coupon analysis before definitive recommendations regarding suggested materials of construction can be made.
- The chemical composition of the solvent appears to have changed, as indicated by a change in color. Although changes in solvent color are not necessarily indicative of the presence of degradation species, changes in solvent composition due to degradation are often accompanied by color changes similar to those observed for the OptiCap solvent during the subject campaign. Chemical analysis will confirm and quantify the extent of degradation species present in the solvent as a function of time.

- During the OptiCap test campaign, values in the range of 1,100 to 1,125 BTU/lb CO_2 were achieved at a CO_2 removal efficiency of 90 percent. These results are consistent with both lab- and pilot-scale measurements as well as process simulations, which indicate that further energy savings are possible when using a cross-flow heat exchanger optimized for the OptiCap solvent.

The following items describe the conclusions reached from testing of the MTR 1 MW membrane:

- During the shakedown period, several operational issues required resolution. The issues were mainly due to the fact that the system was originally designed and built for testing at the Cholla power plant. The different operating conditions at NCCC required adjustment of the engineering design. With the site service provided by the equipment vendors, MTR and NCCC engineers resolved all the system issues and completed the system shakedown activities.
- Overall, the system removed 78 percent (0.89 metric ton/day) of the CO_2 from post-FGD flue gas that contained 1.15 metric tons/day CO_2. The CO_2 content of the flue gas was reduced from 12.1 percent in the feed to 3.3 percent in the treated gas. Process simulations confirmed that the overall CO_2 removal rate and high CO_2 enrichment factor were consistent with expected performance. MTR plans to slightly reduce the flue gas flow rate in the future test to the design value (1 metric ton/day), to verify that the membrane process can reach the target CO_2 capture rate of 90 percent.

6.3 OXY-COMBUSTION

- The updated study, “Planning Study of a Greenfield Transport Oxy-Combustion System,” compared the economics of the revised TROC greenfield technology to other coal based options for CO_2 capture. The study results indicated that the TROC greenfield case had the lowest levelized cost of electricity of the options considered.
- A second evaluation, “Economic Comparison of Transport Oxy-Combustion and Natural Gas Combined Cycle with Post Combustion CO_2 Capture,” compared the economics of TROC to a natural gas combined cycle with CO_2 capture. The results show that TROC offers a viable economic alternative at capacity factors above 40 percent, and an economic benefit at capacity factors above 70 percent.

6.4 GASIFICATION

- Operation of the gasification process occurred over two test runs during BP3—test runs R06, and R07—for a total of 2,204 hours. At the conclusion of R07, the gasification process had operated for over 16,800 hours on coal.
- Modifications to the distribution of nitrogen gas addition to the bottom of the PDAC feeder improved coal flow rate control and overall nitrogen flow control.
- Improvements to the PDAC feeder control logic included adding a new control point utilizing the feeder conveying line differential pressure transmitter, which has been shown to provide a good instantaneous indication of coal feed rate. This change was successfully

tested during R06 and R07 and served to maintain a more constant feed rate to the gasifier. The controller can be modified in the future to use other coal flow measurement devices.

- Gasifier temperature control enhancements utilizing upper and lower mixing zone air flow adjustments reduced gasifier temperature variation to less than 5°F.
- Gasifier operation was stable during runs R06 and R07, with steady-state carbon conversions comparable to previous test runs.
- During R06, the sapphire thermowell and thermocouple assembly continued to show good responsiveness during gasifier temperature changes, but the temperature indication continued to drift relative to reference thermocouple. Following R06, the thermowell was taken out of service due to this discrepancy in temperature readings. Inspections showed corrosive attack and micro-cracking of the thermocouple, indicating that syngas contact had occurred.
- Performance of the Densflow coal meter on the PDAC feeder during R07 revealed a lower coal feed rate indication when compared to the feed rate calculated from the feeder weigh cells. As a result of this finding and the limited range of operation during the run, PDAC feeder off-line testing was planned to establish flow meter operation over a range of coal feed rates and conveying line velocities.
- The Dynatrol vibration level probes installed in the PDAC feeder dispense vessel and lock vessel have operated for more than 4,700 hours. The probes continued to demonstrate consistent indication of solids levels inside the vessels for both lignite and subbituminous coals.
- The Drexelbrook point sensitive level probe, installed in the top of the PDAC feeder dispense vessel, has continually provided steady solids level control, mitigating the impact of solids level variation on coal feed rate. A total of 3,500 hours of reliable operation has been achieved to date.
- During the gasification test runs, the PCD operation with very high collection efficiency (>99.999 percent) and without any filter element failures. The elements used included various metal elements supplied by Pall, Mott, and Porvair. The elements with the greatest exposure time, which are iron aluminide elements, have accumulated over 15,700 hours of syngas and fine ash particulate exposure to date.
- A number of test coupons of metal alloys being investigated for service in hydrogen separation membranes were installed downstream of the filter elements where they were exposed to particulate free raw syngas in order to assess their resistance to degradation arising from syngas stream impurities, such as hydrogen sulfide. To date, approximately 50 different alloy compositions have been exposed over the course of three gasifier runs with a total exposure time of 2,123 hours. Duplicates were also tested during R07 to ensure repeatability of the results. Preliminary analysis shows that the varied nature of syngas impurities produced significant differences compared to controlled laboratory conditions.
- Testing of the Johnson Matthey palladium-based mercury sorbent indicated no breakthrough of the metals tested, which included mercury, arsenic, and selenium. Johnson Matthey’s sorbent analysis supports near-complete absorption of mercury, as the penetration of mercury and other trace metals was limited to the first two to three layers in the sorbent bed.