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Integrated Operation of a Pressurized Fixed-Bed Gasifier, Hot Gas Desulfurization System, and Turbine Simulator

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4.1 Integrated Operation of a Pressurized Fixed Bed Gasifier, Hot Gas Desulfurization System and Turbine Simulator

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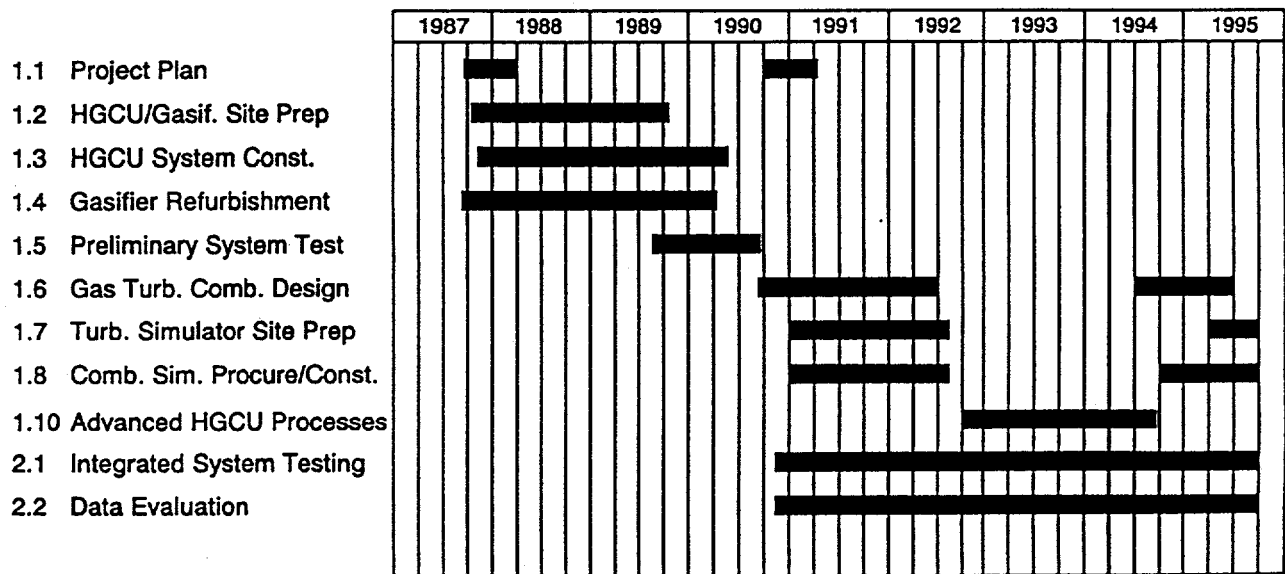
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Period of Performance September 30, 1987 to September 30, 1995

Schedule and Milestones

Project Schedule



Today

OBJECTIVES

The overall objective of the General Electric Hot Gas Cleanup (HGCU) Program is to develop a commercially viable technology to remove sulfur, particulates, and halogens from a high-temperature fuel gas stream using a moving bed, regenerable mixed metal oxide sorbent based process. This technology will ultimately be incorporated into advanced Integrated Gasification Combined Cycle (IGCC) power generation systems. In IGCC applications, HGCU is projected to improve overall power generation cycle efficiencies by 1 to 2% compared with conventional low-temperature cleanup technologies, and will also offer the potential for reducing plant capital and operating costs.

The HGCU Program is based on the design and demonstration of the HGCU system in a test facility made up of a pilot-scale fixed bed gasifier, a HGCU system, and a turbine simulator in Schenectady, NY, at the General Electric Research and Development Center (GE-CRD). A modified GE MS6000 gas turbine combustor and a film-cooled, first-stage LM6000 nozzle assembly are incorporated into the turbine simulator.

The objectives of the turbine simulator testing are (1) to demonstrate the suitability of fuel gas processed by the HGCU system for use in state-of-the-art gas turbines firing at "F" conditions (2350°F rotor inlet temperature) and (2) to quantify the combustion characteristics and emissions on low-Btu (LBtu) fuel gas. The turbine simulator program also includes the development and operation of experimental combustors based on the rich-quench-lean concept (RQL) to minimize the conversion of ammonia and other fuel-bound nitrogen species to NO_x during combustion.

The HGCU system and turbine simulator have been designed to process approximately 8000 lb/hr of low heating value fuel gas produced by the GE fixed bed gasifier. The raw fuel gas is provided to the HGCU and turbine simulator systems at 280 psig and 1000°F. The HGCU system has utilized several mixed metal oxide sorbents — including zinc ferrite, zinc titanate, and Z-Sorb (a proprietary zinc-based sorbent developed by Phillips Petroleum Company) — with the objective of demonstrating good sulfur removal and mechanical attrition resistance as well as economic cost characteristics. Demonstration of halogen removal and the characterization of alkali and trace metal concentrations in the fuel gas are subordinate objectives of the overall program.

BACKGROUND INFORMATION

As a result of the availability of advanced gas turbine combined cycles that fire at temperatures of over 2350°F and the advent of integrated gasification/oxygen separation technology, IGCC plants now offer overall plant efficiencies of 42–44% with extremely low emissions of SO_x , NO_x , and particulate.

Although the capital cost of IGCC technology is higher than conventional coal-fired steam turbine plants, it is rapidly dropping to competitive levels. As IGCC power generation technology reaches the early stage of demonstration and commercialization, the first plants built will use conventional low-temperature gas-cleaning technology. However the goal of HGCU is that, when fully developed, it will represent a "second generation" cleanup technology offering further improvements in IGCC power generation efficiency and operation flexibility, as well as reduced capital and operating costs.

Fuel gas derived from coal by gasification processes contains sulfur in the form of H₂S and COS at concentrations of up to several thousand parts per million (ppm). In order to comply with increasingly stringent environmental regulations and to meet the conditions required for the long life of gas turbine components, it is necessary to reduce both the sulfur and particulate in coal-derived fuel gas to low levels. In the temperature range of approximately 800–1200°F, mixed metal oxides, such as zinc oxide, react with these gaseous sulfur species, forming metal sulfides under reducing conditions. These metal sulfides can then be regenerated to their original oxide state, producing a sulfur-dioxide-rich off-gas suitable for conversion either to sulfuric acid or to elemental sulfur. GE Environmental Systems, Inc. (GEESI) has patented a moving bed process to utilize mixed metal oxide sorbents to remove sulfur from a high-temperature fuel gas stream and regenerate the sorbent for reuse while producing a byproduct stream suitable for the recovery of sulfur.

The history of this project and the concurrent development of high-temperature particulate removal systems based on cyclones has been

documented by Smith et al. (1987), Cook et al. (1988, 1989, 1990, 1991, and 1992), and Bevan et al. (1994a and 1994b). In addition, the previous development of the mixed metal oxide sorbents and turbine simulator have been documented by Ayala et al. (1992, 1993, and 1994) and Feitelberg et al. (1993 and 1994).

PROJECT DESCRIPTION

The HGCU Program is based on the design and demonstration of gas cleaning technologies at GE-CRD's Process Evaluation Facility (PEF) located in Schenectady, NY. This work is being carried out under a current DOE contract, with the most recent schedule shown in the Schedule section at the beginning of this paper. Construction of the basic facility was completed in late 1990, with continuing modifications and test operation taking place since that time. A summary of the test runs performed to date is shown in Table 1. These tests include 13 long duration runs (approximately 100 hours each), incorporating a variety of new hardware features and sorbents. Test 8 is scheduled for later in 1995.

Table 1. Gasifier/HGCU/Turbine Simulator Test Operation — 20 atm, 1000°F

Run	Date	Run Hours	Coal Type	System Configuration
1-2B	12/90-4/92	230	I6	Gasifier/HGCU/GT Valve
3	8/30/92	88	I6	Gasifier/HGCU/GT Valve/MS6000 Simulator
3A	2/7/93	120	Anthracite	Gasifier/HGCU/GT Valve/MS6000 Simulator
4	6/13/93	100	I6, Crown II	Gasifier/HGCU/GT Valve/Simulator/Halogen
5	11/1/93	103	I6, Crown II	Gasifier/HGCU/GT Valve/Halogen/"F" Simulator 2300°F
6	5/1/94	85	I6	Gasifier/Guard Bed/HGCU/GT Valve/Halogen/"F" Simulator 2500°F
7A	8/14/94	99	I6	Gasifier/HGCU/GT Valve/Halogen/"F" Simulator 2500°F
4A	11/13/94	115	I6, Anthracite	Gasifier/HGCU/GT Valve/R-Q-L Sub-Scale
7B-I, 7B-II	3/5/95 3/26/95	} 190	I6	Gasifier/HGCU/GT Valve/CFB Halogen/Simulator 2500°F
8	3Q95	100-200	I6	Gasifier/HGCU/GT Valve/CFB Halogen/RQL Full-Scale

A schematic of the PEF facility, as configured at the conclusion of Test 7B, is shown in Figure 1. The GE-CRD fixed bed, pressurized air-/steam-blown gasifier is shown on the left. The gasifier includes a water-cooled bed stirrer, alternating pressurized coal feed lock-hoppers, and a rotating grate for ash removal. The LBTu coal gas is directed from the gasifier to the primary cyclone located immediately downstream of the gasifier, through the recently installed circulating fluidized bed (CFB) chloride removal vessel, then to the HGCU system, which consists of the absorber and associated upper and lower lock-hoppers, regenerator with recycle gas system, fines screen, and elevator. A secondary cyclone is located downstream of the HGCU system, and the cleaned fuel can be directed either to the MS6000 turbine simulator or to the system flare. Using analytical instrumentation in "real time" to allow process control, both the process fuel gas and the recycle gas are sampled continuously during the run. Solid flows are sampled periodically for later chemical analysis.

RESULTS

Testing of the GE HGCU pilot system has been underway since December 1990. During this period, three sorbents and a variety of constantly improving system configurations have been tested, including zinc ferrite, zinc titanate and Z-Sorb sorbents, a single-/two-stage regenerator with mass flow bottom and annular reaction zones, alternate recycle blower configurations, and dedicated vessels for removing chlorides from the gas streams. Since the last contractor's report, three long duration tests have been completed in the following sequence: 7A, 4A, and 7B (7B was completed in two parts). In each test Z-Sorb sorbent was used for sulfur removal. Test 7A, completed in August 1994, was the second long duration test using Z-Sorb sorbent. Included in the run were operation of the chloride guard bed and the turbine simulator with a scaled fuel nozzle based on the nozzle design for the Tampa Electric Company project. During Test 4A, the sub-scale RQL combustor was operated for the first time on

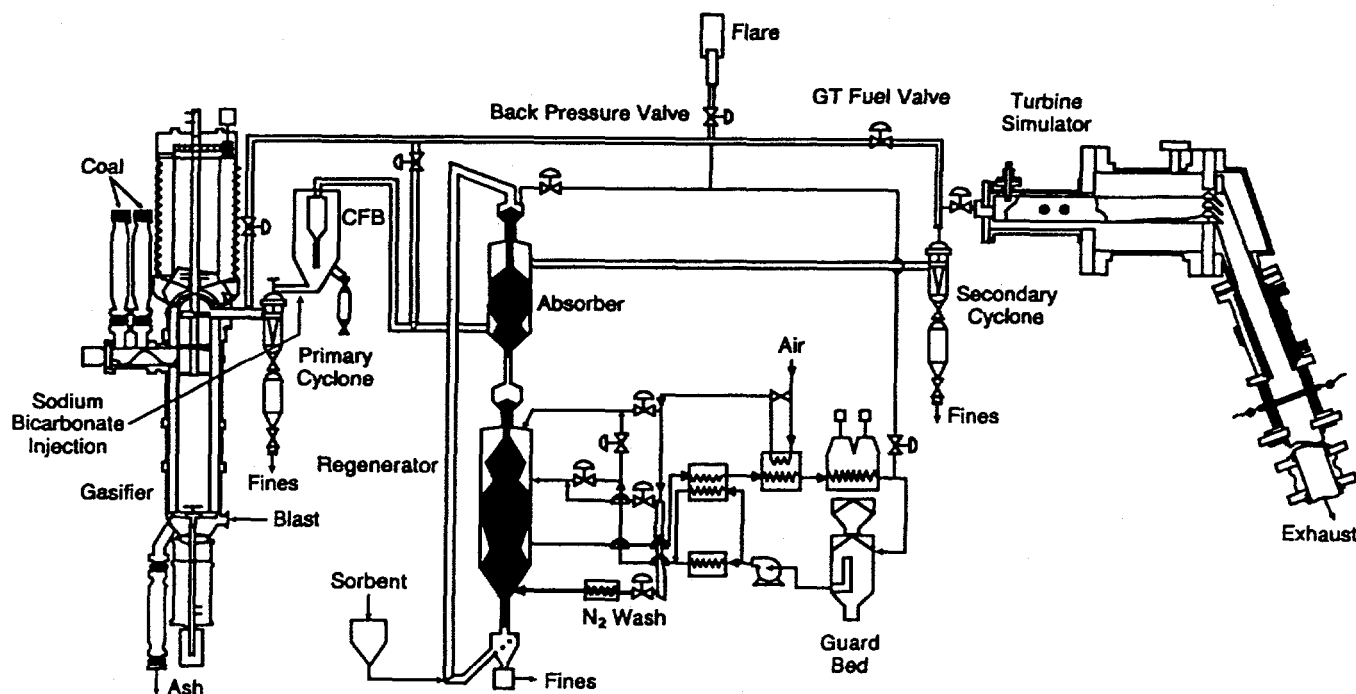


Figure 1. GE Gasifier/Hot Gas Cleanup Process Flow Diagram

coal gas to demonstrate the feasibility of applying the staged combustion concept to minimize conversion of fuel-bound nitrogen to NO_x . In this test the Z-Sorb sorbent used for Tests 6 and 7A was bolstered with fresh makeup to further evaluate its performance under pilot conditions. Tests 7B-I and 7B-II included initial operation of the CFB chloride removal system using sodium bicarbonate injection, in addition to testing of the HGCU on a fresh charge of Z-Sorb sorbent. The turbine simulator was operated with a modified fuel nozzle for improved part-load operation. The hardware modifications, operating conditions, and system performance for each test are discussed in detail in the following sections.

Test 7A - Conditions and Hardware Configuration

Long Duration test 7A was performed at GE-CRD's pilot plant in August 1994 using Z-Sorb sorbent. This sorbent had been used previously for

approximately 80 hours of integrated operation in Test 6 and demonstrated acceptable operation at the 25% sulfur loading tested, with sulfur removal efficiencies of 98-99% achieved at steady state. Illinois 6 coal, nominal 1.7% sulfur, was used as the test coal for Test 7A. Anthracite coal (0.5% sulfur) was used for startup and shutdown to minimize tar condensation from the fixed bed gasifier.

Because test data on absorber samples showed a significant range of sulfur loadings on sorbent sampled at the outlet, the absorber was modified between Tests 6 and 7A to incorporate a mass flow bottom to improve the uniformity of sorbent flow. A similar modification was tested successfully on the regenerator in Test 6 and significantly improved the uniformity of solids loading. The current regenerator configuration and the modified absorber are shown in Figure 2. Off-line testing with sorbent after the modification indicated uniform transfer of solids with little radial movement. The two-stage regenerator configuration

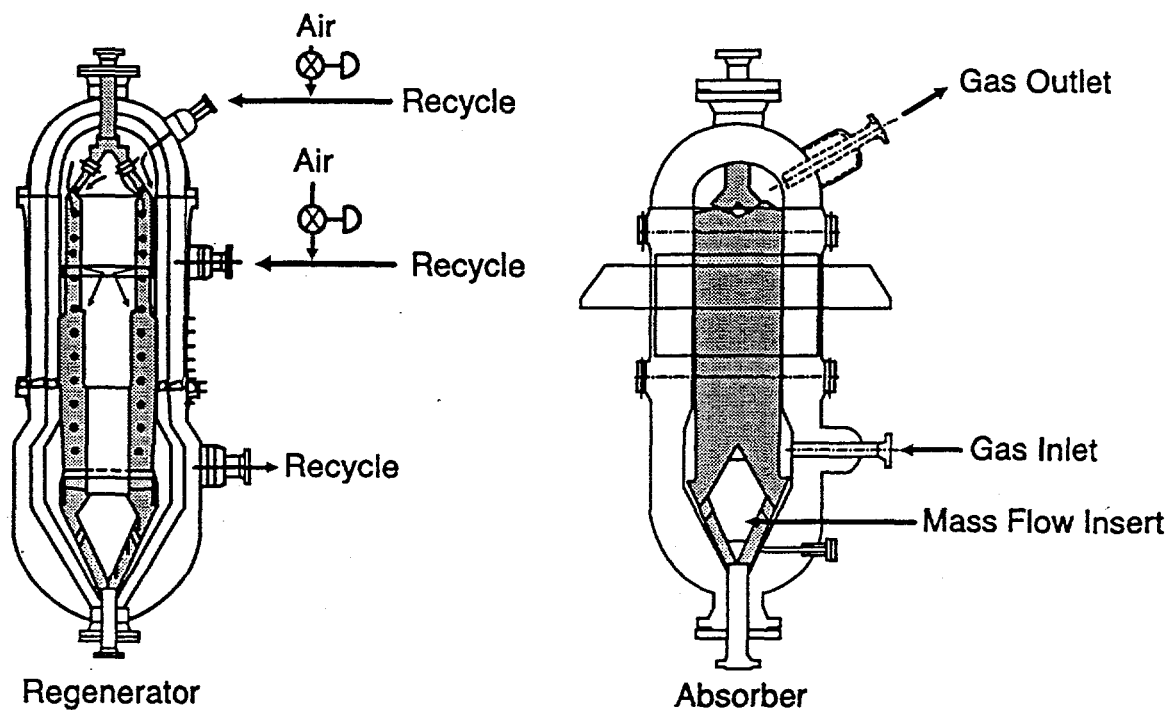


Figure 2. Regenerator and Absorber Vessel Configurations - Test 7A

with automatic control of temperatures to the first and second stages that was developed in previous testing was preserved for Test 7A.

The exotherm generated by the combustion of sulfur in the regenerator is controlled in the GE system by modulating the oxygen concentration to each stage and by controlling the recycle flowrate through the regenerator to provide convective cooling. Data from previous tests indicated that the throughput of the system was limited by the necessity for controlling regenerator temperatures to the 1400°F level anticipated for Z-Sorb sorbent. Modifications were made to the recycle compressor to upgrade the motor to a larger, higher speed drive prior to Test 7A. This upgrade resulted in a 20% increase in available recycle flow, which in turn provided for additional temperature modulation, higher throughput, and improved control of the regeneration process. During operation, a portion of the increased capacity is used to automatically modulate peak temperatures. A high-temperature third-stage nitrogen source was added to the regenerator for control of sulfate formation

As previously reported, chlorides in the coal gas stream cause problems to the HGCU system by absorbing on the sorbent in the absorber with subsequent release in the recycle gas during regeneration. In the regenerator the chlorides react with the sorbent and SO₂ to form zinc sulfate and zinc chloride deposits in the recycle loop heat exchangers, leading to blockage after several days of operation. In Test 6A, a combination of in-duct injection of sodium bicarbonate upstream of the gasifier primary cyclone and a guard bed using sodium bicarbonate pellets in the recycle loop had successfully controlled recycle loop deposits. For Test 7A, a zinc/calcium-based sorbent material manufactured by United Catalyst Inc. was substituted for the sodium bicarbonate pellets to

reduce problems with moisture generation and handling.

Modifications to the MS6000 turbine simulator included a new fuel nozzle patterned after the nozzle being developed by GE Power Generation for the Tampa Electric Company's MS7000F gas turbine.

Test 7A - Results

Test 7A was conducted to obtain additional understanding of the operational characteristics of the Z-Sorb sorbent and, more specifically, to load the sorbent to the 50% of theoretical sulfur levels required for the Tampa Electric Company demonstration project. The gasifier and regenerator were operated for 100 hours, with 84 hours of integrated operation of the HGCU system achieved. The sulfur content of the coal gas ranged from 1000 ppmv H₂S during startup on anthracite to 3500 ppmv on Illinois 6. As indicated in Figure 3, H₂S levels in the fuel gas downstream of the absorber ranged from 50 ppmv during steady state to 700 ppmv during periods of interrupted sorbent transfer or off-design operation.

The HGCU system was started at a sorbent transfer rate equivalent to 25% sulfur loading, as successfully demonstrated in Test 6; then the transfer rate was lowered to increase the residence time and sulfur loading to the 50% target. Problems with a rotary feeder drive early in the test period led to excessively low transfer rates with resulting high outlet H₂S concentrations. The problem was corrected approximately 18 hours into the run, and outlet sulfur levels returned to acceptable levels as sorbent transfer rates equivalent to 25% loading were achieved. As sorbent transfer rates were decreased to achieve higher loadings, however, the outlet H₂S levels

increased, giving rise to the spikes in H₂S levels seen in Figure 3.

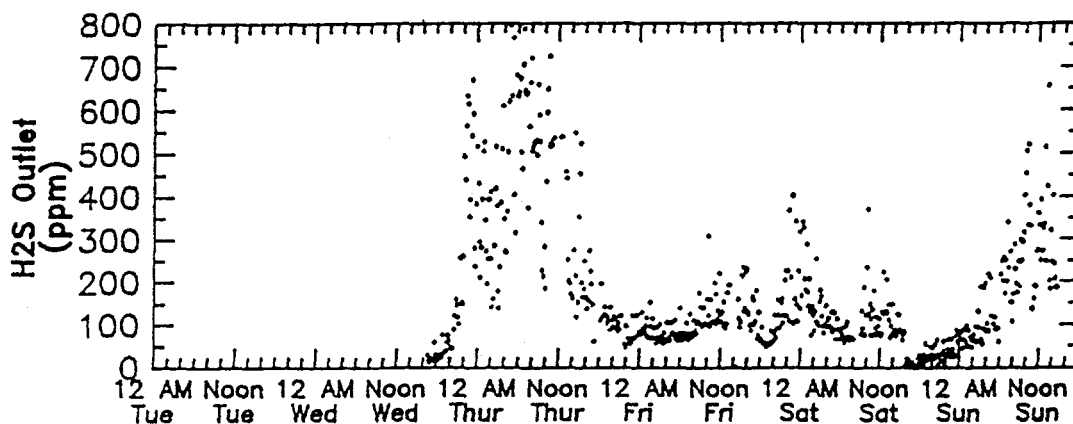
Analysis of sorbent samples collected from the absorber and regenerator outlets during Test 7A indicated sulfur loadings of less than 25% of theoretical sulfur loading independent of sorbent transfer rate. This decrease indicates a loss of capacity of the sorbent and is consistent with the pilot data, which indicated good sulfur removal at low sorbent loadings but a decrease in performance as sorbent sulfur loadings were increased. Bench-scale testing confirmed the loss of reactivity. At the end of Test 7A, the relative capacity was around 40% of that of the fresh material. The source of the degradation was believed to be the presence of water vapor produced by oxidation of the tars deposited on the sorbent combined with the high-temperature in the regenerator.

Analysis of sorbent samples confirmed that good regeneration was achieved. Sorbent leaving the regenerator typically contained less than 0.5% residual sulfur and virtually no carbon. Temperature control in the regenerator was improved over previous runs through upgrades

to the recycle compressor and tuning of the control parameters for first- and second-stage oxygen control. Some spikes were noted above the 1400°F target point, particularly in the first stage, as gasifier throughputs above 75% were attempted.

Effective chloride control was provided by the combination of in-duct injection of sodium bicarbonate upstream of the absorber and use of the zinc/calcium-based sorbent in the recycle loop guard bed. The zinc/calcium-based sorbent did not show any signs of clinkering or degradation over the course of the run. The guard bed material was changed once during the test. HCl levels in the recycle loop were generally under 200 ppmv, as compared to 1000–2000 ppmv without the guard bed. No plugging of the recycle loop heat exchanger was noted. However, since only 35% of the chlorides are being removed upstream of the absorber, chlorides were still being picked up by the sorbent, giving rise to concerns about long-term operability as well as plant emissions.

Performance of the HGCU valving and sorbent transfer equipment continued to be satisfactory. No problems were encountered with



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Figure 3. Absorber Outlet H₂S Concentrations - Test 7A. Inlet Conditions: P=280 psig, T=1020°F, Illinois 6 coal - 3500 ppm H₂S Inlet

transfer of Z-Sorb sorbent during the entire test. A total of 64,800 lb of sorbent was transferred, or slightly under 10 inventory turnovers, at a run-averaged attrition rate of 0.5%, based on a 12 mesh screen.

The turbine simulator was operated for 81 hours, including 50 hours at "F" class turbine conditions. Operation of the new fuel nozzle was satisfactory, with nozzle and liner temperatures within acceptable ranges. Stable combustion was achieved over a wide range of firing temperatures, although some instability was noted at part-load conditions.

Test 4A - Conditions and System Modifications

Long Duration test 4A was performed during the period of November 13 to November 18, 1994. The primary objective was to test the sub-scale RQL staged combustor on coal gas containing approximately 2000 ppmv of ammonia. The secondary objective was to obtain test data on the fresh Z-Sorb sorbent that was added to the existing charge in the ratio of 1 part new to 2 parts old. The existing material had been run in Tests 6 and 7A and was known to have lost significant capacity. Run conditions were modified to limit regenerator temperatures to 1350°F, compared with 1400°F in 7A, and operation was split between anthracite coal and Illinois 6. The low volatile anthracite was chosen to minimize tar production in order to (1) eliminate condensation in the piping at the low gas flowrates required by the RQL combustor and (2) minimize the introduction of water into the regenerator resulting from tar absorption on the sorbent. The second half of Test 4A was run with the MS6000 turbine simulator on-line using a modified version of the fuel nozzle tested in 7A. No sodium bicarbonate injection was performed during Test 4A. The chloride guard bed was operated in the recycle

loop using the zinc/calcium-based sorbent tested successfully in 7A.

Samples of fresh sorbent were placed in a slipstream on the regenerator recycle loop. The samples were located in an oven capable of operating at temperatures from 1300°F to 1500°F to determine the effect of regeneration temperature on sorbent life. Prior to the start of the test, basket samples of fresh sorbent were also placed in the coal gas stream at the absorber inlet and outlet and at one location in the recycle loop. The basket samples were left in for the duration of Test 4A so that they were exposed to both anthracite and Illinois 6 fuel gas for a total of 100 hours.

A change in sorbent transfer procedure was implemented during Test 4A. Smaller increments of sorbent were moved more frequently through the absorber and regenerator to maintain the same hourly rate but provide a more continuous feed of fresh sorbent to the absorber and sulfided material to the regenerator.

Test 4A - Results

A total of 100 hours of uninterrupted HGCU operation was obtained: 52 hours on anthracite and 48 hours on Illinois 6. System throughputs were reduced to approximately 50% for anthracite during RQL testing and increased to 65–75% for Illinois 6. The H₂S removal efficiency of the HGCU for Test 4A is shown in Figure 4. Inlet H₂S concentrations averaged 1000 ppmv for anthracite and 3500 ppmv for Illinois 6. The outlet H₂S during anthracite gasification was around 20 ppmv; during steady state operation on Illinois 6, it was around 50 ppmv. The higher peaks seen on Thursday occurred when sorbent transfer rates lagged the required value for 25% sulfur loading during the transition from 1000 ppmv to 3500 ppmv coal gas. A change in sorbent

transfer procedure was also implemented at this time. Sorbent was moved in smaller increments every 15 minutes through both the absorber and regenerator; previously a single but larger batch of sorbent was transferred once per hour through the absorber.

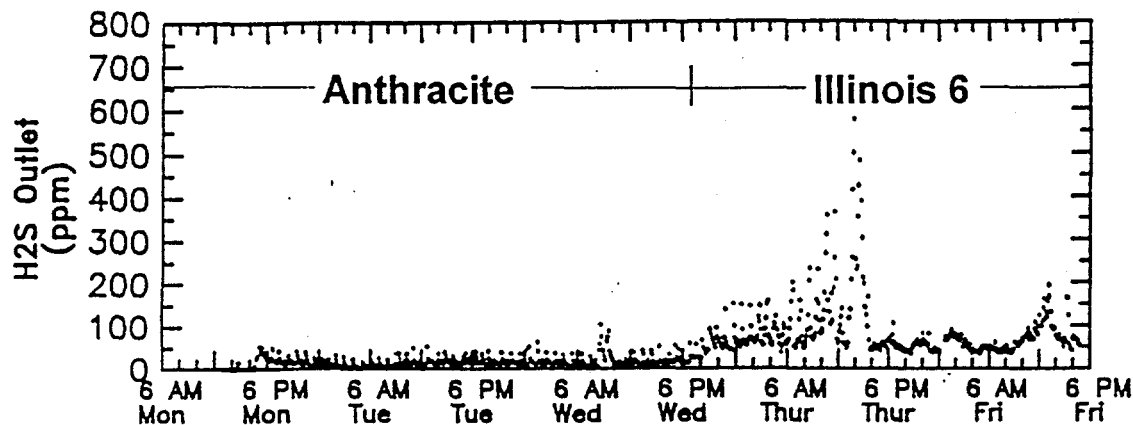
Sulfur levels in the sorbent samples taken at the absorber outlet were around 4% (22% of theoretical) for anthracite and 4.5% (25% of theoretical) for Illinois 6. Both were on target for the test conditions set. Achievement of 50% of theoretical sulfur loading on the sorbent was not an objective of this test since it was known that the sorbent sulfur capacity had deteriorated by the end of Test 7A. The sulfur levels measured in the regenerator samples averaged between 0.5 and 1.0%, slightly higher than the target of 0.5% but sufficient to meet the requirements for desulfurization at the sorbent duty selected. As a result of higher regenerator inlet temperatures, the level of regeneration was slightly better during Illinois 6 operation.

Both stages of regeneration were run under automatic temperature control. Continued tuning

of the control parameters and lowering of the overall high temperature setpoint allowed maximum bed temperatures to be maintained below the prescribed absolute limit of 1400°F under fully automatic modulation of air and recycle flows. Table 2 summarizes the temperature control results. This represents an improvement over Test 7A where occasional spikes to 1500°F were noted at the inlet to the first stage.

Thermo Gravimetric Analysis (TGA) testing of slip stream sorbent samples run at 1300, 1400, and 1500°F for 10 hours indicated no loss in reactivity on the 1300°F sample even in the presence of 2% water in the recycle loop. Although very little change was noted at 1400°F, a significant loss in reactivity was observed at 1500°F, indicating that the combination of 2% H₂O and 1400°F is the practical limit for Z-Sorb sorbent regeneration.

TGA analysis of the sorbent basket samples located at the absorber inlet and outlet indicated that after exposure to 100 hours of coal gas, the inlet samples retained a high sulfur capacity, 90% of theoretical, whereas the outlet samples had lost



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Figure 4. Absorber Outlet H₂S Concentrations - Test 4A. Inlet Conditions: P=280 psig, T=925-1020°F, Illinois 6 - 3500 ppm H₂S Inlet, Anthracite - 1000 ppm H₂S

Table 2. Regenerator Temperature Control - Test 4A

	Anthracite	Illinois 6
Absorption Temperature, °F	900-1000	900-1000
Regenerator Mean Maximum Temperature, °F	<1300	<1300
Peak Maximum Bed Temperatures, °F	1380-1390	1390
Range of Bulk Temperatures in Regenerator, °F	800-1200	800-1300

Table 3. Comparison of Sulfur Capacity on Sorbent Basket Samples Exposed to Coal Gas at Absorber Inlet and Outlet - Test 4A

	Absorber Inlet	Absorber Outlet
2 hour TGA reactivity *	91% of fresh	71% of fresh
Sulfur loading, Wt %	13.9 (77% of theoretical)	3.8 (21% of theoretical)
Carbon loading, Wt %	4.9	1.4

* After regeneration in the laboratory

almost 30% capacity. The data is summarized in Table 3. The reasons for this difference are not fully understood at present.

The actual sulfur loading on the basket sample sorbent pellets, as shown in Table 3, indicates that the loading achieved during Test 4A is dependent on the levels of H₂S to which the pellets were exposed. Seventy-seven percent of theoretical sulfur loading was achieved on the absorber inlet samples at H₂S levels of 1000–3500 ppmv, compared with only 21% of theoretical sulfur loading on the absorber outlet samples where H₂S levels were much lower, averaging 50 ppmv. The exposure time of 100 hours was the same for both locations.

Conclusions from analysis of absorber and regenerator samples collected throughout the run indicated that little if any further degradation occurred in Z-Sorb sorbent reactivity beyond that measured at the end of Test 7A. At the conclusion of Test 4A, fresh sorbent that had been added at the start of the test retained about 70% of theoretical reactivity as measured in a TGA analysis, as compared to only 30–40% of fresh reactivity for samples taken at the end of Test 7A. There were a number of differences in operating conditions for these two tests: In Test 4A peak regenerator temperatures were lower, there was more operational time on the low volatile, low tar anthracite fuel, and no sodium bicarbonate injection was used for chloride control.

Tests 7B-I and 7B-II - Conditions and System Modifications

Long Duration tests 7B-I and 7B-II were performed in March 1995. Test 7B was scheduled as a single 100-hour test, but hardware problems prematurely halted operations after 70 hours. The test was continued as 7B-II during the last week of March. A major focus in Test 7B was the installation and operation of an enhanced circulating fluidized bed (CFB) chloride removal system. The CFB is designed to replace the in-duct injection of sodium bicarbonate with a system capable of high levels of removal efficiency upstream of the absorber. Chloride removal efficiency of the once-through sodium bicarbonate injection averaged about 35%. Modeling work supported by bench-scale experiments in the University of North Dakota Energy and Environmental Center's pressurized drop tube furnace demonstrated that removal rates of over 90% were possible using longer gas/solid residence times, finer powder, and increased stoichiometry. The CFB design incorporates these features in a single vessel.

The CFB, shown schematically in Figure 5, is located downstream of the primary cyclone. It consists of a recycle cyclone located in a pressure vessel sized to provide about a 10-second residence time for the coal gas. Powdered sodium bicarbonate is injected upstream with the LBTu coal gas and enters at the bottom of the CFB vessel. The sodium bicarbonate rapidly calcines to sodium carbonate in the 1000°F gas stream, forming highly porous particles that react with hydrogen chloride in the coal gas to form sodium chloride. Larger particles settle out at the lower CFB bed velocities to form a fluidized bed at the gas inlet while the smaller particles remain entrained with the gas for removal in the internal cyclone. The particulate is recycled to the bed via

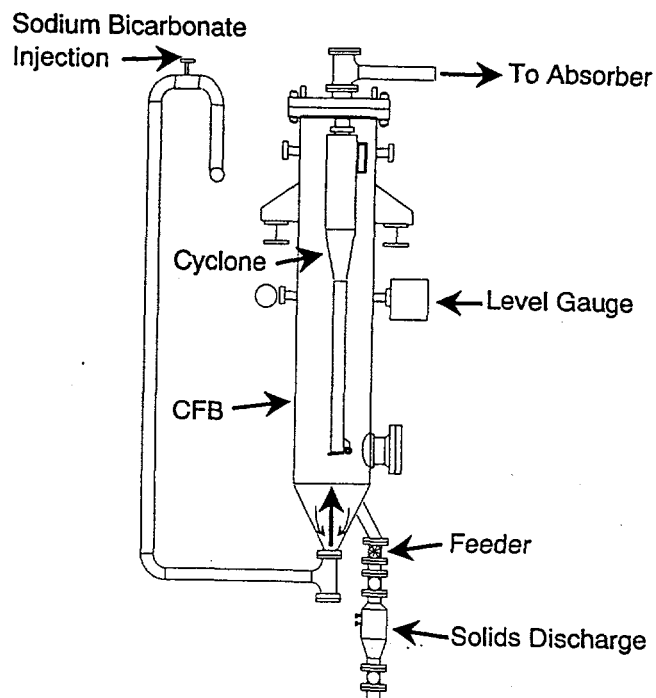


Figure 5. Circulating Fluidized Bed Chloride Removal System

a dipleg and foot valve below the cyclone for increased utilization of sorbent. Clean gas exits the top. Spent sorbent is removed through a solids off-take pipe and lock-hopper. A nuclear level gauge is provided on the internal dipleg to monitor performance.

A second major focus in Test 7B was the evaluation of system performance on a fresh charge of Z-Sorb sorbent. The material was similar in composition to that tested in Tests 6, 7A, and 4A, but with a slightly smaller, 3-mm average pellet size. Prior to the start of fired operation, sorbent was circulated through the system under dry nitrogen to measure background mechanical attrition. Less than 0.2% of <12 mesh material was obtained. The off-line sorbent attrition test also provided the opportunity to test and debug the automatic sorbent transfer software that was implemented for the first time in Test 7A.

Illinois 6 coal (1.8% sulfur) was used as the test coal, producing 3500–4000 ppmv H₂S in the coal gas. The recycle loop guard bed utilized the zinc/calcium-based sorbent used successfully in Tests 4A and 7A.

Modifications to the turbine simulator included a modified version of the Tampa Electric Company fuel nozzle used in the second half of Test 4A.

Test 7B - Results

The HGCU was operated for approximately 70 hours in 7B before being shut down due to failure of the sorbent transfer elevator. Preliminary operation of the CFB during this time indicated elevated levels of chloride capture (75%) as bed inventory was increased. Problems were encountered with blockage of the solids discharge pipe that prevented attainment of steady state operation. The suspected cause of blockage was condensation of steam and tars from the coal gas in combination with the sodium carbonate powder. Chloride concentrations in the recycle loop were significantly lower than seen in the previous run.

Excellent sulfur removal was achieved in the HGCU at sorbent transfer rates equivalent to 30% of theoretical loading. During the first hours of operation on anthracite coal at 1000 ppmv H₂S levels, the absorber outlet H₂S concentrations were less than 10 ppmv. As the inlet coal gas H₂S concentration increased to 3500 ppmv under Illinois 6, the outlet concentration remained under 50 ppmv. At 400–500 lb/hr sorbent transfer rate, the measured sulfur loading reached the target of 30% set for the first part of the test. Analysis of the regenerator samples indicated satisfactory regeneration of the sorbent, with residual sulfur levels dropping to 0.5% as steady state operation

was achieved. Temperatures in the regenerator were successfully controlled to the target of 1350°F through two-stage automatic control of oxygen and recycle flowrates.

TGA analysis of sorbent samples collected at the 30 hour and 50 hour mark, however, indicated that the sorbent had lost significant reactivity in spite of the generally good performance of the HGCU system. This condition was not reflected as a loss in sulfur removal performance because the sorbent still had sufficient reactivity to perform at the 30% level targeted for the start of Test 7B.

Modifications were made to the CFB to remove the rotary feeder on the solids discharge pipe and add additional heat tracing to prevent condensation of tars and water. A coarser sodium bicarbonate feed stock was obtained to improve sorbent flowability during startup. Operating procedures for the CFB were modified to further reduce the chance of solids blockage in the discharge pipe.

Test 7B was continued March 26–April 1 using Illinois 6 coal and Z-Sorb sorbent. A total of 124 hours of additional operation was obtained, of which 80 were fully integrated. Performance of the CFB system was significantly improved during 7B-II. No problems were encountered with discharge of spent sorbent throughout the run. The CFB was started on a coarser USP #5 sodium bicarbonate, then switched to USP #2 (nominal 75-micron) after 40 hours. Chloride removal levels were approximately 50–75% at low CFB sorbent bed concentrations and coarser feed stock but increased to over 95% as sorbent inventory was increased to design levels on USP #2 material. HCl concentration as a function of run time is shown in Figure 6. The CFB was intentionally started at low inventories because of concerns over blockage, then run for up to one

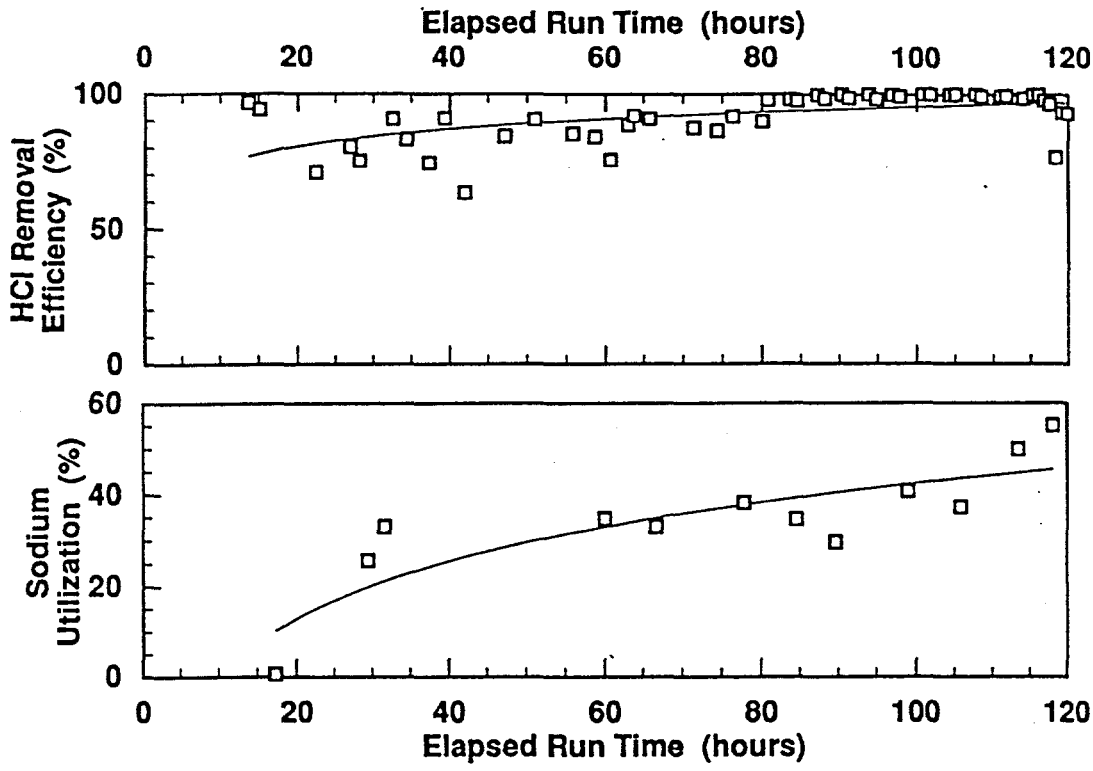
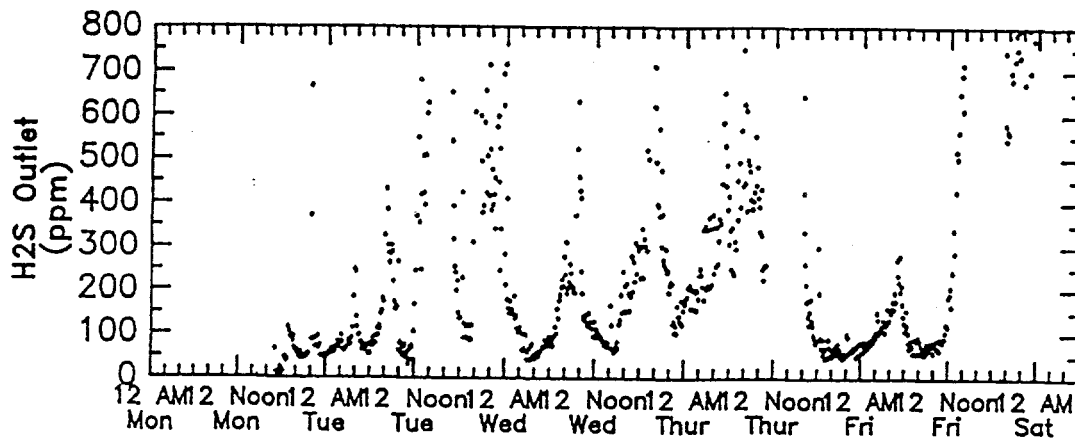


Figure 6. Circulating Fluidized Bed HCL Removal Efficiency - Test 7B

hour with the outlet lockhopper closed. Sorbent utilization approached 55% at the conclusion of the test. Chloride levels in the recycle loop were the lowest recorded to date. No fouling of the heat exchangers was observed.

Sulfur removal performance of the HGCU is shown in Figure 7. Outlet H_2S levels ranged from baseline values of 50 ppmv at sorbent transfer rates equivalent to $\leq 30\%$ of theoretical sulfur loading, to several hundred ppmv H_2S as sorbent



27 March - 1 April 1995

Figure 7. Absorber Outlet H_2S Concentrations - Test 7B. Inlet Conditions: P=280 psig, T=875-950°F, Illinois 6 coal - 3500 ppm H_2S Inlet

transfer rates were slowed, and the gasifier throughput rate was increased in an attempt to reach the target loading of 50%. Absorber operation was intentionally interrupted twice to permit off-line regeneration of the bed prior to attempting 40 and 50% sulfur loadings. The HGCU was run under automatic sorbent transfer and temperature control, with the upper limit on regenerator temperatures set to 1350°F. Temperature control in the regenerator was the best to date.

Post test analysis of sorbent samples collected during Test 7B indicated the sorbent retained approximately 25% to 30% of theoretical capacity. This level of capacity is consistent with the pilot plant data, which showed good performance at sorbent transfer rates equivalent to 30% loading but dropped off as higher sorbent loadings were attempted. The sorbent did not meet the demonstration target of 50% required for commercial operation at the pilot plant conditions.

FUTURE WORK

One additional Long Duration test, Test 8, is included in the current contract for fiscal year 1995. A series of bench-scale tests will be performed to evaluate alternate sorbent candidates, the most promising of which will be selected for operation in the pilot plant during Test 8. The three candidates currently under evaluation include a modified zinc titanate, modified Z-sorb sorbent, and a proprietary sorbent under development by DOE-METC. Hardware modifications for Test 8 will include installation of a stainless steel liner in the upper portion of the absorber to minimize wall effects, modification to the CFB solids discharge to include the outlet rotary feeder, and installation of the full-scale RQL combustor.

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