

SECTION 3 FIELD TESTING OF THE SKID-MOUNTED DSRP UNIT AT PSDF

3.1 CHRONOLOGY OF EVENTS LEADING UP TO THE PSDF TESTING

The concept of testing the skid-mounted DSRP unit at DOE's PSDF was first seriously discussed at a meeting at the PSDF site in July 1996. This was the first visit by RTI personnel to that location, and several options for establishing a working arrangement were proposed and discussed. The idea of canister exposure testing of the fixed-bed catalyst pellets was also considered and scheduled for immediate implementation.

PSDF and RTI staff worked out the specifics of the exposure test plan from February to April 1997. RTI staff fabricated the two canisters that would be placed inside the Westinghouse particulate control device (PCD) on the downstream (clean) side. Two batches of fixed-bed DSRP catalyst were pre-sulfided, following two different protocols, and stored in evacuated, sealed containers in preparation for placing in the gasifier later that year.

In September 1997, RTI conducted bench-scale, laboratory tests of several formulations of fluid-bed catalysts. Using a simulated coal gas mixture and a simulated ROG with 14% SO₂ content, the testing identified one particular material—Catalyst B, as referenced in the paper presented at the Advanced Coal-Based Power and Environmental Systems '98 Conference, included as Appendix A.

The planned August 1997 startup of the PSDF gasifier did not take place as scheduled, causing installation of the exposure canisters to be postponed. However, plans for the field test of the DSRP skid did proceed. The kick-off meeting for that activity took place in December 1997 at the PSDF site, at which the requirements for field test equipment at that site were defined.

Because the gasifier startup schedule was delayed, the DSRP skid modifications to accommodate the PSDF field test proceeded at a modest pace. In April 1999, a rigging contractor moved the process equipment skid out of the RTI shop, into the parking lot. The crew joined the heater control panel (previously intended to be installed at a remote location) to the equipment skid to make a single, larger skid.

With the initial startup of the gasifier finally scheduled for the fall of 1999, RTI shipped the canisters and sealed containers of catalyst in July 1999. On August 16, 1999, PSDF loaded the canisters and placed them on the support ring of the opened Westinghouse PCD. Several days later, that unit was closed up in preparation for the gasifier run.

A second field test kick-off meeting took place in February 2000 at PSDF. The construction of the supporting infrastructure was accelerated to accommodate commissioning schedule for November-December. The exact requirements for the field test were discussed, and the safety standards to be met were more carefully delineated. To meet those requirements, RTI scheduled a design hazard review (DHR) of the RTI-supplied equipment, to be facilitated by an outside consultant. This DHR was conducted during July-August 2000 in coordination with the DHR activities at PSDF. An intensive fabrication and construction

effort ensued in order to incorporate the required design changes and still meet the proposed project schedule.

Finally, in November 2000, contract haulers moved the trailer and the DSRP skid to the PSDF site. December 2000 to March 2001 marked an intensive period of field work, during which RTI personnel made multiple trips to Alabama to set up the equipment for the field test. The commissioning (“shakedown”) of the unit took place in March 2001. At the end of the gasifier run, the exposure test canisters were removed from the PCD, and the catalyst pellets were taken out of the perforated exposure canisters and returned to RTI.

3.2 FEATURES OF THE PSDF FACILITY

The major attraction of the PSDF site in relation to DSRP testing is the “demonstration” scale transport reactor gasifier. This air-blown gasifier has a coal feed rate of approximately 2.5 tons per hour, and a syngas production rate of 15,000-20,000 lb/hr. This gasifier is much larger than the one at Morgantown and has the potential for longer campaigns and, thus, longer on-stream time for the DSRP test unit.

On the other hand, there is no hot-gas desulfurization equipment at the PSDF; therefore, there is no stream of ROG available to feed to the DSRP. A simulated ROG stream (SimROG) had to be provided as part of the DSRP test skid. This was accomplished by including equipment to vaporize liquid sulfur dioxide (LSO₂) into a heated nitrogen stream.

3.3 LIAISON AND SCOPES OF RESPONSIBILITY FOR PSDF AND RTI

RTI personnel interfaced primarily with Southern Company Services (SCS) personnel at the PSDF site, although other organizations were present. The basic philosophy established for the field test was that SCS (and other entities, as required) would provide a place for RTI to set up the equipment, and would provide a heated slipstream of coal gas. RTI would provide all the test equipment, support equipment (Mobile Laboratory), and personnel to operate the equipment.

Figure 3 shows the proposed concept, including the routing of the slipstream line from the fifth floor of the gasifier structure down to the ground-level DSRP skid.

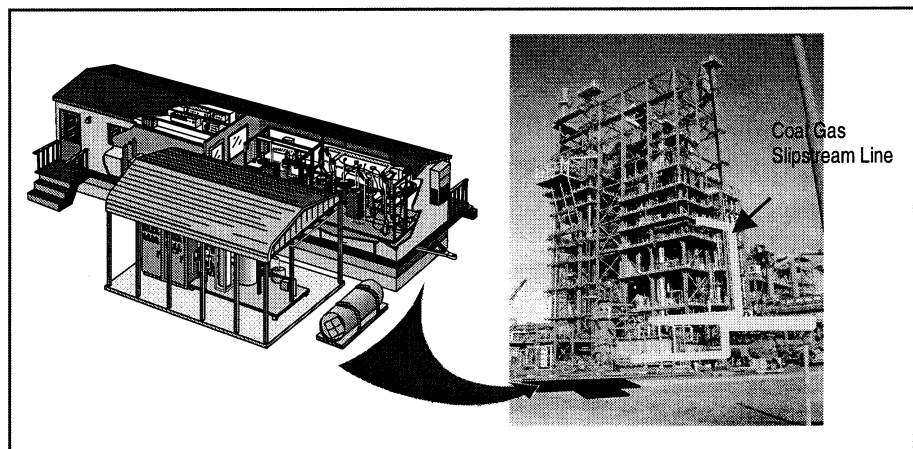


Figure 3. Slipstream Route

Figure 4 shows the final definition of the scopes of responsibility in some detail. In addition to the coal gas line, SCS also provided:

- heated process vent line back to the thermal oxidizer
- cooling water supply and return
- medium pressure nitrogen
- instrument air
- construction assistance to assemble the carport-type roof and to install the trailer and skid
- telephone line
- computer network connection (including e-mail and Internet access)
- access to the gasifier process control system (for monitoring-only of process conditions)
- electrical hookups of the skid and trailer.

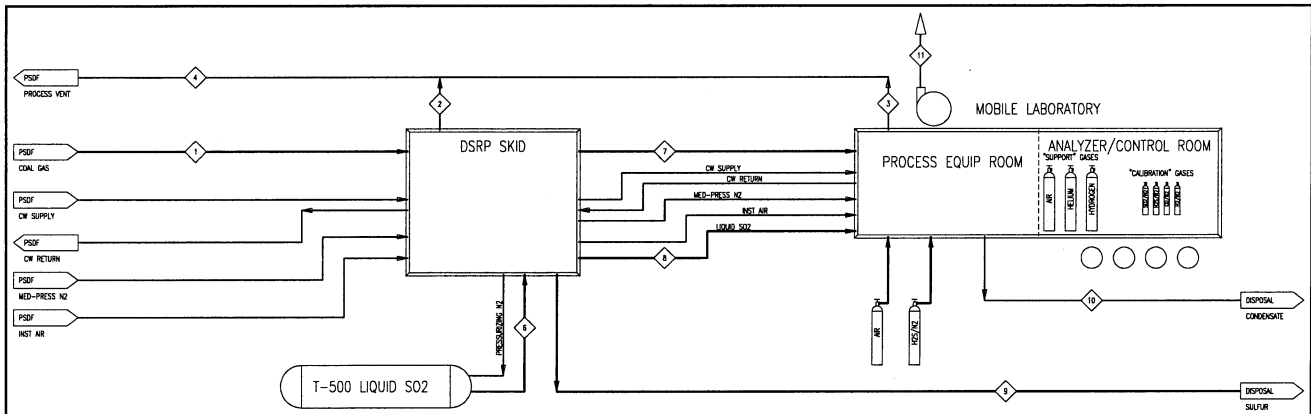


Figure 4. Scope Definition Diagram

In addition to providing the DSRP skid and the Mobile Laboratory, RTI was responsible for

- carport/shed parts
- interconnections between the skid and the trailer
- unpacking and re-assembly of all equipment at the site
- all work on the skid and/or in the trailer
- operating staff.

During the planning and liaison meetings, the possibility of exposing canisters of fixed-bed DSRP catalyst to the PSDF gasifier coal gas was discussed. All parties agreed that it would be fairly straightforward to provide that service, and informal arrangements were made to do so. Section 4, Catalyst Canister Exposure Testing, describes this activity more fully.

3.4 MODIFICATIONS TO DSRP FIELD-TEST UNIT REQUIRED FOR PSDF SITE TESTING

The decision to test the DSRP skid at PSDF meant that the equipment had to be modified considerably from its original design. As initially fabricated for the European site test, the DSRP process equipment skid was a stand-alone device without process control equipment. The heater control panel was to be located remotely from the process equipment, and process control was to be provided by the site's distributed control system. Furthermore, at that site, both actual coal gas and actual ROG were to be provided. Because the ROG was expected to have a low concentration of SO₂ (1-3%), a fixed-bed DSRP reactor was designed and fabricated.

As described in Section 3.3 above, RTI was responsible for the complete operation of the DSRP at PSDF and for providing a SimROG stream. Also at this time (1996-97), greater interest lay with testing ROG streams with higher SO₂ concentrations (up to 14%). As a fluid-bed reactor was expected to be better able to handle the higher heat of reaction of the more concentrated feed stream, the reactor on the skid-mounted DSRP field-test unit had to be rebuilt for use in the fluid-bed mode. Table 1 shows the design material balance for the PSDF testing. The flow rates were specified to achieve the optimum fluidization velocity with a 5-in diameter fluid bed inside the 6-in diameter reactor vessel.

As ROG was not available from PSDF, a system for generating a SimROG stream at high SO₂ concentrations (up to 14%) was incorporated into the skid-mounted equipment using the technique that was used successfully during the Morgantown field tests—vaporization of pressurized liquid SO₂ into a preheated nitrogen stream. An additional furnace, preheater coil, and liquid flow metering controls had to be added, and the relatively large inventory of LSO₂ that would be required caused some safety concerns (described below).

Figure 5 is a process flow diagram showing the final configuration of the skid-mounted DSRP field-test unit tested at PSDF. This diagram includes the changes made to the process (described above) as well as the changes made due to safety-related issues described in the following section.

Table 1. Design Material Balance for DSRP Field Test at PSDF

Compound	Stream									
	Molar Weight (MW)	1 CG Slip-stream	2 Liquid SO ₂	3 Nitrogen	4 Sim-ROG	5 Feed to DSRP	5A Reactor Make	5B Cond. Outlet	6 Sulfur Make	7 DSRP Tail Gas
COMPOSITION IN MOLE FRACTION										
CH ₄	16.043	0.0000								
CO	28.0134	0.1850				0.0812				
CO ₂	44.01	0.0800				0.0351	0.1163	0.1163		0.1258
H ₂ O	18	0.0800				0.0351	0.0966	0.0966		0.1044
H ₂	2.016	0.1400				0.0614				0.0000
H ₂ S	34.08	0.0005				0.0002	0.0021	0.0021		0.002274
SO ₂	64.063		1.0000		0.1401	0.0786	0.0011	0.0011		0.001137
S	32.064					0.0000	0.0755	0.0755	1.0000	0.0000
O ₂	31.9988									
N ₂	28.0134	0.5145		1.0000	0.8599	0.7083	0.7085	0.7085		0.7663
Total		1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000
MW of mixture		24.86	64.06	28.01	33.06	29.46	29.26	29.26	32.06	29.03
FLOW RATE IN GRAMS PER HOUR										
CH ₄	16.043	0				0	0	0		0
CO	28.0134	576				576	0	0		0
CO ₂	44.01	391				391	1296	1296		1296
H ₂ O	18	160				160	440	440		440
H ₂	2.016	31				31	0	0		0
H ₂ S	34.08	2				2	18	18		18
SO ₂	64.063		1275		1275	1275	17	17		17
S	32.064						613	613	613	0
O ₂	31.9988						0	0		0
N ₂	28.0134	1,601		3,423	3,423	5,024	5,024	5,024		5,024
Flow rate (kg/h)		2.761	1.275	3.42	4.70	7.46	7.41	7.41	0.613	6.79
Flow rate (g/s)		0.77	0.35	0.95	1.30	2.07	2.06	2.06	0.17	1.89
Flow rate (lb/hr)		6.09	2.81	7.55	10.36	16.45	16.33	16.33	1.35	14.98
FLOW RATE IN VOLUMETRIC UNITS										
SLPM		41		46	53	95	94	94		87
SCFH		94		104	120	215	215	215		198
SCFM		1.57		1.73	2.01	3.58	3.58	3.58		3.31
gal/hr			0.25						0.091	
gal/day									2.18	
cc/min			15.41						5.66	
Temperature (°C)		538	21	21	599	599	427	135	21	204
Temperature (°F)		1000	70	70	1110	1110	800	275	70	400
Pressure, atm, abs	19.98	24.82	21.41	19.98	19.98	19.71	19.51	1.00	2.02	
Pressure, psig		279	350	300	279	279	275	272	0	15
Density, g/cc			1.379	0.02486	0.00924	0.00823	0.0101	0.0171	1.80	
Density, lb/ft ³				1.5581	0.5794	0.5163	0.6303	1.0692		
ALPM				2.29	8.47	15.10	12.28	7.24		
ACFH				4.84	17.88	31.86	25.91	15.28		

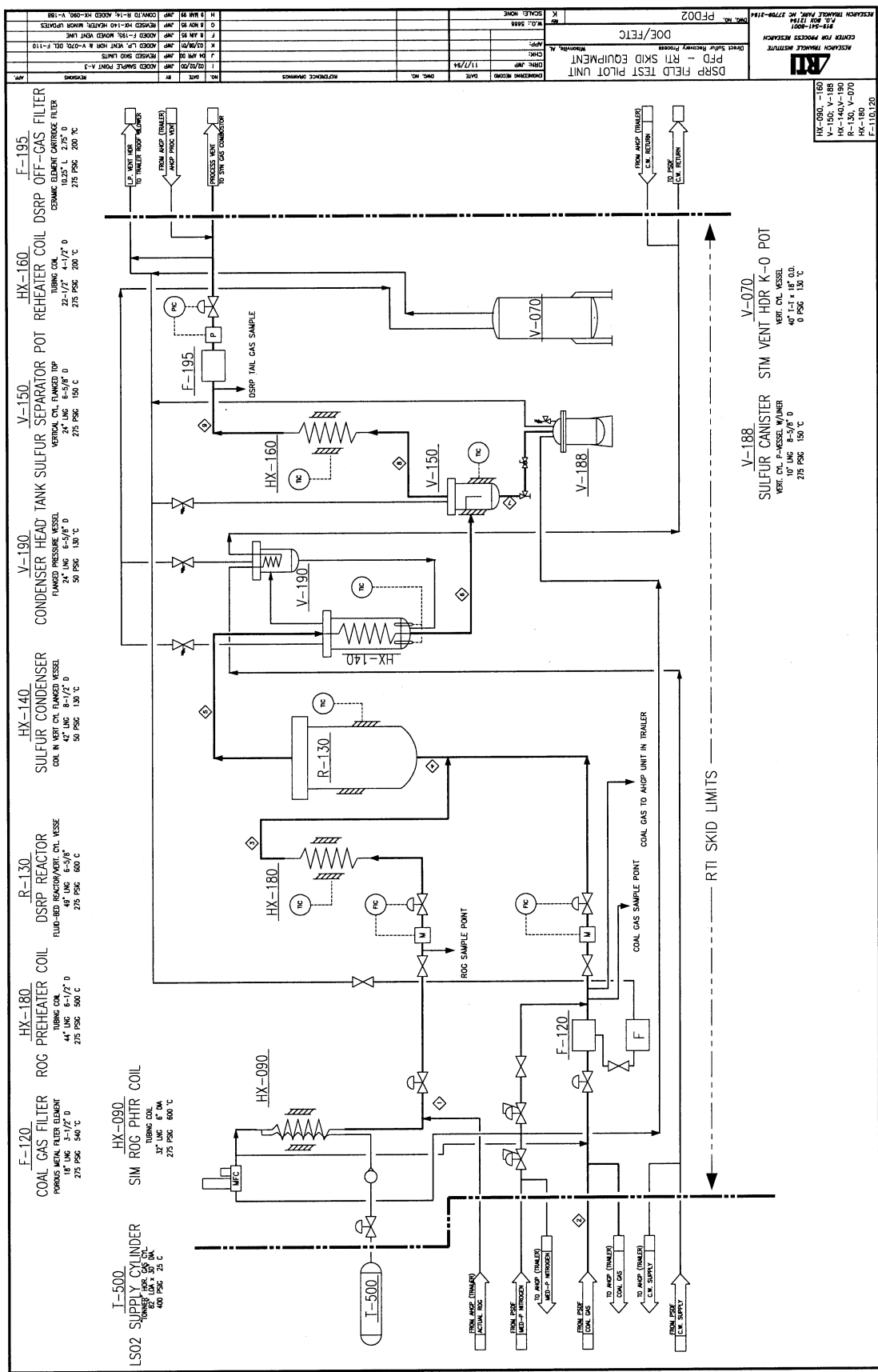


Figure 5. Process Flow Diagram for the Skid-Mounted DSRP Field-Test Unit

3.5 DESIGN HAZARD REVIEW (DHR)

At the request of the SCS personnel at PSDF charged with insuring its safe operation, RTI conducted a detailed DHR of the DSRP field-test unit and the Mobile Laboratory. A rigorous approach was used, such as might be followed for a full-scale process plant. Although research units are legally exempt from the process safety requirement contained in the Occupational Safety and Health Administration (OSHA) rules, SCS personnel felt it prudent to take a detailed look at this relatively unknown equipment to be operated on their site. Thus, RTI was held to the same safety standards as their own personnel. Appendix C reproduces the final report that was generated from the review process.

As a result of the analysis, a number of equipment modifications had to be incorporated for the RTI equipment to be approved for operation at PSDF. The main items are summarized as follows:

1. The pressure relief valves (PSVs) were required to vent to a safe location; therefore, a low-pressure vent header, ducted to trailer roof blower, was added.
2. PSVs on the steam side of the heat exchangers (which might contain water during a pressure relief) were directed to a separate vent header, in which a knockout pot was incorporated.
3. The total height of the test skid was sufficient that OSHA rules governing elevated work platforms came into play, and scaffolding-type permanent platforms with railings were added.
4. The back-pressure in the process vent (to syngas combustor) required additional isolation valves to protect RTI personnel during set-up and maintenance operations.
5. The potential for excessively high pressure in the medium-pressure N₂ supplied from PSDF to RTI meant that additional safety relief valves had to be added in several locations.
6. The potential for exposure to toxic gases from the unit resulted in the need for additional sensor heads to be added to the existing alarm system in the trailer.
7. PSVs had to be added to the analytical system inside the trailer to protect the plastic sample tubing lines.

With these required process and equipment changes, the need for which was only made clear as the dates for field testing approached, RTI staff had to exert a concerted effort to meet the schedule. Appendix D contains the final Piping and Instrumentation Diagrams (P&IDs) for the DSRP field-test unit, including the analytical equipment that was installed inside the Mobile Laboratory trailer. Figure 6 shows the final fabricated DSRP skid with the canopy removed during packing for shipment, and Figure 7 shows the skid and trailer in place at the PSDF in Wilsonville, Alabama.

3.6 DSRP PROCESS CONTROL

As stated above and as described in the 1998 Topical Report (Appendix D), the 6X DSRP test unit did not include process control equipment (hardware and software); for the PSDF testing, a complete, self-contained system was needed. RTI staff spent considerable effort designing and fabricating (including supervising vendor-supplied labor and materials) a control system for the skid-mounted DSRP field-test unit. The core of the system is a programmable logic controller (PLC) with National Instruments' Lookout™ software on a connected personal computer to provide the human-machine interface (HMI). This hardware-software combination is commonly referred to in the process control industry as a Supervisory Control and Data Acquisition (SCADA) system.

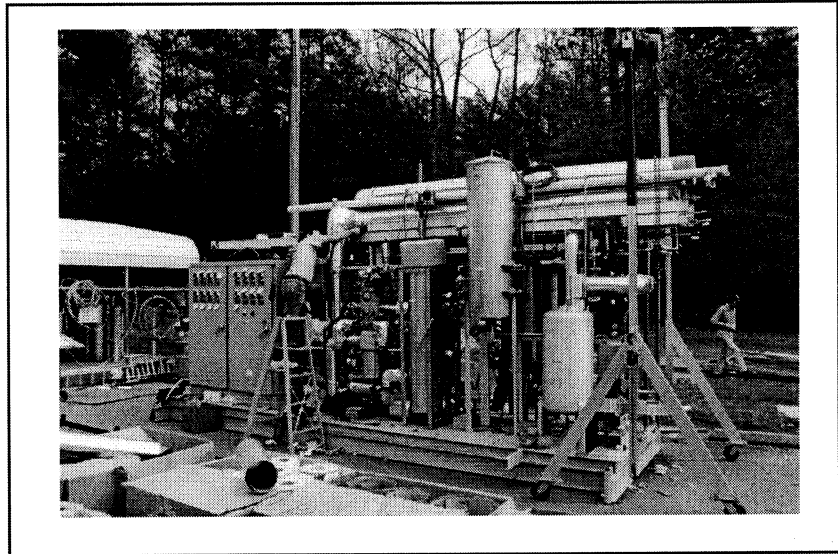


Figure 6. DSRP Skid Without Canopy

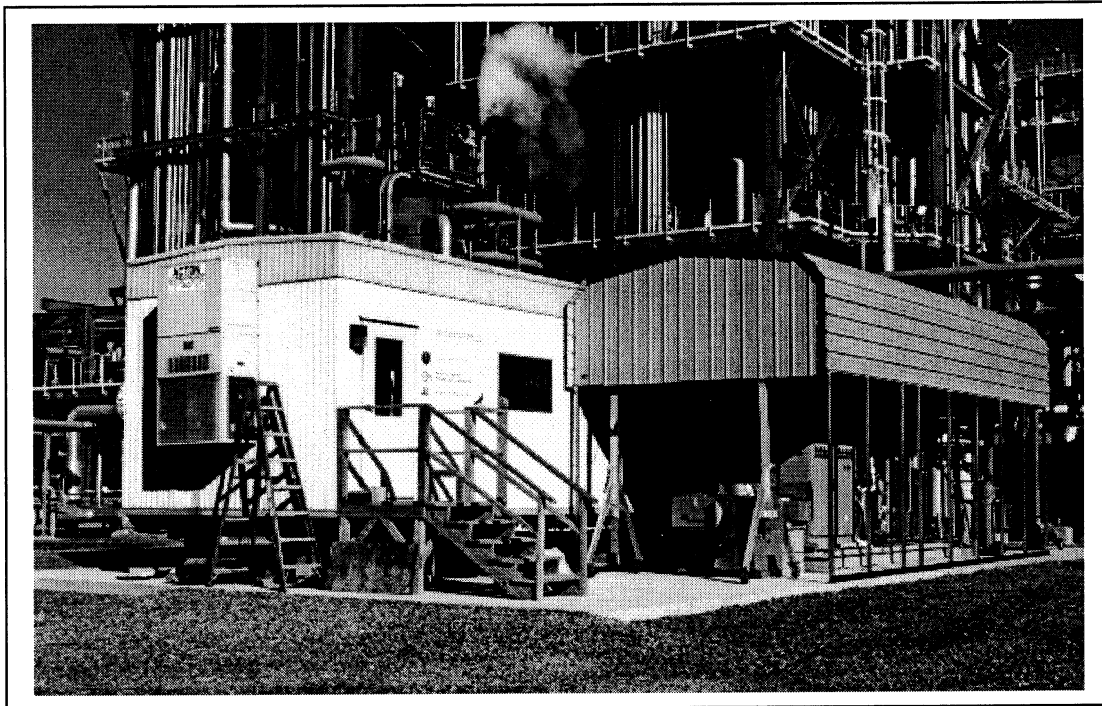


Figure 7. Trailer and Canopy at the PSDF in Wilsonville, AL

The field-test unit SCADA system performs the following functions:

- monitoring (and alarming) of process temperatures and pressures
- remote actuation of three air-operated shutoff valves for ROG, CG, and LSO₂
- modulation of the flow of two streams, ROG and coal gas (CG), through two air-operated flow control valves
- stoichiometric ratio flow control.

The separate heater control panel, mounted on the skid, controls the temperature set points of the furnaces and the heat tracing.

The trickiest control function is that the CG mass flow going to the DSRP reactor must be maintained in precise stoichiometric ratio (*i.e.*, ratio of the chemical components) with the ROG mass flow, taking into account changing chemical composition of both, as well as changing pressure and temperature at the orifice flow meters.

3.6.1 Need for Control of Reaction Stoichiometry

In the early stages of DSRP development, the researchers recognized the need to maintain the reactants in the proper stoichiometric ratio of 2.0. The final report on the bench-scale unit project (DOE Contract No. DE-AC21-90MC27224) makes this point graphically (Gangwal & Chen, p. 17, Figure 6). For example, the percent conversion to sulfur at 14.6 atm was 90% at a H₂S to SO₂ ratio of 1.87 and 84.5% when this ratio was reduced to 1.77.

From an apparatus design standpoint, achieving the correct feed stoichiometry with actual process streams (both ROG and CG), rather than synthetic mixtures prepared from compressed gas cylinders, is challenging. One expects a fair degree of natural fluctuation in composition, temperature, and pressure of actual process streams. Thus, a control system that automatically compensates for these changes (without active operator intervention) is required. As fabricated, the control scheme on the DSRP field-test unit achieves the desired functionality:

- When the operator increases the ROG flow rate, the control system automatically increases the CG flow rate in proportion.
- When the SO₂ content of the ROG decreases randomly, the CG flow rate decreases in proportion.
- When the CO or H₂ content of the coal gas decreases randomly, the CG flow rate increases.
- When the tail gas composition shows less than optimum composition, the operator can change the stoichiometric ratio to change the ratio of CG to ROG flow rate.
- When the temperature and/or pressure of the ROG or CG streams flowing through their respective orifice flow meters change, the flow control valves modulate automatically to maintain the set points based on a mass flow rate value.

3.6.2 Automatic Stoichiometric Ratio Control

The design of the automatic stoichiometric ratio flow control scheme provides the desired functionality by achieving three objectives:

- continuous measurement and control of the mass flow of SO₂ in the ROG (either simulated or actual) to the DSRP reactor
- continuous measurement of the concentration of the reactants in the coal gas (H₂ and CO)
- continuous, automatic adjustment of the mass flow of the coal gas so that the reactants will be introduced into the DSRP reactor in the desired ratio (2:1 mole ratio of reducing components to SO₂) for maximum conversion. This mass flow rate control compensates for any fluctuations in the concentrations of the SO₂, CO, and H₂, as well as fluctuations in the flow rate.

Figure 8 graphically shows the stoichiometric control system logic in a simplified P&ID format.

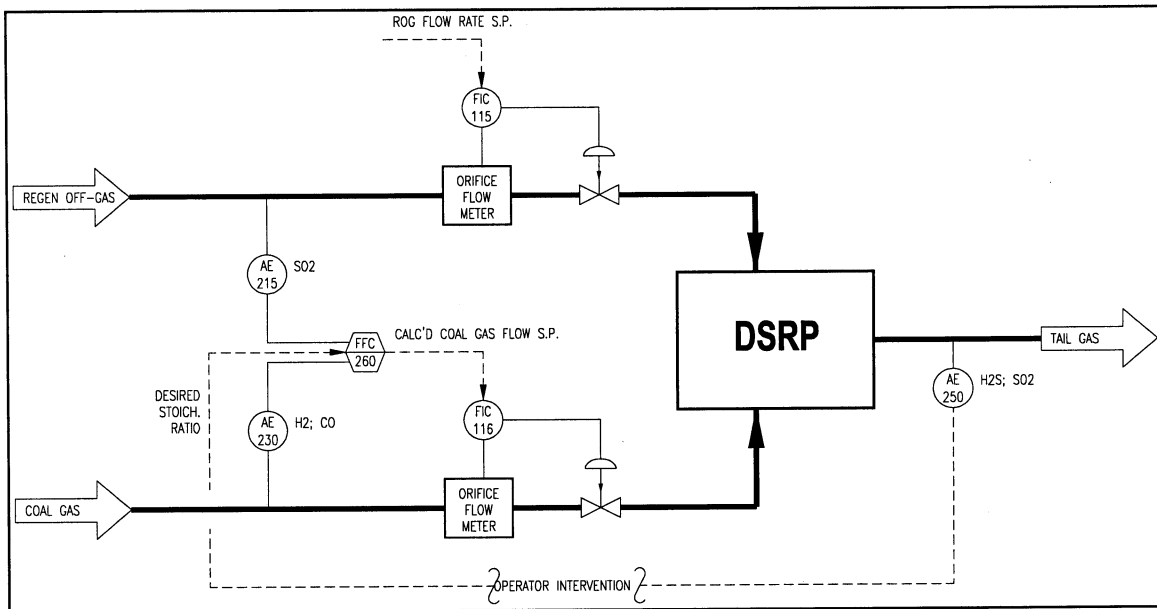


Figure 8. Stoichiometric Ratio PFD

For the purposes of programming the SCADA system, the control system logic can be described stepwise, as follows:

1. Measure the pressure drop across an orifice flow meter in the ROG line.
2. Measure the gas temperature and pressure at the orifice.
3. Based on an approximate molecular weight (keyboard entry by the operator) and pre-defined coefficients for the orifice plate, calculate the mass flow rate of the ROG.
4. Control the mass flow rate of the ROG to the set point value.
5. Measure the volumetric concentration of SO₂ in the ROG.

6. From the mass flow rate and concentration values, calculate the molar flow rate of SO_2 .
7. Calculate the required molar flow of reducing components to achieve the desired stoichiometric ratio. (Nominally this value is 2.0, but the operator can input a different value to fine tune the reactor performance.)
8. Measure the volumetric concentrations of H_2 and CO in the coal gas.
9. Calculate the required mass flow rate of coal gas (using a keyboard input value of the approximate molecular weight) to achieve the required molar flow. This value becomes the set point.
10. Measure the pressure drop across an orifice flow meter in the coal gas line.
11. Measure the gas temperature and pressure at the orifice.
12. Calculate coal gas mass flow rate. This value becomes the “process value” or PV input to a proportional control algorithm.
13. Use the control algorithm to modulate the coal gas flow control valve to change the PV in order to satisfy the set point.

Fine-tuning the process control scheme requires operator intervention; in order to determine if the optimum reactant ratio in the reactor feed has been achieved, the operator monitors the composition of the sulfur components in the tail gas.

Appendix E describes in detail the calculations and logic that were programmed into the PLC and HMI of the SCADA system.

3.6.3 Analytical Equipment

The analytical equipment installed in the trailer serves two functions: Process control and process evaluation. In terms of process control, the stoichiometric flow ratio control system (as explained above) requires composition data of the reactor feed streams in order to set the flow rates. The operator needs information on the tail gas composition to fine-tune the stoichiometric ratio.

Determination of the composition of the reducing components of the coal gas requires both a continuous analyzer and a gas chromatograph (GC). The infrared (IR) continuous analyzer measures the CO content, a reducing component, and feeds a continuous analog input signal to the PLC. The IR analyzer also measures the CO_2 content, but that is for interest only; it is not required for process control.

A GC equipped with a thermal conductivity detector (TCD) measures the hydrogen content of the coal gas. This measurement is intermittent (~15-minute cycle); additional software and hardware in the GC's control computer convert the data point into a continuous (but stepwise) analog input.

Another IR-based analyzer measures the concentration of SO_2 in the SimROG and supplies a continuous analog input to the PLC. This is the third analysis input to the process control scheme.

Two separate instruments monitor the tail gas from the DSRP, but neither is directly connected to the process control logic. An ultraviolet-based analyzer continuously measures the H₂S and SO₂ content. This measurement is very useful for observing trends and catching process upsets, but the absolute accuracy is low because the COS present in the tail gas causes an interference. For accurate measurement of all the sulfur species in the tail gas, a GC equipped with a TCD monitors H₂S, COS, and SO₂ on a 10-minute cycle.

3.6.4 Chronology of the Shakedown/Commissioning Test

The test campaign of the PSDF gasifier, designated as GCT-4, was anticipated to start in early March 2001. This date had been rescheduled several times, but by mid-February it was considered fairly firm. In addition to a lined-out gasifier operating on coal feed, the RTI test program also required that the heated slipstream line be functioning properly. Several false starts occurred during March before both of these requirements were satisfied. Finally, on March 24 the coal feed was started for the beginning of a period of several days of gasification. RTI staff immediately traveled to the site and commenced the start-up procedure.

On Monday, March 26, 2001, the field crew attempted to flow coal gas through the slipstream line. After a few start-up glitches, flow was achieved 4 hours later and checkout of the RTI coal gas analytical equipment commenced. Twelve hours later, in the early morning of March 27, the coal gas sample regulator had to be replaced due to plugging.

Following several hours of successful flow control and analysis of the coal gas slipstream, the crew started up the SimROG flow. The DSRP reaction could not be lined out, apparently due to coal gas flow control problems that surfaced. That afternoon it was discovered that the impulse lines on the coal gas flow orifice meter had become plugged; naphthalene was suspected. Both coal gas and SimROG flows were restarted late that afternoon and the process was run continuously but with erratic tail gas compositions.

In an effort to smooth out the composition changes of the SimROG, a design change was incorporated: During the early morning of March 28, the night shift staff relocated the LSO₂ needle valve flow controller to be downstream of the rotameter. They took the opportunity to unplug the coal gas sample regulator again at this time, and restarted the process after only a few hours of downtime. Control of the process definitely improved.

Later in the morning of March 28, the process tail gas sample line became plugged, presumably with elemental sulfur. That line was re-routed and better analyses of the tail gas ensued. At this time, the best period of operation began.

By late morning of March 28, it seemed that enough LSO₂ had run into the process that a sizeable quantity of elemental sulfur should have collected. The collection canister was isolated and opened to reveal only a very small quantity of elemental sulfur. Apparently, the sulfur condenser and separator pot, as designed, had failed to capture the sulfur mist. One hypothesis was that the vessel heat tracing temperature was too high, causing the condensed sulfur to be re-vaporized. Therefore, after reassembly of the collection canister, the set point was lowered.

The unit continued to operate smoothly during the afternoon, until problems developed in controlling the coal gas flow and the LSO₂ flow had to be shut off as the orifice flow meter was worked on. LSO₂ flow was restarted that evening, but had to be shut down because the coal gas differential pressure transmitter impulse lines were once again completely plugged with naphthalene.

The decision earlier in the day to adjust the set point of the heat tracing had an unintended consequence. Around 02:00 on March 29, it became apparent that the outlet piping of the whole system had become plugged. The field crew worked most of that day to disassemble parts of the apparatus to find the plugs, and no LSO₂ feed was permitted that day due to stack testing of the main PSDF stack. The efforts to unplug the system were unsuccessful. As March 30 was the scheduled end of the gasifier run, no further attempts were made to operate the DSRP.

3.6.5 Observed Performance of the DSRP Reactor During Shakedown

For the shakedown/ commissioning test, a generic fluid-bed catalyst was charged to the reactor. Because it was anticipated that potential upsets in operation during the initial runs might cause the loss of some or all of the catalyst charge, an optimized formulation of a fluid-bed DSRP catalyst was not used. Thus, low conversion efficiencies of around 80% were not unexpected.

The longest period of continuous operation was on March 28, 2001, with 13.5 hours of LSO₂ feed. The best performance of the DSRP reaction was during the period from 11:00 to 14:00; Table 2 summarizes the operating conditions.

Table 2. Operating Conditions on March 28, 2001, During Period of Best Performance

General Operating Conditions	
Reactor pressure	202 psig
Reactor temperature	
Bottom of reactor (inlet)	275 °C
Catalyst bed (1" above frit)	570 °C
Catalyst bed (7" above frit)	515 °C
Catalyst bed (14" above frit)	515 °C
SimROG	
Flow rate	37 SLPM
SO ₂ concentration	10 vol%
Coal Gas	
Flow rate	34 SLPM
CO concentration	9-10 vol %
H ₂ concentration	approx. 8 vol%

During this time period, the tail gas composition showed H₂S to SO₂ in the preferred 2:1 ratio, indicating that the optimum 2:1 ratio of reducing gas to SO₂ had been achieved in the feed. As Figure 9 shows, the tail gas sulfur compound concentrations were somewhat high, however, indicating that the SO₂ conversion was lower than expected. As calculated from the gas analyses, approximately 79-82% of the SO₂ in the inlet was converted to elemental sulfur in the 11:00-14:00 time period.

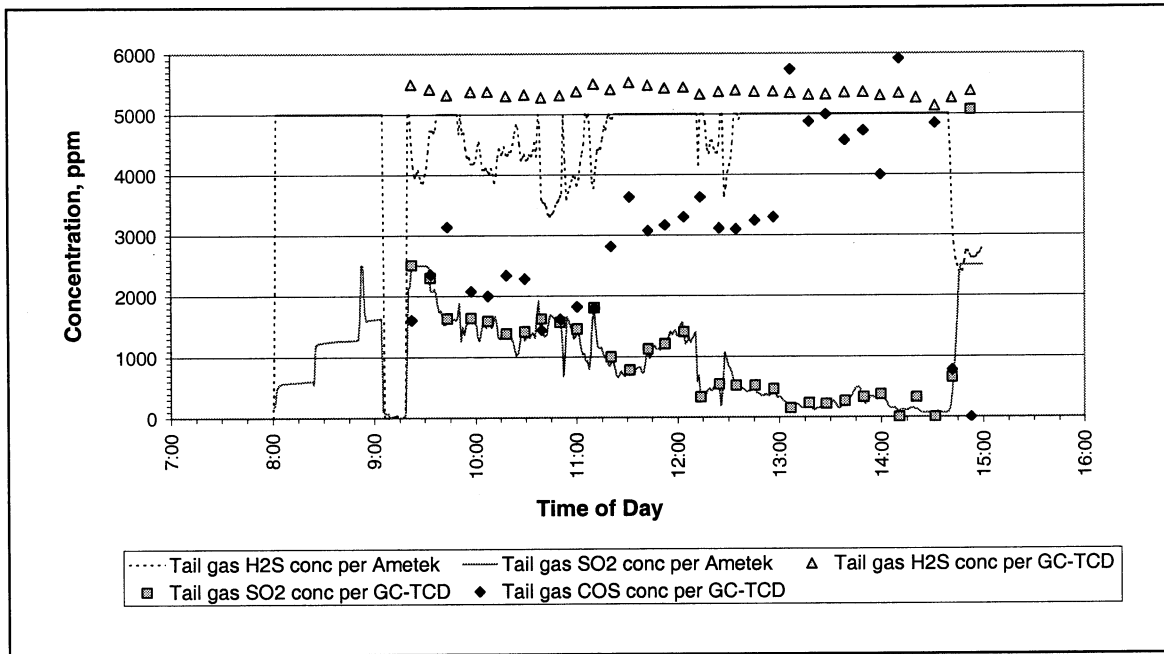


Figure 9. Tail Gas Composition, March 28, 2001

Table 3 presents the material balance derived (in part) from the observed performance of the field-test unit. The measured syngas compositions are used at the actual operating pressure, which was lower than design (202 psig [1.39 MPa] compared to 279 psig [1.92 MPa]). The content of the reducing components in the coal gas, H_2 and CO , was also much lower. To calculate the material balance, the extent of the reaction was inferred from the measured tail gas composition and was used to calculate the reactor outlet stream. The overall effect is that the expected yield of elemental sulfur in this material balance (201 g/h) is considerably lower than the design value (613 g/h).

Because the DSRP reaction is highly exothermic, a significant rise in catalyst bed temperature is expected when LSO_2 feed is started. Figure 10 plots the temperatures logged by the SCADA system for the four thermocouples on the inside of the reactor. A bed temperature rise of $150\text{ }^\circ\text{C}$ (over a period of approximately 3 hours) occurred when the LSO_2 feed was re-started at 02:42. Similarly, a $150\text{ }^\circ\text{C}$ -drop occurred over a period of about 4 hours when the LSO_2 feed was cut off at 16:13. Calculations performed during the DHR suggested that a catalyst bed temperature rise of over $300\text{ }^\circ\text{C}$ should be expected, based on full conversion of the feed SO_2 and minimal heat leak from the reactor. The less-than-expected temperature rise is confirming evidence of the low conversion.

An interesting observation with this test run is the importance of the COS in the tail gas, which accounted for 6 to 7 percentage points of the 20% of the sulfur in the feed that remained in the tail gas.

Table 3. Approximate Material Balance During Operation on March 28, 2001

Compound	Stream									
	Molar Weight (MW)	1 CG Slip-stream	2 Liquid SO ₂	3 Nitrogen	4 Sim-ROG	5 Feed to DSRP	5A Reactor Make	5B Cond. Outlet	6 Sulfur Make	7 DSRP Tail Gas
COMPOSITION IN MOLE FRACTION										
CH ₄	16.043	0.0000								
CO	28.0134	0.1100				0.0579	0.0058	0.0058		0.0060
CO ₂	44.01	0.0800				0.0421	0.0927	0.0927		0.0963
H ₂ O	18	0.0800				0.0421	0.0744	0.0744		0.0773
H ₂	2.016	0.0700				0.0369	0.0037	0.0037		0.0038
H ₂ S	34.08	0.0005				0.0003	0.0003	0.0003		0.000275
SO ₂	64.063		1.0000		0.1001	0.0474	0.0063	0.0063		0.006593
S	32.064					0.0000	0.0381	0.0381	1.0000	0.0008
COS	60						0.0019	0.0019		0.002015
O ₂	31.9988									
N ₂	28.0134	0.6595		1.0000	0.8999	0.7733	0.7767	0.7767		0.8068
Total		1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000
MW of mixture		26.68	64.06	28.01	31.62	29.02	28.99	28.99	32.06	28.87
FLOW RATE IN GRAMS PER HOUR										
CH ₄	16.043	0				0	0	0		0
CO	28.0134	274				274	27	27		27
CO ₂	44.01	313				313	687	687		687
H ₂ O	18	128				128	225	225		225
H ₂	2.016	13				13	1	1		1
H ₂ S	34.08	2				2	2	2		2
SO ₂	64.063		513		513	513	68	68		68
S	32.064						205	205	201	4
COS	60						20	20		20
O ₂	31.9988						0	0		0
N ₂	28.0134	1,644		2017	2017	3661	3661	3661		3661
Flow rate (kg/h)		2.374	0.513	2.02	2.53	4.90	4.90	4.90	0.201	4.70
Flow rate (g/s)		0.66	0.14	0.56	0.70	1.36	1.36	1.36	0.06	1.30
Flow rate (lb/hr)		5.23	1.13	4.45	5.58	10.81	10.80	10.80	0.44	10.35
FLOW RATE IN VOLUMETRIC UNITS										
SLPM		33		27	30	63	63	63		60
SCFH		75		61	68	143	143	143		137
SCFM		1.26		1.02	1.13	2.39	2.38	2.38		2.29
gal/hr			0.10						0.030	
gal/day									0.72	
cc/min			6.20						1.86	
Temperature (°C)		538	21	21.	599.	599.	427.	135.	21.	204.
Temperature (°F)		1000	70	70	1110	1110	800	275	70	400
Pressure, atm, abs		15.29	24.82	21.41	15.29	15.29	14.95	14.75	1.00	2.02
Pressure, psig		210	350	300	210	210	205	202	0	15
Density, g/cc			1.379	0.02486	0.00676	0.00620	0.0076	0.0128	1.80	
Density, lb/ft ³				1.5581	0.4239	0.3890	0.4734	0.8005		
ALPM				1.35	6.24	13.17	10.77	6.37		
ACFH				2.85	13.16	27.80	22.81	13.49		

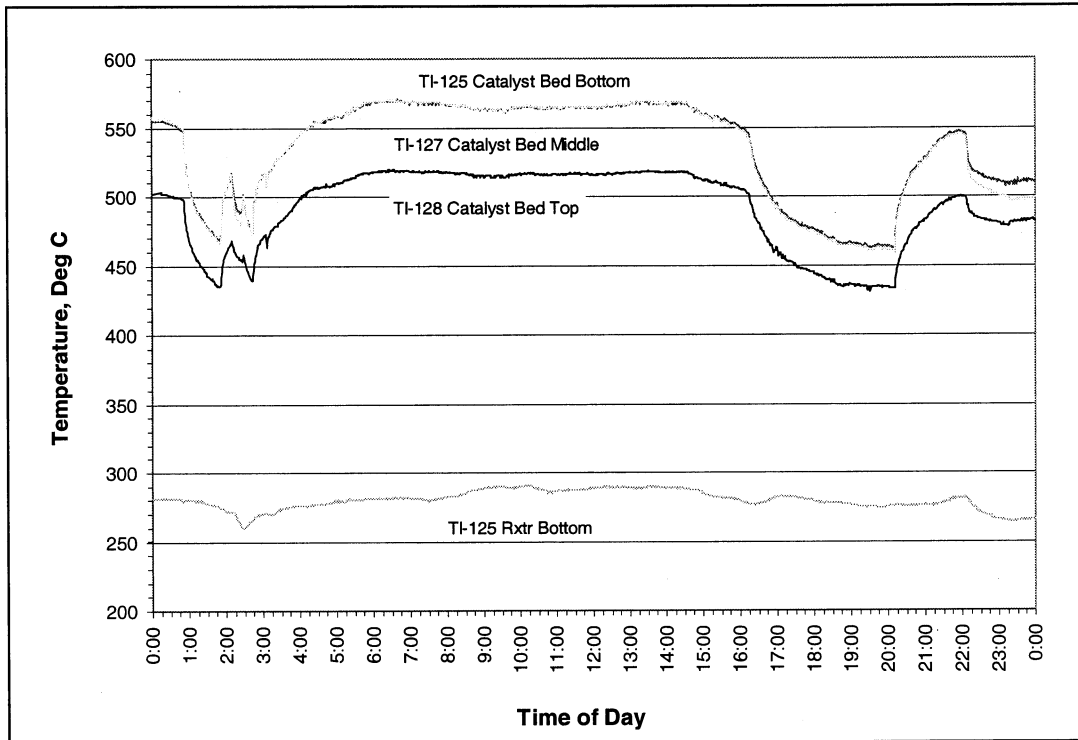


Figure 10. Reactor Temperatures, March 28, 2001

3.6.6 Lessons Learned from the Shakedown/Commissioning

Table 4 summarizes the on-stream time for the shakedown test. Syngas was passed through the fluid-bed reactor in the field-test unit for a total of 54 hours. During that time, SimROG (from vaporization of liquid SO₂) was being fed for 30 hours.

Although the skid-mounted DSRP field-test unit was operated for only a relatively brief period of time, a great deal of valuable information was gathered. That the unit was able to be run at all represents the accomplishment of many significant project milestones by both RTI and Southern Company Services at PSDF:

- completed construction of the skid-mounted DSRP field-test unit at RTI
- completed modification of the Mobile Laboratory (trailer) for use as the control room for the skid-mounted DSRP
- designed and fabricated a PLC-based SCADA-type process control system
- constructed both the skid and the trailer to meet the requirements of the designated test site, PSDF, in terms of safety and interface/operability
- shipped the skid and trailer to the PSDF site in Wilsonville, Alabama
- installed the skid and trailer at the PSDF site
- connected the systems to the PSDF-supplied process and utility lines
- passed coal gas through a long, heat-traced slipstream line and successfully measured its flow and composition

Table 4. Summary of On-Stream Times

Time	Syngas			LSO ₂		
	On	Off	Elapsed	On	Off	Elapsed
3/26/01 14:05	√					
3/26/01 16:15		√	2:10:00			
3/26/01 17:00	√					
3/27/01 5:25		√	12:25:00			
3/27/01 6:00	√					
3/27/01 7:52				√		
3/27/01 13:17					√	5:25:00
3/27/01 13:30		√	7:30:00			
3/27/01 15:50	√					
3/27/01 16:25				√		
3/28/01 0:35					√	8:10:00
3/28/01 1:50				√		
3/28/01 2:24					√	0:34:00
3/28/01 2:42				√		
3/28/01 16:13					√	13:31:00
3/28/01 20:10				√		
3/28/01 23:15					√	3:05:00
3/28/01 23:15		√	31:25:00			
TOTAL			53:30:00			30:45:00

- generated a simulated ROG by safely vaporizing LSO₂
- controlled the skid-mounted DSRP field-test unit remotely using the SCADA system, and logged useful process monitoring data.
- demonstrated that the process control system could continuously monitor the reactor inlet streams and adjust the coal gas flow rate automatically to maintain the required feed stoichiometry.

The shakedown test achieved its intended purpose of identifying areas of improvement as follows:

- The major issue to be resolved will be elimination of naphthalene plugging of the sample and impulse lines.
- The heat tracing in several locations needs to be improved in order to eliminate cold spots and sulfur plugging.
- The main reactor furnace needs to be changed so that the desired reaction temperature of 600-630°C can be achieved.
- The control system instrument parameters need fine-tuning to smooth out the responses.
- The sulfur collection system needs to be improved so that the sulfur mist can be captured effectively.