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TESTS TO PRODUCE AND RECOVER CARBON DIOXIDE
BY BURNING COAL IN OXYGEN AND
RECYCLED FLUE GAS

Black Hills Power and Light Company
Customer Service Center Boiler No. 2,
Rapid City, South Dakota

by

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FOREWORD

This report is one of several that present results obtained by an Argonne National Laboratory program entitled Systems Analysis for Waste Carbon Dioxide Utilization. The program includes engineering research -- as well as economic and institutional assessments -- dealing with issues related to the supply and use of waste carbon dioxide. Studies of supply options emphasize a new method, being investigated by Argonne, of recovering the carbon dioxide that now escapes from smokestacks; this method would avoid emission of the sulfur oxides and nitrogen oxides believed to be precursors of acid rain. Studies of use options relate primarily to increased exploitation of carbon dioxide for enhanced oil recovery.

The new method that Argonne is investigating differs from conventional approaches, which attempt to separate carbon dioxide from flue gas after the fuel has been burned in air. The flue gas produced after such a burn is composed principally of nitrogen and a smaller amount of water vapor, a small amount of "excess" oxygen, and oxides of sulfur and nitrogen, depending on the fuel, in addition to the carbon dioxide. Because sulfur oxides interfere with the separation of carbon dioxide, they (primarily sulfur dioxide) must be removed first. The higher the concentration of sulfur oxides, the more expensive the conventional methods of removing those oxides become.

The new method would separate air into nitrogen and oxygen streams before combustion. The nitrogen stream would be rejected to the atmosphere or captured and sold as a by-product. The oxygen stream would be mixed with an almost pure carbon dioxide stream, and this mixture would be used instead of air in the combustion process. The carbon dioxide would act as a diluent, just as the nitrogen in air does. The postcombustion gas stream would be composed of carbon dioxide (arising from combustion and the use of carbon dioxide as a diluent), water vapor, "excess" oxygen, and depending on the fuel, oxides of sulfur and nitrogen. This stream would be split into product and recycle streams, both composed of almost pure carbon dioxide. The recycle stream would provide the carbon dioxide that is to be mixed with the oxygen entering the combustor, as described above.

The method just described is what Argonne calls the "dry-recycle" method, in that the postcombustion gas stream would be dehydrated before being split into product and recycle streams. In an alternative "wet-recycle" method, only the product stream would be dehydrated. Both methods are being explored.

This new approach raises crucial questions for research. First, will fuel (particularly coal) burn normally in mixtures of carbon dioxide and oxygen or in mixtures of carbon dioxide, oxygen, and water? And, if it will, will normal heat transfer take place with such a burn? Second, what practical problems will be encountered when retrofitting Argonne's new method to an existing furnace being operated by its usual staff? The research reported here addresses both questions, but emphasizes the second. Other reports from this project address the first question in detail.

A 2.2×10^6 Btu/h, coal-fired, stoker-fed boiler was retrofitted for wet-recycle by the staff of Black Hills Corp., the owners and operators of the furnace. Two related

modifications -- sealing the brickwork supporting the boiler and blanketing the coal bunker with carbon dioxide -- were beyond the scope of this retrofit and test, although they would be necessary for practical operation of a stoker furnace retrofitted for recovery of carbon dioxide. Linde Division of Union Carbide provided oxygen and the associated plumbing. Argonne provided instrumentation and staff to monitor the tests.

With respect to equipment retrofit, the tests showed the importance of preventing air infiltration. With respect to combustion, the tests showed good combustion and good stability of operation when switching between air and recycling modes. With respect to staffing, the tests showed that operators who were previously unfamiliar with the recycling system could easily learn to manage it.

Copies of this report and related ones can be obtained from the National Technical Information Service, the U.S. Department of Energy, or the Argonne project manager:

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ABSTRACT

Experiments were conducted using a modified stoker-fired boiler (2.2×10^6 Btu/h) instrumented to examine the feasibility of producing and recovering carbon dioxide by burning coal in oxygen and recycled flue gas in a utility environment. The tests demonstrated that the boiler can be operated in the oxygen-blown/flue-gas-recirculation mode without any noticeable effects on coal combustion, heat delivery to the water, or the coal-feed or ash-handling systems. Pretest calculations showed that a feasible set of operating parameters for a carbon-dioxide-producing combustor system tightly sealed against air infiltration and containing no more than about 5% O₂ (dry basis) at the furnace exit would be a flue-gas recycling ratio between 0.6 and 0.7 and an oxygen feed rate of 1.17 g-moles per g-atom of carbon, yielding an exhaust gas composition (wet basis) of approximately 46.9% CO₂, 50.6% H₂O, and 2.5% O₂. This composition corresponds to a product gas containing 95% CO₂ and 5% O₂ (dry basis). However, because air leaked into the test combustor and the flue-gas handling system, the highest carbon dioxide concentration achieved in the exhaust gas was 48.5% (dry basis). Major sources of inleakage were the furnace brickwork, the gas-handling system, and the coal-feed and ash-extraction systems.

1 INTRODUCTION

Miscible or immiscible carbon dioxide flooding is an enhanced oil recovery (EOR) technique applicable to certain types of depleted oil fields. For such applications, large amounts of carbon dioxide are needed at reasonably low cost. Commercial quantities of this gas are obtained either from natural deposits or from combustion of carbonaceous fuels. Except in locations where oil fields suitable for carbon dioxide flooding and natural deposits of carbon dioxide are in close proximity, combustion of fossil fuels is the only process that can produce the quantities of carbon dioxide required for EOR.

However, conventional combustion in air yields a product gas containing only about 15% carbon dioxide, the rest being primarily nitrogen, small amounts of sulfur oxides and nitrogen oxides, and excess oxygen. For EOR the carbon dioxide would have to be separated from this rather dilute gas mixture, perhaps by means of a high-strength inhibited amine absorber/stripper system.¹

Under a project entitled Systems Analysis for Waste Carbon Dioxide Utilization, Argonne National Laboratory is investigating a new approach for recovering carbon dioxide from fossil-fuel combustion.²⁻²⁶ The fuel is burned in oxygen rather than in air to eliminate the diluent nitrogen from the product gas. Combustion in only stoichiometric pure oxygen, however, results in excessively high furnace temperatures. Therefore, a fraction of the exhaust gas is recycled to the furnace to moderate the flame temperature. Because all fossil fuels contain substantial amounts of hydrogen, the exhaust gas also contains water vapor. The part to be recycled to the furnace can be "dried" first by condensing the water vapor (dry recycling) or returned without drying (wet recycling). The preferability of dry or wet recycling will vary, depending on the specific application.

The technical feasibility of burning coal in a mixture of oxygen and carbon dioxide was investigated experimentally in a laboratory combustor by Weller and associates.² Tests were conducted to provide data for comparing coal combustion in air with coal combustion in mixtures having CO_2/O_2 ratios of 3.65 (simulating the N_2/O_2 ratio in air), 2.42 (giving the same calculated adiabatic flame temperature as that obtained with air), and 2.23 (typifying an even lower rate of carbon dioxide recycle). The resulting heat-transfer data agreed well with computer simulations³ using a one-dimensional model. Both the simulations and the experimental data demonstrated that, with a CO_2/O_2 ratio of between 2.23 and 2.42, the heat transfer from the flame and the hot combustion gases is very similar to what is experienced when coal is combusted in air. Tests of carbon dioxide production in a 10×10^6 Btu/h research combustor fired with pulverized coal are also being conducted, and computer simulation of a 50-MWe utility boiler has been completed.^{27,28}

The purpose of the present study was to further the development of this new method for producing carbon dioxide in three ways: (1) to conduct combustion tests using a significantly larger combustor (2.2×10^6 Btu/h) than the one used in the earlier laboratory tests (0.4×10^6 Btu/h); (2) to use actual combustion exhaust gases for recycling rather than mixtures of carbon dioxide and oxygen to simulate flue gas; and (3) to perform the tests in an electric utility environment, primarily using the utility's own staff of engineers, operators, and maintenance personnel. Because the last of these items presented the greatest uncertainty, these tests emphasized obtaining and evaluating data from the utility's point of view.

In addition to the U.S. Department of Energy, which sponsored the project, three organizations participated in the design and execution of these tests. The test facility and utility staff were provided by Wyodak Resources Development Corporation, a subsidiary of Black Hills Corporation of Rapid City, South Dakota. The liquid oxygen and the oxygen handling and delivery system were provided by Linde Division of Union Carbide Corporation, Danbury, Connecticut. Argonne National Laboratory coordinated the experimental activity and provided most of the analytical instrumentation, analyses, and data acquisition and processing.

2 TEST FACILITY

The combustor used for the carbon dioxide production tests is one of the twin space-heating boilers at the Customer Service Center of the Black Hills Power and Light Company (a subsidiary of Black Hills Corporation) in Rapid City, South Dakota. The boiler provides 15-psi, 180-230°F hot water to the building's space-heating system. The unit is a Keewanee double-pass fire-tube boiler with a Canton (Detroit Stokers) underfed stoker. It burns low-sulfur Wyodak subbituminous "oiled pea coal" (higher heating value is ~8000 Btu/lb) at a design rating of 2.2×10^6 Btu/h. The boiler is located adjacent to the utility's Ben French Power Station. The plant operators and technicians from that station participated in the conduct of the tests.

Figure 1 is a schematic diagram of the gas flows in the combustor prior to modifications for these tests. A forced-draft (FD) fan supplies room air to the windbox, where the air mixes with the coal before coming up through the grate. An induced-draft (ID) fan maintains a slightly negative pressure in the combustion and fire-tube zones. An overfire-air fan is also available to provide secondary air, but it is normally not used. The combustion gases flow through the fire tubes and a cyclone, past an opacity monitor, and through the ID fan, before being exhausted to the atmosphere through a relatively short stack. Automatic damper controls at the intake of the FD fan and the exit from the boiler (upstream of the cyclone and the ID fan) work in tandem so that the boiler operates with a balanced draft.

Coal is fed to the boiler by a horizontal, variable-speed auger. The ash is periodically dumped from the grate into an ash pit below, from which it is removed by an inclined-screw conveyor system that transports the ash to a dumpster located outside the boiler room.

The boiler is normally controlled automatically, cycling off and on, depending on the temperature of the hot water supply. Operators are needed to rake the coal bed on the grate and to clear the grate of ash and clinkers, but only infrequently.

The boiler, as modified for these tests, is shown schematically in Fig. 2, which also indicates the locations of the thermocouples (T), pressure gauges (P), and gas-sampling ports (SP-X) used for the tests. Figure 3 shows the main modification to the system, which was the installation of a recycling line for recirculating flue gas to the combustor. It also shows the gas sparger in the recycling line (input O₂), which allows injection of oxygen ahead of the FD fan. The recycling line had two slide gates that permitted operating the boiler in either the normal air-blown mode or the oxygen/flue-gas-blown mode. One gate near the bypass takeoff from the main exhaust line permitted flue-gas recycling when open and no flue-gas recycling when closed. The other slide gate was on a branch just ahead of the FD fan; when open to the room, it permitted air intake for normal firing of the boiler. In the flue-gas-recycling mode, this second gate was closed to prevent air intake at the FD fan. The flue gas was not dried before being recycled.

FIGURE 1 Schematic Diagram of Service Center Boiler No. 2 before Modification for Carbon Dioxide Production Tests

FIGURE 2 Schematic Diagram of Modified Service Center Boiler No. 2, Showing Locations of Gas-Sampling Points and Temperature and Flow Instrumentation

FIGURE 3 Modifications to Test Combustor

A butterfly damper was installed in the exhaust duct beyond the bypass connection. Changing the setting of this damper permitted varying the percentage of recycled flue gas.

Before the first series of tests, some attempts were made to seal the furnace against inleakage of air. However, air inleakage was still significant and limited the carbon dioxide levels attained during the tests. Leaks ranged from the obvious, such as the ventilated furnace doors, to the obscure, such as the ventilation ports on the opacity meters. After the first series of tests, further attempts were made to stop all leaks.

The boiler setting was sealed; the gaps between the fan shafts and housings were closed; a slide valve was installed in the ash dump system; and the furnace doors were gasketed and sealed. Two major sources of air inleakage still could not be properly addressed: one was the air brought in with the coal through the stoker, and the other was the air flowing through the furnace brickwork on which the boiler rested. No attempt was made to blanket the coal storage bin with carbon dioxide. An attempt was made to seal some of the cracks in the brickwork with a high-temperature cement. Only limited success was achieved; after one thermal cycle, the cracks reopened.

During the first series of tests, it was determined that additional heat-rejection capability would be needed to run the furnace in a steady state mode; it normally runs in an on/off mode. Before the second series of tests, a spray-cooled, forced-air, finned-tube heat exchanger was installed on the hot-water system.

3 OXYGEN SUPPLY AND CONTROL SYSTEM

The oxygen supply system shown in Fig. 4 was provided by Linde Division of Union Carbide Corporation. It was designed to provide a maximum of about 75 ft³/min, which is equivalent to a combustion rate of 2.2×10^6 Btu/h. The equipment consisted of a liquid-oxygen storage tank with its integral safety, pressure-relief, and flow-control devices, and a finned-tube evaporator. The oxygen control system, shown schematically in Fig. 5, consisted of an on/off ball valve, an in-line filter/dryer, a pressure regulator, a globe valve, a pressure-relief valve, a gas-flow control module, and an oxygen sparger. A branch line was provided just ahead of the filter dryer for flushing the system with nitrogen. The flow-control module contained an orifice meter, total and differential pressure gauges, a flow-regulating valve, a solenoid valve, and a check valve. The oxygen sparger was made from a horizontal length of 1-in.-o.d. closed-end tube with 1/8-in. holes drilled in the horizontal plane through its axis at 1-in. intervals along its length.

The oxygen supply system was made entirely of copper and brass pipe and fittings; it was assembled with flanged or silver-soldered joints. Before first use, all new plumbing was cleaned with a degreasing solvent before being dried and flushed with nitrogen. Subsequently, before and after each use of oxygen, the lines were flushed with nitrogen to minimize the volume of piping filled with oxygen between tests.

The solenoid valve in the flow-control module was of the normally closed type and had to be energized to open. During the tests, this valve could be deenergized (closed) either by an alarm condition for a temperature, flow, or gas-analysis signal, or by a manual switch near the data-acquisition system. Further details concerning the alarm system are provided in Sec. 5.

FIGURE 4 Oxygen Supply Tank and Evaporator (from left: G. Teats, ANL; R. Kumar, ANL; A. Wolsky, ANL; D. Sigdestad, Black Hills Corp.; R. Tupper, Black Hills Corp.; and T. Fuller, Black Hills Power and Light Co.)

FIGURE 5 Schematic Diagram of Oxygen Control System for Carbon Dioxide Production Tests

4 INSTRUMENTATION

The only instrumentation already extant on the boiler system was that available on the boiler control panel: windbox and furnace pressure gauges, an exhaust gas temperature (measured just ahead of the ID fan) gauge, and indicators for the opacity, coal-feed-rate, and fan-damper settings. There were also thermometers on the hot-water lines for indicating the temperature of the circulating water.

For the carbon dioxide production tests, instruments were added for measuring temperature, flow, and gas composition, most of which are indicated in Fig. 2. Not shown in Fig. 2 are the boiler-inlet-water thermocouple and the Annubar (Bailey Controls) flow transmitter installed in the water discharge line from the circulation pumps.

Figure 6 shows the instrumentation and gas-conditioning equipment installed by Argonne for these tests. The equipment included the following:

- *Temperature.* Thermocouples were installed
 - in the water inlet line to the boiler;
 - on the furnace grate at three locations (front, middle, and back);
 - in the exhaust gas and recycling lines (one just ahead of the ID fan, a second just downstream of the exhaust line damper, and a third on the recycling line, between the oxygen sparger and the FD fan); and
 - on the recycling duct (skin temperature) (one just downstream of the oxygen sparger and another at the elbow in the duct below the sparger).
- *Pressure.* Magnahelic (F.W. Dwyer Mfg. Co.) pressure gauges were installed on the combustion gas duct just after the FD fan and on the exhaust gas duct just after the ID fan (ahead of the recycling line takeoff).
- *Gas Composition.* Combustion and exhaust gases were analyzed for carbon dioxide, carbon monoxide, oxygen, and nitrogen oxides. Carbon dioxide was measured with a Beckman Model 865 nondispersive infrared (NDIR) gas analyzer. Carbon monoxide was measured with a Beckman Model 864 NDIR analyzer. Oxygen was measured using a Beckman Model F3 paramagnetic oxygen analyzer. Nitrogen oxides were measured with a ThermoElectron Model 10 chemiluminescent analyzer. Certified standard span gases for instrument calibration were obtained from Matheson Co.

FIGURE 6 Gas Analysis Instrumentation

The combustion and exhaust gases were sampled at three locations. Sample point 1 was located just ahead of the ID fan and provided the composition of the exhaust gas before any air inleakage at the ID fan. Sample point 2 was located downstream of the exhaust line damper and provided the composition of the gases being vented from the system as well as the gases being recycled. Sample point 3 was located on the recycling line, between the oxygen sparger and the FD fan, and provided the composition of the combustion gas in the recycling mode. The solenoid valve manifold, which permitted selection of the sampling point for gas analysis, was under the control of the computerized data-acquisition system.

- *Gas Flow Rate.* Purged Pitot tube-type gas-flow sensors were installed downstream of the ID fan and in the recycling line between the oxygen sparger and the FD fan.
- *Data-Acquisition System.* A microcomputer-based data-acquisition system automatically logged the test data during the experiments.

The data-acquisition program scanned the various temperatures, gas compositions, and flow rates at selectable time intervals. In addition, the program actuated the three sample gas solenoid valves to select one of the three gas-sampling points in rotation at desired time intervals. The program could be set for alarm conditions (high or low) on each of the sensed values and could be set to trip the oxygen supply solenoid valve if a data scan produced any reading in the alarm state (see Sec. 5). When such a condition occurred, the oxygen supply was cut off, and the sensor going into the alarm condition was identified on the monitor. The data scan times, sample solenoid switching times, and various alarm set points were always specified before beginning a test run, but could be altered during the run if necessary.

- *Gas Conditioner.* The gas sample was "conditioned" before being analyzed. The sample first passed through a water-cooled condenser to remove most of the water vapor. It next passed through particle filters and a diffusion gas dryer before entering the sample line manifold from which the various instruments were supplied. The gas conditioner was equipped with visual and audible alarms to warn of a low sample gas flow rate or a low air flow rate (for the gas dryer).
- *Miscellaneous.* Other instruments used for these tests included an optical pyrometer to measure the coal-bed and flame temperatures. However, these readings were used only qualitatively because the emissivity of the coal/ash particles was unknown and probably varied from one sighting to the next. In the second series of tests, a sealed view port was used for the pyrometer readings. However, the transmissivity of the viewing window changed with time as soot accumulated on the inside. The combustion temperature readings were useful for qualitative verification of the judgments of the operator on flame brightness.

5 SAFETY

Safety considerations received careful attention, both in the design and execution of the tests and in the equipment modifications for the tests. During the planning stages, staff from the three participating organizations (Wyodak Resources, Linde, and Argonne) jointly discussed their concerns about safety. Later, similar discussions were held independently within each organization. The final equipment modifications and operating procedures reflected the outcome of all of these discussions.

The major safety concerns were (1) the possible, accidental presence of pure oxygen downstream from the sparger and (2) the possible leakage from the furnace to the room of gases having high concentrations of carbon dioxide and possibly carbon monoxide. Because the test boiler was not an open-air facility, precautions were taken so that dangerous levels of either of these gases would not build up within the combustor system or in the boiler room.

Additional concern attached to the possibility of unusually high temperature excursions and for consequent damage to the test boiler and other components of the space-heating system.

The major equipment items related to safe operation were:

- The piping system for delivering oxygen was designed by Linde and installed by Wyodak Resources according to Linde's specifications and guidelines. This approach ensured materials compatibility; proper installation procedures; appropriate "pickling" of the lines with a degreasing solvent prior to hook up; and adequate training of the Wyodak Resources personnel by the Linde personnel on proper techniques for turning on, using, and turning off the oxygen supply system.
- The following major components of the oxygen supply and control system were provided by Linde: oxygen sparger, oxygen flow-control module, and oxygen tank and delivery system. Having Linde responsible for this aspect of the study ensured that the critical items of equipment for handling oxygen had been tested and proven in pure oxygen service.
- A normally closed solenoid valve was provided in the oxygen flow-control module; it had to be energized to permit oxygen to flow to the combustor system. A manual switch near the furnace permitted the operator to shut off the oxygen flow quickly and safely. The same solenoid valve was also wired into the data-acquisition system's alarm interrupts (see below) to automatically stop the oxygen flow under unacceptable conditions. The normally closed solenoid valve ensured that, in the event of a power failure, the oxygen flow would stop automatically.

The operating precautions taken to ensure safety included:

- The data-acquisition software was designed to permit setting of "high" or "low" alarms for each of the measured parameters. Any alarm condition triggered shutdown of the oxygen feed to the combustor. The parameters that were actually set to trip the alarm during the testing were
 - too high a temperature at the grate, or at any of the thermocouple locations on the bypass line;
 - too high an oxygen concentration at any of the sampling points, in particular at sample point 3 (bypass line); and
 - too low a flow in the bypass line.

After gaining some experience with the combustor operated in the recycling mode, the high oxygen concentration alarm and the low bypass flow alarm were deliberately disabled. The former was unworkable in practice because of sample switching between the three sampling points and the instrument response time. In fact, high oxygen concentrations could possibly exist for almost eight minutes before the automatic trip would shut off the oxygen flow. The alarm for low flow in the bypass line was unworkable because the flow sensor proved to be unreliable, either as a result of port plugging or changes in Pitot tube orientation.

Later, another safety interlock was discovered to be desirable -- a common power supply to the boiler FD and ID fans and the oxygen supply solenoid valve. During one of the tests, the boiler fans were tripped by too high an exhaust gas temperature (alarm built into the boiler control system) while oxygen continued to be supplied. The oxygen supply solenoid had to be tripped manually.

- Area monitors for carbon monoxide and carbon dioxide measured the concentrations of these gases in the boiler room, particularly in the back corner of the room, which was poorly ventilated.
- The oxygen supply lines were purged with nitrogen before and after each test. For each test, the oxygen feed was always started at a low rate and gradually increased to the desired rate.
- At least two and generally three or more persons were present in the boiler room during each test -- at least one boiler operator, one or more Argonne staff members, and one or more Wyodak Resources engineers. (After the initial installation, checkout, and operator training, Linde staff members were not actively involved in the conduct of the experiments.)

6 PREDICTED RESULTS

Before conducting the tests, two types of computer calculations were carried out for the purpose of experimental design. First, anticipated operating conditions were determined in terms of combustion temperatures, gas-flow rates, and gas compositions, assuming an air-tight system. Second, the effect of inleakage of air on flue gas composition was computed.

The first type of calculations was used to predict the effects of changing furnace operating conditions on gas composition. The results of one such set of calculations are shown in Fig. 7 for a specified product gas containing 95% CO₂ and 5% O₂ (dry basis). The compositions of the combustion gas and the exhaust gas were calculated as a function of the flue-gas recycling ratio, which is the fraction of the furnace exhaust gas that is recycled to the combustor. In Fig. 7, the solid curves show the wet-basis composition of the combustion gas; the broken lines show the exhaust gas composition. The dotted curve in Fig. 7 is the calculated adiabatic flame temperature under these combustion conditions. The adiabatic flame temperature for coal combustion in 5% excess air is ~2100°C. Figure 7 shows that to obtain a similar adiabatic flame temperature in the carbon-dioxide-production mode, a flue-gas recycling ratio of between 0.6 and 0.7 is needed. The composition of the combustion gas at a recycling ratio of 0.6 would be 34.3% CO₂, 37.1% H₂O, and 28.6% O₂; at a recycling ratio of 0.7, the combustion gas would contain 38% CO₂, 41% H₂O, and 21% O₂. The exhaust gas composition is unaffected by the recycling ratio and is approximately 46.9% CO₂, 50.6% H₂O, and 2.5% O₂ (corresponding to 95% CO₂ and 5% O₂ [dry basis]).

The calculated effect of air inleakage on the purity of the product carbon dioxide gas is shown in Fig. 8. The rate of air inleakage is represented as the ratio of the air inleakage rate to the oxygen feed rate on the x-axis, and the carbon dioxide concentration (dry-basis) in the exhaust gas is represented on the y-axis. As can be seen, an inleakage rate of about 80% of the oxygen feed rate would lower the carbon dioxide concentration in the product gas from 95% to 50%. Thus, at the rated heat rate for this boiler, an air inleakage rate of only ~60 ft³/min would reduce the carbon dioxide concentration in the product gas to 50%. Figure 8 illustrates the importance of eliminating even the smallest leaks from the system.

FIGURE 7 Calculated Gas Compositions and Adiabatic Flame Temperatures as Functions of the Recycling Ratio for 95% CO_2 , 5% O_2 (dry basis) Product Gas (solid curves show the composition of the gas, including water vapor, entering the furnace; broken lines show the combustor outlet gas composition; and the dotted curve shows the adiabatic flame temperature)

FIGURE 8 Effect of Air Inleakage on Carbon Dioxide Level in Exhaust Gas (CO₂ concentration would be 95% for a zero leak rate, as shown in Fig. 7)

7 EXPERIMENTAL RESULTS AND CONCLUSIONS

During the two series of tests -- November 10-14, 1986, and February 2-10, 1987 -- more than 20 test runs were conducted during which data were logged. Of these, some were terminated after a short time for one reason or another. Most of them are described in some detail in App. A, which also presents the test results graphically.

The tests showed that the heating boiler can be operated in a flue-gas recycling mode to produce increased levels of carbon dioxide in the flue gas. Operation in this mode did not have any noticeable effects on the boiler or the space-heating system. Minimal training of the utility operators was required.

The tests also provided useful information for retrofit of existing units for production of carbon dioxide. Mechanical retrofitting is likely to be relatively straightforward; pulverized-coal boilers, especially those with a pressurized furnace, are probably the best candidates for retrofit. Operating the furnace in air-blown mode or in the carbon-dioxide-production mode, and switching from one mode to the other, is not difficult. (The significant operating parameters needing attention during the transition from one mode to the other are the combustion temperature and furnace pressure.) Gas leakage, both of air into the system and of combustion gases out of the system, requires careful attention. For system components that cannot be conveniently sealed against air infiltration, such as coal bunkers, blanketing with carbon dioxide is needed. Also, the plant's ventilation and heating systems require modification to provide adequate safeguards against buildup of carbon monoxide and carbon dioxide in the workplace. Finally, safety systems for handling pure oxygen must be integrated into overall plant process control as well as safety systems and procedures.

The highest concentration of carbon dioxide produced in any of the tests was 48.5%, which is well below the target value for using the product gas in EOR, that is, 95% or higher. The lower-than-desired carbon dioxide concentrations were caused by air leaking into the combustion system. Stoker-fired boilers like the one used in this test typically are not designed for leak-tight boiler settings; indeed, air inleakage serves to cool fixtures, such as the furnace and ash-pit doors. Also, the Customer Service Center Boiler No. 2 has a brickwork hearth design, which provides multitudinous passages for air flow into the negative-pressure furnace volume. Other points of air entry into the system include the ID and FD fan shafts and damper motor linkages, coal-feed and ash-removal augers and ducts, and furnace doors and view ports.

The carbon-dioxide-production mode did not create any operational difficulties. Observations of the fuel bed indicated that the size of the fuel bed affected combustion efficiency. High levels of carbon monoxide were present when the bed was at a higher-than-normal level, even though a substantial concentration of oxygen was observed at the same time in the flue gas. With a small bed, or when the bed burned into the stoker slot (grate), carbon monoxide levels were low and flame conditions were very good. Under these conditions, clinkering of the ash appeared to be less than what had been generally observed during normal air-fired operations. Also, the stack was visually cleaner. However, during these tests, the boiler required more operator attention during oxygen-enriched firing than during normal firing.

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APPENDIX:
DETAILED TEST DESCRIPTIONS AND RESULTS

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DETAILED TEST DESCRIPTIONS AND RESULTS

A.1 INTRODUCTION

The modified boiler was either (1) fired with air, (2) in transition from being fired with air to being fired with oxygen and recycled flue gas (or vice versa), or (3) fired with oxygen and recycled flue gas. Under air-fired conditions, the slide gate at the FD fan was open to the room, the slide gate in the recycling line was closed, and the exhaust damper was fully open. Changing to being fired by oxygen and flue gas was relatively simple because the hot coal bed would "hold the fire" during the transition. Recycling was started by opening the slide gate in the recycling line, closing the slide gate at the FD fan air intake, and partially closing the exhaust damper. Oxygen feed was started at a low level and was gradually increased to the test level. The FD and ID fan settings were then adjusted to achieve the lowest practical negative furnace pressure.

During the two series of tests, which took place over November 10-14, 1986, and February 2-10, 1987, more than 20 test runs were conducted. Some were terminated for various reasons; those that yielded useful information are described below in chronological order. The name, date, start time (in Central Standard Time, which was the clock time on the data-acquisition system), and duration of each run are given. The descriptions include any special events or actions occurring either before, during (indicated as "at $t = x$ min" after the start of the run), or after the run.

Also included are two plots of the data from each run. The first figure shows the temperature at the three gas-sampling points, the temperature at three grate locations, and the temperature of the boiler inlet water, all as functions of time. The second figure shows the composition of the gas at the three gas-sampling points, again as functions of time. Because these instruments have finite response times, the "correct" gas concentration value was not read immediately after the gas-sampling point was switched from one to the next. Thus, after every change of the sampling relay, the gas composition figure typically shows six to eight data points that gradually increase (or decrease) before reaching the correct value for each analysis.

A.2 FIRST TEST SERIES

A.2.1 BASE-1

Date: 11/11/86; Start Time: 14:36; Duration: 180 min

This was the first run in which oxygen was injected, albeit at a low rate, into the combustion system. Figure A.1 shows the temperatures recorded during the run, whereas Fig. A.2 shows the composition of the gases at the three sampling points. The recycling-line slide gate was opened at $t = 9$ min, and the oxygen was first turned on at $t = 26$ min. The oxygen was turned off at $t = 88$ min and then turned back on at $t = 105$ min.

BASE-1

FIGURE A.1 Gas, Grate, and Inlet Water Temperatures Recorded during Test Run BASE-1 on 11/11/86

BASE-1

FIGURE A.2 Gas Compositions Recorded at the Three Sampling Locations during Test Run BASE-1 on 11/11/86

The oxygen was turned off for the last time at $t = 174$ min, and the run terminated after ~180 min. Figure A.1 shows that after recycling began, the temperatures at the three gas-sampling locations were close to each other, gradually increasing from about 150°C to about 180°C . The temperature of the furnace grate also gradually increased from about $100\text{--}120^{\circ}\text{C}$ to about 140°C . In the first series of tests, the water inlet thermocouple was barely in contact with the external surface of the pipe wall. Therefore, the inlet water temperature data show a great deal of scatter but indicate a gradual warming of the water. Figure A.2 shows that the highest concentrations of carbon dioxide in the exhaust gas were well below 20%.

A.2.2 BASE-2

Date: 11/12/86; Start Time: 10:33; Duration: 115 min

This run was conducted to obtain baseline data on boiler performance characteristics under air-fired conditions. However, the boiler was operated in a steady-burn mode, rather than in the usual on/off pattern. Figure A.3 shows that exhaust gas temperatures initially rose very gradually and then dropped after about $t = 73$ min, the time at which the coal feed rate was decreased. Without recycling, the temperature at sample point 3 was the room temperature, or $\sim 0^{\circ}\text{C}$. A significant difference in grate temperature is apparent between the thermocouple at the back and the thermocouples at the middle and front of the grate. The inlet water temperature increased gradually during the run. The gas compositions for this run are shown in Fig. A.4. The carbon dioxide concentration varied between 10% and 20%, whereas the carbon monoxide concentration remained low at all times. (The high oxygen concentration values shown for sample points 1 and 2 were caused by an instrument span check.) Note that the gas in the recycling line (sample point 3) contains an appreciable amount of carbon dioxide, indicating that some flue gas was recirculating in the bypass line even though the slide gate was closed.

A.2.3 BASE-3

Date: 11/12/86; Start Time: 12:34; Duration: 42 min

This run was originally meant to be a continuation of BASE-2, but with oxygen injection. However, the coal stoker developed a problem and had to be shut down at $t = 14$ min for about 15 min. The grate (see Fig. A.5) showed a large temperature difference between the front and back, indicating that the coal bed was not burning evenly. Because of the coal-feed interruption, the inlet water temperature declined. Figure A.6 shows the gas compositions for this run. Again, the high oxygen values were due to a span check of the instruments.

BASE-2

FIGURE A.3 Gas, Grate, and Inlet Water Temperatures Recorded during Test Run BASE-2 on 11/12/86

BASE-2

FIGURE A.4 Gas Compositions Recorded at the Three Sampling Points during Test Run BASE-2 on 11/12/86

BASE-3

FIGURE A.5 Gas, Grate, and Inlet Water Temperatures Recorded during Test Run BASE-3 on 11/12/86

BASE-3

FIGURE A.6 Gas Compositions Recorded at the Three Sampling Points during Test Run BASE-3 on 11/12/86

A.2.4 BASE-4

Date: 11/12/86; Start Time: 13:43; Duration: 220 min

The first two hours or so of this run were a continuation of test runs BASE-2 and BASE-3. At $t = 108$ min, the recycling-line slide valve was opened, and oxygen was introduced at $t = 137$ min. The coal feeder broke a shear pin at $t = 147$ min and was down for the next 10 min or so. The oxygen feed rate was gradually increased to $\sim 37 \text{ ft}^3/\text{min}$, or about half the calculated full load value, by $t = 203$ min. The oxygen flow was cut off at $t = 215$ min. Figure A.7 shows the exhaust gas temperatures rising somewhat after recycling began. The grate temperatures also rose, but this time the coal was burning evenly from front to back. The inlet water temperature rose steadily during the run. Figure A.8 shows that the highest carbon dioxide concentrations were still below 25%; the sum of the three measured gases was only $\sim 33\%$, indicating a nitrogen concentration of $\sim 67\%$ in the exhaust gas.

A.2.5 BASE-5

Date: 11/13/86; Start Time: 9:24; Duration: 56 min

This run was intended to be another oxygen-blown run. However, the elbow thermocouple was not reading correctly because of a defective terminal block in the data-acquisition system. Although the run was terminated to switch the thermocouple to a different terminal block, the temperature and gas-composition data recorded during this run are given in Figs. A.9 and A.10 for completeness.

A.2.6 BASE-6

Date: 11/13/86; Start Time: 10:22; Duration: 80 min

In this run, attempts were made to minimize the air inleakage -- the cause of the low carbon dioxide concentrations in the product gas -- by operating the boiler at as high a pressure as possible. During this run, parts of the gas system were below atmospheric pressure while other parts were above atmospheric pressure. Flue-gas recycling was started at $t = 16$ min. Oxygen was injected soon thereafter, with the feed rate being increased to $\sim 67 \text{ ft}^3/\text{min}$ fairly quickly. The oxygen feed was turned off at $t = 42$ min because of almost untenable air conditions in the boiler room and excessive temperatures in the recycling line. Figure A.11 shows that gas temperatures at the three sampling points were fairly close together during recycling. However, the grate temperatures showed a large disparity, indicating nonuniform burning of coal, which was visually confirmed. As expected, the inlet water temperature rose rapidly during oxygen injection. Figure A.12 shows that some of the nitrogen was eliminated from the system by operating in this mode. The carbon dioxide concentration rose to almost 38%, which was more than 10% higher than the highest value achieved earlier.

BASE-4

FIGURE A.7 Gas, Grate, and Inlet Water Temperatures Recorded during Test Run BASE-4 on 11/12/86

BASE-4

FIGURE A.8 Gas Compositions Recorded at the Three Sampling Points during Test Run BASE-4 on 11/12/86

FIGURE A.9 Gas, Grate, and Inlet Water Temperatures Recorded during Test Run BASE-5 on 11/13/86

BASE-5

FIGURE A.10 Gas Compositions Recorded at the Three Sampling Points during Test Run BASE-5 on 11/13/86

FIGURE A.11 Gas, Grate, and Inlet Water Temperatures Recorded during Test Run BASE-6 on 11/13/86

FIGURE A.12 Gas Compositions Recorded at the Three Sampling Points during Test Run BASE-6 on 11/13/86

A.2.7 OXY-1

Date: 11/14/86; Start Time: 9:20; Duration: 60 min

The recycling mode was started at $t = 7$ min, with the switch to oxygen injection being completed by $t = 14$ min. Figure A.13 shows that the coal was burning unevenly on the grate. Figure A.14 shows that the carbon dioxide concentrations were between 20% and 30%. However, carbon monoxide built up to a fairly high concentration of ~10%.

A.2.8 OXY-2

Date: 11/14/86; Start Time: 10:22; Duration: 100 min

This run was a continuation of test run OXY-1. The oxygen was turned off at $t = 17$ min, and data were logged for approximately the next 80 min of the "cool-down" period. Figure A.15 shows that the coal bed seemed to burn more evenly as the boiler returned to its normal air-blown mode, but uneven burning was not completely eliminated. The gas compositions shown in Fig. A.16 also reverted to normal air-blown values when recycling ceased. (The high oxygen values were again caused by an instrument span check.)

A.2.9 OXY-3

Date: 11/14/86; Start Time: 12:03; Duration: 84 min

This run was similar to the previous oxygen runs. The oxygen was turned on at $t = 14$ min at a feed rate of $\sim 40 \text{ ft}^3/\text{min}$. This rate was maintained until the end of the run at $t = 83$ min. Figure A.17 shows the gradual warming of the entire boiler system as the cooling effect of fresh combustion air is eliminated because of recycling of flue gas. However, the carbon dioxide concentrations were again only ~20% as shown in Fig. A.18.

A.3 SECOND TEST SERIES

During the first series of tests, it was quickly realized that the test boiler had an unacceptably high inleakage of air, which limited the concentrations of carbon dioxide achievable in the exhaust gas. Further, the boiler could not be operated at or near its rated capacity for any length of time without overheating the water system. Overheating occurred even with maximum heat rejection into the space-heating system.

It was clear that the problem of air inleakage had to be solved before making any further attempts to achieve carbon dioxide concentrations of 95% or higher. Therefore, the major sources of air inleakage were identified. Because the carbon dioxide concentration at sample point 2 was generally observed to be lower than that at sample point 1, the ID fan was identified as one source of air inflow. An examination of other boiler components indicated that the coal-feed system, ash-handling system, and various

OXY-1

FIGURE A.13 Gas, Grate, and Inlet Water Temperatures Recorded during Test Run OXY-1 on 11/14/86

OXY-1

FIGURE A.14 Gas Compositions Recorded at the Three Sampling Points during Test Run OXY-1 on 11/14/86

OXY-2

FIGURE A.15 Gas, Grate, and Inlet Water Temperatures Recorded during Test Run OXY-2 on 11/14/86

OXY-2

FIGURE A.16 Gas Compositions Recorded at the Three Sampling Points during Test Run OXY-2 on 11/14/86

OXY-3

FIGURE A.17 Gas, Grate, and Inlet Water Temperatures Recorded during Test Run OXY-3 on 11/14/86

OXY-3

FIGURE A.18 Gas Compositions Recorded at the Three Sampling Points during Test Run OXY-3 on 11/14/86

penetrations (e.g., for the fan damper control levers), as well as the furnace iron and brickwork itself, were important sources of leakage. It was decided to run a second series of tests after additional sealing of the boiler system. The specific actions taken before the second series of tests included: (1) minimizing the clearance between the ID fan shaft and housing; (2) installing an isolation valve in the ash-disposal line; (3) gasketing and sealing all furnace access doors; (4) sealing the coal-feed auger shell; and (5) sealing furnace penetrations to the extent possible.

The problem of inadequate heat rejection was addressed by adding a forced-air finned-tube heat exchanger to the water system. A water spray was added later to increase the rate of heat removal. These modifications and improvements were made, and the second series of tests was conducted over February 2-10, 1987.

A.3.1 2-BASE-1

Date: 2/5/87; Start Time: 12:23; Duration: 175 min

In the first run of the second series, the boiler was operated in the air-fired mode only. On attempting to run at a high heat rate, the inlet water temperature rose rapidly (see Fig. A.19), reaching the alarm value of 95°C even with the increased heat rejection capability added during the boiler modifications. As Fig. A.20 shows, the gas compositions were about typical for this mode of operation.

A.3.2 2-OXY-1

Date: 2/6/87; Start Time: 9:28; Duration: 84 min

The oxygen was turned on at $t = 13$ min, and the furnace temperatures started rising. At $t = 67$ min, the oxygen supply was tripped by too high a temperature at the back thermocouple on the grate. The coal had started to burn unevenly. Figure A.21 shows the grate temperature excursions and the disparity among the back, middle, and front grate temperatures. Figure A.22 shows that carbon dioxide concentrations somewhat lower than 30% were obtained at sample point 1, whereas sample point 2 recorded even lower carbon dioxide levels than before. These results indicated that the air leak at the ID fan had not been sealed adequately.

A.3.3 2-OXY-2

Date: 2/6/87; Start Time: 12:00; Duration: 46 min

This test run was similar to the previous one. Flue-gas recycling was started at essentially $t = 0$ min. However, even before oxygen injection began, a significant but dull fire was burning in the coal bed, indicating that air was entering the system at an appreciable rate. Oxygen injection was started at $t = 23$ min. The oxygen feed solenoid was automatically tripped at $t = 37$ min by too high a temperature at the back of the grate. The grate temperatures in Fig. A.23 show a major temperature excursion for the

2-BASE-1

FIGURE A.19 Gas, Grate, and Inlet Water Temperatures Recorded during Test Run 2-BASE-1 on 2/5/87

2-BASE-1

FIGURE A.20 Gas Compositions Recorded at the Three Sampling Points during Test Run 2-BASE-1 on 2/5/87

2-OXY-1

FIGURE A.21 Gas, Grate, and Inlet Water Temperatures Recorded during Test Run 2-OXY-1 on 2/6/87

2-OXY-1

FIGURE A.22 Gas Compositions Recorded at the Three Sampling Points during Test Run 2-OXY-1 on 2/6/87

2-OXY-2

FIGURE A.23 Gas, Grate, and Inlet Water Temperatures Recorded during Test Run 2-OXY-2 on 2/6/87

back thermocouple, while the other two thermocouples showed a much smaller temperature increase at the same time. Figure A.24 shows that the highest carbon dioxide concentration reached was ~25%. Further examination of the boiler system for air leaks revealed at least two additional openings -- one on the boiler exit plenum (probably a condensate drain hole) and one at the opacity monitor (air bleed designed to keep the glass windows of the monitor clear). These openings were sealed before the next test.

A.3.4 2-OXY-3

Date: 2/6/87; Start Time: 14:46; Duration: 107 min

Oxygen was injected at a high feed rate. Injection was started at $t = 29$ min and gradually increased to $\sim 67 \text{ ft}^3/\text{min}$ by $t = 43$ min. At $t = 81$ min, the rate was increased to the maximum deliverable, or $\sim 70 \text{ ft}^3/\text{min}$. As Fig. A.25 indicates, a temperature excursion occurred at the grate at $t = 62$ min, but raking the coals to spread the fire more evenly sufficed to bring the three grate temperatures close to each other. The inlet water temperature rose steadily after oxygen injection began, but it did not climb high enough to trip the high-temperature alarm. Figure A.26 shows that the carbon dioxide concentrations in the exhaust gas were higher than those obtained earlier, reaching 34.3% at $t = 92$ min. Sample point 2 still showed a lower value (29%), indicating that the ID fan still contributed significant amounts of air to the system. The ash deposition patterns within the boiler indicated that air was leaking through a large crack in the furnace brickwork at the far right back wall, as well as along a ledge that ran down both sides of the furnace. These cracks were sealed with high-temperature cement before the next run.

A.3.5 2-OXY-4

Date: 2/9/87; Start Time: 9:05; Duration: 87 min

This run proved to be one of the two most successful of the test series. Flue-gas recycling was started at $t = 7$ min, and oxygen injection began at $t = 11$ min. The injection rate was gradually increased to the maximum of $\sim 70 \text{ ft}^3/\text{min}$ by $t = 50$ min. Figure A.27 shows that the gas temperatures rose steadily. Uneven burning was indicated by the disparate grate temperatures; however, the coals were raked at about $t = 48$ min, which resulted in the grate temperatures moving closer together. Over the preceding weekend, the water temperature had been allowed to drift down to lower than 40°C ; thus, high inlet water temperatures were not a concern. A carbon dioxide concentration of 48.2% was recorded at $t = 50$ min as shown in Fig. A.28. However, as the gas temperature rose, a thermal cutout on the ID and FD fans tripped, turning off those fans at $t = 53$ min. The oxygen feed was not wired through the fan power supply, so it continued unabated. The fans were restarted, but were tripped again. To avoid a recurrence of continued oxygen flow with tripped fans, the high-temperature alarm for the gas-sampling points was set to 240°C , a value lower than the setting of the thermal cutout on the boiler.

2-OXY--2

FIGURE A.24 Gas Compositions Recorded at the Three Sampling Points during Test Run 2-OXY-2 on 2/6/87

2-OXY-3

FIGURE A.25 Gas, Grate, and Inlet Water Temperatures Recorded during Test Run 2-OXY-3 on 2/6/87

2-OXY-3

FIGURE A.26 Gas Compositions Recorded at the Three Sampling Points during Test Run 2-OXY-3 on 2/6/87

2-OXY-4

FIGURE A.27 Gas, Grate, and Inlet Water Temperatures Recorded during Test Run 2-OXY-4 on 2/9/87

2-OXY-4

FIGURE A.28 Gas Compositions Recorded at the Three Sampling Points during Test Run 2-OXY-4 on 2/9/87

A.3.6 2-OXY-5

Date: 2/9/87; Start Time: 10:40; Duration: 80 min

This run was conducted soon after the previous one ended. However, the crack at the back right side of the furnace had opened up again, probably due to a mismatch of the thermal expansion coefficients of the brickwork and the cement. The oxygen was turned on at about $t = 5$ min and increased to the maximum rate of $\sim 70 \text{ ft}^3/\text{min}$ by $t = 29$ min. Figure A.29 shows the gas, grate, and inlet water temperatures. Because of the high oxygen flow rate and the relatively low recycling ratio, the gas temperature at sample point 3 was substantially lower than that at the other two sampling points. The three grate temperatures remained close to each other. The water inlet temperature climbed steadily, but did not reach the alarm setpoint. As shown in Fig. A.30, the highest carbon dioxide concentration recorded during this run was 42.4% at $t = 68$ min. However, there was something different about the fire during this run. A strong flame was evident even when the oxygen was turned off in the flue-gas recycling mode. Air seemed to be coming in with the coal; a "rat hole" was later discovered in the coal hoppers.

A.3.7 2-OXY-6

Date: 2/9/87; Start Time: 15:52; Duration: 62 min

Although it was finally clear that it would be impossible to achieve the target carbon dioxide concentrations of 95% or higher in this test boiler, this final run was conducted to determine qualitatively how the boiler would behave as the oxygen feed rate and the recycling ratio were changed. The coal bin "rat hole" was neutralized, and oxygen injection was started. The maximum oxygen feed rate was reached at $t = 33$ min. This time the run was terminated by tripping of the high-temperature set point at sample point 1 at $t = 56$ min. Figure A.31 shows the temperature histories during the test, whereas Fig. A.32 shows the gas compositions. The highest carbon dioxide concentration achieved in this run was 48.5% at $t = 38$ min. Other tests on the boiler during this run showed that the recycling ratio could be varied over a wide range. The coal bed and the fire in the coals behaved as expected, and the operators felt that they could control the furnace as desired.

2-OXY-5

FIGURE A.29 Gas, Grate, and Inlet Water Temperatures Recorded during Test Run 2-OXY-5 on 2/9/87

2-OXY-5

FIGURE A.30 Gas Compositions Recorded at the Three Sampling Points Recorded during Test Run 2-OXY-5 on 2/9/87

2-OXY--6

FIGURE A.31 Gas, Grate, and Inlet Water Temperatures Recorded during Test Run 2-OXY-6 on 2/9/87

2-OXY-6

FIGURE A.32 Gas Compositions Recorded at the Three Sampling Points during Test Run 2-OXY-6 on 2/9/87

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