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FOSSIL ENERGY

PIPELINE GAS FROM COAL-HYDROGENATION (IGT HYDROGASIFICATION PROCESS)

Project 9000 Quarterly Report No. 3, January 1—March 31, 1977

May 1977

Work Performed Under Contract No. EX-76-C-01-2434

Institute of Gas Technology
IIT Center, 3424 S. State Street
Chicago, Illinois 60616

ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION

NOTICE

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SUMMARY

This quarterly report covers work conducted between January 1, 1977 and March 31, 1977. A summary of this work is provided below.

After modifications and improvements to the plant, the first hydrogasification test with Peabody No. 10 mine bituminous coal, Test 59, was conducted. A coal feed rate of 2.3 tons/hr was achieved, and the final results of this test are presented here. Two leaks forced termination of the test before steady-state operation could be attained.

A light-oil stripper flow test was carried out, and a surge pot will be installed to improve operation of the light-oil stripper.

An oil circulation test of the liquid-phase methanation unit was completed, as well as a leak test of the entire unit. A motorized nuclear level gauge lifting device was installed and calibrated, and the oil heater was checked out completely. An LDI Catalyst Co. catalyst, LDI X-826, was tested in the IGT fixed-bed process development unit (PDU) and found to be as active and as durable as other high-activity catalysts tested previously. A method for reducing the catalyst was developed and is presented here.

The results of several months of material testing of refractories are given in the Appendix. These results show that high-alumina refractories with calcium-aluminate bonds perform more satisfactorily in a condensing acid-gas atmosphere under given conditions than a material with phosphate bonds would.

The final results of two process development unit tests of the steam/oxygen gasification of Peabody No. 10 mine coal are presented here. These tests show that the coal yielded carbon conversions of $\approx 88\%$ at $1820^{\circ}F$. A fluidization curve for pretreated Peabody coal is also presented.

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INTRODUCTION

The overall program has been divided into the following major tasks:

- Task 1. Pilot plant tests with pretreated bituminous and subbituminous coal
- Task 2. Effluent treatment and water reuse
- Task 3. Methanation tests
- Task 4. Materials testing
- Task 5. Program assessment
- Task 6. Engineering services.

No work was done under Task 5 this quarter. A discussion of work done under Tasks 1 through 4 and Task 6 follows.

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PROGRESS REPORT

Task 1. Pilot Plant Tests With Bituminous Coal

During the month of January, the HYGAS® plant underwent plant modifications and improvements to prepare for tests with a new coal type and for new test objectives. These modifications included –

a. Coal Preparation: Modifications were made on the Sweco screener, oversize chute, and screw conveyor to remove the moisture from the system that caused interruptions in coal feed by plugging the lines and screens.

b. Pretreatment Section: A new pretreater grid design was developed. This grid, if and when necessary, will replace the present pretreater grid. Other modifications included a better venturi scrubber with improved water sprays and additional instruments, which were added to the internal cyclone to monitor its operation and efficiency. The pretreater quench-water effluent system was improved; smoother piping configurations, steam-traced lines, and standby pumps to improve reliability of the section were added.

c. Slurry Preparation and Pumping Area: Because this section had troublefree operation in the past, no major changes were made. A new high-pressure magnetic flowmeter was installed on the coal slurry line to the reactor to measure high-pressure slurry flow directly.

d. Reactor System: The reactor start-up heater was repaired. The weld overlay application and remachining were done on the 24-inch Grayloc reactor flanges and start-up heater flanges. Hydrostatic tests were successfully performed on the start-up heater and its water jacket, after which a castable refractory was installed in the start-up heater. The refractory was then cured by passing steam through it until no visible moisture evolved from the refractory. The start-up heater was installed.

e. Quench Station: The entire section was cleaned.

f. Purification Section: After establishing the foaming agent in the diglycolamine (DGA) system, a reclaiming procedure was developed. Batch reclaiming of DGA in our reclaimer was initiated. Samples of DGA were collected downstream of the reclaimer, and analyses showed that they were consistently nonfoaming.

Modifications of the methanation section and the effluent clean-up section are discussed under Tasks 3 and 2, respectively.

The results of batch pretreatment tests, fluidization studies, and coal-crushing tests (presented in ERDA Report No. FE-2434-6, December 1976) prompted the selection of Peabody coal to be used in the next series of tests with the HYGAS Process. These tests are being conducted to study the effects of carbon conversion, pretreatment severity, and fines recycle, as directed by the ERDA/A.G.A. Operating Committee. Fifteen hundred tons of the Peabody washed coal were ordered early in January. Because

of unusually severe weather and extended sub-zero temperatures experienced throughout Illinois, the Peabody coal washing plant was inoperable. The shipment of 1500 tons could not be delivered as scheduled on January 17. Finally, on January 26, 13 carloads (1000 tons) of washed Peabody No. 10 mine coal were shipped from the mine in Christian County. Seven more carloads were received on February 4, six more were received the following week, and the balance was received the week ending February 18. All 1500 tons were unloaded and stockpiled in the raw-coal storage area.

Coal crushing tests were made on the new coal. A screen analysis and a proximate analysis of the crushed coal are presented here. As a comparison, the raw coal analysis is also listed. (See Table 1.) The coal crushing and drying operation was entirely satisfactory, so the pretreater section was activated for operation. The objective of the pretreater test was to produce nonagglomerating coal for the HYGAS reactor feed. Operating conditions were a temperature of 775°F in the pretreater, a superficial velocity ranging from 0.8 to 0.9 ft/s, a mean residence time longer than 1 hour, and an oxygen-to-coal ratio greater than 2 SCF oxygen per pound of coal. Based on past experience in pretreater operation, the largest feed material for Test 59 was -14 mesh.

Pretreater heat-up began on February 14, when the temperature was brought to 775°F and the coal feed rate was established at 1 ton/hr. Erratic temperature control in the pretreater and pretreater off-gas flow rates indicated a leak in the pretreater internal cooling coil system; a subsequent pressure test on the cooling coil confirmed the observation. The pretreater was shut down, and an inspection of the cooling coil revealed a ruptured pipe at the bottom header on one of the four cooling coils. The coil was removed, repaired, and put back in service. Ice formation in the coil during the previous cold spell is believed to have caused the tube rupture. Pretreater operation resumed on February 19, but due to unusually wet coal present in the 60-ton coal storage hopper, the pretreater coal feeding system periodically plugged up. Finally, this wet coal plugged up the hopper discharge, and pretreater operation had to be interrupted again. The 60-ton hopper was completely emptied and cleaned. A large mass of caked coal had accumulated during the years in the bottom of the hopper. To facilitate future cleaning of the hopper, a 20-inch manway was installed near the bottom of the vessel's 30-foot high side.

Concurrent with the pretreatment operation, the reactor was readied for Test 59. The repair of the start-up heater for the reactor was completed. This involved the weld-overlay and resurfacing of the sealing surfaces. After the start-up heater refractory

Table 1. PEABODY No. 10 MINE BITUMINOUS COAL ANALYSES

Sample	Washed Raw Coal	Crushed Coal
Proximate Analysis	wt %	
Moisture	12.6	5.8
Volatile Matter	37.0	37.1
Ash	8.0	10.0
Fixed Carbon	42.4	47.1
Total	100.0	100.0
Ultimate Analysis		
Ash	9.20	10.61
Carbon	71.50	59.60
Hydrogen	5.09	4.93
Sulfur	4.23	4.32
Nitrogen	1.35	1.32
Oxygen (by Difference)	8.63	9.22
Total	100.00	100.00
Screen Analysis, USS %, retained on		
12	*	15.5
20		24.0
30		10.3
40		11.6
60		12.4
80		6.3
100		3.1
200		7.8
230		1.0
Pan		8.0
Total		100.0

* All raw coal was 1-1/4 in. x 0.

was cured by steaming, it was lifted into place. On February 8, the heat-up cycle on the reactor began. When the reactor pressure reached 750 psig, numerous leaks were detected in Grayloc closures on the reactor. The reactor was depressurized and cooled down to repair the leaks. At the same time, faulty valve instrumentation on the 321 line was discovered and fixed. The reactor was reignited on February 14; after the pressure had been brought up to 1000 psig, a small leak on the cooling water inlet connection to the start-up heater was discovered. Also noted was a high-temperature reading on one spot of the start-up heater attachment to the main reactor. Consequently, the reactor was depressurized and cooled down, the refractory was patched, and a metal disk assembly was fabricated and inserted ahead of the start-up heater to prevent hot gases from circulating to the reactor nozzle wall. The start-up heater and manway 4 assembly were cleaned and reassembled to prepare for the start of Test 59.

The reactor was lit off on March 8, 1977, in preparation for Test 59. Problems with the reactor quench-water pumps and leaky seals delayed the heat-up cycle, and Test 59 was begun on March 11 at 1400 hours. Initial char feed rates averaged between 1 and 1.5 tons/hr. Pretreated coal feed was interrupted three times during the test period: once to repair the screw feeder to the coal slurry mix tank, once when a plug developed in the reactor spent-char discharge line, and once when the reactor spent-char slurry letdown chokes plugged. Toward the end of the test, reactor operation was smooth, and the coal feed rate reached 2.3 tons/hr. Test 59 was terminated before the start-up heater was shut down. A leak was detected at the 40-inch Grayloc closure of the reactor top manhole. Another leak was detected at the 24-inch Grayloc flange on the start-up heater at 1800 hours on March 14. These leaks forced the termination of Test 59. For this first test on Peabody No. 10 mine bituminous coal, a total of 52 tons of pretreated coal was fed to the reactor.

The coal preparation section was started up on March 2 on Peabody No. 10 mine coal. The 60-ton raw-coal storage hopper required a complete cleanup because caked subbituminous and bituminous coal had accumulated in the base of the hopper. Also, the secondary fan began drawing dirty gas from the wet scrubber in the coal mill grinding circuit. Pretreatment operations were resumed on March 3. Operations were conducted at around 775°F, at residence times of 1 to 1-1/2 hours, and at oxygen-to-coal feed ratios of approximately 2 to 2-1/2 cubic feet of oxygen per pound of coal. PDU tests had indicated that these conditions were satisfactory for producing nonagglomerating coal from the Peabody No. 10 mine coal. Operation at these conditions did produce a non-agglomerating char according to the IGT boat test analysis. Therefore, the char was stored in the 15-ton hopper to be fed to the slurry preparation section at the initiation of

Test 59. Operation of the pretreater for Test 59 was interrupted several times by the plugging of the vent line on the feed lockhopper system. This plugging was caused by moist coal particles and/or cold weather. This vent system was later modified by enlarging the line from 1 to 3 inches and placing it on the feed lockhopper itself. This greatly improved the operation of the lockhopper system. Also, the level control probe in the bottom lockhopper was modified to eliminate interference from coal accumulated in the surrounding dead space. These two changes resulted in steady feed rates to the coal pretreater.

The pretreater section was inspected after Test 59. A hard, red clinker had formed in the southwest quadrant of the pretreater, about 1 sq ft in area and 6 inches above the grid. Two smaller clinkers were observed at the east and north wall of the pretreater. The unsteady coal feed to the pretreater made temperature control and product-char flow control difficult, causing these clinkers. Clinkers were also found in the pretreater char cooler, as a result of intermittent solids flow. The clinkers in this area could also be the result of poor fluidization. Superficial velocities of 0.3 ft/s were used in Test 59. Subsequent fluidization tests indicate that a superficial velocity of 0.45 ft/s is required for complete fluidization of Peabody No. 10 pretreated coal.

The pretreater quench unit was also inspected. Tar-like material was found in the venturi scrubber and in the quench tower, as is typical of post-run pretreater quench unit inspection. The pretreater was cleaned and readied for the next test.

The reactor was inspected following Test 59. A small amount of solids, typical of post-run inspection, was observed at the slurry dryer grid area. There was no plugging of grid holes. The dryer section wall was coated with a thin film of powdered coal and tar-like material. Similar material was found in the fresh-coal feed line to the vertical lift reactor (321 line). The lift line and the 322 line, connecting the first-stage and the second-stage reactor, were found to be free of solids. The second-stage reactor was found to be clean, and so was the steam/oxygen reactor. The external start-up heater was also inspected, and some damage was found in the top refractory plug. The reactor was cleaned, samples obtained from the slurry dryer area were sent to the laboratory for analysis, and the refractory was fixed.

Inspection of the two leaky closures that forced termination of Test 59 showed that Manhole 0 was in good condition. The seal ring was found to be undersized, and the 24-inch Grayloc flange on the start-up heater had erosion scratches on its surface. Manhole 0 required no machine work and was reinstalled under the supervision of Gray Tool Company's service technicians, who also discovered that one of the two new seal

rings in stock for Manhole 0 was out of specification. The seal rings were returned to Gray Tool Company for credit. The 24-inch Grayloc closure on the start-up heater was remachined and reassembled. The entire reactor was reassembled and readied for Test 60.

The slurry preparation section and the quench section operated very smoothly for Test 59. The purification section also operated satisfactorily. Some foaming in the diglycolamine absorber occurred, which was controlled by using a Nalco antifoam additive. The light-oil recovery unit and the Edens separator operated satisfactorily for Test 59.

Figures 1 through 9 present the final engineering information for Test 59 on both the pretreater and the reactor.

Task 2. Effluent Treatment and Water Reuse

A light-oil stripper (LOS) flow test was carried out during January. Three input streams and two output streams were activated. Flowmeters and level controls were calibrated and checked for proper operation.

To improve the operation of the LOS, a surge pot was designed and ordered. It was received in late February. This new pot will dampen all fluctuations that may occur upstream of the LOS and bypass gas flow around it, thus preventing operating upsets in the unit. The concrete pads for the vessel and the circulation pump were poured during March. At the end of this quarter, the unit's instrumentation had been partially received, and the remainder was expected to be delivered shortly. The surge pot was expected to be ready for Test 61.

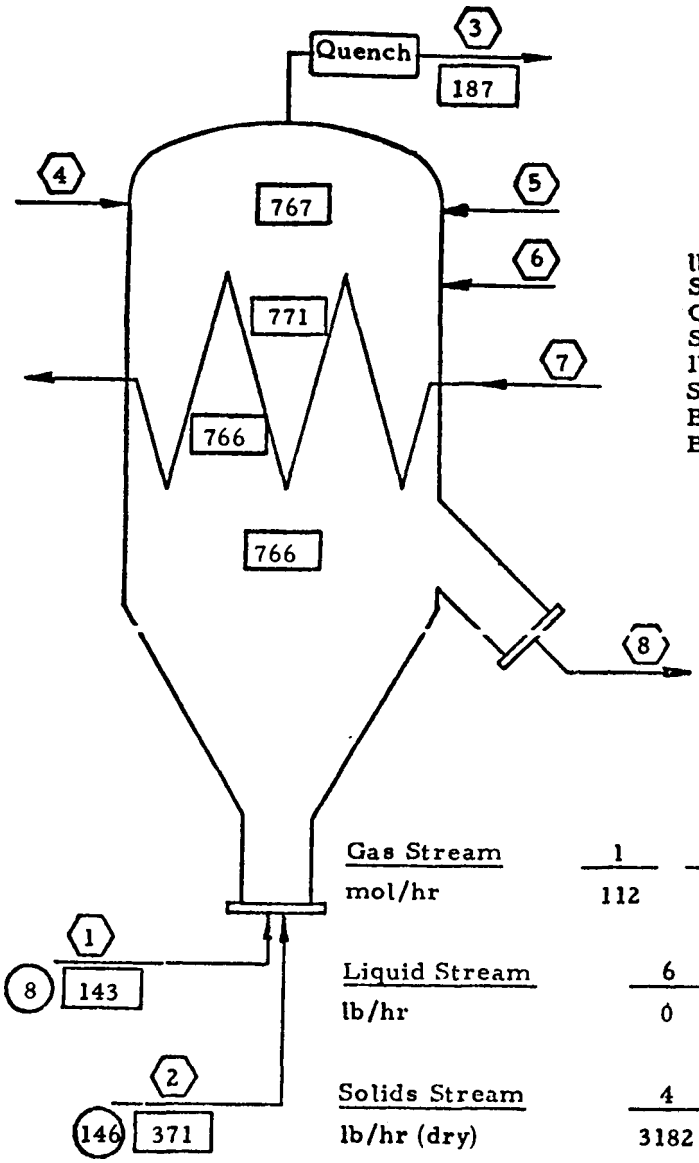
An improved level detection system in the cyclone slurry pot was also installed during January to prevent effluent flow fluctuations. Past experience has shown that solid fines from the cyclone pot collect at the level controller and foul up its operation, thus giving false level indications and, as a consequence, erratic flows out of the pot into the LOS.

The light-oil recovery unit operated satisfactorily for Test 59. The drag chain on the Edens separator was replaced with new links.

Task 3. Methanation Tests

IGT Fixed-Bed Methanation Unit

During February, the leaky heat exchanger in the IGT fixed-bed methanation section was repaired. The IGT methanation unit is on standby.



Stream No.
Pressure, psig
Temperature

lb Char/lb Coal	--
SCF O_2/lb Coal	2.80
Oxygen Breakthrough, % fed	6.64
Superficial Velocity, ft/s	0.95
lb O_2 Reacted/lb Coal	0.22
Solids Residence Time, hr	2.7
Bed Density, lb/cu ft	17
Bed Height, ft	12

Gas Stream	1	2	5
mol/hr	112	114	62

Liquid Stream	6	7
lb/hr	0	360

Solids Stream	4	8
lb/hr (dry)	3182	--

Gas Stream	3
Components, mol %	
H_2	0.06
CO_2	4.14
C_2H_6	0.06
O_2/Ar	1.57
N_2	39.19
SO_2	--
CH_4	0
CO	0.23
H_2O	54.75
mol/hr	313
mol. wt	23

1. Air to Pretreater
2. Steam to Pretreater
3. Pretreater Off-Gas After Quench
4. Raw Coal to Pretreater
5. Gas From Char Cooler
6. Direct Water Quench
7. Cooling Water to Cooling Coil in Pretreater
8. Pretreated Char to Char Cooler

Figure 1. PRETREATMENT DATA FOR TEST 59 FOR STEADY PERIOD FROM 3/13/77 (0300 Hours) TO 3/13/77 (0800 Hours)

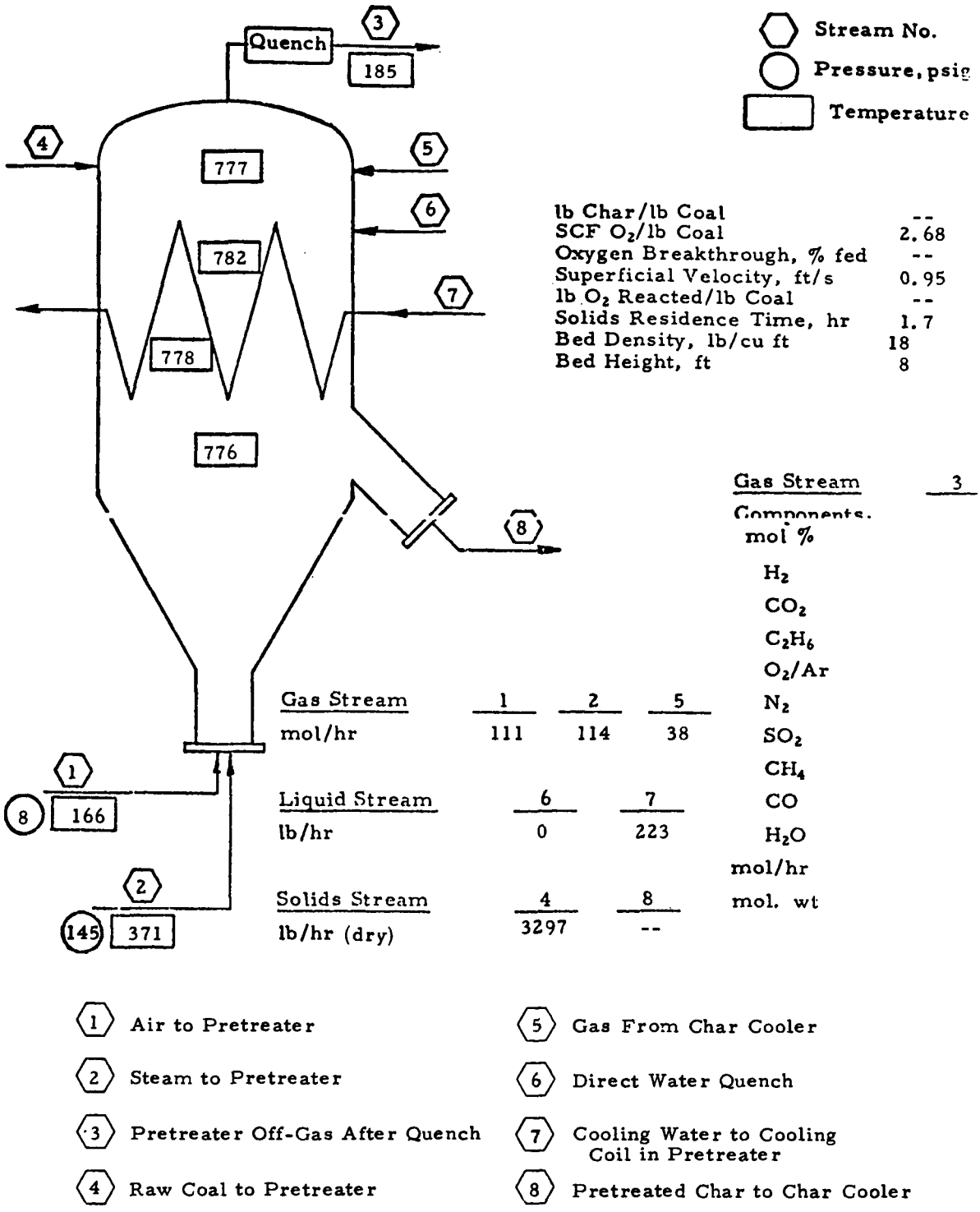
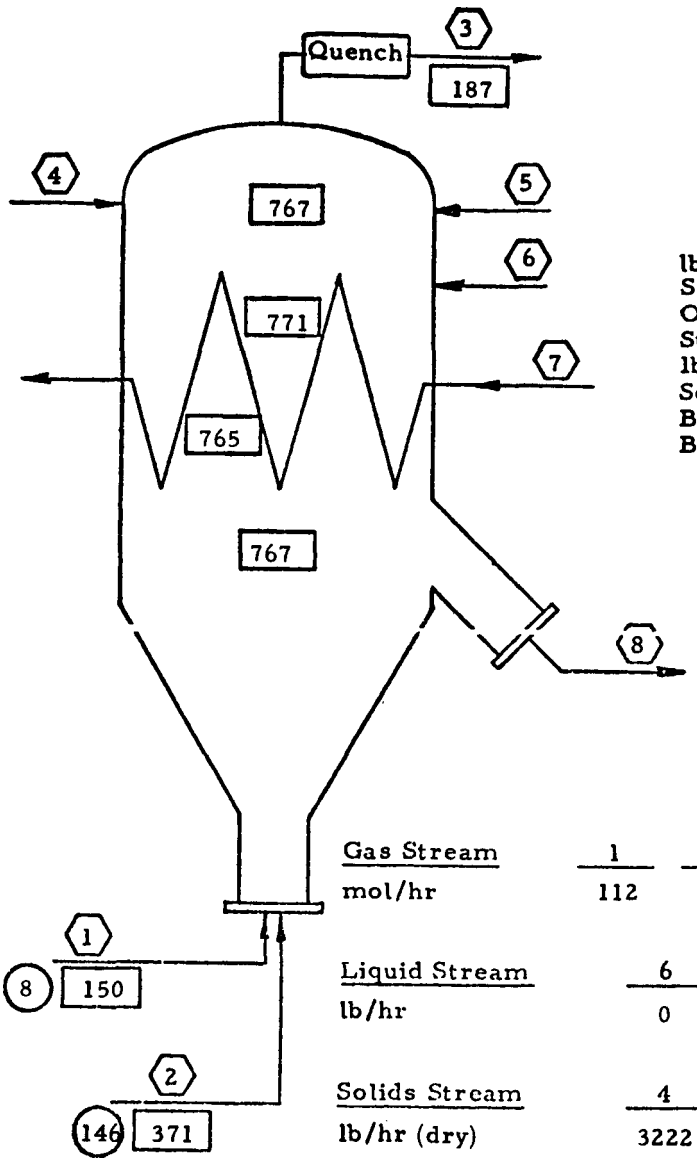


Figure 2. PRETREATMENT DATA FOR TEST 59 FOR STEADY PERIOD FROM 3/12/77 (1400 Hours) TO 3/12/77 (1700 Hours)



Stream No.
Pressure, psig
Temperature

lb Char/lb Coal	--
SCF O_2/lb Coal	2.77
Oxygen Breakthrough, % fed	5.18
Superficial Velocity, ft/s	0.96
lb O_2 Reacted/lb Coal	0.22
Solids Residence Time, hr	2.5
Bed Density, lb/cu ft	16
Bed Height, ft	12

Gas Stream	1	2	5
mol/hr	112	117	55

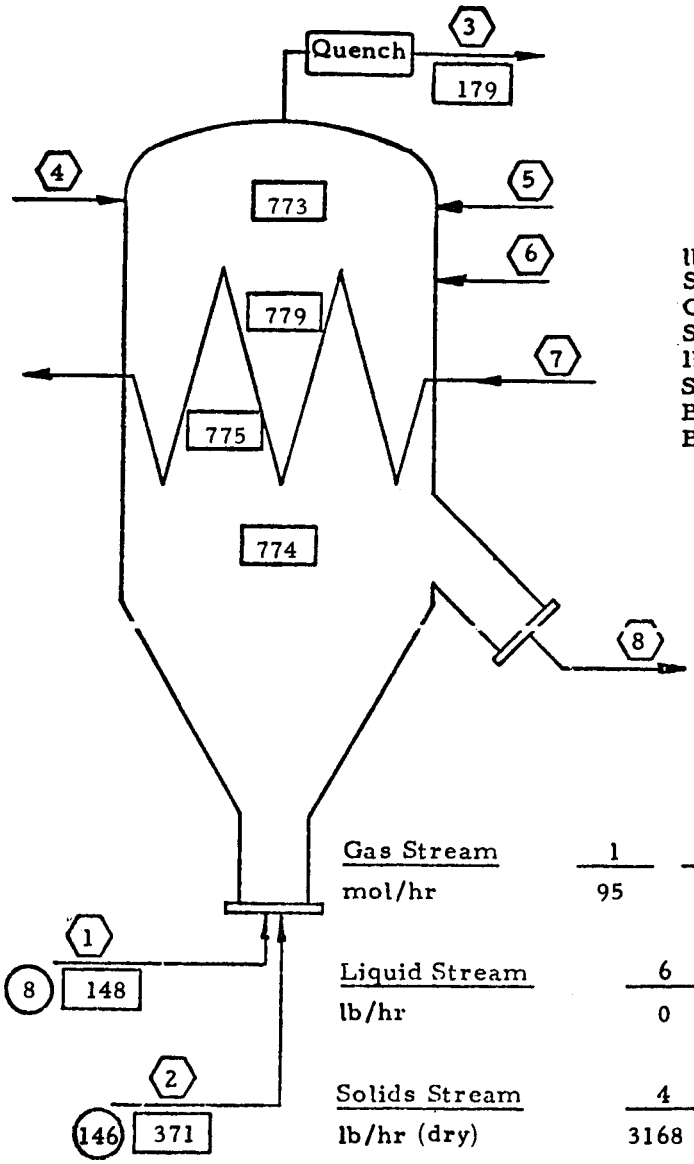
Liquid Stream	6	7
lb/hr	0	302

Solids Stream	4	8
lb/hr (dry)	3222	--

Gas Stream	3
Components, mol %	
H_2	0.06
CO_2	4.14
C_2H_6	0.10
O_2/Ar	1.28
N_2	36.99
SO_2	--
CH_4	0.03
CO	0.52
H_2O	56.88
mol/hr	313
mol. wt	23

1. Air to Pretreater
2. Steam to Pretreater
3. Pretreater Off-Gas After Quench
4. Raw Coal to Pretreater
5. Gas From Char Cooler
6. Direct Water Quench
7. Cooling Water to Cooling Coil in Pretreater
8. Pretreated Char to Char Cooler

Figure 3. PRETREATMENT DATA FOR TEST 59 FOR STEADY PERIOD FROM 3/12/77 (1800 Hours) TO 3/13/77 (0100 Hours)



Stream No.
Pressure, psig
Temperature

lb Char/lb Coal	--
SCF O_2/lb Coal	2.39
Oxygen Breakthrough, % fed	12.23
Superficial Velocity, ft/s	0.89
lb O_2 Reacted/lb Coal	0.18
Solids Residence Time, hr	2.0
Bed Density, lb/cu ft	18
Bed Height, ft	9

Gas Stream	1	2	5
mol/hr	95	116	43

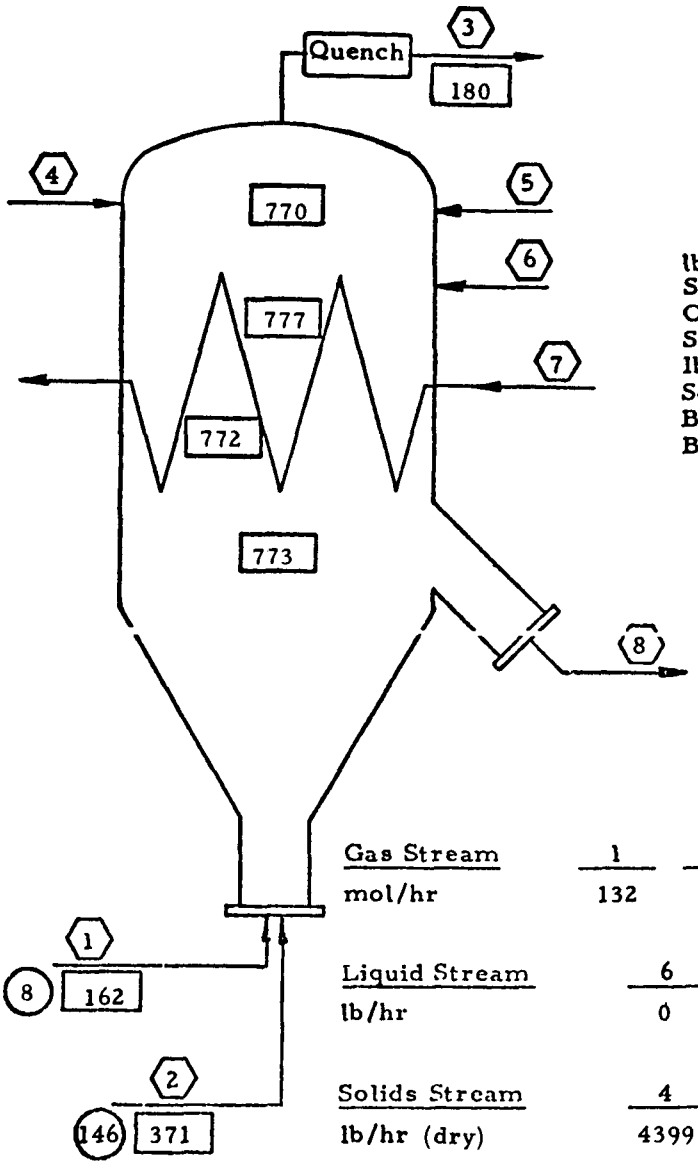
Liquid Stream	6	7
lb/hr	0	145

Solids Stream	4	8
lb/hr (dry)	3168	--

Gas Stream	3
Components, mol %	
H_2	0.07
CO_2	4.87
C_2H_6	0.11
O_2/Ar	2.16
N_2	44.05
SO_2	--
CH_4	0
CO	0.38
H_2O	48.36
mol/hr	228
mol. wt	24

1. Air to Pretreater
2. Steam to Pretreater
3. Pretreater Off-Gas After Quench
4. Raw Coal to Pretreater
5. Gas From Char Cooler
6. Direct Water Quench
7. Cooling Water to Cooling Coil in Pretreater
8. Pretreated Char to Char Cooler

Figure 4. PRETREATMENT DATA FOR TEST 59 FOR STEADY PERIOD FROM 3/13/77 (1900 Hours) TO 3/13/77 (2300 Hours)



Stream No.
Pressure, psig
Temperature

lb Char/lb Coal	--
SCF O_2/lb Coal	2.39
Oxygen Breakthrough, % fed	2.30
Superficial Velocity, ft/s	0.94
lb O_2 Reacted/lb Coal	0.20
Solids Residence Time, hr	1.8
Bed Density, lb/cu ft	18
Bed Height, ft	10

Gas Stream	1	2	5
mol/hr	132	91	70

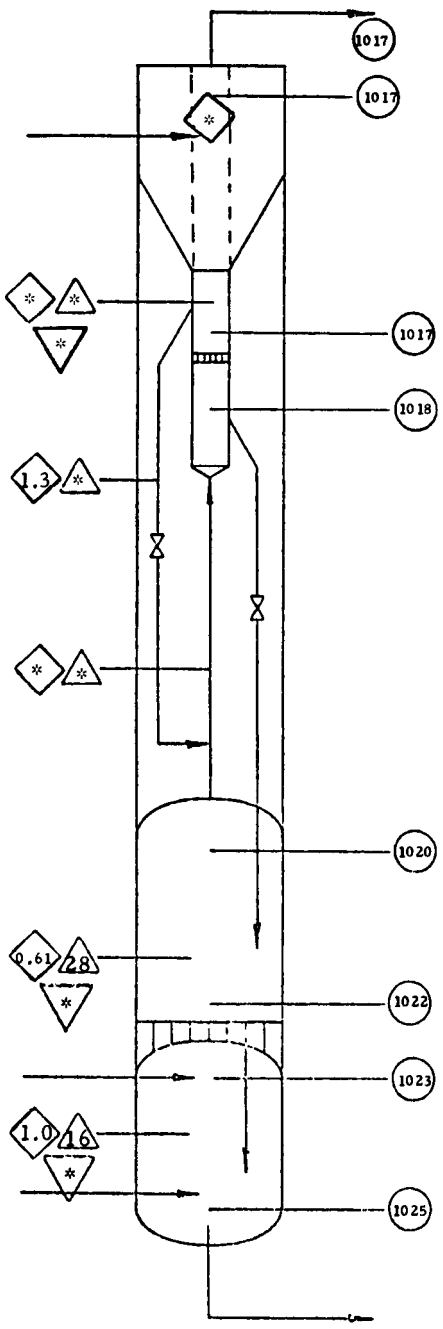
Liquid Stream	6	7
lb/hr	0	802

Solids Stream	4	8
lb/hr (dry)	4399	--

Gas Stream	3
Components, mol %	
H_2	0.08
CO_2	5.03
C_2H_6	0.11
O_2/Ar	1.26
N_2	44.76
SO_2	0
CH_4	0
CO	0.61
H_2O	48.15
mol/hr	296
mol. wt	24

1. Air to Pretreater
2. Steam to Pretreater
3. Pretreater Off-Gas After Quench
4. Raw Coal to Pretreater
5. Gas From Char Cooler
6. Direct Water Quench
7. Cooling Water to Cooling Coil in Pretreater
8. Pretreated Char to Char Cooler

Figure 5. PRETREATMENT DATA FOR TEST 59 FOR STEADY PERIOD FROM 3/14/77 (1530 Hours) TO 3/14/77 (1830 Hours)



Pressure, psig

Density, lb/cu ft

Velocity, ft/s

Mean Residence Time, min

* Not Available

Product Gas – dry, nitrogen- and acid-gas-free basis
Coal Fed – dry basis

lb Oxygen/lb Coal Fed = 0.09
lb Steam/lb Coal Fed = 2.82
lb Hydrogen/lb Coal Fed = 0.10
lb Coal Fed/1000 SCF Product Gas = *

By Ash Balance

Coal Gasified, % = *
Carbon Gasified, % = *

Methane Yield, SCF/lb coal fed = *
Carbon Oxide Yield, SCF/lb coal fed = *

Bed Height (ft)

Slurry Dryer = *

HTR = 12

SOG = 20

Figure 6. HYGAS REACTOR ENGINEERING DATA FOR TEST 59 FOR STEADY PERIOD FROM 3/12/77 (2200 Hours) TO 3/13/77 (0130 Hours)

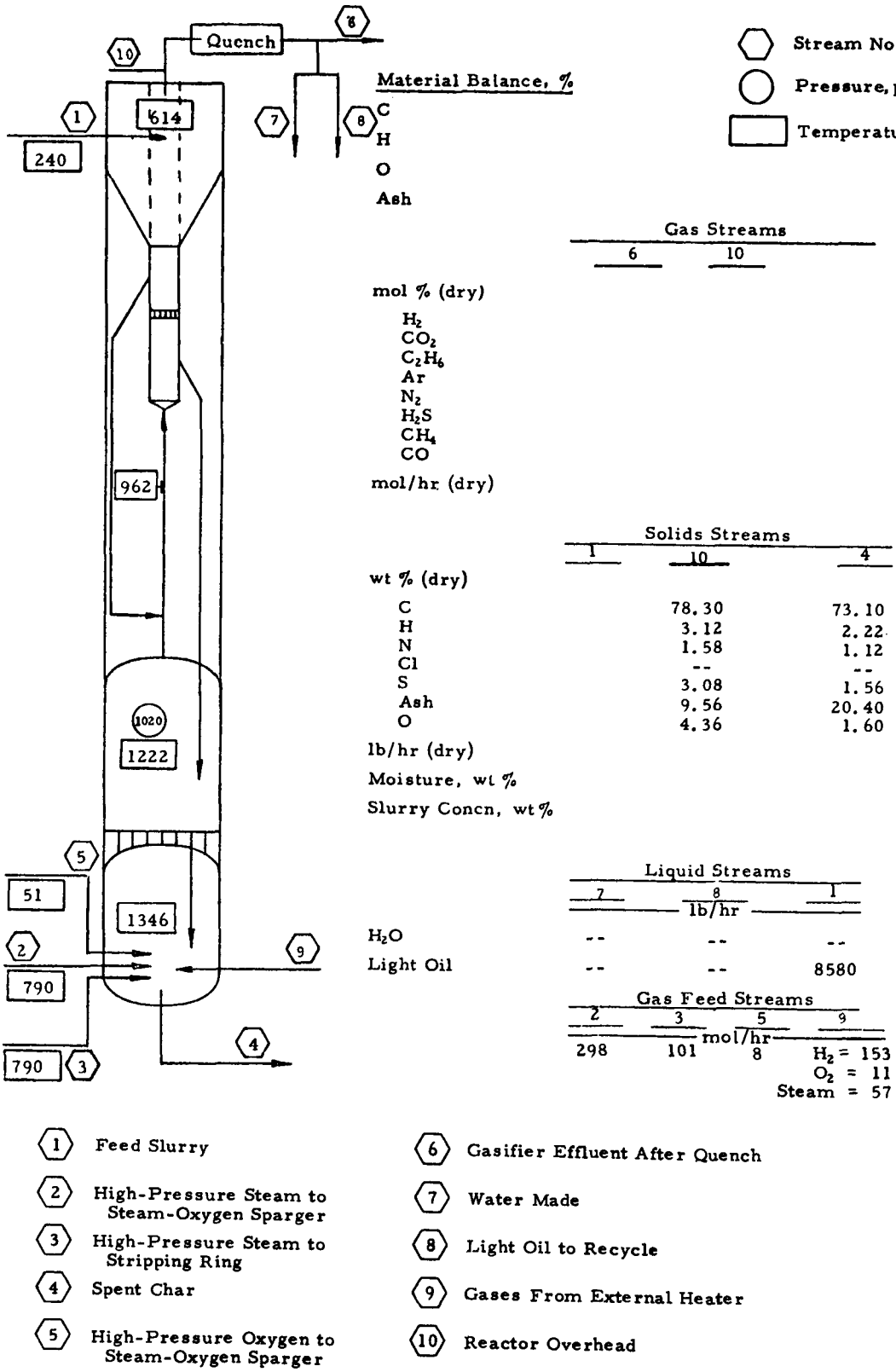


Figure 7. HYGAS REACTOR DATA FOR TEST 59 FOR STEADY PERIOD FROM 3/12/77 (2200 Hours) TO 3/13/77 (0130 Hours)

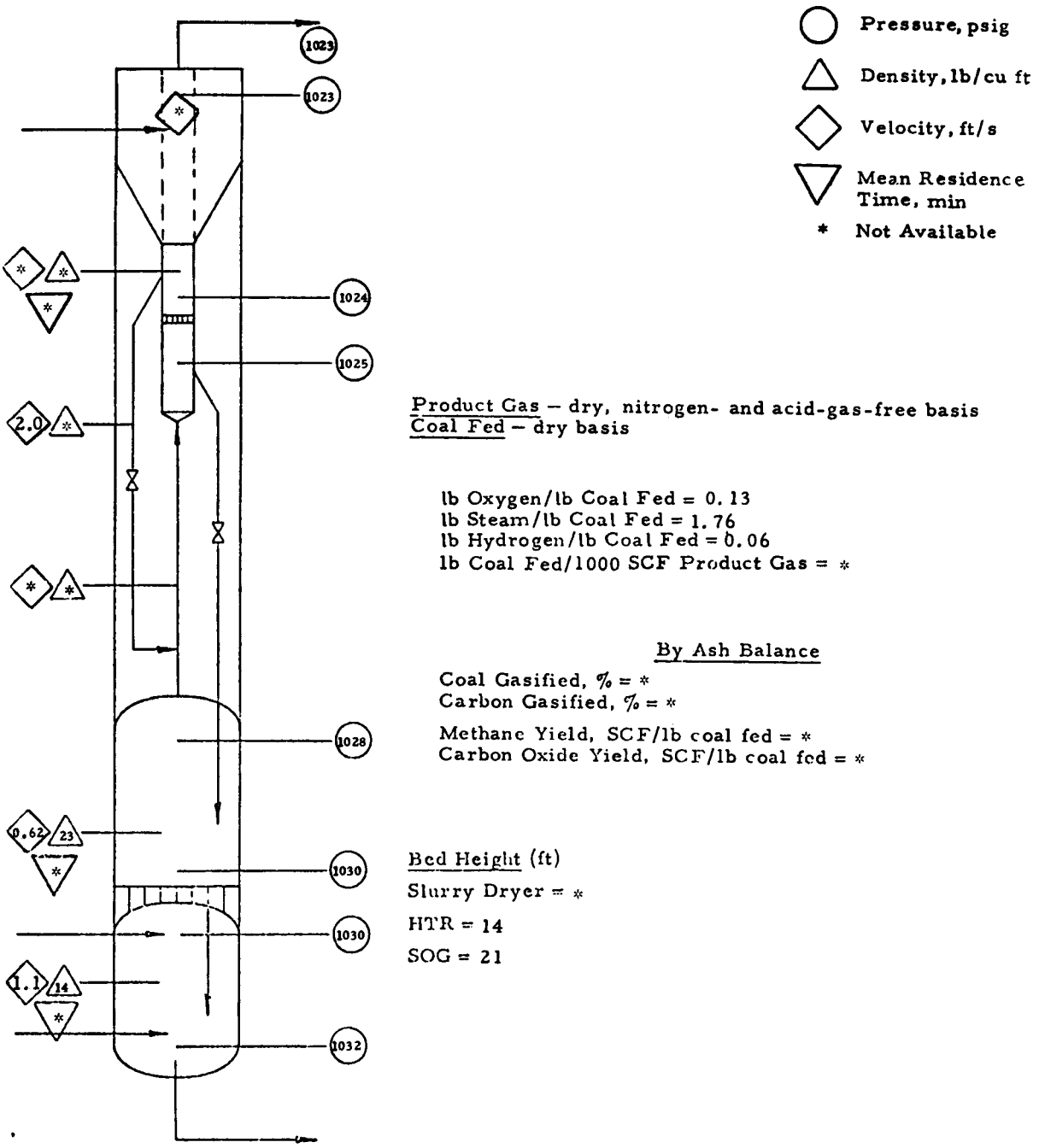
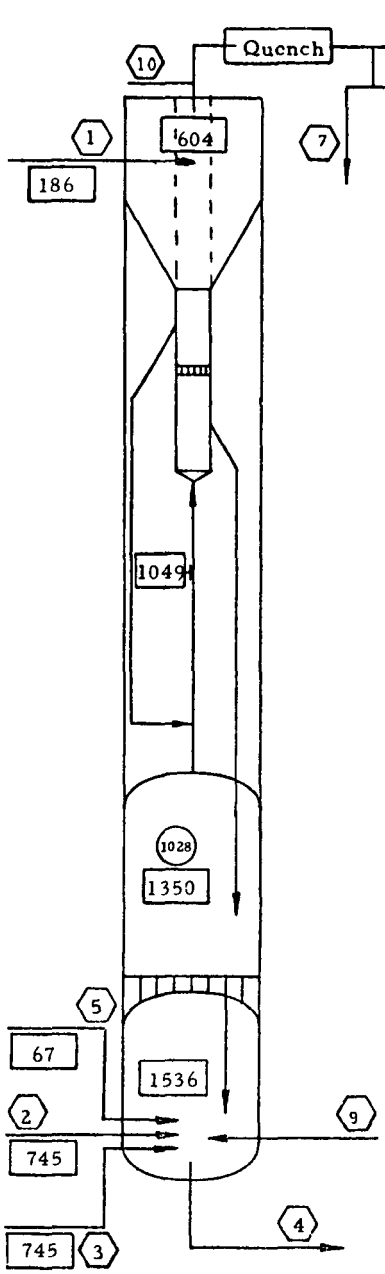


Figure 8. HYGAS REACTOR ENGINEERING DATA FOR TEST 59 FOR STEADY PERIOD FROM 3/14/77 (1430 Hours) TO 3/14/77 (1700 Hours)



Material Balance, %

C
H
O
Ash

Stream No.
Pressure, psig
Temperature, °F

Gas Streams	6	10
mol % (dry)		
H_2		
CO_2		
C_2H_6		
Ar		
N_2		
H_2S		
CH_4		
CO		
mol/hr (dry)		

Solids Streams	1	10	4
wt % (dry)			
C		78.80	73.50
H		2.82	1.27
N		1.33	0.54
Cl		--	--
S		2.77	0.84
Ash		9.98	22.25
O		4.30	1.60
lb/hr (dry)			
Moisture, wt %			
Slurry Concn, wt %			

Liquid Streams	7	8	1
		lb/hr	
H_2O	--	--	--
Light Oil	--	--	10,448

Gas Feed Streams	2	3	5	9
		mol/hr		
	301	85	19	H_2 = 125 O_2 = 6 Steam = 57

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. Gasifier Effluent After Quench
7. Water Made
8. Light Oil to Recycle
9. Gases From External Heater
10. Reactor Overhead

Figure 9. HYGAS REACTOR DATA FOR TEST 59 FOR STEADY PERIOD FROM 3/14/77 (1430 Hours) TO 3/14/77 (1700 Hours)

Liquid-Phase Methanation (LPM) Pilot Unit (Chem Systems, Inc.)

A leak test of the entire LPM unit was completed during January. Because there were too many leaks in the system, individual sections were isolated with blind flanges before complete pressure testing could be done. All leaks were fixed, and this unit was winterized.

An oil circulation test of the unit was made during January. Oil was circulated through the entire oil loop except for the methanation reactor. All related equipment and instrumentation was checked out. There was leaky packing on the oil seal circulation pumps, and it was discovered that the suction and discharge piping of one of the pumps was off-center. Calibration and repairs were made where needed, and oil was stored in the reactor separator vessel.

During January, the oil heater was checked out, and a problem was discovered in the electrical control system that required maintenance and troubleshooting. After repairs, the oil heater was checked out completely, and hot oil at 200°F was successfully circulated. Gas flow was also initiated. The gas heater was used to raise the reactor temperature to 50°F, and an integrated oil and gas circulation test was completed. The oil could not be heated above 500°F until insulation was installed on the exposed flanges of major vessels and the excess oil seal flush was shut off. Then the oil was heated to 560°F quickly, and the system pressure was raised to 300 psig by high-pressure nitrogen. Numerous leaks were discovered at this pressure, the unit was depressurized, and the leaks were fixed. A leak was also discovered at the hydrogen gas preheater heat exchanger for catalyst reduction. The heat exchanger was removed and inspected; the flange face was resurfaced and reinstalled.

The methanation reactor nuclear level gauge lifting device was found to be very hard to operate, and a new motorized device was ordered and received. This device was delivered and installed during February. Technicians from Texas Nuclear Corporation completed calibration of the device during March.

During February, all analytical equipment was installed, and shakedown continued with the activation of various instruments and control circuitry and with the troubleshooting of the system. Catalyst reduction was successfully completed during March.

The unit was ready to receive gas from the HYGAS plant during Test 59, but, due to the early termination of the test, no gas was processed through the unit. At the end of this quarter, the LPM unit was ready to receive product gas from Test 60. Chem

Systems personnel were in the plant on a round-the-clock basis and were assuming operational supervision of the unit.

Methanation Catalyst Evaluation Studies

The evaluation studies of methanation catalysts on the bench-scale reactor were continued during January. The first methanation catalyst supplied by the LDI Catalyst Co., LDI X-825 (the CRG-A catalyst manufactured in the United Kingdom), had been tested during November 1974 (OCR Report No. 122). The performance was poor, and it was found that a bad batch had been received (ERDA Report 125, February 1975). The second LDI catalyst, LDI X-826 (the CRG-A catalyst manufactured in the United States), was tested, and the results are presented here. LDI X-826 is a high-density, high-activity catalyst. For the temperature range (550° to 680°F) studied in a packed-bed reactor (1-inch, Schedule 80, Type-316 stainless steel), equilibrium conversion was achieved at 40,000 SCF/hr-cu ft and 1000 psig. This was the same conversion achieved at 25,000 SCF/hr-cu ft and 500 psig. Near-equilibrium conversion was achieved at 20,000 SCF/hr-cu ft and 200 psig. The conversion decreases rapidly and nonlinearly at pressures below 200 psig. The dependence of conversion on the pressure and space velocity is illustrated in Figure 10.

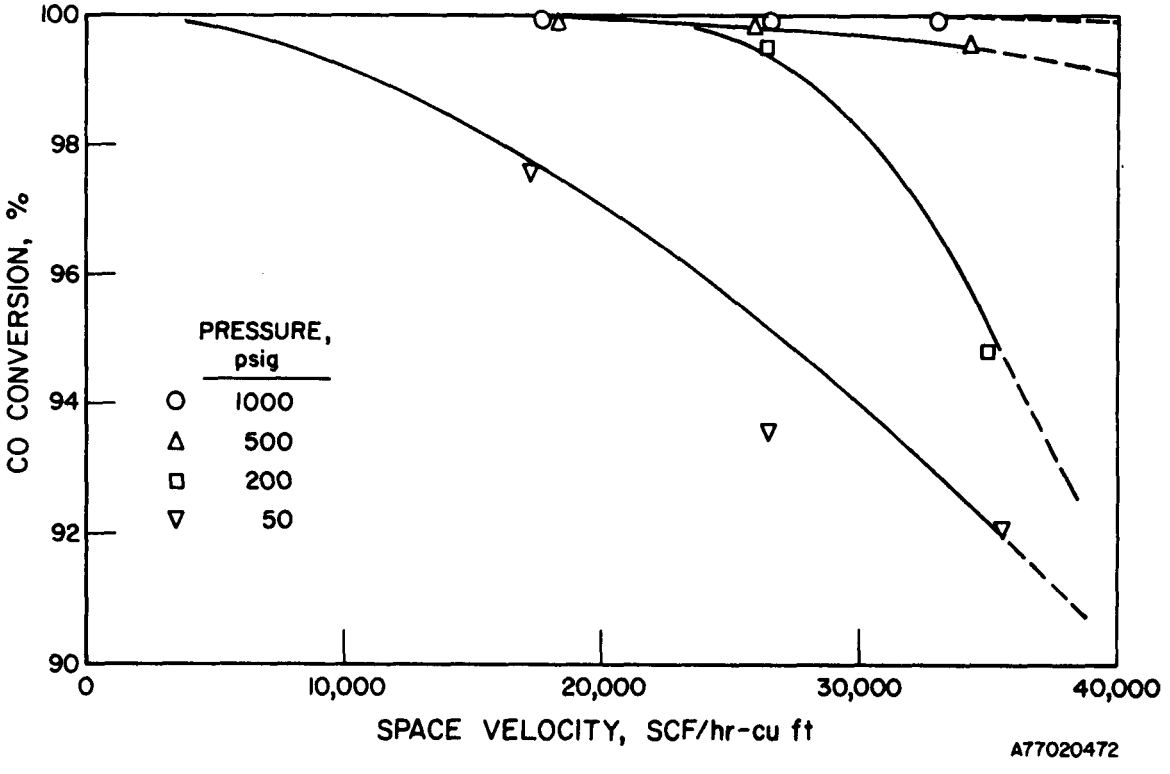


Figure 10. EFFECT OF SPACE VELOCITY ON THE CONVERSION OF CARBON MONOXIDE AT TEMPERATURES FROM 550° to 680°F (LDI X-826 Catalyst, 1/8-Inch Cylinders)

The catalyst was loaded into the reactor on top of alumdum inerts. It was later found that the thermocouple positions were not as indicated, as Table 2 shows. The thermocouples marked "quarter bed" and "middle bed" were actually closer to "inlet" and "1/8 bed," respectively, and the one marked "outlet" was closer to "middle bed". The catalyst was supplied in its oxidized form, making it necessary to reduce it before it contacted the feed-gas mixture. The ideal reduction conditions are 750°F and pure hydrogen flowing at an optimum space velocity so that the hydrogen uptake is maximized and the nickel oxide is reduced to nickel. In a plant, the hydrogen is recycled, and the space velocity is, therefore, not a problem. However, the composition of the reduction gas is usually 99% hydrogen and 1% CO. The concentration of CO decreases to about 0.02% after being recycled a number of times and becomes acceptable. However, it is not anticipated that, in a commercial plant, the methanator can be heated to an initial temperature of 750°F just for start-up. The temperature that can be achieved will more likely be 500°F or less, and thus the catalyst will not be reduced properly. Based on experience with the reduction of other catalysts, the catalyst can be reduced to near "ideal reduction" conditions by the following method:

a. Purge the reactor of air, using an inert gas, while heating the reactor to its maximum temperature. If nitrogen is used as the purge gas, it should be purged out to prevent the formation of ammonia, which will poison the catalyst.

b. Introduce the reducing gas (99% H_2 + 1% CO) from the hydrogen plant and recycle it as quickly as possible, so that the CO concentration is decreased rapidly. Do not introduce this reducing gas at high pressures and low temperatures, because these conditions favor the formation of nickel carbonyl and nickel carbide.

c. The initial methanation of the reducing gas will increase the bed temperature. As the bed temperature increases, increase the feed reducing gas and decrease the recycle ratio so that the methanation reaction will be favored and a higher bed temperature can be achieved.

d. The reduction can be assumed to be complete when the reactor temperatures are stabilized and a reasonable material balance on product water is obtained.

For insurance, it is recommend that a) a layer of prereduced catalyst be installed at the entrance of the methanator and b) a H_2/CO mole ratio of more than 3:1 in the feed gas mixture be used for the first 100 hours of operation.

The feed gas used for this test, a synthetic mixture of high-purity components, was sulfur-free. Both the feed and product compositions as well as the operating conditions are presented in Table 2. The times listed are the periods that the catalyst remained in the reactor, not necessarily under a continuous feed-flow condition. However, the actual run time of each feed-flow condition was usually 8 hours or longer.

Table 2, Part 1. METHANATION CATALYSIS – CATALYST EVALUATION (LDI Catalyst Co. LDI X-826 Catalyst, 1/8-Inch Cylinders, 22.29 g)

Run No.	476		477		478	
Time, hr	95		167		239	
Basis for Analysis	Dry	Wet	Dry	Wet	Dry	Wet
Pressure, psig	1004	1004	1002	1002	999	999
Reactor Temperature, inlet, °F	510	510	425	425	412	412
Reactor Temperature, quarter bed, °F	570	570	553	553	548	548
Reactor Temperature, middle bed, °F	583	583	549	549	535	535
Reactor Temperature, outlet, °F	639	639	653	653	680	680
Furnace Temperature, top zone, °F	490	490	480	480	480	480
Furnace Temperature, bottom zone, °F	550	550	550	550	548	548
Flow Rate, feed, lb-mol/hr	0.03357	0.03357	0.05057	0.05057	0.06286	0.06286
Flow Rate, H_2O, lb-mol/hr	0	0	0	0	0	0
Feed Composition, mol %						
H_2	11.9	11.9	11.5	11.5	12.2	12.2
N_2	2.9	2.9	2.5	2.5	2.6	2.6
CH_4	79.9	79.9	80.9	80.9	80.0	80.0
C_2H_6	0	0	0.3	0.3	0.2	0.2
CO_2	1.6	1.6	1.6	1.6	1.6	1.6
CO	3.1	3.1	3.0	3.0	3.1	3.1
He	0.6	0.6	0.2	0.2	0.3	0.3
H_2O	0	0	0	0	0	0
Total	100.0	100.0	100.0	100.0	100.0	100.0
Flow Rate, product, lb-mol/hr	0.0301	0.0321	0.04581	0.04833	0.05618	0.05898
Flow Rate, H_2O in product, lb-mol/hr	0	0.00191	0	0.00252	0	0.00280
Product Composition, mol %						
H_2	1.8	1.7	1.9	1.9	1.6	1.5
N_2	3.2	3.0	2.8	2.8	2.9	2.8
CH_4	93.8	88.0	93.4	88.5	92.3	87.8
C_2H_6	0	0	0	0	0	0
CO_2	0.7	0.7	0.7	1.6	2.9	2.7
CO*	0	0	0	0	0	0
He	0.5	0.6	0.2	0.2	0.3	0.3
H_2O	0	6.0	0	5.2	0	4.9
Total	100.0	100.0	100.0	100.0	100.0	100.0
CO Consumed, lb-mol/hr	0.001041	--	0.001517	--	0.001949	--
CO_2 Changed, lb-mol/hr	-0.00033	--	-0.00003	--	0.000623	--
H_2 Consumed, lb-mol/hr	0.00345	--	0.00495	--	0.006770	--
H_2O Produced, lb-mol/hr	0.00191	--	0.00252	--	0.00280	--
CH_4 Produced, lb-mol/hr	0.00141	--	0.00187	--	0.001563	--
C_2H_6 Consumed, lb-mol/hr	0	--	0.00015	--	0.000126	--
Space Velocity, SCF/hr-cu ft†	17,511	--	26,385	--	32,808	--

* CO concentrations, which are within the range of 0.0% to 0.1%, cannot be accurately detected by the current calibration of the mass spectrometer and gas partitioner. Therefore, unless otherwise indicated, zero (0) means that equilibrium conversion concentration is achieved.

† Space velocity is calculated based on a measured bulk density of 66.49 lb/cu ft catalyst.

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Table 2, Part 2. METHANATION CATALYSIS – CATALYST EVALUATION (LDI Catalyst Co. LDI X-826 Catalyst, 1/8-Inch Cylinders, 22.29 g)

Run No.	479		480		481	
Time, hr	263		335		359	
Basis for Analysis	Dry	Wet	Dry	Wet	Dry	Wet
Pressure, psig	500	500	200	200	200	200
Reactor Temperature, inlet, °F	475	475	442	442	412	412
Reactor Temperature, quarter bed, °F	568	568	556	556	544	544
Reactor Temperature, middle bed, °F	559	559	548	548	538	538
Reactor Temperature, outlet, °F	622	622	659	659	678	678
Furnace Temperature, top zone, °F	480	480	482	482	482	482
Furnace Temperature, bottom zone, °F	548	548	547	547	549	549
Flow Rate, feed, lb-mol/hr	0.03409	0.03409	0.05019	0.05019	0.06694	0.06694
Flow Rate, H_2O, lb-mol/hr	0	0	0	0	0	0
Feed Composition, mol %						
H_2	12.1	12.1	12.3	12.3	11.6	11.6
N_2	2.4	2.4	2.3	2.3	2.3	2.3
CH_4	79.2	79.2	79.0	79.0	79.9	79.9
C_2H_6	0.2	0.2	0.3	0.3	0.3	0.3
CO_2	2.3	2.3	2.3	2.3	2.3	2.3
CO	3.6	3.6	3.5	3.5	3.5	3.5
He	0.2	0.2	0.3	0.3	0.1	0.1
H_2O	0	0	0	0	0	0
Total	100.0	100.0	100.0	100.0	100.0	100.0
Flow Rate, product, lb-mol/hr	0.03028	0.03194	0.04477	0.04702	0.06061	0.06301
Flow Rate, H_2O in product, lb-mol/hr	0	0.00166	0	0.00225	0	0.00241
Product Composition, mol %						
H_2	0.8	0.8	1.3	1.3	2.0	1.9
N_2	2.7	2.6	2.6	2.5	2.5	2.4
CH_4	93.8	88.9	93.29	88.8	92.4	88.8
C_2H_6	0	0	0	0	0	0
CO_2	2.5	2.4	2.5	2.4	2.8	2.7
CO*	0	0	0.01	0.01	0.2	0.2
He	0.2	0.2	0.3	0.3	0.1	0.1
H_2O	0	5.1	0	4.69	0	3.9
Total	100.0	100.0	100.00	100.00	100.0	100.0
CO Consumed, lb-mol/hr	0.001227	--	0.001752	--	0.002221	--
CO_2 Changed, lb-mol/hr	-0.00003	--	-0.000035	--	0.00015	--
H_2 Consumed, lb-mol/hr	0.003882	--	0.005592	--	0.006553	--
H_2O Produced, lb-mol/hr	0.001662	--	0.002254	--	0.00241	--
CH_4 Produced, lb-mol/hr	0.001398	--	0.002117	--	0.002510	--
C_2H_6 Consumed, lb-mol/hr	0.000068	--	0.000151	--	0.000201	--
Space Velocity, SCF/hr-cu ft†	17,793	--	26,198	--	34,939	--

* CO concentrations, which are within the range of 0.0% to 0.1%, cannot be accurately detected by the current calibration of the mass spectrometer and gas partitioner. Therefore, unless otherwise indicated, zero (0) means that equilibrium conversion concentration is achieved.

† Space velocity is calculated based on a measured bulk density of 66.49 lb/cu ft catalyst.

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Table 2, Part 3. METHANATION CATALYSIS – CATALYST EVALUATION (LDI Catalyst Co. LDI X-826 Catalyst, 1/8-Inch Cylinders, 22.29 g)

Run No.	482		483		484	
Time, hr	383		407		431	
Basis for Analysis	Dry	Wet	Dry	Wet	Dry	Wet
Pressure, psig	200	200	500	500	502	502
Reactor Temperature, inlet, °F	480	480	410	410	446	446
Reactor Temperature, quarter bed, °F	565	565	535	535	554	554
Reactor Temperature, middle bed, °F	558	558	526	526	540	540
Reactor Temperature, outlet, °F	610	610	684	684	657	657
Furnace Temperature, top zone, °F	483	483	482	482	481	481
Furnace Temperature, bottom zone, °F	548	548	547	547	548	548
Flow Rate, feed, lb-mol/hr	0.03305	0.03305	0.06585	0.06585	0.04955	0.04955
Flow Rate, H_2O, lb-mol/hr	0	0	0	0	0	0
Feed Composition, mol %						
H_2	11.2	11.2	13.2	13.2	13.1	13.1
N_2	2.5	2.5	2.7	2.7	2.3	2.3
CH_4	79.8	79.8	77.3	77.3	78.8	78.8
C_2H_6	0.2	0.2	0.3	0.3	0.2	0.2
CO_2	2.4	2.4	2.6	2.6	2.3	2.3
CO	3.4	3.4	3.8	3.8	3.2	3.2
He	0.5	0.5	0.1	0.1	0.1	0.1
H_2O	0	0	0	0	0	0
Total	100.0	100.0	100.0	100.0	100.0	100.0
Flow Rate, product, lb-mol/hr	0.02978	0.03087	0.05801	0.06097	0.04419	0.04657
Flow Rate, H_2O in product, lb-mol/hr	0	0.00109	0	0.00295	0	0.00238
Product Composition, mol %						
H_2	1.2	1.2	1.0	0.9	2.5	2.4
N_2	2.8	2.7	3.0	2.9	2.6	2.4
CH_4	92.71	89.4	93.18	88.6	92.5	87.8
C_2H_6	0	0	0	0	0	0
CO_2	2.7	2.6	2.7	2.6	2.3	2.2
CO*	0.09	0.09	0.02	0.02	0	0
He	0.5	0.5	0.1	0.1	0.1	0.1
H_2O	0	3.51	0	4.88	0	5.1
Total	100.00	100.00	100.00	100.00	100.0	100.0
CO Consumed, lb-mol/hr	0.001097	--	0.002490	--	0.001586	--
CO_2 Changed, lb-mol/hr	0.000011	--	-0.000146	--	-0.000122	--
H_2 Consumed, lb-mol/hr	0.003345	--	0.00811	--	0.005386	--
H_2O Produced, lb-mol/hr	0.001090	--	0.002949	--	0.002379	--
CH_4 Produced, lb-mol/hr	0.001230	--	0.003160	--	0.001839	--
C_2H_6 Consumed, lb-mol/hr	0.000066	--	0.000198	--	0.000099	--
Space Velocity, SCF/hr-cu ft†	17,251	--	34,369	--	25,862	--

* CO concentrations, which are within the range of 0.0% to 0.1%, cannot be accurately detected by the current calibration of the mass spectrometer and gas partitioner. Therefore, unless otherwise indicated, zero (0) means that equilibrium conversion concentration is achieved.

† Space velocity is calculated based on a measured bulk density of 66.49 lb/cu ft catalyst.

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Table 2, Part 4. METHANATION CATALYSIS – CATALYST EVALUATION (LDI Catalyst Co. LDI X-826 Catalyst, 1/8-Inch Cylinders, 22.29 g)

Run No.	485		486		487	
Time, hr	503		527		551	
Basis for Analysis	Dry	Wet	Dry	Wet	Dry	Wet
Pressure, psig	52	52	52	52	52	52
Reactor Temperature, inlet, °F	410	410	471	471	440	440
Reactor Temperature, quarter bed, °F	522	522	565	565	549	549
Reactor Temperature, middle bed, °F	528	528	557	557	540	540
Reactor Temperature, outlet, °F	670	670	618	618	650	650
Furnace Temperature, top zone, °F	480	480	480	480	479	479
Furnace Temperature, bottom zone, °F	550	550	550	550	550	550
Flow Rate, feed, lb-mol/hr	0.06786	0.06786	0.03312	0.03312	0.05078	0.05078
Flow Rate, H_2O, lb-mol/hr	0	0	0	0	0	0
Feed Composition, mol %						
H_2	11.1	11.1	13.7	13.7	13.3	13.3
N_2	2.7	2.7	2.4	2.4	2.4	2.4
CH_4	80.2	80.2	77.6	77.6	77.9	77.9
C_2H_6	0.2	0.2	0.2	0.2	0.2	0.2
CO_2	2.3	2.3	2.5	2.5	2.5	2.5
CO	3.4	3.4	3.5	3.5	3.5	3.5
He	0.1	0.1	0.1	0.1	0.2	0.2
H_2O	0	0	0	0	0	0
Total	100.0	100.0	100.0	100.0	100.0	100.0
Flow Rate, product, lb-mol/hr	0.06025	0.06565	0.02991	0.03090	0.04551	0.04683
Flow Rate, H_2O in product, lb-mol/hr	0	0.005404	0	0.00989	0	0.01319
Product Composition, mol %						
H_2	0.4	0.4	3.3	3.2	3.5	3.4
N_2	3.0	3.0	2.7	2.6	2.7	2.6
CH_4	93.0	85.3	90.8	87.9	90.55	87.9
C_2H_6	0.1	0.1	0.1	0.1	0	0
CO_2	3.1	2.8	2.9	2.8	2.8	2.7
CO*	0.3	0.3	0.09	0.09	0.25	0.24
He	0.1	0.1	0.1	0.1	0.2	0.2
H_2O	0	8.2	0	3.21	0	2.96
Total	100.0	100.0	100.0	100.00	100.00	100.00
CO Consumed, lb-mol/hr	0.002126	--	0.001138	--	0.001664	--
CO_2 Changed, lb-mol/hr	0.00031	--	0.000040	--	0.000005	--
H_2 Consumed, lb-mol/hr	0.007291	--	0.003545	--	0.005159	--
H_2O Produced, lb-mol/hr	0.005405	--	0.000989	--	0.001319	--
CH_4 Produced, lb-mol/hr	0.001609	--	0.001452	--	0.001656	--
C_2H_6 Consumed, lb-mol/hr	0.000075	--	0.000019	--	0.000102	--
Space Velocity, SCF/hr-cu ft†	35,419	--	17,289	--	26,503	--

* CO concentrations, which are within the range of 0.0% to 0.1%, cannot be accurately detected by the current calibration of the mass spectrometer and gas partitioner. Therefore, unless otherwise indicated, zero (0) means that equilibrium conversion concentration is achieved.

† Space velocity is calculated based on a measured bulk density of 66.49 lb/cu ft catalyst.

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This set of experiments showed that, under ideal operating conditions, the LDI X-826 catalyst is as active and as durable as other high-activity catalysts tested before, such as the Harshaw NiO104T, G-87p, and MC-100 catalysts.

Task 4. Materials Testing

The Appendix summarizes the results of material testing of refractories done over the past several months of the project.

It was found that high-alumina refractories with calcium aluminate bonds perform satisfactorily in a condensing acid-gas atmosphere under 1000-psig pressure and a temperature of 540°F. Under the same conditions, a phosphate bond material would show substantial losses of material and strength.

Task 6. Engineering Services

A bench-scale unit simulating the HYGAS steam/oxygen gasifier is being used to help establish the operating conditions for high carbon conversion (90% or more) of the Peabody No. 10 mine coal. The volatile matter content in the raw coal was reduced to about 5% to simulate steam/oxygen gasifier char feed material. The unit being used is a 6-inch reactor with a six-cone assembly steam/oxygen gas distributor. The cones have the same configuration as the HYGAS steam/oxygen gasifier gas distributor.

Test work began on February 22 after sufficient material was prepared. Preheated nitrogen was passed through the reactor for initial heat-up. Feeding of the char was begun when the unit reached 850°F. Oxygen and steam were gradually introduced into the unit as the nitrogen flow was reduced to maintain a constant superficial velocity in the reactor. The reactor temperature was raised to 1800°F, and the pressure was 398 psig. The unit was running very smoothly, and a gas sample was obtained. Three hours after the initial introduction of oxygen into the unit, there was a pressure upset in the reactor as the last bit of nitrogen was cut out. The result was a jammed solids screw feeder. It could not be freed, and the test was terminated. The reactor was opened and inspected. It was found to be clean, and solids samples were obtained to determine the degree of carbon conversion.

A second, successful PDU test was carried out on February 28. Results from these two tests are presented in Table 3. In the second test, low-volatile-matter char, simulating the steam/oxygen reactor feed, was again continuously fed to the 6-inch reactor where steam, oxygen, and nitrogen were introduced to maintain fluidization in the bed. The bed temperature was maintained at about 1800°F. Results of both tests show that Peabody No. 10 mine coal was gasified to yield carbon conversions of about

Table 3. PRELIMINARY RESULTS OF PDU STEAM-OXYGEN GASIFICATION OF PEABODY NO. 10 MINE BITUMINOUS CHAR

	Test 1	Test 2
Spent Char Analysis, %		
Ash	92.22	77.84
Carbon	7.18	20.58
Hydrogen	0.12	0.27
Sulfur	0.40	1.15
Nitrogen	0.08	0.16
Oxygen	0	0
Mean Residence Time, min	22	25
Superficial Gas Velocity, ft/s	0.88	0.75
lb O_2/lb Coal in Steam-Oxygen Gasifier	0.47	0.44
lb Steam/lb Coal in Steam-Oxygen Gasifier	2.45	2.78
Carbon Gasified in Steam-Oxygen Gasifier (By Gas Balance)	88	86
High-Temperature Bed, ^{o}F	1820	1820
Pressure, psig	399	398

88% in the steam/oxygen gasifiers at a temperature level of 1820°F, well within the design temperature of the HYGAS pilot plant.

The fluidization curve was determined for pretreated Peabody No. 10 mine coal. The data show that complete fluidization is observed at 0.45 ft/s. As a result, future char cooler fluidization velocity will be kept above 0.45 ft/s at all times to ensure good fluidization and even temperature distribution in the bed.

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CONCLUSIONS

This quarter was highlighted by the initiation of tests with Peabody No. 10 mine bituminous coal. Nonagglomerating coal was produced by the pretreater for the gasifier feed material. Post-run inspection of the gasifier showed that it was free of clinkers. Further tests will be conducted with this coal.

The objective of this series of tests is to –

- Investigate operating conditions necessary to increase coal conversion to the 90%+ level
- Minimize the degree of pretreatment required to produce a nonagglomerating feed for the gasifier with bituminous coal
- Develop equipment and procedures for reinjecting the dust collected in the overhead hot-gas cyclone into the gasification zone for consumption.

At the end of this quarter, the HYGAS pilot plant was being readied for Test 60.

APPENDIX. Performance of Castable Refractory Materials in Condensing Acid-Gas Atmospheres: Final Summary Report

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OBJECTIVE

The objective of this work was to test the performance of some of the commonly used castable refractory materials in a condensing acid-gas environment, present either in water or gas-cooled high-pressure coal gasification reactors.

BACKGROUND

As construction of large-scale demonstration coal gasification plants is rapidly becoming a reality, several reactor wall refractor designs are possible. A conventional reactor wall could consist of a hard-face castable exposed to temperatures and pressures of up to 2000°F and 1000 psig, backed by layers of insulating brick, lightweight castable, or both. These refractory materials will be encased by a wide variety of reactor wall materials, ranging from carbon-steel to special alloys, depending on the reactor operating conditions. The reactor wall could be either surrounded by an annular jacket for gas- or steam-cooling or simply be exposed to ambient air, as shown in Figures A-1 and A-2.

By virtue of the thermal gradients existing in reactor walls of the type shown in Figure A-2, the refractory in the vicinity of the metal wall is always exposed to condensing acid-gas atmospheres. However, the reactor walls, as illustrated in Figure A-1, could be dry during gasifier operation and have a chance to come in contact with condensates between runs. Therefore it is essential to determine the performance of both the hard-face and the insulating refractories in condensing acid-gas atmospheres at about 1000 psig.

Steel walls are known to react with condensing acid-gas; therefore, commercial gasification plants could employ special claddings.

The equipment and tests used in this investigation were designed for studying the performance of selected castable refractory materials, patching materials, and block and blanket insulating materials that have potential use in the construction of coal gasification reactors.

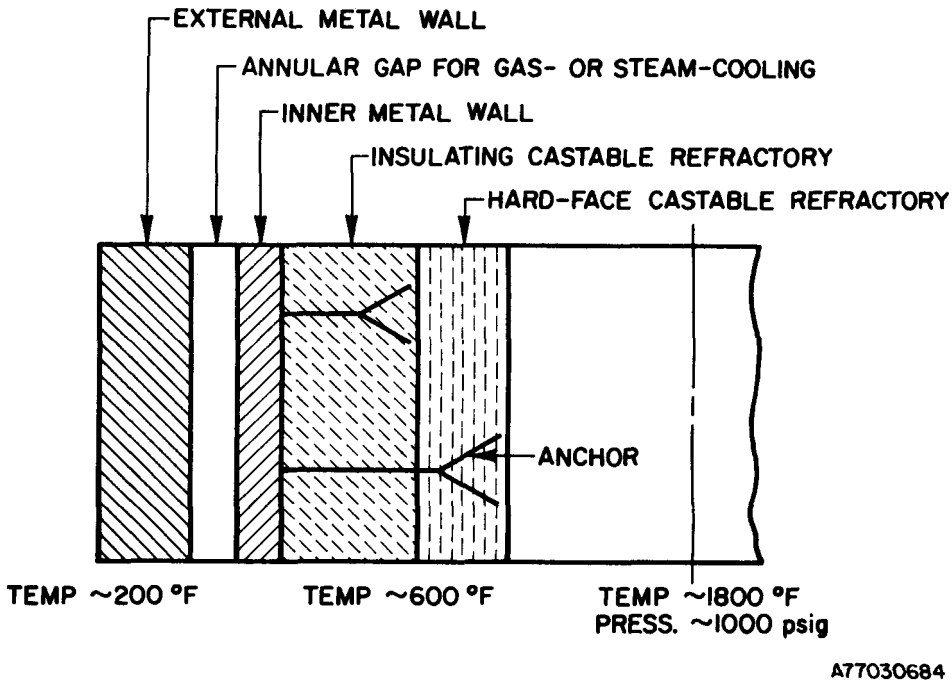

Figure A-1. EXAMPLE OF A GAS-COOLED REACTOR WALL CONSTRUCTION

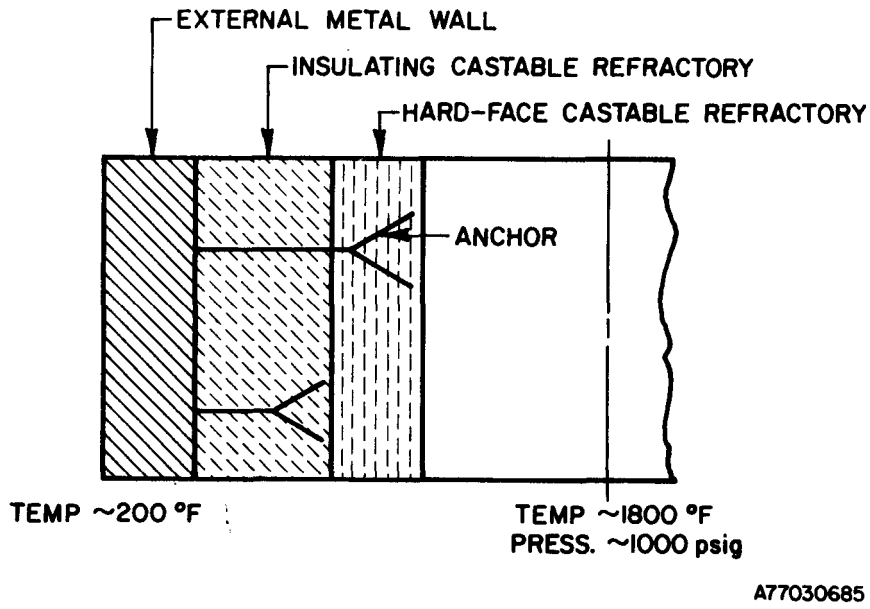

Figure A-2. EXAMPLE OF AMBIENT AIR-COOLED REACTOR WALL CONSTRUCTION

DESCRIPTION OF EXPERIMENTAL EQUIPMENT

The selected materials were cast inside 9-inch-long flanged sections of 6-inch Schedule 40 carbon-steel pipe (Type-A-106). The flanged sections are equipped with a gas stop, four anchor bolts, and a condensate return shield as shown in Figure A-3. The flanged sections containing the cast and cured castable are stacked and installed on top of a 2-inch ID pressure vessel containing water, as illustrated in Figure A-4. The equipment assemblies employed in this investigation are shown in Figures A-4 through A-6. The pressure vessel is designed with adequate wall thickness and surrounded by electrical heaters to maintain the entire test assembly at the required pressure and temperature. A blind flange covering the topmost section has a provision for introducing thermocouples to predetermined locations in the test sections. The test facility is equipped with gas and water feed inlets, sampling ports, and instrumentation to monitor temperature and pressure and to provide temperature control for the electrical heaters.

The test materials were usually cast and cured according to the instructions provided by the manufacturer before being subjected to condensing steam and acid-gas environments.

DESCRIPTION OF THE TEST PROGRAM

Three separate tests were conducted with selected materials characteristic of the general categories of castables that could be used in constructing coal gasifiers. The materials selected and the test conditions are listed in Table A-1. Tests of some materials were repeated to check the reproducibility of their behavior. The assemblies of materials for the three tests are shown in Figures A-2 through A-4. The test conditions were chosen on the basis of expected coal gasification atmospheres and are considered severe enough to reveal the effect of condensing acid-gas on refractories under coal gasification conditions.

At the beginning of each test, the flanged sections containing the cast and cured castable materials were assembled and pressure tested at 540°F and 1000 psig. After cooling, the water container was filled with the required amount of water, then pressurized with carbon dioxide to 185 psig and with hydrogen sulfide to 210 psig. When heated to about 540°F and 1000 psig, the following gas composition was calculated to exist at the beginning of each test:

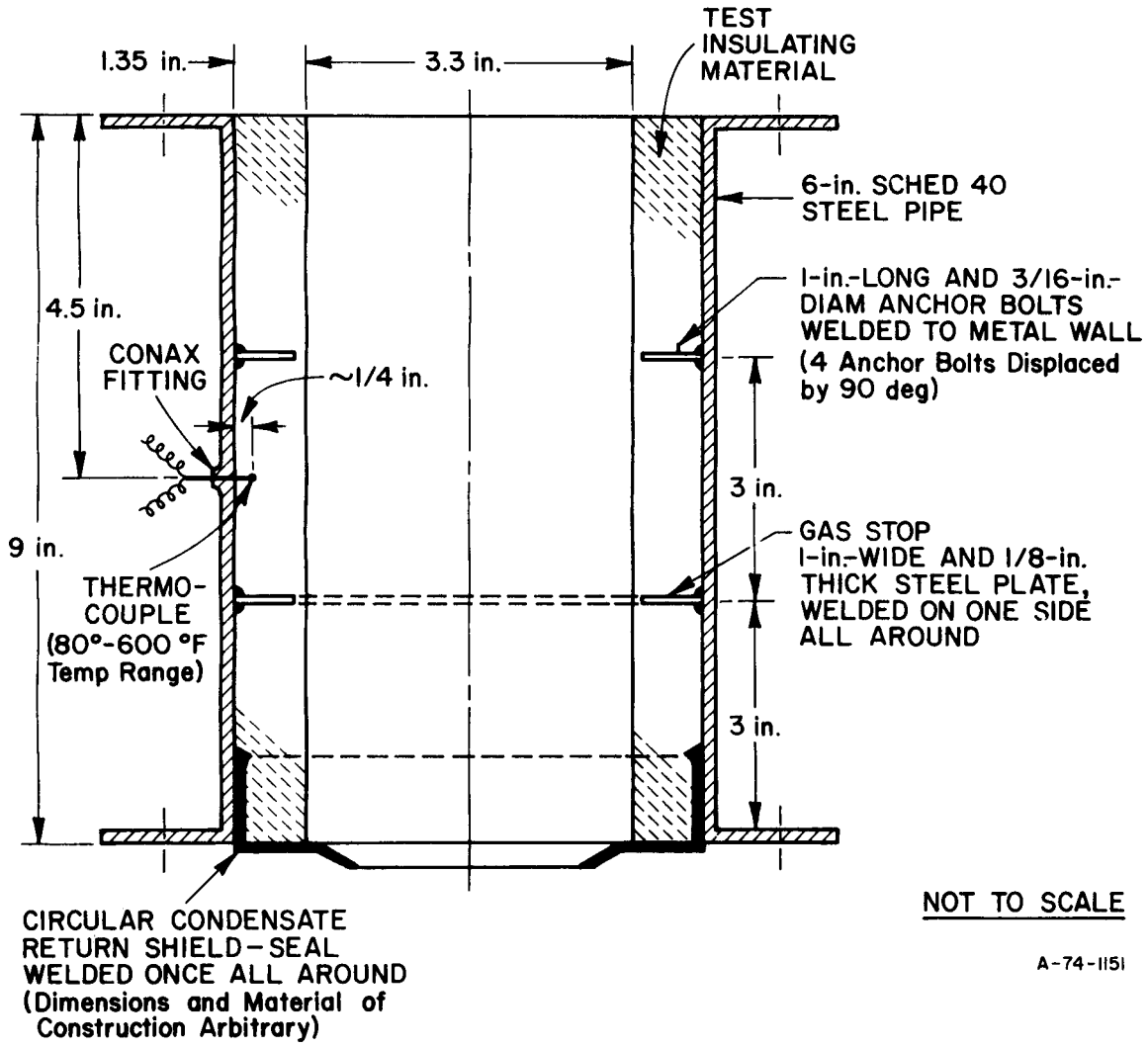

Figure A-3. CONSTRUCTION DETAILS OF THE FLANGED INSULATING MATERIAL TEST SECTION

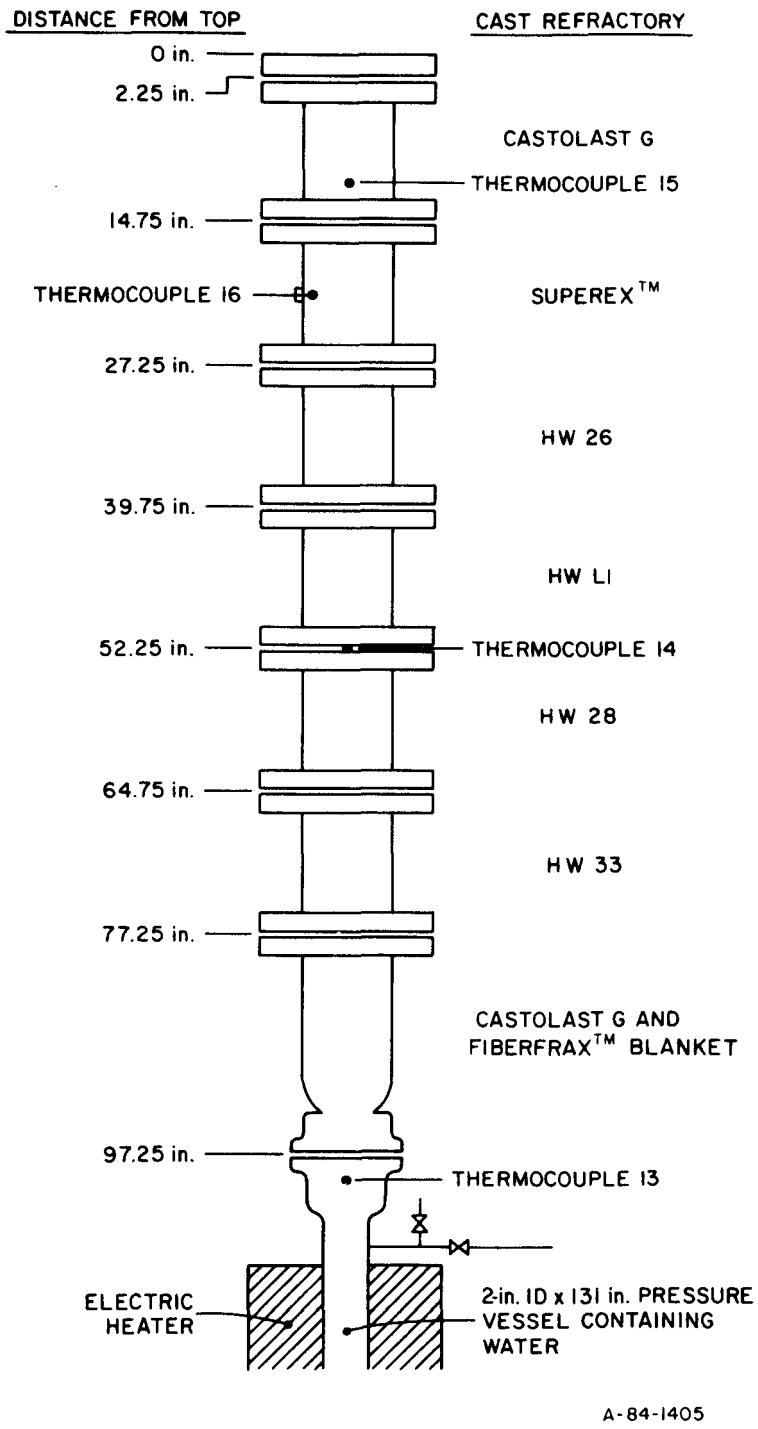

Figure A-4. EQUIPMENT SETUP FOR TESTING CASTABLE REFRACTORIES

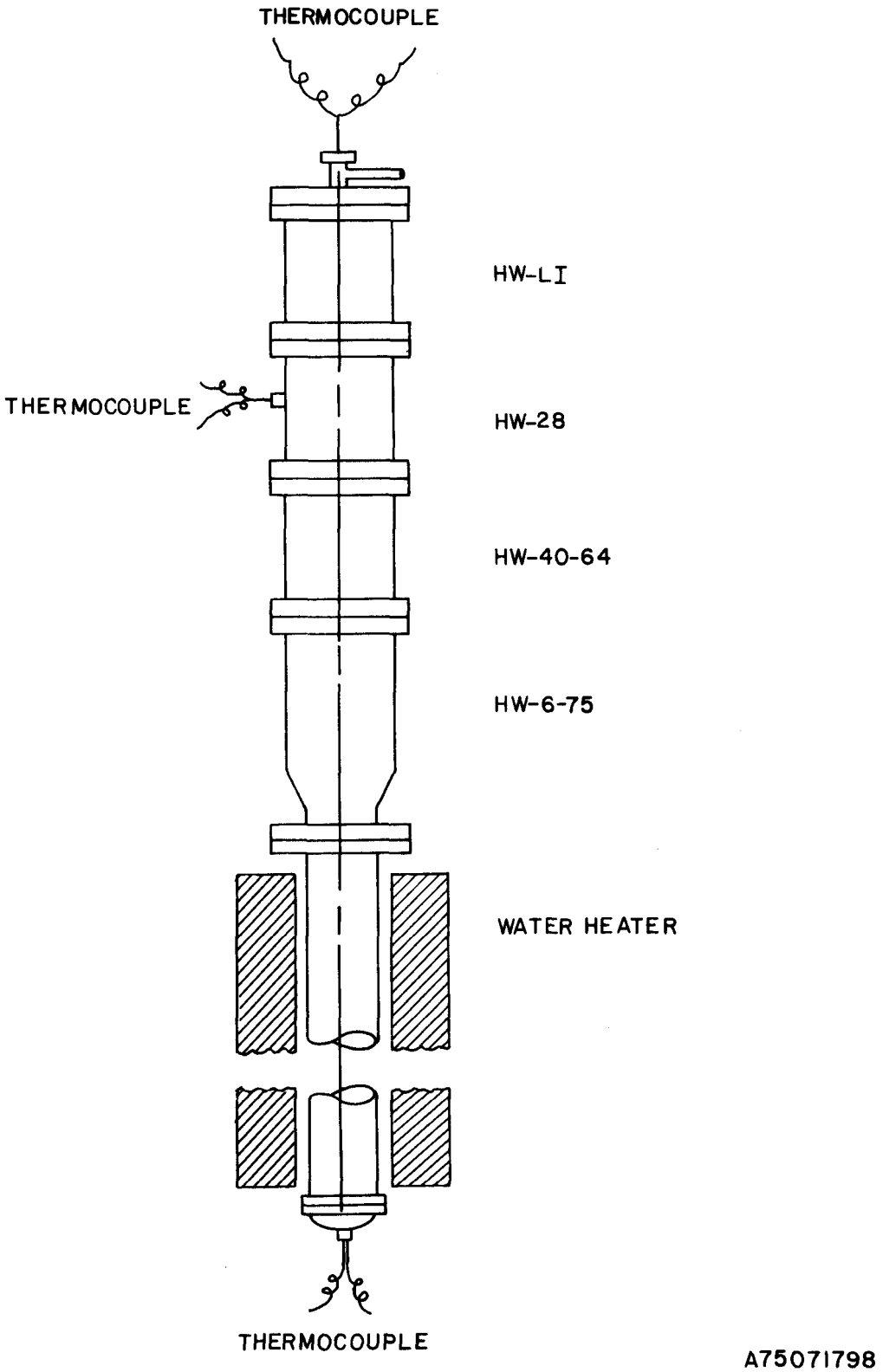

Figure A-5. ASSEMBLY OF CASTABLE REFRACTORY MATERIALS

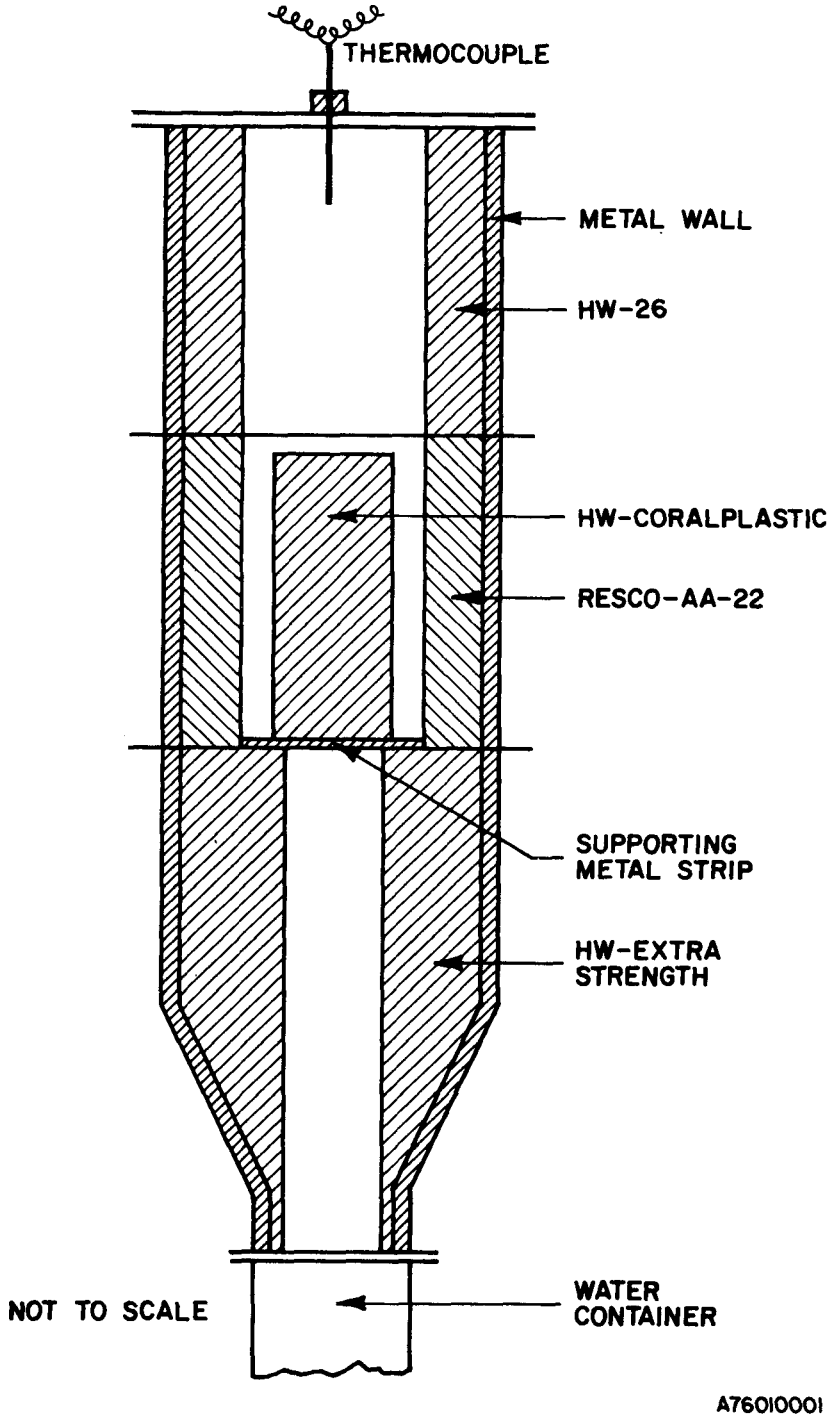

Figure A-6. ASSEMBLY OF CERAMIC REFRACTORY MATERIALS FOR TESTING IN AN ACID-GAS ENVIRONMENT

Table A-1. SELECTED CASTABLE REFRACTORIES AND TEST CONDITIONS

Test	Castable Materials Selected	Test Conditions
I	HW-Castolast G HW-33 HW-28 HW-26 HW-LI Superex™ * Fiberfrax™†	30-day exposure to steam at 1000 psig and its saturation temperature (about 540°F), followed by a 30-day exposure to condensing acid-gas at 540°F and 1000 psig.
II	HW-6-75 HW-40-64 HW-28 HW-LI	30-day exposure to steam at 1000 psig and its saturation temperature (about 540°F), followed by a 30-day exposure to condensing acid-gas at 540°F and 1000 psig.
III	HW-ES HW-26 HW-Coralplastic ‡ Resco AA-22 ‡	30-day exposure to steam at 1000 psig and its saturation temperature (about 540°F), followed by a 30-day exposure to condensing acid-gas at 540°F and 1000 psig.

* Block insulation.

† Blanket insulation.

‡ Could be used as patching materials.

	vol %
Hydrogen Sulfide	≃1
Carbon Dioxide	30
Steam $H_2O(g)$	69

When the test materials are exposed to condensing steam only, the test unit is pressurized with nitrogen to about 210 psig before turning on the electrical heaters.

The test materials are inspected for general appearance and physical coherence after exposure for 30 days to saturated steam and for 30 additional days to condensing acid-gas. At the end of the latter period, the flanged sections are disassembled and dried at room temperature, then sections of samples are cut out for detailed physical and chemical analyses. Beginning with the second test, reference samples were cast and cured under similar conditions for subsequent comparison with samples exposed to steam and condensing acid-gas.

At the beginning, during, and after the tests, gas and water samples were taken for a detailed analysis.

A summary of the results of all the tested castables and other kinds of insulating materials is shown in Tables A-2 through A-4. Although most of the tested materials were from Harbison-Walker Refractories, other refractory materials with a comparable composition are expected to show similar behavior.

DISCUSSION

Test I

Inspection of the refractory sections after the first 30-day exposure to saturated steam at 1000 psig showed that both the block and the blanket insulating materials (Superex™ and Fiberfrax™), were severely attacked. The effect of steam exposure and a subsequent 30-day exposure to condensing acid-gas at 540°F and 1000 psig, is shown in Figures A-7 and A-8. A physical inspection of the castables HW-LI, HW-26, HW-28, and HW-33 did not reveal any visible deformations; several rust-colored areas were seen on their convex surfaces.

At the end of Test I, several sections of the castables were cut and subjected to a detailed physical and chemical analysis at the Garber Research Center, Harbison-Walker

Table A-2. PHYSICAL AND CHEMICAL ANALYSIS OF CASTABLE MATERIALS EXPOSED TO CONDENSING STEAM AND CONDENSING ACID-GAS IN TEST I

Castable Material Sample	HW-33		HW-28		HW-26		HW-LI		HW-Castolast G	
	Used[a]	Reference[b]	Used	Reference	Used	Reference	Used	Reference	Used	Reference
Physical Properties										
Bulk Density,[c] lb/CF	105	96[d]	91	100[d]	63	61[d]	56	55[d]	172	167[d]
Bulk Density,[d] lb/CF	--[g]	--[g]	91	100	65	61	62	55	167	167
Modulus of Rupture, psi	--[g]	--[g]	--[g]	--[g]	--[g]	--[g]	--[g]	--[g]	--[g]	--[g]
Cold-Crushing Strength, psi	--[g]	--	950	1390	470	480	470	550	7860	9060
Chemical Analysis										
Calcined Basis, %[e]										
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_2O_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_2O_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_2O)	0.01	0.20	0.01	1.1	0.01	1.6	0.01	1.8	0.08	0.1
Potash (K_2O)	0.01	0.20	0.06	1.1	0.16	1.6	0.16	1.8	0.02	0.1
Total	99.9	100.0	99.4	100.0	99.8	100.0	99.8	100.0	100.4	100.0
Dry Basis[f]										
Sulfur Trioxide (SO_3), %	0.11	--	1.69	--	1.18	--	0.34	--	0.10	--
Total Carbon (C), %[c]	0.72	--	0.05	--	0.09	--	0.17	--	0.07	--
Loss on Ignition, %	11.55	--	6.51	--	8.51	--	16.33	--	9.10	--
Loss on Ignition, %[d]	--	--	5.88	--	7.72	--	14.20	--	8.72	--
pH	9.10	--	8.35	--	8.10	--	8.25	--	11.10	--

[a] Sample exposed to condensing steam for 30 days and to condensing acid-gas for 30 days at 540°F and 1000 psig.

[b] Analysis based on quality control average values.

[c] As received.

[d] After drying at 230°F.

[e] Alkalies by flame photometer, all others by spectrograph.

[f] Total carbon and sulfur trioxide by Leco furnace.

[g] Insufficient sample to run.

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Table A-3. PHYSICAL AND CHEMICAL ANALYSIS OF CASTABLE MATERIALS EXPOSED TO CONDENSING STEAM AND CONDENSING ACID-GAS IN TEST II

Castable Material Sample	HW 40-64		HW 6-75		HW-LI		HW-28	
	Used[a]	Reference[b]	Used	Reference	Used	Reference	Used	Reference
Physical Properties								
Bulk Density, lb/CF	87	76 (86)[e]	101	94 (100)	62	60 (55)	94	93 (100)
Modulus of Rupture, psi	140	120 (210)	--[f]	70 (560)	--[f]	110 (140)	400	420 (310)
Cold-Crushing Strength, psi	580	200 (810)	1180	160 (1110)	--[f]	470 (550)	1260	970 (1390)
Chemical Analysis[c]								
Calcined Basis, %								
Silica (SiO_2)	44.3	45.2	93.10[g]	97.50[g]	46.9	45.0	37.8	38.8
Alumina (Al_2O_3)	44.9	43.7	1.48	1.69	39.5	40.8	53.4	52.7
Titania (TiO_2)	2.29	2.10	0.05	0.05	0.25	0.25	1.47	1.36
Iron Oxide (Fe_2O_3)	1.10	1.10	0.20	0.18	0.11	0.3	1.18	1.04
Lime (CaO)	5.52	6.05	0.07	0.07	11.8	12.3	4.83	4.85
Magnesia (MgO)	0.21	0.18	0.01	0.01	0.06	0.08	0.07	0.06
Phosphorus Pentoxide (P_2O_5)	--	--	0.10	2.44	--	--	--	--
Soda (Na_2O)	0.14	0.40	0.48	2.33	0.80	0.14	0.10	0.30
Potash (K_2O)	0.59	0.81	0.25	0.09	0.70	0.25	0.29	0.44
Lithia (Li_2O)	0.03	0.08	--	0.01	0.02	0.01	0.02	0.05
Total	99.1	99.6	100.00	100.00	100.1	99.1	99.2	99.6
Dry Basis, %								
Sulfur Trioxide (SO_3)[d]	0.02	0.29	0.22	1.01	0.0	0.23	0.50	0.02
Loss on Ignition	6.35	6.30	1.50	1.20	4.66	16.50	14.70	7.62

[a] Sample exposed to condensing steam for 30 days and to condensing acid-gas for 30 days at 540°F and 1000 psig.

[b] Reference sample cast but uncured.

[c] Alkalies by flame photometer, phosphorous pentoxide by wet chemistry, all others by x-ray flourescence.

[d] Sulfur trioxide by Leco furnace.

[e] Numbers in parentheses are based on quality control average values.

[f] Insufficient sample to run.

[g] Calculated by difference.

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Table A-4. PHYSICAL AND CHEMICAL ANALYSIS OF CASTABLE MATERIALS EXPOSED TO CONDENSING STEAM AND CONDENSING ACID-GAS IN TEST III

Castable Material	HW-ES Castable		HW-26 Castable		Resco AA-22	
Sample	Used[a]	Reference[b]	Used	Reference	Used	Reference
Physical Properties						
Bulk Density, lb/CF	130	127	63	62	173	158-164
Modules of Rupture, psi	--	--	--	--	1050	1490-1780
Cold-Crushing Strength, psi	5310	6290	250	530	3090	6330-9780
Chemical Analysis[c]						
Calcined Basis, %						
Silica (SiO_2)	37.0	39.1	33.6	37.4	0.90	0.66
Alumina (Al_2O_3)	45.2	33.7	59.8	52.8	See Below	See Below
Titania (TiO_2)	2.56	1.9	0.76	0.5	0.04	0.04
Iron Oxide (Fe_2O_3)	3.35	6.9	1.06	0.6	0.14	0.26
Lime (CaO)	10.6	14.8	4.27	7.0	0.36	0.14
Magnesia (MgO)	0.64	1.3	0.21	0.1	3.00	3.13
Phosphorous Pentoxide (P_2O_5)	0.1	--	0.1	--	5.5	5.58
Soda (Na_2O)	0.07	2.3	0.05	1.6	0.02	0.07
Potash (K_2O)	0.42	Total	0.08	Total	0.01	0.01
Lithia (Li_2O)	0.06	Alkalies	0.01	Alkalies	0.01	0.01
Total	100.0	100.0	99.9	100.0	10.0	9.9
By Difference						
Alumina (Al_2O_3)	--	--	--	--	90.0	90.1
Total					100.0	100.0
Dry Basis, %						
Loss on Ignition	7.21	--	7.15	--	1.66	2.61
Sulfur Trioxide (SO_3)[d]	0.68	--	0.81	--	0.04	--
Soluble Phosporous Pentoxide (P_2O_5)	0.0	--	0.0	--	0.2	2.88

[a] Sample exposed to condensing steam for 30 days and to condensing acid-gas for 30 days at 540°F and 1000 psig.

[b] Reference sample cast and cured only.

[c] Alkalies by flame photometer, phosphorous pentoxide by wet chemistry, all others by spectrograph.

[d] Sulfur trioxide by Leco furnace.

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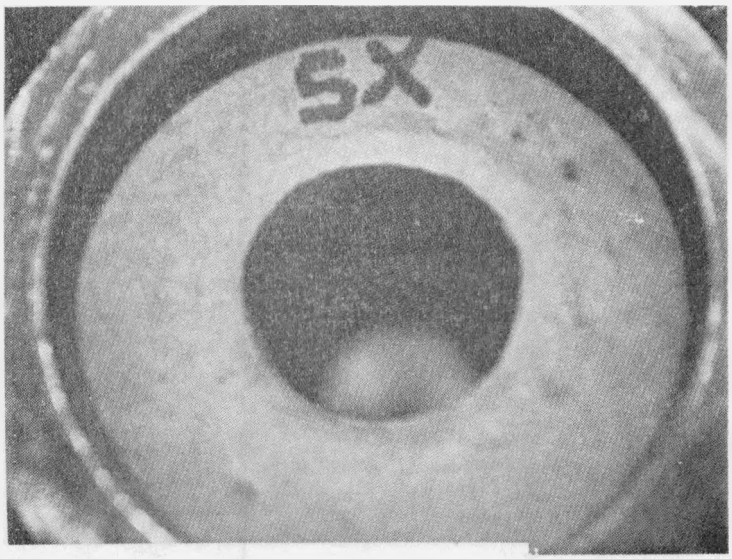

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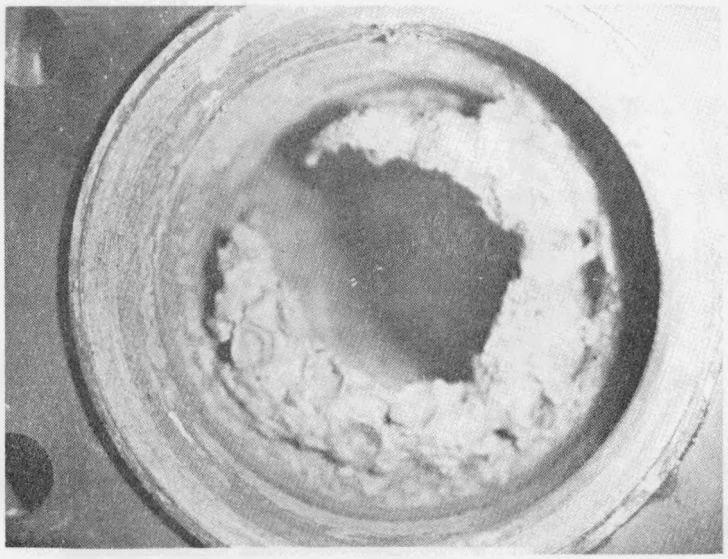

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Figure A-7. PERFORMANCE OF SUPEREX^{TM} INSULATING MATERIAL IN A CONDENSING ACID-GAS (1000 psig and 540°F)

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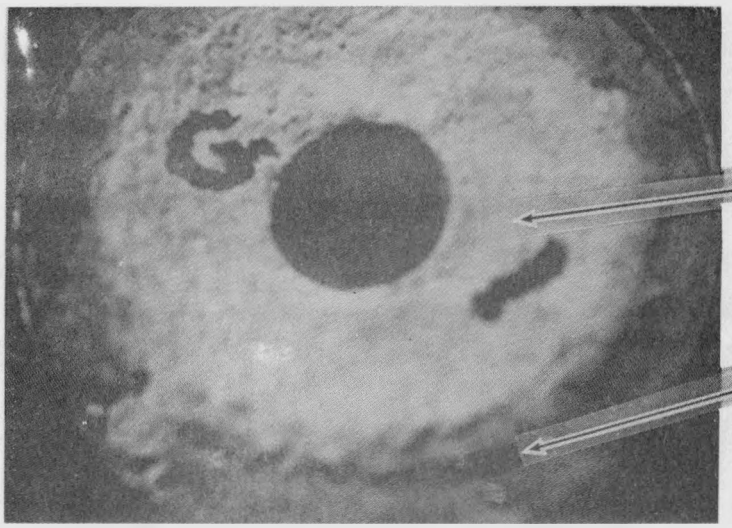

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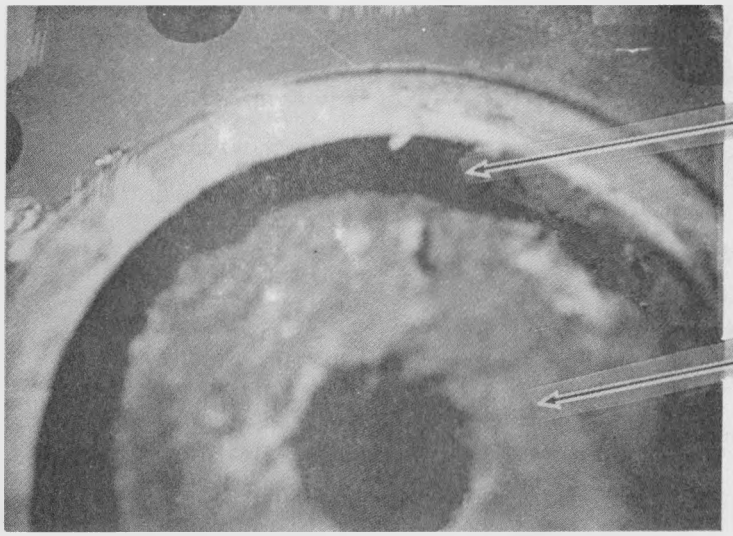

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Figure A-8. PERFORMANCE OF CASTOLAST-G AND FIBERFRAX™ INSULATION MATERIALS IN A CONDENSING ACID-GAS (1000 psig and 540°F)

Refractories, Pittsburgh, Pa. The effects of the condensing acid-gas on the refractory properties can be evaluated from the results shown in Table A-2. There is very little change in the bulk density except for HW-28, which reports a 10% reduction. The observed loss in cold-crushing strength of HW-28, HW-LI, and HW-Castolast G are about 30%, 15%, and 13%.

From the chemical analyses, there is a slight reduction in the silica content of the castables, except for HW-Castolast G. The largest change in silica among the tested castables is in HW-28. The castables HW-33 and HW-Castolast G did not show any significant alteration in their chemical properties.

Table A-2 shows that the soda (Na_2O) and potash (K_2O) contained in the tested castables were almost completely leached by contact with steam and condensing acid-gas. The soda and potash probably form hydroxides and are washed out of the castables in the presence of high-pressure condensing steam. Another possibility involves an interaction between the alkalies and silica, resulting in silicates. This might account for the slight depletion in silica observed in most of the castables. The loss of Na_2O and K_2O is not considered 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 loss of the calcium aluminate bonding and could result in a loss of refractory strength. Of the two castables (HW-26 and HW-28) that show the highest loss in CaO, HW-28 appears to show a 32% reduction in its cold-crushing strength.

An enrichment of the tested refractory materials with iron sulfide, reported as Fe_2O_3 and SO_3, was noticed in HW-26 and HW-28. This increase could be due to the formation of an insoluble iron sulfide compound by the interaction of H_2S in the acid-gas with the carbon-steel walls.

The slight gains in Al_2O_3 in HW-LI, HW-26, and HW-28 could have been the result of the loss in other elements. The castable HW-LI showed a slight loss of SiO_2 and no change in Fe_2O_3 content.

The loss on ignition was determined on as-received samples that contained significant moisture.

Test II

Two of the materials (HW-LI and HW-28) used in Test I were retested in Test II. Of the four tested castables HW 6-75, a vitreous silica refractory, was found to be disintegrated after the first 30-day exposure to saturated steam at 1000 psig. This castable material disintegrated further after another 30-day exposure to condensing

acid-gas. The remaining castables, HW 40-64, HW-LI, and HW-28, did not show any visual decomposition.

In comparing the used and reference analyses (based on quality control averages, shown in Table A-3) note that there is no significant change in bulk density during the test. HW 40-64 shows a reduction in the modulus of rupture and cold-crushing strength compared to the quality control average values. However, this could not be explained, as there was no significant change in the chemical analysis.

Even though the vitreous silica refractory (HW 6-75) did not show any change in the bulk density or cold-crushing strength, it was found to be severely corroded by saturated steam and condensing acid-gas.

A detailed mineralogical examination performed at the Garber Research Center, Pittsburgh, Pa., showed that the castable HW 6-75 was weakened by disruption of the acid-resistant bond and partial devitrification of amorphous SiO_2. This could probably be attributed to the observed loss in P_2O_5, as this castable uses a phosphate bond.

Due to inadequate samples, the modulus of rupture and the cold-crushing strength of HW-LI could not be determined. Comparing the chemical analysis, slight losses in CaO and Al_2O_3 and no change in silica were observed in Test II. In Test I, HW-LI lost only some silica.

The castable HW-28 showed a little reduction in bulk density and cold-crushing strength compared with the quality control average values. However, the difference in values for this material is lower in Test II than in Test I. Also, unlike Test I, this chemical analysis does not show any significant change in the silica content for HW-28. Because the change in chemical analysis is insignificant for this material, the reported high loss on ignition may be due to an improperly dried sample.

The tested sample of castable HW 40-64 has a typical density but a lower modulus of rupture and cold-crushing strength compared with the quality control averages. This, however, could not be explained from the chemical analysis, as this material reports only slight losses in silica and lime.

Test III

Four refractories were involved in Test III. The three castables HW-26, HW-ES, and Resco AA-22 were cast in the flanged carbon-steel cylindrical sections. HW-26 was tested earlier (Test I). The fourth material (HW-Coralplastic), which is commonly used for patching purposes, is available in the form of a slab. It was cut in the shape of a brick and supported on a thin metal strip in the annular speck within the section

containing Resco AA-22. At the end of the 30-day test with saturated steam at 1000 psig, the HW-Coralplastic disintegrated into moist lumps. As a result the Coralplastic pieces were removed from the test section and were, therefore, not exposed to the condensing acid-gas. The failure of Coralplastic could be due to the penetration of high-pressure steam into the voids of the Coralplastic slab and subsequent disintegration of its phosphate bond. Coralplastic is normally worked into holes and gaps of existing refractory linings.

The three castables, HW-26, HW-ES, and Resco AA-22, did not show any physical deformations at the end of Test III, although HW-26 was slightly soft to touch on the surface.

The results in Table A-4 show that the castable HW-ES lost about 15% of its cold-crushing strength, although its bulk density remained unchanged. Although no loss in Al_2O_3 was observed, the reduced CaO content is apparent. It is not certain whether this could be attributed to the loss in cold-crushing strength.

In Test III, castable HW-26 shows about a 50% reduction in cold-crushing strength compared with little or no change in Test I. The bulk density appears to remain unchanged. The chemical analysis shows a significant loss in CaO, which could be responsible for the loss in cold-crushing strength. However, a similar loss of CaO in Test I does not result in any reduction in the cold-crushing strength.

The castable Resco AA-22, which employs a phosphate bond, shows reductions in modulus of rupture and cold crushing strength of at least 30% and 50%; the bulk density is unaffected. There is no change in the chemical analysis of Resco AA-22 at the end of Test III (Table A-4). The loss in strength during the test could not be explained by the reported analysis.

Feedwater and Condensate Analysis

The compositions of feedwater and condensate samples collected during and at the end of the tests were determined by atomic absorption (Table A-5). Although no quantitative conclusions regarding the rate or extent of leaching of the specific test materials can be drawn from these cumulative samples, some general observations can be deduced. From comparing samples 1 and 2 in Table A-6, it appears that saturated steam at 1000 psig is fairly reactive with some of the materials used in Test I, conceivably with the block and blanket insulations. The loss of elements like sodium, potassium, and silicon, in particular from the castables, observed at the end of Test I may explain the enrichment of the condensate with these elements. The higher concentrations of

Table A-5. ANALYSIS OF FEEDWATER AND CONDENSATE FROM REFRACTORY TEST SECTION

Sample No.	Sample ID	Elemental Composition, ppm								Remarks
		Ca	Mg	Al	Na	K	Si	Fe	Others	
					Test I					
1	24878	9.02	0.182	0.44	438.0	0.104	<2.5	--		Feedwater
2	24811	0.56	6.0	6.2	980.0	61.0	590.0	--		Condensate: 15 days contact with steam
3	24875	142.00	24.3	66.8	2280.0	188.0	344	--		Condensate: 30 days contact with steam and 7 days contact with acid-gas
4	25156	325.0	156.0	97.7	2750.0	331.0	1560	--		Condensate: 30 days contact with steam and 23 days contact with acid-gas (pH = 8)
					Test II					
5	28530	38.3	10.3	<1.0	6.3	0.94	<1.0	0.064		Feedwater (pH = 7)
6	28531	0.41	1.9	14.2	1.53	465	0.71	2.64		Condensate: 30 days contact with steam (pH = 12)
7	29074	0.79	0.65	42.2	2590	124	2200	0.25		Condensate: 30 days contact with steam and 30 days contact with acid-gas
					Test III					
8	30480	66.0	20.5	10.6	17.30	0.92	2.9	0.05		Feedwater
9	30481	1.9	4.5	2.8	3600.0	118.00	2.7	0.64		Condensate: 30 days contact with steam and 30 days contact with acid-gas
10	30482	1.8	4.6	2.8	3600.0	117.00	2.7	0.75		Condensate: 30 days contact with steam and 30 days contact with acid-gas

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Table A-6. SUMMARY OF GAS ANALYSIS

Sample No.	Sample ID (Date)	Dry Gas Composition, vol % N_2	CO_2	H_2	H_2S	Others
			Test I			
1	MS7231 (9/20/74)	5.9	88.2	3.2	2.6	0.1
2	MS7230 (9/20/74)	94.1	0.8	0.2	--	4.9
3	MS7281 (9/30/74)	8.8	80.7	9.3	0.93	0.27
			Test II			
4	MS10185 (8/29/75)	17.2	71.6	9.0	1.5	0.7
5	MS10302 (9/16/75)	5.0	76.2	12.8	5.4	0.6
6	MSL1 (9/29/75)	4.1	63.1	27.9	4.1	0.8
			Test III			
10	MSA610 (2/17/76)	14.1	67.1	13.6	4.8	0.4
11	MSA667 (2/25/76)	23.8	58.8	13.4	2.9	1.1
	CL760022 (2/26/76)	--*	--*	--*	--*	(CO = 0.1)
12	MSA843 (3/15/76)	32.1	58.8	7.2	0.3	1.6

* Undetermined.

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calcium, magnesium, and aluminum in the Test I condensate compared with those in Test II or Test III could be due to the decomposition of the block and blanket insulating materials in Test I. The condensates collected at the end of Tests II and III show only the generally observed increase in sodium and potassium. Compared with Tests I and II, the condensate from Test III shows very little silicon; this could be due to the nature of the materials used in this test.

Gas Analysis

A summary of the analysis of gas samples collected during the tests is shown in Table A-6. The analysis of sample 1, reported on a moisture-free basis, is representative of the environment maintained during the test with condensing steam only. The gas composition employed at the beginning of the acid-gas exposure test is shown by the analysis of sample 2. However, with the progress of the test, the batch-operated test unit gradually loses its H_2S, as shown by sample 3. This could be due either to the interaction of H_2S with the test materials or to the exposed carbon-steel surface. As a result, the gas analysis was checked every 10 days, and the H_2S was increased to the required levels by injecting more H_2S into the test assembly.

Interestingly, a significant amount of hydrogen was normally observed in the gas samples taken from the test unit. It is possible that hydrogen could be formed during the test, either by the interaction between steam and the oxidized iron interior surface of the test unit or by the absorption of sulfur from H_2S by the steel walls. This evidence is further supported by the steady increase in hydrogen content with progress in the 30-day tests. Conceivably hydrogen buildup is observed because the tests are conducted in a batch unit – this phenomenon may not even be noticeable in an operating coal gasifier.

The high nitrogen content of samples 10, 11, and 12 could be due to contamination in the feed gas or sampled gases or even due to improper purging of the test unit while switching from steam and nitrogen to steam and acid-gas.

CONCLUSION

- The performance of some commonly used castable materials and a block and an insulating material in a condensing acid-gas atmosphere representative of coal gasification conditions was determined. The operating pressure and temperature were maintained at about 1000 psig and 540°F. At these conditions, the test materials were exposed to condensing steam for 30 days followed by 30 more days of exposure to condensing acid-gas.

- The block and blanket insulating materials were severely decomposed by exposure to condensing steam and acid-gas, and hence are unsuitable for constructing reactor walls susceptible to contact with condensate.

- An acid-resistant phosphate-bonded vitreous silica, HW 6-75 (about 97% SiO_2), was severely decomposed by condensing steam and acid-gas. Almost all of the P_2O_5 contained in this material was lost during the test. Another phosphate-bonded castable, Resco AA-22, showed a loss in the modulus of rupture and cold-crushing strength but did not lose any P_2O_5 during the test. HW-Coralplastic, which also employs a phosphate bond, was disintegrated by high-pressure saturated steam at 1000 psig.

- The calcium aluminate bonded castables such as HW-33 and Castolast G, which contain more than 90% Al_2O_3, showed virtually no change in physical and chemical properties.

- The castables containing 40% to 60% Al_2O_3 and bonded with calcium aluminate include material identified as HW-LI, HW-26, HW-28, and HW-ES. The performance of these materials does indicate possible loss in cold-crushing strength. HW-26 and HW-28, which contain about 50% Al_2O_3, showed losses in cold crushing strength of about 50% and 30%. However, duplicate tests of the samples were not consistent with these results.

- Almost all the materials were leached of their alkalies during the tests. The loss of alkalies is not considered detrimental and could possibly contribute to an increase in their refractory property.

- Some loss in SiO_2 was observed in most of the tested materials.

Recognizing the limitations of this experimental program, some broad generalizations can be made. The high-alumina refractories (greater than 90% Al_2O_3) with calcium aluminate bonds could perform satisfactorily in a condensing acid-gas atmosphere in high-pressure gasifiers. The few samples containing a phosphate bond either lost most of the P_2O_5 or showed a substantial loss in cold-crushing strength and modulus of rupture. With the available information, there is no adequate basis for recommending any particular type of bonding cement for refractories. Both block and blanket insulating materials are unsuitable as gasifier wall construction materials, as they are severely corroded under condensing conditions.

A detailed, systematic experimental program is required to evaluate the suitability of less expensive samples containing moderate amounts (40% to 50%) of Al_2O_3 for gasification refractory wall construction.

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