The National Carbon Capture Center at the Power Systems Development Facility Test Run Summary Report

Test Run R09
November 25, 2012 – December 20, 2012

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List of Abbreviations

CCAD	Continuous Coarse Ash Depressurization
CFAD	Continuous Fine Ash Depressurization
CMS	Carbon Molecular Sieve
DOE	Department of Energy
EPRI	Electric Power Research Institute
HTM	Hydrogen Transport Membrane
MPT	Media & Process Technology
MTR	Membrane Technology & Research
NCCC	National Carbon Capture Center
PCD	Particulate Control Device
PDAC	Pressure Decoupled Advanced Coal
PRB	Powder River Basin
PSDF	Power Systems Development Facility
SCS	Southern Company Services
SCU	Syngas Conditioning Unit
TDL	Tunable Diode Laser
UA	University of Alabama
WGS	Water-Gas Shift
WPI	Worcester Polytechnic Institute
XPS	X-Ray Photoelectron Spectroscopy

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 (DOE), the PSDF is an engineering scale test center located in Wilsonville, Alabama. Since operation began in 1996, the PSDF staff has effectively developed advanced power systems, moving innovative technologies into the marketplace. Building on its previous success, PSDF now hosts the National Carbon Capture Center (NCCC) to address the nation's need for commercially viable CO_2 capture options for flue gas from pulverized coal power plants and syngas from coal gasification power plants.

Test run R09 commenced from November 25 through December 20, 2012, for a total run duration of 545 hours. Progress continued on the development of the pressure decoupled advanced coal (PDAC) feeder, gasifier control automation, sensors, instrumentation, and pre-combustion CO_2 capture technologies in the Syngas Conditioning Unit (SCU). R09 featured testing of Stanford University's Tunable Diode Laser (TDL) sensor for the instantaneous measurement of both syngas moisture and temperature downstream of the particulate control device (PCD). In addition, a proof-of-concept CO_2 measurement sensor was also tested during the run. At the conclusion of R09, the gasification process had operated for over 18,000 hours.

2.0 COAL FEED AND PREPARATION

A total of 1,600 tons of Powder River Basin (PRB) sub-bituminous coal was used in R09. Table 1 lists the average values of coal properties as-received and as-fed (after processing in the coal mill system to achieve the desired moisture concentration and particle size distribution).

Table 1. PRB Coal Properties.

Coal Property	Value
As-Received Carbon, wt %	51.1
As-Received Hydrogen, wt %	3.3
As-Received Nitrogen, wt %	1.0
As-Received Sulfur, wt %	0.3
As-Received Ash, wt %	7.2
As-Received Oxygen, wt % (by difference)	12.1
As-Received Volatiles, wt %	36.5
As-Received Fixed Carbon, wt %	31.3
As-Received Heating Value, Btu/lb	8,740
As-Received Moisture Concentration, wt%	25
As-Fed Moisture Concentration, wt%	20
Moisture Content Reduction, %	20
As-Fed Mass Median Diameter, micron	383
As-Fed Oversize (>1,180 micron) Content, wt%	12
As-Fed Fine (< 45 micron) Content, wt%	9

Both the developmental PDAC feeder and the original coal feeder operated during R09 without any major coal stoppages.

2.1 PDAC Feeder

The PDAC feeder operated for 491 hours during the run at coal feed rates ranging from 1,500 to 3,400 lb/hr. Overall feeder performance during the run was stable over the range of coal feed rates tested.

PDAC Control Logic

Testing of previous control logic improvements continued during the R09 run. The coal feed rate trim controller successfully maintained a consistent coal feed rate to the gasifier by changing the nitrogen feed rate to the feeder as necessary. The feeder conveying line differential pressure transmitter continued to be used as the indication of instantaneous changes in coal feed rate, but the controller could be adapted in the future to use other coal feed measurement devices.

Level Probes

Long term evaluation of two coal feeder solids level probes continued during R09. The Dynatrol vibration level probes installed in the PDAC feeder dispense vessel and lock vessel prior to the R02 test have continued to provide very reliable indication of solids level in both vessels for lignite and sub-bituminous coals. Further optimization of the lock vessel fill cycle has been made possible by the performance of these devices. The level probes have operated for more than 5,960 hours.

Evaluation of the Drexelbrook point sensitive level probe also continued during the run. 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 4,760 hours of operation has been achieved to date.

Coal Flow Meter

Testing of the DensFlow coal flow meter from SWR Engineering continued during R09. The 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.

While the measurement provided by the meter has shown good response to instantaneous changes in coal feed rate when compared to existing conveying line differential pressure readings, the absolute reading has not matched up well with the coal feed rate based on the PDAC feeder surge bin weigh cell device. Although the original design intent was to measure dense flow solids streams at pressure, SWR Engineering has maintained that the device is capable of measuring the more dilute phase flow regime present in the case of the PDAC feeder discharge to the gasifier. Following operation during R07, the decision was made to recalibrate

the instrument following the manufacturer's guidelines and to perform off-line feeder testing at higher coal feed rates than had previously been achieved during on-line operation prior to R08. The goal was to achieve less dilute flow and more closely approach the original design intent of the meter. However, the data collected during the off-line test period revealed that the discrepancy in coal feed rates between the weigh cell calculation and flow meter still existed even at higher rates. This result was further substantiated when observing operations during R09 as illustrated in Figure 1. Collaboration with SWR Engineering personnel regarding the outcome of the data generated will be needed to determine what changes are necessary to address the difference for future testing.

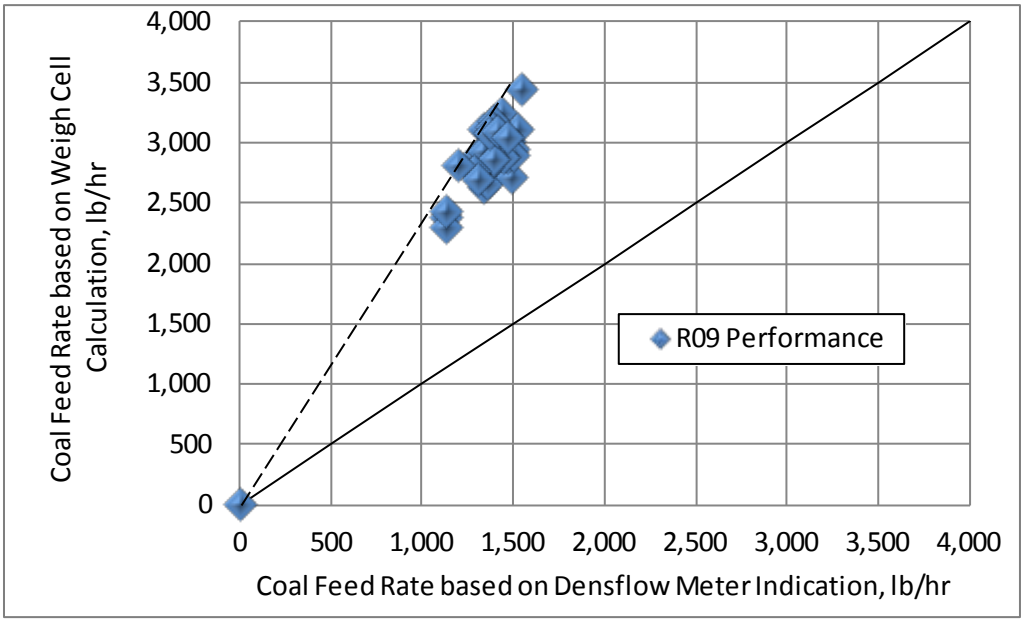

Figure 1. Biomass Feed Rate as a Function of Feeder Speed of Original Coal Feeder.

2.2 Original Coal Feeder

The original rotary coal feeder operated on coal for 537 hours at feed rates varying from 1,100 to 1,525 lb/hr. Overall feeder performance during the run was stable over the range of coal feed rates tested.

3.0 TRANSPORT GASIFIER

R09 operation consisted of 49 steady state operating periods, with recycled syngas utilized for gasifier fluidization during all periods. Gasifier operation during the run yielded high carbon conversions, as shown in Table 2. The historical figures for carbon conversion represent data collected since completion of the 2006 gasifier modifications.

Table 2. Steady-State Carbon Conversion for R09 and Previous PRB Test Runs.

	Maximum	Minimum	Average
R09 PRB Coal Carbon Conversion, %	98.7	97.8	98.1
Historical PRB Coal Carbon Conversion, %	99.6	95.3	98.4

Figure 2 plots the maximum gasifier temperature (the mixing zone temperature), the outlet gasifier temperature and pressure, and the primary gas cooler outlet temperature during the steady state periods. The primary gas cooler operated as designed, with an outlet temperature of about 800°F during the run.

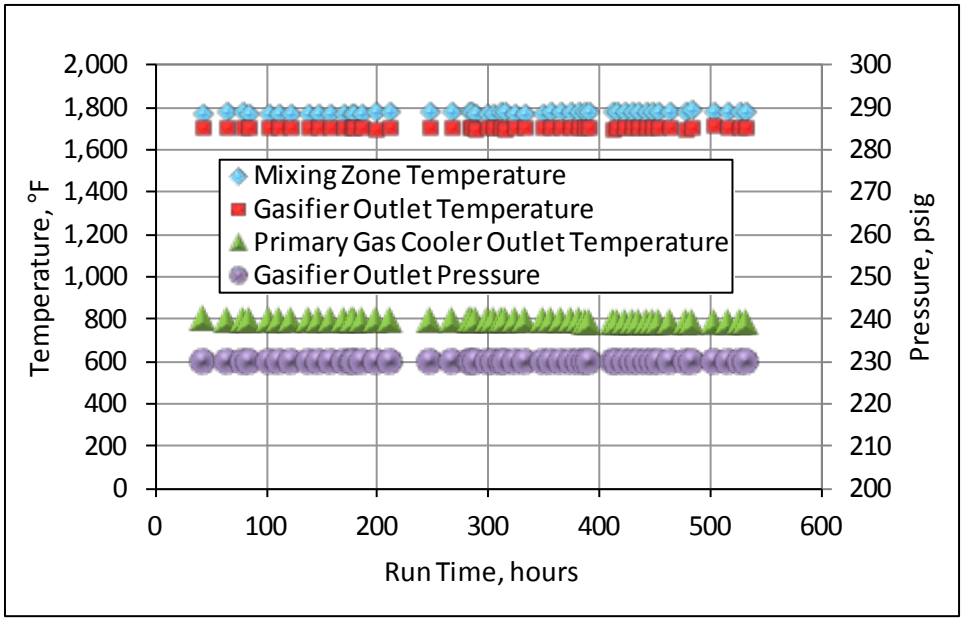

Figure 2. Gasifier Operating Conditions.

Sapphire Thermowell

Testing of the sapphire thermowell technology was suspended for R09 following the second assembly failure at the beginning of R08. NCCC staff has been collaborating with Rosemount to better understand the mode of the two failures and reach a decision regarding future testing. One potential path forward may include testing different sapphire thermowell assembly designs in other gasifier locations. More detailed test plans will be developed after technology collaboration agreements are finalized.

Automated Temperature Control

Gasifier temperature control enhancements utilizing air flow adjustments from the gasifier upper and lower mixing zone continued during R09 through incremental tuning improvements. The controller was in service during the majority of the run and performed well as illustrated in Figure 3. Gasifier temperature variation during the period was less than 2°F.

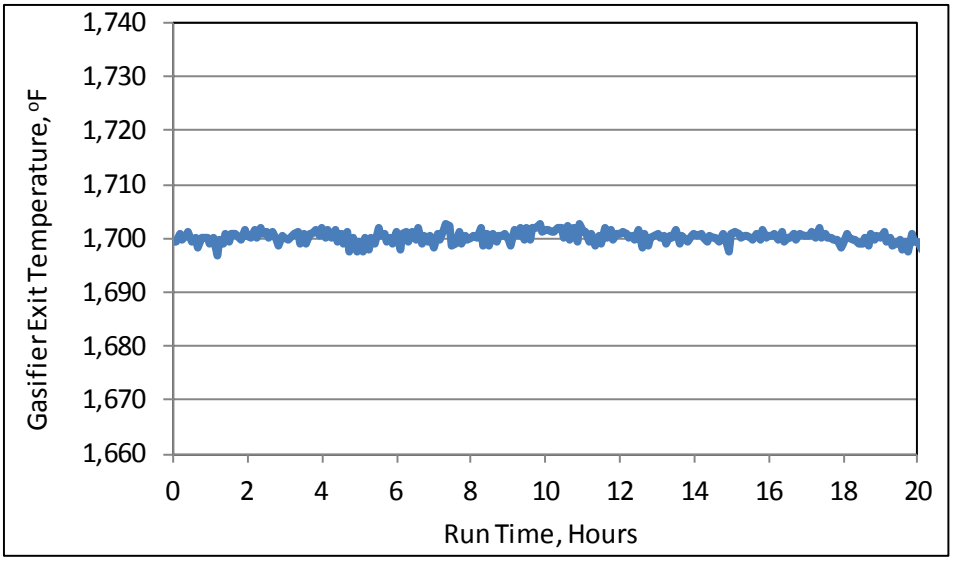

Figure 3. Gasifier Temperature Control Performance.

Refractory Inspections

Gasifier system inspections did not reveal any areas of major concern. The new gasifier standpipe installed earlier in the year continued to demonstrate excellent refractory performance. Figure 4 shows the condition of the standpipe refractory following R09 operation.

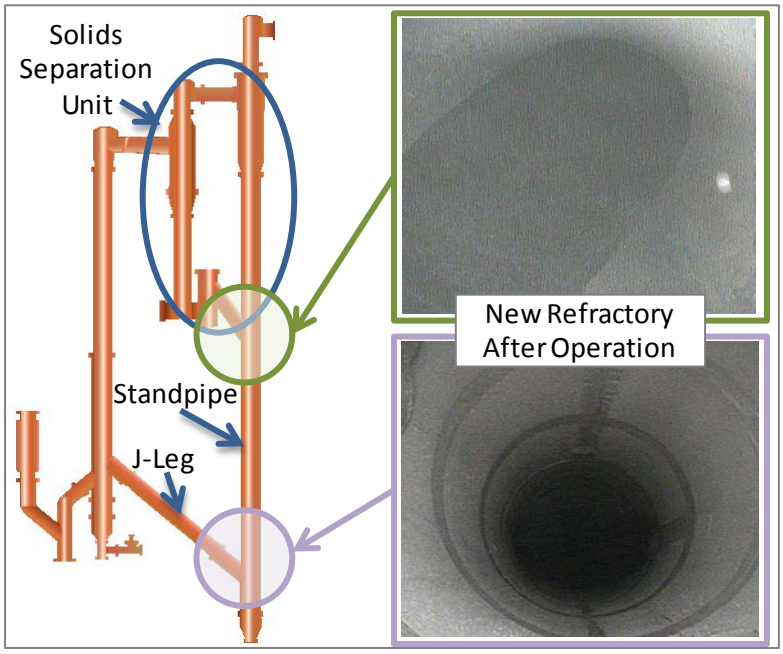

Figure 4. Inspections of Standpipe Refractory.

Monitoring of the gasifier refractory at the mixing zone and J-leg intersection continued during R09. Post-run inspections of the refractory continued to show minimal additional wear with an

acceptable thickness of hard-face refractory remaining (see Figure 5). A skin temperature monitoring system has been maintained for the area during operations, and the area will be closely inspected during future outages.

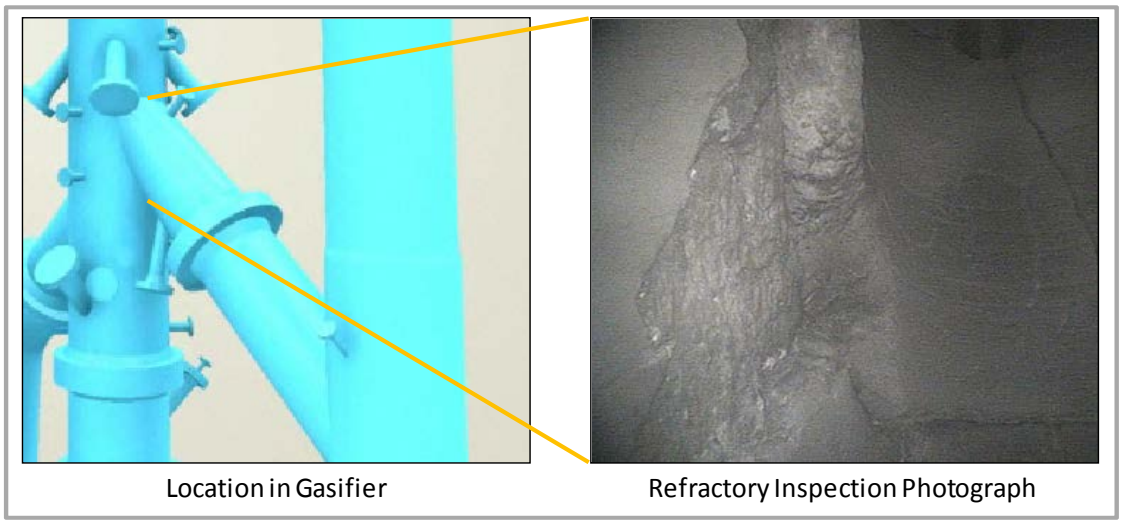

Figure 5. Inspections of Refractory in Gasifier Mixing Zone and J-leg Intersection.

4.0 PRIMARY GAS COOLER

During the R09 start-up, a leak was identified in the primary gas cooler, which is located between the gasifier and the PCD. Hydrostatic pressure testing identified one leaking tube, which was filled with plugging material to prevent further leaking. While the primary gas cooler operated satisfactorily during R09, concern that future tube leaks could develop led to the decision to replace the heat exchanger as soon as practical. An order was issued for a complete replacement of the cooler and should arrive at the NCCC in June 2013.

5.0 STANFORD UNIVERSITY TUNABLE DIODE LASER

The TDL sensor from Stanford University/Electric Power Research Institute (EPRI) was demonstrated on the syngas line downstream of the PCD during R09. Design and installation of the system was completed in November. Figure 6 shows the installed sensor.

Figure 6. TDL Sensor Installed in Gasification Process.

The optical windows and laser system arrived in early November. NCCC personnel led the effort to ensure initial alignment of the optical windows and pitch/catch heads of the laser. The laser system was installed in an analyzer shelter and connected to the field optical devices (see Figure 7). The system was leak checked and placed into service during the R09 test run start-up. The instrument functioned and collected data throughout the R09 run.

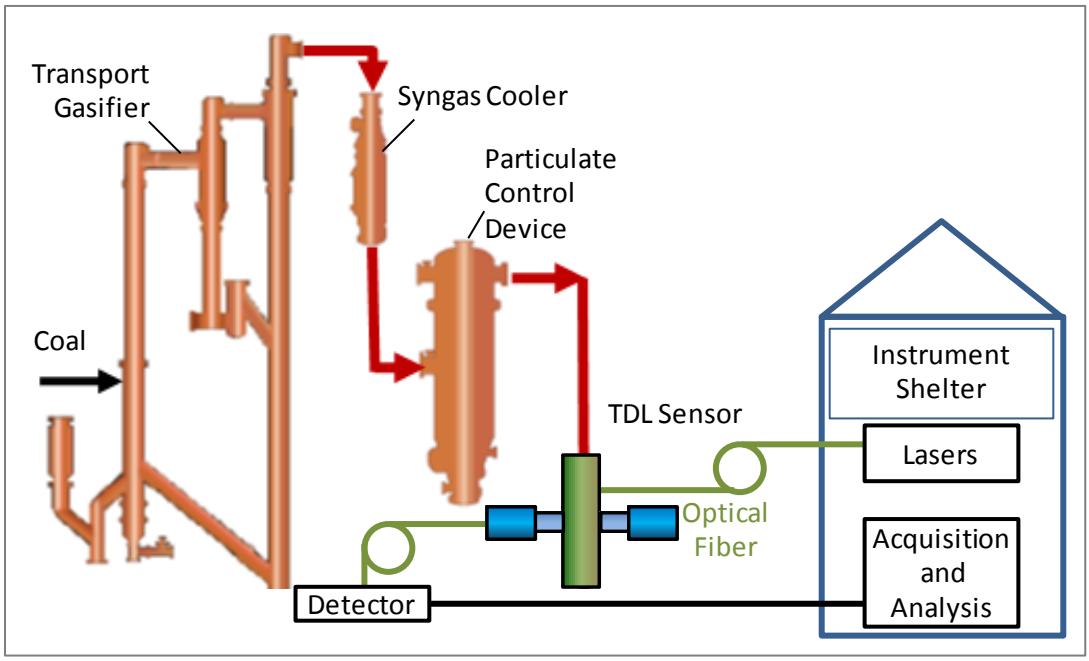

Figure 7. Location of TDL Sensor in NCCC Transport Gasification Process.

The sensor provided continuous, time-resolved measurements of moisture concentration and temperature. In addition, Stanford personnel tested a proof-of-concept CO_2 measurement during start-up. Initial testing focused on testing the sensor during system changes during system heat-up and eventual transition to steady state gasification conditions. The TDL was then set up for long-term monitoring while Stanford personnel were off-site. An internet connection was installed by NCCC personnel to allow for remote monitoring of the instrument by Stanford during this time. The instrument functioned well during both periods, and preliminary results show good agreement with gas analyzer readings and temperature indications near the location of the sensor.

The unit has since been removed and all equipment owned by Stanford has been returned. Post-run inspections of the assembly showed no significant fouling of the sapphire windows or of the associated piping, providing verification of future use for similar testing. The NCCC-provided portion of the test apparatus has also been sealed in preparation for the upcoming R10 run. Discussions are underway to initiate a second test that will likely take place in run R11.

6.0 PARTICULATE CONTROL DEVICE

The PCD operated with a high collection efficiency and stable pressure drop during R09 with particulate outlet loading typically less than 0.1 ppmw. Although research efforts continued to focus on the long-term evaluation of material performance of several types of filter elements and failsafe devices, the decision was made not to re-install the PCD plenums with the longest duration filter elements following R08 due to the compressed timeframe of the outage. Testing continued with the spare filter elements installed from the on-site inventory following the PCD tubesheet repair during R08. The spare filter elements consisted of both new and used iron aluminide and HR160 elements from Pall and used sintered-fiber elements with an inert coating from Porvair. The failsafes utilized 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), and PSDF-designed devices constructed of HR-160 material.

After completion of the R09 test, the filter elements installed in the PCD prior to the tubesheet failure that have the most hours of run time were re-installed in order to continue long-term material evaluation. The elements with the greatest exposure time, which are iron aluminide elements, had accumulated over 16,200 hours of syngas and fine ash particulate exposure prior to the tubesheet failure during R08. Most of the failsafes were cleaned and re-installed during the repair including the both Pall Dynalloy devices and PSDF-designed devices. A new set of failsafes will be ordered in early 2013 for installation following the planned repair and/or replacement of the PCD tubesheet and plenum components.

7.0 ASH REMOVAL SYSTEMS

Both the Continuous Coarse Ash Depressurization and Continuous Fine Ash Depressurization systems operated well during R09, maintaining high reliability and consistent ash removal. Following the discharge blockage that occurred in the coarse ash system during R08, work began on a modification to create greater operational flexibility and safety during runs. The development of this project scope is underway and should be implemented during 2013.

8.0 SYNGAS CONDITIONING AND PRE-COMBUSTION CO_2 CAPTURE TECHNOLOGIES

8.1 Water-Gas Shift Catalyst

Testing of the water-gas shift catalyst previously in runs R07 and R08 continued in R09. Parametric testing for the catalyst was completed during R07, using 50 lb/hr of raw syngas at 480°F and 200 psia for 850 hours. Steam was added to extend the range of steam-to-CO ratios. A fresh batch of catalyst was installed prior to R08. Following 292 hours of operation in R08, the catalyst was blanketed with nitrogen and kept ready for retesting in R09. A further 746 hours of testing was achieved in R09, bringing the exposure time for this batch of catalyst to 1,038 hours.

Test data collected over the three runs are presented in Figure 8. The range of data for R07 is wider because of the steam added during testing. For one pass of syngas, over 80 percent CO conversion was achieved with a steam-to-CO ratio of 2. For the intrinsic steam-to-CO ratio of

around 0.9, CO conversion was as much as 70 percent. Further testing of this catalyst is planned for 2013.

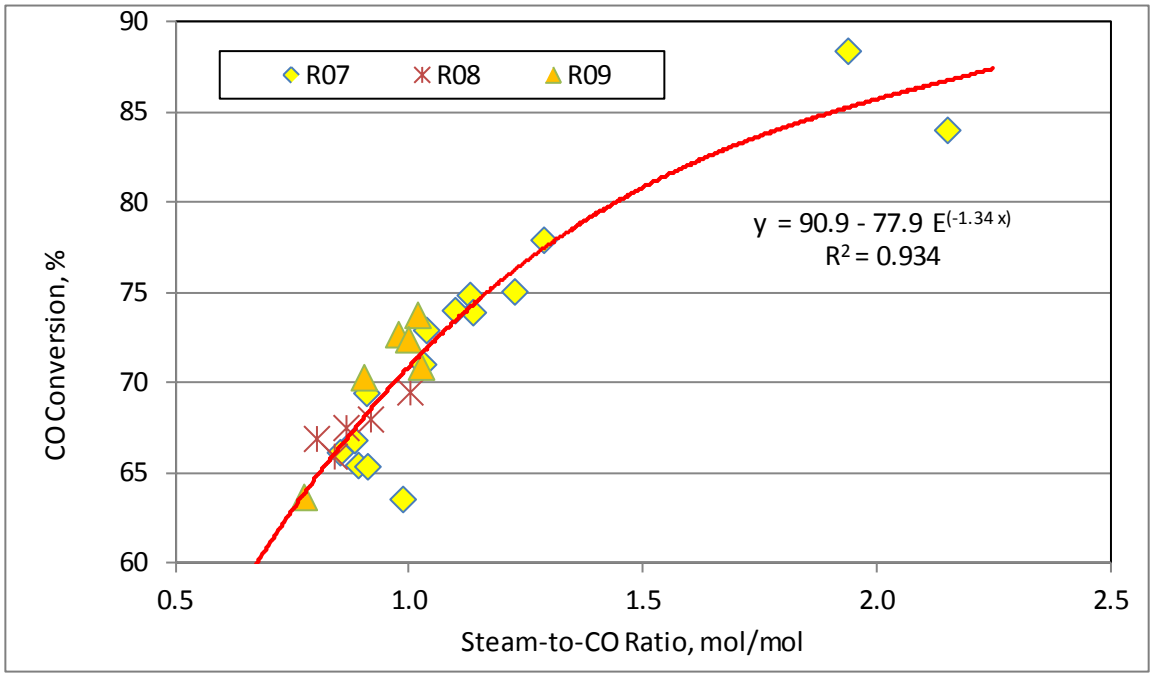

Figure 8. Water-Gas Shift Catalyst Test Results for Runs R07 through R09.

8.2 Worcester Polytechnic Institute Membranes

For R09, Worcester Polytechnic Institute (WPI) tested a ternary alloy membrane coated with the proprietary material. Though the coating was designed to resist sulfur poisoning, testing during R09 was primarily to determine the hydrogen permeance without any sulfur present for comparison to that from a previously tested membrane without the coating. A membrane coupon was also installed inside the membrane module for exposure to the same conditions as the tested membrane. The coupon is currently being analyzed for surface contamination to optimize the material composition and determine appropriate gas pre-treatment requirements.

High product purity and stability of the membrane was demonstrated during the 164 hours of testing. The proprietary coating did not appear to impact hydrogen permeance. Figure 9 plots hydrogen permeance during the R09 test run and shows its stable performance. However, the hydrogen permeance was lower than that measured in the WPI laboratory.

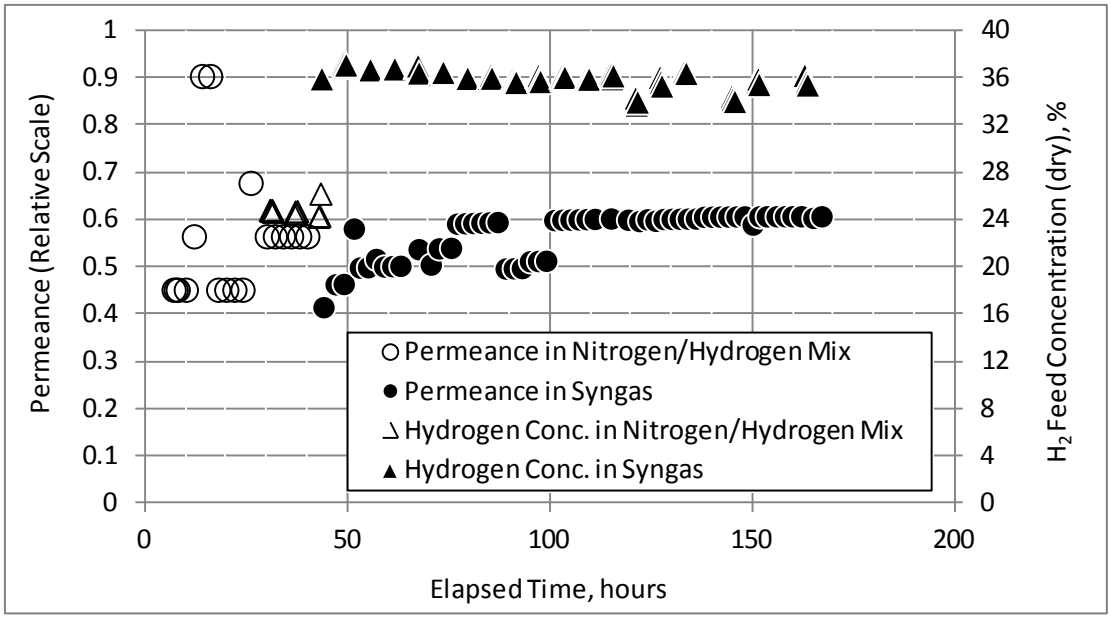

Figure 9. WPI Membrane Permeance for Run R09.

Analysis by X-Ray Photoelectron Spectroscopy (XPS) from a previously tested membrane showed high concentrations of carbon on the surface layer as shown in Figure 10. Elemental carbon concentration at the membrane surface was about 46 percent and gradually decreased as the depth increased. However, even at 1,000 nm depth, the carbon concentration remained at 1 percent. WPI theorized that the presence of unsaturated hydrocarbons in the syngas may have resulted in the carbon deposition on the palladium layer, leading to the loss of active sites for hydrogen permeation. Additional testing and analysis is planned to provide insight of the root causes for the permeance reduction, which has also been observed in previous tests.

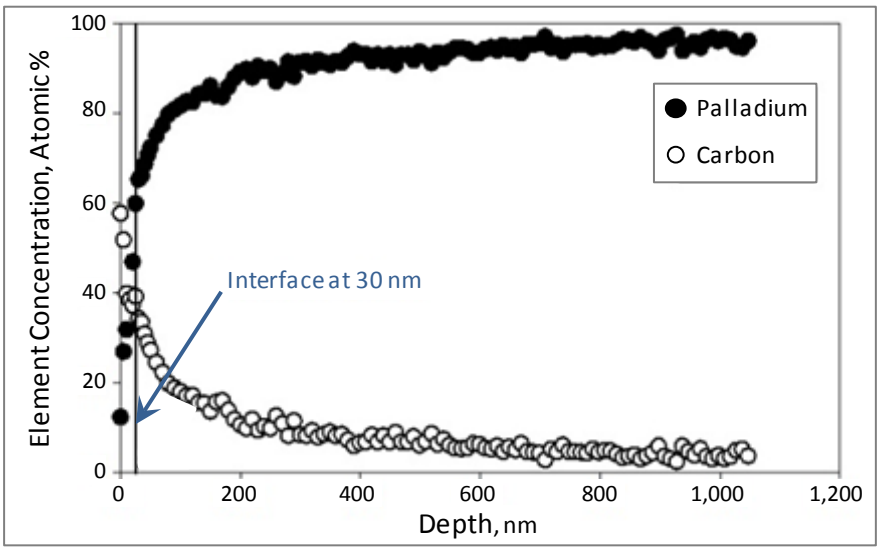

Figure 10. XPS Analysis of WPI Membrane Surface.

8.3 Membrane Technology & Research Membranes

Membrane Technology & Research (MTR) continued evaluation of the Polaris™ CO_2 and Proteus™ hydrogen membranes. MTR scaled up the 50-lb/hr CO_2 membrane module to an integrated system designed for 500 lb/hr syngas (see Figure 11), and the new system was tested for the first time in R09.

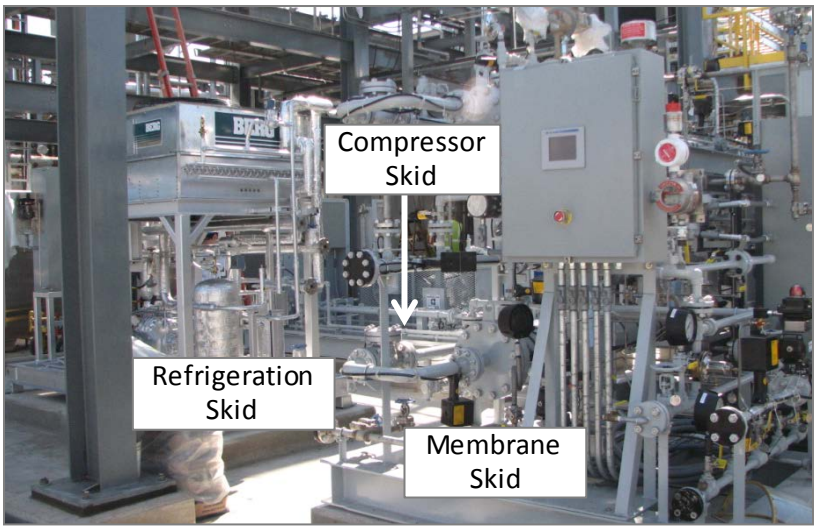

Figure 11. Installed MTR 500 lb/hr CO_2 Membrane System.

During R09, syngas treated in the newly installed syngas cleaning system was fed to the MTR system. The NCCC provided gas composition measurements from four gas chromatographs for continuous data from feed, permeate, and reject streams of the two membranes, as well as measurements from a non-dispersive infrared instrument to measure CO_2 concentration in the liquid CO_2 stream.

The R09 testing accomplished two major milestones. First, the complete system operated successfully as designed on syngas. Second, the process yielded liquid CO_2 with up to 92 percent purity. MTR plans to continue testing of the 500 lb/hr system in 2013. Prior to further testing, several modifications will be incorporated to improve system performance, including a new membrane design, improved CO_2 liquid level control, and reprogramming of the Programmable Logic Controller.

MTR also continued testing the smaller CO_2 membrane module and the hydrogen membrane stamp cell during R09 and completed 186 hours of operation. Testing of the scaled-up hydrogen membrane module at 10 lb/hr was previously completed, and future plans are to scale this H_2 membrane module test to 50 lb/hr. In the meantime, MTR continued materials evaluation with the stamp cell. Results are shown in Figure 12 for the testing of the CO_2 and hydrogen membranes completed in R09.

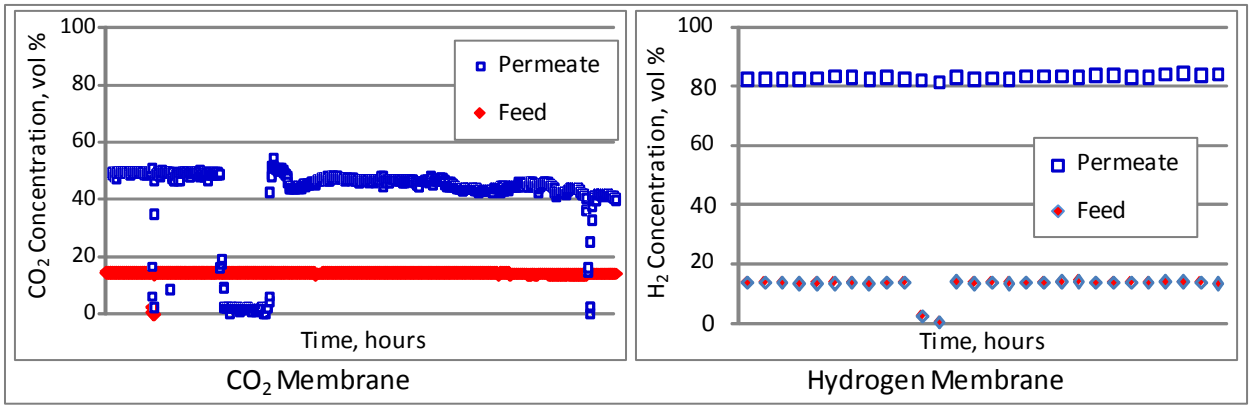

Figure 12. Test Results for MTR 50 lb/hr CO_2 Membrane Module and Hydrogen Membrane Stamps.

The CO_2 membrane was tested in two configurations—as a single membrane module and with two modules connected in series. R09 testing demonstrated CO_2 enrichment from about 15 vol% in the feed to 50 vol% in the permeate stream. The hydrogen membrane stamp cell enriched the hydrogen from a feed concentration of 14 vol% to 80 vol% in the permeate. Both CO_2 and hydrogen membranes performed stably, achieving separation performances equal to or slightly above what had been achieved historically.

8.4 Media & Process Technology Membrane

Media and Process Technology's (MPT's) Carbon Molecular Sieve (CMS) membrane bundle operated for almost 400 hours during R09, including a 300-hour period of continuous operation under NCCC supervision, processing 50 lb/hr of raw flue gas at 190 psia and 500°F. Table 3 provides gas composition data on a dry basis for the two egress streams. For short-duration tests, the syngas hydrogen content was increased to 40 vol% (simulating the syngas produced by a commercial oxygen-blown gasifier), resulting in hydrogen permeate purity of about 95 percent (dry basis). This degree of product enrichment is greater than that achieved for the single tube and pilot-bundle membranes and indicates the improvements made in the quality of the deposited CMS layer.

Table 3. Composition Data for Egress Streams from MPT Membrane during R09.

Syngas Constituent	Permeate Concentration	Reject Concentration
Hydrogen, vol%	95	35
CO_2, vol%	1.3	6
CO, vol%	< 0.3	6
H_2S, ppmv	Below LDL	25
Nitrogen (balance), vol%	3.4	53

High permeate moisture levels limited the accuracy of hydrogen permeance determinations, but helium and nitrogen pure-component permeance measurements were made periodically to give an indication of membrane performance stability. The permeance for helium was about 1.1 $m^3/m^2/hr/bar$ and that for nitrogen was about 0.01 $m^3/m^2/hr/bar$; these values were stable throughout the 300-hour continuous test period and correspond to values obtained from off-line characterization tests for the membrane bundle.

8.5 Eltron Hydrogen Membrane

Under DOE sponsorship, Eltron Research & Development, a technology group specializing in chemical and engineering technologies, is developing a high-temperature hydrogen transport membrane (HTM) for pre-combustion capture of CO_2. The Eltron HTM technology uses a multi-layer metal alloy tube to separate hydrogen from coal-derived syngas (see Figure 13), providing high purity hydrogen and capture of high pressure CO_2 suitable for utilization or sequestration. Eltron's membranes can withstand high differential pressures of up to 1,000 psi, allowing for successful integration with advanced, high-pressure coal gasification processes.

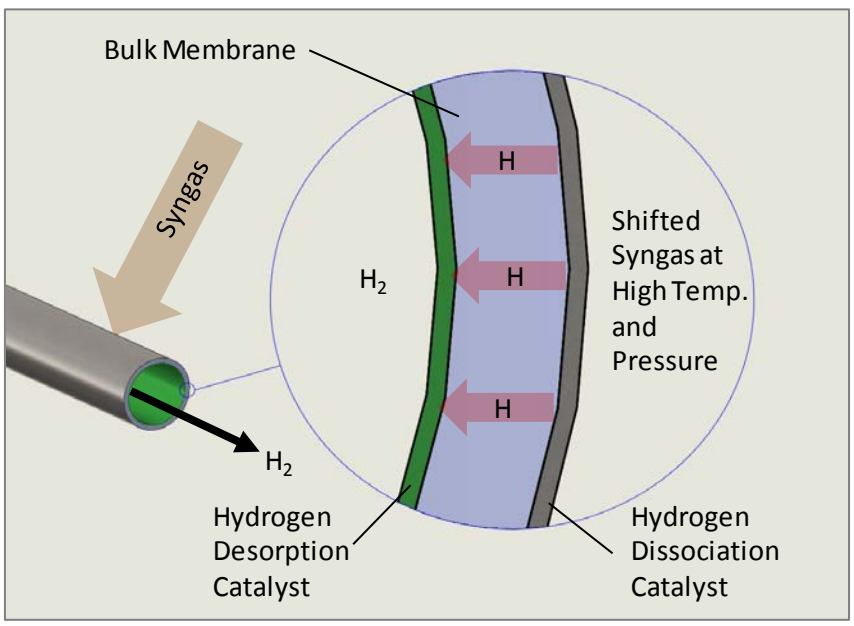

Figure 13. Schematic of Eltron Hydrogen Membrane Tube.

At the direction of DOE, NCCC began discussions with Eltron in early October about testing the HTM at NCCC in run R09. To accommodate the testing within the tight schedule, WPI's existing membrane testing oven was used. A new module and flange connections for the permeate and retentate gas streams were fabricated on-site according to the American Society of Mechanical Engineers' standards. The two membrane tubes tested were of 0.5-inch outer diameter, with one tube 8 inches long and the other, 6 inches long. The 8 inch long tube was installed for permeation testing, and the 6 inch long tube was installed for exposure-only testing. Figure 14 shows these two membrane tubes.

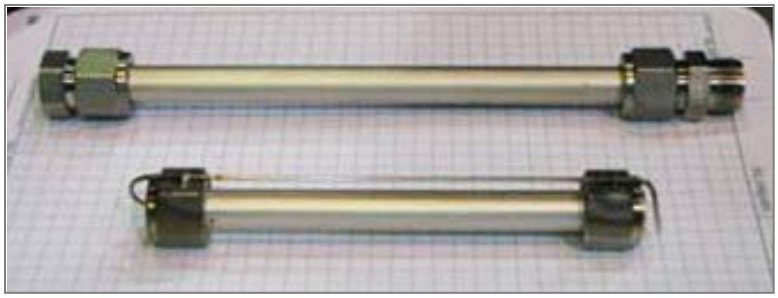

Figure 14. Eltron Membrane Tubes.

The assembled module was installed inside WPI's oven. A new thermocouple was installed to directly measure syngas temperature from the retentate stream in addition to an oven temperature indicator. The oven heating program was modified to accommodate Eltron's proprietary startup process, and testing began on December 1.

The membranes were tested at about 640°F for a total of 204 hours, including 183 hours of syngas operation with hydrogen enrichment and 21 hours with bottled hydrogen/nitrogen gases. The system operated with 10 lb/hr shifted and sweetened syngas (less than 1 ppmv sulfur). With hydrogen enrichment, the hydrogen concentration of the syngas reached about 38 to 40 vol% (dry basis). During the testing period, the membrane consistently demonstrated a hydrogen flux of about 20 scfh/ft^2, as shown in Figure 15. The step increase in flux at around 50 hours was the result of replacing a rotameter with a higher resolution one. Hydrogen purity in the permeate stream was greater than 99.87 percent during the entire testing period, which indicated excellent membrane integrity without leakage.

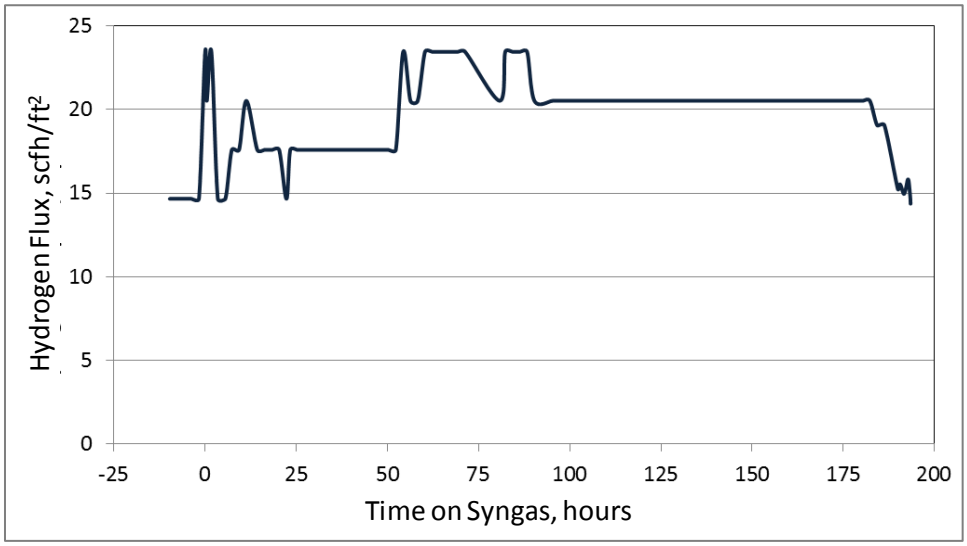

Figure 15. Eltron Membrane Hydrogen Flux during R09 Testing.

Eltron plans to conduct post-operation analysis of the two membranes, including laboratory permeation measurements at conditions similar to those of NCCC testing and surface impurities analysis using various techniques available in-house such as X-ray diffraction, X-ray

fluorescence, and scanning electron microscope/microprobe. For higher sensitivity analysis to detect low levels of impurities, samples from each of these tubes will be sent to an independent lab with instrumentation and expertise appropriate for surface analysis such as Auger electron spectroscopy or X-ray photoelectron spectroscopy. Depth profiling will be conducted as well.

8.6 Membrane Coupon Testing

Material testing efforts continued during R09 with a series of test coupons of metal alloys being investigated for hydrogen separation membranes. The coupons were placed in the PCD downstream of the filter elements where they were exposed to particulate free raw syngas in order to assess their resistance to degradation from syngas stream impurities, such as hydrogen sulfide. Preliminary analysis has shown that the varied nature of syngas impurities produced significant differences compared to controlled laboratory conditions. Coupon suppliers included both the National Energy Technology Laboratory and United Technologies Research Center.

8.7 CO_2 Capture Solvent Testing in Batch Reactor

The Bara Research group of the University of Alabama (UA) has been researching the use of alkylimidazoles, a group of low volatility and low viscosity liquids, as physical solvents for economical CO_2 separation from processes such as gasification. UA provided an alkylimidazole solvent for testing in the SCU stirred batch reactor during R09 using bottled gases and syngas. Results indicated that the solvent performed as expected in regard to the solvent's capacity for CO_2. All tests were performed at 70°F. CO_2 capture was evaluated using bottled gases with 35 and 40 vol% CO_2 at 150 and 300 psig. CO_2 capacity of the UA solvent as a function of partial pressure is compared with other physical solvents in Figure 16.

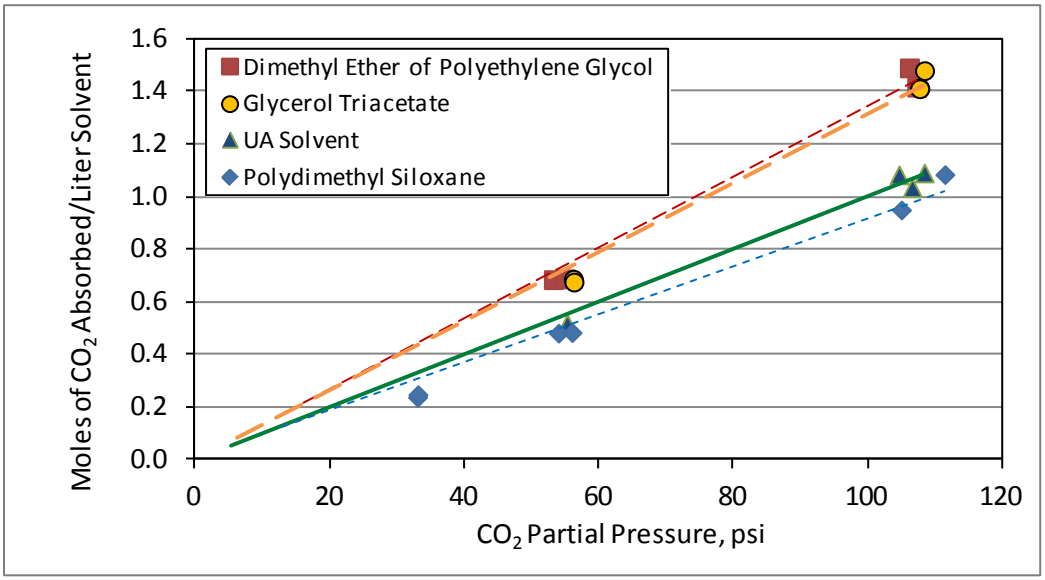

Figure 16. CO_2 Capacity as a Function of CO_2 Partial Pressure for UA Solvent.

Absorption of H_2S from the syngas is indicated by Figure 17. This graph shows the reactor outlet concentration of H_2S in the syngas and indicates that with fresh solvent essentially all of the H_2S was collected for over an hour.

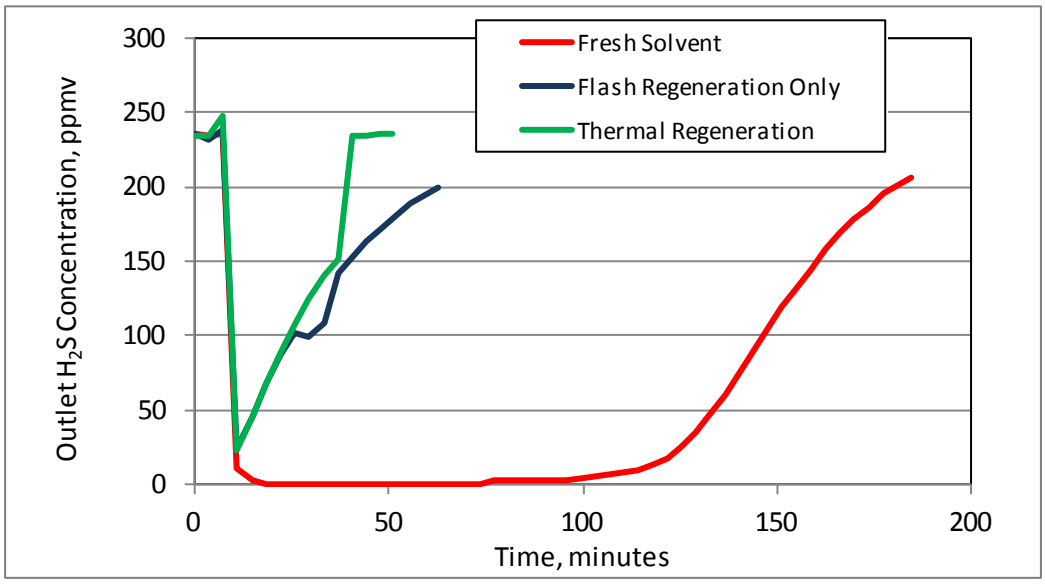

Figure 17. H_2S Absorption for University of Alabama Solvent.

The solvent was saturated with CO_2 after about 20 minutes, but this did not affect the H_2S capacity. Also shown in Figure 17 are data from subsequent H_2S absorption tests after regeneration by various techniques. Flash regeneration released all of the CO_2 from the sample but was not very effective at removing H_2S from the solvent (restoring less than 10 percent of the original capacity). Flash regeneration followed by thermally soaking the solvent at 212°F while purging with nitrogen did not appear to remove any additional H_2S. This behavior seems to indicate that it acts as a physical solvent for CO_2 but reacts chemically with H_2S irreversibly. This interaction will be studied more closely in future testing. For the next test campaign, planned for 2013, a blend of the base and a new solvent will be evaluated for improvements, and water will be added to the solvent to observe the effect on the H_2S interaction.