THE NATIONAL CARBON CAPTURE CENTER
AT THE POWER SYSTEMS DEVELOPMENT FACILITY

TEST RUN SUMMARY REPORT

TEST RUN R06
MAY 18, 2011 – AUGUST 21, 2011

DOE Cooperative Agreement

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LIST OF ABBREVIATIONS

CCAD—Continuous Coarse Ash Depressurization
CFAD—Continuous Fine Ash Depressurization
DEPG—Dimethyl Ethers of Polyethylene Glycol
DOE—Department of Energy
EDX—Energy Dispersive X-Ray
FTIR—Fourier Transform Infrared
GTA—Glycerol Triacetate
NCCC—National Carbon Capture Center
MMD—Mass Median Diameter
MTR—Membrane Technology & Research
PCD—Particulate Control Device
PDAC—Pressure Decoupled Advanced Coal
PDMS—Poly Dimethyl Siloxane
PRB—Powder River Basin
PSDF—Power Systems Development Facility
R01 through R06—Tests Runs 1 through 6
SCS—Southern Company Services
SCU—Syngas Conditioning Unit
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 (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.

The facility conducted test run R06 beginning on May 18, 2011, but the run was interrupted by the failure of a nitrogen tank in the on-site vendor-operated nitrogen plant. Operation resumed on July 7, and the gasification process ran continuously until August 21, 2011. The total run duration was 1,207 hours. Improvements to coal feeder operation and gasifier temperature control were achieved during the run, and a variety of pre-combustion CO_2 capture-related technologies were completed.

2.0 COAL PREPARATION

The fuel for R06 was Powder River Basin (PRB) sub-bituminous coal, which was processed in the mill system to achieve the desired moisture concentration and particle size distribution. A total of 2,700 tons of PRB coal were processed. Table 1 lists the average coal properties as-received and as-fed (after processing in the coal mill).

Table 1. PRB Coal Properties.

Coal Property	Average Value
As-Received Carbon, wt %	52.5
As-Received Hydrogen, wt %	3.5
As-Received Nitrogen, wt %	0.4
As-Received Sulfur, wt %	0.3
As-Received Ash, wt %	6.1
As-Received Oxygen, wt % (by difference)	12.6
As-Received Volatiles, wt %	32.0
As-Received Fixed Carbon, wt %	37.2
As-Received Heating Value, Btu/lb	8,955
As-Received Moisture Concentration, wt%	25
As-Fed Moisture Concentration, wt%	17
Moisture Content Reduction, %	32
As-Fed Mass Median Diameter, micron	414
As-Fed Oversize (>1,180 micron) Content, wt%	14
As-Fed Fine (< 45 micron) Content, wt%	10

3.0 COAL FEED

Both the developmental Pressure Decoupled Advanced Coal (PDAC) feeder and the original coal feeder operated during R06 without any major coal stoppages.

3.1 PDAC FEEDER

The PDAC feeder operated for 942 hours, including independent operations for 12 hours. The feeder operated at coal feed rates ranging from 460 to 4,800 lb/hr. Overall feeder performance during the run was stable over the range of coal feed rates tested.

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 coal feed rate to the gasifier, this improvement in nitrogen flow control also resulted in decreased coal feed rate variability. The decreased variability in nitrogen flow is illustrated by Figure 1.

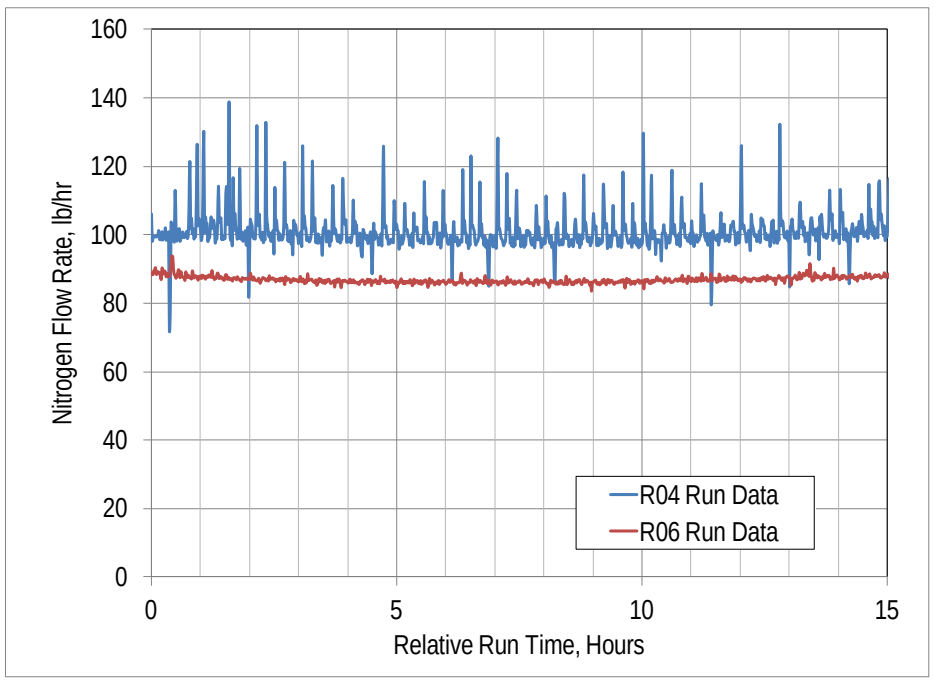

Figure 1. PDAC Feeder Nitrogen Flow Rate during R04 and R06 Operation.

Periodic coal feed stoppages were experienced due to the accumulation of large coal particles in the bottom of the feeder and the oversize material trap in the discharge line, which is shown in Figure 2. In order to reduce the accumulation of larger coal particles and the frequency of coal stoppages, the oversize material trap was bypassed, and an insert was installed in the bottom of the feeder to reduce coal particle segregation. While these steps did reduce the frequency of coal stoppages, additional modifications will be considered following R06 to further improve feeder performance.

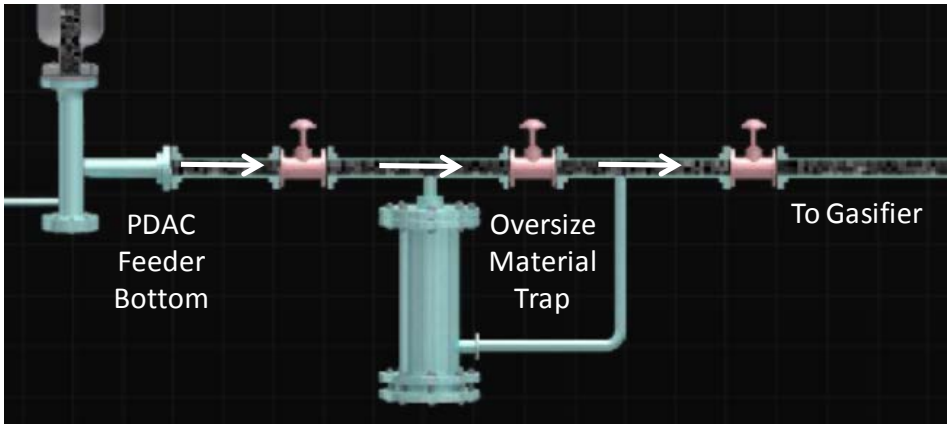

Figure 2. PDAC Feed Line.

Improvements to the control logic continued during R06 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. The new addition to controller was successfully tested during the run and served to maintain a more constant feed rate to the gasifier.

Vibration level probes provided by Dynatrol and installed in both the feeder lock vessel and dispense vessel continued to perform well and have now achieved more than 3,700 hours of consistent solid level indications. The point sensitive level probe provided by Drexelbrook and installed in the feeder dispense vessel has continued to facilitate steady solids level control, mitigating the impact of solids level variation on coal feed rate. More than 2,500 hours of operation have now been achieved utilizing this probe for dispense vessel level control.

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. The meter will be returned prior to the R07 run, and a new installation procedure will be implemented to prevent future damage to the meter.

3.2 ORIGINAL COAL FEEDER

The original rotary coal feeder operated for 1,044 hours at feed rates varying from 1,200 to 3,500 lb/hr. Overall feeder performance during the run was stable over the range of coal feed rates tested.

4.0 TRANSPORT GASIFIER

R06 operation consisted of 54 steady state operating periods, all with recycle syngas utilized for gasifier fluidization. Gasifier operation during the run yielded high carbon conversions, as

shown in Table 2. The historical data for carbon conversion represents data collected since completion of the 2006 gasifier modifications.

Table 2. Steady State Carbon Conversion for R06 and Previous PRB Test Runs.

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

Figure 3 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.

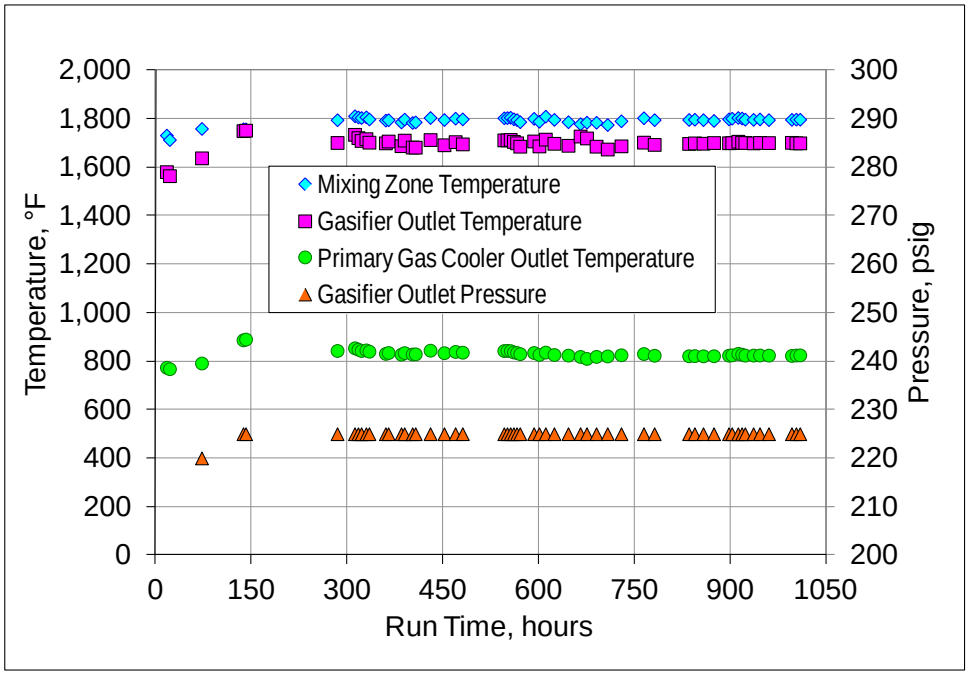

Figure 3. Gasifier Operating Conditions.

Gasifier temperature control enhancements utilizing upper and lower mixing zone air flow adjustments continued during R06. Since gasifier temperatures respond rapidly to changes in air feed rate, further tuning of the individual and total air flow controllers to prevent temperature overshoot was required. This control strategy worked well while maintaining steady state operation but did not respond as well to sudden operational changes such as variation in coal feed rate. As a result, a more aggressive controller was designed and implemented to compensate for these changes by increasing the rate of change of the total air flow rate set point. The improvement observed from implementation of this new control feature is illustrated in Figure 4.

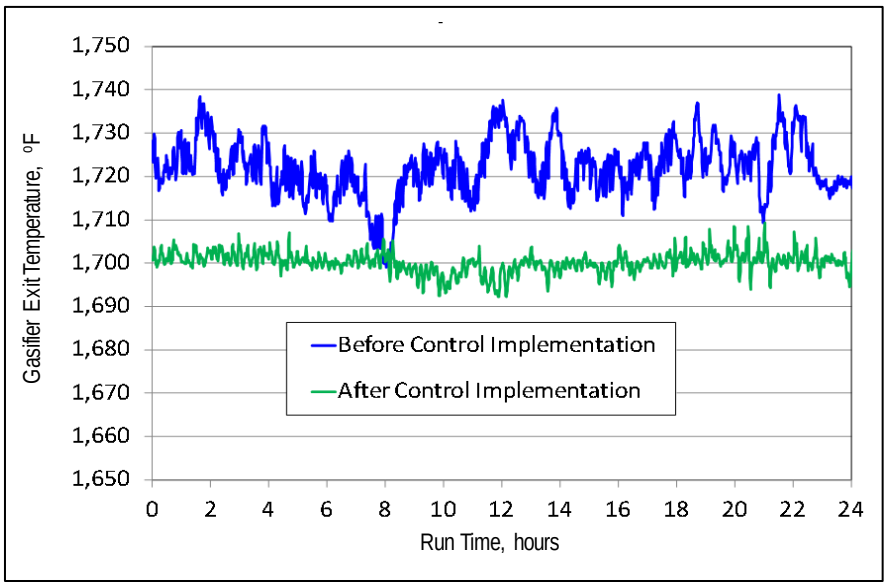

Figure 4. Gasifier Temperature Control during Non-Steady State Operation.

Gasifier sensor development continued during R06 with the long-term evaluation of a sapphire thermowell installed in the gasifier riser. The thermowell has now been exposed to over 2,700 hours of gasification operation. While the sapphire thermowell and thermocouple assembly has continued to show good responsiveness during gasifier temperature changes, the temperature indication continued drift relative to nearby thermowell/thermocouple assemblies (see Figure 5).

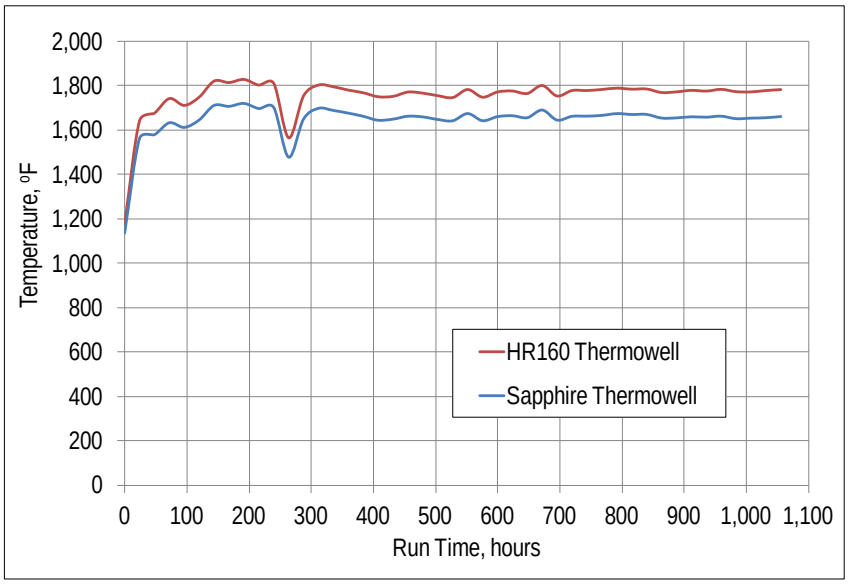

Figure 5. Comparison of Temperature Readings for Sapphire and HR-160 Thermowells.

Analysis of possible causes of the temperature drift, such as inspection of the temperature transmitter, were performed during the outage prior to the R06 run but did not reveal any obvious problems. As a result, the sapphire thermowell will be sent back to the manufacturer for further analysis to determine a root cause for the drift in temperature indication. Post-run inspections of the thermowell revealed no signs of erosion or ash deposition.

5.0 PARTICULATE CONTROL DEVICE

The particulate control device (PCD) operated with high collection efficiency and stable pressure drop. PCD research continued to focus on material testing. Several types filter elements and failsafe devices were tested during R06. 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 manufactured by Porvair, iron aluminide and HR-160 elements from Pall, and sintered powder filter elements with a high alloy material from Mott. In addition, several filter media materials and a few catalyst materials provided by various third parties were installed downstream of the filter elements to assess material performance in the presence of syngas at commercially relevant operating conditions. These materials were removed for inspection following R06. The filter elements and failsafe devices will be removed from the PCD following R07 testing. The Plastite protective coating on the tubesheet will also be inspected at that time.

6.0 ASH REMOVAL SYSTEMS

Both the Continuous Coarse Ash Depressurization (CCAD) and Continuous Fine Ash Depressurization (CFAD) systems operated well during R06. The change in piping configuration to eliminate intermediate transport equipment for the CFAD system, installed prior to R03, continued to maintain the high reliability that has been historically demonstrated by the system. Inspection of the discharge piping and associated off-centered elbows showed some evidence of line erosion due to high conveying velocity during the run. The identified areas of concern will continue to be monitored, and additional design changes may be proposed in the future. A similar change in piping configuration is planned for the CCAD system during 2012 to eliminate additional transport equipment.

7.0 SYNGAS CONDITIONING UNIT

7.1 BATCH REACTOR CO_2 CAPTURE

Batch reactor testing using bottled gases had determined the CO_2 capture characteristics of the physical solvent glycerol triacetate (GTA). During R06, testing was completed to confirm these CO_2 capture results with syngas and to evaluate the absorption and regeneration characteristics of H_2S.

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. Repeat regeneration tests at 212°F are planned for R07 to determine if cycling affects the CO_2 and H_2S absorption capacity of the GTA.

Figure 6 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 dimethyl ethers of polyethylene glycol (DEPG), and both were superior to capture with poly dimethyl siloxane (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.

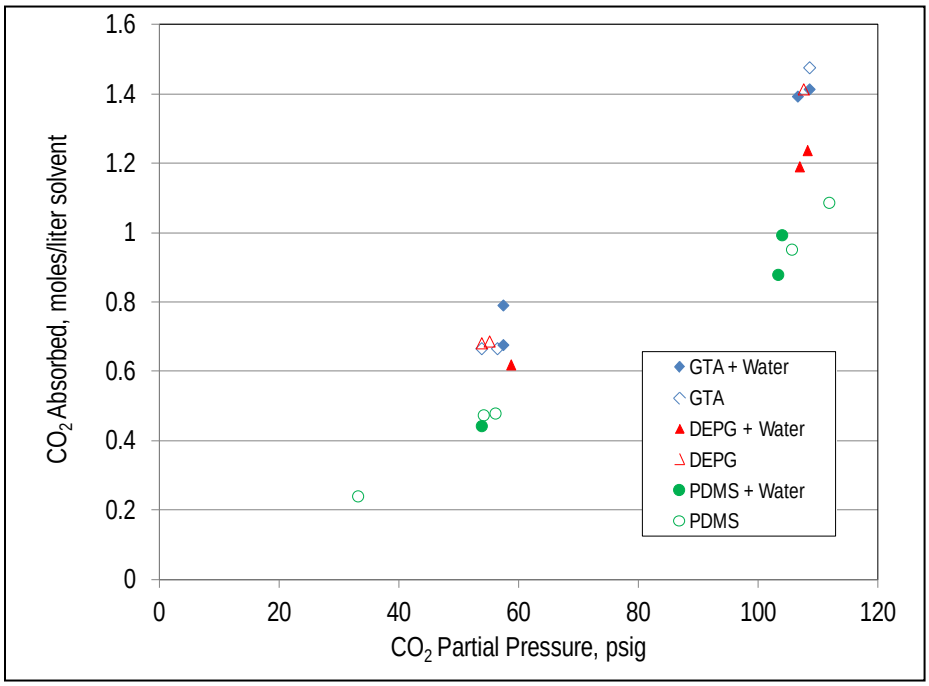

Figure 6. Absorption of CO_2 with and without Water Addition for Three Solvents.

7.2 WATER-GAS SHIFT CATALYST

To improve the data quality from water-gas shift (WGS) testing, several minor modifications were incorporated prior to R06. The on-line reactor syngas inlet moisture analyzers had not provided reliable data in past runs when compared to the moisture contents determined by condensation from the in-situ sampling performed at the outlet of the PCD. To determine the reactor syngas outlet moisture content, a cooling coil was installed to condense water at a known temperature, which was then collected and weighed. The dry syngas volumetric flow rate was measured by a flow meter, and the two measurements were used to determine the water content. To further reduce heat losses from the reactors and improve the heat balance closures, a modified

control strategy for the heating jackets was developed. Some heat loss is tolerated to avoid operating the jackets at too high a load and adding heat to the reactor.

For R06, a previously untested catalyst was installed in the WGS reactor. Figure 7 presents preliminary conversion data for the new catalyst. Variations in the syngas inlet temperature and flow rate had no significant effect on the conversion. The modified moisture measurement procedures were successful in improving heat and mass balance closures for the data, increasing confidence in the results. A new catalyst from a commercial developer will be tested in R07.

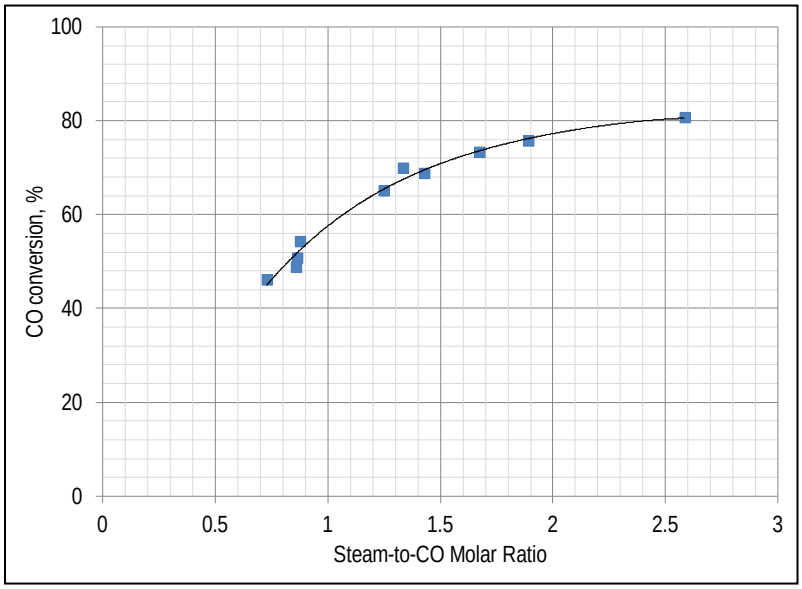

Figure 7. Preliminary WGS Catalyst CO Conversion Data.

7.3 WORCESTER POLYTECHNIC INSTITUTE MEMBRANES

The testing in R06 was the first time the Worcester Polytechnic Institute (WPI) membranes operated on coal-derived syngas. Figure 8 presents a view of the palladium-based membrane module inside its oven. To provide the driving force for hydrogen permeation, bottled gas is used to enrich the syngas hydrogen concentration from less than 10 vol% to 33 vol%. To minimize corrosion of the palladium, syngas sulfur content is reduced to below 1 ppmv by a combination of hydrolysis to convert the carbonyl sulfide to hydrogen sulfide, and the absorption of nearly all hydrogen sulfide by zinc oxide. The syngas is heated to operating temperature (about 840°F) in the coil prior to entering the housing containing the membrane. The unit is started up using a nitrogen/hydrogen flow.

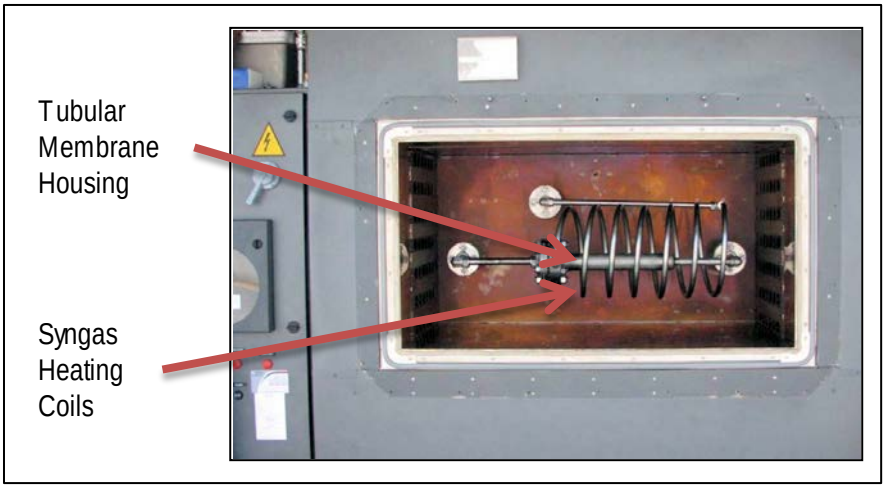

Figure 8. WPI Membrane Installed in Oven.

Three different palladium-based hydrogen membranes were tested for a total exposure time of 550 hours in hydrogen-enriched syngas and 140 hours in a nitrogen/hydrogen mixture. The first membrane consisted only of palladium and operated for over 250 hours, 165 hours on hydrogen-enriched syngas and 86 hours on a nitrogen/hydrogen mixture. The permeate stream was close to pure hydrogen, and initially the targeted 2 lb/day hydrogen production rate was achieved. The flux decreased but stabilized during the test period. The variation of permeate hydrogen content with time for this run is presented in Figure 9.

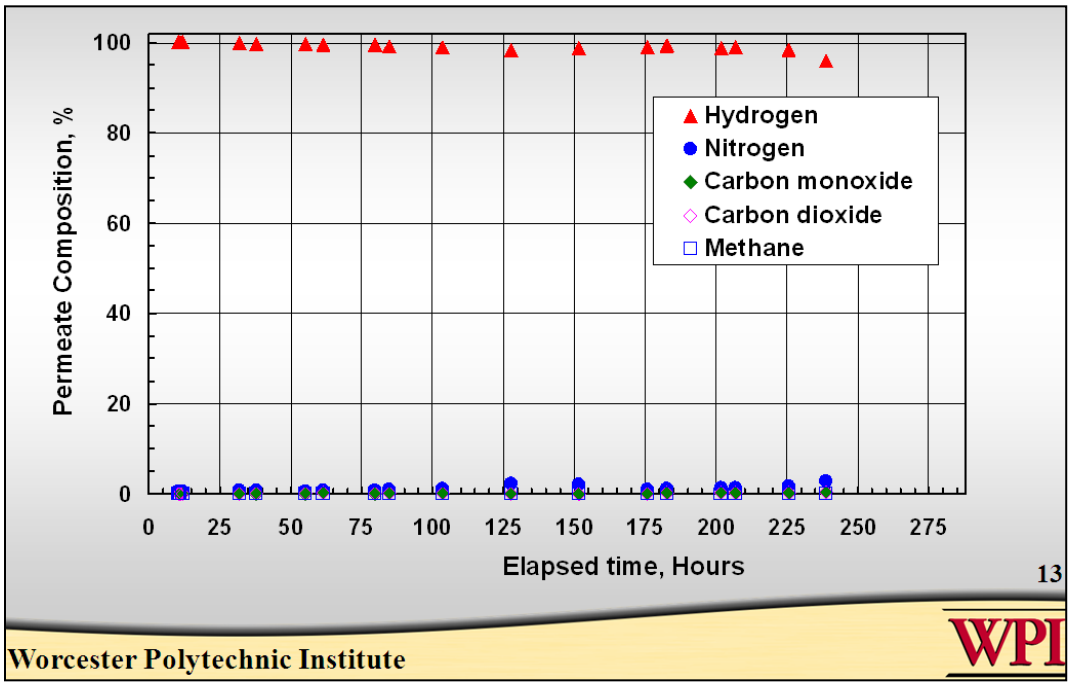

Figure 9. WPI Membrane Permeate Composition.

The other two membranes were palladium coated with different thicknesses of gold to inhibit sulfur attack from any residual sulfur present in the syngas. Figure 10 presents a view of a plain membrane and a gold coated one. All three membranes produced near pure hydrogen but all suffered from a reduction in flux. The first two developed leaks at the weld joining the porous tube to the solid tube and they were replaced. The third membrane did not leak.

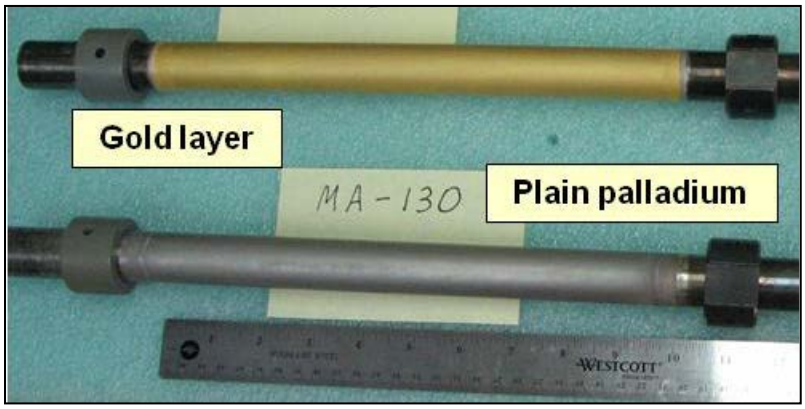

Figure 10. Gold-Coated and Uncoated Palladium Membranes.

After exposure, the membrane surfaces were black, which could be coke produced by the Boudouard reaction resulting in the disproportionation of carbon monoxide into carbon dioxide and graphite, as follows:

$$2CO = C + CO_2$$

To avoid this reaction in R07, a WGS reactor will be installed to minimize the CO concentration in the syngas upstream of the membrane. Because of the higher syngas temperature leaving the WGS reactor, a higher temperature zinc oxide sulfur sorbent will need to be used. In support of its fundamental research program, WPI will install a membrane coupon inside the membrane module. In this way, both components will see the same syngas conditions, allowing for more accurate comparisons of the materials. In R07, WPI will continue to test both pure palladium membranes and ones with different thickness of gold coatings.

7.4 MEMBRANE TECHNOLOGY & RESEARCH MEMBRANES

Previous testing of the Membrane Technology & Research (MTR) polymeric hydrogen membrane was with a small flat sheet of material operating at a flow rate of 1 lb/hr of hydrogen-enriched syngas. Using the test data, materials were selected for the manufacture of spiral-wound membranes operating at a flow rate of 10 lb/hr of hydrogen-enriched syngas. The design is similar to that of the CO_2 membrane and representative of the proposed commercial design, thus the performance results obtained will better support scale-up to demonstration-sized modules. Figure 11 presents an end view of the membrane showing the membrane layers.

Figure 11. End View of MTR Spiral-Wound Hydrogen Membrane.

Figure 12 presents a view of the MTR membrane module in its oven. The syngas was heated to the operating temperature of 280°F in three coils in series prior to entering the housing containing the membrane. The first two membranes tested performed stably but developed leaks. They were returned to MTR for analysis, which resulted in modifications for the third membrane. Analysis suggested that pressure pulses from a malfunctioning flow control valve could be contributing to the mechanical damage, and the valve was replaced. The third membrane was successfully tested, and it concentrated hydrogen from about 12 vol% in the feed stream to about 65 vol% in the permeate stream.

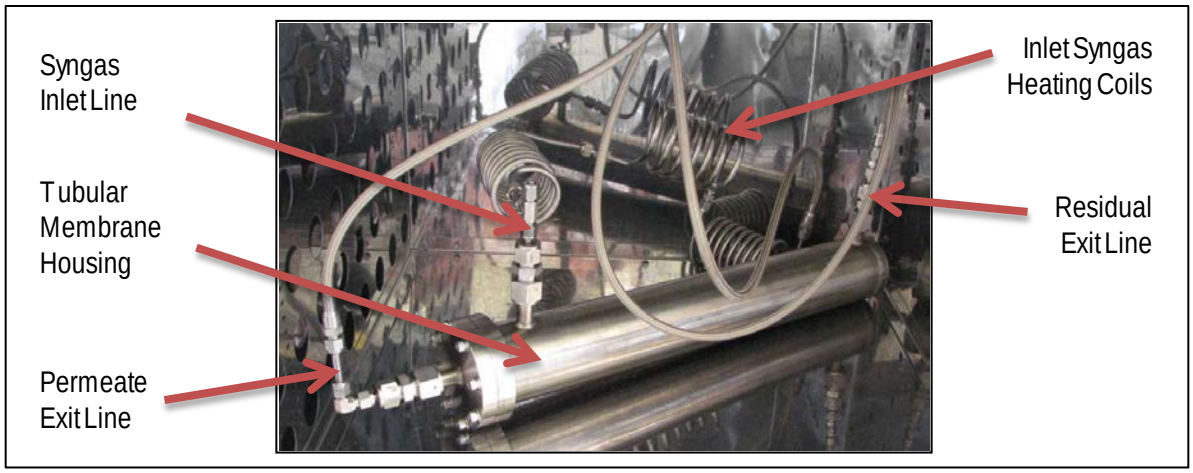

Figure 12. MRT Membrane Installed in Oven.

Three CO_2 membranes were tested for a combined total of 660 hours They performed stably for the majority of the time and concentrated the CO_2 from about 12 to 15 vol% in feed to over 40% in the permeate. This result agreed with MTR's membrane design expectation.

During R06, the syngas hydrocarbon removal system was commissioned and operated to protect the MTR membranes from contamination. The syngas is maintained above 400°F to avoid

condensing the high-boiling point hydrocarbons in the inlet pipe work to the vessel. The syngas was then bubbled through water chilled to 55°F to condense as much hydrocarbon as possible. A filter in the chiller circuit removed much of the condensed material and the remainder was drained off with water condensed from the syngas. Once commissioned and operating stably, this new process removed over 90 percent of the naphthalene and almost all other poly-aromatic hydrocarbons. Much of the benzene present remained in the gas phase, but this passed through the membrane without causing contamination. For R07, an activated carbon filter will be installed upstream of the membrane to remove residual naphthalene and reduce the syngas benzene content.

7.5 JOHNSON MATTHEY MERCURY SORBENT

Johnson Matthey tested its 2.5% palladium-based sorbent for mercury and trace metals removal for close to 1,000 hours during R06. Previous testing has been with sorbents containing 5% palladium. To determine the mercury collection efficiency, samples of syngas upstream and downstream of the reactor were taken at intervals during the run using a modified EPA Method 29. These were sent to an external laboratory for analysis, and results will be forthcoming when available. The sorbent was removed in layers to determine the penetration of mercury and other heavy metals. These samples were sent to Johnson Matthey for analysis.