**POWER SYSTEMS DEVELOPMENT FACILITY
TOPICAL REPORT**

**GASIFICATION TEST CAMPAIGN TC24
FEBRUARY 16, 2008 – MARCH 19, 2008**

DOE Cooperative Agreement
DE-FC21-90MC25140

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September 2008

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ABSTRACT

In support of technology development to utilize coal for efficient, affordable, and environmentally clean power generation, the Power Systems Development Facility (PSDF), located in Wilsonville, Alabama, routinely demonstrates gasification technologies using various types of coals. The PSDF is an engineering scale demonstration of key features of advanced coal-fired power systems, including a KBR Transport Gasifier, a hot gas particulate control device, advanced syngas cleanup systems, and high-pressure solids handling systems.

This report summarizes the results of TC24, the first test campaign using a bituminous coal as the feedstock in the modified Transport Gasifier configuration. TC24 was conducted from February 16, 2008, through March 19, 2008.

The PSDF gasification process operated for about 230 hours in air-blown gasification mode with about 225 tons of Utah bituminous coal feed. Operational challenges in gasifier operation were related to particle agglomeration, a large percentage of oversize coal particles, low overall gasifier solids collection efficiency, and refractory degradation in the gasifier solids collection unit. The carbon conversion and syngas heating values varied widely, with low values obtained during periods of low gasifier operating temperature.

Despite the operating difficulties, several periods of steady state operation were achieved, which provided useful data for future testing. TC24 operation afforded the opportunity for testing of various types of technologies, including dry coal feeding with a developmental feeder, the Pressure Decoupled Advanced Coal (PDAC) feeder; evaluating a new hot gas filter element media configuration; and enhancing syngas cleanup with water-gas shift catalysts. During TC24, the PSDF site was also made available for testing of the National Energy Technology Laboratory's fuel cell module and Media Process Technology's hydrogen selective membrane.

ACKNOWLEDGEMENT

The authors wish to acknowledge the contributions and support provided by various project managers, including Morgan Mosser of the Department of Energy, Tim Challand of KBR, and John Wheeldon of the Electric Power Research Institute. The project is sponsored by the U.S. Department of Energy National Energy Technology Laboratory under cooperative agreement DE-FC21-90MC25140.

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1.0 EXECUTIVE SUMMARY

Test campaign TC24 was a demonstration of the Power Systems Development Facility (PSDF) gasification process with bituminous coal from the Dugout Canyon Mine in Wellington, Utah. TC24 began on February 16, 2008, and lasted until March 19, 2008, achieving 237 hours of operation in air-blown gasification mode. In addition to characterizing operation of the modified gasifier and related equipment with the Utah bituminous coal, test objectives included developmental coal feeder testing, continued testing of instrumentation enhancements, hot gas filter element and failsafe testing, and performing a preliminary evaluation of multi-functional filter elements. The PSDF also provided support for the objectives of commissioning by Media and Process Technology (MPT) of a hydrogen membrane and of installing and shakedown testing of the Fuel Cell Module developed at the National Energy and Technology Laboratory (NETL).

1.1 PSDF Overview

The PSDF, located near Wilsonville, Alabama, was established to support the U.S. Department of Energy's effort to develop cost-competitive and environmentally acceptable coal-based power generation technologies. This effort promotes fuel diversity—a key component in maintaining national security—while meeting the highest environmental standards. The PSDF is developing environmentally friendly technologies that will allow the continued use of coal, the United States' most abundant and least expensive fuel source.

The PSDF is operated by Southern Company Services. Other project participants currently include the Electric Power Research Institute, KBR (formerly Kellogg Brown & Root), the Lignite Energy Council, and Peabody Energy. The facility is a highly flexible test center where researchers can evaluate innovative power system components on a semi-commercial scale at a low cost. Development of advanced power systems at the PSDF is focused specifically on identifying ways to reduce capital cost, enhance equipment reliability, and increase efficiency while meeting strict environmental standards. Current testing involves pressurized feed systems, coal gasifier optimization using a variety of fuels, sensor development, hot gas particulate removal, and advanced syngas cleanup.

1.2 Process Description

The PSDF gasification process, shown in Figure 1-1, features key components of an integrated gasification combined cycle (IGCC) plant. These include high pressure solids feed systems; a KBR Transport Gasifier; syngas coolers; a hot gas filter vessel, the particulate control device (PCD); continuous ash depressurization systems developed at the PSDF for ash cooling and removal; a novel piloted syngas burner; a slipstream syngas cleanup unit to test various pollutant control technologies; and a recycle syngas compressor.

The coal used as the gasifier feedstock is processed on site, first crushed and then pulverized to a nominal particle diameter between 250 and 400 microns. Coal may be fed to the gasifier using two systems, the original coal feed system and a secondary coal feed system. The original coal feed system is a lock hopper, horizontal pocket feeder design with a "rotofeed" dispenser. It consists of two pressure vessels, with the coal pressurized in the upper lock vessel and then

gravity fed into a dispense vessel, which is always pressurized. The material is fed out of the dispense vessel by the rotofeed dispenser, which is driven by a variable speed electric motor and delivers the material into the discharge line where it is conveyed by air or nitrogen into the gasifier. The secondary coal feeder is a developmental test unit designed to evaluate different feeder mechanisms. Types of mechanisms that can be tested with this system include auger-style, fluid bed, and a higher pressure rotary feeder. Coal is fed at a nominal rate of 4,000 lb/hr.

Figure 1-1. PSDF Gasification Process Flow Diagram.

A sorbent feeder is available to feed material into the gasifier for in-situ sulfur capture or to address ash chemistry issues. For sulfur capture, either limestone or dolomite is fed after being crushed and pulverized to a nominal particle diameter of 10 to 100 microns. The sorbent feeder utilizes the same design as the original coal feeder, but for a lower feed rate of nominally 100 lb/hr.

The start-up burner is a direct propane-fired burner operated to heat the gasifier to about 1,200°F. The burner is typically started at a system pressure of 60 psig, and can operate at pressures up to 135 psig.

The Transport Gasifier, a pressurized, advanced circulating fluidized bed reactor, consists of a mixing zone, riser, solids separation unit, seal leg, standpipe, and J-leg. The gasifier is equally capable of using air or oxygen as the gasification oxidant. Steam and either air or oxygen are mixed together and fed into the mixing zone at different elevations and orientations to evenly distribute heat generated from the partial combustion of the circulating solids. The oxygen from the air or pure oxygen feed is completely consumed in this section of the gasifier. The coal and sorbent are fed at a higher elevation in the mixing zone where the atmosphere is reducing, or oxygen-free.

As the coal devolatilizes and chemical reactions occur to generate syngas, the gas and solids move up the riser and enter the solids separation unit. This unit contains two solids separation devices, which use cyclonic action to remove particles. Between the first and second solids separation devices is the seal leg, which prevents backflow of solids. The solids collected by the solids separation unit are recycled back to the gasifier mixing zone through the standpipe and J-leg. The gasifier solids inventory is controlled by removing gasification ash through the continuous coarse ash depressurization (CCAD) system, which cools and depressurizes the solids. The nominal gasifier operating temperature is 1,800°F, and the gasifier system is designed to have a maximum operating pressure of 294 psig with a thermal capacity of about 41 MBtu/hr.

The syngas exits the Transport Gasifier, passes through the primary gas cooler where the gas temperature is reduced to about 750°F, and enters the PCD for final particulate removal. The metal or ceramic filter elements used in the PCD remove essentially all the particulate from the gas stream. The PCD utilizes a tube sheet holding up to 91 filter elements, which are attached to one of two plenums. Process gas flows into the PCD through a tangential entrance, around a shroud, and through the filter elements into the plenums. Failsafe devices are located downstream of the filter elements to stop solids leakage by plugging in the event of element failures. High pressure nitrogen back-pulsing, typically lasting 0.2 seconds, is used to clean the filters periodically to remove the accumulated gasification ash and control the pressure drop across the tube sheet. The solids fall to the bottom of the PCD and are cooled and removed through the continuous fine ash depressurization (CFAD) system.

After exiting the PCD, a small portion of the syngas, up to 100 lb/hr, can be directed to an advanced syngas cleanup system downstream of the PCD. The syngas cleanup system is a specialized, flexible unit, capable of operating at a range of temperatures, pressures, and flow rates, and provides a means to test various pollutant control technologies, including removal of sulfur, nitrogen, chlorine, and mercury compounds. The syngas cleanup slipstream can also be used to test other power technologies such as fuel cells.

A portion of the syngas can also be directed to the piloted syngas burner (PSB), a gas turbine combustor designed to burn coal-derived syngas with a lower heating value below 100 Btu/SCF. After syngas combustion in the burner, the flue gas passes through a four MW turbine before exiting the turbine stack. An associated generator can supply power from the turbine to the electric transmission grid.

The main stream of syngas is then cooled in a secondary gas cooler, which reduces the temperature to about 450°F. Some of this gas may be compressed and sent to the gasifier for

aeration to aid in solids circulation. The recycle gas compressor is a vertically mounted centrifugal compressor which operates at high temperature, nominally 500 to 600°F, and was designed for a throughput of about 2,000 to 3,000 lb/hr.

The remaining syngas is reduced to near atmospheric pressure through a pressure control valve. The gas is then sent to the atmospheric syngas combustor which burns the syngas components. The flue gas from the atmospheric syngas combustor flows to a heat recovery boiler, through a baghouse, and then is discharged out a stack. A flare is available to combust the syngas in the event of a system trip when the atmospheric syngas combustor is offline.

A brief description of the PSDF gasification testing history can be found in Appendix A.

1.3 Major Test Campaign Objectives

Evaluation of Bituminous Coal Operation. The PSDF gasification process operated for 237 hours in air-blown gasification mode using a total of about 225 tons of a Utah bituminous coal. Gasifier operations were challenging due to a number of factors, such as particle agglomeration; a large percentage of oversize coal particles (greater than 1,180 microns); lower overall gasifier solids collection efficiency; and degradation of refractory in the solids separation unit. The carbon conversion ranged from 75 to 95 percent, with the lower values attained during periods when the gasifier operating temperature was low due to limited solids circulation.

Coal Feeder Testing. The original coal feed system operated well after the conveying velocity was increased to address momentary plugging in the coal feed line caused by a high percentage of oversize particles. A developmental coal feeder, Pressure Decoupled Advanced Coal (PDAC) feeder, was successfully operated for 36 hours to feed coke breeze to the gasifier for start-up and re-starts after system trips. The PDAC feeder was installed at the bottom of the secondary coal feed system in place of a rotary table.

Continued Sensor Development. Development continued with real-time particulate monitors, the PCME DustAlert-90 and the Process Metrix Process Particle Counter. Both monitors operated acceptably. Thermowell material testing continued. Unfortunately, thermowells in the gasifier showed significant wear during TC24, especially in the riser section. The observed wear was much more than observed in any test campaign since the gasifier modifications in 2006.

Two test rods, composed of a ceramic blend of molybdenum, silicon, and boron, developed at Oak Ridge National Laboratory were tested in the riser during TC24. The test rods were exposed to 440 hours of solids circulation, including 274 hours of solids feed to the gasifier, when the temperature was nominally 1,825°F. Inspection of the rods showed significant wear similar to that observed on the thermowells in this area.

An infrared gas analyzer installed at the outlet of the WGS reactor provided real-time measurements of carbon monoxide (CO) and carbon dioxide (CO₂). Improvements to the gas chromatograph (GC) sampling system, which provided outlet WGS reactor concentrations of hydrogen (H₂), nitrogen (N₂), CO, CO₂, and methane (CH₄), increased the data points collected and reduced the required operating time.

Filter Element and Failsafe Testing. A new component of PCD testing was the addition of a higher collection efficiency version of the Pall Dynalloy HR-160 sintered metal fiber filter elements tested previously. The new filter elements, constructed with a significantly finer fiber size than the previous Dynalloy version, demonstrated good collection efficiency during operation. Inspection and flow testing of the new elements following TC24 showed no indication of corrosion or high pressure drop across the element media.

Advanced Syngas Cleanup Testing. A system utilizing catalytic filter elements for the WGS reaction was developed at the PSDF and successfully tested with syngas for about 40 hours during TC24. To evaluate the performance of the catalytic filter elements, several of the operating parameters were varied, including inlet temperature, pressure, face velocity, and total water-to-CO molar ratio. Under these operating conditions, a wide range of CO conversion, from about 5 to 94 percent, was achieved.

Slipstream Testing by Outside Researchers. A hydrogen selective carbon molecular sieve (CMS) membrane developed by researchers at MPT in cooperation with NETL was installed at the PSDF to evaluate the material stability when utilized with coal-derived syngas. The membrane had been extensively tested at the NETL facility in Pittsburgh, PA, using bottled gases to simulate syngas. Some commissioning activities were completed during TC24, including safely integrating the membrane test unit with the PSDF slipstream unit. Functional tests were also completed. Completion of commissioning activities and initial testing is planned for the next test campaign.

NETL completed installation and shakedown testing of the Fuel Cell Module, which contained 12 individual solid oxide fuel cells. Shakedown testing was conducted with hydrogen for over 100 hours. Five of the cells operated well, although a sealing problem hindered operation of the remaining cells. Modifications to the sealing method needed to achieve 100 percent cell operation were identified during the testing, as were several other design improvements.

1.4 Report Structure

The following report presents the operational data and results of gasification technology development at the PSDF during TC24, compiled in the sections listed below.

- Section 2 Solids Feed — Presents coal characteristics, operation of the original coal feed system and the PDAC feeder operation with coke breeze. Gives coal moisture values and particle sizes and their effect on coal feed system performance.
- Section 3 Transport Gasifier —Includes the major gasifier operating parameters and the gasifier performance as indicated by solids and gas analyses.
- Section 4 Sensor Development — Discusses testing of real-time particulate monitors and results of gasifier instrumentation and gas analysis improvements.
- Section 5 Particulate Control Device — Describes the hot gas filter particulate characteristics, PCD performance, and failsafe and filter element testing.

Section 6   Advanced Syngas Cleanup — Describes testing of the catalytic filter elements, the NETL fuel cell module, and the MPT hydrogen membrane.

Section 7   Support Equipment — Describes operation of the start-up burner and recycle gas compressor.

Section 8   Conclusions — Summarizes the major conclusions and lessons learned from TC24 operation.

Appendix A gives a brief history of gasification operation at the PSDF. Appendix B shows the steady state operating periods and the major system operating conditions for each period. Appendix C details the solids and gas sampling and analysis performed to assess PSDF process performance. Material and energy balances are shown in Appendix D, and Appendix E lists the abbreviations and units used in this report.

2.0 SOLIDS FEED

During TC24, the original coal feed system and a developmental system, the PDAC feed system were operated. Like the original feeder, the PDAC system is a lock hopper-based feeder, but uses conveying gas flow to control the solids feed rate. The original coal feed system was used to feed the bituminous coal, and the PDAC system was used to feed coke breeze to the gasifier.

2.1 Coal Characteristics

The coal used exclusively during TC24 was a western bituminous coal from the Dugout Canyon Mine located in Wellington, Utah. The composition was typical for a bituminous coal classified as High Volatile C, with an as-received heating value of about 12,800 Btu/lb and moisture content of about 4 percent. Due to the low moisture content of the coal, little moisture removal occurred during the milling process. Table 2-1 shows the composition and heating value of the coal as sampled from the original coal feeder. Hydrogen in the coal was reported separately from hydrogen in moisture.

Table 2-1. As-Fed Utah Bituminous Coal Characteristics.

	Average Value	Minimum Value	Maximum Value	Standard Deviation
Moisture, wt %	2.1	1.9	2.5	0.2
Carbon, wt %	73.1	70.2	74.4	0.9
Hydrogen, wt %	5.0	4.9	5.1	0.0
Nitrogen, wt %	1.4	1.3	1.4	0.0
Oxygen, wt %	7.6	7.3	7.9	0.2
Sulfur, wt %	0.5	0.5	0.6	0.0
Ash, wt %	10.2	9.1	13.3	1.0
Volatiles, wt %	37.1	35.7	38.6	1.1
Fixed Carbon, wt %	50.5	48.3	52.1	1.2
Heating Value, As Received, Btu/lb	12,911	12,482	13,153	151
CaO, wt % in Ash	5.9	5.2	6.1	0.3
SiO ₂ , wt % in Ash	58.2	57.7	60.0	0.9
Al ₂ O ₃ , wt % in Ash	18.2	17.7	18.5	0.3
MgO, wt % in Ash	1.5	1.4	1.6	0.1
Na ₂ O, wt % in Ash	1.2	1.0	1.4	0.1

The mass median diameter (MMD) and Sauter mean diameter (SMD) particle sizes of the coal sampled from the original coal feeder are shown in Figure 2-1. The coal particle size varied from 300 to 530 microns MMD and from 180 to 245 microns SMD, except for one sample, which measured 830 microns MMD and 390 microns SMD.

Figure 2-1. Coal Particle Sizes.

Figure 2-2 shows the percentage of oversize coal (above 1,180 microns), and the percentage of fine coal (below 45 microns). The oversize particles ranged between 7 and 33 weight percent of the coal, much higher than previous operation with bituminous coal due to removal of a top size screen in the coal mills. Fines concentrations were acceptable at less than 15 percent.

Figure 2-2. Coal Fines and Oversize Particles.

2.2 Original Coal Feeder Operation

The original coal feeder operated throughout TC24, achieving rates up to 4,000 lb/hr. The Spheri valves on the original feeder cycled over 2,000 times without failure. The feed system operated reliably after initial problems with the discharge line plugging were addressed. Increasing the conveying velocity from 40 to 50 ft/s allowed for smoother operation and prevented plugging in the feeder discharge line. Nitrogen was the coal conveying gas for the duration of TC24.

Coal Feeder Operating Envelope. Based on TC24 operation, the original coal feed system operating range of coal moisture content and particle size was evaluated. Figure 2-3 shows the variation in particle size and coal moisture contents as compared to the previously defined operating envelope for test campaigns TC20 through TC23. TC24 operation was problematic when the particle size was greater than 500 microns, even at moisture contents as low as 2 percent due to the existing hardware. This was due in part to a high percentage of oversize particles (greater than 1180 microns) in the feed material.

Figure 2-3. Coal Feeder Operating Envelope.

Figure 2-4 compares the percentage of oversize particles over a range of particle sizes for the bituminous coals tested in TC09, TC17 and TC24 and the Power River Basin subbituminous coal tested in TC20. During TC24, the particle size at times was greater than 400 microns MMD, and the percentage of oversize particles exceeded 15 percent, which was higher than in previous tests.

Figure 2-4. Coal Oversize Content versus Particle Size.

2.3 Pressure Decoupled Advanced Coal Feeder Operation

The first on-line operation of the PDAC feeder occurred in TC24. Installed to support high pressure coal feeder development, the PDAC feeder is a non-mechanical feed control device with no moving parts which combines some of the successful concepts developed with the PSDF continuous ash depressurization systems with traditional designs for flow rate control. The driving force for solids flow is a pressure differential, and the solids flow is metered by the nitrogen conveying gas.

The PDAC feeder concept was first tested in the PSDF cold flow unit, and following this initial concept validation, the system was scaled up to a size meaningful for testing in the PSDF gasification process. The feed device was installed on the secondary coal feeder and was first commissioned in an off-line test system which allowed testing of dry coal feed systems at operating pressures up to 500 psig and coal feed rates up to 20,000 lb/hr.

During TC24, the PDAC feed system was successfully operated for 36 hours, feeding coke breeze to the gasifier during start-up and hot re-starts. Coke breeze was fed to heat the gasifier from the maximum start-up burner temperature of 1,200°F to a temperature high enough for coal feed (about 1,700°F for the bituminous coal). Table 2-2 gives the composition, ash fusion temperature, and average particle size of the coke breeze.

The PDAC system operated well, feeding about 20 tons of coke breeze to the gasifier at relatively low feed rates of about 1,100 lb/hr. The controls system operated well, with the feed rate responding as desired to changes in the conveying nitrogen flow rate. During initial operation, several trips occurred due to a problem with the interlock logic. Logic modifications prevented further trips, and thereafter, the feed system was always available for operation.

Table 2-2. As-Fed Coke Breeze Characteristics.

	Value
Moisture, wt %	0.5
Ash, wt %	8.8
Fixed Carbon, wt %	90.7
CaO, wt % in Ash	3.4
SiO ₂ , wt % in Ash	48.6
Al ₂ O ₃ , wt % in Ash	23.0
Fe ₂ O ₃ , wt % in Ash	17.5
Na ₂ O, wt % in Ash	0.7
Initial Ash Fusion Temperature in Reducing Environment , °F	2,106
Particle Size (MMD), microns	270

3.0 TRANSPORT GASIFIER

Prior to TC24, the modified gasifier had only been operated with the low-rank fuels, subbituminous and lignite coals. Characterizing gasifier operation with bituminous coal was a major test objective for TC24, and operation with this coal was expected to differ in several respects. For example, since bituminous coals are less reactive relative than subbituminous and lignite coals, higher gasifier operating temperatures are required to achieve comparable carbon conversions. In addition, due to the lower reactivity, bituminous coals may produce higher concentrations of organics in the syngas if sufficient energy is not supplied to complete the hydrocarbon cracking reactions during the gasification process.

Fully assessing gasifier operation with the Utah bituminous coal was precluded by various factors. Gasifier operation was complicated by a high percentage of oversize coal particles, refractory degradation in the first solids separation device, and poor solids circulation. In addition, problems with support equipment, including the start-up burner and the waste heat boiler caused delays in system start-ups. Due to these challenges, the test campaign extended over a month and consisted of three distinct operating periods, designated as Parts A, B, and C (TC24A, TC24B, and TC24C).

Start-up of TC24A began on February 12, 2008, although coal feed was not initiated until February 16, 2008, due to problems lighting the start-up burner. The system was shut down on February 18, 2008, due to a high PCD inlet temperature caused by fouling in the primary gas cooler. Operational data indicated that the primary gas cooler fouling was caused by tar formation during start-up. To prevent fouling in subsequent start-ups, coke breeze was fed to bring the gasifier to a higher temperature before initiating coal feed.

Start-up of TC24B began on February 27, 2008, but was delayed by a leak in the waste heat boiler downstream of the atmospheric syngas combustor. The leak was repaired, and coke breeze feed and later coal feed were started on March 3, 2008. On the same day, a blockage formed in the seal leg, necessitating system shutdown. During the inspection, several large pieces of dislocated refractory were found, and samples from the gasification ash remaining in the gasifier showed some agglomeration.

TC24C started on March 8, 2008, with coke breeze feed and then coal feed begun on March 10, 2008. The system was shut down on March 19, 2008, due to operating problems caused by a blockage in the mixing zone.

There were sixteen steady state operating periods during TC24, five during TC24A and eleven during TC24C. All of the steady state periods were in air-blown gasification mode with bituminous coal feed. Recycle syngas operation was achieved in all the TC24C operating periods. The steady state periods are defined based on maintaining gasifier operating conditions within defined ranges. The steady state operating periods and major operating parameters are shown in Appendix B. Gasifier performance was evaluated through extensive gas and solids sampling and analyses. The details of the sampling locations and methods are given in Appendix D.

3.1 Transport Gasifier Operations Part A

TC24A lasted only 49 hours due to fouling in the primary gas cooler which caused higher than normal temperature of syngas entering the PCD. Since the syngas temperature at the PCD inlet was above the design limit of some of the PCD internal components, ambient temperature nitrogen was added directly to the PCD inlet gas to reduce the temperature. Gasification operation was continued with nitrogen cooling to determine if the primary gas cooler performance would improve over time. The gas cooler performance stabilized but did not improve, so the process was shut down. It was determined that the primary gas cooler fouling was caused by tar deposition on the heat exchange surface during the low temperature operation at start-up. The primary gas cooler heat transfer coefficient (U) during TC24A is plotted in Figure 3-1. The figure shows the immediate loss of heat transfer below the design of $40.85 \text{ BTU/hr}\cdot\text{ft}^2\cdot^\circ\text{F}$ during the first three hours of coal feed.

Figure 3-1. TC24A Primary Gas Cooler Heat Transfer.

During a typical start-up of the PSDF gasification process, a propane fired start-up burner is fired to heat the gasifier and circulating solids to about $1,200^\circ\text{F}$ before coal feed is initiated. The coal feed rate is minimal as the gasifier temperature increases and the O_2 content in the flue gas decreases. The coal feed rate is gradually increased to maintain the rate of increase in gasifier temperature while the O_2 and CO content are closely monitored. The coal feed rate and air flow rate are adjusted to maintain the O_2 concentration below 2 mole percent while in an oxidizing environment and the CO concentration below 2 mole percent in a reducing environment. When the gasifier temperature reaches about $1,650^\circ\text{F}$, the start-up burner is shut down, and the coal feed rate is increased to transition into gasification mode. With lower reactivity coals, the possibility of tar formation is high during the transition from an oxidizing to a reducing environment due to coal addition at the lower temperature. Although the Utah coal was classified as High Volatile and considered relatively reactive for a bituminous coal, tar formation

during the transition to gasification mode was high enough to result in heat exchanger fouling as the tars condensed.

3.1.1 Operating Parameters

Figure 3-2 gives the gasifier temperature and pressure, and the primary gas cooler outlet temperature data during TC24A steady state periods. The mixing zone temperature ranged from 1,835 to 1,847°F, the gasifier outlet temperature ranged from 1,726 to 1,738°F, and the primary gas cooler outlet temperature ranged from 1,041 to 1,062°F. The gasifier outlet pressure was held constant at 200 psig for all of these steady state periods. The primary gas cooler outlet temperature was notably higher than usual during all of the steady state operating periods due to the fouling that occurred during start-up.

Figure 3-2. TC24A Gasifier and Primary Gas Cooler Operating Parameters.

Flow rates of the major feed streams to the gasifier during TC24A are shown in Figure 3-3. The coal feed rates were calculated from the feeder weigh cells, and the air, nitrogen, and steam flow rates were taken from flow indicators. The reported nitrogen flow rates were decreased from the flow indicator rates by 1,500 lb/hr to account for ash transport nitrogen. The coal feed rate was maintained at a low rate, approximately 1,200 lb/hr, because of the reduced heat transfer in the primary gas cooler previously discussed. The steam rate was held constant at an initial start-up rate of 1,300 lb/hr. Generally, steam consumption is minimized; however, the short duration of this portion of the test did not allow for optimization. The air rate was maintained at nominally 7,300 lb/hr to control the gasifier temperature, and the nitrogen rate was approximately 7,700 lb/hr, which is higher than in typical operation. Higher nitrogen concentrations occur when nitrogen is used for gasifier aeration and coal transport, and during times of low coal feed rate, since the amount of nitrogen used for gasifier instrument purges is independent of the coal feed rate. Any nitrogen added to the gasifier requires heat of combustion to heat it to gasifier temperature, so the air-to-coal ratio is increased with increased nitrogen use. As a result of the

high nitrogen flow rate and low coal feed rate, the air to coal ratio was higher than usual, ranging from 6.1 to 6.5 lb/lb.

Figure 3-3. TC24A Flow Rates to Gasifier.

Figure 3-4 shows the standpipe levels (measured as differential pressures) and the riser differential pressure. The standpipe level was held nearly constant at about 160 inH₂O, and the riser differential pressure was also fairly constant at about 100 inH₂O.

Figure 3-4. TC24A Gasifier Differential Pressures.

3.1.2 Gasifier Performance, Solids Analysis

The gasifier solids chemical composition and particle size analyses presented in the following sections represent both the circulating gasifier solids sampled from the gasifier standpipe and the solids exiting the gasifier, filtered in the PCD, and sampled from the CFAD system.

Solids Chemical Analyses. The solids chemical analyses were used to monitor transition of the solids inventory from the start-up bed material, sand, to gasification ash and to characterize operation of the gasifier solids collection devices. The chemical analyses of the gasifier circulating solids as sampled from the standpipe and the PCD solids sampled from the CFAD system are presented in Table 3-1. The gasifier circulating solids were composed mainly of silicon dioxide (SiO_2) from the start-up bed material during TC24A since the coal feed rate was low and the operating duration was short. The carbon conversion was high; thus, the gasifier circulating solids organic carbon content was low and the solids heating value was essentially zero. Only one PCD sample was taken for analysis due to the short duration of operation. This sample was mainly composed of SiO_2 (about 35 percent) and organic carbon (about 36 percent). The heating value of the PCD solids was about 4,600 Btu/lb.

Table 3-1. TC24A Gasifier Circulating and PCD Solids Analyses.

	Gasifier Circulating Solids				PCD Solids
	Average	Standard Deviation	Minimum Value	Maximum Value	Value
SiO_2 , wt%	79.3	2.3	76.6	80.8	34.9
Al_2O_3 , wt%	10.3	1.2	9.0	11.3	12.6
Fe_2O_3 , wt%	1.9	0.6	1.5	2.5	2.1
Other Inerts (P_2O_5 , Na_2O , K_2O , BaO , TiO_2), wt%	3.6	0.7	3.2	4.4	3.7
CaS , wt%	0.2	0.1	0.1	0.3	0.8
CaO , wt%	3.4	0.8	2.7	4.2	3.6
MgO , wt%	0.8	0.2	0.6	1.0	1.5
Carbon, wt%	2.2	0.1	2.1	2.4	35.5
Heating Value, As Received, Btu/lb	< 100	---	---	---	4,588

Solids Physical Analyses. The TC24A particle sizes (SMD and MMD) of the gasifier circulating solids and PCD solids are shown in Figure 3-5. The SMD of the gasifier circulating solids gradually decreased to about 125 microns, and the MMD of the gasifier circulating solids decreased to about 120 microns as gasification ash replaced a portion of the initial bed inventory of sand, which has an MMD of about 145 microns. The PCD solids SMD was about 10 microns and the MMD was about 14 microns, confirming that the gasifier solids collection devices were effective in retaining the larger particle sizes, allowing only very fine material to flow to the PCD.

Figure 3-5. TC24A Particle Sizes of Gasifier Circulating Solids and PCD Solids.

The average particle size distributions (PSDs) for the gasifier circulating solids and the PCD solids during TC24A are provided in Figure 3-6. The range of PSD data for TC24A was relatively narrow.

Figure 3-6. TC24A PSD Curves for Gasifier Circulating Solids and PCD Solids.

Bulk densities of the gasifier circulating solids and PCD solids are shown in Figure 3-7. The bulk density of the circulating solids gradually decreased from 93 lb/ft³, the bulk density of the start-up sand, to 86 lb/ft³ due to the replacement of bed start-up material with gasification ash.

The rate of sand replacement and thus rate of density decrease was slower than typical due to the low coal feed rate. The PCD solids bulk density varied from about 16 to 21 lb/ft³.

Figure 3-7. TC24A Bulk Densities of Gasifier Circulating Solids and PCD Solids.

Figure 3-8 presents optical microscope photographs of the gasifier circulating solids. The solids were fairly uniform in size and color and did not show indications of particle agglomeration.

Figure 3-8. Photomicrographs of TC24A Gasifier Circulating Solids.

Gasification Ash Removal. Figure 3-9 shows the solids rates for the fine gasification ash removed from the PCD by the CFAD system and for the coarse gasification ash removed from the gasifier by the CCAD system. The PCD solids rates were determined from the in-situ sampling at the PCD inlet, and the ash removal rates for CCAD were determined by a system ash balance. The CFAD system discharge rate averaged 92 lb/hr. The CCAD discharge rate averaged 55 lb/hr,

which accounted for about 37 percent of the total gasification ash removal rate. The ash removal rates were relatively low due to the low coal feed rates.

Figure 3-9. TC24A Gasification Ash Removal from the Gasifier and PCD.

3.1.3 Gasifier Performance, Gas Analysis

Continuous extractive syngas sampling was performed between the primary gas cooler and the PCD inlet, and the syngas constituents were analyzed using continuous analyzers and gas chromatography. Manual in-situ samples of syngas moisture were made at the PCD outlet during the particulate sampling.

Syngas Composition. Concentrations of the major syngas components for the steady state operating periods are given in Figure 3-10. The concentrations of H_2 , CO, CO_2 , CH_4 and H_2O are shown on a molar basis. The H_2 , CO, CO_2 , and CH_4 concentrations were measured by a GC on a moisture free basis and converted to wet gas concentrations using the water concentration. The water concentration for steady state periods was estimated based on the PCD outlet sampling and a mathematical correlation based on the water-gas shift reaction equilibrium. The CO and H_2 concentrations were low, at less than 3 percent, because of the low coal feed rate and a relatively high nitrogen concentration.

Figure 3-10. TC24A Concentrations of Major Syngas Components.

Minor constituents in the syngas include reduced sulfur compounds such as hydrogen sulfide (H₂S), carbonyl sulfide (COS), and carbon disulfide (CS₂), and reduced nitrogen compounds such as NH₃ and hydrogen cyanide (HCN). Some of the sulfur (typically less than 15 percent) is captured in the solid phase by forming compounds with the calcium in the coal ash, and the remaining gas phase sulfur is mostly in the form of H₂S. The H₂S concentration ranged from about 265 to 302 ppm on a wet molar basis. Ammonia concentrations were not obtained during TC24A since the FTIR was being used to support the gas cleanup unit test.

Syngas Heating Value. The syngas lower heating value (LHV) was very low at around 24 Btu/SCF on a wet basis for the steady state periods during TC24A due to the low coal feed rate and high nitrogen concentration.

Carbon Conversion. The carbon conversion was about 96 percent for all of the steady state periods during TC24A.

Gasification Efficiency. The cold gasification efficiency was 32 percent, and the hot gasification efficiency was 77 percent. Both were lower than typical due to the low coal feed rates, which produced lower than typical syngas heating values.

3.1.4 Gasifier Inspections

The primary gas cooler was inspected after the system was shut down. Figure 3-11 shows the primary gas cooler inlet during inspection and a ceramic ferrule that was removed. A light, fairly loose powder coated the inside of the ceramic ferrule, and a dark coating was evident below this solids coating. The combination of tar and solids deposition apparently resulted in the loss of heat transfer.

Figure 3-11. Post-TC24A Inspection of Primary Gas Cooler Inlet.

Figure 3-12 shows the shell side (water side) of the primary gas cooler. Inspections of the shell side showed the tubing and baffle arrangement was clean and intact as designed, confirming that heat transfer limitations were not related to the water side of the syngas cooler.

Figure 3-12. Post-TC24A Inspection of Primary Gas Cooler Shell Side.

3.2 Transport Gasifier Operations Part B

After the primary gas cooler was cleaned and reinstalled, the system was restarted. However, on February 27, 2008, during start-up of the support equipment in preparation for coal feed, a tube leak developed in the waste heat boiler. Figure 3-13, shows the atmospheric syngas combustor and waste heat boiler with an arrow noting the general area of the leak. The waste heat boiler had operated over 27,000 hours since the initial start-up in 1996 without any leaks. The leaks were repaired, and start-up of the process resumed.

Figure 3-13. Atmospheric Syngas Combustor and Waste Heat Boiler.

Coke breeze feed to the gasifier began on March 1, 2008, increasing the gasifier temperature to about 1800°F before starting coal feed. Gasification operation was stable until a blockage occurred in the gasifier, and solids circulation was severely hindered. The location of the blockage, shown in Figure 3-14, occurred in the bottom of the first solids separation device and the inlet of the seal leg.

Figure 3-14. TC24B Seal Leg Blockage Location.

Figure 3-15 is a plot of the seal leg temperatures and differential pressures on the morning of March 3, 2006, when the solids circulation became restricted. The seal leg temperatures and differential pressures rapidly decreased due to the loss of solids flow into the seal leg. Attempts to clear the blockage and reestablish solids flow to the seal leg were unsuccessful, so the system was shut down.

Figure 3-15. TC24B Seal Leg Temperatures and Differential Pressures.

After the system was sufficiently cool, the gasifier was opened and solids were removed from the first solids separation device. During the solids removal process, several pieces of separated refractory were found. The two largest pieces recovered are shown in Figure 3-16 and were about 6 inches long, 8 inches wide and 2.5 inches thick. The refractory condition at the inlet of the first solids separation device was of particular concern due to the large refractory pieces found and the previous loss of refractory in this area found after TC23, as noted in the TC23 Topical Report.

Figure 3-16. Recovered Refractory Pieces from Post-TC24B Inspection.

Inspection of the first solids separation device revealed that the refractory loss at the inlet had progressed. Figure 3-17 shows the progression of refractory loss at the inlet from post-TC23 to post-TC24B.

Figure 3-17. Inlet of the First Solids Collection Device.

During the gasifier modifications in 2006, Actchem VC refractory was installed in the first solids separation device to evaluate its performance at these conditions. The Actchem VC properties include a high erosion resistance and high coefficient of thermal expansion, which make it resistant to wear but susceptible to cracking. The Actchem VC refractory life is heavily

dependent on the number of thermal cycles and is rated for about 30 thermal cycles (a 10 year commercial life). The thermal cycles for the Actchem VC refractory prior to TC24 were calculated as 11 thermal cycles, almost half of the estimated useful life. A full thermal cycle is defined as a temperature increase from ambient temperature to 1800°F combined with a decrease in temperature below 200°F at a rate of 100°F/hr. A half temperature cycle was designated as a temperature decrease below 1000°F at a rate of 100°F/hr. During TC24A and B, there were 2 thermal cycles, bringing the total to 13 thermal cycles at the conclusion of TC24B. Short trips where the temperature did not decrease below 1,000°F and the temperature rate of change less than 100°F/hr also impact the true equivalent thermal cycles.

Additional findings from the inspection were agglomerated particles in the solids that were removed from the first solids collection device. The agglomerates were fairly soft and easily broken apart. Laboratory testing and solids analysis were performed to determine the cause of the agglomeration. Four different mixes of coke breeze and sand (10, 15, 20, and 25 weight percent coke breeze mixed with sand) were baked at 1,825°F in a standard laboratory furnace. There was no consolidation in these samples after baking; however, a solids sample taken from the circulating solids in the standpipe in a subsequent start-up was almost completely consolidated when it was baked in the furnace at 1,875°F, as shown in Figure 3-18.

Figure 3-18. TC24B Gasifier Solids Sample after Baking at 1,875°F.

The solids sample from the standpipe was examined with a scanning electron microscope (SEM), and an Energy Dispersive X-Ray Spectroscopy (EDS) was performed. The SEM image, Figure 3-19, shows two particles, Particle One (P1) and Particle Two (P2) and the area between the particles where bonding occurred. The EDX analyses for P1, P2, and the area between the particles are shown in Figure 3-20. The only component noted that might have bonded the particles together was potassium. There were small amounts of potassium coating the sand particles that might have formed some potassium silicates which have adhesive properties that could have bonded some of the particles together.

Figure 3-19. SEM Image of Gasifier Solids.

Figure 3-20. EDS Analysis of Gasifier Particles P1 and P2 and the Area between the Particles.

Based on the inspections and solids analysis, the blockage formation was attributed to the combination of the refractory pieces partially blocking the solids outlet of the first separation device and particle agglomeration. The refractory pieces initially fell and partially blocked the solids flow into the seal leg. Uneven gas and solids flow, local hot spots, and the sand and coke breeze chemical interactions caused the agglomeration of particles, which then fully blocked the solids flow in the seal leg.

3.3 Transport Gasifier Operations Part C

After a thorough gasifier inspection and a comprehensive review of the refractory integrity were completed, the system was restarted. A monitoring program was established to take skin temperature measurements with an infrared gun to obtain point-specific temperature readings and to perform thermal scans with an uncooled radiometric camera to generate high resolution thermal images of the inlet to the first solids separation device. The gasifier temperature ramp rate was slower than typical due to concerns over the refractory condition. Coke breeze feed was initiated on March 10, 2008. Unfortunately, a partial blockage in the seal leg occurred within a few hours. Figure 3-21 shows the seal leg temperatures and differential pressures. Similar to the previous blockage, a rapid decrease in the temperature and differential pressures occurred. Gas flows were varied to dislodge the material, and the blockage was successfully cleared; as seen by the increase in loop seal temperatures and differential pressures in Figure 3-21 at around 16:20.

Figure 3-21. Seal Leg Temperatures and Differential Pressures on March 10.

Coke breeze feed was re-started later that day and followed by the addition of coal to the gasifier designated as Hour 50. Process operations were stable during the transition from coke breeze to coal feed and during the first few days of gasification operation. However, after a few hours of coal feed the air flow to the mixing zone below the J-leg entry, referred to as the lower mixing zone (LMZ), became restricted, forcing a lower percentage of the total air to be added into the LMZ. Figure 3-22 shows the decline in the percentage of air added to the LMZ for the first 100 hours of operation after coal feed was started.

Figure 3-22. TC24C Air Flow to Lower Mixing Zone.

At Hour 74, about 24 hours after coal feed was started, a temperature measurement in the LMZ dropped below the actual operating temperature. Several other temperature measurements in the LMZ began erroneously indicating low temperatures over the subsequent days of operation at Hour 112 and 225. The temperature trends in the LMZ during the first 100 hours of operation are plotted in Figure 3-23.

Figure 3-23. TC24C Lower and Upper Mixing Zone Temperatures.

Temperature measurement losses also occurred in the mixing zone above the J-leg entry, designated as the upper mixing zone (UMZ). The temperature indications in the UMZ and riser

from Hour 50 to 200 are plotted in Figure 3-24. In addition, the gasifier circulating solid samples showed a marked change in particle size and appearance as operation progressed. Section 3.3.2 discusses these changes in the solids physical properties.

Figure 3-24. TC24C Upper Mixing Zone and Maximum Gasifier Temperatures.

The erroneously low temperature indications throughout the gasifier mixing zone and the appearance of the circulating solids in the gasifier, made evident that particle agglomeration was occurring in the gasifier. The particle agglomeration was likely caused by high carbon content in the LMZ, since the gas velocities are relatively low in this region, and high carbon content can cause localized hot spots. Carbon in the LMZ could have come from the coke breeze and/or the bituminous coal. The addition of coke breeze at start-up and at times where coal feed was interrupted contributed to a high carbon content in the circulating solids. The low carbon conversion of the bituminous coal also contributed to the high carbon content. Chemical contamination of the coke breeze may have also contributed to the agglomeration.

The agglomeration in the LMZ likely occurred within the first few hours of coal feed addition. The agglomeration grew and eventually restricted the air flow to the LMZ. This then resulted in a non-uniform distribution of air flow into the gasifier, which compounded the potential for hot spots in the UMZ with the high carbon content circulating solids. .

Another indication of degrading gasifier operation was the decrease in solids circulation rate and the increase in the ratio of UMZ and riser differential pressures. Figure 3-25 plots relative circulation rate and the ratio of the differential pressures in the UMZ and riser. The circulation rate steadily dropped from Hour 50 to 200 and decreased significantly at Hours 200 and 230.

Figure 3-25. TC24C Ratio of Mixing Zone and Riser Differential Pressures.

At around Hours 200 and 225, there was a significant increase in the gasifier temperature differential (DT), calculated as the difference in the maximum gasifier temperature and the gasifier outlet temperature, which is normally about 100°F. Poor solids circulation, as indicated by the high gasifier temperature differential, resulted in low riser temperatures which then led to tar formation and deposition in the primary gas cooler. A high percentage of oversize coal particles compounded the problem, as the larger coal particles have a greater tendency to form tars since they require additional residence time and temperature to be completely converted into syngas. The gasifier and the primary gas cooler DTs are plotted in Figure 3-26.

Figure 3-26. TC24C Gasifier and Primary Gas Cooler Differential Temperatures.

The primary gas cooler performance decreased due to tar deposition on the heat transfer surfaces, causing the primary gas cooler temperature differential to decrease significantly at around Hour 200. The resulting high PCD inlet temperature forced a process shutdown on March 19, 2008.

3.3.1 Operating Parameters

Despite the operating challenges, there were 11 steady state operating periods in TC24C; however, there was only one after Hour 200 due to the increasing agglomeration in the gasifier. Figure 3-27 gives the gasifier temperature and pressure, and the primary gas cooler outlet temperature during TC24C steady state periods. The mixing zone temperature ranged from 1772 to 1,905°F and the gasifier outlet temperature ranged from 1,756 to 1,825°F. The gasifier temperatures were varied to quantify the effect of temperature on tar formation. The primary gas cooler operated as designed, with an outlet temperature below 1,000°F for the majority of TC24C; however, the temperature increased significantly around Hour 200 (reaching 1,126°F) as the gasifier temperature changed and the cooler performance declined. The gasifier outlet pressure was varied from 170 to 204 psig to maintain the desired velocities in the gasifier.

Figure 3-27. TC24C Gasifier and Primary Gas Cooler Operating Parameters.

Flow rates to the gasifier during TC24C are shown in Figure 3-28. Coal feed rates were calculated from a system carbon balance since the feeder weigh cells were not reading properly, and the air, nitrogen, steam, and recycle gas flow rates were taken from flow indicators. The reported nitrogen flow rates were reduced from the indicator rates by 500 lb/hr to account for ash transport nitrogen. The coal feed rate was decreased significantly during last operating period to reduce the load on the primary gas cooler and thereby help moderate the PCD inlet temperature. Steam rates were varied based on localized cooling requirements, and the recycle gas flow rate was held nearly constant.

Figure 3-28. TC24C Flow Rates to the Gasifier.

Figure 3-29 shows the standpipe levels (measured as differential pressures) and the riser differential pressure. The standpipe level varied from 105 to 175 inH₂O, and the riser differential pressure varied from 14 to 44 inH₂O. Standpipe level variations were dependent upon CCAD operation. Flow restrictions at the inlet of the CCAD system prevented steady control of the standpipe level. Under normal operating conditions, the riser differential pressure has a positive correlation with the standpipe level; however, during TC24C the riser differential pressure gradually decreased throughout the test campaign even when the standpipe level increased. The riser differential pressure was also notably lower in TC24C than in TC24A likely due to the decrease in solids circulation rate.

Figure 3-29. TC24C Gasifier Standpipe and Riser Differential Pressures.

3.3.2 Gasifier Performance, Solids Analysis

Solids Chemical Analyses. The solids chemical analyses were used to evaluate the gasifier operations and performance of the solids collection devices. The chemical analyses of the gasifier circulating solids ash for TC24C are given in Table 3-2. The carbon content of the circulating solids was as high as 45 percent, higher than in any previous testing. Typically, the carbon content is less than 1 percent, and the solids heating value is negligible, but in TC24C, the high carbon content resulted in a heating value of about 6,600 Btu/lb. Between Hours 50 and 150, the carbon content increased from 14 to 45 percent, were then between 30 and 35 percent, and then decreased to 12 percent. The higher carbon content was due in part to the addition of coke breeze during startup and during coal feed interruptions. The other main constituents, SiO₂ and alumina dioxide (Al₂O₃), were similar in concentration to that of the coal ash.

Table 3-2. TC24C Gasifier Circulating Solids Analysis.

	Average	Standard Deviation	Minimum Value	Maximum Value
SiO ₂ , wt%	50.5	8.2	41.6	61.3
Al ₂ O ₃ , wt%	13.7	2.4	11.1	16.2
Fe ₂ O ₃ , wt%	3.1	0.7	2.3	3.7
Other Inerts (P ₂ O ₅ , Na ₂ O, K ₂ O, BaO, TiO ₂), wt%	4.7	1.0	3.6	5.6
CaS, wt%	0.5	0.2	0.3	0.9
CaO, wt%	4.4	1.4	3.0	5.8
MgO, wt%	1.2	0.3	0.9	1.5
Carbon, wt%	19.8	9.8	7.5	34.1
Heating Value, As-Received, Btu/lb	2,751	1,633	565	5,100

The chemical analyses of the PCD solids for TC24C are given in Table 3-3. The PCD solids were composed mainly of carbon. The carbon content, much higher than in TC24A, was about 78 percent until Hour 125, and then decreased to about 70 percent until Hour 191. The heating value of the PCD solids was as high as 11,600 Btu/lb.

Table 3-3. TC24C PCD Solids Analysis.

	Average	Standard Deviation	Minimum Value	Maximum Value
SiO ₂ , wt%	16.9	4.8	11.7	24.2
Al ₂ O ₃ , wt%	6.4	1.7	4.6	9.1
Fe ₂ O ₃ , wt%	1.2	0.3	0.9	1.7
Other Inerts (P ₂ O ₅ , Na ₂ O, K ₂ O, BaO, TiO ₂), wt%	1.6	0.5	1.2	2.3
CaS, wt%	1.0	0.1	0.8	1.3
CaO, wt%	1.2	0.5	0.6	1.9
MgO, wt%	0.4	0.1	0.3	0.5
Carbon, wt%	70.9	5.4	56.3	77.6
Heating Value, As-Received, Btu/lb	10,456	712	8,464	11,601

Solids Physical Analyses. The TC24C particle sizes of the gasifier circulating solids and PCD solids are shown in Figure 3-30. The MMD of the gasifier circulating solids varied from 100 to 275 microns while the SMD of the gasifier circulating solids varied from about 100 to almost 500 microns. The PCD solids MMD and SMD steadily decreased during the test campaign from about 23 to 15 microns and 17 to 10 microns, respectively.

Figure 3-30. TC24C Particle Sizes of Gasifier Circulating Solids and PCD Solids.

The range of PSDs for the gasifier circulating solids and PCD solids during TC24C are provided in Figure 3-31. The wide range of gasifier solids PSD data was due to particle agglomeration.

Figure 3-31. TC24C Range of PSDs for Gasifier Circulating Solids and PCD Solids.

Bulk densities of the gasifier circulating solids and PCD solids are shown in Figure 3-32. The bulk density of the circulating gasifier solids initially decreased from about 64 to 36 lb/ft³, but the density started to increase around Hour 120. The increase was gradual up to about 60 lb/ft³, but around Hour 210 there was a sharp increase up to 90 lb/ft³. The changes in density were due to the particle agglomeration and flow restriction in the mixing zone. The limited PCD solids bulk density data showed little variation, with an average value of about 15 lb/ft³.

Figure 3-32. TC24C Bulk Densities of Gasifier Circulating Solids and PCD Solids.

Figure 3-33 includes micrographs of the gasifier circulating solids sampled at Hours 59 and 67. The Hour 59 sample was collected about 10 hours after coal feed started in TC24C and looked similar to the solids sample taken near the end of TC24A. The Hour 67 sample showed an increase in particle size and shape and appeared to be agglomerating and forming large round particles. Figure 3-34 compares the PSD of the gasifier circulating solids at Hours 59 and 67.

Figure 3-33. Photomicrographs of Gasifier Circulating Solids at Hours 59 and 67.

Figure 3-34. PSD Curves of Gasifier Circulating Solids for Hours 59 and 67.

Figure 3-35 presents the photomicrographs of the gasifier solids at Hour 83, 107, 137, and 147. The general particle appearance was similar for all of the samples, although the particle size distribution varied somewhat. Figure 3-36 gives the PSD for the gasifier circulating solids at Hours 67, 83, 107, 137, and 147.

Figure 3-35. Photomicrographs of Gasifier Circulating Solids for Hours 83, 107, 137, and 147.

Figure 3-36. PSD Curves of Gasifier Circulating Solids for Hours 67, 83, 107, 137, and 147.

The Hour 155 sample appeared to have more gasification ash present, and the LOI was much lower than previous samples. Figure 3-37 shows the photomicrographs of the gasifier solids at Hours 155, 170, 195, and 199. Figure 3-38 gives the PSD of the gasifier circulating solids at Hours 147, 155, 170, 195, and 199. The appearance again changed at Hour 199, and the LOI increased from 8 percent at Hour 195 to 45 percent at Hour 199. The Hour 199 sample was taken during a period of coke breeze feed.

Figure 3-37. Photomicrographs of Gasifier Circulating Solids for Hours 155, 170, 195, and 199.

Figure 3-38. PSD Curves of Gasifier Circulating Solids for Hours 147, 155, 170, 195, and 199.

The sample appearance continued to change as the blockage in the mixing zone affected gasifier operation and coal conversion. Samples taken at Hours 207 and 223 are shown in Figure 3-39. The LOI decreased from 50 percent at Hour 207 to 3 percent at Hour 223. The PSDs are given in Figure 3-40.

Figure 3-39. Photomicrographs of Gasifier Circulating Solids at Hours 207 and 223.

Figure 3-40. PSD Curves of Gasifier Circulating Solids for Hours 199, 207, and 223.

Gasification Ash Removal. Figure 3-41 shows the removal rates for the fine PCD solids removed by the CFAD system and for the coarse gasifier solids removed by the CCAD system. The PCD solids rates were determined from the PCD inlet in-situ sampling, and the rates for CCAD were determined by a system ash balance. The PCD solids rate varied from about 400 to 900 lb/hr, and were higher than previous operation at the same relative coal feed rates. This was likely due to inefficiency in the first solids separation device due to the refractory damage. The gasifier solids removal rate was initially comparable to previous operation; however, as testing progressed, the removal rate was much less due to the restrictions that formed at the CCAD inlet.

Figure 3-41. TC24C Gasification Ash Removal from the Gasifier and PCD.

3.3.3 Gasifier Performance, Gas Analysis

The syngas sampling and analysis during TC24C was the same as in TC24A and discussed in Section 3.1.3.

Syngas Composition. Concentrations of the major syngas components for the steady state operating periods are given in Figure 3-42. The concentrations of H_2 , CO , CO_2 , CH_4 and H_2O are shown on a molar basis. The H_2 , CO , CO_2 , and CH_4 concentrations were measured by a GC on a moisture free basis and converted to wet gas concentrations using the water concentration. The water concentration for steady state periods was estimated based on the in-situ PCD outlet sampling and a mathematical correlation based on the water-gas shift reaction equilibrium. The CO and H_2 concentrations varied from about 3 to 8 mole percent because of variations in the coal feed rate and steam flow rate. The H_2O content also had a large variation from about 7 to 11 mole percent due to the changes in steam flow rate.

Figure 3-42. TC24C Concentrations of Major Syngas Components.

Figure 3-43 compares the measured H_2S concentration and the equilibrium concentration calculated at the gasifier outlet. The H_2S concentration ranged from about 274 to 476 ppm on a wet molar basis showing that about 66 to 80 percent of the coal bound sulfur was converted to H_2S and was above the equilibrium concentration for all of the steady state periods except the last one when the coal feed rate was low. Ammonia concentrations were not obtained during TC24C since the FTIR was being used to support the gas cleanup unit test.

Figure 3-43. TC24C Measured and Calculated Equilibrium H₂S Concentrations.

Syngas Heating Value. The syngas lower heating value (LHV) on a wet basis varied from 22 Btu/SCF at a coal feed rate of about 1,700 lb/hr up to 63 Btu/SCF at a coal feed rate of about 3,700 lb/hr. Plotted in Figure 3-44 are the raw lower heating values for the steady state periods.

Figure 3-44. TC24C Raw Syngas LHV.

Carbon Conversion. The carbon conversion was low at about 73 to 81 percent for all of the steady state periods during TC24C due to the low overall solids collection efficiency. Plotted in Figure 3-45 is the carbon conversion during TC24C, which generally increased as operation

progressed due to slightly higher solids collection efficiency brought about by changes in operating conditions and coal feed rates.

Figure 3-45. TC24C Carbon Conversion.

Gasification Efficiency. The cold gasification efficiency was between 38 and 46 percent, and the hot gasification efficiency was between 69 and 74 percent.

3.3.4 Gasifier Inspections

Detailed inspections of the gasifier and related equipment were performed following TC24C. Visual inspections confirmed that significant agglomeration had occurred in the LMZ and UMZ. Figure 3-46 presents photographs of the agglomeration in the LMZ and UMZ.

Figure 3-46. Post-TC24C Inspection of Mixing Zone.

Samples of the agglomerated material were analyzed to evaluate the formation mechanism and the time the agglomeration occurred. The ash mineral analyses of the agglomerations collected

from different sections of the LMZ are shown in Table 3-4. The coal ash mineral analysis is also listed for comparison. There was a trend of decreasing silicon oxide and increasing alumina oxide, calcium oxide, and sodium oxide from the agglomerations formed in the lower to upper LMZ. The sample from the upper section of LMZ is closer to the coal ash mineral analysis. The mass fraction of start-up sand in the agglomeration gives an indication when the formation may have started. Based on the gasification ash removal rate and gasification ash composition, it was estimated that the agglomeration formation in the LMZ started within 3 hours of the start of coal feed. The agglomeration had grown upward within 20 hours, and then the growth rate stabilized.

Table 3-4. Ash Mineral Analysis of Agglomerations in the LMZ and Bituminous Coal Ash.

	Lower	Middle	Upper	Coal Ash
Al ₂ O ₃ , wt%	4.4	15.1	18.4	17.7
BaO, wt%	0.0	0.1	0.1	0.2
CaO, wt%	1.3	4.0	6.6	7.8
Fe ₂ O ₃ , wt%	2.8	4.4	4.0	3.6
MgO, wt%	0.4	1.9	1.6	2.3
P ₂ O ₅ , wt%	0.1	0.2	0.3	1.1
K ₂ O, wt%	0.3	1.3	0.7	0.9
SiO ₂ , wt%	90.5	71.8	66.4	59.0
Na ₂ O, wt%	0.2	0.7	1.2	1.6
SO ₂ , wt%	0.02	0.07	0.05	4.72
Ti ₂ O ₅ , wt%	0.22	0.62	0.60	0.78

Several pieces of refractory were discovered during the solids removal process following TC24C. The largest piece recovered, pictured in Figure 3-47, was about 12 inches long, 6 inches wide and 2 inches thick. Figure 3-48 compares the refractory pieces recovered following TC24B and C. The refractory condition in the first solids separation device was of concern due to the previously noted cracking and material loss. At the conclusion of TC24, the refractory life was curtailed since the thermal cycles then totaled 15. Inspection of the first stage solids collection revealed that the refractory loss at the inlet had progressed. Figure 3-49 shows the additional loss of refractory in several locations of the inlet from post-TC24B to post-TC24C. The extent of refractory damage made necessary a complete replacement. Refractory in other sections of the gasifier was found in good condition.

Figure 3-47. Refractory Piece Recovered after TC24C.

Figure 3-48. Refractory Pieces Recovered after TC24B and TC24C.

Figure 3-49. Post-TC24B and TC24C Inspections of the First Solids Separation Device Inlet.

The primary gas cooler was visually inspected as well. Figure 3-50, a photograph of the cooler inlet, shows that some loose material had accumulated. The material was easily removed, and all of the tubes were open. However, there was a dark coating on the inside tube surfaces. This same coating was observed after TC24A and was due to tar deposition on the tubes, consistent with the increase in PCD temperature observed at the end of the run. Deposition in TC24C likely occurred during the last hours of operation due to the low operating temperature in the riser as discussed earlier. The primary gas cooler was cleaned and re-inspected. This inspection showed that the tubes were clean and the cooler was ready for service.

Figure 3-50. Post -TC24C Inspection of the Primary Gas Cooler Inlet.

During the inspection the inlet line to the CCAD system contained significant agglomeration. The inlet vessel contained many small, 2-inch pieces of refractory and agglomerations, and there was one large piece of refractory, which was about 6 inches long. The CCAD system was found to be in a normal condition during inspections with the borescope.

The secondary gas cooler was also visually inspected since about 10 percent of the tubes were plugged prior to TC24 due to tube leaks. As depicted in Figure 3-51, inspections revealed scale formation on the water-side and corrosion near the tube sheet. Additionally, some holes were found in the tubes and in the tube connections to the tube sheet. Based on the inspection findings, tube bundle replacement was planned for the post-TC24 outage.

Figure 3-51. Post -TC24C Inspection of Secondary Gas Cooler.

4.0 SENSOR DEVELOPMENT

4.1 Real-Time Particle Monitors

Although the in-situ measurements used to evaluate PCD outlet particulate concentrations at the PSDF give the most accurate results possible, it is useful to have real-time, automated instruments to indicate PCD collection performance and particulate penetration. Two real-time particulate monitors, the PCME DustAlert-90 and the Process Particle Counter (PPC) by Process Metrix, were evaluated in TC24.

4.1.1 PCME DustAlert-90

The PCME DustAlert-90 particulate monitor (referred to as the PCME) was operational throughout TC24. However, the outlet particulate concentration during TC24 did not reach a level that resulted in any indication by the instrument. Although the measured PCD outlet emissions were as high as 25 ppmw (indicated by in-situ measurements), the combination of corrosion product and condensed organic compounds at the PCD outlet was made up of particles/droplets too small for the PCME to detect. Because of the nature of the instrument, particles smaller than about 50 microns are not detected. This means that the PCME will detect gross leaks that have a particle size distribution similar to the PCD inlet, but not the concentrations during TC24.

When the instrument was removed from the process stream at the end of TC24, the probe was heavily coated with condensed hydrocarbon material, but this had no obvious effect on the operation of the instrument. The nitrogen purge kept the insulators clean, which was important since previous operational experience indicated that the instrument signal shorted if these become coated with hydrocarbons. While the instrument remained operational, its ability to detect high concentrations of particulate was not tested with injected dust during TC24 because of the high baseline outlet concentration.

4.1.2 Process Metrix Process Particle Counter

The PPC did not work well during TC24, as the high concentration of condensable hydrocarbon materials caused operational problems with the optical cell of the instrument. There was also a problem with the flow control valve on the syngas extraction probe hot-nitrogen sheath flow. This hot-nitrogen sheath served to heat the outside of the extraction probe and helps to deliver the syngas to the measurement cell at temperatures above the dewpoint. These two problems combined to limit the operation of the PPC to only minutes after cleaning of the optical cell, and the instrument could not be used to collect any useful data during TC24. During the outage, the nitrogen sheath valve will be replaced to alleviate that problem.

4.2 Thermowell Materials

Ceramic and HR-160 metal thermowell materials were tested during TC24 for continued evaluation of thermowell durability and longevity. All of the five ceramic-tipped thermocouples failed during the test campaign. Of the 51 HR-160 metal-tipped thermocouples, 15 failed, and 8 were noticeably worn. Shortly after coal feed began in TC24C, agglomerations in the gasifier covered several of the thermowells, creating an insulating effect that caused abnormally low

temperature readings. The low temperature indications began first in the LMZ, then in the UMZ, and later in the riser.

The thermowells in the gasifier were removed for inspection and analysis. A majority of the thermowells in the mixing zone and riser showed some wear. Thermowell wear was characterized in terms of severity and is charted on the gasifier schematic in Figure 4-1. There was a notable difference in the wear severity based on the location in the gasifier. In Zone A, some wear was noted and one thermocouple was severely eroded. Thermowells in Zone B showed only minimal wear; however, thermowell wear in Zone C (above the coal feed nozzle at the transition from the UMZ to riser and at the bottom of the riser) was severe. The degradation was severe at the tips and apparent on the inner sheaths, and one thermowell tip was completely missing. Most of the thermowells in Zone D (the upper riser) showed moderate to significant wear. Figure 4-2 shows three of the thermowells that were removed from the LMZ. Several of the riser thermowells are shown Figure 4-3.

The wear was apparently due to erosion and possibly corrosion caused by a number of factors such as the operating temperature, large abrasive circulating solids and local zones of high velocities due to agglomeration formation restricting the flow paths. Thermowells insulated by agglomerations were typically in better condition than others in similar locations. Metallurgical analysis is underway to further investigate the root cause of the material degradation.

Figure 4-1. Gasifier Schematic with Thermowell Wear Characterization.

Figure 4-2. LMZ Thermowells after TC24C.

Figure 4-3. Riser Thermowells after TC24C.

4.3 Gas Analysis

The gas analysis system utilized in support of the water-gas shift reaction unit testing significantly improved the quantity of data collected and reduced the time required for analysis. An infrared analyzer installed on the outlet of the water-gas shift reaction unit prior to TC24 provided real time measurement of CO and CO₂. The infrared analyzer is shown in Figure 4-4. Installation of sample gas conditioning system improved the operation of the gas chromatograph (GC) system, which provides H₂, N₂, CO, CO₂, and CH₄ concentrations at the outlet of the water-gas shift reaction unit. The sample gas condition system is pictured in Figure 4-5. The instantaneous gas composition feedback improved the overall operation and collection of data from the test units. About 120 data points were collected during TC24 as compared to 18 data points collected in a previous test campaign during a comparable testing duration.

Figure 4-4. Infrared Analyzer.

Thermoelectric Chiller

Custom-Built Impinger and Valve Assembly

Figure 4-5. Gas Sample Conditioning System.

5.0 PARTICULATE CONTROL DEVICE

For TC24, the particulate characteristics and performance of the PCD with Utah bituminous coal were quantified by particulate sampling and analyses. Long-term evaluation of filter element and failsafe materials was continued. A new component of PCD testing for TC24 was a higher efficiency version of the Pall Dynalloy HR-160 sintered metal fiber filter elements tested previously. The previous version of the HR-160 Dynalloy filter elements demonstrated corrosion resistance and durability, but did not have as high a particulate collection efficiency as some other elements. Therefore, the Dynalloy HR-160 element was constructed with a significantly finer fiber size and were installed for initial testing in TC24.

5.1 PCD Particulate Collection Performance

In-situ particulate sampling was performed at the PCD inlet and outlet using the in-situ batch sampling systems described in previous reports. The inlet particulate measurements were used to quantify the particulate loading to the PCD and were used in a later section to calculate transient drag. The outlet measurements indicated the collection performance of the PCD with the installed filter elements.

5.1.1 PCD Inlet Mass Loadings

Particulate mass concentrations measured at the PCD inlet and calculated particulate mass flow rates are given in Table 5-1. The first two inlet tests during initial coal feed and coke breeze operation can be disregarded because of sample contamination. After stable gasifier operation was obtained on bituminous coal (Runs 3 through 6), particulate mass concentrations were higher than normal.

Table 5-1. In-Situ Particulate Measurements.

Test Date	PCD Inlet					PCD Outlet				
	Run Number	Start Time	End Time	Particulate Loading,		Run Number	Start Time	End Time	Water Vapor, vol %	Particulate Loading, ppmw
				ppmw	lb/hr					
Bituminous Coal Gasification										
2/18/2008	1	10:15	10:30	5,530	92	1	09:00	13:00	11.9	15.6*
Coke Breeze Combustion										
3/10/2008	2	10:30	10:45	8,090	130	2	10:15	13:15	1.9	25.6*
Bituminous Coal Gasification										
3/11/2008	3	12:30	12:45	36,600	892	3	10:00	14:00	8.0	0.56**
3/12/2008	4	11:00	11:10	37,500	924	4	10:30	12:27	10.2	0.52**
3/13/2008	5	09:30	09:40	24,700	676	5	08:30	12:15	10.5	0.13**
3/14/2008	6	12:15	12:30	24,600	530	6	10:30	13:30	5.7	0.15**
3/17/2008	--	--	--	--	--	7	12:45	13:30	8.3	1.51**
* A large fraction of the sample mass appeared to be corrosion product and not gasification ash.										
** Contained both corrosion product and condensed organic material in addition to any gasification ash.										

5.1.2 PCD Outlet Mass Loadings

Particulate concentrations measured at the PCD outlet are included in Table 5-1. As discussed in previous reports, it is common to see an elevated particulate concentration at the outlet of the

PCD during the first few days of a test campaign. However, in TC24, the particulate loadings remained above 0.1 ppmw (the minimum measurement resolution) for the entire test campaign. Although there may have been some minor corrosion or leakage contributing to the overall outlet concentration, the vast majority of the measured mass at the PCD outlet during TC24 gasification was condensation of vapors from organic compounds in the coal.

These organic compounds apparently passed through the PCD filter elements as vapors, which then condensed downstream after contact with relatively cool PCD backpulse gas or other cool structures/purges. Organic contamination produced a mass concentration background level of 0.13 to 1.5 ppmw. Since the particulate penetration expected with the finer fiber Dynalloy elements would have been less than the lower limit of that range, measuring the penetration through those new filter elements was not possible. Fortunately, these filter elements appeared to be in good condition following the test campaign, and further testing in future test campaigns was planned.

5.1.3 Failsafe Performance Tests

During TC24 the new valve-activated failsafe tester that was first used in test campaign TC19 was scheduled for use to simulate the effect of a minor filter element failure on a Pall reversed-media, metal fiber fuse. This type of fuse was previously tested with a high particulate loading in TC20. Following TC20, a 0.25-inch orifice was installed on the failsafe tester to limit particulate flow to the failsafe for evaluation of failsafe performance under simulated low leak rate conditions.

The contamination by condensed organic material at the PCD outlet precluded accurate measurement of particulate concentration and subsequent quantification of failsafe performance, so the failsafe test was deferred until a future test campaign when meaningful results could be obtained.

5.2 PCD Solids Analysis

Particulate characteristics that can affect PCD pressure drop include particle size distribution, density, porosity, surface area, composition, and flow resistance. These parameters were evaluated for characterization of PCD pressure drop performance.

5.2.1 Particle Size Distributions

A Microtrac X-100 particulate size analyzer was used to measure the size distributions of the in-situ particulate samples collected at the PCD inlet and the PCD hopper sample used for the lab drag measurements. Figure 5-1 compares the differential mass percentage distributions for the in-situ samples with the hopper composite sample used for the TC24 lab drag measurements. (Although the in-situ samples are a more accurate representation of the particulate entering the PCD at a given time, the quantity of particulate collected is far too small of be useful for drag measurements.) The one hopper composite was blended from samples collected during the period of best operation from March 11 through March 13, 2008.

Although there are some minor differences in the ends of the distributions, these differences are minor and should not affect the flow resistance properties of the dustcake significantly. Therefore, the composite hopper sample is appropriate for simulation of the pressure drop characteristics of the PCD during operation in TC24.

Figure 5-1. Comparison of TC24 Particle Size Distributions.

5.2.2 Dustcake Observations and Thickness Measurements

The residual dustcake retained after TC24 was heavier than normal. While a typical residual dustcake thickness is about 0.01 inches, the TC24 dustcake thickness varied from about 0.02 to 0.11 inches, as shown in Table 5-2. While condensed material was not visible on the dustcake, deposition of condensable organic vapors may have affected the dustcake.

Table 5-2. Dustcake Thicknesses Measured After TC24

Element Location	Element Type	PSDF No.	Hrs Before TC24	Hrs After TC24*	Thickness, in.	
					Top	Bottom
T-1	FEAL	1522	9959	10196	0.1160	0.0785
T-6	FEAL	1442	5922	6159	0.0212	0.0302
T-14	FEAL	2040	2282	2519	0.0290	0.0227
Average of Top Plenum (FEAL)					0.0554	0.0438
B-1	HR-160	2208	0	237	0.0180	0.0203
B-6	HR-160	2213	0	237	0.0335	0.0359
B-14	HR-160	2221	0	237	0.0388	0.0430
Average of Bottom Plenum (HR-160)					0.0301	0.0331

* Hours on coal only. Does not include hours on coke breeze.

Interesting differences were noted in the dustcakes on the top plenum and bottom plenum. On the fine-fiber HR-160 elements on the bottom plenum, the dustcakes were generally uniform and smooth, with dustcake thicknesses in the range of 0.02 to 0.04 inches. On the iron aluminide elements on the top plenum, the dustcakes were much less uniform, with thicknesses in the range of 0.02 to 0.11 inches. The thickest dustcake was observed on the oldest iron aluminide element, which had 10,196 hours of exposure after TC24. The dustcakes on the older iron aluminide elements, with exposure times of 7,396 to 10,196 hours, also had a more lumpy appearance than did the dustcakes on the younger elements. All of the iron aluminide elements with 2,282 or fewer hours appeared to have smooth dustcakes.

The variations in dustcake thickness and appearance with the age of the element suggest that dustcake thickness and uniformity are influenced by the condition of the element on which the dustcake is collected. Some of this effect may be attributable to changes in the roughness of the element surface brought on by corrosion and wear. Plugging caused by ash penetration and sulfidation could also play a role. These effects will be closely monitored in future testing with the objective of better understanding the effect of filter element condition on the formation and thickness of the dustcake.

5.2.3 Particulate Physical Properties and Chemical Compositions

This section discusses the physical properties and chemical compositions of the in-situ samples collected at the PCD inlet, the PCD hopper sample used for the laboratory drag measurements, and the dustcake samples.

In-situ Samples. Table 5-3 gives the physical properties and chemical compositions of the in-situ samples collected at the PCD inlet and the hopper sample selected for laboratory drag measurements. Run Number 1 was omitted from these tables because it was obtained during a period of abnormally low solids carryover to the PCD. As noted in the tables, Run Number 2 was collected during operation on coke breeze. All of the other samples were collected during operation on the Utah bituminous coal, and they generally have fairly consistent physical properties and chemical compositions. The one exception is the relatively low LOI of Run Number 5. Since it is inconsistent with the other LOI data and also inconsistent with the non-carbonate carbon content, the value appeared to be the result of an analytical error.

Table 5-3. Physical Properties and Chemical Composition of In-Situ Samples and Sample Used for Lab Measurements.

Run Number	Bulk Density, g/cc	True Density, g/cc	Bulk Porosity, %	Surface Area, m ² /g	MMD, microns	LOI, %	CaCO ₃ , wt %	CaS, wt %	CaO, wt %	NCC, wt %	Inerts, wt %
In-Situ Samples—Coke Breeze											
2	0.45	2.13	78.9	32.8	16.4	69.06	0.80	1.67	0.40	66.5	30.63
In-Situ Samples—Utah Bituminous Coal											
3	0.29	2.03	85.7	86.0	23.8	78.06	0.09	1.04	0.87	75.19	22.80
4	0.31	2.02	84.7	57.1	27.9	76.16	0.16	1.18	1.15	73.58	23.93
5	0.27	2.01	86.6	74.8	22.3	62.83	0.18	1.05	2.51	76.48	19.78
6	0.28	2.01	86.1	19.5	18.0	82.36	0.05	1.11	0.60	79.39	18.85
Hopper Sample Used for Lab Drag Measurements											
--	0.31	2.02	84.7	47.4	20.8	78.5	0.07			74.59	

The most striking feature of the TC24 samples was the unusually high non-carbonate carbon (NCC). This was a result of the relatively low carbon conversion achieved with the Utah bituminous coal as discussed in the section on gasifier operations. Past observation indicated an increase in surface area with increasing carbon content. As shown in Figure 5-2, the data from TC24 seem to be similar to previous data obtained during operation with Illinois Basin bituminous coal.

Figure 5-2. Effect of Carbon Content on Specific Surface Area of Gasification Ash.

Composite Hopper Sample. For the lab drag measurements, a composite hopper sample using individual hopper samples from steady state periods was prepared. As shown in Table 5-3, the physical properties and chemistry of the composite hopper sample were similar to those of the in-situ samples, suggesting that the composite hopper sample was a representative sample for the lab drag measurements.

Dustcake Samples. Table 5-4 gives the physical properties and chemical composition of the dustcake samples taken after TC24. The samples included a bulk sample from the bottom plenum (fine-fiber HR-160) elements, a bulk sample of the smooth dustcake from the top plenum (iron aluminide) elements, and a bulk sample of the lumpy dustcake from the top plenum (iron aluminide) elements. (The lumpy dustcake was present on the older iron aluminide elements; while the younger iron aluminide elements had smooth dustcakes.)

Table 5-4. Physical Properties and Chemical Composition of Dustcake Samples.

Sample ID	Bulk Density, g/cc	True Density, g/cc	Bulk Porosity, %	Surface Area, m ² /g	MMD, microns	LOI, %	CaCO ₃ , wt %	CaS, wt %	CaO, wt %	NCC, wt %	Inerts, wt %
Bulk Dustcake from Bottom Plenum											
AB24831	0.30	2.11	85.8	32	16.3	58.14	0.52	0.92	0.61	54.64	43.31
Smooth Dustcake from Top Plenum											
AB24832	0.29	2.11	86.3	43	17.3	62.76	0.61	0.87	--	58.93	39.59
Lumpy Dustcake from Top Plenum											
AB24833	0.22	2.03	89.2	16	11.8	66.29	0.39	0.91	0.19	62.15	36.36

As shown in the table, similar physical properties and chemistry were obtained for the smooth dustcakes from both the fine-fiber HR-160 elements and the younger iron aluminide elements. However, the lumpy dustcake from the older iron aluminide elements had a significantly smaller mean particulate size (12 microns versus 16 to 17 microns for the smooth dustcakes) and a significantly lower surface area (16 m²/g versus 32–43 m²/g for the smooth dustcakes). The carbon content of the lumpy dustcake was also higher than that of the smooth dustcakes (62 versus 55 to 59 weight percent for the smooth dustcakes). The higher carbon content with lower surface area was counter to the established trend of increasing surface area with increasing carbon content. However, this trend was established primarily with PRB coal and may not apply to bituminous coals. Also, in this case, the differences in carbon content are relatively small, and all of the carbon contents are relatively high compared to past data for PRB coal.

Past inspections showed that the residual dustcake had a finer particulate size than the in-situ samples collected at the PCD inlet. This relationship was not clear in TC24, since the final in-situ sample had a MMD particle size of 18 microns, and the smooth dustcakes were 16 to 17 microns MMD. The lumpy dustcake had a finer MMD size (12 microns), but that could be related to the condition of the element and the interaction between the element surface and the dustcake mentioned earlier. One possible explanation is that the roughening of the element surface by corrosion and wear resulted in greater retention of fine particulate within the residual dustcake.

5.2.4 PCD Pressure Drop Performance

5.2.4.1 Transient PCD Drag

The pressure rise within a cleaning cycle of the PCD is a direct measure of the characteristics of the particulate being collected at that time. Under stable operation, the vast majority of this particulate is removed from the filter elements during cleaning, so this is referred to as the transient pressure drop. Since pressure drop is a function of the gas velocity, temperature (gas viscosity), particulate loading, and the flow resistance of the particulate, describing PCD operation in terms of pressure drop makes comparison of different conditions and particulate difficult. Instead, a value of normalized drag is calculated, which is pressure drop that is normalized to 1 ft/min face velocity, 1 lb/ft² areal particulate loading, and gas viscosity of air at 70°F. The result is a fundamental parameter that describes the flow resistance of the collected dustcake.

For each in-situ sample at the PCD inlet, the PCD transient drag was calculated using the measured particulate concentration along with the pressure drop increase and face velocity during the period of the in-situ test. All of the particulate measured at the PCD inlet is assumed to be collected on the filter elements and to contribute to pressure drop.

The inputs and results of the transient drag calculations are shown in Table 5-5. The calculated transient drag at PCD conditions is listed under the column heading "PCD." The corresponding value of transient drag normalized to the viscosity of air at room temperature (70°F) is listed under the heading "PCD@RT". These values are comparable to the lab drag measurements discussed in a later section and are also comparable to other test campaigns that operated at different temperatures. The comparison of these values is shown in a later section. The drag values for TC24 were low, consistent with previous experience with bituminous coal operation.

Table 5-5. Transient Drag Determined from PCD Pressure Drop and from Lab Measurements.

Run No.	$\Delta P/\Delta t$, inwc/min	$\Delta(AL)/\Delta t$, lb/ft ² /min	FV, ft/min	MMD, μm	LOI, %	Drag, inwc/(lb/ft ²)/(ft/min)		
						PCD	PCD@RT	Lab
3	2.62	0.069	5.36	23.8	78.1	38	22	16
4	2.99	0.071	5.66	27.9	76.2	42	23	14
5	3.55	0.052	6.04	22.3	62.8	68	38	18
6	2.00	0.041	4.77	17.9	82.4	49	27	23
Avg	2.79	0.06	5.46	23.0	74.9	49	28	18
Lab drag data calculated from linear regression to MMD of lab drag samples.								

Normalized PCD transient drag is plotted as a function of carbon content in Figure 5-3. As seen in previous test campaigns, transient drag increased with increasing carbon content in the gasification ash. However, the relationship between drag and carbon was weak for the bituminous coals relative to the lower rank coals tested at the PSDF. This correlation shows scatter in the data because it does not take into account the effect of particle size, which will be considered in a later section.

Figure 5-3. PCD Transient Drag versus Carbon Content of In-Situ Samples.

5.2.4.2 Baseline Pressure Drop Analysis

Figure 5-4 shows the peak and baseline PCD pressure drop measurements for the TC20 test campaign, and the TC20 baseline data are compared to previous test campaigns in Figure 5-5. While these pressure drop values are normalized for temperature and gas velocity, they are not normalized for particulate concentration as are the drag data discussed in the previous section.

Both baseline and peak pressure drop were moderate during TC24 especially considering the high particulate mass rate and subsequent elevated areal loading on the filter elements that would have resulted. The moderate pressure drop is consistent with the low values of dustcake drag measured in the lab.

For TC24, the bottom plenum was installed with finer fiber version of the Pall Dynalloy filter elements. These finer fiber elements were expected to provide improved collection efficiency but increased pressure drop. However, neither this effect nor the effect of the old FEAL filter elements installed in the top plenum was obvious from the pressure drop data.

Figure 5-4. Normalized PCD Pressure Drop.

Figure 5-5. Comparison of Normalized Baseline Pressure Drop from TC20 through TC24.

5.2.5 Prediction of PCD Drag and Pressure Drop

5.2.5.1 Lab Drag Measurements

To investigate the characteristics of the TC24 particulate more completely, drag measurements were made in the lab flow resistance test device on the composite hopper sample. This lab apparatus uses a series of cyclones between the particulate generator and the dustcake collection

surface to vary the particle size distribution of the dustcake. The results are illustrated in Figure 5-6 with normalized drag plotted against the MMD of the collected dustcake.

In the past, the drag of a particulate sample has been related to both the particle size and to the non-carbonate carbon (NCC) content or Loss on Ignition (LOI). However, since there was only one lab sample available and the carbon contents were very consistent between the various size fractions of the sample, the multiple linear correlation did not calculate a meaningful result. Therefore, the TC24 data was related only to particulate size as shown in the equation below.

$$\text{Drag} = 10^{(2.796 - (1.150 * \text{Log}(\text{MMD})))}, \text{ with an } r^2 = 0.999$$

Figure 5-6 gives the values of PCD transient drag calculated for each of the in-situ samples from Table 5-6. All of the PCD data points fell above the lab drag measurements despite lower values of bulk carbon. It has been observed from previous data that drag increases with carbon content. However, the TC24 lab samples had carbon (LOI) values of 84 to 86 percent, while the in-situ samples ranged from 60 to 80 percent. Therefore, the PCD drag values were expected to fall below the lab measurements. The fact that it did not indicated that some of the mass entering the PCD fell to the bottom of the PCD without first being collected on the filter elements, which would consistent with the large particle size. Because of the lower than expected areal loading on the PCD filter elements, the calculated PCD drag was higher than expected.

Figure 5-6. Lab-Measured Drag as a Function of Particle Size.

The results of regression predictions for each individual value of PCD transient drag are shown in the rightmost column of Table 5-7. These calculations use the MMD of each in-situ sample to predict the transient drag of the PCD during that test. The lab predictions are higher than the actual PCD data for both the individual values and the average for TC24.

5.2.5.2 Comparison of Lab Measurements with Transient Drag

Average laboratory and PCD drag values for all gasification test campaigns are summarized in Table 5-8. The comparison shows excellent overall historical agreement (average difference of about 10 percent). For TC24, the difference was large, at negative 44 percent. The results for all gasification test campaigns are plotted in Figure 5-7 and continue to show data points scattered around the perfect agreement line. The average lab-measured drag ($18 \text{ inH}_2\text{O}/(\text{ft}/\text{min})/(\text{lb}/\text{ft}^2)$) was the lowest measured to date, and the actual drag value of $28 \text{ inH}_2\text{O}/(\text{ft}/\text{min})/(\text{lb}/\text{ft}^2)$ was one of the lowest ever measured. These drag values are consistent with the coarser particle size distributions combined with characteristically low drag of bituminous coal gasification ash.

Table 5-6. Average Drag Values Determined from PCD and Lab Measurements.

Run	Coal	Average Transient Drag Determined from PCD Performance, $\text{inwc}/(\text{lb}/\text{ft}^2)/(\text{ft}/\text{min})$	Average Drag Determined from RAPTOR Lab Measurements, $\text{inwc}/(\text{lb}/\text{ft}^2)/(\text{ft}/\text{min})$	Difference from Mean Value*, %
GCT2	PRB	29	21	-33.5
GCT3	PRB	80	93	14.5
GCT4	PRB	66	57	-15.2
TC06	PRB	89	81	-9.6
TC07	PRB	48	50	4.3
TC08	PRB	47	50	7.3
TC09	Hiawatha	29	23	-21.8
TC10	PRB	45	58	25.2
TC11	Falkirk Lignite	16	36	76.2
TC12	PRB	58	61	4.7
TC13	Freedom Lignite	34	39	13.6
TC14	PRB	47	42	-13.0
TC15	PRB	55	76	33.3
TC16	PRB + Limestone	49	52	4.8
TC16	Lignite + Dolomite	26	42	47.1
TC17	IL Basin	25	19	-27.8
TC18	PRB	59	82	32.6
TC19**	PRB	64	72	11.8
TC20**	PRB	78	108	32.3
TC21**	ND Lignite	19	32	51.0
TC22**	MS Lignite	27	40	38.8
TC24	Utah Bit.	28	18	-43.5
Average		46	52	10.6
* $D = (R1-R2)/(R1+R2)/2 \times 100$				
** Technique modified to use carbon content of lab drag sample				

Figure 5-7. Comparison of PCD Transient Drag with Lab Measurements.

5.3 Analysis of PCD Filter Element Condition

After shutdown of TC24, a complete set of measurements was made on the filter elements to determine the effect of exposure hours on pressure drop and to determine if condensed material affected the filter elements or dustcakes.

Following shutdown of the gasifier at the end of TC24, the PCD backpulse system was operated for several hours to clean the PCD filter elements to the extent possible. Nonetheless, when the filter elements were removed from the PCD, they were found to have thicker dustcakes than typical. For flow testing, the elements are typically air blown to remove excess dustcake and achieve a uniform residual dustcake for testing. In the case of TC24, the dustcake thicknesses were quite variable even after air blowing. The particulate appeared to be somewhat tacky (likely related to the condensed material) and had to be lightly brushed to achieve uniformity.

The pressure drop at fixed face velocities was measured on each of the filter elements both with the light residual dustcake and after pressure washing to remove all particulate. The data are plotted as a function of gasification exposure hours for the FEAL filter elements in Figure 5-8. These are not historical data in the plot, but are a snapshot of the filter elements actually installed in TC24. Since the fine-fiber Dynalloy elements were used for the first time in TC24, these elements all have the same age, 237 hours. The pressure drop at 3.5 ft/min face velocity for the Dynalloy elements ranged from 2.6 to 1.4 inH₂O in the dirty condition, and from 0.4 to 0.7 inH₂O in the cleaned condition.

Figure 5-8. Pressure Drop versus Exposure Hours for Iron Aluminide Filter Elements.

The FEAL filter elements represented in Figure 5-8 continued to show the previously established trend of increasing pressure drop with time for both clean and dirty elements. The limited data for the new fine-fiber Dynalloy filter elements did not indicate pressure drop problems. These elements will continue to be monitored to assess their long-term performance.

6.0 ADVANCED SYNGAS CLEANUP

Since enhancing the syngas CO_2 and H_2 concentrations is desirable for applications such as CO_2 capture and fuel cell operation, testing of water-gas shift systems and catalysts, begun in TC23, continued in TC24. The advanced syngas cleanup unit was used in TC24 to test two WGS catalysts installed in the same type of filter elements used in the PCD. The syngas cleanup slipstream was also used to support outside researchers from the DOE National Energy Technology Laboratory and from Media Process Technology.

6.1 Water-Gas Shift Reaction Catalyst

A system which utilizes catalytic filter elements for the WGS reaction was developed at the PSDF and successfully tested with syngas for about 40 hours during TC24. Two catalytic filter elements were installed in parallel to test the Sud-Chemie T-2822 shift catalyst and the Johnson Matthey Katalco K8-11 shift catalyst. Table 6-1 lists the catalyst properties. During the outage preceding TC24, the catalysts were pre-sulfided using a gas mixture of 5 percent H_2S and 95 percent H_2 to convert the cobalt and molybdenum, the active ingredients of the catalyst, to the sulfided form. The pre-sulfidation was performed at 200 psig and 660°F. After the pre-sulfidation was completed, the catalysts were pulverized and sieved to a particle size of 106 to 212 microns and then packed in iron aluminide filter elements.

Table 6.1. Water-Gas Shift Catalyst Properties.

Catalyst Supplier	Sud-Chemie	Johnson Matthey
Catalyst Trade Name	T-2822	Katalco K8-11
Chemical Composition		
Aluminum Oxide Content, wt %	50-70	
Magnesium Oxide Content, wt %	15-35	
Molybdenum Oxide Content, wt %	5-15	10
Cobalt Oxide Content, wt %	1-10	4
Calcium Oxide Content, wt %	3-7	
Physical Properties		
Shape	Powder	Powder
Size, microns	100-200	100-200
Density, lb/ft ³	50	50

While operating on syngas, the operating conditions were varied to evaluate the performance of the catalytic filter elements. The inlet temperature was varied from 450 to 850°F, and the pressure ranged from 150 to 160 psig. The face velocity was varied between 1 and 3.6 ft/min, and the H_2O to CO molar ratio was varied from 0.8 to 6.2 mole/mole.

The CO conversions ranged from 5.3 to 94.4 percent for the T-2822 catalyst during 30 hours of testing, while the CO conversion for the Katalco K8-11 catalyst ranged from 10.5 to 75.4 percent during 10 hours of testing. The CO conversion was dependent upon operating temperature, face velocity and catalyst type. Figure 6-1 presents the data collected during the T-2822 catalyst testing, which shows a positive linear relationship for two different face velocities with a $\text{H}_2\text{O}/\text{CO}$ ratio of 4.5 to 6.0 mole/mole. The data demonstrated the expected trends of increasing

conversion with increasing temperature and increasing conversion with decreasing face velocity. Figure 6-2 shows the positive linear relationship for the K8-11 catalyst at a face velocity of 1.3 to 1.9 ft/s and a $\text{H}_2\text{O}/\text{CO}$ ratio of 4.5 to 6.0 mole/mole.

Figure 6-1. Sud-Chemie T-2822 Shift Catalyst Performance.

Figure 6-2. Johnson Matthey Katalco K8-11 Shift Catalyst Performance.

Figure 6-3 compares the performance of the two catalysts over a range of temperatures at a face velocity of 1.3 to 1.9 ft/s and a $\text{H}_2\text{O}/\text{CO}$ mole ratio of 4.5 to 6.0. Based on this data, the T-2822 catalyst performed slightly better than the K8-11.

Figure 6-3. T-2822 and Katalco K8-11 Shift Catalyst Performance Comparison.

6.2 NETL Fuel Cell Module

The NETL Fuel Cell Module was installed, and shakedown tests were successfully completed. Figure 6-4 is a photograph of the Fuel Cell Module during the completion of installation. The Fuel Cell Module is a multi-cell array mobile platform developed to test different solid oxide fuel cells in parallel on coal-derived syngas. The unit is designed to enable testing for up to 12 individual fuel cells simultaneously over a range of electric load conditions for extended periods of time to provide data on the influence of trace coal contaminants such as arsenic, phosphorous, selenium, and mercury on fuel cell performance. This information is critical for development of fuel cells for coal-based power generation.

The shakedown tests were performed on hydrogen and lasted for over 100 hours. The test cells were operated at an average temperature of $1400^{\circ}\text{F} \pm 60^{\circ}\text{F}$ with 68 mole percent H_2 and 32 mole percent H_2O . At initial hydrogen feed, eight cells produced output indicative of proper operation; however, only five cells were functioning properly by the end of the 100-hour test. Cell 7, which was typical of the five operating cells, had a current density of about $150 \text{ mA}/\text{cm}^2$ and steadily maintained a voltage of about 0.73V.

After the initial shakedown tests were completed, new button cells were loaded. The module was then re-started. The two subsequent re-starts initially demonstrated good cell response, but through the first 24 hours, cell failure reduced the number of operable cells. Diminishing cell operation was attributed to seal failure and thermal degradation of the current collecting wires. Improvements to the seal method were identified and will be completed before future testing to increase cell operating life.

Figure 6-4. NETL Fuel Cell Module.

6.3 MPT Carbon Molecular Sieve Membrane Commissioning

An advanced CMS membrane was installed and commissioned during TC24. Working in cooperation with NETL, researchers at MPT developed the membrane which is highly selective for hydrogen and particularly well suited for coal-derived syngas. The membrane separates hydrogen from syngas with membrane materials that were extensively lab-tested. The objective of the testing at PSDF is to evaluate material stability of the membrane under gasification conditions with particulate-free coal-derived syngas containing both major and minor contaminants including hydrocarbons at the parts per million level.

After installation, the membrane unit was connected to a slip stream of syngas from the process that utilized bottled H_2 to increase the hydrogen content of the syngas. Upon review of the system, modifications to the control system were made. Two low flow switches were installed and interlocked with the existing trip-monitoring device to stop syngas flow to the unit if purge nitrogen flow was lost to the main process cabinet or to the H_2 bubbler flow meter box. In addition, the emergency shutdown logic was reprogrammed to ensure a proper shutdown in the case of loss of power. Due to delays associated with modifying the system and with gasifier operation, testing with syngas was postponed. The membrane unit will be further tested in the laboratory and modified before continuing testing planned for TC25.

7.0 SUPPORT EQUIPMENT

7.1 Startup Burner

Start-up of TC24A was delayed by problems lighting the pilot flame of the startup burner. Prior to operation, a new shroud assembly was installed on the pilot as preventive maintenance. After several failed attempts to light the pilot, the burner pilot assembly was removed and inspected. The inspection revealed that the pilot assembly was not fabricated the same as the previous assembly and was missing an opening that allows the ignition spark to light the pilot. The assembly was modified to include the ignitor opening, and the ignitor was relocated closer to the pilot tip, as indicated in Figure 7-1. A number of other repairs were made during the initial attempts to light the pilot such as repairing a failed trip switch, repairing the flame rod, replacing the igniter spark plug and transformer, and verifying the flame detection system operation. Later, adjustments were made to increase the supply propane pressure. Several options are being evaluated to enhance startup burner operation in the future.

Figure 7-1. Startup Burner Assembly.

7.2 Recycle Gas Compressor

The recycle syngas compressor supplied syngas for gasifier aeration for 171 hours during TC24C. The compressor was not used during TC24A or B due to gasifier operational instability. The system operated well and experienced no major problems. There were no compressor related trips. Steady state operating conditions at the recycle gas compressor outlet as well as the recycle syngas flow rate to the gasifier are shown in Figure 7-2. The control trip logic for the recycle syngas compressor was modified prior to TC24 to increase the availability of the recycle syngas system. The trip condition related to the main air compressor discharge pressure that was eliminated worked properly so that a gasifier trip caused by a low main air compressor discharge pressure did not trip the recycle syngas compressor during TC24.

Figure 7-2. Recycle Gas Compressor Operating Conditions.

8.0 CONCLUSIONS

A major focus of test campaign TC24 was characterization of gasifier operation and performance with bituminous coal following the modifications completed in 2006. These modifications were the most significant changes to the PSDF Transport Gasifier since its installation in 1996. The purpose of the modifications was to improve residence time and solid collection efficiencies, two critical parameters affecting the gasifier performance with high rank fuels. Although gasifier operations were challenging, several other objectives were achieved during TC24.

Lessons Learned. The following list shows the main points gained from TC24 operation.

- During WGS testing with new methods of contacting syngas with catalysts, CO conversions of up to 94 percent were achieved. The CO conversion increased as the operating temperature was increased.
- Process integration issues were identified and addressed in preparation for testing the Media and Process Technology hydrogen selective CMS membrane in the next test campaign, TC25.
- NETL's fuel cell module was commissioned and successfully integrated with the PSDF process. The seal method utilized on the fuel cell module was identified as inadequate, and a re-design was developed based on the testing conducted in TC24.
- Higher conveying velocities are needed to prevent coal feeder discharge line plugging when feeding material with a particle size (SMD) greater than 400 microns and an oversize percentage greater than 10 percent.
- The larger gasifier circulating solids particle size may have impacted thermowell wear rates.
- Gas sampling system improvements were effective in increasing the data points collected.
- Tar formation and deposition in the primary gas cooler can occur during operation with bituminous coal if the operating temperature is too low and the coal feed particle size is too large. To achieve higher carbon conversions with bituminous coal, a higher gasifier operating temperature is required.
- The Actchem VC refractory is erosion resistance but is susceptible to cracking and should not be utilized in a developmental application where multiple start-ups and shutdowns occur.
- PCD testing of finer-fiber HR-160 elements showed good operation without a discernible increase in the PCD pressure drop.

APPENDIX A OPERATING HISTORY

System commissioning of the KBR Transport Reactor train and the first five test campaigns (TCs) were performed in combustion mode. Approximately 5,000 hours of combustion operation were completed from 1996 to 1999. The system was transitioned to gasification operation in late 1999. Four gasification commissioning tests (GCTs), each lasting nominally 250 hours, were completed by early 2001. At the conclusion of TC24, 19 gasification test campaigns were completed, each nominally 250 to 1,500 hours in duration, for a total of about 10,840 hours of coal gasification operation. Powder River Basin subbituminous coal is the most extensively tested fuel, although several bituminous and lignite coals have also been tested. The Transport Gasifier has operated successfully in both air-blown and oxygen-blown modes.

Table A-1 summarizes the gasification testing completed at the conclusion of TC24. The table lists the duration, number of hours on coal, fuel type, and major objectives of each test. More information about the individual test campaigns may be found in the test campaign reports, located on the PSDF website, <http://psdf.southernco.com>.

Table A-1. Gasification Operating History.

Test	Start Date	Duration (hrs)	Fuel Type*	Comments
GCT1	September 1999	233	PRB, Illinois #6, Alabama	First gasification testing
GCT2	April 2000	218	PRB	Stable operations
GCT3	February 2001	184	PRB	Loop seal commissioning
GCT4	March 2001	242	PRB	Final gasification commissioning test
TC06	July 2001	1,025	PRB	First long duration test campaign
TC07	April 2002	442	PRB, Alabama	Lower mixing zone commissioning
TC08	June 2002	365	PRB	First oxygen-blown testing First on-line failsafe testing
TC09	September 2002	309	Hiawatha	New mixing zone steam system
TC10	October 2002	416	PRB	Developmental coal feeder
TC11	April 2003	192	Falkirk Lignite	First lignite testing
TC12	May 2003	733	PRB	Fuel cell testing
TC13	September 2003	501	PRB, Freedom Lignite	Syngas to combustion turbine
TC14	February 2004	214	PRB	Syngas to combustion turbine CFAD commissioning
TC15	April 2004	200	PRB	Improved oxygen feed distribution
TC16	July 2004	835	PRB, Freedom Lignite	Fuel cell testing High pressure O ₂ -blown operation
TC17	October 2004	313	PRB, Illinois Basin	Bituminous coal testing
TC18	June 2005	1,342	PRB	Recycle gas compressor commissioning
TC19	November 2005	518	PRB	CCAD commissioning
TC20	August 2006	870	PRB	Gasifier configuration modifications
TC21	November 2006	388	Freedom Lignite	First lignite test following the gasifier modifications
TC22	March 2007	543	Mississippi Lignite	High moisture lignite testing
TC23	August 2007	481	PRB, Freedom Lignite	High sodium lignite testing
TC24	February 2008	23	Utah	First bituminous coal test following the gasifier modifications

*Note: PRB is subbituminous coal; Illinois #6, Alabama, Hiawatha, Utah, and Illinois Basin coals are bituminous coals.

APPENDIX B STEADY STATE OPERATING PERIODS AND MAJOR OPERATING PARAMETERS

There were sixteen steady state operating periods during TC24, five periods (TC24-1 through TC24-5) during TC24A and eleven (TC24-6 through TC24-16) during TC24C. These periods are given in Table B-1, along with the major operating parameters for each period. The steady state periods are defined based on maintaining gasifier operating conditions within defined ranges.

All of the steady state periods were in air-blown gasification mode with bituminous coal feed. Recycle syngas operation was achieved in all the TC24C operating periods. The coal feed rates were calculated from the feeder weigh cells, and the air, steam, nitrogen, and recycle syngas flow rates were taken from flow indicators. The PCD solids rates were determined from the in-situ sampling at the PCD inlet, and the ash removal rates for CCAD were determined by a system ash balance.

Table B-1. Steady State Operating Periods and Major Operating Parameters.

Operating Period	Run Time Hours	Gasifier Outlet Temperature °F	Gasifier Outlet Pressure psig	Coal Feed Rate lb/hr	Air Feed Rate lb/hr	Steam Feed Rate lb/hr	Nitrogen Feed Rate lb/hr	Recycle Syngas Rate lb/hr	Syngas Rate lb/hr	Gasifier Solids Removal Rate lb/hr	PCD Solids Removal Rate lb/hr
TC24-1	12	1,730	200	1,160	7,350	1,260	7,670	0	17,110	60	90
TC24-2	16	1,740	200	1,140	7,440	1,260	7,820	0	17,330	50	90
TC24-3	25	1,740	200	1,170	7,170	1,270	7,420	0	16,620	50	90
TC24-4	31	1,730	200	1,180	7,140	1,270	7,590	0	16,810	50	90
TC24-5	37	1,730	200	1,180	7,210	1,270	7,780	0	16,620	60	90
TC24-6	70	1,760	178	3,730	13,110	1,820	6,470	1,440	25,000	300	900
TC24-7	94	1,770	186	3,660	12,670	1,920	5,500	1,200	23,320	340	790
TC24-8	107	1,760	186	3,810	14,300	2,140	6,340	1,220	27,240	420	680
TC24-9	132	1,770	186	2,610	10,850	700	6,680	1,090	21,370	220	530
TC24-10	143	1,790	186	2,330	10,230	1,280	6,840	660	20,890	110	510
TC24-11	154	1,790	186	2,280	9,980	1,320	5,610	810	19,500	80	490
TC24-12	166	1,810	204	3,090	12,250	2,180	5,710	650	23,130	140	670
TC24-13	171	1,810	204	3,060	12,230	2,180	5,780	650	23,210	140	660
TC24-14	180	1,820	204	3,140	12,420	2,190	5,780	680	23,450	140	680
TC24-15	191	1,830	204	3,130	12,470	2,180	5,850	710	23,410	130	680
TC24-16	219	1,780	170	1,750	8,550	1,640	10,370	900	22,310	110	380

APPENDIX C SOLIDS AND GAS SAMPLING AND ANALYSES

To assess and optimize system performance, extensive solids and gas sampling and analysis are routinely performed during gasification operation. Figure C-1 shows the sample locations, labeled A through M. These locations are referenced in the following sections.

Figure C-1. Sampling Locations of the PSDF Gasification Process.

Coal and Sorbent (Locations A and B). Coal samples taken at various locations in the coal preparation area are used to assess coal mill operation and the extent of particle size segregation in the equipment. Samples of coal and sorbent are also taken from the surge bins of the feeders. These samples are used to develop operating envelopes for the feeders as well as to characterize the feed material. Figure C-2 shows an example of a feeder sample system, which incorporates an auger device located on the original coal feeder surge bin. Particle size analyses are performed in the on-site laboratory using sieve analysis for the coal and using a Microtraz

analyzer for the sorbent. Moisture values are also derived on-site using an MF-50 moisture analyzer. Chemical analyses are obtained through an outside certified laboratory.

Figure C-2. Solids Sample System at Coal Feeder Surge Bin.

Gasifier Solids (Locations C, D, E, and F). Gasifier solids are taken from the riser, seal leg, and standpipe portions of the gasifier. These samples are used to characterize gasifier performance and to identify operating conditions that could lead to agglomeration formation. An example of the gasifier solids sampling systems is shown in Figure C-3. These water-cooled systems use collection vessels that are filled by exposure to gasifier pressure. Particle size is determined on-site using a Microtrac X-100 analyzer or by sieve analysis. Samples are sent an outside certified laboratory for chemical analyses.

Figure C-3. Gasifier Solids Sampling System.

In-Situ Gasification Ash (Locations H and I). In-situ sampling at the PCD inlet is used to measure the concentration of gasification ash exiting the gasifier with the syngas. The solids concentration, typically 10,000 to 20,000 ppmw, as well as the LOI values and particle sizes, are measured on-site. In-situ samples are also taken at the PCD outlet, which measure solids concentrations down to a resolution of about 0.1 ppmw. During outlet sampling, which takes typically four hours, the condensate from the syngas is collected and measured to provide syngas moisture concentration.

The in-situ sampling systems, an example of which is shown in Figure C-4, are isokinetic samplers designed and operated by Southern Research Institute.

Figure C-4. In-Situ Solids Sample System.

Extractive Gasification Ash (Location J). Bulk samples of gasification ash (PCD solids) are taken at the inlet of the CFAD system. This sample system is similar to the extractive systems on the gasifier, but does not require cooling. Solids are extracted by exposing a collection vessel to the system pressure at the CFAD inlet. Particle sizes are determined by on-site analysis using a Microtrac X-100 instrument. LOI analysis is also performed on site, and chemical analysis is completed by an off-site certified laboratory.

Solids Analyses Techniques. Table C-1 lists the ASTM standard techniques used for chemical analyses of the solids samples.

Table C-1. ASTM Standards Used in Solids Chemical Analyses.

Test Component	Standard	Standard Title
Ash Minerals	ASTM D 3682	Standard Test Method for Major and Minor Elements in Combustion Residues from Coal Utilization Processes
Carbon, Hydrogen, and Nitrogen	ASTM D 5373	Standard Test Methods for Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Laboratory Samples of Coal and Coke
Carbon Dioxide	ASTM D 1756	Standard Test Method for Determination as Carbon Dioxide of Carbonate Carbon in Coal
Heating Value	ASTM D 5865	Standard Test Method for Gross Calorific Value of Coal and Coke
Moisture Content	ASTM D 5142	Standard Test Methods for Proximate Analysis of the Analysis Sample of Coal and Coke by Instrumental Procedures
Sulfur Content	ASTM D 4239	Standard Test Methods for Sulfur in the Analysis Sample of Coal and Coke Using High- Temperature Tube Furnace Combustion Methods
Volatile Content	ASTM D 5142	Standard Test Methods for Proximate Analysis of the Analysis Sample of Coal and Coke by Instrumental Procedures

Syngas Bomb Sampling (Locations C and G). Syngas composition data for difficult or non-routine samples is obtained by using bomb sampling techniques. These techniques allow for the analysis of samples that would otherwise be impossible or problematic for continuous analysis. Pictured in Figure C-5 are bomb sample cylinders. The cylinder on the left of the figure was treated with a Sulfinert performance coating for use in sampling sulfur compounds, and the bomb sample cylinder on the right is a standard cylinder used for general syngas quality samples.

Figure C-5. Bomb Sampler Cylinders.

Bomb samples from the gasifier riser and the upper mixing zone are captured using a sample system consisting of a 30-microns sample filter, a flow orifice, and high temperature valves. Once the samples are obtained, they are analyzed on an Applied Automation Optichrome Advance GC. Measurements are taken for O₂, N₂, H₂, CO, CO₂, and CH₄.

Bomb samples are also frequently obtained at the advanced syngas cleanup unit. These samples are much easier to capture due to their lower temperature and lack of particulate. Samples from this slipstream may be analyzed for syngas quality (identical to the gasifier samples mentioned above) or for sulfur compounds. If measurements are made for sulfur compounds, specially coated bombs (collection vessels) and valves are used to minimize sample/bomb interactions. These sulfur compounds include SO₂, H₂S, COS, and CS₂. Analyses for SO₂ for specific tests are made using a Rosemount XStream analyzer; all other sulfur compounds are analyzed using an Agilent 5890 GC equipped with a flame photometric detector.

Gas Analyzers (Locations H, K, L, and M). Both extractive and in-situ gas sampling systems are utilized at the PSDF. Although extractive sampling takes a longer time to gain results (at least one minute delay compared to nearly instant), the presence of particulate and the high temperature, high pressure conditions at most locations in the gasification process require that the gas be extracted and conditioned prior to analysis.

Most of the syngas quality measurements are taken at PCD inlet, downstream of the primary gas cooler, which is the first location in the process where the temperature is suitably low. Gas is sampled here to give the earliest possible indication of process changes. GC units are used in addition to the continuous analyzers.

The system for conditioning the gas prior to analysis is depicted in Figure C-6. The system uses reflux probes, which are demarked with a blue mark. The high pressure canister filter (marked with green), which is shared by both probes, can be seen at the top of the photograph. The probes remove moisture and long chain hydrocarbons from the syngas, and the filter removes any residual particulate matter. The probes and filters are further described below. This system provides uninterrupted sample flow for all of the major syngas components.

Figure C-6. Gas Conditioning System.

The temperature control set up for the gas conditioning system, shown in Figure C-7, includes temperature control thermocouples (blue), a control valve (yellow), and a vortex chiller (red). With this control system in place, the outlet sample temperature is maintained between 70 and 100°F.

Figure C-7. Gas Conditioning System with Temperature Control.

A disassembled reflux probe is shown in Figure C-8. The cooling coil/fins, along with the demister pad can be seen on the left. Cooling air/nitrogen from the vortex chiller flows through the

interior of this section. The main body of a reflux probe (minus the insulation) is displayed on the right. This section houses the cooling coils and also has its own cooling jacket.

Figure C-8. Disassembled Reflux Probe.

Figure C-9 shows a disassembled canister filter. This type of filter is used to remove particulate from the PCD inlet prior to gas sampling. During operation, the filter is replaced once a week.

Figure C-9. Disassembled Canister Filter.

Figure C-10 is a photograph of the on-line laboratory GC. This Agilent GC, adapted for on-line operation, analyzes for varying levels of H_2S , COS , and CS_2 . The analytical method and GC integration were developed at the PSDF. This instrument, and others like it, have been valuable in the successful evaluation of the performance of H_2S sorbents and COS hydrolysis catalysts.

Figure C-10. On-Line Laboratory GC for PCD Inlet Gas Analysis.

The continuous PCD inlet analyzers are shown in Figure C-11. This analyzer cabinet houses the fast response analyzers used for gasifier monitoring. The response time for these instruments is typically less than 5 minutes. This bank of analyzers consists of four Rosemount XStream units (blue and gray) along with an oxygen analyzer (bottom of photo) which is used as a backup. The Rosemount units provide redundant analyses for O₂, CO, and CO₂. A customized sample system (built on-site) can be seen at the top of the cabinet.

Figure C-11. Continuous Analyzer Cabinet for PCD Inlet Gas Analysis.

In-situ analyzers are located at the outlet of the atmospheric syngas combustor. Shown in Figure C-12, the in-situ system features an infrared analyzer (blue) manufactured by Procal Analytics. The system measures the flue gas at the syngas combustor outlet for percent levels of moisture and CO₂, as well as for SO₂, nitrogen oxides (NO_x), and CO in the ppm range. The auto

zero/calibration unit (green), a new addition to this instrument, was designed and built at the PSDF.

Figure C-12. In-Situ Flue Gas Analyzer.

Measurements of water vapor, ammonia, and hydrocarbons in the syngas are made by the on-line Fourier Transform Infrared (FTIR) system, which was manufactured by Temet Gasmet. This system, pictured in Figure C-13, is located at the advanced syngas cleanup unit. The primary enclosure (red) contains the laser and infrared sources, the measurement cell, and associated electronics (power supplies, processors, and relays). The sample interface unit (blue) on the right contains components related to temperature and valve control. The analyzer at the bottom of the photo is an SO₂ analyzer used for direct oxidation tests.

Figure C-13. FTIR Gas Analysis System.

Also located at the advanced syngas cleanup unit are analyzers for assessing CO₂ separation, direct oxidation of H₂S, and water-gas shift reactions. These analyzers are shown in Figure C-14. The Rosemount XStream analyzer at the top of this bank is the main instrument used to monitor water-gas shift reactions. The other XStream is used to monitor CO₂ separation testing. The oscilloscope at the bottom of this rack is used to set up and test FTIR parameters. The

sample system (valves and flow meters) is used to calibrate/zero both of the XStream analyzers. Figure C-15 shows a new process GC used for syngas quality measurements.

Figure C-14. CO₂ Capture and Water-Gas Shift Reaction Analyzers.

Figure C-15. Syngas Quality Gas Chromatograph.

Sample conditioning at the syngas cleanup unit is provided by thermoelectric chillers. Essentially, these chiller units provide the same service to the cleanup unit that the reflux probes provide to the gasification process unit. Chiller units manufactured by Baldwin/Permapure are utilized, but the impinger portions of the systems were designed and built on-site to

accommodate the higher pressures. Most of the impinger units are coated with Sulfinert to allow for accurate H_2S and SO_2 analyses. A coated impinger and drain valve assembly is shown in Figure C-16. A Baldwin/Permapure chiller unit is displayed in Figure C-17.

Figure C-16. High Pressure Impinger Set.

Figure C-17. Thermoelectric Chiller.

APPENDIX D MATERIAL AND ENERGY BALANCES

The material and energy balances showed reasonable accuracy given the diversity of the measurements used for their calculation. A gasifier mass balance for the TC24 steady state operating periods is shown in Figure D-1. The mass balance documents the accuracy of the solids and gas rates at the inlet and outlet of the gasifier. The data agree within 5 percent.

Figure D-1. Mass Balance.

The overall energy balance for the gasifier is shown in Figure D-2. A gasifier heat loss of 3.5 MMBtu/hr was assumed for the energy balance. This balance verifies the accuracy of the gasification efficiencies, and shows agreement within about 10 percent.

Figure D-2. Energy Balance.

The carbon balance documents the accuracy of the carbon conversions, and is shown for TC24A in Figure D-3. The carbon balances for the steady periods in TC24A fell within a 6 percent error range. The carbon balance is perfect for TC24C since the carbon balance was used to determine the coal rates for all steady periods in TC24C when carbon rates were above 1,000 lb/hr.

Figure D-3. Carbon Balance.

APPENDIX E LIST OF ABBREVIATIONS AND UNITS

Abbreviations

ASTM—American Society for Testing and Materials	MMD—Mass Median Diameter
CCAD—Continuous Coarse Ash Depressurization	MPT—Media and Process Technology
CFAD—Continuous Fine Ash Depressurization	NETL—National Energy Technology Lab
CMS—Carbon Molecular Sieve	PCD—Particulate Control Device
DOE—Department of Energy	PDAC—Pressure Decoupled Advanced Coal
EDS—Energy Dispersive X-Ray Spectrometry	PPC—Process Particle Counter
FEAL—Iron Aluminide	PRB—Powder River Basin
FTIR—Fourier Transform Infrared	PSD—Particle Size Distribution
GCT—Gasification Commissioning Test	PSDF—Power Systems Development Facility
IGCC—Integrated Gasification Combined Cycle	SEM—Scanning Electron Microscope
GC—Gas Chromatograph	SMD—Sauter Mean Diameter
LHV—Lower Heating Value	SRI—Southern Research Institute
LMZ—Lower Mixing Zone	TC—Test Campaign
LOI—Loss on Ignition	UMZ—Upper Mixing Zone
MCA—Multi-Cell Array	WGS—Water Gas Shift

Units

Btu—British thermal units	MMBtu—million British thermal units
°F—degrees Fahrenheit	mol—mole
ft—feet	μm—microns or micrometers
ft ³ —cubic feet	MW—megawatts
g/cm ³ or g/cc—grams per cubic centimeter	ppm—parts per million
hr—hours	ppmv—parts per million by volume
inH ₂ O—inches of water	ppmw—parts per million by weight
in—inches	psi—pounds per square inch
inwc—inches of water column	psig—pounds per square inch gauge
lb—pounds	s or sec—second
min—minutes	SCF—standard cubic feet
mm—millimeters	wt—weight