THE NATIONAL CARBON CAPTURE CENTER
AT THE POWER SYSTEMS DEVELOPMENT FACILITY

TEST RUN SUMMARY REPORT

TEST RUN R04
APRIL 1, 2010 –APRIL 28, 2010

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LIST OF ABBREVIATIONS

CCAD—Continuous Coarse Ash Depressurization
CFAD— Continuous Fine Ash Depressurization
DOE—Department of Energy
EDX—Energy Dispersive X-Ray
FTIR—Fourier Transform Infrared
IC—Ion Chromatography
ICP/MS—Inductively Coupled Plasma/Mass Spectrometry
NCCC—National Carbon Capture Center
NFPA—National Fire and Protection Association
NMR—Nuclear Magnetic Resonance
MMD—Mass Median Diameter
MPT—Media & Process Technology
MTR—Membrane Technology & Research
PCD—Particulate Control Device
PDAC—Pressure Decoupled Advanced Coal
PRB—Powder River Basin
PSDF—Power Systems Development Facility
R01 through R05—Tests Runs 1 through 5
SCS—Southern Company Services
SCU—Syngas Conditioning Unit
WGS—Water-Gas Shift

R04 SUMMARY REPORT

The DOE-sponsored National Carbon Capture Center (NCCC) at the Power Systems Development Facility (PSDF) has continued to support the advancement of clean coal technologies through testing under realistic industrial conditions. In April 2010, the PSDF operated its gasification process in test run R04. The major objectives of R04 were testing of co-gasification of biomass and coal and testing of syngas conditioning technologies.

1.0 EXECUTIVE SUMMARY

Gasification test run R04 was planned as a nominally 500-hour test of gasifier operation with high moisture Mississippi lignite and with biomass fuel. Coal feed commenced from April 1 to April 28, 2010, for 509 hours of gasification operation, which included about 400 hours of coal only, 106 hours of coal/biomass co-feeding, and 3 hours of biomass only. Operation with biomass co-feed commenced without significant feeder- or gasifier-related issues and yielded carbon conversions typically 99 percent or greater.

The major test objectives of R04 included:

- CO_2 capture testing in the Syngas Conditioning Unit (SCU) batch reactor using two solvents: the amino acid proline and potassium carbonate
- Water-gas shift testing using catalytic filter elements and a fixed bed reactor in the SCU with varying parameters of H_2O-to-CO ratios, temperatures, and face velocity
- Continued optimizing of the developmental Pressure Decoupled Advanced Coal (PDAC) feeder
- Continuing long-term material assessment of hot gas filter elements
- Testing of a new type of thermowell in the gasifier
- Providing a syngas test site for operation of gas membranes from Media & Process Technology (MPT) and Membrane Technology Research (MTR)

Operation of the gasification process during R04 allowed successful completion of all test objectives. The following sections highlight operation of the major equipment.

2.0 COAL AND BIOMASS PREPARATION

The coal used for R04 operation was high moisture lignite from the Red Hills mine in Ackerman, Mississippi. Prior to milling, the lignite moisture concentration was reduced by processing the coal in the PSDF fluid bed drying system. Table 1 lists the average coal properties as-received and as-fed (after processing in the dryer and coal mill).

Table 1. Mississippi Lignite Coal Properties.

As-Received Carbon, wt %	33
As-Received Hydrogen, wt %	2.2
As-Received Nitrogen, wt %	0.6
As-Received Sulfur, wt %	0.4
As-Received Ash, wt %	11
As-Received Oxygen, wt % (by difference)	9.8
As-Received Volatiles, wt %	24.8
As-Received Fixed Carbon, wt %	21.0
As-Received Heating Value, Btu/lb	5,611
As-Received Moisture Concentration, wt%	43
As-Fed Moisture Concentration, wt%	16
Moisture Content Reduction, %	63
As-Fed Mass Median Diameter, micron	325
As-Fed Oversize (>1,180 micron) Content, wt%	13
As-Fed Fine (< 45 micron) Content, wt%	19

Fluid bed drying system operations were stable during the run, and a total of 2,130 tons of lignite were processed, bringing the total tons of lignite processed in the dryer to 7,910 since the system was commissioned in early 2008. The dryer coal feed rate was varied from 5,100 lb/hr to 11,500 lb/hr. Figure 1 illustrates the consistency of coal moisture concentration at the dryer outlet for R04 when compared to previous test runs which utilized the fluid bed dryer (TC25 and R01). The standard deviation for the coal moisture content for the three runs was essentially the same (about 1.3 percent).

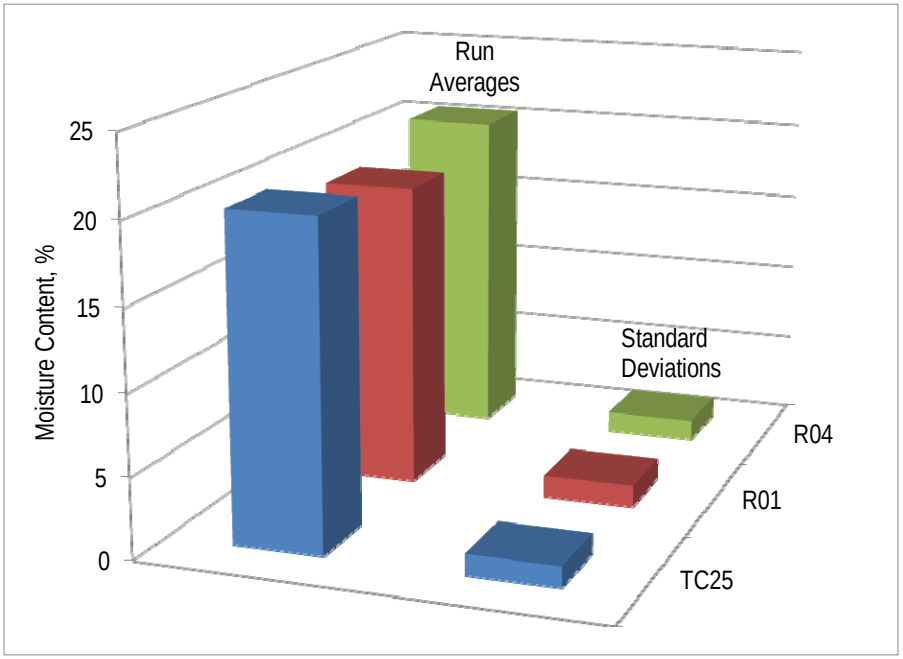

Figure 1. Fluid Bed Dryer Outlet Coal Moisture Concentration for Three Runs.

The biomass fuel used consisted of wood pellets from Green Circle BioEnergy. The biomass fuel was also processed in the mill system to reduce the moisture content and control the particle size distribution. Table 2 lists the average as-received and as-fed coal moisture and size data.

Table 2. Biomass Properties.

As-Received Moisture Concentration, wt%	7
As-Fed Moisture Concentration, wt%	4
Moisture Content Reduction, %	43
As-Fed Mass Mean Diameter, micron	830
As-Fed Oversize (>1,180 micron) Content, wt%	29
As-Fed Fine (< 45 micron) Content, wt%	1

To avoid problems conveying biomass that were experienced during previous biomass operation (in run R03), modifications were made to the dense phase conveying system that transfers the biomass from the pulverized coal storage silo to the coal feeder. The transfer line plugging problems previously experienced were due to piping configuration and insufficient conveying gas (nitrogen) supply. The piping layout was modified to remove excess elbows and other areas of unnecessary pressure drop. Additionally, the conveying gas supply to the dense phase feeder was increased and booster gas was added to elbows in the transfer line. For the transfer line booster gas additions, new Kates flow controllers were installed that maintain a constant volumetric flow of gas by using an internal regulating valve to counterbalance downstream fluctuations in pressure. These changes resulted in improved system operation when compared to R03 operation, maintaining solids velocity requirements high enough for consistent transfer of the low density biomass material. Figure 2 shows some of the changes made.

Booster Gas at Elbow | Kates Flow Controllers

Figure 2. Modifications to the Dense Phase Transfer Line for Biomass Conveying.

3.0 COAL FEEDING

Both the developmental and the original coal feeders operated well during R04. The developmental PDAC feeder was used to feed coal exclusively while the original rotary feeder

fed coal initially before transitioning to biomass operations during the co-gasification portion of the run.

PDAC Feeder. The PDAC feeder operated for 464 hours, including independent operations for 13 hours. The feeder operated at coal feed rates ranging from 770 to 5,700 lb/hr. Overall feeder performance during the run was stable over the range of coal feed rates specified.

Several modifications and improvements were made to the feeder system prior to R04 and were tested during the run. A new coal flow measuring device, the DensFlow meter from SWR Engineering, was installed in the feeder discharge line to the gasifier as shown in Figure 3. 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.

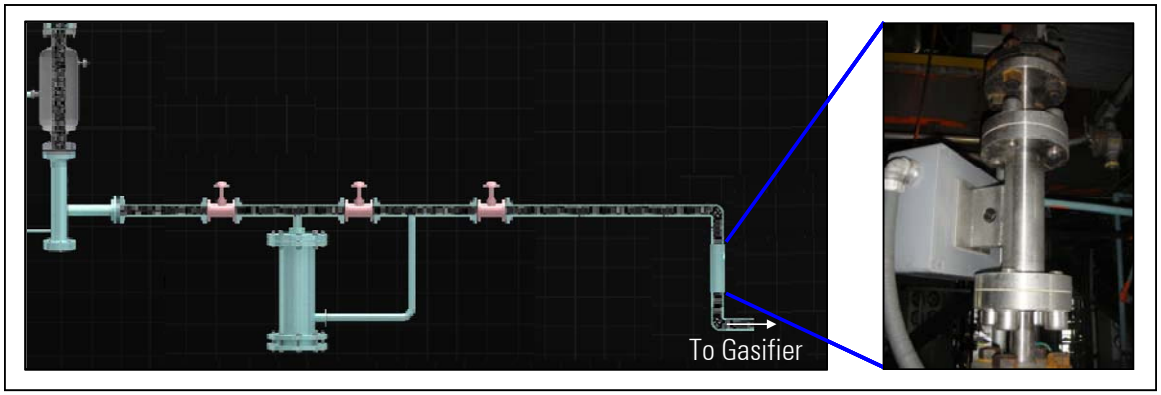

Figure 3. DensFlow Coal Flow Meter Installed on PDAC Feeder Outlet Piping.

As shown in Figure 4, the new DensFlow meter showed good agreement during the run with the flow rate calculated from the existing weigh cells. The measurement was also sensitive to instantaneous changes in coal feed rate when compared to existing conveying line differential pressure readings. While the meter was installed with the coal flowing in the vertical and down direction as suggested by the vendor, the feeder off-line system was also modified to permit testing of the flow meter in the vertical and up flow direction in the future.

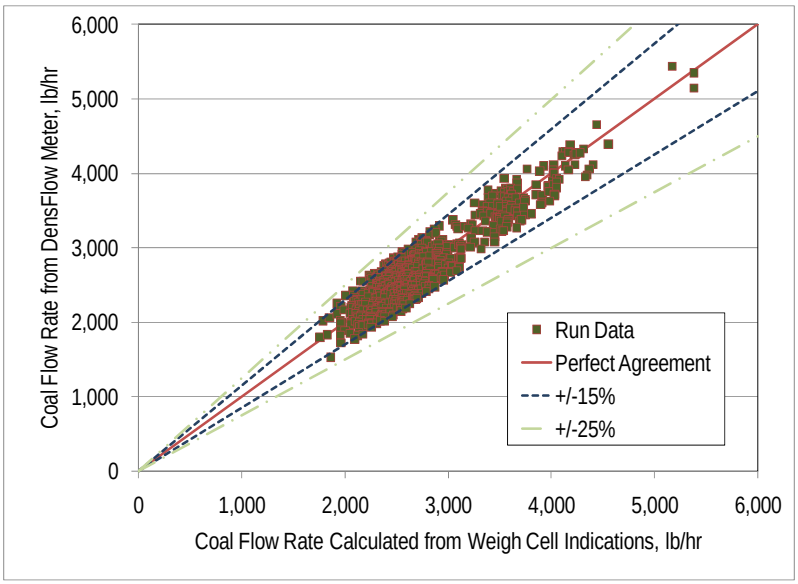

Figure 4. Comparison of Coal Feed Rates from DensFlow Meter and from Weigh Cell Calculation.

Long term evaluation of the Dynatrol vibration level probes continued during R04. The probes were installed to replace older, less reliable capacitance probes in several locations including both the lock vessel and dispense vessel of the feeder, as shown in Figure 5. The probes continued to demonstrate consistent operation. The vibration probes have a wide range of material measurement capability, from low density flakes or powders to heavy granules and pellets, and there is no required field adjustment of the probes after installation.

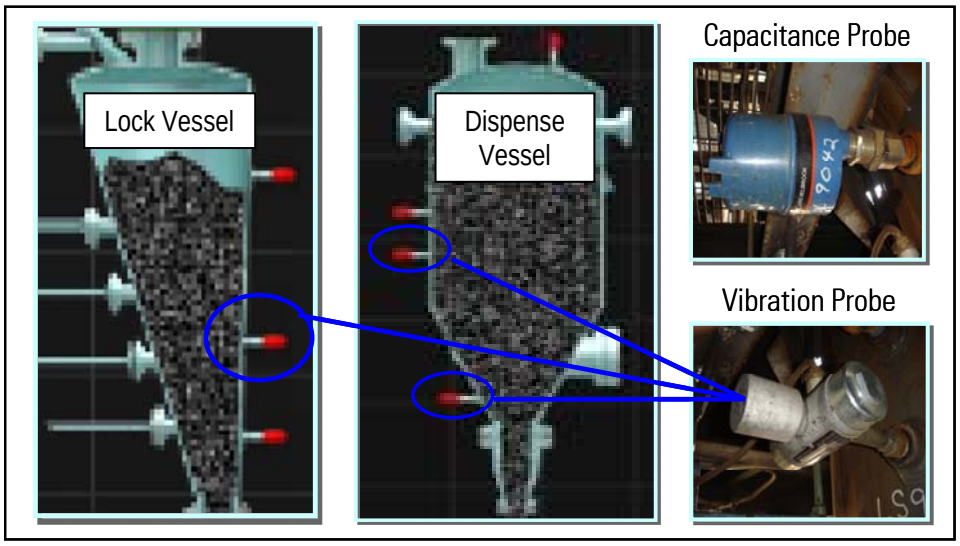

Figure 5. Location of Vibration Level Probes on PDAC Lock Vessel and Dispense Vessel.

Additional level probe technology was tested in the PDAC feeder dispense vessel during R04. Observations of previous feeder operation revealed a decrease in coal feed rate slightly before a

subsequent lock vessel fill cycle occurs. Data analysis of these instances indicated that a deep funnel was forming inside the dispense vessel resulting in the drop in coal feed rate. In order to prevent this occurrence, the level inside the dispense vessel needed to be controlled at a higher level. Therefore, a vertical level probe was installed in the top of the dispense vessel to permit the lock vessel fill cycle to occur sooner, maintaining higher level control.

A Drexelbrook point sensitive level probe was selected for this service. The operating basis of the probe is similar to a capacitance probe but combines a radio frequency signal with circuitry shielding technology to ignore the effects of material buildup on the probe. Initial field calibration of the probe was required using a simple potentiometer adjustment. The results achieved during the run, shown in Figure 6 and Figure 7, indicate that previous assumptions of funnel formation were correct. The probe will be inspected during the outage, and additional testing is planned for future runs.

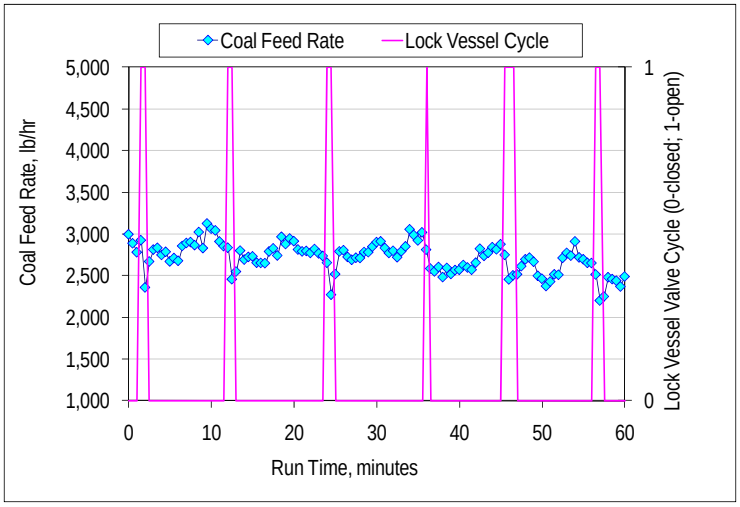

Figure 6. PDAC Coal Feed Rate before Dispense Vessel Vertical Level Probe Control.

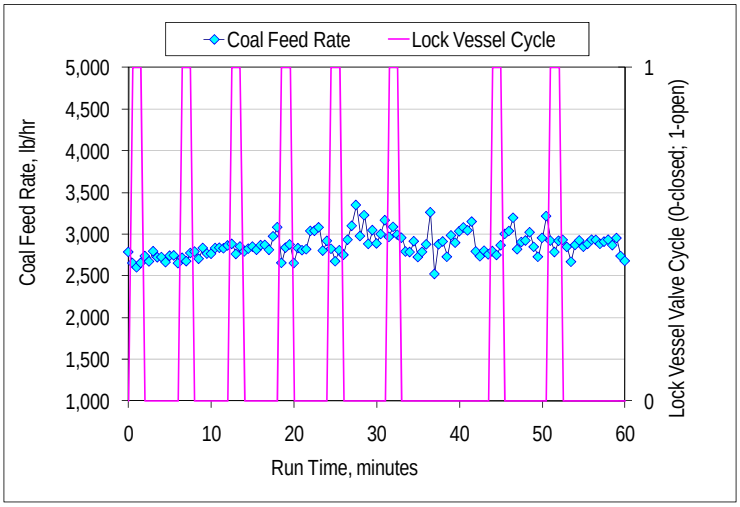

Figure 7. PDAC Coal Feed Rate after Dispense Vessel Vertical Level Probe Control.

PDAC feeder control logic improvements continued during the R04 test run. Additional tuning was performed on the coal feed rate trim controller to further improve response to instantaneous changes in feed rate as indicated by the conveying line differential pressure indication. Future testing will involve the incorporation of the new DensFlow coal flow meter mentioned previously into the control scheme. Control changes were also made to increase the operating level of the feeder atmospheric storage silo. Controlling the silo at a higher level promoted a mass flow regime from the bin, decreasing material segregation that can lead to coal feed rate fluctuations. Finally, additional tuning was also performed on the temperature controller implemented during R03 to control gasifier temperature by adjusting the PDAC coal feed rate. Further optimization of this controller will include incorporation into the gasifier air flow control scheme.

Original Coal Feeder. The original rotary coal feeder operated on coal for 388 hours at feed rates varying from 1,100 to 3,400 lb/hr. The feeder was then transitioned to feed biomass for 109 hours at feed rates ranging from 475 to 2,700 lb/hr. The biomass feed rate accounted for an average of 17 weight percent of the total mass feed rate to the gasifier. A period of biomass only operation was conducted for a short time during the last few hours of the run.

4.0 TRANSPORT GASIFIER

Figure 8 plots the averages of the maximum gasifier temperature (the mixing zone temperature), the outlet gasifier temperature and pressure, and the primary gas cooler outlet temperature during the 31 steady state operating periods. The primary gas cooler operated as designed, with an outlet temperature of about 750°F during the run.

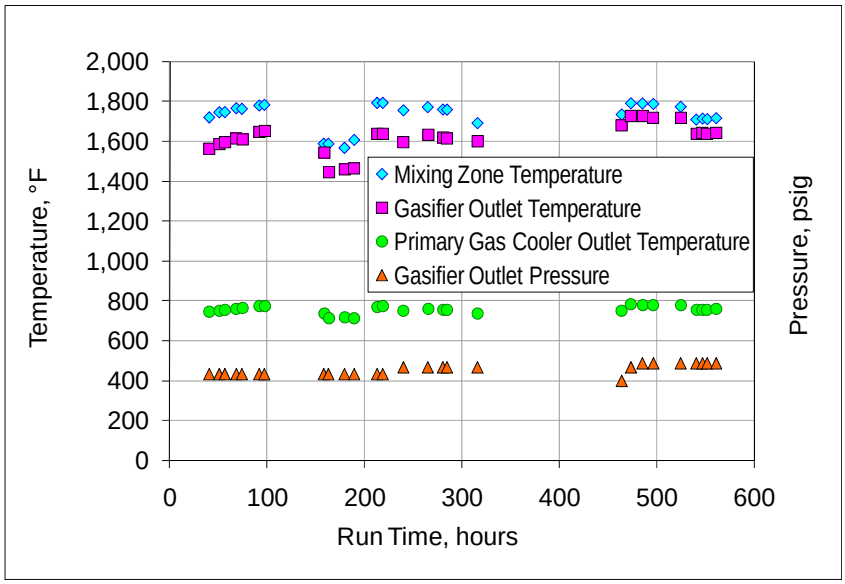

Figure 8. Steady State Gasifier Operating Conditions.

Parametric testing was performed during gasifier startup (before coal feed) to determine if standpipe fluidization could be used exclusively to control gasifier solids circulation rate to low

levels. Test data indicated that gasifier solids circulation rate could be controlled to low levels by varying only the fluidization to the bottom of the standpipe. Figure 9 illustrates the relationship that was demonstrated during this testing.

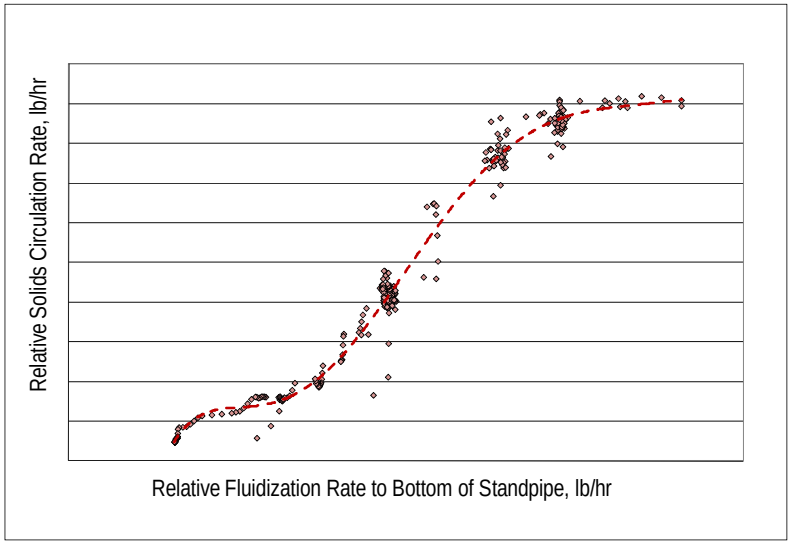

Figure 9. Gasifier Solids Circulation Rate versus Fluidization Rate to Bottom of Standpipe.

Gasifier operations remained stable for most of the R04 test producing consistent quality syngas for various tests at the syngas cleanup unit. Coal-only feed was maintained for the first 400 hours of the test run before transitioning to a period of co-feed operations with both coal and biomass for the final 100 hours. Inspections of gasifier ash samples taken during both these periods did not indicate agglomeration (see Figure 10). Additionally, the primary gas cooler performance remained stable, and condensate sampling did not indicate any evidence of tar formation.

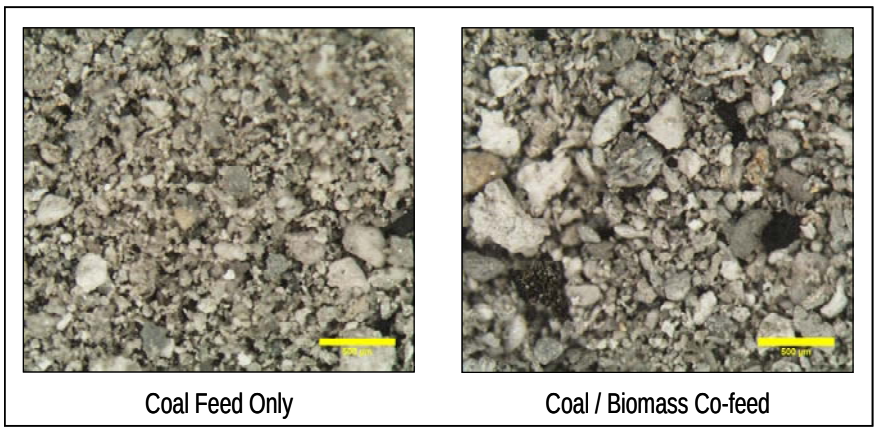

Figure 10. Gasifier Ash Samples during Coal-Only Feed and during Biomass Co-Feed.

During the final hours of the test run, a period of biomass feed only was tested. Primary gas cooler performance began to deteriorate soon after establishing biomass only feed to the gasifier,

as the cooler outlet temperature began to rise (evidence of tube fouling). Eventually, this decrease in performance resulted in the end of gasifier operations for R04. Post-run inspections revealed fouling in the primary gas cooler tubes, as shown in Figure 11. Samples were collected for analysis, and results will be forthcoming. Preliminary indications suggest the presence of tars, but fouling of the exchanger due to the alkali metal content of the biomass could not be ruled out at the time of this report.

Figure 11. Post-Run Inspection of Primary Gas Cooler Tubes.

Gasifier sensor development efforts continued during R04. A sapphire thermowell was installed in the gasifier riser 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. Figure 12 illustrates the agreement of the two indications.

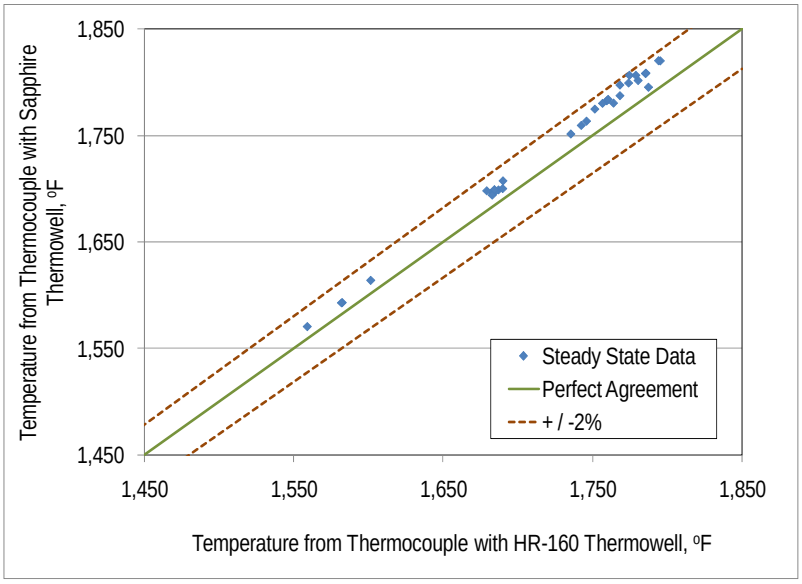

Figure 12. Comparison of Temperature Readings for Sapphire and HR-160 Thermowells.

Post-run inspections revealed no observable signs of erosion. Figure 13 shows both the side and end-on views of the sapphire thermowell, which was left installed in the gasifier.

End-on View of Thermowell Side View of Thermowell

Figure 13. Inspection of Sapphire Thermowell.

Inspections of the gasifier refractory showed that the hot-face refractory at the J-leg / standpipe intersection, which had been repaired prior to R02, was still in good condition, as shown in Figure 14. A slightly damaged area in the upper standpipe / solids separation unit intersection, which had been discovered prior to R01, continued to show acceptable condition, as shown in Figure 15. The damage noted appeared to be related to the extra stress that intersecting areas of refractory receive. The gasifier standpipe, including these two areas of concern, is scheduled to be replaced during the 4th quarter of this year.

Prior to R02 Post-R04 Inspections

Figure 14. Inspection of Refractory in J-leg / Standpipe Intersection.

Prior to R01 Post-R04 Inspections

Figure 15. Inspection of Refractory in Upper Standpipe / Solids Separation Intersection.

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. 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. The elements were left installed for further exposure in R05.

6.0 ASH REMOVAL SYSTEMS

The new piping configuration for the Continuous Fine Ash Depressurization (CFAD) system (installed prior to R03) that routes the fine ash directly to the ash silo without intermediate transport equipment was operated for the entire R04 gasification run. Operation of the new configuration has maintained the high reliability historically demonstrated by the CFAD system. Prior to R04, an oxygen analyzer was installed in the ash silos to continuously monitor the silo oxygen concentration and ensure compliance with the National Fire Protection Association (NFPA) 69, Standard on Explosion Prevention Systems. Previously, additional nitrogen was added to the ash silos as a conservative measure to comply with the standard since the oxygen concentration could not be continuously measured and there was some possibility of the presence of a small amount of syngas in the transport gas. As a result of this oxygen analyzer installation, the extra nitrogen consumption at the ash silos was eliminated and realization of the project savings (approximately $9,000 annual cost reduction) began during R04.

7.0 SYNGAS CONDITIONING

CO_2 Capture Testing. The batch reactor system was operated to capture CO_2 from syngas. Initial tests with two new solvents were carried out, selected properties for which are tabulated in Table 3. In addition, measurements were made to speciate compounds formed during CO_2 capture with ammonia.

Table 3. CO_2 Sorbent Test Parameters.

Sorbent	Molecular Weight	Concentration		Absorption Temperature, ºF
		Molar	wt%	
Ammonium hydroxide	35	7.6	14	100
Potassium carbonate	138	1.7	20	210
Proline	153	5.1	56	100

Testing was performed with the potassium salt of proline, an amino acid. The solution had good collection efficiency and kinetics but had limited capacity. However, proline is not volatile and indications are that it does not degrade over the short-term testing conducted to date. In addition to the CO_2 capture, co-capture of H_2S with high efficiency was also observed during testing of proline. Preliminary indications are that the H_2S can be thermally regenerated along with the CO_2. However, deep regeneration of the proline appeared to be difficult without significant loss of water. Samples of the absorption and regeneration solutions are being analyzed to quantify the level of regeneration, and additional testing is planned to minimize the water loss.

Testing was also initiated using potassium carbonate. The potassium carbonate also is stable at the temperatures used and was shown to maintain CO_2 capacity over repeated absorption-regeneration cycles. Although the potassium carbonate has good capacity, its reaction kinetics are slow, and overall collection efficiency is not as high as other materials. To approach 100 percent CO_2 capture, the reactor was operated at 210ºF rather than the typical operating temperature of 100ºF. Future testing is planned with the use of additives to improve the reaction kinetics. As with the proline, regeneration tests were also successfully conducted with the potassium carbonate. Figure 16 show the CO_2 capture efficiency for the three solvents.

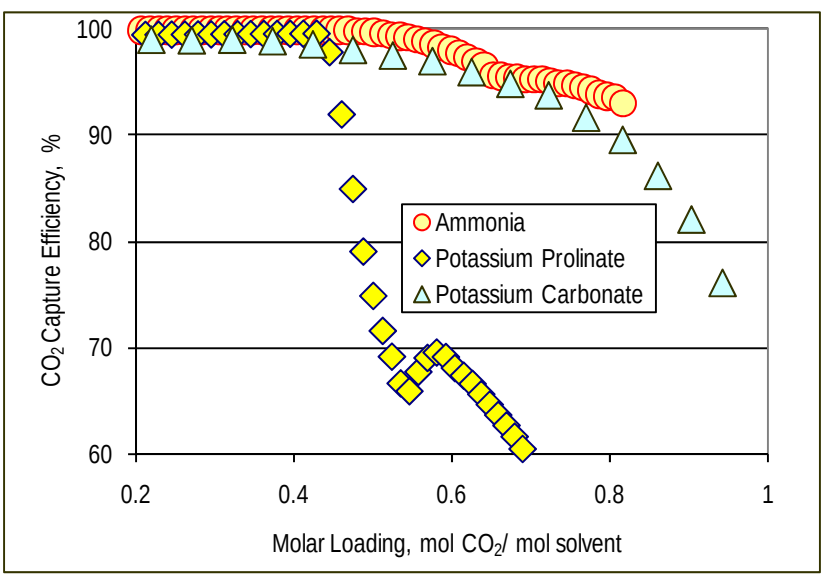

Figure 16. CO_2 Capture Efficiency of Sorbents Tested in R04.

During the run, a Raman spectrometer with a high temperature, high pressure probe was utilized in the batch reactor to continuously monitor the progress of the CO_2 capture reaction. The

instrument was provided by Kaiser Optical Systems. There is currently no library of spectra for the compounds formed, so NCCC/PSDF staff is working with Kaiser to develop a model that can relate the spectra obtained to the concentrations of the chemical species present.

As the CO_2 capture reactions proceeded in the aqueous ammonia solution, Raman spectra were obtained, which are given in Figure 17. In the figure, different line colors denote spectra obtained at different times during the reaction; red arrows indicate significant Raman spectral shifts that appear to be correlated with the concentrations of ammonia, hydroxide, carbamate, carbonate, and bicarbonate. While the spectra were generated, samples were collected for analysis by nuclear magnetic resonance (NMR). NMR has been shown previously to produce reliable results. Results obtained from the Kaiser probe will be compared to the NMR data and to computer simulation model predictions.

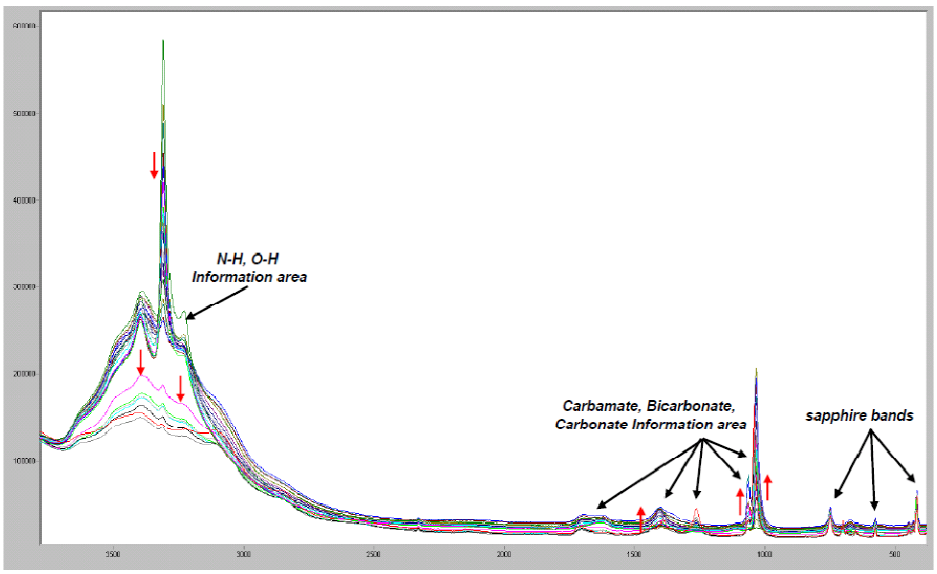

Figure 17. Raman Spectra Obtained during CO_2 Capture by Aqueous Ammonia.

Water-Gas Shift Testing. Water-gas shift (WGS) tests were performed using catalytic filter elements and a fixed bed reactor. Parametric tests for both types of tests involved H_2O-to-CO ratios and temperatures, and face velocity was also varied for the filter element testing. Both tests utilized 50 lb/hr of syngas. Parameters varied included:

- Space velocities for fixed-bed reactor from 2,000 to 3,000 hr^{-1}
- Face velocities for catalytic filters from 1. 8 to 3.0 ft/min
- Steam-to-CO molar ratios from 0.8 to 2.0
- Bed temperatures from 500 to 650ºF

Data analysis is underway.

Near the end of R04, there was an oxygen breakthrough event for both the WGS test vessels that was initially triggered by a coal feeder trip. After careful study, actions being taken to prevent a

reoccurrence of an oxygen breakthrough at the SCU include several safety interlock/control changes as well as changes in operating procedures.

Media Process Technology Membrane. MPT continued its testing of the Carbon Molecular Sieve hydrogen membrane. The membrane apparatus consisted of a bundle of ten 30-inch by 0.225 inch outer diameter tubes installed in a 4-inch pipe. To prevent deposition on the membrane surface, naphthalene was removed from the syngas in a cooled isopropanol bubbler upstream of the membrane skid. The cleaned syngas was then re-heated to the required operating temperature.

The nominal syngas flow rate of about 10 lb/hr was spiked with pure hydrogen in order to raise the hydrogen concentration to about 40 percent, the typical hydrogen concentration in syngas produced from an oxygen-blown gasifier and treated in a water-gas shift reactor. About 40 percent of the hydrogen in the syngas was recovered in the permeate when the membrane temperature was maintained at 300°F. The hydrogen concentration in the permeate was about 90 percent. Results with a single tube assembly tested at the NCCC in 2008 achieved 30 percent recovery of with 98 percent hydrogen in the permeate. Reasons for the difference have not yet been determined.

For future testing, MPT is planning to test 14 membrane tubes held in position by fused glass mounted in a ceramic collar that fits inside a 3-inch tube. The assembly, shown in Figure 18, is designed to handle a maximum syngas flow of 20 lb/hr. Improved trace heating will be applied to achieve the design operating temperature of 520ºF.

Figure 18. MPT's Carbon Molecular Sieve 14-Tube Assembly.

Johnson Matthey Mercury Capture Sorbent. Johnson Matthey had previously tested a high temperature mercury sorbent with 5 wt% palladium at the NCCC/PSDF (in 2008 and 2009), and demonstrated high capture efficiencies of mercury, as well as arsenic and selenium. To optimize the palladium loading and reduce the cost of sorbent materials, Johnson Matthey is evaluating lower palladium loading in the sorbent. During R04, sorbent with 2 wt% palladium loading was tested in syngas for 500 hours.

The testing commenced with 10 pounds of sorbent at 10 lb/hr of syngas flow. Before syngas was introduced, the sorbent was pre-reduced on site with a hydrogen/nitrogen mix according to Johnson Matthey's instructions. Four sets of gas samples taken at the reactor inlet and outlet were collected using a modified EPA Method 29 for trace metal analysis.

Membrane Technology and Research Membranes. MTR continued testing of a hydrogen membrane and a CO_2 membrane, both of which had been tested previously during R03 in 2009. The CO_2 membrane used 50 lb/hr syngas (scaled up from the previous test), while the hydrogen membrane employed 1 lb/hr. In this test, sulfur was not removed from the syngas as it was in R03. The syngas passed through the water-gas shift reactor, a cooler, and a knockout pot before entering the membrane skid.

Figure 19 plots the performance of the CO_2 and hydrogen membranes. As shown, the CO_2 membrane increased the CO_2 concentration from about 12 percent in the feed syngas to 50 to 60 percent in the permeate stream. The hydrogen membrane increased the hydrogen concentration from about 12 percent in feed syngas to 70 to 80 percent in the permeate stream. MTR staff will evaluate the sulfur impact to the membrane performances, although initial results suggest that any impact is not significant.

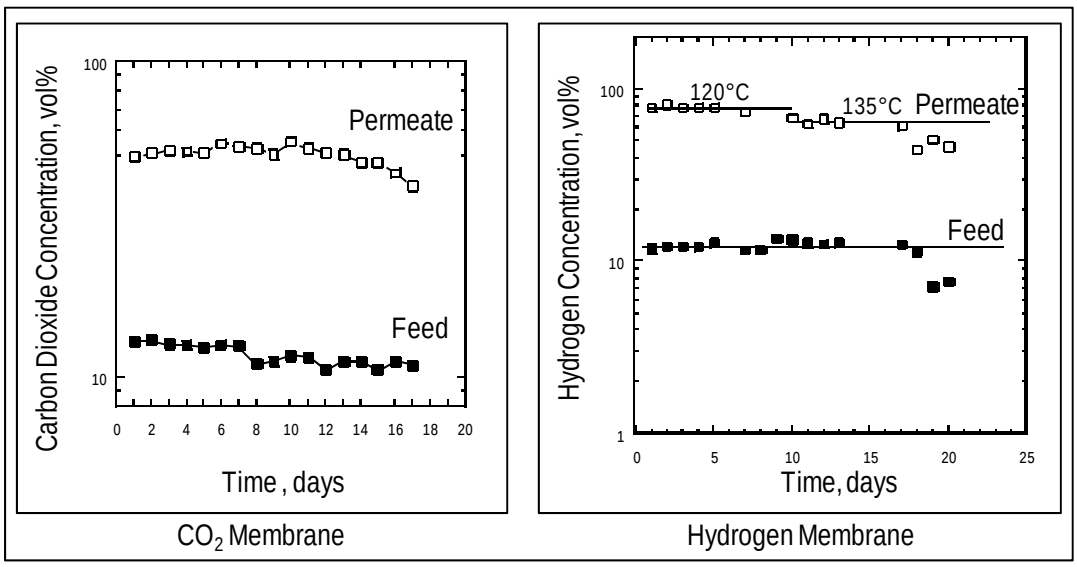

Figure 19. Performance of MTR's CO_2 and Hydrogen Membranes.