THE NATIONAL CARBON CAPTURE CENTER
AT THE POWER SYSTEMS DEVELOPMENT FACILITY

TEST RUN SUMMARY REPORT

TEST RUN R07
OCTOBER 20, 2011 – DECEMBER 12, 2011

DOE COOPERATIVE AGREEMENT
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Prepared by:
Southern Company Services, Inc.
Power Systems Development Facility
P.O. Box 1069, Wilsonville, AL 35186
Phone: 205-670-5840
Fax: 205-670-5843
http://www.NationalCarbonCaptureCenter.com

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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
NCCC—National Carbon Capture Center
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 R07—Tests Runs 1 through 7
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 R07 from October 20 to December 12, 2011. The total run duration was 996 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 tested.

2.0 COAL PREPARATION

The fuel for R07 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 2,280 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 %	51.2
As-Received Hydrogen, wt %	3.2
As-Received Nitrogen, wt %	0.7
As-Received Sulfur, wt %	0.3
As-Received Ash, wt %	7.0
As-Received Oxygen, wt % (by difference)	12.6
As-Received Volatiles, wt %	31.4
As-Received Fixed Carbon, wt %	36.7
As-Received Heating Value, Btu/lb	8,739
As-Received Moisture Concentration, wt%	25
As-Fed Moisture Concentration, wt%	19
Moisture Content Reduction, %	24
As-Fed Mass Median Diameter, micron	402
As-Fed Oversize (>1,180 micron) Content, wt%	9
As-Fed Fine (< 45 micron) Content, wt%	8

3.0 COAL FEED

Both the developmental Pressure Decoupled Advanced Coal (PDAC) feeder and the original coal feeder operated during R07 without any major coal stoppages.

3.1 PDAC FEEDER

The PDAC feeder operated for 805 hours, including independent operations for 22 hours. The feeder operated at coal feed rates ranging from 500 to 2,800 lb/hr. Overall feeder performance during the run was stable over the range of coal feed rates tested.

PDAC Modifications. Modification of nitrogen gas distribution to the feeder resulted in decreased nitrogen flow rate variability and improved overall nitrogen flow control during the run. The internal configuration of the feeder bottom was also changed, reducing coal particle segregation and providing more consistent coal feed to the gasifier. Control logic modifications were also successfully tested during the run. The coal feed rate trim controller, which changes nitrogen feed rate as needed to maintain coal feed to the gasifier, was successfully modified to use the feeder conveying line differential pressure as an indication of instantaneous changes in coal feed rate. This controller performed well and can be modified in the future with other coal flow measurement devices. Results of the testing are demonstrated by Figure 1.

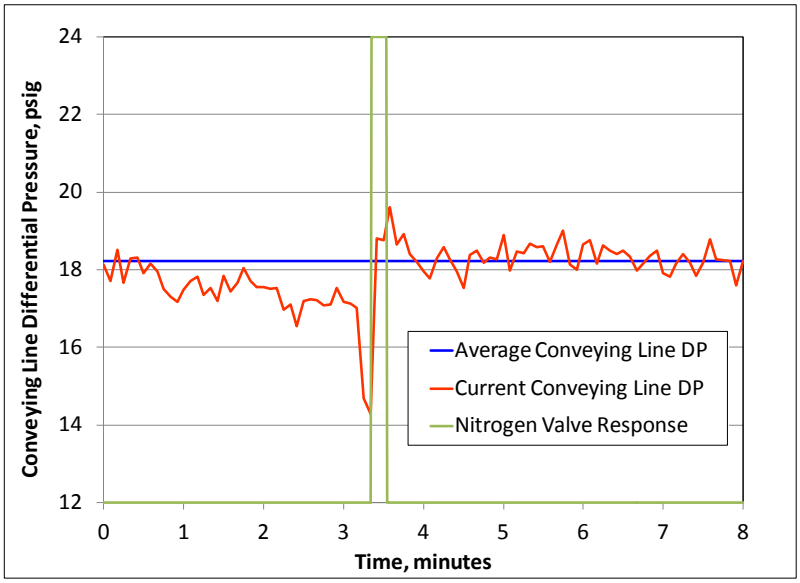


Figure 1. PDAC Feeder Trim Controller Response to Change in Coal Feed Rate.

Level Probes. Long-term evaluation of the Dynatrol vibration level probes continued during 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 solids level indication.

Evaluation of the Drexelbrook point sensitive level probe also continued during 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.

Coal Flow Meter. 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 offline testing will be conducted during the 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.

3.2 ORIGINAL COAL FEEDER

The original rotary coal feeder operated for 975 hours at feed rates varying from 1,000 to 3,400 lb/hr. Overall feeder performance during the run was stable over the range of coal feed rates tested.

4.0 TRANSPORT GASIFIER

R07 operation consisted of R07 operation consisted of 46 steady state operating periods, with recycled syngas utilized for gasifier fluidization for 37 of those steady state 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 R07 and Previous PRB Test Runs.

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

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.

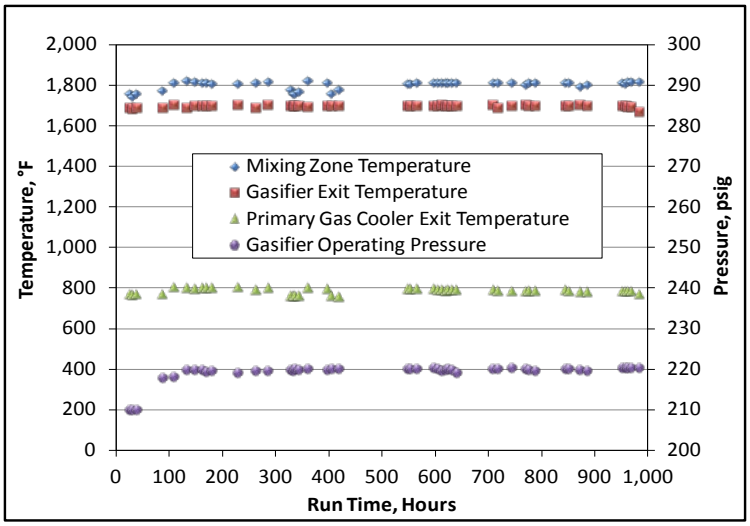


Figure 2. Gasifier Operating Conditions.

Automated Temperature Control. Gasifier temperature control enhancements utilizing air flow adjustments from the gasifier upper and lower mixing zone continued during R07. Since gasifier temperatures respond rapidly to changes in air feed rate, tuning of the individual and total air flow controllers to prevent temperature fluctuations was to maintain steady-state operation during previous runs. Beginning during 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 3 illustrates the improvement in gasifier exit temperature control achieved by these controllers. Gasifier temperature variation has been reduced to less than 5°F.

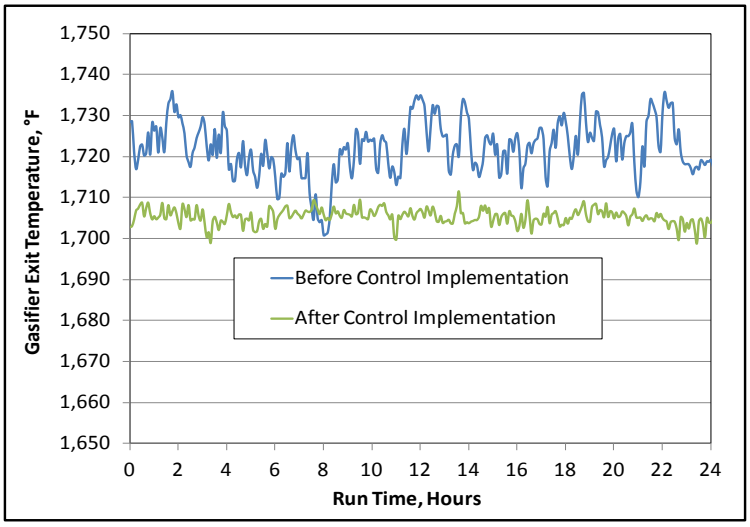


Figure 3. Gasifier Temperature Control during Non-Steady-State Operation.

Sapphire Thermowell. Testing of the sapphire thermowell ceased prior to R07 due to a continued drift observed in its measurements. The thermowell had been exposed to over 2,700 hours of gasification operation throughout which a slow but consistent decline in its measurement was observed, as shown in Figure 4. However, the instrument still responded well to temperature changes at a rate similar to that of nearby thermocouples.

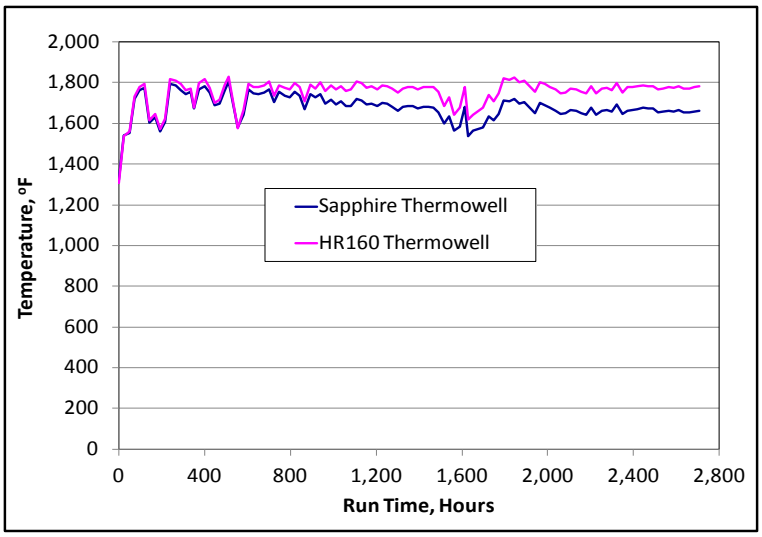
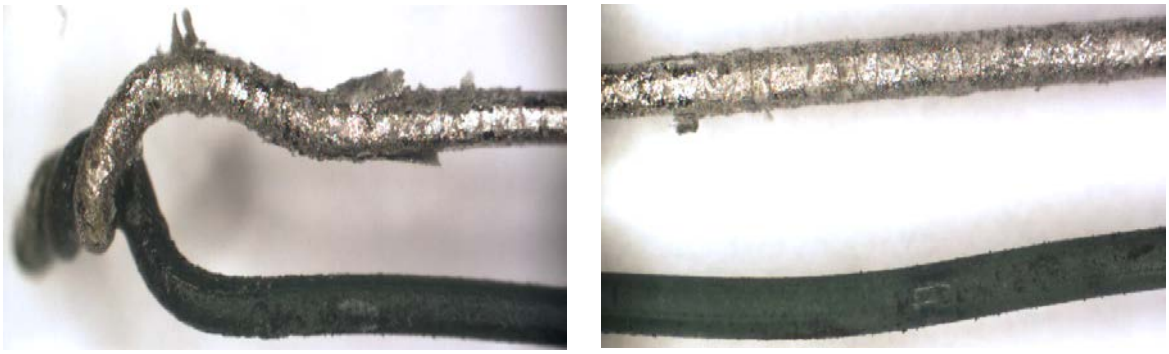


Figure 4. Drift in Sapphire Thermowell Temperature Indication during Gasifier Operation.

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 offsite 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 5, indicating that syngas contact had occurred.



View Near Tip of Thermocouple View Further from Tip of Thermocouple

Figure 5. 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 R08. They will be onsite for assistance during the initial inspection and installation of the assembly. Discussions are also currently underway to look at potential tests of alternate sapphire thermowell designs in other gasifier locations.

5.0 PARTICULATE CONTROL DEVICE

Operation of the particulate control device (PCD) was achieved during R07 with high collection efficiency 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 R07. 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.

6.0 ASH REMOVAL SYSTEMS

Both the Continuous Coarse Ash Depressurization (CCAD) and Continuous Fine Ash Depressurization (CFAD) systems operated well during R07. 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. 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 WATER-GAS SHIFT CATALYST

During R07, a new 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, pressure,

and space velocity. 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 indicates that good mass balance closures were achieved, giving confidence in the data collected. Over 80 percent CO conversion was achieved at a steam-to-CO ratio of 1.0, which agreed well with the vendor's laboratory test data. The catalyst performance did not deteriorate over the test period, and post-test inspections showed no physical degradation.

7.2 WORCESTER POLYTECHNIC INSTITUTE MEMBRANES

Two membranes from Worcester Polytechnic Institute (WPI) were tested in R07 for a combined 646 hours (582 hours in hydrogen-enriched syngas, with the balance in a nitrogen/hydrogen mixture). This length of time is a milestone in palladium membrane syngas operation. The syngas used was passed through sulfur sorbent and COS hydrolysis beds and any sulfur present was below the 1 ppmv detection limit of the gas chromatographs. Although essentially sulfur free, the syngas still contained hydrocarbons.

The two membranes were tested in R06, and were subsequently polished and recoated with the palladium or palladium/gold layer. No leaks developed during R07 and as a result, high hydrogen purity was achieved in the permeate stream —greater than 99.5 percent. WPI also continued its fundamental research program by installing a membrane coupon inside the membrane module. In this way, both components were exposed to the same syngas conditions, allowing for unbiased comparisons of the materials.

The first WPI membrane was pure palladium with a thickness of 9.6 microns and the second was a palladium-gold membrane with 8.7-micron thick palladium covered with a 0.5-micron thick layer of gold. The palladium membrane showed stable hydrogen flux of around 20 scfh/ft^2 in a nitrogen/hydrogen atmosphere. After nitrogen was replaced with syngas, this increased to about 36 scfh/ft^2 due to a hydrogen feed concentration increase from 24.2 to 30.5 percent. Unlike the previous run, no significant decline in initial hydrogen flux was observed. The palladium-gold membrane showed a stable hydrogen flux of 37 scfh/ft^2 in a nitrogen/hydrogen atmosphere that increased to about 60 scfh/ft^2 after nitrogen was replaced with syngas. Again, no significant decline in initial hydrogen flux was observed, but after 470 hours operation the average flux was reduced to 50 scfh/ft^2, producing 1.4 lb of hydrogen per day.

Membrane performance measurements at WPI facilities before and after the field test showed a 35 to 40 percent reduction in hydrogen permeance, the majority of which appeared to be unrecoverable. The most likely cause is thought to be contamination of the palladium. Preliminary X-ray photo-electron spectroscopic analysis of a palladium coupon placed inside the palladium membrane tube reveals the presence of a large amount of carbon with some sulfur, and mercury. It is hypothesized that the carbon present originated from polyaromatic hydrocarbons present in the syngas.

7.3 MEMBRANE TECHNOLOGY & RESEARCH MEMBRANES

During R07, Membrane Technology & Research (MTR) tested a CO_2 membrane (Polaris™), and a hydrogen membrane (Proteus™) for a total of 807 hours. The syngas was processed to remove

the hydrocarbons but still contained hydrogen sulfide. Based on its design data and operating experience, MTR projects that with further improvement its membranes will approach the DOE target of 10 percent increase in the levelized cost of electricity.

The Polaris membrane modules were housed in a pressure vessel that treats 30 to 45 lb/hr syngas. A total of four 4-inch diameter membrane modules were tested, two individually and two connected in series. They varied in total membrane areas and stage cuts (percentage of feed gas permeates across the membrane). Based on the test results, MTR concluded that:

- CO_2 content increased from about 10 percent in the feed to 30 to 50 percent in the permeate.
- The separation performance was stable during the entire period of normal operation for the four membrane modules, indicating that there was no performance deterioration after extended exposure to the raw syngas.
- The CO_2 permeance ranged from 200 to 400 gpu (gas permeation unit, representing the volumetric flow rate per unit cross sectional area for a given pressure differential), CO_2/H_2 selectivity from 3.0 to 5.7, and CO_2/N_2 selectivity from 6 to 27.
- The membrane modules were stable in the presence of sulfur and other containments.
- The two modules that used a modified membrane structure and were connected in series had better selectivity than the other modules despite having only a slightly lower stage cut. Future testing will be based on this new module configuration.

Two 2.5-inch diameter lab-scale Proteus™ membrane modules were tested treating about 5 lb/hr of syngas. The first module was tested near the end of R06 but with limited test hours, and the second one was not previously tested. Normal operating temperature was 275°F, and tests were also carried out at 300°F. Some flow issues affected the test results. Based on the test results, MTR concluded:

- The hydrogen content was enriched from 12.5 percent in the feed to 48 to 65 percent in the permeate.
- The field test results were in good agreement with the laboratory performance results.
- The hydrogen permeance ranged from 130 to 160 gpu, and H_2/CO_2 selectivity was about 14.
- The modules can be used at temperatures up to 300°F for extended periods and were stable in the presence of sulfur and other contaminants.
- Future tests will continue to determine the membrane stability for long-term operation.

7.4 MEDIA & PROCESS TECHNOLOGY MEMBRANE

During R07, Media & Process Technology (MPT) evaluated its full-scale 86-tube Carbon Molecular Sieve (CMS) membrane bundle processing syngas without pretreatment to remove hydrocarbons or hydrogen sulfur. Also included in this test campaign was MPT's palladium/copper alloy membrane for hydrogen enrichment of the permeate leaving the CMS membrane. The run verified the performance and stability of the CMS membrane, with feed rates as high as 600 scfh. With over 300 hours of performance testing completed with two full-scale membrane bundles, the CMS membrane permeance was unchanged. Operating the membrane at 480°F effectively prevented hydrocarbon deposition on the membrane and module.

The CMS bundle configuration and housing design employed for these tests have proved adequate for the selected operating conditions, i.e., 480 to 570°F and greater than 200 psi. As previously demonstrated in smaller scale NCCC testing, no membrane degradation in the presence of H_2S or other gasifier syngas contaminants was observed. The permeate contains over 80 percent hydrogen, with nitrogen and some CO and hydrogen sulfide. Before passing to the palladium membrane the hydrogen sulfide is removed from the CMS permeate in a sorbent bed. The permeate from the palladium membrane consisted of over 99 percent pure hydrogen. No performance degradation was noted with this membrane in the 10 hours of testing.

The hydrogen permeance of the full-scale CMS bundle operating on syngas was lower than anticipated. Observations suggest that this arises from insufficient syngas reaching the tubes in the interior of the bundle. MPT engineers are currently investigating this issue in their laboratory and exploring ways to improve syngas distribution using internal baffles and increased tube spacing.

7.5 TDA RESEARCH CO_2 SORBENT

During R07 TDA commissioned and tested its CO_2 sorbent system in syngas, and initial post-run data analysis has been completed. The four-bed pressure-swing adsorption system completed more than 1,200 cycles using shifted, sweet syngas, with hydrocarbons present achieving more than 98 percent CO_2 removal from syngas at temperatures in excess of 390°F. The sorbent regenerations were carried out at pressures greater than 70 psia, resulting in recovery of CO_2 at pressure. Figure 6 shows typical field test data for the CO_2 concentration in the feed and the product syngas. The inlet CO_2 concentration was taken from a single data point from a gas chromatograph. The figure shows about 98.4 percent CO_2 removal from coal-derived syngas with a bed temperature ranging from 410 to 445°F, adsorption pressure at 203 psia, and desorption pressure of 73 psia. The sorbent maintained its capacity throughout the test period.

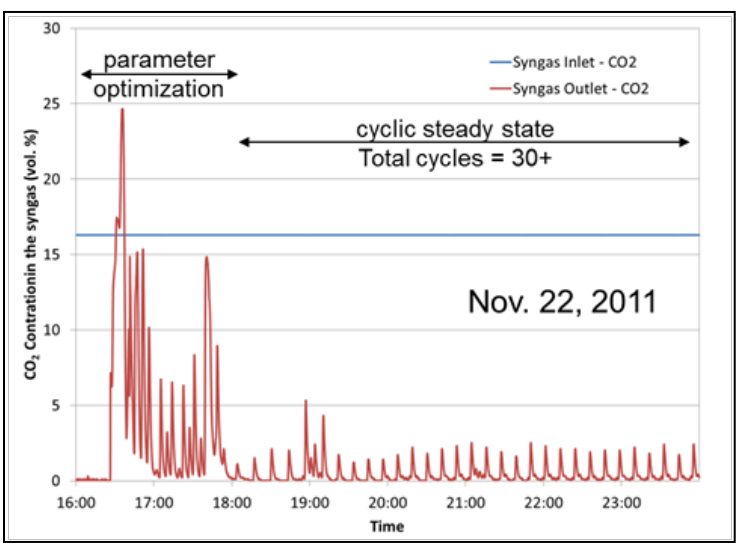


Figure 6. Typical Field Test Data for the TDA Research CO_2 Sorbent.