

PIPELINE GAS FROM COAL-HYDROGENATION (IGT HYDROGASIFICATION PROCESS)

**Project 8907 Interim Report No. 2
For the Period July 1974 to June 1975**

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**UNITED STATES ENERGY RESEARCH
AND
DEVELOPMENT ADMINISTRATION**

Under Contract No. E(49-18)-1221

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FORWARD

This report covers the period from July 1974 to June 1975 and describes the work carried out under ERDA Contract No. E(49-18)-1221. This contract is a continuation of earlier OCR Contracts Nos. 14-32-0001-1221 and 14-01-0001-381, 381(1), and 381(2).

The overall objective of this project is the development of an economically attractive process for producing pipeline gas from coal to supplement natural gas supplies. The immediate objective in the design, construction, and operation of a large integrated pilot plant is to obtain scale-up data and operating experience.

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SUMMARY

During this reporting period, 10 tests were conducted in the HYGAS pilot plant, using Montana lignite as feed.

In June 1974, Test 28 signified the beginning of the program wherein steam-oxygen gasification was combined with hydrogasification to produce SNG from coal, steam, and oxygen. In this test, the operation of the newly installed external burner was found to be successful in providing heat for start-up without causing any slagging of coal dust, as had happened previously with a burner inside the gasifier. The heat was generated by combusting oxygen in excess hydrogen. During the course of the test, this external heat supply was reduced and process oxygen was introduced on two occasions into the fluidized-bed reactor to initiate steam-oxygen gasification reaction. The test was terminated due to problems in the hydrogen plant, which resulted in shutting off the external burner.

During the two tests that followed, solid flow behavior through the high-temperature reactor (HTR) and the steam-oxygen gasifier (SOG) were examined. Among the modifications made to improve solids flow and control were straightening the standpipe that transfers solids from the HTR to the SOG, extending this line to act as an overflow pipe for the HTR, and installing a new and improved valve on this transfer line to provide a full opening to the line. Also in the SOG, the bottom solids discharge section was reduced in diameter from 24 to 12 inches to improve fluidization and prevent the excessive use of steam. The distributor rings were resized to give proper pressure drops to cover start-up and design flow conditions. The bottom solids discharge valve was improved by eliminating short-circuiting around the valve.

As a result of the changes made, Test 31 showed a much-improved solids flow into and out of the steam-oxygen gasifier, indicating good fluidization throughout. An extended period of process oxygen feed to the SOG was logged. Toward the end of the test, the cyclone operation had improved, resulting in a clean quench system. The test was terminated when the upper transfer lines were plugged. Good material balance and engineering data were collected.

Test 33, the sixth test in this program, provided a continuous and extended operation of the HYGAS plant in which over 500 tons of coal were fed to the gasifier. The entire plant was on-stream, and the methanator behaved extremely well.

Test 34, made in April 1975, represented one of the most important runs in the program. During this test, a critical milestone was achieved as the entire plant was operated from coal feeding through methanation with no external hydrogen addition and no supplemental heat added to the gasifier. This was the first time a U. S. coal was processed in an integrated plant to produce substitute natural gas. On a nitrogen-free basis, the product gas after methanation was 95% CH₄ and 5% H₂ with a heating value of over 950 Btu/SCF. Substantial information was gathered from the test. Material and heat balances were obtained for the entire plant.

With Tests 34 35, and 36, important operating parameters for the system were established, to enable the HYGAS/steam-oxygen gasification program with lignite as the feedstock to be successfully concluded with Test 37 in June 1975.

Test 37 represented the longest, self-sustained operation of HYGAS/steam-oxygen gasification with coal feed rates of up to 3 tons/hr. A total of 395 hours of continuous operation was logged, during which over 790 tons of dried coal were fed. The technical feasibility of the process was demonstrated, and valuable information was obtained to contribute to the design of the demonstration and commercial HYGAS plant. Excellent material and heat balances were established (on long steady-state periods) throughout the plant. They closed to 95% or better. For the first time, it was possible to quantify the net oil made from the system due to the addition of a light-oil recovery unit. Pipeline-quality gas was produced from the methanator. The test was voluntarily terminated and post-test inspection showed the reactor to be completely clean and in excellent condition.

Other improvements and achievements made during the year include —

- a. A light-oil stripping unit was built and activated to recover oil from plant effluent water and solids. Clean oil was recovered, and this unit played an important role in defining HYGAS Process' oil production in Test 37.
- b. The hydraulic system that operates the two solids flow control valves in the reactor was modified by reducing the hydraulic operating pressure from 2500 to 1200 psi. As a result, the operation of both valves was smooth, and the hydraulic hammering that existed before was eliminated.
- c. A high-temperature, high-pressure thermowell with weephole and nitrogen purge was developed to monitor the steam-oxygen gasifier bed temperature. It has worked extremely well under the severe atmosphere used.
- d. In the coal mill area, a new water quench dust scrubber was installed in place of the old baghouse, which has been a problem area. The new scrubber behaved well and thus improved the operation of the coal mill. Other modifications were improvements made to the lubrication system in the mill, to the mill ductwork, and to the wind box. These all contributed to the reliable crushing and drying operation of the mill.

The study on ash-agglomerating gasification to improve the coal conversion efficiency of the HYGAS operation continued. Twenty-eight tests were made during this period. Coke breeze was used as the feed material, except in Tests 33 and 40 where FMC char was used. High ash agglomerates were successfully produced. Operating parameters to yield high-ash agglomerates from coke breeze were established for both the air-stream gasifying mixture and the oxygen-steam gasifying mixture. FMC char was selected to be the next feed material.

Ten support studies were carried out to improve the HYGAS plant's operation. The major ones included a) studies on various castable insulations to check their performance under high-pressure, acidic condensing conditions, b) coal-water partial oxidation to establish the feasibility of pretreating coking coal in a water slurry, and c) the behavior of coal in oil to determine if the slurry oil affects the properties of coal.

HYGAS OPERATION

Introduction

The overall objective of the HYGAS Process is to make synthetic pipeline-quality gas from coal.

This report describes the integrated steam-oxygen/HYGAS gasification test program. This phase of the program began in June 1974 with Test 28. For start-up, external heat was provided by burning hydrogen with oxygen. By Test 34, the gasifier was operated in a completely integrated fashion without the addition of hydrogen and external heat. Test 37 in June 1975 marked the successful conclusion of the lignite program. It proved the technical feasibility of the HYGAS Process, integrated with the steam-oxygen gasifier. Pipeline-quality gas was produced in this extended run. Design data were obtained from steady-state operation, including material and energy balances that closed within 95%. Such data are being used in the design of demonstration or commercial plants.

Description of Tests

Gasification Test 28, July 1974

Conducted in July, Test 28 successfully demonstrated, for the first time, the operation of the HYGAS Process integrated with the steam-oxygen gasification of hydrogasified residue char.

Test 28 began with coal feed on June 30. Steady solids flows were established preparatory to introducing oxygen into the steam-oxygen gasifier. However, when a severe electrical storm struck the Chicago area, lightning struck the incoming 12-KV power lines, resulting in a total power failure and shutdown of the plant.

After power was restored, the gasifier was heated up again and lignite feed reestablished on July 5. At 2000 hours on July 6, 1974, oxygen was introduced to the steam-oxygen gasification zone for the first time. Oxygen feed was maintained for 2 hours until poor temperature control, caused by unsteady solids feed rates, forced it to be cut off.

While reestablishing smooth solids flow throughout the reactor, a solenoid valve in the high-pressure carrier steam system failed, shutting down the steam superheater, thus interrupting the coal feed early on July 7. The start-up heater operated throughout the above events, and the reactor was maintained at operating pressure. At 1745 hours on July 7, another solenoid valve in the hydrogen plant safety system malfunctioned, causing a shutdown of the hydrogen plant and of the start-up heater.

These problems were corrected and the reactor started again at 1150 hours on July 8; lignite coal feed was begun at 0820 hours on July 9. Feed rates were stabilized, and at 0200 hours on July 10, oxygen was again introduced to the steam-oxygen gasification zone. Oxygen feed was maintained under steady-state conditions for 7 hours until 0900 hours on July 10.

Lignite coal was fed at a rate of 2 tons/hr, the oxygen feed rate was 7400 SCF/hr (40% of design), and temperatures were controlled in the 1700°F to 1800°F range. Heat input through the start-up heater was reduced to 500,000 Btu/hr (20% of the start-up heat input) by simultaneously reducing the hydrogen and oxygen rates to the heater. Solids flow and bed levels were maintained throughout the reactor. This steady-state operating period with steam-oxygen gasification is a highly significant milestone in the HYGAS program, demonstrating the first high-pressure HYGAS operation utilizing gasified char residue as the hydrogen source.

A lack of high-pressure nitrogen for balanced pressure operation and the aeration of solids diplegs forced the test's termination. A mechanical breakdown of the transport truck delivering the liquid nitrogen to our plant caused the problem.

After the reactor was cooled down and opened, the inside was inspected. The steam-oxygen gasifier stage was in excellent condition with no sign of any attack on the refractory or the metal components in the reaction stage. The solids sample from this stage was highly gasified. Both of the hydro-gasification stages of the reactor were in good shape with no accumulation of any lumps or agglomerates.

The hydraulic system for controlling the valves in the nitrogen jacket was modified to allow the use of much lower hydraulic pressure instead of the current 2500-psig level. This was intended to minimize hydraulic hammering and extend the operating life of the system components.

A number of thermocouples installed in the steam-oxygen gasifier stage were replaced after this test. The thermocouple sheaths broke, causing erratic temperature readings near the end of the test. The sheaths were probably attacked by the gas atmosphere rather than by the high temperature because, from the appearance of the residue char in the gasifier, it is certain that no slagging had taken place. The thermowells that contain the thermocouples have a small weep hole for pressure equalization. Small nitrogen purges were added to these to maintain a neutral atmosphere without affecting the temperature reading.

Table 1 shows the results of Test 28 for the steady-state period from 0600 to 0900 hours on July 10, 1974.

Annual Plant Turnaround, July 1974

The HYGAS plant was shut down from July 14 until July 28 for the operating staff's annual vacation period. During the shutdown, the wind box on the coal mill was replaced. It was buckled by high temperatures several tests back. A new water-quench dust scrubber was installed in place of the old baghouse. The bags in the baghouse were removed a long time ago because of unsatisfactory operation. A wet scrubber will be able to clean up the total recycle gas to the furnace and provide better operation. The 60-ton storage hopper was modified to receive a live bottom with a bin vibrator. This will facilitate solids discharge from the hopper, which had been a problem before, particularly in the winter.

Table 1. RESULTS OF TEST 28

Gases to Start-Up Burner, lb/hr	
O ₂	184
H ₂	130
Steam	400
Gases to the Steam-Oxygen Distributor, lb/hr	
O ₂	505
Steam	4274
Gases to the Bottom Stripping Ring, lb/hr	
Steam	486
Toluene Flow to Slurry Tank, lb/hr	6980
Coal Feed Rate, lb/hr	2700
Solids in Slurry Feed, wt %	28
Dry Product Gas After the Quench Section at 78,400 SCF/hr, mol %	
H ₂	47.5
CO ₂	28.3
C ₂ H ₆	0.6
H ₂ S	0.1
Ar/O ₂	0.3
N ₂	6.1
CH ₄	11.5
CO	5.6
Total	100.0
Molecular Weight of Dry Product Gas After the Quench Section	18.8
Excess Water From Quench Tower + Fines, lb/hr	6990
Carbon Gasification, %	
By Gas Analysis	70
By Estimated Ash Analysis	80
Temperatures in the Bed, °F	
Slurry Dryer Bed	600
Spouting Bed	1220
Lift Line	1300
High-Temperature Reactor	1510
Oxygasifier Bed	1100-1800

	Bed Height, ft	Superficial Velocity in Bed, ft/s
Lift Line		>20
High-Temperature Reactor	7	0.4
Oxygasifier	20	0.68
	<u>Actual</u>	<u>Design</u>
SCF Methane/lb Coal (Dry)	3.34	3.6
SCF Carbon Oxides/lb Coal (Dry)	9.8	13.3
lb Coal Fed/1000 CF Product Gas	34.4	37.7
lb O ₂ /lb Coal (Dry)	0.18	0.26
lb O ₂ /lb Steam	0.10	0.22

The suction piping to the recirculating coal slurry pump was enlarged to increase the net available suction head to the pump. Experiments conducted on the slurry pumping section indicated that the original design for the slurry velocity in the pipes was conservative and could be reduced. Increasing this suction piping reduced the pressure drop on the suction line and improved pump operation.

Tuck-pointing of the stack refractory in the hydrogen plant was completed. A new quench desuperheater was installed on the hydrogen plant. Both the high-pressure and low-pressure boilers were inspected and were in excellent condition.

Gasification Test 29, August 1974

Following the annual plant shutdown, the gasifier reactor system was reassembled, pressure-tested, and heated up on August 3 in preparation for Test 29. A leak in the high-pressure steam line interrupted the heat-up once. After coal feed began on August 8, heat-up was interrupted once more by a cracked Conax fitting at a thermocouple connection in the start-up heater, which caused a process-gas leak. The reactor was drained of solids and depressurized so that the Conax fitting could be repaired.

Coal feed was restarted late on August 9; however, the control valve in the transfer line from the slurry dryer to the first stage was not operable. After working on this system for the whole weekend, it was depressurized and the nitrogen jacket inspected. A leaking hydraulic cylinder piston seal caused the hydraulic fluid to leak across the seal and not stroke the valve. This was repaired, the reactor was closed once more, and heat-up was resumed. Then coal feed to the reactor was begun on August 14. After feeding around 30 tons of coal, the coal belt feeder jammed, and coal feed had to be stopped to repair it.

Oxygen was introduced to the gasifier for brief periods during Test 29. For one period, oxygen flowed steadily for about 12 hours and was interrupted because solids discharge from the second-stage gasifier into the steam-oxygen bed became sluggish. The second oxygen-feeding period lasted for about 8 hours and was interrupted by a combination of control valve sluggishness and the failure of the coal mill described below.

Because the coal mill required major repairs, the test was ended so that the control valves from the second-stage gasifier and from the steam-oxygen bed could be examined to see what was binding the valve movement. About 120 tons of coal were fed during the entire test. The gasifier performed satisfactorily, except for the valves. Temperature control in the second stage was excellent.

The cyclone at the top of the gasifier would perform properly if a small flow of gas were allowed to bleed down the dipleg. Therefore, the vent pipe was rerouted from the dust-quench pot at the base of the cyclone to allow a small flow of vent gas into the downstream quench unit. This seemed to improve cyclone operation significantly. The butterfly valve, located directly at the cyclone outlet, was also removed and replaced with a spool piece. The butterfly valve was no longer needed, and its removal eliminated a potential spot for dust to accumulate.

The new hydraulic system for controlling the valves in the nitrogen jacket operated very well. By keeping the hydraulic pressure at 1200 psig instead of 2500 psig, the system was quiet: All hydraulic hammering has been eliminated.

In coal preparation section, the new water-quench dust scrubber performed according to expectations. The live bottom on the 60-ton hopper together with the new Vibra-screw feeder provided very smooth solids feed. The collection of coal between the belt and the feeder housing — a situation which has been rectified — had jammed this belt feeder.

Some damage was discovered in the lubrication system for the Williams mill. This was repaired on a rush basis by combining parts flown in from Williams with parts salvaged from the mill. But later, the coal mill had to be shut down because its main shaft bearing failed. The shaft began to wobble, and a set of belts burned out. After finding pieces of the bearing retainer in the grease chamber and after consulting with the manufacturer, the main shaft bearing was disassembled to replace the damaged parts. This was a major undertaking because, essentially, the entire internals had to be removed to get the shaft out to examine the bearing at both ends of the shaft.

The slurry system operated well during the short feeding period and is was put on standby. The enlarged suction piping smoothed out the discharge flow and increased the discharge pressure from the circulating pump.

The gasifier reactor was opened for inspection after terminating Test 29. It was clean except for a ring of clinkers between the steam-oxygen distributor and the gasifier wall. This did not interfere with the discharge of solids from the steam-oxygen bed, but might have contributed to the sluggish motion of the valves controlling the flow from the second-stage gasifier to the steam-oxygen bed. These valves were cleaned and adjusted in preparation for the next test.

The pressure-equalizing line between the gasifier reactor and the steam-water separator on the water jacket was rerouted. This will avoid the accumulation of dust in the water jacket from the "breathing" of the reactor system.

Gasification Test 30, September 1974

The gasifier reactor was closed up for a pressure test on August 31, and heat-up in preparation for Test 30 was begun on September 1. At a 400-psig system pressure, a leak developed at the top of the reactor closure, thus necessitating depressurizing and disassembling the top head. After polishing the seating surface of the Gray closure, the system was tested again. It leaked. A replacement seal ring was installed after the excess web material on that ring was machined off. The reactor was again heated up on September 5.

While the reactor pressure was 200 psig with the hydrogen-oxygen start-up burner in operation, a sudden increase in the pressure differential between the hydrogen inlet pipe on the start-up heater and the reactor side

was noted. This pressure spike exceeded the instrument's 100-psi range and came down immediately. This was caused by a sudden blockage of the hydrogen orifices at the tip of the burner, which, in turn, raised the pressure on the hydrogen side of the burner. The expansion joint on the oxygen line, which was concentric with the hydrogen pipe, failed under the high pressure differential. (This expansion joint was designed for a 150-psi differential pressure.) When this happened, oxygen and hydrogen mixed and started to burn in the burner pipe. Although the oxygen flow was shut off, combustion had gone on for several minutes and eventually caused the failure of the hydrogen pipe. Hydrogen escaped from the rupture and caught fire. Hydrogen flow to the system was shut off and large quantities of nitrogen were introduced to extinguish the fire. The system was depressurized smoothly and secured. No one was injured, and no significant damage was done.

Because of the design of this Zink burner, the expansion joint was a matter of concern during the design phase. The instrumentation on the system was such that the difference in pressure was not allowed to exceed 25 psi without having controllers automatically cut back the hydrogen flow. Even if the orifices were partially blocked, the pressure was expected to rise gradually, so that the controllers could take action. As it turned out, the pressure surge was so rapid that the expansion joint had already failed before the controller could cut back the hydrogen flow.

The burner assembly was removed and replaced by a modified version of the spare burner assembly. A sleeve was added to the oxygen inlet pipe to carry the oxygen flow past the expansion joint section. The annular space between the burner inside and the sleeve will be purged with nitrogen. If the expansion joint should fail, hydrogen will not contact the oxygen directly, except at the tip. (The use of the sleeve to carry the oxygen all the way to the tip of the burner should eliminate a recurrence of this type of failure.) Furthermore, an automatic shutdown from the differential-pressure controller was installed to shut off the oxygen immediately if there is a pressure surge of 80 psi. A filter on the hydrogen line was also added, even though particulates were not expected to plug the burner tip.

The alternative proposed by the John Zink Company, even though it had not been tried under similar circumstances, was to use a packing gland arrangement around the 1-1/2 inch pipe that carries the oxygen. This would not be safe because it is likely that hydrogen would leak through the packing with a 1000-psi differential pressure. Once a leak develops, there is no way to shut it off. The next burner assembly will incorporate expansion joints good for a 300-psi differential pressure. Other burner design companies are trying to produce a different burner design that would not require the use of an expansion joint.

A 1/2-inch-diameter pipe was welded as a sleeve into the 1-1/2 inch diameter oxygen inlet pipe. The 1/2-inch pipe extends through the expansion joint to within 1 inch of the burner tip. Before this arrangement could be installed, experiments were conducted to make sure that the flame-front generator, designed by the John Zink Company, would operate with a 1/2-inch pipe. Zink said that this generator would not work for a pipe

smaller than 1 inch in diameter. After exhaustive tests with different pipe sizes and lengths, a flame front was generated, which had a 4 to 6-inch-long flame at the tip, with all combinations. Using this information, the burner assembly was modified and the expansion joint tested at an 85-psi differential pressure.

The mating part of the burner closure attached to the 24-inch-diameter head on the start-up burner assembly got hot, although not excessively, during the burner failure. All dimensions of the closure head were measured, but no sign of warping was visible. There was no sign of any scaling or change in color. After consulting with Grayloc and Struthers-Wells Corp., the closure assembly was successfully hydrotested at 2200 psig. Following the hydrotest, dye penetrant was used on the welds, and no signs of cracks appeared. Therefore, the head assembly was reused.

Then the reactor system was closed up and pressure-tested. The burner was successfully lit off on September 12 in preparation for the start of Test 30. But during heat-up, leaks were discovered in the quench-water exchanger. The reactor had to be depressurized and the exchangers opened. Checking through the tubes and pressure-testing revealed that three tubes leaked. These were plugged off, and the exchanger was headed up again.

The start-up burner was successfully lit off once again on September 18, and coal feed for Test 30 began late on September 20. It had continued for about 12 hours when leaks were again discovered in the quench-water heat exchangers. The makeup pumps could not handle the great water loss, and the proper water level could not be maintained in the quench system; therefore, the test was interrupted, and the system brought down in pressure. A decision to retube the two quench-water exchangers, which operate in series, resulted from an examination of the exchangers.

After the leaks were discovered, replacement tubes were located in the local area. Lasker Boiler also agreed to work around-the-clock on retubing these exchangers. The two tube bundles were pulled and sent to Lasker on September 24.

These exchangers have been used for 4 years. A preliminary examination showed pitting on the outside of the tube, which had been in contact with the Chicago Sanitary District canal water. This water is not the cleanest available. Conversion to a closed-loop cooling-water system next year will eliminate this problem. Sections of the tubes were cut and metallographically examined.

The new tube bundles were received on September 28, and the exchangers were reassembled and successfully hydrotested in 1 week. Samples of the old tubes were examined by the Institute of Gas Technology (IGT) and were also sent to C. F. Braun for examination and analysis.

The reactor system was heated up, beginning September 30. Because an extended test was planned, one of the early objectives was to establish full solids flow through the reactor system before introducing oxygen. This was to ensure adequate control of bed levels before adding oxygen.

The goal was to maintain steady flow for at least 8 hours before introducing oxygen. Coal feed was started on October 2. The reactor had operated smoothly for 8 hours, when a construction worker accidentally shut off an instrument line on the hydrogen plant, causing loss of the hydrogen feed and subsequent plant shutdown. The problem was corrected within 15 minutes, but the shutdown required depressurization and reignition of the hydrogen burner.

The reactor was restarted and repressurized, and coal feed was reintroduced on October 4. Since then, steady coal feed periods were maintained, at times up to a 3 tons/hr design rate, for almost 8 hours. However, transfer valves from the second-stage gasifier to the steam-oxygen bed and from the steam-oxygen bed to the discharge slurry line would periodically hang up and not control the solids level properly. A number of methods to improve this situation, such as decreasing the reactor system pressure from 1000 to 750 psig and to 500 psig to increase fluidizing velocity in the bed, were tried. This seemed to help for some periods, but did not solve the problem completely.

Probably the transfer valve from the second stage to the steam-oxygen bed, now buried in the steam-oxygen bed solids, has its movement hampered by the bed material. In previous operations before the steam-oxygen gasifier was added, this valve discharged solids into the freeboard space of the heat-exchange bed, which required only a shallow bed depth. Because a deeper bed was needed for steam-oxygen gasification, this valve had to be immersed in solids. This hypothesis was tested by operating the second-stage gasifier empty so that the solids from the first-stage gasifier dropped directly through the transfer pipe into the steam-oxygen bed. Operating in this manner, the control valve can be left in the fully open position, and the transfer pipe from the second stage will be sealed in a bed of solids and will act as a sealed dipleg. However, this arrangement did not solve the problem completely, in that solids flow was still erratic at times.

As for the transfer valve from the steam-oxygen bed to the discharge line, formerly a mixture of hydrogen and steam was used to fluidize the lower area of the heat-exchange bed. Currently, only steam is used. Even though the steam is superheated, because of the heat loss at the bottom of the reactor, there is a possibility of incipient condensation, which may cause solids to stick. A high-pressure nitrogen line was added to blend some nitrogen with the steam and thereby reduce the dew point at the bottom of the reactor. This arrangement helped solids flow, but did not solve the problem completely.

On October 9, an unexplained momentary flow stoppage on the hydrogen line automatically shut off the oxygen flow. This necessitated restarting the start-up burner and repressurizing the reactor system. Coal feed was later resumed, and further attempts were made to define the solids flow control difficulties between the high-temperature gasifier stage and the steam oxygen bed and from the steam-oxygen bed to the exit line. Using different fluidizing velocities in the bed by operating at pressures ranging from 500 to 1000 psig and varying the amounts of steam to the steam-oxygen distributor, steady solids flow could be maintained for short periods of

time, including several periods of oxygen feed. But such steady operation could not be carried on for long periods.

These tests led to the conclusion that the transfer valves needed to be examined and modified. Therefore, the test was terminated on October 16. During Test 30, about 280 tons of lignite were fed at an overall average feed rate of 2 tons/hr. Oxygen was introduced for a total of 57 hours, but this represented several periods, the longest of which was 44 hours at a relatively low oxygen feed rate.

A number of modifications were planned for the solids transfer lines and valves. In the transfer line from the high-temperature gasifier to the steam-oxygen bed, the flapper valve was removed, the slight bend in the transfer pipe was eliminated, and the transfer pipe operated as a sealed dipleg in this steam-oxygen bed. This transfer pipe was extended by 10 feet to act as an overflow pipe to control the level in the high-temperature gasification stage. This arrangement was similar to that of the transfer pipe from the first-stage gasifier to the second stage, which has operated completely successfully.

In regard to the transfer valve from the steam-oxygen gasifier to the exit line, a false metal bottom was installed to reduce the cross section near the discharge pipe's diameter from 24 to 12 inches. This allows higher velocities to be used without using excessive amounts of steam. The bottom tee and the transition piece above that were also removed. With repeated depressurizations necessary to initiate solids flow, a short circuit was established through the refractory, perhaps through some cracks in it, which allowed the leakage of gas and solids and, therefore, lessened the control valve's ability to operate properly. A solid 1-inch-thick plate was welded to the transition piece from the metallic pipe below the valve to make it impossible for gas to pass through any cracks in the refractory.

Once these transfer valves' operation was corrected, however, the steam-oxygen bed was expected to operate on a long-term basis in the next test. Despite the start-and-stop nature of this particular run, the introduction of oxygen to the bed was smooth on each occasion. The temperature rise was gradual as expected, and good gasification rates were observed. The start-up heater, after being modified following the last test, also performed very well. This burner had to be restarted on several occasions, each time without difficulty.

The gasifier was cooled down and opened for inspection. The various stages were all clear with no signs of any clinkers or slagging. The work outlined above continued. The bottom tee and the transition piece from the reactor bottom to the tee were removed by October 23. This required ingenious rigging and working in tight quarters inside the reactor skirt. The transition piece, containing the valve, LV 340, showed precisely the predicted failure of the seal plate weld at the 3-inch pipe. The valve was taken out and cleaned, and measurements taken. In addition to welding on a 1-inch plate to block gas bypass, a new aeration tap was added to the transition piece so that the zone directly above valve LV 340 will be properly aerated. This area has been troublesome on many occasions.

The cyclone on top of the reactor operated well, but it also plugged at times. When the dipleg is plugged, the extra load of fines in the quench system has caused the loss of interface control. Once the cyclone is operating well, the quench system can be controlled; however, the overall operation of the cyclone at the top of the reactor was not satisfactory. The quench separator and the prequench tower were filled with coal dust when they were opened. Dr. F. A. Zenz was consulted on this point. He believed that there was a need to improve the venting of gases from the top of the dust slurry tank at the base of the cyclone dipleg. There now is a control valve to adjust the flow of gas. However, the vent line is long and has several horizontal runs. The control valve, which is the most likely place for plugging, will be eliminated, and the run will be shortened to minimize horizontal sections. This rearrangement should improve the cyclone operation.

The "glop" collected at the interface of the quench separator was blown down several times a day. This material was fed to a steam-stripping section. A low-pressure stripper will easily separate oil overhead and allow water and coal dust to collect in the bottom. With that equipment, oil can be recovered, and the oil balance around the plant can be closed.

The mill has been operated satisfactorily since it was repaired after the last test. The shaft assembly was removed, revealing that the main bearing failed and broke in two. There was grease in the bearing, so the failure was not caused by a lack of lubrication. The system was cleaned, checked, and reassembled. The lubrication system was simplified so that the automatic Trabon system lubricates the main bearing and the upper shaft sleeve only. All other lubrication points will be done by hand on a weekly basis. This will make it much easier to spot a lubrication failure.

The slurry system operated satisfactorily. However, one or two plungers on the Wilson-Snyder reciprocating pump finally developed some wear after a long period of service. These were changed before Test 31.

The interface control and level-control float chambers in the quench separator were removed and cleaned. A great deal of dust had accumulated there, which interfered with the free movement of the float arm. This contributed to the unstable control on the interface level during the last test. Modifications to the control loop have resulted in sharper interface control. Additional block valves were also installed to permit isolating the quench-water exchangers from the process, if necessary.

There was a strike at Chemetron Corporation, which supplies liquid nitrogen and oxygen. Fortunately the plant was not operating, because the necessary gases could not be obtained. The nitrogen supply was tight, and the oxygen supply was on 50% allocation for a few weeks. The repairs in the HYGAS plant proceeded as fast as possible with the hope that the strike would be settled shortly so that the supply of cryogenic gases could be reestablished.

The hydrocarbon and sulfur gas chromatographs were overhauled and upgraded. The new control unit for the hydrocarbon chromatograph improved its reliability, and a similar unit, together with an active filter, was designed for the sulfur chromatograph.

The results of Test 30 are shown in Figures 1 through 5. Figures 1, 2, and 3 refer to the longest steady period for which chemical analyses of solid streams were available. By forcing the ash balance and oxygen balance, the resulting carbon and hydrogen balances were quite good. This indicates that the collection of data has been much improved. Because of difficulties in discharging residue from the steam-oxygen gasifier, the lignite feed rate was kept low. Figures 4 and 5 refer to a period when the steam-oxygen gasifier temperature was higher, and the gas composition from the reactor showed the correspondingly greater methane concentration and carbon oxide yield. However, solids analysis was not available to allow a material balance to be made.

Gasification Test 31, November 1974

Following Test 30, a number of modifications were made to improve solids flow control into and out of the steam-oxygen gasifier. The transition piece at the bottom of the reactor was disassembled, and a 1-inch-thick seal plate was welded. New refractory was cast around the valve, and the valve housing was machined for a larger clearance to avoid any possibility of binding caused by thermal expansion. A new purge connection directly above the valve was machined in the transition piece.

Inside the reactor, the transfer line was modified from the second-stage gasifier to the steam-oxygen bed. A revised and improved control valve, which would be held open during the test to allow the transfer line to act as a sealed standpipe, was installed at the end of the pipe. For start-up, however, there was concern about the possibility of continued gas short-circuiting up an open line before a seal could be established. For start-up this valve could be closed to establish the initial seal. Once the seal is made, the bed should operate on an overflow control from the stage above. In modifying that valve, the location of the transfer line at the center of the steam-oxygen bed was retained, so that there is no bend in the pipe at all. The redesigned valve will enable the flapper to be completely pulled back to allow a completely open discharge from the transfer line and thus eliminate any possible obstruction by the flapper near the entrance of the pipe. These were some of the objections to the original valve design at its previous location. Moving the valve to the center made this modification possible.

After successfully pressure-testing the reactor system at 100 and 500 psig, coal feed began on November 10, starting Test 31. It became immediately apparent that solids feeding through the system was well under control — much improved over the previous experience. The feed rate was increased to about 3 tons/hr, and oxygen was introduced to the steam-oxygen gasifier on the same day. In several steps, the oxygen flow was increased to 50% of

INPUT		C	H	O	N	S	Cl	ASH	OTHER	TOTAL
Coal	% (Dry)	61.20	3.66	21.85	0.94	0.94	0.04	11.37		100
	PPH (Dry)	1534	92	547	24	24	1	285		2507
	Moist.		38	302						340
Sparger	Oxygen			141						141
	Steam		341	2727						3068
Burner	Oxygen			312						312
	Steam		111	892						1003
	Hydrogen	26	284	35	29	5				379
Fluidizing Ring	Steam		141	1127						1268
Nitrogen					776					776
Seal Flush			27	217						244
Total (PPH)		1560	1034	6300	829	29	1	285		10,038
OUTPUT										
Spent Char	% (Dry)	69.12	2.72	4.95	0.78	0.38	0.05	22.00		100
	PPH (Dry)	896	35	64	10	5	1	285		1296
Product Gas	% (Dry)	22.07	12.20	33.46	32.26					100
	PPH (Dry)	626	346	949	915	*				2836
Water Out			661	5287						5948
Total (PPH)		1522	1042	6300	925	5	1	285		10,080
Net (Out-In)		-38	8	0	96	-24	0	0		42
% Balance		97.6	100.8	100.0	111.6		100.0	100.0		100.4

Toluene	
lb In	7346
lb Out	7489

*Sulfur analysis not available in gas stream.

Figure 1. MATERIAL BALANCE SUMMARY FOR HYGAS GASIFIER,
TEST 30, 10/13/75 (0406 hr) TO 10/14/74 (0799 hr)

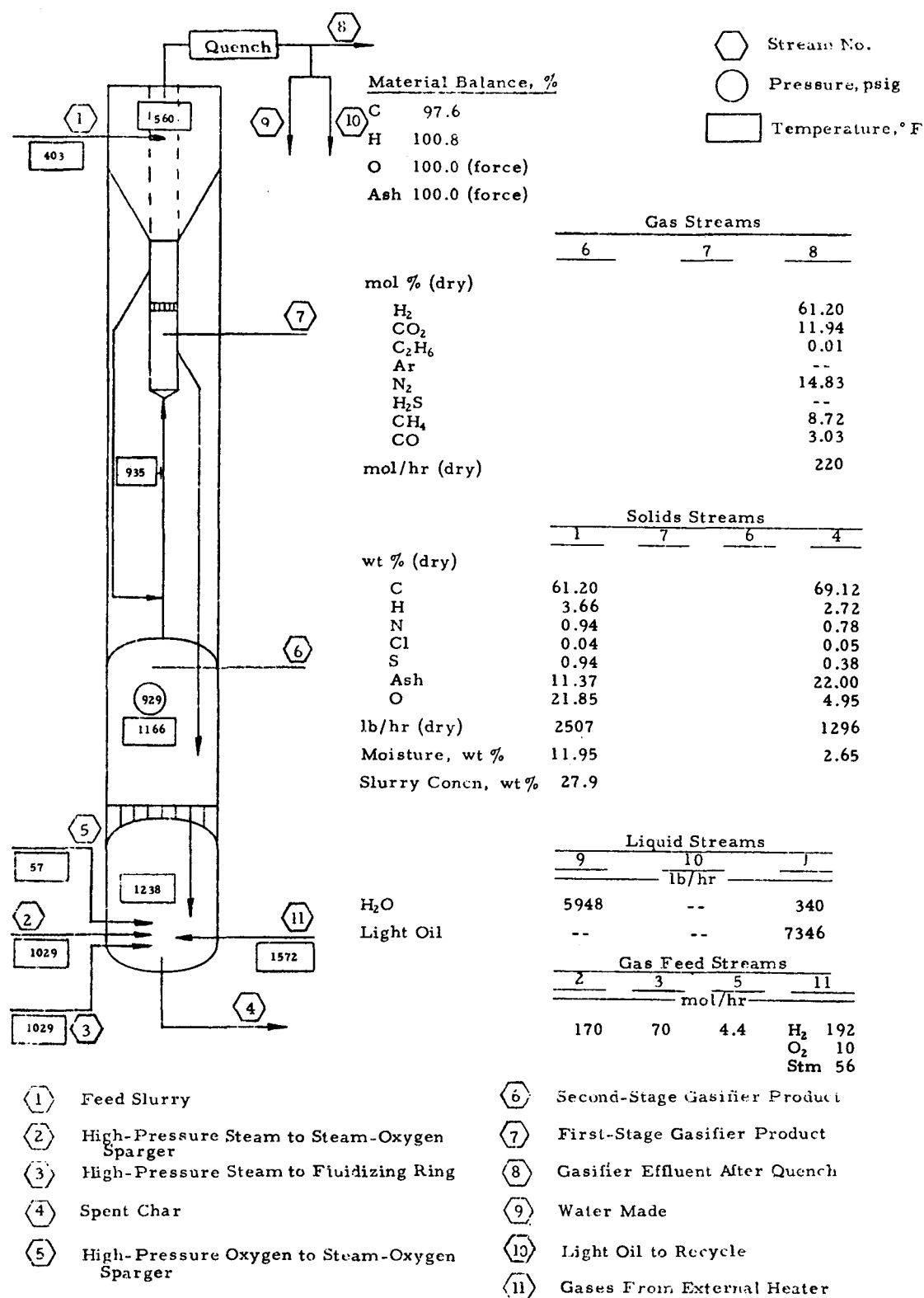


Figure 2. HYGAS REACTOR DATA FOR TEST 30 FOR THE STEADY PERIOD FROM 10/13/74 (0406 hr) TO 10/14/74 (0799 hr)

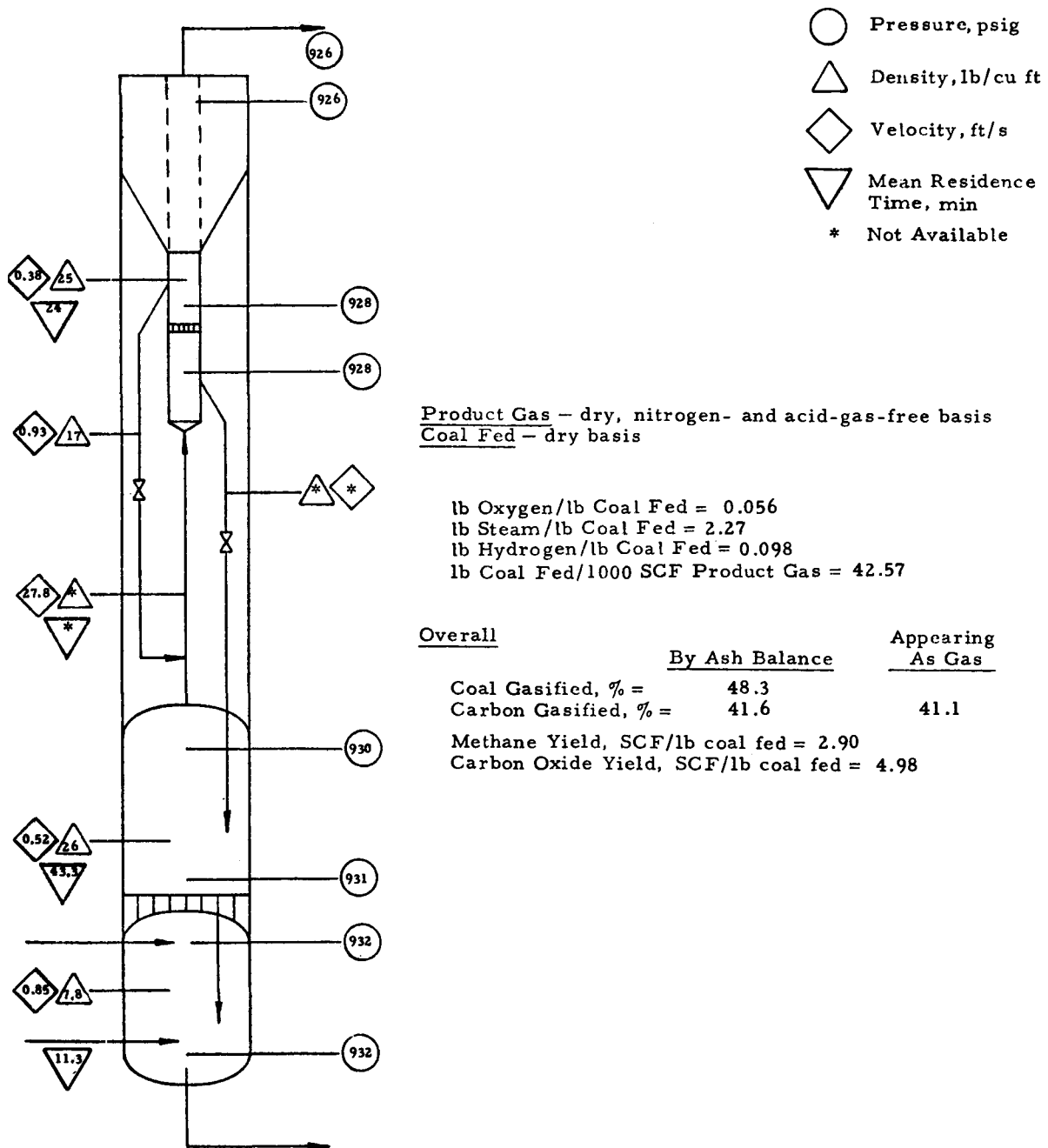


Figure 3. HYGAS REACTOR ENGINEERING DATA FOR TEST 30 FOR THE STEADY PERIOD FROM 10/13/74 (0406 hr) TO 10/14/74 (0799 hr)

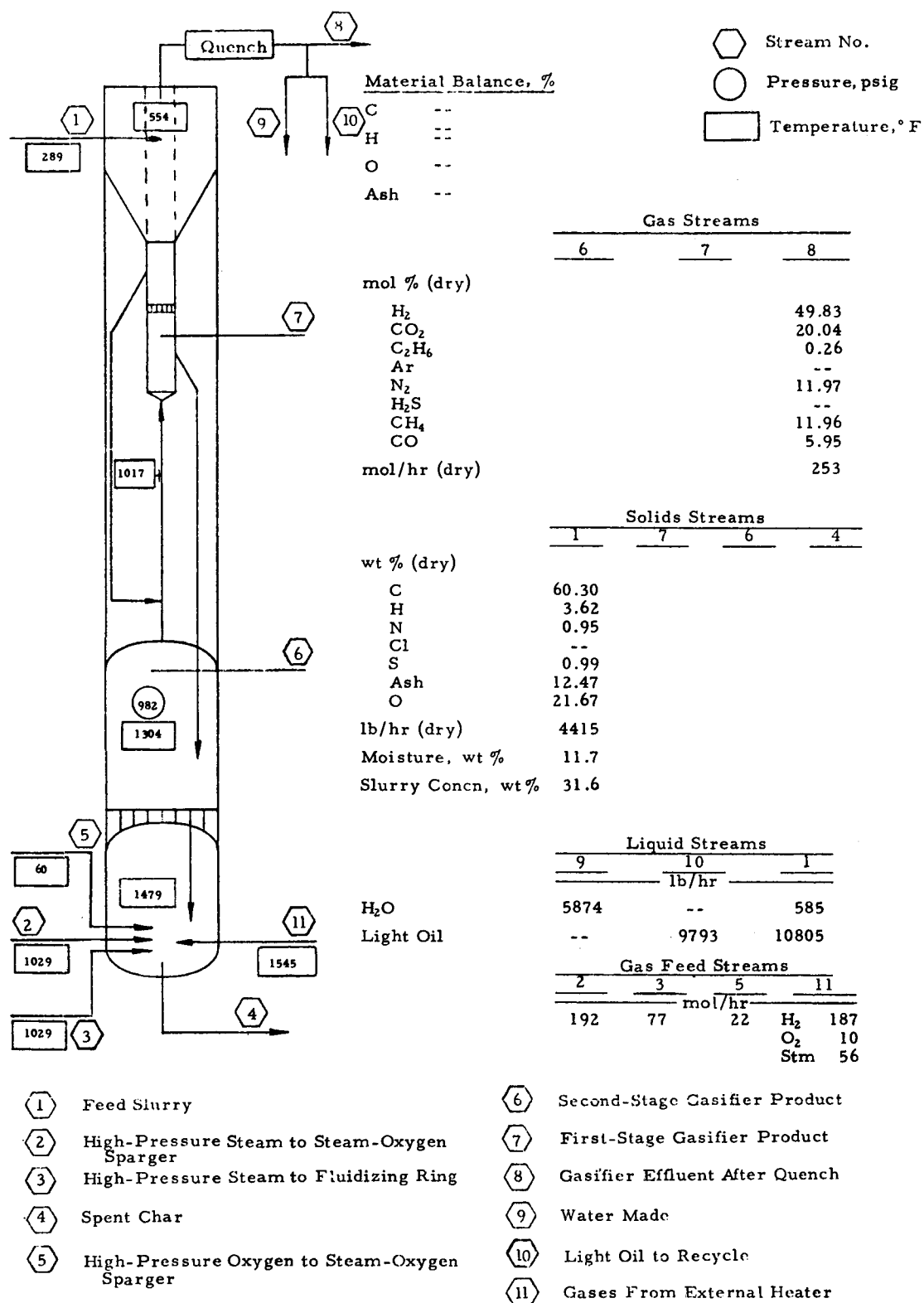


Figure 4. HYGAS REACTOR DATA FOR TEST 30 FOR THE STEADY PERIOD FROM 10/13/74 (1401 hr) TO 10/13/74 (1697 hr)

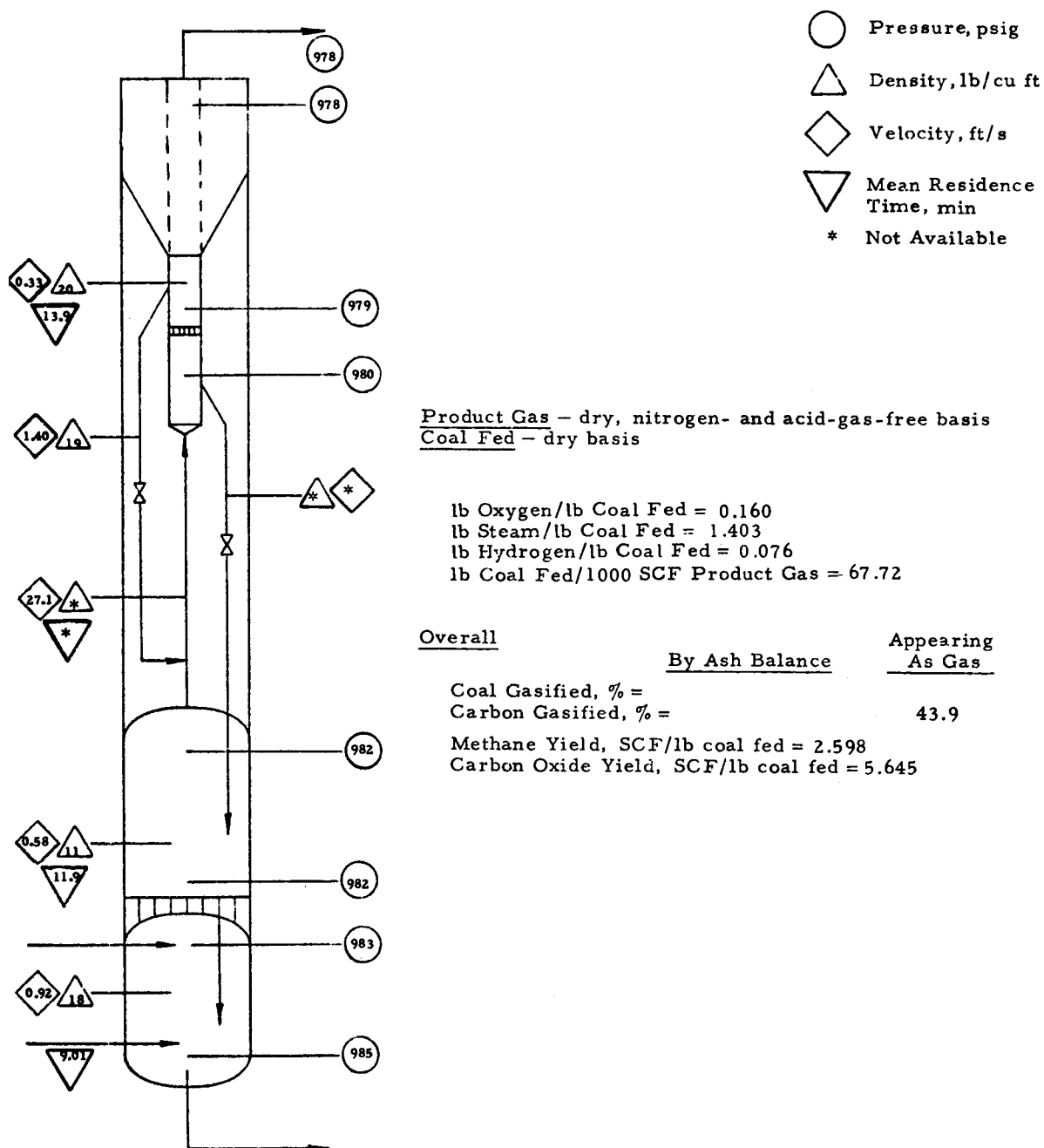


Figure 5. HYGAS REACTOR ENGINEERING DATA FOR TEST 30 FOR THE STEADY PERIOD FROM 10/13/74 (1401 hr) TO 10/13/74 (1697 hr)

the full design flow, and the start-up hydrogen flow was reduced to 25% of the initial rate. After feeding in this manner for 8 hours, the failure of a pressure controller on the feedwater pump to the high-pressure boiler caused the boiler to go down on low water input. As a result, coal feed was interrupted.

Coal feed was reestablished on November 11, and oxygen was introduced. After about 8 hours of operation, a plug developed in the line between the slurry dryer and the lift-line gasifier because of too rapid a reduction of hydrogen flow in the start-up heater, thus resulting in a lack of adequate fluidization in the slurry dryer bed. This plug was cleared, solids feed was resumed on November 12, and smooth feeding continued for 48 hours. During this time the oxygen flow was increased gradually, and solids movement through the system was under good control. The discharge of solids from the slurry dryer bed to the lift-line reactor was on automatic control. The bed densities and standpipe densities were constant and within the expected range.

On November 14, a momentary hang-up of the spent char control valve caused a surge of solid to discharge into the slurry system and plug the discharge line. Since this occurred while the bed level in the steam-oxygen bed was being brought from a high bed to the design level, a larger amount of solids was discharged than would normally be expected. Coal feed was interrupted on November 15 to clear the discharge line. The gasifier system was put back on-stream after a leaking flange on the steam line to the bottom of the reactor was repaired. On November 17, coal feed was established once more and Test 31 continued. Steam-oxygen operation was maintained until November 19. The gasifier operated well with all flows under control at the desired bed levels; however, intermittent operation of the cyclone and the dust-slurry tank resulted in poor separation in the quench system. The recycle oil, mixed with water, limits the amount of slurry that can be pumped to the gasifier, which has a given capacity for slurry evaporation. As a result, the coal feed rate had to be kept at about 1 ton/hr.

Coal feed was stopped on November 20 to clear the entire quench system. It was resumed late that day, after which cyclone operation was carefully monitored. The level control of water in the dust-quench tank was adjusted to minimize the surging that had been noticed previously. This, plus periodic venting from the tank to the atmosphere, kept the cyclone in good overall operation. As a direct indication of the improved performance of the cyclone system, the quench water and oil have been exceptionally clear.

Test 31 was terminated late on November 22, when the transfer line between the first- and second-stage hydrogasifier developed a plug. This caused solids, which were carried up the lift line, to accumulate in the disengagement space above the lift line. The solids blocked off gas flow to the slurry dryer distributor grid, and as a result, the pressure increased in the lower half of the gasifier system until the rupture disks that protect the water jacket were blown to equalize the pressure. With coal dust being discharged into the nitrogen jacket, the upper equalizing line gradually became plugged. The resulting pressure buildup then stopped the hydrogen and steam flow to the reactor and terminated the test.

During the test a total of 240 tons of lignite were fed and, more significantly, the steam-oxygen gasifier operated for 127 hours. In the four tests run since the steam-oxygen gasifier was installed, there has been no difficulty in operating this new reaction stage. Flow control has been excellent. The temperature was uniform throughout the bed, and there were no indications of hot spots or clinker formation in the steam-oxygen bed. The primary purpose for conducting Tests 28 through 31 with lignite was to gain familiarity with the steam-oxygen gasifier operation, using a coal that had been used before. In these four tests, the difficulties encountered were outside the steam-oxygen gasifier. Therefore, shakedown and initial operation of the steam-oxygen gasifier have been accomplished.

It was highly encouraging that near the end of Test 31 the dust cyclone and the quench system performed very well for more than 36 hours, as evidenced by the fact that the entire quench system showed no accumulation of dust after the test.

After the test, the reactor was opened up for inspection. Because the rupture disks had blown to equalize the pressure, the nitrogen jacket had a great deal of coal dust and condensed steam. Many of the lines were cleaned out and checked, and no significant repairs had to be made. A pressure buildup, caused by an accumulation of solids, pushed the slurry dryer grid out of position. The steam-oxygen distributor in the steam-oxygen gasifier was damaged when the steam flow to that distributor was cut off for about 10 minutes by a freeze-up in the flow transmitter signal line. Steam was later added by manual control, and the run was continued for several days. Thus, even with the distributor damaged by a period of high oxygen concentration, the steam-oxygen gasifier performed well, illustrating the basic stability of that reactor.

Prior to starting Test 31, the vent line from the dust slurry tank at the base of the cyclone was modified to minimize flow restrictions in that line. During the test, the quench system operated well when the cyclone was able to remove the dust. The vent line from the cyclone became plugged on two occasions, each time causing dust to be carried over into the quench system, a situation that caused interruption of coal feed on November 19. After the quench system was cleared and the coal feed restarted, the cyclone system performed very well because of a combination of constantly monitoring the cyclone pressure drop and briefly venting to the atmosphere each time the cyclone pressure drop decreased.

Although the small pilot-plant cyclone can be kept operating satisfactorily, an intensive effort was made to examine modifications of the dust slurry tank design to allow smoother operation and to avoid plugging the vent line from that tank. The best solution appeared to be the installation of a larger tank to more thoroughly wash the vent gas. There were plans to modify a vertical separator vessel in the electrothermal gasification system, which was not being used. This modification involved substantial repiping and welding.

The coal mill has been operating steadily. Although the Sweco screen had its counterweight shaken out of position, which stopped the coal feed to the slurry tank for less than 2 hours on November 21, the coal slurry

feed to the reactor never stopped. The Sweco screen was put back in operation.

The purification section was put on-stream on November 21 and remained on-stream for more than 1 day. Signs of foaming were indicated by pressure drops across the packing in the absorber. The addition of polyglycol, which was very effective previously, was not effective this time. Other possible causes of foaming, such as the accumulation of small amounts of dust and hydrocarbon oil, were investigated. The reclaimer was set up to operate at high pressure to concentrate and regenerate the diglycolamine solution.

Following Test 31, the reactor system was opened and cleaned up; all of the lines were verified and cleared. Line 322 between the top of the lift line and the second-stage gasifier was indeed plugged by damp coal. The moisture apparently came from a water spray nozzle for temperature control in the area above line 322. This valve was activated for a short time on November 22 when the slurry mud pump stopped for a short period.

In the section below the slurry dryer grid and the top of the lift line, three "dimples" were noticed in the 30-inch-diameter reactor internal section. These were caused by the pressure differential between the reactor and the nitrogen jacket at the end of Test 31. The metal was checked with a dye-penetrant test, but no cracks were found. The dimples were all at the same elevation, equally spaced around the periphery. Because the metal appeared to be intact, to save time, this section was not replaced.

The slurry dryer grid was reinstalled and no part of the reactor system was modified. The steam-oxygen distributor ring was replaced. The oxygen-feed line to the steam-oxygen distributor had some coal dust backed into it when the reactor pressure gradually increased at the end of Test 31. This line was cut apart, brushed, chemically cleaned, and put back into service.

The reactor system was reassembled for a pressure test. During this test, a leak occurred in the section between the slurry dryer and the lift line caused by an unused pressure tap that had broken off. Because of the awkward position of this pressure tap, the top of the reactor had to be opened once more, the slurry dryer grid removed, to close off this opening from the inside of the reactor. This was done by December 11; then the reactor was closed and was successfully pressure-tested. The water jacket was also tested at a 40-psi differential pressure and was found to be intact.

Results for Test 31, completed in November 1974, are presented in Table 2 and in Figures 6 and 7. The delay in reporting the results arises from the delay in receiving the sample analyses. The period chosen to represent Test 31 was short, but was one of steady operation with a good coal feed rate and gas production.

Table 2. MATERIAL BALANCE SUMMARY FOR HYGAS GASIFIER FOR TEST 31

INPUT		C	H	O	N	S	Cl	Ash	Total
Coal	% (Dry)	62.87	4.11	18.81	0.77	0.72	0.02	12.70	100.00
	pph (Dry)	2543	166	760	31	30	1	514	4045
	Moisture		59	476					535
Sparger	Oxygen			607					607
	Steam		391	3128					3519
Burner	Oxygen			314					314
	Steam		93	743					836
	Hydrogen	27	295	35	47				404
Fluidizing Ring	Steam		142	1134					1276
Nitrogen					400				400
Seal Flush			66	528					594
Total pph		2570	1212	7725	478	30	1	514	12,530
OUTPUT									
Reactor Overhead	% (Dry)	60.22	3.26	9.76	0.84	0.69	0.07	25.16	100.00
	pph (Dry)	918	50	149	13	10	1	384	1525
Spent Char	% (Dry)	46.94	0.97	4.53	0.21	0.45	0.04	46.86	100.00
	pph (Dry)	130	3	13	1	2	0	130	279
Product Gas	pph (Dry)	1522	540	2215	446	*			4723
Water Out			668 [†]	5348 [†]					6016
Total pph		2570	1261	7725	460	12	1	514	12,543
Net (Out - In)		0	49	0	-18	-18	0	0	13
% Balance		100	104	100	96.2	*	100	100	100.1

Toluene	pph In	11,318
	pph Out	11,027

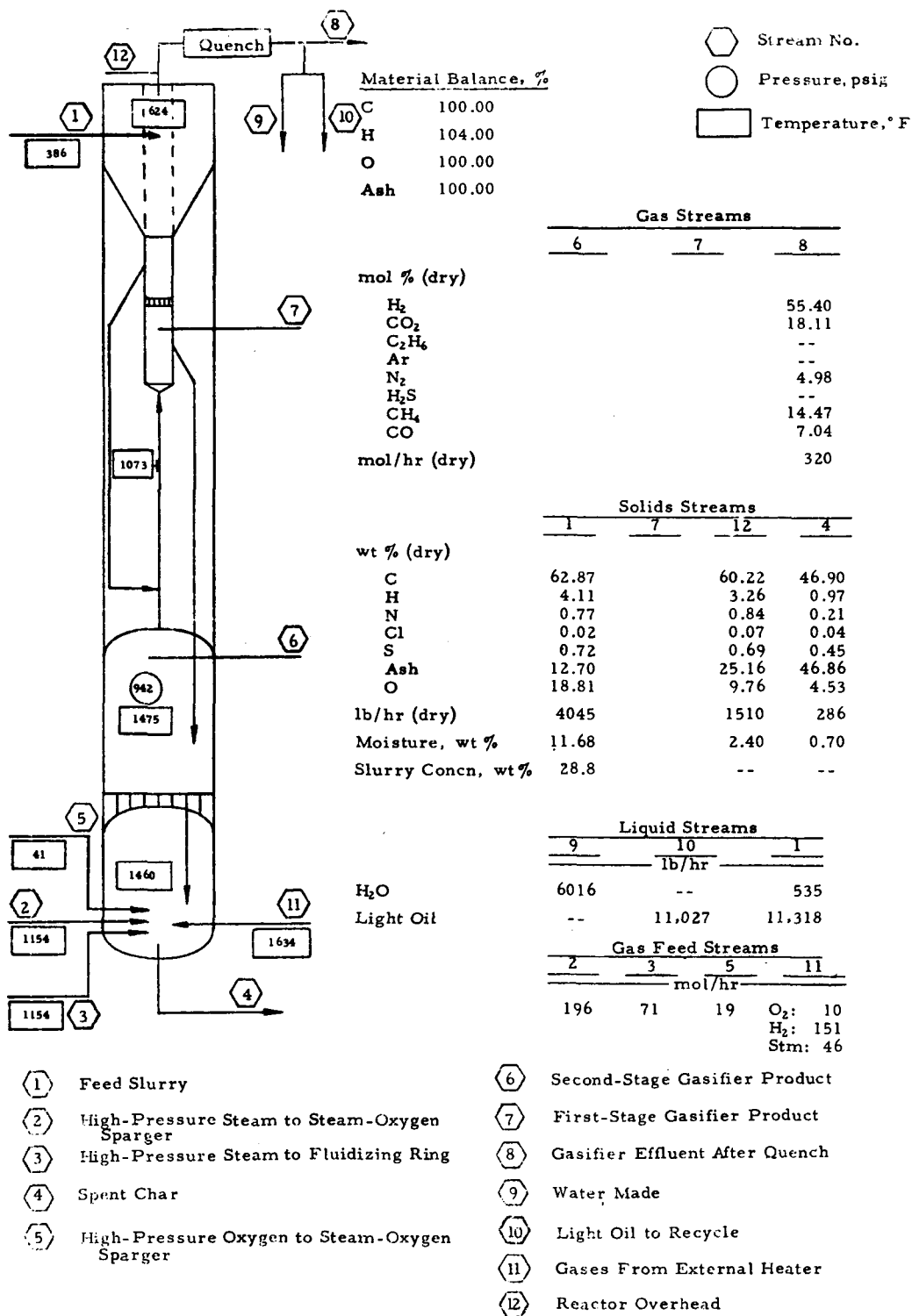
Test: 31

From: 11/21/74, 1905 hours

To: 11/21/74, 2244 hours

* Sulfur analysis not available.

† Includes estimated water for interface blowdown.



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Figure 6. HYGAS REACTOR DATA FOR TEST 31 FOR THE STEADY PERIOD FROM 11/21/74 (1905 hr) TO 11/21/74 (2244 hr)

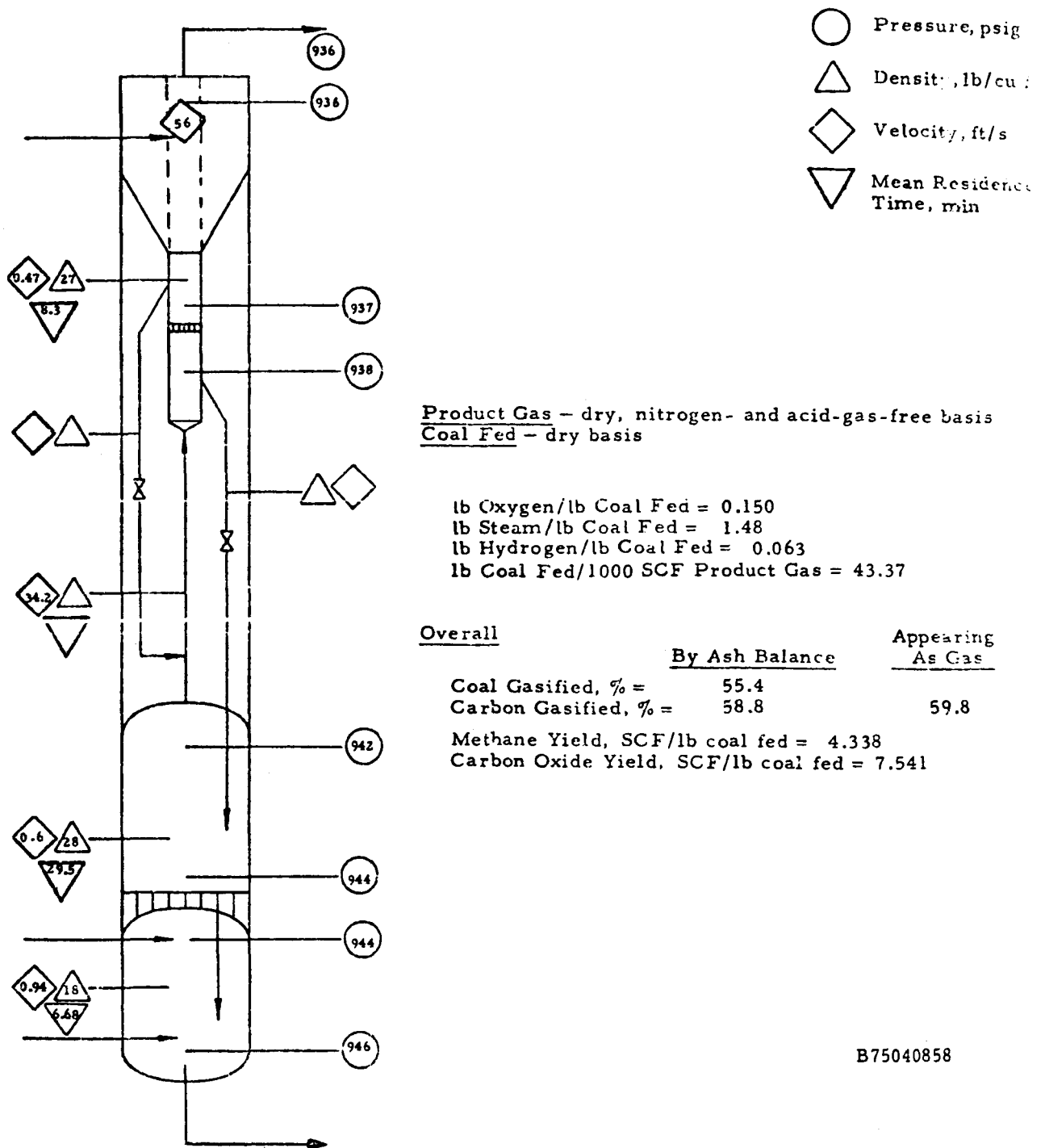


Figure 7. HYGAS REACTOR ENGINEERING DATA FOR TEST 31 FOR THE STEADY PERIOD FROM 11/21/74 (1905 hr) TO 11/21/74 (2244 hr)

Gasification Test 32, December 1974

Test 32, which began with lignite feed at 8 p.m. on December 19, was interrupted in the afternoon of December 20 by a shutdown of the high-pressure boiler. The boiler feedwater pump was not able to keep up with the combined demand of the high-pressure boiler itself and the boiler feedwater for the reactor water jacket. This situation was brought about when the circulating pump for the water jacket on the start-up heater had to be taken off-line for repair. To keep the circulation going, boiler feedwater was injected into the water jacket. This extra demand led to the lack of water supplied to the boiler.

Prior to this occurrence, solids flow had been established to all of the stages, and oxygen had been introduced to the steam-oxygen bed. After restarting the high-pressure boiler, solids flow could not be initiated through line 322, between the top of the lift line and the second-stage hydrogasifier. After working on this line for 3 days, trying various combinations of purging and blasting with high-pressure nitrogen, the test was stopped on December 23.

After cooling off and purging the reactor system, the reactor was opened up for inspection and cleaning. The 30-inch-diameter section between the top of the lift line and the slurry dryer grid had collapsed. This is the area where the three "dimples" were noticed after Test 31. The integrity of the metal at these dimpled areas was checked, and Test 32 was begun without going through the long process of replacing this section. The strength of this metal section had apparently been impaired, and together with the presence of the dimples, was enough to cause this section to collapse under moderate differential pressures.

There was no choice but to replace this section of the reactor internals. The insulation on the outside of this section was removed and plans were made to cut out the collapsed section and replace it with a new one. Fortunately, practically all of the necessary material was on hand.

The slurry system operated well. During Test 32, the slurry feed was momentarily stopped twice. Once the coal-feed Sweco screen had a few holes in it, it apparently allowed some larger particles to fall through, thus causing stoppage. Each time, however, the coal feed was put back on-stream within a short time.

The installation of the high-pressure steam connection to the diglycolamine reclaimer was completed, and the reclaimer was operated at higher temperatures to obtain a better reclaiming rate. Although the diglycolamine solution was reclaimed at higher operating temperatures, laboratory filter tests showed that activated carbon effectively eliminated the foaming tendency of the solution. The feasibility of installing a carbon cleanup filter on a side stream of the solution was considered.

The utilities operated routinely. The operating procedure was modified to minimize the demand on the high-pressure boiler feedwater pump. The high-pressure boiler package was purchased in 1967, when steam-oxygen gasification with its high steam demand was not part of the project plan.

Even though steam-oxygen gasification can be accommodated, the high-pressure boiler and its associated equipment are being called on to operate at high demand with a smaller margin of reserve capacity than is desirable.

Following Test 32, the collapsed section (Figure 8) of the reactor internals was removed. The cuts in the photo were made during the removal process. Considerable difficulty was experienced in removing the damaged section, because of its odd shape and the extremely tight quarters existing in the reactor. The replacement, prefabricated in 30-inch-diameter sections, was installed, and all of the required construction welds were completed on January 17. Internal fitting of the various transfer lines, slurry nozzle inlet, and **pressure tap connections**, as well as the slurry dryer grid and

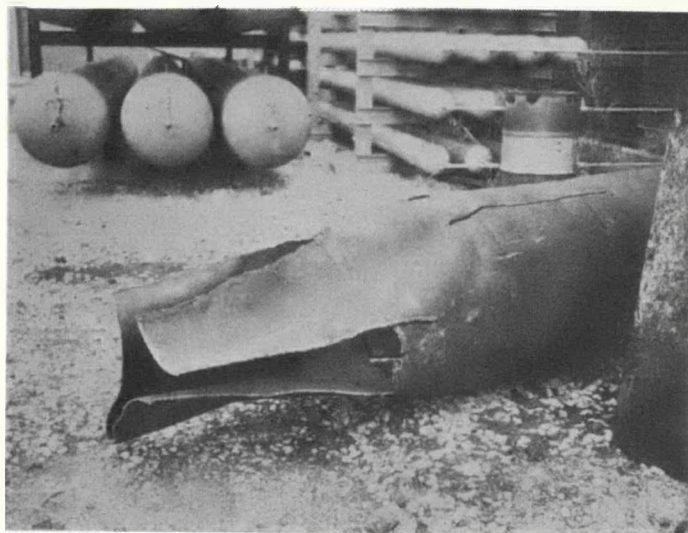


Figure 8. COLLAPSED SECTION OF REACTOR INTERNALS

its hold-down clamps, were completed by January 20. Three reinforcing rings were added around the 30-inch-diameter section for stiffening. The insulators began their work on January 20 and completed it on January 25. Then the pipefitters came back to make the connections to pressure taps and quench-water lines outside the insulation. Overall, all participants in this rebuilding project, including the subcontractors, have done an excellent job, which reduced the originally scheduled completion date of January 31 by 1 week.

Gasification Test 33, February, 1975

Test 33, which was completed in March, was highly significant, producing the longest period of stable operation since the steam-oxygen gasifier was integrated with the HYGAS plant. The gasifier operated continuously for 162 hours, with uninterrupted coal slurry feed to the top stage and steam and oxygen to the last stage. During this period, the methanation section was on-stream continuously for 62 hours. Thus, the entire plant, the HYGAS Process combined with steam-oxygen gasification, was placed on-stream to successfully demonstrate the operability of the process on a pilot plant scale.

The capacity of the gasifier was only limited by the output of the coal mill (discussed in a later section). Because of this restriction, the

coal feed rate was held to 1.5 tons/hr. Over 700 tons of lignite were fed to the plant, and over 500 tons of dry lignite were fed to the gasifier. Test 33, which began on February 2, lasted over 5 weeks, during which the resiliency of the process was demonstrated by reestablishing the feed to the reactor following a number of interruptions. In the end, several elements that were not part of the process development program (namely, the coal mill, boilers, and hydraulic valve operator) necessitated the test's termination.

Test 33 was begun with the introduction of coal feed on February 5. Smooth solids flow was established throughout the unit for the steady-state period prior to the introduction of oxygen in the steam-oxygen gasification zone. A 0°F cold wave plus strong winds on the night of February 6 caused some freezing and sluggish instrument response. Lacking significant flow and differential pressure readings, solids flow was interrupted to clear these lines.

Coal feed was resumed on February 8 after the necessary instruments on the reactor structure that were affected by the cold weather were fixed. However, the cold spell continued, and on February 8 the temperature dropped to -8°F. The 6-inch-diameter, waste-gas header going to the flare froze, despite a continuous flow of process gas through the line, and the resulting back pressure caused the hydrogen plant to automatically shut down. No problems occurred in this line before. Since then, steam tracing was added to this line, even though by all calculations such freeze-ups should not have occurred.

During this period when instrument readings on the reactor were affected by the cold weather, the pressure in the nitrogen jacket was not properly indicated, which resulted in a blown rupture disk between the nitrogen jacket and the process side of the gasifier. Moisture entered the jacket and condensed. Two manways in the nitrogen jacket had to be opened up to drain the water and dry out the insulation. The thermocouple lead wires were also damaged by the water and had to be replaced.

Test 33 continued with heat-up and reactor pressurization on February 16 and 17. Coal feed was introduced to the reactor at 0600 hours on February 17, and process oxygen feed was introduced to the reactor at 1640 hours on the same day. Both coal and oxygen feed rates continued over a steady-state period until 1510 hours on February 18. No unusual solids feed or oxygen feed problems were apparent during this period, and the steady operation was very encouraging. However, control valve problems within the reactor jacket on the solids transfer line interrupted coal and oxygen feed. Leaks developed at the seals on the hydraulic valve operators, causing a loss of hydraulic oil and rendering the valves inoperable. The reactor was depressurized, the jacket opened, and all seals on the control valves replaced. These were the new two-way seals from Parker-Hannifin, which have been performing well for longer than 6 months. The upper seals appeared to have been damaged by the coal dust and water that entered the nitrogen jacket when the rupture disk blew.

The run was resumed with reactor heat-up and pressurization. Coal feed was initiated at 0530 hours on February 21. At 0730 hours, a leak

developed at the integral flow transmitter in the line ahead of the control valve used for venting the char-slurry mix drum (vessel 3.06-45). Coal feed was interrupted, the line ahead of the control valve replaced, and the control valve and its operating mechanism reinstalled. Coal feed was resumed at 1430 hours on February 21.

From February 21, coal feed was continuous for 162 hours until 0900 hours on the morning of February 28. During this period, 244 tons of coal were fed at an average rate of approximately 1.5 tons/hr. The feed rate varied from 1 ton to a maximum of 2.2 tons/hr over this period and was limited by the output from the coal mill. Oxygen was introduced to the reactor at 1730 hours on February 21 and was continuously fed for 154 hours until 0300 hours on February 28. The oxygen feed rate varied from 4000 to 7000 SCF/hr — about 75% of the planned oxygen feed rate for the equivalent coal feed rate during this period. This was the most stable period of gasifier operation since the integration of the steam-oxygen gasifier.

Oxygen and coal feeds were interrupted on February 28 when there was difficulty in withdrawing solids from the reactor's steam-oxygen bed through the bottom flow control valve. Agglomerate or clinker formation in the steam-oxygen bed was one possibility; another could have been plugging of the discharge slurry letdown chokes. We cleared the chokes on February 28 and emptied the reactor of solids by depressurizing the discharge slurry tank. The run was continued, introducing coal and later oxygen into the reactor.

Coal feed was resumed on February 28 and steam and oxygen flow soon after. It was continued until March 1, a period of 30 hours. Continuing trouble with the bottom discharge from the reactor interrupted coal and oxygen feed to the unit. Coal feed was resumed on March 2 and continued in an intermittent fashion until March 3. During this period, solids feed through the reactor was sluggish with plugging problems in the solids flow from the first to the second stage and from the second stage to the steam-oxygen bed, in addition to problems with discharging solids through the bottom of the reactor. The primary difficulty in moving solids from the bottom of the reactor was diagnosed as a collection of small-size (4 to 8 mesh) ash agglomerates that accumulated in the bottom of the reactor during the week of February 24. The ease with which agglomerates formed indicates that selectively removing ash from the bottom later on in the program could be entirely feasible.

After clearing the reactor of the agglomerates as much as possible, coal feed was resumed on March 4 and continued for a 34-hour period until March 6. Steam and oxygen were also introduced to the reactor during this period. On March 6, when the high-pressure boiler went off-line, coal feed and steam and oxygen feed were stopped. (A discussion of the problems with the high-pressure boiler follows.)

When the high-pressure boiler was put back in service, it took almost 2 days to get the solids out of the reactor. Some of the small agglomerates, which were occasionally found in the discharge slurry stream, were still present in the discharge slurry tank. As many as possible were removed

without completely depressurizing or purging the reactor system and opening up the discharge slurry tank. Coal feed was resumed early on March 10; however, shortly after that, a position indicator that supplies feedback signals to the hydraulic valve operator on line 321 between the slurry dryer and the lift line failed. Without feedback, that control valve could be operated either completely open or completely closed, and only through a great deal of effort could it be made to operate manually in an intermediate position. Level control in the slurry dryer bed, therefore, was erratic. In addition, on March 13, the low-pressure boiler went off-line. Although the boiler was brought back on-stream, the test was terminated on March 14 to repair some of the mechanical equipment.

During Test 33, the coal mill could not produce more than 1.5 tons/hr of coal feed to the gasifier, which limited the capacity of the gasifier. All during the test, the amount of firing in the mill furnace was limited by the high temperature in the connecting duct between the furnace and the coal mill. The necessity of keeping that temperature below the upper limit of 1500°F fixed the rate of feed to the mill. Extensive pressure profile measurements were taken as well as Pitot tube measurements for checking the capacity of the primary and secondary air fans. Oxygen concentration measurements were made at a number of points in the system. These eventually led to the conclusion that air was leaking into the mill circuit. The hot duct between the furnace and the mill had a number of sizable cracks. These problems were discussed with Mr. Williams, President of the Williams Patent Crusher Co., who agreed with this approach completely. The ductwork in this area was rebuilt. The construction work was completed by the end of March, and a coal-drying test was conducted to confirm the mill's ability to supply at least 3 tons/hr of dried coal solids for the next run.

The purification section operated satisfactorily during Test 33. The high-pressure amine pump was taken off-line twice to have its seals replaced. The activated carbon filter for the diglycolamine solution was regenerated, but no further maintenance activities were done on this section of the plant.

The methanation section was activated early during Test 33 for 66 hours and was placed on standby during the last part of the test. The check valves on the recycle compressor were also replaced during the test. No maintenance work was done on this section of the plant during this shut-down period. The hydrogen plant performed satisfactorily during the entire test.

One of the two plant air compressors, which had a mini-lube system added to it, was put into service in January. Immediately a 50°F to 75°F drop in discharge temperature was noted — apparently the result of the elimination of friction. There were filters downstream to trap the oil vapor before air got into the instrument air dryer.

The strike at Chemetron, which lasted over 3 months, was finally settled on January 30. During this time, through various arrangements, the supply of nitrogen or oxygen was not held up. With the settlement of the strike, the shipments of cryogenic gases returned to normal.

A shutoff valve on the sight gage on the high-pressure boiler developed a leak, and moisture and condensate from this leaking valve, which could not be repacked or replaced under pressure, caused electrical shorting in the flame safeguard system on the high-pressure boiler, thus forcing a shutdown on the morning of March 6. The high-pressure boiler was taken out of service, the steam valve repaired, and the damaged electrical controls on the boiler replaced. The boiler was put back into service on the evening of March 6 and operated routinely.

In the low-pressure boiler system, operations were limited by the output of the demineralizer system. In an effort to conserve demineralized water, the induced draft fan on the boiler was switched over from turbine drive to motor drive on March 13; however, the heaters on the motor starter tripped during the night, shutting down the low-pressure boiler. The demineralizer operation was reviewed to increase its capacity for demineralized boiler feedwater. A modified cycle on the demineralizer operation might accomplish this purpose.

The discharge slurry filter that separates the char residue from water for disposal had not been performing up to design capacity for some time: The disk filter elements could not hold the cake on the disks. An Edens separator was ordered, which should remove most of the particles that settle easily. The disk filter will be replaced with a drum filter to clean up the effluent from the Edens separator.

All accumulated solids were cleaned out of the char-slurry retention pond, and the maintenance work on the char-slurry filter to bring it up to design solids-removal capacity was finished. In case of a filter failure, a clean retention pond will allow several weeks of operation at design solids throughput before it accumulates enough solids to become an effluent cleanup problem.

Gasification Test 34, April 1975

The gasifier reactor was pressure-tested, purged, and relighted for heat-up late on April 11. Coal feed was introduced to the reactor by noon of April 12 to begin Test 34. The coal feed rate was as high as 3 tons/hr for substantial periods. Controls on the gasifier were excellent, and its operation was stable.

Coal feed was interrupted on April 15 when one of the quench-water circulating pumps stopped because the original pump shaft size was not big enough to handle the torque generated by the quench water plus some coal dust. A redesigned shaft, shaft sleeve, and seal arrangement have been developed by the Institute of Gas Technology and were ready for installation. The stoppage of the pump required activating the spare quench pump, thus upsetting the pressure in the reactor because of the volume reduction in the gas phase brought about when vapors were condensed in the quench tower. This transient period was handled well; the pressure differential was equalized. However, the solids feed rate through the transfer lines, 321 and 322, was sluggish until the solids were gradually removed from the bed to obtain a fresh start. This was done on April 16, and coal feed was resumed.

A critical milestone was reached in the HYGAS program during Test 34, when the entire plant was operated through the methanation section without supplemental heat supply or hydrogen. Operation was completely self-sustained, using only steam-oxygen gasification to supply the necessary heat of reaction as well as to produce the hydrogen-rich gas for gasification. Operation was smooth and stable with automatic control of solids transfer in the gasifier and complete methanation of the total product gas. The only inert gas in the final product was nitrogen, which was added to the system for instrument purges. On a nitrogen-free basis, the product gas reached 96.5% methane and 3.5% hydrogen, with a heating value of over 970 Btu/CF. The successful operation in this mode culminates the HYGAS group's intensive effort over the last few years during which many modifications and improvements were incorporated into the plant. A complete, new generation, coal-to-SNG plant was demonstrated in an integrated fashion for the first time anywhere.

Test 34 was continued for 29 hours without external heating. The last 20 hours of this period was without any external hydrogen supply. Test 34 was interrupted when a faulty pressure switch on the oxygen supply system (Chemetron) erroneously locked out the electric power to the oxygen pad. By the time the difficulty was diagnosed and corrected, the oxygen flow to the gasifier had become insufficient and could not maintain temperatures; therefore, the bed could not be refluidized. The test was interrupted while the solids were drained out of the reactor. The system was ready and was reheated to resume Test 34.

The transfer valve, LV 321, between the slurry dryer and the first-stage gasifier stuck in a 65% open position. Operation continued with that valve position until the test was interrupted. Fortunately, the valve position allowed up to 2.5 tons of coal to be put through. Before the test was restarted, the reactor was depressurized to allow entrance into the nitrogen jacket to repair that valve. The repeated motion of the hydraulic operator had unthreaded the end connection on the hydraulic shaft. This shaft was pinned, and operation resumed.

When solids in the reactor were removed, and LV 321 that controls discharge from the slurry dryer was repaired, reactor heat-up was begun once more on April 28, and coal feed was introduced to the reactor on April 29.

By 1 p.m., April 30, all of the hydrogen was removed from the start-up heater. Once more, the operation became completely self-sustained, using only steam-oxygen gasification to supply the necessary heat of reaction to the process. Operation continued in this mode for 25 hours until difficulties with solids discharge from the second-stage hydrogasifier to the steam-oxygen stage forced an interruption of steam-oxygen gasification.

For the second time then, the HYGAS steam-oxygen gasification system was operated on a completely self-sustained basis with no supplemental heat or hydrogen added to the reactor. It only took 5 hours to cut out the hydrogen in steps, and the transition was made smoothly. During the coal feed period, LV 321 operated satisfactorily: A coal feed rate of approximately 3 tons/hr was achieved and maintained.

Test 34 was terminated on May 6 after three unsatisfactory attempts to feed coal to the gasifier were made from May 3 to 6. Valve 339, which controls the flow of solids through the transfer line from the second stage to the steam-oxygen zone, could not be operated satisfactorily, and external indications were that it was stuck in the 85% open position. The test had to be terminated because this valve would not operate and transfer line 339 became plugged.

During the time the coal feed began on April 12 to the first interruption on April 22, a total of 203 hours of feed to the gasifier was logged. After we resumed coal feed on April 29, we logged an additional 70 hours of coal feed for a total of 273 hours during the run. A total of 457 tons of coal was fed to the gasifier, representing about 600 tons of the as-received coal to the plant.

During the first period, there was a 99-hour stretch when the methanation section was on-stream, the last 29 hours of which operated without the start-up heater in service. No hydrogen was added to the gasifier for the last 20 hours of this 29-hour period. A typical gas analysis, made during this 20-hour period, is shown in Table 3.

Table 3. GAS ANALYSIS FOR TEST 34, APRIL 21, 1975

<u>Components</u>	<u>After Quench</u>	<u>After Purification</u>	<u>After Methanation</u>	<u>After Methanation (Nitrogen-Free)</u>
H ₂	0.3209	0.4595	0.0409	0.0498
CO ₂	0.3113	0	0	0
C ₂ H ₆	0.0019	0.0114	0	0
H ₂ S	0.0043	0	0	0
N ₂	0.0714	0.1079	0.1797	--
CH ₄	0.1827	0.2641	0.7794	0.9502
CO	0.1075	0.1571	0	0
Molecular Weight	22.4	12.9	17.6	15.3
Heating Value, Btu/SCF	325	484	798	973

Note that of the total amount of methane produced, 65% is produced in the gasifier, with the balance being produced in the methanation section. This split agrees with the predictions of our kinetic model that about two-thirds of the total methane would be produced in the HYGAS reactor. Also note that the hydrogen-to-carbon monoxide ratio entering the methanator section was roughly 3:1, ideal for methanation. Despite the fact that there is no water-gas shift section in the plant, the gasifier output indicates that at least with lignite, a shift section may not be required.

Material and heat balances, based on average data for the period from April 21 to 22, when the external hydrogen and supplemental heat were cut out, are presented in Figures 9 through 12 and in Tables 4 through 9. These show balances around the gasifier, purification, and methanation sections,

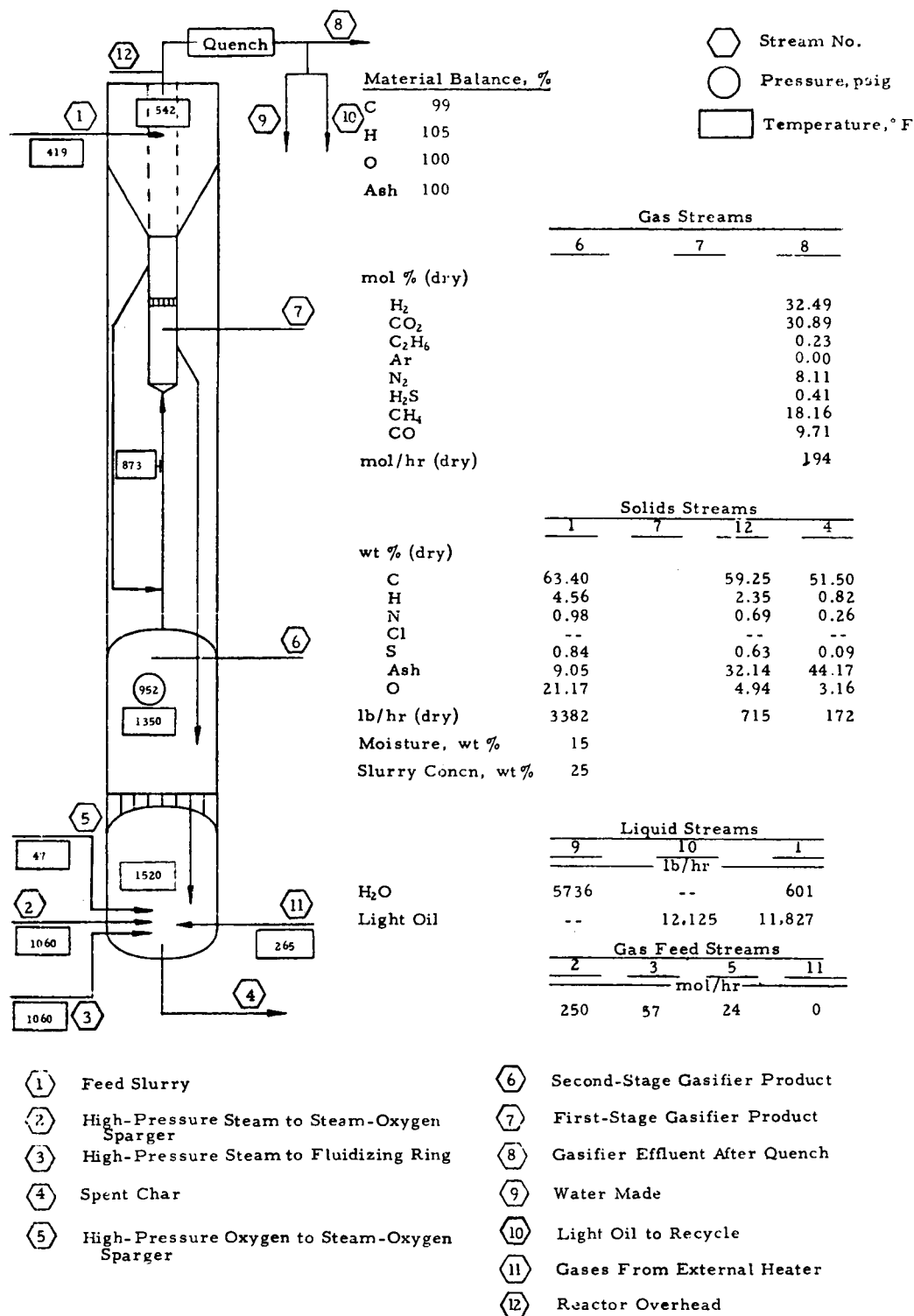


Figure 9. HYGAS REACTOR DATA FOR TEST 34 FOR THE STEADY PERIOD FROM 4/21/75 (0707 hr) TO 4/22/75 (0197 hr)

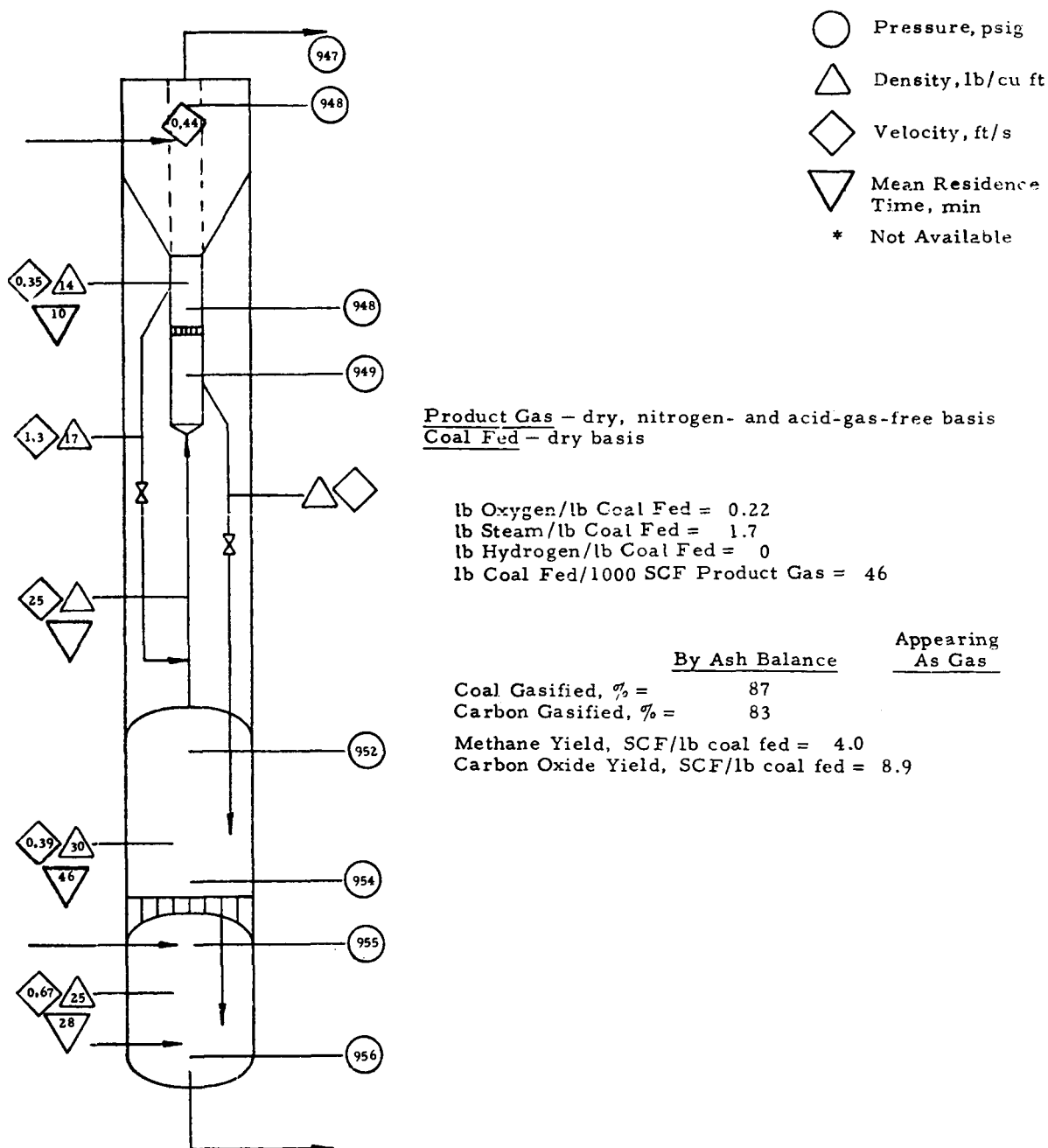
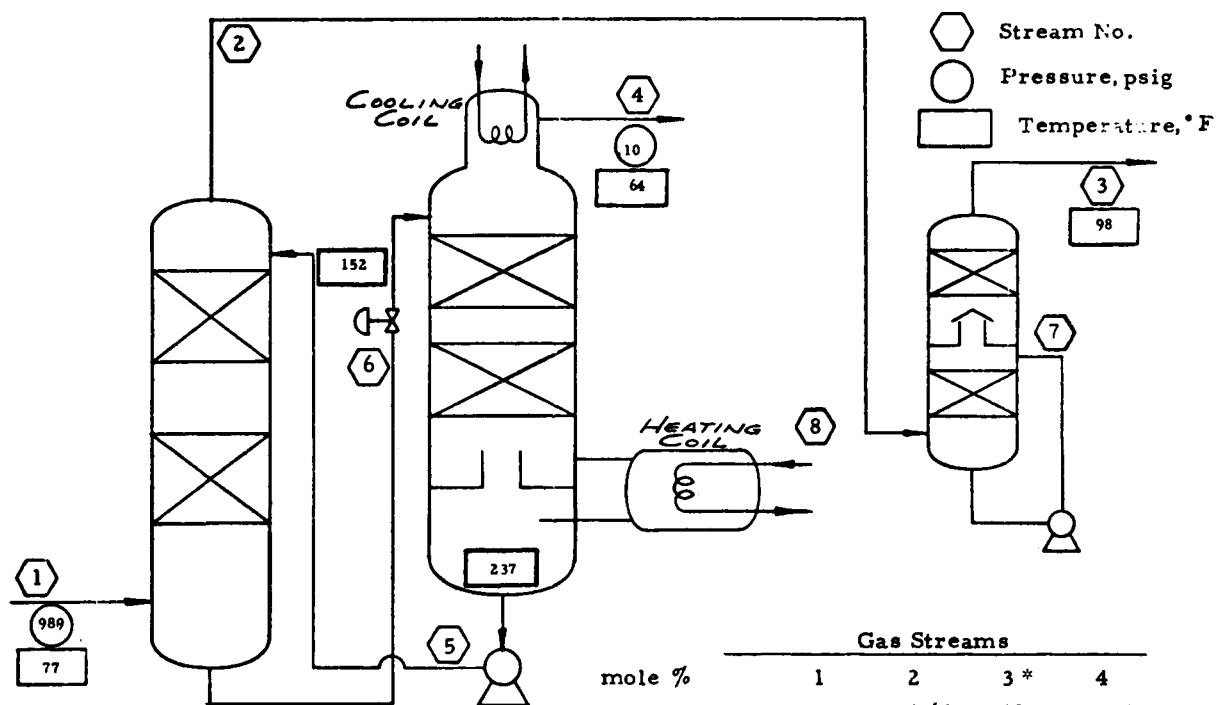


Figure 10. HYGAS REACTOR ENGINEERING DATA FOR TEST 34 FOR THE STEADY PERIOD FROM 4/21/75 (0707 hr) TO 4/22/75 (0197 hr)



Material Balance, %

C	97
H	99
O	91

Liquid Streams

	5	6	7	8
% Amine	36	36	--	--
% Caustic	--	--	7	--
lb/hr	50,200	51,400	5,070	3,965

Liquid Streams

	5	6
Acid gas loading of DGA, lb mole/gal	--	--

* The mole % in this stream is obtained independently by IR spectrometers.

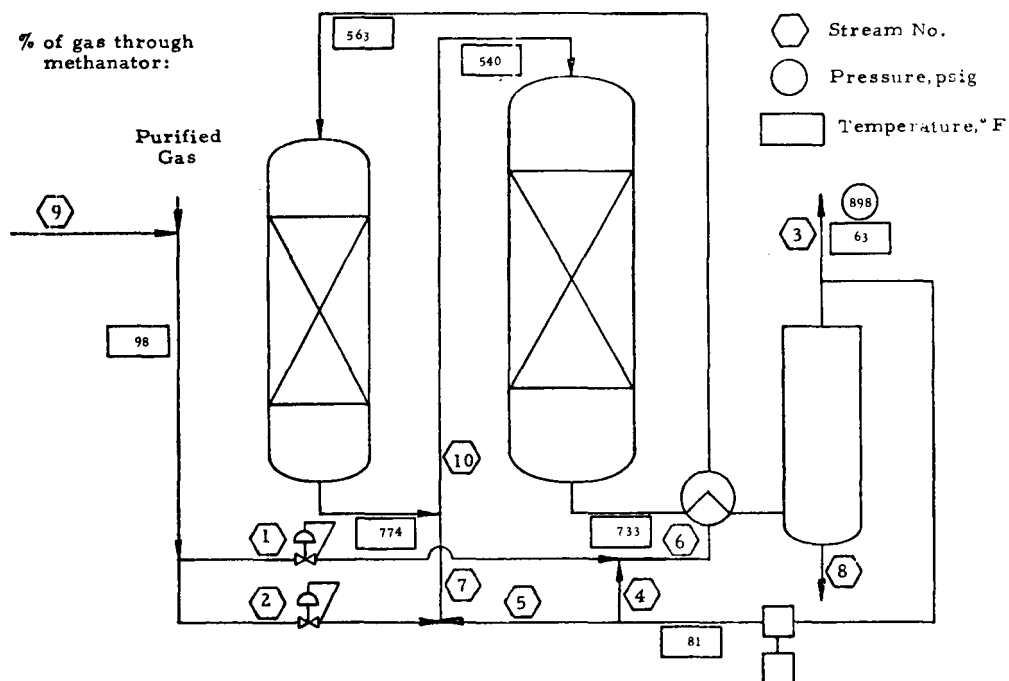
Gas Streams

mole %	1	2	3 *	4
H ₂	32.49	45.69	49	3.22
CO ₂	30.89	0.06	0	92.25
C ₂ H ₆	0.23	1.15	--	0.10
Ar	0.00	0.00	--	0.00
N ₂	8.11	12.53	--	1.71
H ₂ S	0.41	0.00	--	0.40
CH ₄	18.16	26.31	--	2.32
CO	9.71	14.25	14	0.00
mol/hr	100	65	30	30
Sulfur Analysis (ppm)				
CH ₃ SH		0.0014	0.0003	
CH ₃ SCH ₃ -CH ₃ CH ₂ SH		0.0059	0.0002	
CS ₂		0	0	
(CH ₃) ₂ CHSH		0	0	
CH ₃ SCH ₂ CH ₃		0	0	
H ₂ S		0.1027	0.0004	
COS		0.0010	0.0088	
CH ₃ SSCH ₃		0	0	

- ① Gas to Purification
- ② Scrubbed Gas
- ③ Purified Gas
- ④ Regenerator Effluent

- ⑤ Lean DGA
- ⑥ Rich DGA
- ⑦ Caustic Wash
- ⑧ 40 lb Steam

Figure 11. GAS PURIFICATION DATA FOR TEST 34 FOR THE STEADY PERIOD FROM 4/21/75 (0707 hr) TO 4/22/75 (0197 hr)

**Material Balance, %**

C 104
H 102
O 99

	Gas Streams									
mol %	1	2	3	4	5	6	7	8	9	10
H ₂	49.82	49.82	13.30	13.30	13.30	21.63	33.02		98.24	19.13
CO ₂	0.06	0.06	0.02	0.02	0.02	0.03	0.06		0.00	0.04
C ₂ H ₆	1.06	1.06	0.00	0.00	0.00	0.24	0.57		0.00	0.16
Ar	0.00	0.00	0.00	0.00	0.00	0.00	0.00		0.00	0.00
N ₂	11.50	11.50	18.79	18.79	18.79	17.14	14.89		0.51	17.72
H ₂ S	0.00	0.00	0.00	0.00	0.00	0.00	0.00		0.00	0.00
CH ₄	24.31	24.31	67.90	67.90	67.90	57.95	44.35		0.41	60.93
CO	13.17	13.17	0.00	0.00	0.00	3.00	7.11		0.84	2.02
mol/hr	38.1	32.7	42.9	128.9	27.8	167.0	60.5	9.3	5.4	212.4
H.V. (N ₂ , acid gas free)	528	528	895	895	895	805	689		325	839

	First Stage	Second Stage	Overall
Recycle Ratio	3.4	0.85	2.2
Space Velocity Based on Total Bed, hr ⁻¹	4030	2560	--
Pressure Drop (Inches H ₂ O)	7	29	36
% Bed Utilized (From Temp Profile)	46	12	24
Space Velocity Based on Bed Utilized, hr ⁻¹	8800	20,500	--

① Purified Gas to First Stage	⑥ First-Stage Feed Gas
② Purified Gas to Second Stage	⑦ Recycle and Fresh Feed to Second Stage
③ Product Gas	⑧ Condensate
④ Recycle Gas	⑨ Makeup Hydrogen
⑤ Recycle Gas	⑩ Second-Stage Feed Gas

Figure 12. METHANATION DATA FOR TEST 34 FOR THE STEADY PERIOD FROM 4/21/75 (0707 hr) TO 4/22/75 (0197 hr)

Table 4. MATERIAL BALANCE SUMMARY FOR HYGAS GASIFIER FOR TEST 34

Basis = 1 hr. All units in lb unless noted otherwise.										
INPUT		C	H	O	N	S	Cl	ASH	OTHER	TOTAL
Coal Feed	Wt % (Dry)	63.4	4.56	21.17	0.98	0.84	--	9.05		100
	Coal (Dry)	2144	155	716	33	28		306		3382
	Moisture		67	534						601
Sparger	Oxygen			752						752
	Steam		501	4005						4506
Burner	Oxygen									0
	Steam									0
	Hydrogen									0
Stripping Ring	Steam		115	919						1034
Nitrogen from purges					471					471
Pump seal flush			56	444						500
TOTAL INPUT		2144	894	7370	504	28		306		11246
OUTPUT										
Reactor Overhead	Wt % (Dry)	59.25	2.35	4.94	0.69	0.63		32.14		100
	Dust (Dry)	424	17	35	5	4		230		715
Spent Char	Wt % (Dry)	51.5	0.82	3.16	0.26	0.09		44.17		100
	Char (Dry)	89	1	5	1	0		76		172 ^a
Product Gas	Total (Dry)	1383	271	2226	441	27				4348
	Components H ₂		126							126
	CO ₂	721		1924						2645
	C ₂ H ₆	10	3							13
	H ₂ S		1			27				28
	N ₂				441					441
	CH ₄	425	141							566
Water Out	CO	227		302						529
		33	615	4868	23	2				5541 ^b
Toluene storage tank vent gases		190	44	236	107					577 ^c
TOTAL OUTPUT		2119	948	7370	577	33		306		11353
Net (Output-Input)		-25	54	0	73	5		0		107
% Balance (Output/Input)		99	106	100	114	117		100		101

Toluene	
In	11827 ^d
Out	12125
% Balance	
Output/Input	102

Test #	34
From	4/21/75, 0707 hr
To	4/22/75, 0197 hr

^a The spent char stream rate is obtained by ash balance. This will be resolved in the future by a more effective sampling procedure, namely, a continuous sampler.

^b The oxygen in this stream is obtained by estimating the amount of "glop" blowdown. The rest of the components are obtained by actual analyses done on the water samples from this section. With the recent incorporation of the glop stripper actual amounts of oil, water, and solids will be measured.

^c The amount of gases vented out of the toluene storage tank is measured by a Rootsmeter and gas analyses are done by mass spectrometer.

^d The excess toluene that comes from the gasifier may be due to the addition of clean toluene from a separate source.

Table 5. HEAT BALANCE SUMMARY ON HYGAS GASIFIER

Test No. 34 (4/21/75, 7.07 hrs to 4/22/75, 1.97 hrs)

Basis: 1 hr

Datum temperature 77 °F, liquid water

<u>INPUT</u>		<u>1000 Btu</u>
Coal (1)	Heat of Combustion	36.08
Feed Streams (1), (2), (3), (5)	Sensible Heat + Latent Heat	<u>10.51</u>
Total Input		46.59
<u>OUTPUT</u>		
Char (4) (12)	Heat Combustion and Gas Streams (8)	
Heat Combustion		36.39
Outlet Streams (8), (9), (10), (4)	Sensible Heat + Latent Heat	<u>9.80</u>
Total Output		46.19

The difference in the heat is within the limits of expected heat loss from the gasifier. Refer to Figure 1 for stream numbers.

Table 6. MATERIAL BALANCE SUMMARY FOR PURIFICATION SECTION

Basis = 1 hr. All units in lb unless noted otherwise.

INPUT		C	H	O	N	S	TOTAL
Product Gas From Quench	Components H ₂		65				65
	CO ₂	370		986			1356
	C ₂ H ₆	6	1				7
	H ₂ S		1			13	14
	N ₂				227		227
	CH ₄	217	73				290
	CO	116		155			271
TOTAL INPUT		709	140	1141	227	13	2230
OUTPUT							
Product Gas From Purification	Total (Dry)	344	134	143	230	0	851
	Components H ₂		60				60
	CO ₂	1		1			2
	C ₂ H ₆	18	5				23
	H ₂ S						0
	N ₂				230		230
	CH ₄	206	69				275
	CO	119		142			261
Acid Gas From Regenerator	Total (Dry)	343	5	892	14	4	1258
	Components H ₂		2				2
	CO ₂	334		892			1226
	C ₂ H ₆	1					1
	H ₂ S					4	4
	N ₂				14		14
	CH ₄	8	3				11
	CO						0
Dissolved Gases							
TOTAL OUTPUT		687	139	1035	244	4	2109
Net (Output-Input)		-22	-1	-106	17	-9	-121
% Balance (Output/Input)		97	99	91	107	31	95

Test # 34From 4/21/75, 0707 hr
To 4/22/75, 0197 hr

Table 7. HEAT BALANCE SUMMARY OF PURIFICATION SECTION

Test No. 34 (4/21/75, 7.07 hrs to 4/22/75, 1.97 hrs)

Basis: 1 hr

Datum temperature 77 °F, liquid water

<u>INPUT</u>	<u>1000 Btu</u>
Gas Stream 1 Heat of Combustion	12.22
40 lb Steam Latent Heat	<u>3.65</u>
Total Input	15.87
 <u>OUTPUT</u>	
Gas Streams 2 , 4 Heat of Combustion	12.27
Heat Exchangers, Sensible Heat	<u>3.72</u>
Total Output	15.99

Refer to Figure 3 for stream numbers.

Table 8. MATERIAL BALANCE SUMMARY FOR METHANATION SECTION

Basis = 1 hr. All units in lb unless noted otherwise.

INPUT		C	H	O	N	S	TOTAL
Purified gas to methanation	Components H ₂		70				70
	CO ₂	1		1			2
	C ₂ H ₆	18	5				23
	H ₂ S						0
	N ₂				229		229
	CH ₄	207	69				276
	CO	111		149			260
TOTAL INPUT		337	144	150	229		860
OUTPUT							
Condensate from Methanator			19	148			167 ^a
Final Product Gas	Total (Dry)	350	128	0	227		705
	Components H ₂		11				11
	CO ₂						0
	C ₂ H ₆						0
	H ₂ S						0
	N ₂				227		227
	CH ₄	350	117				467
	CO						0
TOTAL OUTPUT		350	147	148	227		872
Net (Output-Input)		13	3	-2	-2		12
% Balance (Output/Input)		104	102	99	99		101

Heating Value	Btu/SCF (N ₂ , Acid-gas free)
Gas to methanator	528
Final product gas	895

Test #	34
From	4/21/75, 0707 hr
To	4/22/75, 0197 hr

^a Condensate is calculated.

Table 9. HEAT BALANCE SUMMARY FOR METHANATION SECTION

Test No. 34 (4/21/74, 0707 hrs to 4/22/75, 0197 hrs)

Basis: 1 hr

Datum temperature 77 °F, liquid water

<u>INPUT</u>	<u>1000 Btu</u>
Streams ① and ② Heat of Combustion	12.50
<u>OUTPUT</u>	
Stream ③ Heat of Combustion	11.84
Sensible Heat	<u>0.73</u>
Total Output	12.57

Refer to Figure 4 for stream numbers.

which are good and suitable for design. Around the gasifier, further improvements in discharge char sampling should allow closure of the ash balance. Commissioning the light-oil stripper section should allow closure of the oil (toluene) and water balances. In the purification section, improvements in the regenerator effluent gas analysis and flow rate should tighten the mass balance. The methanation section mass balance is very good. A small amount of hydrogen was added to the methanation feed gas to compensate for the absence of a water-gas shift reactor. This addition, represented by stream 9 in Figure 12, is not necessary when the hydrogen/carbon monoxide ratio exceeds 3:1. The data in Table 3 were obtained when, on several occasions, stream 9 was cut off. However, for most of the 20-hour period, a small hydrogen flow was maintained to protect against upsets. The excess hydrogen diluted the product-gas heating value to 895 Btu/SCF from the 973 Btu/SCF figure shown in Table 3, both on a nitrogen-free basis. In future tests, this hydrogen addition will be monitored more closely and will be eliminated whenever possible.

In regard to heat balances, Tables 5, 7, and 9 show that total input and output for each section are essentially in balance. Independent estimates of heat losses will be obtained to refine the heat balance. In Table 5, around the gasifier section, the slurry oil is not included in the heat balance; when the oil balance is closed, the heat balance will also improve.

The gasifier reactor was opened for inspection following the completion of Test 34. The steam-oxygen gasifier contained a few clinkers. Also, a number of the bricks in the manway (to which the start-up heater is attached) had fallen into the bed. Clinkers had formed around some of these bricks. It was not clear if the bricks had fallen in first, causing the flow distribution of gas to be altered, which then led to the clinkers being built up around the bricks. Possibly the clinkers formed when the oxygen flow to the gasifier was increased. Small beads were never observed in the discharge slurry, as they were during Test 33, and therefore, the formation of clinkers in the gasifier was not suspected at that time.

A clinker lodged at the valve opening of LV 339 and prevented it from closing. There was nothing wrong with either the valve or with the line 339 from the second-stage gasifier to the steam-oxygen bed. The line was straight, and the valve was operable once the clinker was removed. To prevent the bricks from falling into the reactor, a metallic sleeve was installed. Racks for the metal coupons were also installed in the gasifier wherever possible. The rack for the steam-oxygen gasifier had not been received from IITRI as yet.

After the cracked piping on the coal mill system was repaired, the mill was operated successfully for 2 to 4-hour test periods with up to 5 tons/hr of crushed, dried coal. Three modifications and changes in operating procedures improved operation. First, the speed of the secondary circulation fan was increased by 18% from 1500 rpm to about 1770 rpm. This circulated a much greater volume of cool, inert gas back through the coal mill furnace and into the wind box. Second, the air-to-gas ratio on the combustion chamber of the furnace was increased from 100% of stoichiometric to 150% of stoichiometric. Third, an internal velocity control cone in the upper part of the Williams mill was adjusted to give maximum velocity and minimum retention time for ground coal in the mill.

The coal mill's overall operation was satisfactory: The analysis of the coal product showed a good distribution, without too much fines. A tremendous rainstorm hit Chicago on April 18 during which almost 4 inches of rain was measured in the area. The coal pile became very wet, which made feeding into the coal mill difficult. During this period, the repeated sticking of solids in the rotary feed valve kept the capacity of the mill down to about 2 tons/hr of useful product. However, even with 2 tons/hr, the entire plant was maintained at a self-sustained state of operation. While waiting for the plant to be restarted, three journals to the mill, which had been taken out earlier in an attempt to further increase the mill's capacity, were put back in operation. Initial tests indicate that the capacity of the mill can be maintained at 5 tons/hr. Enlarging the primary and secondary fans on the mill system to further improve its performance was also considered.

The revamp work on the pretreater was essentially complete. The refractory was installed in the reactor, as were the cyclone diplegs. The reactor was cleaned up inside, and the instruments were checked.

The slurry system operated well, although the reciprocating slurry pumps were plugged for short times on three occasions. Excessive vibration in the discharge piping caused one such interruption. The piping was checked and the addition of special pulsation dampeners was considered to solve this difficulty. The normal diaphragm in pulsation dampeners is made of elastomers, which will not withstand the action of the light oil. If a resistant elastomer can be found, the dampener will be purchased and installed at the appropriate location.

The purification system operated well, producing sulfur-free feed gas to the methanation section throughout the test. The methanation section operated well. It was on-stream for 99 hours continuously, producing a completely methanated product gas, which had reached a composition of 3.5% hydrogen and 96.5% methane on a nitrogen-free basis. The only sources of nitrogen were from the numerous pressure taps and instrument purges throughout the system. The ratio of hydrogen to carbon monoxide was almost exactly 3:1, so that no shift adjustment was required. Although there was no water-gas shift section in the plant, with lignite, the H_2/CO ratio was naturally about 3:1, an ideal methanator feed gas.

The utilities in the plant are in operation. A higher capacity city water booster pump to increase the throughput of water to the demineralizer was ordered. This should relieve the problem of high water demand during the regeneration cycle of the demineralizer operation. The turbine-driven, low-pressure boiler feedwater pump had to be taken off-line for repair. However, operation continued with the spare, electric motor-driven pump.

Gasification Test 35, May 1975

Test 35 began on May 20 with the initiation of coal feed to the gasifier. Operation was smooth, and successive sections of the plant were being brought on-stream when the high-pressure boiler tripped out on May 21, interrupting the test. With the sudden stoppage of high-pressure steam

flow to the gasifier, the bed defluidized. All of the solids were drained out from the lower stages; however, line 321 between the slurry dryer bed and the first-stage gasifier was plugged, possibly because of the condensation of moisture in that line. Test 35 was terminated.

The expansion joint in that line apparently was overextended probably because of some hot gases bypassing up through the line. As a result, the line below the solids control valve, LV 321, bent, and the expansion joint cracked. The expansion of the line apparently pushed the gaskets on the flanges on LV 321 together to the point where the ball in the ball valve seized, resulting in the shearing of a keyway on the shaft extension.

Control valve LV 321, the expansion joint, and the bent pipe below the expansion joint were replaced. The solids in the pipe were tightly packed, presumably because of the condensation of moisture.

While line 321 was being repaired, the manway on the steam-oxygen gasifier was opened up for inspection. Everything was in excellent shape: The reactor was clean, and the metallic sleeve installed in the manway to hold the bricks in place appeared to be operating properly because all the bricks were in place and intact.

The light-oil stripper section was completed during the week of May 9. It was shaken down during the following week and commissioned during Test 35. Operation was quite smooth, and the results were gratifying. A clean, light oil was recovered from the overhead condenser separator and returned to the seal flush tank. Surplus oil can be produced from the plant when this recovery section is on-stream. This section was carefully monitored to ensure complete oil recovery and material balance.

The slurry system operated well overall. On two occasions the slurry flow to the reactor was interrupted because of a pressure shutoff of the discharge from the reciprocating pump.

During Test 35, the purification section was put on-stream for a short time before the run was interrupted. Afterwards, it was put on standby.

The utilities operated well overall. During Test 35, a lack of feedwater to the boiler shut off the high-pressure boiler. This was most likely the cause of a sudden change in pressure on the deaerator that fed the boiler feedwater pumps. With the deaerator operating at the boiling point under 7-psig pressure, possibly a dip in the deaerator pressure could have caused flashing, and hence cavitation, at the boiler feedwater pump. The pressure-control sensing point was moved from the input steam line to a nozzle on the deaerator and a faster-acting controller installed. The vendor also worked to see if additional improvements could be made to eliminate pressure surges and thus avoid flashing in the feed line.

Gasification Test 36, June 1975

Test 36 began with coal feed at 0200 hours on June 5. Operation was stabilized, hydrogen was cut back in steps, the purification section was brought on-stream, and the methanation section was ready to be activated.

At around 0200 hours on June 6, the feed slurry concentration appeared to suddenly increase, despite the fact that operation had been stable for over 10 hours. This increase caused the slurry line on the discharge side of the Wilson-Snyder reciprocating pump to become plugged, thus interrupting the coal feed.

The plug in the coal-slurry feed line was cleared, and the test was continued on June 7. On June 8, the bed-density and bed-level instrumentation line for the steam-oxygen gasification bed became plugged, and coal feed was interrupted to clear these lines. During the clearing operation purging with high-pressure nitrogen blew the lower rupture disk, and the reactor had to be depressurized to 200 psig to replace the ruptured disk. The external heater was kept in service, and the reactor remained on-stream.

The reactor was then pressurized, and coal feed resumed on the afternoon of June 9. Smooth operation was established, and oxygen was introduced at 6 p.m. The auxiliary heater was shut down at 10 p.m., and all external hydrogen was shut off at 10:30 p.m. Self-sustained gasification was continued from June 9 at 10:30 p.m. until June 10 at 5 p.m. The purification and methanation sections were in operation during this period. Coal feed varied from 1.5 to 2 tons/hr, and the methanated product-gas heating value was 980 Btu/SCF on a nitrogen-free basis.

Steam-oxygen bed temperatures ran between 1500°F and 1600°F. About 1 p.m. on June 10, the thermocouple 3 feet above the steam-oxygen inlet indicated a temperature rise to 1800°F. The oxygen flow was reduced slightly and the temperature returned to normal, but evidently sintering had occurred because during the next 3 hours, operation of the second-stage discharge became increasingly difficult, and intermittent temperature excursions continued. The run was terminated at 5 p.m., when the second-stage discharge valve operation was limited to 20% of the full stroke and it became obvious that clinker formation was building up. The reactor was cooled with the bed materials kept in place in the various stages.

This test achieved the second, self-sustained HYGAS plant operation that lasted approximately 19 hours, with the entire train on-stream. Valuable data were gathered at the permissible temperature levels in the steam-oxygen zone and will be used to guide the selection of future test parameters.

When the reactor manways were opened following Test 36, an inspection revealed the presence of clinker formation in the steam-oxygen gasification zone and high-ash-content material throughout the bed. Clinkers had formed around the 339 valve operator, effectively reducing the stroke of that valve to about 20% of its full operating range. Eighty percent of the clinkers were in the form of loose material that could be broken up by hand; 20% were rather hard, glassy, high-temperature clinkers. There was no damage to line 339, valve 339, or the steam-oxygen sparger ring; all were in good operating condition. The site of the initial clinkers could not be determined from this location; but it was assumed they started forming somewhere near the end of valve 339. This condition, once initiated, made it unable to fluidize and maintain good mixing in the bed, and the

clinkers propagated. The start-up heater refractory area and the brickwork were all in good shape, with no damage to any of the refractory walls of the steam-oxygen gasification zone. No problems occurred in any of the reactor sections, with the exception of the expansion joint on the dry-coal feed line from the slurry dryer area to the first-stage gasifier (line 321). The expansion joint on line 321 was stressed slightly out of its normal position, but it remained intact throughout the test. This expansion joint was replaced with a double joint to double the expansion capability of this line to move more freely during the next test. The coal mill operated satisfactorily, and the gasifier coal feed rates were easily maintained. The refractory in the pretreater was cured, and the unit was put on standby. The slurry system operated well with no problems. The discharge valves on one of the Wilson-Snyder slurry pumps were modified to complete the ball-check design that was found to be successful in the HYGAS plant. Now both pumps are equipped with ball-check designs on the discharge ends of the pumps.

The purification section continued to do a good job removing carbon dioxide and sulfur with no apparent problems. The methanation section handled the feed gases with complete carbon monoxide conversion with no problems. The light-oil stripper section, which appeared to work well during the short time of Test 35, was activated during Tests 36 and 37. Some operating problems occurred with it because of solids carry-over from the stripper tower into the condenser and condensate receiver. Adding some auxiliary recycle to the top of the stripper tower overcame this problem. While the stripper was in operation, a light-oil inventory was maintained without requiring oil from storage.

The utilities operated routinely. Since the pressure-sensing point was relocated on the deaerator, the pressure control on the deaerator was much more stable.

Gasification Test 37, June 1975

Test 37 was initiated at midnight on June 21, 1975, with the introduction of slurry feed to the reactor. The purification and methanation sections were brought on-stream within the next 24 hours. Methanation was started at 0200 on June 23, the start-up heater was cut out at 0830 on June 23.

Test 37 continued in a self-sustained mode without supplemental heat or external hydrogen supply until it was voluntarily terminated on July 8. The lignite supply was essentially exhausted, or would have been in 1 more day. An operator's error in measuring the slurry tank inventory interrupted the slurry flow and, therefore, the feed to the gasifier. Although the system was cleared and the test resumed, there was not a sufficient coal supply to allow the plant to reach steady state again; therefore, we terminated the test voluntarily.

In all, Test 37 was the most successful to date in the program with 362.5 hours of self-sustained operation. During this period, 757 tons of dried lignite were fed to the gasifier. From the beginning of the coal feeding period, the test operated for 395 hours with a 795-ton total dried

lignite feed. More than 1000 tons of lignite were fed into the plant. During the period of self-sustained operation, the gas purification system was on-stream for 349 hours; the methanation section was on-stream for 333 hours.

The gasifier operated very well. The gasification system adapted to the several interruptions caused by slurry pumping problems. On two occasions following the slurry flow stoppage, the temperature in the steam-oxygen stage was raised from 900°F up to operating temperature. The resiliency of the gasifier system in handling upsets and interruptions has been amply demonstrated. Solids flow control through the various stages was consistently good.

The light-oil stripper section operated well during most of the run. An oil balance was established, which showed net production of oil from the process. Clean oil was returned to storage to be reused as seal flush. The stripper section was improved during its downtime for better cooling of the discharge and to increase the stripper's capacity.

Opening the gasifier reactor system, following the successful completion of Test 37, revealed that all vessels were in excellent shape. In the gasifier, some residual coal was found only on the slurry dryer grid, a condition to be expected because the contents between the grid and the discharge pipe could never be fully drained. These solids were left over from Tests 35 and 36, since the top manway was not opened prior to the beginning of Test 37. The lift line, the spouting bed at the top of the lift line, and the transfer lines from the spouting line to the second stage were all completely clean. Similarly, the second-stage gasifier and the transfer pipe from that stage to the steam-oxygen bed were clean. The steam-oxygen bed was exceptionally clean. The steam-oxygen sparger was completely intact with no deposits anywhere. No chipping or spalling occurred in the refractory. Indeed, this was the best gasifier condition following a test seen so far, and it was returned to service immediately.

The coal mill operated satisfactorily throughout the test and produced an adequate amount of feed material. There was sufficient excess capacity in the mill's operation to shut down at regular intervals for routine lubrication and maintenance.

The slurry system operated well overall; however, the vibration and the discharge-pressure fluctuations at the Wilson-Snyder pumps kept the feed rate from being increased much above 2.5 tons/hr for extended periods. Discussions with other users of the pump led to the tentative conclusion that the suction pressure on the Wilson-Snyder pumps should be increased to minimize the possibility of momentary flashing during the pump's suction stroke. The adaptability of a pulsation dampener to the discharge side of the Wilson-Snyder pump was investigated. On several occasions during Test 37 when attempts were made to get the coal feed rate up to 3 tons/hr, excessive vibration and pressure fluctuations caused the pumps to be shut down by pressure spikes that reached the shutoff pressure set at the discharge pressure switch. The final test period was conducted with the coal feed rate between 2 and 2.5 tons/hr without attempting to increase the rate to 3 tons/hr. During this time, the fluctuations, although still present, were tolerable.

The purification section operated well throughout the test. There was one short interruption due to problems with the caustic pump and another due to a shutdown of the amine-recycle pump from a faulty low-pressure shutoff switch at the suction of the pump. Otherwise, operation was smooth. The first charge of activated carbon for filtering the amine system was exhausted during this run and had to be replaced.

The methanation section operated well throughout the test. It was interrupted each time the purification section had to be taken off line. Complete carbon monoxide conversion was achieved throughout the test. Approximately 1000 hours have been accumulated on the methanation section in the test program so far. There was some gradual deactivation of the catalyst in the first-stage methanator. The hydrogen plant was on standby, and the utilities operated well during the test.

Material balances for five steady-state periods at coal feed rates ranging from 2 to 3 tons/hr were established and are shown in Tables 10 through 24 and Figures 13 through 47. Overall material balances for the three major sections — gasification, purification, and methanation — were excellent. The results around the gasification section, where measurements of process streams were obtained under a difficult environment, were particularly gratifying. The balances were from 98% to 102% for the five periods.

Heat Balances From Test 37

Heat balances have been calculated for five steady-state operating periods from Test 37, for which material balance data were reported previously. The basis for these calculations is summarized in Table 10. The data are reported in Figures 34 through 43. Heat balance closures ranged from 96% to 103% indicating no abnormalities in the basic mass input-output data. The basis for the heat balance calculations is as follows:

Basis: liquid water at 77°F

Data Source:

- a. Heat of Combustion: Chemical Engineers Handbook, 5th ed , Perry & Chilton, 1973.
- b. Calorific Value of Feed Coal: experimentally determined by ASTM D271.
- c. Steam Enthalpy: 1967 ASME steam tables.
- d. Specific Heat: Physical and Thermodynamic Properties of Elements and Compounds, Chemetron Corporation, 1969.

Table 10. MATERIAL BALANCE SUMMARY FOR HYGAS GASIFIER
FOR TEST 37 FROM 6/25/75 (1206 hr) TO 6/26/75 (1996 hr)

Basis = 1 hr. All units in lb unless noted otherwise.

INPUT		C	H	O	N	S	Cl	ASH	OTHER	TOTAL
Coal Feed	Wt % (Dry)	62.30	4.59	20.65	1.01	0.82	--	10.63		100
	Coal (Dry)	2037	150	675	33	27	--	347		3269
	Moisture		68	546						614
Sparger	Oxygen			615						615
	Steam		481	3849						4330
Burner	Oxygen			--						0
	Steam		--	--						0
	Hydrogen		--							0
Stripping Ring	Steam		142	1135						1277
Nitrogen from purges					560					560
Pump seal flush			59	474						533
Water to cyclone pot			500	4001						4501
Light oil in		9341	890							10,231
TOTAL INPUT		11,378	2290	11,295	593	27		347		25,930
OUTPUT										
Reactor Overhead	Wt % (Dry)	54.00	2.03	5.51	0.63	0.35	--	37.48		100
	Dust (Dry)	281	11	29	3	2	--	194		520
Spent Char	Wt % (Dry)	45.70	1.26	5.11	0.30	0.27	--	47.36		100
	Char (Dry)	156	4	17	1	1	--	162		341
Product Gas	Total (Dry)	1004	205	1805	615	10				3639
	Components H ₂		114							114
	CO ₂	621		1657						2278
	C ₂ H ₆	14	4							18
	H ₂ S		1			10				11
	N ₂				615					615
	CH ₄	258	86							344
	CO	111		148						259
Water Out + dissolved materials *		62	1108	8859	3	3				10,035
Toluene storage tank vent gases		262	22	540	110	3				937
Stripper vent gas *		21	1	45	14					81
Light oil out †		9540	909							10,449
TOTAL OUTPUT		11,326	2260	11,295	746	19	--	356		26,002
Net (Output-Input)		-52	-30	0	153	-8	--	9		72
% Balance (Output/Input)		100	97	100	126	70	--	103		100

* Oxygen balance forced.

† Obtained by using average values from other periods because light-oil stripper was not in operation during this period.

**Table 11. MATERIAL BALANCE SUMMARY FOR PURIFICATION SECTION
FOR TEST 37 FROM 6/20/75 (1480 hr) TO 6/26/75 (1996 hr)**

Basis = 1 hr. All units in lb unless noted otherwise.

INPUT		C	H	O	N	S	TOTAL
Product Gas From Quench	Components H ₂		75				75
	CO ₂	406		1083			1489
	C ₂ H ₆	9	2				11
	H ₂ S					7	7
	N ₂				402		402
	CH ₄	168	56				224
	CO	73		97			170
TOTAL INPUT		656	133	1180	402	7	2378
OUTPUT							
Product Gas From Purification	Total (Dry)	248	127	92	390	--	857
	Components H ₂		69				69
	CO ₂ *	--		--			0
	C ₂ H ₆	23	6				29
	H ₂ S		--			--	0
	N ₂				390		390
	CH ₄	156	52				208
	CO	69		92			161
Acid Gas From Regenerator	Total (Dry)	365	6	958	10	3	1342
	Components H ₂		2				2
	CO ₂	359		958			1317
	C ₂ H ₆	1	1				2
	H ₂ S		1			3	4
	N ₂				10		10
	CH ₄	5	2				7
	CO	--		--			0
Dissolved Gases in Caustic		1		2			3
TOTAL OUTPUT		614	133	1052	400	3	2202
Net (Output - Input)		-42	0	-128	-2	-4	-176
% Balance (Output/Input)		94	100	89	100	43	93

* CO₂ removed by caustic wash.

Test # 37

Table 12. MATERIAL BALANCE SUMMARY FOR METHANATION SECTION
FOR TEST 37 FROM 6/25/75 (1206 hr) to 6/26/75 (1996 hr)

Basis = 1 hr. All units in lb unless noted otherwise.

INPUT		C	H	O	N	S	TOTAL
Purified gas to methanation	Components H ₂		69				69
	CO ₂	--		--			0
	C ₂ H ₆	23	6				29
	H ₂ S		--			--	0
	N ₂				390		390
	CH ₄	156	52				208
	CO	69		92			161
TOTAL INPUT		248	127	92	390	--	857
OUTPUT							
Condensate from Methanator *			12	92			104
Final Product Gas	Total (Dry)	271	124	--	404	--	799
	Components H ₂		34				34
	CO ₂	--		--			0
	C ₂ H ₆	--	--				0
	H ₂ S		--			--	0
	N ₂				404		404
	CH ₄	271	90				361
	CO	--		--			0
TOTAL OUTPUT		271	136	92	404	--	903
Net (Output-Input)		23	9	0	14	--	46
% Balance (Output/Input)		109	107	100	104	--	105

Heating Value Btu/SCF (N₂, Acid-gas free)

Gas to methanator	515
Final product gas	718

* Condensate calculated assuming 100% CO conversion.

Table 13. MATERIAL BALANCE SUMMARY FOR HYGAS GASIFIER FOR
TEST 37 FROM 6/27/75 (2007 hr) TO 6/29/75 (0192 hr)

Basis = 1 hr. All units in lb unless noted otherwise.

INPUT		C	H	O	N	S	Cl	ASH	OTHER	TOTAL
Coal Feed	Wt % (Dry)	61.80	4.54	20.77	0.99	0.81	--	11.09		100
	Coal (Dry)	2474	182	831	40	32	--	444		4003
	Moisture		73	584						657
Sparger	Oxygen			665						665
	Steam		480	3842						4322
Burner	Oxygen			--						0
	Steam		--	--						0
	Hydrogen		--							0
Stripping Ring	Steam		141	1132						1273
Nitrogen from purges					379					379
Pump seal flush			74	592						666
Water to cyclone pot			501	4008						4509
Light oil in		9717	925							10,642
TOTAL INPUT		12,191	2376	11,654	419	32	--	444		27,116
OUTPUT										
Reactor Overhead	Wt % (Dry)	54.30	2.20	5.30	0.63	0.37	--	37.20		100
	Dust (Dry)	209	9	20	2	1	--	143		384
Spent Char	Wt % (Dry)	45.60	0.90	4.47	0.26	0.12	--	48.65		100
	Char (Dry)	291	6	28	2	1	--	310		638
Product Gas	Total (Dry)	1265	252	2170	490	14				4191
	Components H ₂		133							133
	CO ₂	721		1921						2642
	C ₂ H ₆	11	3							14
	H ₂ S		1			14				15
	N ₂				490					490
	CH ₄	346	115							461
	CO	187		249						436
Water Out + dissolved materials		70	1194	9543	3	3				10,813
Toluene storage tank vent gases		214	13	431	29					687
Stripper vent gas		24	1	51	16					92
Light oil out		9896	943							10,839
TOTAL OUTPUT		11,969	2418	12,243	542	19	--	453		27,644
Net (Output-Input)		-222	42	589	123	-13	--	9		528
% Balance (Output/Input)		98	102	105	129	59	--	102		102

Table 14. MATERIAL BALANCE SUMMARY FOR PURIFICATION SECTION
FOR TEST 37 FROM 6/27/75 (2007 hr) TO 6/29/75 (0192 hr)

Basis = 1 hr. All units in lb unless noted otherwise.

INPUT		C	H	O	N	S	TOTAL
Product Gas From Quench	Components H ₂		74				74
	CO ₂	402		1072			1474
	C ₂ H ₆	6	2				8
	H ₂ S		1			8	9
	N ₂				274		274
	CH ₄	193	64				257
	CO	104		139			243
TOTAL INPUT		705	141	1211	274	8	2339
OUTPUT							
Product Gas From Purification	Total (Dry)	306	136	135	261	--	838
	Components H ₂		70				70
	CO ₂ *	--		--			0
	C ₂ H ₆	20	5				25
	H ₂ S		--			--	0
	N ₂				261		261
	CH ₄	184	61				245
	CO	102		135			237
Acid Gas From Regenerator	Total (Dry)	379	5	998	14	16	1412
	Components H ₂		2				2
	CO ₂	374		998			1372
	C ₂ H ₆	--	--				0
	H ₂ S		1			16	17
	N ₂				14		14
	CH ₄	5	2				7
	CO	--		--			0
Dissolved Gases in Caustic		1		2			3
TOTAL OUTPUT		686	141	1135	275	16	2253
Net (Output-Input)		-19	0	-76	1	8	-86
% Balance (Output/Input)		97	100	94	100	200	96

* CO₂ is removed by caustic wash.

Table 15. MATERIAL BALANCE FOR METHANATION SECTION
FOR TEST 37 FROM 6/27/75 (2007 hr) TO 6/29/75 (0192 hr)

Basis = 1 hr. All units in lb unless noted otherwise.

INPUT		C	H	O	N	S	TOTAL
Purified gas to methanation	Components H ₂		70				70
	CO ₂	--		--			0
	C ₂ H ₆	20	5				25
	H ₂ S		--			--	0
	N ₂				261		261
	CH ₄	184	61				245
	CO	102		135			237
TOTAL INPUT		306	136	135	261	--	838
OUTPUT							
Condensate from Methanator *			17	135			152
Final Product Gas	Total (Dry)	323	127	--	263	--	713
	Components H ₂		19				19
	CO ₂	--		--			0
	C ₂ H ₆	--	--				0
	H ₂ S		--			--	0
	N ₂				263		263
	CH ₄	323	108				431
	CO	--		--			0
TOTAL OUTPUT		323	144	135	263	--	865
Net (Output-Input)		17	8	0	2	--	27
% Balance (Output/Input)		106	106	100	101	--	103

Heating Value	Btu/SCF (N ₂ , Acid-gas free)
Gas to methanator	518
Final product gas	824

* Condensate calculated assuming 100% CO conversion.

Table 16. MATERIAL BALANCE SUMMARY FOR HYGAS GASIFIER
FOR TEST 37 FROM 6/29/75 (2001 hr) TO 6/29/75 (2399 hr)

Basis = 1 hr. All units in lb unless noted otherwise.

INPUT		C	H	O	N	S	Cl	ASH	OTHER	TOTAL
Coal Feed	Wt % (Dry)	61.70	4.58	20.40	0.96	0.75	--	11.61		100
	Coal (Dry)	3196	237	1057	50	39	--	601		5180
	Moisture		68	546						614
Sparger	Oxygen			777						777
	Steam		486	3891						4377
Burner	Oxygen			--						0
	Steam		--	--						0
	Hydrogen		--							0
Stripping Ring	Steam		143	1142						1285
Nitrogen from purges					356					356
Pump seal flush			74	592						666
Water to cyclone pot			496	3971						4467
Light oil in		10,055	958							11,013
TOTAL INPUT		13,251	2462	11,976	406	39	--	601		28,735
OUTPUT										
Reactor Overhead	Wt % (Dry)	57.00	1.97	5.46	0.67	0.37	--	34.53		100
	Dust (Dry)	164	6	16	2	1	--	99		288
Spent Char	Wt % (Dry)	54.30	0.70	3.25	0.27	0.11	--	41.37		100
	Char (Dry)	692	9	42	3	1	--	527		1274
Product Gas	Total (Dry)	1576	301	2618	383	17				4895
	Components H ₂		145							145
	CO ₂	860		2293						3153
	C ₂ H ₆	18	4							22
	H ₂ S		1			17				18
	N ₂				383					383
	CH ₄	454	151							605
	CO	244		325						569
Water Out + dissolved materials		32	1081	8621	15	2				9751
Toluene storage tank vent gases		178	15	371	--					564
Stripper vent gas		21	2	45	13					81
Light oil out		10,231	974							11,205
TOTAL OUTPUT		12,894	2388	11,713	416	21	--	626		28,058
Net (Output-Input)		-357	-74	-263	10	-18	--	25		-677
% Balance (Output/Input)		97	97	98	102	54	--	104		98

Table 17. MATERIAL BALANCE SUMMARY FOR PURIFICATION SECTION
FOR TEST 37 FROM 6/29/75 (2001 hr) TO 6/29/75 (2399 hr)

Basis = 1 hr. All units in lbs unless noted otherwise.

INPUT		C	H	O	N	S	TOTAL
Product Gas From Quench	Components H ₂		85				85
	CO ₂	503		1342			1845
	C ₂ H ₆	10	3				13
	H ₂ S		1			10	11
	N ₂				224		224
	CH ₄	265	88				353
	CO	143		190			333
TOTAL INPUT		921	177	1532	224	10	2864
OUTPUT							
Product Gas From Purification	Total (Dry)	425	173	185	235	--	1018
	Components H ₂		80				80
	CO ₂ *	--		--			0
	C ₂ H ₆	29	7				36
	H ₂ S		--			--	0
	N ₂				235		235
	CH ₄	258	86				344
	CO	138		185			323
Acid Gas From Regenerator	Total (Dry)	503	7	1316	24	3	1853
	Components H ₂		4				4
	CO ₂	494		1316			1810
	C ₂ H ₆	1	--				1
	H ₂ S		--			3	3
	N ₂				24		24
	CH ₄	8	3				11
	CO	--		--			0
Dissolved Gases in Caustic		1		3			4
TOTAL OUTPUT		929	180	1504	259	3	2875
Net (Output-Input)		8	3	-28	35	-7	11
% Balance (Output/Input)		101	102	98	116	30	100

* CO₂ removed by caustic wash.

Table 18. MATERIAL BALANCE SUMMARY FOR METHANATION SECTION
FOR TEST 37 FROM 6/29/75 (2001 hr) TO 6/29/75 (2399 hr)

Basis = 1 hr. All units in lb unless noted otherwise.

INPUT		C	H	O	N	S	TOTAL
Purified gas to methanation	Components H ₂		80				80
	CO ₂	--		--			0
	C ₂ H ₆	29	7				36
	H ₂ S		--			--	0
	N ₂				235		235
	CH ₄	258	86				344
	CO	138		185			323
TOTAL INPUT		425	173	185	235	--	1018
OUTPUT							
Condensate from Methanator *			23	185			208
Final Product Gas	Total (Dry)	440	160	--	247	--	847
	Components H ₂		13				13
	CO ₂	--		--			0
	C ₂ H ₆	--	--				0
	H ₂ S		--			--	0
	N ₂				247		247
	CH ₄	440	147				587
	CO	--		--			0
TOTAL OUTPUT		440	183	185	247	--	1055
Net (Output-Input)		15	10	0	12	--	37
% Balance (Output/Input)		104	106	100	105	--	104

Heating Value	Btu/SCF (N ₂ , Acid-gas free)
Gas to methanator	545
Final product gas	908

* Condensate calculated assuming 100% CO conversion.

Table 19. MATERIAL BALANCE SUMMARY FOR HYGAS GASIFIER
FOR TEST 37 FROM 6/30/75 (1480 hr) to 7/2/75 (0598 hr)

Basis = 1 hr. All units in lb unless noted otherwise.

INPUT		C	H	O	N	S	Cl	ASH	OTHER	TOTAL
Coal Feed	Wt % (Dry) --	61.6	4.21	20.84	0.98	0.84		11.53		100
	Coal (Dry)	2685	183	908	43	37		502		4358
	Moisture		84	675						759
Sparger	Oxygen			754						754
	Steam		489	3909						4398
Burner	Oxygen			--						0
	Steam		--	--						0
	Hydrogen		--							0
Stripping Ring	Steam		144	1152						1296
Nitrogen from purges					364					364
Pump seal flush			74	592						666
Water to cyclone pot			542	4334						4876
Light oil in		10,075	960							11,035
TOTAL INPUT		12,760	2476	12,324	407	37		502		28,506
OUTPUT										
Reactor Overhead	Wt % (Dry)	54.16	1.96	5.44	0.67	0.42	--	37.35		100
	Dust (Dry)	279	10	28	3	2	--	192		514
Spent Char	Wt % (Dry)	49.21	0.95	3.83	0.28	0.16	--	45.57		100
	Char (Dry)	312	6	24	2	1	--	288		633
Product Gas	Total (Dry)	1409	276	2386	413	15				4499
	Components H ₂		139							139
	CO ₂	792		2112						2904
	C ₂ H ₆	17	4							21
	H ₂ S		1			15				16
	N ₂				413					413
	CH ₄	395	132							527
	CO	205		274						479
Water Out + dissolved materials		41	1235	9842	19	3				11,140
Toluene storage tank vent gases		240	18	493	27					778
Stripper vent gas		45	3	88	27					163
Light oil out		10,321	983							11,304
TOTAL OUTPUT		12,647	2531	12,861	491	21	--	480		29,031
Net (Output-Input)		-113	55	537	84	-16	--	-22		525
% Balance (Output/Input)		99	102	104	120	57	--	96		102

Table 20. MATERIAL BALANCE SUMMARY FOR PURIFICATION SECTION
FOR TEST 37 FROM 7/1/75 (1300 hr) TO 7/2/75 (0600 hr)

Basis = 1 hr. All units in lb unless noted otherwise.

INPUT		C	H	O	N	S	TOTAL
Product Gas From Quench	Components H ₂		89				89
	CO ₂	512		1365			1877
	C ₂ H ₆	10	2				12
	H ₂ S		1			9	10
	N ₂				250		250
	CH ₄	259	86				345
	CO	138		184			322
TOTAL INPUT		919	178	1549	250	9	2905
OUTPUT							
Product Gas From Purification	Total (Dry)	363	158	158	226	--	905
	Components H ₂		79				79
	CO ₂ *	--		--			0
	C ₂ H ₆	26	6				32
	H ₂ S		--			--	0
	N ₂				226		226
	CH ₄	219	73				292
	CO	118		158			276
Acid Gas From Regenerator	Total (Dry)	570	7	1485	22	5	2089
	Components H ₂		3				3
	CO ₂	557		1485			2042
	C ₂ H ₆	2	--				2
	H ₂ S		--			5	5
	N ₂				22		22
	CH ₄	11	4				15
	CO	--		--			0
Dissolved Gases in Caustic		1		2			3
TOTAL OUTPUT		934	165	1645	248	5	2997
Net (Output-Input)		15	-13	96	-2	-4	92
% Balance (Output/Input)		102	93	106	99	56	103

* CO₂ removed by caustic wash.

Table 21. MATERIAL BALANCE SUMMARY FOR METHANATION SECTION
FOR TEST 37 FROM 7/1/75 (1300 hr) TO 7/2/75 (0600 hr)

Basis = 1 hr. All units in lb unless noted otherwise.

INPUT		C	H	O	N	S	TOTAL
Purified gas to methanation	Components: H ₂		79				79
	CO ₂	--		--			0
	C ₂ H ₆	26	6				32
	H ₂ S		--			--	0
	N ₂				226		226
	CH ₄	219	73				292
	CO	118		158			276
TOTAL INPUT		363	158	158	226	--	905
OUTPUT							
Condensate from Methanator*			20	158			178
Final Product Gas	Total (Dry)	373	139	--	222	--	734
	Components: H ₂		15				15
	CO ₂	--		--			0
	C ₂ H ₆	--	--				0
	H ₂ S		--			--	0
	N ₂				222		222
	CH ₄	373	124				497
	CO	--		--			--
TOTAL OUTPUT		373	159	158	222	--	912
Net (Output-Input)		10	1	0	-4	--	7
% Balance (Output/Input)		103	101	100	98	--	101

Heating Value	Btu/SCF (N ₂ , Acid-gas free)
Gas to methanator	527
Final product gas	879

* Condensate calculated assuming 100% CO conversion.

Table 22. MATERIAL BALANCE SUMMARY FOR HYGAS GASIFIER
FOR TEST 37 FROM 7/5/75 (1506 hr) TO 7/7/75 (0694 hr)

Basis = 1 hr. All units in lb unless noted otherwise.

INPUT		C	H	O	N	S	Cl	ASH	OTHER	TOTAL
Coal Feed	Wt % (Dry)	61.27	4.20	20.1	0.97	0.88		12.58		100
	Coal (Dry)	2163	148	710	34	31		444		3530
	Moisture		74	590						664
Sparger	Oxygen			696						696
	Steam		501	4005						4506
Burner	Oxygen			--						0
	Steam		--	--						0
	Hydrogen		--							0
Stripping Ring	Steam		144	1155						1299
Nitrogen from purges					388					388
Pump seal flush			89	711						800
Water to cyclone pot			593	4748						5341
Light oil in		8728	831							9559
TOTAL INPUT		10,891	2380	12,615	422	31		444		26,783
OUTPUT										
Reactor Overhead	Wt % (Dry)	49.61	2.24	5.58	0.71	0.48	--	41.38		100
	Dust (Dry)	490	22	55	7	5	--	408		987
Spent Char	Wt % (Dry)	34.49	0.76	3.26	0.19	0.17	--	61.13		100
	Char (Dry)	32	1	3	0	0	--	57		93
Product Gas	Total (Dry)	1156	229	2087	398	13				3883
	Components H ₂		132							132
	CO ₂	701		1868						2569
	C ₂ H ₆	6	1							7
	H ₂ S		1			13				14
	N ₂				398					398
	CH ₄	285	95							380
	CO	164		219						383
Water Out + dissolved materials		77	1196	9566	15	3				10,857
Toluene storage tank vent gases		194	14	403	27	6				644
Stripper vent gas		45	2	91	27					165
Light oil out		8945	852							9797
TOTAL OUTPUT		10,939	2316	12,205	474	27	--	465		26,426
Net (Output-Input)		48	-64	-410	52	-4	--	21		-357
% Balance (Output/Input)		100	97	97	112	87	--	105		99

Table 23. MATERIAL BALANCE SUMMARY FOR PURIFICATION SECTION
FOR TEST 37 FROM 7/6/75 (1405 hr) TO 7/7/75 (0694 hr)

Basis = 1 hr. All units in lb unless noted otherwise.

INPUT		C	H	O	N	S	TOTAL
Product Gas From Quench	Components H ₂		79				79
	CO ₂	411		1095			1506
	C ₂ H ₆	3	1				4
	H ₂ S		1			7	8
	N ₂				229		229
	CH ₄	170	56				226
	CO	102		135			237
TOTAL INPUT		686	137	1230	229	7	2289
OUTPUT							
Product Gas From Purification	Total (Dry)	267	127	126	218	--	738
	Components H ₂		71				71
	CO ₂ *	--		--			0
	C ₂ H ₆	15	4				19
	H ₂ S		--			--	0
	N ₂				218		218
	CH ₄	158	52				210
	CO	94		126			220
Acid Gas From Regenerator	Total (Dry)	378	4	988	12	1	1383
	Components H ₂		2				2
	CO ₂	370		988			1358
	C ₂ H ₆	1	--				1
	H ₂ S		--			1	1
	N ₂				12		12
	CH ₄	7	2				9
	CO	--		--			0
Dissolved Gases in Caustic		1		2			3
TOTAL OUTPUT		646	131	1116	230	1	2124
Net (Output-Input)		-40	-6	-114	1	-6	-165
% Balance (Output/Input)		94	96	91	100	14	93

* CO₂ removed by caustic wash.

Table 24. MATERIAL BALANCE SUMMARY FOR METHANATION SECTION
FOR TEST 37 FROM 7/6/75 (1405 hr) TO 7/7/75 (0694 hr)

Basis = 1 hr. All units in lb unless noted otherwise.

INPUT		C	H	O	N	S	TOTAL
Purified gas to methanation	Components H ₂		71				71
	CO ₂	--		--			0
	C ₂ H ₆	15	4				19
	H ₂ S		--			--	0
	N ₂				218		218
	CH ₄	158	52				210
	CO	94		126			220
TOTAL INPUT		267	127	126	218	--	738
OUTPUT							
Condensate from Methanator *			16	126			142
Final Product Gas	Total (Dry)	304	125	--	228	--	657
	Components H ₂		24				24
	CO ₂	--		--			0
	C ₂ H ₆	--	--				0
	H ₂ S		--			--	0
	N ₂				228		228
	CH ₄	304	101				405
	CO	--		--			0
TOTAL OUTPUT		304	141	126	228	--	799
Net (Output-Input)		37	14	0	10	--	61
% Balance (Output/Input)		114	111	100	105	--	108

Heating Value	Btu/SCF (N ₂ , Acid-gas free)
Gas to methanator	497
Final product gas	790

* Condensate calculated assuming 100% CO conversion.

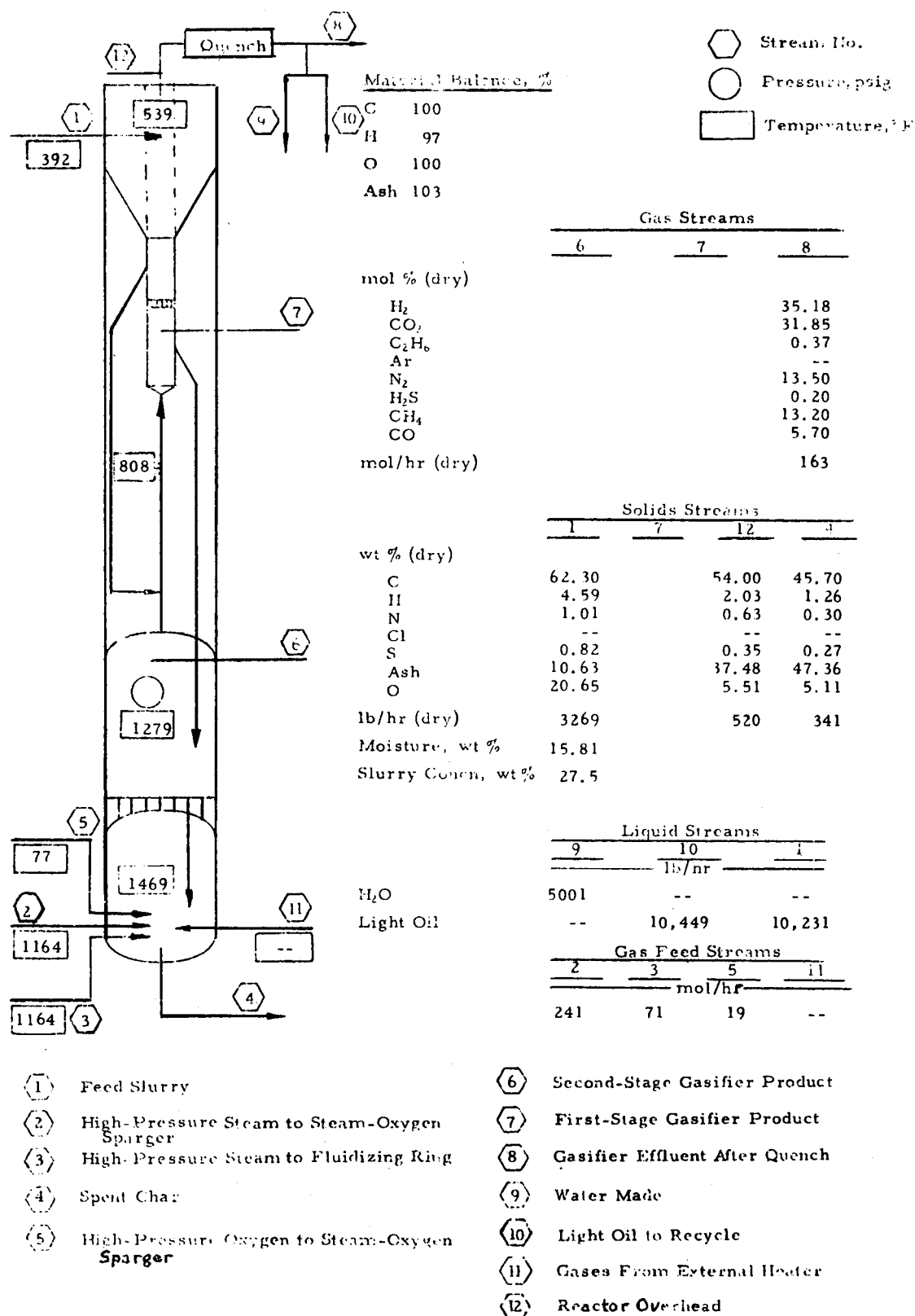


Figure 13. HYGAS REACTOR DATA FOR TEST 37 FROM 6/25/75 (1206 hr) TO 6/26/75 (1996 hr)

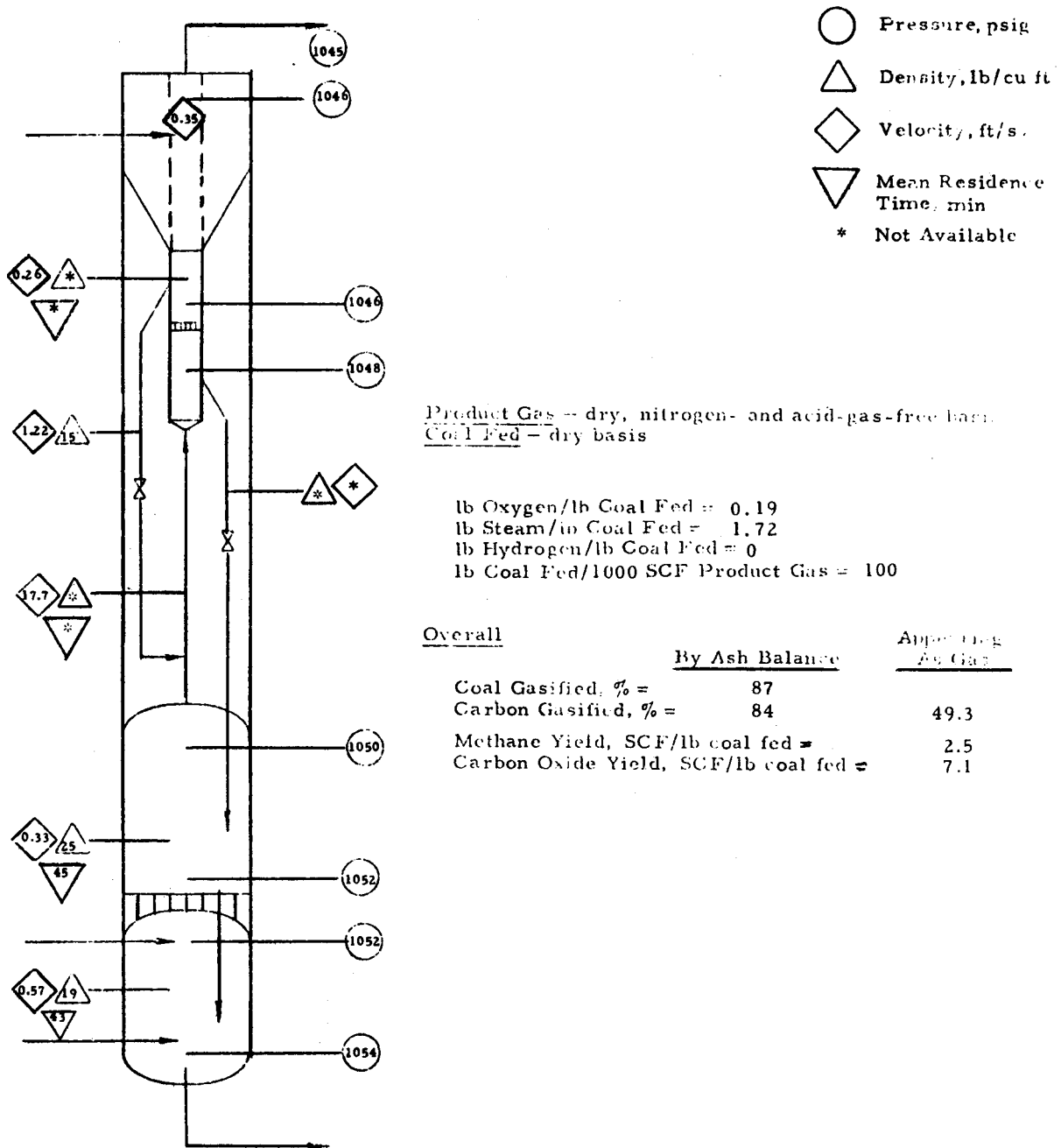
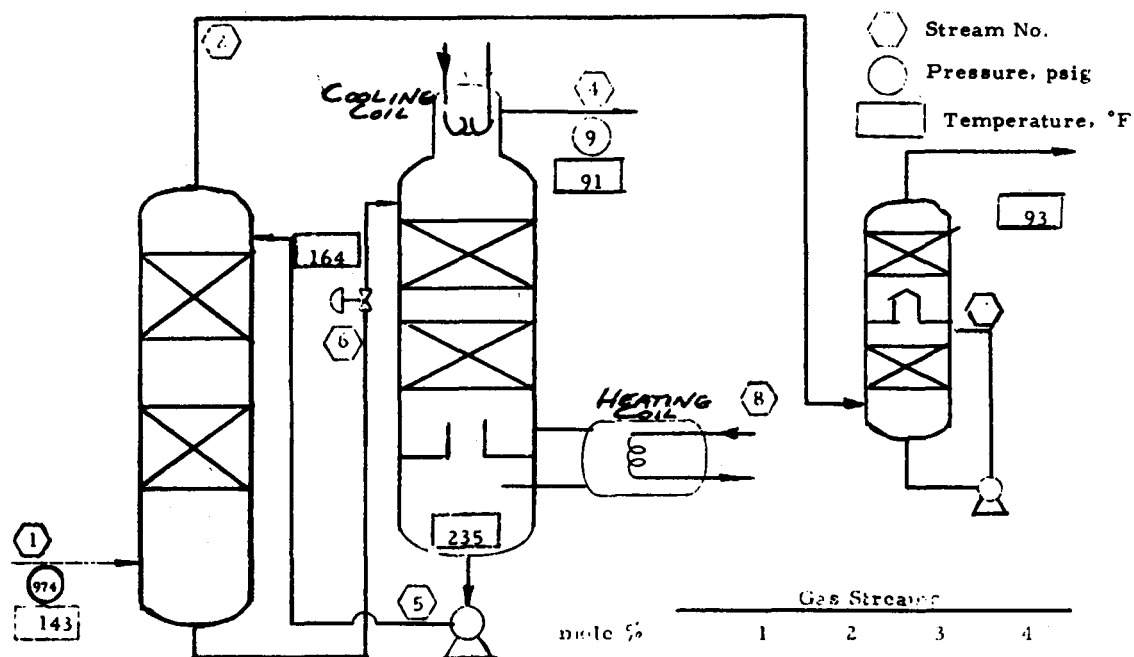


Figure 14. HYGAS REACTOR ENGINEERING DATA FOR TEST 37
FROM 6/25/75 (1206 hr) TO 6/26/75 (1996 hr)



Material Balance, %

C	94
H	100
O	89

Liquid Streams

	5	6	7	8
% Amine	39.5	39.5	--	--
% Caustic	--	--	7.0	--
lb/hr	49,085	50,194	5593	3567

Liquid Streams

	5	6
Acid gas loading of DGA, lb mole/gal	--	--

mole %

	1	2	3	4
H ₂	35.18	50.45	55	2.70
CO ₂	31.85	0.08	0	94.40
C ₂ H ₆	0.37	1.43		0.10
Ar	--	--		--
N ₂	13.50	20.44		1.14
H ₂ S	0.20	0		0.31
CH ₄	13.20	19.12		1.35
CO	5.70	8.48	8	0
mol/hr	106	68		32

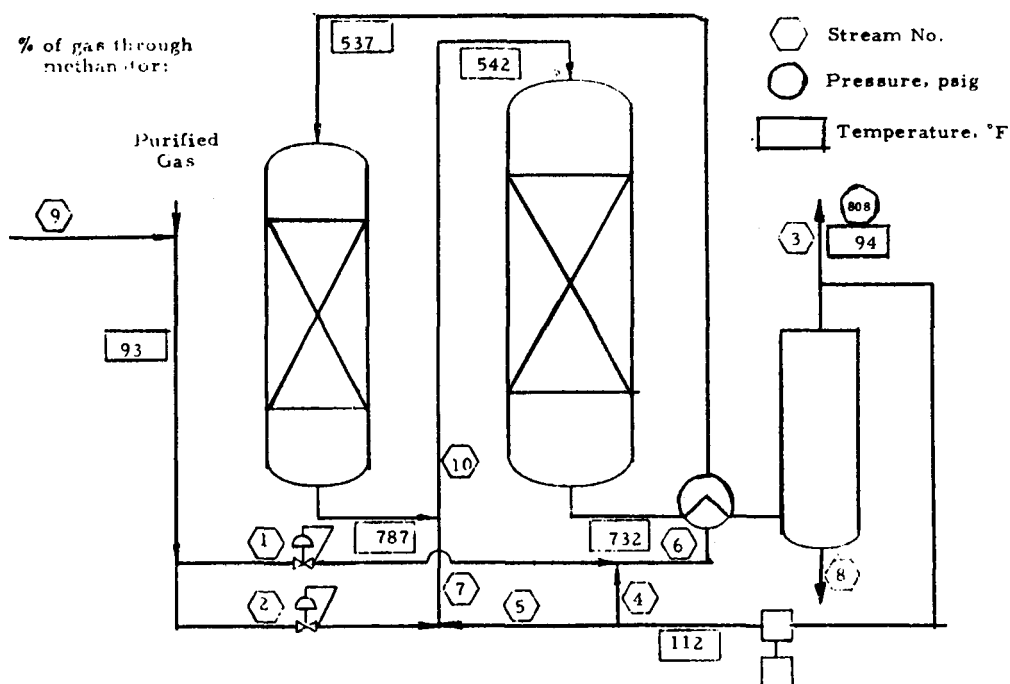
Sulfur Analysis (ppm)

CH ₃ SH	0.1013	0.0058
CH ₃ SCH ₃ -CH ₃ CH ₂ SH	0.0031	0.0009
CS ₂	0	0
(CH ₃) ₂ CHSH	0	0
CH ₃ SCH ₂ CH ₃	0	0
H ₂ S	0.2441	0.0011
COS	0.0047	0.0492
CH ₃ SSCH ₃	0	0

- ① Gas to Purification
- ② Scrubbed Gas
- ③ Purified Gas
- ④ Regenerator Effluent

- ⑤ Lean DGA
- ⑥ Rich DGA
- ⑦ Caustic Wash
- ⑧ 40 lb Steam

Figure 15. GAS PURIFICATION DATA FOR TEST 37
FROM 6/25/75 (1206 hr) TO 6/26/75 (1996 hr)



Material Balance, %

C	109
H	107
O	100

	Gas Streams									
mol %	1	2	3	4	5	6	7	8	9	10
H ₂	50.5	50.5	31.1	31.1	31.1	37.3	42.6			35.83
CO ₂	0.1	0.1	0	0	0	0	0			0
C ₂ H ₆	1.4	1.4	0	0	0	0.5	0.8			0.23
Ar	--	--	--	--	--	--	--			--
N ₂	20.4	20.4	26.8	26.8	26.8	24.8	23.0			25.31
H ₂ S	0	0	0	0	0	0	0			0
CH ₄	19.1	19.1	42.1	42.1	42.1	34.7	28.6			37.23
CO	8.5	8.5	0	0	0	2.7	5.0			1.40
mol/hr	40	27	53	85	19	125	46			164
H.V. (N ₂ , acid gas free)	515	515	718	718	718	649	594			667

	First Stage	Second Stage	Overall
Recycle Ratio	2.13	0.70	1.55
Space Velocity Based on Total Bed, hr ⁻¹	3018	1979	--
Pressure Drop (Inches H ₂ O)	3.79	13.99	17.78
% Bed Utilized (From Temp Profile)	62.50	12.50	29.17
Space Velocity Based on Bed Utilized, hr ⁻¹	4829	15,832	--

① Purified Gas to First Stage	⑥ First-Stage Feed Gas
② Purified Gas to Second Stage	⑦ Recycle and Fresh Feed to Second Stage
③ Product Gas	⑧ Condensate
④ Recycle Gas	⑨ Makeup Hydrogen
⑤ Recycle Gas	⑩ Second-Stage Feed Gas

Figure 16. METHANATION DATA FOR TEST 37
FROM 6/25/75 (1206 hr) TO 6/26/75 (1996 hr)

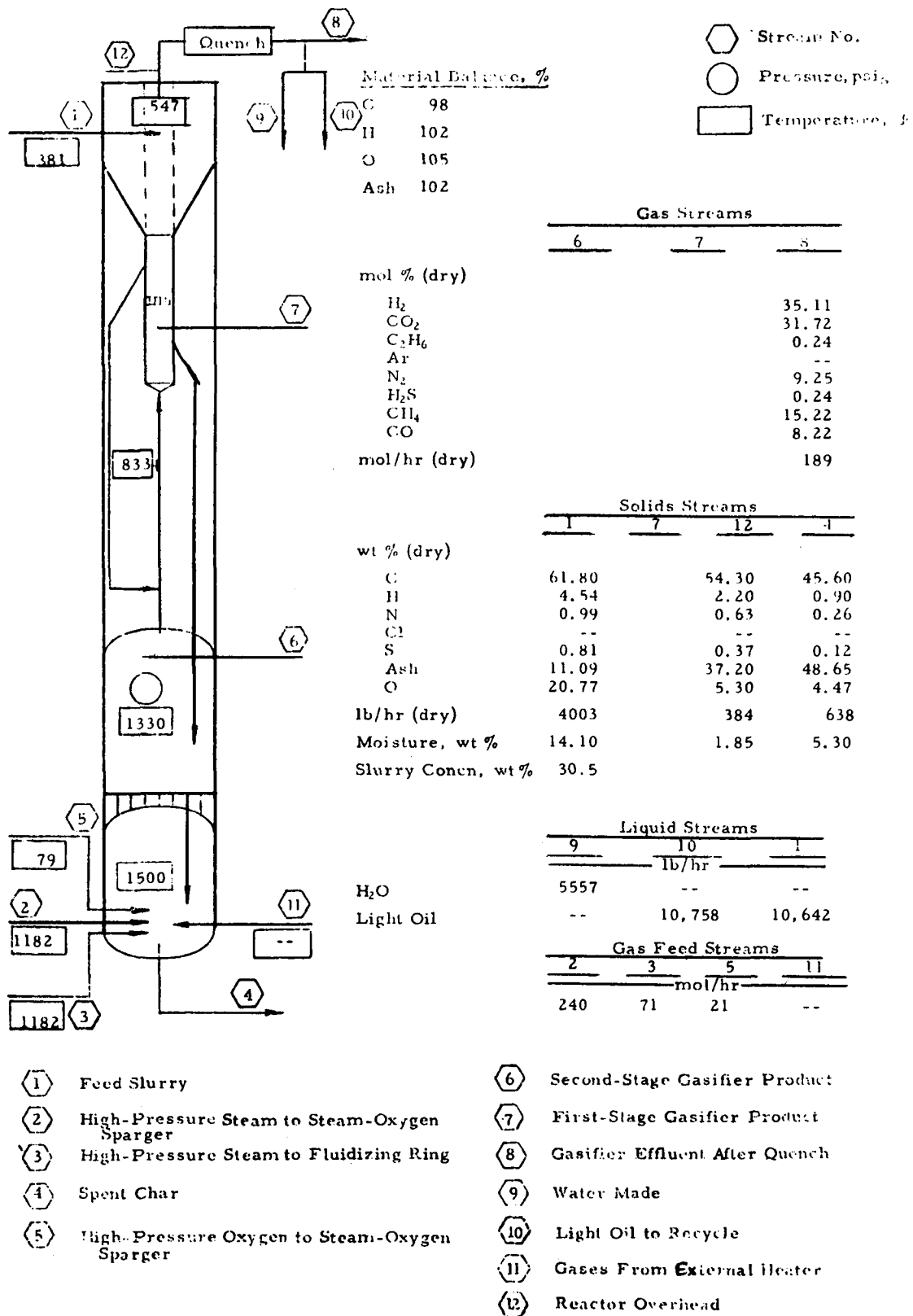


Figure 17. HYGAS REACTOR DATA FOR TEST 37
FROM 6/27/75 (2007 hr) TO 6/29/75 (0192 hr)

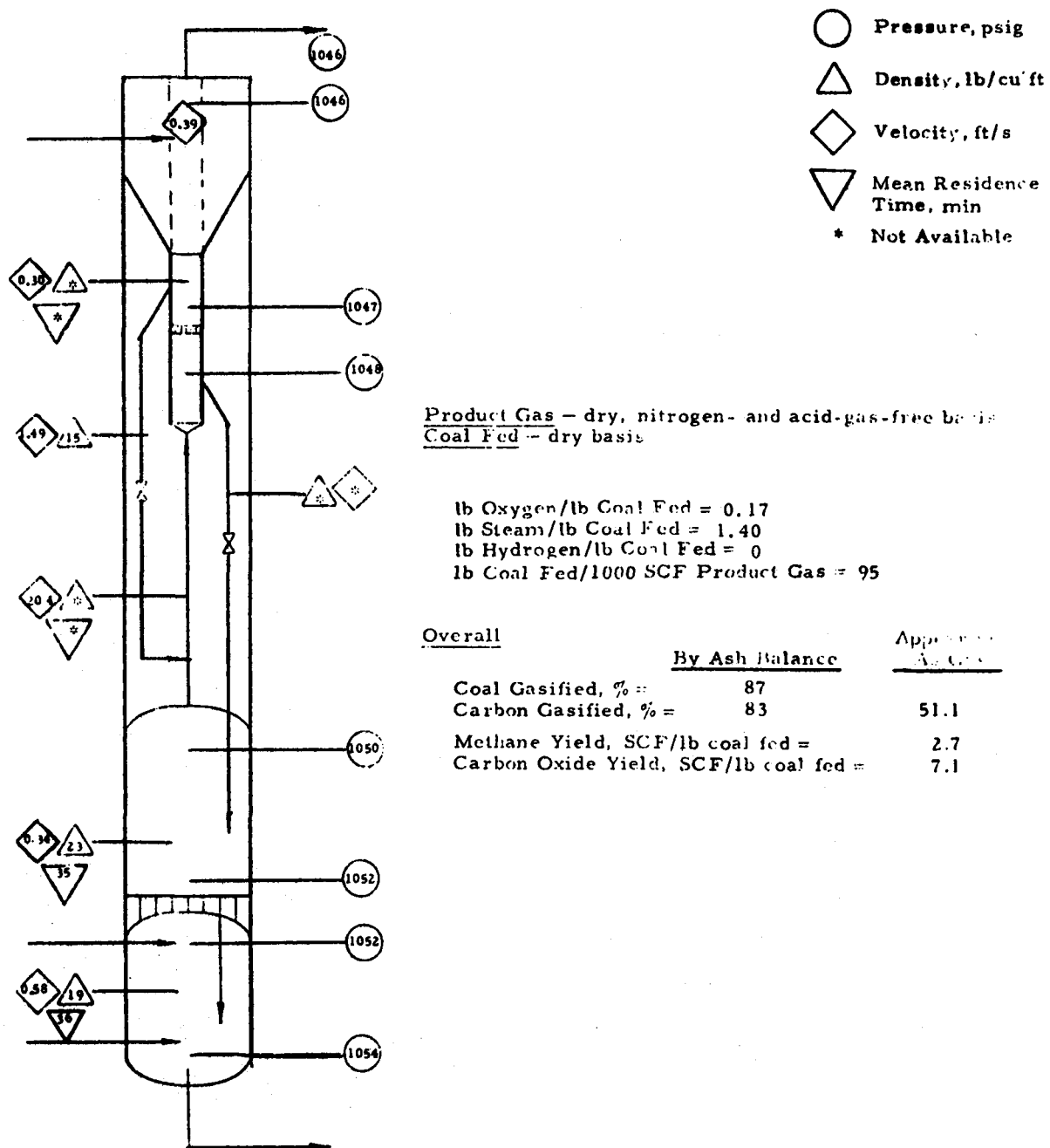


Figure 18. HYGAS REACTOR ENGINEERING DATA FOR TEST 37
FROM 6/27/75 (2007 hr) TO 6/29/75 (0192 hr)

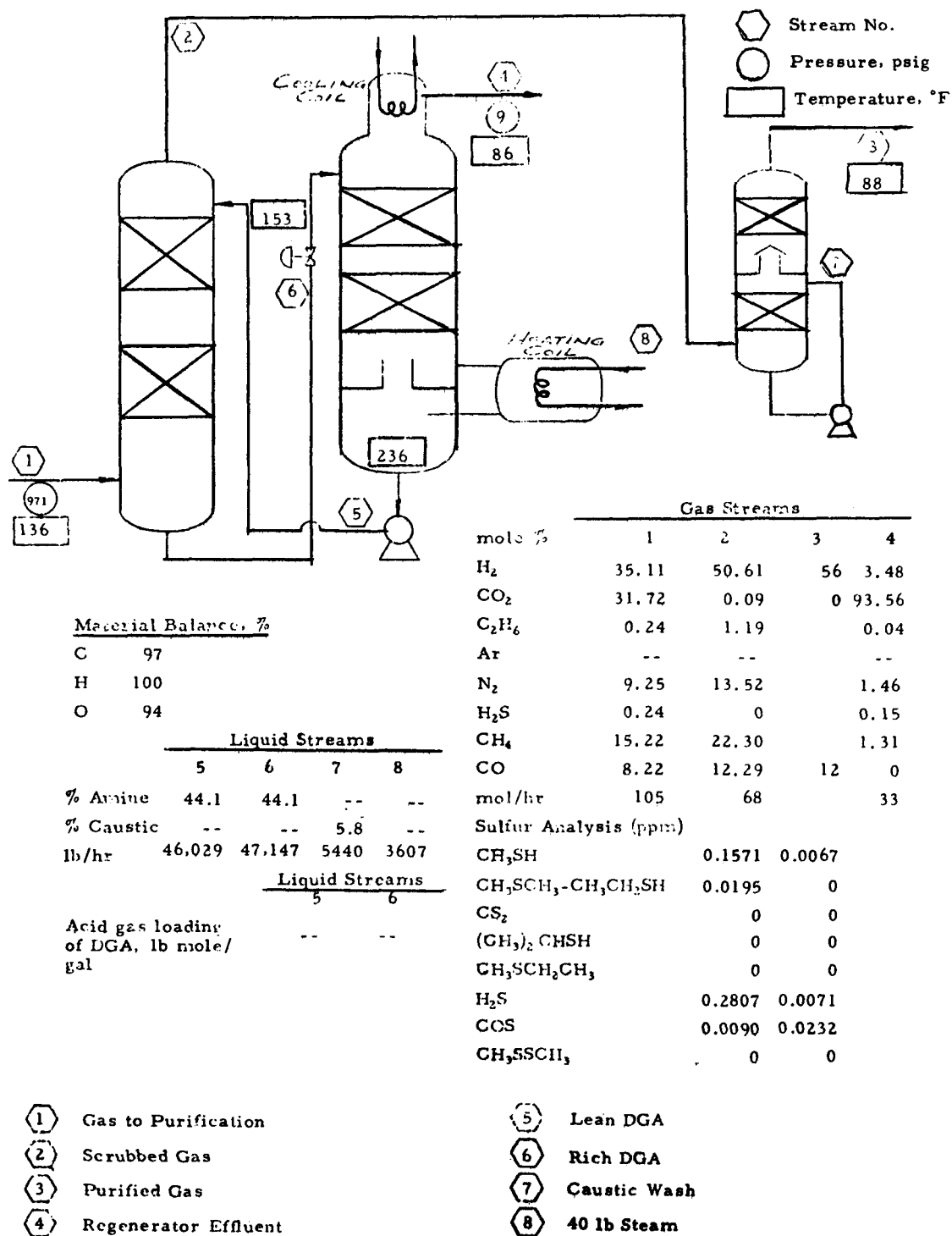
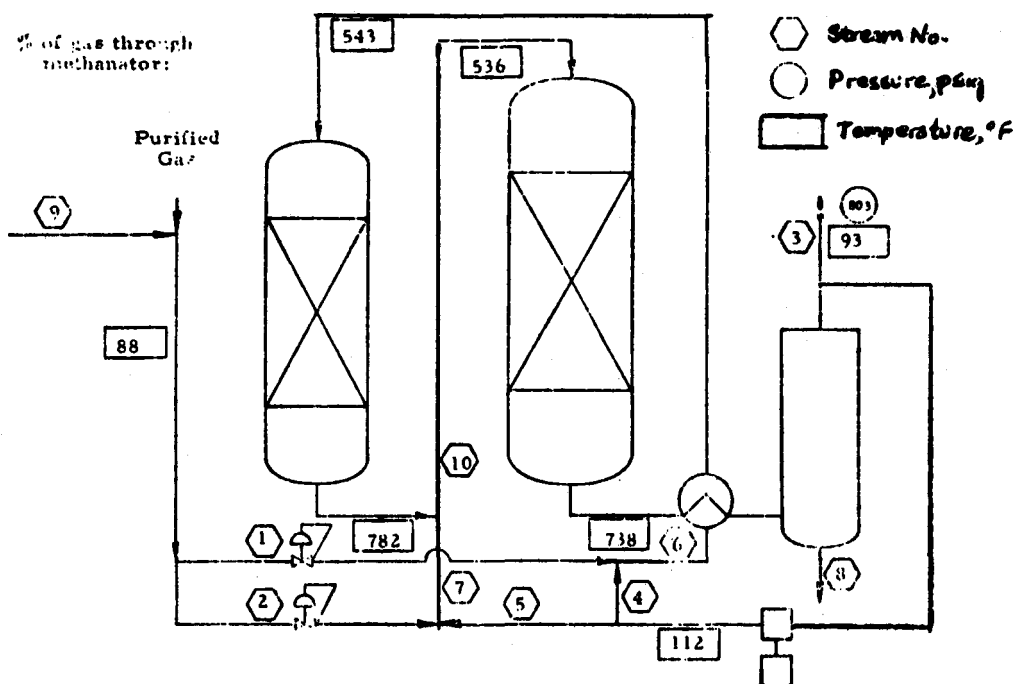


Figure 19. GAS PURIFICATION DATA FOR TEST 37
FROM 6/27/75 (2007 hr) TO 6/29/75 (0192 hr)

**Material Balance, %**

C 106
H 106
O 100

	Gas Streams									
mol %	1	2	3	4	5	6	7	8	9	10
H ₂	50.7	50.7	21.3	21.3	21.3	28.1	34.5			24.9
CO ₂	0	0	0	0	0	0	0			0
C ₂ H ₆	1.2	1.2	0	0	0	0.3	0.5			0.2
Ar	--	--	--	--	--	--	--			--
N ₂	13.6	13.6	20.4	20.4	20.4	18.7	17.3			19.7
H ₂ S	0	0	0	0	0	0	0			0
CH ₄	22.2	22.2	58.3	58.3	58.3	50.0	42.1			53.6
CO	12.3	12.3	0	0	0	2.9	5.6			1.6
mol/hr	39	29	46	131	35	170	63			219
H.V. (N ₂ , acid gas free)	518	518	824	824	824	751	682			788

	First Stage	Second Stage	Overall
Recycle Ratio	3.3	1.2	2.4
Space Velocity Based on Total Bed, hr ⁻¹	4110	1940	--
Pressure Drop (Inches H ₂ O)	5.7	54.4	60.1
% Bed Utilized (From Temp Profile)	66.7	16.7	33.3
Space Velocity Based on Bed Utilized, hr ⁻¹	6165	11,638	--

① Purified Gas to First Stage	⑥ First-Stage Feed Gas
② Purified Gas to Second Stage	⑦ Recycle and Fresh Feed to Second Stage
③ Product Gas	⑧ Condensate
④ Recycle Gas	⑨ Makeup Hydrogen
⑤ Recycle Gas	⑩ Second-Stage Feed Gas

**Figure 20. METHANATION DATA FOR TEST 37
FROM 6/27/75 (2007 hr) TO 6/29/75 (0192 hr)**

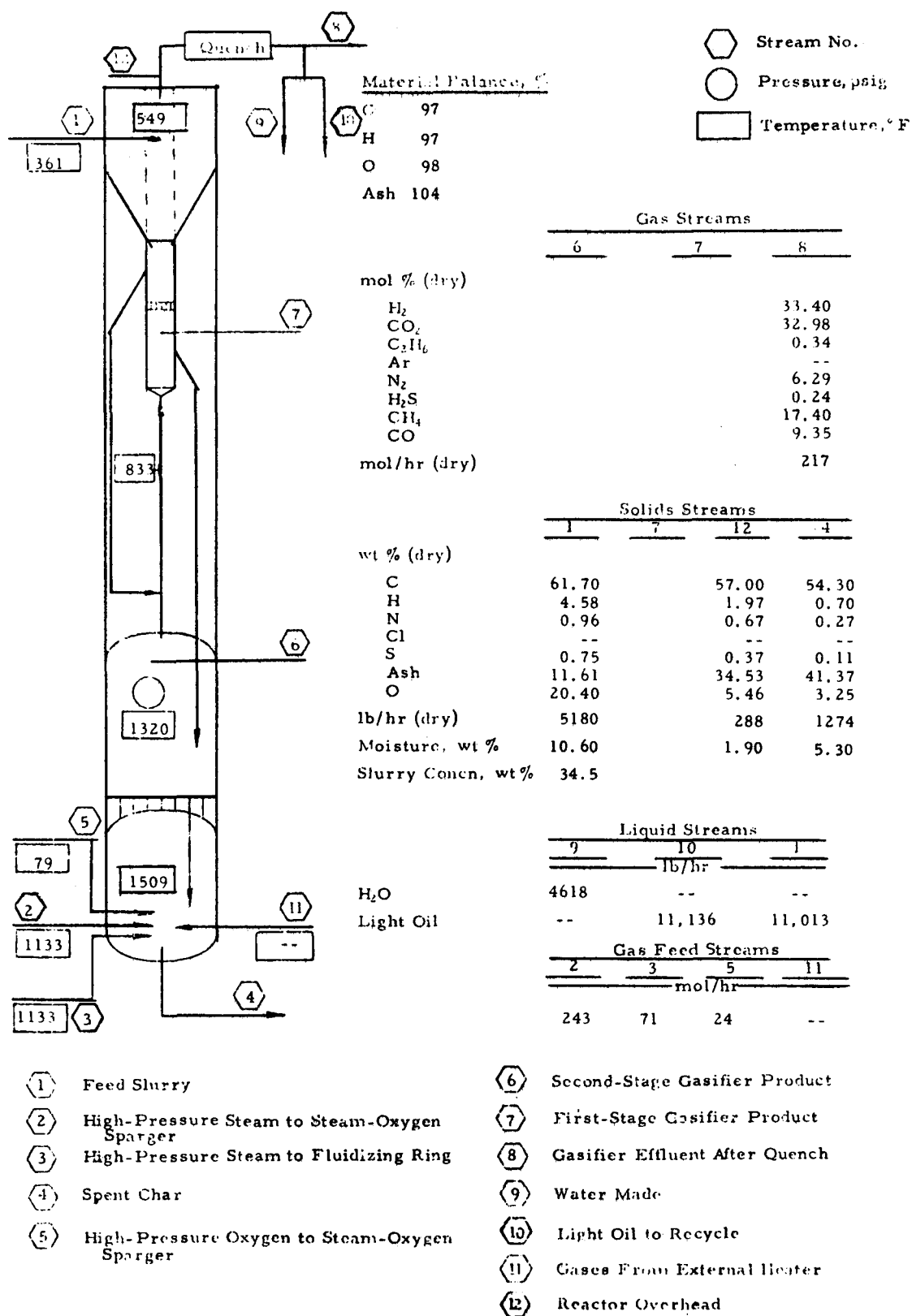


Figure 21. HYGAS REACTOR DATA FOR TEST 37
 FROM 6/29/75 (2001 hr) TO 6/29/75 (2399 hr)

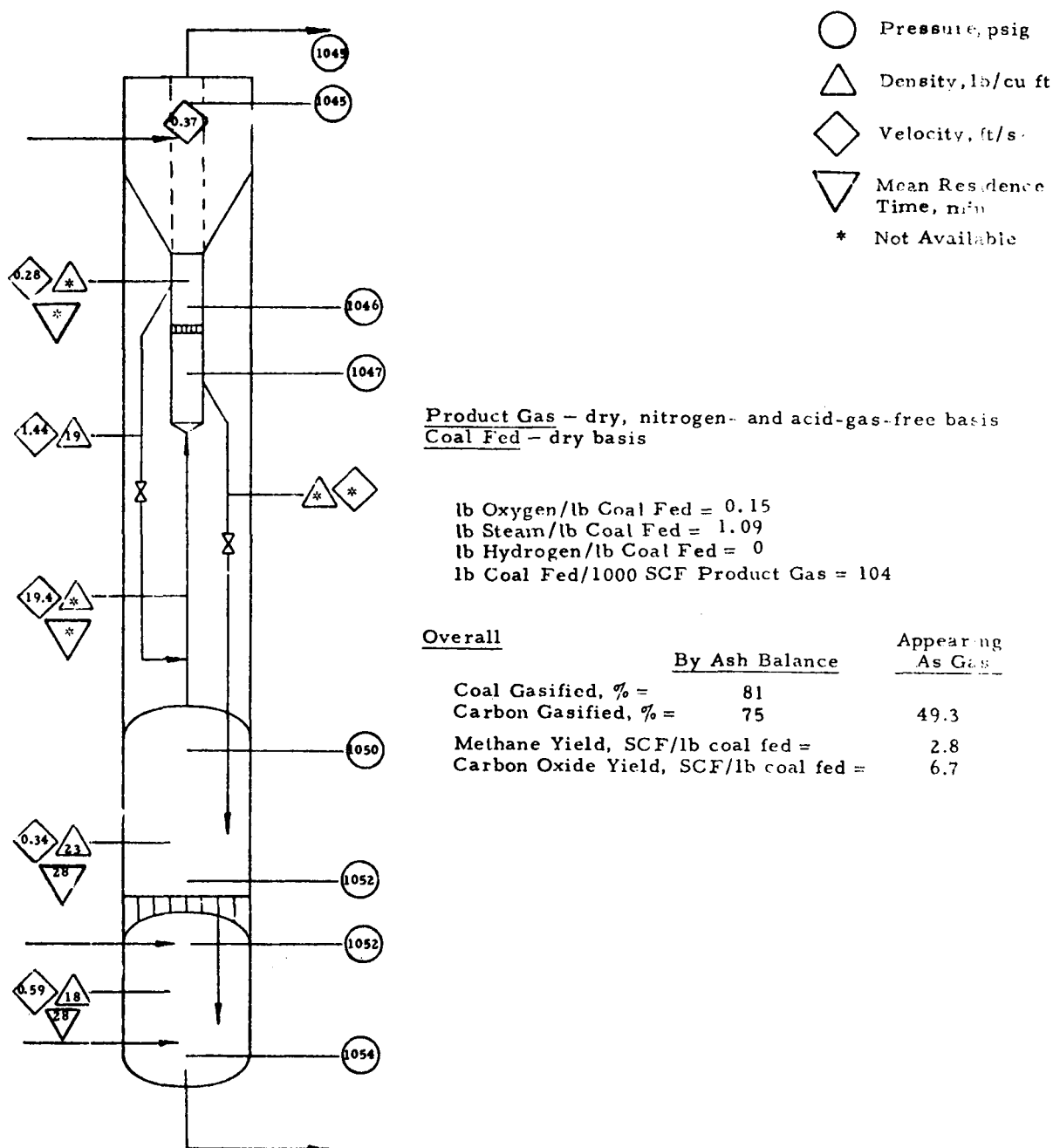
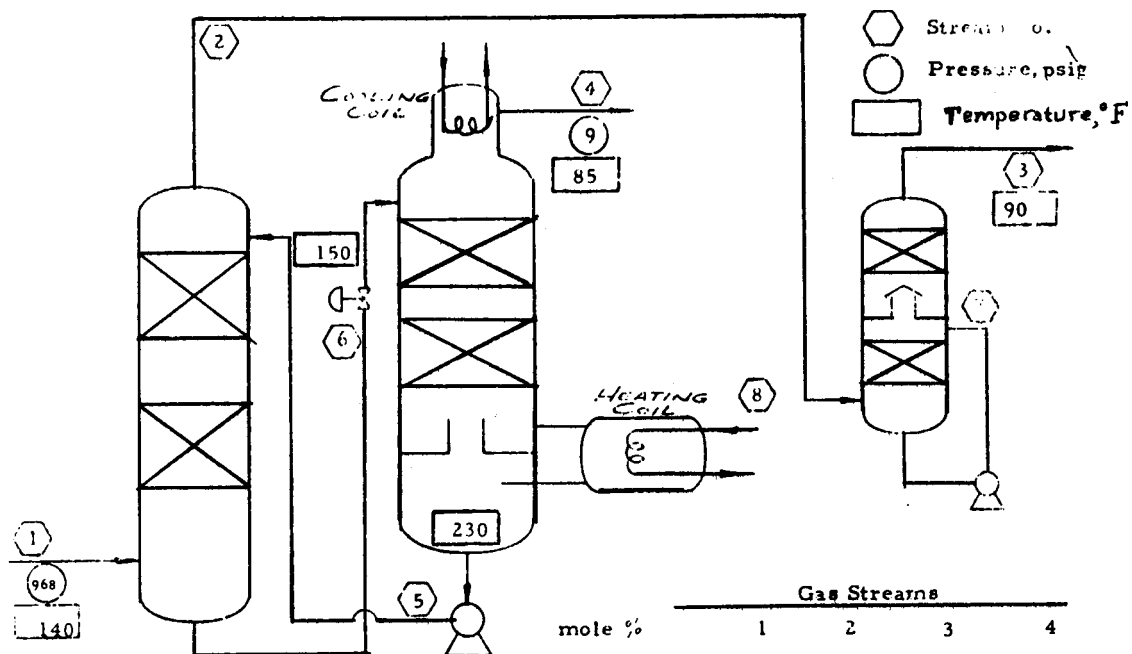


Figure 22. HYGAS REACTOR ENGINEERING DATA FOR TEST 37
FROM 6/29/75 (2001 hr) TO 6/29/75 (2399 hr)



Material Balance, %

C	101
H	102
O	98

Liquid Streams

	5	6	7	8
% Amine	43.0	43.0	--	--
% Caustic	--	--	3.6	--
lb/hr	53,965	55,575	5522	3632

Liquid Streams

	5	6
Acid gas loading of DGA, lb mole/gal	--	--

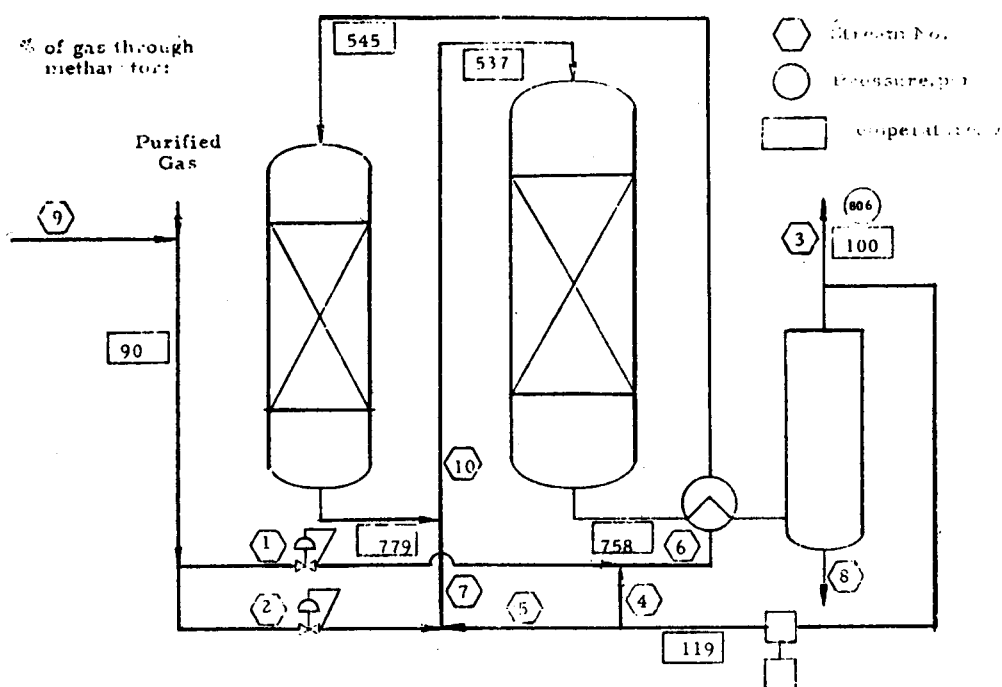
Gas Streams

mole %	1	2	3	4
H ₂	33.40	48.44	54	4.53
CO ₂	32.98	0.11	0	91.90
C ₂ H ₆	0.34	1.46		0.05
Ar	--	--		--
N ₂	6.29	10.12		1.91
H ₂ S	0.24	0		0.20
CH ₄	17.40	25.96		1.41
CO	9.35	13.91	14	0
mol/hr	126	82		45
Sulfur Analysis (ppm)				
CH ₃ SH		0.0063	0.0025	
CH ₃ SCH ₃ -CH ₃ CH ₂ SH		0	0	
CS ₂		0	0	
(CH ₃) ₂ C=SH		0	0	
CH ₃ SCH ₂ CH ₃		0	0	
H ₂ S		0.5685	0	
COS		0.0020	0.0222	
CH ₃ SSCH ₃		0	0	

- ① Gas to Purification
- ② Scrubbed Gas
- ③ Purified Gas
- ④ Regenerator Effluent

- ⑤ Lean DGA
- ⑥ Rich DGA
- ⑦ Caustic Wash
- ⑧ 40 lb Steam

Figure 23. GAS PURIFICATION DATA FOR TEST 37
FROM 6/29/75 (2001 hr) TO 6/29/75 (2399 hr)

**Material Balance, %**

C 104
H 106
O 100

	Gas Streams									
mol %	1	2	3	4	5	6	7	8	9	10
H ₂	48.4	48.4	12.3	12.3	12.3	19.8	30.0			17.0
CO ₂	0	0	0	0	0	0	0			0
C ₂ H ₆	1.5	1.5	0	0	0	0.3	0.7			0.2
Ar	--	--	--	--	--	--	--			--
N ₂	10.1	10.1	17.0	17.0	17.0	15.6	13.7			16.1
H ₂ S	0	0	0	0	0	0	0			0
CH ₄	26.1	26.1	70.7	70.7	70.7	61.4	48.9			64.8
CO	13.9	13.9	0	0	0	2.9	6.8			1.9
mol/hr	43	38	52	167	39	211	77			270
H.V. (N ₂ , acid gas free)	545	545	908	908	908	828	724			851

	First Stage	Second Stage	Overall
Recycle Ratio	3.9	1.0	2.5
Space Velocity Based on Total Bed, hr ⁻¹	5094	3248	--
Pressure Drop (Inches H ₂ O)	7.8	79.7	87.5
% Bed Utilized (From Temp Profile)	62.5	14.6	30.6
Space Velocity Based on Bed Utilized, hr ⁻¹	8150	22,250	--

- ① Purified Gas to First Stage
② Purified Gas to Second Stage
③ Product Gas
④ Recycle Gas
⑤ Recycle Gas

- ⑥ First-Stage Feed Gas
⑦ Recycle and Fresh Feed to Second Stage
⑧ Condensate
⑨ Makeup Hydrogen
⑩ Second-Stage Feed Gas

Figure 24. METHANATION DATA FOR TEST 37
FROM 6/29/75 (2001 hr) TO 6/29/75 (2399 hr)

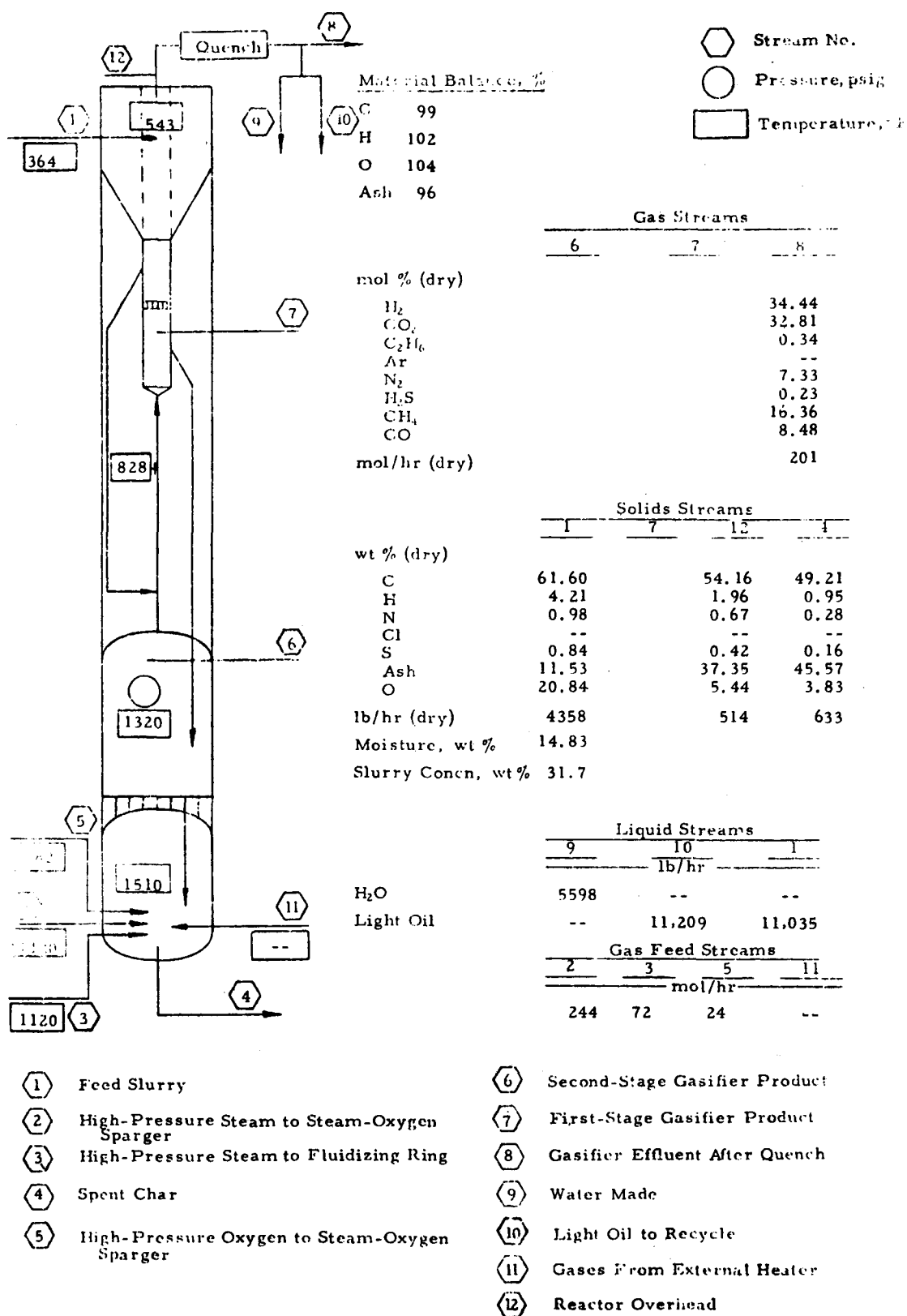


Figure 25. HYGAS REACTOR ENGINEERING DATA FOR TEST 37
FROM 6/30/75 (1480 hr) TO 7/2/75 (0598 hr)

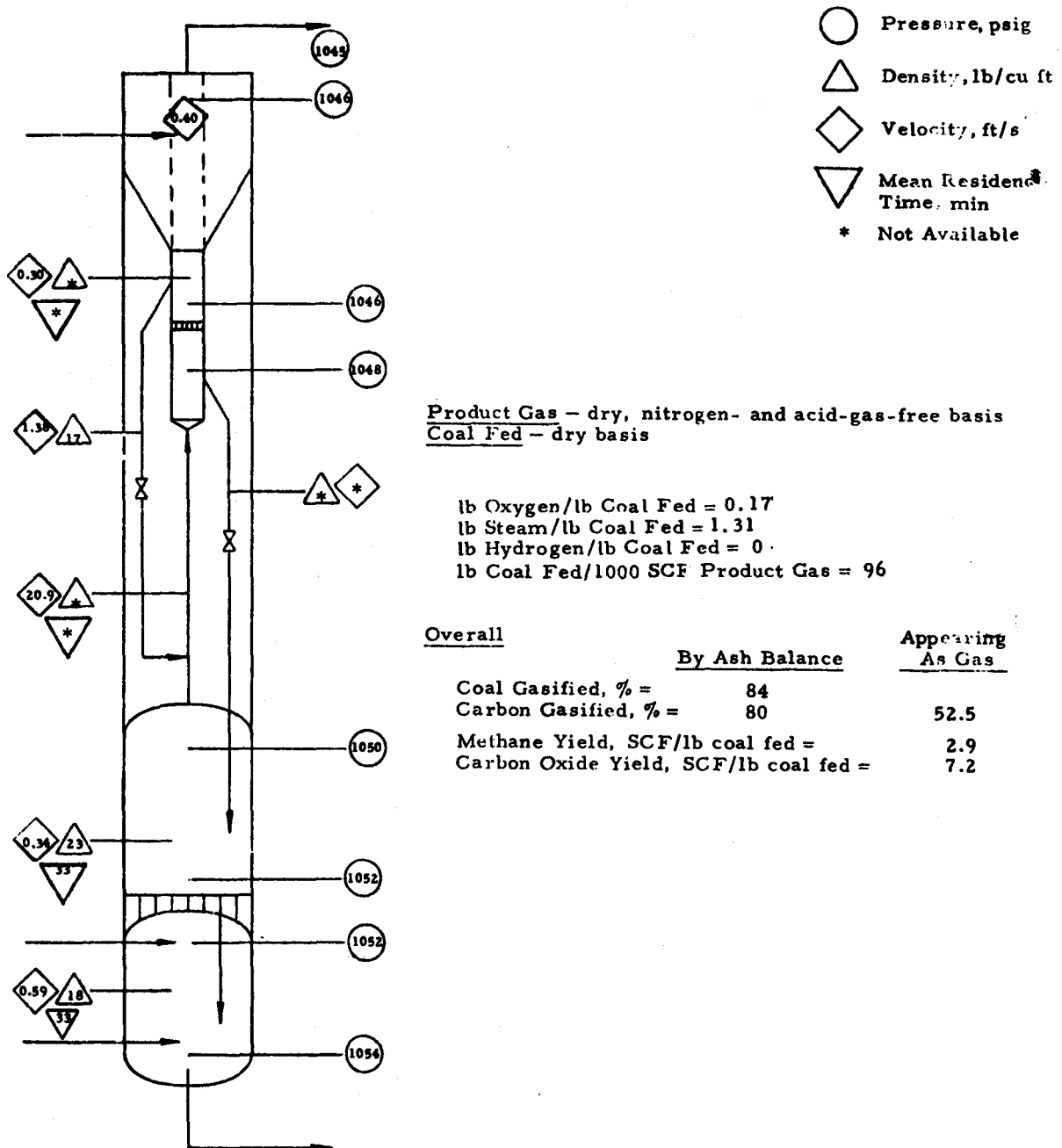
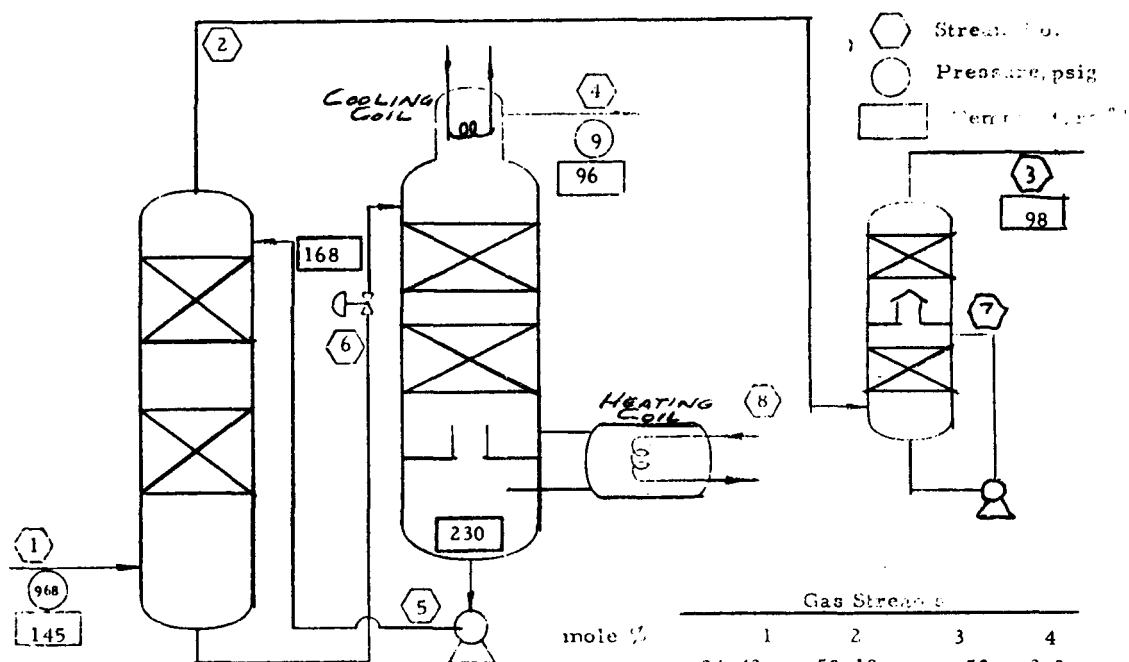


Figure 26. HYGAS REACTOR ENGINEERING DATA FOR TEST 37
FROM 6/30/75 (1480 hr) TO 7/2/75 (0598 hr)



Material Balance, %

C	102
H	93
O	106

Liquid Streams

	5	6	7	8
% Amine	43.6	43.6	--	--
% Caustic	--	--	9.6	--
lb/hr	50,640	52,487	6014	3131
Liquid Streams				
	5	6	7	8
Acid gas loading of DGA, lb mole/ gal	--	--	--	--

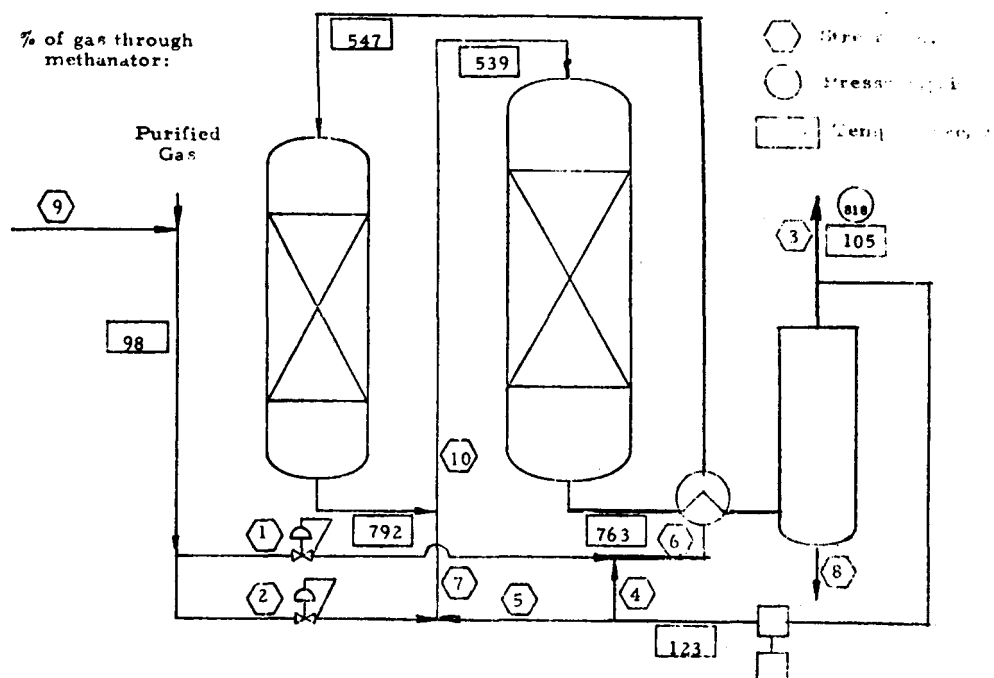
Gas Streams

mole %	1	2	3	4
H ₂	34.43	50.18	50	3.3
CO ₂	32.81	0.09	0	92.1
C ₂ H ₆	0.31	1.41		0.9
Ar	--	--		--
N ₂	6.86	10.78		1.5
H ₂ S	0.23	0		0.3
CH ₄	16.52	24.43		1.9
CO	8.83	13.11	13	0
mol/hr	130	75		50
Sulfur Analysis (ppm)				
CH ₃ SH		0.0357	0.0019	
CH ₃ SCCH ₃ -CH ₃ CH ₂ SH		0.0264	0	
CS ₂		0	0	
(CH ₃) ₂ CSH		0	0	
CH ₃ SCCH ₂ CH ₃		0	0	
H ₂ S		0.5254	0.0029	
COS		0.0157	0.0450	
CH ₃ SSCH ₃		0	0	

- ① Gas to Purification
- ② Scrubbed Gas
- ③ Purified Gas
- ④ Regenerator Effluent

- ⑤ Lean DGA
- ⑥ Rich DGA
- ⑦ Caustic Wash
- ⑧ 40 lb Steam

Figure 27. GAS PURIFICATION DATA FOR TEST 37
FROM 7/1/75 (1300 hr) TO 7/2/75 (0600 hr)



Material Balance, %

C 103
H 101
O 100

	Gas Streams									
mol %	1	2	3	4	5	6	7	8	9	10
H ₂	51.6	51.6	15.5	15.5	15.5	23.9	33.8		98.4	21.4
CO ₂	0	0	0	0	0	0	0		0	0
C ₂ H ₆	1.4	1.4	0	0	0	0.3	0.7		0	0.2
Ar	--	--	--	--	--	--	--		--	--
N ₂	10.5	10.5	17.1	17.1	17.1	15.6	13.8		0.9	16.1
H ₂ S	0	0	0	0	0	0	0		0	0
CH ₄	23.7	23.7	67.4	67.4	67.4	57.2	45.3		0	60.3
CO	12.8	12.8	0	0	0	3.0	6.4		0.7	2.0
mol/hr	40	35	46	133	34	174	69		2	227
H.V. (N ₂ , acid gas free)	527	527	879	879	879	790	693		322	818

	First Stage	Second Stage	Overall
Recycle Ratio	3.3	1.0	2.2
Space Velocity Based on Total Bed, hr ⁻¹	4200	2800	--
Pressure Drop (Inches H ₂ O)	4.1	30.2	34.3
% Bed Utilized (From Temp Profile)	62.5	12.5	29.2
Space Velocity Based on Bed Utilized, hr ⁻¹	6720	22,400	--

① Purified Gas to First Stage	⑥ First-Stage Feed Gas
② Purified Gas to Second Stage	⑦ Recycle and Fresh Feed to Second Stage
③ Product Gas	⑧ Condensate
④ Recycle Gas	⑨ Makeup Hydrogen
⑤ Recycle Gas	⑩ Second-Stage Feed Gas

Figure 28. METHANATION DATA FOR TEST 37
FROM 7/1/75 (1300 hr) TO 7/2/75 (0600 hr)

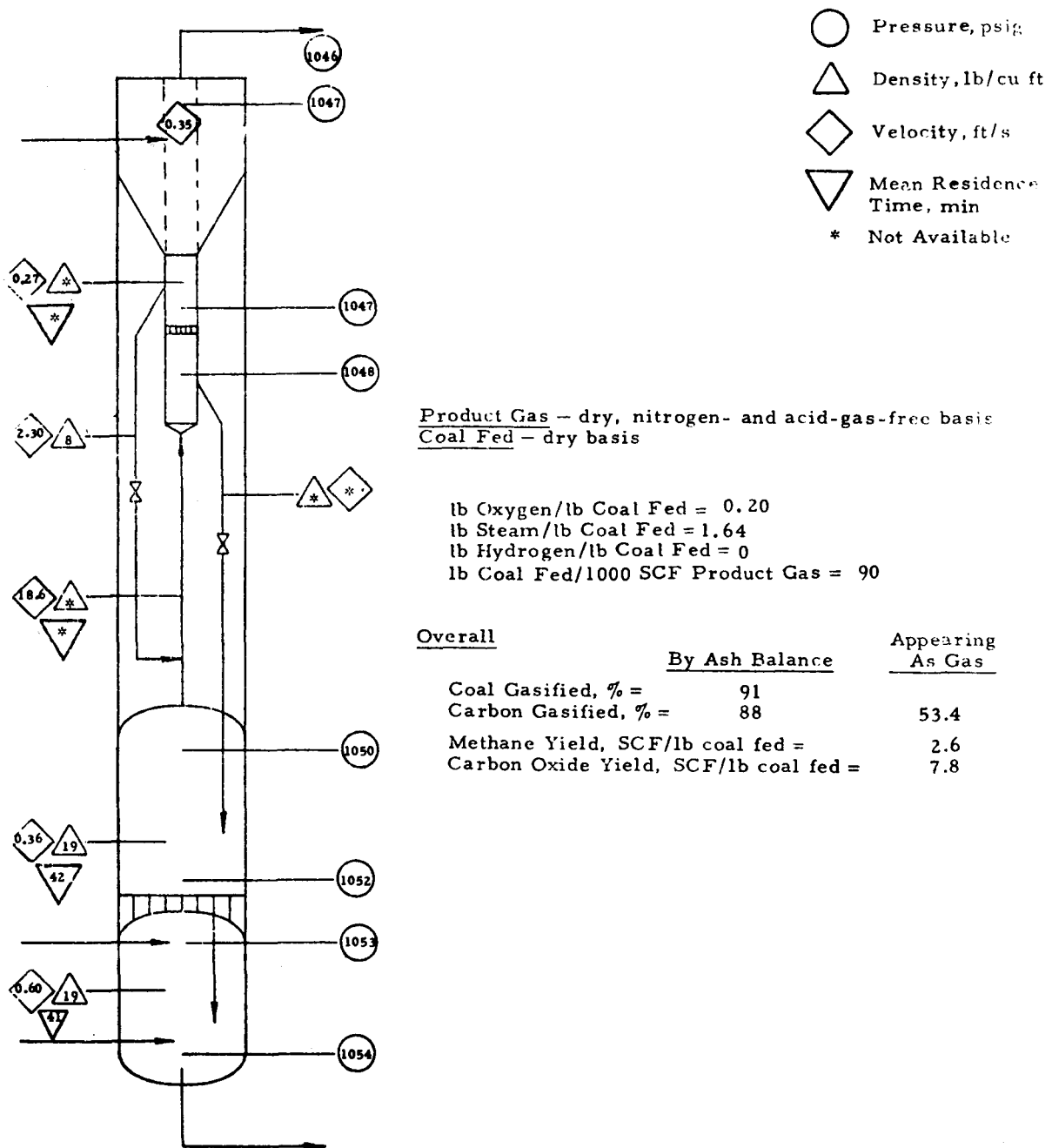


Figure 30. HYGAS REACTOR ENGINEERING DATA FOR TEST 37
FROM 7/5/75 (1506 hr) TO 7/7/75 (0694 hr)

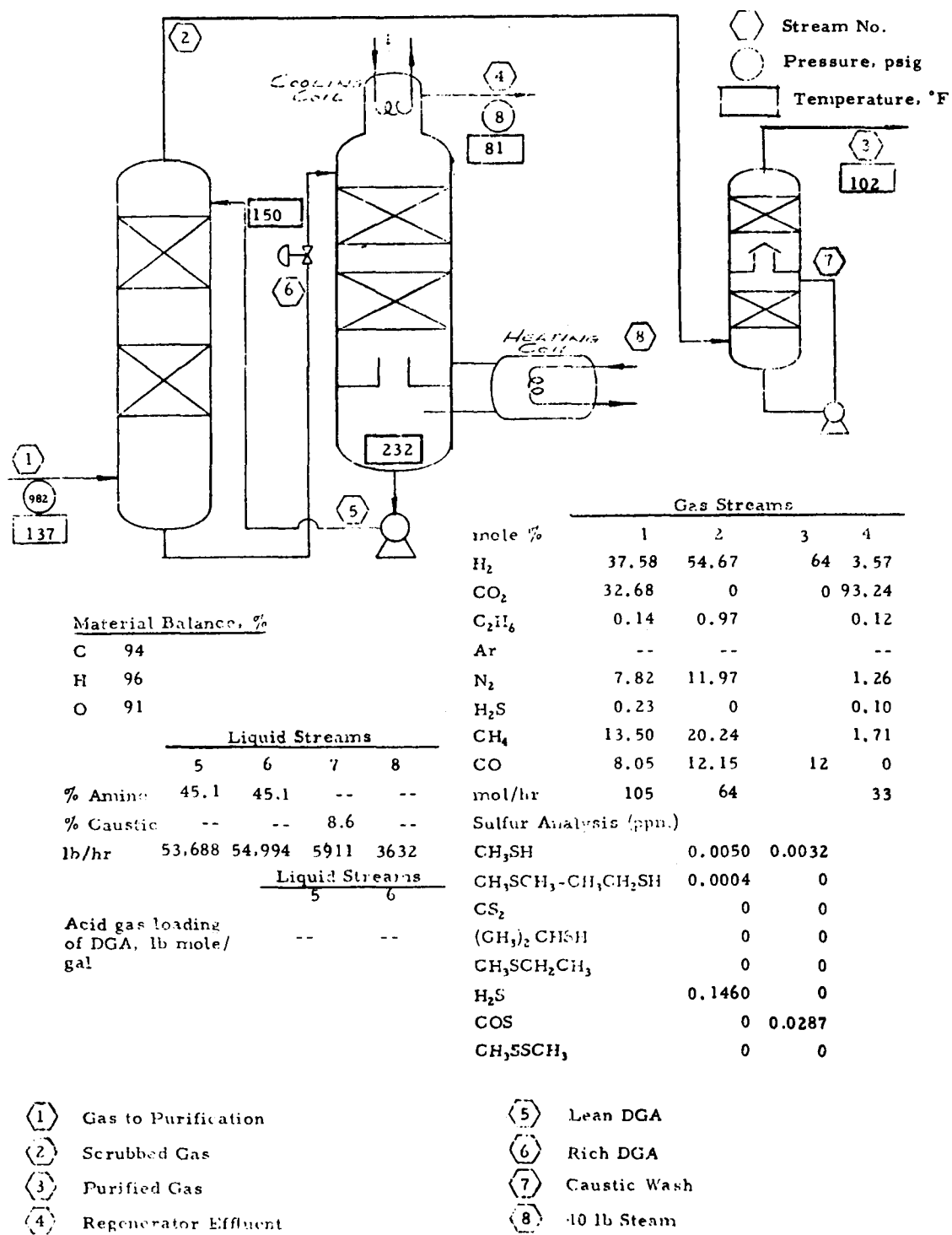
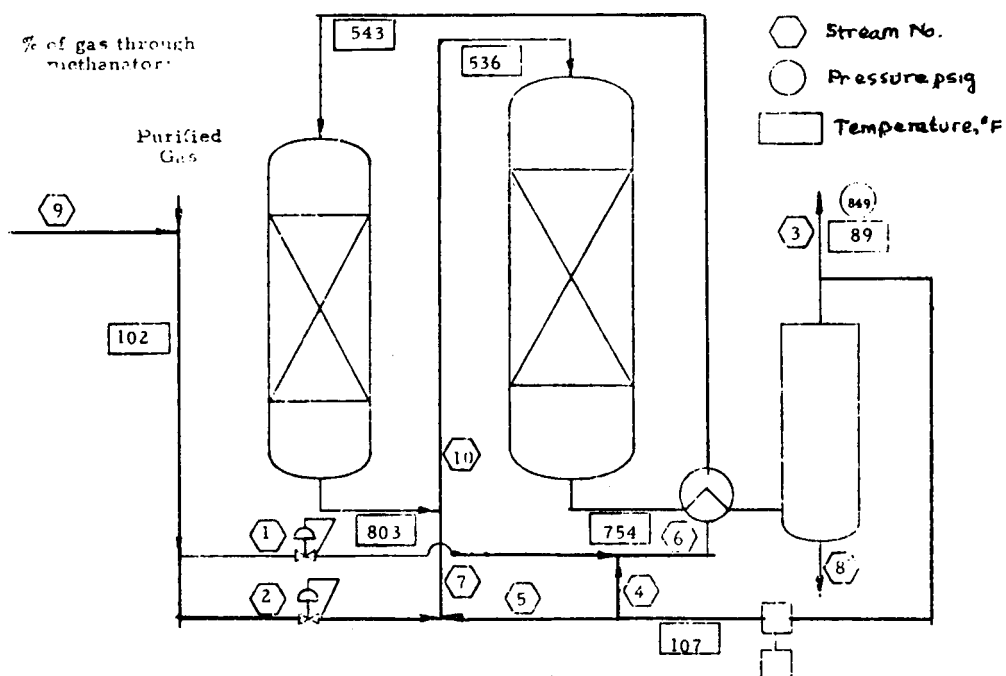


Figure 31. GAS PURIFICATION DATA FOR TEST 37
FROM 7/6/75 (1405 hr) TO 7/7/75 (0694 hr)



Material Balance, %

C 114

H 111

O 100

mol %	Gas Streams									
	1	2	3	4	5	6	7	8	9	10
H ₂	54.7	54.7	26.3	26.3	26.3	33.2	39.8			30.6
CO ₂	0	0	0	0	0	0	0			0
C ₂ H ₆	1.0	1.0	0	0	0	0.2	0.5			0.1
Ar	--	--	--	--	--	--	--			--
N ₂	12.0	12.0	17.9	17.9	17.9	16.5	15.1			17.2
H ₂ S	0	0	0	0	0	0	0			0
CH ₄	20.2	20.2	55.8	55.8	55.8	47.1	38.9			50.3
CO	12.1	12.1	0	0	0	3.0	5.8			1.8
mol/hr	35	28	45	109	31	144	60			191
H.V. (N ₂ , acid gas free)	497	497	790	790	790	715	646			740

	First Stage	Second Stage	Overall
Recycle Ratio	3.1	1.1	2.1
Space Velocity Based on Total Bed, hr ⁻¹	3476	2305	--
Pressure Drop (Inches H ₂ O)	2.8	11.0	13.8
% Bed Utilized (From Temp Profile)	54.2	16.7	29.2
Space Velocity Based on Bed Utilized, hr ⁻¹	6413	13,802	--

- ① Purified Gas to First Stage
- ② Purified Gas to Second Stage
- ③ Product Gas
- ④ Recycle Gas
- ⑤ Recycle Gas

- ⑥ First-Stage Feed Gas
- ⑦ Recycle and Fresh Feed to Second Stage
- ⑧ Condensate
- ⑨ Makeup Hydrogen
- ⑩ Second-Stage Feed Gas

Figure 32. METHANATION DATA FOR TEST 37
FROM 7/6/75 (1405 hr) TO 7/7/75 (0694 hr)

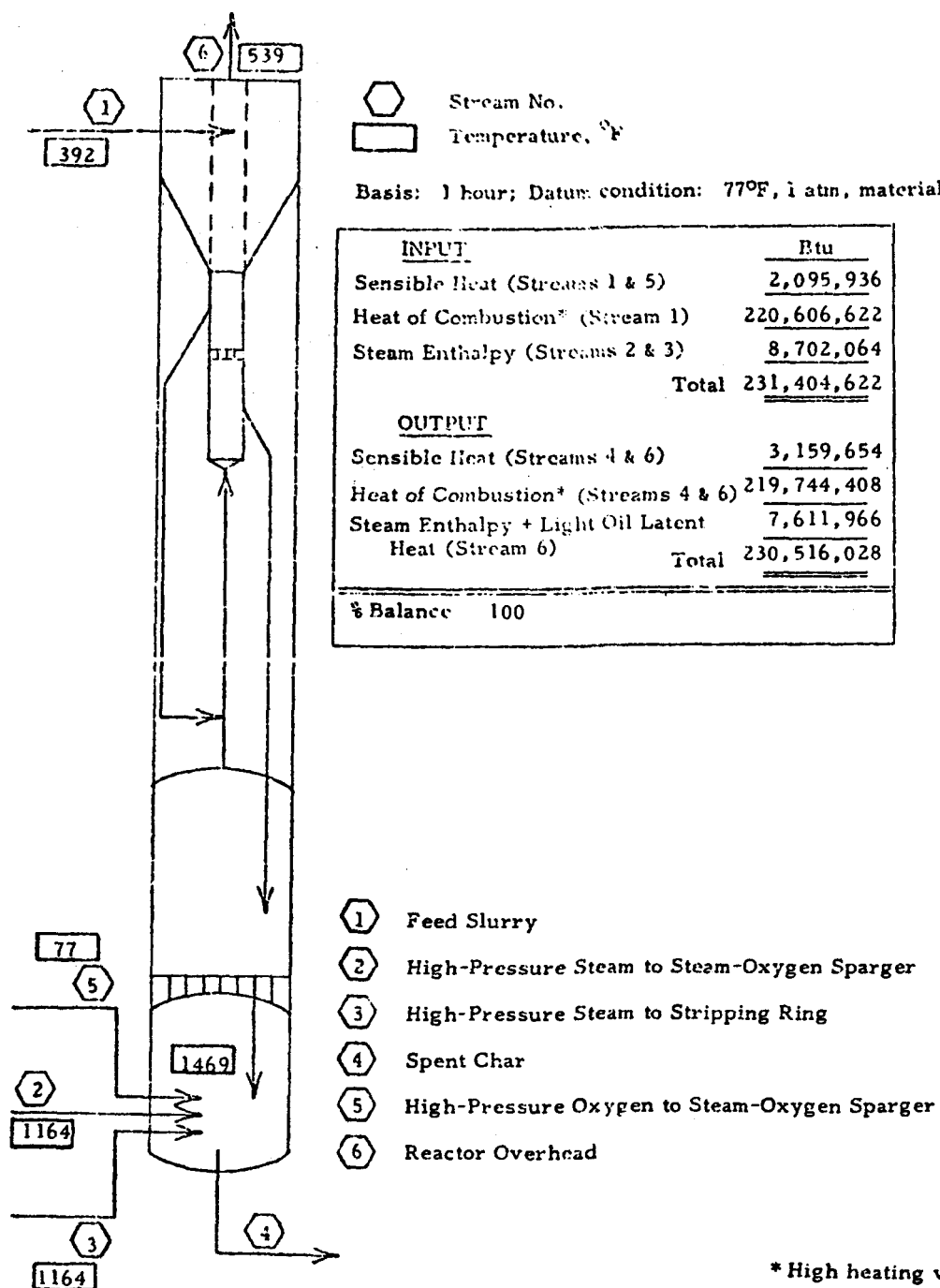
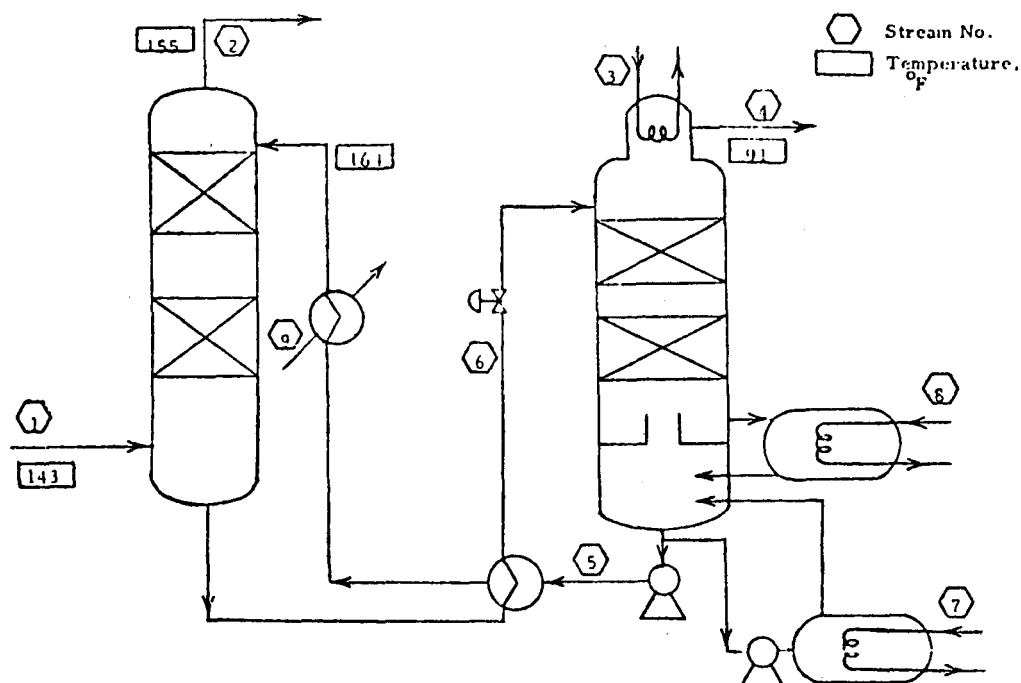


Figure 33. HYGAS REACTOR HEAT BALANCE DATA SHEET FOR TEST 37
 FROM 6/25/75 (1206 hr) TO 6/26/75 (1996 hr)

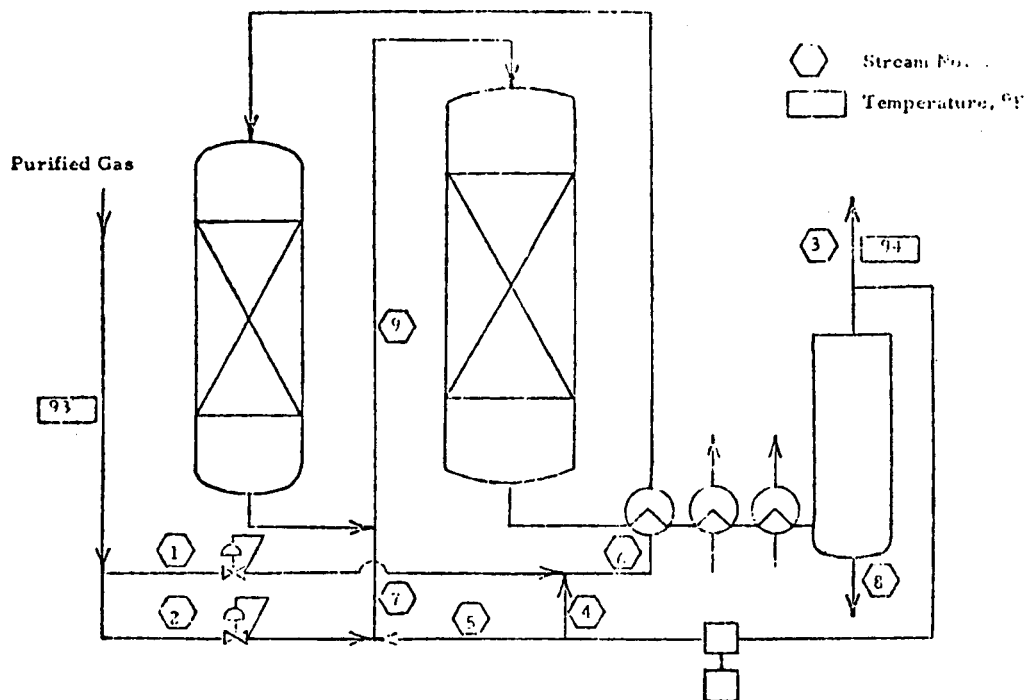


Basis: 1 hour: Datum condition: 77°F, 1 atm, material in standard state.

<u>INPUT</u>		<u>Btu</u>
Sensible Heat (Stream 1)		54,577
Heat of Combustion* (Stream 1)		10,946,472
Latent Heat (Streams 7 & 8)		3,541,714
Total		14,542,763
<u>OUTPUT</u>		
Sensible Heat (Streams 2 & 4)		42,990
Heat of Combustion* (Streams 2 & 4)		10,848,941
Sensible Heat (Streams 3 & 9)		3,455,759
Total		14,347,690
% Balance	99	

① Gas to purification	⑥ Rich. DGA
② Scrubbed gas	⑦ 200 lb Steam
③ Cooling water to reflux condenser	⑧ 40 lb Steam
④ Regenerator gas effluent	⑨ Cooling water to heat exchanger
⑤ Lean DGA	
- * High heating value.

Figure 34. GAS⁴ PURIFICATION HEAT BALANCE DATA SHEET FOR TEST 37 FROM 6/25/75 (1206 hr) TO 6/26/75 (1996 hr)



Basis: 1 hour; Datum condition: 77°F, 1 atm, material in standard state.

INPUT		Btu
Sensible Heat (Streams 1 & 2)		7,966
Heat of Combustion* (Streams 1 & 2)		10,515,390
Total		10,523,356
OUTPUT		
Sensible Heat (Stream 3)		6,958
Heat of Combustion* (Stream 3)		10,686,393
Sensible Heat (Heat Exchangers)		432,729
Total		11,126,080
% Balance	106	

(1) Purified Gas to First Stage	(6) First-Stage Feed Gas
(2) Purified Gas to Second Stage	(7) Recycle and Fresh Feed to Second Stage
(3) Product Gas	(8) Condensate
(4) Recycle Gas	(9) Second-Stage Feed Gas
(5) Recycle Gas	

* High heating value.

Figure 35. METHANATION HEAT BALANCE DATA SHEET FOR TEST 37
FROM 6/25/75 (1206 hr) TO 6/26/75 (1996 hr)

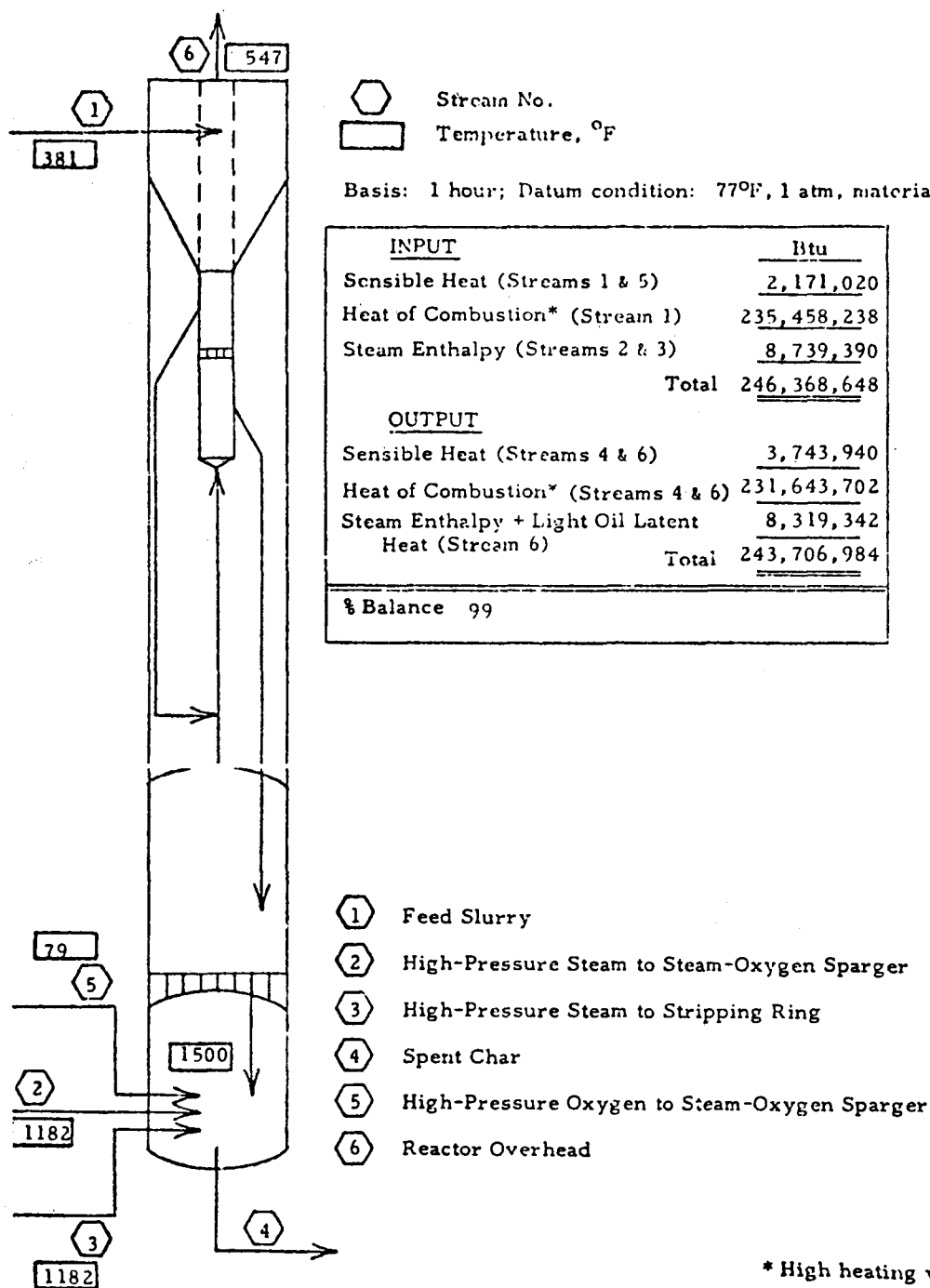
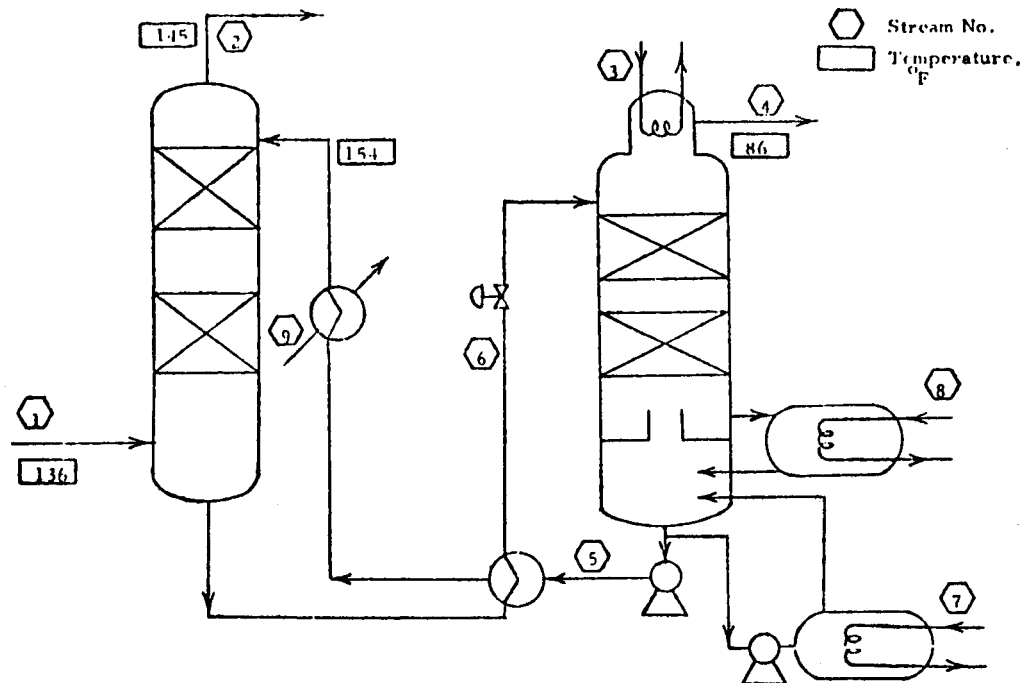


Figure 36. HYGAS REACTOR HEAT BALANCE DATA SHEET FOR TEST 37
FROM 6/27/75 (2007 hr) TO 6/29/75 (0192 hr)



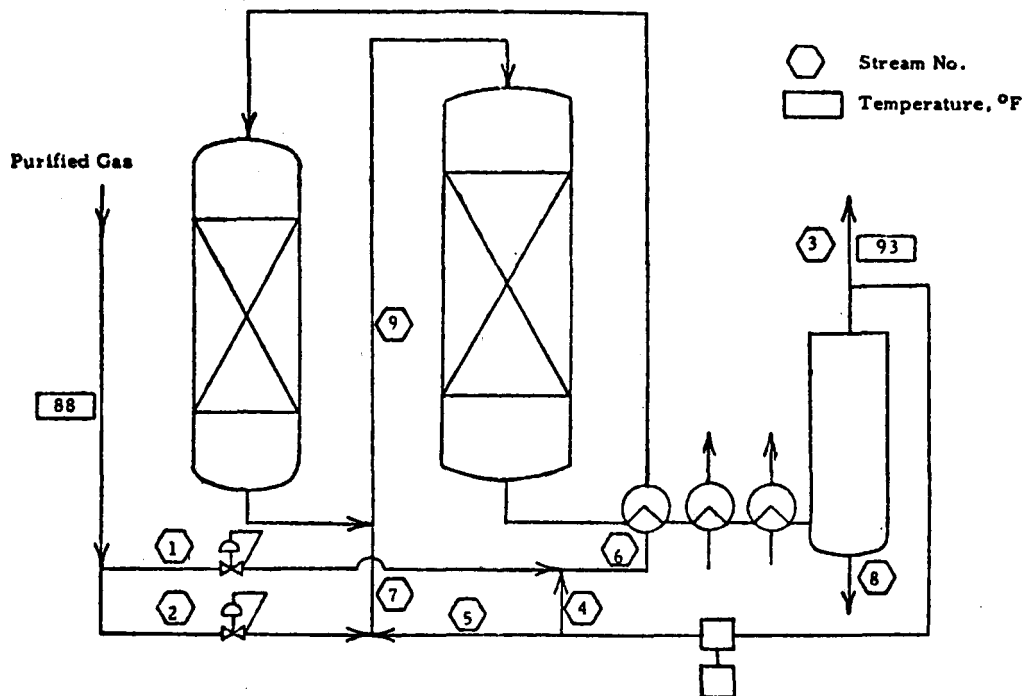
Basis: 1 hour; Datum condition: 77°F, 1 atm, material in standard state.

INPUT		Btu
Sensible Heat (Stream 1)		48,600
Heat of Combustion* (Stream 1)		11,936,251
Latent Heat (Streams 7 & 8)		3,581,850
Total		15,566,701
OUTPUT		
Sensible Heat (Streams 2 & 4)		37,290
Heat of Combustion* (Streams 2 & 4)		11,989,076
Sensible Heat (Streams 3 & 9)		3,591,285
Total		15,617,651
% Balance	100	

① Gas to purification	⑥ Rich DGA
② Scrubbed gas	⑦ 200 lb Steam
③ Cooling water to reflux condenser	⑧ 40 lb Steam
④ Regenerator gas effluent	⑨ Cooling water to heat exchanger
⑤ Lean DGA	

* High heating value.

Figure 37. GAS PURIFICATION HEAT BALANCE DATA SHEET FOR TEST 37 FROM 6/27/75 (2007 hr) TO 6/29/75 (0192 hr)



Basis: 1 hour; Datum condition: 77°F, 1 atm, material in standard state.

INPUT		Btu
Sensible Heat (Streams 1 & 2)		5,565
Heat of Combustion* (Streams 1 & 2)		11,700,133
Total		11,705,698
OUTPUT		
Sensible Heat (Stream 3)		5,825
Heat of Combustion* (Stream 3)		11,442,293
Sensible Heat (Heat Exchangers)		692,645
Total		12,140,763
% Balance	104	

① Purified Gas to First Stage	⑥ First-Stage Feed Gas
② Purified Gas to Second Stage	⑦ Recycle and Fresh Feed to Second Stage
③ Product Gas	⑧ Condensate
④ Recycle Gas	⑨ Second-Stage Feed Gas
⑤ Recycle Gas	

* High heating value.

Figure 38. METHANATION HEAT BALANCE DATA SHEET FOR TEST 37
FROM 6/27/75 (2007 hr) TO 6/29/75 (0192 hr)

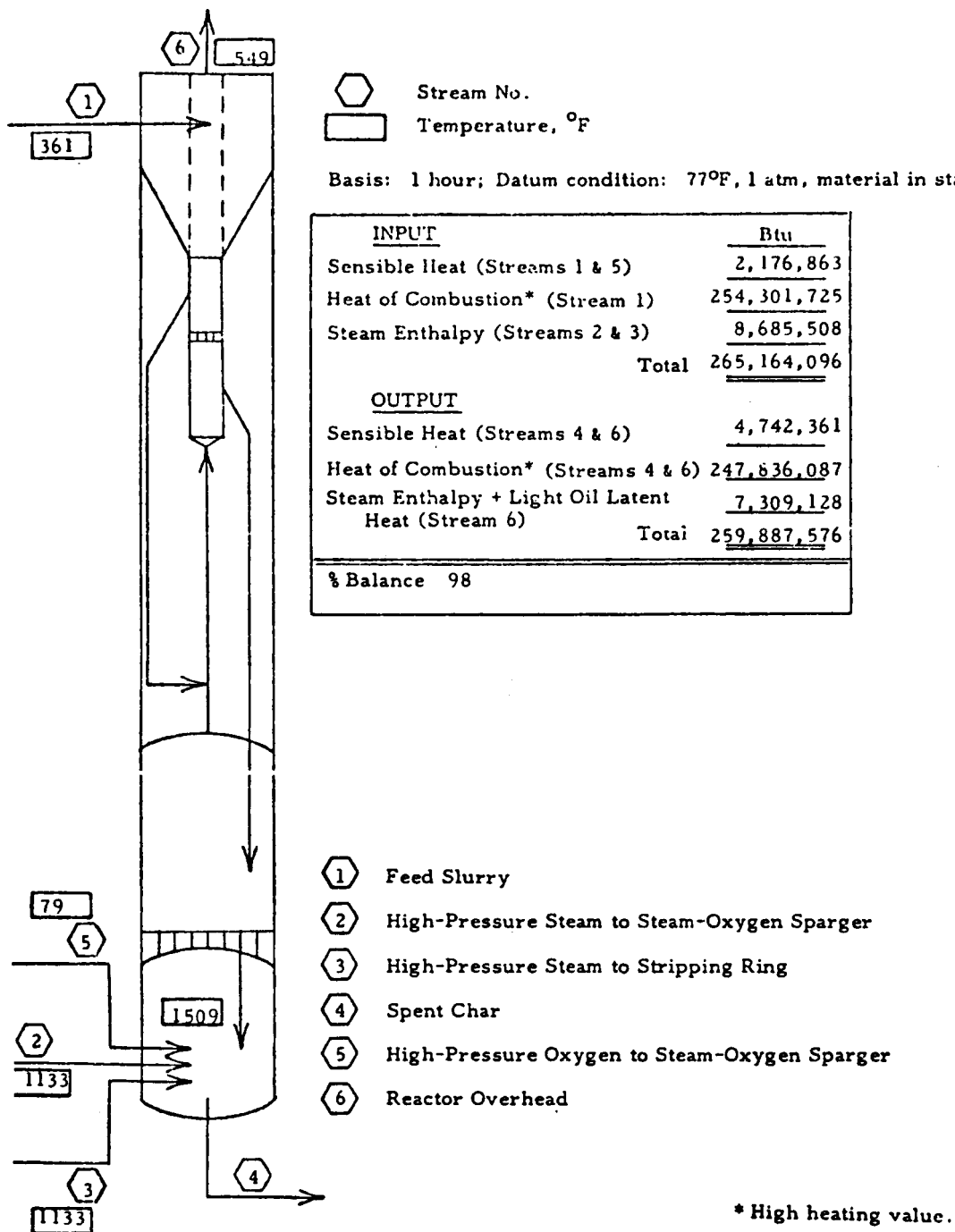
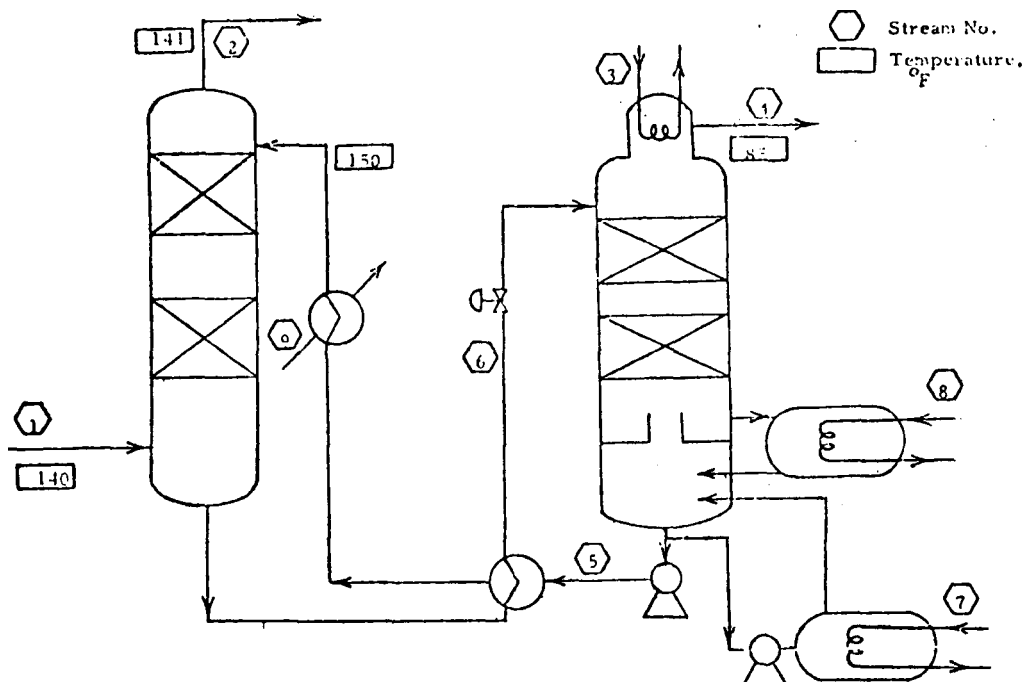


Figure 39. HYGAS REACTOR HEAT BALANCE DATA SHEET FOR TEST 37
FROM 6/29/75 (2001 hr) TO 6/29/75 (2399 hr)

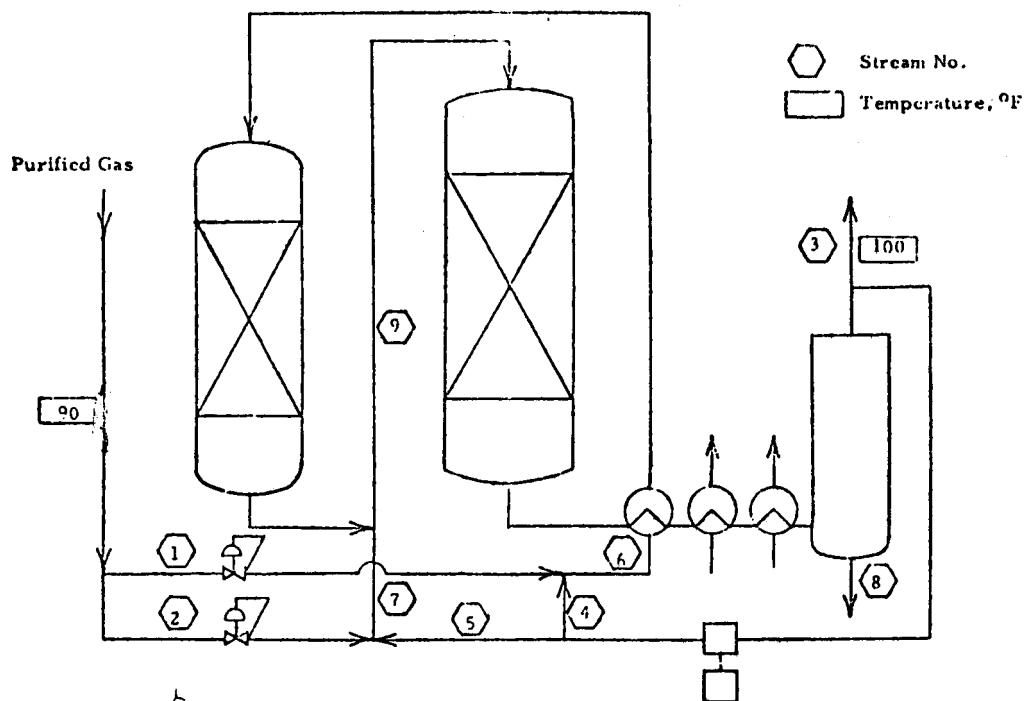


Basis: 1 hour; Datum condition: 77°F, 1 atm, material in standard state.

INPUT		Btu
Sensible Heat (Stream 1)		63,022
Heat of Combustion* (Stream 1)		15,413,049
Latent Heat (Streams 7 & 8)		3,608,100
Total		19,084,261
OUTPUT		
Sensible Heat (Streams 2 & 4)		42,633
Heat of Combustion* (Streams 2 & 4)		15,557,016
Sensible Heat (Streams 3 & 9)		3,729,919
Total		19,329,568
% Balance	101	

① Gas to purification	⑥ Rich DGA
② Scrubbed gas	⑦ 200 lb Steam
③ Cooling water to reflux condenser	⑧ 40 lb Steam
④ Regenerator gas effluent	⑨ Cooling water to heat exchanger
⑤ Lean DGA	
- * High heating value.

Figure 40. GAS PURIFICATION HEAT BALANCE DATA SHEET FOR TEST 37 FROM 6/29/75 (2001 hr) TO 6/29/75 (2399 hr)



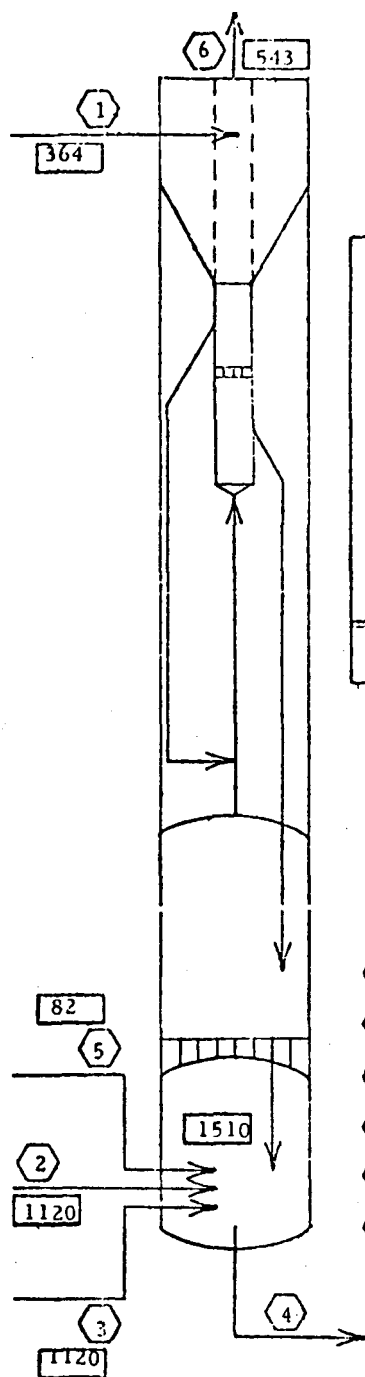
Basis: 1 hour; Datum condition: 77°F, 1 atm, material in standard state.

<u>INPUT</u>		Btu
Sensible Heat (Streams 1 & 2)		7,973
Heat of Combustion* (Streams 1 & 2)		15,290,880
Total		15,298,853
<u>OUTPUT</u>		
Sensible Heat (Stream 3)		9,743
Heat of Combustion* (Stream 3)		14,798,861
Sensible Heat (Heat Exchangers)		1,011,349
Total		15,819,953
% Balance	103	

① Purified Gas to First Stage	⑥ First-Stage Feed Gas
② Purified Gas to Second Stage	⑦ Recycle and Fresh Feed to Second Stage
③ Product Gas	⑧ Condensate
④ Recycle Gas	⑨ Second-Stage Feed Gas
⑤ Recycle Gas	

* High heating value.

Figure 41. METHANATION HEAT BALANCE DATA SHEET FOR TEST 37
FROM 6/29/75 (2001 hr) TO 6/29/75 (2399 hr)



Stream No.
 Temperature, °F

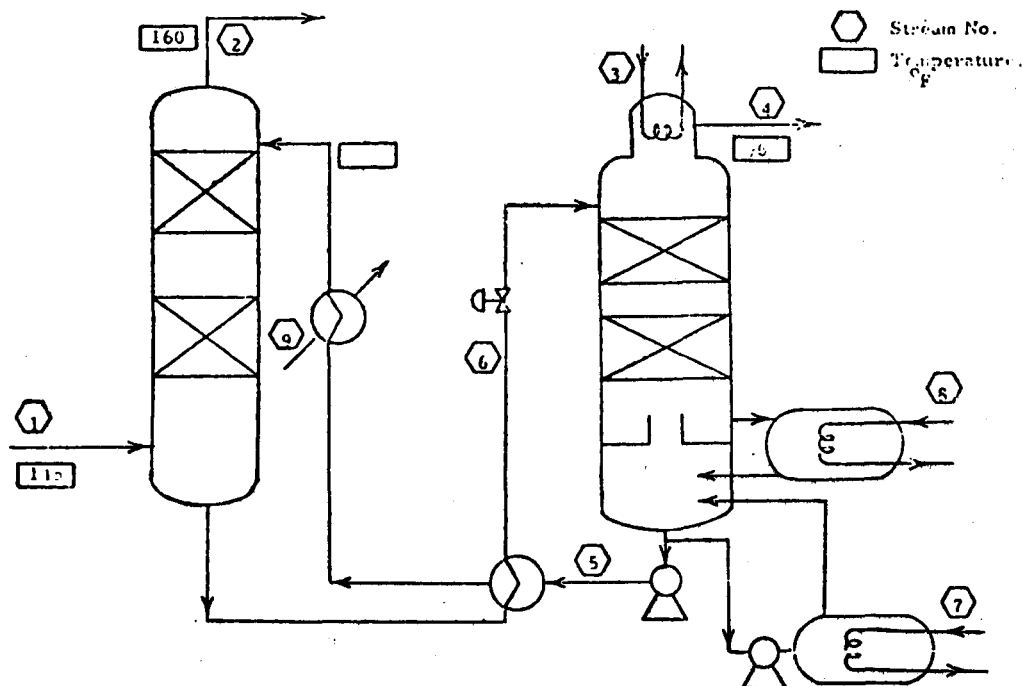
Basis: 1 hour; Datum condition: 77°F, 1 atm, material in standard state.

INPUT		Btu
Sensible Heat (Streams 1 & 5)		2,160,985
Heat of Combustion* (Stream 1)		246,220,975
Steam Enthalpy (Streams 2 & 3)		8,694,738
Total		257,076,698
OUTPUT		
Sensible Heat (Streams 4 & 6)		3,880,065
Heat of Combustion* (Streams 4 & 6)		243,794,954
Steam Enthalpy + Light Oil Latent Heat (Stream 6)		8,450,028
Total		256,125,047
% Balance		100

- 1 Feed Slurry
- 2 High-Pressure Steam to Steam-Oxygen Sparger
- 3 High-Pressure Oxygen to Steam-Oxygen Sparger
- 4 Spent Char
- 5 High-Pressure Oxygen to Steam-Oxygen Sparger
- 6 Reactor Overhead

* High heating value.

Figure 42. HYGAS REACTOR HEAT BALANCE DATA SHEET FOR TEST 37
FROM 6/30/75 (1480 hr) TO 7/2/75 (0598 hr)



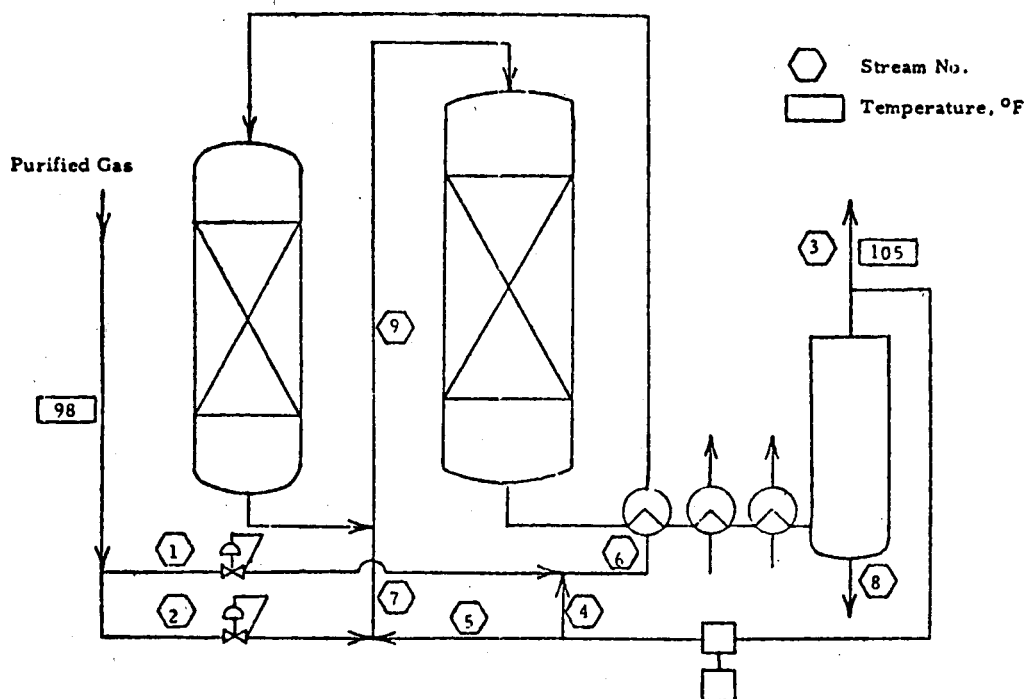
Basis: 1 hour: Datum condition: 77°F, 1 atm, material in standard state.

INPUT		Btu
Sensible Heat (Stream 1)		69,312
Heat of Combustion* (Stream 1)		15,389,343
Latent Heat (Streams 7 & 8)		3,149,932
Total		18,608,587
OUTPUT		
Sensible Heat (Streams 2 & 4)		55,910
Heat of Combustion* (Streams 2 & 4)		14,313,973
Sensible Heat (Streams 3 & 9)		3,433,097
Total		17,802,980
Balance		96

① Gas to purification	⑥ Rick DGA
② Scrubbed gas	⑦ 200 lb Steam
③ Cooling water to reflux condenser	⑧ 40 lb Steam
④ Regenerator gas effluent	⑨ Cooling water to heat exchanger
⑤ Lean DGA	

* High heating value.

Figure 43. GAS PURIFICATION HEAT BALANCE DATA SHEET FOR TEST 37 FROM 7/1/75 (1300 hr) TO 7/2/75 (0598 hr)



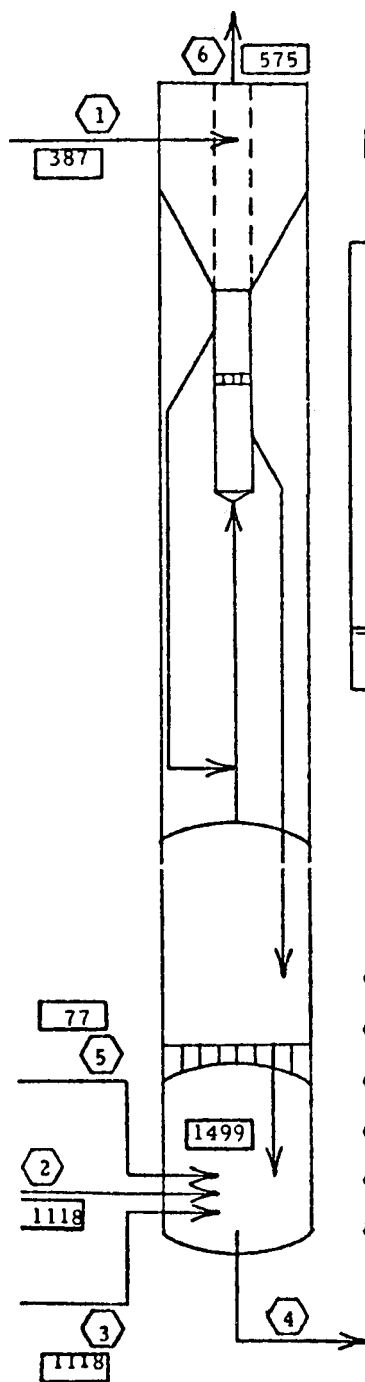
Basis: 1 hour; Datum condition: 77°F, 1 atm, material in standard state.

INPUT		Btu
Sensible Heat (Streams 1 & 2)		11,960
Heat of Combustion* (Streams 1 & 2)		13,695,766
Total		13,707,726
OUTPUT		Btu
Sensible Heat (Stream 3)		10,518
Heat of Combustion* (Stream 3)		12,773,287
Sensible Heat (Heat Exchangers)		809,774
Total		13,593,579
% Balance		99

① Purified Gas to First Stage	⑥ First-Stage Feed Gas
② Purified Gas to Second Stage	⑦ Recycle and Fresh Feed to Second Stage
③ Product Gas	⑧ Condensate
④ Recycle Gas	⑨ Second-Stage Feed Gas
⑤ Recycle Gas	

* High heating value.

Figure 44. METHANATION HEAT BALANCE DATA SHEET FOR TEST 37
FROM 7/1/75 (1300 hr) TO 7/2/75 (0600 hr)



 Stream No.
 Temperature, °F

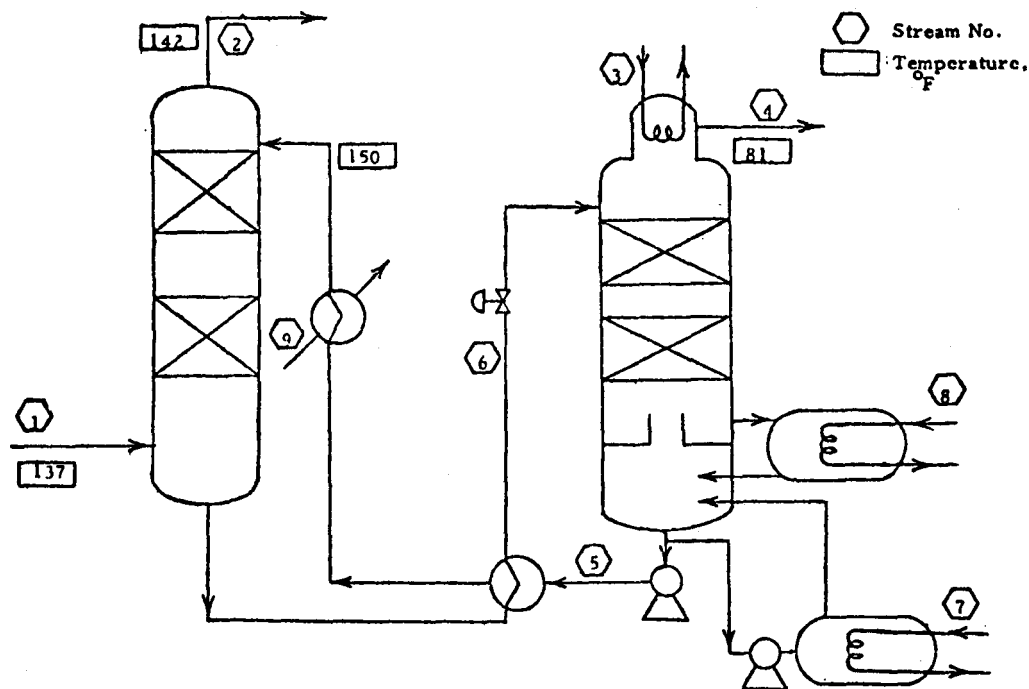
Basis: 1 hour; Datum condition: 77°F, 1 atm, material in standard state.

INPUT		Btu
Sensible Heat (Streams 1 & 5)		2,007,610
Heat of Combustion* (Stream 1)		210,346,415
Steam Enthalpy (Streams 2 & 3)		8,858,430
Total		221,212,455
OUTPUT		
Sensible Heat (Streams 4 & 6)		3,134,141
Heat of Combustion* (Streams 4 & 6)		211,120,427
Steam Enthalpy + Light Oil Latent Heat (Stream 6)		7,290,612
Total		221,545,180
% Balance		100

-  1 Feed Slurry
-  2 High-Pressure Steam to Steam-Oxygen Sparger
-  3 High-Pressure Steam to Stripping Ring
-  4 Spent Char
-  5 High-Pressure Oxygen to Steam-Oxygen Sparger
-  6 Reactor Overhead

* High heating value.

Figure 45. HYGAS REACTOR HEAT BALANCE DATA SHEET FOR TEST 37
FROM 7/5/75 (1506 hr) TO 7/7/75 (0694 hr)



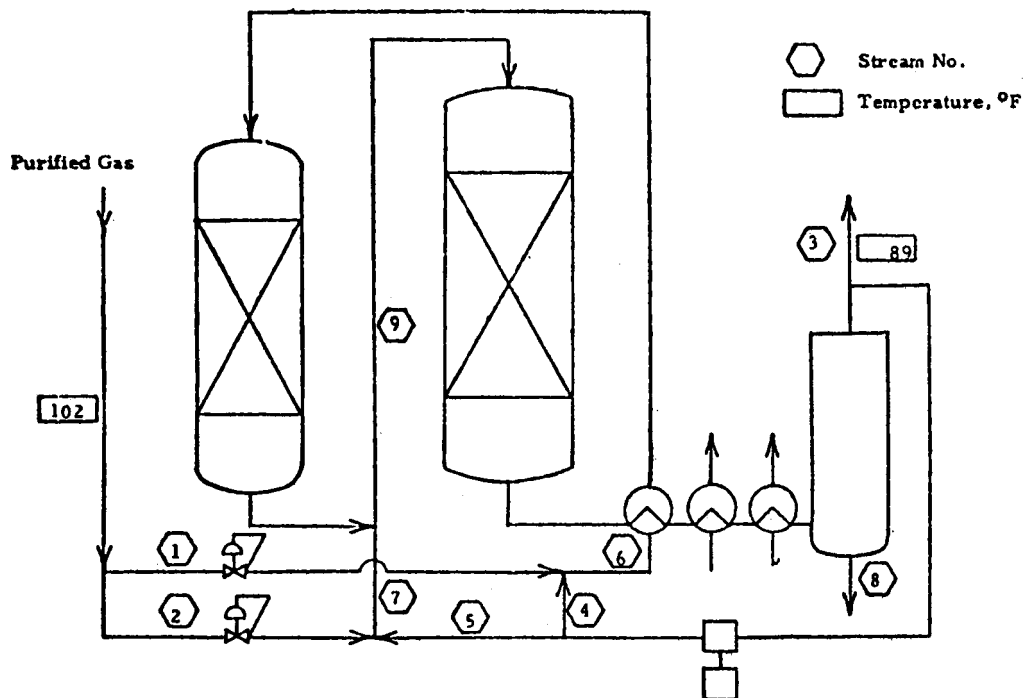
Basis: 1 hour; Datum condition: 77°F, 1 atm, material in standard state.

INPUT		Btu
Sensible Heat (Stream 1)		48,955
Heat of Combustion* (Stream 1)		11,379,508
Latent Heat (Streams 7 & 8)		3,619,071
Total		15,047,534
OUTPUT		
Sensible Heat (Streams 2 & 4)		31,598
Heat of Combustion* (Streams 2 & 4)		11,083,815
Sensible Heat (Streams 3 & 9)		3,518,090
Total		14,633,503
% Balance	97	

① Gas to purification	⑥ Rich DGA
② Scrubbed gas	⑦ 200 lb Steam
③ Cooling water to reflux condenser	⑧ 40 lb Steam
④ Regenerator gas effluent	⑨ Cooling water to heat exchanger
⑤ Lean DGA	

* High heating value.

Figure 46. GAS PURIFICATION HEAT BALANCE DATA SHEET FOR TEST 37 FROM 7/6/75 (1405 hr) TO 7/7/75 (0694 hr)



Basis: 1 hour; **Datum condition:** 77°F, 1 atm, material in standard state.

<u>INPUT</u>	<u>Btu</u>
Sensible Heat (Streams 1 & 2)	11,901
Heat of Combustion* (Streams 1 & 2)	10,718,284
Total	10,730,185
 <u>OUTPUT</u>	
Sensible Heat (Stream 3)	4,251
Heat of Combustion* (Stream 3)	11,126,697
Sensible Heat (Heat Exchangers)	639,966
Total	11,770,914
Balance 110	

①	Purified Gas to First Stage	⑥	First-Stage Feed Gas
②	Purified Gas to Second Stage	⑦	Recycle and Fresh Feed to Second Stage
③	Product Gas	⑧	Condensate
④	Recycle Gas	⑨	Second-Stage Feed Gas
⑤	Recycle Gas		

* High heating value.

Figure 47. METHANATION HEAT BALANCE DATA SHEET FOR TEST 37
FROM 7/6/75 (1405 hr) TO 7/7/75 (0694 hr)

ASH-AGGLOMERATING GASIFICATION

Introduction

Twenty-eight tests have been made since July 1, 1974. Coke breeze continued to be the feed material. Since Test 17, the conditions were developed using air-steam gasifying mixture to successfully produce agglomerates without process or operating problems. From Tests 25 to 34, mechanical modifications were developed so that dust was successfully and continuously recirculated to the fluid bed. The changes included means of providing oxygen-enriched air to promote gasification of the dust. Changing the venturi diameter and removing the +6 mesh material from the coke breeze promoted the formation of smaller agglomerates.

Next, the proper conditions were found using oxygen-steam mixtures to produce high-ash agglomerates. After installing a new internal cyclone, tests were run to establish conditions for continuous high-ash agglomerate production along with dust reinjection, while continuing to use oxygen-steam mixtures. Two tests were made with FMC char made from Illinois No. 6 coal. Neither test produced agglomerates; however, the gasifier will be modified before running this char again so that lower velocities can be employed. The dust losses from this char were too high as its particle size averaged one-quarter that of the coke.

Methods of data taking were developed and accuracy improved so that heat and mass balances were regularly included. Another sheet is also furnished, listing operational parameters and particle size ranges of solids streams. An account of the tests made since July 1, 1974, follows.

Description of TestsTest 15

The first-stage cyclone dipleg was rewelded in position. Four gussets were added at the welded juncture for additional strength. Strips of 10-gauge stainless steel were welded at the junction of the stabilizing bars and the internal channels attached to the reactor. These strips are intended to act as "spring-type" dampeners for the dipleg against the vibrations caused by the fluid bed.

While feeding coke to the reactor, an unusually high differential pressure was observed across the venturi, indicating an obstruction. After several unsuccessful attempts to clear this blockage by surging air through the venturi, the test was terminated.

After letting the reactor cool for several hours, suddenly the entire bed began to flow through the venturi. Two hours later, after the reactor was empty and sufficiently cooled, the manways were opened, but no evidence of obstructions and/or clinkers was found.

Test 16

Since no reason was found for the high differential pressure across the venturi in Test 15, Test 16 was started immediately using the same start-up procedures as in Tests 14 and 15. Accordingly, a preheated process-gas temperature of 600°F was used, and a 12% oxygen concentration was maintained at the grid. A 5-1/2 foot bed was built up with no difficulty in the reactor before the start-up burner was ignited. The reaction became autogenous at 900°F, and the reactor temperatures indicated good fluidization. The bed temperature rose to a maximum of 1890°F. Although the material discharged from the venturi appeared to be rich in ash (later confirmed by analysis), no agglomerates were formed.

This test was terminated when clinker formation was observed through the sight glass on the bed pressure tap. Most of the material in the bed was removed through the venturi by lowering the venturi velocity. After cooling the reactor, it was found that the first-stage cyclone dipleg had broken off just below the newly added gussets. The collar on the dipleg, which holds the stabilizer, had also split and fallen. This probably accentuated the vibration of the dipleg, resulting in fatigue failure at the weld.

An analysis of the discharged bed material indicated that the ash content in the bed had built up to an average of 19% over a period of 34 hours. Figure 48 is a graph of the calculated ash content in the bed based upon the feed rate, venturi discharge rate, and the gasification rate determined by monitoring the carbon monoxide and carbon dioxide contents of the product gas. In Test 11, agglomerates began to form after 16 hours when the average ash content in the bed reached approximately 22%. Also in Test 14, agglomerates were produced after 18 hours as the average ash content of the bed reached 22%. However, mechanical failure of the first-stage cyclone ended the test shortly after the appearance of the agglomerates. No agglomerates were produced in Test 16 because a sufficiently high (22%) ash content in the bed was never reached, as can be seen from the graph.

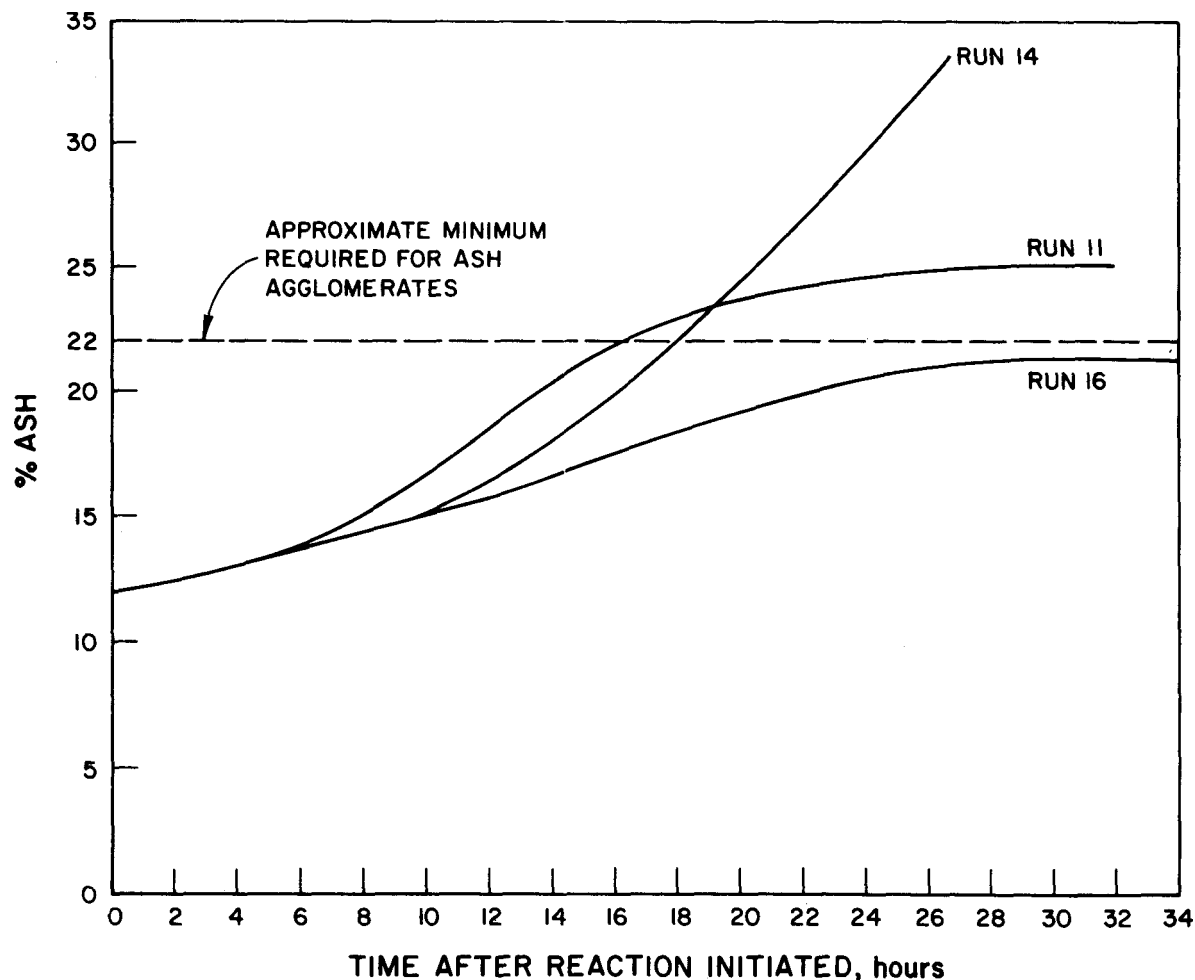


Figure 48. AVERAGE ASH CONTENT IN THE BED

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Test 17

A new first-stage cyclone dipleg was welded in position. The trickle valve at the bottom of the dipleg was modified to a symmetrical double-cone arrangement similar to a valve successfully used by FMC Corporation in Project COED. The 22-1/2 degree slope grid was replaced by a 45-degree-slope grid of similar design. Grid nozzles were drilled to obtain a reactant gas flow perpendicular to the grid surface.

A 5-foot bed was accumulated, and the oxygen concentration at the grid was maintained at 12%. Approximately 22 hours after the test began, a decrease of pressure drop across the first-stage cyclone was noted. The dust collected in the second-stage cyclone was also noticeably coarser than in earlier samples.

In this test, however, ash agglomerates were successfully formed for over 24 hours, utilizing complete dust reinjection, through the venturi, of fines collected by the second-stage cyclone. The agglomerates produced in this test were uniform and spherical. Preliminary indications are that the carbon level in the bed was high, which is extremely significant in that it provides operational stability to handle any fluctuations in oxidant flow. A bed with a low-carbon inventory is less able to handle fluctuations without a corresponding change in temperature, which, in turn, leads to gross slagging or agglomeration in the bed.

The test had to be terminated because of problems emanating from the solids feed system. Concurrently, the discharge through the venturi was reduced considerably, indicating an obstruction.

After cooling the reactor, it was discovered that the entire cyclone had fallen onto the grid. Two stabilizing bars had broken off from the collar and lodged in the venturi, creating the blockage. An inspection of the unit after shutdown revealed a hole located between the cyclone barrel and the dust disengaging hopper of the first-stage cyclone; this hole resulted in gas bypassing the cyclone and in solids carry-over. The hole was probably caused by ignition and burning of the solids, which had collected and plugged the first-stage cyclone.

From the run data, after the cyclone broke off, operation was maintained for at least 10 hours without any difficulties. An inspection of the quench unit before the secondary cyclone showed that dust accumulation stabilized at an acceptable level without causing any blockage. The secondary cyclone was refractory lined to take the abrasive solids directly for the next test. Therefore, the primary cyclone will not be reinstalled. The cyclone was removed and the reactor reassembled.

Figure 49 shows the calculated ash content of the bed for Test 17 where agglomerates averaging 90% ash (10% carbon) were produced for a 12-hour period. The ash content of the bed rose 22% to 25%, the level apparently

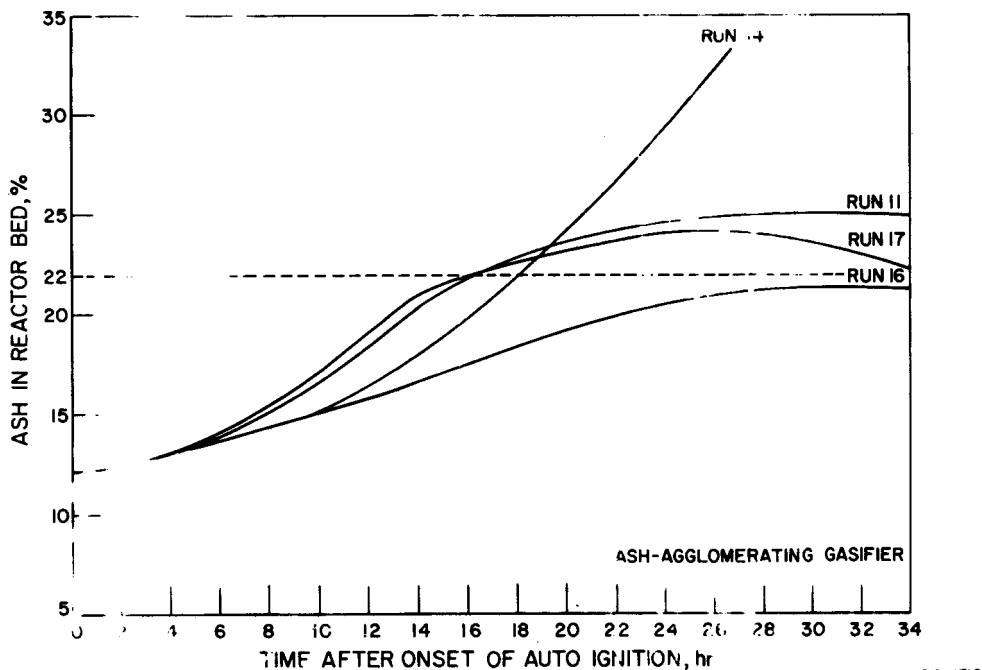


Figure 49. CALCULATED ASH CONTENT IN THE BED

necessary to produce agglomerates. This graph (without the Test 17 line) was referred to in the July 1974 OCR Report No. 118 to explain the presence of agglomerates in Test 11 and 14, and their absence in Test 16. An analysis of bed samples taken at the termination of Test 16 revealed an average 19% ash content, thus substantially verifying the calculated line shown in Figure 49.

Tables 25 and 26 show data covering a portion of the operating periods for Tests 16 and 17. Table 29 shows that in Test 16 a sufficiently low level of carbon in the ash to form agglomerates was approached, but not reached. In fact, no agglomerates were observed before Test 16 was terminated.

Agglomerating conditions and agglomerates were present in Test 17, as shown in Table 26 beginning at 0230 hours on July 10, 1974. Beginning at 1630 hours on July 10, 1974, the venturi velocity was reduced to 65 ft/s, and as a result, gasified, but not agglomerated, large coke particles began to be discharged. While the bed height readings were correct, a high back pressure at some pressure taps provided erroneously high bed-density values. Finally, the loss of the first-stage dust collector resulted in high dust losses from the system and an increased feed rate (versus Tests 14 and 16) to maintain a steady bed level.

Test 18

This test was conducted in August after completely removing the first-stage cyclone dust collector, which had been badly damaged in Test 17. Similar to Test 17, after a 5-foot-deep bed was accumulated, the start-up burner was fired and autoignition achieved. Agglomerates were observed 15 hours later. This test was continued until the system was voluntarily shut down on August 2 at 1600 hours. The bed was completely emptied from the unit into drums. An internal inspection after the reactor cooled revealed no accumulation whatsoever of char or coke and no evidence of clinkers. Ash agglomerates were produced continuously for over 36 hours in this test.

A feed rate of 600 lb/hr (versus 350 to 400 lb/hr in previous tests) was required to maintain a 5-foot-bed level. From examining the dust samples from the cyclone, it appeared that the material was quite coarse and that probably the single cyclone being used was allowing a considerable amount of dust to escape.

Test 19

While preparing to convert the process gas input to a steam-oxygen mixture instead of the steam-air mixture, another steam-air test was made during which plans were to operate at reduced superficial velocities to minimize fines carry-over and to obtain data for a complete material balance around the system. In Test 18, a superficial velocity of 2 to 5 ft/s was used as had been the case in all previous tests with two stages of cyclone separation. Although a 5-foot-deep fluidized bed was satisfactorily accumulated, autoignition achieved, and a reactor temperature of 1860°F attained, the test had to be terminated. The large dust losses that occurred again with only one stage of dust collection interfered with the operation of the product-gas incinerator and shut it down. When forced to divert all of the dusty product gas to the bypass stack, a large back pressure was put on the reactor in excess of 5 psig so that feed material would not flow from the surge hopper to the reactor screw feeder. Since coke could not be fed to the reactor, the test was terminated.

Table 25. OPERATING CONDITIONS IN THE ASH-AGGLOMERATING GASIFIER DURING TEST 16

Date	6/19/74					6/20/74						
Time	1600	1800	2000	2200	2400	0200	0400	0600	0800	1000	1200	1400
Bed Temperature, °F	1863	1869	1881	1890	1895	1885	1883	1883	1885	1892	1876	1914
Air Flow Rate, lb/hr	1510	1510	1510	1510	1510	1510	1510	1510	1510	1510	1310	1510
Steam Flow Rate, lb/hr	410	410	410	410	410	410	410	410	410	340	340	340
Coke Feed Rate, lb/hr	250	250	250	250	250	325	325	325	325	325	285	285
Venturi Velocity, ft/s	86	87	87	88	87	90	93	93	95	96	53	73
Superficial Reactor Velocity, ft/s	2.25	2.25	2.25	2.25	2.25	2.25	2.25	2.25	2.25	2.14	1.93	2.14
Bed Height, ft	4.89	4.73	4.73	4.49	4.47	4.32	4.49	4.6	4.55	4.81	4.89	4.98
Bed Density, lb/cu ft	35.3	35.3	35.3	35.3	33.8	35.3	35.3	35.7	36.1	35.3	35.3	35.3
Ash Discharge Rate, lb/hr	24	22	20	22	16	25	29	35	40	38	49	20
Carbon in Ash, %	78.5	77.3	75.7	74.2	69	64.6	64.5	61	55.2	62.3	45.7	26.9
Carbon Conversion, % (based on ash analysis)	45.3	49.7	54.0	57.9	66.8	73.8	74.1	77.8	82.5	76.4	88.2	94.9
Reactor Gas, % (dry basis)												
N ₂	68.3	67.4	68.0	67.1	--	63.0	61.8	61.4	64.1	70.4	63.0	62.7
CO	7.6	7.9	7.9	8.1	--	10.8	12.0	12.7	10.4	12.7	11.5	12.3
CO ₂	15.8	16.0	15.7	16.1	--	15.6	15.7	15.1	15.0	4.8	14.6	14.0
H ₂	8.3	8.7	8.4	8.7	--	9.8	9.8	9.9	9.8	10.3	10.2	10.3
Ar	--	--	--	--	--	0.7	0.7	0.7	0.7	0.7	0.7	0.7
CH ₄	--	--	--	--	--	0.1	0.0	0.2	0.0	1.1	0.0	0.0

Table 26. OPERATING CONDITIONS IN THE ASH-AGGLOMERATING GASIFIER DURING TEST 17

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Date	7/10/74										
Time	0230	0430	0630	0830	1030	1230	1430	1630	1830	2030	2230
Bed Temperature, °F	1928	1909	1920	1919	1922	1895	1903	1887	1883	1887	1912
Air Flow Rate, lb/hr	1720	1720	1720	1720	1720	1610	1610	1520	1520	1520	1610
Steam Flow Rate, lb/hr	410	410	410	410	410	410	410	410	410	410	410
Coke Feed Rate, lb/hr	485	485	485	485	485	485	485	485	485	485	485
Venturi Velocity, ft/s	85	85	85	85	85	74	74	65	65	65	74
Superficial Reactor Velocity, ft/s	2.47	2.47	2.47	2.47	2.47	2.36	2.36	2.26	2.26	2.26	2.36
Bed Height, ft	4.58	4.81	4.73	4.72	4.73	4.73	4.66	4.61	4.62	4.66	3.42
Bed Density, lb/cu ft	47.2	46.4	47.2	44.3	43.8	43.8	46.4	48.5	48.0	48.5	44.3
Ash Discharge Rate, lb/hr	11	13	16	19	24	38	47	66	40	42	25
Carbon in Ash, %	13.2	10.7	10.0	9.2	5.1	6.3	11.0	27.2	20.2	30.9	7.7
Carbon Conversion, % (based on ash analysis)	97.8	98.3	98.4	98.6	99.2	99.0	98.3	94.6	96.4	93.7	98.8
Reactor Gas, % (dry basis)											
N ₂	--	67.6	--	60.9	--	60.9	--	64.9	--	65.9	--
CO	--	9.3	--	11.2	--	11.4	--	8.7	--	9.8	--
CO ₂	--	11.5	--	14.1	--	14.1	--	14.7	--	12.0	--
H ₂	--	10.5	--	12.9	--	12.6	--	10.4	--	11.4	--
Ar	--	0.73	--	0.64	--	0.64	--	0.68	--	0.68	--
CH ₄	--	0.2	--	0.2	--	0.4	--	0.3	--	0.1	--

After the test, a second-stage cyclone dust collector was added directly following the existing cyclone. This carbon-steel cyclone was available onsite, and this process equipment change was begun immediately. Provisions were also made to reinject the dust from this new cyclone to the reactor venturi.

Test 20

The addition of the second-stage cyclone dust collector following Test 19 was completed in 3 days, thus allowing this evaluation test to be conducted. Once again, agglomerates were produced approximately 24 hours after autogenous operation was reached. High dust losses from the system still occurred, which were caused either by plugging of the first-stage cyclone dipleg or by an excessive upflow of aeration gas through its dipleg. Both conditions existed alternately and created visually high dust losses. During the transition between these cyclone operating conditions, the stack was quite clean. It was necessary to maintain a full, but not flooded, column of dust in the first-stage cyclone dipleg. Therefore, a butterfly valve, controlled from the panelboard, was installed in the dipleg, together with taps, transmitter, and a recorder to measure and control the dust column height in the dipleg.

Test 21

Test 21 was initiated and autogenous operation achieved. The primary objective was to obtain the proper conditions for dust collection and return. Proper dipleg aeration conditions to maintain a stable solids leg in the dipleg were achieved. However, there was insufficient time to determine its dust collection efficiency because the air lock seal valves on the coke feed system began leaking reactor gases, thus preventing feeding and forcing termination of the test.

Test 22

After repairing and reseating the feed air lock, Test 22 was begun. This test was aborted during the preliminary heat-up stage because of localized premature autogenous reactions occurring within the poorly fluidized bed. Dumping the bed revealed a clean reactor (no clinkers), so the manways were closed and the test restarted.

Test 23

Again, a bed was built up and autogenous operation achieved. Control of dipleg material height in the first-stage cyclone was easily maintained. Yet its effectiveness in minimizing dust losses from stack observation was not apparent. Fatigue failure of the reactor screw-feeder motor shaft forced the termination of this test.

More coke was dried for charging to the storage system. At the same time, it was discovered that the primary cyclone, which had been the secondary cyclone when an internal primary cyclone was used on the gasifier, was severely worn near the inlet area. Even the hard-face refractory that was cast behind the steel shell was heavily worn. This was the result of the

high-velocity inlet gas containing large-particle solids at high dust-loading levels. This was the primary reason that the cyclone's collection efficiency had not been up to expectations. A properly sized primary cyclone replacement was designed, built, and installed. At the same time, the vent gas line was enlarged so that, in the case of incinerator shutdown, the gas would be vented to the atmosphere without creating large back pressure on the gasifier.

Test 24

Steady-state operation was reached 36 hours after the start of the test. The new first-stage cyclone provided a lower pressure drop (approximately 10 inches of water); however, large dust losses from the system still occurred. This dust was coarse and could not be reinjected up the venturi to the fluid bed. The discharged ash agglomerates contained a significant amount of dust. The large loss of dust apparently plugged the product-gas line for the bypass stack had to be used to continue the test. Twenty-four hours after diverting the product gas, the test was terminated.

During Test 24, there were problems with the feed system similar to those in Test 21. These might have resulted from the back flow of purge gas used at the reactor screw-feeder shaft seals.

After the reactor was cooled down, a portion of the product gas line, which was plugged with dust, was readily vacuumed out. The J-valve dust reinjector had holes that allowed the dust to be driven down to the ash hoppers rather than up the venturi to the bed. No wear was apparent on the new first-stage cyclone. However, it was discovered that the outlet tube on the second-stage cyclone was too long and may have been responsible for considerable dust reentrainment. Accordingly, this outlet tube was shortened.

To help evaluate the cyclone performance, double-Y sight ports and knife gate valves with nitrogen-blast connections were installed at the highest point on each cyclone dipleg. A redesigned 1-1/2 inch outlet dust reinjection J-valve, complete with four metered nitrogen purges, was also installed.

Test 25

After bringing the reactor up to the steady-state temperature of 1850°F to 1900°F for a 16-hour period, all dust (first- and second-stage cyclones) was intentionally bypassed into collecting drums, which were weighed and sampled. Dust was collected, 180 lb/hr and 10 lb/hr of dust, respectively, from the first- and second-stage dust collectors. The particle size of the second-stage dust was significantly finer than the first-stage dust.

First- and second-stage cyclone dust was then recycled to the J-valve reinjector. However, the 450 lb/hr coal feed rate before fines reinjection could not be reduced to maintain the same bed level after the fines were reinjected. Approximately 50 lb/hr of the dust appeared in the ash discharge. By continuous monitoring, a first-stage dipleg height of approximately 15 feet (3 feet above the calculated level required for a seal) was maintained;

still the dust loss was not reduced. A mass balance conducted over a 16-hour period showed a loss of 127 lb/hr of material.

After the test, Dr. F. A. Zenz was consulted on observation of fines return to the reactor. Based on the experiments in Test 25, fines must be hung up in the diplegs, and additional standpipe height would be helpful. As a result, experiments were conducted to see if dust could be allowed to build up in a fluidized standpipe all the way down to the point where the fines are returned to the venturi throat. This gave another 12 feet of seal height to ensure against any backblowing of gas up the dipleg. The modified J-valve configuration, found to be successful in previous tests, was used.

Test 26

Reactor start-up was readily achieved within 24 hours. Relocating the dipleg butterfly control 12 feet lower considerably stabilized the control of the cyclone dipleg height. There was a tremendous reduction in the stack-dust loss. Preliminary calculations showed that the dust loss from the system was reduced from about 200 lb/hr to less than 30 lb/hr. This reflected in a substantially reduced coal feed rate necessary to maintain the same bed level.

With the larger dust load that had to be reinjected, a considerable amount was not reentrained into the gasifier, but dropped into the discharge hopper. Fifty pounds of dust per hour and 30 pounds of agglomerates per hour were discharged.

With the large dust return, the phenomenon of a cyclically fluctuating bed temperature was also experienced for the first time. This temperature cycling could be related to a mechanical malfunction in the gas quencher. However, the quencher water input rate also fluctuated on the same cycle, with the flow increasing with a falling bed temperature. The quencher was disassembled before the test, but no sign of damage or cause for malfunctioning could be found. Therefore, the temperature fluctuations had to be related to the changing dust inventory in the bed.

Dr. F. A. Zenz was again consulted about dust reinjection. The concept of air conveying was adapted to ensure that the dust is well dispersed at the point of its introduction to the venturi process-gas stream. In addition, the reinjection nozzle was modified to ensure an upward velocity component to this dispersed dust at this same juncture.

Test 27

Again, the reactor was started up with no difficulty and autogenous operation achieved. However, as bed temperatures reached 1600°F, the temperature cycling experienced in Test 26 became evident. The air-conveying method of dust reinjection worked satisfactorily so that no dust appeared in the material discharged down through the venturi, and the gas effluent from the system was as clear as any ever experienced in this test program.

Operation continued with and without dust reinjection as well as with a reduced superficial bed velocity in an effort to reduce the temperature cycling and to increase the bed temperature to initiate ash agglomeration. Complete removal of dust from the system quickly led to a temperature rise and agglomerate production. Dust reinjection (recirculation) resulted in reduced and cycling bed temperatures and a gradual cessation of ash-agglomerate production.

More heat had to be added to the reactor to make up for the extra heat load created by quenching and reheating the dust inventory. The circulating dust was cooled to about 500°F before being reinjected, and this amount of dust had to be reheated to the reactor temperature each time it circulated. The system was modified slightly to allow the injection of a small amount of oxygen to enrich the air, without significantly increasing the volumetric flow of gas through the reactor. This provided the extra heat without increasing the fluidizing velocity. In addition, a properly designed cyclone was installed to replace the makeshift second-stage cyclone.

Table 27 is a mass balance summary for the four different conditions used in Test 27 (two superficial velocities with and without dust reinjection). Three conditions show good mass balance, but the fourth shows a 22% imbalance. This last state of operation was maintained only for approximately 8 hours, perhaps not long enough for a stable system to be achieved. This last operating condition has the most severe dust recirculation rate. Preliminary calculations, based on quench-water requirements with and without dust removal, indicate that when dust was recirculated at a superficial velocity of 2.25 ft/s, its rate in the gasifier may be as high as 2000 lb/hr.

Table 27. MASS BALANCE FOR FOUR DIFFERENT RUN CONDITIONS

<u>Superficial Velocity, ft/s</u>	<u>Carbon Gasified</u>	<u>Bed Accumu- lation</u>	<u>Ash Discharge</u>	<u>Dust Removed</u>	<u>Total Output</u>	<u>Feed Rate</u>
			lb/hr			
2.5	276	--	40	201	517	505
2.25	174	50	25	--	249	320
2.0	228	-30	27	152	377	377
1.9	131	122	22	--	275	276

Test 28

This test was the first using air enriched with oxygen to see how much enrichment was required to make up for the heat load presented by the circulating inventory of fines. Molal ratios equivalent to 35% oxygen and 65% nitrogen were used. Because the test was interrupted twice, once when the coal ran out and a second time when the coal-feeder motor burned out, there was only a short time to operate with enriched air. The results were not conclusive. The enriched air was fed only to the venturi of the reactor. A 12% oxygen content was still maintained at the grid by raising the steam concentration to compensate for the added oxygen.

Oxygen was added following a relatively steady 12-hour period of total dust (first- and second-stage cyclones) reinjection to the venturi. During this 12-hour period, the bed temperatures cycled between 1500°F and 1730°F. During the 8-hour period when oxygen was added, air enrichment to 25% oxygen (versus standard air, 21% oxygen) was tried, to 30% oxygen 4 hours later, and to 35% oxygen for a final 2-hour period. No agglomerates were observed during this 8-hour period, yet a gradual increase in bed temperature was noted so that its temperature fluctuations ranged from 1650°F to 1750°F accompanied by a small increase in carbon monoxide and hydrogen production, basically from 4% to 6% carbon monoxide and from 0% to 2% hydrogen.

Test 29

Twelve hours after autogenous operation was attained, agglomerates began appearing in the venturi discharge material as all first-stage cyclone dust was removed.

After a steady 12 hours of agglomerates production, dust recirculation was begun, and simultaneously switched to 30% oxygen-enriched air at the venturi. As in Test 28, 12% oxygen was maintained at the grid by a corresponding steam increase in this stream. For the next 26 hours, agglomerates, roughly 1/4-inch in size and containing a minimal amount of dust, were produced. For the first 4 hours after the above change, the bed temperature remained near 1900°F, and the dry product-gas analyzers showed 16.5% hydrogen and 24.5% carbon monoxide. Thereafter, the bed temperature began falling and an increase in the quench-water rate was noted. These higher rates always prevailed when dust was recirculated in Tests 27 and 28 signaling that the dust-solids recirculation rate was increasing. Apparently, the dust was not being eliminated in the bed or venturi as fast as it was formed. The production of dust-free agglomerates continued for the next 12 hours, but the hydrogen output dropped to an average 2.5% and the carbon monoxide to 7% as the bed temperature settled near 1750°F.

A final change to 35% oxygen at the venturi was initiated, still holding 12% at the grid, by first withdrawing dust for only 45 minutes to raise the bed temperature to 1935°F. The final 5 hours of this test indicated that the bed temperature was falling and that carbon monoxide and hydrogen production was off. Yet agglomerates still continued to be produced.

An attempt was made to calculate what oxygen content is necessary for the system to sustain the 1900°F temperature required to yield a gas that has a high percentage of carbon monoxide and hydrogen and to completely consume all reinjected dust at the same rate that it is emitted from the bed. Test 29 was the most successful test to date. A good material balance was achieved, and the agglomerates were uniform and had a very smooth surface.

Following Test 29, operations were suspended while the process-air superheater was replaced. This heater had several tubes that failed and has been a source of problems in the past. An investigation of sources of different feed materials for the ash-agglomeration gasifier continued and FMC char was used as the next feed.

Table 28 presents heat and material balances for Test 29 made in December 1974. All are for periods when agglomerates were being discharged. During two periods, all dust was withdrawn and only air and steam were used for gasification. The other two periods used 30% and 35% oxygen at the venturi simultaneously with complete dust reinjection. Both dust reinjection periods revealed a material loss from the system that had to occur at the second-stage dust collector. Because there were no means to measure the dust recirculation rate to the venturi, the last two entries on the table show it calculated by two methods. The block diagram, Figure 50, covers the 4-hour period when 30% oxygen was used, when conditions were relatively stable.

Test 30

Calculations indicated that a concentration of 40% oxygen and 60% nitrogen feed to the venturi at a volume sufficient to yield a velocity of 96 ft/s should provide sufficient heat to maintain a 1900°F bed temperature. This condition should give a high carbon monoxide yield and, hopefully, the combustion of the recycled dust to agglomerates. This test was brought to the agglomerating state readily within 12 hours of autogenous operation by the regular procedure of removing dust from the first-stage cyclone as fast as it was collected.

Five hours after starting Test 30, oxygen addition was begun using a 40% concentration at the central venturi and the standard 12% at the grid. This oxygen feed was maintained for 5 hours; however, the recirculating dust load (which was reinjected at the same time) slowly increased, and the bed temperature began cycling to as low as 1850°F. When the required quench-water rate had tripled in quantity (indicating a high recirculating dust load), dust was again withdrawn and air-steam operation resumed to reestablish stable conditions and raise the bed temperature. Upon seeing "conglomerates" (clusters of agglomerates) in the discharged ash, oxygen and the dust reintroduction was delayed until they were no longer present.

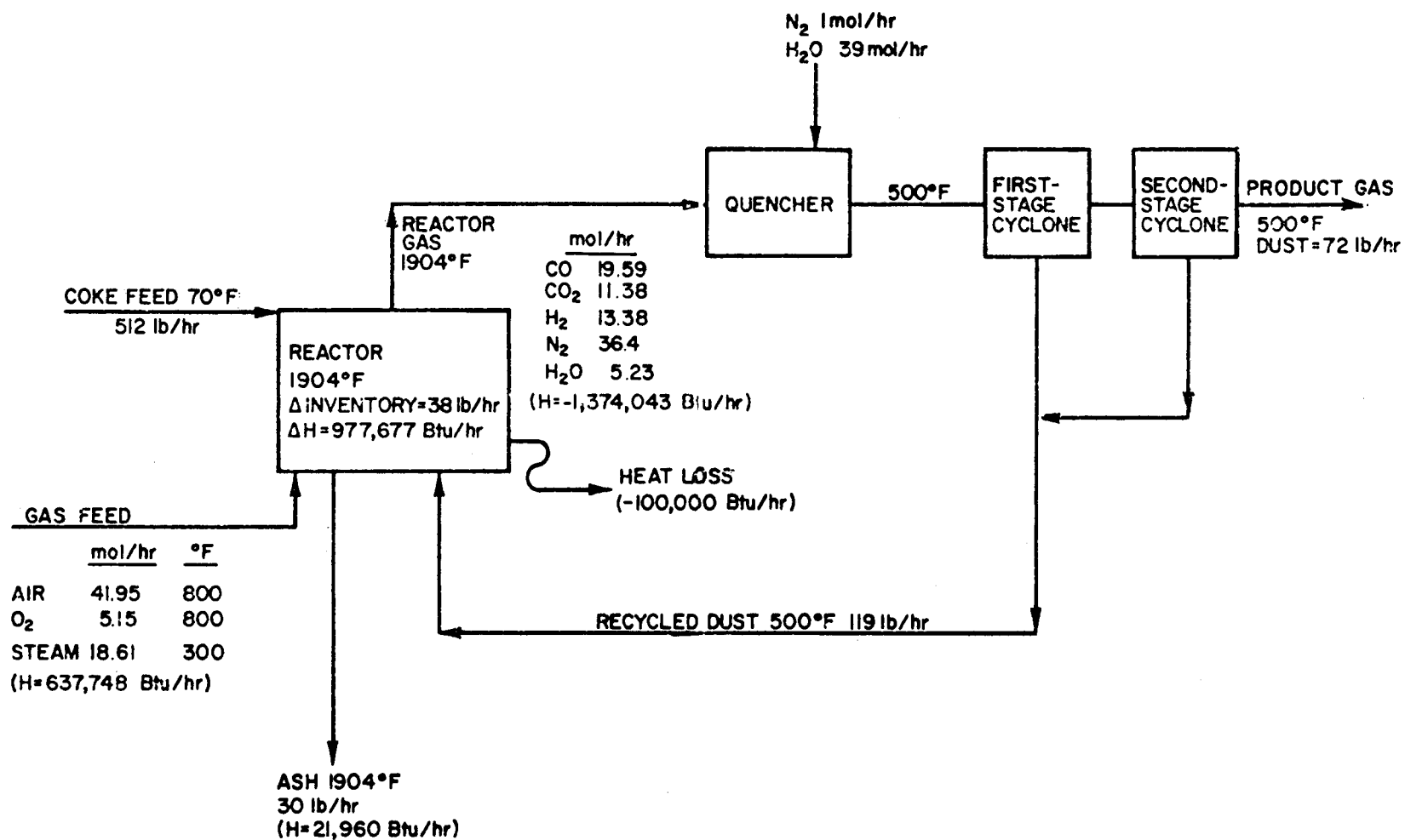
Next a 45% oxygen mixture was used at the venturi, still retaining 12% oxygen at the grid. For the first time, a bed temperature near 1900°F could be maintained. This period lasted 8 hours, and the bed temperature averaged 1905°F, with approximately 20°F deviation. The test was terminated by a blocked venturi section that forced a premature shutdown because the ash could not be discharged.

When the reactor was opened, a large clinker that had formed over the entire grid area immediately above the venturi section was discovered; part of it extended down into the venturi throat. Preignition of the recycle dust by the 45% oxygen-enriched air that was used to convey the dust into the venturi area caused this clinker formation. The method of dust reinjection was changed, from enriched air to steam.

Material and heat balances calculated for Test 30 are given in Table 29 and Figure 51. The period covered in this test includes the use of oxygen at 40% concentration at the venturi.

Table 28. HEAT AND MATERIAL BALANCES FOR ASH-AGGLOMERATING
GASIFIER TEST 29

Date	12/12/74	12/12/74	12/12/74	12/13/74
Time Period	0200- 0600	0800- 1200	1600- 2000	0400- 0800
Bed Temperature, TE 9206-6, °F	1920	1890	1904	1760
Bed Height, ft	3.4	3.82	3.91	4.0
Air Flow Through Grid, mol/hr	24.82	17.58	12.06	12.06
Air Flow Through Venturi, mol/hr	24.48	23.27	23.4	23.44
Air Flow With Recycled Dust, mol/hr	7.93	8.79	9.23	8.17
Total Air to the Reactor, mol/hr	57.23	49.64	41.95	41.37
Additional O ₂ to the Reactor, mol/hr	0	0	5.15	5.62
Steam to the Grid, mol/hr	18.61	13.33	18.61	18.61
N ₂ Purge to the Reactor, mol/hr	3.26	3.26	3.26	3.26
Coke Feed Rate, lb/hr	461	512	512	310
Ash Discharge Rate, lb/hr	20	50	30	34
Reactor Gas, mol/hr				
CO	12.45	12.23	19.59	4.73
CO ₂	10.61	8.99	11.38	12.81
H ₂	8.84	8.27	13.38	2.22
N ₂	48.47	42.47	35.4	35.94
H ₂ O	9.77	5.06	5.23	16.39
Bed Density, lb/cu ft	30.27	31.70	32.86	43.24
Increase in Bed Inventory, lb/hr	23	48	38	0
Rate of Carbon Combustion, lb/hr	277	255	394	211
Rate of First-Stage Dust Collection, lb/hr	197	157	0	0
Unaccounted Material Loss, lb/hr	-56	2	72	65
Enthalpy of Inlet Gases, Btu/hr	690,407	540,356	637,748	637,259
Enthalpy of Reactor Gas, Btu/hr	1,564,876	1,231,773	1,374,043	1,359,571
Wall Losses, Btu/hr	100,000	100,000	100,000	100,000
Heat of Reaction, Btu/hr	1,163,547	908,483	977,677	1,950,291
Enthalpy of Discharged Ash, Btu/hr	16,560	36,400	21,960	23,120
Enthalpy of Unaccounted Dust Loss + Cyclone, Btu/hr	104,340	115,752	52,700	44,200
Unaccounted Heat Loss, Btu/hr	68,178	-35,000	66,722	1,084,849
Dust Recycle Rate Based on Unaccounted Heat, lb/hr			119	2150
Dust Recycle Rate Based on Heat Balance Across the Quencher, lb/hr			132	1830



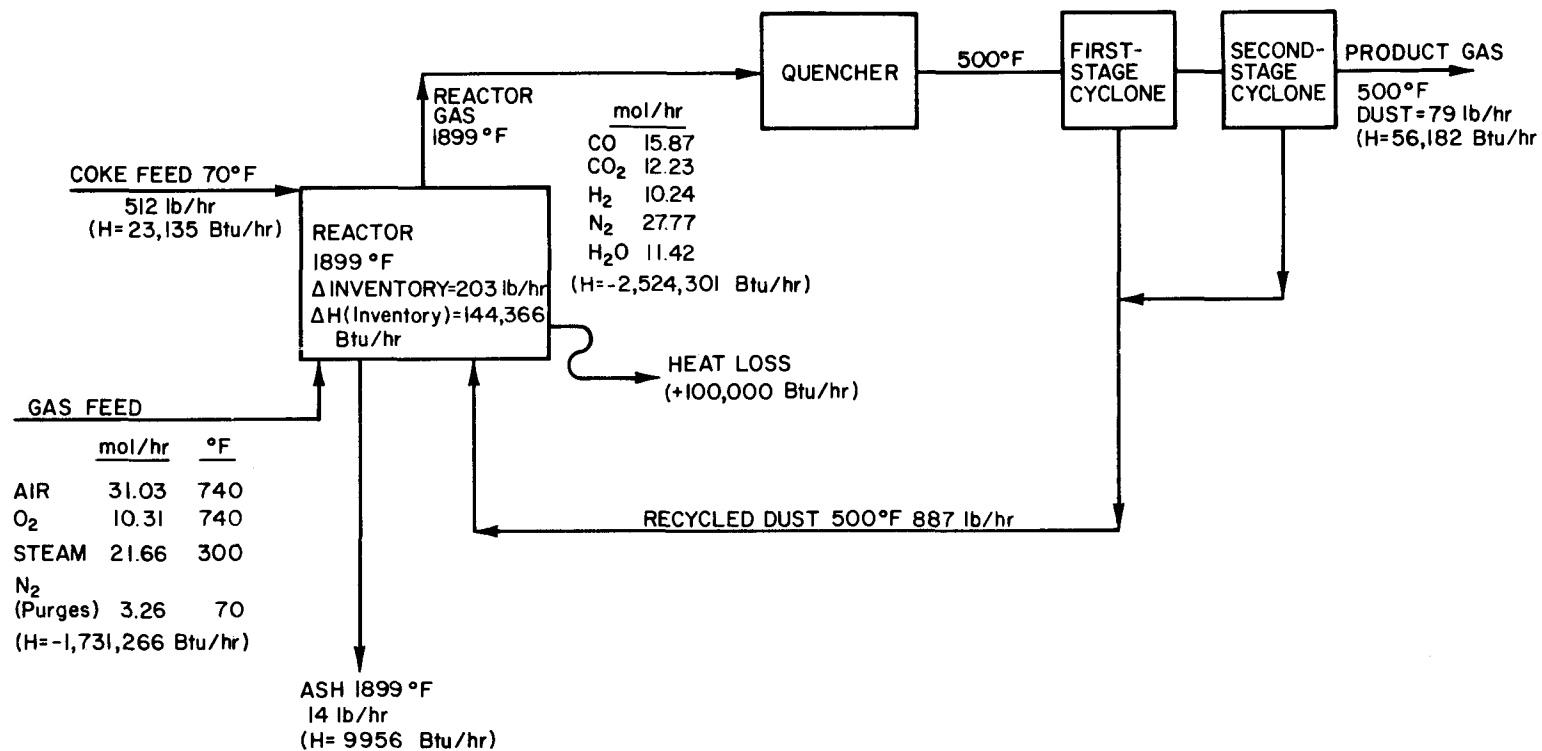
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Figure 50. ASH-AGGLOMERATING GASIFIER

Table 29. HEAT AND MATERIAL BALANCES FOR ASH-AGGLOMERATING
GASIFIER TEST 30

Date	1/9/75	
Time Period	10 am-1 pm	
Bed Temperature, TE 9206-6, °F	1899	
Bed Height, ft	3.81	
Bed Density, lb/cu ft	35.02	
Total Air to Reactor, FR 995, at 740°F, mol/hr	31.03	
Total Oxygen to Reactor, FR 938, at 70°F, mol/hr	10.31	
Total Steam to Reactor, FR 903, at 300°F, mol/hr	21.66	
Total Nitrogen Purge to Reactor at 70°F, mol/hr	3	
Coke Feed Rate, lb/hr	633	
Ash Discharge Rate, lb/hr	14	
Increase in Bed Inventory, lb/hr	203	
Reactor Gas	<u>% Dry</u>	<u>mol/hr</u>
CO	24.0	15.87
CO ₂	18.5	12.23
H ₂	15.5	10.24
N ₂	42.0	27.77
H ₂ O	--	11.42
Rate of Carbon Combustion, lb/hr	337	
Rate of First-Stage Dust Collection, lb/hr	0	
Unaccounted Material Loss, lb/hr	79	
Enthalpy of Input Materials, Btu/hr	-1,708,091*	
Enthalpy of Reactor Gas, Btu/hr	-2,524,301*	
Wall Losses, Btu/hr	100,000	
Enthalpy Change of Increased Bed Inventory, Btu/hr	+144,366	
Enthalpy of Discharged Ash, Btu/hr	9,956	
Enthalpy of Unaccounted Material Loss, Btu/hr	56,182	
Unaccounted Heat Loss, Btu/hr	505,706	
Dust Recycle Rate Based on Unaccounted Heat, lb/hr	887	

* Gas enthalpies relative to elements at 0 K from "Physical and Thermodynamic Properties of Elements and Compounds," Chemetron Corp., 1969. All solid enthalpies taken as solid graphite.



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Figure 51. ASH-AGGLOMERATING GASIFIER SYSTEM MATERIAL AND ENERGY BALANCE FOR TEST 30

Test 31

For this test, a monitor was operating to determine the second-stage dipleg depth. Changes in the cycle time of the Gemco dust air locks have also been made to enable the second-stage dust dipleg level to be controlled. Agglomerate formation began on January 22 and continued smoothly until January 23. Forty-five percent oxygen-enriched air was used in the venturi throat, and the temperature was steadily maintained between 1800°F and 1900°F throughout the test, accompanied by the steady production of agglomerates. The balance point has been reached in the addition of oxygen to account for the additional heat load presented by the reheating of recycled dust. Operation was conducted with a total recycle of the dust.

On January 23, while switching the emergency generator in the HYGAS plant back on-line following overhaul maintenance work, the air blower in the ash-agglomerating gasifier section was shut off. This was anticipated, and it was restarted within seconds. However, despite the precaution taken to switch back to air from enriched air, a procedure used successfully before, this time the momentarily slumped bed could not be refluidized. Therefore, the test was terminated and the reactor cooled down.

Test 32

In this test, the intention was to operate the gasifier at an average bed temperature of 1900°F using up to 40% enriched air (40% oxygen, 60% nitrogen) at the venturi as required to agglomerate the recirculated fines. Twenty-four hours after attaining autogenous operation, no agglomerates were observed in the venturi discharge material while operating with air. It was decided to promote agglomerate growth by enriching the air to the venturi while continuing to withdraw dust from the cyclone and not recycle it. This procedure, using 28.5% oxygen at the venturi and 15% at the grid, immediately raised the bed temperature by 60°F to 1920°F. During a 3-1/2 hour period, the dust removal rate rose 50% to 170 lb/hr. When agglomerates began to appear, dust recirculation was instituted simultaneously while raising the oxygen concentration at the venturi to 35%. Although the bed temperature remained above 1900°F for 6 hours, the dust recirculation rate, as noted by the quench-water flow, continued to increase. Because of a malfunction of the dust return system, a large amount of dust fell out with the ash discharge and was removed from the system instead of being recycled. The bed temperature also cycled over a 100°F range as the dust recirculation reached high levels. The test was ended when the feed material ran out. Because of the dust loss, there was trouble in measuring the true ash downflow rate, and consequently, enough ash may not have been discharged. The clinker, found in the reactor after cooling, was attributed to the collection of bed-ash residue.

Test 33

Knowing that the reactivity of the Bethlehem coke breeze that has been used is low compared with that of char and that a significant amount of this coke is larger than 10 mesh, this next test run was conducted using an available supply of about 14 tons of FMC char. This test run was relatively short because of the limited feed supply, and therefore, its results are qualitative. This material fluidized readily at lower superficial

velocities so that operation at 1.5 ft/s was possible. Nevertheless, the dust entrainment rate was still quite high at over 300 lb/hr compared with 200 lb/hr when withdrawing dust in the coke tests. The run was continued until the coke supply was exhausted, during which time a) the bed temperature stabilized at 1750°F using air and steam and rose to 1850°F using 30% oxygen at the venturi and 12% at the grid, b) agglomerates never appeared although the venturi discharge material did become brown after adding the oxygen, and c) the added oxygen did raise the dust rate to 400 lb/hr.

Test 34

The feed for this test was the -6 mesh Bethlehem coke breeze. It was operated at 1.5 ft/s superficial velocity to reduce fines carry-over. Twelve hours after autogenous operation was reached, the bed temperature had not exceeded 1870°F, and there was no evidence of agglomerate formation. Accordingly, the venturi gas was enriched to 35% oxygen and the grid mixture allowed to rise to 15% oxygen. Within a 2 to 4-hour period following this change, the bed temperature rose to 1920°F and then to 1940°F. However, twice during this same period upward excursions in the hydrogen, carbon monoxide, and product-gas flow were experienced simultaneously with a reduction in reactor bed temperature. These two phenomena lasted approximately 15 minutes each.

For the balance of this run (30 hours), operation continued with 80 ft/s and 30% oxygen at the venturi and 1.4 to 1.5 ft/s overall superficial velocity. During most of this period, the bed temperature stayed above 1850°F and was slowly rising when the test was terminated because of the inability to sustain a continuous discharge from the central venturi. Only the beginnings of agglomerate formation were evident at the test's termination.

Upon opening the reactor, there was a large bed of coke with numerous agglomerates overlaying a large clinkered mass. It was concluded that the recorded excursions of reactor gas composition, gas flow, and bed temperature had signaled clinker formation. This development occurred shortly after introducing oxygen to the reactor. A decision was made to confirm with Test 35 (using only air) whether agglomerates could be produced at these lower superficial velocities and in what form.

Complete data sheets (except for analytical results) for Test 34 and the corresponding heat and mass balances for selected periods are given in Table 30 and Figures 52 and 53.

Test 35

Upon reaching autogenous operation, Test 35 continued for approximately 54 hours. Although agglomerates were produced and their presence was quite evident during the last 12 hours of the test, the samples were never quite free of fines or carbon. To encourage agglomerate formation, the residue discharge rate was kept below the calculated ash input rate, so as not to deplete the ash content of the bed. To achieve this, at a residue discharge rate of about 20 lb/hr, it was necessary to operate at venturi velocities of up to 150 ft/s.

Table 30, Part 1. ASH-AGGLOMERATING GASIFIER DATA SUMMARY FOR TEST 34

Date	2/26/75							
Time	0000	0200	0400	0600	0800	1000	1200	1400
TE 9206.6								
Bed Temperature, °F	1759	1815	1858	1865	1864	1926	1941	1927
TE 9206.18								
Outlet Gas Temp Process Gas Heater, °F	768	765	772	764	765	758	754	762
TE 9206.1								
Gas Temp Entering Venturi, °F	630	570	624	613	623	848	692	502
TE 9206.2								
Gas Temp Entering Grid, °F	488	490	487	492	487	352	348	347
PI 913 & PDR 910								
Pressure in Venturi, psig	3.1	3.1	3.0	3.1	3.0	3.0	3.1	2.8
PI 905								
Pressure at FE 905 & FE 906, psig	13.6	13.8	13.5	13.5	11.4	12.3	12.3	12.5
FI 909								
Air/Stm Flow With Dust Return, lb/hr	290	290	290	290	290	290	290	290
FR 903 at 40 psig & 300°F								
Steam Flow to Grid, lb/hr	220	235	235	235	235	430	430	430
FR 905								
Enriched Air to Grid, lb/hr	595	606	606	606	573	276	276	276
FR 906								
Enriched Air to Venturi, lb/hr	184	206	206	206	198	207	207	117
FR 995								
Total 21% Air to Process Gas Heater, lb/hr	771	805	805	810	779	247	247	227
FR 938								
Total Oxygen to Process Gas Heater, lb/hr	0	0	0	0	0	286	280	228
Nitrogen Purge to Reactor, lb/hr	102	102	102	102	102	102	102	102
FR 918								
Rate of Quench Water, lb/hr	120	180	260	280	300	400	430	400
PDR 912/PDR 913 X 3.33								
Bed Height, ft	4.22	4.22	3.81	3.93	4.14	4.37	4.13	3.9
PDR 913 X 43.3								
Bed Density, lb/cu ft	32.48	32.5	30.3	28.6	28.6	27.7	25.1	25.1
Coke Feed Rate, lb/hr (average)	333	333	333	333	410	410	410	410
Reactor Gas Composition, % (moisture-free)								
CO ₂ (ANR 975)	16.7	16.2	15.5	15.0	14.0	20.0	13.0	19.2
CO (ANR 977)	8.0	10.0	12.0	12.5	13.5	25.7	35.5	24.5
H ₂ (ANR 981)	7.0	8.7	10.5	11.0	10.7	22.0	26.7	22.5
O ₂ (ANR 978)	0	0	0	0	0	0	0	0
N ₂ (By Difference)	68.3	65.1	62.0	61.5	61.8	32.3	24.8	33.8
1st-Stage Dust Removal Rate, lb/hr	125	125	125	125	125	125	125	125
2nd-Stage Dust Removal Rate, lb/hr	11	11	11	11	11	11	11	11
C-4 Discharge Rate of Ash, lb/hr	82	57	52	47	45	15	2	3
Feed Composition:								
%C								85.4
%Ash								11.7
Ash Composition:								
%C								
%Ash								
TE 9206.16								
Gas Temp Entering Quencher, °F	942	1079	1216	1281	1342	1398	1456	1461
PIC 901								
Blower Outlet Pressure, psig	14.3	14.3	14.3	14.6	12.2	12.2	12.2	12.1
TI 9203								
Blower Outlet Temperature, °F	250	250	247	245	218	200	199	195
PI 9122								
Oxygen Supply Pressure, psig	--	--	--	--	--	44	42	45
TE 9206.4								
Oxygen Supply Temperature, °F	--	--	--	--	--	45	48	49

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Table 30, Part 2. ASH-AGGLOMERATING GASIFIER DATA SUMMARY FOR TEST 34

Date	2/26/75					2/27/75	
Time	1600	1800	2000	2200	2400	0200	0400
TE 9206.6							
Bed Temperature, °F	1885	1866	1865	1878	1886	1884	1726
TE 9206.18							
Outlet Gas Temp Process Gas Heater, °F	770	764	765	771	773	769	769
TE 9206.1							
Gas Temp Entering Venturi, °F	513	699	792	897	962	988	692
TE 9206.2							
Gas Temp Entering Grid, °F	411	424	424	425	423	424	423
PI 913 & PDR 910							
Pressure in Venturi, psig	2.5	2.5	2.55	2.55	2.55	2.5	2.5
PI 905							
Pressure at FE 905 & FE 906, psig	10.6	10.7	10.6	10.7	10.6	10.8	10.4
FI 909							
Air/Stm Flow With Dust Return, lb/hr	0	0	0	0	0	0	0
FR 903 at 40 psig & 300°F							
Steam Flow to Grid, lb/hr	335	370	370	370	370	370	370
FR 905							
Enriched Air to Grid, lb/hr	420	471	471	471	471	471	471
FR 906							
Enriched Air to Venturi, lb/hr	251	195	195	195	195	195	195
FR 995							
Total 21% Air to Process Gas Heater, lb/hr	558	558	558	558	543	543	661
FR 938							
Total Oxygen to Process Gas Heater, lb/hr	138	134	134	134	134	134	0
Nitrogen Purge to Reactor, lb/hr	102	102	102	102	102	102	102
FR 918							
Rate of Quench Water, lb/hr	300	360	280	380	360	360	300
PDR 912/PDR 913 X 3.33							
Bed Height, ft	4.05	4.18	4.27	4.27	4.16	4.23	4.35
PDR 913 X 43.3							
Bed Density, lb/cu ft	26	27.3	27.7	27.7	27.7	27.3	28.1
Coke Feed Rate, lb/hr (average)	364	364	364	364	364	364	360
Reactor Gas Composition, % (moisture-free)							
CO ₂ (ANR 975)	17.7	18.7	18.7	18.7	25.5	24.2	22.0
CO (ANR 977)	20.7	18.5	18.5	19.5	25.7	24.5	10.7
H ₂ (ANR 981)	20.0	19.0	18.0	18.5	18.0	18.5	9.5
O ₂ (ANR 978)	0	0	0	0	0	0	0
N ₂ (By Difference)	41.6	43.8	44.8	43.3	30.8	32.8	57.8
1st-Stage Dust Removal Rate, lb/hr	125	125	125	125	125	125	125
2nd-Stage Dust Removal Rate, lb/hr	5	5	5	5	5	5	5
C-4 Discharge Rate of Ash, lb/hr	5	25	30	33	40	53	73
Feed Composition: %C				86.3			85.1
%Ash				11.0			12.4
Ash Composition: %C	51.7		59.3		60.0		67.8
%Ash	47.9		39.8		39.3		37.1
TE 9206.16							
Gas Temp Entering Quencher, °F	1447	1444	1449	1455	1461	1460	1425
PIC 901							
Blower Outlet Pressure, psig	11.2	11.2	11.2	11.2	11.4	11.4	11.4
TI 9203							
Blower Outlet Temperature, °F	200	200	195	190	190	185	187
PI 9122							
Oxygen Supply Pressure, psig	38.6	38.7	40	40.5	41.3	42	--
TE 9206.4							
Oxygen Supply Temperature, °F	51	53	48	45	42	40	77

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Table 30, Part 3. ASH-AGGLOMERATING GASIFIER DATA SUMMARY FOR TEST 34

Date	2/27/75						
Time	0600	0800	1000	1200	1400	1600	1800
TE 9206.6 Bed Temperature, °F	1841	1859	1870	1885	1899	1921	1885
TE 9206.18 Outlet Gas Temp Process Gas Heater, °F	772	778	775	773	765	764	764
TE 9206.1 Gas Temp Entering Venturi, °F	704	779	601	569	512	508	453
TE 9206.2 Gas Temp Entering Grid, °F	463	465	467	467	463	451	468
PI 913 & PDR 910 Pressure in Venturi, psig	2.8	2.8	2.8	3.0	3.1	3.0	2.7
PI 905 Pressure at FE 905 & FE 906, psig	9.7	10.3	10.5	10.0	9.9	9.9	9.9
FI 909 Air/Stm Flow With Dust Return, lb/hr	0	0	0	0	0	0	0
FR 903 at 40 psig & 300°F Steam Flow to Grid, lb/hr	305	305	305	305	305	315	245
FR 905 Enriched Air to Grid, lb/hr	579	567	567	567	567	526	526
FR 906 Enriched Air to Venturi, lb/hr	241	243	255	304	369	331	331
FR 995 Total 21% Air to Process Gas Heater, lb/hr	707	722	722	814	855	737	843
FR 938 Total Oxygen to Process Gas Heater, lb/hr	93	82	86	85	81	147	0
Nitrogen Purge to Reactor, lb/hr	102	102	102	102	102	102	102
FR 918 Rate of Quench Water, lb/hr	340	380	340	390	420	440	340
PDR 912/PDR 913 X 3.33 Bed Height, ft	4.28	4.74	4.57	4.6	4.64	4.37	3.75
PDR 913 X 43.3 Bed Density, lb/cu ft	30.3	28.6	29	29.4	28.6	27.7	27.7
Coke Feed Rate, lb/hr (average)	360	360	360	360	360	341	341
Reactor Gas Composition, % (moisture-free)							
CO ₂ (ANR 975)	--	--	16.2	15.7	15.0	15.7	12.5
CO (ANR 977)	--	--	17.0	16.5	17.5	20.7	16.0
H ₂ (ANR 981)	--	--	16.2	14.0	14.0	17.2	14.5
O ₂ (ANR 978)	--	--	0	0	0	0	0
N ₂ (By Difference)	--	--	50.6	53.8	53.5	46.4	57.0
1st-Stage Dust Removal Rate, lb/hr	125	125	125	125	125	125	125
2nd-Stage Dust Removal Rate, lb/hr	5	5	5	5	5	5	5
C-4 Discharge Rate of Ash, lb/hr	60	46	23	20	9	8	2
Feed Composition:							
%C							
%Ash							
Ash Composition:							
%C		63.7					
%Ash		35.6					
TE 9206.16 Gas Temp Entering Quencher, °F	1414	1442	1456	1477	1500	1514	1511
PIC 901 Blower Outlet Pressure, psig	11.4	11.0	11.0	11.0	11.0	11.2	11.2
TI 9203 Blower Outlet Temperature, °F	186	184	185	200	205	205	210
PI 9122 Oxygen Supply Pressure, psig	39.5	40.3	39.5	39.5	39.5	39.8	41.5
TE 9206.4 Oxygen Supply Temperature, °F	47	51	56	67	66	62	65

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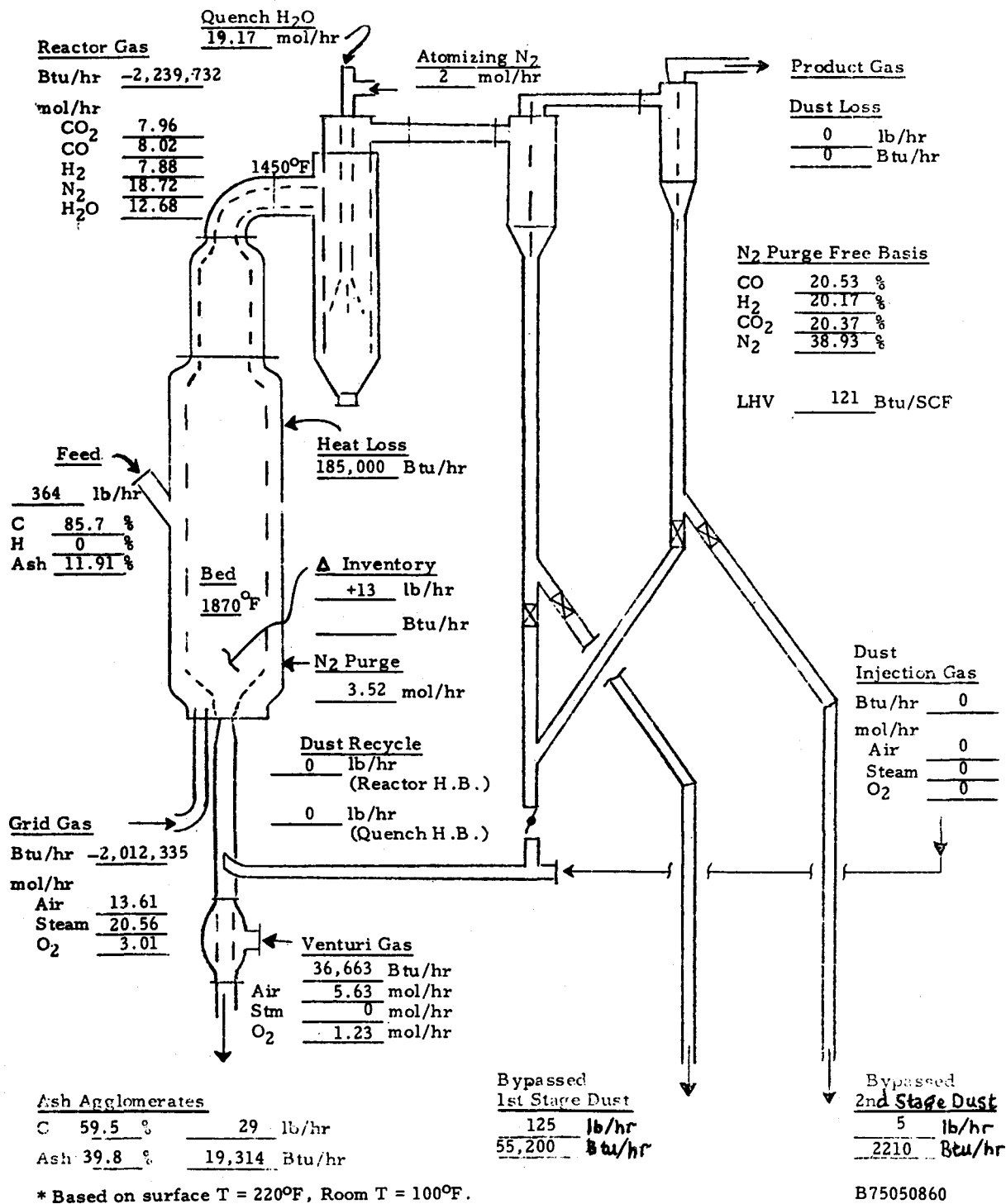


Figure 52. ASH-AGGLOMERATING GASIFICATION FOR TEST 34
(February 26, 1975; 1800 to 2200 Hours)

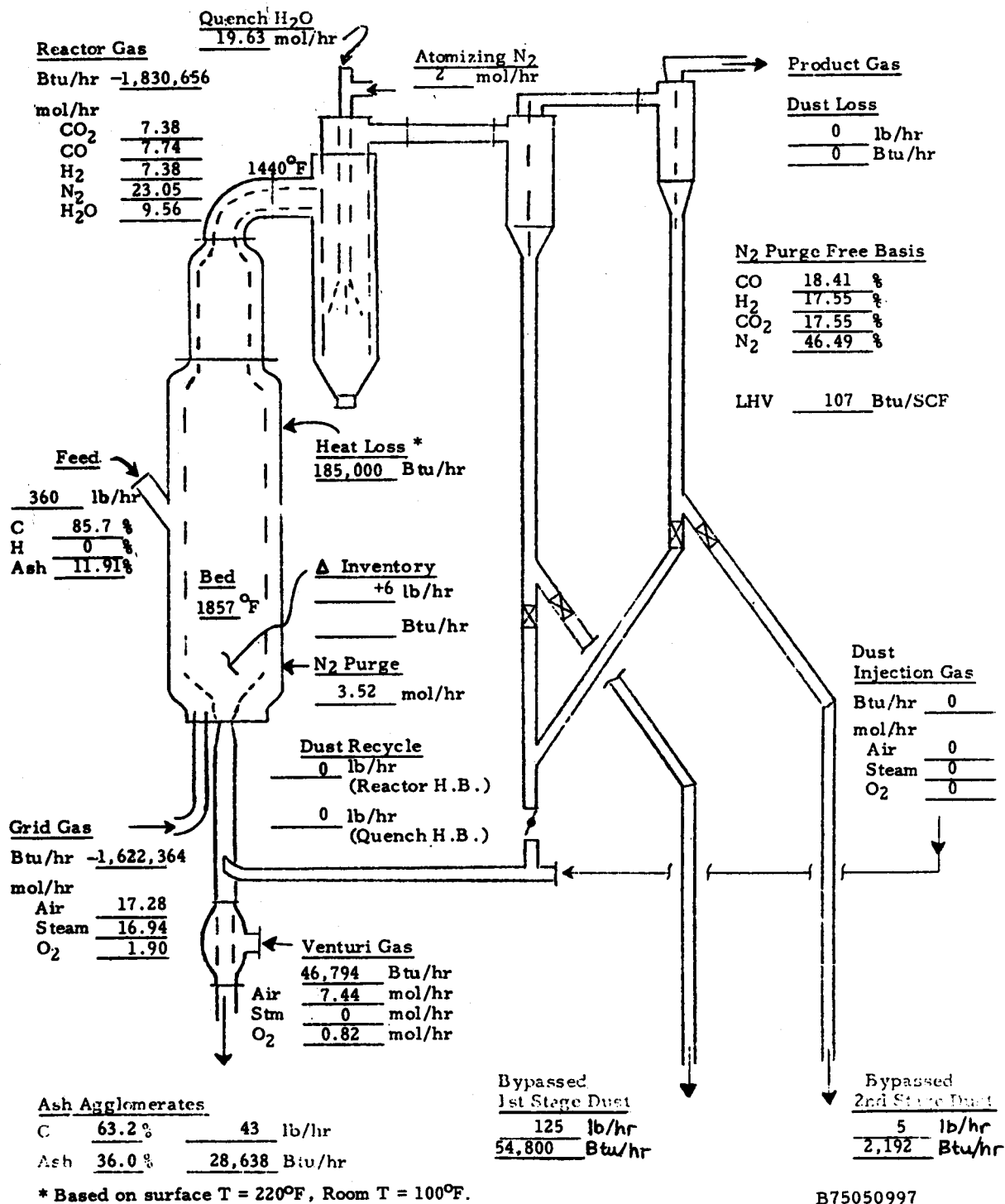


Figure 53. ASH-AGGLOMERATING GASIFICATION FOR TEST 34
(February 27, 1975; 0600 to 1000 Hours)

Two graphs, Figures 54 and 55 illustrating the fuel-gas-producing relationship between FMC char and metallurgical coke are included. Both conditions represent the use of air and sufficient steam as feed gas to the reactor. Heat and material balances for Test 35 are also included in Figures 56 through 59 and Table 31.

Test 36

After a routine start-up, including the attainment of autogenous operation, Test 36 was operated at 2.0 ft/s rather than the 1.5 ft/s superficial velocity used in Test 35. Although the bed temperature was not significantly higher than that in Test 35 (20°F), the dust elutriation rate was 50% (210 vs. 140 lb/hr) higher. Agglomerates began to appear within 16 hours after autogenous operation, and 24 hours thereafter, the discharge material was essentially all agglomerates. Operations continued this way for an additional 10 hours, producing well-defined (1/8 to 3/16-inch spheres) agglomerates at a venturi (2-inch-diameter) velocity of 160 ft/s. Then the bed depth was raised from 4 to 8 feet by increasing the feed rate. It took 12 hours to reach the 8-foot bed depth. Correspondingly, it was necessary to raise the venturi velocity to try to maintain the same ash downflow rate as that which occurred during the 4-foot-depth operation; however, because of the available pressure limit of the blower, it could not be raised sufficiently to reduce the material downflow rate to that level. Table 32 compares the 4-foot with the 8-foot bed-depth operating conditions.

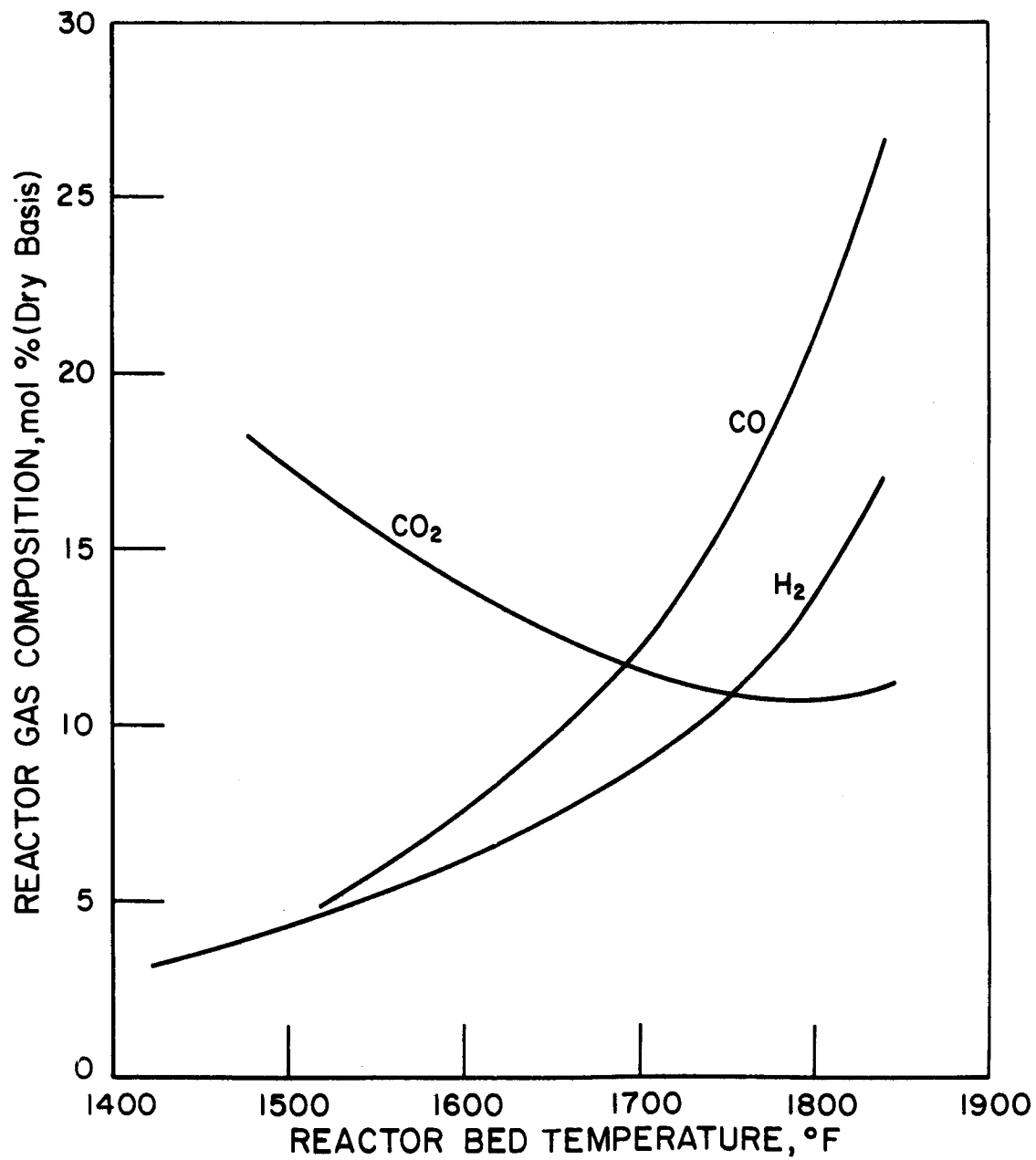
Heat and material balances are included for Test 36 in Figures 60 and 61 and Table 33. Cumulative size analyses graphs of the coke feed and cyclone dust for Test 36 are shown in Figures 62 through 66. These analyses include the subsieve-size data from a Coulter counter.

Test 37

With the successful production of agglomerates with the 2-inch venturi line and operation at an 8-foot-deep bed level in Test 36, it was time to try a test using only steam and oxygen for gasification agglomeration. Test 37 was started up in the usual manner using air. Twelve hours after attaining autoignition of the bed, the switchover to steam and oxygen was begun. The changeover was completed in 4 hours without incident and with every indication that proper fluidization prevailed. After operating for an additional 8 hours, a plug in the coke-feed chute forced a shutdown. At this time, there was still no evidence of agglomerates. The reactor screw feeder purge seal had failed, allowing high-moisture product gas to flow up the feed chute and condense and cake the entering feed coke.

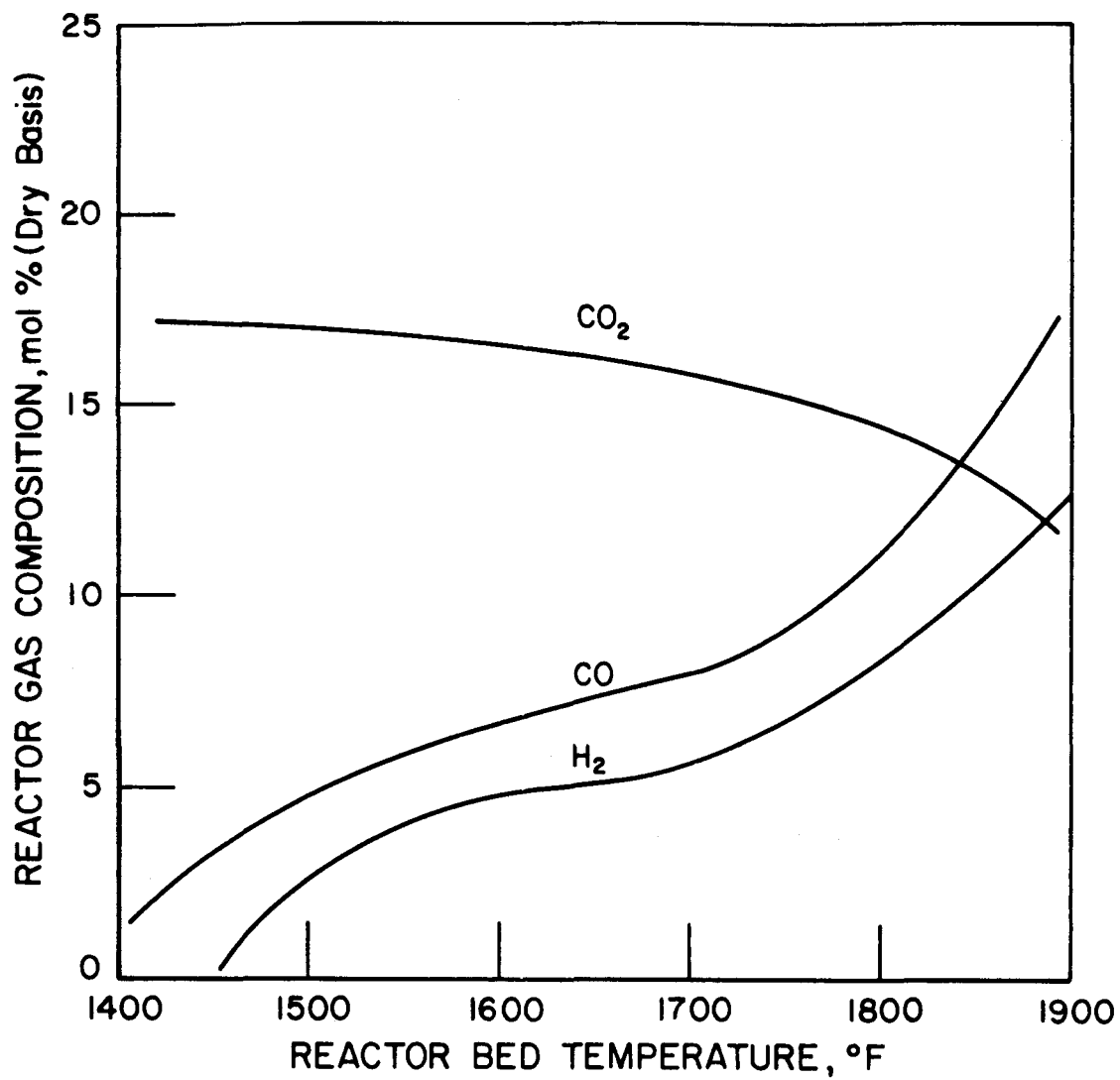
Test 38

Upon repairing the screw feeder shaft seal and making provisions to monitor the feed chute temperature, Test 38 began. Air was once again used for start-up with a 2.0 ft/s fluidization rate. Eight hours after reaching the autogenous state, the changeover was completed with the introduction of 21% oxygen-steam mixture to the gasifier; approximately 12 hours later, agglomerates appeared in the central venturi discharge. The agglomerates continued to improve in quality (growing in size) and quantity so that, 18



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Figure 54. ASH-AGGLOMERATING GASIFICATION FOR TEST 33
USING FMC CHAR FEED



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Figure 55. ASH-AGGLOMERATING GASIFICATION FOR TEST 35
USING -6 MESH METALLURGICAL COKE FEED

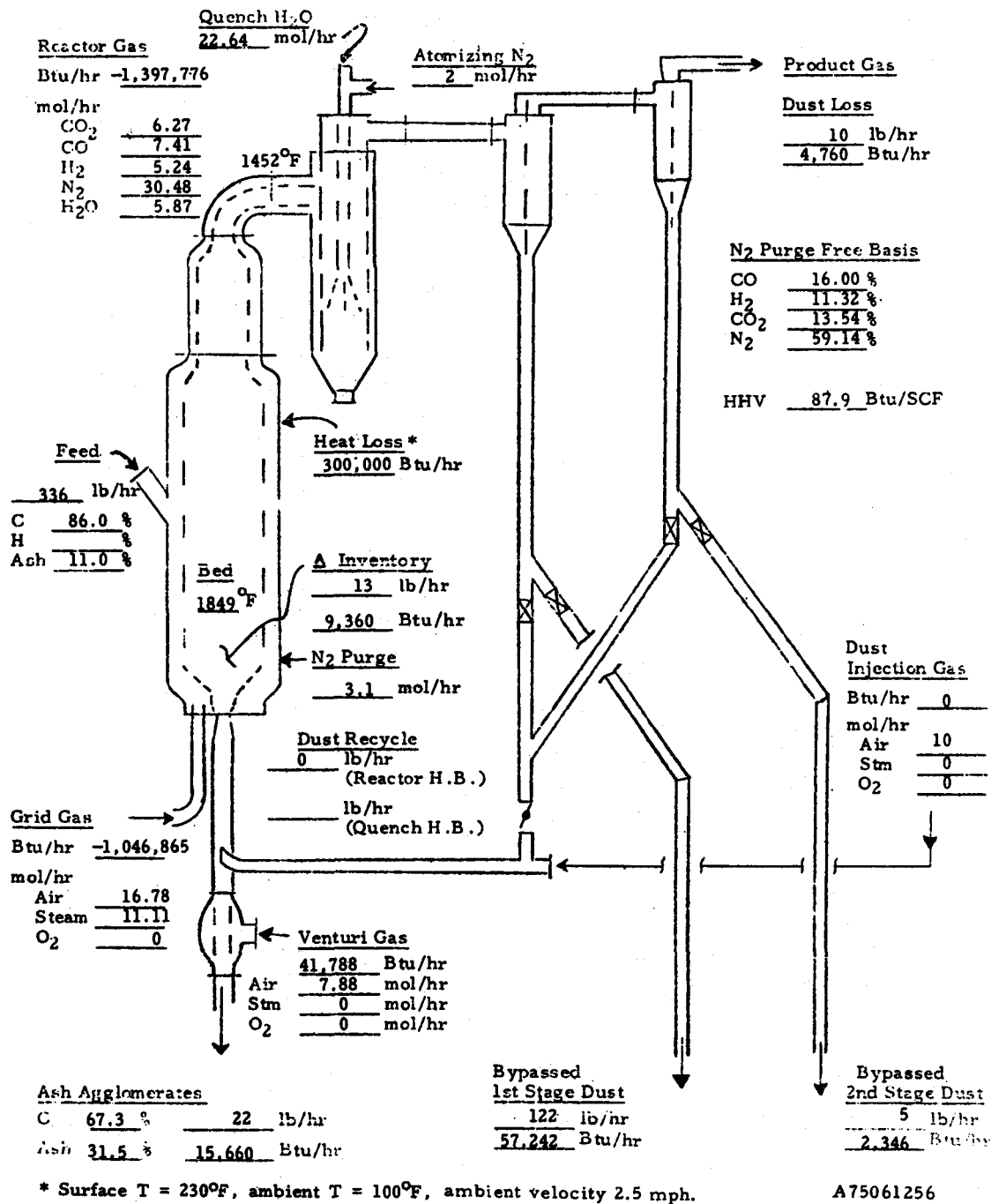


Figure 56. ASH-AGGLOMERATING GASIFICATION FOR TEST 35
 (March 11, 1975; 1600 to 2200 Hours)

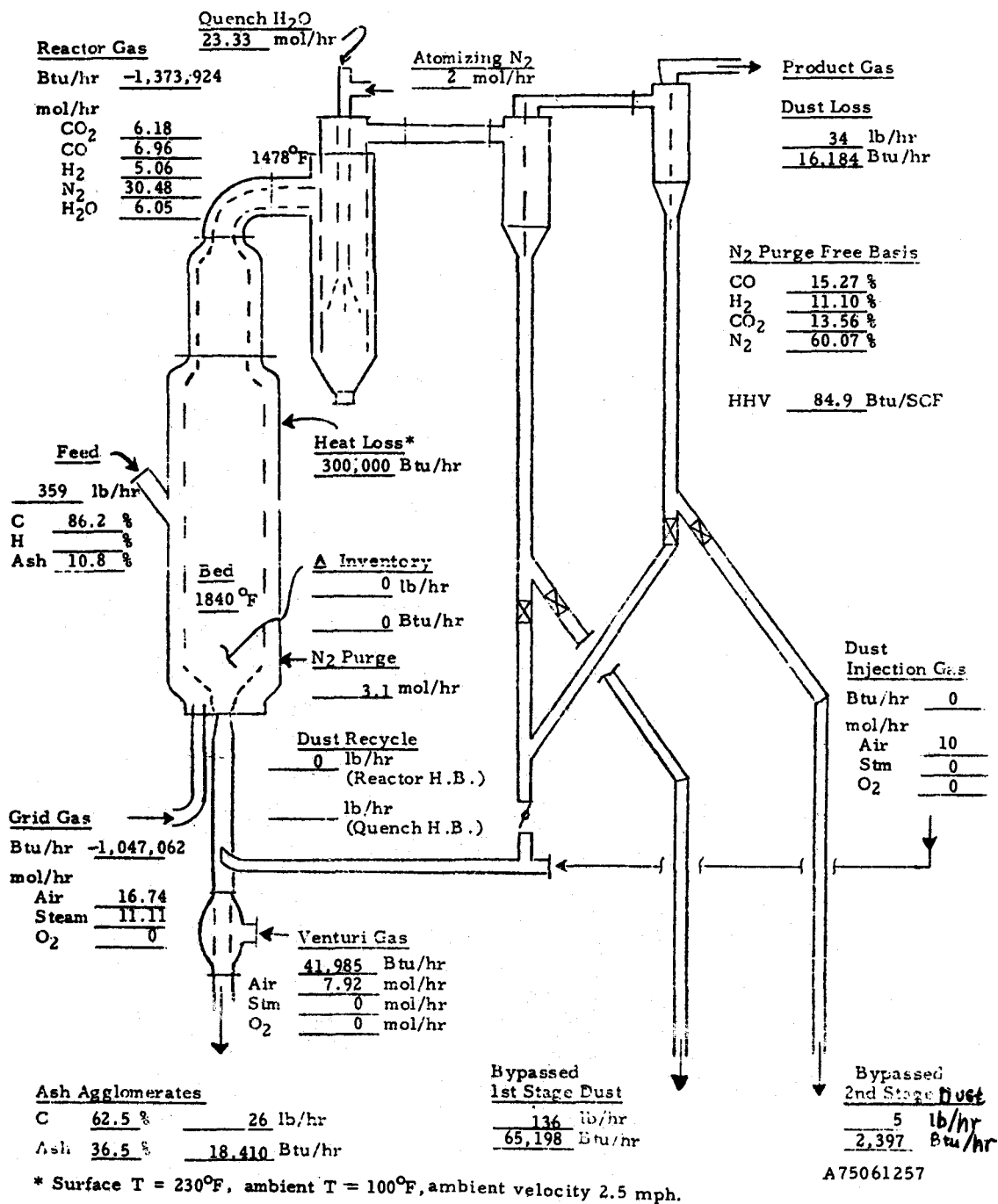


Figure 57. ASH-AGGLOMERATING GASIFICATION FOR TEST 35
(March 12, 1975; 0000 to 0600 Hours)

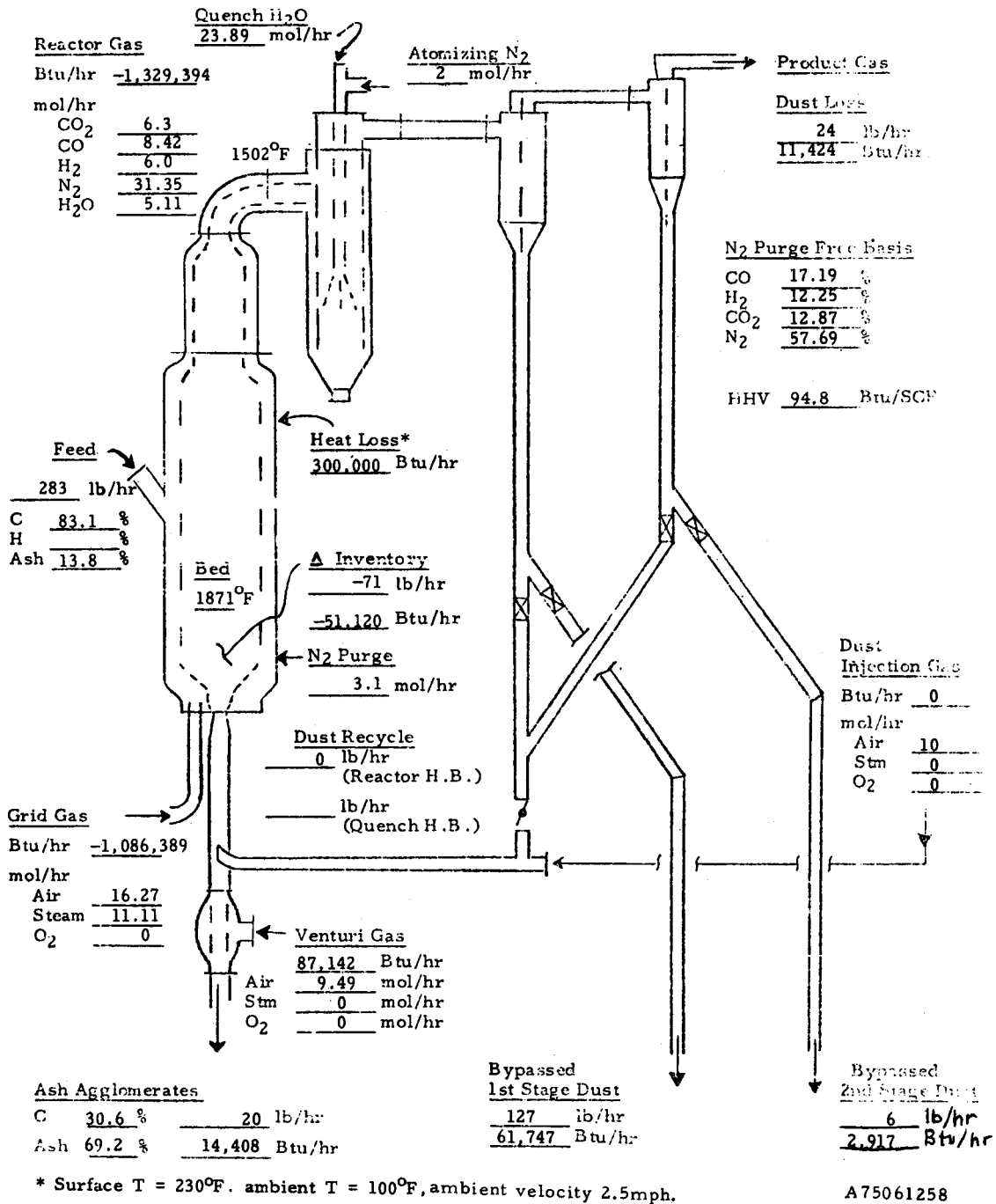


Figure 58. ASH-AGGLOMERATING GASIFICATION FOR TEST 35
(March 12, 1975; 0800 to 1200 Hours)

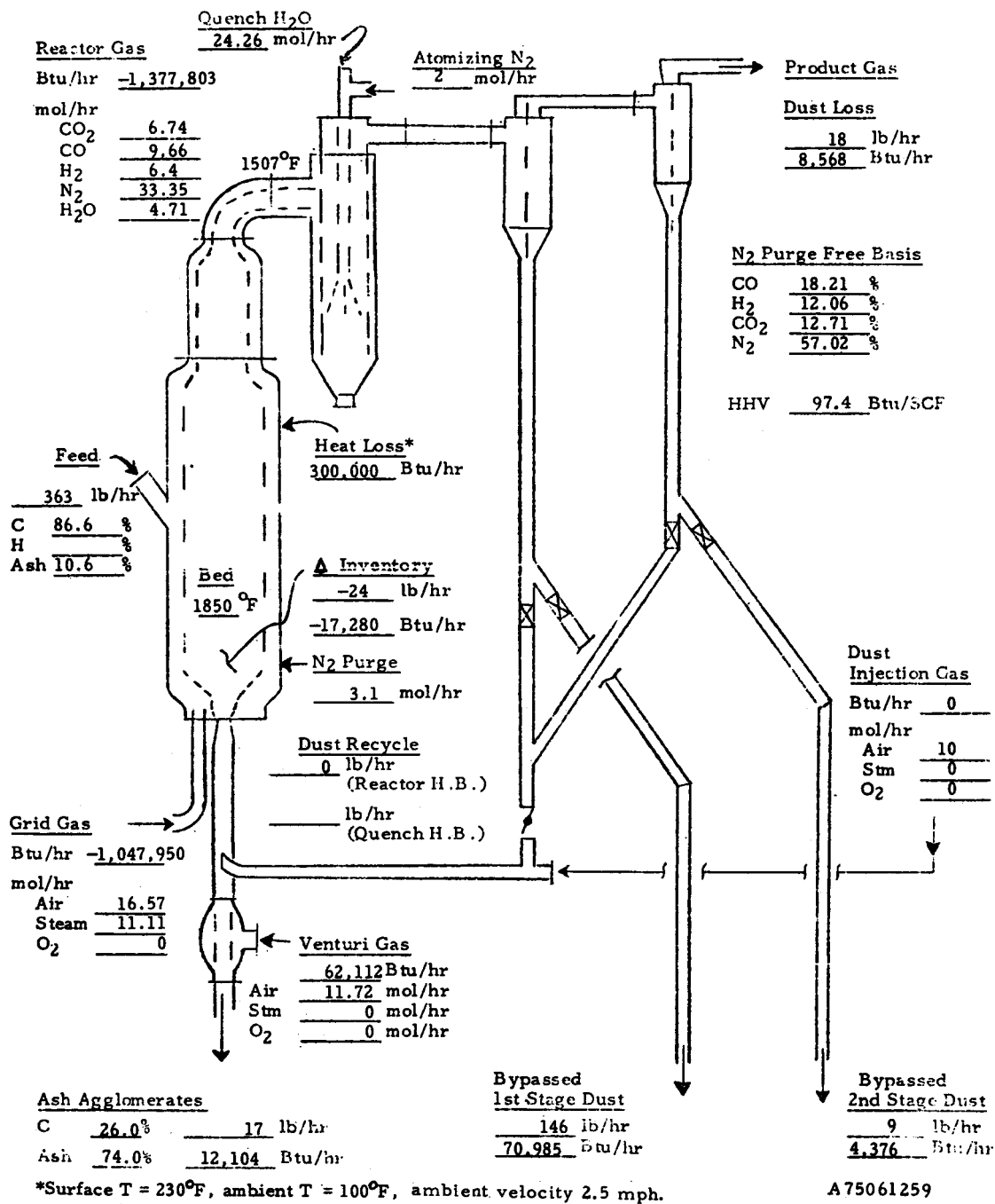


Figure 59. ASH-AGGLOMERATING GASIFICATION FOR TEST 35
(March 12-13, 1975; 2200 to 0200 Hours)

**Table 31, Part 1. ASH-AGGLOMERATING GASIFIER DATA SUMMARY
FOR TEST 35**

Time	0000	0200	0400	0600	0800	1000	1200	1400
TE 9206.6 Bed Temperature, °F	1750	1801	1839	1853	1859	1857	1854	1859
TE 9206.18 Gas Temperature-Air Heater, °F	770	707	728	727	729	798	801	773
TE 9206.1 Gas Temp. Entering Venturi, °F	546	545	570	523	507	545	558	506
TE 9206.2 Gas Temp. Entering Grid, °F	487	461	471	458	461	491	496	483
PI 913 & PDR 910 Pressure in the Venturi, psig	2.6	3.1	2.9	3.2	3.3	3.2	3.2	3.3
PI 905 Press. at Air Heater Exit, psig	12	12	12	12	12	10.1	10.1	10.1
FI 909 Air/Stm Flow with Dust Return, lb/hr	295	295	295	295	290	290	290	290
FR 903 @ 40 psig & 300°F N ₂ /Stm Flow to Grid, lb/hr	235	235	235	200	200	200	200	200
FR 905 Enriched Air to Grid, lb/hr	610	643	632	490	561	490	490	501
FR 906 Enriched Air to Venturi, lb/hr	268	282	277	326	269	235	235	240
FR 995 Total 21% Air to Air Heater, lb/hr	843	850	852	759	785	714	726	726
FR 938 Total O ₂ to Air Heater, lb/hr	0	0	0	0	0	0	0	0
N ₂ Purge to Reactor, mole/hr	3.31	3.31	3.31	3.31	3.1	3.1	3.1	3.1
FR 918 Rate of Quench Water, lb/hr	170	260	310	330	350	350	365	390
PDR 912/PDR 913 X 3 33 Bed Height, ft	4.13	4.34	4.4	4.11	4.12	3.98	3.93	3.94
PDR 913 X 43.3 Bed Density, lb/cu ft	32.04	31.17	29.44	29.44	29.01	28.57	28.57	28.14
Coke Feed Rate, lb/hr (average)	333	333	333	333	315	315	315	315
Reactor Gas Composition, % (Dry) CO ₂ (ANR 975)	15.7	15.2	14.5	13.2	13.2	13.2	13.2	12.7
CO (ANR 977)	8.7	11.0	12.5	13.2	13.2	15.0	15.0	16.0
H ₂ (ANR 981)	6.5	8.5	10.0	10.5	10.5	10.7	10.7	10.7
O ₂ (ANR 978)	0	0	0	0	0	0	0	0
N ₂ (By Difference)	69.1	65.3	63.0	63.1	63.1	61.1	61.1	60.6
1st Stage Dust Removal Rate, lb/hr	144	144	144	144	115	115	115	115
2nd Stage Dust Removal Rate, lb/hr	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5
C-4 Discharge Rate, lb/hr	34	32	27	18	12	22	21	20
Reactor Bed Composition:	%C					79.8		
%Ash						18.8		
Feed Composition:	%C	83.1				83.0		
%Ash		14.2				14.2		
Ash Composition:	%C		78.1			71.9		
%Ash			20.7			26.9		
TE 9206.16 Gas Temp. Entering Quencher, °F	892	1067	1202	1273	1333	1370	1396	1423
PIC 901 Blower Outlet Pressure, psig	13.2	13.2	13.2	13.2	13.2	11.2	11.2	11.2
TI 9203 Blower Outlet Temperature, °F	235	235	228	228	230	200	200	200
PI 9122 O ₂ Supply Pressure, psig	--	--	--	--	--	--	--	--
TE 9206.4 O ₂ Supply Temperature, °F	--	--	--	--	--	--	--	--

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Table 31, Part 2. ASH-AGGLOMERATING GASIFIER DATA SUMMARY
FOR TEST 35

Time	1600	1800	2000	2200	0000	0200	0400	0600
TE 9206.6 Bed Temperature, °F	1857	1848	1846	1843	1835	1846	1840	1839
TL 9206.18 Gas Temperature-Air Heater, °F	795	799	799	802	781	785	785	792
TE 9206.1 Gas Temp. Entering Venturi, °F	509	512	548	547	535	545	551	558
TE 9206.2 Gas Temp. Entering Grid, °F	487	494	496	497	489	488	488	489
PI 913 & PDR 910 Pressure in the Venturi, psig	3.1	3.1	3.2	3.2	3.2	3.2	3.2	3.2
PI 905 Press. at Air Heater Exit, psig	10.1	10.3	10.1	10.1	10.1	10.2	10.3	10.3
FI 909 Air/Stm Flow with Dust Return, lb/hr	290	290	290	290	290	290	290	290
FR 903 @ 40 psig & 300°F N ₂ /Stm Flow to Grid, lb/hr	200	200	200	200	200	200	200	200
FR 905 Enriched Air to Grid, lb/hr	500	500	500	500	508	506	506	503
FR 906 Enriched Air to Venturi, lb/hr	235	235	235	235	239	238	238	237
FR 995 Total 21% Air to Air Heater, lb/hr	715	715	715	715	715	715	715	715
FR 938 Total O ₂ to Air Heater, lb/hr	0	0	0	0	0	0	0	0
N ₂ Purge to Reactor, mole/hr	3.1	3.1	3.1	3.1	3.1	3.1	3.1	3.1
FR 918 Rate of Quench Water, lb/hr	400	400	410	420	420	420	420	400
PDR 912/PDR 913 X 3.33 Bed Height, ft	3.89	3.89	3.89	4.03	4.23	4.23	4.23	4.23
PDR 913 X 43.3 Bed Density, lb/cu ft	28.14	28.14	28.14	28.57	28.57	28.57	28.57	28.57
Coke Feed Rate, lb/hr (average)	336	336	336	336	359	359	359	359
Reactor Gas Composition, % (Dry) CO ₂ (ANR 975)	12.7	12.7	12.7	12.7	13.2	12.5	12.5	12.7
CO (ANR 977)	16.0	15.1	14.5	14.5	13.7	14.5	14.5	14.5
H ₂ (ANR 981)	10.6	10.6	10.6	10.6	10.1	10.6	10.6	10.1
O ₂ (ANR 978)	0	0	0	0	0	0	0	0
N ₂ (By Difference)	60.7	61.6	62.2	62.2	63.0	62.2	62.2	62.7
1st Stage Dust Removal Rate, lb/hr	122	122	122	122	136	136	136	136
2nd Stage Dust Removal Rate, lb/hr	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5
C-4 Discharge Rate, lb/hr	20	20	22	23	24	27	28	25
Reactor Bed Composition:	%C	72.6				71.4		
%Ash		26.0				28.1		
Feed Composition:	%C	86.0				86.2		
%Ash		11.0				10.8		
Ash Composition:	%C	68.4		66.2				62.5
%Ash	30.4		32.6					36.4
TE 9206.16 Gas Temp. Entering Quencher, °F	1437	1449	1457	1466	1473	1476	1479	1484
PIC 901 Blower Outlet Pressure, psig	11.2	11.2	11.2	11.2	11.2	11.2	11.2	11.2
TI 9203 Blower Outlet Temperature, °F	210	210	210	210	210	210	210	210
PI 9122 O ₂ Supply Pressure, psig	--	--	--	--	--	--	--	--
TE 9206.4 O ₂ Supply Temperature, °F	--	--	--	--	--	--	--	--

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Table 31, Part 3. ASH-AGGLOMERATING GASIFIER DATA SUMMARY
FOR TEST 35

Time	0800	1000	1200	1400	1600	1800	2000	2200
TE 9206.6 Bed Temperature, °F	1842	1895	1875	1836	1824	1840	1840	1847
TE 9206.18 Gas Temperature-Air Heater, °F	777	868	779	821	830	828	833	825
TE 9206.1 Gas Temp. Entering Venturi, °F	535	493	482	472	512	531	512	498
TE 9206.2 Gas Temp. Entering Grid, °F	480	496	487	494	501	502	505	497
PI 913 & PDR 910 Pressure in the Venturi, psig	3.5	3.3	3.4	3.5	3.5	3.5	3.5	3.5
PI 905 Press. at Air Heater Exit, psig	10.1	10.1	10.1	10	10	10	10	10
FI 909 Air/Stm Flow with Dust Return, lb/hr	290	290	290	290	290	290	290	290
FR 903 @ 40 psig & 300°F N ₂ /Stm Flow to Grid, lb/hr	200	200	200	200	200	200	200	200
FR 905 Enriched Air to Grid, lb/hr	506	470	503	487	483	484	482	485
FR 906 Enriched Air to Venturi, lb/hr	295	275	295	344	342	342	341	343
FR 995 Total 21% Air to Air Heater, lb/hr	764	727	750	810	810	812	816	822
FR 938 Total O ₂ to Air Heater, lb/hr	0	0	0	0	0	0	0	0
N ₂ Purge to Reactor, mole/hr	3.1	3.1	3.1	3.1	3.1	3.1	3.1	3.1
FR 918 Rate of Quench Water, lb/hr	430	430	430	420	420	420	420	420
PDR 912/PDR 913 X 3 33 Bed Height, ft	4.28	3.83	3.72	3.57	3.77	4.0	3.9	4.06
PDR 913 X 43.3 Bed Density, lb/cu ft	30.31	28.57	29.01	29.44	32.47	32.47	32.47	31.17
Coke Feed Rate, lb/hr (average)	283	283	283	487	487	400	400	363
Reactor Gas Composition, % (Dry) CO ₂ (ANR 975)	12.7	11.7	12.0	12.5	12.7	12.5	12.5	12.0
CO (ANR 977)	15.0	16.5	17.0	15.5	15.5	16.5	16.0	17.0
H ₂ (ANR 981)	10.5	12.2	11.8	11.0	11.0	11.0	11.2	11.0
O ₂ (ANR 978)	0	0	0	0	0	0	0	0
N ₂ (By Difference)	61.8	59.6	59.2	61	60.8	60	60.3	60
1st Stage Dust Removal Rate, lb/hr	127	127	127	127	158	158	158	158
2nd Stage Dust Removal Rate, lb/hr	4.5	4.5	8.7	8.7	8.7	8.7	8.7	8.7
C-4 Discharge Rate, lb/hr	25	17	18	16	19	18	18	17
Reactor Bed Composition:								
% C		69.7				68.5		
% Ash		29.4				30.7		
Feed Composition:								
% C			83.1				85.5	
% Ash			13.8				11.7	
Ash Composition:								
% C			30.6			31.9		29.6
% Ash			69.2			68.1		71.6
TE 9206.16 Gas Temp. Entering Quencher, °F	1489	1509	1507	1503	1492	1492	1500	1502
PIC 901 Blower Outlet Pressure, psig	11.2	11.2	11.2	11.3	11.3	11.3	11.3	11.3
TI 9203 Blower Outlet Temperature, °F	210	210	210	210	210	208	205	200
PI 9122 O ₂ Supply Pressure, psig	--	--	--	--	--	--	--	--
TE 9206.4 O ₂ Supply Temperature, °F	--	--	--	--	--	--	--	--

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**Table 31, Part 4. ASH-AGGLOMERATING GASIFIER DATA SUMMARY
FOR TEST 35**

Temp	0000	0200							
TE 9206.6									
Bed Temperature, °F	1852	1851							
TE 9206.18									
Gas Temperature-Air Heater, °F	815	820							
TE 9206.1									
Gas Temp. Entering Venturi, °F	484	480							
TE 9206.2									
Gas Temp. Entering Grid, °F	495	497							
PI 913 & PDR 910									
Pressure in the Venturi, psig	3.4	3.3							
PI 905									
Press. at Air Heater Exit, psig	10	10							
FI 909									
Air/Stm Flow with Dust Return, lb/hr	290	290							
FR 903 @ 40 psig & 300°F									
N ₂ /Stm Flow to Grid, lb/hr	200	200							
FR 905									
Enriched Air to Grid, lb/hr	489	487							
FR 906									
Enriched Air to Venturi, lb/hr	346	345							
FR 995									
Total 21% Air to Air Heater, lb/hr	824	814							
FR 938									
Total O ₂ to Air Heater, lb/hr	0	0							
N ₂ Purge to Reactor, mole/hr	3.1	3.1							
FR 918									
Rate of Quench Water, lb/hr	430	460							
PDR 912/PDR 913 X 3 33									
Bed Height, ft	3.93	3.94							
PDR 913 X 43.3									
Bed Density, lb/cu ft	30.74	30.31							
Coke Feed Rate, lb/hr (average)	363	363							
Reactor Gas Composition, % (Dry)									
CO ₂ (ANR 975)	11.7	12.1							
CO (ANR 977)	17.0	17.5							
H ₂ (ANR 981)	11.5	11.6							
O ₂ (ANR 978)	0	0							
N ₂ (By Difference)	59.8	58.8							
1st Stage Dust Removal Rate, lb/hr	140	140							
2nd Stage Dust Removal Rate, lb/hr	8.7	8.7							
C-4 Discharge Rate, lb/hr	16	17							
Reactor Bed	%C	67.9							
Composition:	%Ash	31.0							
Feed Composition:	%C	86.6							
	%Ash	10.6							
Ash Composition:	%C	24.3							
	%Ash	75.1							
TE 9206.16									
Gas Temp. Entering Quencher, °F	1507	1510							
PIC 901									
Blower Outlet Pressure, psig	11.3	11.3							
TI 9203									
Blower Outlet Temperature, °F	190	190							
FI 9122									
O ₂ Supply Pressure, psig	--	--							
TE 9206.4									
O ₂ Supply Temperature, °F	--	--							

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Table 32. COMPARISON OF OPERATING CONDITIONS AT 4-FOOT
AND 8-FOOT BED DEPTHS DURING TEST 36

Test Date	3/26/75	3/27/75
Time	0000-1000	1400-2000
Bed Height, ft	4.00	8.00
Bed Temperature, °F	1900	1855
Bed Density, lb/cu ft	29.23	34.64
Total Air (21%) to Reactor, mol/hr	48.23	55.30
Total Steam to Reactor, mol/hr	20.27	17.98
Rate of Dust Removal From First Stage, lb/hr	226	335
Rate of Dust Removal From Second Stage, lb/hr	12	22
Reactor Gas Composition (Moisture Free), %		
CO	15.91	13.42
CO ₂	13.40	13.87
Ash Discharge, lb/hr	34.0	14.3*
Venturi Velocity, ft/s	152	220*
Overall Velocity, ft/s	2.04	1.97
Solid Feed Rate, lb/hr	567	667
Carbon Combusted, lb/hr	246	252

* Ash discharge contains many fine particles.

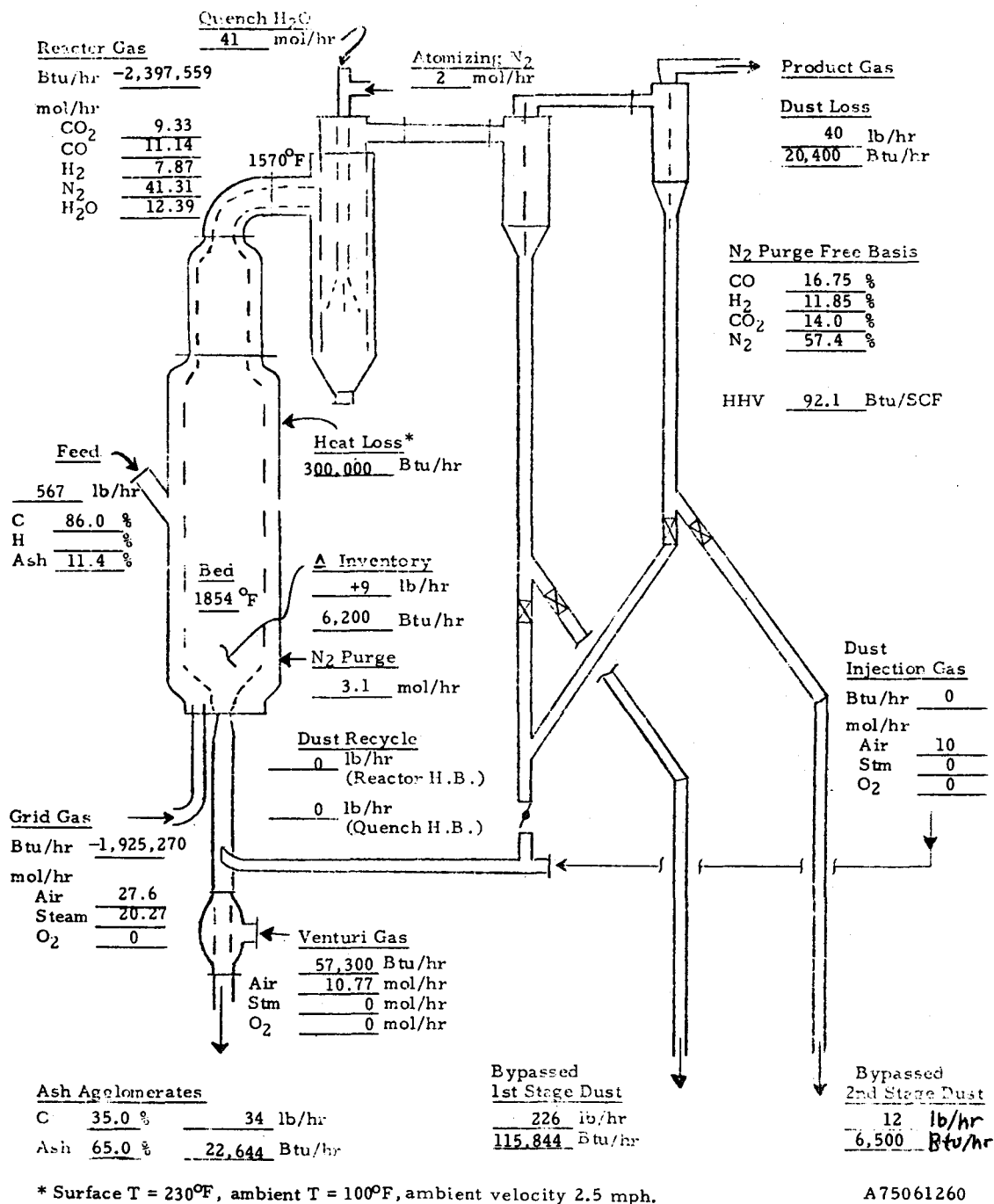


Figure 60. ASH-AGGLOMERATING GASIFICATION FOR TEST 36
(March 26, 1975; 0000 to 1000 Hours)

Table 33, Part 1. ASH-AGGLOMERATING GASIFIER DATA SUMMARY
FOR TEST 36

Time	1400	1600	1800	2000	2200	0000	0200	0400
TE 9206.6								
Bed Temperature, °F	1826	1862	1858	1865	1856	1857	1854	1856
TE 9206.18								
Gas Temperature-Air Heater, °F	803	796	795	802	799	800	804	796
TE 9206.1								
Gas Temp. Entering Venturi, °F	604	619	546	535	556	535	575	565
TE 9206.2								
Gas Temp. Entering Grid, °F	528	533	537	538	538	538	539	539
PI 913 & PDR 910								
Pressure in the Venturi, psig	4.1	4.5	4.5	4.5	4.5	4.6	4.6	4.6
PI 905								
Press. at Air Heater Exit, psig	10.5	10.1	10.0	10.1	10.2	10.1	10.1	10.1
FI 909								
Air/Stm Flow with Dust Return, lb/hr	290	290	290	290	290	290	290	290
FR 903 @ 40 psig & 300°F								
N ₂ /Stm Flow to Grid, lb/hr	285	365	365	365	365	365	365	365
FR 905								
Enriched Air to Grid, lb/hr	--	--	--	800	800	800	800	800
FR 906								
Enriched Air to Venturi, lb/hr	300	300	300	300	300	300	300	300
FR 995								
Total 21% Air to Air Heater, lb/hr	1031	1181	1186	1192	1192	1192	1192	1192
FR 938								
Total O ₂ to Air Heater, lb/hr	0	0	0	0	0	0	0	0
N ₂ Purge to Reactor, mole/hr	3.1	3.1	3.1	3.1	3.1	3.1	3.1	3.1
FR 918								
Rate of Quench Water, lb/hr	440	520	660	680	705	705	720	740
PDR 912/PDR 913 X 3.33								
Bed Height, ft	4.39	4.25	3.99	3.9	3.94	3.93	3.91	3.91
PDR 913 X 43.3								
Bed Density, lb/cu ft	31.17	28.14	28.14	27.71	28.14	28.57	29.44	29.44
Coke Feed Rate, lb/hr (average)	364	364	600	600	600	567	567	567
Reactor Gas Composition, % (Dry)								
CO ₂ (ANR 975)	14.0	13.6	13.6	13.6	13.6	13.3	13.6	13.3
CO (ANR 977)	14.0	16.5	16.5	17.0	17.0	16.5	16.0	16.0
H ₂ (ANR 981)	9.2	11.0	11.5	11.5	11.5	11.5	11.5	11.5
O ₂ (ANR 978)	0	0	0	0	0	0	0	0
N ₂ (By Difference)	62.8	58.9	58.4	57.9	57.9	58.7	58.9	59.2
1st Stage Dust Removal Rate, lb/hr	189	189	189	189	189	226	226	226
2nd Stage Dust Removal Rate, lb/hr	6	6	6	6	12	12	12	12
C-4 Discharge Rate, lb/hr	28	25	24	25	31	31	38	36
Reactor Bed Composition:								
%C		82.0		75.3				70.8
%Ash		16.5		23.5				28.2
Feed Composition:								
%C	84.8				85.6			
%Ash	12.4				11.3			
Ash Composition:								
%C			70.0		51.3			37.2
%Ash			29.2		48.7			62.6
TE 9206.16								
Gas Temp. Entering Quencher, °F	1273	1410	1468	1508	1528	1541	1558	1570
PIC 901								
Blower Outlet Pressure, psig	11.7	11.7	11.5	12	12	12.1	12.1	12.2
TI 9203								
Blower Outlet Temperature, °F	220	223	220	220	220	220	220	215
PI 9122								
O ₂ Supply Pressure, psig	--	--	--	--	--	--	--	--
TE 9206.4								
O ₂ Supply Temperature, °F	--	--	--	--	--	--	--	--

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Table 33, Part 2. ASH-AGGLOMERATING GASIFIER DATA SUMMARY
FOR TEST 36

Time	0600	0800	1000	1200	1400	1600	1800	2000
TE 9206.6 Bed Temperature, °F	1848	1853	1853	1854	1854	1855	1828	1820
TE 9206.18 Gas Temperature-Air Heater, °F	777	776	778	799	800	794	795	795
TE 9206.1 Gas Temp. Entering Venturi, °F	546	536	545	541	547	481	571	623
TE 9206.2 Gas Temp. Entering Grid, °F	528	523	523	530	529	530	533	529
PI 913 & PDR 910 Pressure in the Venturi, psig	4.5	4.6	4.6	4.7	5.0	5.0	5.1	5.3
PI 905 Press. at Air Heater Exit, psig	10.0	10.0	10.0	10.1	10.1	10.1	10.5	10.1
FI 909 Air/Stm Flow with Dust Return, lb/hr	290	290	290	290	290	290	290	290
FR 903 @ 40 psig & 300°F N ₂ /Stm Flow to Grid, lb/hr	365	365	365	365	365	365	365	365
FR 905 Enriched Air to Grid, lb/hr	784	784	784	770	770	772	772	772
FR 906 Enriched Air to Venturi, lb/hr	341	341	341	365	405	405	405	455
FR 995 Total 21% Air to Air Heater, lb/hr	1228	1228	1228	1247	1267	1267	1276	1316
FR 938 Total O ₂ to Air Heater, lb/hr	0	0	0	0	0	0	0	0
N ₂ Purge to Reactor, mole/hr	3.1	3.1	3.1	3.1	3.1	3.1	3.1	3.1
FR 918 Rate of Quench Water, lb/hr	770	750	750	760	785	770	800	800
PDR 912/PDR 913 X 3.33 Bed Height, ft	4.04	3.92	3.9	3.95	3.77	3.98	4.66	5.16
PDR 913 X 43.3 Bed Density, lb/cu ft	28.58	29.44	30.31	30.31	32.48	31.18	32.48	34.64
Coke Feed Rate, lb/hr (average)	567	567	567	567	567	840	840	840
Reactor Gas Composition, % (Dry) CO ₂ (ANR 975)	13.5	13.5	13.2	13.2	13.0	13.0	14.0	14.0
CO (ANR 977)	15.5	16.0	15.5	15.5	15.5	15.5	13.0	13.3
H ₂ (ANR 981)	11.0	11.0	11.4	11.4	11.4	11.4	9.5	10.0
O ₂ (ANR 978)	0	0	0	0	0	0	0	0
N ₂ (By Difference)	60.0	59.5	59.9	59.9	60.1	60.1	63.5	62.7
1st Stage Dust Removal Rate, lb/hr	226	226	226	226	226	323	323	323
2nd Stage Dust Removal Rate, lb/hr	12	12	12	12	12	12	14	14
C-4 Discharge Rate, lb/hr	31	35	33	30	26	25	43	43
Reactor Bed Composition:	%C			66.3		69.3		71.3
%Ash				32.8		29.7		27.6
Feed Composition:	%C	86.0			84.7			
%Ash		11.4			12.5			
Ash Composition:	%C		32.5		12.5		25.5	
%Ash			67.5		72.8		73.4	
TE 9206.16 Gas Temp. Entering Quencher, °F	1580	1583	1575	1590	1596	1599	1590	1588
PIC 901 Blower Outlet Pressure, psig	12.1	12.5	12.5	12.5	12.5	12.5	12.5	12.5
TI 9203 Blower Outlet Temperature, °F	225	225	218	225	225	225	235	235
PI 9122 O ₂ Supply Pressure, psig	--	--	--	--	--	--	--	--
TE 9206.4 O ₂ Supply Temperature, °F	--	--	--	--	--	--	--	--

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Table 33, Part 3. ASH-AGGLOMERATING GASIFIER DATA SUMMARY
FOR TEST 36

Time	2200	0000	0200	0400	0600	0800	1000	1200
TE 9206.6 Bed Temperature, °F	1804	1798	1788	1823	1795	1784	1809	1795
TE 9206.18 Gas Temperature-Air Heater, °F	792	789	797	794	794	792	794	793
TE 9206.1 Gas Temp. Entering Venturi, °F	652	699	699	697	663	663	654	647
TE 9206.2 Gas Temp. Entering Grid, °F	533	532	528	529	529	518	536	524
PI 913 & PDR 910 Pressure in the Venturi, psig	5.8	6.1	6.3	6.3	6.1	6.1	6.5	6.5
PI 905 Press. at Air Heater Exit, psig	10.0	10.5	10.6	10.6	10.6	10.4	14.5	13.5
FI 909 Air/Stm Flow with Dust Return, lb/hr	290	290	290	290	290	290	290	290
FR 903 @ 40 psig & 300°F N ₂ /Stm Flow to Grid, lb/hr	367	367	367	367	367	367	367	367
FR 905 Enriched Air to Grid, lb/hr	762	784	784	784	784	753	817	750
FR 906 Enriched Air to Venturi, lb/hr	488	500	511	511	511	511	613	603
FR 995 Total 21% Air to Air Heater, lb/hr	1357	1367	1335	1355	1355	1311	--	1429
FR 938 Total O ₂ to Air Heater, lb/hr	0	0	0	0	0	0	0	0
N ₂ Purge to Reactor, mole/hr	3.1	3.1	3.1	3.1	3.1	3.1	3.1	3.1
FR 918 Rate of Quench Water, lb/hr	850	850	860	820	900	870	840	940
PDR 912/PDR 913 X 3.33 Bed Height, ft	6.04	6.87	7.49	7.99	7.17	7.66	7.99	7.91
PDR 913 X 43.3 Bed Density, lb/cu ft	34.64	34.64	34.64	34.64	33.77	34.64	34.64	34.64
Coke Feed Rate, lb/hr (average)	840	840	840	800	800	800	800	667
Reactor Gas Composition, % (Dry) CO ₂ (ANR 975)	14.0	14.0	14.0	12.5	14.0	14.5	13.3	13.3
CO (ANR 977)	13.3	13.3	12.5	17.0	13.0	12.0	13.7	13.7
H ₂ (ANR 981)	9.5	9.5	9.0	11.0	9.4	9.4	10.5	10.7
O ₂ (ANR 978)	0	0	0	0	0	0	0	0
N ₂ (By Difference)	63.2	63.2	64.5	59.5	63.6	64.1	62.5	62.3
1st Stage Dust Removal Rate, lb/hr	323	323	323	323	323	323	335	335
2nd Stage Dust Removal Rate, lb/hr	14	14	14	14	14	14	14	22
C-4 Discharge Rate, lb/hr	41	43	34	40	44	32	22	15
Reactor Bed Composition:	%C			73.2				72.5
	%Ash			25.6				26.4
Feed Composition:	%C	85.4			84.5			
	%Ash	12.1			12.7			
Ash Composition:	%C	35.5	46.3		36.8		55.3	
	%Ash	65.2	53.6		63.1		44.6	
TE 9206.16 Gas Temp. Entering Quencher, °F	1604	1607	1624	1637	1637	1631	1658	1658
PIC 901 Blower Outlet Pressure, psig	13.0	13.0	13.2	13.5	13.5	12.5	16.3	16.0
TI 9203 Blower Outlet Temperature, °F	235	245	245	245	240	235	300	285
PI 9122 O ₂ Supply Pressure, psig	--	--	--	--	--	--	--	--
TE 9206.4 O ₂ Supply Temperature, °F	--	--	--	--	--	--	--	--

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Table 33, Part 4. ASH-AGGLOMERATING GASIFIER DATA SUMMARY
FOR TEST 36

Time	1400	1600	1800	2000	2200			
TE 9206.6								
Bed Temperature, °F	1798	1801	1788	1787	1786			
TE 9206.18								
Gas Temperature-Air Heater, °F	797	794	793	800	797			
TE 9206.1								
Gas Temp. Entering Venturi, °F	612	579	573	570	555			
TE 9206.2								
Gas Temp. Entering Grid, °F	508	507	494	494	492			
PI 913 & PDR 910								
Pressure in the Venturi, psig	6.5	6.7	6.3	6.3	6.3			
PI 905								
Press. at Air Heater Exit, psig	13.6	14.2	13.1	13.1	13.1			
FI 909								
Air/Stm Flow with Dust Return, lb/hr	290	290	290	290	290			
FR 903 @ 40 psig & 300°F								
N ₂ /Stm Flow to Grid, lb/hr	367	367	367	367	367			
FR 905								
Enriched Air to Grid, lb/hr	631	640	628	626	626			
FR 906								
Enriched Air to Venturi, lb/hr	685	724	663	663	663			
FR 995								
Total 21% Air to Air Heater, lb/hr	1399	1431	1350	1360	1358			
FR 938								
Total O ₂ to Air Heater, lb/hr	0	0	0	0	0			
N ₂ Purge to Reactor, lb/hr	3.1	3.1	3.1	3.1	3.1			
FR 918								
Rate of Quench Water, lb/hr	920	940	900	900	880			
PDR 912/PDR 913 X 3.33								
Bed Height, ft	7.99	7.91	8.03	8.08	7.99			
PDR 913 X 43.3								
Bed Density, lb/cu ft	34.64	34.64	34.64	34.64	34.64			
Coke Feed Rate, lb/hr (average)	667	667	667	667	667			
Reactor Gas Composition, % (Dry)								
CO ₂ (ANR 975)	13.3	13.7	14.5	14.0	14.0			
CO (ANR 977)	13.7	14.5	13.0	12.5	12.5			
H ₂ (ANR 981)	10.7	10.7	10.5	10.7	10.7			
O ₂ (ANR 978)	0	0	0	0	0			
N ₂ (By Difference)	62.3	61.1	62.0	62.8	62.8			
1st Stage Dust Removal Rate, lb/hr	335	335	335	335	335			
2nd Stage Dust Removal Rate, lb/hr	22	22	22	22	12			
C-4 Discharge Rate, lb/hr	14	14	15	14	14			
Reactor Bed Composition:								
%C		71.1		71.3				
%Ash		27.9		27.6				
Feed Composition:								
%C	35.2				85.4			
%Ash	12.2				11.9			
Ash Composition:								
%C	43.1		37.7		43.6			
%Ash	56.4		62.0		56.4			
TE 9206.16								
Gas Temp. Entering Quencher, °F	1658	1664	1653	1653	1656			
PIC 901								
Blower Outlet Pressure, psig	16.0	17.0	15.0	15.5	15.5			
TI 9203								
Blower Outlet Temperature, °F	285	275	250	245	275			
PI 9122								
O ₂ Supply Pressure, psig	--	--	--	--	--			
TE 9206.4								
O ₂ Supply Temperature, °F	--	--	--	--	--			

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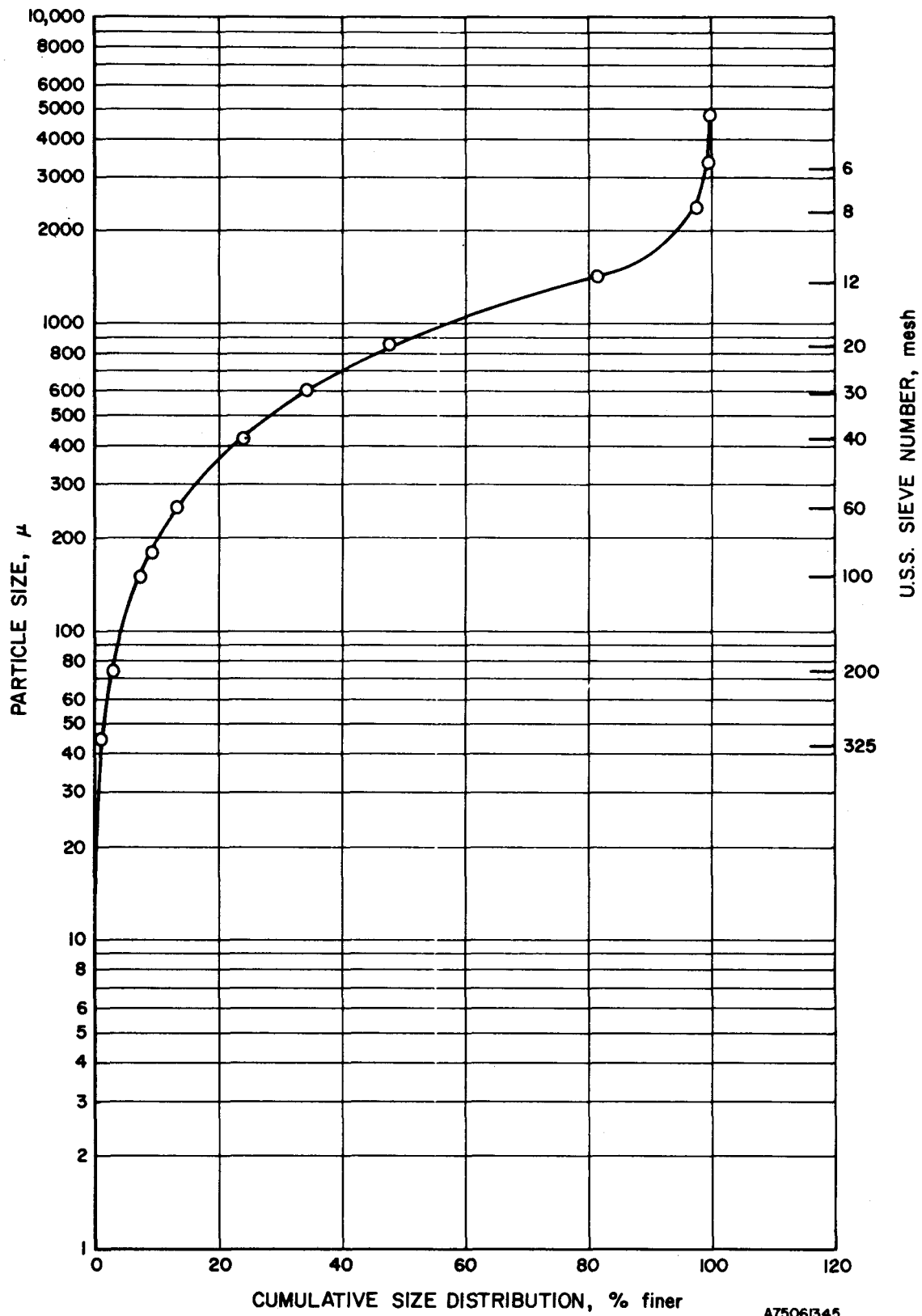


Figure 62. ASH-AGGLOMERATING TEST 36 USING FINE BREEZE COKE REACTOR FEED

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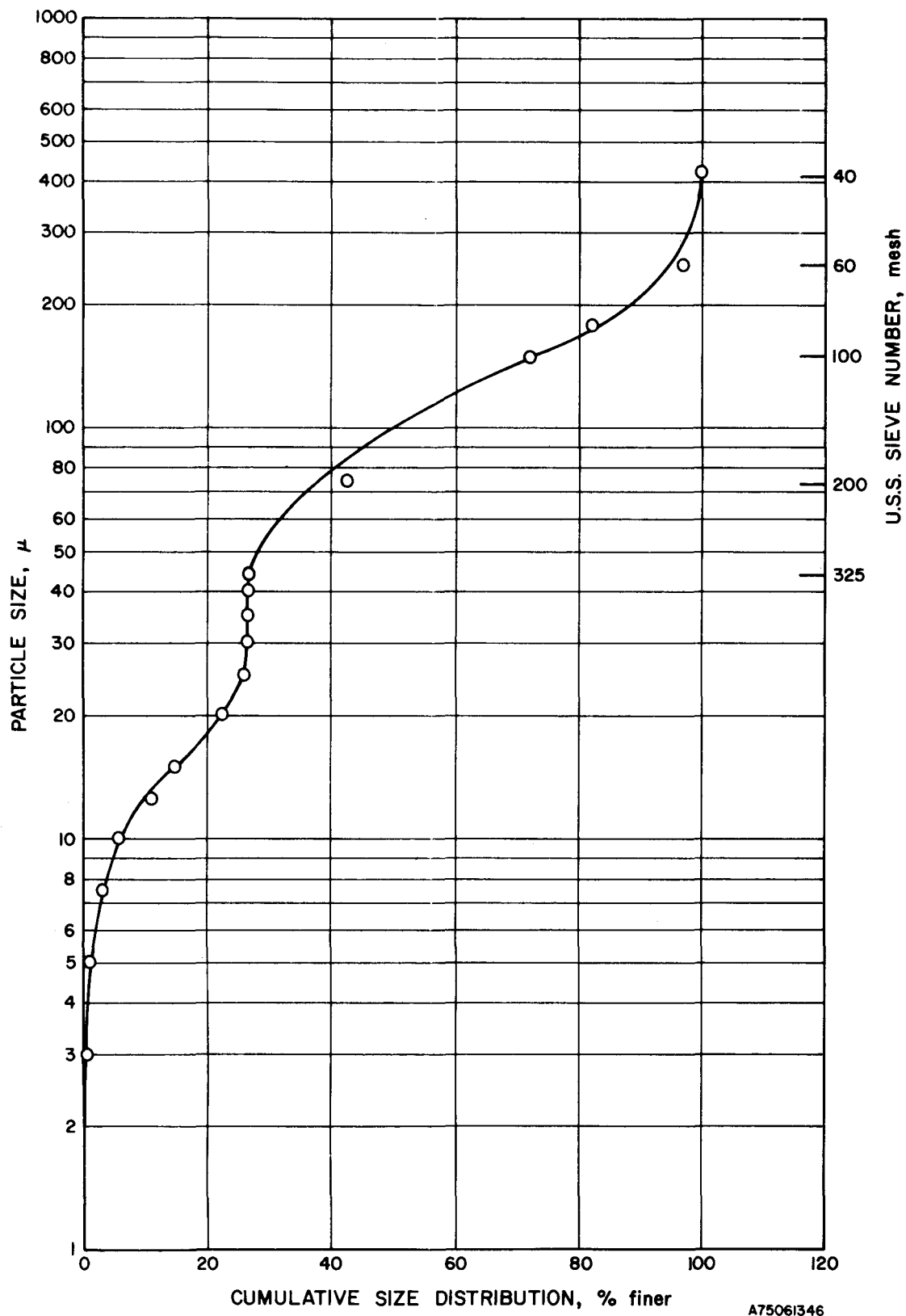


Figure 63. ASH-AGGLOMERATING GASIFIER TEST 36 USING FINE BREEZE COKE REACTOR FEED AND A 4-FOOT-DEEP FLUIDIZED BED (First-Stage Cyclone Dust)

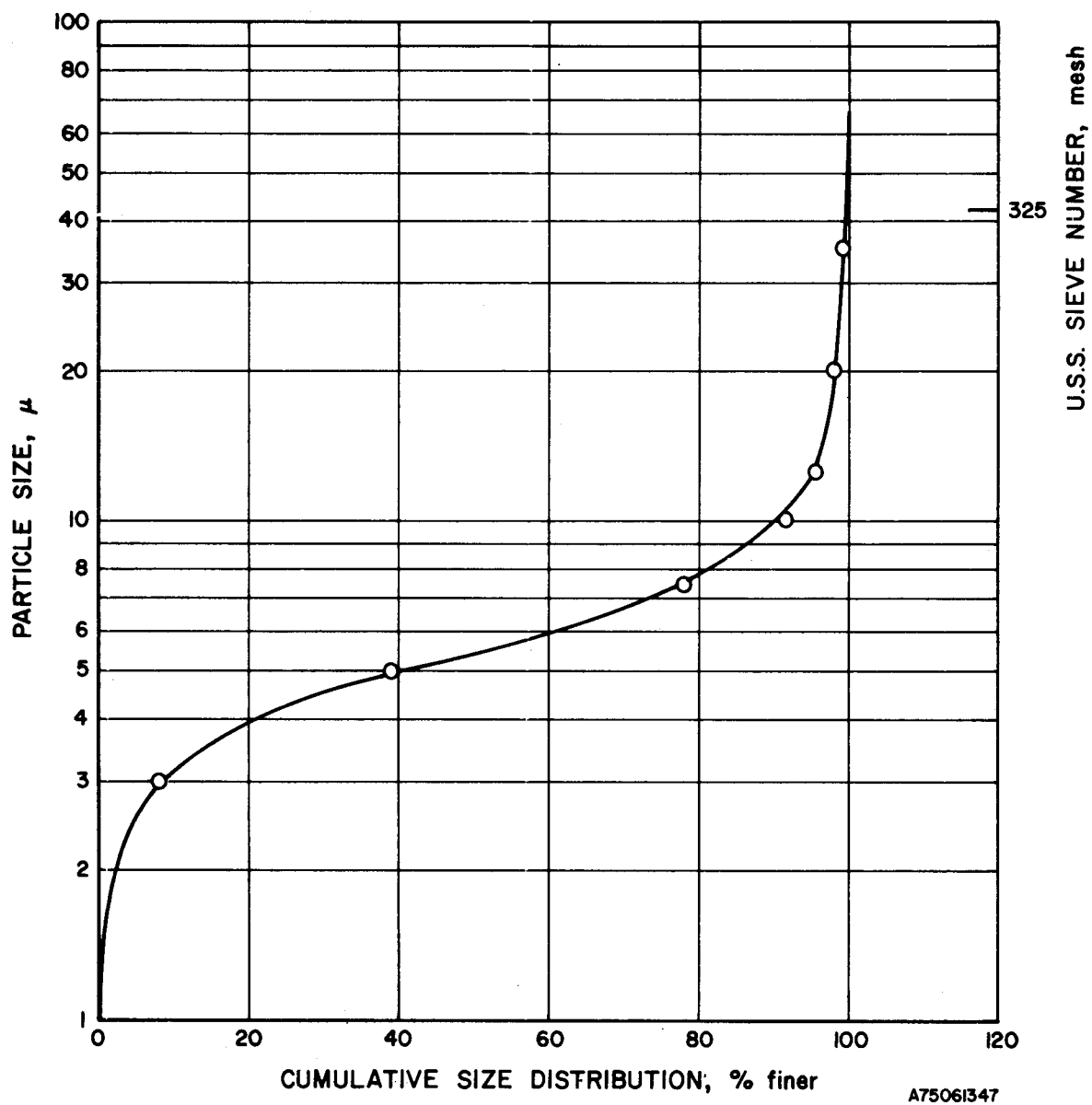


Figure 64. ASH-AGGLOMERATING GASIFIER TEST 36 USING FINE BREEZE COKE REACTOR FEED AND A 4-FOOT-DEEP FLUIDIZED BED (Second-Stage Cyclone Dust)

7-76

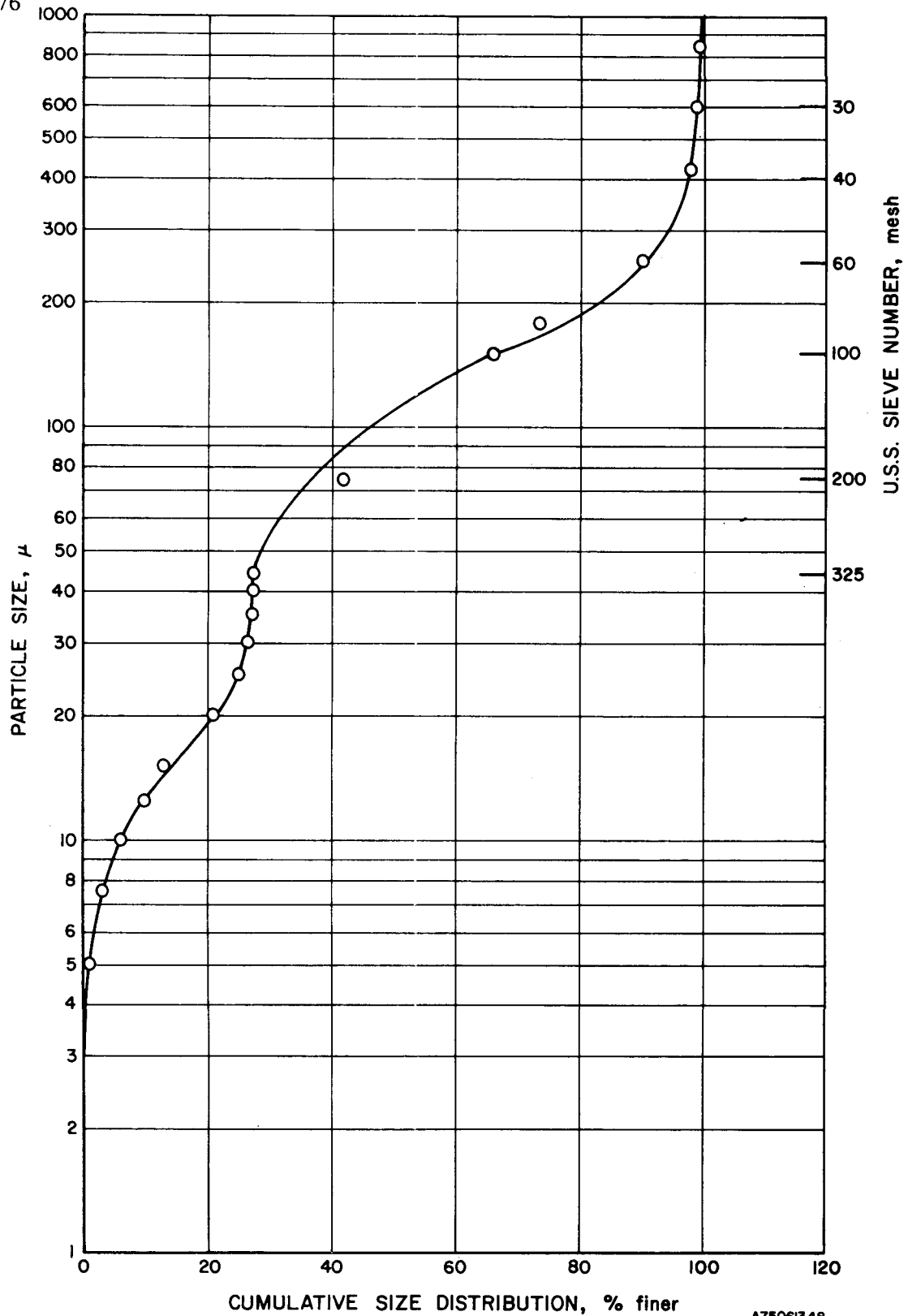


Figure 65. ASH-AGGLOMERATING GASIFIER TEST 36 USING FINE BREEZE COKE REACTOR FEED AND A 8-FOOT-DEEP FLUIDIZED BED (First-Stage Cyclone Dust)

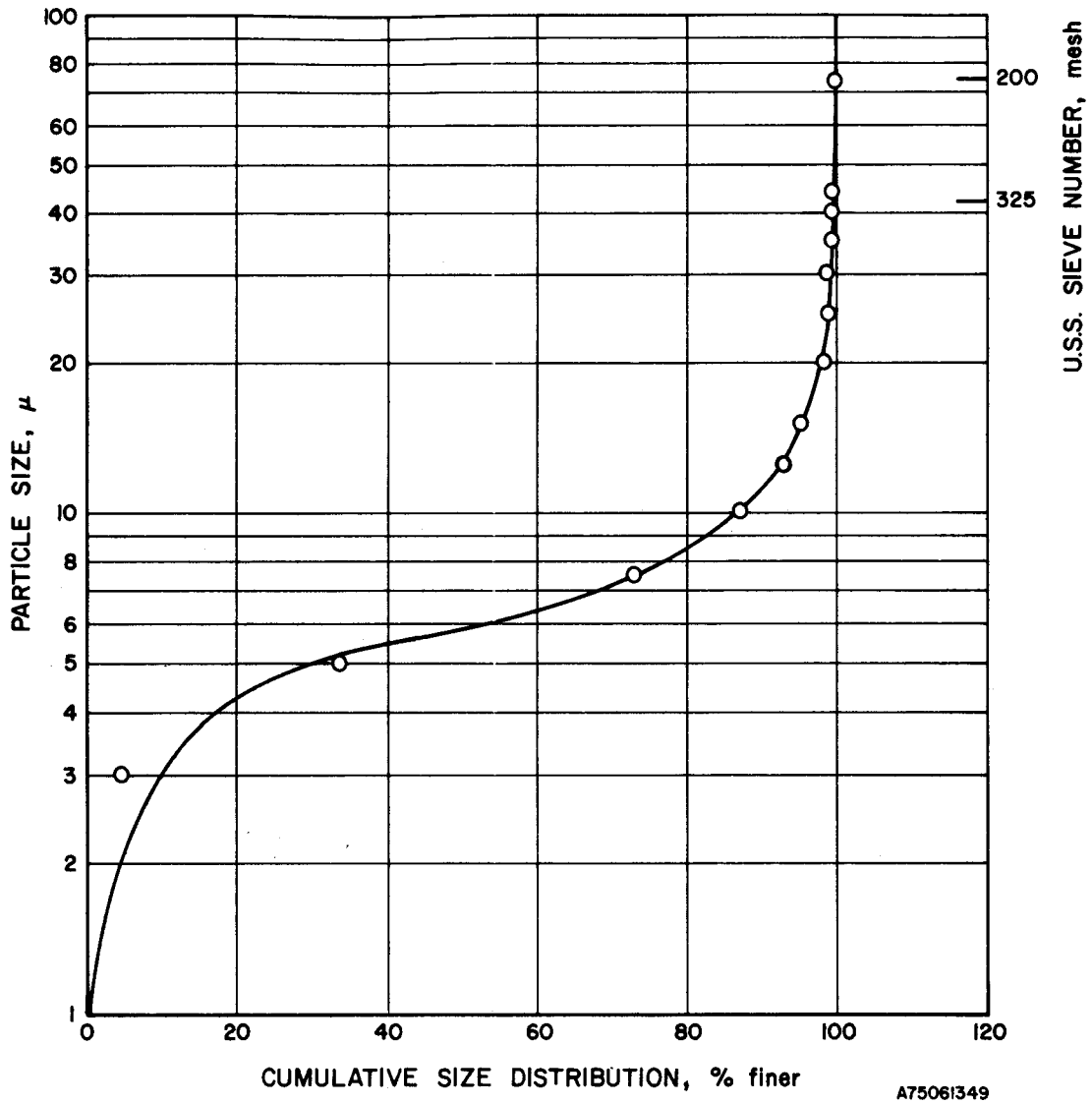


Figure 66. ASH-AGGLOMERATING GASIFIER TEST 36 USING FINE BREEZE COKE REACTOR FEED AND A 8-FOOT-DEEP FLUIDIZED BED (Second-Stage Cyclone Dust)

hours after their appearance, the ash discharge was averaging 80% ash. Minor adjustments were made to the venturi velocity to minimize fines withdrawal and to further improve the percentage of ash. The test was continued without incident for an additional 12 hours to accumulate steady-state data and a drum of ash agglomerates. In producing these agglomerates, no dust was recirculated. The feed rate averaged 500 lb/hr; dust was collected at 130 lb/hr, the balance being gasified or discharged as ash. Heat and material balances for Test 38 are included in Figures 67 and 68 and Table 34.

Test 39

Test 39 proved that agglomerates with a high ash content can be made from the -6 mesh coke breeze using the steam-oxygen mode of gasification and at superficial velocities (1.6 ft/s) lower than those required for steam-air gasification. The dust was collected, but not recirculated.

Test 39 was begun using air, adding steam after autogenous operation was achieved, and switching to steam-oxygen fluidization without incident. The same 21% oxygen/79% steam ratio as in Test 38 was also used. The need to increase the oxygen concentration to obtain an 1875°F bed temperature was anticipated, but it was not necessary. Accordingly, the same total oxygen input in this run as was used in Test 35 was essentially maintained, the sole operational difference being that the nitrogen occurring with the air of Test 35 was replaced by steam, at the same volume concentration as in Test 39. Agglomerates were observed in the venturi discharge material 9 hours after completing the change to steam-oxygen operation. Continuing the test for another 2-1/2 days, the gas throughput was gradually adjusted upward at the venturi and downward at the grid to minimize fines discharge. Ultimately a velocity of 220 ft/s at the venturi was reached, yielding essentially 100% of +12 mesh agglomerates. Heat and material balances for Test 39 are included and also some operating information for the test in Figures 69 through 72 and Tables 35 and 36.

Test 40

Having successfully completed Test 39, it was once again time to try char. Accordingly, the storage system was loaded up with 17 to 18 tons of -12 mesh FMC char. The objective was to operate with steam-oxygen and to use as high an oxygen concentration as was needed to achieve the bed temperatures necessary to yield ash agglomerates. Furthermore, fines carry-over was to be minimized, as much as possible, by using as low a superficial velocity as possible consistent with sufficient fluidized-bed turbulence.

As with coke, autogenous operation was achieved with air and switched to steam-oxygen operation 24 hours later. A 21% oxygen with 79% steam mixture was used for 24 hours. During this same period, the dust removal rate was gradually reduced to 120 lb/hr by reducing the steam-oxygen input, obtaining an operating superficial velocity of 1.0 ft/s. The bed temperature was comparatively low (1600°F) although the reactor gas composition produced was similar to that obtained with coke at temperatures in excess of 1850°F. (See Test 39.)

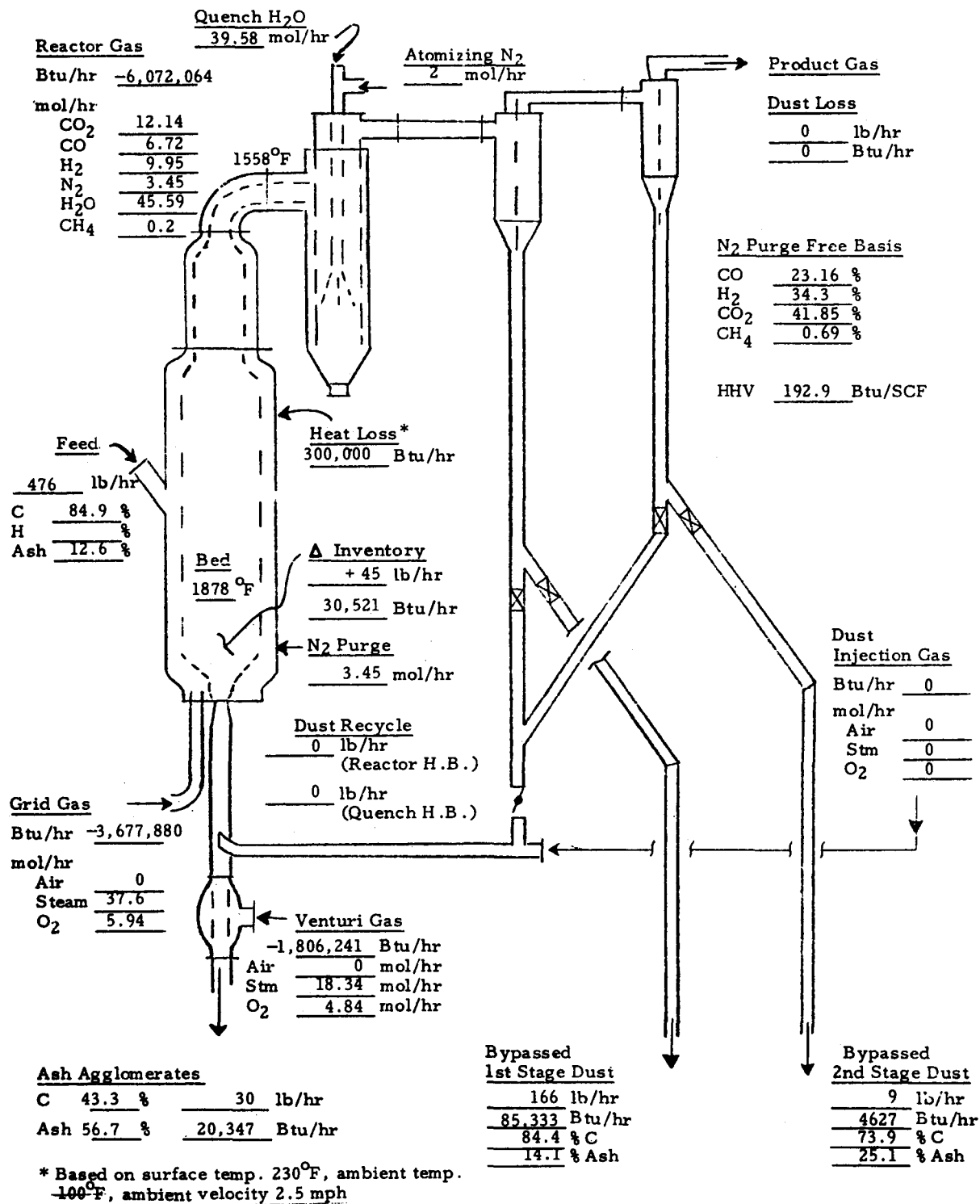


Figure 67. ASH-AGGLOMERATING GASIFIER TEST 38
FROM 0200 to 0800 HOURS (April 17, 1975)

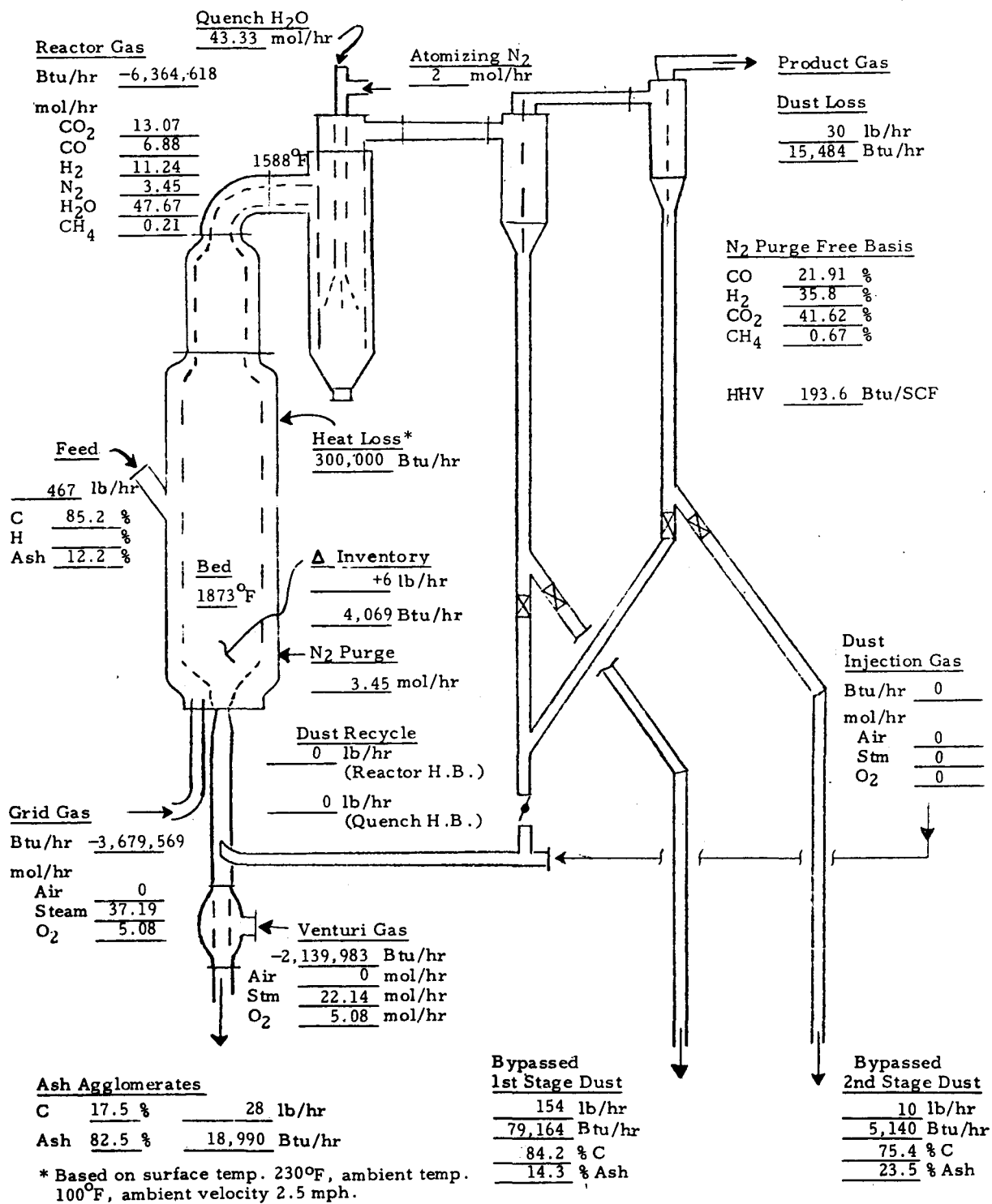


Figure 68. ASH-AGGLOMERATING GASIFIER TEST 38
FROM 0200 TO 0800 HOURS (April 18, 1975)

Table 34, Part 1. ASH-AGGLOMERATING GASIFIER DATA SUMMARY
FOR TEST 38

Date April 16, 1975

Time	0400	0400	0600	0800	1000	1200	1400	1600
TE 9206.6 Bed Temperature, °F	276	290	585	1258	1511	1776	1836	1866
TE 9206.18 Gas Temperature-Air Heater, °F	627	743	699	754	794	791	778	747
TE 9206.1 Gas Temp. Entering Venturi, °F	415	432	430	517	639	703	667	653
TE 9206.2 Gas Temp. Entering Grid, °F	450	495	505	488	546	526	564	565
PI 913 & PDR 910 Pressure in the Venturi, psig	3.4	3.2	3.0	3.0	3.2	3.9	4.2	4.3
PI 905 Press. at Air Heater Exit, psig	11.1	11.1	11.4	11.0	11.1	10.9	14.0	9.3
FI 904 (FR 905 + FR 906 - FR 938) Stm Flow to Air Heater, lb/hr	0	0	0	0	0	0	--	864
FR 903 @ 40 psig & 300°F N ₂ /Stm Flow to Grid, lb/hr	427	427	357	357	290	290	290	290
FR 905 Enriched Air to Grid, lb/hr	926	887	797	779	750	724	--	690
FR 906 Enriched Air to Venturi, lb/hr	700	678	478	608	590	632	--	484
FR 995 Total 21% Air to Air Heater, lb/hr	1330	1260	940	984	1183	--	710	0
FR 938 Total O ₂ to Air Heater, lb/hr	0	0	0	0	0	0	161	310
N ₂ Turge to Reactor, mole/hr	3.45	3.45	3.45	3.45	3.45	3.45	3.45	3.45
FR 918 Rate of Quench Water, lb/hr	0	0	0	0	90	260	400	590
PDR 912/PDR 913 X 3 33 Bed Height, ft	--	4.4	4.32	3.69	4.15	4.12	4.14	3.94
PDR 913 X 43.3 Bed Density, lb/cu ft	--	26.85	32.04	36.37	36.81	36.37	33.77	30.74
Coke Feed Rate, lb/hr (average)	600	0	0	0	280	280	280	280
Reactor Gas Composition, % (Dry) CO ₂ (ANR 975)					17.2	16	22.5	36
CO (ANR 977)					2	7.5	13.5	20.5
H ₂ (ANR 981)					0	6.2	16	30
O ₂ (ANR 978)					0	0	0	0
N ₂ (By Difference)					80.8	70.3	48	13.5
1st Stage Dust Removal Rate, lb/hr	42	42	42	42	141	141	141	141
2nd Stage Dust Removal Rate, lb/hr	7	7	7	7	7	7	7	7
C-4 Discharge Rate, lb/hr	0	0	37	35	31	23	12	22
Reactor Bed Composition: %C								
%Ash								
Feed Composition: %C								
%Ash								
Ash Composition: %C								
%Ash								
TE 9206.16 Gas Temp. Entering Quencher, °F	199	242	341	521	744	977	1201	1368
PIC 901 Blower Outlet Pressure, psig	14.8	14.8	14.8	13.5	13.0	14.5	15.5	--
TI 9203 Blower Outlet Temperature, °F	260	265	260	245	240	265	285	--
PI 9122 O ₂ Supply Pressure, psig	--	--	--	--	--	--	34	40
TE 9206.4 O ₂ Supply Temperature, °F	--	--	--	--	--	--	77	66

Table 34, Part 2. ASH-AGGLOMERATING GASIFIER DATA SUMMARY
FOR TEST 38

Date April 16-17, 1975

Time	1800	2000	2200	0000	0200	0400	0600	0800
TE 9206.6 Bed Temperature, °F	1856	1831	1856	1876	1861	1875	1877	1876
TE 9206.18 Gas Temperature-Air Heater, °F	779	775	772	770	773	777	789	782
TE 9206.1 Gas Temp. Entering Venturi, °F	652	668	679	667	657	658	672	662
TE 9206.2 Gas Temp. Entering Grid, °F	571	569	570	566	571	572	579	579
PI 913 & PDR 910 Pressure in the Venturi, psig	4.0	4.1	4.1	4.1	4.1	4.1	4.0	4.2
PI 905 Press. at Air Heater Exit, psig	10.2	10.2	10.4	10.5	10.2	10.4	10.4	10.6
FI 904 (FR 905 + FR 906 - FR 938) Stm Flow to Air Heater, lb/hr	756	756	746	726	746	726	726	746
FR 903 @ 40 psig & 300°F N ₂ /Stm Flow to Grid, lb/hr	271	271	271	271	271	271	271	271
FR 905 Enriched Air to Grid, lb/hr	586	586	586	586	586	586	586	586
FR 906 Enriched Air to Venturi, lb/hr	495	495	495	495	495	495	495	495
FR 995 Total 21% Air to Air Heater, lb/hr	0	0	0	0	0	0	0	0
FR 938 Total O ₂ to Air Heater, lb/hr	325	325	335	355	335	355	355	335
N ₂ Purge to Reactor, mole/hr	3.45	3.45	3.45	3.45	3.45	3.45	3.45	3.45
FR 918 Rate of Quench Water, lb/hr	620	620	670	700	700	700	700	750
PDR 912/PDR 913 X 3 33 Bed Height, ft	3.78	4.04	4.08	4.13	3.86	3.95	3.95	3.95
PDR 913 X 43.3 Bed Density, lb/cu ft	31.6	32.04	32.47	32.04	32.04	32.04	32.04	36.04
Coke Feed Rate, lb/hr (average)	400	500	500	500	457	457	457	457
Reactor Gas Composition, % (Dry) CO ₂ (ANR 975)	37.7	39	39	37	37.7	37.7	37	37
CO (ANR 977)	19.5	19.5	20	20.5	21.2	20	20.5	20.5
H ₂ (ANR 981)	29.2	26.5	28.5	30.5	30.5	30.5	30.5	32
O ₂ (ANR 978)	0	0	0	0	0	0	0	0
N ₂ (By Difference)	13.6	15	12.5	12.0	10.6	11.8	12.0	10.5
1st Stage Dust Removal Rate, lb/hr	181	181	181	181	166	166	166	166
2nd Stage Dust Removal Rate, lb/hr	7	7	7	7	7	7	10	10
C-4 Discharge Rate, lb/hr	21	21	21	25	24	27	33	34
Reactor Bed Composition:	%C							
	%Ash							
Feed Composition:	%C							
	%Ash							
Ash Composition:	%C							
	%Ash							
TE 9206.16 Gas Temp. Entering Quencher, °F	1418	1462	1491	1530	1550	1555	1563	1564
PIC 901 Blower Outlet Pressure, psig	--	--	--	--	--	--	--	--
TI 9203 Blower Outlet Temperature, °F	--	--	--	--	--	--	--	--
PI 9122 O ₂ Supply Pressure, psig	40	41	41	40	39	40	41	40.5
TE 9206.4 O ₂ Supply Temperature, °F	59	54	52	52	50	52	54	57

Table 34, Part 3. ASH-AGGLOMERATING GASIFIER DATA SUMMARY
FOR TEST 38

Date: April 17, 1975

Time	1000	1200	1400	1600	1800	2000	2200	0000
TE 9206.6 Bed Temperature, °F	1866	1870	1874	1872	1878	1884	1880	1875
TE 9206.18 Gas Temperature-Air Heater, °F	786	790	781	777	781	778	779	776
TE 9206.1 Gas Temp. Entering Venturi, °F	649	651	630	623	630	631	629	638
TE 9206.2 Gas Temp. Entering Grid, °F	580	579	570	565	568	568	567	566
PI 913 & PDR 910 Pressure in the Venturi, psig	4.3	4.3	4.4	4.4	4.4	4.4	4.4	4.4
PI 905 Press. at Air Heater Exit, psig	10.9	11	12	12	12.5	12.4	12.2	12.1
FI 904 (FR 905 + FR 906 - FR 938) Stm Flow to Air Heater, lb/hr	760	760	786	786	786	784	784	786
FR 903 @ 40 psig & 300°F N ₂ /Stm Flow to Grid, lb/hr	271	271	271	271	271	271	271	271
FR 905 Process Gas to Grid, lb/hr	573	573	556	556	556	556	556	556
FR 906 Process Gas to Venturi, lb/hr	507	507	556	556	556	556	556	556
FR 995 Total 21% Air to Air Heater, lb/hr	0	0	0	0	0	0	0	0
FR 938 Total O ₂ to Air Heater, lb/hr	320	320	326	326	326	328	328	326
N ₂ Surge to Reactor, mole/hr	3.45	3.45	3.45	3.45	3.45	3.45	3.45	3.45
FR 918 Rate of Quench Water, lb/hr	730	730	760	770	770	770	770	770
PDR 912/PDR 913 X 3 33 Bed Height, ft	3.95	4.0	4.14	4.08	4.0	3.95	3.91	3.87
PDR 913 X 43.3 Bed Density, lb/cu ft	32.48	32.48	32.04	32.48	32.48	32.48	32.48	32.04
Coke Feed Rate, lb/hr (average)	480	480	480	480	467	467	467	467
Reactor Gas Composition, % (Dry) CO ₂ (ANR 975)	37	37	37	39	39	39	39	39
CO (ANR 977)	20	19.5	19.5	19.5	20	20	19.5	19.5
H ₂ (ANR 981)	31	32	32	32	31	32	32	32
O ₂ (ANR 978)	0	0	0	0	0	0	0	0
N ₂ (By Difference)	12.0	11.5	11.5	9.5	10	9	9.5	9.5
1st Stage Dust Removal Rate, lb/hr	173	173	173	173	174	174	174	174
2nd Stage Dust Removal Rate, lb/hr	10	10	10	10	10	10	10	10
C-4 Discharge Rate, lb/hr	29	30	24	18	20	20	21	27
Reactor Bed Composition:	%C							
	%Ash							
Feed Composition:	%C							
	%Ash							
Ash Composition:	%C							
	%Ash							
TE 9206.16 Gas Temp. Entering Quencher, °F	1571	1574	1577	1580	1584	1590	1588	1587
PIC 901 Blower Outlet Pressure, psig	--	--	--	--	--	--	--	--
TI 9203 Blower Outlet Temperature, °F	--	--	--	--	--	--	--	--
PI 9122 O ₂ Supply Pressure, psig	42	42	74	72	75	76	74	74
TE 9206.4 O ₂ Supply Temperature, °F	65	68	74	79	80	72	68	65

Table 34, Part 4. ASH-AGGLOMERATING GASIFIER DATA SUMMARY
FOR TEST 38

Date April 18, 1975

Time	0200	0400	0600	0800	1000	1200	1400	1600
TE 9206.6 Bed Temperature, °F	1874	1871	1870	1877	1877	1881	1887	1884
TE 9206.18 Gas Temperature-Air Heater, °F	775	773	775	779	768	801	803	807
TE 9206.1 Gas Temp. Entering Venturi, °F	629	628	634	634	629	621	629	629
TE 9206.2 Gas Temp. Entering Grid, °F	565	561	566	568	563	574	581	584
PI 913 & PDR 910 Pressure in the Venturi, psig	4.4	4.4	4.4	4.5	4.5	4.6	4.6	4.6
PI 905 Press. at Air Heater Exit, psig	12.1	12.1	12.1	12.1	12.5	13.2	12.9	12.8
FI 904 (FR 905 + FR 906 - FR 938) Stm Flow to Air Heater, lb/hr	797	782	786	782	785	777	790	800
FR 903 @ 40 psig & 300°F N ₂ /Stm Flow to Grid, lb/hr	271	271	271	271	271	271	271	271
FR 905 Process Gas to Grid, lb/hr	556	556	556	556	552	552	552	552
FR 906 Process Gas to Venturi, lb/hr	556	556	556	556	563	561	574	574
FR 995 Total 21% Air to Air Heater, lb/hr	0	0	0	0	0	0	0	0
FR 938 Total O ₂ to Air Heater, lb/hr	315	330	326	330	330	336	336	326
N ₂ Surge to Reactor, mole/hr	3.45	3.45	3.45	3.45	3.45	3.45	3.45	3.45
FR 918 Rate of Quench Water, lb/hr	780	770	780	780	780	780	780	780
PDR 912/PDR 913 X 3 33 Bed Height, ft	4.08	4.13	4.17	4.17	4.11	4.06	4.02	3.98
PDR 913 X 43.3 Bed Density, lb/cu ft	32.48	32.48	32.48	32.48	31.61	31.61	31.18	31.18
Coke Feed Rate, lb/hr (average)	467	467	467	467	467	467	467	467
Reactor Gas Composition, % (Dry) CO ₂ (ANR 975)	37	38	38	37	36	35.2	35.2	35.2
CO (ANR 977)	20	19.5	19.5	20	20	21.5	20	20.7
H ₂ (ANR 981)	33.2	32	32	32	31	32.5	32	32.5
O ₂ (ANR 978)	0	0	0	0	0	0	0	0
N ₂ (By Difference)	9.8	10.5	10.5	11	13	10.8	12.8	11.6
1st Stage Dust Removal Rate, lb/hr	154	154	154	154	154	154	154	154
2nd Stage Dust Removal Rate, lb/hr	10	10	10	10	10.5	10.5	10.5	10.5
C-4 Discharge Rate, lb/hr	26	27	28	30	27	27	27	27
Reactor Bed Composition: %C								
%Ash								
Feed Composition: %C								
%Ash								
Ash Composition: %C								
%Ash								
TE 9206.16 Gas Temp. Entering Quencher, °F	1586	1588	1589	1593	1593	1594	1594	1594
PIC 901 Blower Outlet Pressure, psig	--	--	--	--	--	--	--	--
TI 9203 Blower Outlet Temperature, °F	--	--	--	--	--	--	--	--
PI 9122 O ₂ Supply Pressure, psig	72	72	73	73	73	72	74	73
TE 9206.4 O ₂ Supply Temperature, °F	64	65	65	65	70	70	69	70

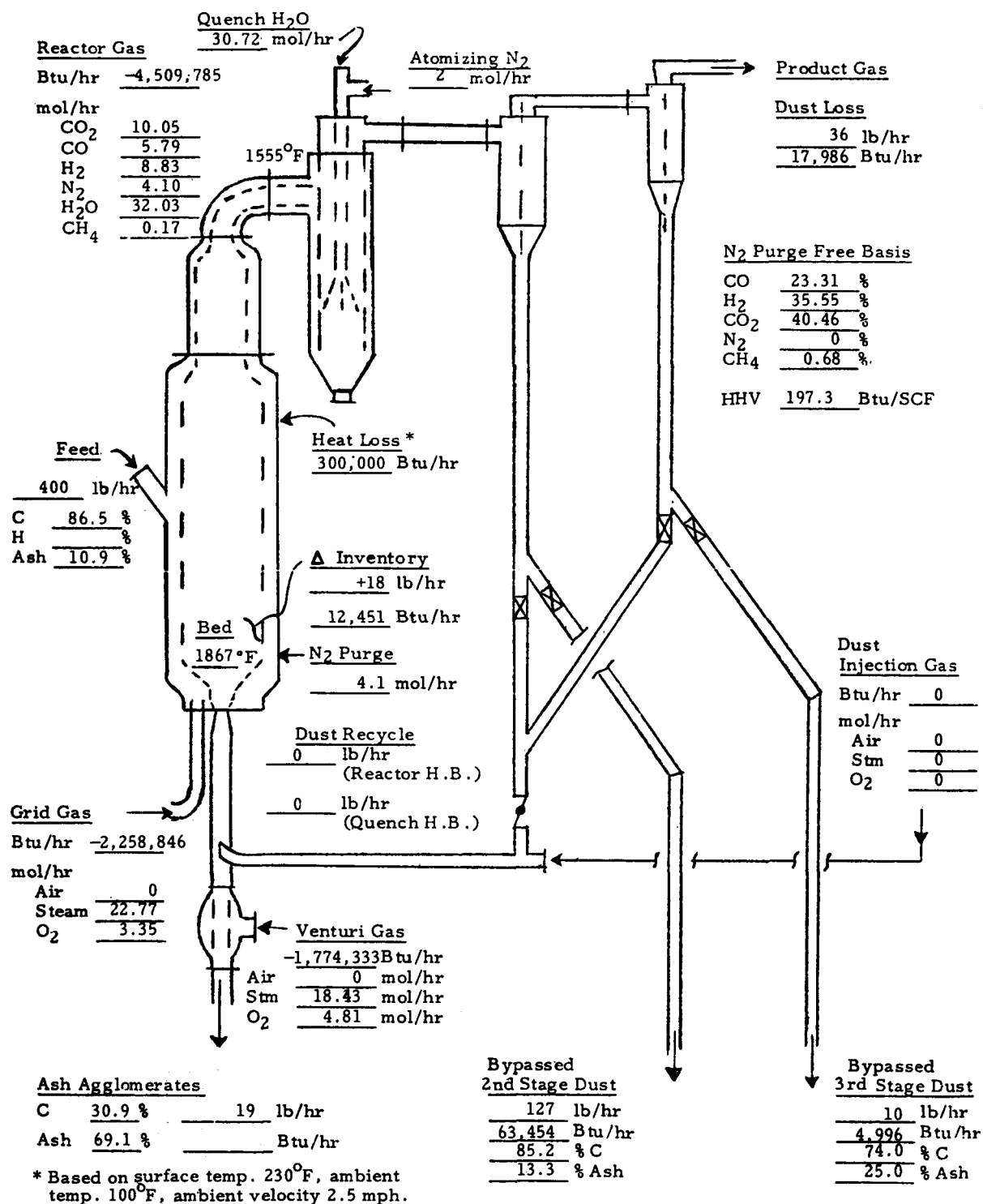


Figure 69. ASH-AGGLOMERATING GASIFIER TEST 39
FROM 0200 TO 0600 HOURS (April 30, 1975)

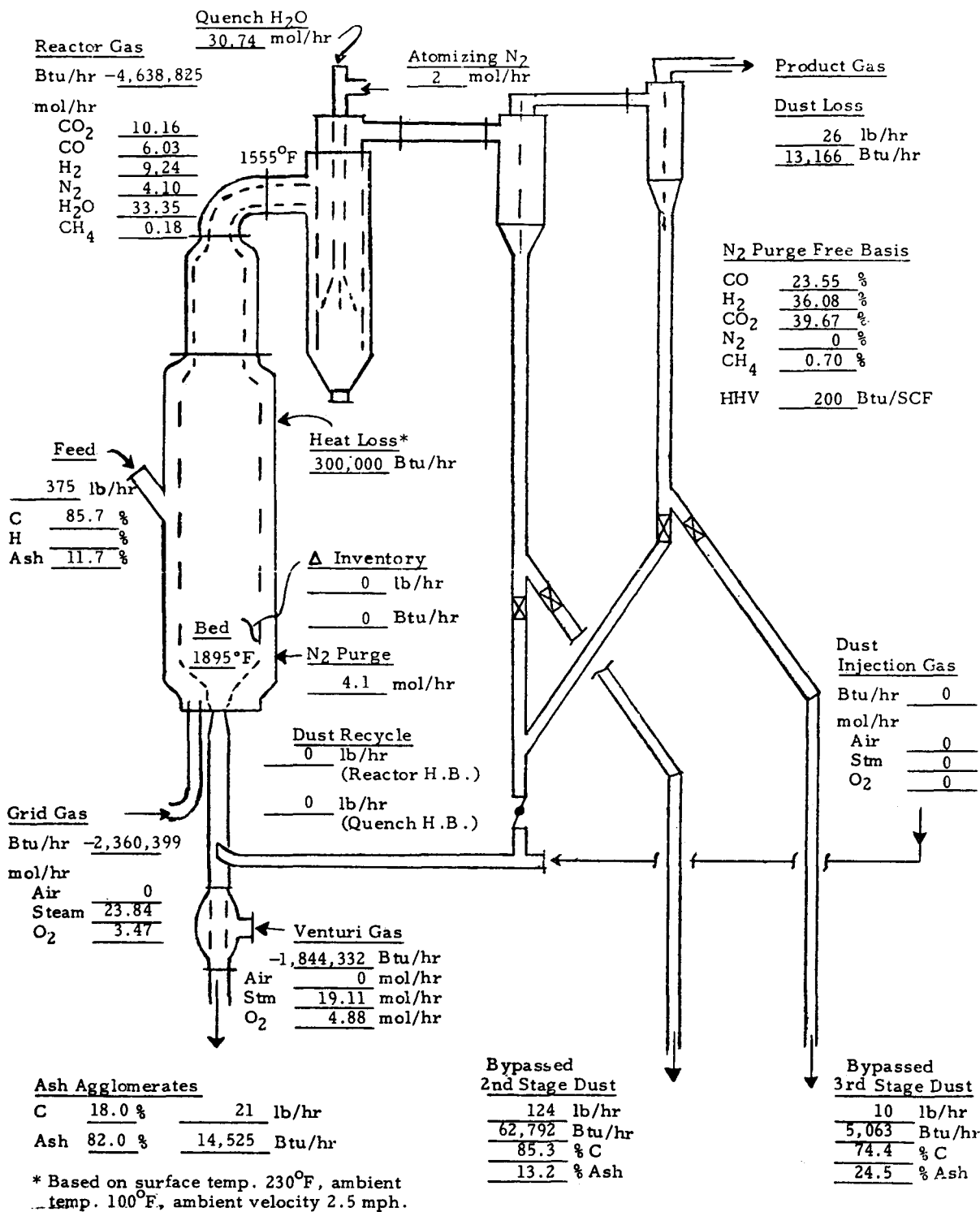


Figure 70. ASH-AGGLOMERATING GASIFIER TEST 39
FROM 1200 TO 1600 HOURS (April 30, 1975)

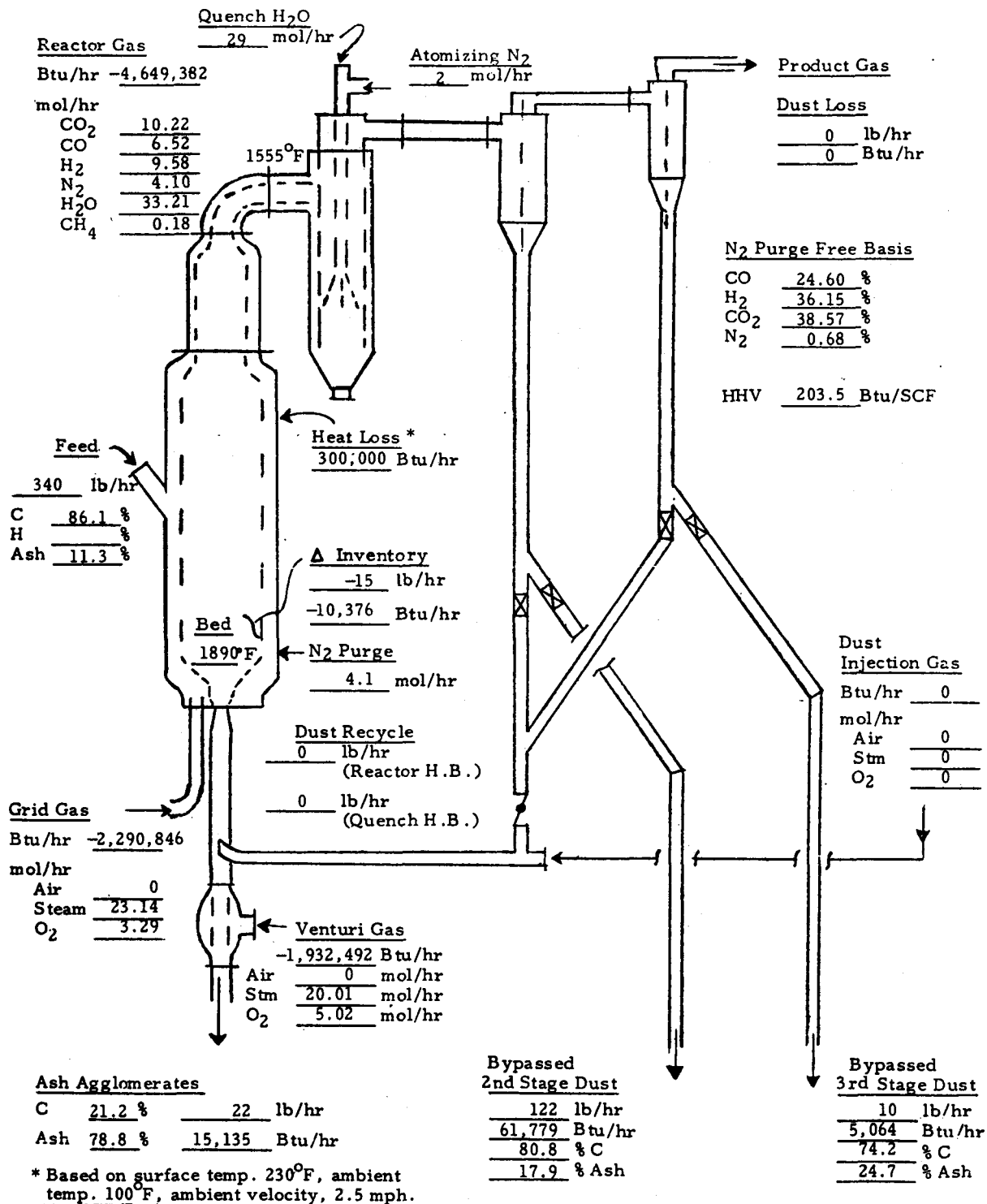


Figure 71. ASH-AGGLOMERATING GASIFIER TEST 39
FROM 0400 TO 1000 HOURS (May 1, 1975)

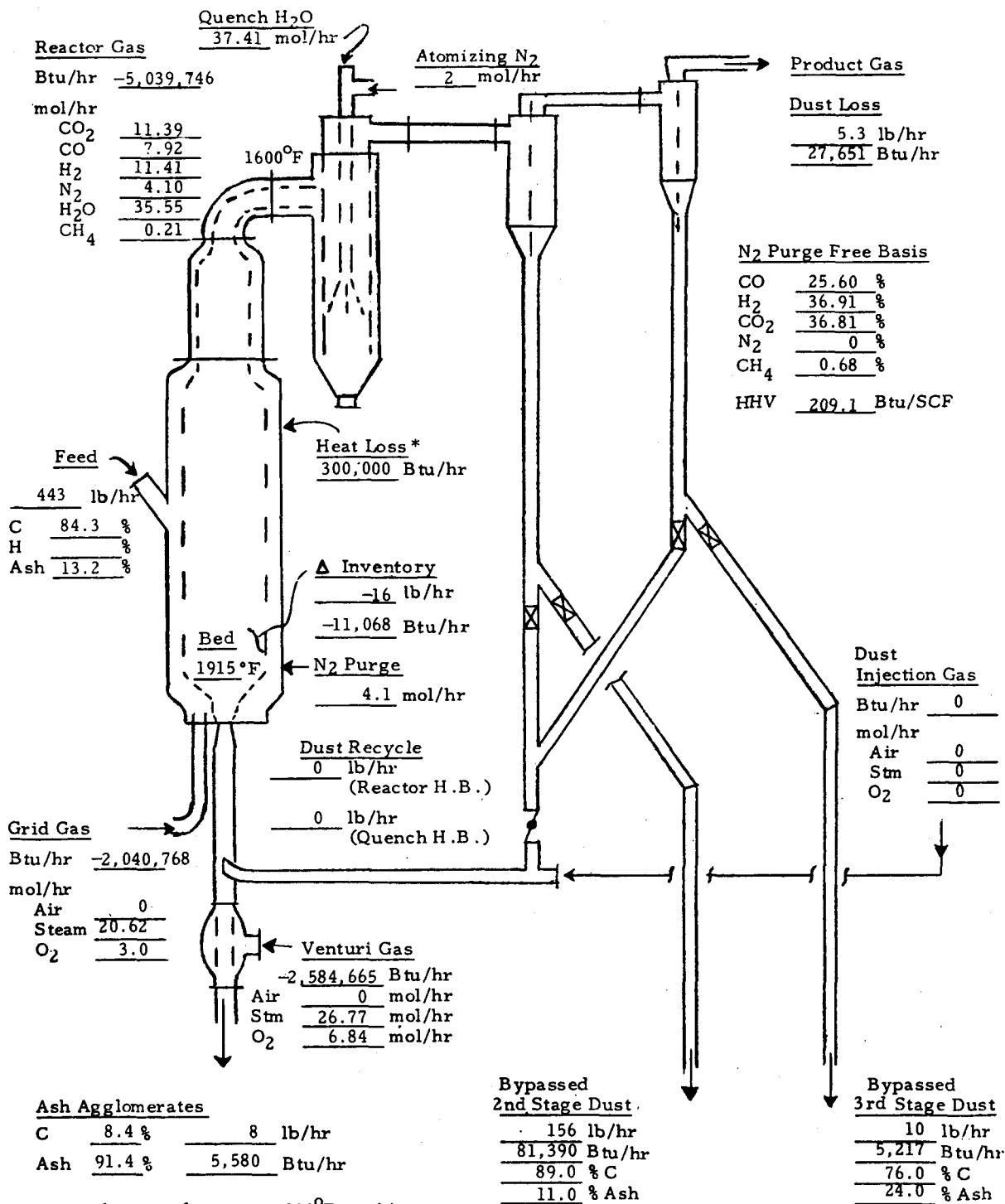


Figure 72. ASH-AGGLOMERATING GASIFIER TEST 39
FROM 1200 TO 1600 HOURS (May 2, 1975)

Table 35, Part 1. ASH-AGGLOMERATING GASIFIER DATA SUMMARY
FOR TEST 39

Date 4/29/75-4/30/74

Time	1600	1800	2000	2200	0000	0200	0400	0600
TE 9206.6								
Bed Temperature, °F	1861	1872	1867	1878	1871	1864	1861	1876
TE 9206.18								
Gas Temperature-Air Heater, °F	781	784	784	777	776	777	778	781
TE 9206.1								
Gas Temp. Entering Venturi, °F	694	664	626	623	612	614	633	636
TE 9206.2								
Gas Temp. Entering Grid, °F	554	549	539	539	531	534	534	534
PI 913 & PDR 910								
Pressure in the Venturi, psig	3.4	3.4	3.4	3.4	3.4	3.4	3.5	3.5
PI 905								
Press. at Air Heater Exit, psig	7.9	8.5	9.4	9.6	10.5	10.3	10.3	10.3
FI 904								
Steam Flow to Air Heater, lb/hr	586	594	596	596	569	567	559	559
FR 903 @ 70° psig & 300°F								
Steam Flow to Grid, lb/hr	225	180	180	180	180	180	180	180
FR 905								
Enriched Air to Grid, lb/hr	454	408	378	378	338	338	338	338
FR 906								
Enriched Air to Venturi, lb/hr	408	443	479	489	492	490	482	482
FR 995								
Total 21% Air to Air Heater, lb/hr	0	0	0	0	0	0	0	0
FR 938								
Total O ₂ to Air Heater, lb/hr	276	257	261	261	261	261	261	261
N ₂ Purge to Reactor, mole/hr	4.1	4.1	4.1	4.1	4.1	4.1	4.1	4.1
FR 918								
Rate of Quench Water, lb/hr	550	540	550	560	550	540	560	560
PDR 912/PDR 913 X 3 33								
Bed Height, ft	3.95	3.93	3.87	3.96	4.05	4.32	4.5	4.5
PDR 913 X 43.3								
Bed Density, lb/cu ft	30.31	31.18	32.04	32.04	32.04	32.04	32.04	32.04
Coke Feed Rate, lb/hr (average)	400	400	400	400	400	400	400	400
Reactor Gas Composition, % (Dry)								
CO ₂ (ANR 975)	35.2	34.5	35.2	34.5	34.5	34.5	35.2	34.5
CO (ANR 977)	19.5	19.5	19.5	20.5	20.5	20	20	20
H ₂ (ANR 981)	30	30	30	31.0	31.0	30.5	30.5	30.5
CH ₄ (ANR 978)	0.6	0.6	0.6	0.6	0.6	0.6	0.6	0.6
N ₂ (By Difference)	14.7	15.4	14.7	13.4	13.4	14.4	13.7	14.4
1st Stage Dust Removal Rate, lb/hr	122	122	122	122	122	127	127	127
2nd Stage Dust Removal Rate, lb/hr	10	10	10	10	10	10	10	10
C-4 Discharge Rate, lb/hr	29	23	18	17	16	17	19	20
Reactor Bed Composition:	%C							
Feed Composition:	%C							
Ash Composition:	%Ash							
TE 9206.16								
Gas Temp. Entering Quencher, °F	1513	1513	1516	1524	1521	1530	1534	1541
PIC 901								
Blower Outlet Pressure, psig	--	--	--	--	--	--	--	--
TI 9203								
Blower Outlet Temperature, °F	--	--	--	--	--	--	--	--
PI 9122								
O ₂ Supply Pressure, psig	76	78	73	77	78	73	75	75
TE 9206.4								
O ₂ Supply Temperature, °F	84	82	72	60	61	63	65	63

Table 35, Part 2. ASH-AGGLOMERATING GASIFIER DATA SUMMARY
FOR TEST 39

Date 4/30/75

Time	0800	1000	1200	1400	1600	1800	2000	2200
TE 9206.6 Bed Temperature, °F	1897	1904	1896	1893	1896	1903	1887	1885
TE 9206.18 Gas Temperature-Air Heater, °F	777	797	800	796	795	801	800	802
TE 9206.1 Gas Temp. Entering Venturi, °F	632	610	617	628	633	620	608	621
TE 9206.2 Gas Temp. Entering Grid, °F	533	541	542	544	541	537	531	532
PI 913 & PDR 910 Pressure in the Venturi, psig	3.7	3.6	3.5	3.5	3.5	3.5	3.5	3.5
PI 905 Press. at Air Heater Exit, psig	10	10.2	10.1	10.2	10.3	10.5	10.6	10.5
FI 904 Steam Flow to Air Heater, lb/hr	585	588	588	588	588	591	588	588
FR 903 @ 70° psig & 300°F Steam Flow to Grid, lb/hr	185	185	185	185	185	185	185	185
FR 905 Enriched Air to Grid, lb/hr	358	355	355	355	355	338	338	338
FR 906 Enriched Air to Venturi, lb/hr	494	500	500	500	500	520	520	520
FR 995 Total 21% Air to Air Heater, lb/hr	0	0	0	0	0	0	0	0
FR 938 Total O ₂ to Air Heater, lb/hr	267	267	267	267	267	267	270	270
N ₂ Purge to Reactor, mole/hr	4.1	4.1	4.1	4.1	4.1	4.1	4.1	4.1
FR 918 Rate of Quench Water, lb/hr	610	610	550	510	600	570	520	520
PDR 912/PDR 913 X 3 33 Bed Height, ft	4.19	3.98	3.89	3.94	3.89	3.89	3.82	3.91
PDR 913 X 43.3 Bed Density, lb/cu ft	32.04	31.18	31.18	30.74	31.18	31.18	32.48	32.48
Coke Feed Rate, lb/hr (average)	311	311	375	375	375	375	400	400
Reactor Gas Composition, % (Dry) CO ₂ (ANR 975)	33.5	32.7	34.2	34.2	34.2	34.2	34.5	34.5
CO (ANR 977)	20.7	20.5	20	20.2	20.7	20.7	20.7	22
H ₂ (ANR 981)	31	31	31.2	31	31.2	31.2	31.2	31.2
CH ₄ (ANR 978)	0.6	0.6	0.6	0.6	0.6	0.6	0.6	0.6
N ₂ (By Difference)	14.2	15.2	14	14	13.3	13.3	13	11.7
1st Stage Dust Removal Rate, lb/hr	127	124	124	124	124	114	114	114
2nd Stage Dust Removal Rate, lb/hr	10	10	10	10	10	10	10	10
C-4 Discharge Rate, lb/hr	20	19	18	22	22	19	20	20
Reactor Bed Composition:	%C							
Feed Composition:	%C							
Ash Composition:	%Ash							
TE 9206.16 Gas Temp. Entering Quencher, °F	1554	1561	1558	1554	1556	1556	1553	1549
PIC 901 Blower Outlet Pressure, psig	--	--	--	--	--	--	--	--
TI 9203 Blower Outlet Temperature, °F	--	--	--	--	--	--	--	--
PI 9122 O ₂ Supply Pressure, psig	73	76	73	74	77	78	76	74
TE 9206.4 O ₂ Supply Temperature, °F	62	72	75	74	73	68	58	57

Table 35, Part 3. ASH-AGGLOMERATING GASIFIER DATA SUMMARY
FOR TEST 39

Date 5/1/75

Time	0000	0200	0400	0600	0800	1000	1200	1400
TE 9206.6 Bed Temperature, °F	1882	1878	1878	1881	1894	1895	1895	1887
TE 9206.18 Gas Temperature-Air Heater, °F	799	797	799	797	796	794	794	797
TE 9206.1 Gas Temp. Entering Venturi, °F	622	623	620	618	617	599	594	586
TE 9206.2 Gas Temp. Entering Grid, °F	535	534	534	534	534	526	529	527
PI 913 & PDR 910 Pressure in the Venturi, psig	3.7	3.7	3.7	3.7	3.7	3.7	3.6	3.6
PI 905 Press. at Air Heater Exit, psig	10.5	11	10.6	10.8	10.5	11.1	11	11
FI 904 Steam Flow to Air Heater, lb/hr	597	597	602	594	598	593	588	588
FR 903 @ 70 psig & 300°F Steam Flow to Grid, lb/hr	190	190	190	190	190	190	190	190
FR 905 Enriched Air to Grid, lb/hr	341	345	342	344	343	323	323	323
FR 906 Enriched Air to Venturi, lb/hr	520	525	521	523	520	535	535	535
FR 995 Total 21% Air to Air Heater, lb/hr	0	0	0	0	0	0	0	0
FR 938 Total O ₂ to Air Heater, lb/hr	264	273	261	273	265	265	270	270
N ₂ Purge to Reactor, mole/hr	4.1	4.1	4.1	4.1	4.1	4.1	4.1	4.1
FR 918 Rate of Quench Water, lb/hr	520	500	510	500	550	530	490	500
PDR 912/PDR 913 X 3 33 Bed Height, ft	4.0	4.17	4.22	4.22	4.17	4.0	3.87	3.88
PDR 913 X 43.3 Bed Density, lb/cu ft	32.48	32.48	32.48	32.48	32.48	32.48	32.04	31.61
Coke Feed Rate, lb/hr (average)	400	400	340	340	340	340	356	356
Reactor Gas Composition, % (Dry) CO ₂ (ANR 975)	33.5	33.5	33.5	33.5	33.2	33.2	33.2	33.2
CO (ANR 977)	21.2	21.2	21.2	21.2	21.5	21.5	21.7	21.5
H ₂ (ANR 981)	31.7	31.7	30.5	31.2	31.7	31.7	31.7	31.7
CH ₄ (ANR 978)	0.6	0.6	0.6	0.6	0.6	0.6	0.6	0.6
N ₂ (By Difference)	13	13	14.2	13.5	13	13	12.8	13
1st Stage Dust Removal Rate, lb/hr	114	114	114	114	129	129	129	129
2nd Stage Dust Removal Rate, lb/hr	10	10	10	10	10	10	10	10
C-4 Discharge Rate, lb/hr	21	20	20	24	24	20	18	19
Reactor Bed Composition:	%C	%C	%C	%C	%C	%C	%C	%C
Feed Composition:	%C	%C	%C	%C	%C	%C	%C	%C
Ash Composition:	%C	%C	%C	%C	%C	%C	%C	%C
TE 9206.16 Gas Temp. Entering Quencher, °F	1550	1548	1546	1552	1559	1560	1560	1559
PIC 901 Blower Outlet Pressure, psig	--	--	--	--	--	--	--	--
TI 9203 Blower Outlet Temperature, °F	--	--	--	--	--	--	--	--
PI 9122 O ₂ Supply Pressure, psig	71	76	68	75	72	72	75	77
TE 9206.4 O ₂ Supply Temperature, °F	61	54	53	48	62	64	64	67

Table 35, Part 4. ASH-AGGLOMERATING GASIFIER DATA SUMMARY
FOR TEST 39

Date 5/1/75-5/2/75

Time	1600	1800	2000	2200	0000	0200	0400	0600
TE 9206.6								
Bed Temperature, °F	1893	1893	1897	1895	1901	1899	1892	1894
TE 9206.18								
Gas Temperature-Air Heater, °F	794	795	796	794	793	798	794	797
TE 9206.1								
Gas Temp. Entering Venturi, °F	594	612	594	599	587	582	593	593
TE 9206.2								
Gas Temp. Entering Grid, °F	529	531	520	517	515	514	515	517
PI 913 & PDR 910								
Pressure in the Venturi, psig	3.5	3.6	3.7	3.7	3.6	3.7	3.7	3.7
PI 905								
Press. at Air Heater Exit, psig	10.9	10.7	10.8	10.9	12	12.5	12.5	12.5
FI 904								
Steam Flow to Air Heater, lb/hr	588	588	585	584	596	605	604	607
FR 903 @ 70 psig & 300°F								
Steam Flow to Grid, lb/hr	190	190	190	190	190	190	190	190
FR 905								
Enriched Air to Grid, lb/hr	326	326	296	296	300	303	303	303
FR 906								
Enriched Air to Venturi, lb/hr	530	530	561	561	575	580	585	582
FR 995								
Total 21% Air to Air Heater, lb/hr	0	0	0	0	0	0	0	0
FR 938								
Total O ₂ to Air Heater, lb/hr	268	268	272	273	279	278	279	278
N ₂ Purge to Reactor, mole/hr	4.1	4.1	4.1	4.1	4.1	4.1	4.1	4.1
FR 918								
Rate of Quench Water, lb/hr	500	530	550	520	560	560	770	590
PDR 912/PDR 913 X 3 33								
Bed Height, ft	3.77	3.91	3.95	3.94	4.08	3.97	3.97	4.02
PDR 913 X 43.3								
Bed Density, lb/cu ft	32.48	32.48	32.48	32.91	32.91	33.77	33.77	33.34
Coke Feed Rate, lb/hr (average)	356	400	400	400	400	400	400	420
Reactor Gas Composition, % (Dry)								
CO ₂ (ANR 975)	33.2	33.5	33.5	33.5	32.7	32.7	32.7	32.7
CO (ANR 977)	21.2	20.7	22	22.5	23	22.7	23	22.5
H ₂ (ANR 981)	31.7	31.7	31.7	31.7	31.7	31.7	31.7	32.5
CH ₄ (ANR 978)	0.6	0.6	0.6	0.6	0.6	0.6	0.6	0.6
N ₂ (By Difference)	13.3	13.5	12.2	11.7	12	12.3	12	11.7
1st Stage Dust Removal Rate, lb/hr	129	129	129	129	156	156	156	156
2nd Stage Dust Removal Rate, lb/hr	10	10	10	10	10	10	10	10
C-4 Discharge Rate, lb/hr	20	22	19	16	17	17	18	19
Reactor Bed Composition:								
%C								
%Ash								
Feed Composition:								
%C								
%Ash								
Ash Composition:								
%C								
%Ash								
TE 9206.16								
Gas Temp. Entering Quencher, °F	1557	1553	1556	1558	1561	1563	1564	1564
PIC 901								
Blower Outlet Pressure, psig	--	--	--	--	--	--	--	--
TI 9203								
Blower Outlet Temperature, °F	--	--	--	--	--	--	--	--
PI 9122								
O ₂ Supply Pressure, psig	76	76	77	77	80	79	80	79
TE 9206.4								
O ₂ Supply Temperature, °F	73	74	63	58	53	51	55	57

Table 35, Part 5. ASH-AGGLOMERATING GASIFIER DATA SUMMARY
FOR TEST 39

Date 5/2/75

Time	0800	1000	1200	1400	1600			
TE 9206.6 Bed Temperature, °F	1877	1898	1915	1914	1916			
TE 9206.18 Gas Temperature-Air Heater, °F	796	790	786	785	786			
TE 9206.1 Gas Temp. Entering Venturi, °F	593	579	584	584	587			
TE 9206.2 Gas Temp. Entering Grid, °F	518	527	533	530	532			
PI 913 & PDR 910 Pressure in the Venturi, psig	3.8	4.1	4.3	4.2	4.3			
PI 905 Press. at Air Heater Exit, psig	12.1	17	17	17	16.6			
FI 904 Steam Flow to Air Heater, lb/hr	587	693	693	693	693			
FR 903 @ 70° psig & 300°F Steam Flow to Grid, lb/hr	190	160	160	160	160			
FR 905 Enriched Air to Grid, lb/hr	290	307	307	307	307			
FR 906 Enriched Air to Venturi, lb/hr	568	695	701	701	701			
FR 995 Total 21% Air to Air Heater, lb/hr	0	0	0	0	0			
FR 938 Total O ₂ to Air Heater, lb/hr	271	309	315	315	315			
N ₂ Purge to Reactor, mole/hr	4.1	4.1	4.1	4.1	4.1			
FR 918 Rate of Quench Water, lb/hr	520	600	710	670	640			
PDR 912/PDR 913 X 3 33 Bed Height, ft	3.98	4.01	3.9	3.91	3.87			
PDR 913 X 43.3 Bed Density, lb/cu ft	35.51	35.94	35.51	34.64	34.64			
Coke Feed Rate, lb/hr (average)	423	480	443	443	443			
Reactor Gas Composition, % (Dry) CO ₂ (ANR 975)	32.7	33.5	32.7	32.5	32.5			
CO (ANR 977)	20.7	21.5	22.5	22.7	22.5			
H ₂ (ANR 981)	30.7	31.2	32	32.5	33.2			
CH ₄ (ANR 978)	0.6	0.6	0.6	0.6	0.6			
N ₂ (By Difference)	15.3	13.2	12.2	11.7	11.2			
1st Stage Dust Removal Rate, lb/hr	156	156	156	156	156			
2nd Stage Dust Removal Rate, lb/hr	10	10	10	10	10			
C-4 Discharge Rate, lb/hr	19	14	5	8	10			
Reactor Bed Composition: %C								
%Ash								
Feed Composition: %C								
%Ash								
Ash Composition: %C								
%Ash								
TE 9206.16 Gas Temp. Entering Quencher, °F	1562	1574	1593	1599	1602			
PIC 901 Blower Outlet Pressure, psig	--	--	--	--	--			
TI 9203 Blower Outlet Temperature, °F	--	--	--	--	--			
PI 9122 O ₂ Supply Pressure, psig	77	67	70	70	71			
TE 9206.4 O ₂ Supply Temperature, °F	67	69	73	81	73			

Table 36. ASH-AGGLOMERATING GASIFICATION DATA SUMMARY
FOR TEST 39

Unit Size: 4-ft diameter x 21-ft high

Venturi Diameter 2 inches

Feed Material < 6M Coke Breeze

Cyclone Dust Collectors: 0 internal 2 external

1	Superficial Velocity, ft/s	1.7
2	Avg. Freeboard Temperature, °F	1825
3	Freeboard Pressure, psig	1.6
4	Grid Pressure Drop, psi	1.0
5	Venturi Pressure Drop, psi	0.6
6	Venturi Velocity, ft/s	204
7	Temperature of Gas Entering Venturi, °F	600
	Grid Gas Composition	
8	% O ₂	12.6
9	% N ₂	--
10	% Steam	87.4
	Venturi Gas Composition	
11	% O ₂	20.0
12	% N ₂	--
13	% Steam	80.0
14	Fluidized Bed Density, lb/CF	32.5
15	Fluidized Bed Height, ft	4.2
16	Avg. Fluidized Bed Temperature, °F	1890
17	Feed Composition, %C/% ash	86.1 / 11.3
18	Avg. Fluidized Bed Composition, %C/% ash	71.0 / 28.1
19	Venturi Discharge Composition, %C/% ash	21.2 / 78.8
	<u>1st</u> Stage Dust Composition, %C/% ash	80.8 / 17.9
	<u>2nd</u> Stage Dust Composition, %C/% ash	74.2 / 24.7
	Superficial Velocity/Minimum Fluidization Velocity	5.7

Mesh Size	6	12	20	40	70	100	140	200	270	40μ	20μ	10μ	5μ	-5μ
Feed	0	17.2	48.6	26.7	2.9		3.2		0.8	0.6				
Ash	53.4	36.5	5.8	1.0	0.7	2.6								
<u>1st</u> Stage Dust					2.9	16.2	20.9	13.9	9.6	0.7	17.0	13.7	4.6	0.6
<u>2nd</u> Stage Dust									1.0	49.4	2.7	7.4	22.1	17.4

Because there was no evidence of agglomerates during this 24-hour period of 21% oxygen operation, the concentration was raised to 25% oxygen, resulting in the bed temperature being raised to approximately 1680°F by the end of another 24-hour period. Again, there was no evidence of agglomerates, although the material falling through the venturi acquired a grayish cast. The increased oxygen throughput also required increased char feed to maintain the standard 4-foot bed level. There were also changes in product gas composition: carbon dioxide decreased from 30% to 27.5%, carbon monoxide increased from 21.5% to 24.5%, and hydrogen remained unchanged.

The oxygen concentration was then raised to 30%. (These changes are for the flow to the venturi only; the standard 12% oxygen concentration at the grid was not changed.) Again, the product gas hydrogen concentration was not changed, but the carbon dioxide decreased to 24.5% and the carbon monoxide increased to 28.0% within 8 hours of making this process change. At the time, the oxygen had to be cut back because the increased char feed requirements exceeded the feed conveyor capacity. The increased oxygen resulted in 30% increase in dust loss relative to that experienced when using a 21% oxygen concentration. The reactor bed temperature also showed little sign of rising past 1720°F.

With only 24 hours of run time remaining, the fluidized-bed level was slowly lowered, anticipating that a reduction in gas contact time would result in less carbon monoxide formed from the reaction of carbon dioxide and carbon. However, although the bed was lowered to approximately 30 inches from 48 inches and its temperature did rise to 1780°F, the bed ash content apparently was too high. The test was terminated after a sudden defluidization and clinkering. Gas analyzer data show, however, that at this time the carbon dioxide content (28%) exceeded that of carbon monoxide by approximately 1%.

Apparently, the right direction was being followed to raise the bed temperature, but a visual examination of the venturi discharge material failed to note its high ash content. Operation with char at lower bed levels could be successful in producing agglomerates if the bed ash content is held below the critical level.

A new high-temperature cyclone was installed in the reactor freeboard to return hot dust to the bed. Heat and material balances are included in Table 37 and Figures 73 and 74. Some operating information for Test 40 is also given in Table 38.

Test 41

The start-up with unscreened coke was routine, yet this test was terminated before agglomerates could be produced because a piece of refractory blocked the venturi. There was time to note, however, that only 50 lb/hr of dust was obtained at the first external (now second-stage) dust collector rather than the 120 lb/hr or more experienced earlier without the internal cyclone. The blocked venturi caused high-temperature, localized clinkering that overstressed and cracked the Incoloy grid. The missing refractory that had fallen was replaced, the grid rewelded, and the next test prepared.

**Table 37, Part 1. ASH-AGGLOMERATING GASIFIER DATA SUMMARY
FOR TEST 40**

Date 5/13/75

Time	0200	0400	0600	0800	1000	1200	1400	1600
TE 9206.6 Bed Temperature, °F	1636	1630	1681	1647	1614	1603	1611	1618
TE 9206.18 Gas Temperature-Air Heater, °F	633	820	715	807	500	837	828	826
TE 9206.1 Gas Temp. Entering Venturi, °F	773	784	746	759	764	878	826	785
TE 9206.2 Gas Temp. Entering Grid, °F	461	505	478	503	461	529	532	533
PI 913 & PDR 910 Pressure in the Venturi, psig	1.9	1.9	1.9	1.9	1.8	2.4	2.7	2.7
PI 905 Press. at Air Heater Exit, psig	10.2	10.5	10.2	10.2	1.6	2.1	2.5	3.7
FI 904 Steam Flow to Air Heater, lb/hr	0	0	0	0	371	317	349	349
FR 903 @ 70 psig & 300°F Steam Flow to Grid, lb/hr	234	234	234	218	224	184	199	199
FR 905 Enriched Air to Grid, lb/hr	544	506	535	531	311	297	291	301
FR 906 Enriched Air to Venturi, lb/hr	243	225	234	225	170	138	167	171
FR 995 Total 21% Air to Air Heater, lb/hr	835	778	806	778	0	0	0	0
FR 938 Total O ₂ to Air Heater, lb/hr	0	0	0	0	177	155	160	155
N ₂ Purge to Reactor, mole/hr	2.87	2.87	2.87	2.87	2.87	2.87	2.87	2.87
FR 918 Rate of Quench Water, lb/hr	240	240	240	260	280	180	260	260
PDR 912/PDR 913 X 3 33 Bed Height, ft	4.31	4.31	3.94	4.04	4.11	4.39	4.32	3.82
PDR 913 X 43.3 Bed Density, lb/cu ft	19.1	19.1	19.1	20.4	20.4	20.4	20.4	20.8
Coke Feed Rate, lb/hr (average)	348	348	348	348	348	348	250	250
Reactor Gas Composition, % (Dry) CO ₂ (ANR 975)	12.7	13.1	11.2	12.8	26	25.5	29.8	29.8
CO (ANR 977)	14.1	14	16.5	14.1	20.1	18.5	22	21.7
H ₂ (ANR 981)	13.2	13.2	15.5	14	30.5	29.7	37	36.2
CH ₄	0.2	0.2	0.2	0.2	0.2	0.8	0.8	0.9
H ₂ S	0	0	0	0.1	0.1	0.1	0.1	0.1
N ₂ (By Difference)	59.8	59.5	56.6	58.8	23.1	25.4	10.3	11.3
1st Stage Dust Removal Rate, lb/hr	161	161	161	111	111	111	111	111
2nd Stage Dust Removal Rate, lb/hr	6	6	6	6	6	6	6	6
C-4 Discharge Rate, lb/hr	15	14	13	17	27	39	26	19
Reactor Bed Composition:								
%C		61.0				59.2		
%Ash		36.2				37.8		
Feed Composition:								
%C							72.8	
%Ash							21.2	
Ash Composition:								
%C			43.2				42.1	
%Ash			54.1				54.8	
TE 9206.16 Gas Temp. Entering Quencher, °F	1171	1200	1234	1243	1261	1235	1235	1230
PIC 901 Blower Outlet Pressure, psig	11.5	11.5	11.5	11.5	--	--	--	--
TI 9203 Blower Outlet Temperature, °F	220	220	220	220	--	--	--	--
PI 9122 O ₂ Supply Pressure, psig	--	--	--	--	41	40	37	34
TE 9206.4 O ₂ Supply Temperature, °F	--	--	--	--	70	72	76	76

Table 37, Part 2. ASH-AGGLOMERATING GASIFIER DATA SUMMARY
FOR TEST 40

Date 5/13-14/75

Time	1800	2000	2200	2400	0200	0400	0600	0800
TE 9206.6 Bed Temperature, °F	1622	1618	1609	1613	1600	1608	1620	1628
TE 9206.18 Gas Temperature-Air Heater, °F	800	797	796	792	796	796	799	826
TE 9206.1 Gas Temp. Entering Venturi, °F	717	731	727	717	742	737	797	784
TE 9206.2 Gas Temp. Entering Grid, °F	523	519	516	512	513	512	524	536
PI 913 & PDR 910 Pressure in the Venturi, psig	2.9	2.9	2.9	3.0	2.9	3.0	2.8	2.7
PI 905 Press. at Air Heater Exit, psig	4.2	4.2	4.3	4.3	4.4	4.4	5.0	5.4
FI 904 Steam Flow to Air Heater, lb/hr	371	371	371	371	371	371	371	371
FR 903 @ 70' psig & 300°F Steam Flow to Grid, lb/hr	199	199	199	199	199	199	199	199
FR 905 Enriched Air to Grid, lb/hr	302	302	302	302	302	302	328	328
FR 906 Enriched Air to Venturi, lb/hr	207	207	207	207	207	207	185	185
FR 995 Total 21% Air to Air Heater, lb/hr	0	0	0	0	0	0	0	0
FR 938 Total O ₂ to Air Heater, lb/hr	166	166	168	168	168	168	172	172
N ₂ Purge to Reactor, mole/hr	2.87	2.87	2.87	2.87	2.87	2.87	2.87	2.87
FR 918 Rate of Quench Water, lb/hr	260	260	260	260	260	260	260	260
PDR 912/PDR 913 X 3 33 Bed Height, ft	3.73	3.84	4.03	4.1	4.23	4.35	4.03	3.93
PDR 913 X 43.3 Bed Density, lb/cu ft	21.7	22.5	22.5	22.5	22.5	22.5	22.5	21.7
Coke Feed Rate, lb/hr (average)	326	326	326	326	326	326	275	275
Reactor Gas Composition, % (Dry)								
CO ₂ (ANR 975)	31.2	31.2	31.2	30.5	31.2	30.5	29.8	29.8
CO (ANR 977)	21.5	21.7	21.5	21.7	21.5	21.7	22.5	22.5
H ₂ (ANR 981)	34.7	34.7	34.7	35.3	34.7	34.7	35.5	35.5
CH ₄	0.9	0.9	0.9	0.9	0.9	0.9	0.9	0.9
H ₂ S	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
N ₂ (By Difference)	11.6	11.4	11.6	11.5	11.6	12.1	11.2	11.2
1st Stage Dust Removal Rate, lb/hr	126	126	126	126	126	126	126	126
2nd Stage Dust Removal Rate, lb/hr	6	6	6	6	6	6	6	6
C-4 Discharge Rate, lb/hr	13	17	19	20	17	21	27	24
Reactor Bed Composition:								
%C		50.7						50.3
%Ash		46.8						46.8
Feed Composition:								
%C					73.5			
%Ash					20.9			
Ash Composition:								
%C				31.4				32.6
%Ash				66.2				65.1
TE 9206.16 Gas Temp. Entering Quencher, °F	1240	1240	1239	1244	1248	1247	1248	1252
PIC 901 Blower Outlet Pressure, psig	--	--	--	--	--	--	--	--
TI 9203 Blower Outlet Temperature, °F	--	--	--	--	--	--	--	--
PI 9122 O ₂ Supply Pressure, psig	35	34	33	36	34	35	34	35
TE 9206.4 O ₂ Supply Temperature, °F	72	67	63	62	61	60	60	62

Table 37, Part 3. ASH-AGGLOMERATING GASIFIER DATA SUMMARY
FOR TEST 40

Date 5/14/75

Time	1000	1200	1400	1600	1800	2000	2200	2400
TE 9206.6 Bed Temperature, °F	1642	1656	1645	1667	1673	1676	1680	1674
TE 9206.18 Gas Temperature-Air Heater, °F	614	614	608	805	835	794	788	787
TE 9206.1 Gas Temp. Entering Venturi, °F	699	656	648	651	697	697	717	719
TE 9206.2 Gas Temp. Entering Grid, °F	427	400	396	453	476	470	468	467
PI 913 & PDR 910 Pressure in the Venturi, psig	2.9	3.2	3.4	3.4	3.4	3.4	3.5	3.5
PI 905 Press. at Air Heater Exit, psig	3.0	4.4	4.7	5.4	5.4	5.4	5.5	5.6
FI 904 Steam Flow to Air Heater, lb/hr	333	333	333	333	333	333	333	333
FR 903 @ 70 psig & 300°F Steam Flow to Grid, lb/hr	231	231	231	231	231	231	231	231
FR 905 Enriched Air to Grid, lb/hr	267	278	263	246	243	246	248	249
FR 906 Enriched Air to Venturi, lb/hr	211	230	241	254	253	249	261	261
FR 995 Total 21% Air to Air Heater, lb/hr	0	0	0	0	0	0	0	0
FR 938 Total O ₂ to Air Heater, lb/hr	187	203	203	208	202	205	213	213
N ₂ Purge to Reactor, mole/hr	2.87	2.87	2.87	2.87	2.87	2.87	2.87	2.87
FR 918 Rate of Quench Water, lb/hr	180	280	310	300	240	280	260	260
PDR 912/PDR 913 X 3 33 Bed Height, ft	3.72	3.65	3.77	3.65	3.58	3.45	3.56	3.79
PDR 913 X 43.3 Bed Density, lb/cu ft	22.1	22.5	22.9	22.5	22.9	23.8	24.7	25.1
Coke Feed Rate, lb/hr (average)	285	285	339	339	339	339	436	436
Reactor Gas Composition, % (Dry) CO ₂ (ANR 975)	28.7	28.7	29	27.5	27	27	27.2	27
CO (ANR 977)	23.2	24.5	23.2	25	25	25	26.5	25.7
H ₂ (ANR 981)	36	36	35.2	36.2	35.5	35.5	35.5	35.5
CH ₄	0.8	0.8	0.8	0.7	0.7	0.7	0.7	0.7
H ₂ S	0.1	0.1	0.1	0.1	0.1	0.2	0.2	0.3
N ₂ (By Difference)	11.2	9.9	11.7	10.5	11.7	11.6	9.9	10.8
1st Stage Dust Removal Rate, lb/hr	137	137	137	137	137	152	152	152
2nd Stage Dust Removal Rate, lb/hr	6	6	6	6	6	6	6	6
C-4 Discharge Rate, lb/hr	14	13	11	6	8	10	6	7
Reactor Bed Composition:	%C			40.5				32.7
%Ash				57.3				66.0
Feed Composition:	%C							
%Ash								
Ash Composition:	%C			39.8				26.7
%Ash				56.9				70.7
TE 9206.16 Gas Temp. Entering Quencher, °F	1252	1263	1270	1266	1267	1271	1273	1280
PIC 901 Blower Outlet Pressure, psig	--	--	--	--	--	--	--	--
TI 9203 Blower Outlet Temperature, °F	--	--	--	--	--	--	--	--
PI 9122 O ₂ Supply Pressure, psig	33	42	42	42	40	40	43	42
TE 9206.4 O ₂ Supply Temperature, °F	67	74	80	80	86	74	64	64

Table 37, Part 4. ASH-AGGLOMERATING GASIFIER DATA SUMMARY
FOR TEST 40

Date 5/15/75									
Time	0200	0400	0600	0800	1000	1200	1400	1600	
TE 9206.6 Bed Temperature, °F	1667	1671	1682	1680	1713	1719	1700	1704	
TE 9206.18 Gas Temperature-Air Heater, °F	788	787	787	786	797	783	822	830	
TE 9206.1 Gas Temp. Entering Venturi, °F	731	732	725	723	751	656	771	766	
TE 9206.2 Gas Temp. Entering Grid, °F	470	461	462	465	436	434	430	436	
PI 913 & PDR 910 Pressure in the Venturi, psig	3.6	3.6	3.6	3.6	3.6	3.8	3.6	3.6	
PI 905 Press. at Air Heater Exit, psig	5.7	5.7	5.6	5.5	6.4	7.1	5.9	6.0	
FI 904 Steam Flow to Air Heater, lb/hr	333	333	333	333	305	327	288	288	
FR 903 @ 70 psig & 300°F Steam Flow to Grid, lb/hr	231	231	231	231	226	247	259	259	
FR 905 Enriched Air to Grid, lb/hr	243	243	252	252	213	206	198	198	
FR 906 Enriched Air to Venturi, lb/hr	259	259	264	259	295	323	270	270	
FR 995 Total 21% Air to Air Heater, lb/hr	0	0	0	0	0	0	0	0	
FR 938 Total O ₂ to Air Heater, lb/hr	207	207	207	204	239	237	212	210	
N ₂ Purge to Reactor, mole/hr	2.87	2.87	2.87	2.87	2.87	2.98	2.98	2.98	
FR 918 Rate of Quench Water, lb/hr	260	260	280	310	260	310	260	260	
PDR 912/PDR 913 X 3 33 Bed Height, ft	4.0	4.05	3.94	3.93	3.77	3.54	3.59	3.64	
PDR 913 X 43.3 Bed Density, lb/cu ft	26.0	26.0	26.0	26.4	26.0	26.8	28.1	28.1	
Coke Feed Rate, lb/hr (average)	436	325	325	220	406	406	406	396	
Reactor Gas Composition, % (Dry) CO ₂ (ANR 975)	27.5	26.7	26	26.7	25	25.2	25.5	25.2	
CO (ANR 977)	25	25	26.5	26.2	29.5	29.5	27.2	28	
H ₂ (ANR 981)	35.5	35.5	35.5	35.5	36.2	36.2	36.2	36.2	
CH ₄	0.7	0.7	0.7	0.7	0.7	0.6	0.6	0.6	
H ₂ S	0.1	0.1	0.1	0.1	0.1	0.4	0.4	0.4	
N ₂ (By Difference)	11.2	12.0	11.2	10.8	8.5	8.1	10.1	9.6	
1st Stage Dust Removal Rate, lb/hr	152	152	152	152	157	157	157	157	
2nd Stage Dust Removal Rate, lb/hr	6	6	6	6	6	6	6	6	
C-4 Discharge Rate, lb/hr	9	7	6	7	4	2	4	12	
Reactor Bed Composition:	%C	33.7		35.1		27.5		22.3	
	%Ash	64.6		63.5		71.4		76.8	
Feed Composition:	%C		72.1						
	%Ash		21.9						
Ash Composition:	%C			17.4		12.4		9.2	
	%Ash			80.0		85.7		88.8	
TE 9206.16 Gas Temp. Entering Quencher, °F	1286	1289	1287	1296	1307	1316	1305	1302	
PIC 901 Blower Outlet Pressure, psig	--	--	--	--	--	--	--	--	
TI 9203 Blower Outlet Temperature, °F	--	--	--	--	--	--	--	--	
PI 9122 O ₂ Supply Pressure, psig	43	42	46	42	41	41	41	40	
TE 9206.4 O ₂ Supply Temperature, °F	62	60	57	66	67	69	77	71	

Table 37, Part 5. ASH-AGGLOMERATING GASIFIER DATA SUMMARY
FOR TEST 40

Date 5/15-16/75

Time	1800	2000	2200	2400	0200			
TE 9206.6								
Bed Temperature, °F	1707	1715	1727	1740	1779			
TE 9206.18								
Gas Temperature-Air Heater, °F	837	798	800	798	793			
TE 9206.1								
Gas Temp. Entering Venturi, °F	677	512	513	518	519			
TE 9206.2								
Gas Temp. Entering Grid, °F	478	483	493	501	495			
PI 913 & PDR 910								
Pressure in the Venturi, psig	6.2	3.7	3.7	3.7	3.7			
PI 905								
Press. at Air Heater Exit, psig	7.5	5.5	5.3	5.0	4.9			
FI 904								
Steam Flow to Air Heater, lb/hr	280	338	338	336	336			
FR 903 @ 70 psig & 300°F								
Steam Flow to Grid, lb/hr	254	233	233	233	233			
FR 905								
Enriched Air to Grid, lb/hr	252	267	281	294	294			
FR 906								
Enriched Air to Venturi, lb/hr	206	244	222	215	215			
FR 995								
Total 21% Air to Air Heater, lb/hr	0	0	0	0	0			
FR 938								
Total O ₂ to Air Heater, lb/hr	214	209	209	206	208			
N ₂ Purge to Reactor, mole/hr	2.98	2.98	2.98	2.98	2.98			
FR 918								
Rate of Quench Water, lb/hr	310	280	310	310	260			
PDR 912/PDR 913 X 3 33								
Bed Height, ft	3.96	3.53	3.28	2.97	2.72			
PDR 913 X 43.3								
Bed Density, lb/cu ft	27.3	28.1	28.1	28.1	28.1			
Coke Feed Rate, lb/hr (average)	396	325	325	325	325			
Reactor Gas Composition, % (Dry)								
CO ₂ (ANR 975)	24.2	25.2	25.5	25.5	26			
CO (ANR 977)	28	28.7	29.5	27.2	27.2			
H ₂ (ANR 981)	36.2	36.2	36.2	36.2	35.5			
CH ₄	0.6	0.6	0.6	0.6	0.6			
H ₂ S	0.4	0.4	0.4	0.4	0.4			
N ₂ (By Difference)	10.6	8.9	7.8	10.1	10.3			
1st Stage Dust Removal Rate, lb/hr	157	157	152	152	152			
2nd Stage Dust Removal Rate, lb/hr	6	6	6	6	6			
C-4 Discharge Rate, lb/hr	2	4	5	3	4			
Reactor Bed								
Composition: %C		18.2		12.2				
%Ash		82.0		87.4				
Feed Composition: %C								
%Ash								
Ash Composition: %C		9.5	7.3	5.2	2.2			
%Ash		90.0	91.0	94.3	97.6			
TE 9206.16								
Gas Temp. Entering Quencher, °F	1299	1309	1308	1307	1297			
PIC 901								
Blower Outlet Pressure, psig	--	--	--	--	--			
TI 9203								
Blower Outlet Temperature, °F	--	--	--	--	--			
PI 9122								
O ₂ Supply Pressure, psig	42	44	44	42	43			
TE 9206.4								
O ₂ Supply Temperature, °F	69	63	62	60	58			

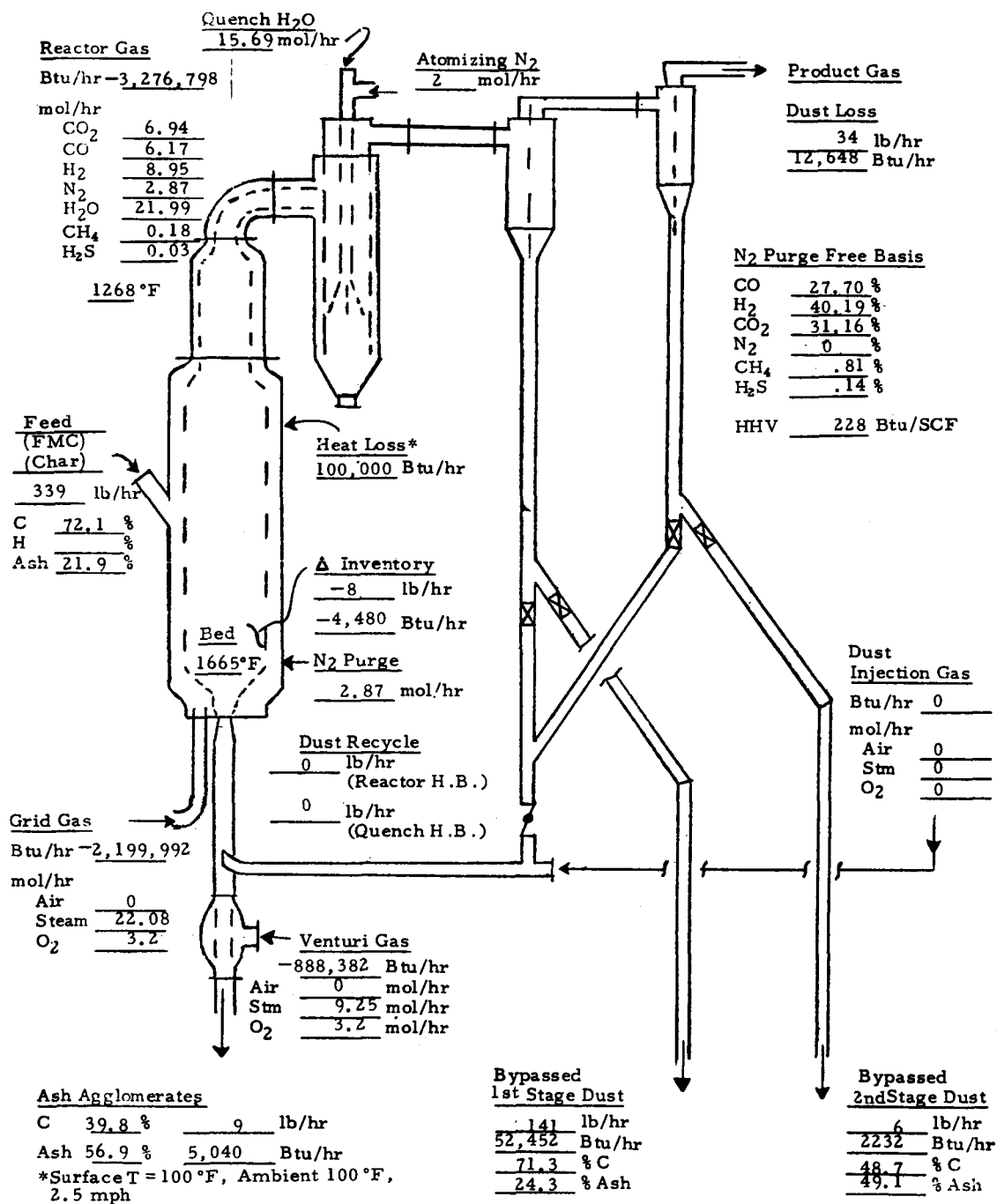


Figure 73. ASH-AGGLOMERATING GASIFIER TEST 40
 FROM 1400 TO 2000 HOURS (May 14, 1975)

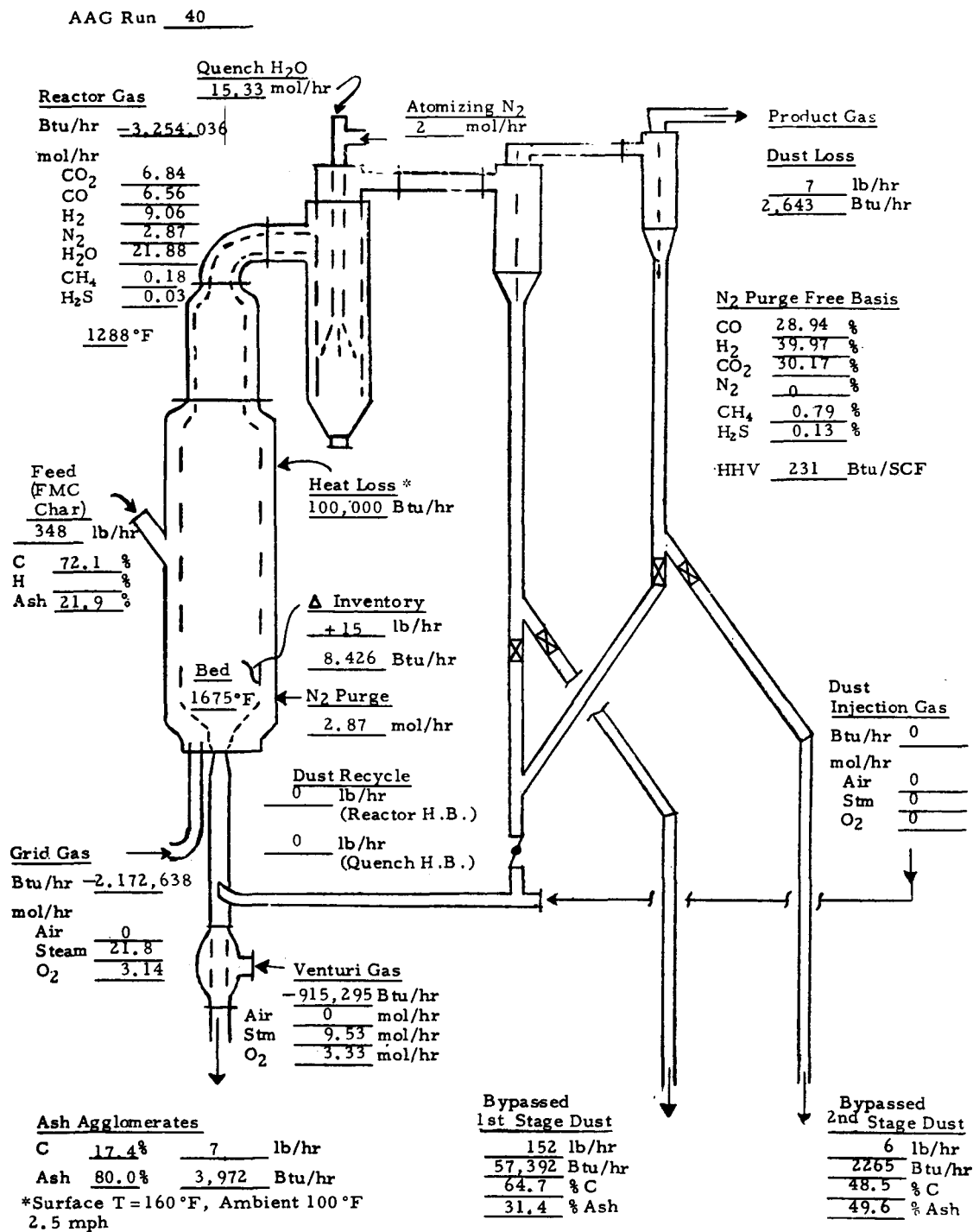


Figure 74. ASH-AGGLOMERATING GASIFIER TEST 40
FROM 0000 TO 0800 HOURS (May 15, 1975)

Table 38, ASH-AGGLOMERATING GASIFICATION DATA SUMMARY
FOR TEST 40

Unit Size: 4-ft diameter x 21-ft high

Venturi Diameter 2 inches

Feed Material FMC Char

Cyclone Dust Collectors: 0 internal 2 external

1	Superficial Velocity, ft/s	1.0
2	Avg. Freeboard Temperature, °F	1600
3	Freeboard Pressure, psig	2.3
4	Grid Pressure Drop, psi	0.5
5	Venturi Pressure Drop, psi	0.25
6	Venturi Velocity, ft/s	102
7	Temperature of Gas Entering Venturi, °F	725
	Grid Gas Composition	
8	% O ₂	12.6
9	% N ₂	0
10	% Steam	87.4
	Venturi Gas Composition	
11	% O ₂	25.8
12	% N ₂	0
13	% Steam	74.2
14	Fluidized Bed Density, lb/CF	26.0
15	Fluidized Bed Height, ft	4.0
16	Avg. Fluidized Bed Temperature, °F	1675
17	Feed Composition, %C/% ash	72.1 / 21.0
18	Avg. Fluidized Bed Composition, %C/% ash	34.9 / 64.0
19	Venturi Discharge Composition, %C/% ash	17.4 / 80.0
	<u>1st</u> Stage Dust Composition, %C/% ash	64.7 / 31.4
	<u>2nd</u> Stage Dust Composition, %C/% ash	48.5 / 49.6
	Superficial Velocity/Minimum Fluidization Velocity	10

Mesh Size	6	12	20	40	70	100	140	200	270	40μ	20μ	10μ	5μ	-5μ
Feed	0	1.0	34.1	26.8	17.0		9.8		5.0		6.3			
Ash	0.2	1.4	25.4	40.4	24.1				8.5					
<u>1st</u> Stage Dust	0	0	0	0	3.6	12.6	22.1	14.8	12.8	23.2	6.2	3.4	1.2	0.1
<u>2nd</u> Stage Dust	0	0	0	0	0	0	0	0	0	0	2.5	3.5	70.5	23.5

) for, using the fine breeze unscreened coke again. Heat and material balances are included in Table 39 and in Figures 75 and 76. Also included is operating data, Table 40.

Test 42

Once again start-up was normal, and the expected operating temperature of 1900°F was reached 12 hours after bed ignition. Four hours later, agglomerates began to appear. The agglomerating state of operation was maintained while adjusting the process gas input to increase the agglomerates' size, and within 24 hours, agglomerates of 6 mesh and larger predominated. However, shortly thereafter, Test 42 was terminated by a blocked venturi, which was later found to have been caused by a trickle-valve guide bar, which had been broken loose by the repeated striking of the flapper. The most significant information obtained from this test was the recorded high rate of dust collected at the second-stage cyclone. This rate had stabilized to around 120 lb/hr before the test was terminated. This dust rate was 2 to 3 times that experienced in Test 41 and led to the conclusion that the first-stage internal cyclone was plugged. This conclusion was verified when the top of the reactor was lifted revealing a partially dust-filled cyclone. Dust had accumulated to a level that caused vortex reentrainment. The cyclone is a standard ME design by Ducon Company, having an integral vortex disengaging hopper. When the hopper portion was cut open, caked material, plugging the hopper, was found and loose fines accumulated up into the cyclone itself. The first-stage (internal) cyclone was modified to provide a more free-flowing exit for dust being returned to the fluid bed. Heat and material balances are included in Figure 77 and Table 41. Operating data for Test 42 are listed in Table 42.

Table 39, Part 1. ASH-AGGLOMERATING GASIFIER DATA SUMMARY
FOR TEST 41

Date 6/3/75

Time	0400	0600	0800	1000	1200	1400	1600	1800
TE 9206.6 Bed Temperature, °F	1539	1677	1752	1820	1839	1857	1816	1860
TE 9206.18 Gas Temperature-Air Heater, °F	779	827	817	827	810	815	870	859
TE 9206.1 Gas Temp. Entering Venturi, °F	646	687	683	567	536	525	598	518
TE 9206.2 Gas Temp. Entering Grid, °F	536	532	518	523	504	504	526	544
PI 913 & PDR 910 Pressure in the Venturi, psig	3.7	4.1	4.45	4.4	4.35	4.1	3.9	3.72
PI 905 Press. at Air Heater Exit, psig	11.5	11.9	12.1	12.0	11.8	11.6	9.6	--
FI 904 Steam Flow to Air Heater, lb/hr	--	--	--	--	--	--	--	--
FR 903 @ 70 psig & 300°F Steam Flow to Grid, lb/hr	425	430	430	400	405	405	405	325
FR 905 Air to Grid, lb/hr	976	893	797	771	712	708	676	--
FR 906 Air to Venturi, lb/hr	613	636	647	643	629	574	473	--
FR 995 Total 21% Air to Air Heater, lb/hr	1684	1602	1476	1466	1379	1351	1194	--
FR 938 Total O ₂ to Air Heater, lb/hr	--	--	--	--	--	--	--	--
N ₂ Purge to Reactor, mole/hr	3.2	3.2	3.2	3.2	3.2	3.2	3.2	3.2
FR 918 Rate of Quench Water, lb/hr	475	500	540	590	590	590	530	525
PDR 912/PDR 913 X 3.5 Bed Height, ft	4.2 ^a	4.41	4.48	4.42	4.43	4.37	4.25	4.35
PDR 913 X 41.14 Bed Density, lb/cu ft	36.2	37.75	37.75	37.34	36.94	36.20	36.20	35.30
Coke Feed Rate, lb/hr (average)	431	431	431	250	250	250	250	250
Reactor Gas Composition, % (Dry) CO ₂ (ANR 975)	17.5	17.5	16.0	15.0	--	15.0	15.5	14.0
CO (ANR 977)	3.0	5.0	6.5	9.0	--	10.5	9.0	12.5
H ₂ (ANR 981)	0.5	3.0	6.0	8.5	--	10.5	9.0	10.5
CH ₄	0.2	0.2	0.2	0.2	--	0.2	0.2	0.2
N ₂ (By Difference)	78.8	74.3	71.3	67.3	--	64.0	66.5	63.0
1st Stage Dust Removal Rate, lb/hr	70	70	70	70	70	70	41	41
2nd Stage Dust Removal Rate, lb/hr	9.0	9.0	9.0	9.0	9.0	9.0	9.0	9.0
C-4 Discharge Rate, lb/hr	34	28	17	5	1	0	29	2
Reactor Bed Composition:	%C					82.7		
	%Ash					15.9		
Feed Composition:	%C							
	%Ash							
Ash Composition:	%C							
	%Ash							
TE 9206.16 Gas Temp. Entering Quencher, °F	1229	1425	1532	1611	1641	1668	1634	1657
PIC 901 Blower Outlet Pressure, psig	15.8	16.0	16.0	15.5	15.0	15.0	12.2	13.2
TI 9203 Blower Outlet Temperature, °F	285	300	290	300	295	300	270	--
PI 9122 O ₂ Supply Pressure, psig	--	--	--	--	--	--	--	--
TE 9206.4 O ₂ Supply Temperature, °F	78	84	80	85	94	96	99	99

Table 39, Part 2. ASH-AGGLOMERATING GASIFIER DATA SUMMARY
FOR TEST 41

Date 6/3-4/75

Time	2000	2200	2400	0200	0400	0600	0800	1000
TE 9206.6								
Bed Temperature, °F	1859	1865	1859	1853	1861	1857	1848	1847
TE 9206.18								
Gas Temperature-Air Heater, °F	779	779	794	797	795	803	805	807
TE 9206.1								
Gas Temp. Entering Venturi, °F	509	459	436	447	434	420	429	424
TE 9206.2								
Gas Temp. Entering Grid, °F	520	511	518	519	517	520	522	522
PI 913 & PDR 910								
Pressure in the Venturi, psig	3.6	3.65	3.6	3.16	3.2	3.05	3.3	3.3
PI 905								
Press. at Air Heater Exit, psig	10.0	10.3	11.0	11.0	11.0	11.2	11.1	11.1
FI 904								
Steam Flow to Air Heater, lb/hr	--	--	--	--	--	--	--	--
FR 903 @ 70 psig & 300°F								
Steam Flow to Grid, lb/hr	325	325	325	325	325	325	325	325
FR 905								
Enriched Air to Grid, lb/hr	701	705	710	710	710	704	705	705
FR 906								
Enriched Air to Venturi, lb/hr	385	391	322	322	322	284	288	288
FR 995								
Total 21% Air to Air Heater, lb/hr	1146	1140	1086	1075	1075	1037	1037	1037
FR 938								
Total O ₂ to Air Heater, lb/hr	--	--	--	--	--	--	--	--
N ₂ Purge to Reactor, mole/hr	3.2	3.2	3.2	3.2	3.2	3.2	3.2	3.2
FR 918								
Rate of Quench Water, lb/hr	490	510	490	490	460	470	470	450
PDR 912/PDR 913 X 3.5								
Bed Height, ft	4.33	4.29	4.16	4.21	4.16	4.08	4.16	4.22
PDR 913 X 41.14								
Bed Density, lb/cu ft	34.48	34.07	34.48	34.07	34.48	34.48	35.70	35.70
Coke Feed Rate, lb/hr (average)	241	241	241	241	241	241	241	241
Reactor Gas Composition, % (Dry)								
CO ₂ (ANR 975)	14.0	14.0	14.0	13.5	14.0	14.0	14.0	14.5
CO (ANR 977)	12.5	13.0	12.0	12.0	12.0	12.0	11.0	11.0
H ₂ (ANR 981)	11.5	11.5	11.5	11.5	11.5	11.5	10.5	10.5
CH ₄	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
N ₂ (By Difference)	61.8	61.3	62.3	62.8	62.3	62.3	64.3	63.8
1st Stage Dust Removal Rate, lb/hr	41	41	41	41	41	41	55	55
2nd Stage Dust Removal Rate, lb/hr	9.0	9.0	9.0	9.0	9.0	9.0	9.0	9.0
C-4 Discharge Rate, lb/hr	6.0	0.5	1.0	7.0	3.0	4.0	8.0	5.0
Reactor Bed Composition:	%C	97.4				77.3		
	%Ash	21.4				21.5		
Feed Composition:	%C		86.9				86.6	
	%Ash		10.5				10.8	
Ash Composition:	%C		73.3			66.8		
	%Ash		25.0			32.4		
TE 9206.16								
Gas Temp. Entering Quencher, °F	1664	1672	1664	1666	1667	1663	1646	1653
PIC 901								
Blower Outlet Pressure, psig	12.7	12.7	13.4	13.3	13.3	13.3	13.2	13.2
TI 9203								
Blower Outlet Temperature, °F	265	260	265	265	265	260	265	270
PI 9122								
O ₂ Supply Pressure, psig	--	--	--	--	--	--	--	--
TE 9206.4								
O ₂ Supply Temperature, °F	92	87	84	79	80	80	81	82

**Table 39, Part 3. ASH-AGGLOMERATING GASIFIER DATA SUMMARY
FOR TEST 41**

Date 6/4/65

Time	1200	1400	1600	1800	2000	2200		
TE 9206.6 Bed Temperature, °F	1879	1864	1883	1886	1886	1884		
TE 9206.18 Gas Temperature-Air Heater, °F	815	815	816	810	815	816		
TE 9206.1 Gas Temp. Entering Venturi, °F	436	449	447	521	541	555		
TE 9206.2 Gas Temp. Entering Grid, °F	550	550	544	492	489	489		
PI 913 & PDR 910 Pressure in the Venturi, psig	3.95	3.8	3.55	4.25	3.7	4.1		
PI 905 Press. at Air Heater Exit, psig	10.1	9.6	9.5	12.0	11.3	11.5		
FI 904 Steam Flow to Air Heater, lb/hr	--	--	--	--	--	--		
FR 903 @ 70 psig & 300°F Steam Flow to Grid, lb/hr	370	370	370	370	370	370		
FR 905 Enriched Air to Grid, lb/hr	866	858	856	600	591	593		
FR 906 Enriched Air to Venturi, lb/hr	304	315	315	639	632	629		
FR 995 Total 21% Air to Air Heater, lb/hr	1248	1248	1242	1265	1239	1243		
FR 938 Total O ₂ to Air Heater, lb/hr	--	--	--	--	--	--		
N ₂ Purge to Reactor, mole/hr	3.0	3.0	3.0	3.0	3.0	3.0		
FR 918 Rate of Quench Water, lb/hr	530	560	560	580	575	570		
PDR 912/PDR 913 X 3.5 Bed Height, ft	4.25	4.23	4.11	3.89	4.03	4.12		
PDR 913 X 41.14 Bed Density, lb/cu ft	34.47	33.25	32.83	33.64	32.00	32.00		
Coke Feed Rate, lb/hr (average)	242	242	242	242	242	242		
Reactor Gas Composition, % (Dry) CO ₂ (ANR 975)	13.5	14.0	13.5	14.0	14.5	14.0		
CO (ANR 977)	12.0	11.5	12.0	11.5	11.5	12.5		
H ₂ (ANR 981)	10.5	10.5	11.0	10.5	11.0	10.5		
CH ₄	0.2	0.2	0.2	0.2	0.2	0.2		
N ₂ (By Difference)	63.8	63.8	63.3	63.8	62.8	62.8		
1st Stage Dust Removal Rate, lb/hr	55	55	55	55	55	55		
2nd Stage Dust Removal Rate, lb/hr	9.0	9.0	9.0	9.0	9.0	9.0		
C-4 Discharge Rate, lb/hr	6	3	0	0	0	0		
Reactor Bed Composition:	%C	74.2				78.2		
	%Ash	24.7				20.6		
Feed Composition:	%C				88.6			
	%Ash				9.7			
Ash Composition:	%C	65.7						
	%Ash	33.6						
TE 9206.16 Gas Temp. Entering Quencher, °F	1693	1691	1701	1706	1698	1697		
PIC 901 Blower Outlet Pressure, psig	14.2	12.6	12.5	14.7	14.4	14.4		
TI 9203 Blower Outlet Temperature, °F	270	275	275	305	295	295		
PI 9122 O ₂ Supply Pressure, psig	--	--	--	--	--	--		
TE 9206.4 O ₂ Supply Temperature, °F	87	98	102	104	102	103		

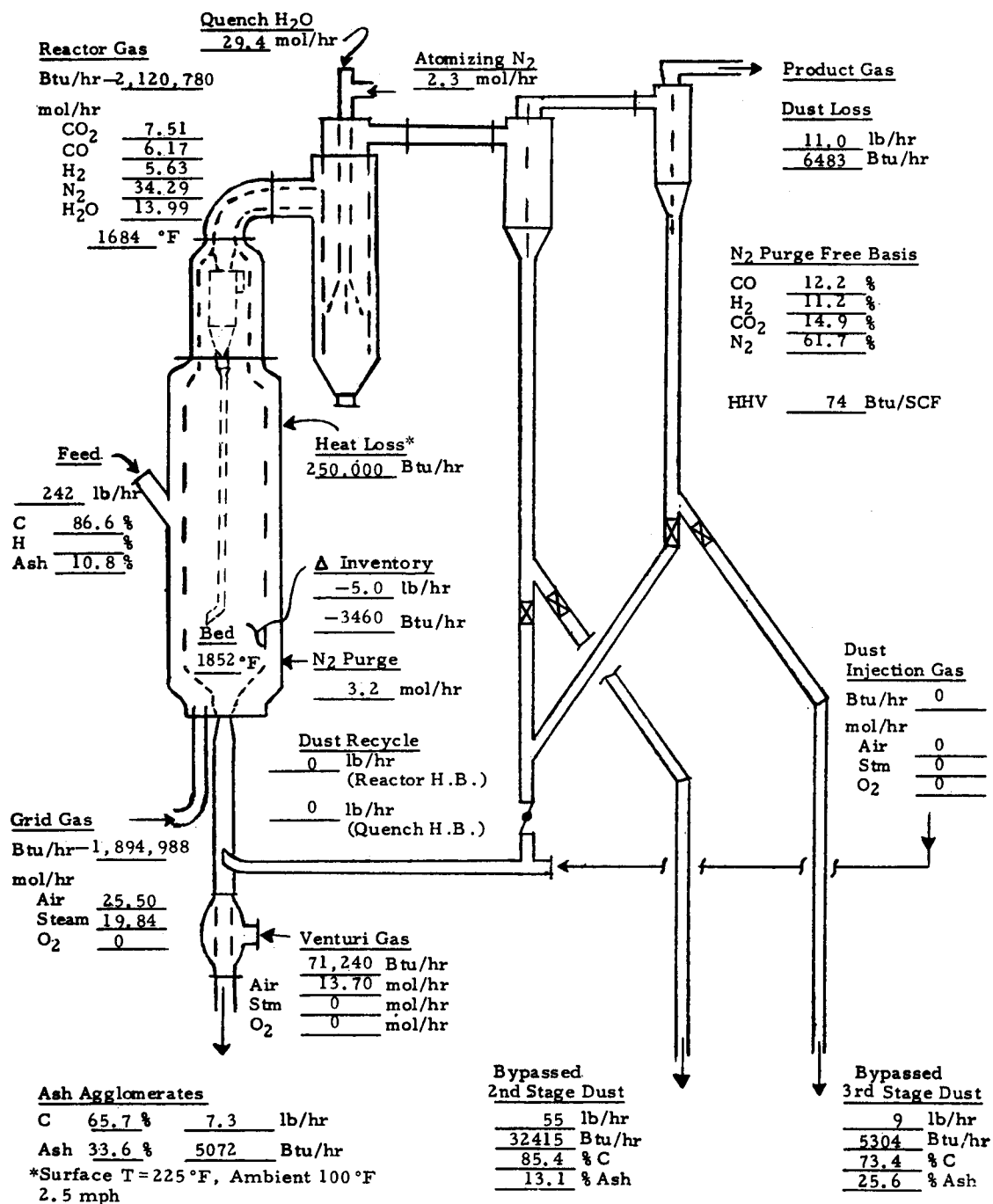


Figure 75. ASH-AGGLOMERATING GASIFIER TEST 41
FROM 0800 TO 2000 HOURS (June 4, 1975)

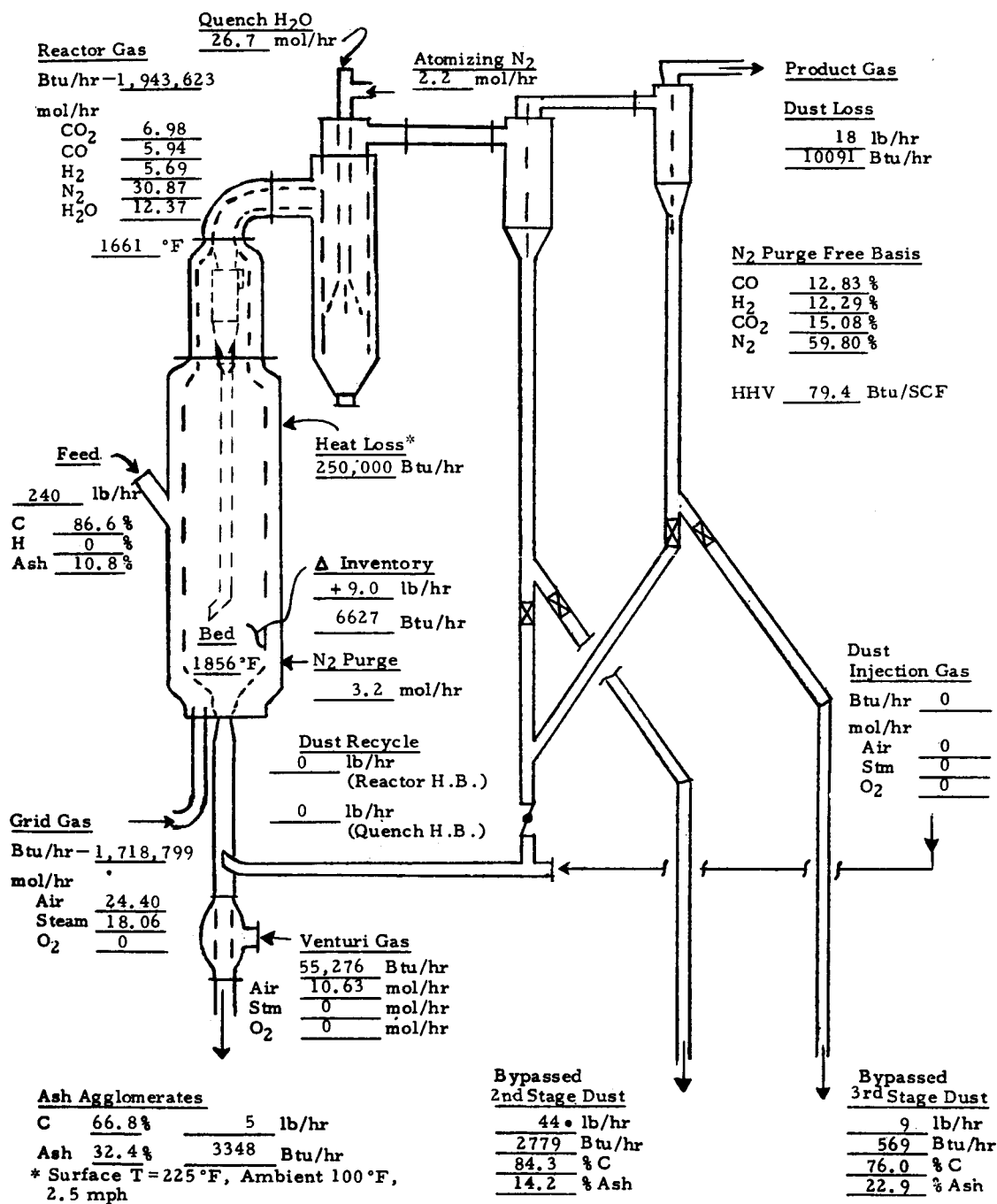


Figure 76. ASH-AGGLOMERATING GASIFIER TEST 41
FROM 0000 TO 0800 HOURS (June 4, 1975)

Table 40, ASH-AGGLOMERATING GASIFIER OPERATING DATA
FOR TEST 41

Run No. 41 Date 6/4/75 Time Period 0000 to 0800

Unit Size: 4-ft diameter x 21-ft high

Venturi Diameter 2 inches

Feed Material Coke Breeze

Cyclone Dust Collectors: 1 internal 2 external

1	Superficial Velocity, ft/s	1.70
2	Avg Freeboard Temperature, °F	1850
3	Freeboard Pressure, psig	1.4
4	Grid Pressure Drop, psi	2.0
5	Venturi Pressure Drop, psi	0.4
6	Venturi Velocity, ft/s	75
7	Temperature of Gas Entering Venturi, °F	430
	Grid Gas Composition	
8	% O ₂	12.2
9	% N ₂	45.3
10	% Steam	42.5
	Venturi Gas Composition	
11	% O ₂	21.0
12	% N ₂	79.0
13	% Steam	
14	Fluidized Bed Density, lb/CF	34.5
15	Fluidized Bed Height, ft	4.1
16	Avg Fluidized Bed Temperature, °F	1880
17	Feed Composition, %C/% ash	86.6 / 10.8
18	Avg Fluidized Bed Composition, %C/% ash	77.3 / 21.5
19	Venturi Discharge Composition, %C/% ash	66.8 / 32.4
	2nd Stage Dust Composition, %C/% ash	84.3 / 14.2
	3rd Stage Dust Composition, %C/% ash	76.0 / 22.9
	Superficial Velocity/Minimum Fluidization Velocity	4.2

Mesh Size	6	12	20	40	70	100	140	200	270	40μ	20μ	10μ	5μ	-5μ
Feed	16.5	31.0	22.2	15.0	8.5		4.3		1.6		0.9			
Ash	20.4	44.7	24.0	10.1	0.2	0.6								
2nd Stage Dust								1.7	10.2	10.3	32.6	29.1	13.7	2.4
3rd Stage Dust									0.8	3.0	1.0	7.6	39.6	48.2

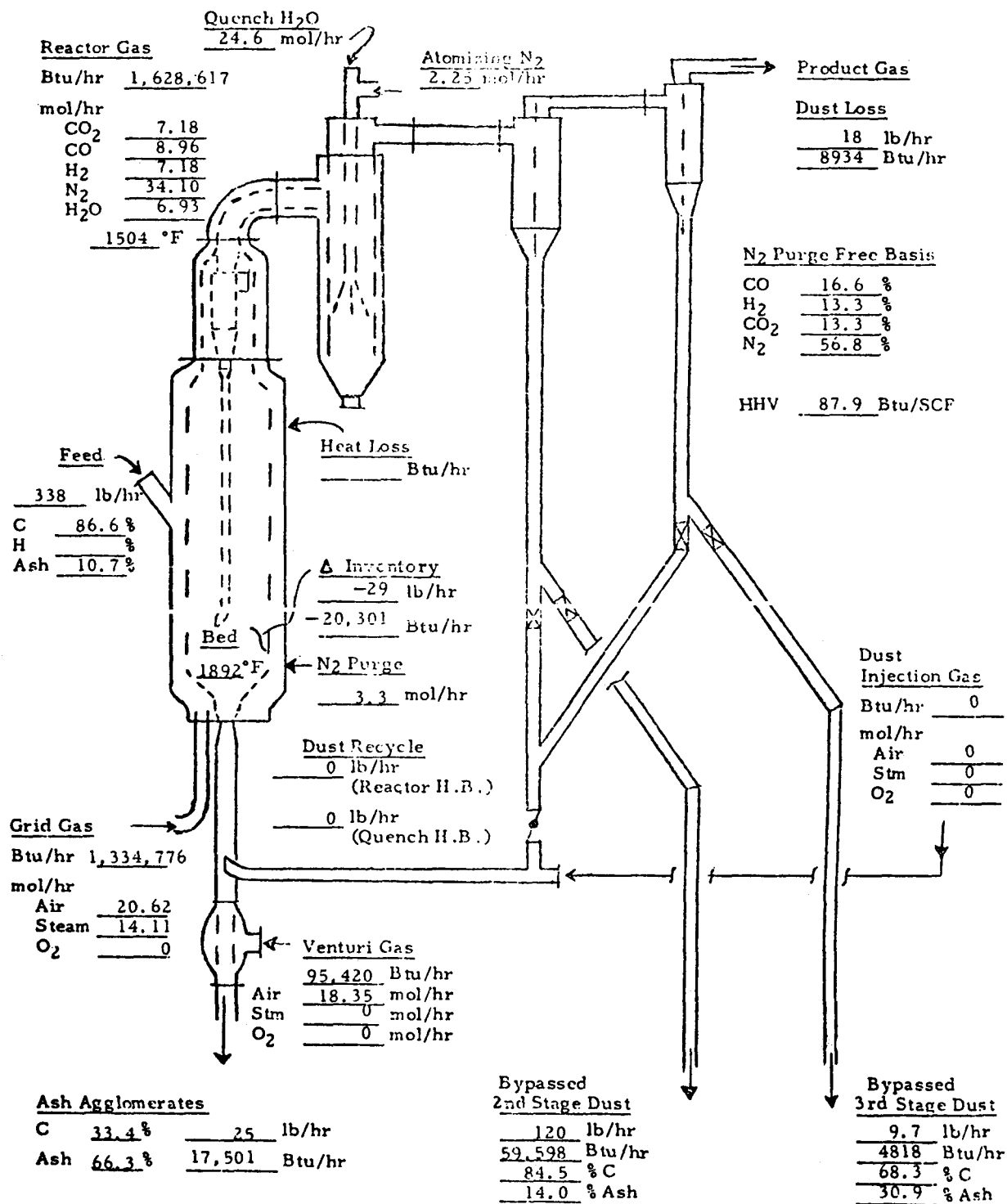


Figure 77. ASH-AGGLOMERATING GASIFIER TEST 42
FROM 0800 TO 1400 HOURS (June 18, 1975)

Table 41. ASH-AGGLOMERATING GASIFIER DATA SUMMARY
FOR TEST 42

Date: 6-17-75	0200	0400	0600	0800	1000	1200	1400	1600
TE 9206.6 Bed Temperature, °F	1591	1668	1782	1862	1893	1907	1911	1904
TE 9206.18 Gas Temperature-Air Heater, °F	779	785	783	778	775	769	776	790
TE 9206.1 Gas Temp. Entering Venturi, °F	662	722	809	770	699	689	645	584
TE 9206.2 Gas Temp. Entering Grid, °F	513	513	515	513	519	523	499	489
PI 913 & PDR 910 Pressure in the Venturi, psig	3.15	3.95	4.4	4.2	3.8	3.45	3.2	2.94
PI 905 Press. at Air Heater Exit, psig	9.6	9.9	10.0	10.0	11.0	10.8	11.3	10.0
PI 904 Steam Flow to Air Heater, lb/hr	0	0	0	0	0	0	0	0
FR 903 @ 70 psig & 300°F Steam Flow to Grid, lb/hr	430	430	430	430	325	300	350	325
FR 905 Air to Grid, lb/hr	870	873	876	857	772	771	673	592
FR 906 Air to Venturi, lb/hr	580	562	604	570	515	514	518	502
FR 995 Total 21% Air to Air Heater, lb/hr	1555	1545	1616	1535	1376	1329	1275	1165
FR 948 Total O ₂ to Air Heater, lb/hr	0	0	0	0	0	0	0	0
N ₂ Purge to Reactor, mole/hr	3.3	3.3	3.3	3.3	3.3	3.3	3.3	3.3
FR 918 Rate of Quench Water, lb/hr	13.3	17.2	23.3	27.8	25.0	27.8	27.8	24.4
PDR 917/PDR 913 X 3.5 Bed Height, ft	3.8	4.0	4.8	4.6	4.5	4.2	3.8	3.6
PDR 913 X 41.14 Bed Density, lb/cu ft	34.6	36.2	36.2	34.7	30.9	28.8	26.3	25.1
Coke Feed Rate, lb/hr (average)	550	550	550	306	306	306	306	371
Reactor Gas Composition, % (Dry)								
CO ₂ (ANR 975)	16.8	16.8	16.0	15.0	13.4	12.5	13.0	13.7
CO (ANR 977)	2.7	4.5	7.5	10.5	14.2	15.0	15.0	14.0
H ₂ (ANR 981)	1.5	3.8	6.5	10.0	11.5	12.0	13.0	12.5
CH ₄ (ANR 978)	--	--	--	--	--	--	--	--
N ₂ (By Difference)	79.0	74.9	70.0	64.5	60.9	60.5	59.0	59.8
1st Stage Dust Removal Rate, lb/hr	227	227	227	114	114	114	114	122
2nd Stage Dust Removal Rate, lb/hr	9.7	9.7	9.7	9.7	9.7	9.7	9.7	9.7
C-4 Discharge Rate, lb/hr	--	48	43	32	18	22	18	14
Reactor Bed Composition:					85.0			82.8
%Ash					13.5			15.8
Feed Composition:					85.1			
%Ash					12.4			
Ash Composition:							48.1	
%C	76.0							
%Ash	22.9						51.9	
TE 9206.16 Gas Temp. Entering Quencher, °F	1003	1125	1284	1385	1432	1468	1501	1500
PIC 901 Blower Outlet Pressure, psig	15.2	15.2	16.4	15.6	15.5	15.0	15.0	13.5
TI 9203 Blower Outlet Temperature, °F	280	280	275	300	285	285	275	265
PI 9122 O ₂ Supply Pressure, psig	--	--	--	--	--	--	--	--
TI 9206.4 O ₂ Supply Temperature, °F	--	--	--	--	--	--	--	--

Table 41, Cont. ASH-AGGLOMERATING GASIFIER DATA SUMMARY
FOR TEST 42

Date: 6-17-75 - 6-18-75

Time	1800	2000	2200	2400	0200	0400	0600	0800
TE 9206.16 Bed Temperature, °F	1902	1891	1899	1899	1892	1883	1868	1877
TE 9206.18 Gas Temperature-Air Heater, °F	773	789	777	775	767	775	763	773
TE 9206.1 Gas Temp. Entering Venturi, °F	576	597	599	583	581	591	656	652
TE 9206.2 Gas Temp. Entering Grid, °F	513	514	512	505	506	506	506	506
PI 913 & PDR 910 Pressure in the Venturi, psig	2.85	2.85	2.85	2.80	2.85	2.80	2.80	3.0
PI 905 Press. at Air Heater Exit, psig	10.0	9.9	10.0	9.9	9.9	10.0	10.0	10.0
FI 904 Steam Flow to Air Heater, lb/hr	0	0	0	0	0	0	0	0
FR 903 @ 70 psig & 300°F Steam Flow to Grid, lb/hr	252	254	254	254	254	254	254	254
FR 905 Air to Grid, lb/hr	596	591	595	595	596	596	598	596
FR 906 Air to Venturi, lb/hr	505	501	504	504	505	505	456	531
FR 995 Total 21% Air to Air Heater, lb/hr	1165	1165	1165	1165	1169	1173	1130	1200
FR 936 Total O ₂ to Air Heater, lb/hr	0	0	0	0	0	0	0	0
N ₂ Purge to Reactor, mole/hr	3.3	3.3	3.3	3.3	3.3	3.3	3.3	3.3
FR 918 Rate of Quench Water, lb/hr	23.9	23.3	23.9	23.3	23.3	22.2	23.3	23.4
PDR 912/PDR 913 X 3.5 Bed Height, ft	3.6	3.8	3.8	3.7	3.9	3.9	4.0	4.0
PDR 913 X 41.14 Bed Density, lb/cu ft	25.9	26.7	26.7	26.7	26.7	27.2	28.8	30.0
Coke Feed Rate, lb/hr (average)	371	371	446	446	446	446	446	338
Reactor Gas Composition, % (Dry) CO ₂ (ANR 975)	13.0	13.4	13.0	13.0	13.4	13.4	13.7	13.2
CO (ANR 977)	15.2	14.7	15.5	15.5	14.2	14.2	13.7	14.7
H ₂ (ANR 981)	12.3	12.0	12.3	12.3	12.0	12.0	11.5	12.0
CH ₄ (ANR 978)	0	0	0	0	0	0	0	0
N ₂ (By Difference)	59.5	59.9	59.2	59.2	60.4	60.4	61.1	60.1
1st Stage Dust Removal Rate, lb/hr	122	122	133	133	133	133	133	120
2nd Stage Dust Removal Rate, lb/hr	9.7	9.7	9.7	9.7	9.7	9.7	9.7	9.7
C-4 Discharge Rate, lb/hr	14	20	21	24	21	25	41	34
Reactor Bed Composition:								
%C			73.0			62.0		
%Ash			26.0			16.8		
Feed Composition:								
%C	86.6				85.6			
%Ash	10.8				11.9			
Ash Composition:								
%C			45.0			35.1		
%Ash			55.0			64.9		
TE 9206.16 Gas Temp. Entering Quencher, °F	1484	1482	1486	1491	1491	1495	1496	1490
PIC 901 Blower Outlet Pressure, psig	13.5	13.5	13.5	13.5	13.5	13.5	13.6	13.6
T1 9203 Blower Outlet Temperature, °F	265	265	265	265	260	255	250	260
PI 9122 O ₂ Supply Pressure, psig	--	--	--	--	--	--	--	--
TE 9206.4 O ₂ Supply Temperature, °F	--	--	--	--	--	--	--	--

Table 42. ASH-AGGLOMERATING GASIFIER OPERATING DATA
FOR TEST 42

Run No. 42 Date 6/18/75 Time Period 0800 to 1400
 Unit Size: 4-ft diameter x 21-ft high
 Venturi Diameter 2 inches Feed Material Coke Breeze
 Cyclone Dust Collectors: 1 internal 2 external

1	Superficial Velocity, ft/s	1.73
2	Avg. Freeboard Temperature, °F	1720
3	Freeboard Pressure, psig	1.5
4	Grid Pressure Drop, psi	1.6
5	Venturi Pressure Drop, psi	0.4
6	Venturi Velocity, ft/s	150
7	Temperature of Gas Entering Venturi, °F	605
	Grid Gas Composition	
8	% O ₂	12.4
9	% N ₂	46.6
10	% Steam	41.0
	Venturi Gas Composition	
11	% O ₂	21.0
12	% N ₂	79.0
13	% Steam	
14	Fluidized Bed Density, lb/CF	28.5
15	Fluidized Bed Height, ft	4.0
16	Avg. Fluidized Bed Temperature, °F	1891
17	Feed Composition, %C/% ash	86.6 / 10.7
18	Avg. Fluidized Bed Composition, %C/% ash	81.5 / 17.0
19	Venturi Discharge Composition, %C/% ash	33.4 / 66.3
	Stage Dust Composition, %C/% ash	84.5 / 14.0
	Stage Dust Composition, %C/% ash	68.3 / 30.9
	Superficial Velocity/Minimum Fluidization Velocity	4.3

Mesh Size	6	12	20	40	70	100	140	200	270	40μ	20μ	10μ	5μ	-5μ
Feed	17.0	33.6	23.1	12.8	5.8		3.4		2.5	1.8				
Ash	42.2	48.8	6.6	1.0	0.3	1.1								
2nd Stage Dust								52.6	12.0	4.2	14.5	12.1	3.8	0.8
3rd Stage Dust									0.3	15.3	2.8	12.0	41.4	36.0

SUPPORT STUDIES

Ozonation of PhenolIntroduction

Several laboratory tests have been performed to elucidate the reaction of ozone with phenol. These tests were run to assess the feasibility of using ozone to clean up the excess condensate from the HYGAS Process at the pilot plant level.

Experimental

The setup used for the study is shown in Figures 78 and 79. Oxygen or air is passed through the ozone generator at a specified rate indicated by a rotameter (FI). The quantity and concentration of ozone in the product gas are dependent upon the oxygen partial pressure and total flow rate. Oxygen has exclusively been used in this particular generator (C2P-3C minipak, Pollution Control Industries), at a flow rate of approximately 5 SCF/hr. This produces an ozone content in the product gas from the generator of approximately 30 mg/l. This flow rate was picked for two reasons: 1) it is small enough for good mass transfer to be assumed and 2) it is large enough for cooling in the ozone generator. Larger flow rates (up to 30 SCF/hr of oxygen or air) can be used to increase ozone generation, but the ozone concentration would decrease.

The flow of product gas from the ozone generator is regulated by two micrometering valves, 1 and 3 in Figure 78. Valve 3 regulates the bypass flow and, at the same time, maintains sufficient back pressure on the system to help stabilize the metered flow through metering valve 1.

Two three-port valves, 2 and 4, are used to simplify the determination of the ozone concentration in the product gas from the generator. One of the three-port valves, 2, diverts the ozone-containing gas through the potassium iodide, KI (1), absorber for oxidation of the I^- solution to iodine. After the gas passes through this absorber, the other three-port valve, 4, diverts the gas stream back through the caustic scrubber and out the wet-test meter so that an accurate total flow of gas through the KI (1) absorber can be determined. Initial experiments showed that 100% of the ozone reacted with potassium iodide at total gas flow rates up to and including 1 l/min. This was determined by replacing the caustic scrubber before the wet-test meter with a potassium iodide solution and consequently absorbing any residual from the KI (1) absorber. Within the experimental accuracy of the test procedure, no ozone residual was found in the exit gas from the first absorber.

After determining the ozone concentration in the ozone generator product gas, the two three-port valves are switched back so that gas flows back to the reaction vessel. Figure 80 shows a more detailed drawing of the reaction system. Liquid feed is pumped from a feed reservoir with a peristaltic pump that can regulate the flow from approximately 5 to 500 cc/min. The feed material then proceeds through the coil in the constant-temperature bath so that the temperature differences between the feed material and reaction material can be kept to a minimum. The liquid in the reaction vessel is pushed out by the pressure created in the reaction vessel by the gas flow and the presence of the two downstream absorbers and wet-test meter. The

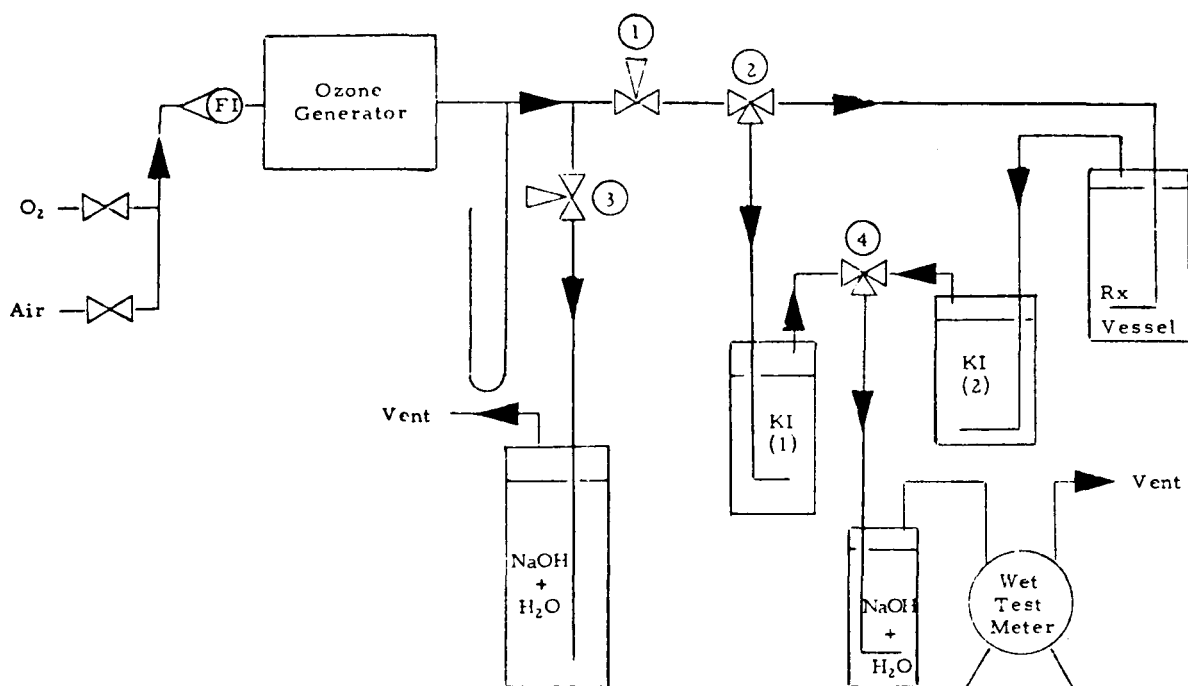


Figure 78. APPARATUS FOR OZONE REACTION STUDIES

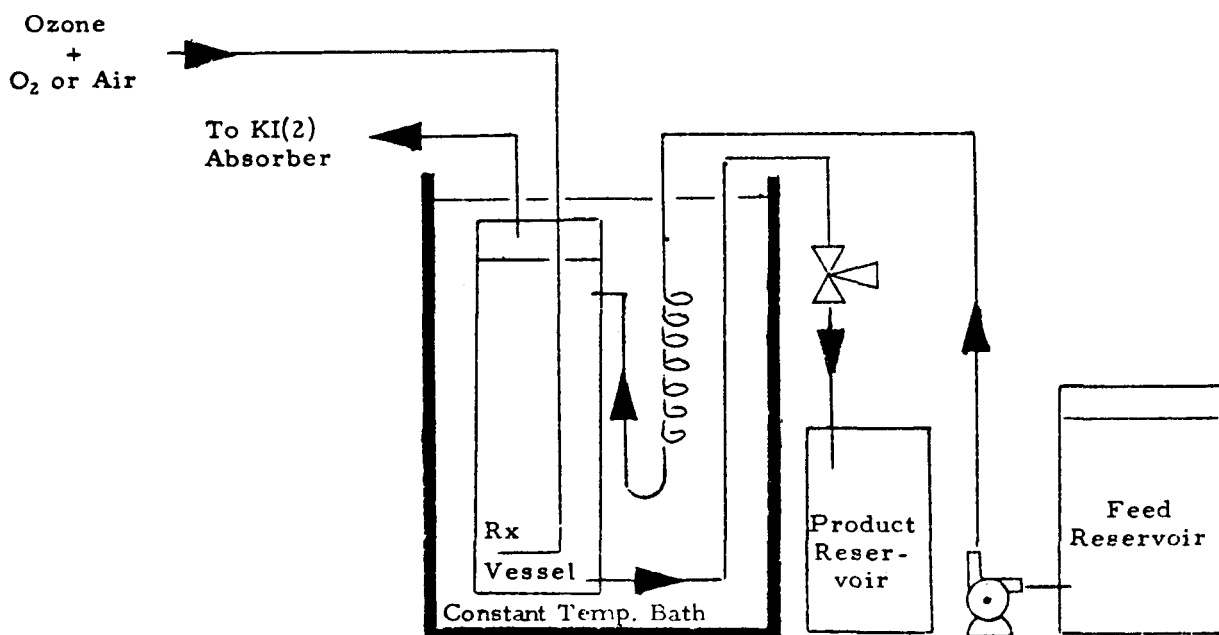


Figure 79. REACTION VESSEL SETUP FOR OZONATION EXPERIMENTS

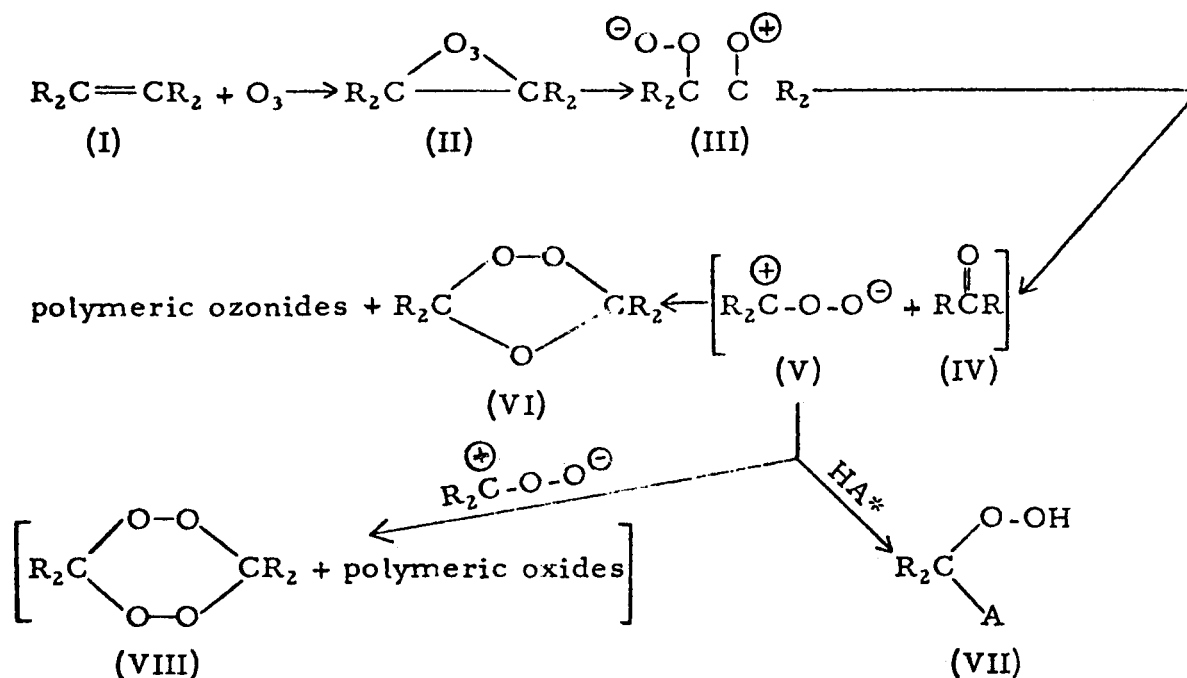
liquid level in the reaction vessel is maintained at a constant level by using a micrometering valve on the product flow from the reaction vessel to the product reservoir. During a run, since the volume of the reaction vessel is approximately 500 cc, the first 500 to 1000 cc of product is discarded so that nonsteady-state material does not influence the results. After this, the next 500 to 1000 cc of material is collected and analyzed for pH, chemical oxygen demand, and phenol content, and the KI (2) absorber is analyzed to determine the amount of unreacted ozone. Flow rates of the liquid and gas to the reaction vessel are estimated during the run by calibrating the liquid pump (i.e., potentiometer setting and tubing size vs. water flow rate) and the speed of the wet-test meter. More accurate average flow rates are determined a) by timing the total flow of liquid to the reaction vessel and determining the change of volume of feed material in the reservoir and b) by taking an initial and final wet-test meter reading for the gas flow.

Conventional analytical methods were used for ozone, pH, and chemical oxygen demand. Ozone was determined by reaction with a 10 weight percent potassium iodide solution to form oxygen and iodine. The amount of iodine formed was back-titrated with sodium thiosulfate using a starch indicator for an end-point determination. A blank of the same starting potassium iodide solution was also run to correct for a possible iodine buildup from standing and for the presence of other substances, which might be reduced by sodium thiosulfate. The pH of the feed and product material was performed on a conventional laboratory pH meter calibrated with standard buffers immediately before pH measurement. The chemical oxygen demand was determined by the standard APHA method, a dichromate and sulfuric acid reflux in the presence of silver sulfate for 30 minutes, followed by titration of the remaining dichromate with ferrous ammonium sulfate to an end point determined by ferroin indicator. Phenol analyses for the runs were performed by two different methods, titration and gas chromatography. The titration method is the same as that used to standardize phenol solutions of relatively high concentrations. Phenol is reacted with a bromate-bromide solution until excess bromine appears and is back-titrated with sodium thiosulfate with a starch indicator as an end point determinator. The gas chromatograph analysis was done with a column, which was able to detect as low as 10 ppm of phenol in water with ease. None of the solutions analyzed was even close to this limit; therefore, we assume that the determinations are reasonably accurate.

Results and Discussion

Most investigators have reviewed the ozonation of phenol from the viewpoint of a reduction in phenol levels. These studies do not adequately reflect the need to know the overall reaction scheme when using ozone as a waste-water treatment agent. Incomplete oxidation of the phenolic species may make it necessary to incorporate further measures to remove pollutants. At the same time, knowledge of factors affecting the overall reaction might save substantially on operating costs. Therefore, in this preliminary laboratory evaluation, both engineering and process aspects could be studied simultaneously.

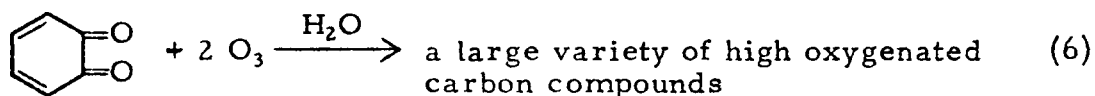
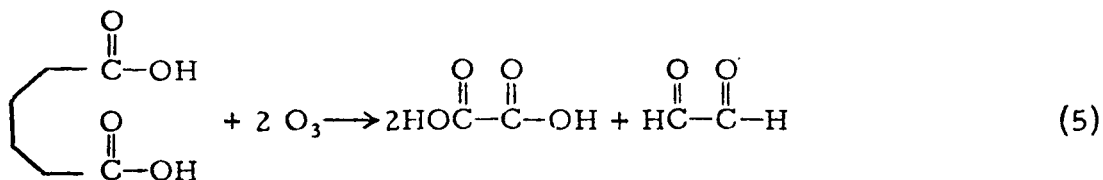
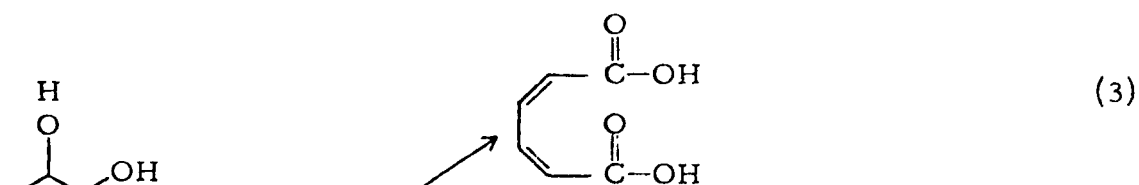
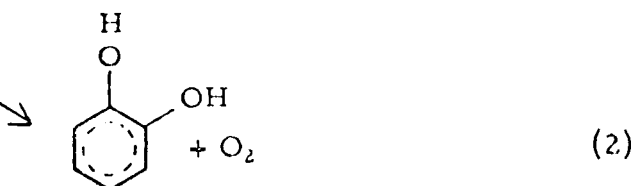
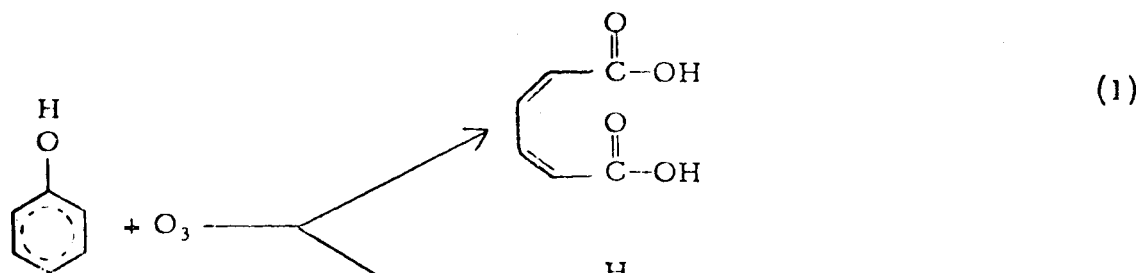
Bailey² has given an excellent review on the ozonation of organic compounds. The most important subject described in his review in relation to this work is the Criegee mechanism of ozonolysis of unsaturated compounds. The general mechanism is as follows:



* A = OH, OR, or OCOR

The first product (III), whose structure has not been fully confirmed, is highly unstable, very quickly cleaving to a zwitterion (V) and an aldehyde or ketone (IV). The fate of the zwitterion is largely determined by its environment. In the presence of a reactive solvent (HA), a great percentage of its reaction products are given by structure (VII). If the solvent is inert, then the zwitterion must either react with itself or with (IV). Reaction with (IV) usually results in the predominant formation of a monomeric ozonide (VI) when (IV) is an aldehyde. Reaction with itself usually produces a product high in (VIII) with small amounts of rearrangement products and polymeric oxides. So, as can be seen, the overall reaction of ozone to destroy phenol is quite complicated. In addition, note that ozone reacts quite slowly with C-C σ bonds. Complete phenol oxidation would take a considerable effort if ozone was used exclusively.

In view of the above discussion on the Criegee mechanism and some experimental findings, it was assumed that the following reactions tend to occur readily between phenol and ozone and their products:



However, once the reaction has proceeded to the point specified by the product of either reaction 5 or 6, double bonds are no longer available for rapid-rate attack, and the reaction should slow considerably at this point.

Eisenhauer⁵ stated that phenol oxidation by ozone was first order in ozone partial pressure. This is shown in Figure 80 by a group of lines originating at 50 mg/l phenol concentration. Changes in ozone addition rates from 7.5 to 15.5 mg/min produced linear changes in phenol reduction rates. In addition, since a plot of the log of the phenol concentration

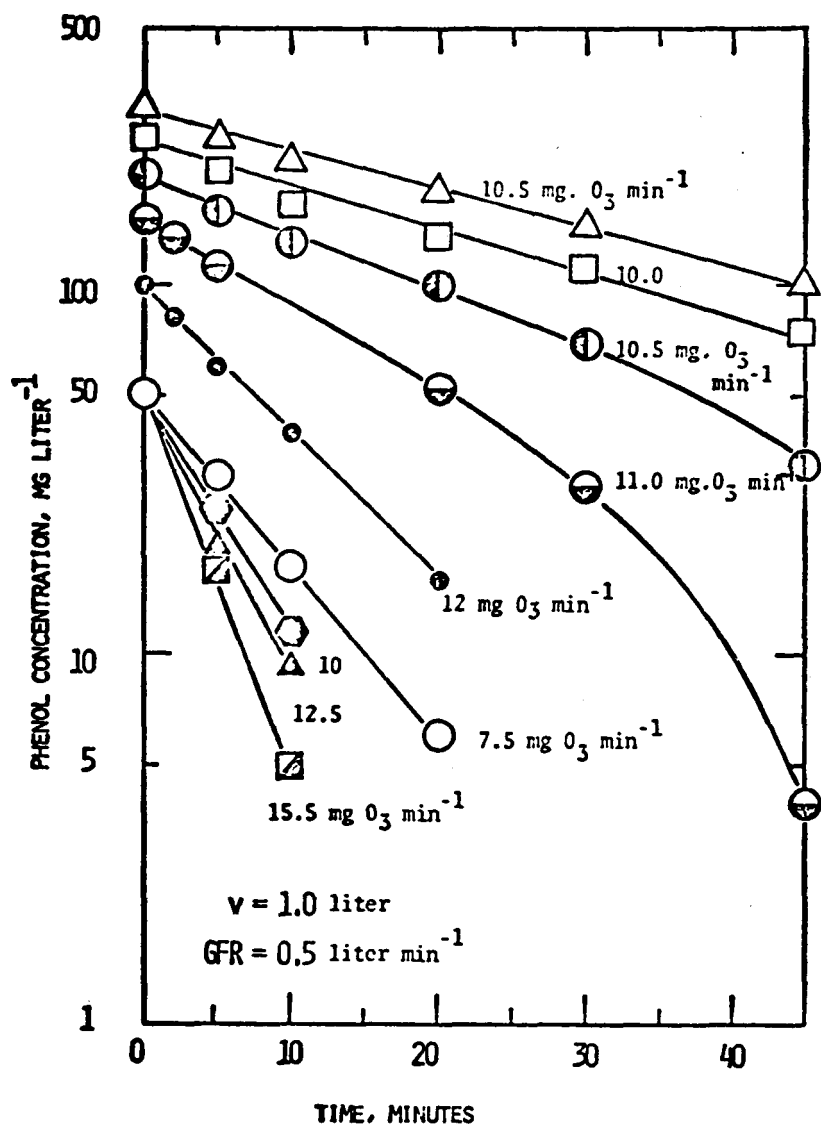


Figure 80. PHENOL OXIDATION REPORTED BY EISENHAUER⁵

versus the reaction time produces a reasonably straight line, the reaction is also first order in phenol concentration. The first-order dependence upon phenol concentration is also reflected in the work of English⁶ (Figure 81). Therefore the reaction rate can be expressed by the equation —

$$\frac{d(\phi\text{-OH})}{dt} = k [\phi\text{-OH}] P_{O_3} \quad (7)$$

where —

$$\begin{aligned} [\phi\text{-OH}] &= C_6H_5OH \\ [\phi\text{-OH}] &= \text{equilibrium concentration of phenol in solution, mg/l} \\ P_{O_3} &= \text{equilibrium concentration of } O_3 \text{ in the gas phase, mg/l.} \end{aligned}$$

In addition, English noted a dramatic change in reaction rate with an increase in pH at equivalent levels of phenol. From this he deduced that there is a reduction in the oxidation potential of phenol as the pH increases.

Eisenhauer shows a psuedo-zero-order reduction in phenol content as a result of the total depletion of ozone in the reaction zone. From his experiments, approximately 0.48 milligram of phenol is destroyed per milligram of ozone consumed. In molar quantities this is about 4.1 to 4.2 moles of ozone per mole of phenol. Since there is no indication of pH in Eisenhauer's work, his starting materials were assumed to be neutral water and phenol. Therefore, this ozone utilization corresponds to a reaction with neutral species.

To calculate a rate constant from Eisenhauer's data, two conditions must be assumed for the reaction: 1) first-order ozone partial pressure and phenol concentration and 2) an ozone consumption of 2.07 mg/l mg of phenol consumed (when oxidizing neutral species). Using these constraints, the following rate constant is based on a batch test:

$$k = \frac{(GFR) \ln \left\{ \frac{[\phi\text{-OH}]_{(f)}}{[\phi\text{-OH}]_{(i)}} \right\}}{O_3 (\Delta t) - 2.07 \{ [\phi\text{-OH}]_{(f)} - [\phi\text{-OH}]_{(i)} \}} \quad (8)$$

where—

$$\begin{aligned} GFR &= \text{gas flow rate, l/min} \\ \Delta t &= \text{total reaction time, min} \\ O_3 &= \text{ozone addition rate, mg/min} \\ [\phi\text{-OH}] &= \text{phenol concentration, mg/l.} \end{aligned}$$

Using the above equation and Eisenhauer's data, the value of k is shown in Table 43. Note that the value of k decreases with an increase in the log mean concentration of phenol in the batch tests, seeming to indicate that the reaction may not be quite first order in phenol concentration.

However, at reasonably constant levels of phenol, the rate constant decreases with a decrease in flow rate, so apparently the reaction between

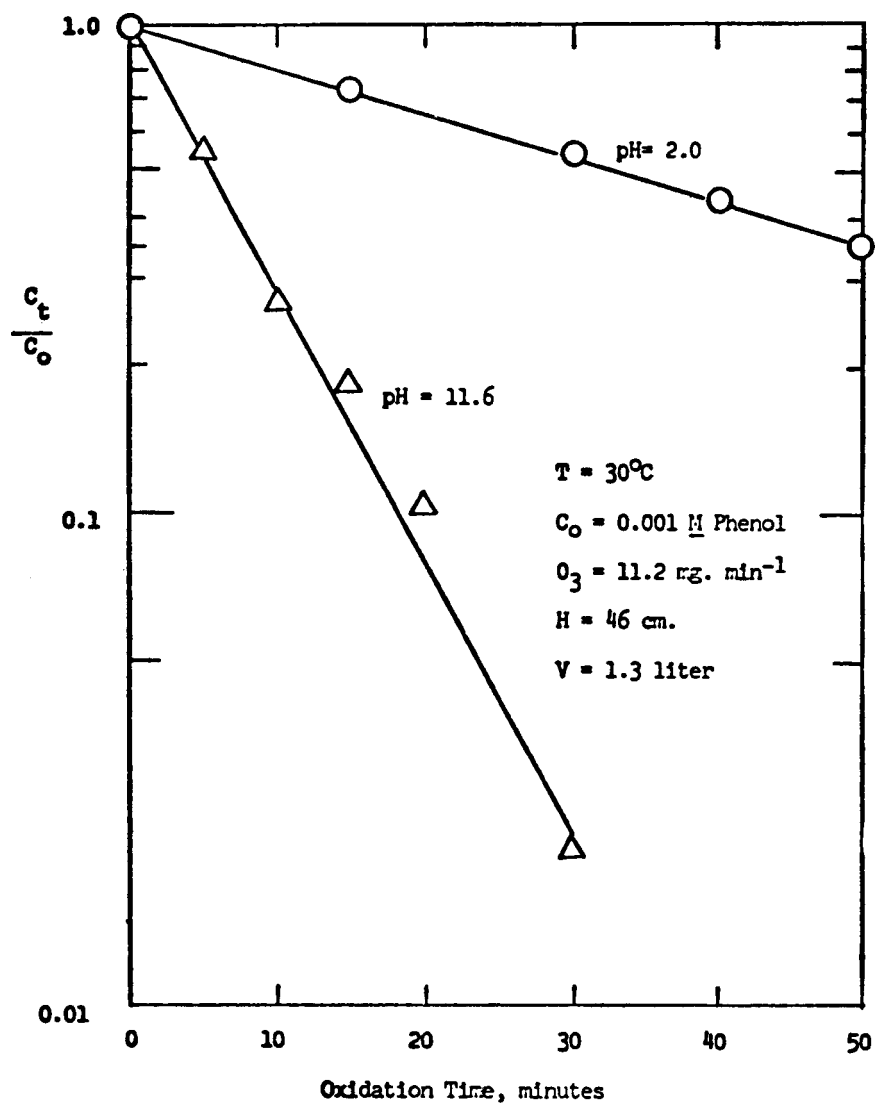


Figure 81. PHENOL OXIDATION REPORTED BY ENGLISH⁶

Table 43. PHENOL OZONATION RATE CONSTANT
AS DETERMINED FROM EISENHAUER'S⁵ DATA

<u>Inlet Ozone Feed Rate</u> —mg/min—	<u>Total Gas Flow Rate</u> —l/min—	<u>Total Reaction Time</u> —min—	<u>Initial Phenol Concentration</u> —mg/l—	<u>Final Phenol Concentration</u> —mg/l—	<u>Reaction Rate Constant</u> —l ² /mc-min—
10.7	0.5	40	248.5	102.0	-6.09×10^{-4}
10.7	0.5	20	299.0	180.5	-5.49×10^{-4}
10.25	0.5	58	229.0	39.5	-8.90×10^{-4}
10.25	0.5	30	249.0	109.0	-6.92×10^{-4}
10.5	0.5	45	198.0	32.0	-1.12×10^{-3}
10.5	0.5	30	151.1	28.6	-1.46×10^{-3}
11.8	0.5	20	99.2	16.2	-2.22×10^{-3}
15.6	0.5	10	49.7	5.1	-4.56×10^{-3}
15.6	0.5	5	49.7	17.1	-3.64×10^{-3}
12.35	0.5	10	49.7	9.3	-4.02×10^{-3}
10.25	0.5	10	49.6	11.0	-4.10×10^{-3}
7.4	0.5	20	50.5	6.0	-4.41×10^{-3}
8.08	0.4	10	50.8	15.4	-3.08×10^{-3}
8.08	0.4	20	50.8	4.15	-3.86×10^{-3}
5.07	0.3	20	50.2	10.1	-2.59×10^{-3}
5.07	0.3	10	50.2	22.5	-2.21×10^{-3}
2.92	0.2	20	50.2	14.7	-1.86×10^{-3}
1.40	0.1	30	49.8	23.1	-7.90×10^{-4}
1.45	0.1	45	49.8	13.8	-9.15×10^{-4}

Table 44. PHENOL OXIDATION RATES WITH OZONE

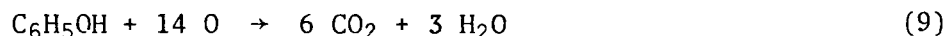
<u>O₃ Concentration</u>		<u>Total Gas Flow</u> —l/min—	<u>Average Phenol Concentration</u> —mg/l—	<u>Average COD*</u> Concentration —mg/l—	<u>Solution pH</u>	<u>Phenol Reduction Rate</u> —mg/min—	<u>COD Reduction Rate</u> —mg/min—
<u>Inlet</u>	<u>Outlet</u>						
30.24	0.0	0.248	525	1800	9.71	6.70	4.31
30.24	0.0	0.248	670	1900	10.21	7.20	4.97
30.24	0.0	0.248	742	1945	10.43	7.70	6.28
30.24	0.0	0.248	815	2000	10.67	8.50	7.83
30.24	0.0	0.248	893	2080	10.97	10.00	9.97
30.24	0.0	0.248	936	2130	11.16	11.30	12.14
30.24	0.0	0.248	958	2160	11.26	12.30	13.60
30.24	0.0	0.248	982	2200	11.37	14.60	15.51
29.6	8.90	0.273	510	2040	2.25	2.73	4.72
31.0	6.86	0.258	450	1748	2.72	2.73	2.90
31.3	7.00	0.247	510	1715	2.6	2.44	3.11
29.8	8.16	0.470	650	2055	2.7	3.84	2.76

* Entire COD value from phenol and intermediate products.

phenol and ozone occurs at the interface between the bubble and the liquid. This is merely the result of the low solubility of ozone in water with respect to its partial pressure in the gas at ambient temperature and pressure. Therefore, the largest amount of phenol and ozone combines at the interface. This may also be a partial explanation for the deviation from first-order dependence at high phenol concentrations, for, at these conditions, some diffusional limitations may occur, and the phenol concentration at the interface may be less than expected. This phenomenon is too complex to study in our simple laboratory setup and, therefore, is only conjecture.

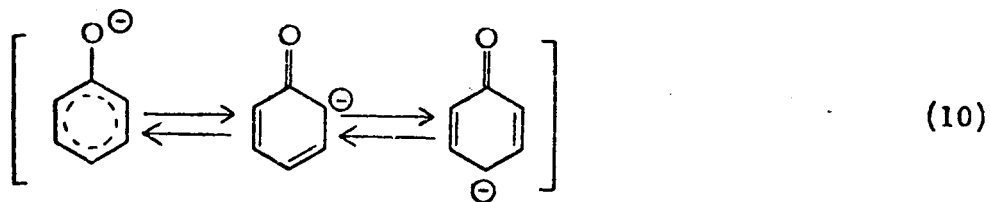
In view of the above literature analysis, certain additional information was needed. The results of the lab tests to determine that information are shown in Table 44.

First of all, very little mention was ever made about how far the reaction of ozone with phenol would proceed at a reasonable rate for the economical use of ozone. If the oxidation of phenol was complete, a minimum of 14 atoms of oxygen would be consumed for every mole of phenol:



In terms of theoretical chemical oxygen demand, this is 2.38 milligrams of chemical oxygen demand per milligram of phenol; therefore, full oxidation requires that the chemical oxygen demand reduction rate be about 2.38 times the phenol reduction rate. As can be seen in Table 44, this does not hold true. In the case of the bottom four runs, the average reduction in chemical oxygen demand was only 48% of that expected for full oxidation. Excluding the last run, the average was 57% of the total possible oxidation value. Assuming that the reaction proceeded as described in Reactions 5 and 6 and then stopped, the oxidation percentage should be 57.1% of the theoretical maximum. So, as can be seen, the reaction scheme offered by Reactions 1 to 6 approximates the oxidation of phenol actually occurring. Therefore, more than likely, only a 57% reduction in its chemical oxygen demand contribution can be economically expected. Unless the phenol was at very low levels to begin with, additional cleanup of phenol ozonation products would be required. In addition, the laboratory experiments confirmed that this waste water will have a slight yellowish cast from the stable diketone product, glyoxal. Finally, to achieve this 57% reduction in chemical oxygen demand plus total phenol destruction, the studies showed (in confirmation of Eisenhower's work) that at least 2.07 pounds of ozone would be needed for each pound of phenol, which is very costly.

Another question answered was the reason for an increased reaction rate with an increase in pH reported by English⁶ and the subsequent reduced ozone requirement reported by Niegowski.¹⁷ Caustic was added to the feed material to study the effect of pH. From the results, it is obvious that the reaction is much faster since no residual ozone was found compared with the amount at the low pH. However, the chemical oxygen demand reduction rate compared with the phenol reduction rate was also found to be much less than that found in the low-pH runs. Thus, phenol oxidation occurs at a faster rate than one or more of the secondary reactions, probably because at a sufficiently high pH, phenol ionizes to form the phenolate ion, $\text{C}_6\text{H}_5\text{O}^-$. This species should have a much lower oxidation potential because of stabilized localization of the double bonds in the benzene ring as a result of the following resonance structures:



This makes the double bonds more suitable for attack by ozone, thus increasing the speed of the reaction; therefore, the overall effect is an increase in the removal of phenol from solution, but a decrease in secondary reactions. This could also be seen from the fact that the product solution was brown instead of yellow. The intermediate *o*-quinone is red, and its combination with glyoxal could produce a brownish cast to the water. This brownish cast appears if the reaction is allowed to continue to the point where the secondary ozonation reactions are able to take place. However, to do this, the total consumption of ozone per pound of phenol would again be 2.07 pounds; thus, the only thing gained by the high pH is the initial ozonation rate of phenol.

Finally, to interpret the reaction kinetics described by the laboratory studies, a good approximation of the bubble size should be made so that some estimate of scale-up can be made. The bubble sizes averaged around 1 millimeter in diameter. The total height of liquid in the reaction column was 8-1/2 inches, and the column diameter was 1/2 inch. Considering the system as a continuous stirred reaction, the bubble size and the surface area per volume of solution are important. Unfortunately, the surface area per volume of solution is difficult to accurately determine, but a good conservative approximation for the experiments is about 1 sq cm/cc of volume, since there was approximately a 5-cc holdup of gas with a 0.25 l/min flow of gas through the reactor. There was no appreciable increase in holdup volume or a significant difference in bubble size when putting a larger volume of gas through; therefore, the experimentally determined reaction rate should be independent of the flow rate.

The average rate constant (assuming a first-order reaction for both ozone partial pressure and phenol concentration) for the four runs with a neutral phenolic species in water was $7.23 \times 10^{-4} \text{ l}^2/\text{mg-min}$, which agrees fairly well with that of Eisenhauer, considering the complexity of the system's effects. In terms of a reaction rate constant per unit of surface area between the gas and liquid, a good estimate would be about $3.0 \times 10^{-6} \text{ l}^2/\text{mg-min-sq cm}$, i.e. —

$$\frac{d(\phi\text{-OH})}{dt} = 3.0 \times 10^{-6} (A) [\phi\text{-OH}] P_{\text{O}_3}, \text{ mg/min} \quad (11)$$

where—

- (A) = surface area of gas-liquid contact, sq cm/cc
 $[\phi\text{-OH}]$ = equilibrium concentration of phenol in solution, mg/l
 P_{O_3} = equilibrium concentration of ozone in the gas phase, mg/l.

However, remember that the actual reaction rate on the actual solution may differ because of the other contaminants in the water that produce surface effects different from those in the laboratory study. Also, if the equilibrium solution in the reactor has a pH between 8 and 11, a more extensive rate determination will be needed because this is the transition region where there are appreciable amounts of both neutral phenolic species and phenolate ions, causing competitive reactions to take place.

Finally, as expected from the reaction scheme for ozonation, the reaction products are highly acidic, resulting in a decrease in the pH of the solution as the reaction proceeds. The amount of acid products per mole of phenol oxidized were quantitatively estimated from the last run. From the change in pH with a decrease in phenol concentration, two equivalent moles of acid are formed for each mole of phenol destroyed. Because of the presence of acid gases, etc., in the waste water being treated, the effect that this will have on the pH cannot be estimated without considering the total composition of the waste stream in question.

Conclusions

Ozonation does not seem to be a desirable means for the removal of phenolics at high concentrations for a number of reasons:

- a. The amount of ozone required for destruction of phenol is 2.07 lb/lb, assuming maximum utilization of the ozone produced.
- b. Only about 57% of the chemical oxygen demand equivalent of the phenolic species initially present will be reduced, thereby leaving appreciable levels of reaction products other than carbon dioxide and water and making additional cleanup necessary.
- c. Since the reaction rate is first order in phenol concentration and large phenol reductions are desired, more than one stage of operation would be needed for maximum ozone utilization.
- d. If a 0.1 ppm effluent is desired, it will take a considerable gas-liquid surface area for any appreciable reaction rate to occur at such a low phenol concentration. Thus, a considerable amount of ozone will be left in the gas coming from the reactor. (See rate Equation 11.)
- e. In addition, because of the unstable nature of ozone and its decomposition on solid surfaces, packed columns would probably increase the total amount of ozone required per mole of phenol destroyed.
- f. The acid products from phenol ozonation will probably result in a need to neutralize the product waste water, thus requiring an additional system and increased caustic consumption.

Therefore, unless phenol has already been sufficiently reduced to low levels by some other means such as extraction, ozonation by itself does not appear to be adequate as a means to remove phenol.

Devolatilization in the Slurry Dryer

The devolatilization of low-rank coal at temperatures up to 600°F (about that of the slurry dryer bed in the HYGAS Process) was investigated. The amount and compositions of gaseous products evolved by a lignite and a subbituminous coal at 400°F and 600°F were determined. The lignite used as feed to the HYGAS pilot plant was from the Savage mine, Montana. The subbituminous coal was from the Colstrip mine, Montana. Two samples of the subbituminous coal were tested: One had been retained from a hydrogasification test of the coal in 1970 in the 4-inch-diameter process development unit; the other had been recently obtained from C. F. Braun in connection with its design study.

Carbon dioxide and water were determined gravimetrically by heating a 0.5 to 1 gram sample in a tube in a furnace and sweeping the evolved gases into absorption bulbs with nitrogen. Some tar was formed in the 600°F runs; glass wool was placed in the tube downstream from the furnace to intercept it and prevent its fouling the water absorption bulb. Sample weight loss was also determined, and the results are shown in Table 45.

In a separate experiment, the gases evolved at 600°F were analyzed by mass spectrometry. Yields in weight percent of the original sample were obtained from the gravimetric carbon dioxide yield and the component-to-carbon dioxide ratios of the gas analysis. Total recovery ranged from 69% to 94% of the measured weight loss.

The measured weight losses of the lignite and the older subbituminous coal agree well with the results of some earlier devolatilization experiments (OCR Report No. 111, January 1974). However, the fresh sample of subbituminous coal lost only about 60% as much weight at 600°F as the older sample. This is probably caused by weathering of the older sample.

Coal-Water Partial Oxidation

A synopsis of the experiments conducted during June and July is given in Table 46.

The analytical data of experimental Runs 22 through 37 are summarized in Table 47. The particle counts of reflectant and nonreflectant particles by petrographic examination for Runs 22, 27 through 34, 36, and 37 are presented in Table 48. Based on the analytical results, the distribution of the oxidizing medium over the 170-mesh, Type-316 stainless-steel screen used as a gas distributor is nonuniform. The reactor design was therefore modified to accommodate a 1/16-inch-thick, Type-316, stainless-steel, perforated distributor plate. The assembly of the reactor with a distributor plate, shown in Figure 82, illustrates the use of a split ring to hold the distributor in place.

The equipment was improved in October. A modified flow sheet for bench-scale coal-water partial oxidation studies is illustrated in Figure 83. Improvements in equipment setup include a knockout pot for trapping condensates in the reactor off-gases and a 1/16-inch-thick, stainless-steel plate, gas distributor to ensure uniform gas distribution.

Table 45. DEVOLATILIZATION OF LIGNITE AND SUBBITUMINOUS COAL AT 400°F AND 600°F

	Montana Lignite [†]		Montana Subbituminous Coal [‡]			
	1973 Sample		1970 Sample		1974 Sample	
	400°F	600°F	400°F	600°F	400°F	600°F
	wt %, dry basis					
Gravimetric Analysis						
Water of Decomposition						
Trial 1	3.66	4.89	1.94	4.18	0.67	1.71
Trial 2	3.34	5.13	2.41	3.49	0.89	1.81
Average	3.50	5.01	2.18	3.84	0.78	1.76
Carbon Dioxide						
Trial 1	0.56	2.80	0.56	2.69	0.31	1.11
Trial 2	0.43	2.99	0.67	2.43	0.33	0.97
Average	0.50	2.90	0.62	2.56	0.32	1.04
Total Loss						
Trial 1	3.95	9.12	2.56	7.31	1.32	4.88
Trial 2	4.53	9.72	3.08	7.52	1.33	3.94
Average	4.24	9.42	2.82	7.42	1.32	4.41
Mass Spectrometer Analysis*						
Hydrocarbons						
Methane	--	0.014	--	0.007	--	0.005
Ethane	--	0.011	--	0.019	--	0.011
Propane	--	0.002	--	0.014	--	0.012
Propylene	--	0.012	--	0.019	--	0.000
Pentenes	--	0.000	--	0.011	--	0.000
Benzene	--	0.002	--	0.022	--	0.002
Toluene	--	0.016	--	0.008	--	0.002
Nonhydrocarbons						
Acetone	--	0.008	--	0.016	--	0.005
Carbon Monoxide	--	0.28	--	0.36	--	0.15
Hydrogen Sulfide	--	0.018	--	0.011	--	0.002
Methanol	--	0.018	--	0.003	--	0.000
Methyl Mercaptan	--	0.015	--	0.003	--	0.004
Sulfur Dioxide	--	0.050	--	0.084	--	0.042
Total Recovery	--	90	--	0.94	--	69

* Water-free and calculated to original sample basis through gravimetric carbon dioxide yield and component-to-carbon dioxide ratios of the mass spectrometer analysis.

[†] Savage mine.

[‡] Colstrip mine.

Table 46. EXPERIMENTS CONDUCTED DURING JULY

<u>Test No.</u>	<u>Date</u>	<u>Operating Conditions</u>	<u>Remarks</u>
26	6/28/74	80 g Ireland mine coal + 160 g water + 0.4 CF/min compressed air at 1000 psig. Reaction temperature = 550° to 650°F. Reaction time = 5 min.	Water evaporated and the residue dry. Could not control reactor temperature.
27	7/01/74	80 g Ireland mine coal + 160 g water + 0.4 CF/min compressed air at 1000 psig. Reaction temperature = 500° to 510°F. Reaction time = 8 min.	Residue submerged in water.
28	7/02/74	80 g Ireland mine coal + 160 g water + 0.25 CF/min compressed oxygen at 1000 psig. Reaction temperature = 500° to 525°F. Reaction time = 11 to 13 min.	Residue submerged in water.
29	7/08/74	80 g Illinois No. 6 coal + 160 g water + 0.4 CF/min compressed air at 1000 psig. Reaction temperature = 500° to 515°F. Reaction time = 12 min.	Residue submerged in water.
30	7/09/74	80 g Illinois No. 6 coal + 160 g water + 0.25 CF/min compressed oxygen at 1000 psig. Reaction temperature = 500° to 530°F. Reaction time = 14 min.	Residue submerged in water.
31	7/10/74	80 g Illinois No. 6 coal + 218 g water + 0.4 CF/min compressed air at 1000 psig. Reaction temperature = 520° to 530°F. Reaction time = 4 min.	Experiment stopped after 4 min because of a plug in back-pressure regulator.
32	7/11/74	80 g Illinois No. 6 coal + 193 g water + 0.4 CF/min compressed air at 1000 psig. Reaction temperature = 500° to 510°F. Reaction time = 11 min.	Residue submerged in water.

Table 46, Cont. EXPERIMENTS CONDUCTED DURING JULY

<u>Test No.</u>	<u>Date</u>	<u>Operating Conditions</u>	<u>Remarks</u>
33	7/12/74	80 g Illinois No. 6 coal + 180 g water + 0.4 CF/min compressed oxygen at 1000 psig. Reaction temperature = 500° to 540°F. Reaction time = 9 to 10 min.	Considerable amount of water evaporated. Residue was submerged under water.
34	7/15/74	80 g Illinois No. 6 coal + 180 g water + 0.4 CF/min compressed air at 1250 psig. Reaction temperature = 500° to 530°F. Reaction time = 26 min.	Almost all the water evaporated but the residue was moist and granular.
35	7/19/74	80 g Illinois No. 6 coal + 180 g water. 0.4 CF/min of compressed oxygen at 1250 psig. Reaction temperature = 540°-560°F. Reaction time = 14 min.	Reactor temperature increased abruptly at the end of a 14-minute reaction time. Residue was dry and partly fused.
36	7/23/74	80 g Illinois No. 6 coal + 180 g water. 0.4 CF/min of compressed oxygen at 1250 psig. Reaction temperature = 540°-560°F. Reaction time = 10-11 min.	Most of the water was lost, but the residue was moist and granular.
37	7/24/74	80 g Illinois No. 6 coal + 180 g water. 0.25 CF/min of compressed oxygen at 1250 psig. Reaction temperature = 520°-560°F. Reaction time = 16-17 min.	Residue found submerged in water.
38	7/25/74	80 g Illinois No. 6 coal + 180 g water. 0.25 CF/min of compressed oxygen at 1250 psig.	The exit-gas line plugged up. and the test was aborted after 5 min.
39	7/29/74	50 g Illinois No. 6 coal + 150 g water. 0.25 CF/min of compressed oxygen at 1250 psig. Reaction temperature = 530°-550°F.	Reactor temperature increased abruptly after 11 min, indicating total loss of water. The residue was dry.

Table 47. ANALYTICAL RESULTS OF UNTREATED AND TREATED COALS

Test No.	Proximate-Analysis, wt % (Moisture-Free Basis)			% Loss in Volatile Matter	Ultimate Analysis, wt % (Moisture-Free Basis)		% Loss in Hydrogen	Agglom- eration Test	Free Swelling Index
	Volatile Matter	Ash	Fixed Carbon		Carbon	Hydrogen			
Untreated Ireland Mine Coal									
	39.6	10.56	49.84	--	70.6	5.01	--	Fused	7
Treated Ireland Mine (Pittsburgh Seam No. 8) Coal									
22	35.81	7.63	55.56	9.57	74.5	5.13	--	Fused	5.5
27	35.44	8.25	56.31	10.51	72.6	4.73	5.59	Fused	5
28	35.26	8.25	56.49	10.96	70.1	4.24	15.37	Caked	2.5
Untreated Illinois No. 6 Coal									
	37.76	7.99	54.25	--	74.57	5.10	--	Fused	7
Treated Illinois No. 6 Coal									
29	35.45	7.68	56.87	6.12	73.8	4.91	3.73	Fused	3
30	35.29	8.23	56.48	6.54	71.3	4.53	11.18	Caked	2
31	35.59	8.41	56.00	5.75	73.7	4.96	2.75	Fused	3
32	34.73	8.25	57.02	8.02	73.0	4.89	4.12	Fused	3
33	35.52	8.80	55.68	5.93	70.7	4.58	10.20	Caked	2.5
34	35.61	8.57	55.82	5.69	71.4	4.62	9.41	Fused	3
36	28.02	12.20	59.78	25.79	70.2	3.76	26.27	Caked	0.5
37	35.94	9.69	54.37	4.82	69.3	4.25	16.67	Fused	2.5

Table 48. PETROGRAPHIC ANALYSIS OF TREATED COAL SAMPLES

Test No.	Type of Coal	Nonagglomerating Particles	Particles Devolatilized, but Without Reflectant Borders	Untreated Particles
			%	
22	Ireland Mine	1.3	75.7	23.0
27	Ireland Mine	42.2	12.0	45.8
28	Ireland Mine	93.0	4.7	2.3
29	Illinois No. 6	79.3	8.7	12.0
30	Illinois No. 6	68.7	5.9	25.4
31	Illinois No. 6	1.3	64.0	34.7
32	Illinois No. 6	75.4	10.3	14.3
33	Illinois No. 6	66.3	1.0	32.7
34	Illinois No. 6	83.0	4.3	12.7
36*	Illinois No. 6	77.8	5.6	4.0
37	Illinois No. 6	69.2	13.5	17.3

*12.6% of these particles were of unusually high reflectance, indicating localized combustion.

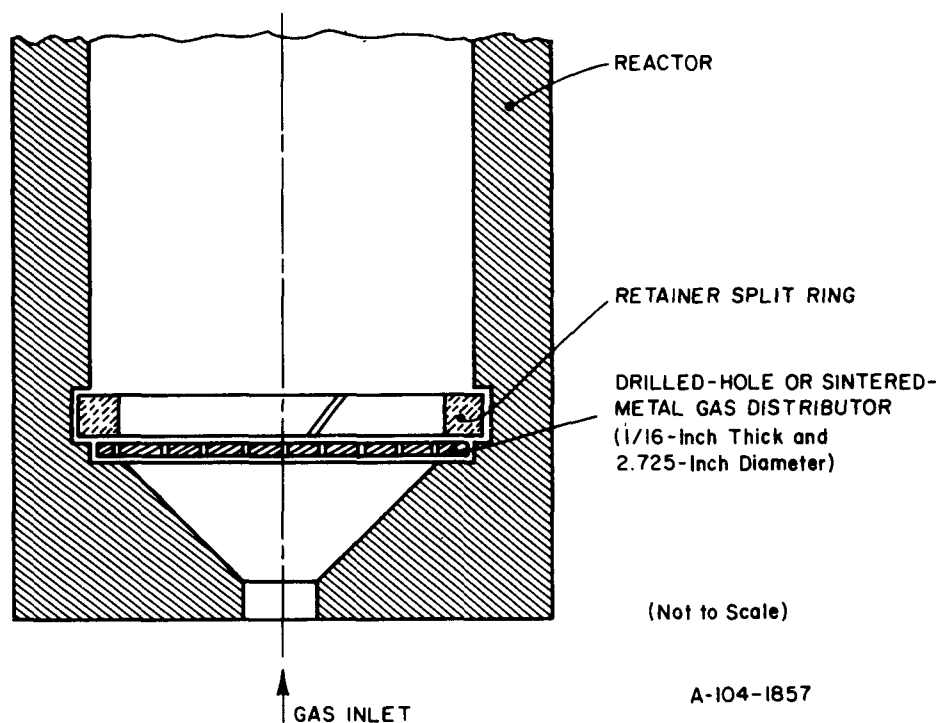


Figure 82. GAS DISTRIBUTOR ASSEMBLY

LEGEND

BPR BACK PRESSURE
REGULATOR

BPV BYPASS VALVE

BR BUBBLER

CV CHECK VALVE

DR DRYER

FBH FLUIDIZED-BED
HEATER

GS GAS SAMPLING

HE HEAT EXCHANGER

M MOTOR

MM MANOMETER

MS MAGNETIC STIRRER

NV NEEDLE VALVE

OAN OXYGEN ANALYZER

P PRESSURE GAGE

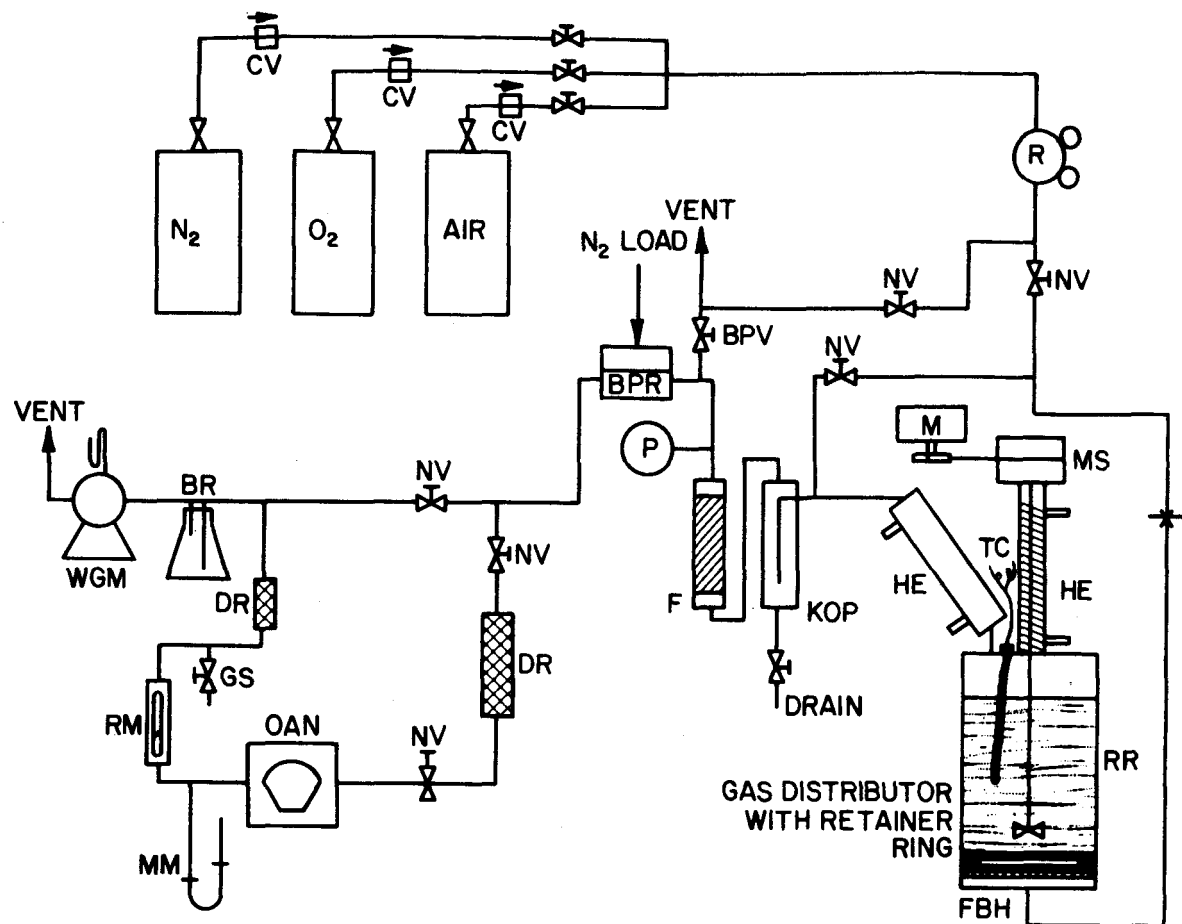
R REGULATOR

RM ROTAMETER

RR REACTOR

TC THERMOCOUPLE

WGM WET-GAS METER



A-114-2030

Figure 83. MODIFIED EXPERIMENTAL SETUP FOR BENCH-SCALE COAL-WATER PARTIAL OXIDATION STUDIES

Two runs were made in the improved experimental setup illustrated in Figure 83. Excessive carry-over of condensate, observed in the knockout pot, can be attributed to the gas superficial velocities exceeding 0.5 ft/s through the 1/4-inch-diameter copper tubing in the heat exchanger used for cooling exit gases. To circumvent the problem of condensate carry-over and to promote better refluxing, the 1/4-inch gas outlet was capped, and the gases were withdrawn through the 1/2-inch, Schedule 80, stainless-steel pipe, shown in Figure 84. The 1/2-inch pipe, enclosing the magnetic stirrer, was cooled by dry ice to return the condensate to the reactor. The dry-ice cooling jacket did not perform adequately. As a result, checking the heat-transfer calculations for the reflux section led to the conclusion that it is essential to prevent the formation of "rat-holes" in the dry-ice section for it to perform satisfactorily. Therefore, it is desirable to cool the off-gases with a liquid coolant, and for this purpose, a refrigerated-methanol cooling system was designed.

The experiments conducted in November are summarized in Table 49, and analytical data of the experiments conducted during November and December are reported in Tables 50 and 51. Note that the free swelling index (FSI) was reduced from about 7 to 1 or 1.5 from the controlled oxidation of coal with only a small loss of volatile matter. This is a highly encouraging result. Coals with an FSI of 2 or less have been fed directly to gasifiers without encountering agglomeration problems. The laboratory test for agglomeration¹⁴ is very severe. It was developed for small test equipment, but is unnecessarily harsh for large pilot-plant units. The next set of experiments will be oriented toward reducing the reaction time with oxygen.

A heat exchanger designed to condense and reflux the water contained in the reactor off-gases performed satisfactorily. Six experiments were conducted during January to determine the effect of residence time and oxygen flow rate on the extent of pretreatment. A summary of the experiments and a part of the analysis of the treated samples are reported in Tables 52 and 53, respectively.

In February, two experiments were conducted to determine partial oxidation on Ireland mine coal in a water slurry. Details of the experimental conditions are shown in Table 54.

The agglomerating properties of high-volatile bituminous coals (Illinois No. 6 and Ireland mine coals) were reduced by coal-water partial oxidation at 1000 psig and 540°F. The free swelling index (FSI) was reduced from 7 to 1 in 15 to 25 minutes of reaction with an associated decrease in volatile matter content from 37.8% to 35% for the Illinois No. 6 coal and from 39.6% to about 34% for the Ireland mine coal. If desired, the FSI could be reduced to zero, with a further loss in volatile matter.

This method produces a nonagglomerating char, as measured by the criterion by Gasior *et al.*¹² (i.e., FSI ≤ 1.5). However, the char is not free-flowing as measured by the more severe IGT agglomeration test.

The pretreated material was sufficiently surface-oxidized, as characterized by 80% to 90% reflectant particles. The material did not swell up as did the product from the atmospheric pressure pretreatment. It contained as much as 95% of the original total carbon and hydrogen, thus indicating its potential to produce excellent methane yields.

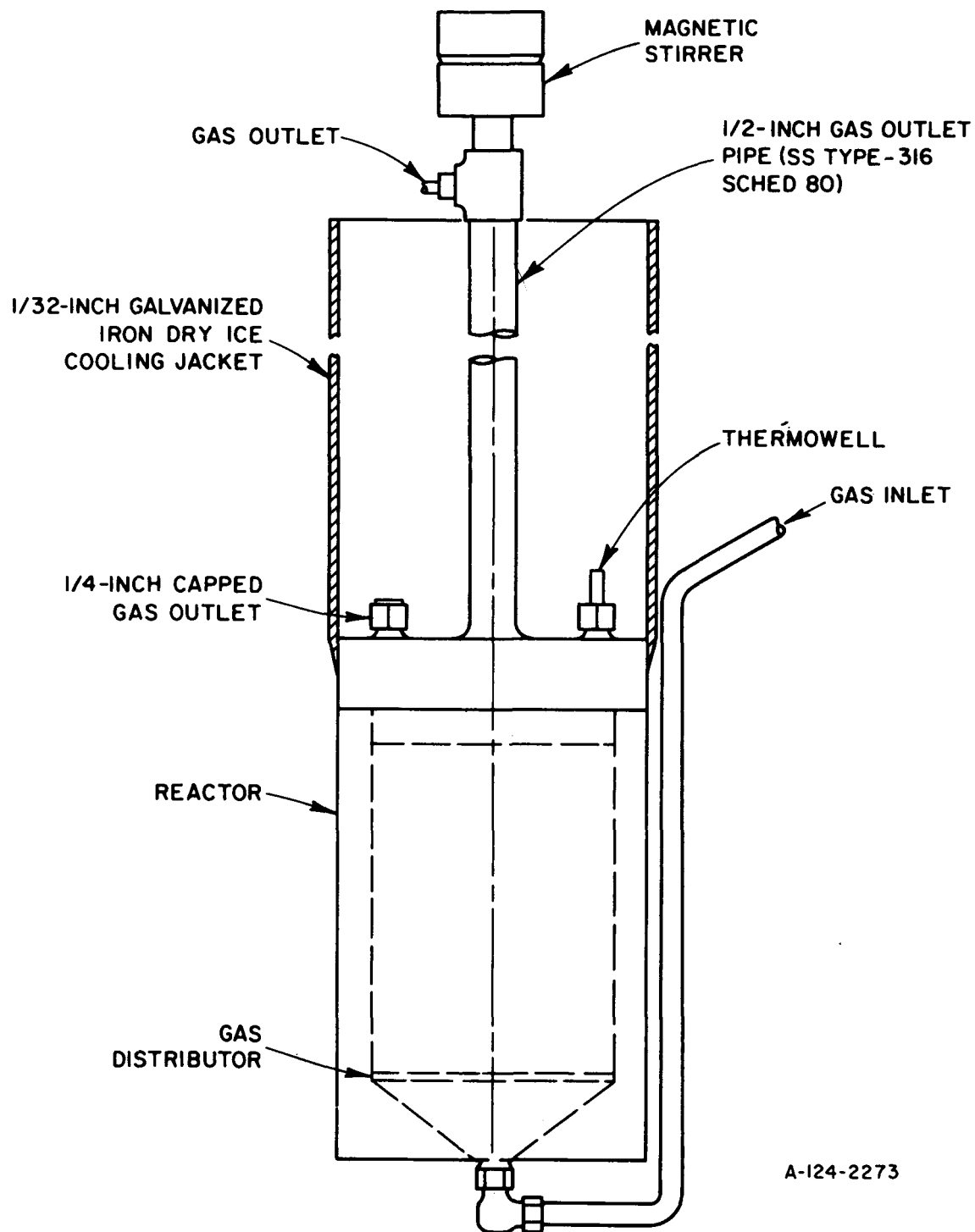


Figure 84. MODIFIED REACTOR ASSEMBLY

Table 49. SUMMARY OF EXPERIMENTS

<u>Run No.</u>	<u>Date</u>	<u>Operating Conditions</u>	<u>Remarks</u>
40	11/08/74	50 g Illinois No. 6 coal + 150 g water. 0.25 CF/min compressed air at 1000 psig. Reaction temperature = 520°F. Reaction time = 6 minutes.	At the end of 6 minutes, about 120 ml of water were collected in the knockout pot, and the reaction was terminated. The moist and granular coal sample was submitted for analysis.
41	11/11/74	50 g Illinois No. 6 coal + 150 g water. 0.25 CF/min compressed air at 1000 psig. Reaction temperature = 505°F. Reaction time = 15 minutes.	At the end of the reaction period, the coal was dry and partially burnt. When measured, the condensate amounted to about 150 ml.
42	11/14/74	50 g Illinois No. 6 coal + 175 g water. 0.20 CF/min compressed air at 1000 psig. Reaction temperature = 525°F. Reaction time = 31 minutes.	With the changes in the gas outlet, good refluxing was achieved and the reaction was terminated after 31 minutes.
43	11/15/74	50 g Illinois No. 6 coal + 175 g water. 0.20 CF/min compressed air at 1000 psig. Reaction temperature = 515° to 520°F. Reaction time = 30 minutes.	Good reproducibility of Run 42. Reaction terminated at the end of 30 minutes.
44	11/18/74	50 g Illinois No. 6 coal + 175 g water. 0.17 CF/min compressed oxygen at 1000 psig. Reaction temperature = 530°F. Reaction time = 30 minutes.	Good refluxing was achieved and about 37 ml of condensate was collected in the knockout pot.
45	11/19/74	50 g Illinois No. 6 coal + 175 g water + 0.17 CF/min compressed oxygen at 1000 psig. Reaction Temperature = 530°F. Reaction Time = 16 min.	The run was discontinued after 16 min. because of the distillation of the water contained in the slurry. This is attributed to inadequate cooling by dry ice. The moist residue was submitted for analysis.
46	11/20/74	80 g Illinois No. 6 coal + 175 g water + 0.17 CF/min compressed oxygen at 1000 psig. Reaction Temperature = 535° to 540°F. Reaction Time = 13 min.	Poor cooling of off-gases resulted in the total distillation of the water contained in the slurry. The dry and charred residue was discarded.
47	11/21/74	80 g Illinois No. 6 coal + 175 g water + 0.1 CF/min compressed oxygen at 1000 psig. Reaction Temperature = 530°F. Reaction Time = 30 min.	A plug that developed in the off-gases line is responsible for the reduction in gas flow. The treated slurry was submitted for analysis.
48	11/25/74	80 g Illinois No. 6 coal + 175 g water + 0.1 CF/min compressed oxygen at 1000 psig. Reaction Temperature = 530°F. Reaction Time = 25 min.	About 118 ml of the water in the slurry was collected in the knockout pot at the end of 25 min. The wet residue was submitted for analysis.

Table 50. ANALYTICAL RESULTS OF UNTREATED AND TREATED
ILLINOIS NO. 6 COALS

Coal	Proximate Analysis*			Ultimate Analysis*		Agglomeration Test	Free Swelling Index
	Volatile Matter	Ash	Fixed Carbon	C	H ₂		
Untreated Ill. No. 6	37.81	8.68	53.51	--	--	Fused	7
Treated Ill. No. 6							
Test 40	34.24	8.71	57.05	72.8	4.67	Fused	1
Test 42	33.91	8.22	57.87	71.2	4.28	Caked	1
Test 43	35.23	8.43	56.34	71.6	4.59	Caked	1.5
Test 44	35.13	10.76	54.11	68.2	4.14	Caked	1
Test 45	36.0	8.21	55.79	71.7	4.47	Caked	3
Test 47	35.18	8.8	56.02	71.7	4.39	Caked	1.5
Test 48	36.0	8.41	55.59	70.7	4.25	Caked	1

* Moisture-free basis.

Table 51. PETROGRAPHIC ANALYSIS OF TREATED ILLINOIS NO. 6 COAL

Test No.	Nonagglomerating Particles	Particles Devolatilized but Without Reflectant Borders	Untreated Particles
		%	
40	0	31.1	68.9
42	49.2	14.8	36.0
43	84.4	4.7	10.6
44	61.7	13.7	24.6
45	73.6	3.7	22.7
47	98.3	0	1.7
48	53.8	12.4	33.8

Table 52. SUMMARY OF EXPERIMENTS

Run No.	Date	Operating Conditions	Remarks
49	12/23/74	80 g Illinois No. 6 coal + 185 g water + 0.123 CF/min compressed oxygen at 1000 psig. Reaction Temperature = 525°F to 530°F. Reaction Time = 50 min.	Gas flow was irregular. The remaining coal, totally submerged in water, was submitted for analysis.
50	12/27/74	80 g Illinois No. 6 coal + 185 g water + 0.20 CF/min compressed oxygen at 1000 psig. Reaction Temperature = 525°F to 530°F. Reaction Time = 24 min.	--
51	12/30/74	80 g Illinois No. 6 coal + 185 g water + 0.24 CF/min compressed oxygen at 1000 psig. Reaction Temperature = 530°F. Reaction Time = 16 min.	--
52	1/02/75	80 g Illinois No. 6 coal + 185 g water + 0.30 CF/min compressed oxygen at 1000 psig. Reaction Temperature = 520°F. Reaction Time = 12 min.	--
53	1/09/75	80 g Illinois No. 6 coal + 185 g water + 0.24 CF/min compressed oxygen at 1000 psig. Reaction Temperature = 530°F. Reaction Time = 60 min.	--
54	1/21/75	80 g Illinois No. 6 coal + 185 g water + 0.24 CF/min compressed oxygen at 1000 psig. Reaction Temperature = 530°F to 535°F. Reaction Time = 60 min.	Run No. 53 was repeated.

Table 53. ANALYTICAL RESULTS OF UNTREATED AND TREATED
ILLINOIS NO. 6 COALS

Coal	Proximate Analysis*			Ultimate Analysis*		Agglomeration Test	Free Swelling Index
	Volatile	Ash	Fixed				
	Matter		Carbon	C	H		
	wt %						
Untreated							
Ill. No. 6	37.81	8.68	53.51	--	--	Fused	7
Treated							
Ill. No. 6							
Test 49	35.15	11.89	52.96	68.2	4.45	Caked	2
Test 50	36.36	8.71	54.93	70.4	4.50	Caked	2
Test 51	35.79	8.61	55.60	70.9	4.38	Caked	1.5
Test 52	36.65	9.00	54.35	70.9	4.58	Caked	3.5
Test 53	32.90	16.70	50.40	63.8	4.02	Partially caked	0

* Moisture-free basis.

Table 54. SUMMARY OF EXPERIMENTS

<u>Test No.</u>	<u>Date</u>	<u>Operating Conditions</u>	<u>Remarks</u>
55	2/5/75	80 g Ireland mine coal + 170 g water + 0.25 CF/min compressed oxygen at 1000 psig. Reaction temperature = 535°F. Reaction time = 30 min.	Satisfactory experimental run. The treated slurry submitted for analysis.
56	2/7/75	80 g Ireland mine coal + 165 g water + 0.25 CF/min compressed oxygen at 1000 psig. Reaction temperature = 535°F to 540°F. Reaction time = 60 min.	Satisfactory experimental run. The treated slurry submitted for analysis.

Table 55 contains analyses of Illinois No. 6 and the Ireland mine coal samples treated in experimental Test 54, 55, and 56. The petrographic analyses of coals to determine the extent of surface oxidation are shown in Table 56.

Summary Report on Coal-Water Partial Oxidation

A coal-water partial oxidation process was developed, based on a patent by Lee and Schora,¹⁵ to reduce the agglomerating properties of high-volatile bituminous coals during hydrogasification. The process essentially consists of treating a coal-water slurry with an oxygen-containing gas at elevated temperature and pressure to effect surface oxidation of the coal, which is thus rendered nonagglomerating and is suitable as feed material for a pressurized hydrogasification process.

The submerged partial oxidation process results in a smaller loss of volatile matter compared with the fluidized-bed pretreatment methods. In addition, pumping coal-water slurries into high-pressure reactors has several advantages over the available dry solids feeding methods; therefore, a coal-water partial oxidation process can be included as an integral part of any hydrogasification process that uses caking coals.

Coal slurries, ranging in consistency from 20% to 30% solids, by weight, were charged into a pressure vessel, kept agitated with a magnetic stirrer, and pressurized with nitrogen at 1000 to 1500 psig. The reactor was heated rapidly for 3 to 5 minutes to the boiling point of water at the operating pressure by immersion in a sand fluidized bed, preheated to about 850°F to 900°F. Partial oxidation was carried out at temperatures ranging from 450°F to 560°F in the presence of either air or oxygen for reaction times up to 60 minutes. The types and analyses of coals tested in the different sets of experiments are reported in Table 57.

Table 55. ANALYTICAL RESULTS OF UNTREATED AND TREATED COALS

Coal	Proximate Analysis*			Ultimate Analysis*		Agglomeration Test	Free Swelling Index
	Volatile Matter	Ash	Fixed Carbon wt %	C	H		
Untreated Ill. No. 6	37.81	8.68	53.51	74.2	5.0	Fused	7
Treated Ill. No. 6							
Test 54	31.54	20.69	47.77	60.7	3.87	Partially caked	0
Untreated Ire- land Mine	39.60	10.56	49.84	70.6	5.01	Fused	7
Treated Ire- land Mine							
Test 55	34.97	18.49	46.54	59.7	3.52	Partially caked	0
Test 56	35.68	24.12	40.20	52.8	3.1	Partially caked	0.5

* Moisture-free basis.

Table 56. PETROGRAPHIC ANALYSIS OF TREATED ILLINOIS NO. 6
AND IRELAND MINE COALS

Test No.	Nonagglomerating Particles	Particles Devolatilized, but Without Reflectant Borders %	Untreated Particles
49	82.7	4.3	13.0
50	45.3	14.7	40.0
51	71.5	6.4	22.1
52	89.6	0.0	10.4
53	77.6	8.5	13.9
54	93.9	0.0	6.1
55	92.4	5.2	2.4
56	97.2	1.2	1.6

Table 57. TYPES AND ANALYSES OF HIGH-VOLATILE BITUMINOUS COAL TEST SAMPLES

Type of Coal	Coal Used as Test Samples in Experimental Tests*	Proximate Analysis (Dry Basis)			Ultimate Analysis (Dry Basis)		Agglomer- ation Test	Free Swelling Index
		Volatile	Ash	Fixed				
		Matter		Carbon	C	H		
				wt %				
Ill. No. 6 (Source Unknown) hvCb	12, 13, 17, 19	38.26	9.15	52.59	70.2	4.78	Caked	1
Ill. No. 6 (Sahara Mine, Saline Co., Ill.) hvBb	29, 30, 32-34 36, 37	37.76	7.99	54.25	74.57	5.10	Fused	7
Ill. No. 6 (Sahara Mine, Saline Co., Ill.) hvBb	40, 42-45, 47-54	37.81	8.68	53.51	74.20	5.00	Fused	7
Pittsburgh Seam No. 8 (Ireland Mine, Marshall Co., W. V.) hvAb	6, 9, 10, 14, 15, 18, 21, 27, 28, 55, 56	39.6	10.56	49.84	70.6	5.01	Fused	7

* The excluded run numbers correspond to aborted experiments.

The completed experimental tests and the analyses of treated samples using the three types of coal are summarized in Tables 58, 59, and 60. The data obtained from the partial oxidation experiments are arranged in these tables in the order of the corresponding contact time between the coal and the oxidizing medium.

Discussion of Results

The Illinois No. 6 coal of unknown source, shown in Table 57, was severely weathered because of the low free swelling index (FSI). Pre-treatment with air at 1500 psig at about 520°F to 560°F for 20 minutes reduced the coal's volatile matter content from 38% to 35%, Table 58. No changes in the IGT agglomeration test or FSI were observed even though about 74% of the particles had been pretreated. A brief description of the FSI, of the IGT agglomeration test, and of the method of characterizing the agglomeration tendency are presented later.

It is apparent in Table 59 that the FSI of Illinois No. 6 high-volatile B bituminous coal (hvBb) was reduced from about 7 to 0 by partial oxidation of the coal in a coal-water slurry with air and oxygen. The FSI of Illinois No. 6 hvBb coal can be changed from 7 to 1 by reducing the volatile matter content from 37.8% to about 35.0%. Table 59 also indicates that this coal's volatile matter content was reduced to about 32% when its FSI was changed to 0. The volatile matter contents of the partially oxidized Pittsburgh seam, Ireland mine coals (Table 60) were reduced from 39.6% to about 34% or 35% as their FSI diminished from 7 to 1 or 0. Using similar coals at an atmospheric pressure, fixed-bed pretreater, Gasior *et al.*¹², found that the volatile matter content of the Illinois No. 6 and the Pittsburgh seam coals have to be reduced from 35.5% to about 22.5% to change the FSI to 1.5. Note that even though the coal-water partial oxidation process can produce a material that meets the nonagglomeration requirements (i.e., FSI ≤ 1.5) determined by Gasior *et al.*¹², it did not result in a pretreated coal that would be free-flowing in IGT's more severe agglomeration test.¹⁴

The severity of the IGT agglomeration test could be reduced if solids were fed to a 30-inch-diameter reactor in the HYGAS pilot plant with unheated walls. Because even fixed-bed gasifiers can operate successfully with a coal that has an FSI less than 2, Figures 85 and 86 indicate that the coal-water partial oxidation process can produce a coal with a FSI less than 1.5 while maintaining the volatile matter content of high-volatile bituminous coals at about 32% to 35%.

Note in Figures 87 and 88 that a 15 to 25-minute reaction time is required to reduce the FSI of high-volatile bituminous coals from about 7 to 1 by the coal-water partial oxidation process in the experimental setup used for this study. Experiments conducted for longer than 25 or 30 minutes, as shown in Tables 59 and 60, were attempted in a deliberate effort to over-pretreat the coal samples to obtain a free-flowing sample using the IGT test.

Table 58. SUMMARY OF EXPERIMENTAL DATA FROM THE PARTIAL OXIDATION
OF ILLINOIS NO. 6 hvCb COAL (Source: Unknown)

<u>Run No.</u>	<u>13</u>	<u>12</u>	<u>17</u>	<u>19</u>
Oxidizing Medium	Air	Air	Air	Air
Oxidizing Medium Flow Rate, CF/min*	0.5	0.26	0.46	0.48
Temperature, °F	550	520	450	500-560
Pressure, psig	1500	1000	1000	1500
Reaction Time, min	11	14	16	20
Analysis of Treated Coal (Dry Basis), wt %				
Volatile Matter	36.79	36.71	36.49	35.36
Ash	8.19	8.33	8.22	8.61
Fixed Carbon	55.02	54.96	55.29	56.03
C	71.8	71.0	70.5	70.7
H	4.57	4.44	4.52	4.41
Petrographic Analysis, %				
Nonagglomerating Particles	22.4	16.8	55.7	65.8
Devolatilized Particles	32.3	43.7	1.8	8.0
Unreacted Particles	45.3	39.5	42.5	26.2
IGT Agglomeration Test	Caked	Caked	Caked	Caked
Free Swelling Index	1	1	1	1

* At 70°F and 1 atm.

Table 59. SUMMARY OF EXPERIMENTAL DATA FROM THE PARTIAL OXIDATION
OF ILLINOIS NO. 6 hvBb COAL (Sahara Mine, Saline County)

Run No.	40	33	36	32	29	52	30	45	51	37
Oxidizing Medium	Air	Oxygen	Oxygen	Air	Air	Oxygen	Oxygen	Oxygen	Oxygen	Oxygen
Oxidizing Medium Flow Rate, CF/min*	0.25	0.4	0.4	0.4	0.4	0.3	0.25	0.17	0.24	0.25
Temperature, °F	520	500-540	540-560	500-510	500-515	520	500-530	530	530	520-560
Pressure, psig	1000	1000	1250	1000	1000	1000	1000	1000	1000	1250
Reaction Time, min	6	9-10	10-11	11	12	12	14	16	16	16-17
Analysis of Treated Coal (Dry Basis), wt %										
Volatile Matter	34.24	35.52	28.02	34.73	35.45	36.65	35.29	36.0	35.79	35.94
Ash	8.71	8.8	12.2	8.25	7.68	9.00	8.23	8.21	8.61	9.69
Fixed Carbon	57.05	55.68	59.78	57.02	56.87	54.35	56.48	55.79	55.60	54.37
C	72.8	70.7	70.2	73.0	73.8	70.9	71.3	71.7	70.9	69.3
H	4.67	4.58	3.76	4.89	4.91	4.56	4.53	4.47	4.58	4.25
Petrographic Analysis, %										
Nonagglomerating Particles	0.0	66.3	90.4	75.4	79.3	89.6	68.7	73.6	71.5	69.2
Devolatilized Particles	31.1	1.0	5.6	10.3	8.7	0.0	5.9	3.7	6.4	13.5
Unreacted Particles	68.9	32.7	4.0	14.3	12.0	10.4	25.4	22.7	22.1	17.3
IGT Agglomeration Test	Fused	Caked	Caked	Fused	Fused	Caked	Caked	Caked	Caked	Fused
Free Swelling Index	1	2.5	0.5	3	3	3.5	2	3	3.5	2.5

* At 70°F and 1 atm,

B75050993a

Table 59. Cont. SUMMARY OF EXPERIMENTAL DATA FROM THE PARTIAL OXIDATION
OF ILLINOIS NO. 6 hvBb COAL (Sahara Mine, Saline County)

Run No.	50	48	34	43	44	47	42	49	53	54
Oxidizing Medium	Oxygen	Oxygen	Air	Air	Oxygen	Oxygen	Air	Oxygen	Oxygen	Oxygen
Oxidizing Medium Flow Rate, CF/min*	0.2	0.1	0.4	0.2	0.17	0.1	0.2	0.123	0.24	0.24
Temperature, °F	525-530	530	500-530	515-520	530	530	525	525-530	530	530
Pressure, psig	1000	1000	1250	1000	1000	1000	1000	1000	1000	1000
Reaction Time, min	24	25	26	30	30	30	31	50	60	60
Analysis of Treated Coal (Dry Basis), wt %										
Volatile Matter	36.36	36.0	35.61	35.23	35.13	35.18	33.91	35.15	32.9	31.74
Ash	8.71	8.41	8.57	8.43	10.76	8.8	8.22	11.89	16.7	20.69
Fixed Carbon	54.93	55.59	55.82	56.34	54.11	56.02	57.87	52.96	50.4	47.57
C	70.4	70.7	71.4	71.6	68.2	71.7	71.2	68.2	63.8	60.7
H	4.5	4.25	4.62	4.59	4.14	4.39	4.28	4.45	4.02	3.87
Petrographic Analysis, %										
Nonagglomerating Particles	45.3	53.8	83.0	84.7	61.7	98.3	49.2	82.7	77.6	93.9
Devolatilized Particles	14.7	12.4	4.3	4.7	13.7	0.0	14.8	4.3	8.5	0.0
Unreacted Particles	40.0	33.8	12.7	10.6	24.6	1.7	36.0	13.0	13.9	6.1
IGT Agglomeration Test	Caked	Caked	Fused	Caked	Caked	Caked	Caked	Caked	Partially caked	Partially caked
Free Swelling Index	2	1	3	1.5	1	1.5	1	2	0	0

* At 70°F and 1 atmosphere.

B75050993b

Table 60. SUMMARY OF EXPERIMENTAL DATA FROM THE PARTIAL OXIDATION
OF PITTSBURGH SEAM NO. 8 hvAb COAL (Ireland Mine, Marshall County, W. Va.)

Run No.	27	28	6	21	15	9	10	18	55	56
Oxidizing Medium	Air	Oxygen	Oxygen	Oxygen	Air	Air	Air	Air	Oxygen	Oxygen
Oxidizing Medium Flow Rate, CF/min*	0.4	0.25	0.3	0.4	0.5	0.3	0.3	0.36	0.25	0.25
Temperature, °F	500-510	500-525	450	500	500	470	500	530-560	540	540
Pressure, psig	1000	1000	1000	1000	1000	1000	1000	1500	1000	1000
Reaction Time, min	8	12	15	17	18.5	20	20	20	30	60
Analysis of Treated Coal (Dry Basis), wt %										
Volatile Matter	35.44	35.26	34.71	35.77	35.13	37.69	34.42	34.53	34.97	35.68
Ash	8.25	8.25	7.54	8.76	8.65	10.95	10.11	8.4	18.49	24.12
Fixed Carbon	56.31	56.49	57.75	55.47	56.22	51.36	55.47	57.07	46.54	40.2
C	72.6	70.10	72.7	69.2	71.7	71.4	70.5	71.6	59.7	52.8
H	4.73	4.24	4.49	4.07	4.48	4.83	4.39	4.41	3.52	3.1
Petrographic Analysis, %										
Nonagglomerating Particles	42.2	93.0	†	40.4	2.1	†	†	74.2	92.4	97.2
Devolatilized Particles	12.0	4.7	†	8.3	30.9	†	†	12.3	5.2	1.2
Unreacted Particles	45.8	2.3	†	51.3	64.3	†	†	13.5	2.4	1.6
IGT Agglomeration Test	Fused	Caked	Fused	Caked	Fused	Fused	Fused	Fused	Partially caked	Partially caked
Free Swelling Index	5	2.5	1	1	1	6.5	1	1	0	0.5

* At 70°F at 1 atmosphere.

† Not available.

B75050994

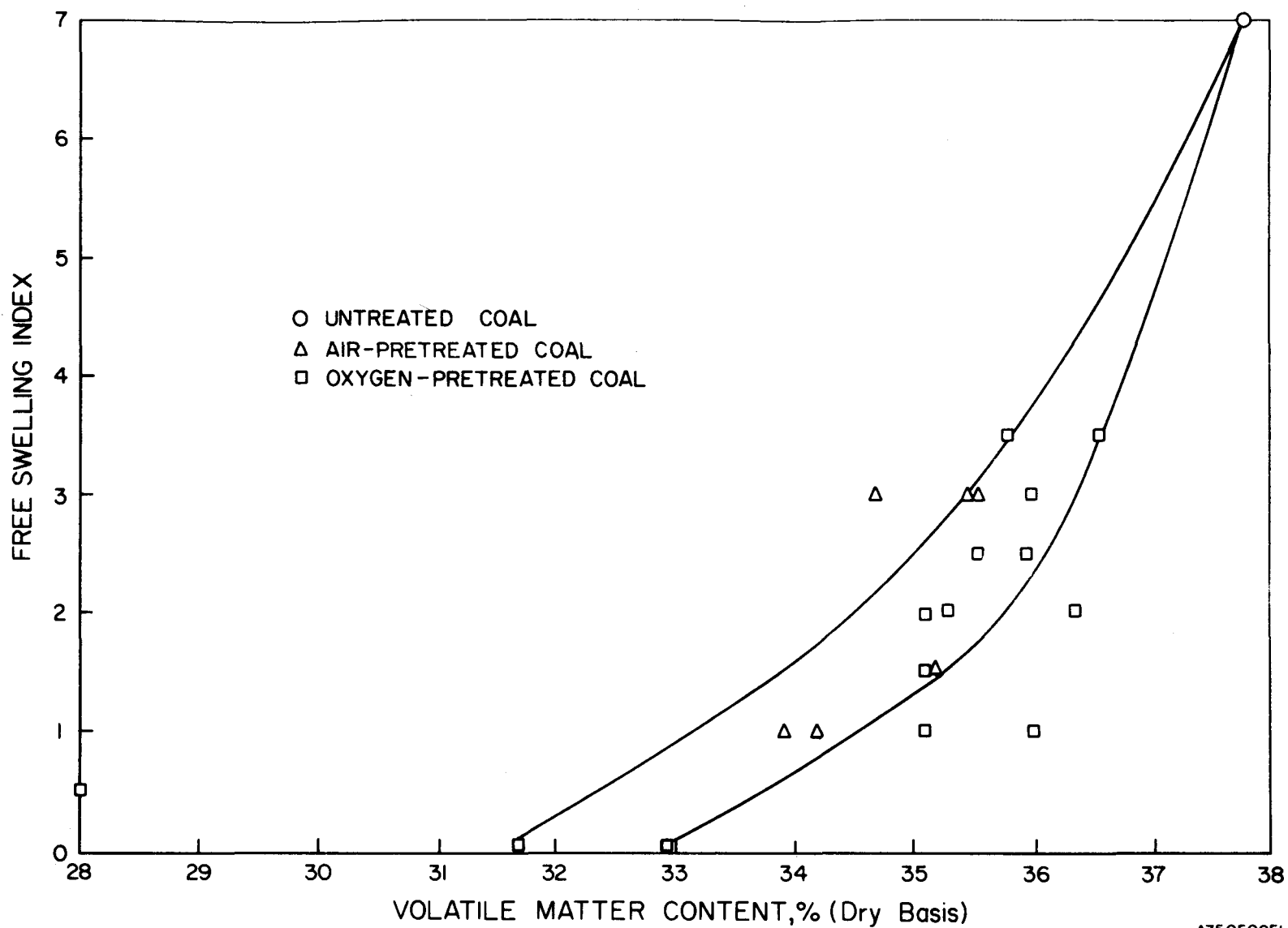
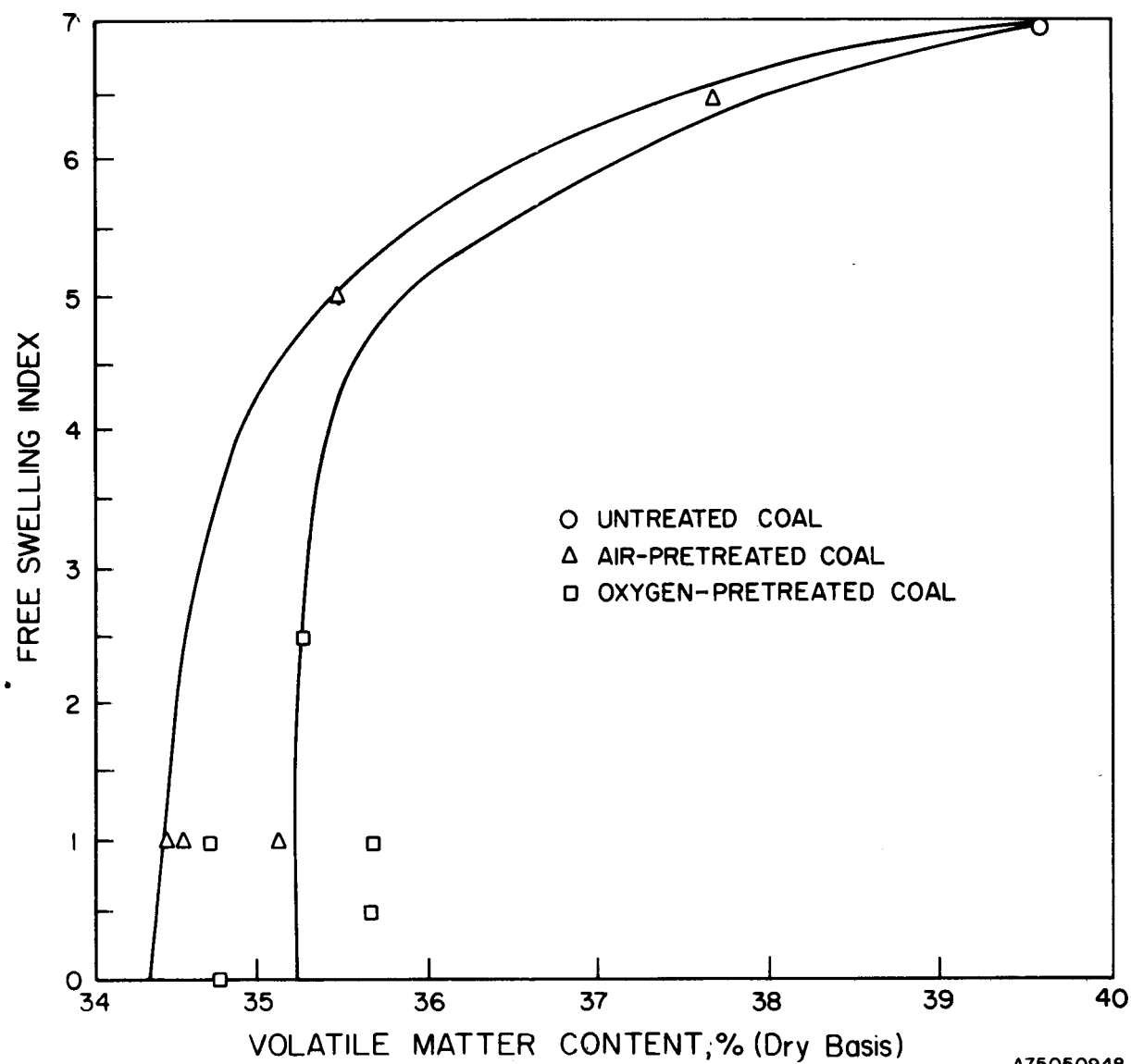


Figure 85. RELATIONSHIP BETWEEN THE FREE SWELLING INDEX AND THE VOLATILE MATTER CONTENT OF ILLINOIS NO. 6 hvBb COAL

A75050951



A75050948

Figure 86. RELATIONSHIP BETWEEN THE FREE SWELLING INDEX AND THE VOLATILE MATTER CONTENT OF PITTSBURGH SEAM, IRELAND MINE hvAb COAL

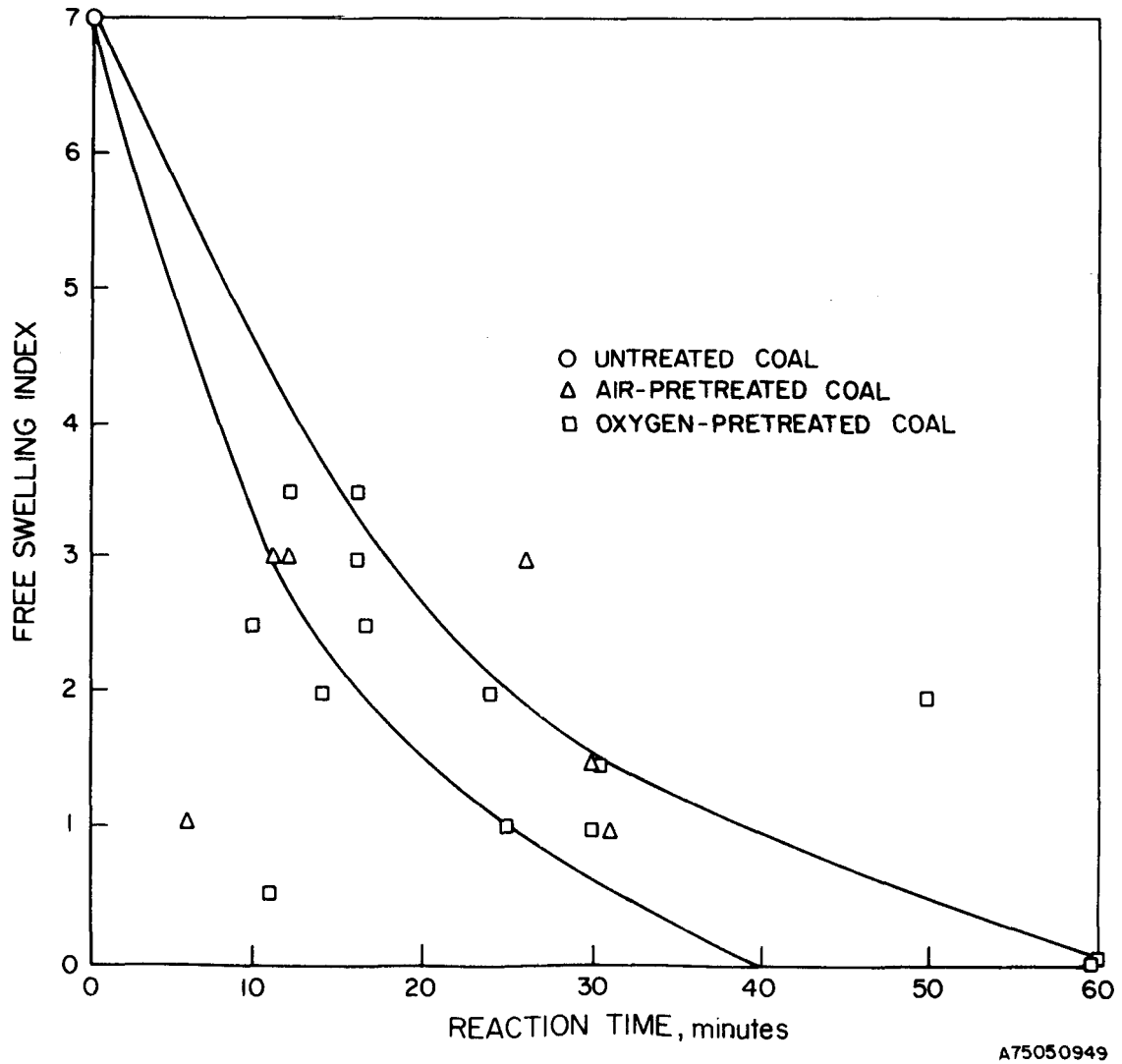
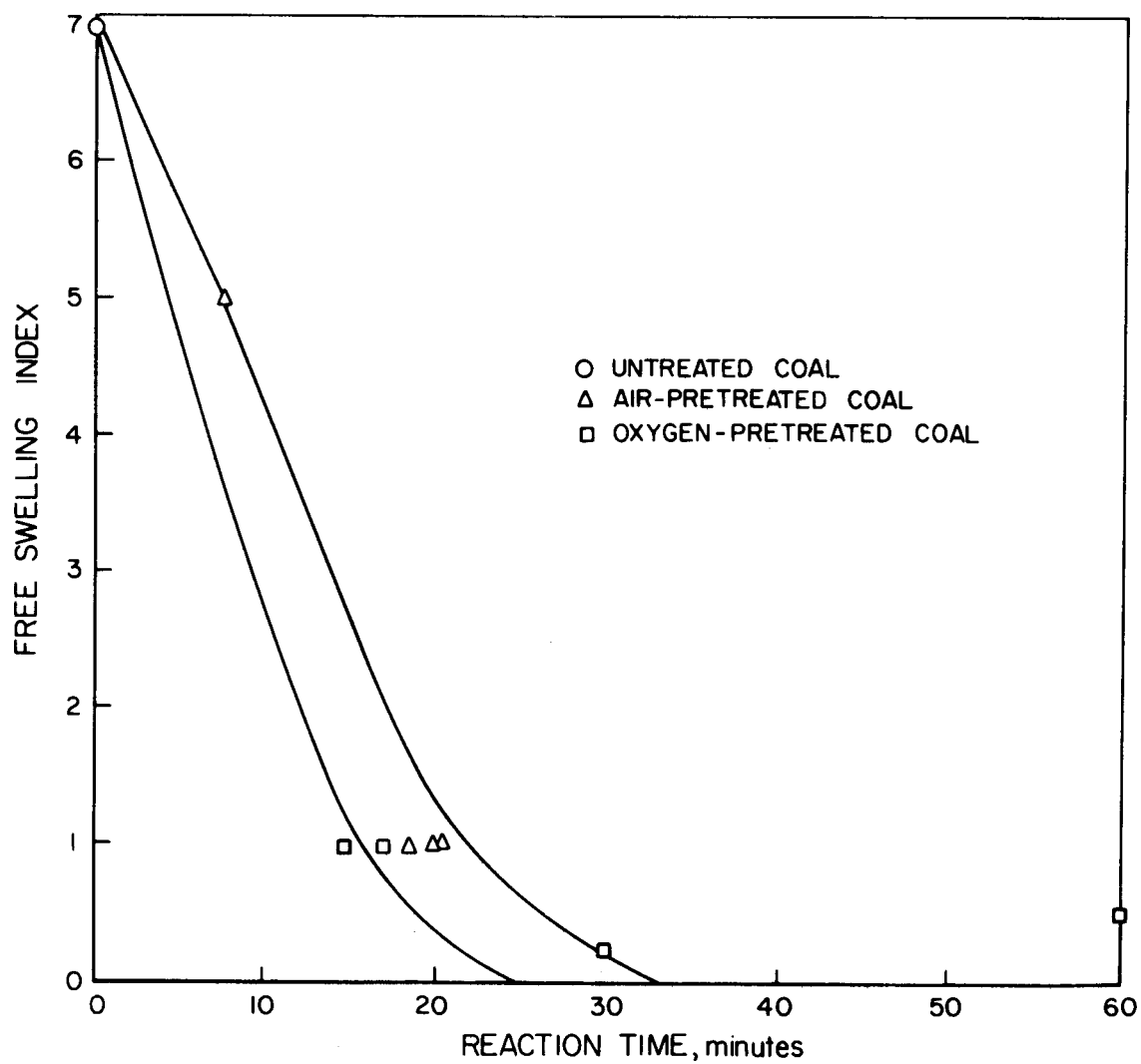


Figure 87. RELATIONSHIP BETWEEN THE FREE SWELLING INDEX AND THE REACTION TIME WITH THE OXIDIZING MEDIUM FOR ILLINOIS No. 6 hvBb COAL



A75050950

Figure 88. RELATIONSHIP BETWEEN THE FREE SWELLING INDEX AND THE REACTION TIME WITH THE OXIDIZING MEDIUM FOR THE PITTSBURGH SEAM, IRELAND MINE hvAb COAL

Tables 58 through 60 show that the treated coals in the coal-water partial oxidation process contain more total carbon and hydrogen than the pretreated samples of Gasior *et al.*,¹² Forney *et al.*,¹² and Kavlick and Lee.¹⁴ This shows that the material produced in the coal-water partial oxidation process has a potential for greater methane yields when compared with coal pretreated by the other methods.

Compared with the atmospheric pressure pretreatment processes, coal-water partial oxidation has resulted in a char with little or no visible swelling. This is attributed to the operating pressure (1000 to 1500 psig) of coal-water pretreatment and is considered an advantage when slurry feeding coal into a high-pressure reactor.

The petrographic analyses of the pretreated coals reported in Tables 58 through 60 show that good surface oxidation could be carried out in the coal-water partial oxidation process. This is indicated by the fact that more than 80% of the treated particles have a reflectant border in experimental Runs 28, 29, 34, 36, 43, 47, 49, 52, 54, 55, and 56. When the method of conducting the partial oxidation process was improved, the amount of undesirable devolatilized particles was reduced, while the amount of particles with reflectant borders, indicative of good surface oxidation of the coal, was increased.

The material balances determined for some of the partial oxidation experiments together with the oxygen consumption rates are shown in Table 61. The agreement between the measured weight loss and the loss in weight calculated from the ash balance is obviously very poor for most of the runs, particularly for reaction times of 30 minutes or less. At this time, no particular reason can be given for this discrepancy. Possibly the dissolution of coal's mineral matter in water under the partial oxidation experimental conditions could be one reason. As a result, the filtered slurry at the end of the pretreatment experiment was analyzed for minor and trace elements. The analytical results in Table 62 show that iron is the only mineral that appears to dissolve, in very small quantities, in the partial oxidation process. The enrichment of the residual water from the pretreatment process with nickel, copper, and zinc is attributed to the possible dissolution effect of water at 1000 psig and 540°F on the reactor assembly and its associated tubing. Therefore, the discrepancy in arriving at an acceptable material balance is either the result of improper sampling of the untreated and treated coals or of incomplete recovery or loss of the treated samples. Some of these experiments will be repeated to correct these inadequacies and to estimate, with reproducibility, the loss of coal on partial oxidation. Residual water from the coal-water partial oxidation experiments is acidified by the formation of weak carboxylic acids from coal known as "humic acids."

In reviewing the material balances shown in Table 61, a matter of concern is the inability of the coal-water partial oxidation process to convert a bituminous coal into a free-flowing sample, as determined by the IGT agglomeration test, in spite of losing nearly 60% of the coal. The reflectant borders produced in this investigation were also quite comparable with the oxidized film produced in the atmospheric pretreatment experiments conducted by Kavlick and Lee.¹⁴ The major difference between the coal-

Table 61. WEIGHT LOSS AND RATE OF OXYGEN CONSUMPTION
DURING PARTIAL OXIDATION

Run No.	Reaction Time, min	Oxygen Flow Rate,* CF/min	Rate of Oxygen Consumption,* CF/min-lb of coal	Weight Loss			IGT Agglomer-ation Test	FSI
				Measured	Calculated From Oxygen Consumption %	Calculated From HSL Balance		
Illinois No. 6 (hvBb) Coal								
42	31	0.042	--	27.54	--	--	Caked	1
43	30	0.042	--	25.10	--	1.07	Caked	1.5
44	30	0.17	--	--	--	--	Caked	1
45	16	0.17	--	13.65	--	--	Caked	3
47	30	0.1	--	16.39	--	5.23	Caked	1.5
48	25	0.1	--	9.59	--	0.84	Caked	1
49	50	0.123	--	43.58	--	29.86	Caked	2
50	24	0.2	0.2	13.07	17.85	4.25	Caked	2
51	16	0.24	--	15.72	--	3.13	Caked	3.5
52	12	0.3	0.27	21.01	14.94	7.33	Caked	3.5
53	60	0.24	0.25	63.98	56.88	50.06	Partially caked	0
54	60	0.24	0.26	63.66	59.73	59.69	Partially caked	0
Pittsburgh Seam No. 8 Ireland Mine (hvAb) Coal								
55	30	0.25	--	16.07	--	42.89	Partially caked	0
56	60	0.25	0.18	41.05	40.10	56.22	Partially caked	0.5

* At 70°F and 1 atm.

water partial oxidation method and the atmospheric pretreatment process described above is the temperature at which the coal is contacted with the oxidizing medium. In the latter processes, the reaction temperatures ranged from 750°F to 800°F, and in the present investigation the maximum temperature was only 560°F. To determine whether the reaction temperature is critical and also to understand the mechanism of agglomeration, pretreated samples will be studied under a heating stage microscope at temperatures approaching 1400°F.

Table 62. MINERAL CONSTITUENTS IN ILLINOIS NO. 6 COAL AND THE RESULTING WATER SAMPLE FROM THE COAL-WATER PARTIAL OXIDATION PROCESS

Component	Analysis			
	Illinois No. 6 Coal	Residual Water From Run 44	Residual Water From Run 45	Reference Water Sample
		ppm		
Li	33.0	<0.01	<0.01	<0.01
Na	1,400.0	6.56	4.60	5.30
K	1,700.0	1.33	0.52	0.96
Mg	570.0	3.79	1.02	11.40
Ca	3,500.0	3.58	3.10	35.50
Cr	15.0	ND*	ND	<0.05
Mn	48.0	0.95	0.98	<0.01
Fe	14,000.0	16.20	9.09	<0.02
Ni	15.0	0.73	0.74	<0.02
Cu	19.0	1,250.00	361.0	0.20
Zn	49.0	29.2	8.37	<0.05
Al	--	ND	ND	<0.50
Si	20,000.0	1.0	1.0	<0.50
pH	--	4	4	7

* ND = no data.

During the partial oxidation experiments, the only gas analysis carried out was the determination of oxygen content in the reactor off-gases to estimate oxygen breakthrough. Because very little coal gasification occurs at operating temperatures of about 540°F, no further analysis was conducted. However, a complete analysis of the coal-water partial oxidation off-gases will be made when some of the experiments will be repeated to check the material balances.

The estimated oxygen consumption rates for partial oxidation, shown in Table 61, are greater than the oxygen requirements reported by Gasior et al.,¹² Kavlick and Lee,¹⁴ and Forney et al.¹⁰ A direct comparison of oxygen requirements is not reasonable because of the difference in the scale on which these experiments were conducted. For a proper comparison, larger scale, continuous coal-water partial oxidation experiments must be conducted.

Tests to Determine Agglomeration Tendency

The free swelling index and the IGT agglomeration test¹⁴ were selected from the several available methods for characterizing the agglomerating properties of treated and untreated coals.^{3,16} A brief description of these methods follows.

- a. Free Swelling Index (FSI): To determine the FSI, 1 gram of a -60 mesh coal sample is placed in a translucent silica crucible and heated at a specified rate in a specially designed gas burner assembly.¹

After heating the sample to 800°C, the residue is cooled, and the projected cross section is compared with standard profiles (Figure 89) to assign a FSI. For coke residues that do not conform to the standard profiles, the FSI is determined from the relationship between the projected area and the FSI shown in Figure 90.

If the residue is not swollen and can support a 500-gram weight, or simply cracks into two or three hard, coherent pieces, an FSI of 1 is reported. An FSI of 0.5 is assigned if the residue disintegrates under the load of the 500-gram weight. The residue is considered to have an FSI of 0 if it is totally noncoherent.

- b. IGT Agglomeration Test: This test is a modification of the method developed by Forney et al.¹⁰ A sample of the test material is placed in a stainless-steel wire-mesh boat and is heated in a quartz tube in the presence of flowing nitrogen by an electric furnace. At 1400°F, the gas flow is switched to hydrogen, and the heating at this temperature is continued for 30 minutes, before cooling down the sample with nitrogen. The residue in the boat is then inspected for its agglomerating tendency and classified in one of the following six categories:
 1. Free-Flowing (FF) — The residual sample is noncoherent and flows easily when the boat is tilted.
 2. Very Lightly Caked (VLC) — A few small agglomerated particles are scattered through less than 25% of the sample. The agglomerates break up easily under slight pressure, or when tumbled.
 3. Lightly Caked (LC) — About one-half of the sample in the boat consists of moderate-sized agglomerates that do not break up easily under pressure.
 4. Partially Caked (PC) — More than 75%, but less than all of the sample consists of moderate- or large-sized agglomerates that do not disintegrate under pressure.

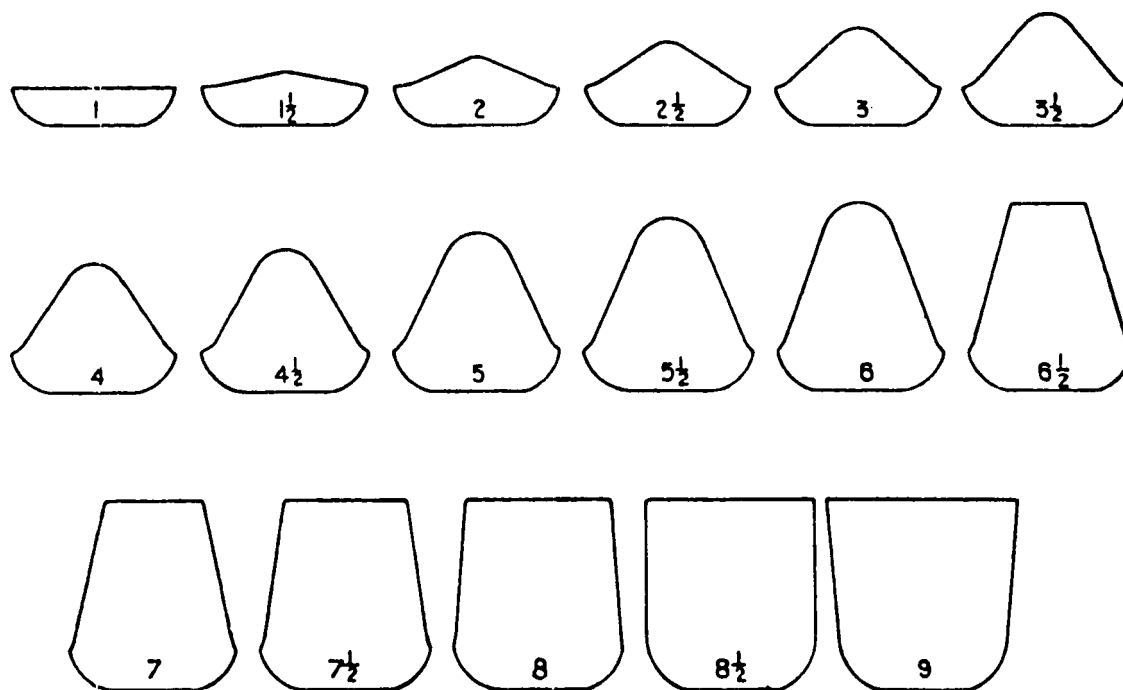


Figure 89. STANDARD PROFILES FOR DETERMINING THE FREE SWELLING INDEX¹⁵

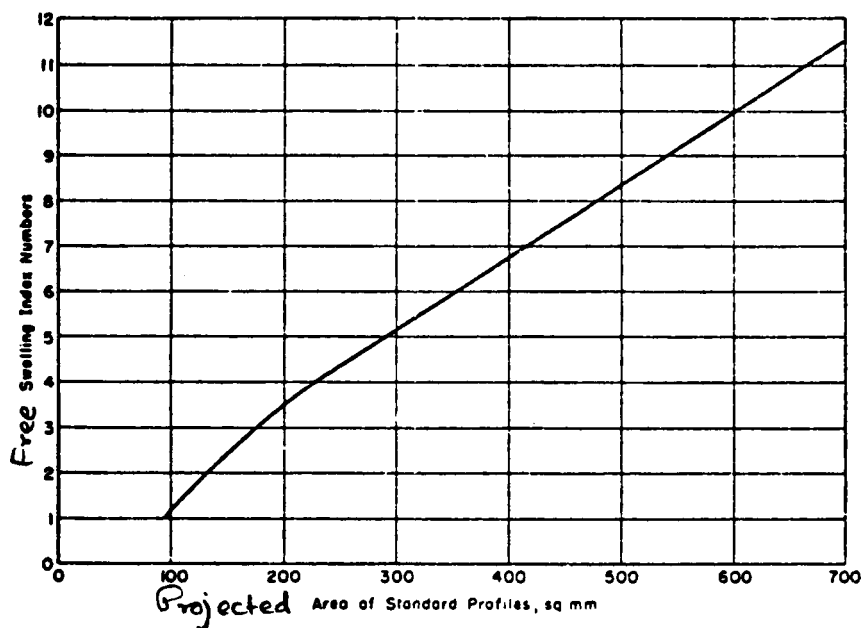


Figure 90. RELATIONSHIP BETWEEN THE PROJECTED AREA OF THE SWELLING INDEX RESIDUE AND THE FSI¹⁵

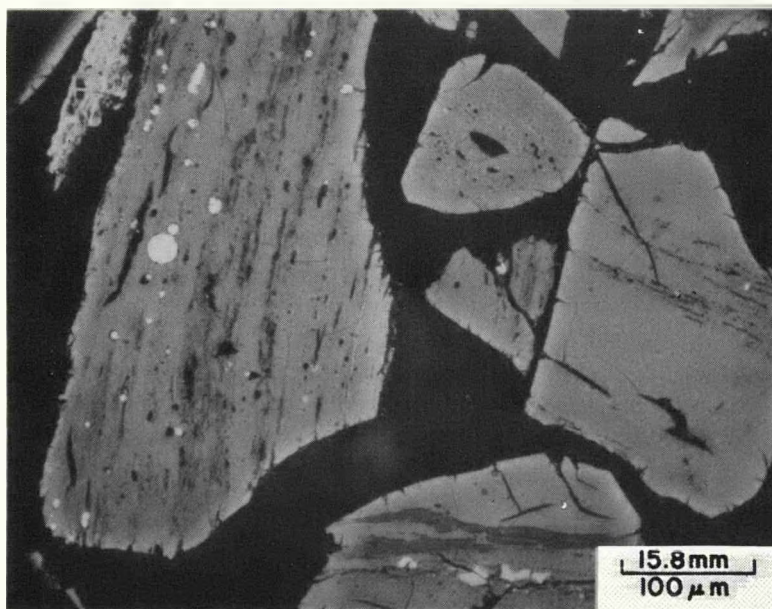
5. Caked — The residue is coherent, hard, either swollen or unswollen, and the individual particles are discretely visible.
6. Fused — The residue in the boat is a fused, swollen, shiny, and hard homogeneous mass, with no discernible particles.

Further insight into the mechanism of pretreatment was obtained by analyzing the photomicrographs from petrographic studies and by observing the behavior of treated and untreated coal particles in a heating-stage microscope. Controlled surface oxidation in a slurry at 540°F appears to be milder than the atmospheric pressure fluidized-bed pretreatment oxidation. Slurry oxidation produces a char that could be acceptable as a hydrogasifier feed; however, such pretreatment does not produce a distinctly bright and hard crust on the particles that positively prevents agglomeration in a hydrogasifier.

Coal-water partial oxidation experiments, done in March show that the high-volatile bituminous coals were successfully pretreated to reduce their free swelling index (FSI) from about 7 to 1. However, the pretreated samples did not result in a free-flowing sample in the IGT agglomeration test. Further insight into the mechanism of pretreatment can be obtained by analyzing the photomicrographs from the petrographic studies and by observing the behavior of treated and untreated coal particles in a heating-stage microscope.

The photomicrographs of some of the pretreated coal particles from the coal-water partial oxidation experiments are shown in Figures 91 through 94. Similar photomicrographs of free-flowing high-volatile bituminous coals, pretreated in a 4-inch fluidized bed at atmospheric pressure and about 780°F, are shown in Figures 95 and 96. Apparently the pretreated samples from the coal-water partial oxidation show few or no pores compared with the atmospheric-pressure fluidized-bed pretreated samples. However, the reflectant borders of the particles shown in Figures 91 through 94 are diffuse and less bright when compared with the oxidized borders of the pretreated samples shown in Figures 95 and 96. The fine cracks visible in the reflectant borders of the coal-water partial oxidation product are absent in the fluidized-bed pretreated samples.

These observations led to the conclusion that the controlled surface oxidation achieved at about 540°F in the coal-water partial oxidation experiments was milder than the fluidized-bed pretreatment oxidation. The slurry pretreatment product may be acceptable as a hydrogasifier feed; however, such pretreatment does not produce a distinctly bright and hard crust that positively prevents agglomeration when the samples are heated to higher temperatures under hydrogasification conditions. It is worth noting that the extent of required pretreatment for a commercial hydrogasification process has not yet been determined. The requirements to produce a free-flowing sample in the IGT agglomeration test are based on experience feeding coal through a 1-inch-diameter tube to a 4-inch-diameter fluidized-bed hydrogasifier. Commercial coal gasifiers are expected to successfully handle samples with an FSI equal to 1 or samples that become partially caked in the IGT agglomeration test. (See March 1975, ERDA Report No. 126.)



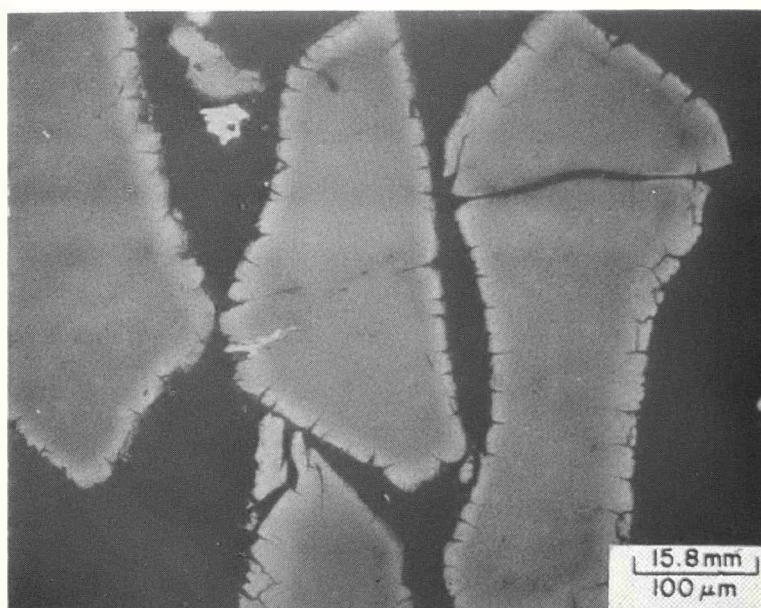
P75061196

Figure 91. PHOTOMICROGRAPH OF ILLINOIS NO. 6 hvBb COAL
SUBJECTED TO COAL-WATER PARTIAL OXIDATION (Test 43)



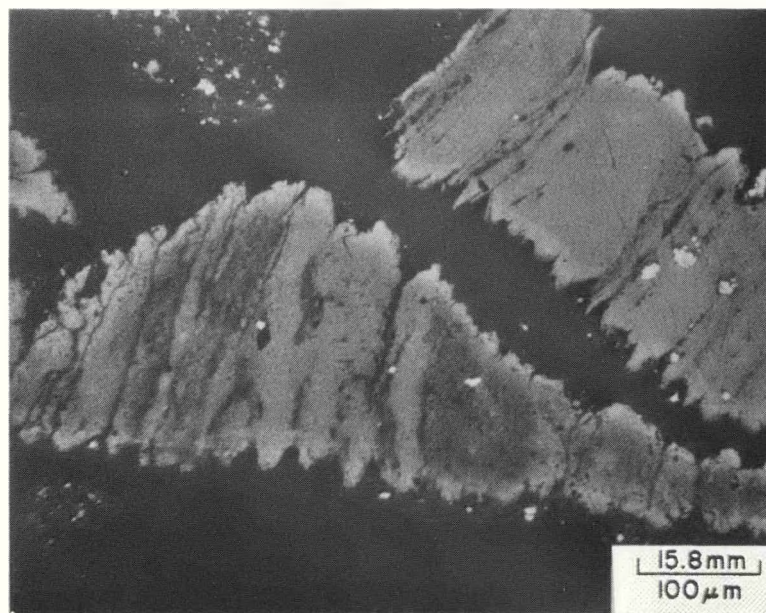
P75061197

Figure 92. PHOTOMICROGRAPH OF ILLINOIS NO. 6 hvBb COAL
SUBJECTED TO COAL-WATER PARTIAL OXIDATION (Test 47)



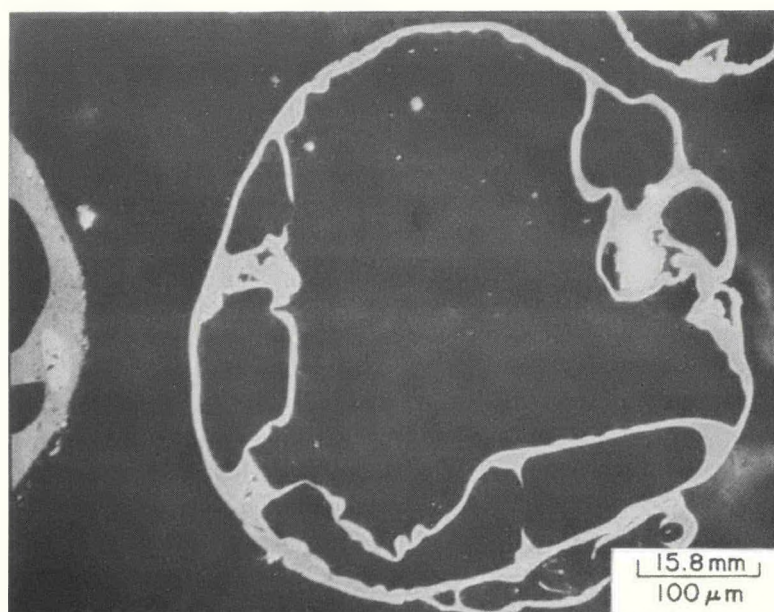
P75061198

Figure 93. PHOTOMICROGRAPH OF ILLINOIS NO. 6 hvBb COAL
SUBJECTED TO COAL-WATER PARTIAL OXIDATION (Test 52)



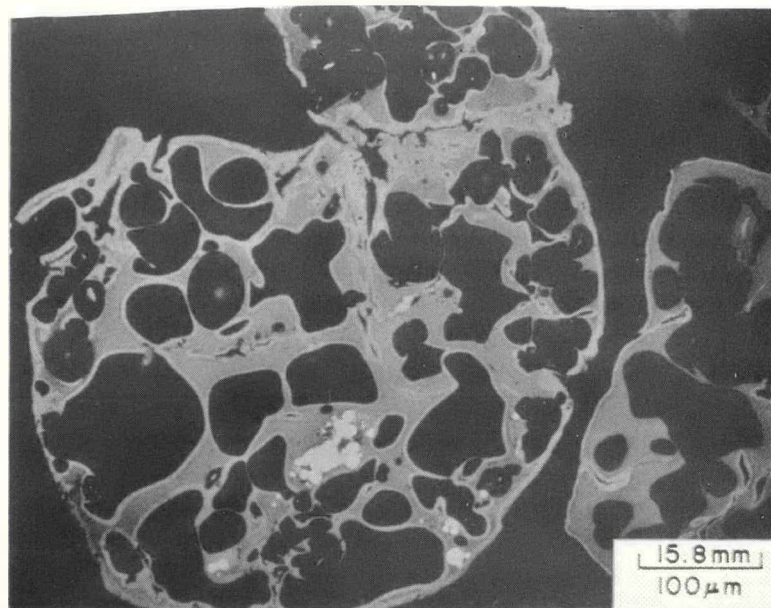
P75061199

Figure 94. PHOTOMICROGRAPH OF ILLINOIS NO. 6 hvBb COAL
SUBJECTED TO COAL-WATER PARTIAL OXIDATION (Test 54)



P75061200

Figure 95. PHOTOMICROGRAPH OF PITTSBURGH SEAM, IRELAND MINE,
hvAb COAL SUBJECTED TO FLUIDIZED-BED PRETREATMENT
AT 1 ATMOSPHERE AND 780°F (Test FP-74)



P75061201

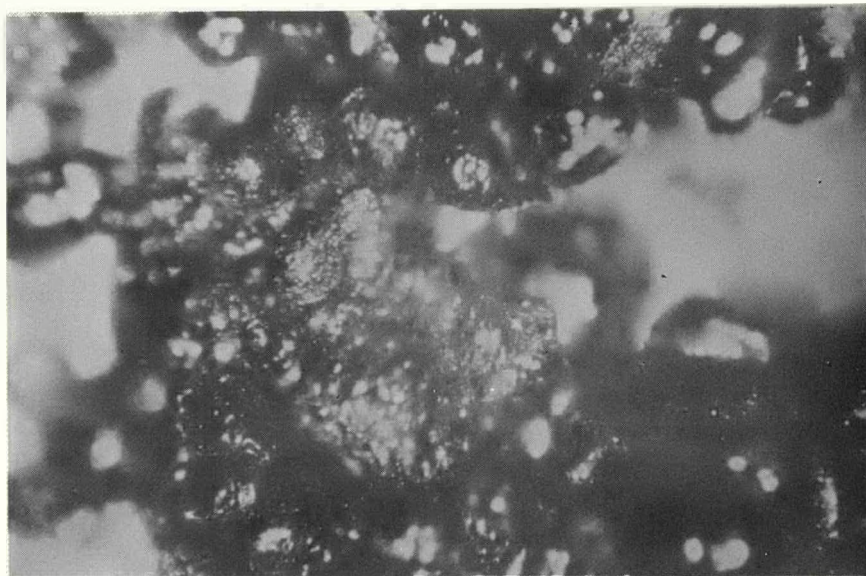
Figure 96. PHOTOMICROGRAPH OF ILLINOIS NO. 6 hvBb COAL
SUBJECTED TO FLUIDIZED-BED PRETREATMENT AT 1 ATMOSPHERE
AND 780°F (Test FP-105)

To further examine the pretreatment mechanism, the behavior of untreated and pretreated coals from the fluidized-bed experiments and coal-water partial oxidation studies, was observed with a Leitz, 1000°C, heating-stage microscope at the Illinois Geological Survey, Urbana, Illinois. A few grains of the coal sample were placed on a 3/16-inch quartz disk, which, in turn, was enclosed in a stainless-steel heating stage provided with a glass window. The first set of experiments was conducted while maintaining a nitrogen flow through the heating stage, as the quartz disk containing the sample was being heated by an electric heater underneath the sample. The second and final set of experiments consisted of observing the behavior of coal particles heated in the presence of hydrogen. A heating rate of about 250°F to 280°F/min was attained with nitrogen, while the presence of hydrogen reduced the heating rate to 200°F/min for the same power input.

The untreated high-volatile B and C coals start evolving gases and aromatic liquids at 450°F and 550°F, respectively. Substantial agitation of particles, combined with the distillation of liquid and gaseous hydrocarbons, continues until about 900°F, after which the agglomerated particles do not show further movement on the disk. When the flow through the heating stage was switched to hydrogen, even though the heating rate dropped to 200°F/min, the gases and liquids distilled at the same temperatures that were observed with nitrogen. The residue of the high-volatile B, Illinois No. 6 coal, heated in the presence of hydrogen, is shown in Figure 97.

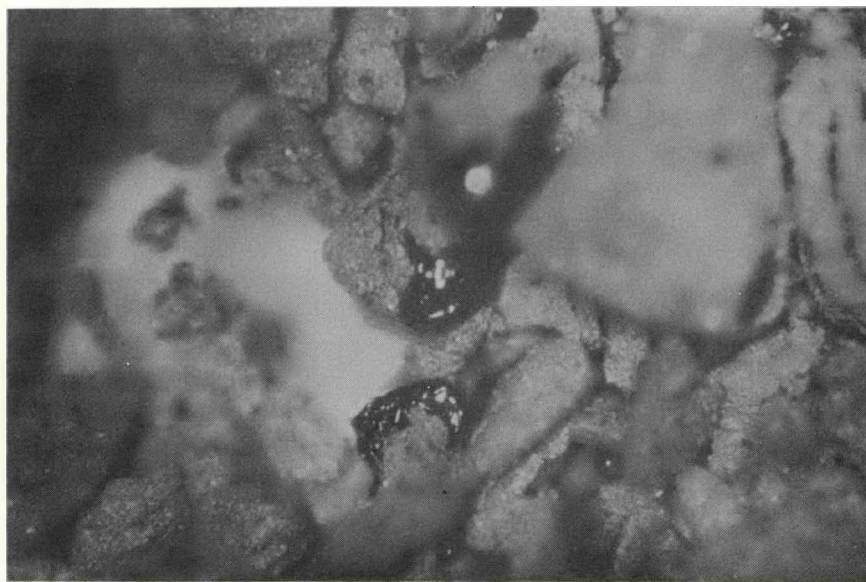
When heated in the presence of nitrogen and hydrogen, the pretreated coal samples from the coal-water partial oxidation experiments behaved slightly differently than untreated coals. When the particles were heated to about 510°F, layers of oxidized coal disintegrated, followed by the liberation of a liquid matter from a few cracks up to a temperature of about 900°F. The residues of these experiments are shown in Figures 98, 99a, and 99b.

The behavior of the free-flowing samples, from the 4-inch fluidized-bed pretreatment experiments, was very different in the heating-stage microscope when compared with that of untreated and pretreated coals. The free-flowing samples, when heated in the presence of either nitrogen or hydrogen, did not release any liquid substance or show signs of agglomeration, even when the temperature was raised to about 1500°F. The usual minor shifting and settling of particles, attributed to the release of gases during heating, were the only phenomena observed. The liberated and solidified liquid material, seen in Figures 98, 99a, and 99b could conceivably be responsible for the agglomeration of individual particles in the IGT agglomeration test. Even though the amount of liquid evolved from the coal-water partial oxidation pretreated coals is very small in comparison with untreated coals, their general behavior in the heating-stage microscope indicates that further pretreatment of the coal is required if the gasification process demands a free-flowing feed. The results reported in the March 1975, ERDA Report No. 126 show that a) partial oxidation beyond 30 minutes does not show any improvement in reducing the agglomerating tendency and b) possibly temperatures in excess of 540°F may be required to obtain a free-flowing sample. However, with a coal-water system, the operating pressure has to be increased to about 300 psig to reach a possible maximum temperature of 700°F.



P75061204

Figure 97. PHOTOMICROGRAPH OF THE RESIDUE OF ILLINOIS NO. 6 hvBb COAL FROM THE HEATING-STAGE MICROSCOPE TEST



P75061218

Figure 98. PHOTOMICROGRAPH OF THE RESIDUE OF PRETREATED ILLINOIS NO. 6 hvBb COAL FROM THF HEATING-STAGE MICROSCOPE TEST (Pretreated Sample from Test 54)



(a)

P75061202



(b)

P75061203

Figure 99. PHOTOMICROGRAPH OF THE RESIDUE OF PRETREATED
PITTSBURGH SEAM, IRELAND MINE, hvAb COAL FROM THE
HEATING-STAGE MICROSCOPE TEST (Pretreated Sample from Test 56)

In conclusion, because most of the hydrogasification processes are being developed to operate at about 1000 psig, it is desirable to devise a scheme to deagglomerate coals at pressures no higher than 1000 psig with a minimum loss of the original volatile matter. Oxidation in a water slurry is limited to a temperature of 540°F and produces a pretreated coal with a low free swelling index. Commercial gasifiers are expected to be able to accept such pretreated coal as feed. However, if a completely free-flowing coal is necessary for the operation of the hydrogasifier — an unlikely requirement — temperatures higher than 540°F may be needed. In that case, pretreatment would not be done in a slurry, but in a gas-solid system, for example, with steam and oxygen or air at 1000 psig and about 750°F to 780°F. Such pretreatment under pressure was demonstrated at the Institute of Gas Technology,¹⁶ and is presently being developed for the SYNTHANE Process by the ERDA Pittsburgh Energy Research Center at Bruceton, Pennsylvania.⁹

Five experiments were conducted during May to check the reproducibility and material balances for the deagglomeration of coal in a slurry by partial oxidation. These experiments are synopsized in Table 63. It was learned that ERDA's Pittsburgh Energy Research Center has successfully pretreated the Eastern high-volatile bituminous coals at 70 atmospheres in a 1-inch-inside-diameter fluidized-bed reactor. The pretreatment was carried out by contacting coal with oxygen concentrations of about 1.0 SCF/lb of coal for about 3 minutes.

Table 63. SUMMARY OF EXPERIMENTS

<u>Run No.</u>	<u>Date</u>	<u>Operating Conditions</u>	<u>Remarks</u>
57	5/2/75	80 g Illinois No. 6 coal + 175 g water + 0.24 CF/min compressed oxygen at 1000 psig. Reaction temperature = 540°F. Reaction time = 30 minutes.	Treated coal totally submerged under water
58	5/6/75	Same as Run No. 57.	Treated coal totally submerged under water
59	5/7/75	80 g Illinois No. 6 coal + 175 g water + 0.24 CF/min compressed oxygen at 1000 psig. Reaction temperature = 540°F. Reaction time = 20 minutes.	Treated coal totally submerged under water
60	5/12/75	Same as Run No. 59.	Treated coal totally submerged under water.
61	5/13/75	80 g Illinois No. 6 coal + 175 g water + 0.24 CF/min compressed oxygen at 1000 psig. Reaction temperature = 540°F. Reaction time = 60 minutes.	Treated coal totally submerged under water

Mass-spectrometric analyses of gas samples collected during experimental Runs 57 through 61 are shown in Table 64.

Table 64. ANALYSES OF COAL-WATER PARTIAL OXIDATION
GAS SAMPLES

Run No.	Minutes After Introducing O ₂	Reported Gas Composition					
		N ₂	CO	O ₂	CO ₂	H ₂	Ar
		vol %					
57	5	72.6	0.2	25.6	1.4	0.1	0.1
	10	39.1	0.6	58.1	1.8	0.1	0.3
	15	9.8	0.4	86.3	3.2	0.1	0.2
	20	15.1	--	80.8	3.7	0.1	0.3
	25	1.3	--	93.1	5.3	0.1	0.2
	31	4.4	0.2	89.6	5.5	0.1	0.2
58	5	69.6	0.4	27.8	1.8	0.3	0.1
	10	36.1	0.4	61.0	2.0	0.1	0.4
	15	3.8	0.5	92.6	2.7	0.2	0.2
	20	7.4	0.7	89.2	2.4	0.1	0.2
	25	2.4	0.4	94.2	2.7	0.1	0.2
	32	0.4	0.3	96.2	2.8	0.1	0.2
59	5	66.0	0.2	32.7	1.0	--	0.1
	10	24.0	0.4	73.6	1.8	--	0.2
	15	6.9	0.3	89.8	2.8	--	0.2
	21	2.7	0.4	92.6	4.1	--	0.2
60	5	94.0	--	5.6	0.3	0.1	--
	10	32.3	0.3	65.5	1.8	--	0.1
	15	6.3	0.3	90.1	3.2	--	0.1
	21	1.4	0.4	94.1	3.9	--	0.2
61	10	21.8	0.2	74.2	3.6	0.1	0.1
	20	2.8	0.6	88.3	8.0	0.1	0.2
	30	3.1	0.5	89.6	6.6	--	0.2
	40	0.8	0.7	92.6	5.6	0.1	0.2
	50	1.4	0.7	92.7	4.9	0.1	0.2
	61	0.9	0.5	92.8	5.5	0.1	0.2

Analytical results of untreated and treated coal samples from Runs 57 to 61 are summarized in Table

Table 65. SUMMARY OF ANALYTICAL RESULTS

	Untreated Coal	Product				
		Run 57	Run 58	Run 59	Run 60	Run 61
		Illinois No. 6 Coal				
Proximate Analysis (Dry Basis), % by weight						
Volatile Matter	37.86	35.42	34.93	35.06	34.96	34.35
Ash	8.56	8.25	9.13	8.54	9.00	9.79
Fixed Carbon	54.58	56.33	55.94	56.40	56.04	55.86
Ultimate Analysis (Dry Basis), % by weight						
Carbon	74.5	73.20	73.10	72.70	72.90	70.90
Hydrogen	5.1	4.74	4.89	4.69	4.71	4.61
Nitrogen	1.55	1.57	1.47	1.54	1.57	1.56
Sulfur	2.75	2.50	2.54	2.53	2.55	2.40
Oxygen	7.54	9.74	8.87	10.00	9.27	10.74
Ash	8.56	8.25	9.13	8.54	9.00	9.79
Free Swelling Index	4	2	2	2	1	1
IGT Boat Test	Fused	Caked	Caked	Caked	Partially caked	
Reaction Time, min	--	30	30	20	20	60
Weight Loss, %	--	10.4	7.2	11.0	8.7	19.9
Petrographic Analysis, %						
Nonagglomerating Particles	--	31.0	18.2	23.7	23.7	--
Devolatilized Particles	--	60.7	53.0	62.0	57.0	--
Unreacted Particles	--	8.3	28.8	14.3	19.3	--

B75092308

Results analyzed in March 1975 showed a significant weight loss during the partial oxidation of coal. To check the material balances, five experiments were repeated and the results summarized in Table 66. An analysis of the gas samples collected during the partial oxidation experiments, and shown in Table 1 of the June 1975 Memorandum Report, indicates that CO₂ is the primary product gas.

The summary of experimental data in Table 66 shows that the free swelling index of Illinois No. 6 hvBb coal was reduced from 4 to 1 by reacting coal in a slurry with oxygen at 540°F and 1000 psig. The associated loss in weight, Table 67 ranges up to about 20% for 60 minutes of contact with the oxidizing medium. The agglomerating properties of coal can be reduced to a free swelling index of 2 by the consumption of about 11%, by

Table 66. SUMMARY OF EXPERIMENTAL DATA FROM THE PARTIAL OXIDATION
OF ILLINOIS NO. 6 hvBb COAL (Sahara Mine, Saline County)

Run No.	Untreated Coal	57	58	59	60	61
Oxidizing Medium	--	Oxygen	Oxygen	Oxygen	Oxygen	Oxygen
Oxidizing Medium Flow Rate, CF/min*	--	0.24	0.24	0.24	0.24	0.24
Temperature, °F	--	540	540	540	540	540
Pressure, psig	--	1000	1000	1000	1000	1000
Reaction Time, min	--	30	30	20	20	60
Analysis of Treated Coal (Dry Basis), wt %						
Volatile Matter	37.86	35.42	34.93	35.06	34.96	34.35
Ash	8.56	8.25	9.13	8.54	9.00	9.79
Fixed Carbon	54.58	56.33	55.94	56.40	56.04	55.86
C	74.50	73.20	73.10	72.70	72.90	70.90
H	5.10	4.74	4.89	4.69	4.71	4.61
N	1.55	1.57	1.47	1.54	1.57	1.56
S	2.75	2.50	2.54	2.53	2.55	2.40
O (by difference)	7.54	9.74	8.87	10.00	9.27	9.79
Increase in Oxygen, wt %	--	15.80	9.20	18.00	12.20	4.00
Petrographic Analysis, %						
Nonagglomerating Particles	--	31.00	18.20	23.70	23.70	40.80
Devolatilized Particles	--	60.70	53.00	62.00	57.00	43.00
Unreacted Particles	--	8.30	28.80	14.30	19.30	16.20
IGT Agglomeration Test	Fused	Caked	Caked	Caked	Partially caked	Partially caked
Free Swelling Index	4	2	2	2	1	1

* At 70°F and 1 atmosphere.

B75112727

Table 67. WEIGHT LOSS AND RATE OF OXYGEN CONSUMPTION
DURING PARTIAL OXIDATION

Run No.	Reaction Time, min	Oxygen Flow Rate, * CF/min	Rate of Oxygen Consumption* CF/min-lb of Coal	Weight Loss			IGT Agglomeration Test	FSI
				Measured	Calculated From Oxygen Consumption† %	Calculated From Ash Balance		
57	30	0.24	0.17	10.4	15.2	--	Caked	2
58	30	0.24	0.10	7.2	8.7	6.2	Caked	2
59	20	0.24	0.10	11.0	5.5	--	Caked	2
60	20	0.24	0.09	8.7	4.9	4.9	Partially caked	1
61	60	0.24	0.10	19.9	26.9	12.6	Partially caked	1

* At 70°F and 1 atmosphere.

A75112728

† Oxygen added to the coal was subtracted from the oxygen assumed to be converted to CO₂ and H₂O.

weight, of feed coal. In this series of experiments, feed and treated samples were handled with extreme care and weighed to the nearest tenth of a gram. The measured weight loss and the loss in weight calculated from the oxygen consumed and the ash balance agree fairly well.

The volatile matter, calculated on a moisture-free basis, was reduced from about 38% to 34%. Combined with the measured loss in weight, this corresponds roughly to a 27% reduction in the original volatile matter contained in coal. The material produced in the coal-water partial oxidation process, by virtue of its volatile matter and hydrogen content, has a potential for greater methane yields compared with the coals pretreated in a fluidized bed of steam and oxygen. (See the March 1975 Memorandum.) A complete ultimate analysis of the treated coals from Runs 57 through 61 indicates that during the partial oxidation process some oxygen was added to the coal. This addition of oxygen to the coal, shown in Table 66, was calculated from the ultimate analysis and ranges up to about 18%, by weight, of the original oxygen content. This leads to the possibility that the volatile matter in treated coals includes oxygenated carbon and hydrogen bonds, which may not contribute to methane formation on subsequent hydrogasification.

A comparison of the analyses of the treated Illinois No. 6, hvBb, Sahara mine coals from Runs 57 through 61 with the treated samples reported in the March 1975 Memorandum Report shows that even though the proximate analyses are similar to those obtained from 30 minutes of reaction time, the carbon and hydrogen contents are slightly higher in the former case. This could be attributed to improper distribution of the oxidizing medium in the reactor. The malfunction of the magnetic stirrer, which could aid gas bypassing, is another possibility. The petrographic analysis of the treated coal shows that Runs 57 through 61 resulted in fewer nonagglomerating particles or particles with bright rims compared with similar experiments reported in the March 1975 Memorandum Report. However, since the percentages of unreacted particles are the same order of magnitude in both the cases, the free swelling indexes are similar.

Poor gas distribution could also be responsible for the lower weight loss observed in Runs 57 through 61 compared with those reported in the March 1975 Memorandum Report. Poor contact between the submerged coal and the oxidizing medium appears to be pronounced in Run 61, which shows a 20% loss in weight compared with the 50% or more reported in the March 1975 Memorandum Report for similar experiments.

The estimated oxygen consumption rates for Runs 57 through 61, given in Table 67, are generally lower than the values reported in the March 1975 Memorandum Report. This observation agrees with suspected bypassing of the oxidizing medium during the partial oxidation experiment.

Conclusions

An analysis of the results of exploratory experiments for partial oxidation of coals suspended in water has shown that the agglomerating properties of Eastern high-volatile bituminous coals can be reduced by treatment with air or oxygen at 1000 psig and 540°F. Tests conducted in a small, semibatch, 500-milliliter reactor showed that the free swelling index of untreated coals equal to or less than 7 could be reduced to 2 or 1, with about 10% loss in feed coal, during a 20 to 30 minute reaction time. In some experiments, however, a weight loss of 50% was noticed. An increase in oxygen content, possibly by the addition of oxygen to carbon and hydrogen in coal, was noted and may not enhance methane or hydrogen production in subsequent coal gasification steps. Carbon dioxide is the primary gaseous product of coal-water partial oxidation.

The concept of a coal-water slurry scheme for coal preparation and handling for coal gasification has several advantages over the dry-coal handling methods. Even though the coal-water partial oxidation process could be an integral part of the coal-water slurry scheme, the results of these small-scale experiments thus far do not provide a basis for recommending this process over the steam-oxygen fluidized pretreatment process. Experiments conducted in a large-scale pilot plant with a continuous flow of slurry and oxidizing medium may aid in optimizing reaction time, oxygen requirements, and coal consumption. With the progress of pilot plant gasification experiments with bituminous coal at IGT and elsewhere, the minimum pretreatment required for fluidized-bed gasification of agglomerating coals will be better defined. The coal-water partial oxidation experiments will be reevaluated for their practical utility when more quantitative information on the agglomerating properties of feed coals becomes available.

Castable Insulation in Acidic Condensing Atmosphere

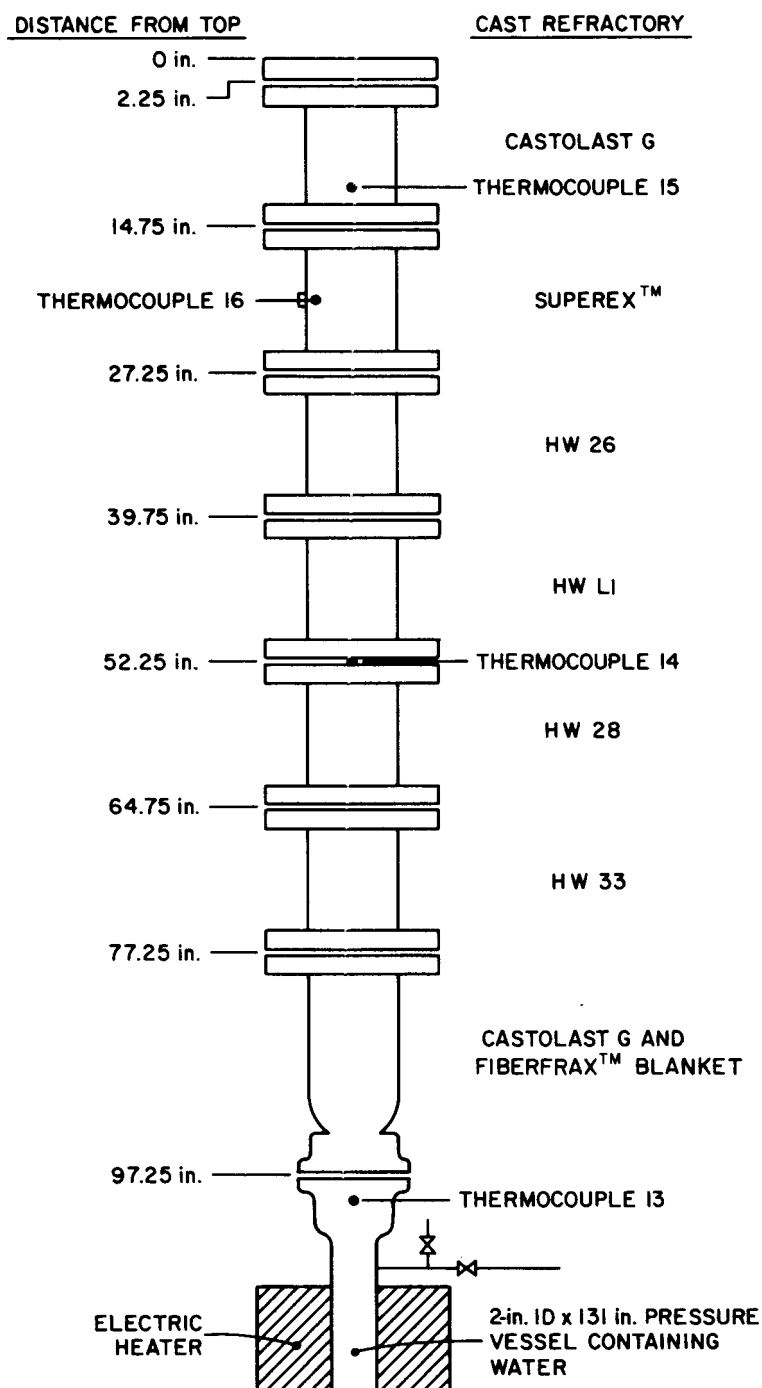
The following seven insulating materials were cast into seven flanges and assembled on top of a water heater as shown in Figure 100.

1. HW-Lightweight Castable LI
2. HW-26
3. HW-28
4. HW-33
5. Castolast G
6. SUPEREXTM Block Insulation
7. FIBERFRAXTM

The cast refractories were cured to 250°F in the presence of nitrogen.

The test section containing seven types of insulating materials was pressure-tested and heated in the presence of saturated steam at pressures ranging from 900 to 1000 psig. During the test period, the temperatures ranged from 540°F at thermocouple No. 13 to 350°F at thermocouple No. 15. The refractory was heated for 15 days, and then the test section was dismantled. The refractories were visually inspected and were undisturbed except for the disassociation of the few patches of insulating material applied by hand to cover the unfilled gaps on the surface of the cast test sections. The water drained from the test section was analyzed by atomic absorption spectroscopy to determine the constituents of the refractories leached by the condensing steam.

The test section was reassembled and pressure-tested at about 530°F and 1000 psig. After it cooled down and was depressurized, a fresh batch of about 19 pounds of water was added. The test section was then pressurized with carbon dioxide to about 185 psig and with hydrogen sulfide from 185 to 210 psig.



A-84-1405

Figure 100. EQUIPMENT SETUP FOR TESTING CASTABLE REFRACTORIES

The test section was cooled, depressurized at the end of 4 weeks of contact with acid-gas in a condensing atmosphere at about 1000 psig and 530°F, and dismantled for a physical and chemical analysis of the refractory materials. The analysis of water samples taken from the test section is shown in Table 68.

Table 68. ANALYSIS OF CONDENSATE FROM REFRACTORY TEST SECTION

	Water Samples		
	No. 1	No. 2	No. 3
	(#24878)	(#24811)	(#24875)
	ppm		
Elements Present			
Ca	9.02	0.56	142.0
Mg	0.182	6.0	24.3
Al	0.440	6.2	66.8
Na	438.0	980.0	2280.0
K	0.104	61.0	188.0
Si	<2.5	590.0	344.0
Remarks	Feedwater	Condensate at the end of 15 days contact with steam	Condensate at the end of 7 days contact with acid-gas and steam

An inspection of the refractory sections showed that the SUPEREX and FIBERFRAX had been severely attacked, while the castables (HW-LI, -26, -28, -33, and Castolast-G) maintained their original physical dimensions and shapes in the condensing acid-gas environment at about 535°F and 1000 psig. Several sections of the treated castables were cut and set aside for detailed physical and chemical analyses. An analysis of a water sample taken at the end of a 23-day test period is shown in Table 69.

Table 69. ANALYSIS OF CONDENSATE FROM REFRACTORY TEST SECTION

	Feedwater	Condensate
	(Lab. No. 24878)	(Lab. No. 25156)
	ppm	
Elements Present		
Ca	9.02	325.0
Mg	0.182	156.0
Al	0.440	97.7
Na	438.0	2750.0
K	0.104	331.0
Si	<2.5	1560.0
Remarks	A fine suspension suspected to be silica gel was observed in the condensate. pH = 8.	

When the refractory test section was dismantled and inspected, Figure 100 the block insulation (SUPEREXTM) and the blanket insulation (FIBERFRAXTM) were obviously completely corroded by the 30-day exposure to the condensing acid gas at 1000 psig and 540°F. These and the castable refractory materials, before and after the test, are shown in photographs in Figures 101 to 107. Although these test sections were from Harbison-Walker Refractories Co., other refractory materials with a comparable composition are expected to show similar behavior.

A physical inspection of the castable refractory materials did not reveal any visible deformations. HW-LI, -26, and -28 were slightly darker on the convex surface adjacent to the metal wall and showed several rust-colored areas. The HW-33 and Castolast G also became slightly darker, and each had a deposition of fine, white powder on its concave surface. A chemical analysis showed that most of this white powder was silica, leached by the condensing acid gas from the test sections on top of HW-33, shown in Figure 100.

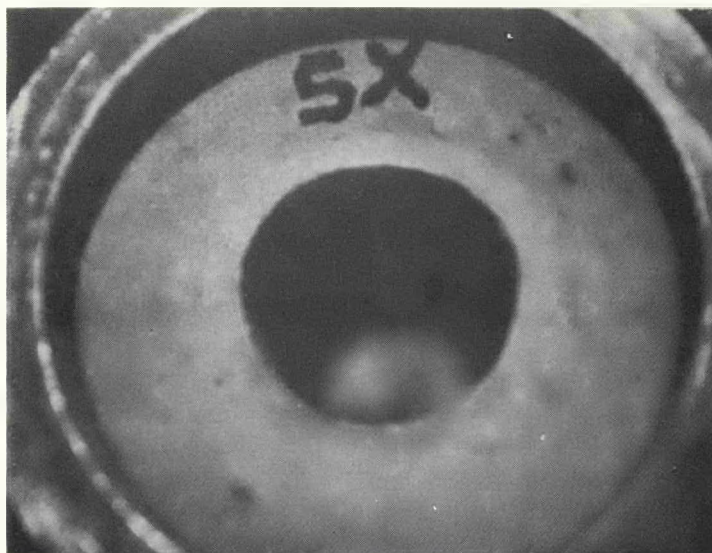
The effects of the condensing acid gas on the physical and chemical properties of the HW refractories can be seen by comparing the analyses of samples before and after testing, Table 70. There is very little change in the bulk density and cold-crushing strength, except for HW-28, which reports a 10% reduction in bulk density and about a 32% loss in cold-crushing strength. However, Harbison-Walker laboratories will repeat these measurements after first drying the treated samples to 230°F for a better comparison with the original sample analyses.

Under chemical analyses, the silica content of all of the refractory samples did not alter very much, the largest change being evident in HW-28. The gain in silica in Castolast G can be attributed to the fine silica powder deposited on the concave surface. A representative analysis of Castolast G will be repeated after washing the surface free of the deposited silica powder. The slight gains in alumina (Al_2O_3) noticed in HW-LI, -26, and -28 could have resulted from the loss of silica, lime (CaO), magnesia (MgO), and the alkaline oxides.

An enrichment of the tested refractory materials with iron sulfide, reported as Fe_2O_3 and SO_3 , was noticed in HW-26, -28, and -33. The increase in iron sulfide is believed to be the result of the reaction of the hydrogen sulfide (H_2S) in the acid gas with the steel walls of the container.

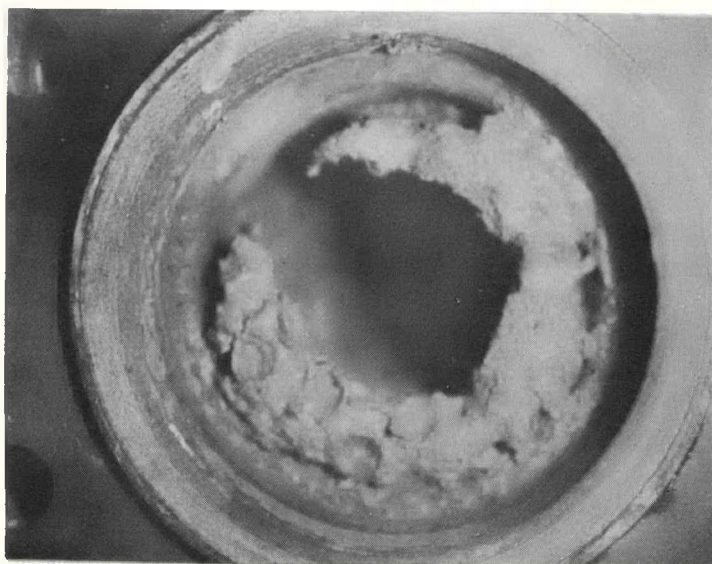
It is apparent from Table 70 that the soda (Na_2O) and potash (K_2O) contained in the tested refractory materials were almost completely leached by contact with the condensing acid gas. The loss of Na_2O and K_2O is not detrimental to the physical strength of the refractory and, in fact, is supposed to improve its refractory qualities. Any loss in CaO, however, may represent a reduction in calcium-aluminate bonding and could result in a loss of refractory strength. HW-26, which showed a CaO loss of about 14%, lost only 2% of its cold-crushing strength. On the other hand, HW-28, with a 31% CaO loss, suffered a 32% reduction in its cold-crushing strength. Measurements of the cold-crushing strength and the modulus of rupture, performed on dried samples, provided additional information on the relationship between the loss of CaO and the strength of the refractory materials.

BEFORE



P75040608

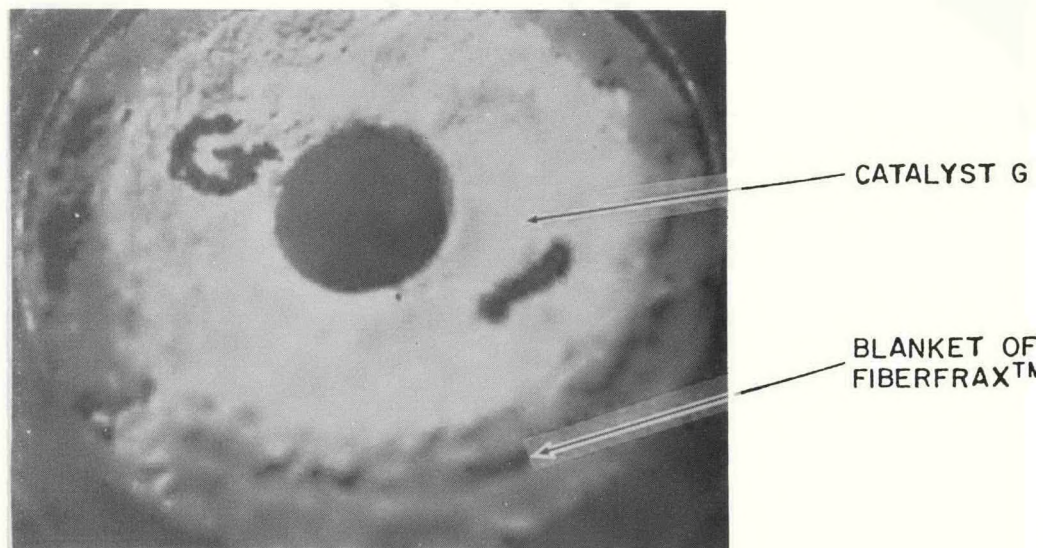
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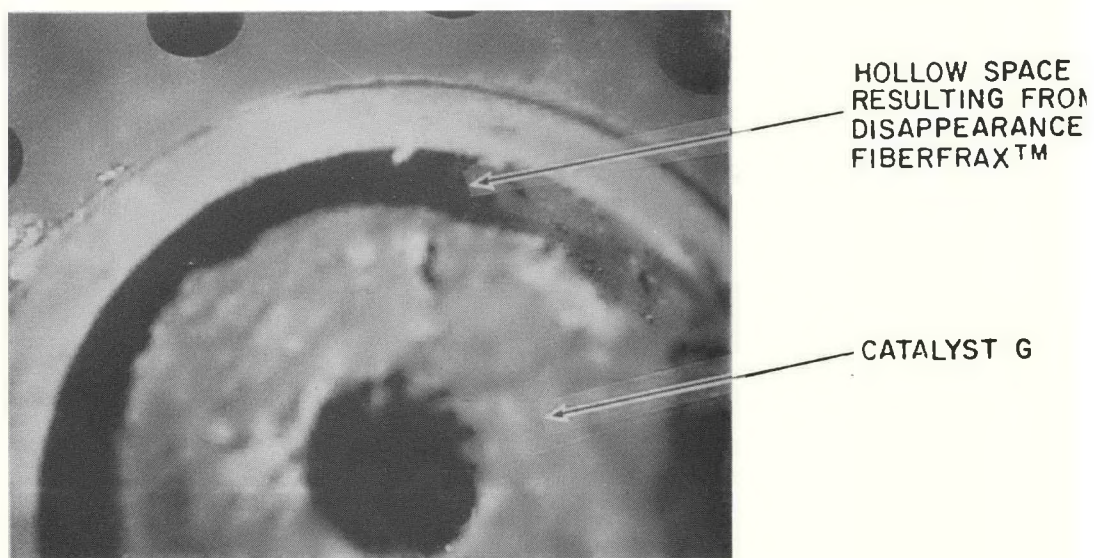
P75040609

Figure 101. PERFORMANCE OF SUPEREXTM INSULATING MATERIAL
IN A CONDENSING ACID GAS (1000 psig and 540°F)

BEFORE



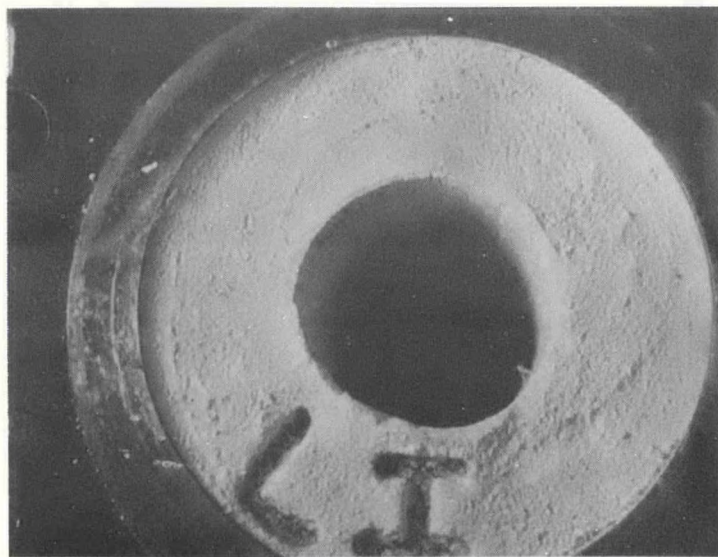
AFTER



P75040611

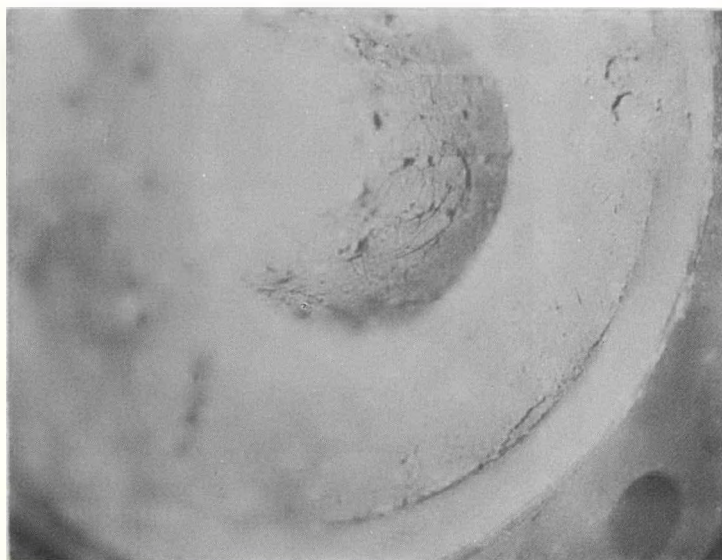
Figure 102. PERFORMANCE OF CASTOLAST G AND FIBERFRAX™ INSULATION MATERIALS IN A CONDENSING ACID GAS (1000 psig and 540°F)

BEFORE



P75040612

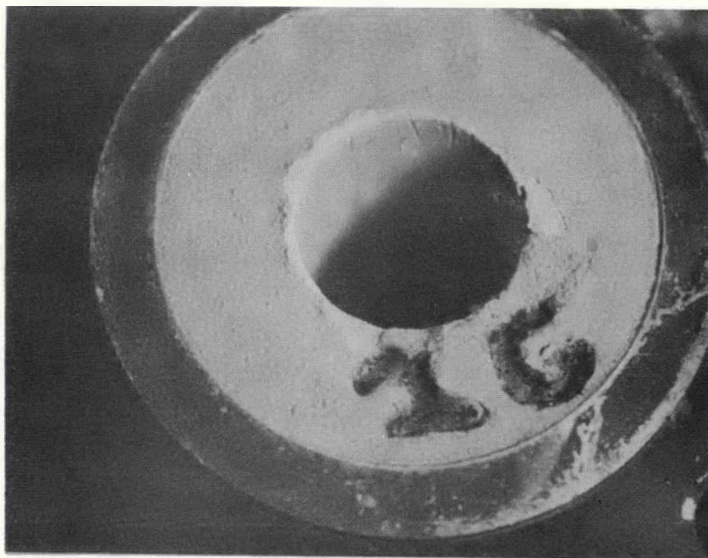
AFTER



P75040613

Figure 103. PERFORMANCE OF HW-LI REFRACTORY MATERIAL IN A
CONDENSING ACID GAS (1000 psig and 540°F)

BEFORE



P75040614

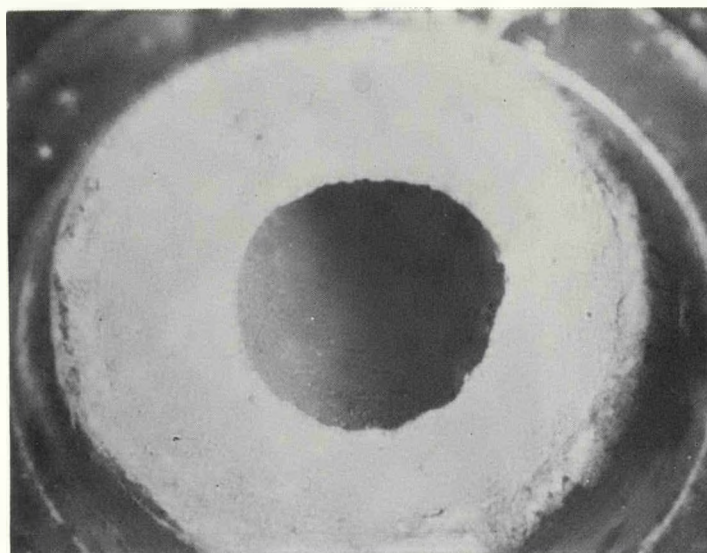
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P75040615

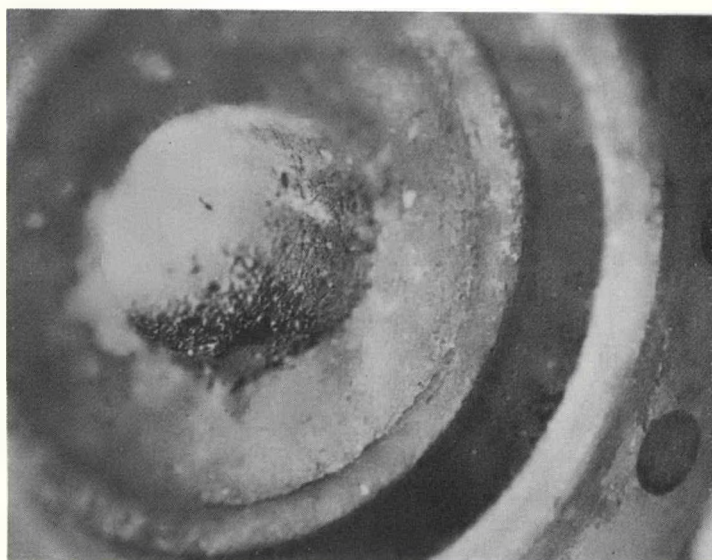
Figure 104. PERFORMANCE OF HW-26 REFRACTORY MATERIAL IN A
CONDENSING ACID GAS (1000 psig and 540°F)

BEFORE



P75040616

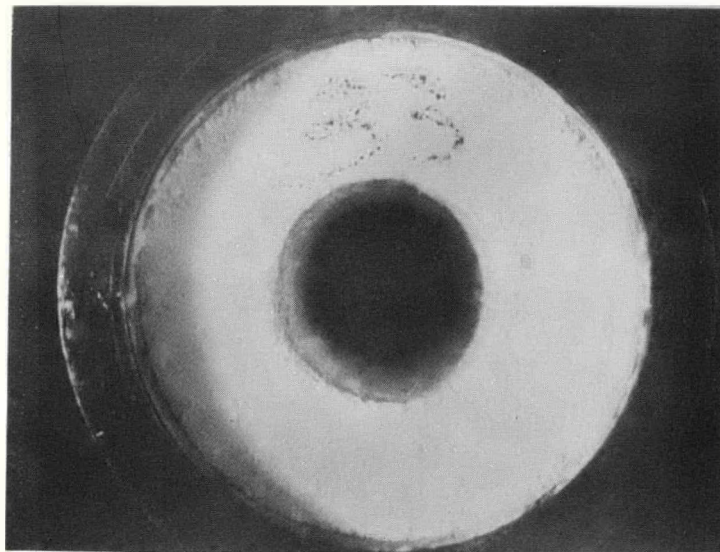
AFTER



P75040617

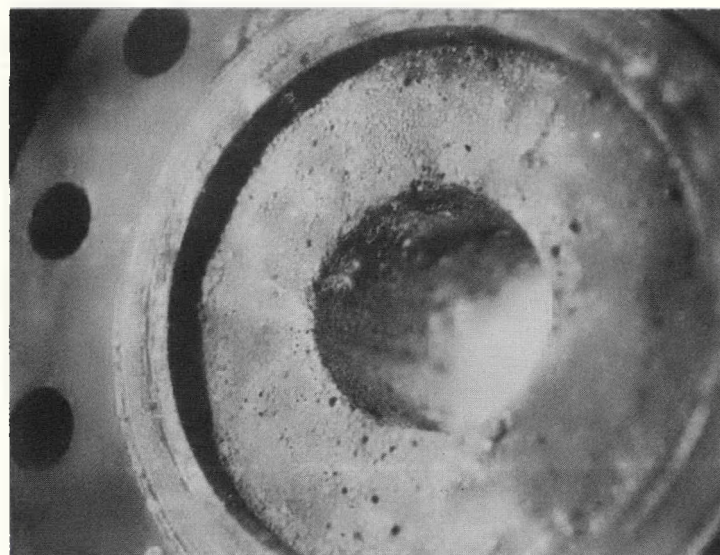
Figure 105. PERFORMANCE OF HW-28 REFRACTORY MATERIAL IN A
CONDENSING ACID GAS (1000 psig and 540°F)

BEFORE



P75040618

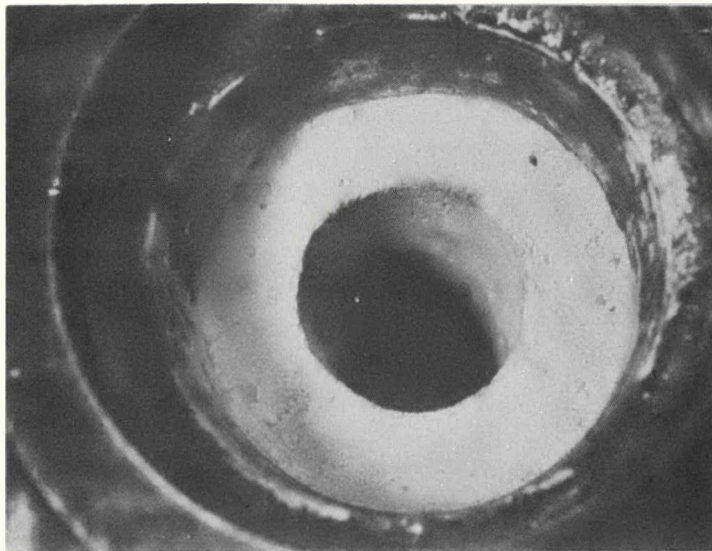
AFTER



P75040619

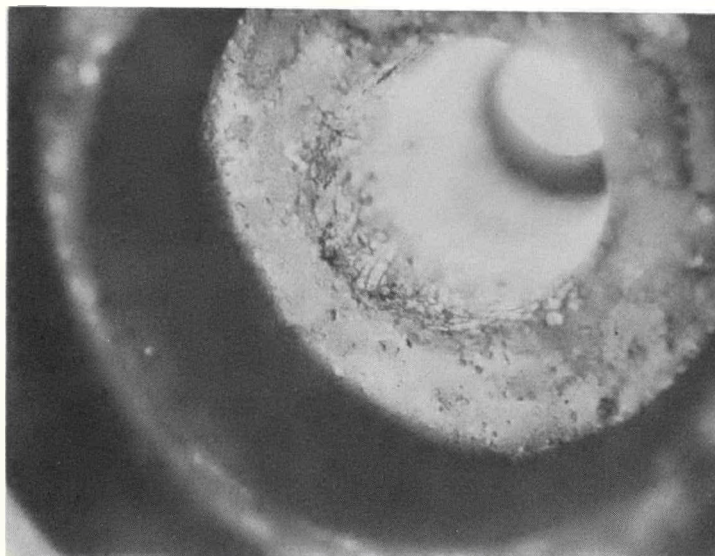
Figure 106. PERFORMANCE OF HW-33 REFRACTORY MATERIAL IN A
CONDENSING ACID GAS (1000 psig and 540°F)

BEFORE



P75040620

AFTER



P75040621

Figure 107. PERFORMANCE OF CASTOLAST G REFRACTORY MATERIAL
IN A CONDENSING ACID GAS (1000 psig and 540°F)

Table 70. PHYSICAL AND CHEMICAL ANALYSES OF CASTABLE REFRACTORY MATERIALS BEFORE (1973 Q.C. Average) AND AFTER TESTING (PD)
(Reported by Harbison-Walker Refractories Co., February 14, 1975)

Laboratory Number	1973 Q.C.		1973 Q.C.		1973 Q.C.		1973 Q.C.		1973 Q.C.	
	PD 0555A	Average	PD 0555B	Average	PD 0555C	Average	PD 0555D	Average	PD 0555E	Average
Brand	HW Lightweight Castable 33		HW Lightweight Castable 28		HW Lightweight Castable 26		HW Lightweight Castable LI		Castolast G	
Bulk Density, lb/cu ft (as received)	105	96 ^d	91	100 ^d	63	61 ^d	56	55 ^d	172	167 ^d
Bulk Density, g/cu cm (as received)	1.68	1.54	1.46	1.60	1.01	0.98	0.90	0.88	2.75	2.67
Cold-Crushing Strength, psi (as received)	-- ^c	--	950	1390 ^d	470	480 ^d	470	550 ^d	7860	9060 ^d
Cold-Crushing Strength, kg/sq cm (as received)			66.9	7.8	33.1	33.8	33.1	38.7	553	638
Chemical Analysis, % (calcined basis) ^a										
Silica (SiO ₂)	0.26	0.40	33.70	41.0	35.90	37.4	46.10	47.9	0.28	0.1
Alumina (Al ₂ O ₃)	92.20	92.60	58.30	50.7	56.90	52.8	40.40	38.8	93.20	93.7
Titania (TiO ₂)	0.01	0.02	1.54	1.2	0.69	0.5	0.26	0.2	<0.01	0.1
Iron Oxide (Fe ₂ O ₃)	0.06	0.03	1.39	0.8	1.03	0.6	0.51	0.6	0.20	0.3
Lime (CaO)	7.25	6.80	4.20	4.9	4.85	7.0	11.90	10.5	6.58	5.6
Magnesia (MgO)	0.12	--	0.19	0.3	0.32	0.1	0.46	0.2	0.07	0.1
Soda (Na ₂ O)	<0.01	{ 0.20	<0.01	{ 1.1	<0.01	{ 1.6	<0.01	{ 1.8	0.08	{ 0.1
Potash (K ₂ O)	<0.01		0.06		0.16		0.16		0.02	
Total	99.92	100.05	99.39	100.0	99.86	100.0	99.80	100.0	100.44	100.0
Chemical Analysis, % (dry basis) ^b										
Sulfur Trioxide (SO ₃)	0.11	--	1.69	--	1.18	--	0.34	--	0.10	--
Total Carbon (C)	0.72	--	0.05	--	0.09	--	0.17	--	0.07	--
Loss on Ignition	11.55	--	6.51	--	8.51	--	16.33	--	9.10	--
pH	9.10	--	8.35	--	8.10	--	8.25	--	11.10	--

^a Alkalies by flame photometer, all others by spectrograph.

^b Total carbon and sulfur trioxide by Leco furnace.

^c Insufficient sample to run.

^d After drying at 230 °F.

B75050992

The results from Test 1, evaluating the performances of HW-LI, -26, -28, -33, Castolast G, SUPEREXTM and FIBERFRAXTM were summarized in the March 1975, ERDA Report No. 126. A recent report from Harbison-Walker Refractories Co. discussed some possible mechanisms for the loss of certain constituents in these samples following the 30-day test at 1000 psig, 540°F, and under acidic condensing conditions. The samples exhibited satisfactory densities and cold-crushing strengths. All of them, with the exception of HW-Lightweight Castable 26, showed very little lime depletion, which is the primary constituent in the high-purity cement binders of these products. An appreciable loss of lime in an acidic environment is expected to result in a significant loss of castable strength.

The chemistries revealed a reduced alkali content in all test samples. According to one theory, the soda and potash are probably forming hydroxides and are being washed out of the castable in the presence of the high-pressure condensing steam. Another theory proposed that the alkalies might be attacking the free silica and forming silicates: This might account for the depletion of silica in several of the products. The water samples reveal there is a corresponding increase of these constituents in the condensate. A depletion of the alkalies should not be detrimental to the refractory composition.

The LOI's (loss on ignition) for each test sample were abnormally high. This is probably caused by the pickup of moisture during the testing. The samples were not dried prior to examination. Harbison-Walker determined the densities and LOI's on additional dried samples and is able to verify this observation. In addition, the chemistry of the white powdery buildup seen on the face of the Castolast G will be checked. It also looks similar to silica and may be from the decomposition of the FIBERFRAXTM or calcium silicate SUPEREXTM block insulation.

In summary, Harbison-Walker does not feel that the conditions imposed on the castables tested resulted in significant deleterious effects on the refractories. Neither HW-Lightweight Castable 33 nor Castolast G showed evidence of significant chemical alteration, and evidently, leaching of constituents from the other lightweight castables was not appreciable enough to affect their physical properties.

The treated castables were dried at 250°F for 18 hours at the Garber Research Center of Harbison-Walker Refractories. Part of the analysis conducted on these samples is reported in Table 71. Compared with the analysis of the as-received samples, there was no apparent change either in the bulk density or in a loss-on-ignition of the dried samples.

Table 71. PROPERTIES OF DRIED CASTABLE SAMPLES

	<u>Dried Sample</u>	<u>As-Received Sample*</u>	<u>1973 - Q.C. Average*</u>
HW-Castolast-G			
Bulk Density, lb/cu ft	167	172	167
Loss on Ignition, %	8.72	9.1	--
HW-Lightweight Castable 26			
Bulk Density, lb/cu ft	65	63	61
Loss on Ignition, %	7.72	8.51	--
HW-Lightweight Castable LI			
Bulk Density, lb/cu ft	62	56	55
Loss on Ignition, %	14.2	16.33	--
HW-Lightweight Castable 28			
Bulk Density, lb/cu ft	91	91	100
Loss on Ignition, %	5.88	6.51	--

* Analysis reported in the March 1975, ERDA Report No. 126.

The analysis of the fine white powder deposited on the Castolast-G test section during the treatment with the condensing acid gas is given in Table 72. This powdery coating was composed primarily of silica, alumina, lime, and magnesia. Previous monthly reports listed the FIBERFRAXTM and SUPEREXTM test sections, which were seriously attacked by the condensing acid gas, as the suspected sources of the white powdery coating on Castolast-G. However, a comparison of the analysis of the white powdery substance with the representative analysis of FIBERFRAXTM and SUPEREXTM shown in Table 72. raises an interesting question about the pickup of magnesia in the powdery substance. Its possible sources were studied and a mechanism to explain this phenomenon was sought. Work continued to interpret the mechanism of loss of silica, soda, potash, and lime observed with the castable materials tested.

Table 72. COMPARISON OF CHEMICAL ANALYSIS OF THE WHITE POWDERY COATING ON CASTOLAST G WITH FIBERFRAXTM AND SUPEREXTM BLOCK INSULATING MATERIALS

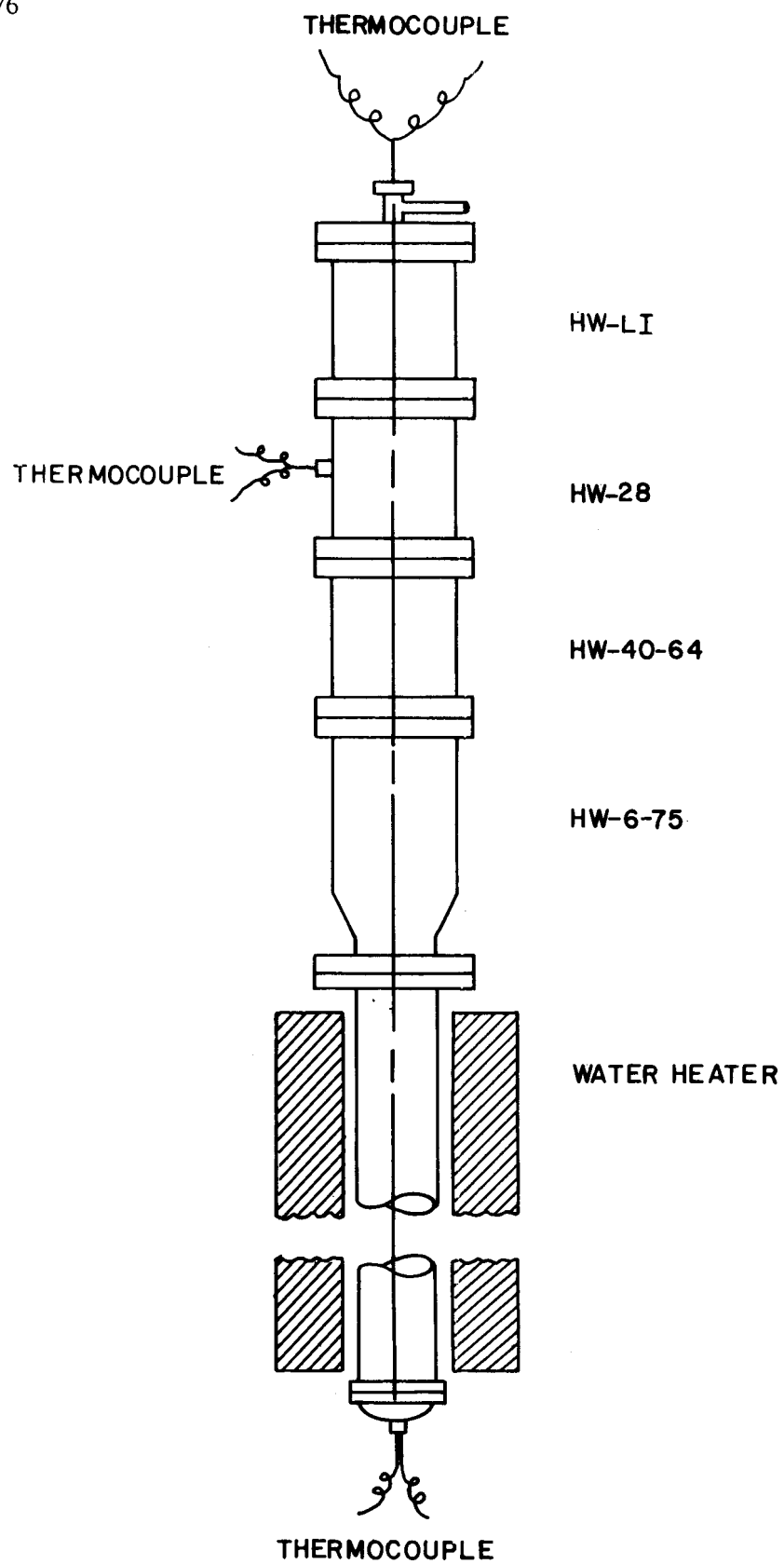
Chemical Analysis (Dry Basis [*])	White Powdery Coating on Castolast G	Representative Analysis	
		FIBERFRAX TM	SUPEREX TM †
		wt %	
Silica (SiO ₂)	34.4	74.8	47.6
Alumina (Al ₂ O ₃)	11.6	4.3	51.7
Titania (TiO ₂)	<0.01	--	--
Iron Oxide (Fe ₂ O ₃)	0.20	2.0	0.02
Chromic Oxide (Cr ₂ O ₃)	<0.1	--	--
Lime (CaO)	7.95	6.0	--
Magnesia (MgO)	25.1	0.8	--
Soda (Na ₂ O)	0.07	2.7	0.3
Potash (K ₂ O)	0.02	--	--
Loss on Ignition	<u>20.6</u>	<u>9.1</u>	<u>--</u>
Total	99.9	99.7	99.62
Sulfur Trioxide (SO ₃)	0.39	--	--
Total Carbon (C)	1.87	--	--

* Alkalies by flame photometer and total carbon and sulfur trioxide by Leco. All others by spectrographic analysis.

† SUPEREXTM also contains 0.15 weight percent B₂O₃ in addition to the listed chemical constituents.

By June 1975, all of the test castable refractory samples were received from Harbison-Walker Refractories. Because of the limited capacity of the electric heater that is available for Test 2, only four samples were cast in the cylindrical flanged sections, and these were HW 6-75 (vitreous silica), HW 40-64, HW-28, and HW-LI. Simultaneously with the cylindrical test sections, reference samples, in the form of 3 x 3 x 9-inch bricks, were also cast and set aside. All of the cast samples were air dried and cured according to the instructions furnished by Harbison-Walker Refractories.

The cylindrical, flanged test sections were assembled as shown in Figure 108. The assembled section was pressure-tested, after which the castables were contacted with condensing steam at 1000 psig and 540°F for 30 days.



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Figure 108. ASSEMBLY OF CASTABLE INSULATING MATERIALS

Economic Summary

Table 73 summarizes investment costs for Cases 1, 2, 3A, and 3B. The total investment increases from \$358 million in Case 1 to \$370 million in Case 3A and to \$385 million in Case 3B. Total installed equipment costs excluding contingencies rise from \$232.4 million in Case 1 to \$240.5 million in Case 3A and to \$250.2 million in Case 3B. The general conclusion is that, although elimination of the LTR simplifies the operation and reduces the cost of the hydrogasifier, it is not desirable from an overall economic standpoint because of increased costs in other sections of the plant.

Elimination of the HTR saves \$10.54 million in the cost of the hydrogasifier-oxygasifier reactor. But everything else costs the same or more because of the reduction of direct methane formation. For Case 3A the added plant costs amount to \$18.61 million over Case 1. Of this total, the increased cost for the oxygen plant is about one-third, or \$6.2 million. The rest of the difference is spread among the different plant sections, with the slurry oil vaporizer, the quench system, purification, carbon monoxide conversion, and methanation being the major items. For Case 3B the added plant costs amount to \$28.26 million.

Annual operating costs and revenue requirements are presented in Table 74. The unit cost of Montana coal was increased from 15¢ (used in previous studies) to 18¢/million Btu. The operating labor rate was increased to \$5.50/hr. Percentage factors for operating costs are the same as before, except for the 10.5% return on the rate base and a small change in the percent of maintenance.

By-product credits are much larger than previously reported because of large increases in market prices during the past 1 to 2 years. Unit by-product values used previously and those used in this study are summarized in Table 75, together with annual by-product credits for each case. Because of the rather capricious record of sulfur prices, they were kept at \$10/ton. Benzene and ammonia values were increased conservatively, actually less, percentagewise than the market price. The unit values of these by-products were always kept below the market price because they are values at the pipeline gas plant, so some allowance is necessary to move them to market. To be conservative, the values used in this study, with the exception of the make oil, were increased less than the market price. Because of the great jump in all oil prices, even residual oil is selling for more than 10¢/gal. Since fuel oil is used everywhere, 10¢/gal is readily justified. Total by-product values reduce gas price by about 12¢/million Btu.

By eliminating the LTR, the overall efficiency of the process is reduced by 2.8% because of an increased oxygen requirement, motive power, and more carbon monoxide conversion and methanation. The gas price is 3¢/million Btu higher in Case 3A and 7¢/million Btu higher in Case 3B.

Mechanism of Ash-Agglomerate Formation

The structure, composition, and mechanism of formation of the solid discharge product from the ash-agglomerator unit, sampled during Run 17 was investigated. The product consists predominantly of rounded particles

Table 73, EFFECT OF SINGLE-STAGE vs. TWO-STAGE HYDROGASIFICATION
ON CAPITAL INVESTMENTS FOR 25 BILLION Btu/DAY PIPELINE GAS PLANTS,
HYGAS PROCESS USING STEAM-OXYGEN GASIFICATION OF MONTANA COAL

Case	1	2	3A	3B
	Installed Cost, \$10 ⁶			
Coal Storage and Reclaiming	3.51	3.55	3.63	3.69
Coal Grinding and Drying	8.15	8.28	8.62	8.62
Slurry Feed System	6.77	6.84	7.01	8.69
Slurry Oil Vaporizers	5.43	5.76	7.87	7.57
Hydrogasification				
Low-Temperature Transport				
Reaction Zone	16.02	16.44	--	--
High-Temperature Hydrogasi- fication Zone	16.53	14.25	16.59	16.59
Char Gasification Zone	13.41	14.01	18.83	18.83
Oxygen Plant	23.20	25.20	29.40	29.40
Oxygasifier Residue Handling and Ash Disposal	2.00	2.00	2.00	2.00
Quench System	16.20	16.46	17.22	20.29
Benzene Recovery	3.86	3.90	4.05	4.05
Prepurification I (Hot K ₂ CO ₃ + Act. Carbon + ZnO)	18.66	19.35	20.75	20.75
CO Conversion	2.13	2.13	2.29	2.29
Prepurification II (Hot Carbonate)	9.59	9.61	10.48	10.48
Methanation and Drying	10.09	10.16	11.15	11.15
Methanation and CO Shift Waste-Heat Recovery	4.45	4.48	4.98	4.98
Turbogenerator	6.61	6.76	7.22	8.25
Power Distribution	6.71	6.85	7.35	8.17
Process and Turbine Steam Generation	22.83	23.10	23.12	26.25
Cooling and Makeup Water	4.65	4.83	5.44	5.60
Sulfur Recovery (Stretford)	3.98	4.08	4.19	4.19
Wastewater Treatment	10.52	10.66	11.05	11.05
Particulate Emission Control	3.39	3.43	3.52	3.52
General Facilities and Miscellaneous	13.75	13.75	13.75	13.75
Subtotal Excontingencies	232.44	235.88	240.51	250.16
Contingencies (15%)	34.87	35.38	36.08	37.52
Total Plant Cost	267.31	271.26	276.59	287.68
Contractors' Overhead + Profits (10%)	26.73	27.13	27.66	28.77
Subtotal	294.04	298.39	304.25	316.45
Interest During Construction (9% X 1.5)	39.70	40.28	41.07	42.72
Start-Up Cost (5% of Total Plant Cost)	13.37	13.56	13.83	14.38
Fixed Investment	347.11	352.23	359.15	373.55
Working Capital	10.44	10.59	10.85	11.22
Total Investment	357.55	362.82	370.00	384.77

Table 74. EFFECT OF SINGLE-STAGE vs. TWO-STAGE HYDROGASIFICATION ON ANNUAL OPERATING COSTS FOR 250 BILLION Btu/DAY PIPELINE GAS PLANTS; HYGAS PROCESS USING STEAM-OXYGEN GASIFICATION OF MONTANA COAL AND USING A.G.A./OCR ACCOUNTING PROCEDURE (90% Stream Factor, Timing Mid-1974)

<u>Case</u>	<u>1</u>	<u>2</u>	<u>3A</u>	<u>3B</u>
	<u>Annual Operating Cost, \$</u>			
Coal Cost, 18¢/10 ⁶ Btu*	24,121,900	24,483,200	25,275,400	25,926,200
Other Direct Materials	2,031,500	2,067,700	2,261,600	2,282,100
Direct Operating Labor (\$5.50/hr, 8760 hr/yr)	2,794,400	2,794,400	2,794,400	2,794,400
Maintenance†	7,716,600	7,821,200	7,954,200	8,263,000
Supplies (15% of Maintenance)	1,157,500	1,173,200	1,193,100	1,239,500
Supervision, 10% of Direct Operating Labor	279,400	279,400	279,400	279,400
Payroll Overhead (10% of Direct Operating Labor + Supervision)	307,400	307,400	307,400	307,400
General Overhead (50% of Direct Operating Labor + Maintenance + Supplies + Super- vision)	5,974,000	6,034,100	6,110,600	6,288,200
Depreciation (5% of Fixed Investment)	17,355,500	17,611,500	17,957,500	18,677,500
Local Taxes and Insurance (3% of Total Plant Cost)	8,019,300	8,137,800	8,297,700	8,630,400
Operating Expenses Subtotal	69,757,500	70,709,900	72,431,300	74,688,100
Contingencies (2%)	1,395,200	1,414,200	1,448,600	1,493,800
Total Operating Expenses	71,152,700	72,124,100	73,879,900	76,181,900
Less By-Product Credits	- 9,816,800	- 9,957,400	-10,366,500	-10,366,500
Net Operating Expense	61,335,900	62,166,700	63,513,400	65,815,400
Return-On-Rate Base at 10.5% (20-yr Average)	18,407,400	18,678,200	19,054,100	19,808,400
Federal Income Tax (48%, 20-yr Average)	6,855,800	6,956,600	7,096,800	7,377,000
Total Revenue Requirements (20-yr Average)	86,599,100	87,801,500	89,664,300	93,000,800
Annual Gas Production, 10 ⁹ Btu (250.4/stream day)	82,256.4	82,256.4	82,256.4	82,256.4
20-Year Average Pipeline Gas Price, ¢/10 ⁶ Btu	105.3	106.7	109.0	113.1

* An additional 2% coal has been included in all cases to correct the energy balance.

† Turbogenerator, oxygen plant, and steam generation at 2.5%; the remaining sections at 3% of the total plant cost including contingencies.

Table 75. SUMMARY OF BY-PRODUCT CREDITS

By-Product	Unit Value				Annual Value		
	1971-72	This	Current Market		Case		
	<u>Studies</u>	<u>Study</u>	<u>Price</u>		<u>1</u>	<u>2</u>	<u>3A and B</u>
						\$1000	
Benzene, ¢/gal	15	25	65-75	Industrial or	3298.6	3338.4	3477.5
(gal/day)				nitration	(40,166)	(40,650)	(42,344)
Sulfur, \$/long ton	10	10	26-27	Chicago	256.9	260.5	271.7
(long tons/day)			12-15	Alberta, Canada	(78.2)	(79.3)	(82.7)
Make Oil, ¢/gal	3.5	10	10¢ and up, all grades		5465.7	5550.2	5775.7
(gal/day)					(166,383)	(168,956)	(175,819)
Ammonia, \$/ton	25	35	110-130	Anhydrous	795.6	808.3	841.6
(short tons/day)				fertilizer	(69.2)	(70.3)	(73.2)
Total Annual Value, \$1000					9816.8	9957.4	10,366.5

(beads), ranging from 1/8 to 1/4 inch in diameter, although about 12 weight percent is in the form of angular particles in about the same size range.

For microscopic observation, the beads were mounted in epoxy resin, sectioned, and polished. A composite photomicrograph of the whole 1-inch briquet, taken with vertical illumination, is shown in Figure 109. Residual coke appears as light areas here, both as the main constituent of the angular particles and as the very small constituent particles of the rounded beads. These small particles are situated mostly on the periphery of the beads, although a few are buried in the interior.

The many rounded black areas in the bead are empty vesicles. Others, connected by passageways to the exterior, were filled with the epoxy resin during mounting. Then the system is evacuated before covering the sample with the resin, and the still-liquid resin subsequently is forced into such vesicles by the readmitted atmospheric pressure.

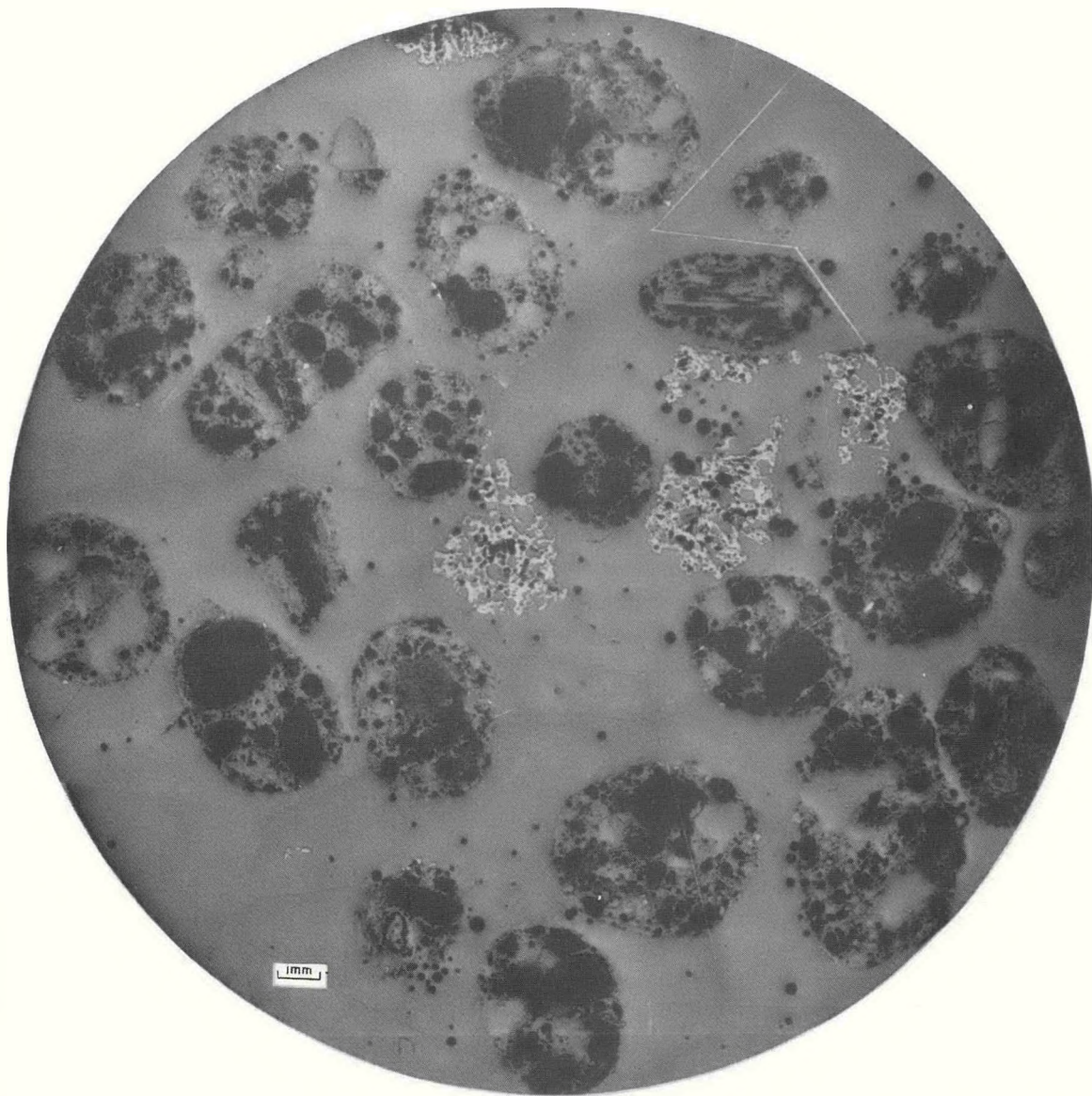
A composite photomicrograph of the same briquet taken with oblique illumination is shown in Figure 110. With this illumination, the continuous phase of glassy melted ash in the rounded beads can be distinguished from many of the embedded unmelted particles.

The unmelted ash particles have not been identified, except that the ones with the highest reflectance are particles of high-alumina refractory from the walls of the unit. When the reflectances of some of these high-reflectance particles were measured, they agreed with that of the refractory. Very small vesicles in the melted ash appear as pinpoints of reflected light. Some of the large, empty vesicles are partly illuminated on one side.

The distribution of the residual carbon indicated by microscopic observation was substantiated by further investigation: A sample of the residue was separated by hand into beads and angular particles; analyses for carbon then showed only 1.9 weight percent in the beads and 70.4 weight percent in the angular particles. That most of the residual carbon in the beads is not embedded and is available for reaction was shown by igniting a sample of the beads for 1/2 hour at about 1550°F, followed by an analysis for unburned carbon. Only 0.36 weight percent remained.

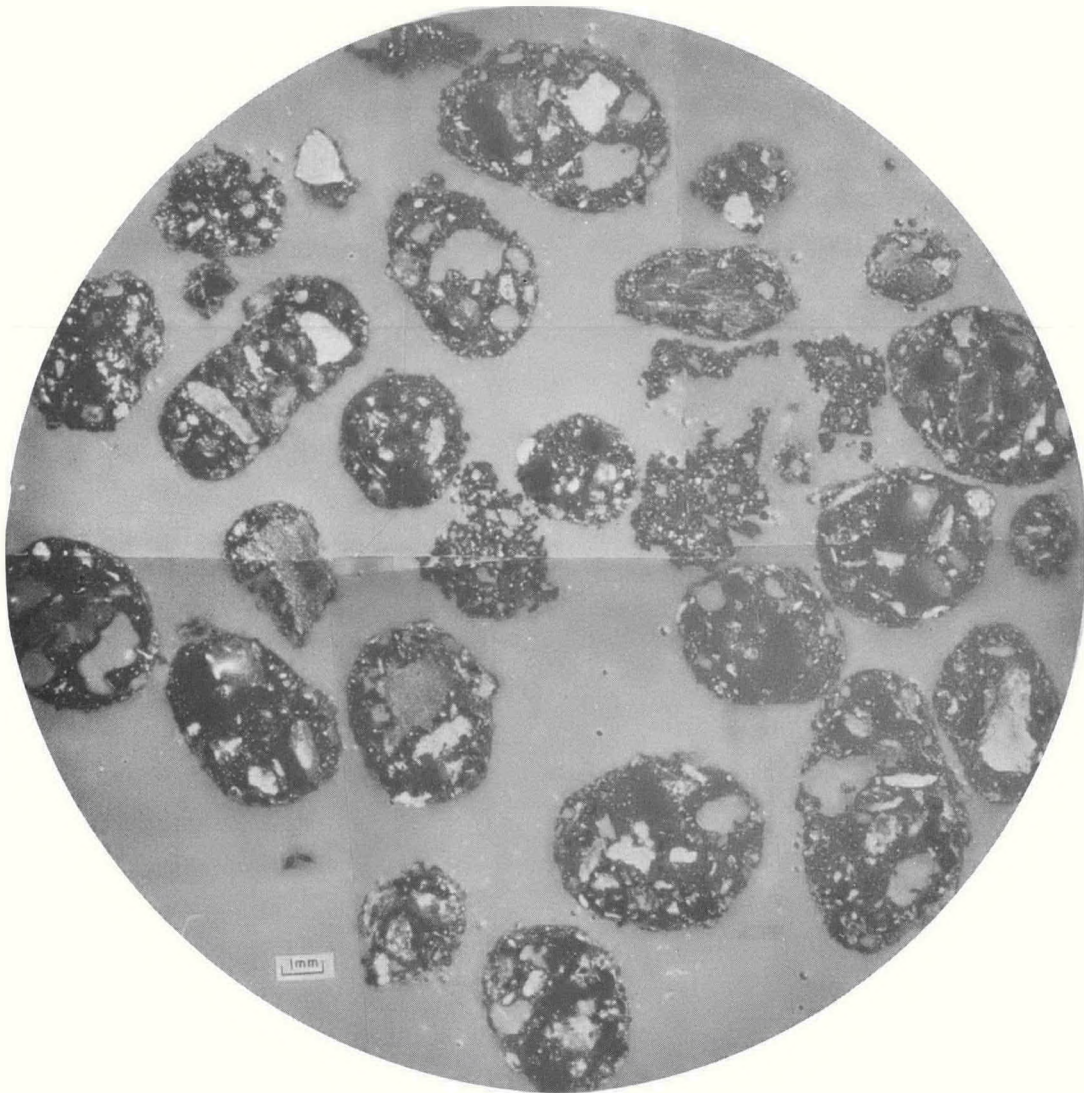
The composition of the beads, particularly the glassy phase, was investigated. Microscopic observations of polished sections (at higher magnification than shown in Figures 109 and 110) show the presence of crystals in the glassy phase that probably exsolve during cooling of the beads. The view in the photomicrograph of Figure 111 is typical, but occasional areas are found where the crystals are larger and comprise a larger fraction of the crystal-in-glass composite. A few crystals of ferrous oxide and metallic iron have been found and identified by measuring their reflectance.

X-ray emission spectra of the crystals shown in Figure 111 and obtained with a scanning electron microscope indicate that they are composed of ferrous aluminate, a spinel. Similarly, iron, aluminum, and silica were major components of the matrix or continuous phase; titanium, potassium, and calcium were also detected in it.



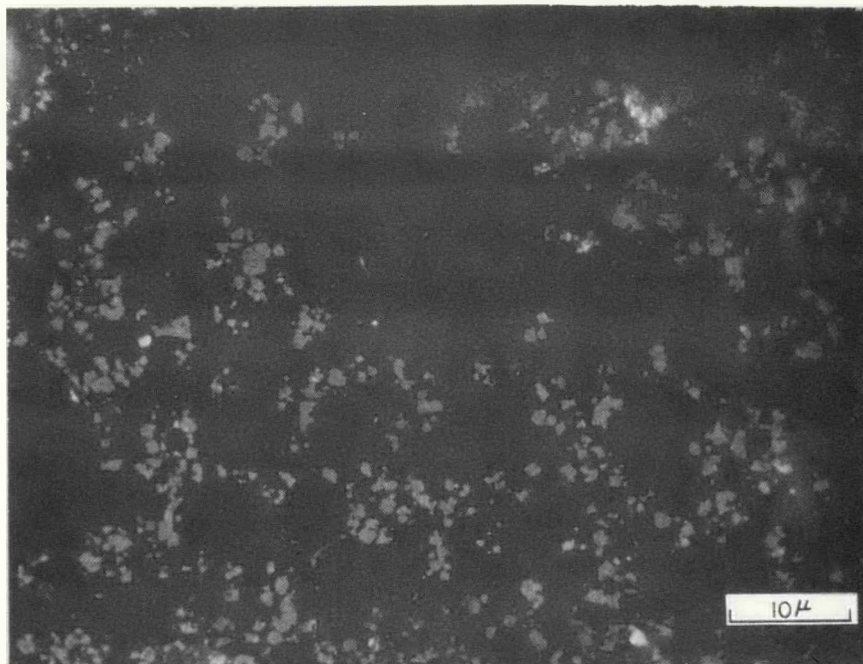
P-114-2025

Figure 109. ASH-AGGLOMERATOR RESIDUE PARTICLES —VERTICAL ILLUMINATION



P-114-2026

Figure 110. ASH-AGGLOMERATOR RESIDUE PARTICLES — OBLIQUE ILLUMINATION



P-114-2023

Figure 111. CRYSTALS OF $\text{FeO} \cdot \text{Al}_2\text{O}_3$ IN MELTED AND RESOLIDIFIED ASH

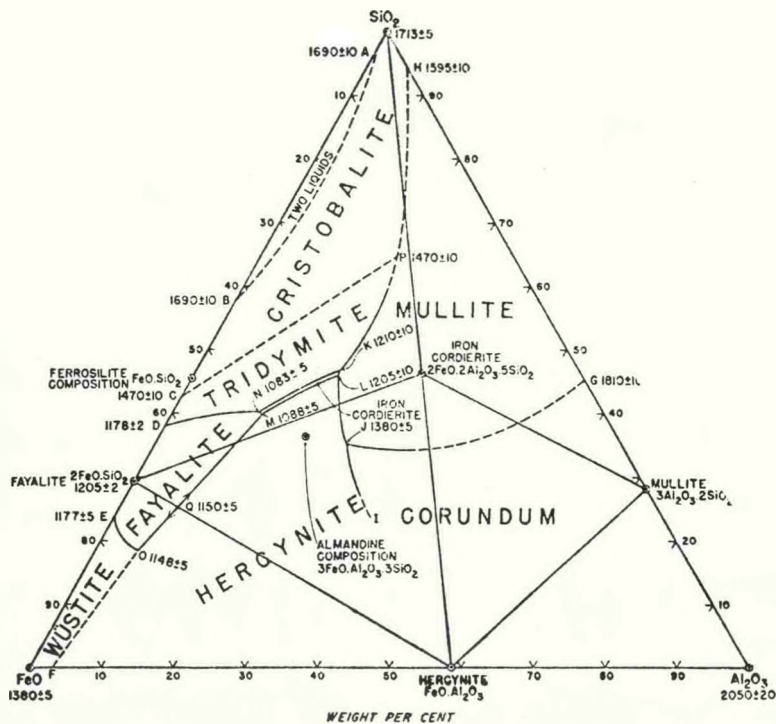


Figure 112. EQUILIBRIUM DIAGRAM OF THE SYSTEM $\text{FeO} \cdot \text{Al}_2\text{O}_3$ - SiO_2 SHOWING FIELDS OF PRIMARY CRYSTALLINE PHASES, TIE LINES, AND INVARIANT POINTS
Source: Schairer, J. F. and Yagi, K., "The System $\text{FeO} \cdot \text{Al}_2\text{O}_3$ - SiO_2 ,"
Am. J. Sci., 471-512 (1952)

An inspection of the phase diagram of the $\text{FeO-Al}_2\text{O}_3\text{-SiO}_2$ system shown in Figure 112 indicates a minimum liquidus temperature of $1990^\circ\text{F} + 9^\circ\text{F}$ at a composition of about 47:13:40 by weight, for equilibrium among fayalite ($2\text{FeO}\cdot\text{SiO}_2$), iron cordierite ($2\text{FeO}\cdot 2\text{Al}_2\text{O}_3\cdot 5\text{SiO}_2$), hercynite ($\text{FeO}\cdot\text{Al}_2\text{O}_3$), and liquid. This diagram is for strongly reducing conditions, i.e., the presence of metallic iron. Even so, about 1% of the iron in the liquid is ferric instead of ferrous. In a slightly more oxidizing atmosphere, the minimum liquidus temperature rises: At an 0.001 oxygen atmosphere, it is about 2471°F .

The presence of other elements as oxides will depress the liquidus temperatures. Analyses of the feed and residue, indicating possible contributors to this effect, in addition to titanium and potassium, are shown in Table 76.

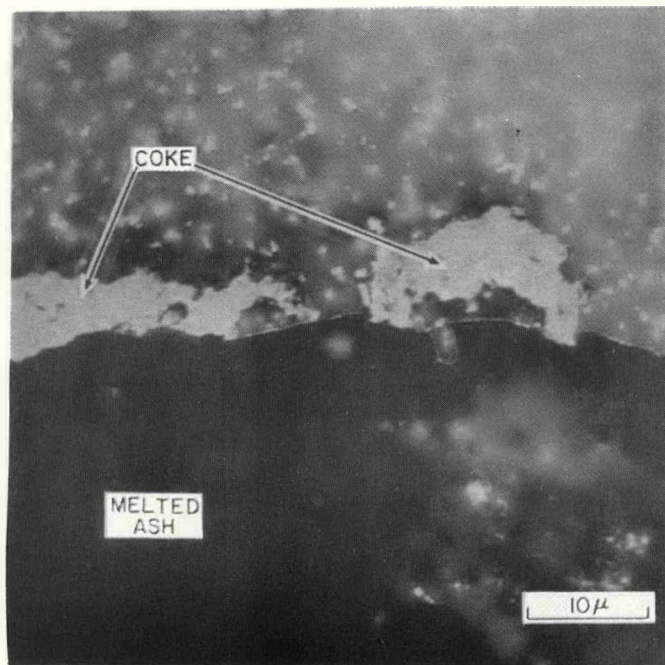
Table 76. ELEMENTAL ASH ANALYSIS

Component	Feed	Residue (Composite)
	wt %	
SiO_2	46.7	45.2
FeO	16.9	20.8
Al_2O_3	20.6	18.5
CaO	3.22	2.39
K_2O	1.69	1.68
MgO	1.00	0.80
TiO_2	0.79	0.65
Zn	--	Minor (spectrochemical)
V	--	Minor (spectrochemical)
Mn	--	Minor-trace (spectrochemical)
B	--	Minor-trace (spectrochemical)
Na	--	Minor-trace (spectrochemical)
Sr	--	Minor-trace (spectrochemical)

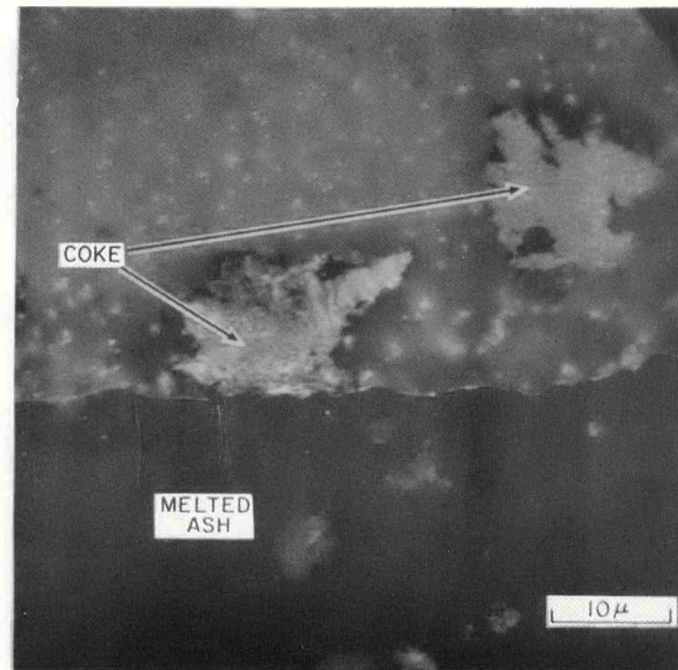
The surface energy of a melt of this kind is high compared with that of carbonaceous solids. For example, the surface tension of lime-soda glass is about 150 dynes/cm; therefore, the melted ash could not be expected to wet the low-energy surface of the coke. This is confirmed by the observation (under the microscope) that coke particles appear to float on the surface of the glassy phase, as shown in the photomicrographs of Figure 113. The observed apparent contact angle between coke and the glassy phase varies from about 90 degrees to much more than that. This explains why so little coke is entrapped inside the melted ash.

Lockhopper Valve Test Stand

By arrangement with Rockwell Mfg. Co., a new valve design, which can potentially serve as lockhopper valves, was prepared for testing. Rockwell assembled the test rig, shown in Figure 114 and IGT set this up with a cyclic feed and discharge system. The unit will be automatically timed and cycled for operating at pressures up to 1000 psig.

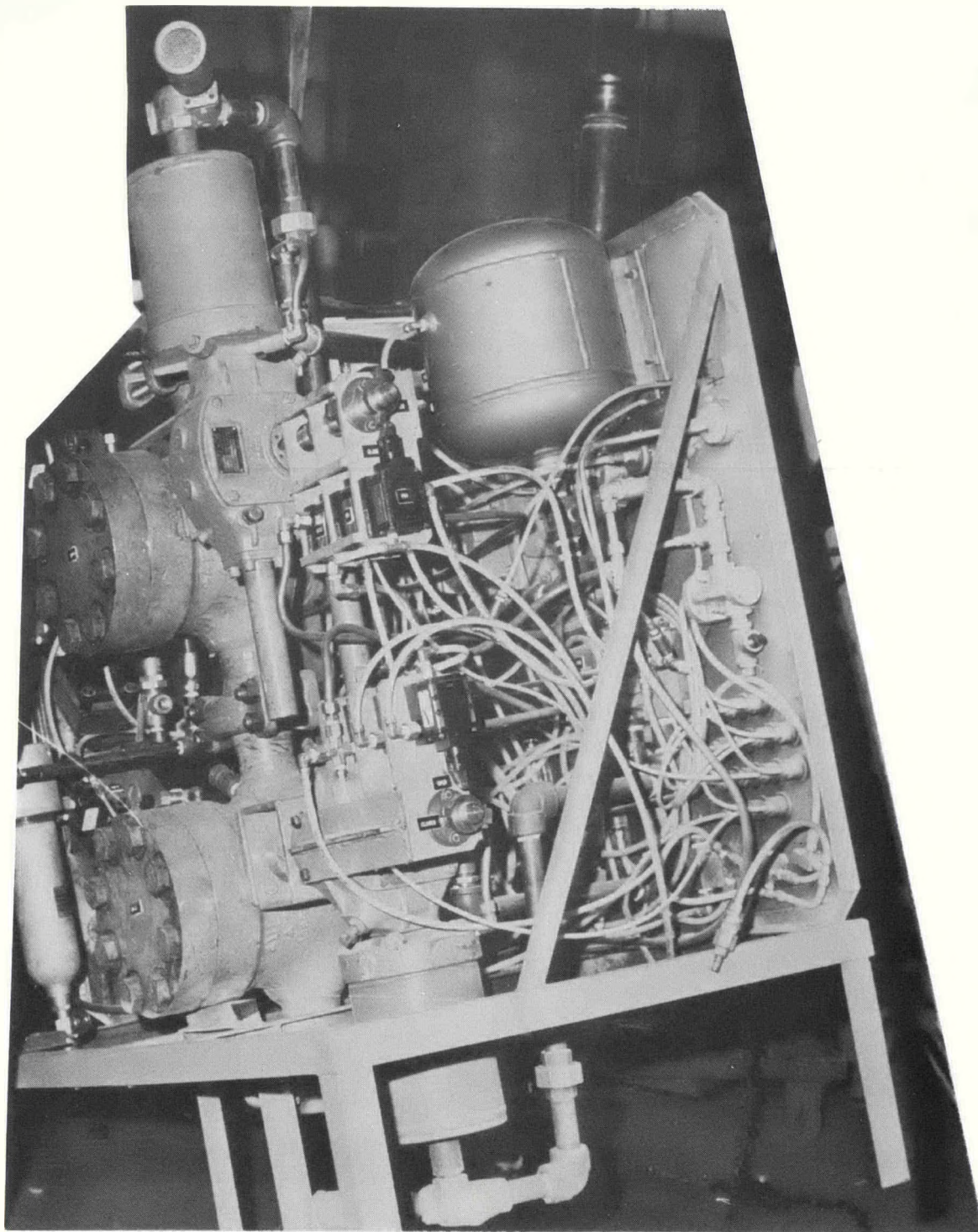


P-114-2024



P-114-2022

Figure 113. COKE PARTICLES ON SURFACE OF MELTED ASH



P-114-2088

Figure 114. LOCKHOPPER VALVE TEST STAND

In November 1974, the test stand for investigating the new lockhopper valve was checked out, and testing was begun. After 280 cycles between 0 and 1000 psig, the valves showed no leaks when pressure-tested. The test will continue to about 10,000 cycles.

By December, the Rockwell lockhopper valve underwent 3500 cycles at pressures between 0 and 1000 psig, with coal being fed through the valves. The top valve showed no leaks so far; the bottom valve showed only a minor leak, which was reduced to one-half by stronger purging of the valve seat.

A total of 4750 cycles, between 1000 psig and atmospheric pressure, of the lockhopper pressurization system were completed by January 1975. Three hundred and fifty of these cycles were made without coal passing through the valves. These cycles were used to check out and time the valve sequence, check the pneumatic controls, and clean and adjust packing of various control valves. The following results of the 4400 cycles with coal were obtained.

- a. No gas leak was found after pressure testing at 30 and 280 cycles.
- b. After 760 cycles, the bottom lock valve had the following leakage rate -

<u>Leakage Rate, SCF/hr</u>	<u>Differential Pressure, psi</u>
38.6	1000
25.2	775
11.2	500
6.8	360
4.0	240

No leak was observed in the other valves.

- c. After 2000 cycles, the top lock valve had a gas leak of 1.6 SCF/hr at 1000 psi. The bottom lock valve leak rate was -

<u>Leakage Rate, SCF/hr</u>	<u>Differential Pressure, psi</u>
41.0	1000
23.8	750
12.6	480
7.6	340
3.8	240

- d. After an additional 1300 cycles, or a total of 3300 cycles with coal, it was decided that the flow of gas purging the seat of the bottom lock valve was insufficient to keep the seat clean, so the gas purge was increased by increasing the gas pressure from 1050 psig to 1100 psig. This resulted in increasing the ΔP for the purge of the bottom valve from 50 to 100 psi, and the ΔP for the top lock valve purge from 1050 to 1100 psi. The gas-vent valve releasing the lockhopper pressure from 1000 psig to atmospheric pressure started to leak after a total of 3565 cycles. At that time, no leak was observed with the top lock valve, and the bottom lock valve leak rate had decreased to -

<u>Leakage Rate, SCF/hr</u>	<u>Differential Pressure, psi</u>
21.8	1000
16.2	750
8.2	500
5.0	350
2.4	250

- e. After replacing the leading gas valve, cycling was continued to a total of 4400 cycles. The leak rate of the top lock valve increased to -

<u>Leakage Rate, SCF/hr</u>	<u>Differential Pressure, psi</u>
10.2	1000
8.6	725
7.4	500
4.8	350
4.6	250

The bottom lock valve increased to -

<u>Leakage Rate, SCF/hr</u>	<u>Differential Pressure, psi</u>
234	1000
187	750
103	500
76	350
49	250

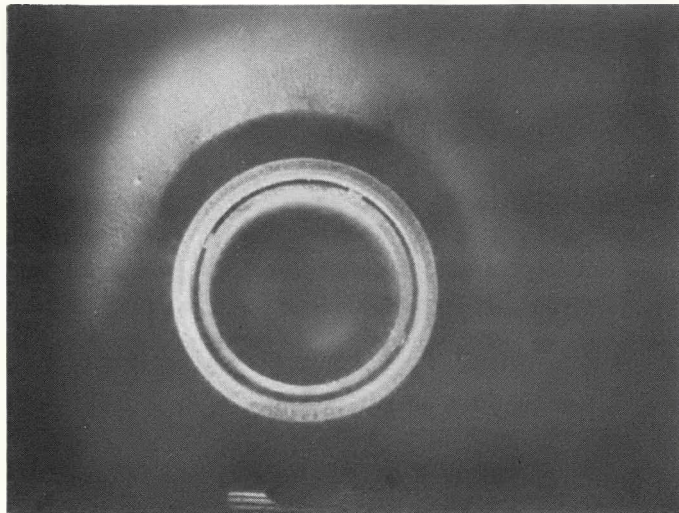
It is possible to operate the system with a leak in the bottom lock valve. The system can also operate with a small leak in the top lock valve, although with difficulty. But it is almost impossible to operate with a leak in the gas-vent valve. The unit was disassembled, the lock valves inspected, and the valves and operation modified before further testing.

The various valves are shown in Figures 115 through 118. Note the pitting on the bottom valve seat in Figure 116 even though the top valve seat was essentially intact. The erosion on the vent valve stem is particularly noticeable in Figure 117.

In March, the valves were disassembled and returned to Rockwell for inspection and modification. By June Rockwell decided to stop further testing of the lockhopper valves. It lapped the valve seats to try to reestablish a tight seal, but found that the task was limited by the size of the valves. As a result, this study was terminated and the valve assembly shipped back to Rockwell.

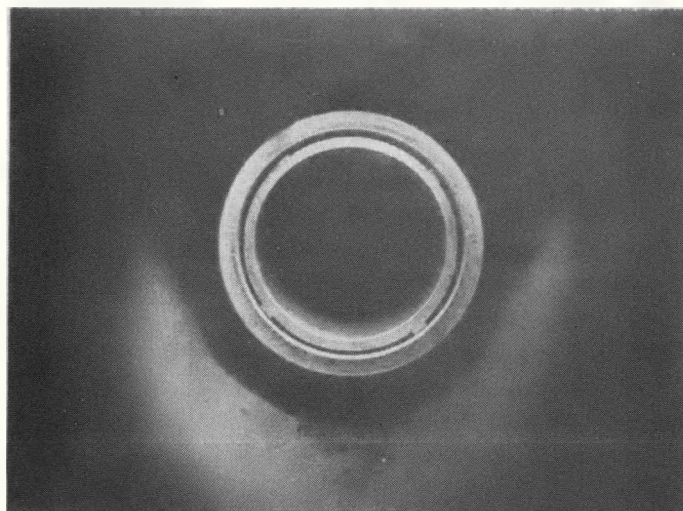
High-Pressure Methanation

The performance of Girdler's G-87 promoted methanation catalyst was further evaluated. The effects of high carbon monoxide and high ethane concentrations in the feed on the methanation, water-gas shift, and hydrogenation



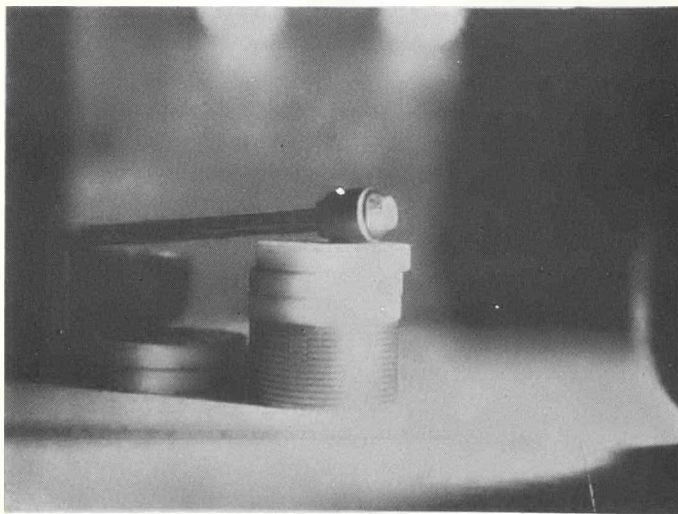
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Figure 115. TOP LOCK VALVE SEAT AFTER 4400 SOLIDS-FEEDING CYCLES



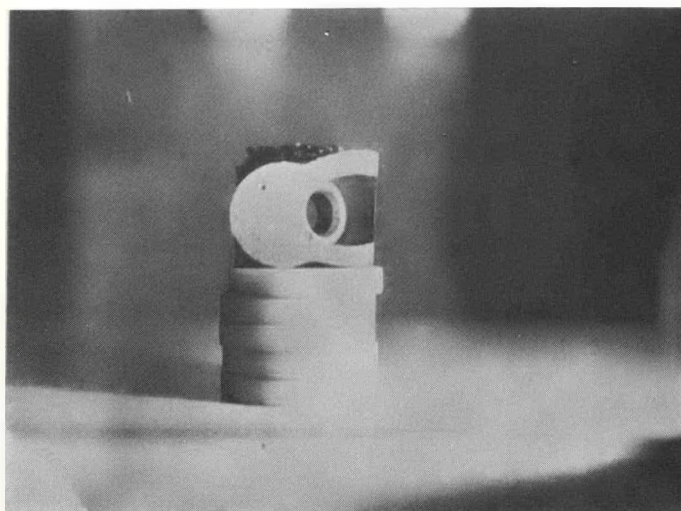
P75010182

Figure 116. BOTTOM LOCK VALVE SEAT AFTER 4400 SOLIDS-FEEDING CYCLES



P75010183

Figure 117. GAS-VENT VALVE STEM AFTER 3565 CYCLES



P75010184

Figure 118. GAS-VENT VALVE SEAT AFTER 3565 CYCLES

reactions were studied, and the results are presented in Table 77. Carbon monoxide, carbon dioxide, and ethane were all hydrogenated at the conditions studied here, and the water-gas shift reaction was not a competing reaction and negligible in extent. Because deactivation occurred during resistance tests (Tests 363-c and 364-c) the upper limit of the carbon monoxide concentration was suspected to be about 10 mole percent. Girdler Chemicals, Inc., analyzed the spent catalyst to determine the cause of this deactivation.

The data collected on the G-87 catalyst were analyzed and are presented in Figures 119, 120, and 121. The rate of carbon monoxide conversion was used instead of the rate of methane formation in this analysis because the latter includes the rate of ethane hydrogenation and that of carbon dioxide hydrogenation. The order of carbon monoxide and that of hydrogen were 0.26 and 0.22 as shown in Figures 119 and 120. The performance of the G-87 promoted catalyst can be described by the expression --

$$-r_{CO} = \frac{k P_{CO}^{0.26} P_{H_2}^{0.22}}{1 + 0.05 P_{CH_4}}$$

where

$-r_{CO}$ = the rate of carbon monoxide

k = the rate constant

P_i = partial pressure of component i .

Figure 120 indicates the fit of the experimental data to this equation.

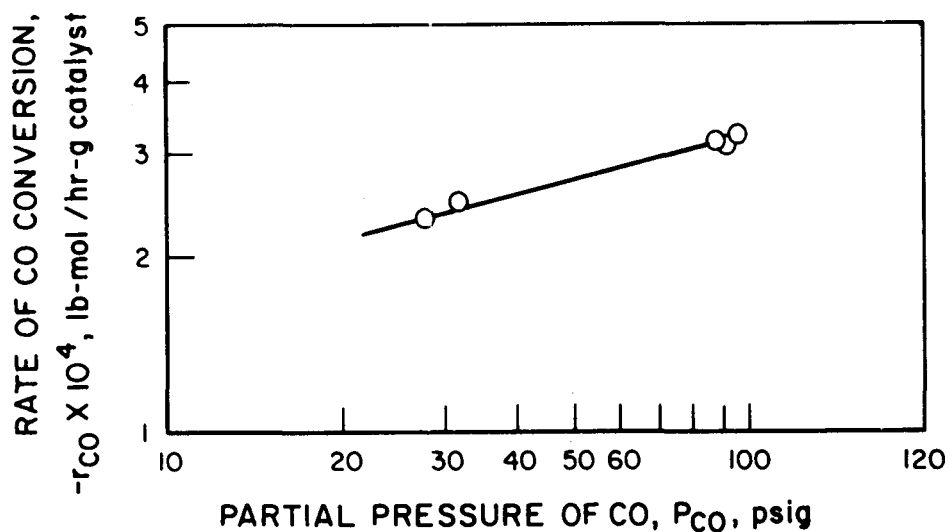
The Laporte-Davison methanation catalyst, LDI-X825, was also evaluated, and the experimental results are presented in Table 78. This catalyst was very active, but the upper temperature limit was a bit low, as shown in Figure 122. The spent catalyst was analyzed by Laporte-Davison, Inc., to determine the change in its physical properties. The initial report from Laporte-Davison was that the batch of catalyst submitted was from a bad lot. Recent experimental work by Laporte-Davison confirmed the poor upper temperature limit that had been determined. The experimental data were analyzed and were found to fit the equation developed in previous work, Figure 123.

In February, the Laporte-Davison methanation catalyst, LDI-X825, which had been tested previously and was suspected of being a bad batch, was checked by the steam deactivation method. The nickel surface area was nearly completely lost, having fallen from the original 25 sq m/g to less than 1 sq m/g. This loss of surface area showed the instability of this lot of catalyst. Laporte-Davison has given assurance that no other batch of catalyst behaves this way. Figure 124 shows the behavior of this bad batch of catalyst before and after steaming, while Figure 125 shows the normal behavior of LDI catalyst. The difference is obvious and striking.

Table 77. METHANATION DATA — CATALYST EVALUATION
(Girdler G-87 Promoted Catalyst, 1/8-Inch Extrusions 4.0584 g)

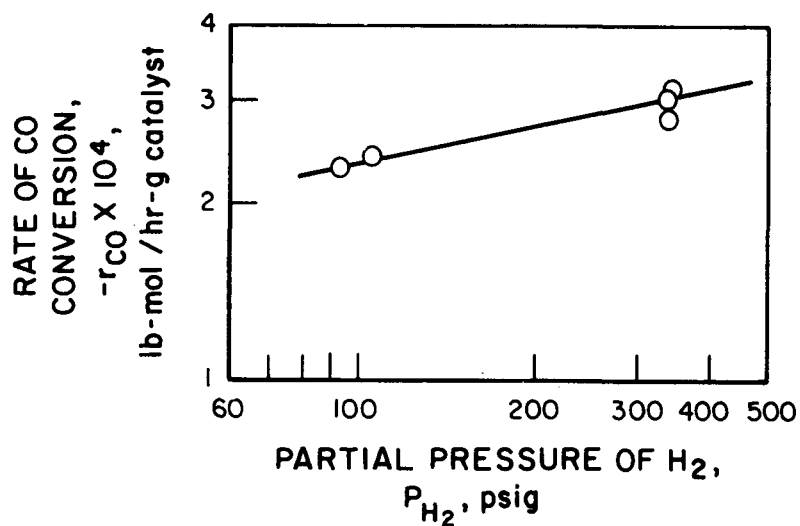
Run No.	363-a	363-b	363-c	364-a	364-b	364-c	365
Time, hr	4	76	100	5	24	96	4
Feed Gas Rate, SCF/hr	15.6699	9.4388	9.4388	9.4266	13.9366	5.7522	5.8573
Feed Gas Composition, mol %							
Nitrogen	2.2	2.3	2.3	2.3	2.4	2.0	1.5
Helium	0.05	0.05	0.05	0.05	0.05	0.03	0.05
Carbon Monoxide	8.9	8.8	8.8	9.1	8.8	10.4	13.4
Carbon Dioxide	2.8	2.7	2.7	2.8	2.8	2.2	1.8
Hydrogen	33.2	36.0	36.0	34.7	34.1	55.9	53.25
Methane	50.25	47.65	47.65	48.45	49.25	27.36	28.3
Ethane	2.6	2.5	2.5	2.6	2.6	2.1	1.7
Propane	--	--	--	--	--	0.01	--
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Reactor Temperature, °F	590	588	588	588	580	587	685
Reactor Pressure, psig	1007	1000	1000	1005	1002	1000	1002
Product Gas Rate, SCF/hr	13.7560	8.9417	9.4388	7.776	11.6208	4.7966	5.8573
Product Gas Composition, mol %							
Nitrogen	2.4	2.4	2.3	2.8	2.7	2.4	1.5
Helium	0.06	0.05	0.05	0.06	0.06	0.04	0.05
Carbon Monoxide	7.0	7.5	8.8	4.8	6.4	6.8	13.4
Carbon Dioxide	2.7	2.7	2.7	2.8	3.3	2.0	1.8
Hydrogen	26.1	31.1	36.0	20.4	20.44	47.04	53.25
Methane	59.04	53.65	47.65	66.14	64.0	39.9	28.3
Ethane	2.7	2.6	2.5	3.0	3.1	1.8	1.7
Propane	--	--	--	--	--	0.02	--
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Feed H ₂ /CO Ratio	3.73	4.09	4.09	3.81	3.88	5.38	3.97
Water Collected, lb-mol /hr	0.000015	0.00031	--	0.00032	0.00037	0.00049	--
Rate of CO Conversion, lb-mol /hr-g catalyst X 10 ⁴	2.60	1.05	0.0	3.19	3.15	1.81	--

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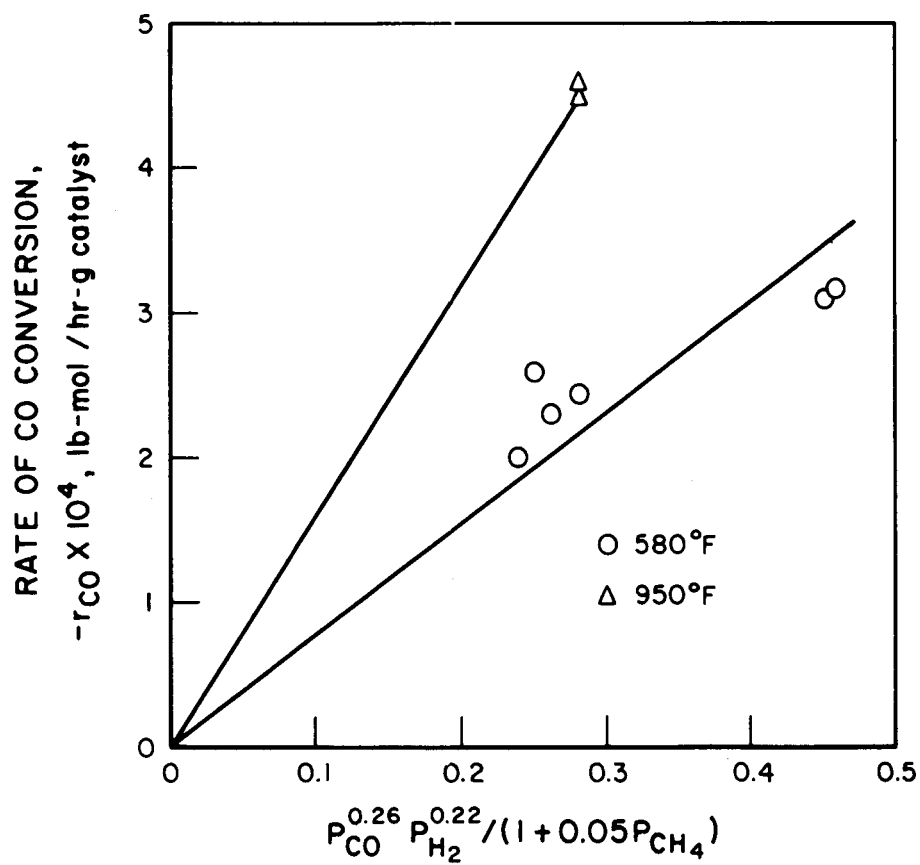
A-124-2278

Figure 119. RATE OF CARBON MONOXIDE CONVERSION AS A FUNCTION OF THE PARTIAL PRESSURE OF CARBON MONOXIDE AT 580°F (G-87 Promoted Methanation Catalyst)



A-124-2277

Figure 120. RATE OF CARBON MONOXIDE CONVERSION AS A FUNCTION OF THE PARTIAL PRESSURE OF HYDROGEN AT 580°F (G-87 Promoted Methanation Catalyst)



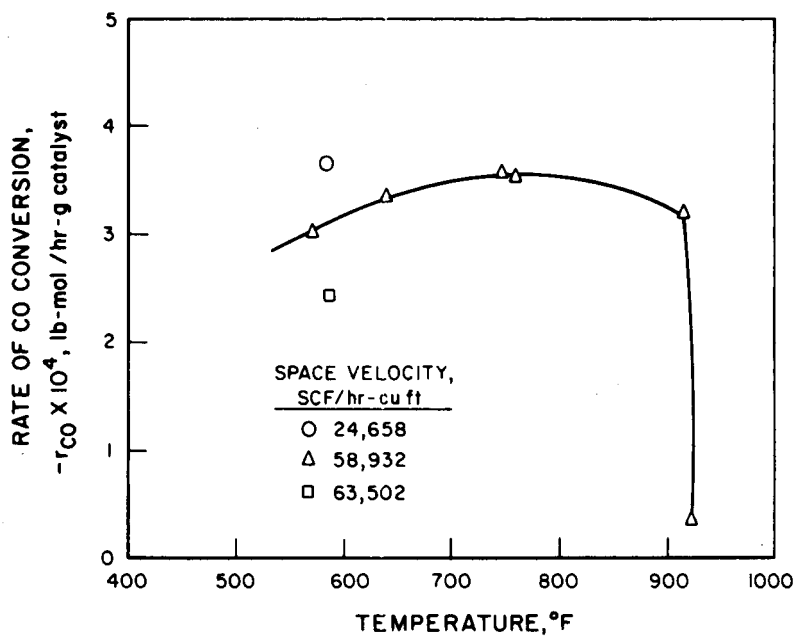
A-124-2275

Figure 121. RATE DATA ANALYSIS FOR THE G-87 PROMOTED METHANATION CATALYST

Table 78. METHANATION DATA — CATALYST EVALUATION
(Laporte-Davison LDI-X825 Catalyst, 1/8-Inch Pellets, 4.0101 g)

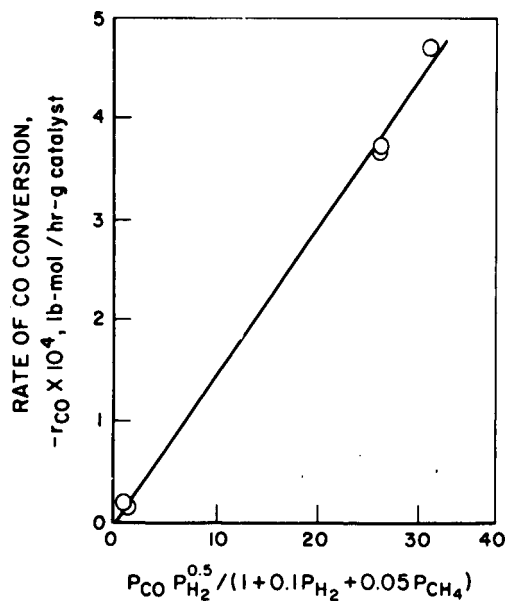
Run No.	366-a	366-b	366-c	366-d	366-e	366-f	366-g	366-h	366-i
Time, hr	4	96	120	143	145	147	167	170	267
Feed Gas Rate, SCF/hr	9.4362	13.4743	3.9600	9.4643	9.4643	9.4643	10.1982	10.1982	5.8335
Feed Gas Composition, mol %									
Nitrogen	2.5	2.7	2.0	2.4	2.4	2.4	2.8	2.8	1.7
Helium	0.05	0.07	0.04	0.04	0.04	0.04	0.04	0.04	0.04
Carbon Monoxide	10.0	8.8	13.0	8.9	8.9	8.9	9.0	9.0	11.3
Carbon Dioxide	2.7	3.0	1.7	2.9	2.9	2.9	2.9	2.9	1.5
Hydrogen	34.4	28.6	51.7	32.06	32.06	32.06	31.66	31.66	55.96
Methane	47.85	54.03	29.96	50.9	50.9	50.9	50.7	50.7	27.9
Ethane	2.5	2.8	1.6	2.8	2.8	2.8	2.9	2.9	1.6
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Reactor Temperature, °F	597	596	594	573	640	745	752	918	907
Reactor Pressure, psig	1004	1003	997	1004	1004	1004	1000	1000	998
Product Gas Rate, SCF/hr	7.2712	11.6285	2.7731	7.8193	7.6859	7.5052	8.2502	8.5861	5.6513
Product Gas Composition, mol %									
Nitrogen	3.2	3.4	2.8	2.9	3.0	3.0	3.5	3.3	1.7
Helium	0.06	0.08	0.06	0.05	0.05	0.05	0.05	0.05	0.05
Carbon Monoxide	3.0	5.3	5.5	4.9	4.2	3.9	4.6	4.9	10.7
Carbon Dioxide	3.8	3.3	1.8	2.7	2.8	2.5	2.5	3.1	1.7
Hydrogen	14.0	15.2	31.0	16.35	15.05	13.15	13.95	17.65	54.65
Methane	73.64	70.62	57.44	71.0	72.8	75.3	73.4	69.0	29.6
Ethane	2.3	2.1	1.4	2.1	2.1	2.1	2.0	2.0	1.6
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Feed H ₂ /CO Ratio	3.44	3.25	3.98	3.60	3.60	3.60	3.52	3.52	4.95
Water Collected, lb-mol /hr	0.00098	0.00140	0.00060	0.00058	0.00058	0.00058	0.00058	0.00058	0.00058
Rate of CO Conversion, lb-mol /hr-g catalyst X 10 ⁴	4.71	3.69	2.35	2.99	3.34	3.54	3.48	3.19	0.34

B-124-2287



A-124-2276

Figure 122. EFFECT OF TEMPERATURE ON THE ACTIVITY OF METHANATION CATALYST LDI-X825



A-124-2274

Figure 123. RATE DATA ANALYSIS FOR METHANATION CATALYST LDI-X825

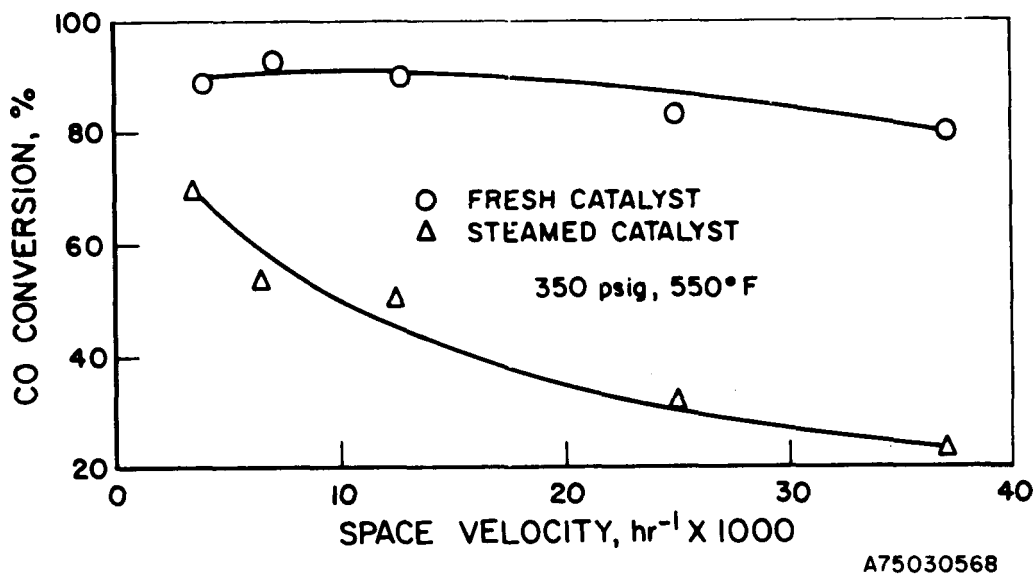


Figure 124. COMPARISON OF THE ACTIVITIES OF THE FRESH AND STEAM-DEACTIVATED LDI-X825 METHANATION CATALYSTS

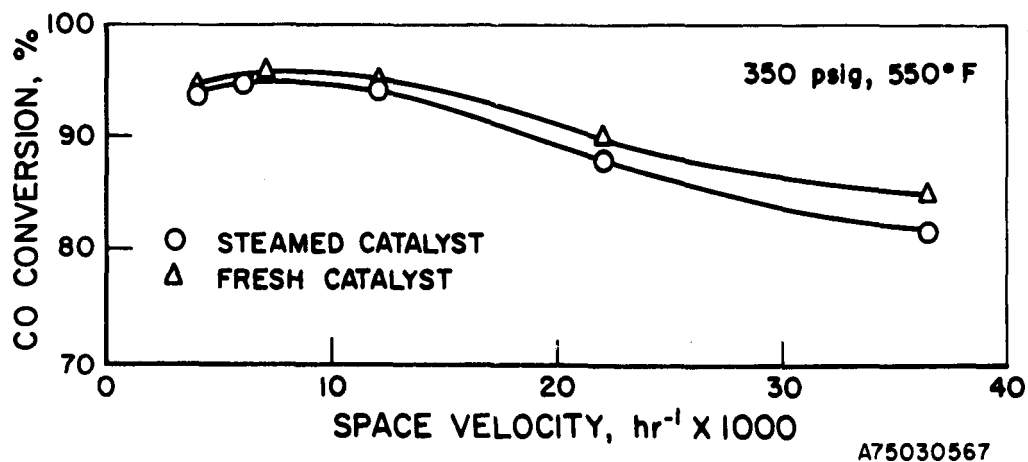


Figure 125. COMPARISON OF THE ACTIVITIES OF A NORMAL LDI METHANATION CATALYST BEFORE AND AFTER STEAMING

Catalysts and Chemicals, Inc.'s, methanation catalyst, C150-4-03, was also evaluated. The results are presented in Tables 79 and 80 and in Figure 126. It has high activity at low temperatures, but a relatively low upper temperature limit of less than 878°F (Runs 368-e and 368-f). The catalyst was inadvertently exposed to 100% carbon monoxide after Run 367-e and deactivated. Attempts to rejuvenate it failed (Runs 367-f to 367-i). The spent catalysts from Runs 363, 364, and 369 were analyzed, and a high sulfur content was found in all cases. Some of the feeds used were probably not up to specifications. Charcoal beds were installed to avoid sulfur poisoning in future experiments.

The temperature effect on Girdler's G-87 promoted methanation catalyst was studied; the results are presented in Tables 81 and 82.

Analyzing the data obtained (Figure 127) from Runs 360, 361 (OCR Report No. 114, March 1974), 362 (OCR Report No. 115, April 1974), 363, 364, 365 (OCR Report No. 122, November 1974), 369, and 370 (ERDA Report No. 125, February 1975) produced the following rate of expression:

$$r = \frac{k P_{\text{CO}}^{0.26} P_{\text{H}_2}^{0.22}}{1 + 0.05 P_{\text{CH}_4}}$$

where

r = rate of CO conversion, lb-mol /hr-g $\times 10^{-4}$

k = $165 \exp (-3380/T)$ = rate constant

P = partial pressure, psig

T = temperature, °R.

An average deviation of 2.6% was found when the calculated rates were compared with the experimental values. This rate expression applies for —

- Temperatures from 550°F to 925°F
- Pressures up to 1000 psig
- An H_2/CO ratio in the feed gas between 2.85 and 4.0
- Ethane and propane in the feed gas being fully hydrogenated.

Five different batches of the G-87P catalyst were used for the study. The variation among the batches and the strong effect of temperature on the rate, combined with the imperfection in the proposed rate expression, caused the scatter in the data in Figure 127. In Figure 128 each experimental point was plotted together with the average values at each temperature level. The activation energy was thus determined to be 6730 Btu/lb-mol (3.71 kcal/g-mol), which was included in the rate expression above. The low value of the activation energy shows that the methanation reaction is diffusion controlled.

Table 79. METHANATION DATA — CATALYST EVALUATION (Catalysts and Chemicals, Inc., CCI C150-4-03 Catalyst; 1/8 x 1/8-Inch Pellets; 4.0143 g)

7-76

Run No.	367-a	367-b	367-c	367-d	367-e	367-f	367-g	367-h	367-i
Time, hr	2	3	6	73	78	98	146	170	245
Feed Gas Rate, SCF/hr	9.1486	9.1486	9.1486	9.7015	9.7015	9.9940	9.1654	5.1742	9.1944
Feed Gas Composition, mol %									
Nitrogen	2.5	2.5	2.5	2.2	2.2	3.3	2.9	2.6	2.7
Helium	0.05	0.05	0.05	0.02	0.02	0.02	0.05	0.05	0.04
Carbon Monoxide	8.4	8.4	8.4	9.8	9.8	6.8	8.2	7.3	10.0
Carbon Dioxide	2.2	2.2	2.2	1.6	1.6	3.2	2.6	2.6	2.6
Hydrogen	41.25	41.25	41.25	54.98	54.98	26.9	32.45	40.25	29.16
Methane	43.6	43.6	43.6	29.9	29.9	56.28	51.3	44.9	53.1
Ethane	2.0	2.0	2.0	1.5	1.5	3.5	2.5	2.3	2.4
Propane	--	--	--	--	--	--	--	--	--
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Reactor Temperature, °F	566	589	588	568	599	580	579	580	579
Reactor Pressure, psig	1003	1003	1003	1004	1004	1005	500	499	802
Product Gas Rate, SCF/hr	6.6204	6.7672	6.8931	6.8604	6.8527	9.5113	8.9242	4.9390	9.1944
Product Gas Composition, mol %									
Nitrogen	3.3	3.2	3.2	3.3	3.1	3.4	3.0	2.7	2.7
Helium	0.07	0.07	0.07	0.03	0.03	0.02	0.05	0.05	0.04
Carbon Monoxide	2.1	1.8	2.2	3.3	3.3	6.0	7.8	6.4	10.0
Carbon Dioxide	2.2	2.1	2.1	1.6	1.6	3.4	2.6	2.5	2.6
Hydrogen	21.53	21.63	21.43	31.07	33.47	24.8	31.55	37.65	29.16
Methane	69.0	69.3	69.1	58.8	56.9	59.68	52.4	48.3	53.1
Ethane	1.8	1.9	1.9	1.9	1.6	2.7	2.6	2.4	2.4
Propane	--	--	--	--	--	--	--	--	--
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Feed H ₂ /CO Ratio	4.91	4.91	4.91	5.61	5.61	3.95	3.95	5.5	2.92
Water Collected, lb-mol /hr	0.00294	0.00294	0.00294	0.00330	0.00330	0.00077	0.00046	0	0
Rate of CO Conversion, X 10 ⁴ lb-mol /hr-g catalyst	4.03	4.18	3.99	4.79	4.87	0.64	0.26	0.38	0

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Table 80. METHANATION DATA — CATALYST EVALUATION (Catalysts and Chemicals, Inc.,
CCI C150-4-03 Catalyst; 1/8 x 1/8-Inch Pellets; 4.0122 g)

7-76

Run No.	368-a	368-b	368-c	368-d	368-e	368-f
Time, hr	4	72	101	147	173	265
Feed Gas Rate, SCF/hr	9.8984	9.3649	9.9705	8.9952	9.8702	9.5192
Feed Gas Composition, mol %						
Nitrogen	3.7	2.6	3.8	3.9	3.9	3.2
Helium	0.09	0.06	0.09	0.13	0.07	0.07
Carbon Monoxide	10.8	11.9	10.4	9.7	9.3	10.9
Carbon Dioxide	2.4	1.7	2.4	2.6	2.7	2.4
Hydrogen	33.21	48.54	34.11	29.37	31.63	33.13
Methane	48.4	34.2	48.4	53.1	50.8	48.4
Ethane	1.4	1.0	0.8	1.2	1.6	1.9
Propane	--	--	--	--	--	--
Total	100.00	100.00	100.00	100.00	100.00	100.00
Reactor Temperature, °F	590	589	585	690	763	878
Reactor Pressure, psig	1003	998	1000	499	1003	999
Product Gas Rate, SCF/hr	8.2691	7.7001	8.0696	7.4532	8.0515	8.8322
Product Gas Composition, mol %						
Nitrogen	4.4	3.2	4.7	4.7	4.8	3.4
Helium	0.1	0.07	0.1	0.15	0.09	0.07
Carbon Monoxide	5.8	7.8	5.3	5.8	5.0	9.5
Carbon Dioxide	2.8	1.9	2.6	2.4	2.5	2.4
Hydrogen	18.8	37.83	18.5	13.95	14.81	28.13
Methane	66.4	48.1	67.8	72.2	72.2	54.6
Ethane	1.7	1.1	1.0	0.8	0.6	1.9
Propane	--	--	--	--	--	--
Total	100.0	100.00	100.00	100.00	100.00	100.00
Feed H ₂ /CO Ratio	3.08	4.08	3.28	3.03	3.40	3.04
Water Collected, lb-mol /hr	0.00145	0.00121	0.00144	0.00191	0.00187	0.00098
Rate of CO Conversion, X 10 ⁴ lb-mol /hr-g catalyst	3.81	3.16	3.98	2.85	3.34	1.30

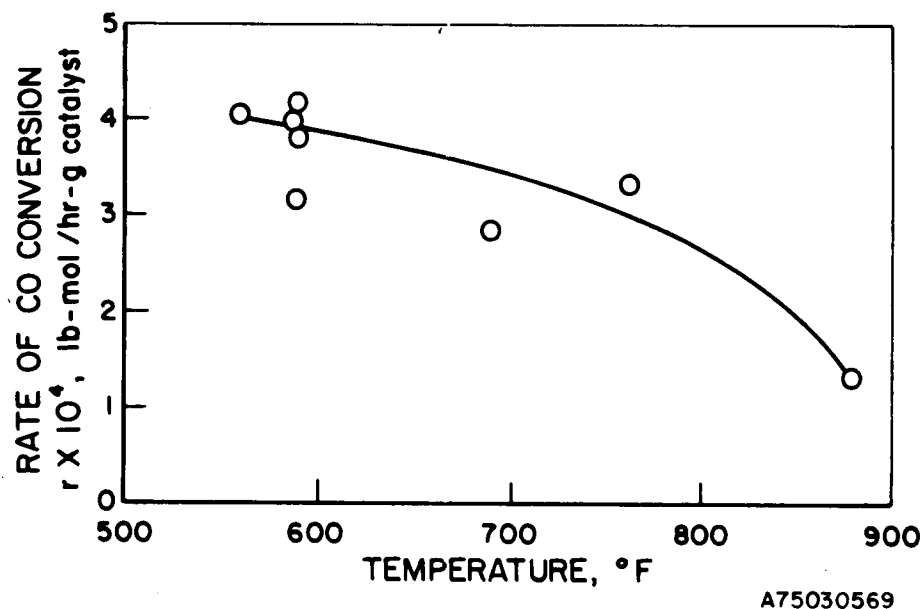


Figure 126. EFFECT OF TEMPERATURE ON THE ACTIVITY OF METHANATION CATALYST C150-4-03 AT 1000 psig

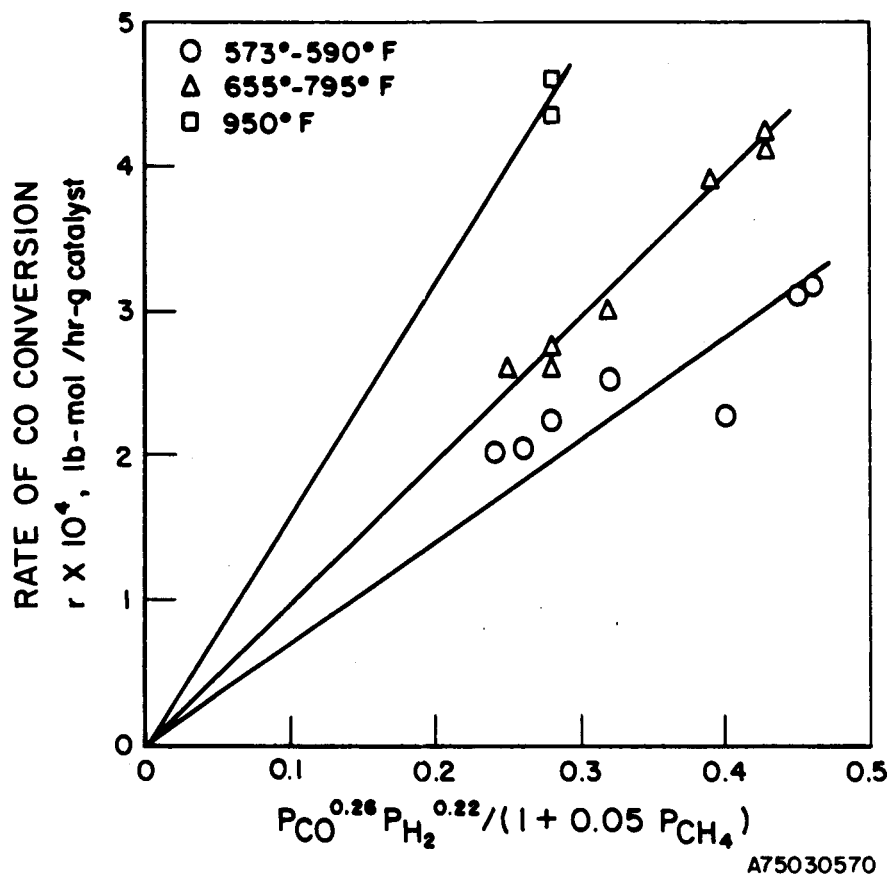


Figure 127. ANALYSIS OF METHANATION DATA FOR G-87 PROMOTED CATALYST

Table 81. METHANATION DATA — CATALYST EVALUATION (Girdler G-87 Promoted Catalyst; 1/8-Inch Extrusions; 4.0048 g)

7-76

Run No.	369-a	369-b	369-c	369-d	369-e	369-f	369-g
Time, hr	4	40	48	73	158	172	192
Feed Gas Rate, SCF/hr	10.0747	10.4062	10.4062	9.7210	9.3133	9.3133	8.7421
Feed Gas Composition, mol %							
Nitrogen	2.8	3.2	3.2	3.5	2.9	2.9	3.9
Helium	0.08	0.09	0.09	0.1	0.07	0.07	0.09
Carbon Monoxide	5.7	4.9	4.9	8.5	9.2	9.2	5.5
Carbon Dioxide	2.7	3.1	3.1	3.1	2.8	2.8	3.4
Hydrogen	35.92	28.31	28.31	27.9	31.83	31.83	22.01
Methane	50.7	58.0	58.0	54.6	51.1	51.1	62.6
Ethane	2.1	2.4	2.4	2.3	2.1	2.1	2.5
Propane	--	--	--	--	--	--	--
Total	100.00	100.00	100.00	100.0	100.00	100.00	100.00
Reactor Temperature, °F	559	570	751	755	750	790	900
Reactor Pressure, psig	998	1000	1000	1002	1002	1002	500
Product Gas Rate, SCF/hr	9.0462	8.5001	8.3350	7.8076	7.3096	7.4066	8.4428
Product Gas Composition, mol %							
Nitrogen	3.1	3.9	4.0	4.3	3.7	3.6	3.9
Helium	0.09	0.1	0.1	0.12	0.09	0.09	0.09
Carbon Monoxide	2.5	1.4	0.6	2.9	3.0	2.7	4.5
Carbon Dioxide	2.4	2.2	1.6	3.8	3.6	3.5	3.3
Hydrogen	27.81	11.4	8.5	10.38	12.51	11.61	18.21
Methane	61.8	78.6	84.5	76.5	75.6	77.6	67.7
Ethane	2.3	2.4	0.7	2.0	1.5	0.9	2.3
Propane	--	--	--	--	--	--	--
Total	100.00	100.0	100.0	100.00	100.00	100.00	100.00
Feed H ₂ /CO Ratio	6.30	5.77	5.77	3.10	3.46	3.46	4.00
Water Collected, lb-mol /hr	0.00015	0.00256	0.00256	0.00206	0.00196	0.00196	0.00103
Rate of CO Conversion, X 10 ⁴ lb-mol /hr-g catalyst	2.26	2.54	2.98	3.89	4.13	4.24	0.65

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Table 82. METHANATION DATA — CATALYST EVALUATION Girdler G-87 Promoted
Catalyst; 1/8-Inch Extrusions; 4.0098 g

Run No.	370-a	370-b	370-c	370-d	370-e
Time, hr	8	102	132	162	191
Feed Gas Rate, SCF/hr	10.2018	9.4854	9.2882	10.0410	10.3847
Feed Gas Composition, mol %					
Nitrogen	3.2	2.9	3.2	3.2	3.9
Helium	0.1	0.1	0.1	0.1	0.1
Carbon Monoxide	4.2	7.3	3.5	2.6	5.8
Carbon Dioxide	2.3	2.4	2.4	2.4	3.2
Hydrogen	23.2	28.2	11.2	9.1	20.4
Methane	64.9	57.1	77.7	80.5	64.2
Ethane	2.1	2.0	1.9	2.1	2.1
Propane	--	--	--	--	0.3
Total	100.0	100.0	100.0	100.0	100.0
Reactor Temperature, °F	650	700	700	850	875
Reactor Pressure, psig	500	1000	500	1002	1000
Product Gas Rate, SCF/hr	8.9624	7.8488	8.3165	9.2779	8.5565
Product Gas Composition, mol %					
Nitrogen	3.6	3.4	3.6	3.4	4.7
Helium	0.1	0.12	0.1	0.1	0.1
Carbon Monoxide	0.5	4.0	0.3	0.3	0.3
Carbon Dioxide	2.7	2.4	2.7	2.4	3.7
Hydrogen	12.1	12.88	3.7	1.5	3.3
Methane	78.8	75.5	88.0	90.2	85.5
Ethane	2.2	1.7	1.6	2.1	2.2
Propane	--	--	--	--	--
Total	100.0	100.00	100.0	100.0	100.0
Feed H ₂ /CO Ratio	5.5	3.4	3.2	3.5	3.5
Water Collected, lb-mol /hr	0.00103	0.00093	0.00082	0.00112	--
Rate of CO Conversion, X 10 ⁴ lb-mol /hr-g catalyst	2.49	2.43	1.97	1.50	3.75

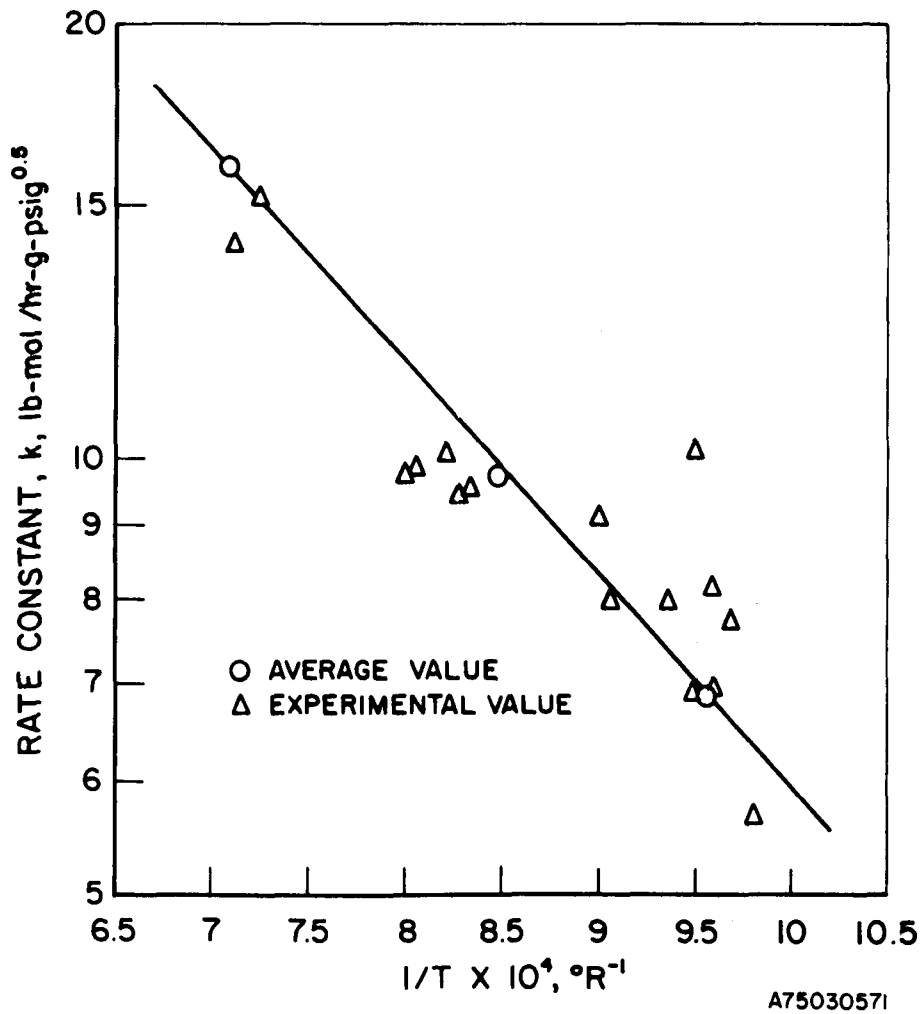


Figure 128. ARRHENIUS PLOT SHOWING THE TEMPERATURE DEPENDENCE OF PROMOTED G-87 METHANATION CATALYST

Toluene Adsorption in Coal

Interest developed in the adsorption of toluene on the coal in the slurry dryer bed because of the possibility of its being transported to the first-stage hydrogasification reactor, where it can be lost by dealkylation to benzene. Adsorption isotherms of toluene were obtained on Montana lignite that had been devolatilized at 600°F and on pretreated Illinois No. 6 coal from the Sahara No. 6 mine, with the results shown in Table 83.

The amount of toluene adsorbed under the conditions in the slurry dryer bed can be estimated from these data by the Polanyi adsorption potential theory, if the composition of the gaseous phase in the pores of the coal is assumed to be equal to that of the product gas leaving the dryer bed. According to this theory, which is applicable when adsorption is dependent on dispersion forces only, the thickness of adsorbate (and thus its liquid volume) is solely a function of the chemical potential term, $RT \ln p_o/p$, where p is the equilibrium pressure (or fugacity) of vapor and p_o is the vapor pressure (or fugacity) of the pure liquid at the adsorption temperature.¹³ The greatest uncertainty in applying this theory is in estimating the proper adsorbate density, especially when the temperature approaches the critical value or is above it. At temperatures equal to or below the normal boiling point, the density is that of the saturated liquid at the temperature at which

Table 83. ADSORPTION ISOTHERMS OF TOLUENE ON LIGNITE AND ILLINOIS NO. 6 COAL

Lignite			Pretreated Illinois No. 6		
Pressure		Amt Adsorbed,	Pressure		Amt Adsorbed,
mm Hg	p/p_o	g/100 g Coal	mm Hg	p/p_o	g/100 g Coal
1.85	0.061	0.295	3.60	0.120	0.287
4.51	0.150	0.423	5.23	0.175	0.346
7.36	0.245	0.536	8.07	0.270	0.420
10.50	0.342	0.658	12.19	0.408	0.506
13.67	0.454	0.768	14.02	0.470	0.586
15.12	0.502	0.850			

the vapor pressure is equal to the adsorption pressure. Various methods of estimating the density have been used at higher temperatures.

Plots of adsorption isotherms with the function $T/V_n \log p_o/p$ as the abscissa are shown in Figure 129. The divisor, V_n , the molar volume at the normal boiling point, is used by some investigators to yield a single correlation curve for the members of a homologous series of hydrocarbons.

The following slurry dryer bed conditions chosen for the estimation were the same as in Test 27 during the period from February 12, 1974, to February 14, 1974: pressure, 996 psig; temperature, 649°F; 21.8 mole percent light oil, calculated as toluene. The value of the correlating function (abscissa in Figure 129) was 1.97, with an adsorbate (toluene) density equal to that of the saturated liquid at a temperature at which the vapor pressure is equal to the partial pressure of toluene in the product gas.

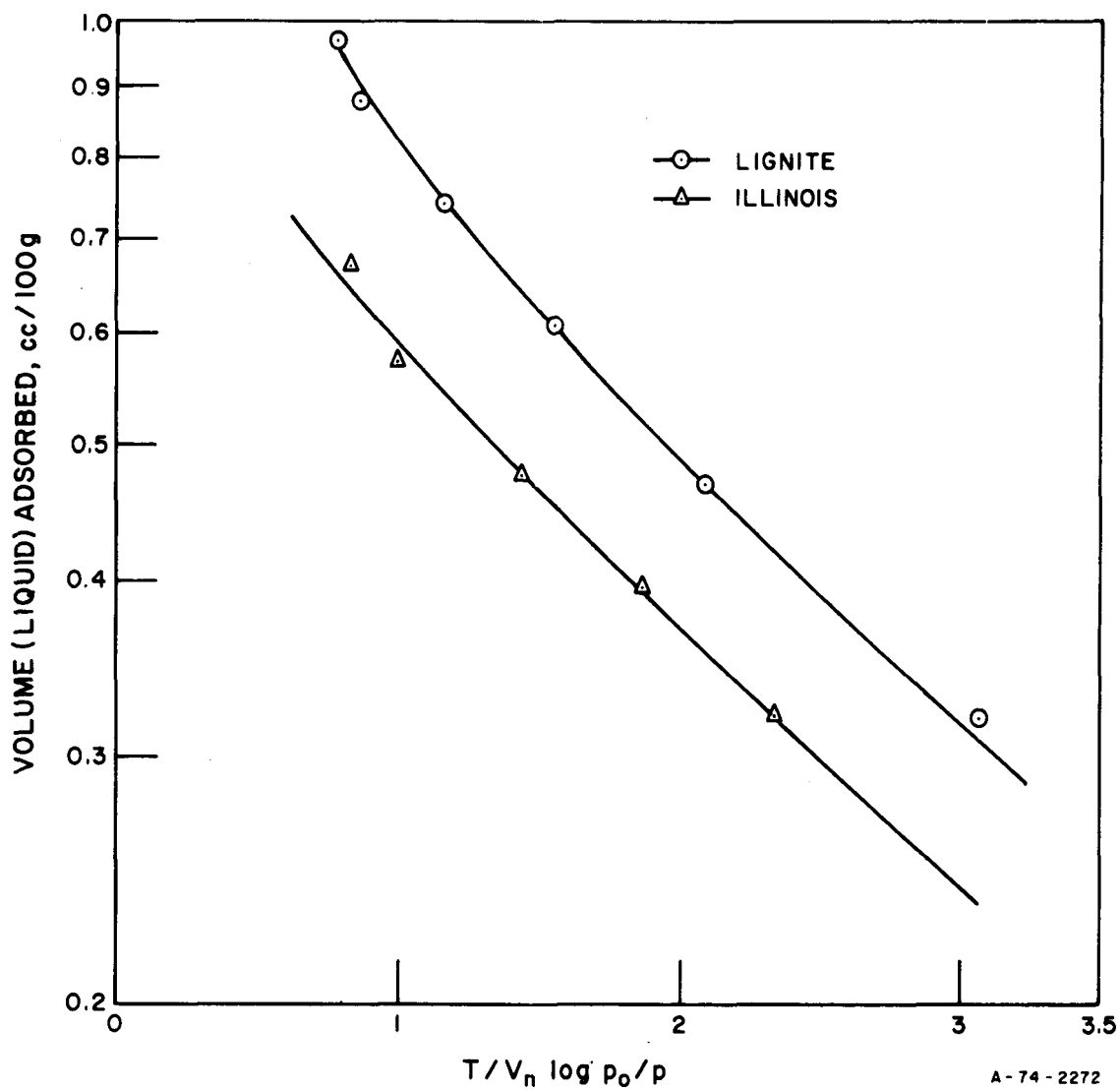


Figure 129. ADSORPTION OF TOLUENE ON MONTANA LIGNITE AND PRETREATED ILLINOIS NO. 6, SAHARA MINE, COAL

The calculated amount of adsorption was 0.30 gram of toluene per 100 grams of lignite and 0.23 gram of toluene per 100 grams of pretreated Illinois No. 6 coal. About 10% greater adsorption is estimated when the adsorbate density is calculated according to the method of Cook and Basmadjian.⁴

The amount of toluene in the gaseous phase in the pores of the coal can also be estimated. The pore volume of the sample of coal from the Sahara No. 6 mine was 0.0734 ml/g on a mineral-matter-free basis, as determined by Gan *et al.*¹¹ The pore volume of the same coal with 13.92 weight percent ash was 0.061 ml/g, and the amount of toluene in the gas phase was estimated to be 0.16 g/100 g of coal. The estimated compressibility factor of the gas was 0.88.

Thus, based on the original assumption that the gaseous phase in the pores of the coal has the same composition as the product gas, the total amount of toluene carried by the coal particles from the slurry dryer into the gasifier is less than 0.5 g/100 g of coal. The expected toluene production is much greater than this, and a net production of toluene should result.

At the other extreme, if one assumes that the pores of the coal contain only toluene, undiluted by product gas, then the amount of toluene in the pores is estimated to be 2.4 g/100 g of coal. With adsorption on the surface, the amount of toluene carried into the gasifier would be even greater. Thus, the diffusion rate through the pores and the residence time provided in the slurry dryer become important parameters. Considering that the coal residence time in the dryer is 10 minutes or more, the assumption of an equilibrated pore gas appears more reasonable.

Slurry Liquid Penetration Into Coal Pores

The penetration of water into the pores of coal when a coal-water slurry is subjected to pressure was investigated. Pretreated coal and dried nonagglomerating coal are expected to contain air or other gas in their pores when they are slurried with water, so that the pores do not fill with water until the slurry is pumped to feeding pressure and the pore gas is compressed to a negligible volume. Thus, a water content adequate for slurrying at atmospheric pressure may be inadequate under the elevated pressure required for feeding to a hydrogasification reactor.

An apparatus and procedure was developed to experimentally determine the amount of water penetration when the coal is subjected to pressure. A schematic diagram of the apparatus is shown in Figure 130. A 1 to 4-gram sample of coal is weighed into the bulb of the two-piece pycnometer. Water is added to cover the coal, and the slurry is stirred to release the air bubbles. The graduated stem is fitted onto the bulb, and more water is added, up to the bottom of the stem. The pycnometer is then rotated to ensure the release of air bubbles, especially with light samples that tend to float. Water is added to bring the slurry up to a reference level at the top of the stem. The pycnometer is weighed and transferred to a Jerguson gage, and readings of water level are taken as gas pressure is applied, in steps, up to about 1000 psig. In some cases, readings were also taken as the pressure was released. With a calibrated pycnometer, the apparent (particle) volume and density can be calculated in addition to the direct determination of the amount of water penetration.

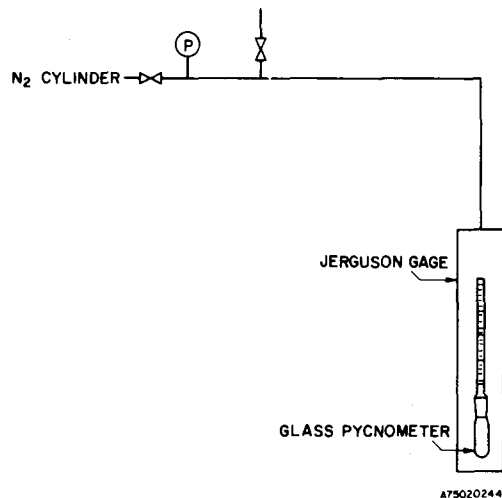


Figure 130. DIAGRAM OF PRESSURE PENETRATION APPARATUS

Tests were made on untreated and pretreated Illinois No. 6 coal (Sahara mine No. 6), on Montana lignite dried to 11.2 weight percent moisture, and on a sample of a Pittsburgh No. 8 seam coal (Montour 4 mine) that was pretreated at about 1000-psig pressure in 1964. Bulk densities and true (helium) densities — the latter on dry samples — were also determined. Results are shown in Table 84.

Volume-pressure readings for a run on the pretreated Illinois No. 6 coal are shown in Figure 131. The behavior can be accounted for by Boyle's law and the compressibility of water, the latter amounting to about 0.42% by volume per 1000 psi.

The true density of the coals on a dry basis was calculated from the apparent density, penetration, and moisture content, as shown in Table 84. Comparisons with helium densities determined on a Beckman pycnometer show good agreement, except for lignite. No reason for the difference is readily apparent. For the other coals it appears that the amount of penetration can be calculated from the true and apparent densities with considerably simplified apparatus and procedure.

The amount of penetration into the raw Illinois No. 6 coal was very small, about 0.01 ml/g of dry coal after correction for compressibility of the water and the coal in the pycnometer; as expected, penetration into the pretreated coal was much greater. This and other volumes of interest for the slurring operation were calculated from the data on the pretreated coal, as follows:

Table 84. DENSITY AND PRESSURE PENETRATION OF COAL

Moisture wt %	Ash (dry basis)	True (Helium) Density (dry basis) g/ml	Bulk Density	Run No.	Apparent (Particle) Density, g/ml	Penetration Test			Calculated True Density (dry basis), g/ml
						Penetration (moist basis)			
						100 psig	500 psig	1000 psig	
Illinois No. 6 Coal, Sahara No. 6 Mine (IGT Lab No. 23832)									
3.92	8.6	1.39	0.82	1	1.30	0.018	0.022	0.028	1.35
Pretreated Illinois No. 6 Coal, Sahara No. 6 Mine (IGT Lab No. 26007)									
3.30		1.46; 1.48	0.50	1	0.72	0.669	0.714	0.725	1.49
				2	0.73	0.626	0.668	0.682	1.42
				3	0.70	0.703	0.753	0.769	1.47
				Avg	0.72	0.666	0.712	0.725	1.46
Montana Lignite, Savage Mine (IGT Lab No. 23147)									
11.2; 10.6	9.9	1.50; 1.49	0.80	1	1.31	0.091	0.106	0.111	1.61
				2	1.34	0.087	0.101	0.107	1.65
				3	1.38	0.061	0.070	0.074	1.61
				Avg	1.34	0.080	0.092	0.097	1.62
Pressure-Pretreated Pittsburgh No. 8 Coal, Montour No. 4 Mine (R-PP-4)									
1.57	10.8	1.42	0.76	1	1.32	0.037	0.043	0.051	1.40
				2	1.32	0.040	0.043	0.047	1.40
				3	1.27	0.059	0.063	0.067	1.39
				4	1.32	0.047	0.052	0.057	1.42
				Avg	1.31	0.046	0.050	0.056	1.40

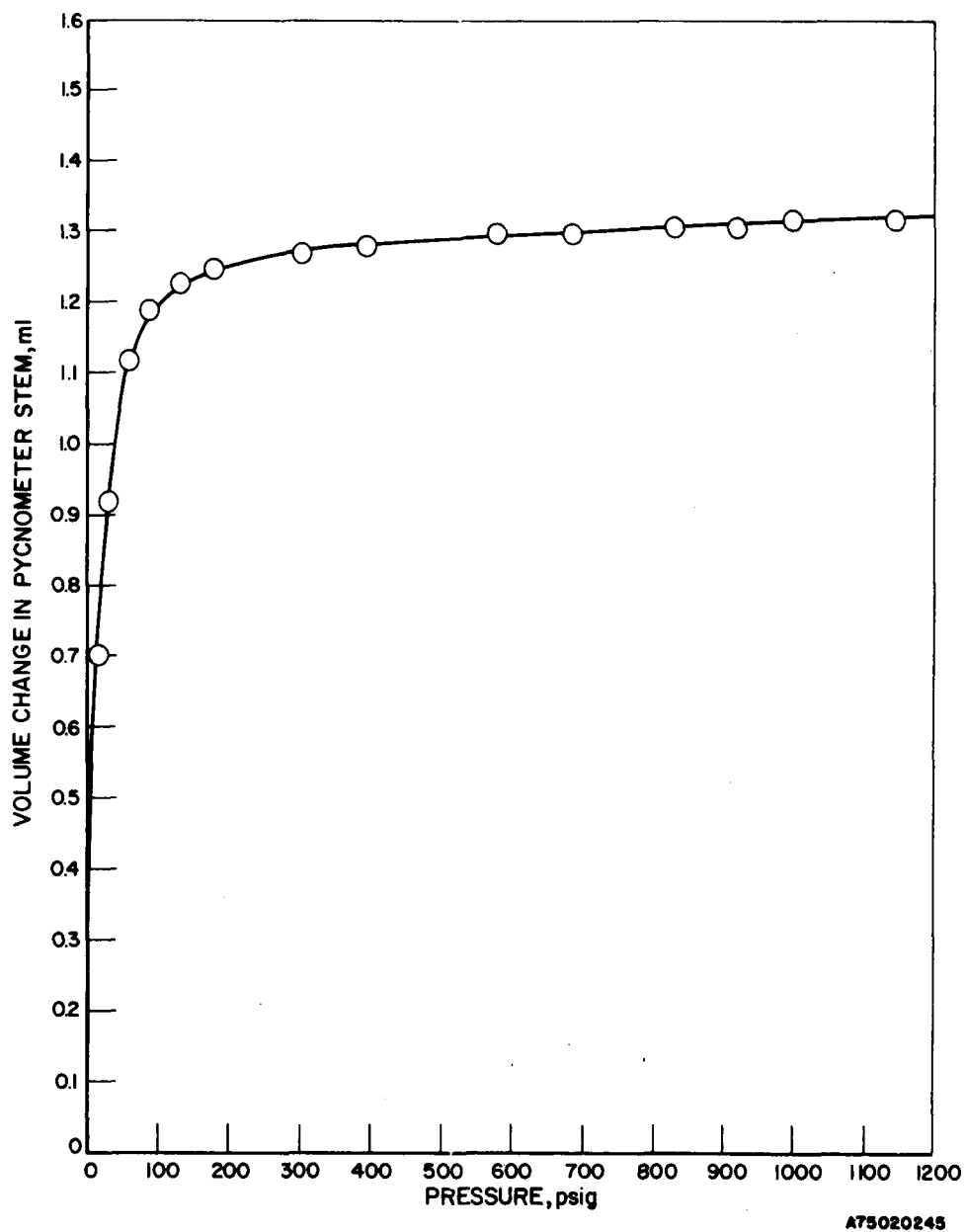


Figure 131. PRESSURE PENETRATION OF 1.712-GRAM SAMPLE OF PRETREATED ILLINOIS NO. 6 COAL

	<u>ml/g of Dry Coal</u>
Volume of Unfilled Pores (Penetration)	0.72
Volume of Solid	0.68
Volume of Moisture	0.03
Total Volume of Particles	1.43
Total Volume of Particles and Interstices	2.07
Volume of Interstices Between Particles	0.64

The volume of interstices is based on the bulk density of the coal measured in air, with the assumption that the coal does not swell when placed in the slurring liquid.

Thus, a volume of 1.45 ml of slurry liquid per gram of dry coal (17 gal/100 lb) must be added before any separation of particles occurs. In correlating slurry properties, the correlating parameter should be the volume of liquid above the amount required to fill pores and interstices.

The potential advantage of pretreatment under pressure is shown by the results on pretreated coal from Run PP-4 made at a 1025-psig pressure and a 725°F average temperature (Project PB-23a May 1964 Report). The product was nonagglomerating when tested in 1964 at a temperature of 1148°F and when tested recently by the regular agglomerating test at 1300°F. Penetration is no more than could be accounted for by the likely pore volume of the raw coal.

Another peculiarity of low-rank coals has come to our attention. Evans has pointed out that brown coal and lignite naturally occur as colloidal gels, that is, the solid is dispersed in a continuous water medium.^{7, 8} As is true of other gels, their volume shrinks as the medium (water) is removed. This is shown for a lignite and a subbituminous C coal by bulk density data obtained by Paulson et al.¹⁸ as these coals were dried. Their data, recalculated to show the loss of volume of a given amount of coal (dry basis) as water is removed, are shown in Figure 132. Except for one point on the subbituminous coal curve, the data show very strikingly the loss of close to 1 milliliter of volume for each gram of moisture removed. However, part of this volume loss may be caused by breakage and slacking.

This effect prompts the question of whether dried lignite swells when placed in water or other slurry liquid, thus increasing the amount of the latter required. The penetration experiments reported here offer no information whatsoever on this point. Other experiments were planned to investigate this question.

Data reported in the January 1975 ERDA Report No. 124 indicated that pretreatment under pressure yielded a product having a substantially higher bulk density (and thus requiring less liquid for slurring) than atmospheric-pressure-treated coal. To provide additional data, a sample of coal that had been pretreated at 1000 psi at the ERDA Pittsburgh Energy Research Center was obtained. Its test data on the original coal, high-volatile C Bituminous from Illinois No. 6 seam, River King mine, and on the pretreatment product are shown in Table 85.

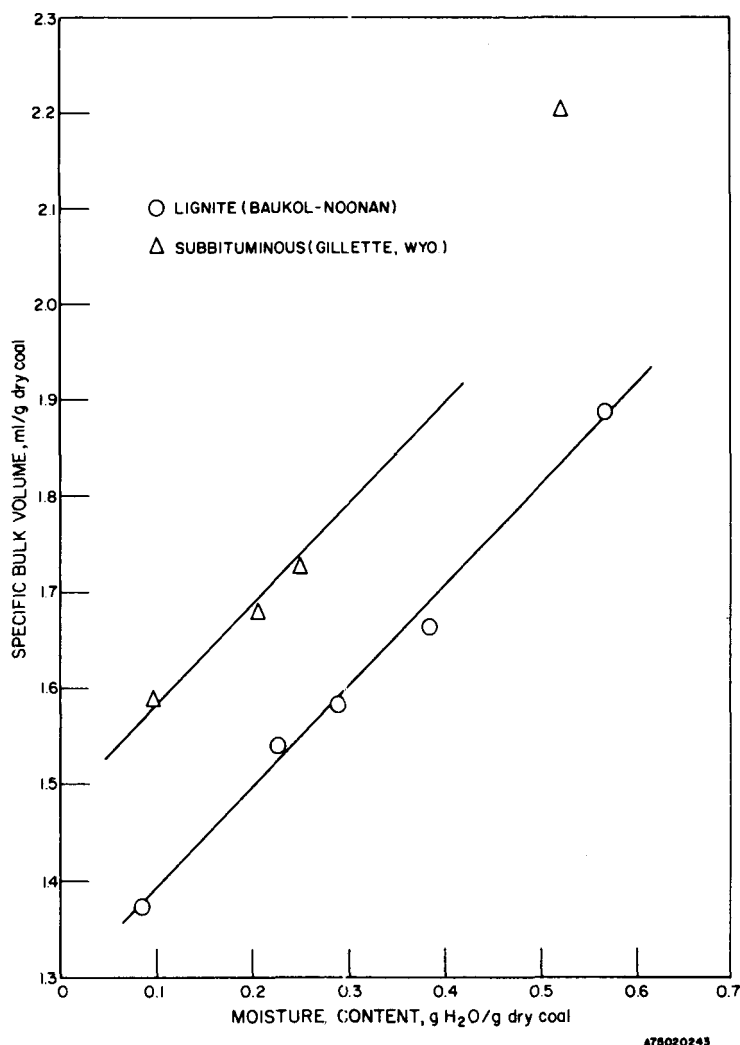


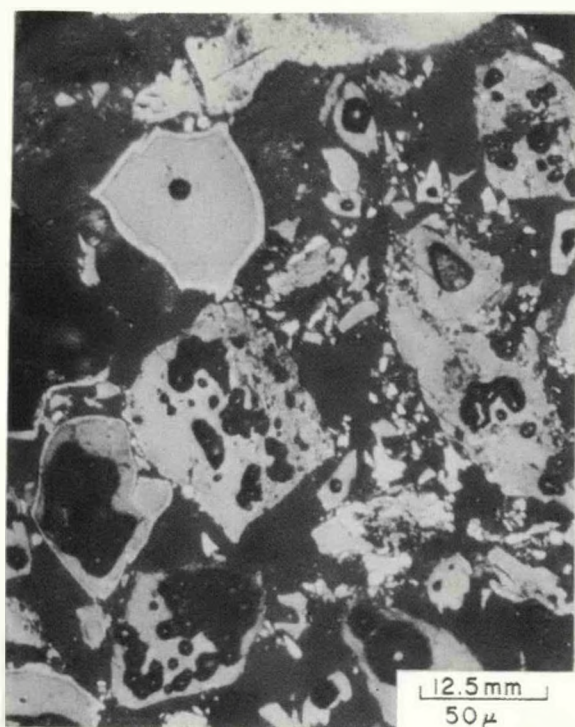
Figure 132. SHRINKAGE OF LOW-RANK COAL WITH DRYING

A petrographic examination of the pretreated coal showed the presence of oxidation rims varying in thickness from particle to particle, Figure 133. Vesicle formation was less extensive than in coal of the same rank pretreated at atmospheric pressure.

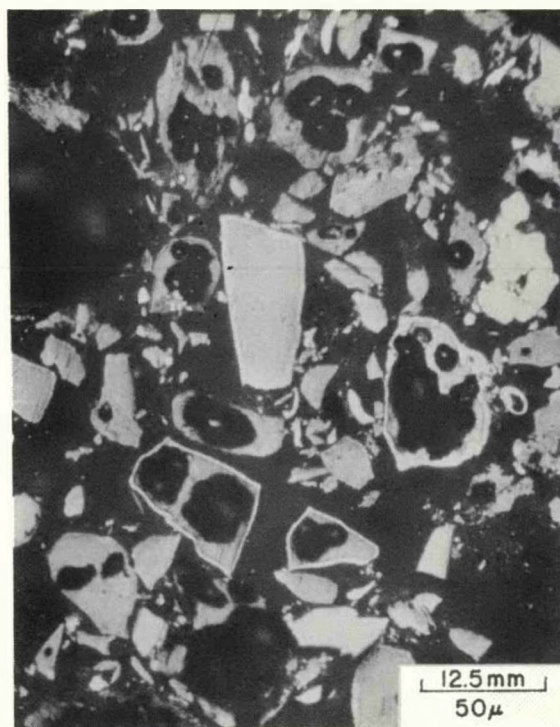
Results of density and penetration tests on this coal are shown in Table 86 together with data previously reported. Volatile matter on these samples has also been added. The bulk density, particle density, and penetration on the Bureau of Mines sample confirm that pressure pretreatment yields a less porous product. Furthermore, the amount of volatile matter in this sample and that in IGT pressure-pretreated sample indicate that less volatile matter is lost in pressure pretreatment than in pretreatment at atmospheric pressure.

Table 85. PITTSBURGH ENERGY RESEARCH CENTER
 TEST DATA ON FEED TO AND PRODUCT FROM
 PRESSURE-PRETREATED ILLINOIS NO. 6 COAL, RIVER KING MINE

<u>Proximate Analysis, wt %</u>	<u>Coal</u>	<u>Pretreated Coal Test 70 PI 6</u>
Moisture	4.6	0.4
Volatile Matter	39.8	29.1
Fixed Carbon	43.1	54.0
Ash	12.5	16.5
<u>Ultimate Analysis, moist basis, wt %</u>		
Carbon	63.4	65.0
Hydrogen	5.0	5.7
Nitrogen	1.2	1.3
Sulfur	3.6	3.9
Oxygen	14.3	7.6
Ash	12.5	16.5
FSI	2.5	--
Bulk Density, lb/cu ft	46.0	41.6
<u>Screen Analysis, wt %</u>		
+20	0.3	0.3
+50	17.2	8.4
+100	18.5	27.0
+140	22.6	19.8
+200	11.1	14.0
+325	12.8	14.2
-325	17.5	16.3



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Figure 133. PHOTOMICROGRAPHS OF COAL AFTER PRESSURE
PRETREATMENT AT THE ERDA PITTSBURGH ENERGY RESEARCH CENTER

Table 86. DENSITY AND PRESSURE PENETRATION OF COAL

Moisture	Volatile Matter (dry basis) wt %	Ash (dry basis)	True (Helium) Density (dry basis) g/ml	Bulk Density	Run No.	Apparent (Particle) Density, g/ml	Penetration Test			Calculated True Density (dry basis), g/ml
							Penetration (moist basis)			
							100 psig	500 psig	1000 psig	
Illinois No. 6 Coal, Sahara No. 6 Mine (IGT Lab No. 23832)										
3.92	37.9	8.6	1.39	0.82	1	1.30	0.018	0.022	0.028	1.35
Pretreated Illinois No. 6 Coal, Sahara No. 6 Mine (IGT Lab No. 26007)										
3.30	23.9	9.3	1.46; 1.48	0.50	1	0.72	0.669	0.714	0.725	1.49
					2	0.73	0.626	0.668	0.682	1.42
					3	0.70	0.703	0.753	0.769	1.47
					Avg	0.72	0.666	0.712	0.725	1.46
Pressure-Pretreated Pittsburgh No. 8 Coal, Montour No. 4 Mine (R-PP-4) ³										
1.57	28.3	10.8	1.42	0.76	1	1.32	0.037	0.043	0.051	1.40
					2	1.32	0.040	0.043	0.047	1.40
					3	1.27	0.059	0.063	0.067	1.39
					4	1.32	0.047	0.052	0.057	1.42
					Avg	1.31	0.046	0.050	0.056	1.40
Bureau of Mines Pressure-Pretreated Illinois No. 6 Coal, River King Mine ⁺										
1.29	29.2	16.5	1.47	0.72	1	0.99	--	--	--	--
				(0.67) ⁺	2	1.08	0.140	0.184	0.213	1.36
					3	1.05	0.189	0.251	0.288	1.39
					Avg	1.04				
Montana Lignite, Savage Mine (IGT Lab No. 23147)										
11.2; 10.6	40.7	9.9	1.50; 1.49	0.80	1	1.31	0.091	0.106	0.111	1.61
					2	1.34	0.087	0.101	0.107	1.65
					3	1.38	0.061	0.070	0.074	1.61
					Avg	1.34	0.080	0.092	0.097	1.62

^{*} Feed coal had 31.7 weight percent volatile matter on a dry basis.

⁺ Feed coal had 41.7 weight percent volatile matter on a dry basis.

^{*} From Table 4, data reported by Pittsburgh Energy Research Center.

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