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FOSSIL ENERGY

PIPELINE GAS FROM COAL-HYDROGENATION (IGT HYDROGASIFICATION PROCESS)

Project 9000 Interim Report No. 1, July 1, 1976—June 30, 1977

April 1978
Date Published

Work Performed Under Contract No. EF-77-C-01-2434

Institute of Gas Technology
Chicago, Illinois

MASTER

U. S. DEPARTMENT OF ENERGY

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Project 9000 Interim Report No. 1

For the Period July 1, 1976 Through June 30, 1977

Prepared by

Institute of Gas Technology
IIT Center, 3424 S. State Street
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Date Published — April 1978

Prepared for the

UNITED STATES DEPARTMENT OF ENERGY

Under Contract No. EF-77-C-01-2434

REA

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FOREWORD

This is the first Interim Report prepared for work conducted under ERDA Contract No. EF-77-C-01-2434 [originally E(49-18)-2434]. It covers the period from July 1, 1976 through June 30, 1977.

All work reported in this volume was sponsored by ERDA and the American Gas Association.

SUMMARY

On July 1, 1976, IGT's program to produce pipeline gas from coal-hydrogenation (the IGT Hydrogasification Process) was continued under ERDA Contract No. E(49-18)-2434 (later changed to EF-77-C-01-2434). The objective of the continuation was to advance pilot plant studies in order to demonstrate the feasibility of the HYGAS process and to provide data to aid in developing a commercial coal gasification process.

This Interim Report covers the work conducted between July 1, 1976 and June 30, 1977. During this time, 10 tests were made with a variety of coals. Highlights of this year included demonstrating the technical feasibility of the HYGAS process with highly caking bituminous coal (during Test 54) and several long tests with subbituminous coal (Tests 55 through 58). Data on maximum carbon conversion were also obtained in another test series with mildly caking bituminous coal. Test 61, in which 90%+ carbon conversion was achieved, represents the best carbon conversion of bituminous coal during this reporting period.

At the beginning of the period covered by this report, the HYGAS process was shown to be technically feasible with pretreated bituminous coal during Test 54. This test, voluntarily terminated in July 1976, included 9-1/2 days of self-sustained operation. It was the last in a series of tests with Illinois No. 6 bituminous coal from Sahara Mine and was the most successful test with this coal feed. Pretreater operation improved dramatically over previous tests. The gasifier reactor was operated at 3.0 tons/hr under steady conditions for the first time.

During the fall of 1976, a series of tests was conducted using Montana Rosebud seam subbituminous coal. Test 55, the first in this series, was very successful in terms of trouble-free operation with a new coal type. Five and one-half days of self-sustained operation were achieved before the test was terminated because of a power failure resulting from an electrical short circuit.

Tests 56 and 57 were completed during October 1976 and were shut down after 11 and 48 hours, respectively, of steady-state operation. Both tests were terminated when a plug developed in the coal transfer line between the slurry dryer bed and the first stage reactor.

Test 58, the most successful test with Rosebud subbituminous coal, extended over 11-1/2 days in November 1976. All sections of the plant were activated for this test and a 3.5 ton/hr feed rate was reached for a short period of time. Earlier problems with the slurry feed system and purification system were solved. After Test 58, IGT was directed by the ERDA/A.G.A. Operating Committee to return to the study of the HYGAS process with a mildly caking bituminous coal from the Illinois Basin.

During December and January, several modifications and improvements were made to the plant to ready it for the first test with Peabody No. 10 Mine bituminous coal. The test series with this coal extended through June 1977 and was conducted to investigate the operating conditions necessary for maximum carbon conversion and for minimum pretreatment.

In March 1977, Test 59 was conducted. Two leaks forced termination of this test before steady-state operations could be achieved. Test 60, in April 1977, achieved 186 hours of self-sustained operation and coal conversions of 60% to 70%. A leak also forced termination of this test.

Carbon conversions of over 90% were achieved for the first time with bituminous coal during Test 61 in May 1977. Gasifier operation was also extremely smooth during Test 61, which was eventually terminated because of a leak.

Test 62 lasted for 3 days in early June 1977, and was terminated because of operational problems in the product-gas quench system. Test 63, the last test conducted during the period covered by this Interim Report, included 7 days of self-sustained operation and carbon conversions of around 80%.

A variety of support studies were also conducted throughout the first year of this contract, including work in plant effluent processing, methanation testing, and engineering services. Notably, in June 1977, IGT began constructing a cold-flow model to increase understanding of the initiation of solids flow to the steam-oxygen bed and of the stability of this fluidized bed.

Details of all 10 of the HYGAS tests, of plant maintenance and improvements, and of the support studies, are presented in the body of this report.

PROJECT OBJECTIVES

The objective of the HYGAS project between July 1976 and June 1977 was to continue and advance pilot plant studies directed at the development of a commercial process for the production of pipeline-quality gas from coal and to demonstrate the feasibility of the HYGAS pilot plant to produce substitute natural gas using lignite, bituminous, and subbituminous coal feed stocks.

By June 1977, successful operation had been achieved with all three of these coal types. Tests to acquire additional data for scale-up to a commercial process had been conducted and were continuing.

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INTRODUCTION

The IGT HYGAS Process for hydrogasifying coal is designed for the maximum production of methane in the hydrogasifier. The process uses high temperature (1200° to $1800^{\circ}F$) and high pressure (~1000 psig) and can use caking bituminous coals as well as noncaking lignite and subbituminous coals. A description of the process and its history follow.

History of the Process

The HYGAS Process is based on gasification studies which were started at the Institute of Gas Technology in 1944. Between 1944 and 1964, fundamental concept parameters in hydrogasification were investigated. The process concept was tested in batch laboratory autoclaves, then in various small continuous reactors.

In 1964, the U. S. Department of the Interior's Office of Coal Research (OCR) joined A.G.A. in funding this work, conducted under OCR Contract No. 14-01-0001-381, in order to accelerate the research. Between 1964 and 1967, over a dozen coals were tested in a 4-inch diameter process development unit (PDU).

In 1967, a five-year extension of the OCR contract was awarded, and design and construction of the HYGAS pilot plant began. In May 1971, the construction of the HYGAS pilot plant was completed. After a series of shakedown and equipment tests, the first gasification test was begun in October of the same year.

In 1972, a new contract was awarded; OCR Contract No. 14-32-001-1221, later changed to ERDA Contract No. E(49-18)-1221 . Between August 1972 and June 1976, the HYGAS pilot plant carried out 31 tests with lignite and 17 tests with bituminous coals. Major milestones included —

- Production of pipeline quality gas, during Test 19 in June-July 1973
- Modification of the slurry dryer section to remove fines from the system, which solved solids circulation problems in Test 25, November 1973
- Long integrated operation, achieved during Test 27, March, 1974, using hydrogen prepared from steam reforming of natural gas

- Design and installation of the steam-oxygen gasification step of the HYGAS Process (the first steam-oxygen test, Test 28, was made in June 1974)
- Demonstration of the technical feasibility of operation with lignite, in Test 37, July 1975, during which 15 days of self-sustained continuous operation were achieved, making pipeline quality gas from the methanator (this marked the conclusion of the lignite test program).

Tests 7 through 27, conducted between August 1972 and June 1974, are described in detail in the HYGAS Interim Report No. 1, OCR Contract No. 14-32-001-1221. Tests 28 through 37, conducted between July 1974 and June 1975, are described in detail in the HYGAS Interim Report No. 2, ERDA Contract E(49-18)-1221. Tests 38 through 53, conducted between July 1975 and June 1976, are described in detail in the HYGAS Final Report, ERDA Contract No. E(49-18)-1221.

Tests 54 through 63 were conducted since July 1976 and under a new contract. These tests are described in detail in this report.

Chemistry of the Process

Methane can be formed directly from coal by the following reaction:

$$\text{coal} + 2H_2 \rightarrow CH_4 \quad (1)$$

or indirectly by —

$$\text{coal} + H_2O \rightarrow CO + H_2 \quad (2)$$

$$CO + 3H_2 \rightarrow CH_4 + H_2O \quad (3)$$

Since the thermal efficiency of the overall process is improved by maximizing direct methanation formation, the conditions of the IGT HYGAS Process are tailored toward this end. High temperature is needed to obtain reasonable reaction rates; high pressure is used to increase equilibrium methane yield. The most active fraction of coal is hydrogasified to form methane, while the less active fraction is used to generate hydrogen for hydrogasification. The raw gas from the hydrogasifier contains carbon monoxide and hydrogen. These are converted to methane through Reaction 3 in the catalytic cleanup methanation unit to boost the heating value of the gas and to reduce the carbon monoxide content to the pipeline standard of less than 0.1%. Of the total amount of methane formed, 65% to 70% is formed by the direct route in the hydrogasifier. This is a key feature contributing significantly to the HYGAS Process's high overall thermal efficiency of over 70%. If the process were operated at a lower pressure, more methane would be made by the indirect rather than by the direct route, and losses in efficiency would result.

Process Description

The HYGAS Process, shown in Figure 1, begins with a coal mill which crushes the coal into 1/8-inch or smaller particles and removes most of the moisture. If the coal is a caking bituminous one, it then undergoes pretreatment. The pretreater exposes the coal to air at about 750°F, which causes a controlled oxidation reaction on the surface of the particles. This oxidized outer surface prevents the particles from sticking together later in the process.

The prepared coal is mixed with a light oil to form a feed slurry resembling thin mud. (The oil used is a by-product of the hydrogasification reactions and is recovered when the gas is cleaned.) A plunger pump pressurizes this slurry to about 1000 psi and it is pumped through a preheater and into the HYGAS reactor.

In the reactor's four sections (shown in Figure 2), coal moves down and gas flows up. In the top section (the slurry dryer), the slurry oil is evaporated by hot gas rising up from the hydrogasification stage just below. In the next section, the first stage of hydrogasification takes place as heated coal falling from the dryer section contacts gas rising from the second hydrogasification stage of the reactor. There, the more reactive part of the coal is converted into methane.

In the third section of the reactor, the partially converted coal from above mixes with a hydrogen-rich gas to form more methane and carbon oxides. The bottom, fourth section of the reactor produces the hydrogen-rich gas needed in the third section.

The solid residue, mostly ash, that is left over after gasification, is carried by steam into a tank, mixed with water, and let down in pressure. The solids are then separated by settling and filtering. The gas leaving the top of the reactor goes through a cyclone which removes fine dust particles. Light oil, steam, and fine coal particles are removed by rinsing the gas with water. Then the gas contacts a diglycolamine scrubbing solution

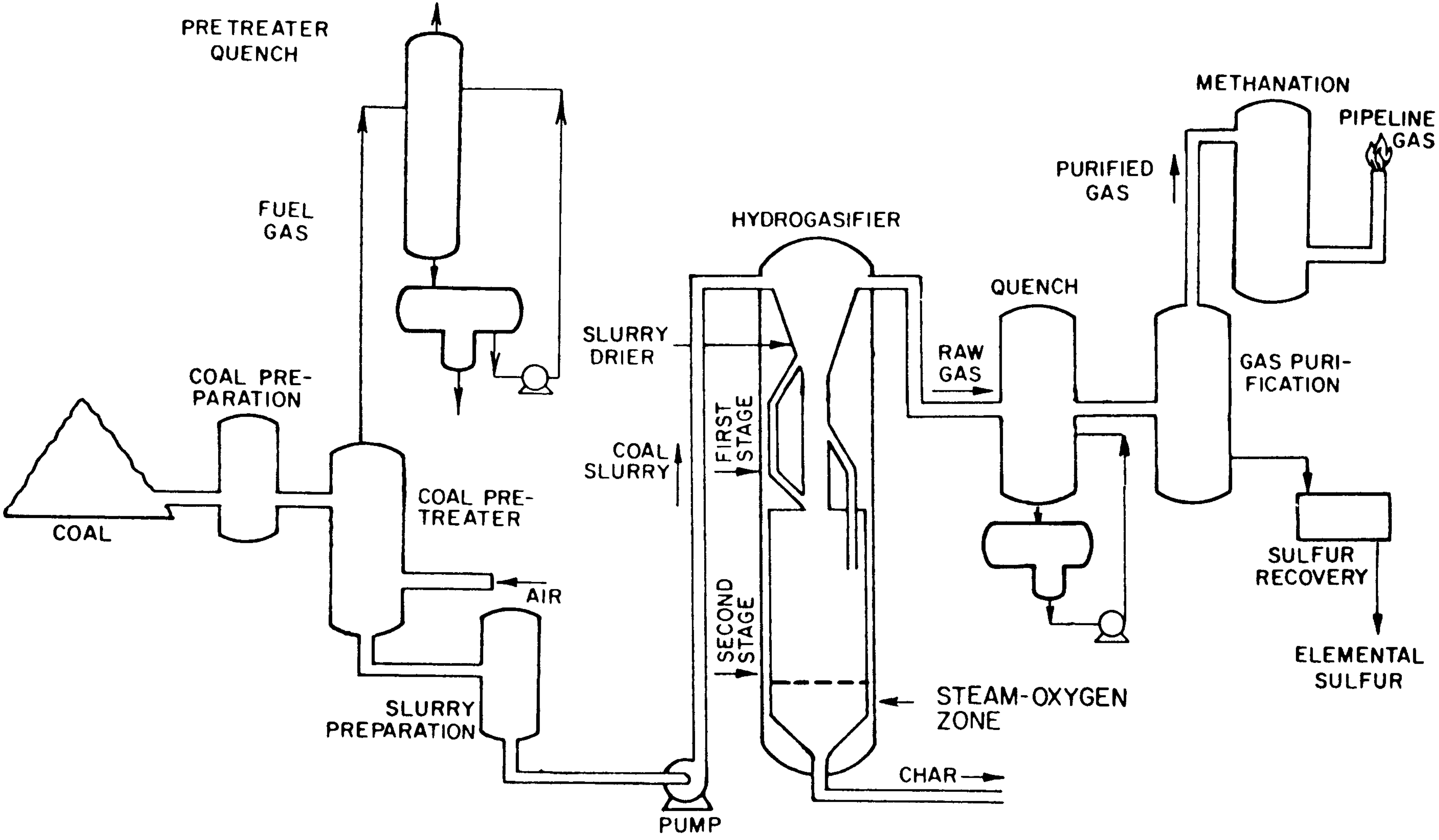


Figure 1. THE HYGAS PROCESS

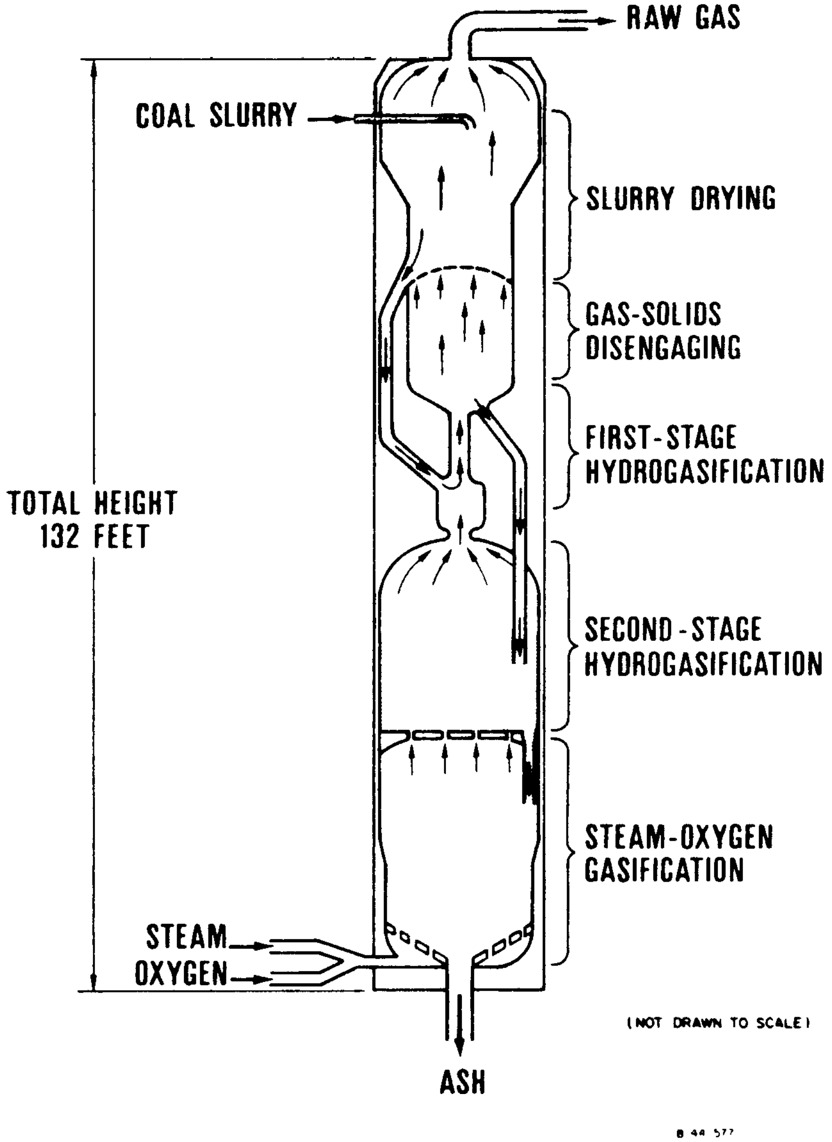


Figure 2. SIMPLIFIED CUTAWAY OF HYGAS REACTOR

which absorbs the carbon dioxide and sulfur gases. Finally, the gas is washed with a caustic solution and then with water. It is then suitable for final processing in the methanation section of the plant.

In the methanation section, all the carbon monoxide and most of the hydrogen contact a catalyst and react to form steam and the remaining 1/3 of the methane. The gas is cooled to condense and remove the steam. This leaves a product gas with a heating value of about 950 Btu/cu ft, which is completely suitable for use in homes and industry.

HYGAS GASIFICATION TESTS

Test 54, July 1976

Highlights

Test 54 established the technical feasibility of operating the HYGAS Process with pretreated bituminous coal. The test was voluntarily terminated on July 11, 1976 after 593 tons of char were fed to the gasifier and about 9-1/2 days of self-sustained operation were achieved.

Test 54 was the last and most successful run in the series of tests with highly caking Illinois No. 6 bituminous coal, initiated in September 1975. It was also the first time that the gasifier reactor was operated at a 3.0 ton/hr level under steady-state conditions.

Details

Experimental operation of the pretreater began on June 27, 1976 with the objective of setting the conditions required to produce nonagglomerating char feed to the reactor. Experimental work in the process development units had established that the +14 mesh (larger coal particles) were more difficult to pretreat than the finer mesh material. More oxygen, higher gas superficial velocity, higher temperatures, and longer residence times were required to obtain a nonagglomerating feed material than were required for the –14 mesh material. A 14-mesh screen was installed on the screen section of the raw-coal feed to the pretreater, and all +14 mesh material was recycled to the coal mill for recrushing. Pretreater operation began at 750°F, with residence times of about 1 hour, raw coal feed rates of 1-1/2 to 2 ton/hr to the pretreater, and oxygen concentrations of 2 CF/lb of coal fed. The 750°F temperature levels did not produce a completely nonagglomerating feed material, as indicated by the IGT boat test, and the temperature levels were raised gradually to 775°F and then to 800°F. The results of the agglomerating tests on the product char produced at 775°F indicated that some of the material was slightly agglomerating and that the material produced at an 800°F average bed temperature was completely nonagglomerating. Therefore, test conditions for pretreating the feed material for Test 54 were set at 800°F, 2 CF of oxygen per pound of coal, and residence times of about 1 hour. These operating conditions were maintained in the pretreater section during all of Test 54.

Early in Test 54, on June 30, material could not be discharged from the second-stage gasifier bed to the steam-oxygen bed. Repeated attempts to establish solids flow through line 339 were unsuccessful. The technique of depressurizing the bottom section of the reactor succeeded in establishing solids flow through line 339. Smooth solids flow was established throughout the entire reactor on July 1, after which the reactor was put on self-sustaining operation. Supplemental hydrogen to the start-up heater was discontinued 14 hours later.

The pretreated-coal feed rate to the reactor was maintained at 2.5 ton/hr, and the reactor operated smoothly at this pretreated-coal feed rate until July 9.

The purification section of the plant was activated and operated satisfactorily until 2300 hours on July 8 when the high-pressure multi-stage diglycolamine circulation pump was taken off-line because of a bad bearing. It was determined that the bearing problem could not be repaired in time to continue operation of the purification section for Test 54, and so the purification section was shut down.

At 0800 hours on July 9 the pretreated-coal feed rate to the reactor was increased to 3.0 ton/hr. A corresponding increase in the feed rate to the pretreater was required to maintain this increased feed rate to the reactor. This is the first time that the reactor was operated at a 3 ton/hr feed rate on any coal feed for an extended period, and the operation was very satisfactory. Smooth solids flow was established throughout the unit, and there was no problem in maintaining the 3 ton/hr feed rate for the next 44 hours.

Test 54 was voluntarily terminated at 0300 hours on July 11 when the supply of Illinois No. 6 bituminous coal was completely exhausted. All of the test objectives for establishing the technical feasibility of operating the HYGAS Process with pretreated bituminous coal had been achieved. During Test 54, 860 tons of raw bituminous coal were fed to the pretreater, beginning with experimental operation of the pretreater at 1300 hours on June 27 and continuing until the test was terminated. During the entire test period, 593 tons of char were fed to the reactor, beginning with operation of the reactor at 0735 hours on June 30 and continuing until the test was terminated at 0330 hours on July 11. Test 54 was self-sustained for a total of 228 hours (about 9-1/2 days); the last 214 consecutive hours of self-sustained operation were obtained with no hydrogen addition to the reactor. During the 214 hours, 640 tons of coal were fed to the pretreatment section.

Inspection of the pretreater and char-cooler sections of the HYGAS plant following Test 54 indicated that the pretreater was completely free of clinkers, agglomerates, and unreacted coal pockets, which had been found in the pretreater reactor or the char-cooler sections during previous tests with bituminous coal. The pretreatment section could not have operated more satisfactorily than it did during Test 54. The change in feed size was responsible for the significant improvement in its operation.

A clinker deposit was found in the steam-oxygen zone of the HYGAS reactor during the post-run inspection following Test 54. The clinker covered 2 of the 15 steam-oxygen sparger outlets and partially blocked off a third. The clinker was eroded and showed evidence of being worn away during operation. It is believed that this clinker formed very early in the test period before steady-state operations were started, when the reactor bottom discharge valve stuck open and discharged a considerable portion of the steam-oxygen fluidized bed, causing a major gasifier upset. The clinker appeared to be reduced in size by erosion during the subsequent steady-state operation. The clinker certainly did not affect the steady-state operation of the gasifier as indicated by smooth bed levels and transfer of solids. The remainder of the gasifier was in excellent condition with no plugs or deposits found in the second stage, first stage transfer line, or slurry dryer bed areas. There was a slight deposit on the slurry dryer grid, although the stilling well and the 321 transfer pipe in the slurry dryer area were completely free and open.

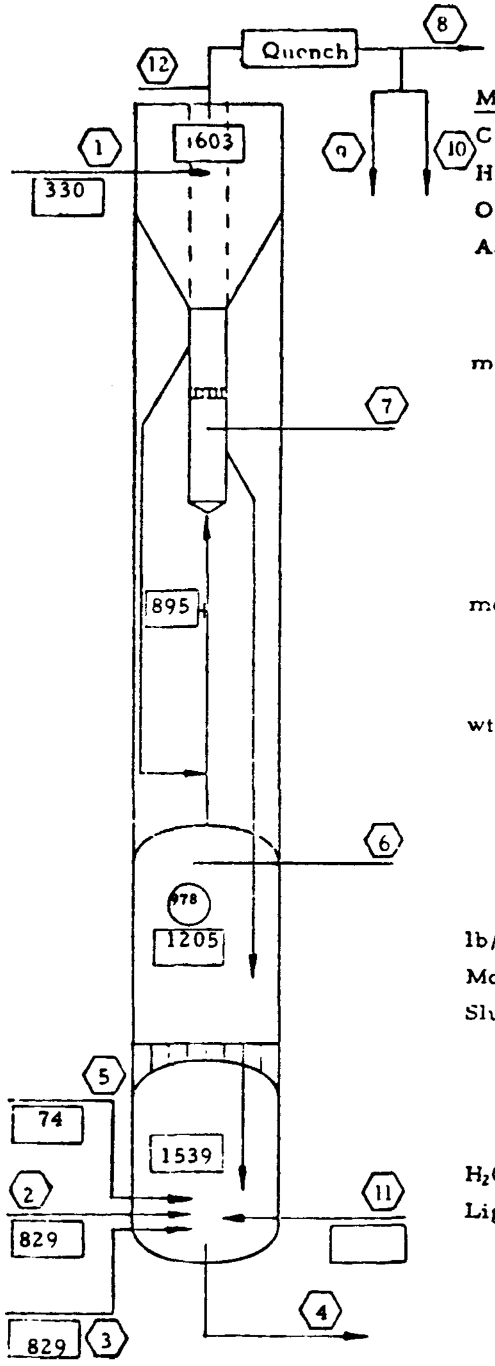
The data for Test 54 are presented in Tables 1 through 4 and Figures 3 through 9. Elemental balances for carbon, hydrogen, and oxygen and the balance for ash all are within the 95% to 105% guidelines for demonstrating technical feasibility of operation. The elemental balance for sulfur is low, and inconsistencies in the sulfur analysis in the effluent liquid streams probably account for this difference.

Table 1. MATERIAL BALANCE SUMMARY FOR TEST 54 FROM 7/2/76 (0500 Hours) TO 7/3/76 (1300Hours)

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

INPUT		C	H	O	N	S	Cl	ASH	OTHER	TOTAL
Coal Feed	Wt. % (Dry)	73.76	3.48	7.89	1.72	2.34	--	10.81		100
	Coal (Dry)	3383	159	362	79	107	--	496		4586
	Moisture		10	78						88
Sparger	Oxygen			610						610
	Steam		458	3666						4124
Burner	Oxygen			0						0
	Steam		0	0						0
	Hydrogen		0							0
Stripping Ring	Steam		191	1532						1723
Nitrogen from purges					435					435
Pump seal flush			74	592						666
Cooling water spray			0	0						0
Water to cyclone pot			530	4238						4768
Light oil in		9206	877							10,083
TOTAL INPUT		12,589	2299	11,078	514	107	--	496		27,083
OUTPUT										
Reactor Overhead	Wt. % (Dry)	82.55	2.79	4.13	1.60	1.93	--	7.00		100
	Dust (Dry)	466	16	23	9	11	--	39		564
Spent Char	Wt. % (Dry)	76.84	1.42	1.56	0.69	0.71	--	18.78		100
	Char (Dry)	1692	31	34	15	16	--	414		2202
Product Gas After Quench	Total (Dry)	610	143	788	243	13				1797
	Components H_2		46							46
	CO_2	271		724						995
	C_2H_6	11	3							14
	H_2S		1			13				14
	N_2				243					243
	CH_4	280	93							373
	CO	48		64						112
Water Out + dissolved materials		9	1289	10,253	34	3				11,588*
Toluene storage tank vent gases		258	23	418	28	5				732
Stripper vent gas*		34	4	76	10	1				125
Light oil out		9744	928							10,672
TOTAL OUTPUT		12,813	2434	11,592	339	49		453		27,680
Net (Output-Input)		224	135	514	-175	-58	--	-43		597
% Balance (Output/Input)		102	106	105	66	46	--	91		102

*Using Sub-period data when light-oil stripper in operation.



⬡ Stream No.
○ Pressure, psig
▭ Temperature, °F

Material Balance, %

C	102
H	106
O	105
Ash	91

	Gas Streams			
	6	7	8	12
mol % (dry)				
H_2			27.76	23.11
CO_2			27.46	35.95
C_2H_6			0.58	0.70
Ar			--	--
N_2			10.55	9.56
H_2S			0.51	0.59
CH_4			28.32	26.17
CO			4.83	3.92
mol/hr (dry)			82	105

	Solids Streams			
	1	7	12	4
wt % (dry)				
C	73.76		82.55	76.84
H	3.48		2.79	1.42
N	1.72		1.60	0.69
Cl	--		--	--
S	2.34		1.93	0.71
Ash	10.81		7.00	18.78
O	7.89		4.13	1.56
lb/hr (dry)	4586		564	2202
Moisture, wt %	1.9			
Slurry Concn, wt %	32			

	Liquid Streams		
	9	10	1
	lb/hr		
H_2O	6820	--	--
Light Oil	--	10,672	10,083

Gas Feed Streams			
2	3	5	11
mol/hr			
229	96	19	0

① Feed Slurry	⑦ First-Stage Gasifier Product
② High-Pressure Steam to Steam-Oxygen Sparger	⑧ Gasifier Effluent After Quench
③ High-Pressure Steam to Stripping Ring	⑨ Water Made
④ Spent Char	⑩ Light Oil to Recycle
⑤ High-Pressure Oxygen to Steam-Oxygen Sparger	⑪ Gases From External Heater
⑥ Second-Stage Gasifier Product	⑫ Reactor Overhead

Figure 3. HYGAS REACTOR DATA FOR TEST 54 FOR STEADY PERIOD FROM 7/2/76 (0500 Hours) TO 7/3/76 (1300 Hours)

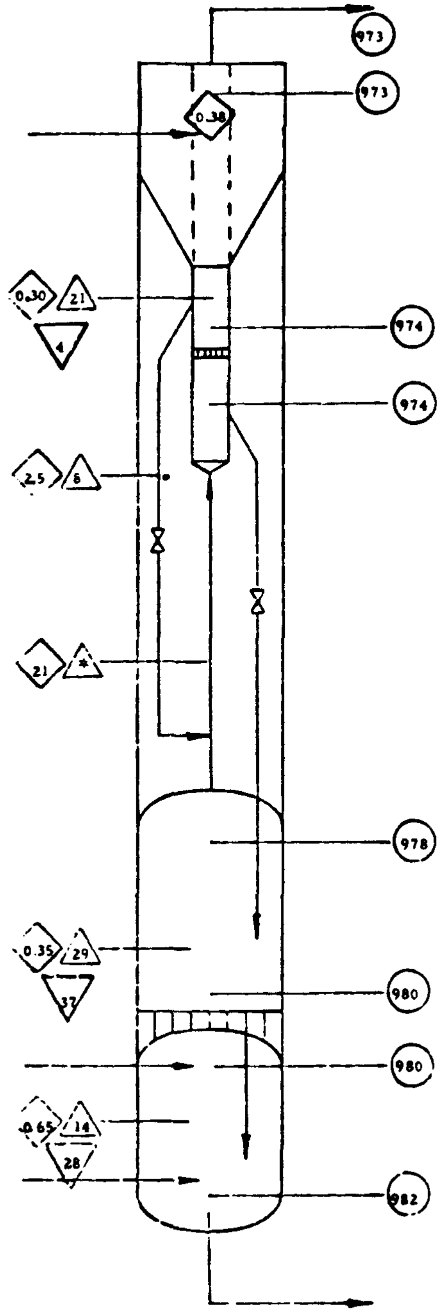
○ Pressure, psig

△ 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 = 0.13
lb Steam/lb Coal Fed = 1.3
lb Hydrogen/lb Coal Fed = 0
lb Coal Fed/1000 SCF Product Gas = 210

By Ash Balance

Coal Gasified, % = 48
Carbon Gasified, % = 40

Methane Yield, SCF/lb coal fed = 2.3
Carbon Oxide Yield, SCF/lb coal fed = 3.5

Bed Height (ft)

Slurry Dryer = *

HTR = 12.5

SOG = 20.4

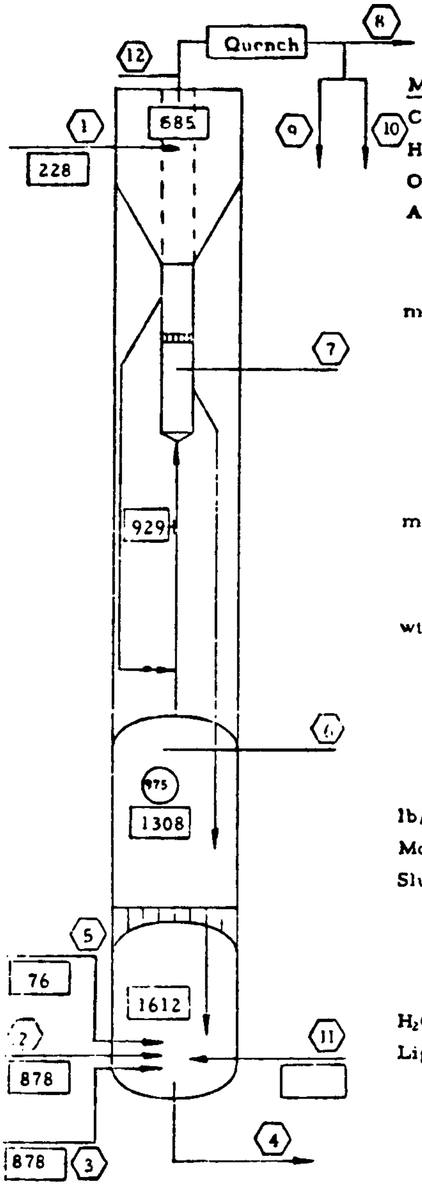
Figure 4. HYGAS REACTOR ENGINEERING DATA FOR TEST 54 FOR STEADY PERIOD FROM 7/2/76 (0500 Hours) TO 7/3/76 (1300 Hours)

Table 2. MATERIAL BALANCE SUMMARY FOR TEST 54 FROM 7/3/76 (1800 Hours) TO 7/7/76 (0030 Hours)

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

INPUT		C	H	O	N	S	Cl	ASH	OTHER	TOTAL
Coal Feed	Wt. % (Dry)	74.14	3.50	8.27	1.74	2.13	--	10.22		100
	Coal (Dry)	3,528	167	393	83	101	--	486		4,758
	Moisture		10	79						89
Sparger	Oxygen			764						764
	Steam		460	3,681						4,141
Burner	Oxygen			0						0
	Steam		0	0						0
	Hydrogen		0							0
Stripping Ring	Steam		211	1,687						1,898
Nitrogen from purges					487					487
Pump seal flush			75	600						675
Cooling water spray			0	0						0
Water to cyclone pot			533	4,261						4,794
Light oil in		9,531	908							10,439
TOTAL INPUT		13,059	2,364	11,465	570	101	--	486		28,045
OUTPUT										
Reactor Overhead	Wt. % (Dry)	81.61	2.71	4.09	1.52	1.84	--	8.23		100
	Dust (Dry)	477	16	24	9	11	--	48		585
Spent Char	Wt. % (Dry)	76.52	1.27	1.12	0.57	0.52	--	20.00		100
	Char (Dry)	1,715	28	25	13	12	--	448		2,241
Product Gas After Quench	Total (Dry)	878	189	1,173	282	18				2,540
	Components H_2		62							62
	CO_2	382		1,019						1,401
	C_2H_6	11	3							14
	H_2S		1			18				19
	N_2				282					282
	CH_4	370	123							493
	CO	115		154						269
Water Out + dissolved materials*		8	1,254	9,974	29	4				11,269
Toluene storage tank vent gases		269	24	439	29	7				768
Stripper vent gas*		47	4	70	8	0				129
Light oil out		10,059	958							11.017
TOTAL OUTPUT		13,453	2473	11,705	370	52	--	496		28,549
Net (Output-Input)		394	109	240	-200	-49		10		504
% Balance (Output/Input)		103	105	102	65	51		102		102

* Obtained from sub-period data when light-oil stripper was in operation.



⬡ Stream No.
○ Pressure, psig
▭ Temperature, °F

Material Balance, %	
C	103
H	105
O	102
Ash	102

	Gas Streams	
	8	12
mol % (dry)		
H_2	27.20	23.64
CO_2	27.78	34.42
C_2H_6	0.40	0.46
Ar	--	--
N_2	8.81	8.27
H_2S	0.49	0.59
CH_4	26.93	25.40
CO	8.39	7.22
mol/hr (dry)	115	138

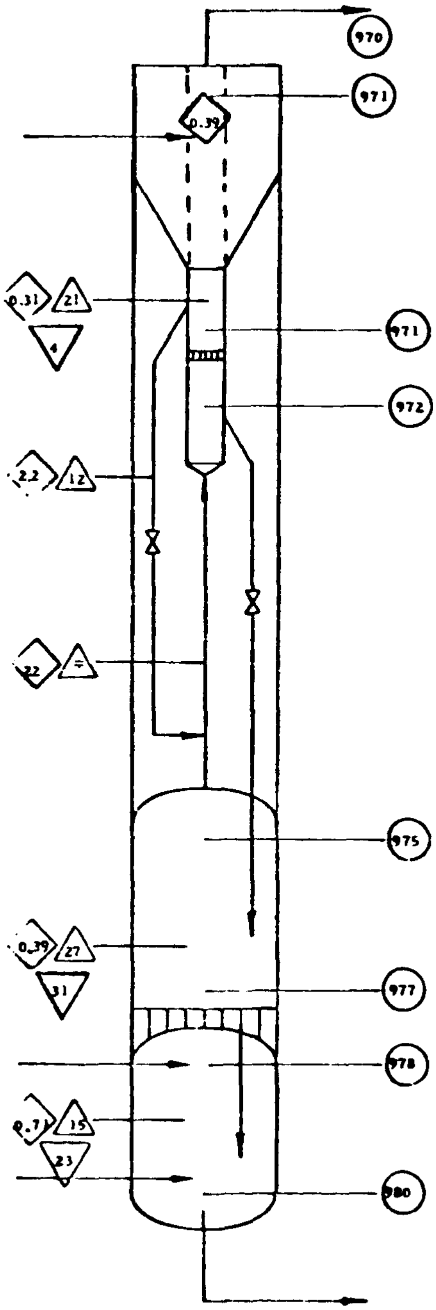
	Solids Streams			
	1	7	12	4
wt % (dry)				
C	74.14		81.61	76.52
H	3.50		2.71	1.27
N	1.74		1.52	0.57
Cl	--		--	--
S	2.13		1.84	0.52
Ash	10.22		8.23	20.00
O	8.27		4.09	1.12
lb/hr (dry)	4758		585	2241
Moisture, wt %	1.8			
Slurry Concn, wt %	32			

	Liquid Streams		
	9	10	1
	lb/hr		
H_2O	6475	--	--
Light Oil	--	11,017	10,439

Gas Feed Streams			
2	3	5	11
mol/hr			
230	105	24	0

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 Second-Stage Gasifier Product
⑦ First-Stage Gasifier Product
⑧ Gasifier Effluent After Quench
⑨ Water Made
⑩ Light Oil to Recycle
⑪ Gases From External Heater
⑫ Reactor Overhead

Figure 5. HYGAS REACTOR DATA FOR TEST 54 FOR STEADY PERIOD FROM 7/3/76 (1800 Hours) TO 7/7/76 (0030 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 = 0.16
lb Steam/lb Coal Fed = 1.3
lb Hydrogen/lb Coal Fed = 0
lb Coal Fed/1000 SCF Product Gas = 158

By Ash Balance

Coal Gasified, % = 54
Carbon Gasified, % = 47

Methane Yield, SCF/lb coal fed = 2.8
Carbon Oxide Yield, SCF/lb coal fed = 4.6

Bed Height (ft)

Slurry Dryer = *

HTR = 13.1

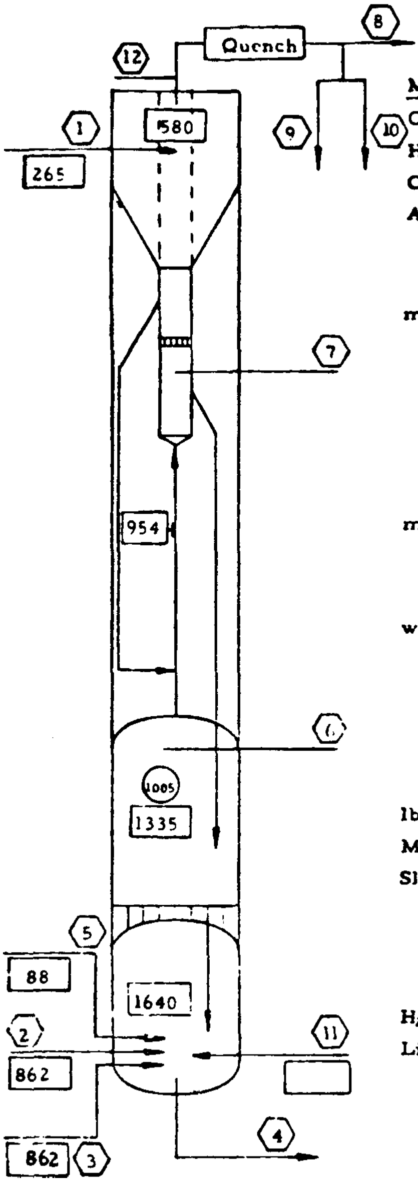
SOG = 19.6

Figure 6. HYGAS REACTOR ENGINEERING DATA FOR TEST 54 FOR STEADY PERIOD FROM 7/3/76 (1800 Hours) TO 7/7/76 (0030 Hours)

Table 3. MATERIAL BALANCE SUMMARY FOR HYGAS GASIFIER FOR TEST 54 FROM 7/10/76 (0000 Hours) TO 7/11/76 (0000 Hours)

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

INPUT		C	H	O	N	S	Cl	ASH	OTHER	TOTAL
Coal Feed	Wt. % (Dry)	74.11	3.33	8.40	1.84	2.15	--	10.17		100
	Coal (Dry)	43 81	197	497	109	127	--	601		5,912
	Moisture		22	175						197
Sparger	Oxygen			824						824
	Steam		473	3,780						4,253
Burner	Oxygen			0						0
	Steam		0	0						0
	Hydrogen		0							0
Stripping Ring	Steam		214	1,710						1,924
Nitrogen from purges					422					422
Pump seal flush			74	592						666
Cooling water spray			0	0						0
Water to cyclone pot			578	4626						5 204
Light oil in		10,398	990							11 388
TOTAL INPUT		14,779	2,548	12,204	531	127	--	601		30,790
OUTPUT										
Reactor Overhead	Wt. % (Dry)	80.49	2.70	4.56	1.58	1.95	--	8.72		100
	Dust (Dry)	384	13	22	8	9	--	41		477
Spent Char	Wt. % (Dry)	76.78	1.28	1.39	0.60	0.62	--	19.33		100
	Char (Dry)	2,100	35	38	16	17	--	529		2,735
Product Gas After Quench	Total (Dry)	1,096	231	1,430	271	30				3,058
	Components H_2		72							72
	CO_2	454		1,210						1,664
	C_2H_6	14	3							17
	H_2S		2			30				32
	N_2				271					271
	CH_4	463	154							617
	CO	165		220						385
Water Out + dissolved materials		6	1,337	10,627	39	2				12,011
Toluene storage tank vent gases		310	27	534	24	6				901
Stripper vent gas		38	3	65	11	2				119
Light oil out		10,955	1,043							11,998
TOTAL OUTPUT		14,889	2689	12716	369	66	--	570		31,299
Net (Output-Input)		110	141	512	-162	-61	--	-31		509
% Balance (Output/Input)		101	105	104	69	52	--	95		102



⬡ Stream No.

○ Pressure, psig

▭ Temperature, °F

Material Balance, %

C	101
H	105
O	104
Ash	95

	Gas Streams			
	6	7	8	12
mol % (dry)				
H_2			26.12	22.73
CO_2			27.57	34.31
C_2H_6			0.40	0.47
Ar			--	--
N_2			7.06	6.68
H_2S			0.69	0.70
CH_4			28.14	26.48
CO			10.02	8.63
mol/hr (dry)			137	164

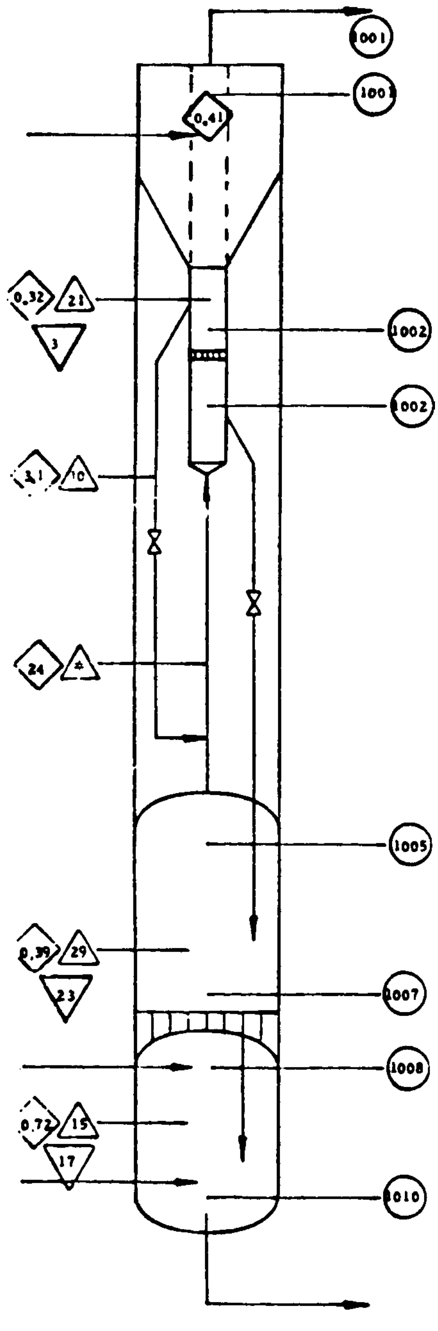
	Solids Streams			
	1	7	12	4
wt % (dry)				
C	74.11		80.49	76.78
H	3.33		2.70	1.28
N	1.84		1.58	0.60
Cl	--		--	--
S	2.15		1.95	0.62
Ash	10.17		8.72	19.33
O	8.40		4.56	1.39
lb/hr (dry)	5912		477	2735
Moisture, wt %	3.2			
Slurry Concn, wt %	35			

	Liquid Streams		
	9	10	1
	lb/hr		
H_2O	6807	--	--
Light Oil	--	11,998	11,388

Gas Feed Streams			
2	3	5	11
mol/hr			
236	107	26	0

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 Second-Stage Gasifier Product
⑦ First-Stage Gasifier Product
⑧ Gasifier Effluent After Quench
⑨ Water Made
⑩ Light Oil to Recycle
⑪ Gases From External Heater
⑫ Reactor Overhead

Figure 7. HYGAS REACTOR DATA FOR TEST 54 FOR STEADY PERIOD FROM 7/10/76 (0000 Hours) TO 7/11/76 (0000 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 = 0.14
lb Steam/lb Coal Fed = 1.0
lb Hydrogen/lb Coal Fed = 0
lb Coal Fed/1000 SCF Product Gas = 162

By Ash Balance

Coal Gasified, % = 53
Carbon Gasified, % = 45

Methane Yield, SCF/lb coal fed = 2.8
Carbon Oxide Yield, SCF/lb coal fed = 4.5

Bed Height (ft)
Slurry Dryer = *
HTR = 12.4
SOG = 19.7

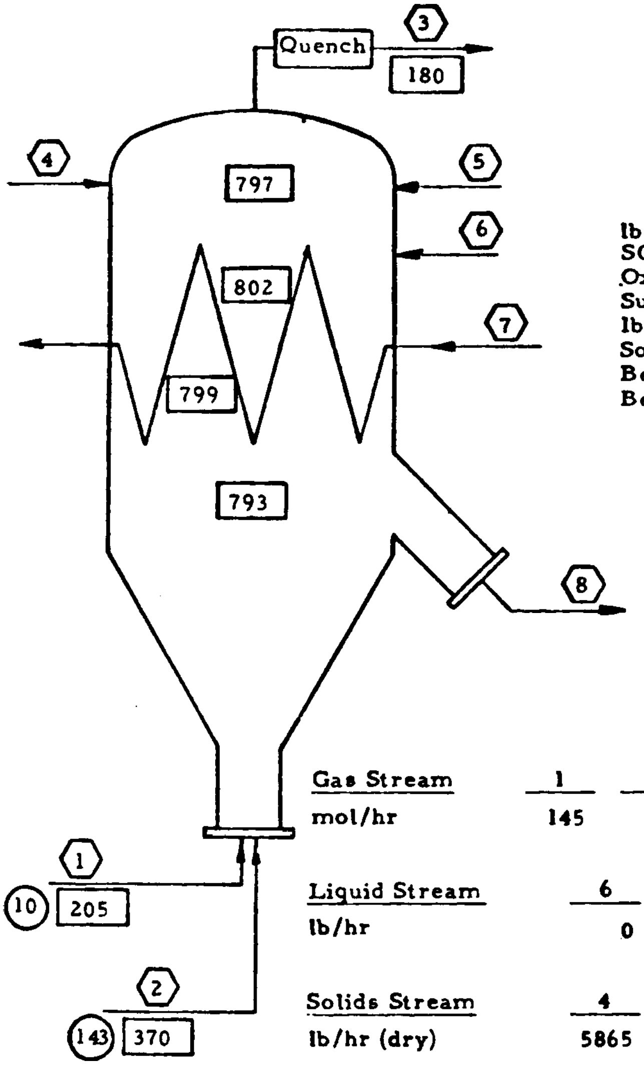
Figure 8. HYGAS REACTOR ENGINEERING DATA FOR TEST 54 FOR STEADY PERIOD FROM 7/10/76 (0000 Hours) TO 7/11/76 (0000 Hours)

Table 4. MATERIAL BALANCE SUMMARY FOR THE PRETREATER SECTION FOR TEST 54 FROM 7/3/76 (1800 Hours) TO 7/7/76 (0030 Hours)

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

INPUT		C	H	O	N	S	Ar	Ash	Other	Total
Coal Feed	Wt.% (Dry)	73.91	5.04	8.28	1.50	2.76	--	8.51		100
	Coal (Dry)	4334	296	486	88	162	--	499		5865
	Moisture		14	115						129
Streams to Pretreater	Air			974	3164		58			4196
	Steam		133	1064						1197
Nitrogen from purges					299					299
Air from purges				9	29					38
H_2O to venturi scrubber			1077	8618						9695
H_2O to quench tower			561	4486						5047
Air to char cooler				164	533		10			707
Cooling water to char cooler			160	1283						1443
TOTAL INPUT		4334	2241	17,189	4113	162	68	499		28,616
OUTPUT										
Pretreated char to gasifier	Wt.% (Dry)	74.14	3.50	8.27	1.74	2.13	--	10.22		100
	Char (Dry)	3528	167	393	83	101	--	486		4758
	Moisture		10	79						89
Slurry waste from quench	Wt.% (Dry)	70.15	2.94	11.69	1.74	2.39	--	11.09		100
	Solids (Dry)	128	6	21	3	4	--	20		182
	*Tars & oils	82	8	5	1	2				98
	H_2O & Dis. materials	10	1634	13,068	6	30				14,748
Quench tower off-gas	Total	316	369	3712	4454	0	68			8919
	Components: H_2		2							2
	CO_2	222		591						813
	C_2H_6	3	1							4
	N_2				4454					4454
	CH_4	25	8							33
	CO	66		88						154
	O_2			168						168
	Ar						68			68
	H_2O		358	2865						3223
TOTAL OUTPUT		4064	2194	17,27	4547	137	68	506		28,794
Net (Output - Input)		-270	-47	79	434	-25	0	7		178
% Balance (Output/Input)		94	98	100	111	85	100	101		101

*Analysis from test 50, typical of product tar for test 54.



⬡ Stream No.

○ Pressure, psig

▭ Temperature, °F

lb Char/lb Coal	0.81
SCF O_2/lb Coal	1.97
Oxygen Breakthrough, % fed	0.41
Superficial Velocity, ft/s	0.91
lb O_2 Reacted/lb Coal	0.17
Solids Residence Time, hr	1.4
Bed Density, lb/cu ft	19
Bed Height, ft	10

Gas Stream	3
Components, mol %	
H_2	0.20
CO_2	4.96
C_2H_6	0.03
O_2/Ar	1.98
N_2	42.71
SO_2	--
CH_4	0.55
CO	1.48
H_2O	48.09
mol/hr	372
mol. wt	24

Gas Stream	1	2	5
mol/hr	145	67	--

Liquid Stream	6	7
lb/hr	0	254

Solids Stream	4	8
lb/hr (dry)	5865	4758

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 9. HYGAS PRETREATMENT DATA FOR TEST 54 FOR STEADY PERIOD FROM 7/3/76 (1800 Hours) TO 7/7/76 (0030 Hours)

The light-oil make was between 10% and 12% by weight of the feed coal. Analysis of the product light-oil indicated that the benzene content increased steadily throughout the run from about 3.7% at the start to 11.2% at the end of the test. Commercial-grade toluene was charged to the plant to initiate test operations. This increase in the benzene content of the light oil had been observed in all of the long steady-state operating periods with both lignite and bituminous coals, indicating that benzene is the predominant portion of the light oil produced.

Preparation for Tests With Subbituminous Coal

Following the successful completion of Tests 54 with Illinois No. 6 bituminous coal, the ERDA/A.G.A. Operating Committee granted IGT permission to proceed to test operations with a Western subbituminous coal. IGT obtained 1300 tons of Montana subbituminous coal from the Rosebud seam. This coal, mined near Colstrip, Montana, was supplied by the Western Energy Company and meets the basic criteria outlined in the C. F. Braun Report FE-1235-1, "Coal Gasification Commercial Concepts Gas Cost Guidelines," January 1976. Table 5 shows analyses and ash fusion temperatures of the coal as received at the HYGAS stockpile. The initial deformation temperature of the ash from Montana subbituminous coal is 2190°F compared with an initial deformation temperature of 2210°F for the Montana lignite coal that was processed in Test 37. The initial ash deformation temperature of Illinois No. 6 bituminous coal was 2520°F.

Reactivity tests conducted with the Montana Rosebud subbituminous coal on the laboratory thermobalance equipment indicated that the coal is slightly less reactive than the lignite coal used previously, but is more reactive than the Illinois No. 6 bituminous coal. Oxygen and steam requirements for complete conversion are somewhat less than those for the Illinois coal. The subbituminous coal is completely nonagglomerating by the IGT standard agglomeration test; therefore, the pretreatment section was bypassed for the initial test series.

Crushing tests were performed on the Montana subbituminous coal supply in IGT's coal preparation section to determine if modifications in mill settings or operating procedures were required to successfully process this new coal. The coal was successfully dried and crushed without excessive fines production at mill temperatures of 270°F with no change in mill setting or speed and at coal feed rates of up to 5 ton/hr. Table 6 illustrates typical operating data from the coal mill test, in which 12.2% of –100 mesh fines and 5.6% of –200 mesh fines were produced. The moisture content was reduced from 20.5% to 9.1%.

Table 5. ANALYSIS OF MONTANA SUBBITUMINOUS COAL

Proximate Analysis, wt % (as received)	
Moisture	20.54
Ash	9.52
Volatile Matter	33.54
Fixed Carbon	36.40
Total	100.00
Ultimate Analysis, wt % (dry)	
Carbon	66.95
Hydrogen	4.57
Nitrogen	0.99
Chlorine	0.03
Sulfur	0.90
Ash	11.98
Oxygen (Difference)	14.58
Total	100.00
Ash Fusion Temperature, °F (reducing)	
Initial Deformation	2190
Softening (H = W)	2245
Softening (H = 1/2 W)	2285
Fluid	2340

* Commercial Testing and Engineering analysis report 71-454891.

A76102078

Table 6. RESULTS OF CRUSHING AND DRYING TESTS ON MONTANA SUBBITUMINOUS COAL

	wt %
Moisture	9.1
Screen Analysis	
Amount Retained on Screen Size	
12	17.2
20	20.7
30	10.5
40	11.0
60	17.7
80	7.6
100	3.1
200	6.6
230	0.8
Pan	4.8
Total	100.0

* IGT Analytical Report No. 34326.

Test 55, September 1976

Highlights

Test 55 was the first HYGAS test with a subbituminous coal. Reactor operation was exceptionally smooth considering this was the first test with a new coal type. A total of 5-1/2 days (126 hours) of self-sustained operation were obtained, including 135 hours of continuous coal feed giving a total feed of 310 tons.

Details

Light-off for Test 55 occurred on August 28, 1976. A series of mechanical problems in peripheral equipment delayed the start of coal feed to the reactor. After coal feed was begun on September 4, a total of 4-1/2 days (126 hours) of self-sustained steady-state operation of the gasifier was obtained. A total of 310 tons of coal was fed, with 276 tons being fed during the steady-state, self-sustained portion of the run. Average feed rates of 2 ton/hr were continued for approximately 4 days, and one 13-hour period of steady-state data was obtained at 3 ton/hr.

The reactor operation during Test 55 was quite smooth, and the unit was able to accept the 3 ton/hr coal feed rate without any process upsets or flow problems within the reactor. In many respects, the reactor operation was better during this test with the Rosebud coal than with the Illinois bituminous pretreated coal. The automatic control mode for reactor operation was used during most of the test period and the entire operation was smooth and trouble-free until the time of the electrical fault that caused the termination of the test.

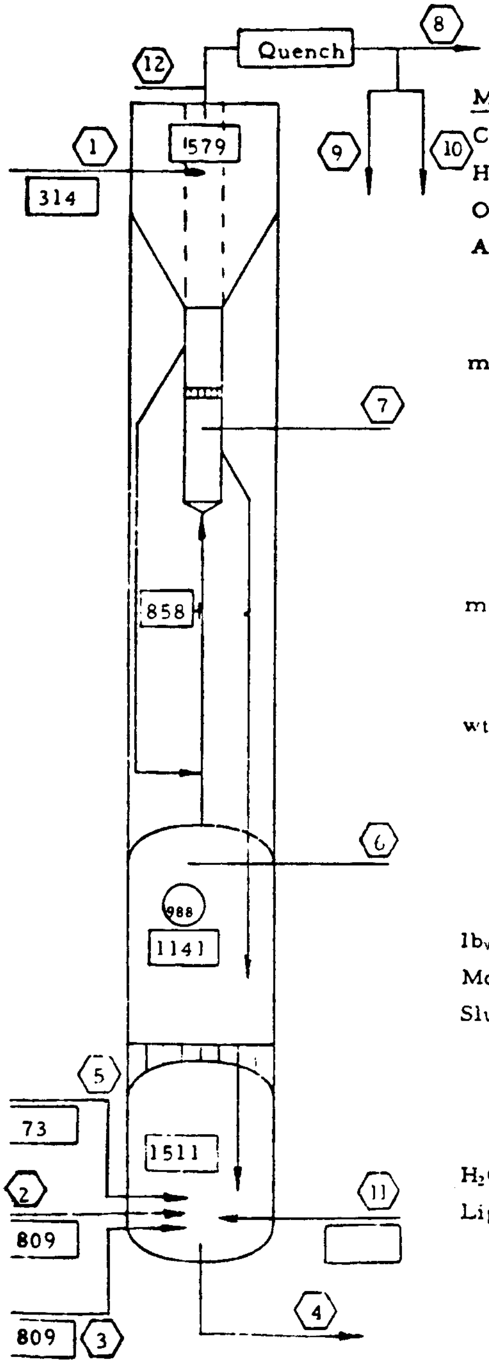
Test 55 was designed to show long-term operability of the process on a Rosebud subbituminous coal and was not designed to obtain maximum carbon conversion or methane production. Temperatures in the steam-oxygen gasification zone were limited to a maximum of $1600^{o}F$, resulting in average temperatures in the 1500^{o} to $1560^{o}F$ range. This was because the initial ash fusion temperature for the Rosebud coal had been analyzed to be between 2000^{o} and $2100^{o}F$. The possiblity of extensive clinker formation was avoided by operating the steam-oxygen gasification zone at a lower temperature in the first test with the subbituminous coal.

At 1200 hours on September 9, 1976, a circuit breaker tripped on a motor control center supplying power to vital high-pressure boiler pumps and circulating pumps. This was caused by a short circuit in the electrical tracing system for the high-pressure cyclone dipleg, which created a ground fault, tripping the main circuit breaker for this motor control center. The power interruption caused both the high- and low-pressure boilers to go off line, which automatically shut off the oxygen flow to the gasifier. The operating crew responded rapidly to this upset and was able to get coal feed back to the gasifier in about 2 hours. However, some local high temperatures were indicated in the steam-oxygen reaction zone upon restart. Operations were not smooth after the restart, and a decision was made to terminate the test when a problem developed with the low-pressure boiler feedwater pump at about 2300 hours on the same evening.

Inspection of the reactor following Test 55 indicated that the steam-oxygen sparger at the manhole 4 location had one of its 15 nozzles burned off, and a large hole about 2 inches in diameter was burned through the 3-inch header pipe. This evidently occurred immediately following the electrical fault which tripped the motor control center and caused the interruption in the test at 1200 hours on September 9, 1976. Inspection of the charts for flow through the sparger indicated a normal pressure drop prior to that time. When steam and oxygen were put back into the sparger in an effort to continue the test, a much lower than expected pressure drop was recorded. Clinker formations attached to the walls of the reactor were also found in the steam-oxygen gasification zone; these clinkers were probably formed because of the uneven oxygen and steam distribution caused by the faulty sparger. The remaining stages of the reactor were found to be in good condition. There was no refractory damage or damage to the 339 valve, 340 valve, the 339 line, or any other internals in the steam-oxygen gasification zone.

Bearings were replaced on the rotor section of the high-pressure amine pump in the purification section; however, the pump was not able to be repaired in time to be put in service for Test 55. Therefore, the methanation section was not put on-line.

Engineering data and calculations for Test 55 are presented in Figures 10 to 15 for representative periods with coal feed rates of 2 and 3 ton/hr.



Stream No.
Pressure, psig
Temperature, ° F

Material Balance, %
C
H
O
Ash

	Gas Streams			
	6	7	8	12
mol % (dry)				
H_2			33.26	
CO_2			32.82	
C_2H_6			1.35	
Ar			--	
N_2			5.42	
H_2S			0.19	
CH_4			21.22	
CO			5.74	
mol/hr (dry)			175	

	Solids Streams			
	1	7	12	4
wt % (dry)				
C	66.64		72.11	66.89
H	4.53		2.11	1.34
N	0.93		0.60	0.38
Cl	--		--	--
S	1.13		0.77	0.20
Ash	9.82		21.64	27.21
O	16.95		2.77	3.98
lb/hr (dry)	3551			
Moisture, wt %	9.84			
Slurry Concn, wt %	34			

	Liquid Streams		
	9	10	1
		lb/hr	
H_2O	--	--	--
Light Oil	--	--	7656

Gas Feed Streams			
2	3	5	11
	mol/hr		
241	90	22	0

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 Second-Stage Gasifier Product
⑦ First-Stage Gasifier Product
⑧ Gasifier Effluent After Quench
⑨ Water Made
⑩ Light Oil to Recycle
⑪ Gases From External Heater
⑫ Reactor Overhead

Figure 10. HYGAS REACTOR DATA FOR TEST 55 FOR STEADY PERIOD FROM 9/4/76 (1400 Hours) TO 9/5/76 (1200 Hours)

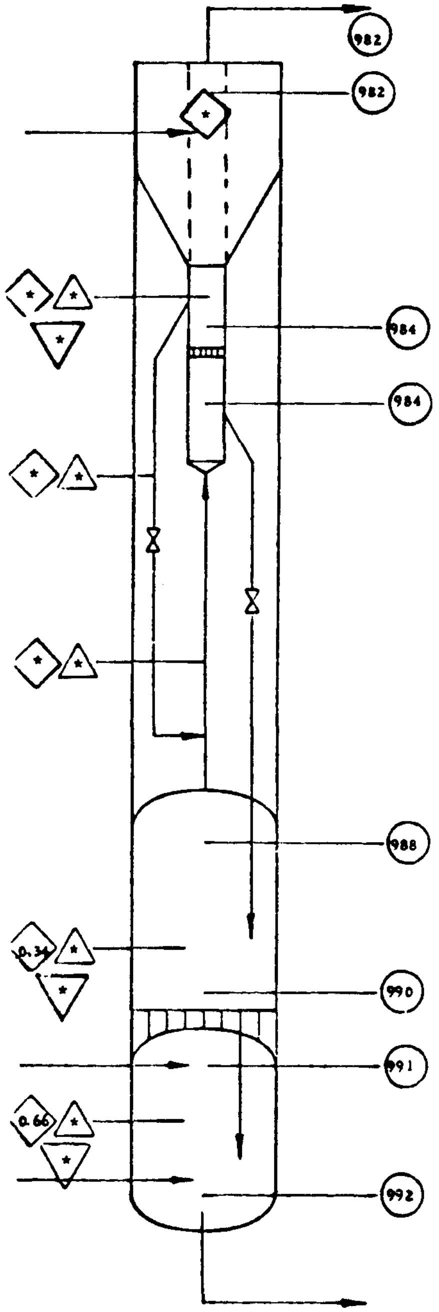
○ Pressure, psig

△ 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 = 0.20
lb Steam/lb Coal Fed = 1.68
lb Hydrogen/lb Coal Fed = 0
lb Coal Fed/1000 SCF Product Gas = *

	By Ash Balance
Coal Gasified, % =	71
Carbon Gasified, % =	64

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

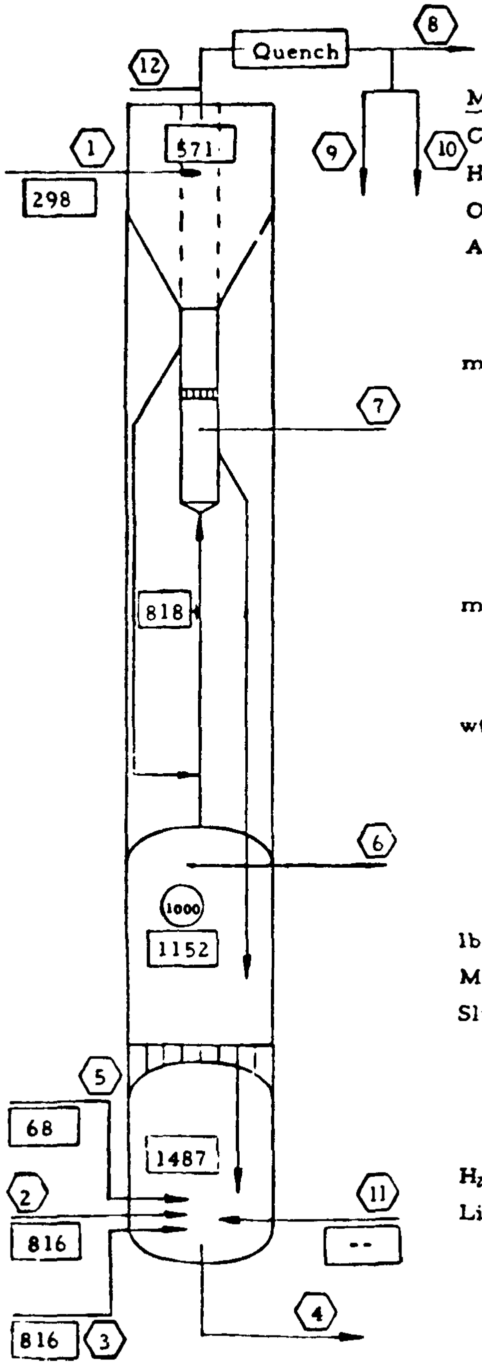
Bed Height (ft)

Slurry Dryer = *

HTR = *

SOG = *

Figure 11. HYGAS REACTOR ENGINEERING DATA FOR TEST 55 FOR STEADY PERIOD FROM 9/4/76 (1400 Hours) TO 9/5/76 (1200 Hours)



Material Balance, %

C

H

O

Ash

	Gas Streams			
	6	7	8	12
mol % (dry)				
H_2			33.29	30.48
CO_2			32.35	36.45
C_2H_6			1.59	1.66
Ar			--	--
N_2			4.86	4.95
H_2S			0.18	0.17
CH_4			21.34	20.55
CO			6.39	5.74
mol/hr (dry)			185	206

	Solids Streams			
	1	7	12	4
wt % (dry)				
C	67.41		73.65	66.21
H	4.45		2.17	1.31
N	0.90		0.62	0.38
Cl	--		--	--
S	0.93		0.66	0.20
Ash	9.65		19.47	27.92
O	16.66		3.43	3.98
lb/hr (dry)	3741			
Moisture, wt %	10.6			
Slurry Concn, wt %	33			

	Liquid Streams		
	9	10	1
		lb/hr	
H_2O	6932	--	--
Light Oil	--	8997	8311

Gas Feed Streams			
2	3	5	11
	mol/hr		
239	84	24	0

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 Second-Stage Gasifier Product
⑦ First-Stage Gasifier Product
⑧ Gasifier Effluent After Quench
⑨ Water Made
⑩ Light Oil to Recycle
⑪ Gases From External Heater
⑫ Reactor Overhead

Figure 12. HYGAS REACTOR DATA FOR TEST 55 FOR STEADY PERIOD FROM 9/5/76 (2200 Hours) TO 9/6/76 (1400 Hours)

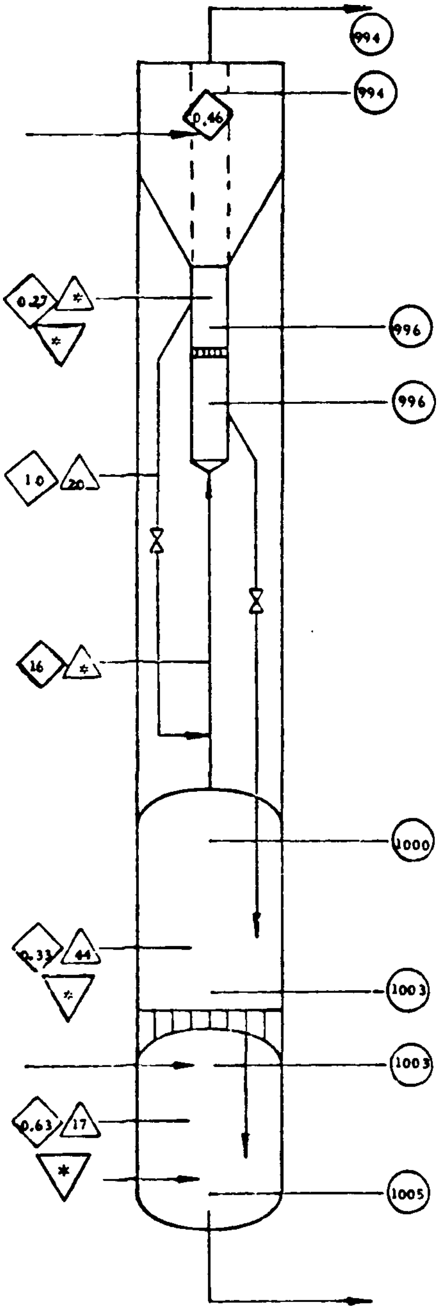
○ Pressure, psig

△ 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 = 0.21
lb Steam/lb Coal Fed = 1.55
lb Hydrogen/lb Coal Fed = 0
lb Coal Fed/1000 SCF Product Gas = 82

By Ash Balance

Coal Gasified, % = 72
Carbon Gasified, % = 66

Methane Yield, SCF/lb coal fed = 4.3
Carbon Oxide Yield, SCF/lb coal fed = 8.8

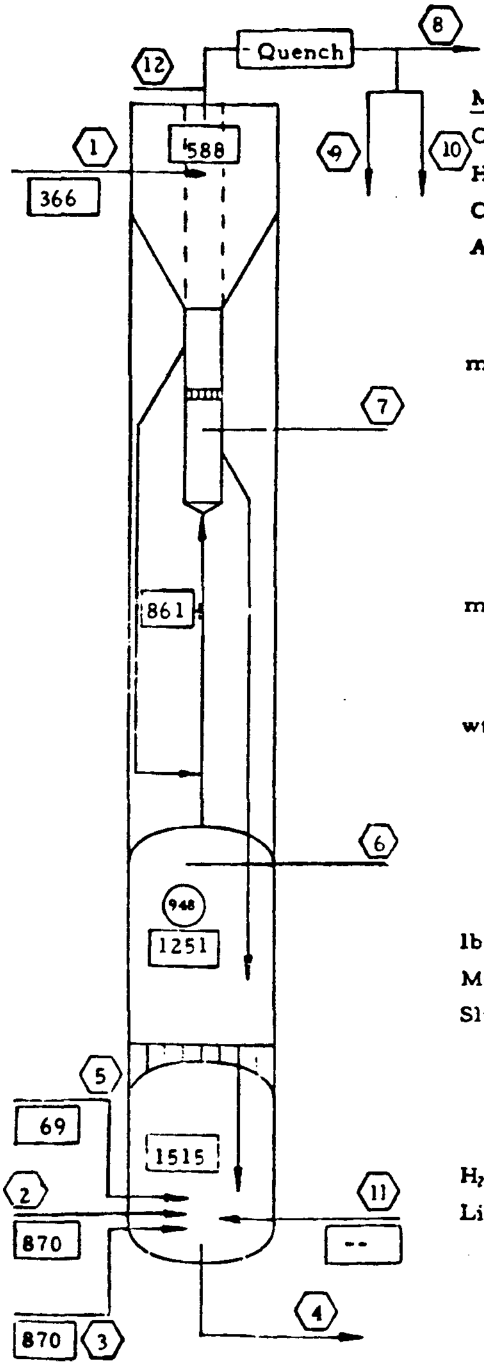
Bed Height (ft)

Slurry Dryer = *

HTR = 10

SOG = 14

Figure 13. HYGAS REACTOR ENGINEERING DATA FOR TEST 55 FOR STEADY PERIOD FROM 9/5/76 (2200 Hours) TO 9/6/76 (1400 Hours)



⬡ Stream No.

○ Pressure, psig

▭ Temperature, °F

Material Balance, %

C

H

O

Ash

	Gas Streams			
	6	7	8	12
mol % (dry)				
H_2			29.64	
CO_2			30.89	
C_2H_6			1.37	
Ar			--	
N_2			4.13	
H_2S			0.22	
CH_4			24.39	
CO			9.36	
mol/hr (dry)			234	

	Solids Streams			
	1	7	12	4
wt % (dry)				
C	67.33		72.47	67.66
H	4.52		2.23	1.22
N	0.95		0.75	0.36
Cl	--		--	--
S	0.87		0.84	0.22
Ash	9.14		19.48	26.48
O	17.19		4.23	4.06
lb/hr (dry)	5443			
Moisture, wt %	11			
Slurry Concn, wt %	36			

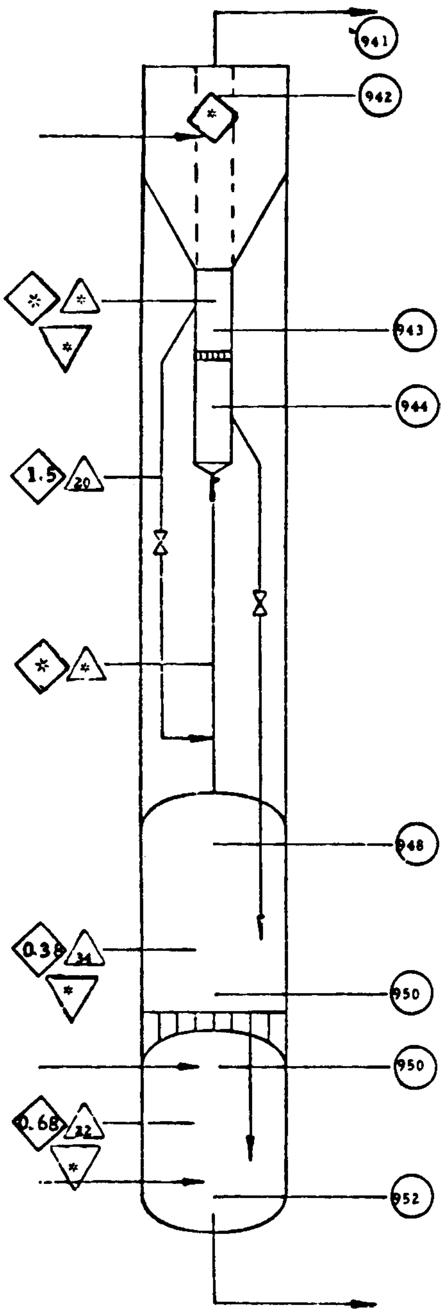
	Liquid Streams		
	9	10	1
		lb/hr	
H_2O	--	--	--
Light Oil	--	11,653	10,934

Gas Feed Streams			
2	3	5	11
	mol/hr		
230	94	27	0

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 Second-Stage Gasifier Product

⑦ First-Stage Gasifier Product
⑧ Gasifier Effluent After Quench
⑨ Water Made
⑩ Light Oil to Recycle
⑪ Gases From External Heater
⑫ Reactor Overhead

Figure 14. HYGAS REACTOR DATA FOR TEST 55 FOR STEADY PERIOD FROM 9/8/76 (2300 Hours) TO 9/9/76 (1200 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 = 0.16
lb Steam/lb Coal Fed = 1.07
lb Hydrogen/lb Coal Fed = 0
lb Coal Fed/1000 SCF Product Gas = 90.4

By Ash Balance

Coal Gasified, % = 72.1
Carbon Gasified, % = 65.3

Methane Yield, SCF/lb coal fed = 4.3
Carbon Oxide Yield, SCF/lb coal fed = 7.8

Bed Height (ft)
Slurry Dryer = *
HTR = 10
SOG = 11

Figure 15. HYGAS REACTOR ENGINEERING DATA FOR TEST 55 FOR STEADY PERIOD FROM 9/8/76 (2300 Hours) TO 9/9/76 (1200 Hours)

Test 56, October 1976

Highlights

During Test 56, self-sustained operation was maintained at a steady feed rate of 2 ton/hr for 11 hours, until a fitting on the high-pressure liquid nitrogen supply blew apart. This interrupted coal and oxygen feed to the reactor, resulting in plugging and subsequent termination of the test.

Details

Coal feed for Test 56 was initiated at 1620 hours on October 1, 1976, and the start-up burner was shut down at 0000 hours on October 3. Self-sustained operation was maintained at a steady coal feed rate of 2 ton/hr until 1300 hours on October 3, when a fitting blew apart on the high-pressure liquid nitrogen pump, shutting down the high-pressure nitrogen supply. Coal and oxygen feeds to the reactor were interrupted to maintain balanced pressure in the upper reactor section while the high-pressure nitrogen system was brought back into service. High-pressure nitrogen service was resumed at 2000 hours on October 3 and repeated attempts were made to bring the reactor back to steady-state conditions. Plugs had developed in the slurry dryer area and in transfer lines 321 and 322. These plugs could not be cleared and Test 56 was terminated at 1300 hours on October 4. Before the high-pressure nitrogen flow was interrupted, the unit had been performing very satisfactorily at an average feed rate of 2 ton/hr.

The reactor was opened following Test 56, and an inspection of the slurry dryer area revealed a layer of about 4 feet of damp coal (wet with light oil), which was then cleared. The second-stage gasification zone was in good condition with no accumulation of solids. The steam-oxygen gasification zone was in good condition with no damage to the steam-oxygen sparger or to its other internals. A small clinker had developed on the wall of the reactor immediately over the steam-oxygen inlet pipe connection through the side of the reactor. It is probable that this clinker was formed during the process upset on October 3, involving the high-pressure nitrogen system.

A leak in the slurry feed line into the slurry dryer, which had allowed slurry to drip into the dead space between the slurry dryer area and the outer reactor pressure shell, was found and repaired. This leak also could have contributed to the wet coal found in the slurry dryer area after shutdown, because liquid from the dead spaces could have leaked into the reactor section during depressurization and cool-off periods following the run. The short, steady-state operating time for Test 56 (11 hours) precludes any detailed analyses or test results for material balance purposes.

Test 57, October 1976

Highlights

Very smooth, self-sustained operation was achieved in Test 57 for about 48 hours, when a plug in the fresh-coal feed line forced termination of the test.

Details

Light-off for Test 57 occurred at 0600 hours on October 17, and coal feed was first initiated at 0115 hours on October 19. The system had to be brought down to 400 psi to repair a leaky sight gage on the high-pressure boiler. During the repair procedure, coal feed was interrupted and a plug developed in solids transfer line 321 from the slurry dryer area to the first-stage lift reactor. The plug was finally cleared by depressurizing the reactor and blasting the transfer line with high-pressure nitrogen. Coal feed was resumed at 0355 hours on October 20, and a steady-state 2 ton/hr feed rate was established at 1300 hours on October 21.

Test 57 ran very smoothly, self-sustained, and at a 2 ton/hr feed rate for about 48 hours. At 1750 hours on October 23, a plug developed in line 321, which transfers fresh coal to the lift-line reactor. Attempts to clear this plugged line by the usual technique of depressurizing the reactor and blasting with nitrogen were not successful. The test was thus terminated at 1400 hours on October 24, and the reactor was opened after cool-down on the morning of October 27. A 10-foot layer of coal containing some caked lumps was found in the slurry dryer area. The high bed level caused some carry-over of coal fines into the overhead product-gas line and into the char cyclone. Neither the dipleg nor the cyclone, however, was plugged.

The second-stage (high-temperature) reactor and the steam-oxygen reactor were in good condition, no clinkers or deposits of coal were found, and all of the valves and piping were in good operating condition. Line 321 was plugged with solids around the flow control valve in the line. The lift line and the lift-line disengaging space were clear.

Test 58, November 1976

Highlights

Test 58 was the most successful test with Rosebud subbituminous coal. The test extended over 11-1/2 days with a total coal feed of 753 tons. All sections of the plant were activated. The reactor smoothly handled solids flow at 3.3 ton/hr for extended periods of time.

Details

Light-off for Test 58 occurred at 1800 hours on November 6. The warm-up and pressurization period was prolonged because of the cold ambient temperatures and pressurization restrictions on the reactor shell at ambient temperatures below 75°F. Coal was introduced to the reactor at 1315 hours on November 9. Coal feed was stabilized at 2 ton/hr at 2300 hours on November 9, and the reactor was self-sustained, with no auxillary heat or hydrogen addition as of that time. The coal feed rate was raised to 3 ton/hr at 0830 hours on November 10 and continued at that nominal feed rate until 1330 hours on November 14. At this point, a flow interruption in the solids transfer from the second stage of hydrogasification to the steam/oxygen zone required the removal of oxygen from the unit. High-pressure, superheated steam was circulated through the unit to maintain pressure, and some process hydrogen was introduced to the unit to aid in fluidization of the upper stages of the reactor. Solids flow was reestablished to the steam/oxygen bed at about 1800 hours, and oxygen was reinjected to the steam-oxygen bed at 2030 hours on the same day. The reactor was brought back to steady-state conditions at 2-1/2 ton/hr of coal feed and all sections of the plant with the exception of the methanation section were brought on-line quickly, following the reintroduction of the oxygen feed.

Test 58 was terminated at 2000 hours on November 21 when a leak developed on the 24-inch Grayloc closure on manhole 4 where the start-up heater is connected to the steam-oxygen gasification zone. The leak was severe enough to cause operations to be interrupted and the reactor had to be depressurized rapidly. Purge nitrogen was introduced to the reactor to prevent flammable gas from escaping through the leak in the manhole. The unit could not be shut down by normal shutdown conditions, in which it is slowly depressurized and hydrogen purge gas is introduced to keep the upper stages of the reactor fluidized. It was also impossible to completely withdraw solids from the upper stages of the reactor, which would normally be attempted in a run shutdown situation.

Many significant achievements were made during Test 58. These included —

1. Excellent operation was obtained with the slurry pumping system. No mechanical problems occurred during the test.

2. The reactor performed very satisfactorily. Several periods of automatic control were achieved at feed rates of 3.3 ton/hr.

3. The purification section experienced initial foaming problems in the absorber. This was controlled partially by the use of antifoam agents (polyalcohols) and partly by raising the absorber temperature. Later in the test, the temperature in the absorber was reduced to 90^{o} to 100^{o}F and control of foaming was maintained by adding small amounts of antifoam agents. During this period the methanation section was put on-line.

4. The methanation section was on-line for 43-1/2 hours, during which complete carbon monoxide conversion and good control of process temperatures temperatures in the catalyst reactor were obtained. Sulfur removal through the zinc oxide guard chamber was excellent, and no indications of sulfur poisoning of the methanation catalyst was noted.

5. The restarting of the reactor, after the 7-hour interruption in coal and oxygen feed which occurred on the seventh day of the self-sustained operating period, was the first time the reactor was able to be restarted after such a long interruption of oxygen feed, without depressurizing the reactor and reinitiating the start-up heater cycle.

Two operating problems occurred during Test 58, which were solved during subsequent tests. These were —

1. The coal feed system gave intermittent service due to operating problems in the area of the top size screener and the oversized return chute. The subbituminous coal was received with a very high moisture content, which was reduced to the 12% moisture range in the coal preparation and drying section. The residual water vapor remaining in the coal feed condensed when it contacted lower ambient temperatures in the area of the top-size screen and oversized return chute. Condensation in these areas caused several interruptions in coal feed in order to remove caked, wet coal and to free these sections so the coal feed could be resumed. Oxygen flow to the reactor had to be reduced during these periods of low coal feed rate.

2. There were several upsets in the light oil stripper (LOS) operation due to carryover of coal fines through the stripping tower and into the separator, plugging that area and causing the stripper to be shut down. The coal fines had to be cleaned out manually before restarting. Toward the end of Test 58 this problem was alleviated somewhat, but oil recovery during most of Test 58 was poor.

Post-run inspection of the reactor showed most sections of the reactor to be in good condition, with the exception of an ash-clinker deposit in the steam-oxygen bed. The slurry dryer area had coal covering the slurry dryer grid up to the stilling well, where coal overflowed and traveled down the 321 line to the first-stage reactor. This was normal after tests with this subbituminous coal. The 321 line itself was plugged for about one-third of its length at the base of the line; this was attributed to the mode of shutdown. The lift line was completely clean, but the line transferring solids from the first-stage disengaging space to the second-stage gasification zone was likewise plugged with solids for about one-third of its length. Both of these plugs were evidentaly caused by the rapid shutdown procedures that were necessary and by the inability to completely drain solids from the upper section of the reactor. There was no indication at any time during the run of any flow problems or accumulation of solids in these areas.

The second-stage gasification zone was entirely clear and free of deposits. The steam-oxygen bed had developed a clinker or ash formation just above the sparger ring and extending 2 to 3 feet up along the reactor walls. This ash deposit was spread around the entire circumference of the inside of the steam-oxygen reactor zone and was probably primarily formed during the process interruption described above that occurred on November 17. It is likely that when the coal flow from the second gasification stage to the steam-oxygen zone was interrupted, high oxygen/coal ratios and over-gasification occurred, with subsequent overheating and ash formation in this bed.

One objective of Test 58 that was not achieved, was the demonstration of coal conversions of over 90%, using subbituminous coal. Coal conversion rates of 70% to 80% were obtained for most of the operating periods. Coal conversion was monitored daily by an analytical technique for determining ash in the bottom discharge from the reactor. Due to limitations in coal feed rates discussed earlier (which caused interruptions and/or cutbacks in oxygen feed rate), the oxygen/coal ratio and the temperatures in the steam-oxygen gasification zone could not be increased to achieve better than 80% coal conversion.

Engineering data for Test 58 are presented in Figures 16 through 33. A material balance for Test 58 was prepared and is presented in Table 7.

Preparation for Tests With Bituminous Coal

After Test 58, the ERDA/A.G.A. Operating Committee directed IGT to return to bituminous coal for the next series of test operations at the HYGAS pilot plant. At this point, a variety of maintenance work and improvements were made in the plant, and work was done in coal selection and preparation for tests with the new coal type. The details of work performed during this period are given below.

Repairs to the Grayloc Closure

Inspection of the 24-inch Grayloc closure on manway 4 of the reactor, following the shutdown of Test 58, indicated the need to remachine the flange face to obtain good sealing on this area. The exact location of the leak at the 24-inch Grayloc closure (flange) is circled in Figure 34. Considerable metal loss resulted from the highly erosive leaking gases, principally on the reactor flange. The entire flange face on both units had to be built up with stainless steel weld overlay and machined to original dimensions.

The application of the weld overlay is shown in Figure 35. The method of weld application had to be qualified, physical testing had to be completed on weld overlay samples, and the entire process had to be conducted in accordance with the required regulations including inspection, in order for the vessel to remain under an ASME code stamp. This overall procedure was very time consuming but necessary to conform to all code regulations.

Improvments in the Coal Preparation Section

Persistent problems occurred in maintaining a smooth coal feed rate during Test 58. Most mechanical problems occurred in the Sweco screen and in the oversized discharge chute from this screening operation. The Montana

Table 7. MATERIAL BALANCE SUMMARY FOR THE HYGAS GASIFIER FOR TEST 58 FROM 11/10/76 (1200 HOURS) TO 11/11/76 (0700 HOURS)

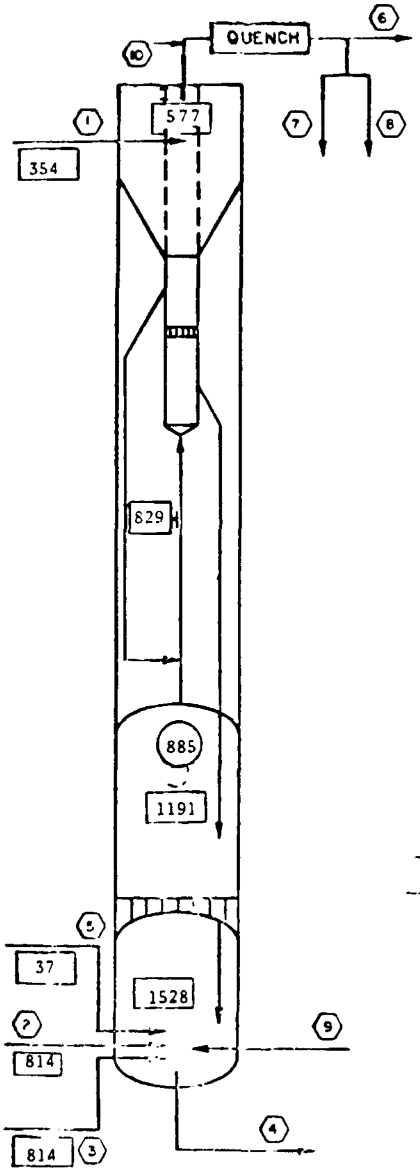
Basis = 1 hr. All units in lbs. unless noted otherwise

INPUT		C	H	O	N	S	Cl	ASH	OTHER	TOTAL
Coal Feed	Wt. % (Dry)	67.78	4.44	16.34	0.92	0.91	--	9.61		100
	Coal (Dry)	3590	235	866	49	48	--	509		5297
	Moisture		70	561						631
Sparger	Oxygen			809						809
	Steam		539	4316						4855
Burner	Oxygen			0						0
	Steam		0	0						0
	Hydrogen		0							0
Stripping Ring	Steam		183	1461						1644
Nitrogen from purges					509					509
Pump seal flush			81	652						733
Cooling water spray			0	0						0
Water to cyclone pot			544	4352						4896
Light oil in		8879	846							9725
TOTAL INPUT		12,469	2498	13,017	558	48	--	509		29,099
OUTPUT										
Reactor Overhead	Wt. % (Dry)	74.08	2.49	3.67	0.65	0.52	--	18.59		100
	Dust (Dry)	307	10	15	3	2	--	77		414
Spent Char	Wt. % (Dry)	67.20	1.15	3.45	0.34	0.16	--	27.70		100
	Char (Dry)	1025	18	53	5	2	--	422		1525
Product Gas After Quench	Total (Dry)	1587	330	2351	243	11				4522
	Components H_2		125							125
	CO_2	803		2143						2946
	C_2H_6	60	15							75
	H_2S		1			11				12
	N_2				243					243
	CH_4	568	189							757
	CO	156		208						364
Water Out + dissolved materials		23	1239	9872	21	54				11,209
Toluene storage tank vent gases		262	24	437	19	0				742
Stripper vent gas		14	1	23	3	0				41
Light oil out		10,149	967							11,116
TOTAL OUTPUT		13,367	2589	12,751	294	69	--	499		29,569
Net (Output-Input)		898	91	–266	–264	21	--	–10		470
% Balance (Output/Input)		107	104	98	53	144	--	98		102

MATERIAL BALANCE, %

C	107
H	104
O	98
ASH	98

○ STREAM No.
○ PRESSURE, psig
☐ TEMPERATURE, °F



GAS STREAMS	6	10
mol % (dry)		
H_2	31.10	28.99
CO_2	33.23	36.41
C_2H_6	1.24	1.34
Ar	--	--
N_2	4.31	4.26
H_2S	0.17	0.17
CH_4	23.50	22.86
CO	6.45	5.97
mol/hr (dry)	201	220

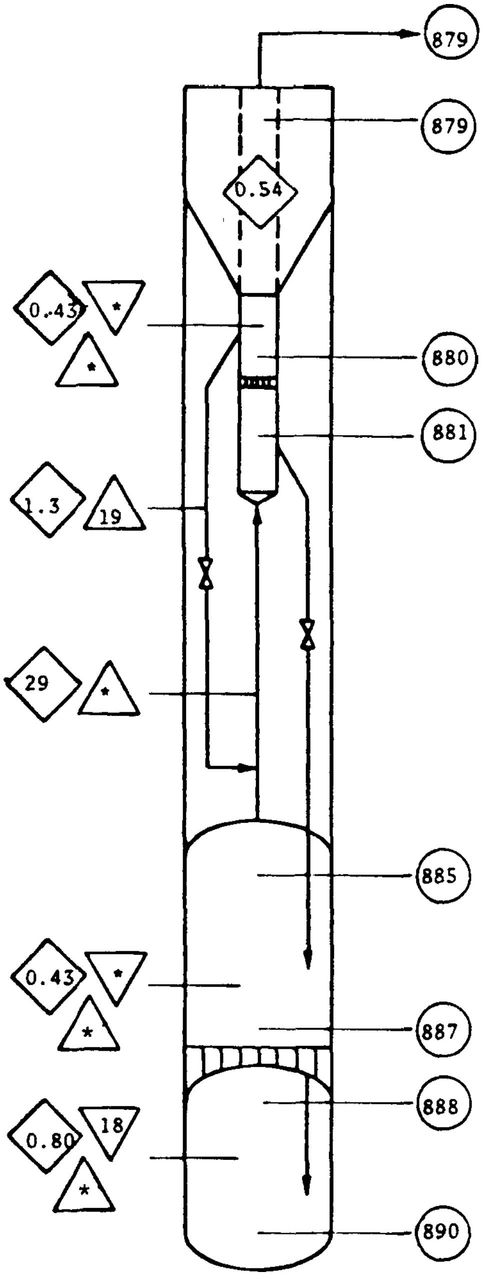
SOLIDS STREAMS	1	10	4
wt % (dry)			
C	67.78	74.08	67.20
H	4.44	2.49	1.15
N	0.92	0.65	0.34
Cl	--	--	--
S	0.91	0.52	0.16
ASH	9.61	18.59	27.70
O	16.34	3.67	3.45
lb/hr (dry)	5297	414	1525
MOISTURE, wt %	11		
SLURRY CONCN, wt %	38		

LIQUID STREAMS	7	8	1
	lb/hr		
H_2O	6577	--	--
LIGHT OIL	--	11,116	9725

GAS FEED STREAMS 2	3	5	9
mol/hr			
270	91	25	0

①	Feed Slurry	⑥	Gasifier Effluent After Quench
②	High-Pressure Steam to Steam-Oxygen Sparger	⑦	Water Made
③	High-Pressure Steam to Stripping Ring	⑧	Light Oil to Recycle
④	Spent Char	⑨	Gases From External Heater
⑤	High-Pressure Oxygen to Steam-Oxygen Sparger	⑩	Reactor Overhead

Figure 16. HYGAS REACTOR DATA FOR TEST 58 FOR THE STEADY PERIOD FROM 11/10/76 (1200 HOURS) TO 11/11/76 (0700 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
Carbon (net) = total carbon in – carbon in overhead

lb Oxygen/lb Carbon (net) =	0.25
lb Steam/lb Carbon (net) =	2.0
lb Oxygen/lb Coal Fed =	0.15
lb Steam/lb Coal Fed =	1.23
lb Hydrogen/lb Coal Fed =	0
lb Coal Fed/1000 SCF Product Gas =	100

By Ash Balance

MAF[†] Carbon Gasified, % = 72
Carbon Gasified, % = 66

Methane Yield SCF/lb coal fed = 3.6
Equivalent Methane Yield, SCF/lb coal fed = 5.3

Bed Height, ft

Slurry Dryer = *
HTR = 10
SOG = 16

[†] Moisture ash free

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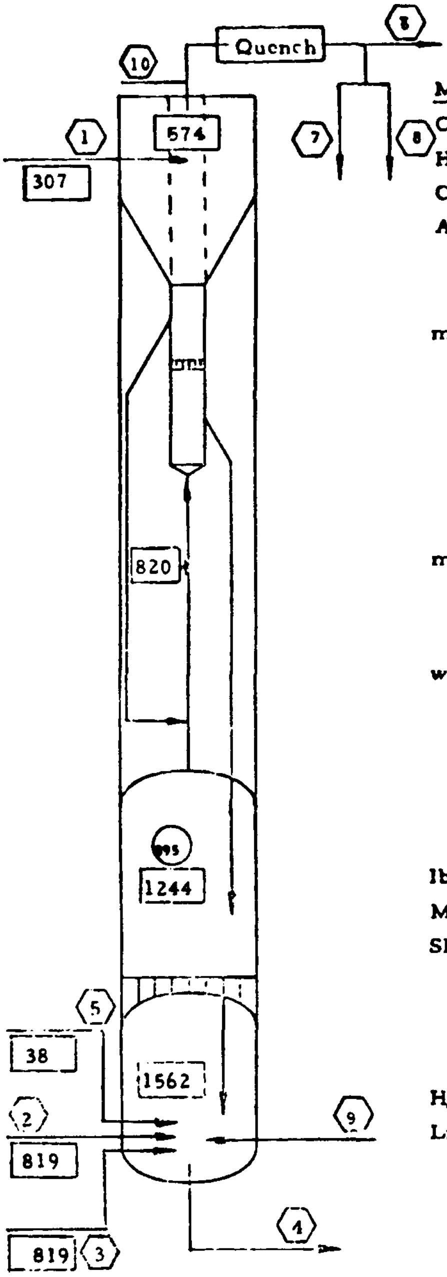
Figure 17. HYGAS REACTOR ENGINEERING DATA FOR TEST 58 FOR THE STEADY PERIOD FROM 11/10/76 (1200 HOURS) TO 11/11/75 (0700 HOURS)



⬡ Stream No.

○ Pressure, psig

☐ Temperature, °F

Material Balance, %

C

H

O

Ash

	Gas Streams	
	6	10
mol % (dry)		
H_2	30.68	29.49
CO_2	31.70	33.52
C_2H_6	1.51	1.51
Ar	--	--
N_2	4.83	4.72
H_2S	0	0.03
CH_4	23.84	23.53
CO	7.44	7.20
mol/hr (dry)	258	271

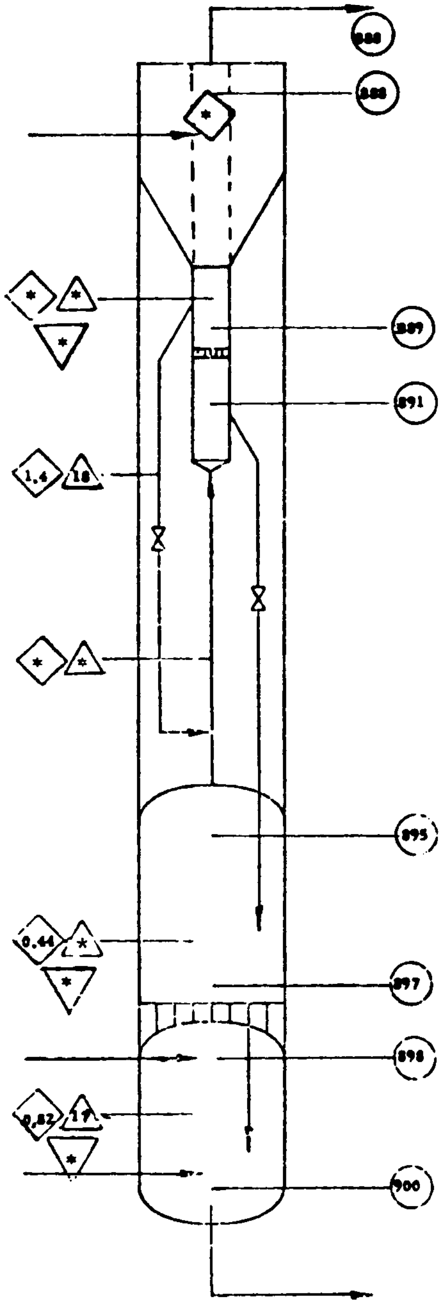
	Solids Streams		
	1	10	4
wt % (dry)			
C	67.80	73.70	69.20
H	4.43	2.49	1.17
N	0.98	0.69	0.27
Cl	--	--	--
S	0.88	0.66	0.11
Ash	9.24	17.99	26.35
O	16.67	4.47	2.90
lb/hr (dry)	5557		
Moisture, wt %	11		
Slurry Concn, wt %	40		

	Liquid Streams		
	7	8	1
	lb/hr		
H_2O	--	--	--
Light Oil	--	--	9145

Gas Feed Streams			
2	3	5	9
mol/hr			
270	91	29	0

⟨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 18. HYGAS REACTOR DATA FOR TEST 58 FOR THE STEADY PERIOD FROM 11/12/76 (1200 Hours) TO 11/12/76 (1800 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 = 0.17
lb Steam/lb Coal Fed = 1.17
lb Hydrogen/lb Coal Fed = 0
lb Coal Fed/1000 SCF Product Gas = 87

By Ash Balance

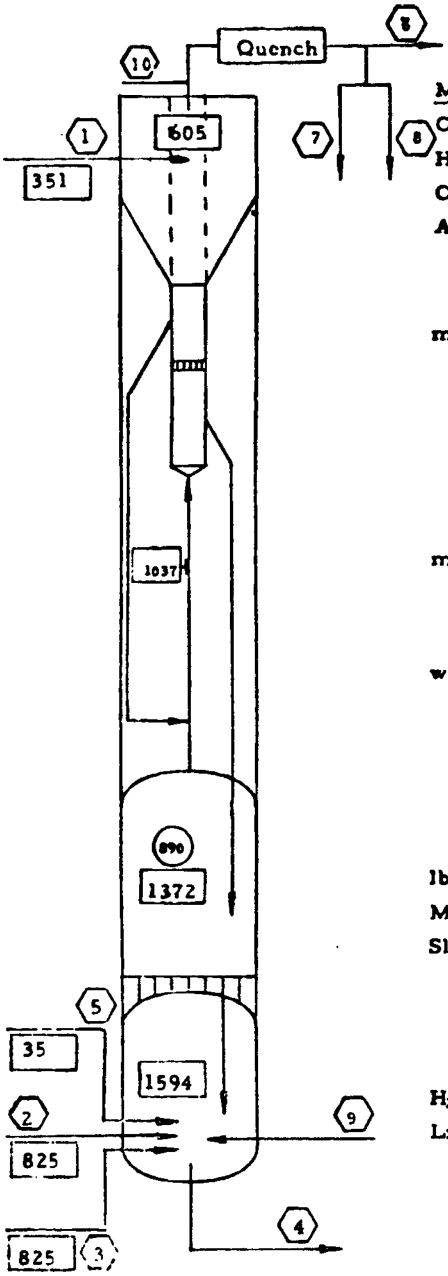
Coal Gasified, % = 72
Carbon Gasified, % = 64

Methane Yield, SCF/lb coal fed = 4.4
Carbon Oxide Yield, SCF/lb coal fed = 7.5

Bed Height (ft)

Slurry Dryer = *
HTR = 10
SOG = 17

Figure 19. HYGAS REACTOR ENGINEERING DATA FOR TEST 58 FOR THE STEADY PERIOD FROM 11/12/76 (1200 Hours) TO 11/12/76 (1800 Hours)



Material Balance, %

C

H

O

Ash

	Gas Streams	
	6	10
mol % (dry)		
H_2	29.65	
CO_2	32.93	
C_2H_6	1.20	
Ar	--	
N_2	4.10	
H_2S	0	
CH_4	21.21	
CO	10.91	
mol/hr (dry)	250	

	Solids Streams		
	1	10	4
wt % (dry)			
C	66.77	71.40	64.93
H	4.19	1.04	0.96
N	0.98	0.46	0.17
Cl	--	--	--
S	0.94	0.30	0.21
Ash	9.65	23.64	30.76
O	17.47	3.16	2.97
lb/hr (dry)	5527		
Moisture, wt %	8.13	1.62	5.75
Slurry Concn, wt %	38		

	Liquid Streams		
	7	8	1
		lb/hr	
H_2O	--	--	--
Light Oil	--	--	9748

Gas Feed Streams			
2	3	5	9
	mol/hr		
293	92	30	0

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

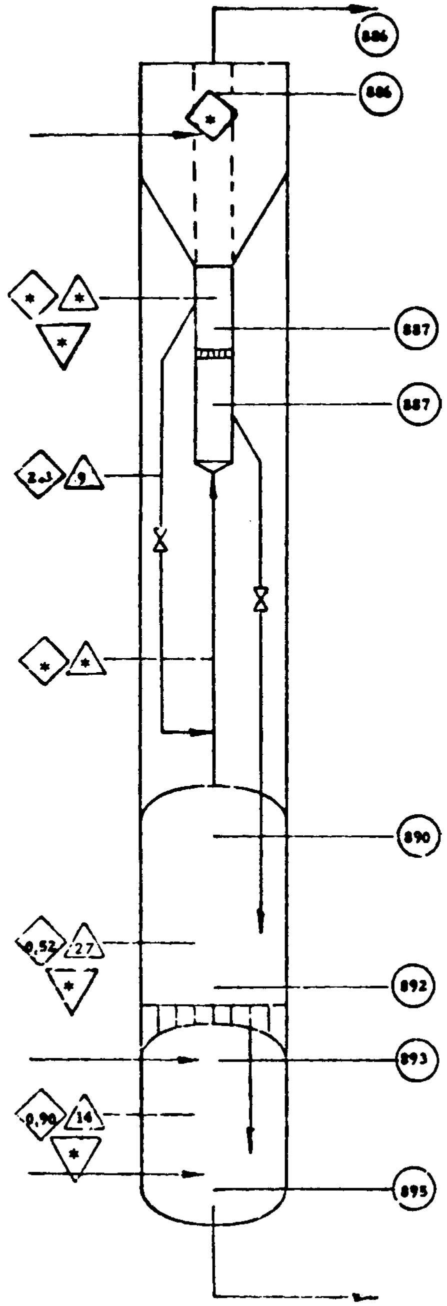
7 Water Made

8 Light Oil to Recycle

9 Gases From External Heater

10 Reactor Overhead

Figure 20. HYGAS REACTOR DATA FOR TEST 58 FOR THE STEADY PERIOD FROM 11/14/76 (1400 Hours) TO 11/15/76 (0800 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 = 0.17
lb Steam/lb Coal Fed = 1.25
lb Hydrogen/lb Coal Fed = 0
lb Coal Fed/1000 SCF Product Gas = *

By Ash Balance

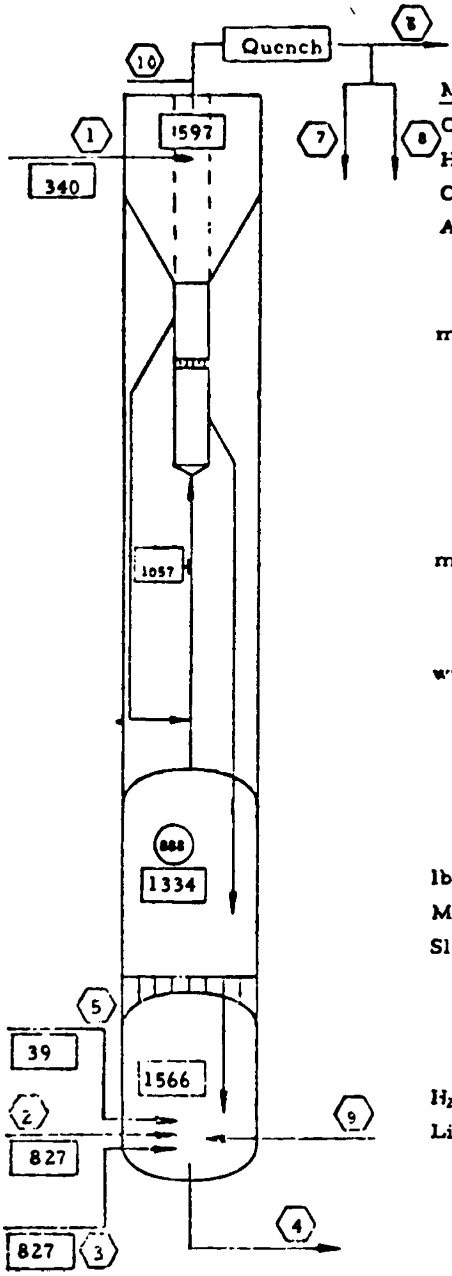
Coal Gasified, % = 76
Carbon Gasified, % = 69

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

Bed Height (ft)

Slurry Dryer = *
HTR = 12
SOG = 19

Figure 21. HYGAS REACTOR ENGINEERING DATA FOR TEST 58 FOR THE STEADY PERIOD FROM 11/14/76 (1400 Hours) TO 11/15/76 (0800 Hours)



⬡	Stream No.
○	Pressure, psig
▭	Temperature, °F

Material Balance, %

C

H

O

Ash

	Gas Streams	
	6	10
mol % (dry)		
H_2	27.97	26.65
CO_2	33.21	35.30
C_2H_6	1.33	1.39
Ar	--	--
N_2	4.48	4.44
H_2S	0	0
CH_4	23.65	23.31
CO	9.36	8.91
mol/hr (dry)	237	252

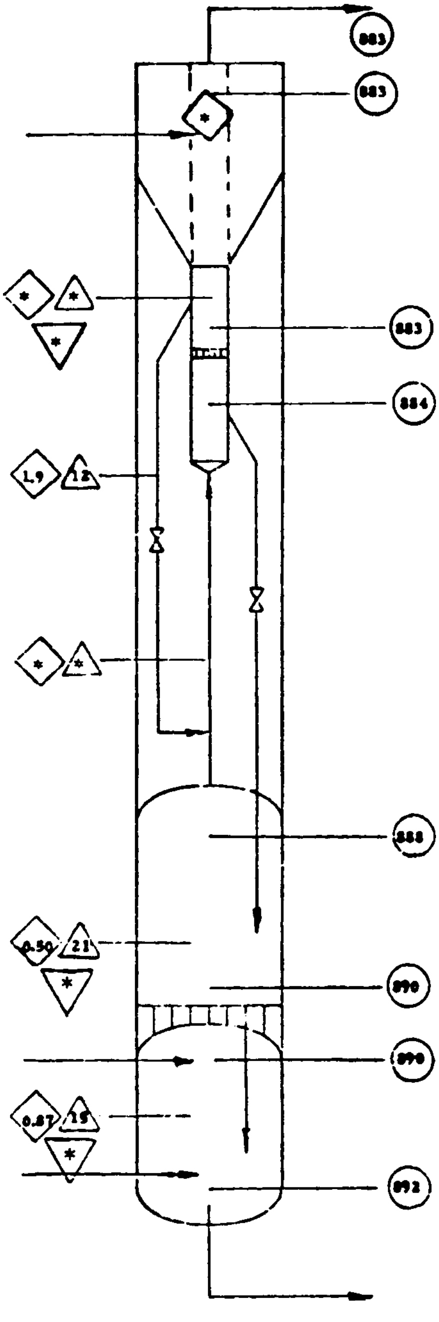
	Solids Streams		
	1	10	4
wt % (dry)			
C	67.49	71.49	66.88
H	4.40	2.81	1.02
N	0.90	0.75	0.24
Cl	--	--	--
S	0.94	0.69	0.16
Ash	9.54	17.47	28.64
O	16.73	6.79	3.06
lb/hr (dry)	5478		
Moisture, wt %	10		
Slurry Concn, wt %	38		

	Liquid Streams		
	7	8	1
	lb/hr		
H_2O	--	--	--
Light Oil	--	--	10,029

Gas Feed Streams			
2	3	5	9
mol/hr			
291	92	30	0

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 22. HYGAS REACTOR DATA FOR TEST 58 FOR THE STEADY PERIOD FROM 11/15/76 (0800 Hours) TO 11/16/76 (0800 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 = 0.17
lb Steam/lb Coal Fed = 1.26
lb Hydrogen/lb Coal Fed = 0
lb Coal Fed/1000 SCF Product Gas = 95

By Ash Balance

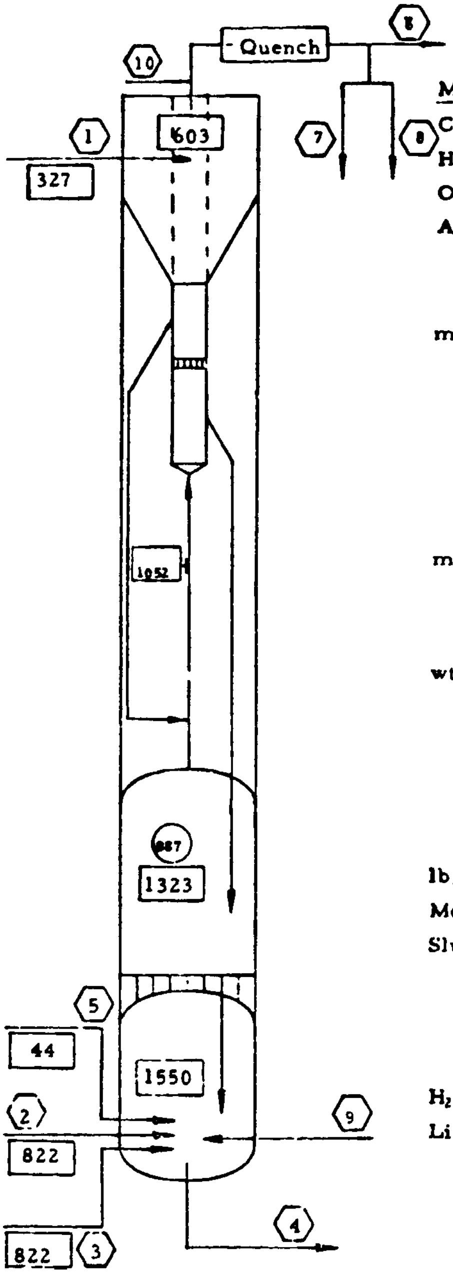
Coal Gasified, % = 74
Carbon Gasified, % = 67

Methane Yield, SCF/lb coal fed = 4.1
Carbon Oxide Yield, SCF/lb coal fed = 7.7

Bed Height (ft)

Slurry Dryer = *
HTR = 15
SOG = 19

Figure 23. HYGAS REACTOR ENGINEERING DATA FOR TEST 58 FOR THE STEADY PERIOD FROM 11/15/76 (0800 Hours) TO 11/16/76 (0800 Hours)



⬡ Stream No.

○ Pressure, psig

▭ Temperature, °F

Material Balance, %

C

H

O

Ash

	Gas Streams	
	6	10
mol % (dry)		
H_2	28.43	26.68
CO_2	31.96	34.77
C_2H_6	1.51	1.63
Ar	--	--
N_2	6.75	6.56
H_2S	0.20	0.22
CH_4	24.19	23.64
CO	6.96	6.50
mol/hr (dry)	235	256

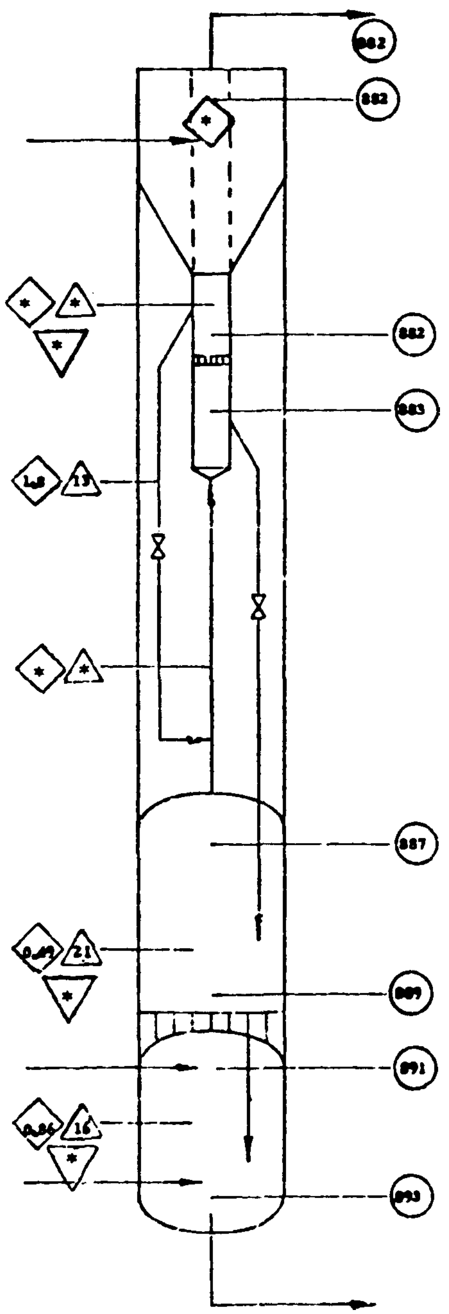
	Solids Streams		
	1	10	4
wt % (dry)			
C	68.00	71.70	67.90
H	4.50	2.94	1.16
N	0.81	0.77	0.34
Cl	--	--	--
S	0.93	0.61	0.13
Ash	9.21	16.69	27.27
O	16.55	7.29	3.20
lb/hr (dry)	5813		
Moisture, wt %	11		
Slurry Concn, wt %	40		

	Liquid Streams		
	7	8	1
	lb/hr		
H_2O	--	--	--
Light Oil	--	--	9648

Gas Feed Streams			
2	3	5	9
mol/hr			
291	92	29	0

⬡1	Feed Slurry	⬡6	Gasifier Effluent After Quench
⬡2	High-Pressure Steam to Steam-Oxygen Sparger	⬡7	Water Made
⬡3	High-Pressure Steam to Stripping Ring	⬡8	Light Oil to Recycle
⬡4	Spent Char	⬡9	Gases From External Heater
⬡5	High-Pressure Oxygen to Steam-Oxygen Sparger	⬡10	Reactor Overhead

Figure 24. HYGAS REACTOR DATA FOR TEST 58 FOR THE STEADY PERIOD FROM 11/16/76 (1800 Hours) TO 11/16/76 (2400 Hours)



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

lb Oxygen/lb Coal Fed = 0.16
lb Steam/lb Coal Fed = 1.18
lb Hydrogen/lb Coal Fed = 0
lb Coal Fed/1000 SCF Product Gas = 102

By Ash Balance

Coal Gasified, % = 73
Carbon Gasified, % = 66

Methane Yield, SCF/lb coal fed = 4.0
Carbon Oxide Yield, SCF/lb coal fed = 6.9

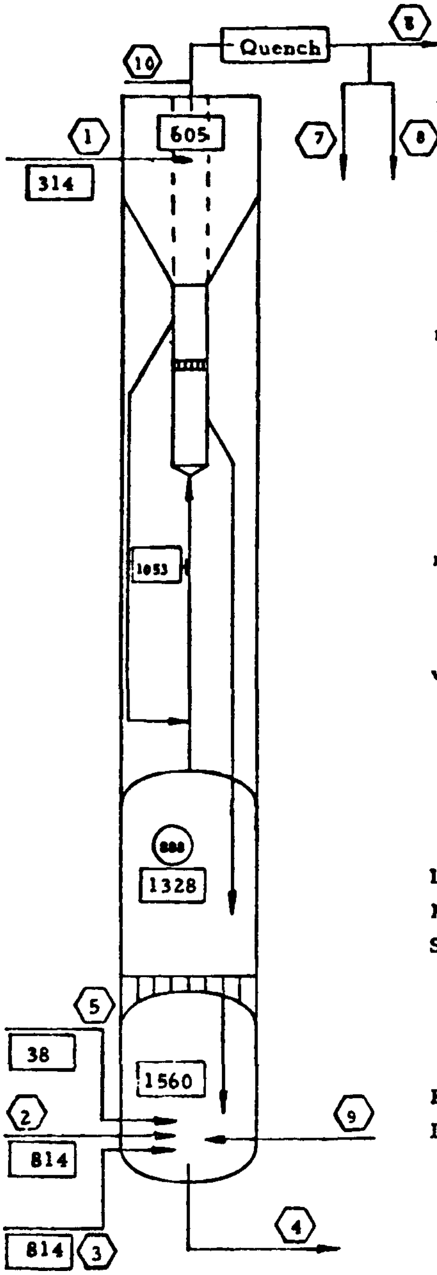
Bed Height (ft)

Slurry Dryer = *

HTR = 16

SOC = 18

Figure 25. HYGAS REACTOR ENGINEERING DATA FOR TEST 58 FOR THE STEADY PERIOD FROM 11/16/76 (1800 Hours) TO 11/16/76 (2400 Hours)



⬡ Stream No.
○ Pressure, psig
▭ Temperature, °F

Material Balance, %

C
H
O
Ash

	Gas Streams	
	6	10
mol % (dry)		
H_2	28.60	26.81
CO_2	32.83	35.61
C_2H_6	1.51	1.62
Ar	--	--
N_2	6.65	6.47
H_2S	0	0.04
CH_4	23.46	22.98
CO	6.95	6.47
mol/hr (dry)	244	265

	Solids Streams		
	1	10	4
wt % (dry)			
C	67.60	70.90	68.30
H	4.46	2.99	1.15
N	0.89	0.76	0.34
Cl	--	--	--
S	0.95	0.70	0.19
Ash	9.51	17.51	26.95
O	16.59	7.14	3.07
lb/hr (dry)	6414		
Moisture, wt %	10		
Slurry Concn, wt %	42		

	Liquid Streams		
	7	8	1
	lb/hr		
H_2O	--	--	--
Light Oil	--	--	9829

Gas Feed Streams			
2	3	5	9
mol/hr			
292	92	30	0

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 26. HYGAS REACTOR DATA FOR TEST 58 FOR THE STEADY PERIOD FROM 11/17/76 (0000 Hours) TO 11/17/76 (0600 Hours)

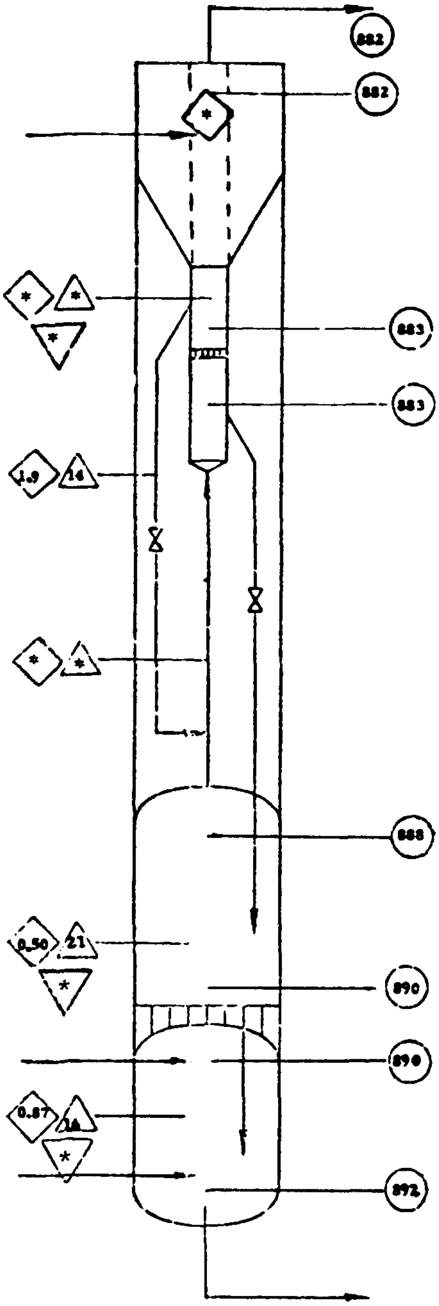
○ Pressure, psig

△ 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 = 0.15
lb Steam/lb Coal Fed = 1.08
lb Hydrogen/lb Coal Fed = 0
lb Coal Fed/1000 SCF Product Gas = 110

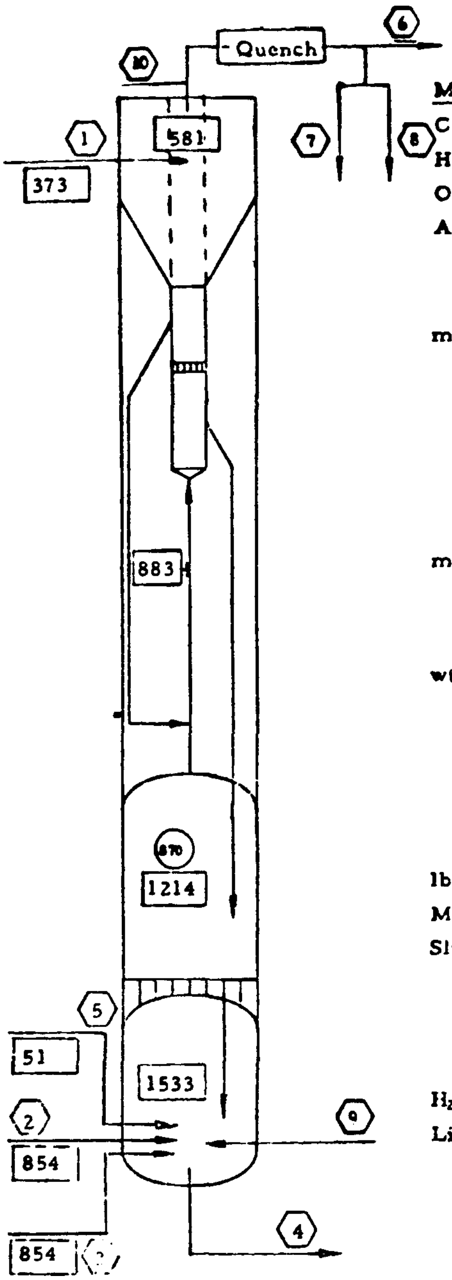
By Ash Balance

Coal Gasified, % = 72
Carbon Gasified, % = 64
Methane Yield, SCF/lb coal fed = 3.6
Carbon Oxide Yield, SCF/lb coal fed = 6.6

Bed Height (ft)

Slurry Dryer = *
HTR = 16
SOG = 18

Figure 27. HYGAS REACTOR ENGINEERING DATA FOR TEST 58 FOR THE STEADY PERIOD FROM 11/17/76 (0000 Hours) TO 11/17/76 (0600 Hours)



⬡ Stream No.
○ Pressure, psig
▭ Temperature, °F

Material Balance, %

C
H
O
Ash

	Gas Streams	
	6	10
mol % (dry)		
H_2	28.92	26.61
CO_2	30.21	34.18
C_2H_6	2.25	2.26
Ar	--	--
N_2	8.06	7.97
H_2S	0	0
CH_4	23.98	23.05
CO	6.58	5.93
mol/hr (dry)	203	225

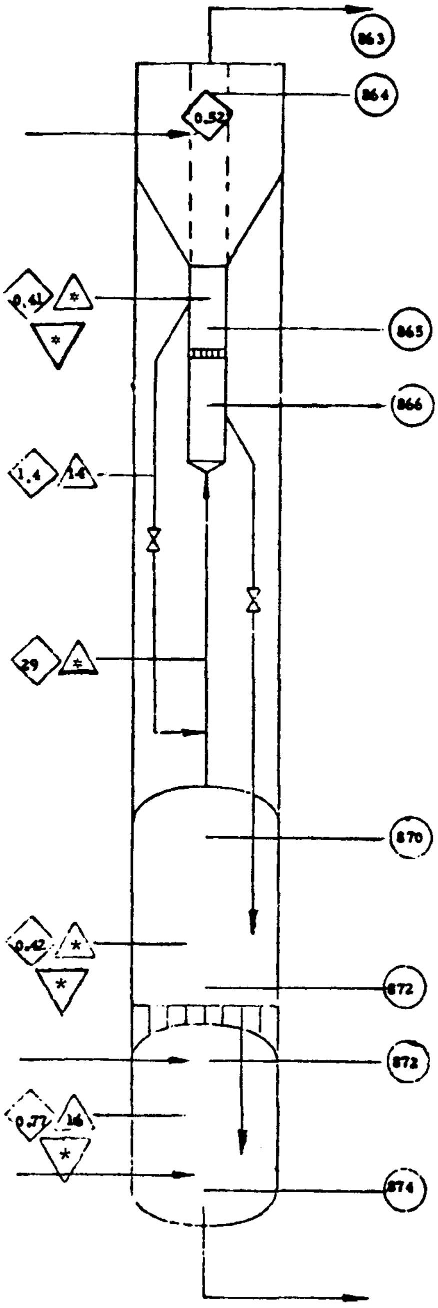
	Solids Streams		
	1	10	4
wt % (dry)			
C	67.56	73.40	69.28
H	4.39	2.17	1.34
N	0.92	0.61	0.36
Cl	--	--	--
S	0.88	0.71	0.18
Ash	9.36	19.31	25.23
O	16.89	3.80	3.61
lb/hr (dry)	4447		
Moisture, wt %	11		
Slurry Concn, wt %	33		

	Liquid Streams		
	7	8	1
	lb/hr		
H_2O	--	--	--
Light Oil	--	--	9913

Gas Feed Streams			
2	3	5	9
mol/hr			
252	90	23	0

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 28. HYGAS REACTOR DATA FOR TEST 58 FOR THE STEADY PERIOD FROM 11/18/76 (1000 Hours) TO 11/19/76 (1800 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 = 0.17
lb Steam/lb Coal Fed = 1.38
lb Hydrogen/lb Coal Fed = 0
lb Coal Fed/1000 SCF Product Gas = 89

By Ash Balance

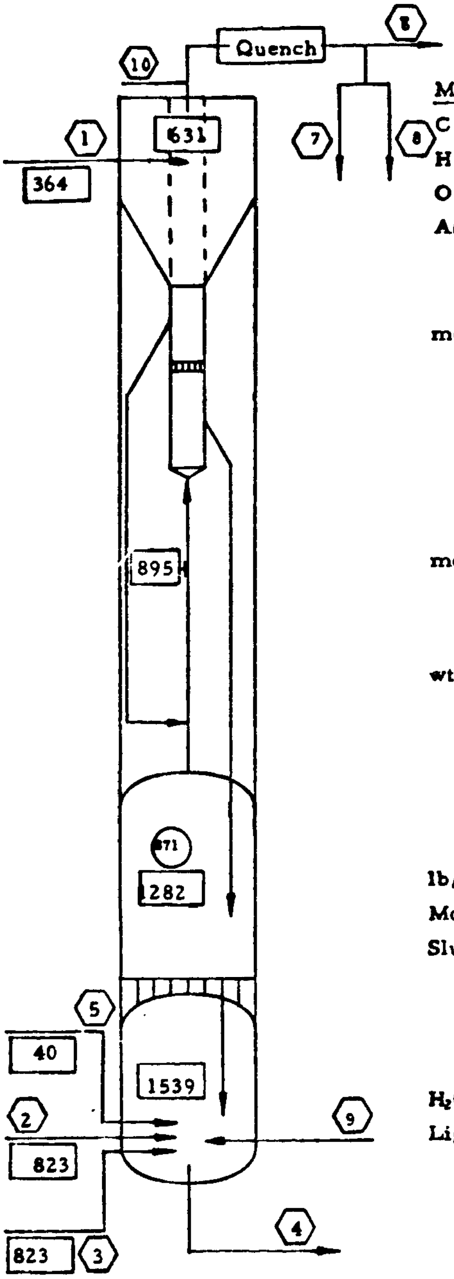
Coal Gasified, % = 69
Carbon Gasified, % = 62

Methane Yield, SCF/lb coal fed = 4.5
Carbon Oxide Yield, SCF/lb coal fed = 7.8

Bed Height (ft)

Slurry Dryer = *
HTR = 10
SOG = 18

Figure 29. HYGAS REACTOR ENGINEERING DATA FOR TEST 58 FOR THE STEADY PERIOD FROM 11/18/76 (1000 Hours) TO 11/19/76 (1800 Hours)



⬡ **Stream No.**
○ **Pressure, psig**
▭ **Temperature, °F**

Material Balance, %

C
H
O
Ash

	Gas Streams	
	6	10
mol % (dry)		
H_2	29.39	26.74
CO_2	32.37	37.00
C_2H_6	1.25	1.34
Ar	--	--
N_2	8.15	7.59
H_2S	.10	0.10
CH_4	22.65	21.72
CO	6.09	5.51
mol/hr (dry)	200	225

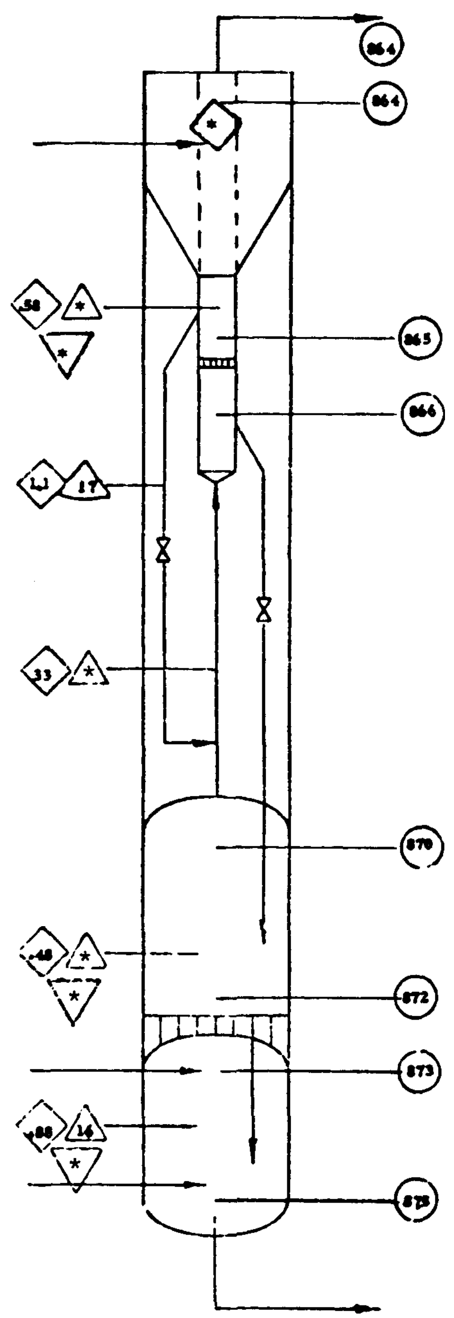
	Solids Streams		
	1	10	4
wt % (dry)			
C	66.80	72.70	69.40
H	4.38	2.25	1.37
N	.91	.61	.41
Cl	--	--	--
S	.97	.82	.19
Ash	9.99	19.64	25.03
O	16.95	3.98	3.60
lb/hr (dry)	4295		
Moisture, wt %	10.1		
Slurry Concn, wt %	34.05		

	Liquid Streams		
	7	8	1
	lb/hr		
H_2O	--	--	--
Light Oil	--	--	9484

Gas Feed Streams			
2	3	5	9
mol/hr			
294	91	26	0

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 30. HYGAS REACTOR DATA FOR TEST 58 FOR THE STEADY PERIOD FROM 11/20/76 (2200 Hours) TO 11/21/76 (0300 Hours)



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

lb Oxygen/lb Coal Fed = 0.20
lb Steam/lb Coal Fed = 1.61
lb Hydrogen/lb Coal Fed = 0
lb Coal Fed/1000 SCF Product Gas = 90.2

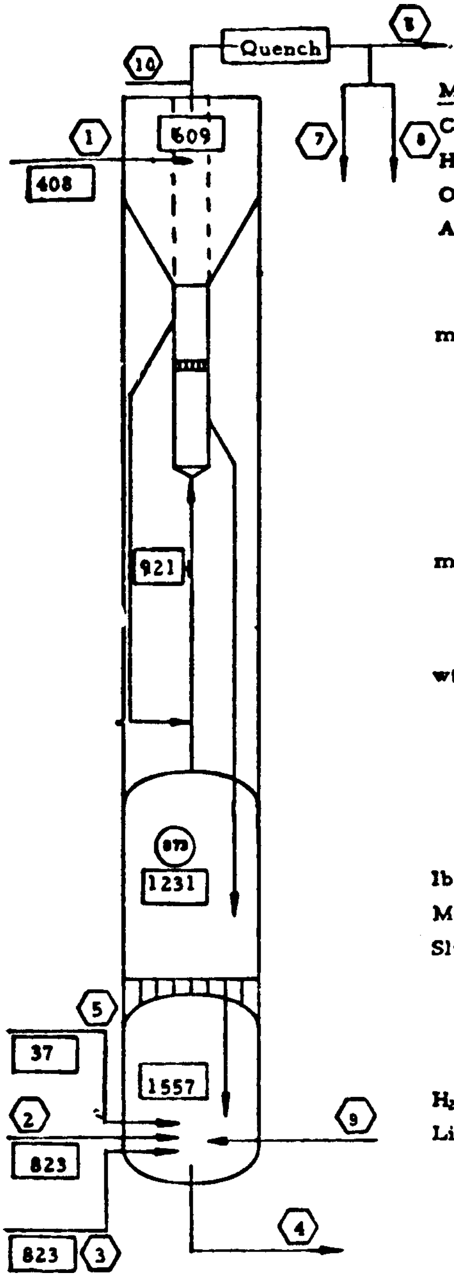
By Ash Balance

Coal Gasified, % = 67
Carbon Gasified, % = 63
Methane Yield, SCF/lb coal fed = 4.3
Carbon Oxide Yield, SCF/lb coal fed = 8.5

Bed Height (ft)

Slurry Dryer = *
HTR = 10.0
SOG = 14

Figure 31. HYGAS REACTOR ENGINEERING DATA FOR TEST 58 FOR THE STEADY PERIOD FROM 11/20/76 (2200 Hours) TO 11/21/76 (0300 Hours)



⬡ Stream No.
○ Pressure, psig
▭ Temperature, °F

Material Balance, %
C
H
O
Ash

	Gas Streams	
	6	10
mol % (dry)		
H_2	32.30	29.64
CO_2	31.90	35.86
C_2H_6	0.98	1.11
Ar	--	--
N_2	7.67	7.32
H_2S	0.10	0.07
CH_4	20.09	19.64
CO	6.96	6.36
mol/hr (dry)	201	227

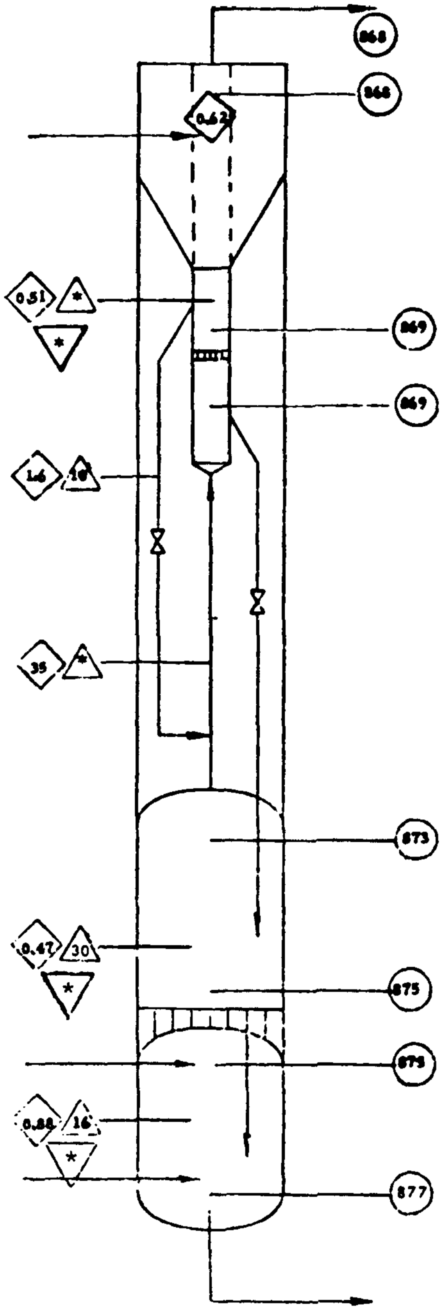
	Solids Streams		
	1	10	4
wt % (dry)			
C	67.44	71.54	66.47
H	4.36	2.36	1.18
N	0.92	0.65	0.29
Cl	--	--	--
S	0.86	0.68	0.17
Ash	9.67	20.26	28.69
O	16.75	4.51	3.20
lb/hr (dry)	4185		
Moisture, wt %	11		
Slurry Concn, wt %	33		

	Liquid Streams		
	7	8	1
	lb/hr		
H_2O	--	--	--
Light Oil	--	--	9397

Gas Feed Streams			
2	3	5	9
mol/hr			
290	91	27	0

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 32. HYGAS REACTOR DATA FOR TEST 58 FOR THE STEADY PERIOD FROM 11/21/76 (0300 Hours) TO 11/21/76 (1800 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 = 0.21
lb Steam/lb Coal Fed = 1.64
lb Hydrogen/lb Coal Fed = 0
lb Coal Fed/1000 SCF Product Gas = 85

By Ash Balance

Coal Gasified, % = 73
Carbon Gasified, % = 67
Methane Yield, SCF/lb coal fed = 4.1
Carbon Oxide Yield, SCF/lb coal fed = 8.8

Bed Height (ft)
Slurry Dryer = *
HTR = 12
SOG = 18

Figure 33. HYGAS REACTOR ENGINEERING DATA FOR TEST 58 FOR THE STEADY PERIOD FROM 11/21/76 (0300 Hours) TO 11/21/76 (1800 Hours)

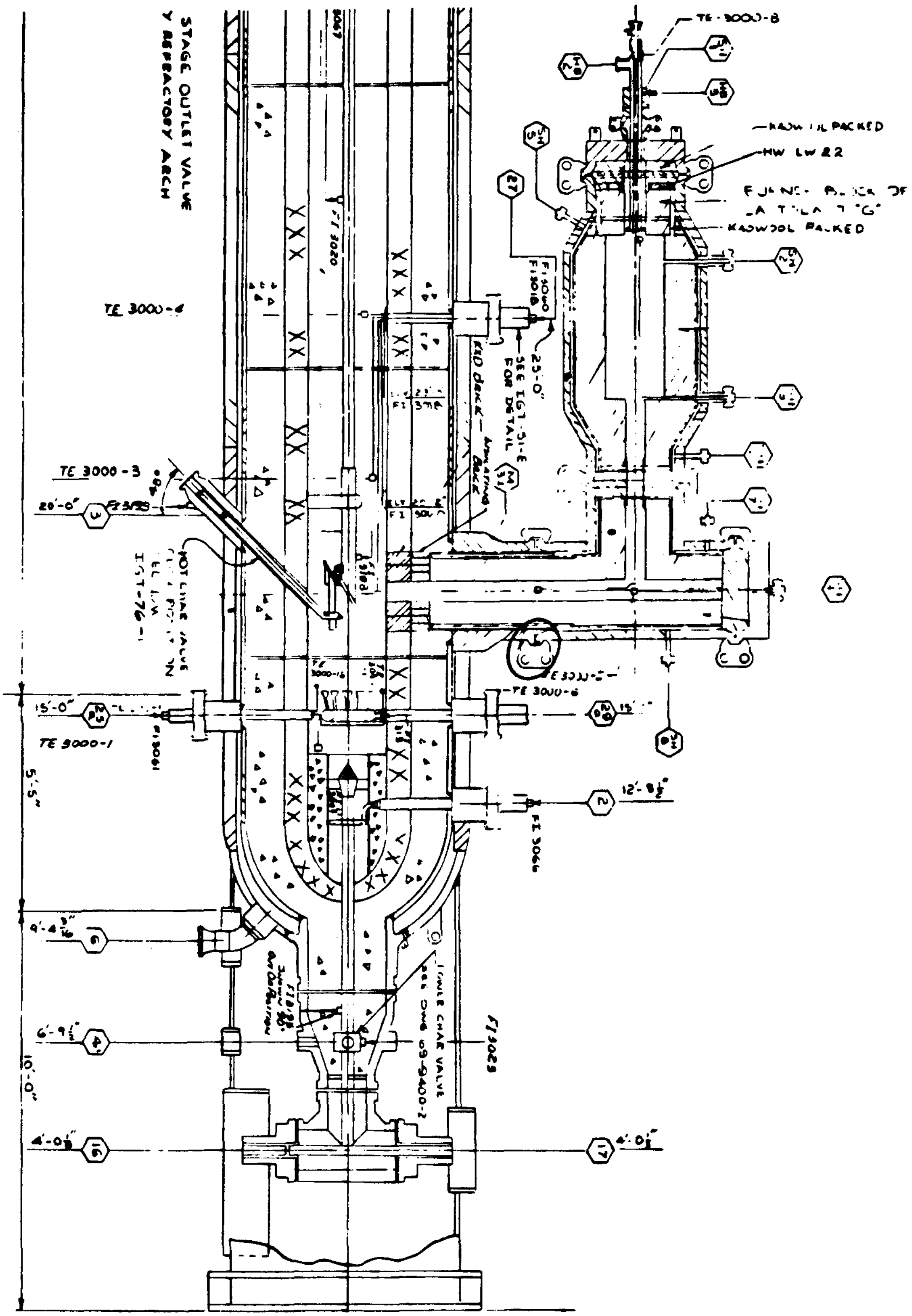


Figure 34. LOCATION OF LEAK AREA ON START-UP HEATER GRAYLOC FLANGE

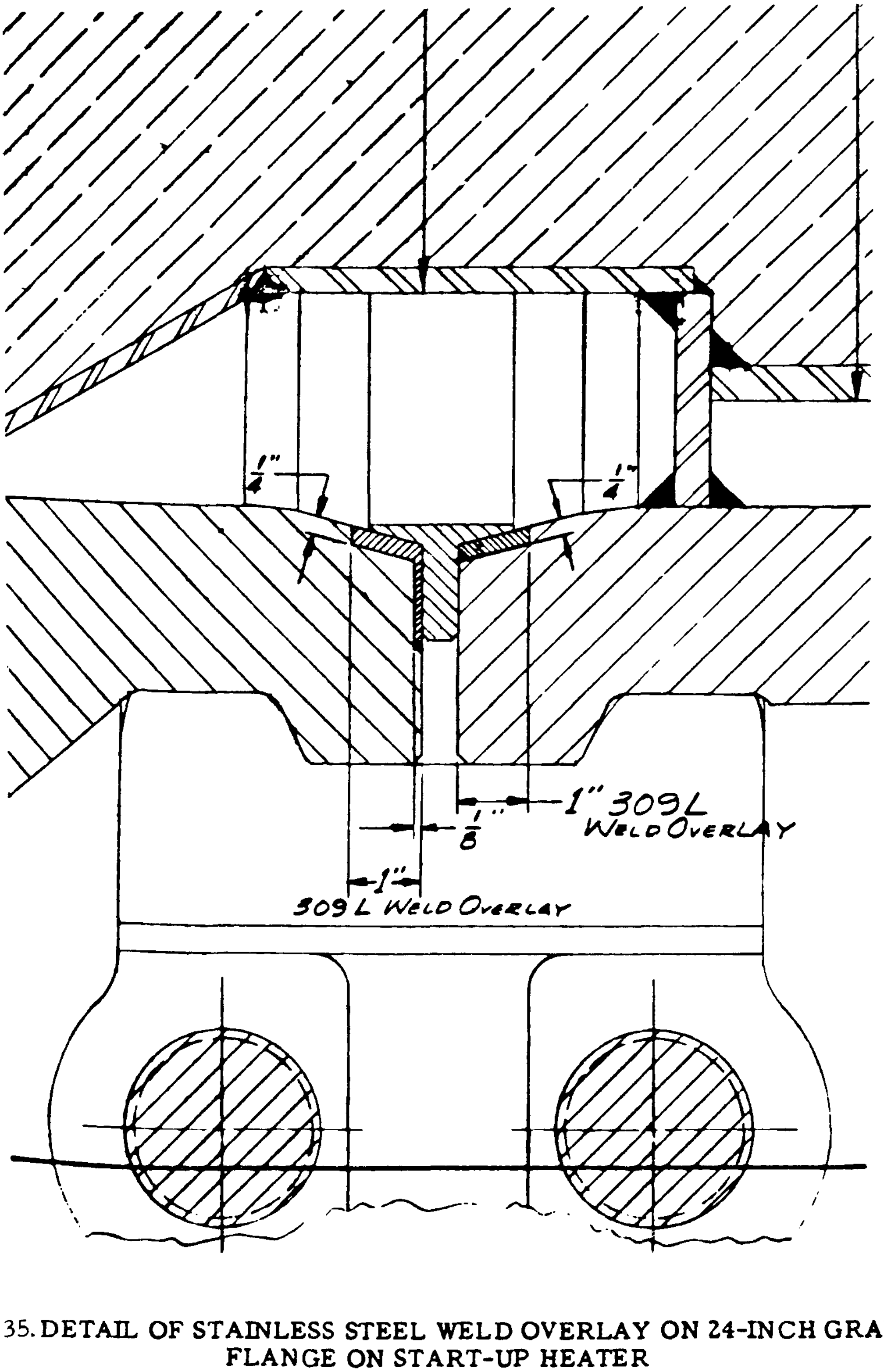


Figure 35. DETAIL OF STAINLESS STEEL WELD OVERLAY ON 24-INCH GRAYLOC FLANGE ON START-UP HEATER subbituminous coal had a moisture content of 12% after the crushing and drying operation. This 12% residual moisture created water vapor that condensed during exposure to low ambient temperatures and caused wet coal to plug the screen and/or the oversized return chute from the screen. During Test 58, the plant was kept in operation by mechanically cleaning this section. After the test, a new cover on the Sweco screener and heat tracing on the oversized return chute and the oversized screw conveyor were installed to improve its operation.

Investigation of Foaming in the Purification Section

Foaming in the purification system was controlled with antifoam agents and during the latter portions of Test 58, the purification system operated satisfactorily. Considerable time was spent during December 1976 in laboratory tests to determine the causes of the foaming that occurred in the DGA absorber during the early stages of Test 58.

The laboratory tests indicated that the foaming agent is a high-boiling compound, a degradation product of the reaction of the DGA with trace elements in the process gas being passed through the absorber system. The laboratory test program indicated that the foaming agent could be removed from the DGA circulating solution by proper use of the high-temperature reclaimer section of the HYGAS pilot plant. The laboratory tests also pinpointed which antifoam agents would be effective in the HYGAS pilot plant if the reclaimer operations would not keep up with the rate of formation of these degradation products. In addition, an operating procedure was developed for batch reclaiming under run conditions for the purification section.

Batch reclaiming of DGA in the reclaimer was initiated. Samples of DGA were collected downstream of the reclaimer, and analysis showed that they were consistently nonfoaming.

Repairs to the Reactor Start-Up Heater

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, making the start-up heater ready for installation.

Improvements in the Pretreater Section

A major modification was made in the water spray inlet to the venturi scrubber to eliminate the plugging that had been observed in this area after extended runs. The quench water circulation piping in the two quench water circulation pumps in the pretreater quench section were modified to result in improved operation of the pretreater quench section and longer onstream reliability.

The instrumentation on the internal cyclones in the pretreater reactor was also improved by adding ΔP taps to both cyclone diplegs to determine the solids level in each dipleg and to determine whether the diplegs are operating satisfactorily during pretreatment reactor operations. This change was important in evaluating the efficiency of the cyclones and to determining whether the cyclones are allowing excessive dust losses from the pretreater reactor.

Selection of a Bituminous Coal

The ERDA/A.G.A. Operating Committee directed IGT to resume testing Illinois Basin bituminous coal, and IGT selected two candidate coals from the State of Illinois. These coals were selected from areas where sufficient reserves exist to support a full-scale demonstration or commercial gasification facility. The coals were from the Consolidation Coal Company Hillsboro Mine (a run-of-mine coal) and from the Peabody Coal Company No. 10 Mine (a washed coal). Evaluation of these two coals included crushing tests, fluidization tests, proximate and ultimate analyses, ash fusion temperature determinations, and batch pretreatment tests.

Table 8 presents a summary of analytical tests (from several different sources) for the Peabody washed coal and Table 9 presents similar information for the Consolidation run-of-mine coal. The principal differences are in the ash and sulfur contents; the washed product from Peabody Coal Company has a much lower ash content than the run-of-mine coal. The sulfur content of the washed product is also slightly lower than for the run-of-mine coal. Ash fusion temperatures (useful in evaluating the tendency of a particular coal type to form clinkers) are about the same for both coals.

Table 8. COMPARATIVE ANALYSIS OF ILLINOIS No. 6 BITUMINOUS COAL FROM No. 10 MINE, PEABODY COAL COMPANY (Washed)

Sample	IGT No. 36388	IGT No. 36137	Commercial Testing 58-1076	Peabody Coal Company Average Analysis
Proximate Analysis, wt %				
Moisture	4.7	3.0	3.78	--
Volatile Matter	37.2	39.5	37.23	--
Ash	11.5	11.0	12.14	--
Fixed Carbon	46.6	46.5	46.85	--
TOTAL	100.0	100.0	100.00	--
Ultimate Analysis (Dry), wt %				
Ash	12.06	11.38	12.62	10.75
Carbon (Total)	68.20	69.00	66.62	69.00
Hydrogen	4.81	4.89	4.81	5.21
Sulfur	4.43	4.26	4.05	4.50
Nitrogen	1.20	1.23	1.24	1.02
Oxygen (Difference)	9.30	9.24	10.66	9.52
TOTAL	100.00	100.00	100.00	100.00
Heating Valve, Btu/lb	12,303	--	12,171	--
Free Swelling Index	--	--	3.5	4.5
Ash Fusion Temperature, Reducing Atmosphere, °F	--	1990	2040	2010
H = W	--	2080	2105	2050
H = 1/2W	--	2100	2155	2090
Fluid	--	--	2215	2150

A77020365

Table 9. COMPARATIVE ANALYSIS OF ILLINOIS No. 6 BITUMINOUS COAL FROM HILLSBORO MINE, CONSOLIDATION COAL COMPANY (Run-of-Mine)

Sample	IGT No. 36242	Commercial Testing No. 58-1073	Consolidated Coal Company (Average Analysis)
Proximate Analysis, wt %			
Moisture	1.2	12.02	--
Volatile Matter	33.0	30.18	--
Ash	26.5	22.83	--
Fixed Carbon	39.3	34.97	--
TOTAL	100.0	100.00	--
Ultimate Analysis, (dry), wt %			
Ash	26.77	25.95	21.39
Carbon (total)	55.50	57.07	61.05
Hydrogen	4.02	4.01	4.29
Sulfur	5.21	5.06	4.30
Nitrogen	0.94	0.98	1.15
Oxygen (Difference)	7.56	6.93	7.82
TOTAL	100.00	100.00	100.00
Heating Value, Btu/lb	--	10198	
Free Swelling Index	--	2	
Ash Fusion Temperature, reducing atmosphere, °F			
Initial Deformation	2040	2045	--
H = W	2090	2125	--
H = 1/2W	2140	2205	--
Fluid	--	2285	--

A77020366

Twenty-five ton shipments of these coals were obtained from the mines and processed through the HYGAS pilot plant crushing and drying system. Both coals crushed easily without any apparent problems in the roller mill operation or the drying and screening operation downstream of the roller mill. The size distributions were similar to those of other bituminous coals that had been processed in the HYGAS pilot plant.

Table 10 gives data on complete fluidization velocities (V_{cf}) for the two coals. V_{cf} is the velocity at which complete backmixing and good agitation exist throughout the dense-phase fluidized bed including the wall area. The V_{cf} is usually several times greater than the minimum fluidization velocity (the velocity at which the characteristic ΔP vs velocity curve shows its first break point). Complete fluidization (V_{cf}) is important in a fluidized-bed contacting processes such as the HYGAS process to ensure good backmixing throughout the bed and adequate particle motion throughout the entire dense phase contacting region. Materials that have a low V_{cf} tend to be completely backmixed and agitate violently throughout the bed at lower velocities than materials that have a high V_{cf}. Therefore, it is desirable for pretreatment operations and for the HYGAS reactor itself to operate at superficial velocities well into the V_{cf} range.

The Hillsboro coal has an experimental V_{cf} of 1.28 and 0.61 ft/sec in the size range of interest (–14 and –20 mesh). Corresponding values for the Peabody coal are 0.78 and 0.41 ft/sec. This very striking difference probably can be attributed to the presence of dense, high-ash particles in the run-of-mine coal after crushing. These dense particles would cause higher V_{cf} velocities than those of coals with a lower particle density.

Batch-scale pretreatement tests were conducted with both coals to give an indication of the degree of severity of pretreatment required to produce a nonagglomerating char feed for the HYGAS reactor. Table 11 indicates the results of these pretreatment tests in which the variables are the temperature of the pretreater reactor, and the oxygen-to-coal ratio in the contacting, fluidizing gases. Residence times were 30 minutes for all tests and the superficial velocity was adjusted to 0.875 ft/sec for all tests. Feed material for both types of coals was the –14 mesh fraction crushed in the HYGAS coal preparation section.

Table 10. COMPLETE FLUIDIZATION VELOCITIES (V_{cf}) FOR TWO ILLINOIS BASIN BITUMINOUS COALS

	Hillsboro Mine		Peabody No. 10 Mine	
Size Fraction	Experimental V_{cf}*	Calculated V_{cf}†	Experimental V_{cf}*	Calculated V_{cf}†
	ft/s			
-8 mesh	2.2	2.28	--	--
-14 mesh	1.28	1.04	0.78	0.55
-20 mesh	0.61	0.41	0.41	0.27

* Experimental values determined in air at 14.7 psia and 70°F.

† Calculated for conditions in the HYGAS pilot unit pretreater reactor of 800°F and 20 psia.

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Table 11. RESULTS OF BATCH PRETREATMENT TESTS WITH ILLINOIS No. 6 BITUMINOUS COAL

Run No.	Hillsboro Coal			Run No.	Peabody Coal		
	Temp, °F	SCF O_2/ lb Cost	Remarks†		Temp, °F	SCF O_2/ lb Cost	Remarks†
HPR7	650	2.0	4	HPR16	650	2.0	4
HPR3	700	1.0	3	HPR11	700	1.0	0
HPR6	700	1.5	0	HPR14	700	1.5	2
HPR4	700	2.0	1	HPR12B	700	2.0	2
—	--	--	--	HPR12	--	--	--
HPR1	750	1.0	1	HPR9	750	1.0	0
HPR2	750	2.0	0	HPR10	750	2.0	0
—	--	--	--	HPR9B	--	--	--
HPR8	750	0	1	HPR15	750	0	2
HPR5	800	0.5	0	HPR13	800	0.5	0

* Residence time = 30 minutes; 1-inch ID reactor size; superficial velocity of 0.875 ft/s; and -14 mesh feed.

† Legend:

0 = no agglomeration
1 = < 25% agglomeration
2 = > 25%, < 50% agglomeration
3 = > 50%, < 75% agglomeration
4 = > 75% agglomeration.

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The degree of agglomeration of the samples, determined by the IGT boat test, is indicated under the column titled "Remarks" in Table 11. The results of the agglomeration test are quantified by a number system with zero assigned to the product that has no agglomerates formed. Successive increments of 25 volume percent of agglomerates formed are numbered through 4, in which the agglomeration is greater than 75% by volume of the product char. The most desirable situation is to have free flowing chars with zero rating, that is, no agglomerate formation when subjected to the IGT boat test. It is probable that chars with a value of 1 would be acceptable for feed material to the HYGAS reactor, but it is doubtful that chars with ratings of 2 or 3 would be acceptable.

The data presented in Table 10 indicate that temperatures of $750^{\circ}F$ and above and oxygen-to-coal ratios from 0.5 to 2 SCF/lb of coal would result in the production of chars with acceptable agglomeration properties to serve as feed to the HYGAS reactor. It is apparent that temperatures below $750^{\circ}F$ could lead to borderline cases with partially agglomerating chars; however, oxygen-to-feed ratios of less than 0.5 are not acceptable. These screening tests served to set the conditions for the first phase of the pretreater operations with the new coal type.

The results of these screening tests indicated that the Peabody coal has a lower ash content and a somewhat lower sulfur content and requires a considerably lower velocity for complete fluidization; therefore, it was the preferred coal for initial tests in the HYGAS pilot plant to study the effects of carbon conversion, pretreatment severity, and fines recycle, as directed by the Operating Committee of ERDA/A.G.A.

In early January, IGT ordered 1500 tons of the Peabody washed coal. 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. 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 in Table 12. As a comparison, the raw coal analysis is also listed. 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 at 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 selected at –14 mesh.

Test 59, February 1977

Highlights

Test 59 was the first test on Peabody No. 10 mine bituminous coal. A total of 52 tons of pretreated coal was fed to the reactor and a smooth coal feed rate of 2.3 ton/hr was achieved before leaks forced termination of the test.

Details

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 fluctuating pretreater product-gas flow rates indicated a leak in the pretreater internal cooling coil system; a subsequent pressure test on the cooling coil confirmed this. 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 (over several 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.

Table 12. PEABODY No. 10 MINE BITUMINOUS COAL ANALYSES

Sample	Washed Raw Coal	Crushed Coal
Proximate Analysis	wt %	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.

Concurrent with the pretreatment operation, the reactor was readied for Test 59. 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 coal preparation section was started up on March 2 on Peabody No. 10 mine coal. Operations were conducted at around $775^{\circ}F$, 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 nonagglomerating 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 to correct this problem.

The reactor was lit off on March 8, 1977, 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 ton/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 ton/hr.

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 during the test.

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 char was fed to the reactor.

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 small 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 indicated 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 very thin film of powdered coal and tar-like material. Similar material was found in the fresh-coal feed line to the vertical lift reactor.

The lift line, and the line connecting the first-stage and the second-stage reactors, 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.

Figures 36 through 44 present the final engineering information for Test 59 for both the pretreater and the reactor.

Plant Modifications

After Test 59, several improvements were made to the HYGAS pilot plant. These included modifying the pretreater feed lockhopper by enlarging the gas vent line on the lower lockhopper from 1 to 3 inches and by reinstalling the lower level probe to eliminate interference from packed coal around the dead space near the base of the probe.

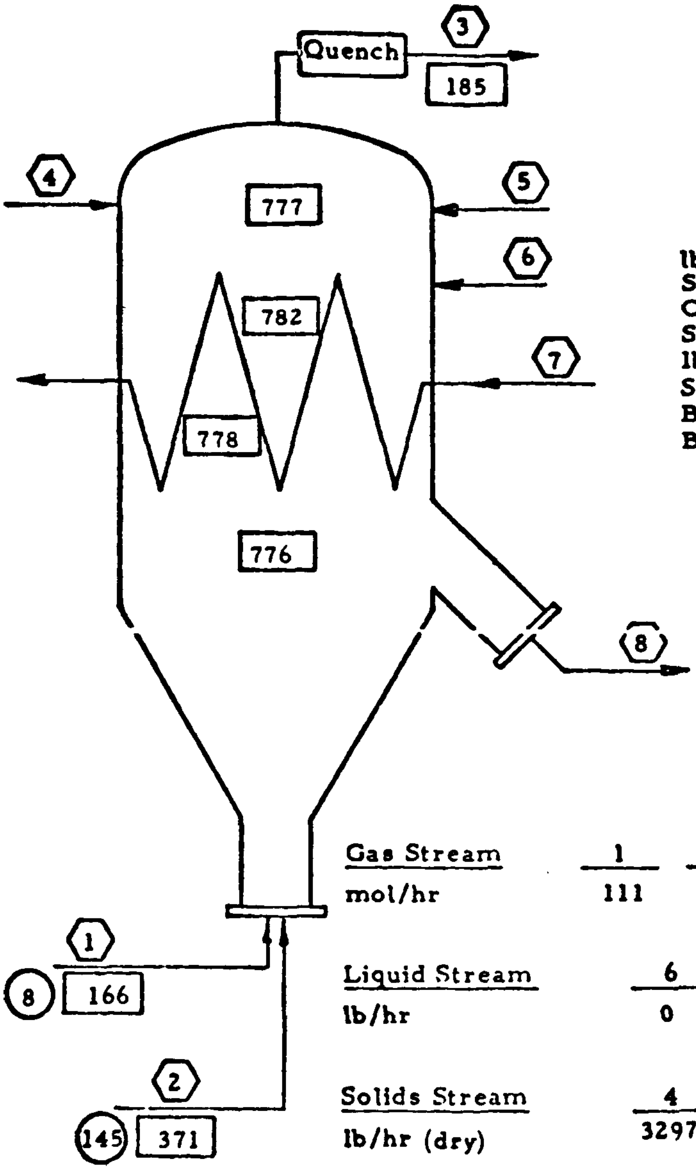
Tests were run on the coal mill, varying solids residence time and lift velocity to determine optimum settings for a satisfactory size consist.

Experiments were conducted which indicated that the char-cooler fluidizing velocity should be increased to 0.45 ft/s to improve heat transfer in the bed and prevent clinker formation.

Test 60, April 1977

Highlights

A total of 186 hours of self-sustained operation was achieved during Test 60 before a leak forced termination of the test. This was the first



⬡ Stream No.

○ Pressure, psig

▭ Temperature

lb Char/lb Coal	--
SCF O_2/lb Coal	2.68
Oxygen Breakthrough, % fed	--
Superficial Velocity, ft/s	0.95
lb O_2 Reacted/lb Coal	--
Solids Residence Time, hr	1.7
Bed Density, lb/cu ft	18
Bed Height, ft	8

Gas Stream	3
Components, mol %	
H_2	
CO_2	
C_2H_6	
O_2/Ar	
N_2	
SO_2 •	
CH_4	
CO	
H_2O	
mol/hr	
mol. wt	

Gas Stream	1	2	5
mol/hr	111	114	38

Liquid Stream	6	7
lb/hr	0	223

Solids Stream	4	8
lb/hr (dry)	3297	--

1 Air to Pretreater

2 Steam to Pretreater

3 Pretreater Off-Gas After Quench

4 Raw Coal to Pretreater

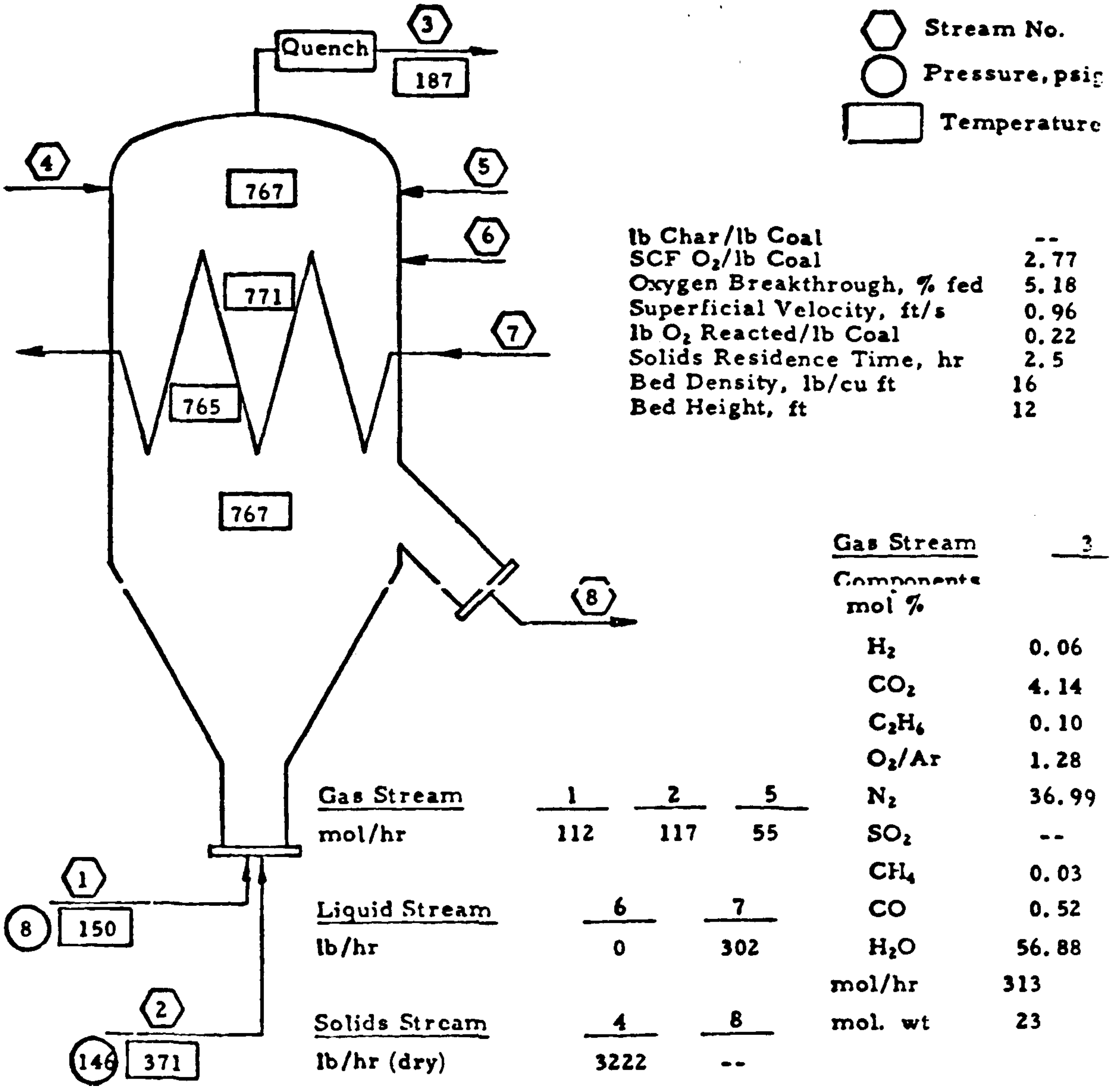
5 Gas From Char Cooler

6 Direct Water Quench

7 Cooling Water to Cooling Coil in Pretreater

8 Pretreated Char to Char Cooler

Figure 36. PRETREATMENT DATA FOR TEST 59 FOR STEADY PERIOD FROM 3/12/77 (1400 Hours) TO 3/12/77 (1700 Hours)



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	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

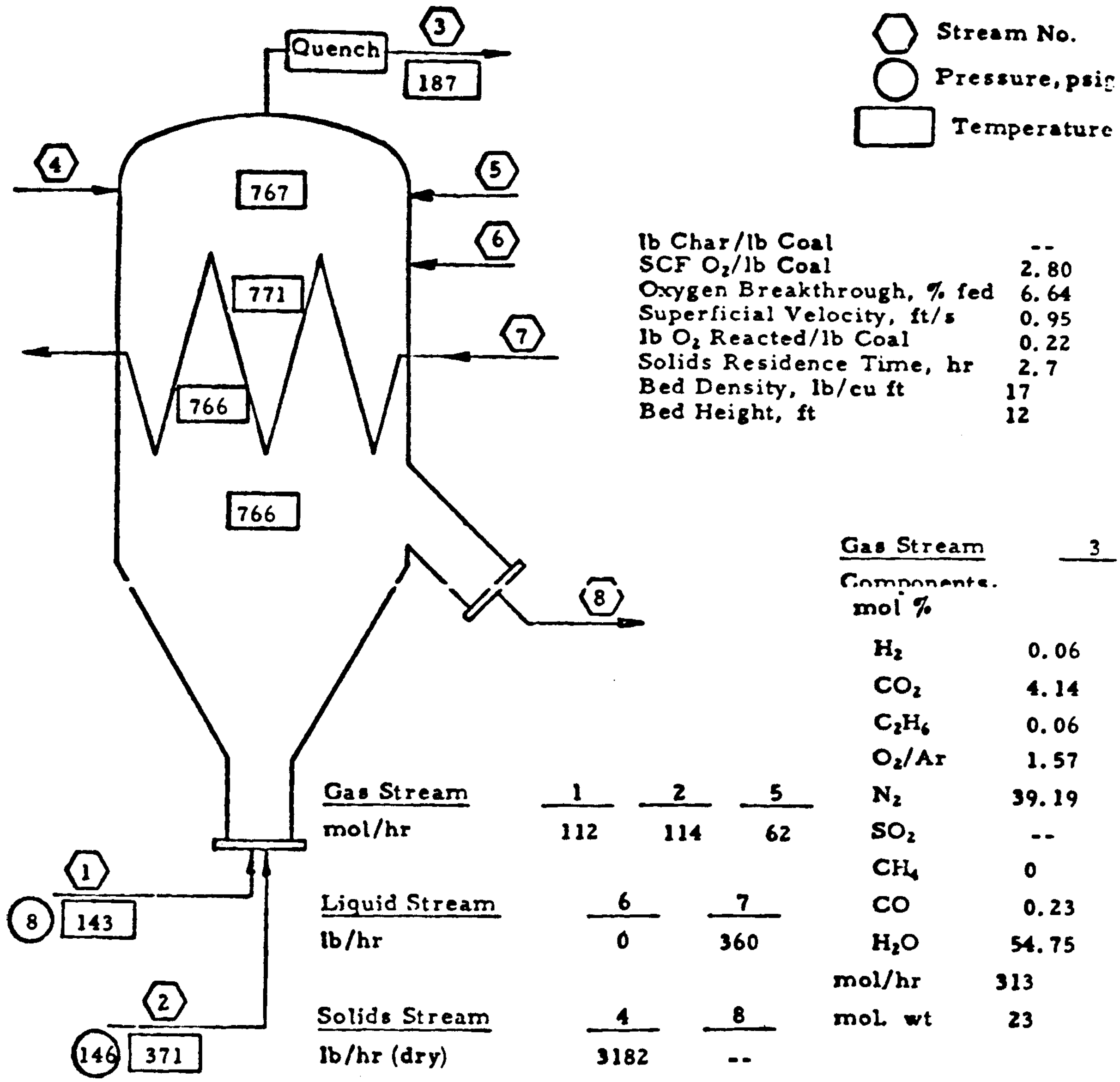
Gas Stream	1	2	5
mol/hr	112	117	55

Liquid Stream	6	7
lb/hr	0	302

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

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

Figure 37. PRETREATMENT DATA FOR TEST 59 FOR STEADY PERIOD FROM 3/12/77 (1800 Hours) TO 3/13/77 (0100 Hours)



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	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

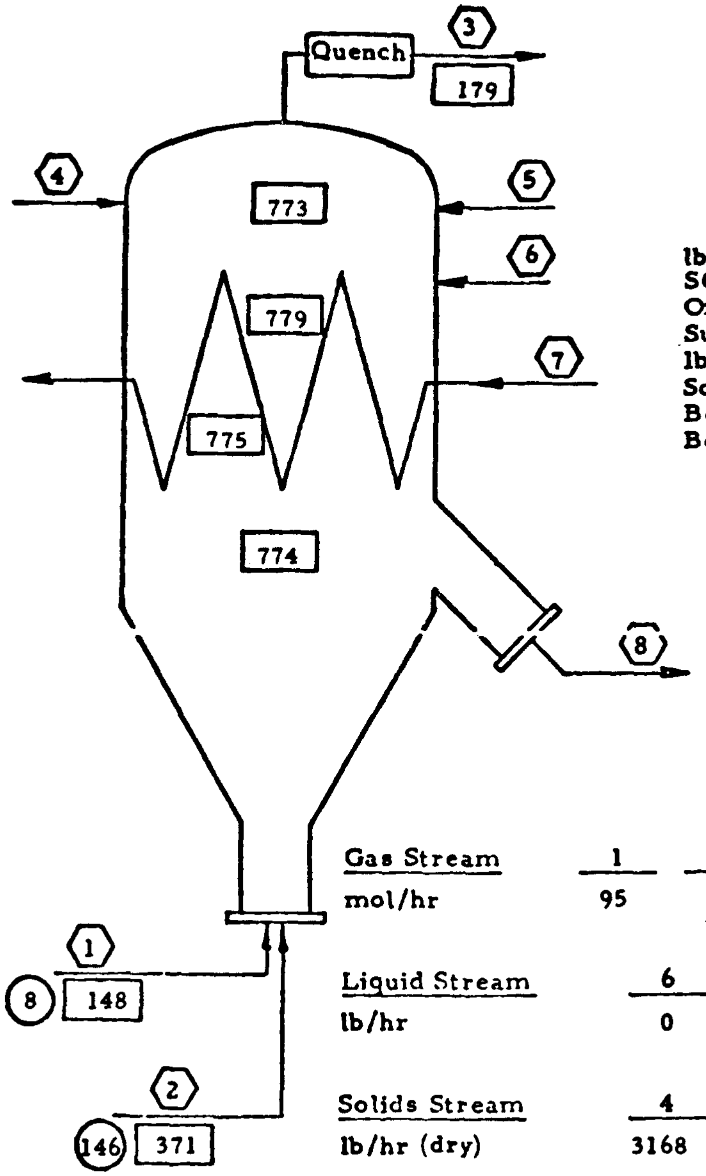
Gas Stream	1	2	5
mol/hr	112	114	62

Liquid Stream	6	7
lb/hr	0	360

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

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



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

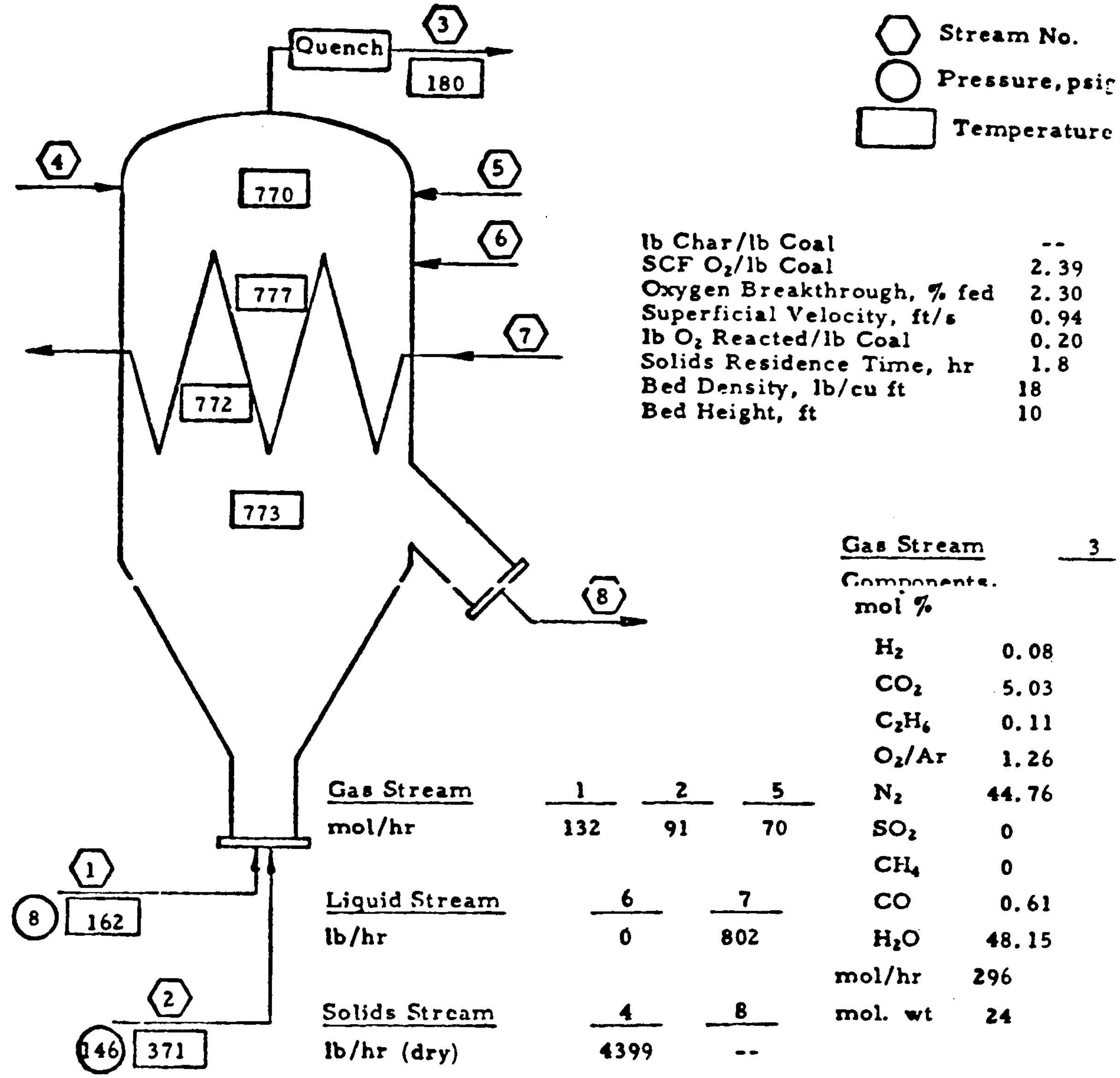
Liquid Stream	6	7
lb/hr	0	145

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



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

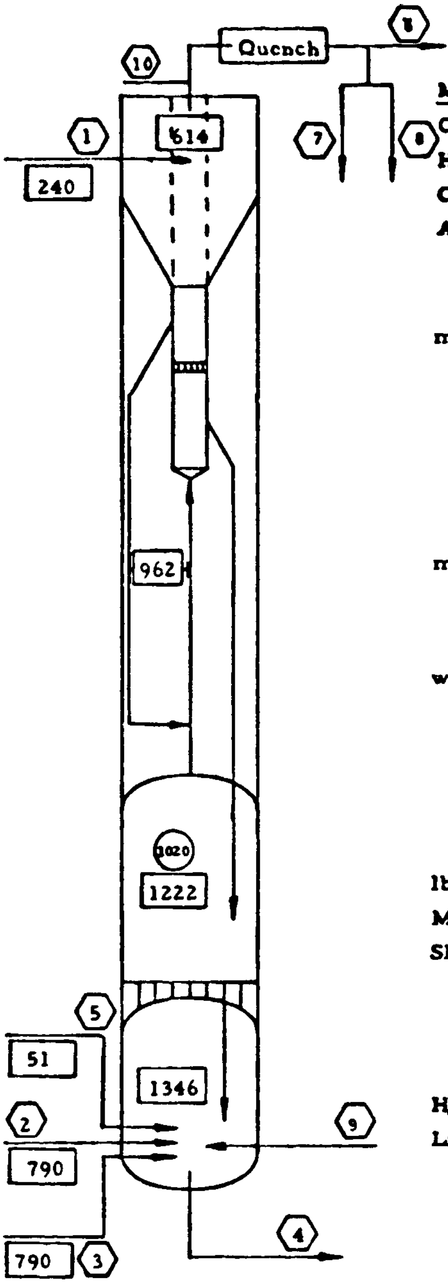
Liquid Stream	6	7
lb/hr	0	802

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



⬡ Stream No.
○ Pressure, psig
☐ Temperature, °F

Material Balance, %
C
H
O
Ash

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

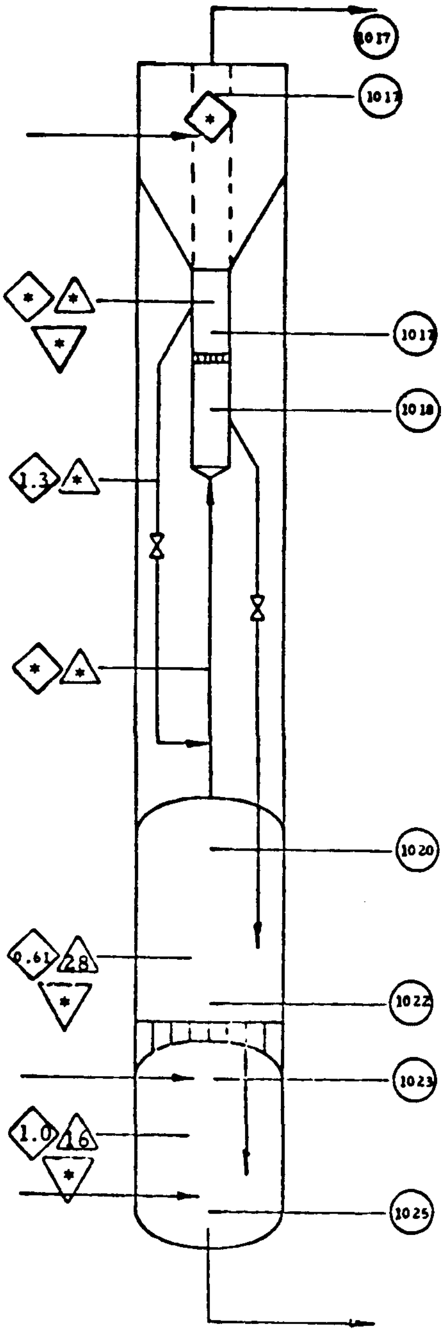
	Solids Streams		
	1	10	4
wt % (dry)			
C		78.30	73.10
H		3.12	2.22
N		1.58	1.12
Cl		--	--
S		3.08	1.56
Ash		9.56	20.40
O		4.36	1.60
lb/hr (dry)			
Moisture, wt %			
Slurry Concn, wt %			

	Liquid Streams		
	7	8	1
	lb/hr		
H_2O	--	--	--
Light Oil	--	--	8580

Gas Feed Streams			
2	3	5	9
mol/hr			
298	101	8	H_2 = 153 O_2 = 11 Steam = 57

1	Feed Slurry	6	Gasifier Effluent After Quench
2	High-Pressure Steam to Steam-Oxygen Sparger	7	Water Made
3	High-Pressure Steam to Stripping Ring	8	Light Oil to Recycle
4	Spent Char	9	Gases From External Heater
5	High-Pressure Oxygen to Steam-Oxygen Sparger	10	Reactor Overhead

Figure 41. HYGAS REACTOR DATA FOR TEST 59 FOR STEADY PERIOD FROM 3/12/77 (2200 Hours) TO 3/13/77 (0130 Hours)



○ Pressure, psig

△ Density, lb/cu ft

◇ Velocity, ft/s

▽ Mean Residence Time, min

* Not Available

<u>Product Gas</u> – dry, nitrogen- and acid-gas-free basis
<u>Coal Fed</u> – 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 = *

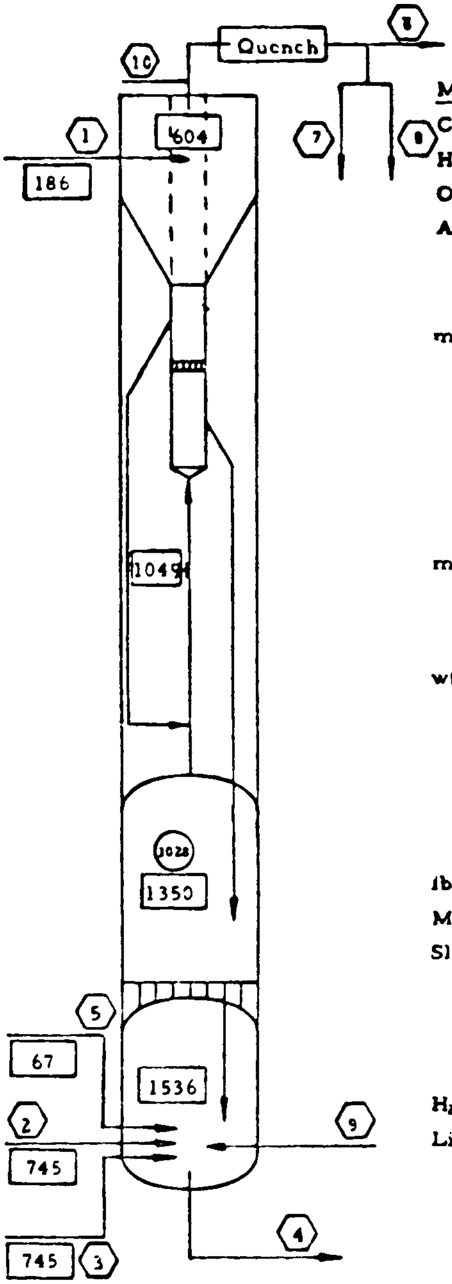
<u>By Ash Balance</u>

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

<u>Bed Height</u> (ft)

Slurry Dryer = *
HTR = 12
SOG = 20

Figure 42. HYGAS REACTOR ENGINEERING DATA FOR TEST 59 FOR STEADY PERIOD FROM 3/12/77 (2200 Hours) TO 3/13/77 (0130 Hours)



⬡ Stream No.
○ Pressure, psig
▭ Temperature, °F

Material Balance, %

C
H
O
Ash

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

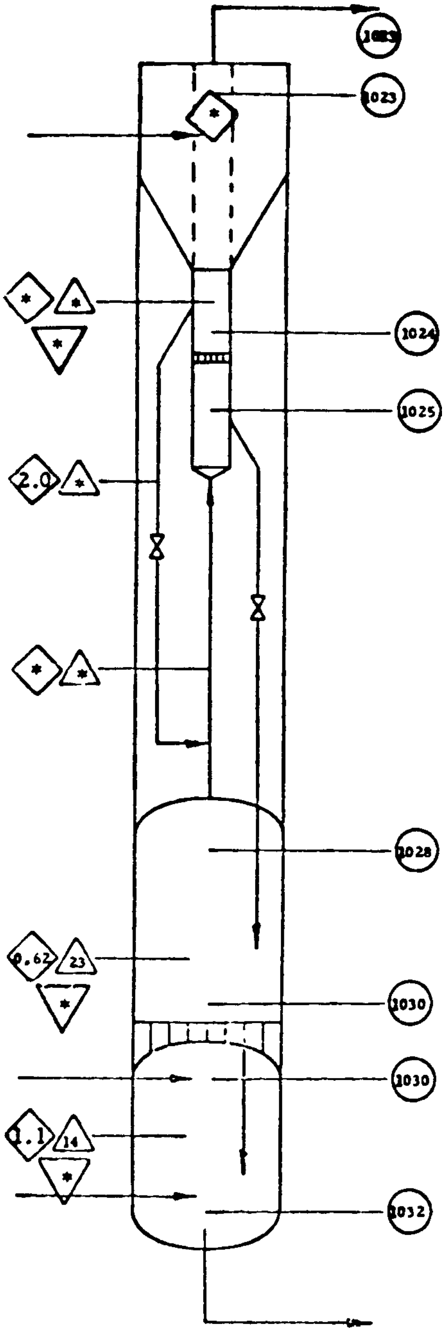
	Solids Streams		
	1	10	4
wt % (dry)			
C		78.80	73.50
H		2.82	1.27
N		1.33	0.54
Cl		--	--
S		2.77	0.84
Ash		9.98	22.25
O		4.30	1.60
lb/hr (dry)			
Moisture, wt %			
Slurry Concn, wt %			

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



○ **Pressure, psig**

△ **Density, lb/cu ft**

◇ **Velocity, ft/s**

▽ **Mean Residence Time, min**

* **Not Available**

<u>Product Gas</u> – dry, nitrogen- and acid-gas-free basis
<u>Coal Fed</u> – dry basis

lb Oxygen/lb Coal Fed = 0.13
lb Steam/lb Coal Fed = 1.76
lb Hydrogen/lb Coal Fed = 0.06
lb Coal Fed/1000 SCF Product Gas = *

<u>By Ash Balance</u>

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

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

<u>Bed Height</u> (ft)

Slurry Dryer = *

HTR = 14

SOG = 21

Figure 44. HYGAS REACTOR ENGINEERING DATA FOR TEST 59 FOR STEADY PERIOD FROM 3/14/77 (1430 Hours) TO 3/14/77 (1700 Hours)

run with this coal type with short, self-sustained, steady-state periods which could be selected for analysis. Also, after the test was ended, additional data was obtained by operating the pretreater at lower temperatures and assessing its performance at those conditions.

Details

Pretreater operation began at 2025 hours on March 29. Operation was interrupted briefly when the motor on the pretreater quench-water pump burned out. Pretreater operation was resumed after the motor was replaced; it began providing nonagglomerating coal to the reactor at 1530 hours on March 30. The pretreater operated satisfactorily during Test 60. The modifications made before the test alleviated most of the coal-feeding problems of Test 59. Coal feed rates to the pretreater ranged from 2.5 to 3 ton/hr, and operating conditions were set at $775^{\circ}F$, with oxygen-to-coal ratios of 2 to 2-1/2 SCF of oxygen per pound of coal.

The heat-up cycle was begun on March 28 at 0620 hours. Slurry feed to the reactor was initiated at 1930 hours on March 30. Smooth solids flow was established on March 31 at 2000 hours, and the external start-up heater was shut off. Coal feed had to be taken out of the gasifier at 0130 hours on April 2 when solids flow from the second-stage gasifier to the steam-oxygen gasifier was stopped. A continuous effort was made to establish solids flow. By late evening on April 2, solids transfer was reestablished, and the solids feed rate ranged from 1 to 2 ton/hr. Mechanical problems with several pumps necessitated stopping the coal feed on April 5. The low-pressure slurry pumps supplying slurry to the high-pressure pumps, the high-pressure water pump supplying quench water to the cyclone pot and the spent char pot, the light-oil recirculating pump for oil to the slurry mixing tank, the high-pressure amine pump, the caustic injection pump for caustic wash, and the spent-char slurry recirculating pump all had mechanical problems. These pumps were repaired and coal feed to the gasifier was resumed at 1800 hours on the same day.

The gas purification section was put on-stream with the gasifier and it removed CO_2 and H_2S satisfactorily. Product gas from the HYGAS reactor system was introduced to the liquid-phase methanation pilot unit at 1930 hours on April 4. Conversion was achieved for 2 hours. At 2130 hours, a process upset in the HYGAS plant allowed the diglycolamine solution to overflow through the absorber system and enter the liquid-phase methanation pilot unit together with the product-gas stream. This terminated test operations on the liquid-phase methanation pilot unit.

Test 60 was terminated at 1300 hours on April 8 when attempts to fix the leak at a Grayloc coupling in the char carrier stream line downstream of the reactor failed. Test 60 had been self-sustained since 1900 hours on March 31 for a total of 186 hours. Two hundred and seventy-eight tons of raw bituminous coal were fed to the pretreater over 102 hours. A total of 165 tons of char was fed to the gasifier.

After Test 60 was ended, the pretreater was operated at 700° and 750°F with coal feed rates of about 2 ton/hr, to find out the pretreater performance at these conditions. Table 13 summarizes the results of the tests, which showed that nonagglomerating feed could be produced at pretreater temperatures of 750°F and higher. (The pretreater temperature for Test 61 was, therefore, set between 750° and 775°F.)

The pretreatment section was inspected after Test 60 and a clinker was found on the north wall of the pretreater. The clinker was a typical hard, red, high-ash-content material. It extended 8 to 10 inches from the wall and was 10 to 12 inches high.

When the char-cooler vessel was opened up, a clinker formation was found covering the entire fluidizing ring. The highest position of the formation was on the north wall. This clinker was also mostly hard, red ash. The clinkers found in both the pretreater and the char cooler could have been caused by the oversized material that escaped the ripped oversize (14 mesh) screen on the Sweco screener. The coal-feed screen analysis received after the test showed that the screen was ripped during the morning of April 1. Vibration problems could have caused the failure of the screen during Test 60 as one of the three springs supporting the screener motor was found to be broken. Following its repair after Test 60, the Sweco screener was tested to see if the screen would rip from normal vibration. It did not.

Table 13. COMPARATIVE PRETREATMENT TEST RESULTS

	Test 59[d]	Test 60[e]	Special Tests[f]	
			Period A	Period B
Temperature, °F	773	762	700	750
Bed Height, ft	10.4	11.8	4.5	5.8
Bed Density, lb/cu ft	18.1	17.2	28.6	22.4
Air, pph	3828	3265	3500	3150
Coal Feed, tons/hr	2.36	2.0	2.11	2.11
Mean Residence Time, min	109	133	78	79
IGT Boat Test Results[a]	0	0	1	1
	0	0	4	1
	0	0	3	0
	0	0	4	1
Volatile Matter, wt %	22.8	21.6	30.0	23.7
SCF O_2/lb Coal[b] (Dry)	2.4	2.3	2.4[b]	2.2[b]
Degree of Pretreatment,[c] %	24	19	14	13

[a] IGT Boat Test: 0 = free-flowing
1 = < 25% agglomerating
2 = > 25%, < 50% agglomerating
3 = > 50%, < 75% agglomerating
4 = > 75%, < 100% agglomerating.

[b] 6% moisture assumed.

[c] Averaged ash, feed coal assumed to have 10.66% ash.

[d] Period from 1530 to 1830 hours (3/14/77).

[e] Period from 0030 to 0830 hours (4/1/77).

[f] Conducted at termination of Test 60.

The pretreater venturi scrubber and quench tower were inspected and a normal buildup of tar and coal fines were found on the wall of the scrubber and a normal accumulation was found at the bottom of the quench tower. The pretreater section was cleaned up and readied for Test 61.

A post-run inspection of the reactor revealed that it was in excellent shape. The slurry dryer was clean except for a small amount of coal dust. The solid transfer lines were all clear, and the second-stage reactor was clean. Pieces of refractory bricks, which had fallen from Manway 3's insulating brick layer, were found in the grid area. The steam-oxygen gasifier was clean, and the steam-oxygen sparger and the stripping-steam ring were both in good condition. The reactor was cleaned and readied for Test 61.

A significant amount of coal had accumulated in all three vessels of the quench system: the prequench tower, the quench tower, and the quench separator. This was expected after Test 60, because there were indications that the quench system had flooded during the test, on April 4, when the gas purification section also flooded. The sequence of events can be traced to overfilling the reactor slurry dryer area, which caused solids overflow into the cyclone section, resulting in a malfunction of the cyclone and eventually in solids buildup in the quench system. The inability to indicate true levels in the quench separator and prequench tower because of excessive solids in the vessel and resultant poor oil-water separation, allowed flooding of the quench tower. This precipitated filling and flooding of the absorber and, eventually, in the carry-over of diglycolamine in the purified gas to the liquid-phase methanation pilot unit.

All the quench section vessels were cleaned and the level control instrumentation on those vessels was cleaned and calibrated. The entire charge of diglycolamine solution in the purification section was decanted so that the absorber, the regenerator, and the reboiler could be cleaned thoroughly for the next test.

Routine maintenance was done on the utility area. A hot spot developed on the hydrogen plant reformer stack. The hydrogen plant was shut down and the reformer furnace burner assembly removed. Scaffold was installed inside the stack to reach the hot-spot area. A 2-foot by 2-foot section of refractory just below the stack dampener had spalled and fallen off. The refractory was replaced, and the hydrogen plant was readied for Test 61.

The results from Test 60 are presented in Figures 45 and 46 and Tables 14 and 15. Material and heat balances are completed for pretreater operation, together with some engineering data. During the process upset early in Test 60, the gas chromatograph monitoring the reactor product gases was rendered inoperable. Therefore, only partial engineering data on the HYGAS reactor were available from Test 60.

Test 61, May 1977

Highlights

During Test 61, a high coal conversion of over 90% was achieved for the first time with a bituminous coal. Gasifier operation was extremely smooth, and excellent cyclone operation helped give good oil-water separation and a clean quench system. Milder pretreatment conditions were used successfully, and the reactor was found to be absolutely clean after the test.

Details

Light-off for Test 61 occurred at 1630 hours on April 30. Coal feed was started to the pretreater at 2040 hours on May 2, and pretreated coal was fed to the gasifier beginning at 0800 hours on May 4. The gasifier operation became self-sustained at 0820 hours on May 5 after a smooth solids flow was established. Pretreated coal feed to the gasifier was interrupted at 1730 hours on the same day when the motor drive for the rotary feeder to the slurry mix tank burned out. The motor was replaced and pretreated coal feed was resumed at 1100 hours on May 6. During this standby period, the gasifier was maintained at temperatures between 850° and $900^{\circ}F$, using superheated steam, and was maintained at pressure by a low flow (15,000 SCF/hr) of hydrogen from the hydrogen plant. The test was restarted by introducing oxygen into the steam-oxygen gasifier after solids feed was resumed.

Test 61 continued until May 9, at 2300 hours, when a bad leak in the 40-inch closure of Manway 0 on the top of the reactor forced termination of the test. After detecting the leak in Manway 0, it was decided to raise the reactor temperature in the steam-oxygen gasifier to $1700^{\circ}F$ for a few

Table 14. MATERIAL BALANCE SUMMARY FOR PRETREATER SECTION FOR TEST 60 FROM 4/4/77 (1000 Hours) TO 4/4/77 (2100 Hours)

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

INPUT		C	H	O	N	S	Ar	Ash	Other	Total
Coal Feed	Wt % (Dry)	68.73	5.00	9.47	1.23	4.70	--	10.87		100
	Coal (Dry)	3242	236	447	58	221	--	513		4717
	Moisture		10	83						93
Streams to Pretreater	Air			963	3129		57			4149
	Steam		185	1479						1664
Nitrogen from purges					69					69
Air from purges				9	28		1			38
H_2O to venturi scrubber			1399	11,190						12,589
H_2O to quench tower			642	5133						5775
Air to char cooler				111	359		7			477
Cooling water to char cooler			142	1138						1280
TOTAL INPUT		3242	2614	20,553	3643	221	65	513		30,851
OUTPUT										
Pretreater* Char	Wt.% (Dry)	70.95	3.44	7.59	1.39	4.08	--	12.55		100
	Char (Dry)	2744	133	294	54	158	--	485		3868
	Moisture		10	81						91
Slurry Waste from Quench	Wt.% (Dry)	64.63	2.64	11.73	1.19	4.87	--	14.74		100
	Solids (Dry)	91	4	16	2	7	--	20		140
	Tars & Oils	99	10	8	1	4		0		122
	H_2O & Dis materials†	24	2082	16,643	2	40				18,791
Quench Tower Off-Gas	Total	556	317	3511	3440	--	63			7887
	Components: H_2		1							1
	CO_2	192		511						703
	C_2H_6	16	4							20
	N_2				3440					3440
	CH_4	26	8							34
	CO	322		430						752
	O_2			135						135
	Ar						63			63
	H_2O		304	2435						2739
TOTAL OUTPUT		3514	2556	20,553	3499	209	63	505		30,899
Net (Output - Input)		272	-58	0	-144	-12	-2	-8		48
% Balance (Output/Input)		108	98	100	96	95	97	98		100

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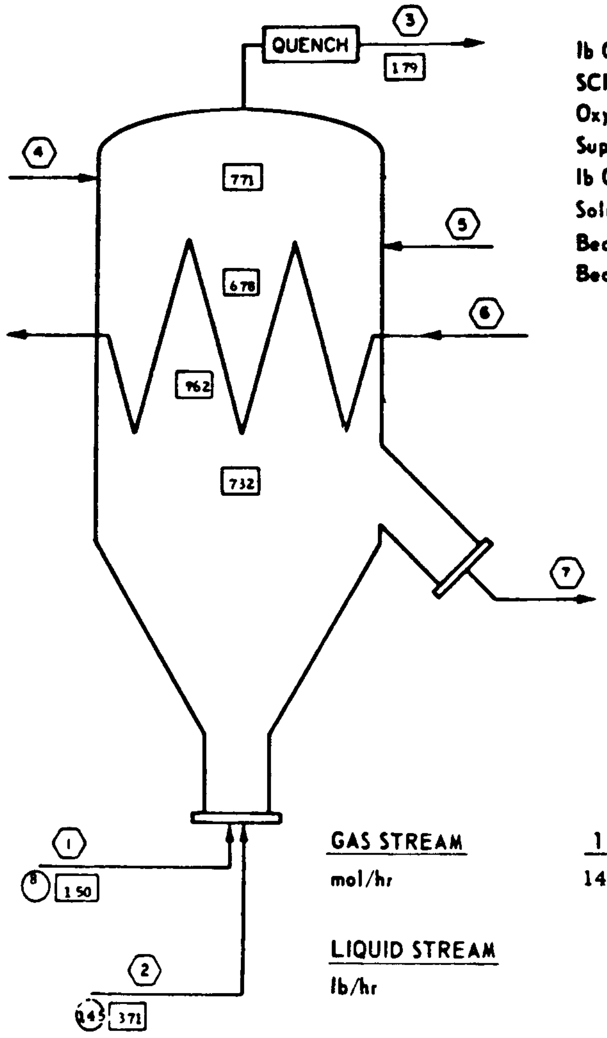
*Solids analyses taken from oil slurry samples.

† Water obtained by O_2 balance because flow measurement could not be made.



⬡ STREAM No.
○ PRESSURE, psig
☐ TEMPERATURE, °F

lb Char/lb Coal = 0.85
SCF O_2/lb Coal = 2.42
Oxygen Breakthrough, % fed = 2.49
Superficial Velocity, ft/s = 0.96
lb O_2 Reacted/lb Coal = 0.20
Solids Residence Time, hr = 1.9
Bed Density, lb/cu ft = 17
Bed Height, ft = 12

GAS STREAM	3
COMPONENTS, mol %	
H_2	0.11
CO_2	4.89
C_2H_6	0.20
O_2	1.29
N_2	37.59
Ar	0.48
CH_4	0.64
CO	8.22
H_2O	46.58
mol/hr	327
mol wt	24

GAS STREAM	1	2
mol/hr	143	92

LIQUID STREAM	5	6
lb/hr	0	826

SOLIDS STREAM	4	7
lb/hr	4810	3959

① AIR TO PRETREATER
② STEAM TO PRETREATER
③ PRETREATER OFF-GAS AFTER QUENCH
④ RAW COAL TO PRETREATER
⑤ DIRECT WATER QUENCH
⑥ COOLING WATER TO COOLING COIL IN PRETREATER
⑦ PRETREATER CHAR TO CHAR COOLER

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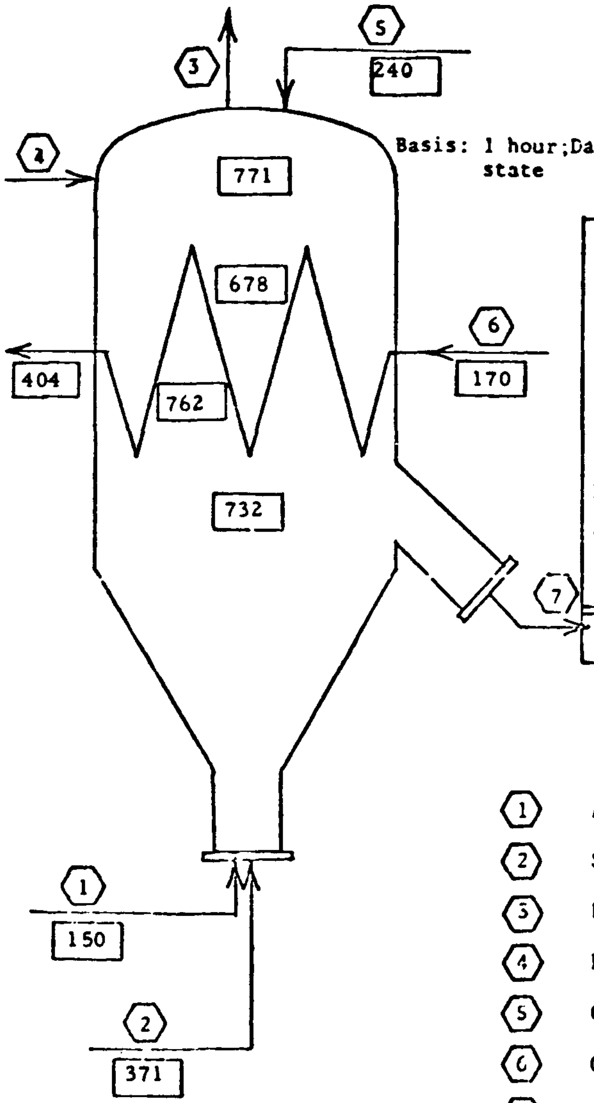
Figure 45. PRETREATMENT DATA FOR TEST 60 FOR STEADY PERIOD FROM 4/4/77 (1000 Hours) TO 4/4/77 (2100 Hours)



◯ Stream No.

▭ Temperature, °F

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

INPUT	Btu
Sensible Heat (Streams 1,2,4,5,6)	497,532
Heat of Combustion (Stream 4)	58,651,178
Steam Enthalpy (Streams 2 & 5)	3,299,200
Total	62,447,910
OUTPUT	
Sensible Heat (Streams 3 & 7)	3,449,253
Heat of Combustion (Streams 3 & 7)	54,834,549
Steam Enthalpy (Streams 3 & 6)	3,858,064
Total	62,141,866
% Balance 100	

①	Air to Pretreater
②	Steam to Pretreater
③	Pretreater Overhead
④	Raw Coal to Pretreater
⑤	Gas from Char Cooler
⑥	Cooling Water to Cooling Coil in Pretreater
⑦	Pretreated Char to Char Cooler

Figure 46. PRETREATER HEAT BALANCE DATA SHEET FOR TEST 60 FOR STEADY PERIOD FROM 4/4/77 (1000 Hours) TO 4/4/77 (2100 Hours)

Table 15. RESULTS OF HYGAS REACTOR TEST 60

	Period 1	Period 2
Date	4/1/77	4/4/77
Time, hr	1900-2200	1500-1800
Coal Gasified by Ash Balance	61	65
Carbon Gasified by Ash Balance	54	59
lb Oxygen/lb Coal Fed (Dry)	0.15	0.21
lb/Steam/lb Coal Fed (Dry)	1.43	1.95
Coal Feed Rate (Dry), tons/hr	2-1/2	2
Steam/Oxygen Gasifier Average Bed Temperature, °F	1595	1563
Superficial Velocity in Steam/Oxygen Gasifier, ft/s	0.80	0.81

hours to determine coal conversion characteristics before the forced shutdown of the test. The oxygen feed rate was gradually increased, starting at 0800 hours on May 9, and in about 4 hours the steam-oxygen gasifier temperature was raised from 1560°F to over 1700°F. The pretreated coal feed rate for this period ranged from 1-1/2 to 2 ton/hr. The reactor was operated in this mode for 8 hours.

The ash balance of the reactor feed material and spent char indicated that, with 1700°F operation in the steam-oxygen gasifier, coal conversion reached 90%. This was confirmed by 80% of the feed carbon appearing in the product gas, with the other 10% carbon accounted for in the oil make.

Post-run inspection of the reactor showed that the entire reactor was clean and free of clinkers. No part was damaged. The quench system was also very clean.

Gray-Serv technicians were called to the plant to be on hand for the opening of the reactor and to completely inspect the leaky Manway 0 closure. Their examination indicated that the closure had been properly installed with the required clearances and bolt tensions in the initial makeup of the closure for Test 61. However, a cut was found in the seal ring in the northwest quadrant of the reactor. Apparently an imperfection in the seal ring (similar to an open void in the casting or an inclusion of some foreign material) had caused the failure. The seal ring was badly eroded in that area and could not be reused. Both the body flange and the blind flange had to be remachined. The damaged seal ring was sent to Standard Oil Company of Indiana for failure analysis to determine the cause of leaks during Test 61.

The purification section was not put on-stream for Test 61 because the motor drive on the low-pressure amine circulation pump was burned out and a replacement was not available in time for the test. No methanation was done during Test 61.

The pretreater operated satisfactorily during Test 61 except that the screen on the Sweco screener periodically ripped, which interrupted the coal feed. The pretreater was operated at 750° to 775°F and provided nonagglomerating coal feed to the reactor. After the termination of Test 61, the pretreater was inspected and a few small clinkers were found throughout the reactor — far fewer than in Test 60. The char cooler had a small clinker on the east side of the vessel. The material was soft and porous. The quench section was in good condition and the tar deposits in the venturi scrubber and the quench tower were noticeably less than in previous tests.

Among the most notable achievements for Test 61 were —

a. The HYGAS reactor achieved a carbon conversion of 90% at a temperature of 1715°F in the steam-oxygen gasifier.

b. The reactor was found to be absolutely clean after the test.

c. Smooth solids flow throughout the reactor was achieved for Test 61. The specified operating bed levels and other operating parameters yielded excellent solids flow and control.

d. Excellent cyclone operation helped in giving good oil/water separation and a clean quench system.

e. Milder pretreatment conditions seemed to yield cleaner quench and separator systems in the pretreater section.

The sole problem area in Test 61 was in the coal-feeding section, involving a continuing problem with the Sweco screener. The oversize screen ripped 3 times during Test 61 and the cleaner ring support plate broke once, interrupting the feed to the pretreater. Samples of the ripped screen were sent to the fabricator and other laboratories to determine the cause of failure. The Sweco screener was completely dismantled and reassembled under the supervision of Sweco's field engineer. He provided advice on adjusting the vibrational mechanism on the Sweco screen. It was decided to begin using Type 316 stainless-steel screens in place of the Type-304 stainless-steel screens that were used earlier, because of a suspicion that the screens had corroded. The Sweco screener was tested after it was reassembled and a 4 ton/hour feed rate was reached.

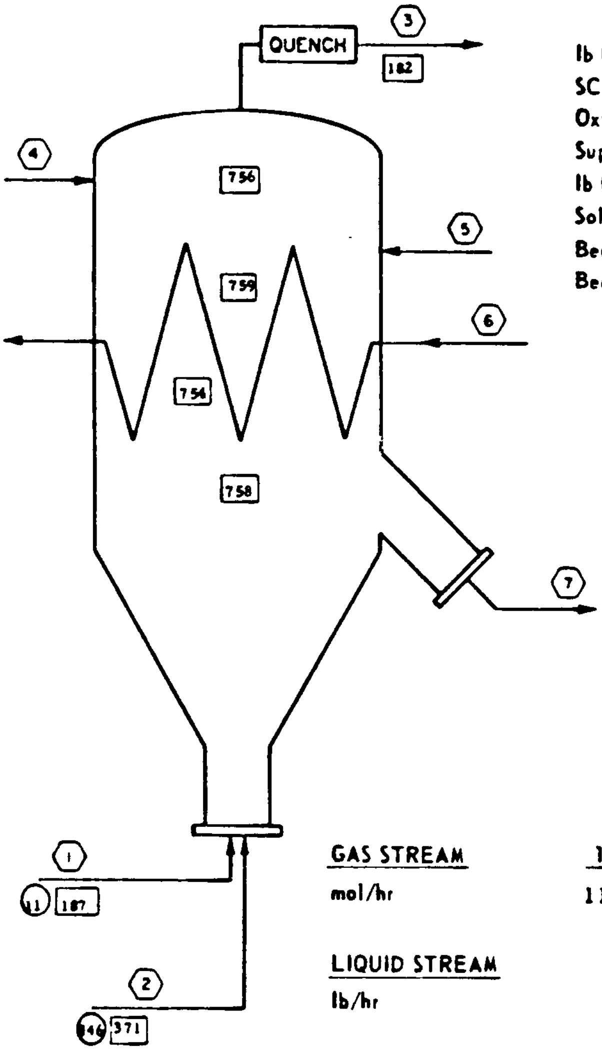
Two sets of pretreater section studies were made. Because of interruptions in the feed to the pretreater caused by problems with ripped screens in the Sweco screener (solved in Test 62), these are relatively short periods. These results are presented in Tables 16 and 17 and Figures 47 through 50. Effluent water from the pretreater quench tower was not measured. Water flow from the quench tower was estimated for both pretreater material

Table 16. MATERIAL BALANCE SUMMARY FOR THE PRETREATER SECTION FOR TEST 61 FROM 5/7/77 (0100 Hours) TO 5/7/77 (0600 Hours)

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

INPUT		C	H	O	N	S	Ar	Ash	Other	Total
Coal Feed	Wt % (Dry)	69.00	4.92	9.67	1.23	4.50	--	10.68		100
	Coal (Dry)	2761	197	387	49	180	--	428		4002
	Moisture		14	114						128
Streams to Pretreater	Air			780	2534		46			3360
	Steam		227	1815						2042
Nitrogen from purges					73					73
Air from purges				9	29					38
H_2O to venturi scrubber			1759	14,076						15,835
H_2O to quench tower			454	3632						4086
Air to char cooler				94	304		6			404
Cooling water to char cooler			64	509						573
TOTAL INPUT		2761	2715	21,416	2989	180	52	428		30,541
OUTPUT										
Pretreater Char	Wt.% (Dry)	69.30	3.45	8.64	1.43	3.99	--	13.19		100
	Char (Dry)	2249	112	280	47	130	--	428		3246
	Moisture		7	52						59
Slurry Waste from Quench	Wt.% (Dry)	64.80	2.68	12.04	1.38	4.14	--	14.96		100
	Solids (Dry)	110	5	20	2	7	--	26		170
	Tars & Oils	55	6	7	0	2				70
	H_2O & Dis materials	17	2275	18,196	2	46				20,536
Quench Tower Off-Gas	Total	141	296	2861	3441		52			6791
	Components: H_2		0							0
	CO_2	96		257						353
	C_2H_6	6	1							7
	N_2				3441					3441
	CH_4	0	0							0
	CO	39		52						91
	O_2			191						191
	Ar						52			52
	H_2O		295	2361						2656
TOTAL OUTPUT		2572	2701	21,416	3492	185	52	454		30,872
Net (Output - Input)		-189	-14	0	503	5	0	26		331
% Balance (Output/Input)		93	99	100	117	103	100	106		101

⬡ STREAM No.
○ PRESSURE, psig
□ TEMPERATURE, °F



lb Char/lb Coal = 0.79
SCF O_2/lb Coal = 2.3
Oxygen Breakthrough, % fed = 12.4
Superficial Velocity, ft/s = 0.96
lb O_2 Reacted/lb Coal = 0.17
Solids Residence Time, hr = 2.6
Bed Density, lb/cu ft = 18
Bed Height, ft = 13

GAS STREAM	3
COMPONENTS, mol %	
H_2	0
CO_2	2.77
C_2H_6	0.08
O_2	2.62
N_2	42.44
Ar	--
CH_4	0
CO	1.12
H_2O	50.97
mol/hr	290
mol wt	23

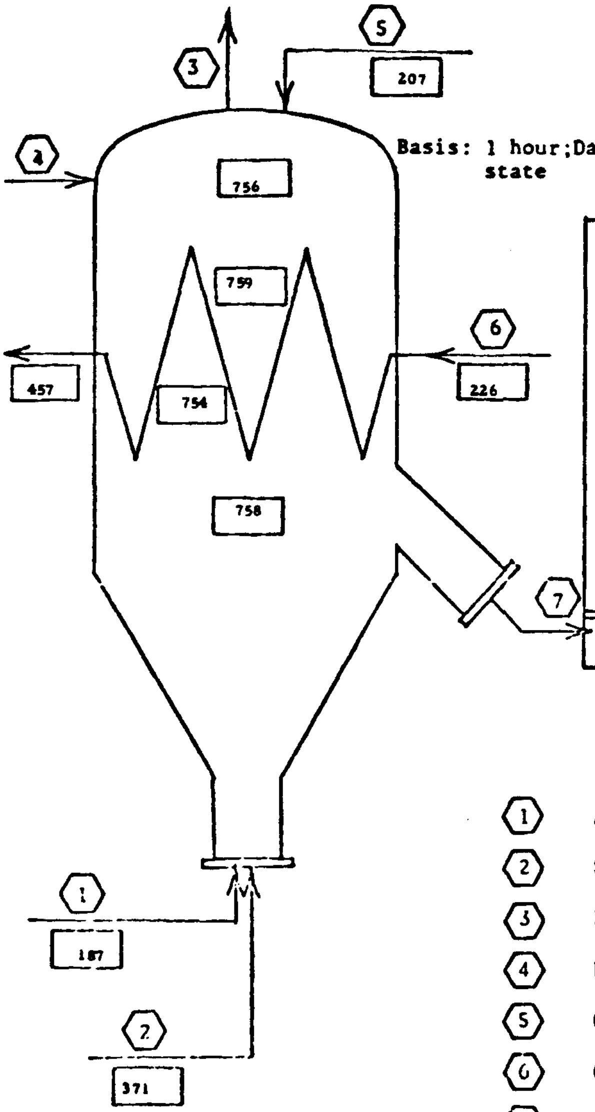
GAS STREAM	1	2
mol/hr	116	113

LIQUID STREAM	5	6
lb/hr	0	739

SOLIDS STREAM	4	7
lb/hr (dry)	4002	3246

① AIR TO PRETREATER
② STEAM TO PRETREATER
③ PRETREATER OFF-GAS AFTER QUENCH
④ RAW COAL TO PRETREATER
⑤ DIRECT WATER QUENCH
⑥ COOLING WATER TO COOLING COIL IN PRETREATER
⑦ PRETREATER CHAR TO CHAR COOLER

Figure 47. PRETREATMENT DATA FOR TEST 61 FOR STEADY PERIOD FROM 5/7/77 (0100 Hours) TO 5/7/77 (0600 Hours)



⬡ **Stream No.**

▭ **Temperature, °F**

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

INPUT	Btu
Sensible Heat (Streams 1,2,4,5,6)	408,854
Heat of Combustion (Stream 4)	49,804,890
Steam Enthalpy (Streams 2 & 5)	2,994,874
Total	53,208,618
OUTPUT	
Sensible Heat (Streams 3 & 7)	2,945,948
Heat of Combustion (Streams 3 & 7)	43,894,854
Steam Enthalpy (Streams 3 & 6)	3,680,034
Total	50,520,836
% Balance 95	

1	Air to Pretreater
2	Steam to Pretreater
3	Pretreater Overhead
4	Raw Coal to Pretreater
5	Gas from Char Cooler
6	Cooling Water to Cooling Coil in Pretreater
7	Pretreated Char to Char Cooler

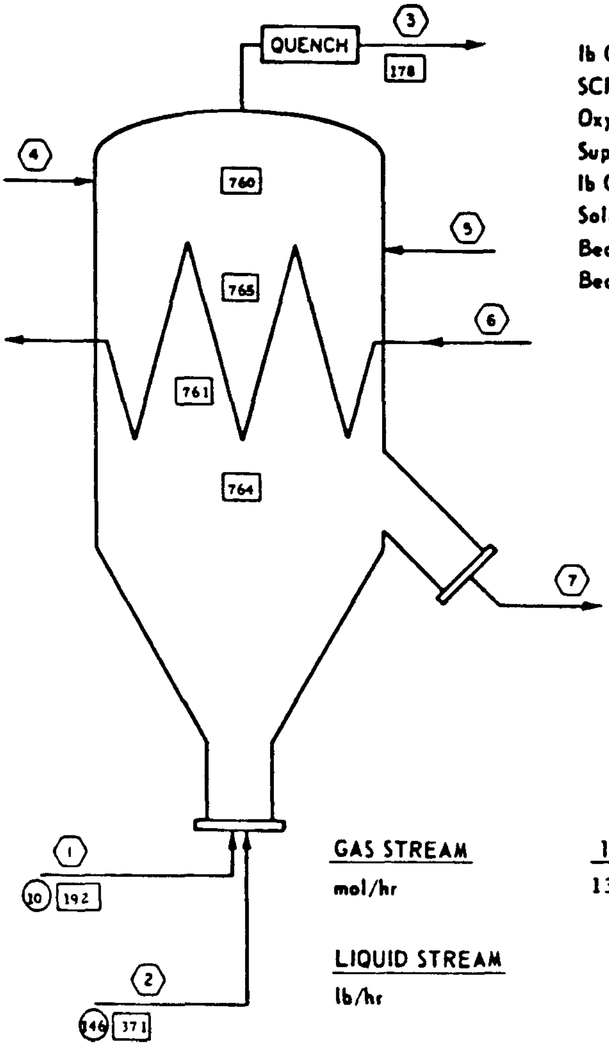
Figure 48. PRETREATER HEAT BALANCE DATA SHEET FOR TEST 61 FOR STEADY PERIOD FROM 5/7/77 (0100 Hours) TO 5/7/77 (0600 Hours)

Table 17. MATERIAL BALANCE SUMMARY FOR THE PRETREATER SECTION FOR TEST 61 FROM 5/8/77 (1300 Hours) TO 5/8/77 (2400 Hours)

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

INPUT		C	H	O	N	S	Ar	Ash	Other	Total
Coal Feed	Wt % (Dry)	68.30	4.84	9.80	1.21	4.71	--	11.14		100
	Coal (Dry)	3887	275	558	69	268	--	634		5691
	Moisture		21	170						191
Streams to Pretreater	Air			905	2943		54			3902
	Steam		166	1326						1492
Nitrogen from purges					87					87
Air from purges				9	28		1			38
H_2O to venturi scrubber			1759	14,069						15,828
H_2O to quench tower			48	387						435
Air to char cooler				103	334		6			443
Cooling water to char cooler			87	696						783
TOTAL INPUT		3887	2356	18,223	3461	268	61	634		28,890
OUTPUT										
Pretreater Char	Wt.% (Dry)	69.03	3.39	9.13	1.42	3.99		13.04		100
	Char (Dry)	3238	159	428	67	187		612		4691
	Moisture		12	93						105
Slurry Waste from Quench	Wt.% (Dry)	64.27	2.76	1200	1.29	4.11		15.57		100
	Solids (Dry)	92	4	17	2	6		22		143
	Tars & Oils	249	25	27	2	10				313
	H_2O & Dis materials	19	1832	14,698	2	47				16,548
Quench Tower Off-Gas	Total	271	291	3010	3717		56			7345
	Components: H_2		2							2
	CO_2	138		368						506
	C_2H_6	4	1							5
	N_2				3717					3717
	CH_4	7	2							9
	CO	122		163						285
	O_2			194						194
	Ar						56			56
	H_2O		286	2285						2571
TOTAL OUTPUT		3869	2323	18,223	3790	250	56	634		29,145
Net (Output - Input)		−18	−33	0	329	−18	−5	0		255
% Balance (Output/Input)		100	99	100	110	93	92	100		101

○ STREAM No.
○ PRESSURE, psig
☐ TEMPERATURE, °F



lb Char/lb Coal = 0.84
SCF O_2/lb Coal= 1.9
Oxygen Breakthrough, % fed= 10.1
Superficial Velocity, ft/s = 0.91
lb O_2 Reacted/lb Coal= 0.14
Solids Residence Time, hr = 1.6
Bed Density, lb/cu ft = 18
Bed Height, ft = 12

GAS STREAM	3
COMPONENTS, mol %	
H_2	0.37
CO_2	3.75
C_2H_6	0.05
O_2	1.98
N_2	43.32
Ar	0.46
CH_4	0.19
CO	3.32
H_2O	46.56
mol/hr	306
mol wt	24

GAS STREAM	1	2
mol/hr	135	83

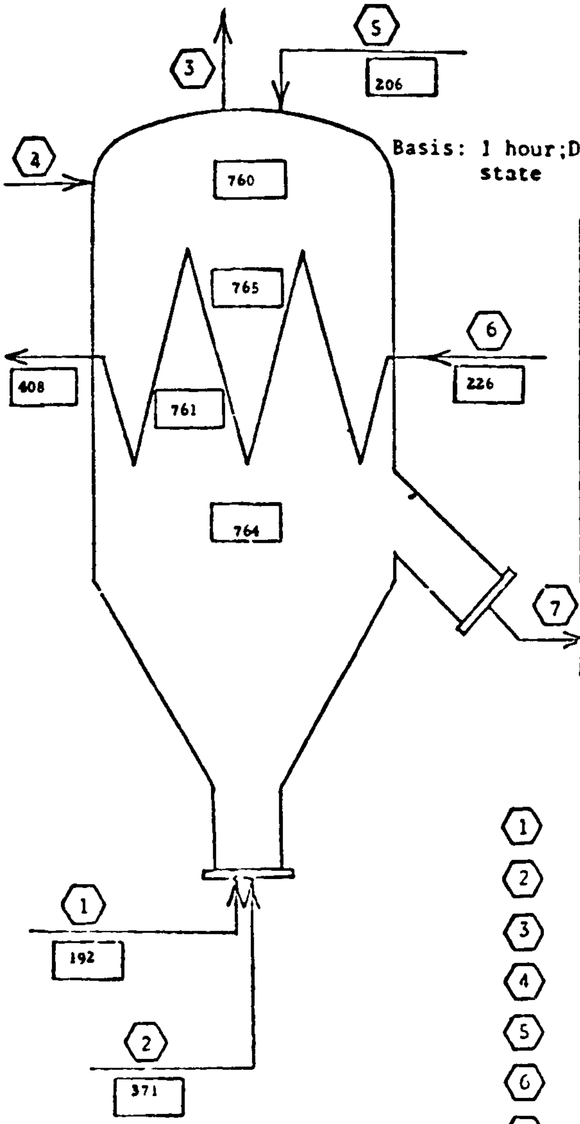
LIQUID STREAM	5	6
lb/hr	0	946

SOLIDS STREAM	4	7*
lb/hr (dry)	5691	4691

*By ash balance.

① AIR TO PRETREATER
② STEAM TO PRETREATER
③ PRETREATER OFF-GAS AFTER QUENCH
④ RAW COAL TO PRETREATER
⑤ DIRECT WATER QUENCH
⑥ COOLING WATER TO COOLING COIL IN PRETREATER
⑦ PRETREATER CHAR TO CHAR COOLER

Figure 49. PRETREATMENT DATA FOR TEST 61 FOR STEADY PERIOD FROM 5/8/77 (1300 Hours) TO 5/8/77 (2400 Hours)



Stream No.

Temperature, °F

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

INPUT	Btu
Sensible Heat (Streams 1,2,4,5,6)	554,418
Heat of Combustion (Stream 4)	70,107,429
Steam Enthalpy (Streams 2 & 5)	2,570,774
Total	73,232,621
OUTPUT	
Sensible Heat (Streams 3 & 7)	3,809,268
Heat of Combustion (Streams 3 & 7)	63,124,776
Steam Enthalpy (Streams 3 & 6)	3,810,154
Total	70,744,198
% Balance 97	

1	Air to Pretreater
2	Steam to Pretreater
3	Pretreater Overhead
4	Raw Coal to Pretreater
5	Gas from Char Cooler
6	Cooling Water to Cooling Coil in Pretreater
7	Pretreated Char to Char Cooler

Figure 50. PRETREATER HEAT BALANCE DATA SHEET FOR TEST 61 FOR STEADY PERIOD FROM 5/8/77 (1300 Hours) TO 5/8/77 (2400 Hours)

balance periods to close the oxygen balance. On May 8, the pretreater char rate from the pretreater was obtained by an ash balance because the pretreater char weigh belt was not in continuous operation during that period.

Reactor operation during the end of Test 61 was of special interest, and five periods were selected for study. Their results are presented in Tables 18 through 22 and Figures 51 through 60. Level control problems in the cyclone slurry quench system caused gas flow surges through the light oil stripper, and the light oil stripper was in intermittent service. Consequently, water and oil balances were estimated for these periods, and spent-char flow rates were estimated by ash balance. Coal conversion during these five periods ranged from 55% to 92%, and carbon conversion ranged from 46% to 90%. The steam-oxygen gasifier temperature during the high-conversion period was 1705°F.

Test 62, June 1977

Highlights

Smooth reactor operation was achieved in Test 62, with a coal feed rate of over 2 ton/hr. However, operational problems in the product-gas quench forced termination of the test 3 days after it was begun.

Details

Light-off for Test 62 was initiated at 0815 hours on May 27, and pretreated coal was fed to the gasifier at 1135 hours on May 30. Reactor operation after initial start-up was smooth. Temperatures ranging from 1700° to 1710°F were observed in the steam-oxygen gasifier. Pretreated char feed rates to the reactor were over 2 ton/hr and reactor operation was self-sustained after 1000 hours on June 1. A total of 68 tons of pretreated char was fed to the reactor.

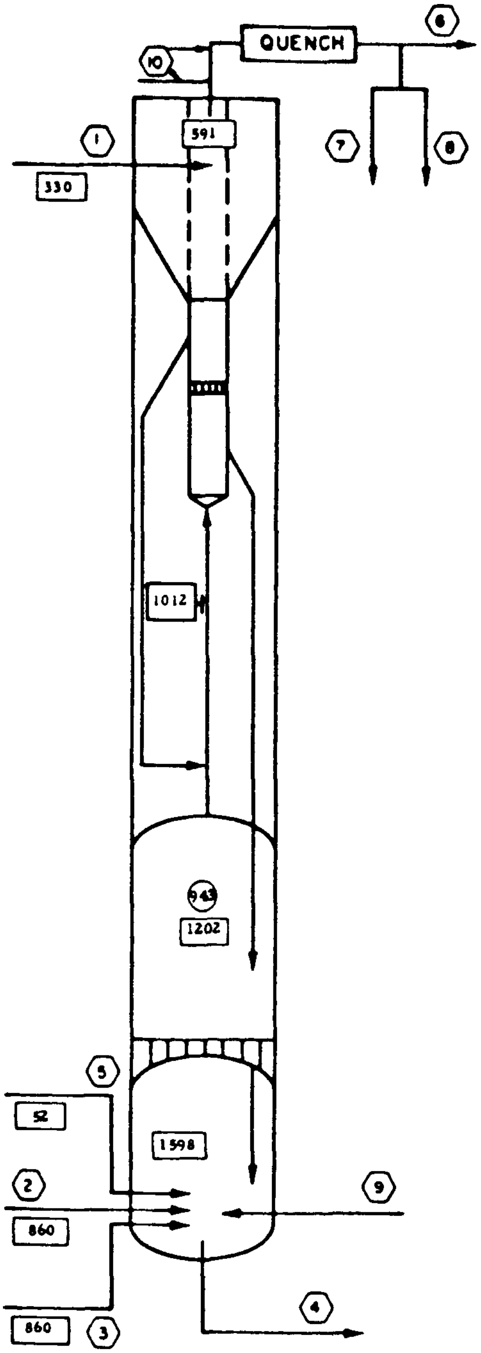
The pretreater operated satisfactorily to produce nonagglomerating char with temperatures ranging from 765° to 775°F. One hundred thirty-one tons of raw Peabody No. 10 bituminous coal was fed to the pretreater at rates up to 3 ton/hr, over a period of 67 hours.

The Sweco screen operated without problems, throughout the test. The problem of ripped screens, seen in the previous tests, had been solved by replacing the Type-304 stainless-steel screen with a Type-316 stainless-steel screen.

Table 18. MATERIAL BALANCE SUMMARY FOR THE HYGAS GASIFIER FOR TEST 61 FROM 5/7/77 (2200 Hours) TO 5/8/77 (1200 Hours)

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

INPUT		C	H	O	N	S	Cl	ASH	OTHER	TOTAL
Coal Feed	Wt % (Dry)	69.08	3.48	8.74	1.39	4.19	--	13.12		100
	Coal (Dry)	2591	130	328	52	157	--	492		3750
	Moisture		9	69						78
Sparger	Oxygen			541						541
	Steam		494	3955						4449
Burner	Oxygen			0						0
	Steam		0	0						0
	Hydrogen		0							0
Stripping Ring	Steam		145	1164						1309
Nitrogen from purges					355					355
Pump seal flush			74	593						667
Cooling water spray			0	0						0
Water to cyclone pot			281	2248						2529
Light oil in		9359	891							10,250
TOTAL INPUT		11,950	2024	8898	407	157	--	492		23,928
OUTPUT										
Reactor Overhead	Wt % (Dry)	79.48	2.77	3.68	1.24	2.74	--	10.09		100
	Dust (Dry)	399	14	18	6	14	--	51		502
Spent Char	Wt % (Dry)	70.65	1.19	0.93	0.55	1.65	--	25.03		100
	Char (Dry)	1245	21	16	10	29	--	441		1762
Product Gas After Quench	Total (Dry)	777	184	985	349	60				2355
	Components H_2		61							61
	CO_2	328		875						1203
	C_2H_6	22	5							27
	H_2S		4			60				64
	N_2				349					349
	CH_4	344	114							458
	CO	83		110						193
Water Out + dissolved materials		13	902	7207	1	21				8144
Toluene storage tank vent gases		343	33	605	61	35				1077
Stripper vent gas		37	3	67	17	1				125
Light oil out		9136	870							10,006
TOTAL OUTPUT		11,950	2027	8898	444	160	--	492		23,971
Net (Output-Input)		0	3	0	37	3	--	0		43
% Balance (Output/Input)		100	100	100	109	102	--	100		100



MATERIAL BALANCE, %

C	110
H	106
O	180
ASH	100

⬡ STREAM No.
○ PRESSURE, psig
▭ TEMPERATURE, °F

	GAS STREAMS	
	6	10
mol % (dry)		
H_2	28.15	23.34
CO_2	25.15	34.02
C_2H_6	0.84	1.01
Ar	--	--
N_2	11.45	10.74
H_2S	1.72	2.12
CH_4	26.36	23.70
CO	6.33	5.07
mol/hr (dry)	109.	142

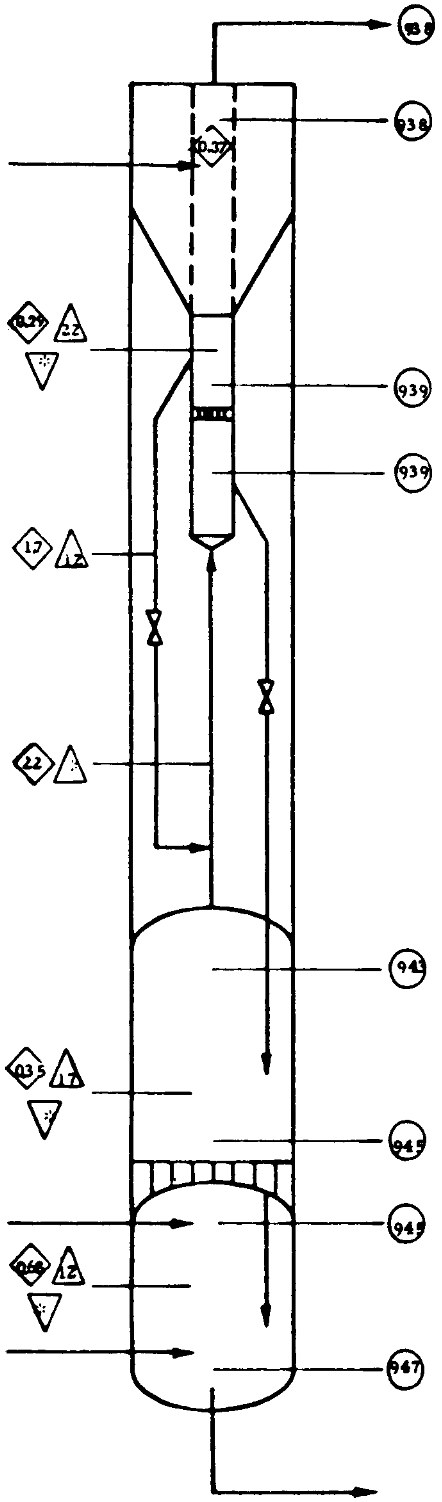
	SOLIDS STREAMS		
	1	10	4
wt % (dry)			
C	69.08	79.48	70.65
H	3.48	2.77	1.19
N	1.39	1.24	0.55
Cl	--	--	--
S	4.19	2.74	1.65
ASH	13.12	10.09	25.03
O	8.74	3.68	0.93
lb/hr (dry)	3750	502	1762
MOISTURE, wt %	2.0		
SLURRY CONCN, wt %	21		

	LIQUID STREAMS		
	7	8	1
	lb/hr		
H_2O	4948	--	--
LIGHT OIL	--	10,006	10,250

GAS FEED STREAMS			
2	3	5	9
mol/hr			
247	73	17	0

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 51. HYGAS REACTOR DATA FOR TEST 61 FOR STEADY PERIOD FROM 5/7/77 (2200 Hours) TO 5/8/77 (1200 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 = 0.14
lb Steam/lb Coal Fed = 1.5
lb Hydrogen/lb Coal Fed = 0
lb Coal Fed/1000 SCF Product Gas = 130

By Ash Balance

Coal Gasified, % = 55
Carbon Gasified, % = 46

Methane Yield, SCF/lb coal fed = 3.4
Equivalent Methane Yield, SCF/lb coal fed = 4.7

Bed Height, ft

Slurry Dryer = 2
HTR = 20
SOG = 22

‡ Light-oil recovery system vent gas composition from Test 60.

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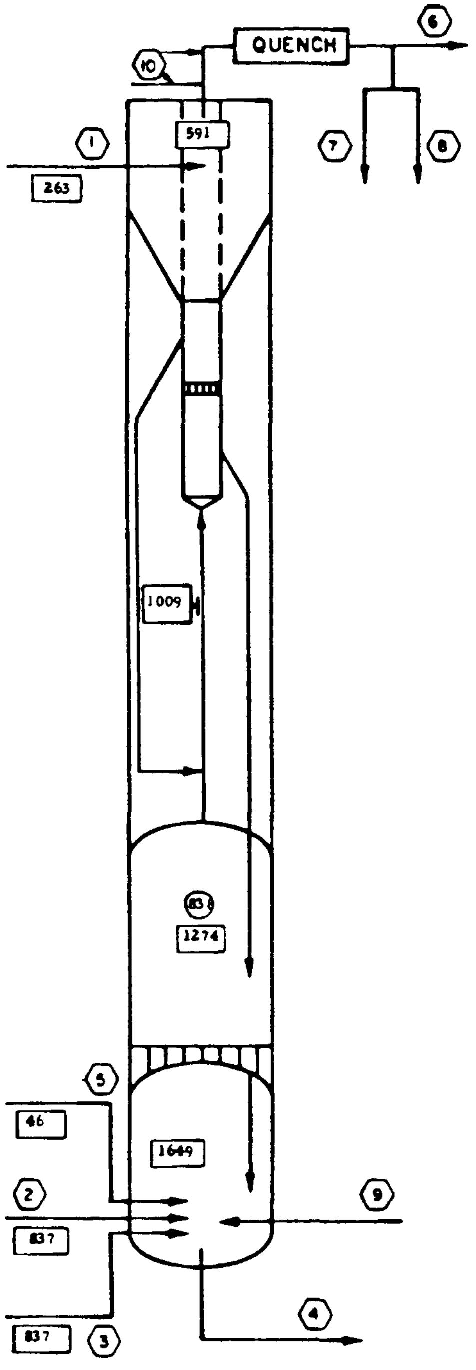
Figure 52. HYGAS REACTOR ENGINEERING DATA FOR TEST 61 FOR STEADY PERIOD FROM 5/7/77 (2200 Hours) TO 5/8/77 (1200 Hours)

Table 19. MATERIAL BALANCE SUMMARY FOR THE HYGAS GASIFIER FOR TEST 61 FROM 5/9/77 (0000 Hours) TO 5/9/77 (0300 Hours)

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

INPUT		C	H	O	N	S	Cl	ASH	OTHER	TOTA
Coal Feed	Wt % (Dry)	68.60	3.32	9.15	1.39	4.13	--	13.41		100
	Coal (Dry)	3281	159	438	66	198	--	642		4784
	Moisture		12	96						108
Sparger	Oxygen			602						602
	Steam		501	4010						4511
Burner	Oxygen			0						0
	Steam		0	0						0
	Hydrogen		0							0
Stripping Ring	Steam		139	1110						1249
Nitrogen from purges					344					344
Pump seal flush			74	593						667
Cooling water spray			0	0						0
Water to cyclone pot			232	1856						2088
Light oil in		8306	791							9097
TOTAL INPUT		11, 587	1908	8705	410	198	--	642		23, 450
OUTPUT										
Reactor Overhead	Wt % (Dry)	79.70	2.54	4.37	1.21	2.78	--	9.40		100
	Dust (Dry)	482	15	26	7	17	--	57		604
Spent Char	Wt % (Dry)	68.40	1.05	1.34	0.52	1.73	--	26.96		100
	Char (Dry)	1484	23	29	11	38	--	585		2170
Product Gas After Quench	Total (Dry)	1000	226	1333	409	87				3051
	Components H_2		75							75
	CO_2	440		1173						1615
	C_2H_6	20	5							25
	H_2S		5			87				92
	N_2				409					409
	CH_4	422	140							562
	CO	118		158						276
Water Out + dissolved materials		34	849	6741	23	26				7673
Toluene storage tank vent gases		293	28	518	52	30				921
Stripper vent gas		32	2	58	15	1				108
Light oil out		8262	787							9049
TOTAL OUTPUT		11, 587	1930	8705	517	199	--	642		23, 576
Net (Output-Input)		0	22	0	107	1	--	0		126
% Balance (Output/Input)		100	101	100	126	101	--	100		101



MATERIAL BALANCE, %

C	100
H	101
O	100
ASH	100

○ STREAM No.
○ PRESSURE, psig
☐ TEMPERATURE, °F

	GAS STREAMS	
	6	10
mol % (dry)		
H_2	27.31	23.92
CO_2	26.72	32.95
C_2H_6	0.60	0.76
Ar	--	--
N_2	10.64	10.26
H_2S	1.96	2.22
CH_4	25.59	23.77
CO	7.18	6.12
mol/hr (dry)	137	165

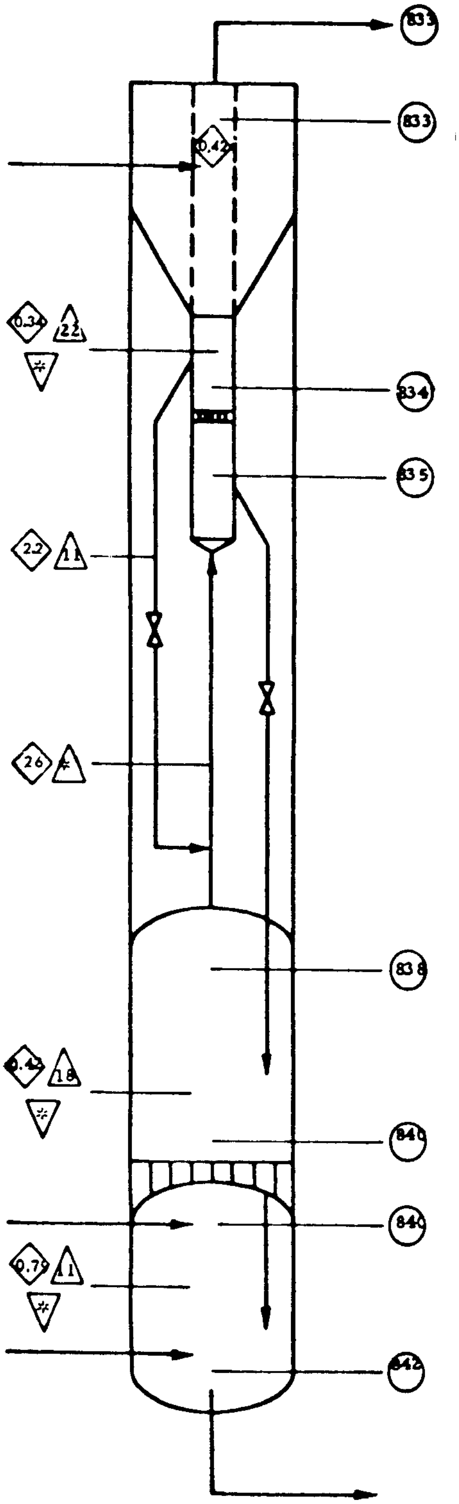
	SOLIDS STREAMS		
	1	10	4
wt % (dry)			
C	68.60	79.70	68.40
H	3.32	2.54	1.05
N	1.39	1.21	0.52
Cl	--	--	--
S	4.13	2.78	1.73
ASH	13.41	9.40	26.96
O	9.15	4.37	1.34
lb/hr (dry)	4784	604	2170
MOISTURE, wt %	2.2		
SLURRY CONCN, wt %	35		

	LIQUID STREAMS		
	7	8	1
	lb/hr		
H_2O	--	--	--
LIGHT OIL	4918	9049	9097

GAS FEED STREAMS			
2	3	5	9
mol/hr			
250	69	19	0

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 53. HYGAS REACTOR DATA FOR TEST 61 FOR STEADY PERIOD FROM 5/9/77 (0000 Hours) TO 5/9/77 (0300 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 = 0.13
lb Steam/lb Coal Fed = 1.20
lb Hydrogen/lb Coal Fed = 0
lb Coal Fed/1000 SCF Product Gas = 139

By Ash Balance

Coal Gasified, % = 58
Carbon Gasified, % = 50

Methane Yield, SCF/lb coal fed = 3.1
Equivalent Methane Yield, SCF/lb coal fed = 4.3

Bed Height, ft

Slurry Dryer = 2
HTR = 18
SOG = 20

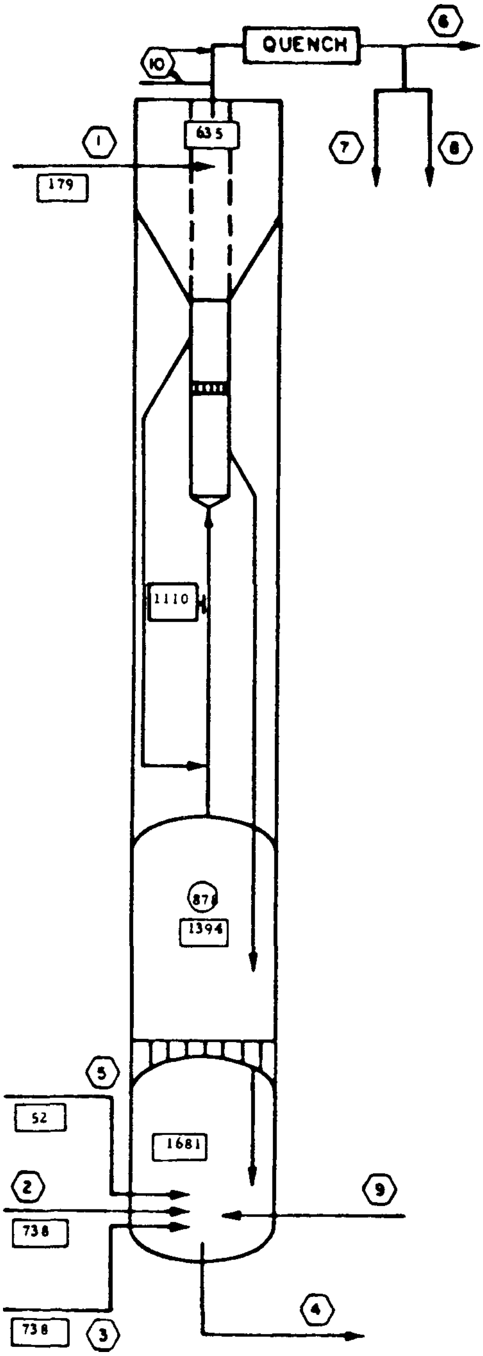
‡ Light-oil recovery system vent-gas composition from Test 60.

Figure 54. HYGAS REACTOR ENGINEERING DATA FOR TEST 61 FOR STEADY PERIOD FROM 5/9/77 (0000 Hours) TO 5/9/77 (0300 Hours)

Table 20. MATERIAL BALANCE SUMMARY FOR THE HYGAS GASIFIER FOR TEST 61 FROM 5/9/77 (0900 Hours) TO 5/9/77 (1200 Hours)

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

INPUT		C	H	O	N	S	Cl	ASH	OTHER	TOTAL
Coal Feed	Wt % (Dry)	69.60	3.22	9.09	1.44	4.04	--	12.61		100
	Coal (Dry)	2819	130	368	58	164	--	511		4050
	Moisture		10	77						87
Sparger	Oxygen			1005						1005
	Steam		757	6055						6812
Burner	Oxygen			0						0
	Steam		0	0						0
	Hydrogen		0							0
Stripping Ring	Steam		173	1386						1559
Nitrogen from purges					333					333
Pump seal flush			74	593						667
Cooling water spray			0	0						0
Water to cyclone pot			354	2833						3187
Light oil in		10,065	959							11,024
TOTAL INPUT		12,884	2457	12,317	391	164	--	511		28,724
OUTPUT										
Reactor Overhead	Wt % (Dry)	76.40	2.66	5.02	1.30	3.16	--	11.46		100
	Dust (Dry)	610	21	40	10	25	--	91		797
Spent Char	Wt % (Dry)	64.00	0.95	0.91	0.37	0.97	--	32.80		100
	Char (Dry)	820	12	12	5	12	--	420		1281
Product Gas After Quench	Total (Dry)	1291	279	1880	379	120				3949
	Components H_2		111							111
	CO_2	609		1624						2233
	C_2H_6	25	6							31
	H_2S		7			120				127
	N_2				379					379
	CH_4	465	155							620
	CO	192		256						448
Water Out + dissolved materials		39	1202	9536	29	33				10,839
Toluene storage tank vent gases		400	39	704	70	41				1254
Stripper vent gas		77	7	145	35	3				267
Light oil out		9647	919							10,566
TOTAL OUTPUT		12,884	2479	12,317	528	234	--	511		28,953
Net (Output-Input)		0	22	0	137	70	--	0		229
% Balance (Output/Input)		100	101	100	135	143	--	100		101



⬡ STREAM No.
○ PRESSURE, psig
☐ TEMPERATURE, °F

MATERIAL BALANCE, %

C	100
H	101
O	100
ASH	100

	GAS STREAMS	
	6	10
mol % (dry)		
H_2	31.05	26.60
CO_2	28.27	34.84
C_2H_6	0.57	0.74
Ar	--	--
N_2	7.53	7.84
H_2S	2.09	2.31
CH_4	21.58	20.27
CO	8.91	7.40
mol/hr (dry)	180	221

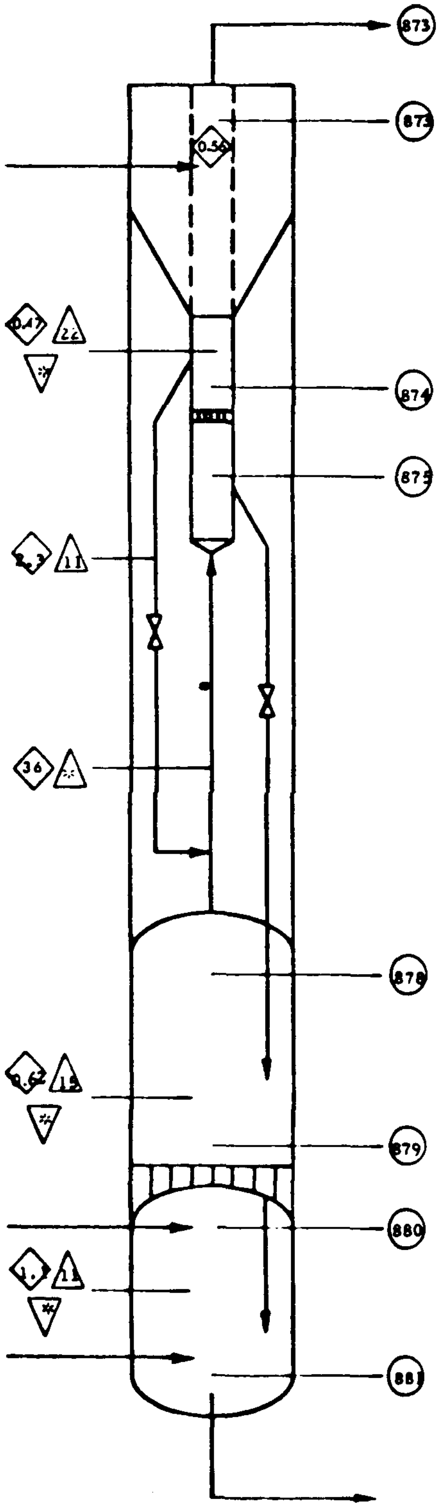
	SOLIDS STREAMS		
	1	10	4
wt % (dry)			
C	69.6	76.4	64.00
H	3.22	2.66	0.95
N	1.44	1.30	0.37
Cl	--	--	--
S	4.04	3.16	0.97
ASH	12.61	11.46	32.80
O	9.09	5.02	0.91
lb/hr (dry)	4050	797	1281
MOISTURE, wt %	2.1 *		
SLURRY CONCN, wt %	34		

	LIQUID STREAMS		
	7	8	1
	lb/hr		
H_2O	6985	--	--
LIGHT OIL	--	10, 566	11, 024

GAS FEED STREAMS			
2	3	5	9
mol/hr			
378	87	31	0

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 55. HYGAS REACTOR DATA FOR TEST 61 FOR STEADY PERIOD FROM 5/9/77 (0900 Hours) TO 5/9/77 (1200 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 = 0.25
lb Steam/lb Coal Fed = 2.1
lb Hydrogen/lb Coal Fed = 0
lb Coal Fed/1000 SCF Product Gas = 87

By Ash Balance

Coal Gasified, % = 70
Carbon Gasified, % = 65

Methane Yield SCF/lb coal fed = 4.2
Equivalent Methane Yield, SCF/lb coal fed = 6.2

Bed Height, ft

Slurry Dryer = *
HTR = 18
SOG = 21

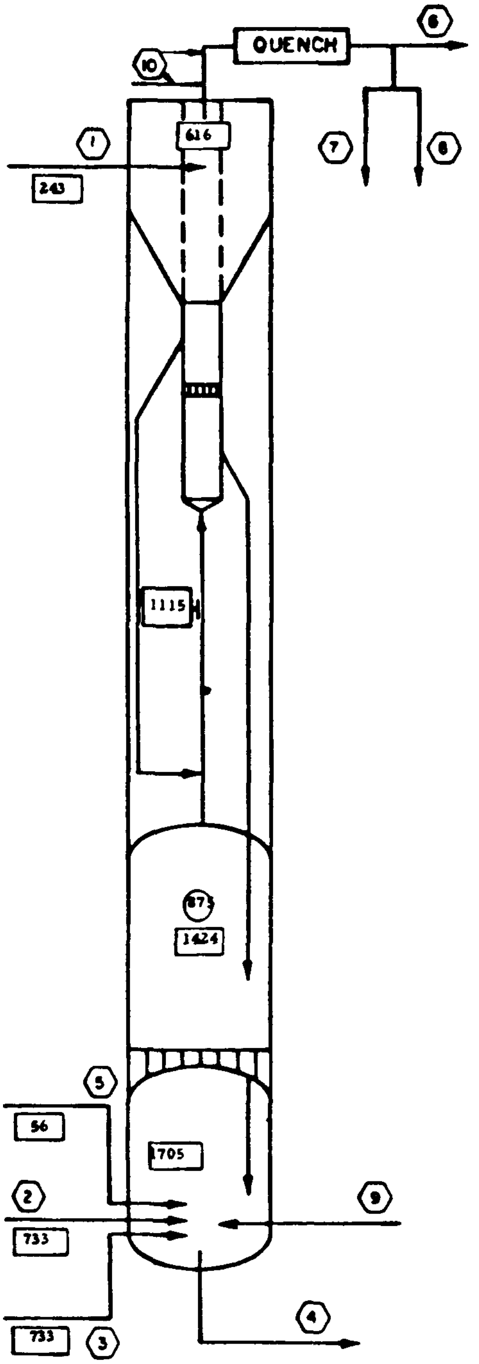
‡ Light-oil recovery system vent gas from Test 60. Toluene storage vent gas from an earlier period during Test 61.

Figure 56. HYGAS REACTOR ENGINEERING DATA FOR TEST 61 FOR STEADY PERIOD FROM 5/9/77 (0900 Hours) TO 5/9/77 (1200 Hours)

Table 21. MATERIAL BALANCE SUMMARY FOR THE HYGAS GASIFIER FOR TEST 61 FROM 5/9/77 (1200 Hours) TO 5/9/77 (1600 Hours)

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

INPUT		C	H	O	N	S	Cl	ASH	OTHER	TOTAL
Coal Feed	Wt % (Dry)	69.10	3.38	8.95	1.36	4.06	--	13.15		100
	Coal (Dry)	2186	107	283	43	129	--	416		3164
	Moisture		6	52						58
Sparger	Oxygen			1059						1059
	Steam		760	6081						6841
Burner	Oxygen			0						0
	Steam		0	0						0
	Hydrogen		0							0
Stripping Ring	Steam		174	1391						1565
Nitrogen from purges					307					307
Pump seal flush			74	593						667
Cooling water spray			0	0						0
Water to cyclone pot			399	3188						3587
Light oil in		11,506	1096							12,602
TOTAL INPUT		13,692	2616	12,647	350	129	--	416		29,850
OUTPUT										
Reactor Overhead	Wt % (Dry)	75.00	2.66	4.39	1.06	2.84	--	14.05		100
	Dust (Dry)	370	13	22	5	14	--	69		493
Spent Char	Wt % (Dry)	33.73	0.56	0	0.15	0.84	--	64.72		100
	Char (Dry)	181	3	0	1	4	--	347		536
Product Gas After Quench	Total (Dry)	1216	261	1840	384	96				3797
	Components H_2		116							116
	CO_2	589		1571						2160
	C_2H_6	22	5							27
	H_2S		6			96				102
	N_2				384					384
	CH_4	403	134							537
	CO	202		269						471
Water Out + dissolved materials		39	1250	9938	30	33				11,290
(earlier time period) Toluene storage tank vent gases		406	39	716	71	41				1273
Stripper vent gas		69	5	131	32	3				240
Light oil make		11,789	1123							12,912
TOTAL OUTPUT		14,070	2694	12,647	523	191	--	416		30,541
Net (Output-Input)		378	78	0	173	62	--	0		691
% Balance (Output/Input)		103	103	100	149	148	--	100		102



○ STREAM No.
○ PRESSURE, psig
☐ TEMPERATURE, °F

MATERIAL BALANCE, %

C	103
H	103
O	100
ASH	100

	GAS STREAMS	
	6	10
mol % (dry)		
H_2	33.22	28.26
CO_2	27.99	34.77
C_2H_6	0.51	0.70
Ar	--	--
N_2	7.82	8.04
H_2S	1.71	2.00
CH_4	19.16	18.29
CO	9.59	7.94
mol/hr (dry)	175	217

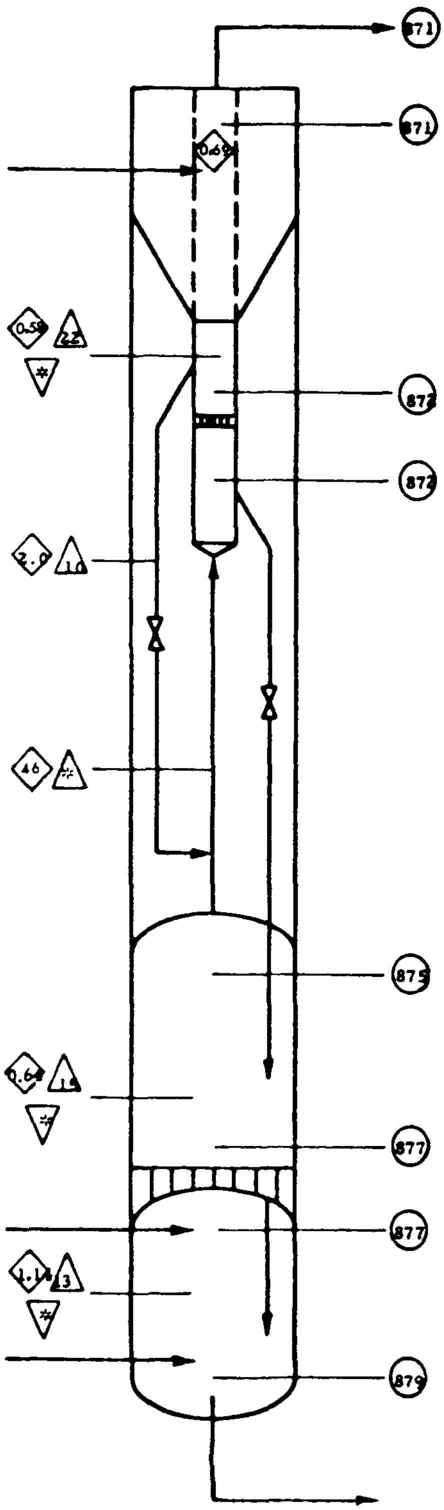
	SOLIDS STREAMS		
	1	10	4
wt % (dry)			
C	69.10	75.00	33.73
H	3.38	2.66	0.56
N	1.36	1.06	0.15
Cl	--	--	--
S	4.06	2.84	0.84
ASH	13.15	14.05	64.72
O	8.95	4.39	--
lb/hr (dry)	3164	493	536
MOISTURE, wt %	1.8		
SLURRY CONCN, wt %	20		

	LIQUID STREAMS		
	7	8	1
	lb/hr		
H_2O	9956	--	--
LIGHT OIL	--	12,912	12,602

GAS FEED STREAMS			
2	3	5	9
mol/hr			
380	87	33	0

① FEED SLURRY	⑥ GASIFIER EFFLUENT AFTER QUENCH
② HIGH-PRESSURE STEAM TO STEAM-OXYGEN SPARGER	⑦ WATER MADE
③ HIGH-PRESSURE STEAM TO STRIPPING RING	⑧ LIGHT OIL TO RECYCLE
④ SPENT CHAR	⑨ GASES FROM EXTERNAL HEATER
⑤ HIGH-PRESSURE OXYGEN TO STEAM-OXYGEN SPARGER	⑩ REACTOR OVERHEAD

Figure 57. HYGAS REACTOR DATA FOR TEST 61 FOR STEADY PERIOD FROM 5/9/77 (1200 Hours) TO 5/9/77 (1600 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 =	0.34
lb Steam/lb Coal Fed =	2.66
lb Hydrogen/lb Coal Fed =	0
lb Coal Fed/1000 SCF Product Gas =	69

By Ash Balance

Coal Gasified, % =	92
Carbon Gasified, % =	90

Methane Yield SCF/lb coal fed =	4.7
Equivalent Methane Yield, SCF/lb coal fed =	7.4

Bed Height, ft

Slurry Dryer =	*
HTR =	17
SOG =	18

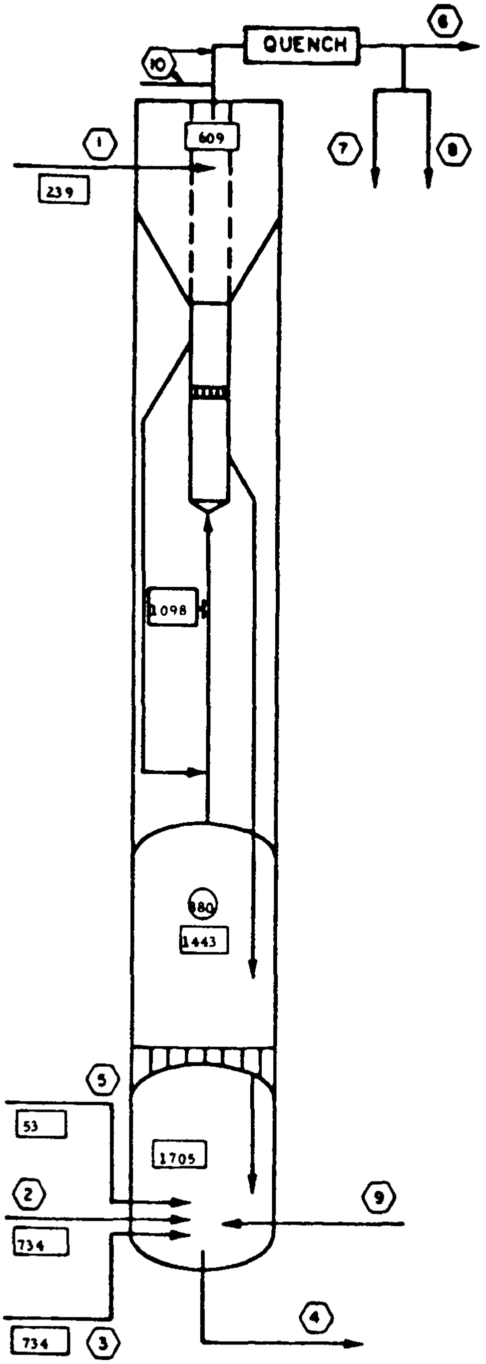
‡Light-oil recovery system off-gas from Test 60. Toluene storage tank vent gas from an earlier time period of Test 61.

Figure 58. HYGAS REACTOR ENGINEERING DATA FOR TEST 61 FOR STEADY PERIOD FROM 5/9/77 (1200 Hours) TO 5/9/77 (1600 Hours)

Table 22. MATERIAL BALANCE SUMMARY FOR THE HYGAS GASIFIER FOR TEST 61 FROM 5/9/77 (1600 Hours) TO 5/9/77 (2000 Hours)

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

INPUT		C	H	O	N	S	Cl	ASH	OTHER	TOTAL
Coal Feed	Wt % (Dry)	68.90	3.47	8.60	1.44	4.31	--	13.28		100
	Coal (Dry)	2971	150	371	62	186	--	573		4313
	Moisture		9	74						83
Sparger	Oxygen			1054						1054
	Steam		758	6060						6818
Burner	Oxygen			0						0
	Steam		0	0						0
	Hydrogen		0							0
Stripping Ring	Steam		173	1388						1561
Nitrogen from purges					302					302
Pump seal flush			74	593						667
Cooling water spray			0	0						0
Water to cyclone pot			400	3198						3598
Light oil in		10,469	997							11,466
TOTAL INPUT		13,440	2561	12,738	364	186	--	573		29,862
OUTPUT										
Reactor Overhead	Wt % (Dry)	73.80	2.64	4.63	1.18	2.96	--	14.79		100
	Dust (Dry)	662	23	41	11	27	--	133		897
Spent Char	Wt % (Dry)	49.70	0.70	0.07	0.23	0.74	--	48.56		100
	Char (Dry)	450	6	1	2	7	--	440		906
Product Gas After Quench	Total (Dry)	1230	267	1860	391	90				3838
	Components H_2		123							123
	CO_2	588		1567						2155
	C_2H_6	21	5							26
	H_2S		6			90				96
	N_2				391					391
	CH_4	401	133							534
	CO	220		293						513
Water Out + dissolved materials		39	1247	9918	31	34				11,269
Toluene storage tank vent gases		450	44	793	79	46				1412
Stripper vent gas		68	5	125	31	3				232
Light oil oil		10,541	1004							11,545
TOTAL OUTPUT		13,440	2596	12,738	545	207	--	573		30,099
Net (Output-Input)		0	35	0	181	21	--	0		237
% Balance (Output/Input)		100	101	100	150	111	--	100		101



MATERIAL BALANCE, %

C
H
O
ASH

⬡ STREAM No.
○ PRESSURE, psig
▭ TEMPERATURE, °F

	GAS STREAMS	
	6	10
mol % (dry)		
H_2	34.11	28.77
CO_2	27.27	34.47
C_2H_6	00.48	0.69
Ar	--	--
N_2	7.78	7.98
H_2S	1.57	1.93
CH_4	18.59	17.82
CO	10.20	8.34
mol/hr (dry)	180	224

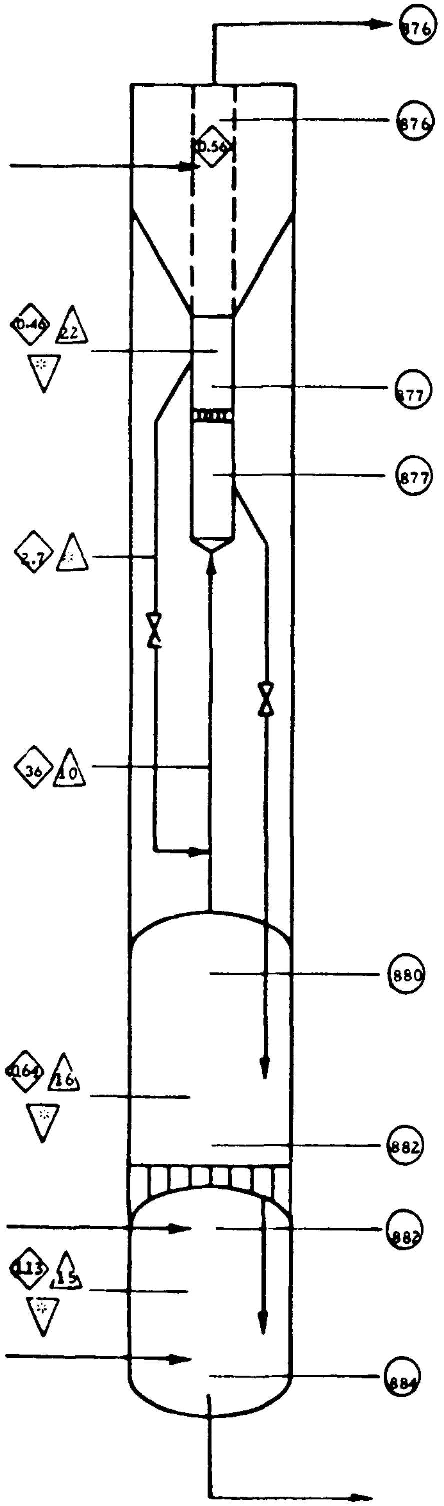
	SOLIDS STREAMS		
	1	10	4
wt % (dry)			
C	68.90	73.80	49.70
H	3.47	2.64	0.70
N	1.44	1.18	0.23
Cl	--	--	--
S	4.31	2.96	0.74
ASH	13.28	14.79	48.56
O	8.60	4.63	0.07
lb/hr (dry)	4313		
MOISTURE, wt %	1.9		
SLURRY CONCN, wt %	27		

	LIQUID STREAMS		
	7	8	1
	lb/hr		
H_2O	6975	--	--
LIGHT OIL	--	11, 545	11,466

GAS FEED STREAMS			
2	3	5	9
mol/hr			
379	87	33	0

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 59. HYGAS REACTOR DATA FOR TEST 61 FOR STEADY PERIOD FROM 5/9/77 (1600 Hours) TO 5/9/77 (2000 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 = 0.24
lb Steam/lb Coal Fed = 1.94
lb Hydrogen/lb Coal Fed = 0
lb Coal Fed/1000 SCF Product Gas = 88

By Ash Balance

Coal Gasified, % = 84
Carbon Gasified, % = 80

Methane Yield SCF/lb coal fed = 3.5
Equivalent Methane Yield, SCF/lb coal fed = 5.6

Bed Height, ft

Slurry Dryer = *
HTR = 16
SOG = 16

†Light-oil recovery system vent-gas from Test 60. Toluene storage off-gas taken from an earlier period during Test 61.

Figure 60. HYGAS REACTOR ENGINEERING DATA FOR TEST 61 FOR STEADY PERIOD FROM 5/9/77 (1600 Hours) TO 5/9/77 (2000 Hours)

The purification and the IGT fixed-bed catalyst methanation sections were not put on-stream because of problems in the quench system. The light-oil recovery unit operated satisfactorily.

Test 62 was terminated 3 days after it began, on June 2 at 1030 hours, because of severe flooding in the product-gas quench tower. The entire quench system was inspected after the test and two lines were plugged by accumulations of char fines from previous tests. The raw product-gas line from the prequench tower to the quench tower had several char deposits in it, and the light-oil liquid line from the prequench tower to the quench separator had a 4-foot solid plug of char in it. These plugged lines could have caused a partial raw product-gas bypass through the quench separator and, consequently, the carry-over of liquids into the quench tower. This resulted in liquids being entrained in the raw product-gas stream and the operating upsets caused the termination of Test 62.

Post-run inspection showed that the prequench tower was very clean, and all of its parts were intact. The quench tower trays were pulled: They were relatively clean except for the bottom six trays, which were badly corroded. The quench separator was clean except for a crust build-up on the dished head on the oil weir side of the vessel.

The reactor was inspected after the test and was found to be free of deposits in the slurry dryer bed and in the first- and second-stage gasifiers. The two solids transfer lines contained some solids but were easily cleared by nitrogen purges. Three clinkers were found in the steam-oxygen gasifier above the steam-oxygen sparger. A high-alumina-content brick used in sealing up the Manhole 4 area had fallen into the steam-oxygen sparger distributor and was lodged across the top of two of the 15 sparger outlets. Exactly when the brick fell was not evident. The brick on the sparger distributor could have caused a maldistribution of the gases and precipitated the clinker formations found in the steam-oxygen bed after the test.

The pretreater was found to be clean after Test 62. A few small clinkers, which were hard but brittle, were found in the southeast quadrant of the pretreater. No grid nozzles were broken. During a routine pressure test, a small leak found on one of the internal cooling coils in the pretreater was repaired. Reddish brown, high-ash-content clinkers were found in the char cooler around the fluidizing ring area. Tar had accumulated in the venturi scrubber and in the pretreater quench system, similar to accumulations during previous tests. The quench tower was very clean with no significant deposits of tar or coal.

Test 63, June 1977

Highlights

The objective of Test 63 was to conduct a long, steady-state test at high coal conversions using Peabody No. 10 mine bituminous coal. Test 63 included 7 days of self-sustained operation, and carbon conversions of around 80%.

Details

Light-off for Test 63 occurred at 1650 hours on June 17. Pretreated coal was introduced to the reactor at 1645 hours on June 19. Some solids flow problems were encountered in establishing the fluidized bed in the steam-oxygen gasifier. The reactor became self-sustained at 1300 hours on June 21 when smooth solids flow was established throughout the reactor and the start-up burner was shut off and hydrogen flow was stopped. The steam-oxygen gasifier was maintained at temperatures ranging from 1600^o to 1680^oF during steady-state operations.

Solids feed to the reactor was interrupted 5 times during Test 63 because of solids flow problems in the reactor, which lasted from 2 to 8 hours. Interruptions were caused by the sluggish flow of solids from the second-stage gasifier to the steam-oxygen gasifier, the difficulty encountered in reestablishing a fluidized bed in the steam-oxygen gasifier, and the sudden loss of the bed in the steam-oxygen gasifier due to problems with the level control on the spent-char slurry pot. During each interruption, oxygen feed to the steam-oxygen sparger was shut off. Gasification was successfully restarted after these interruptions when oxygen was introduced. A total of 266 tons of pretreated char was fed to the reactor over a period of 139 hours.

Pretreater operation was very satisfactory during the test. Four hundred and twelve tons of screened bituminous coal were processed through the pretreater. Pretreatment operation began at 0830 hours on June 19 and continued for more than 10 days, reliably supplying nonagglomerating char feed for the reactor. Pretreater temperature ranged between 750° and $760^{\circ}F$. Coal feed to the pretreater was interrupted twice: once when the 60-ton coal storage hopper bottom feeder was plugged and once when the vent valve on the lockhopper feeding system was plugged. These interruptions were quickly corrected with a minimum loss of feed.

The reactor quench system caused some level control problems early in Test 63. There were indications that the gas line from the prequench tower to the quench tower was plugged. However, this section operated adequately during the remainder of the test.

When the purification section was brought on-line, foaming was evident. It was successfully controlled by the addition of an antifoam agent, the Union Carbide polyglycol UCON SOHB-5100. The purification section was taken out of service when the amine filter elements had to be changed and again when a leak developed in the minimum flow bypass line around the high-pressure lean-amine pump. The methanation section was kept on hot standby but was not put on-line because of the interruptions in the purification section operation.

Toward the end of Test 63, the movement of valve 339, erratic bed temperatures, and sluggish solids flow patterns to and from the steam-oxygen gasifier indicated that clinkers may have formed in the steam-oxygen gasifier. The test was terminated at 1300 hours on June 28 after 7 days of self-sustained operation, when solids could not be transferred out of the reactor and from the second-stage gasifier to the steam-oxygen gasifier.

Post-run inspection of the plant indicated that the coal-handling area was in good shape. The coal mill was clean and the Sweco screener was intact. However, after the 60-ton raw-coal storage hopper was emptied dry caked coal was found near the top of the live bottom section covering 25% of the open area. This deposit was cleaned.

The pretreater reactor was in excellent conditions. A few small pieces of agglomerated coal were found on the gas distributor grid. A check of the gas distributor nozzles showed that they were all clean. Dust that appeared to contain a large amount of ash was also found along the southeast wall of the pretreater. The cyclone diplegs and the hot-char transfer lines to the char cooler were clean. The cooling coil in the pretreater was pressure-tested and found to be in good condition.

A hard clinker was found in the bottom of the char cooler below the solids discharge standpipe. To eliminate this clinker formation, changes were made to mix equal amounts of nitrogen and air for fluidizing gas to the char cooler. The rest of the char cooler was in satisfactory conditions. The pretreater quench tower and the venturi scrubber were in good condition, with the usual amount of coal and tar accumulations. They were cleaned.

An inspection showed that the slurry preparation section was in good condition. When the reactor was opened, the slurry dryer area was found to be clean, containing only the usual amount of dust. Line 321 from the slurry dryer to the lift line and the spouting bed zone of the first-stage gasifier were clear. However, the solids downcomer from the spouting bed to the second-stage gasifier was plugged from the expansion joint down. This plug was believed to have formed during shutdown, because there had been no indication of any plug during Test 63. The plug was cleared by rodding and blasting with nitrogen.

The second-stage gasifier was clean except for the two large pieces of refractory found lying on the grid area. They were approximately 21 x 8 x 2 inches and 10 x 6 x 3 inches. This spalled refractory fell from the freeboard area of the gasifier and could have caused poor gas distribution in the second-stage gasifier. When it fell cannot be deduced from the operating data. The steam-oxygen gasifier had a clinker formation above the gas sparger. Two major clinker areas were found along the southeast and southwest walls of the steam-oxygen gasifier. The one clinker extended up to and around the solid transfer line 339 valve. The upper clinker was porous and spanned the entire steam-oxygen gasifier cross-sectional area. (The steam-oxygen sparger was clean.) Exactly when this clinker formation began is not known. It could have formed during upset conditions experienced in the steam-oxygen gasifier during the test.

The reactor high-pressure cyclone and its slurry pot were examined and found to be clean. When the quench system was inspected, a higher than normal solids buildup was found in the vessels. The prequench tower top tray was full of solids. The lines in this section were all taken apart and cleaned.

The purification section was opened up. The absorber was found to be clean except for the demister at the top of the tower; which was cleaned. An inspection of the regenerator revealed that the packing was in poor conditions.

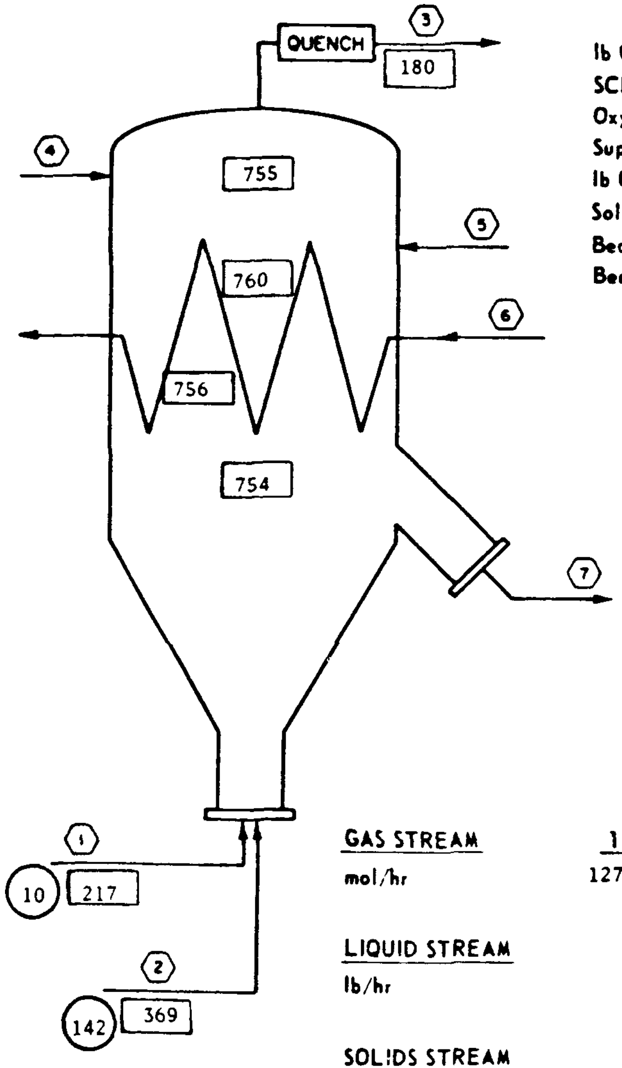
Results from Test 63 are presented in Figures 61 through 78 and Tables 23 through 25.

Table 23. MATERIAL BALANCE SUMMARY FOR THE PRETREATER SECTION FOR TEST 63 FROM 6/21/77 (1700 Hours) TO 6/22/77 (0600 Hours)

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

INPUT		C	H	O	N	S	Ar	Ash	Other	Total
Coal Feed	Wt % (Dry).	68.07	4.88	9.65	1.21	4.71		11.48		100
	Coal (Dry)	3167	227	449	56	219		534		4652
	Moisture		19	148						167
Streams to Pretreater	Air			852	2771		50			3673
	Steam		182	1452						1634
Nitrogen from purges					40					40
Air from purges				9	29					38
H_2O to venturi scrubber			2006	16,051						18,057
H_2O to quench tower			653	5225						5878
Air to char cooler				97	313		6			416
Cooling water to char cooler			98	783						881
TOTAL INPUT		3167	3185	25,066	3209	219	56	534		35,436
OUTPUT										
Pretreated Char to Gasifier	Wt.% (Dry)	67.83	3.49	8.72	1.36	4.30		14.30		100
	Char (Dry)	2596	134	334	52	165		547		3828
	Moisture		10	76						86
Slurry Waste from Quench	Wt.% (Dry)	56.60	2.61	11.27	1.45	4.10		23.97		100
	Solids (Dry)	118	5	24	3	9		50		209
	Tars & Oils	106	10	8	1	4				129
	H_2O & Dis materials	13	2719	21,745	2	42				24,521
Quench Tower Off-Gas	Total	151	284	2879	3314		56			6684
	Components: H_2		1							1
	CO_2	107		287						394
	C_2H_6	3	1							4
	N_2				3314					3314
	CH_4	9	3							12
	CO	32		43						75
	O_2			321						321
	Ar						56			56
	H_2O		279	2228						2507
TOTAL OUTPUT		2984	3162	25,066	3372	220	56	597		35,457
Net (Output – Input)		–183	–23	0	163	1	0	63		21
% Balance (Output/Input)		94	99	100	105	100	100	112		100

⬡ STREAM No.
○ PRESSURE, psig
□ TEMPERATURE,°F



lb Char/lb Coal = 0.78
SCF O_2/lb Coal= 2.2
Oxygen Breakthrough, % fed= 26.3
Superficial Velocity, ft/s= 0.91
lb O_2 Reacted/lb Coal= 0.14
Solids Residence Time, hr= 1.8
Bed Density, lb/cu ft = 20
Bed Height, ft = 10

GAS STREAM	3
COMPONENTS, mol %	
H_2	0.14
CO_2	3.17
C_2H_6	0.05
O_2	4.17
N_2	41.93
Ar	—
CH_4	0.26
CO	0.94
H_2O	49.34
mol/hr	282
mol wt	24

GAS STREAM	1	2
mol/hr	127	91

LIQUID STREAM	5	6
lb/hr	0	1636

SOLIDS STREAM	4	7
lb/hr	4652	3828

① AIR TO PRETREATER
② STEAM TO PRETREATER
③ PRETREATER OFF-GAS AFTER QUENCH
④ RAW COAL TO PRETREATER
⑤ DIRECT WATER QUENCH
⑥ COOLING WATER TO COOLING COIL IN PRETREATER
⑦ PRETREATER CHAR TO CHAR COOLER

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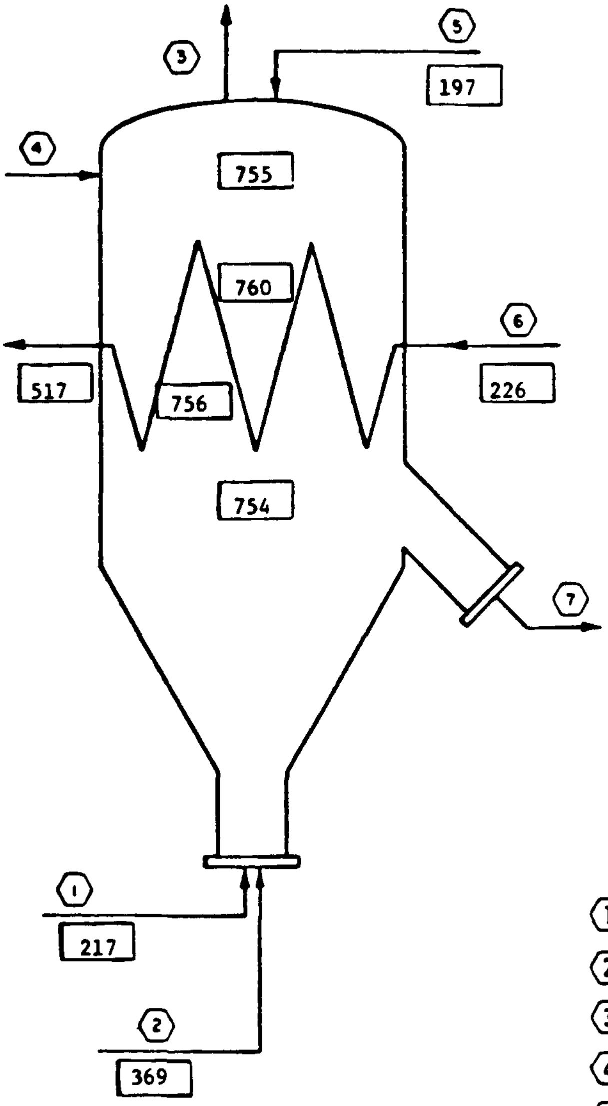
Figure 61. PRETREATMENT DATA FOR TEST 63 FOR STEADY PERIOD FROM 6/21/77 (1/00 Hours) TO 6/22/77 (0600 Hours)

⬡ Stream No.   □ Temperature, °F

Basis: 1 hour

Datum Condition: 77°F, 1 atm, material in standard state.



INPUT	Btu
Sensible Heat (Streams 1, 2, 4, 5, 6)	630,728
Heat of Combustion (Stream 4)	57,168,428
Steam Enthalpy (Streams 2 & 5)	2,803,496
Total	60,602,652
OUTPUT	
Sensible Heat (Streams 3 & 7)	3,204,197
Heat of Combustion (Streams 3 & 7)	50,192,711
Steam Enthalpy (Streams 3 & 6)	4,659,354
Total	58,056,262
% Balance 96	

① Air to Pretreater

② Steam to Pretreater

③ Pretreater Overhead

④ Raw Coal to Pretreater

⑤ Gas From Char Cooler

⑥ Cooling Water to Cooling Coil in Pretreater

⑦ Pretreated Char to Char Cooler

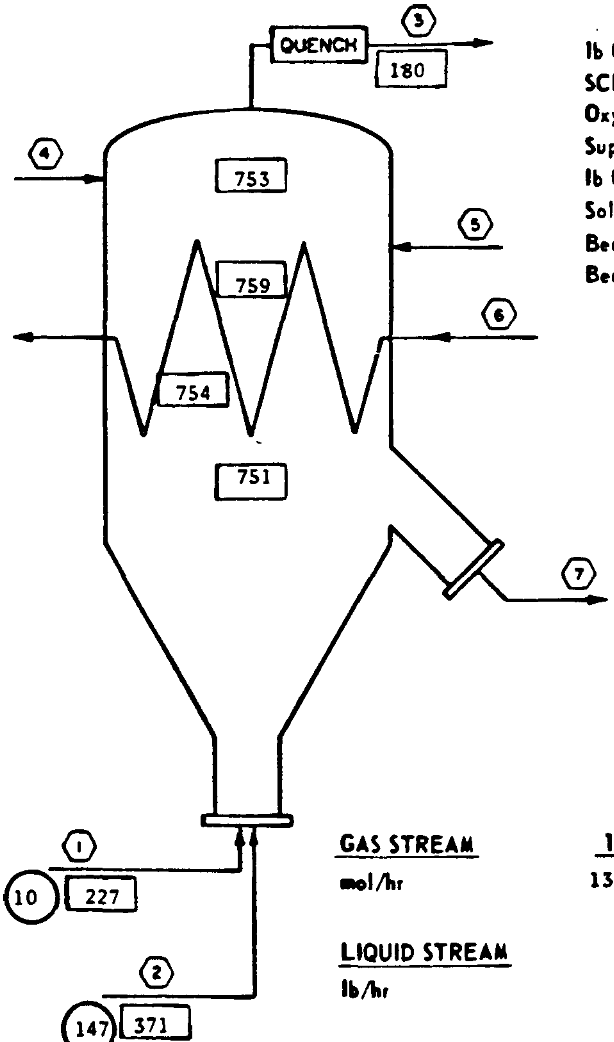
Figure 62. PRETREATER HEAT BALANCE DATA SHEET FOR TEST 63 FOR STEADY PERIOD FROM 6/21/77 (1700 Hours) TO 6/22/77 (0600 Hours)

Table 24. MATERIAL BALANCE SUMMARY FOR THE PRETREATER SECTION FOR TEST 63 FROM 6/25/77 (0100 Hours) TO 6/25/77 (1200 Hours)

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

INPUT		C	H	O	N	S	Ar	Ash	Other	Total
Coal Feed	Wt % (Dry)	67.50	4.95	9.63	1.26	4.89		11.77		100
	Coal (Dry)	3312	243	472	62	240		578		4907
	Moisture		19	154						173
Streams to Pretreater	Air			879	2856		52			3787
	Steam		198	1582						1780
Nitrogen from purges					32					32
Air from purges				9	29					38
H_2O to venturi scrubber			2006	16,050						18,056
H_2O to quench tower			653	5221						5874
Air to char cooler				130	424		8			562
Cooling water to char cooler			95	756						851
TOTAL INPUT		3312	3214	25,253	3403	240	60	578		36,060
OUTPUT										
Pretreated Char to Gasifier	Wt.% (Dry)	67.37	3.53	8.44	1.39	4.35		14.92		100
	Char (Dry)	2562	134	321	53	165		567		3802
	Moisture		6	48						54
Slurry Waste from Quench	Wt.% (Dry)	57.00	2.56	11.62	1.49	4.33		23.00		100
	Solids (Dry)	137	6	28	4	10		55		240
	Tars & Oils	117	12	11	1	4				145
	H_2O & Dis. materials	25	2731	21,847	3	49				24,655
Quench Tower Off-Gas	Total	291	291	2998	3392		58			7030
	Components: H_2		1							1
	CO_2	156		416						572
	C_2H_6	3	1							4
	N_2				3392					3392
	CH_4	5	2							7
	CO	127		169						296
	O_2			120						120
	Ar						58			58
	H_2O		287	229						2580
TOTAL OUTPUT		3132	3180	25,25	3453	228	58	622		35,926
Net (Output − Input)		−180	−34	0	50	−12	−2	44		−13
% Balance (Output/Input)		95	99	100	101	95	97	108		100

⬡ STREAM No.
○ PRESSURE, psig
☐ TEMPERATURE,°F



lb Char/lb Coal = 0.76
SCF O_2/lb Coal= 2.1
Oxygen Breakthrough, % fed= 0
Superficial Velocity, ft/s = 0.95
lb O_2 Reacted/lb Coal= 0.18
Solids Residence Time, hr= 1.5
Bed Density, lb/cu ft = 19
Bed Height, ft = 9

GAS STREAM	3
COMPONENTS, mol %	
H_2	0.05
CO_2	4.42
C_2H_6	0.04
O_2	1.28
N_2	41.22
Ar	0.49
CH_4	0.14
CO	3.59
H_2O	48.77
mol/hr	294
mol wt	24

GAS STREAM	1	2
mol/hr	131	99

LIQUID STREAM	5	6
lb/hr	0	1906

SOLIDS STREAM	4	7
lb/hr	4907	3802

① AIR TO PRETREATER
② STEAM TO PRETREATER
③ PRETREATER OFF-GAS AFTER QUENCH
④ RAW COAL TO PRETREATER
⑤ DIRECT WATER QUENCH
⑥ COOLING WATER TO COOLING COIL IN PRETREATER
⑦ PRETREATER CHAR TO CHAR COOLER

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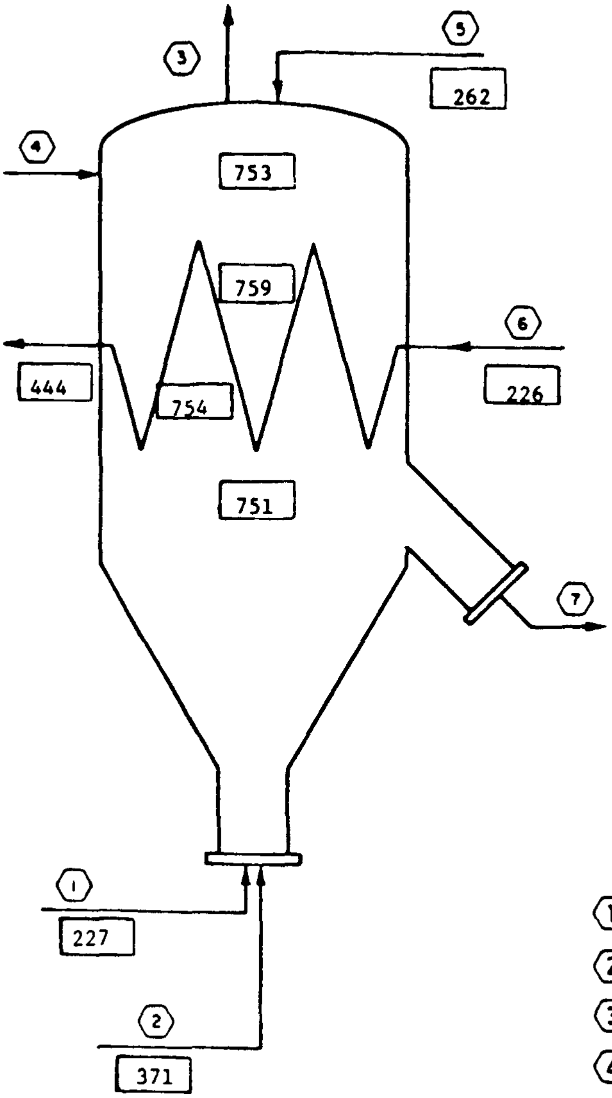
Figure 63. PRETREATMENT DATA FOR TEST 63 FOR STEADY PERIOD FROM 6/25/77 (0100 Hours) TO 6/25/77 (1200 Hours)

○ Stream No. ☐ Temperature, °F

Basis: 1 hour

Datum Condition: 77°F, 1 atm,
material in standard state.



INPUT	Btu
Sensible Heat (Streams 1, 2, 4, 5, 6)	741,150
Heat of Combustion (Stream 4)	59,948,819
Steam Enthalpy (Streams 2 & 5)	3,006,410
Total	63,696,379
OUTPUT	
Sensible Heat (Streams 3 & 7)	3,310,893
Heat of Combustion (Streams 3 & 7)	51,006,009
Steam Enthalpy (Streams 3 & 6)	5,002,884
Total	59,339,786
% Balance	93

① Air to Pretreater

② Steam to Pretreater

③ Pretreater Overhead

④ Raw Coal to Pretreater

⑤ Gas From Char Cooler

⑥ Cooling Water to Cooling Coil in Pretreater

⑦ Pretreated Char to Char Cooler

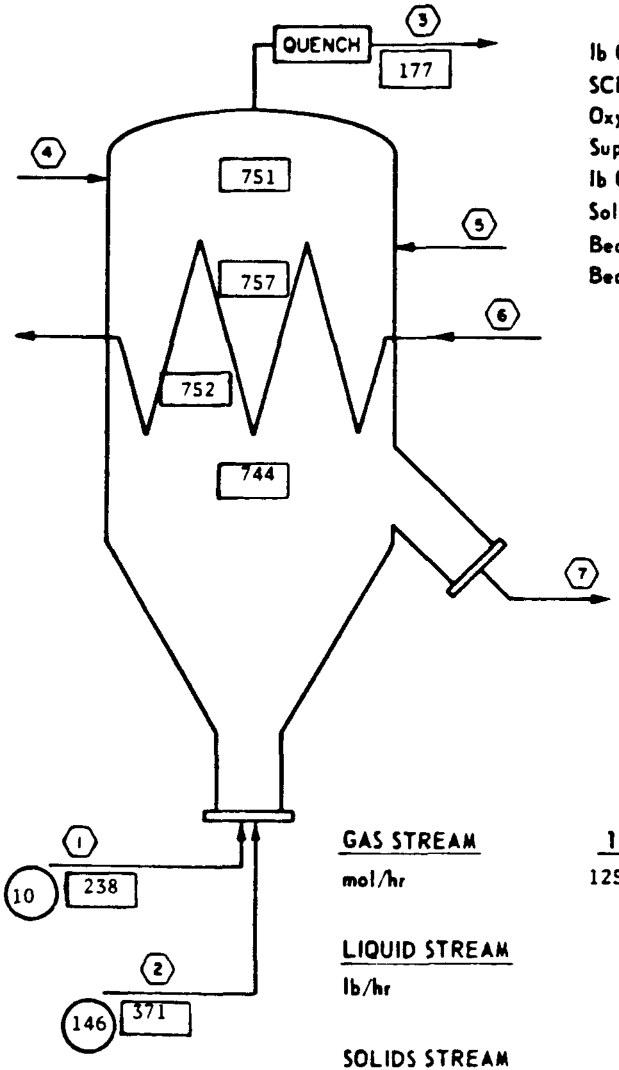
Figure 64..PRETREATER HEAT BALANCE DATA SHEET FOR TEST 63 FOR STEADY PERIOD FROM 6/25/77 (0100 Hours) TO 6/25/77 (1200 Hours)

Table 25. MATERIAL BALANCE SUMMARY FOR THE PRETREATER SECTION FOR TEST 63 FROM 6/25/77 (1400 Hours) TO 6/25/77 (1700 Hours)

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

INPUT		C	H	O	N	S	Ar	Ash	Other	Total
Coal Feed	Wt % (Dry)	67.50	4.82	10.04	1.25	4.72		11.67		100
	Coal (Dry)	3166	226	471	59	221		547		4690
	Moisture		18	147						165
Streams to Pretreater	Air			837	2719		49			3605
	Steam		195	1563						1758
Nitrogen from purges					33					33
Air from purges				9	29					38
H_2O to venturi scrubber			2006	16,048						18,054
H_2O to quench tower			653	5225						5878
Air to char cooler				136	442		8			586
Cooling water to char cooler			71	565						636
TOTAL INPUT		3166	3169	25,001	3282	221	57	547		35,443
OUTPUT										
Pretreated Char to Gasifier	Wt.% (Dry)	66.80	3.43	9.17	1.53	4.33		14.74		100
	Char (Dry)	2505	129	344	57	162		553		3750
	Moisture		9	75						84
Slurry Waste from Quench	Wt.% (Dry)	56.90	2.41	11.91	1.32	4.41		23.05		100
	Solids (Dry)	91	4	19	2	7		37		160
	Tars & Oils	102	11	10	1	3				127
	H_2O & Dis materials	25	2750	21,991	3	50				24,819
Quench Tower Off-Gas	Total	285	238	2562	3327		57			6469
	Components: H_2		1							1
	CO_2	153		407						560
	C_2H_6	3	1							4
	N_2				3327					3327
	CH_4	5	2							7
	CO	124		166						290
	O_2			118						118
	Ar						57			57
	H_2O		234	1871						2105
TOTAL OUTPUT		2008	3141	25,001	3390	222	57	590		35,409
Net (Output - Input)		–158	–28	0	108	–1	0	43		–34
% Balance (Output/Input)		95	99	100	103	100	100	108	.	100

⬡ STREAM No.
○ PRESSURE, psig
▭ TEMPERATURE, °F



lb Char/lb Coal =	0.76
SCF O_2/lb Coal=	2.1
Oxygen Breakthrough, % fed=	0
Superficial Velocity, ft/s =	0.92
lb O_2 Reacted/lb Coal=	0.18
Solids Residence Time, hr =	1.3
Bed Density, lb/cu ft =	21
Bed Height, ft =	7

GAS STREAM	3
COMPONENTS, mol %	
H_2	0.06
CO_2	4.81
C_2H_6	0.04
O_2	1.39
N_2	44.90
Ar	0.54
CH_4	0.16
CO	3.91
H_2O	44.19
mol/hr	265
mol wt	24

GAS STREAM	1	2
mol/hr	125	98

LIQUID STREAM	5	6
lb/hr	0	1546

SOLIDS STREAM	4	7
lb/hr	4690	3750

① AIR TO PRETREATER
② STEAM TO PRETREATER
③ PRETREATER OFF-GAS AFTER QUENCH
④ RAW COAL TO PRETREATER
⑤ DIRECT WATER QUENCH
⑥ COOLING WATER TO COOLING COIL IN PRETREATER
⑦ PRETREATER CHAR TO CHAR COOLER

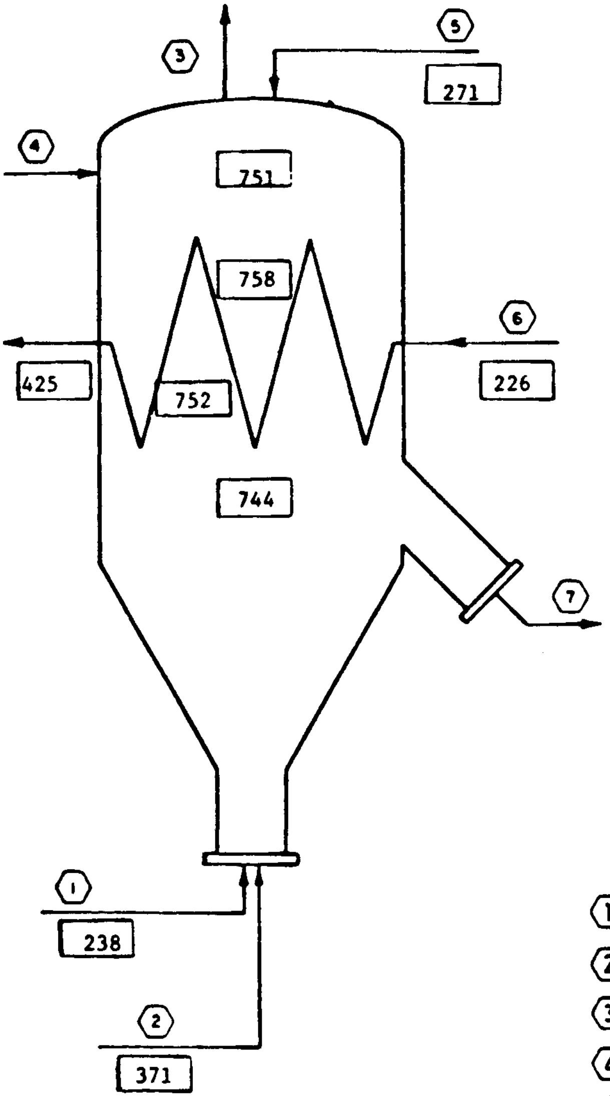
A77051073

Figure 65. PRETREATMENT DATA FOR TEST 63 FOR STEADY PERIOD FROM 6/25/77 (1400 Hours) TO 6/25/77 (1700 Hours)

⬡ Stream No. ☐ Temperature, °F

Basis: 1 hour

Datum Condition: 77°F, 1 atm,
material in standard state.



INPUT	Btu
Sensible Heat (Streams 1, 2, 4, 5, 6)	669,747
Heat of Combustion (Stream 4)	57,391,530
Steam Enthalpy (Streams 2 & 5)	2,728,176
Total	60,789,453
OUTPUT	
Sensible Heat (Streams 3 & 7)	3,080,344
Heat of Combustion (Streams 3 & 7)	48,970,249
Steam Enthalpy (Streams 3 & 6)	4,087,094
Total	56,137,687
% Balance 92	

① Air to Pretreater

② Steam to Pretreater

③ Pretreater Overhead

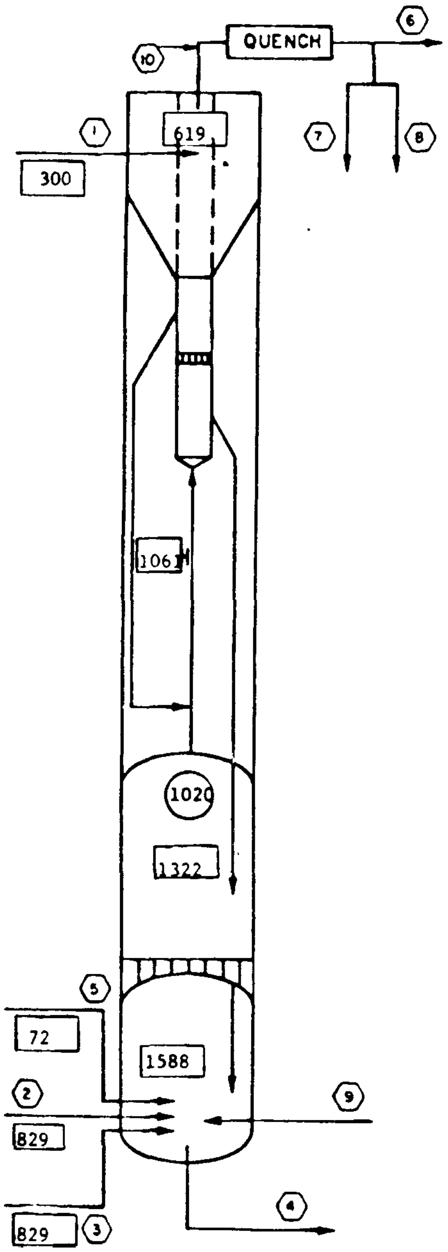
④ Raw Coal to Pretreater

⑤ Gas From Char Cooler

⑥ Cooling Water to Cooling Coil in Pretreater

⑦ Pretreated Char to Char Cooler

Figure 66. PRETREATER HEAT BALANCE DATA SHEET FOR TEST 63 FOR STEADY PERIOD FROM 6/25/77 (1400 Hours) TO 6/25/77 (1700 Hours)



MATERIAL BALANCE, %

C
H
O
ASH

⬡ STREAM No.
○ PRESSURE, psig
☐ TEMPERATURE, °F

	GAS STREAMS	
	6	10
mol % (dry)		
H_2	28.77	24.23
CO_2	28.94	36.63
C_2H_6	0.59	0.70
Ar	—	—
N_2	13.42	11.87
H_2S	1.24	1.59
CH_4	21.54	20.30
CO	5.50	4.68
mol/hr (dry)	124	152

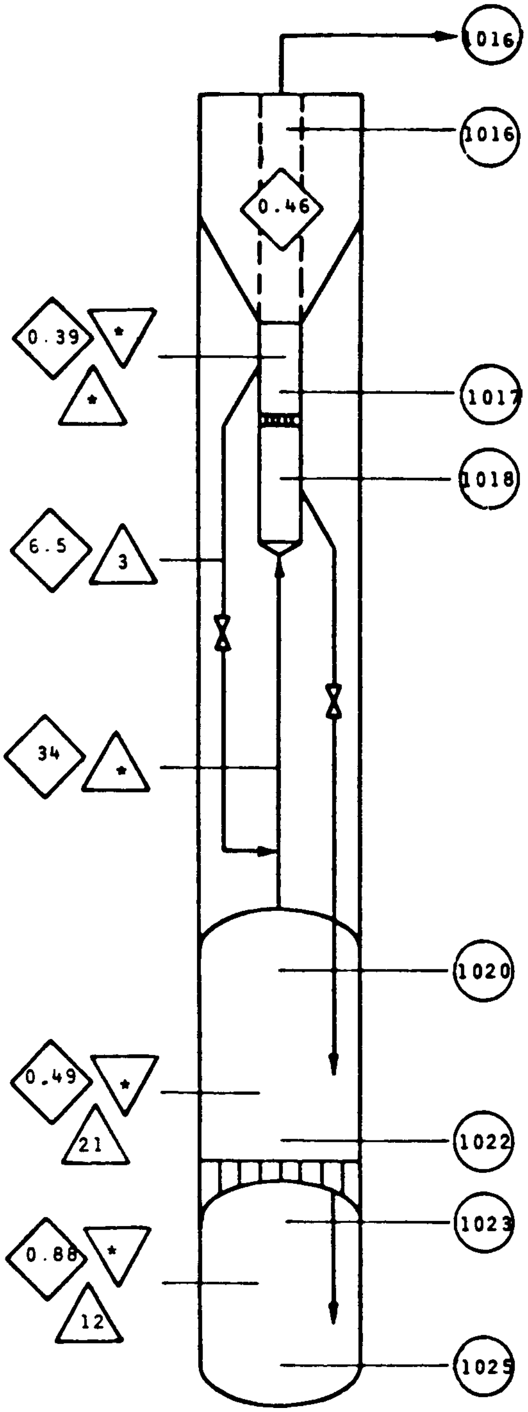
	SOLIDS STREAMS		
	1	10	4
wt % (dry)			
C	67.00	77.10	69.20
H	3.36	2.60	1.28
N	1.29	1.17	0.55
Cl	--	--	--
S	4.52	2.50	1.39
ASH	14.98	12.59	26.70
O	8.85	4.04	0.88
lb/hr (dry)	3889		
MOISTURE, wt %	2.0		
SLURRY CONCN, wt %	29		

	LIQUID STREAMS		
	7	8	1
	lb/hr		
H_2O	7898	--	--
LIGHT OIL	--	--	9954

GAS FEED STREAMS			
2	3	5	9
mol/hr			
366	83	24	0

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 67. HYGAS REACTOR DATA FOR TEST 63 FOR STEADY PERIOD FROM 6/21/77 (1700 Hours) TO 6/21/77 (2300 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
Carbon (net) = total carbon in – carbon in overhead

lb Oxygen/lb Carbon (net) = 0.35
lb Steam/lb Carbon (net) = 3.6
lb Oxygen/lb Coal Fed= 0.20
lb Steam/lb Coal Fed= 2.1
lb Hydrogen/lb Coal Fed= 0
lb Coal Fed/1000 SCF Product Gas= 135

By Ash Balance

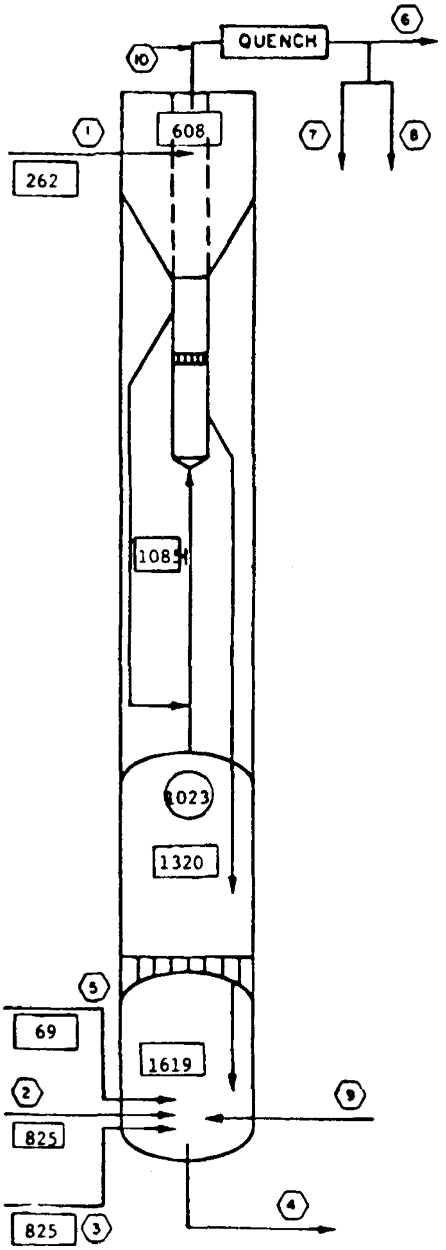
Coal Gasified, %= 52
Carbon Gasified, %= 42

Methane Yield SCF/lb coal fed= 3.0
Equivalent Methane Yield, SCF/lb coal fed = 4.3

Bed Height, ft

Slurry Dryer= *
HTR= 14
SOG= 22

Figure 68. HYGAS REACTOR ENGINEERING DATA FOR TEST 63 FOR STEADY PERIOD FROM 6/21/77 (1700 Hours) TO 6/21/77 (2300 Hours)



⬡ STREAM No.
○ PRESSURE, psig
☐ TEMPERATURE, °F

MATERIAL BALANCE, %

C
H
O
ASH

	GAS STREAMS	
	6	10
mol % (dry)		
H_2	29.63	26.59
CO_2	30.76	35.59
C_2H_6	0.63	0.69
Ar	--	--
N_2	10.04	9.60
H_2S	1.06	1.26
CH_4	21.69	20.78
CO	6.19	5.49
mol/hr (dry)	136	157

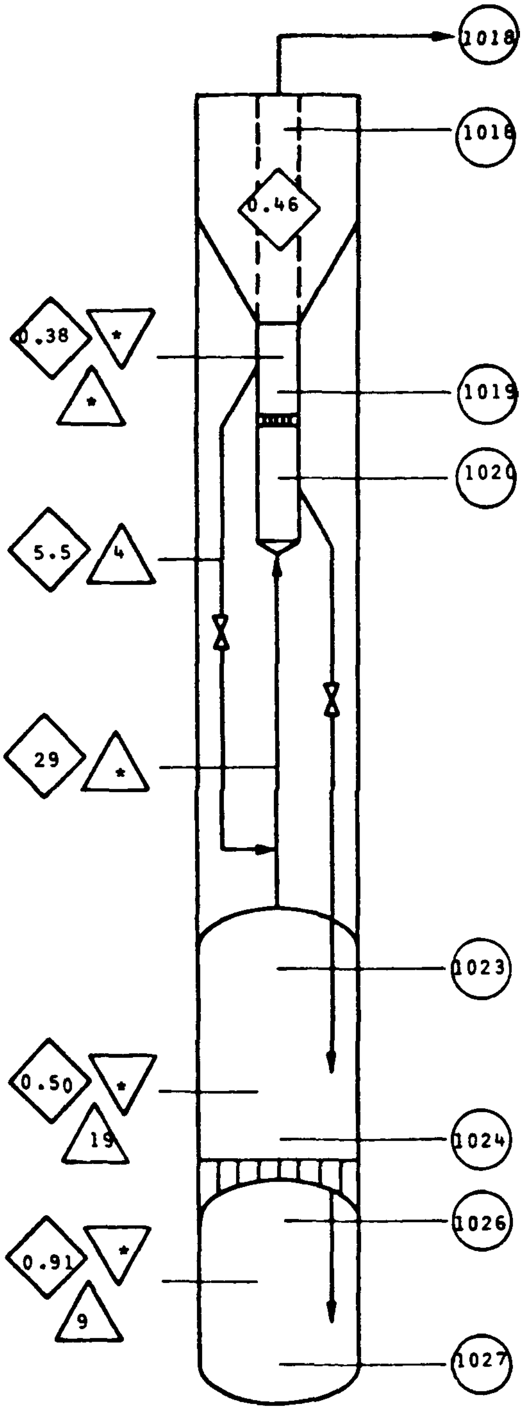
	SOLIDS STREAMS		
	1	10	4
wt % (dry)			
C	67.70	76.20	64.60
H	3.51	3.37	1.16
N	1.37	1.15	0.42
Cl	--	--	--
S	4.26	2.98	1.04
ASH	14.49	13.23	32.08
O	8.67	3.07	0.70
lb/hr (dry)	3664		
MOISTURE, wt %	2.0		
SLURRY CONCN, wt %	26		

	LIQUID STREAMS		
	7	8	1
	lb/hr		
H_2O	7754	--	--
LIGHT OIL	--	--	10,855

GAS FEED STREAMS			
2	3	5	9
mol/hr			
370	83	26	0

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 69. HYGAS REACTOR DATA FOR TEST 63 FOR STEADY PERIOD FROM 6/22/77 (0200 Hours) TO 6/22/77 (0800 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

Carbon (net) = total carbon in – carbon in overhead

lb Oxygen/lb Carbon (net) = 0.36
lb Steam/lb Carbon (net) = 3.5
lb Oxygen/lb Coal Fed= 0.23
lb Steam/lb Coal Fed= 2.2
lb Hydrogen/lb Coal Fed= 0
lb Coal Fed/1000 SCF Product Gas= 115

By Ash Balance

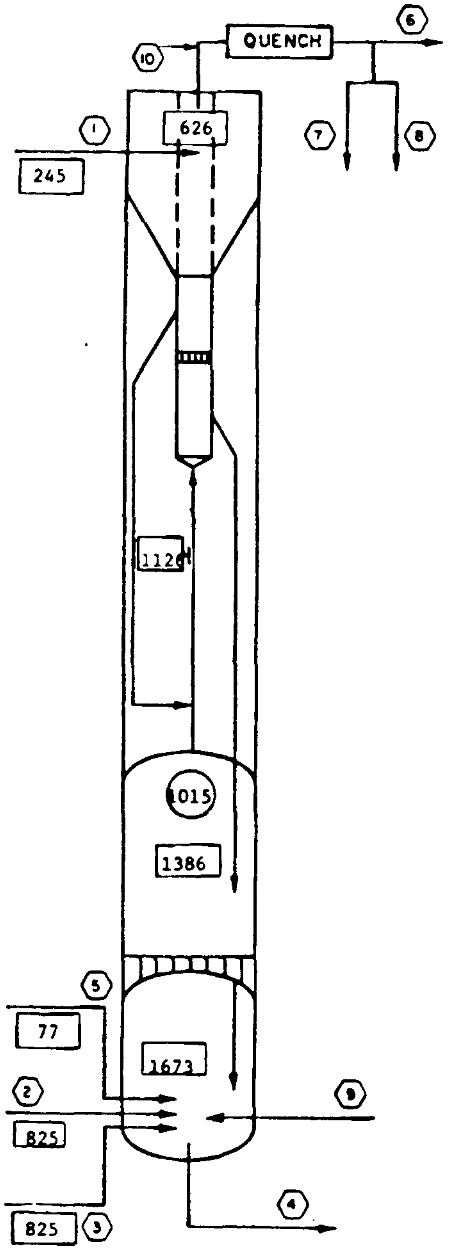
Coal Gasified, %= 64
Carbon Gasified, %= 57

Methane Yield SCF/lb coal fed= 3.4
Equivalent Methane Yield, SCF/lb coal fed = 4.9

Bed Height, ft

Slurry Dryer= *
HTR= 16
SOG= 27

Figure 70. HYGAS REACTOR ENGINEERING DATA FOR TEST 63 FOR STEADY PERIOD FROM 6/22/77 (0200 Hours) TO 6/22/77 (0800 Hours)



MATERIAL BALANCE, %

C

H

O

ASH

	GAS STREAMS	
	6	10
mol % (dry)		
H_2	28.92	25.92
CO_2	31.37	36.19
C_2H_6	0.59	0.68
Ar	--	--
N_2	8.54	8.18
H_2S	1.11	1.40
CH_4	21.40	20.52
CO	8.07	7.11
mol/hr (dry)	189	218

	SOLIDS STREAMS		
	1	10	4
wt % (dry)			
C	66.87	76.50	62.66
H	3.37	2.59	1.06
N	1.45	1.18	0.38
Cl	--	--	--
S	4.29	2.79	1.02
ASH	15.56	13.67	34.13
O	8.46	3.27	0.75
lb/hr (dry)	3999		
MOISTURE, wt %	1.7		
SLURRY CONCN, wt %	30		

	LIQUID STREAMS		
	7	8	1
	lb/hr		
H_2O	6778	--	--
LIGHT OIL	--	--	9737

GAS FEED STREAMS			
2	3	5	9
mol/hr			
359	85	29	0

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

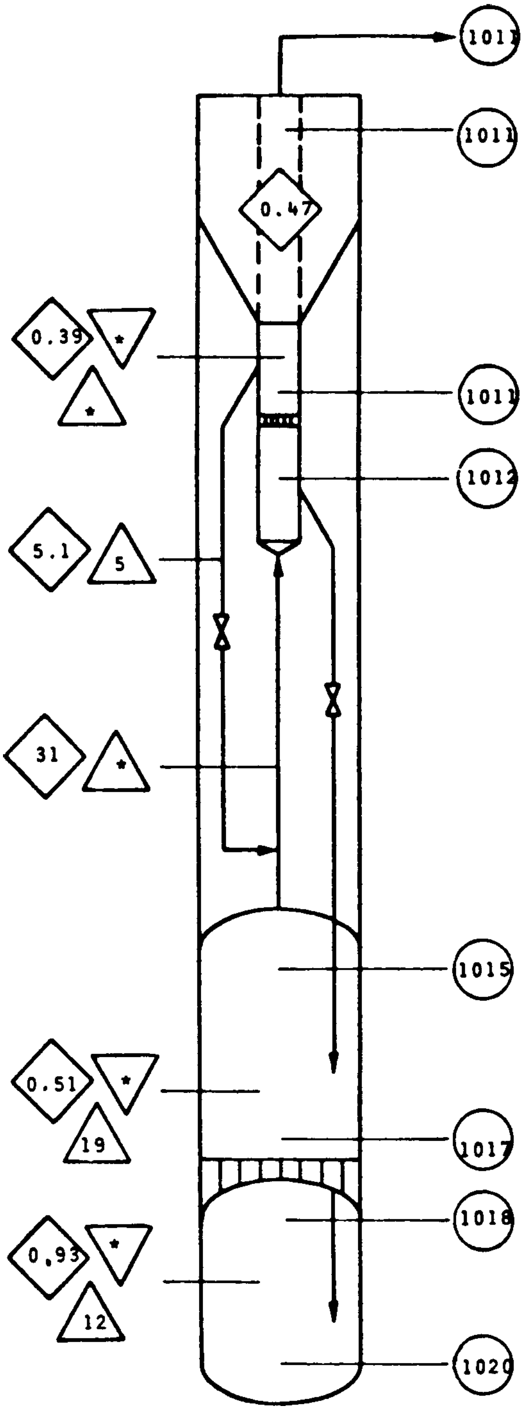
⑦ WATER MADE

⑧ LIGHT OIL TO RECYCLE

⑨ GASES FROM EXTERNAL HEATER

⑩ REACTOR OVERHEAD

Figure 71. HYGAS REACTOR DATA FOR TEST 63 FOR STEADY PERIOD FROM 6/23/77 (1800 Hours) TO 6/24/77 (1300 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
Carbon (net) = total carbon in – carbon in overhead

lb Oxygen/lb Carbon (net) = 0.41
lb Steam/lb Carbon (net) = 3.5
lb Oxygen/lb Coal Fed= 0.23
lb Steam/lb Coal Fed= 2.0
lb Hydrogen/lb Coal Fed= 0
lb Coal Fed/1000 SCF Product Gas= 89

By Ash Balance

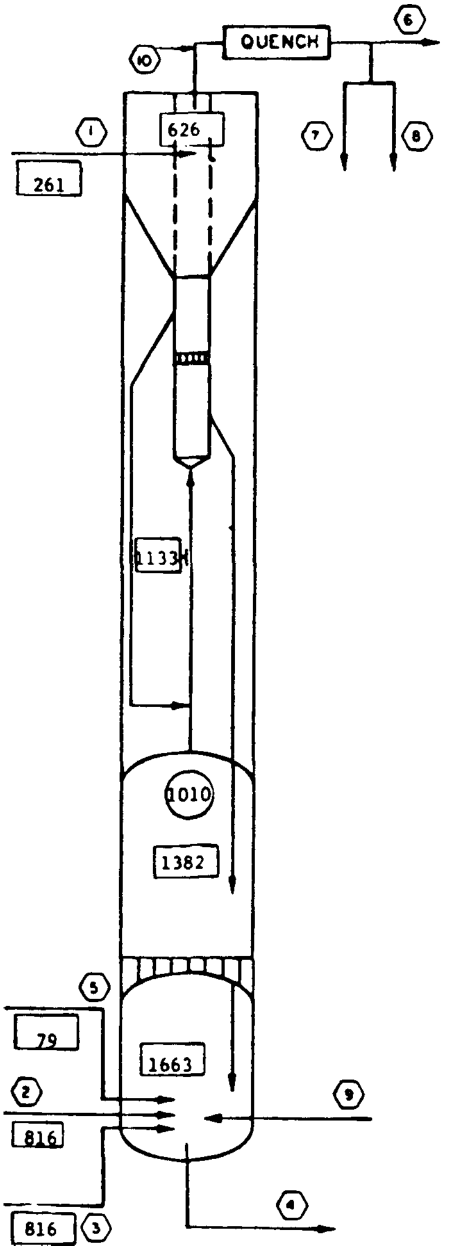
Coal Gasified, %= 64
Carbon Gasified, %= 57

Methane Yield SCF/lb coal fed= 4.2
Equivalent Methane Yield, SCF/lb coal fed = 6.2

Bed Height, ft

Slurry Dryer= *
HTR= 15
SOG= 24

Figure 72. HYGAS REACTOR ENGINEERING DATA FOR TEST 63 STEADY PERIOD FROM 6/23/77 (1800 Hours) TO 6/24/77 (1300 Hours)



MATERIAL BALANCE, %

C

H

O

ASH

GAS STREAMS

	6	10
mol % (dry)		
H_2	29.69	26.51
CO_2	30.80	35.70
C_2H_6	0.59	0.67
Ar	—	—
N_2	8.81	8.52
H_2S	1.26	1.50
CH_4	20.80	19.95
CO	8.05	7.15
mol/hr (dry)	184	213

SOLIDS STREAMS

	1	10	4
wt % (dry)			
C	68.34	75.29	59.32
H	3.49	2.65	1.01
N	1.37	1.15	0.35
Cl	--	--	--
S	4.53	2.82	1.06
ASH	14.71	14.48	33.92
O	7.56	3.61	4.34
lb/hr (dry)	3810		
MOISTURE, wt %	2.1		
SLURRY CONCN, wt %	27		

LIQUID STREAMS

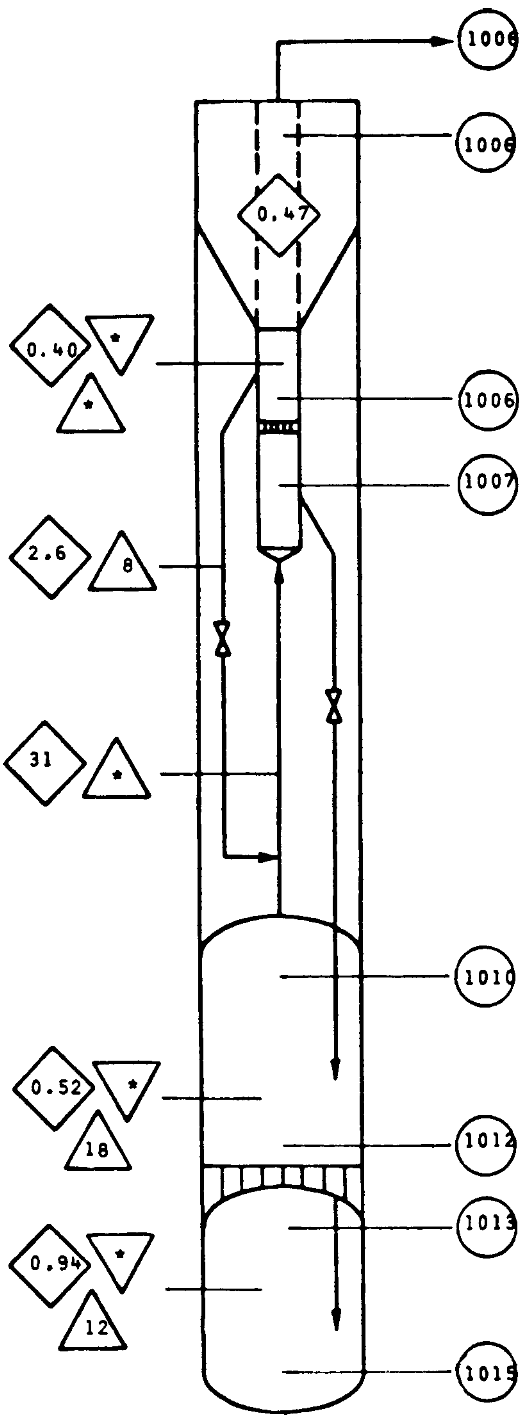
	7	8	1
	lb/hr		
H_2O	6946	--	—
LIGHT OIL	--	--	10,347

GAS FEED STREAMS

2	3	5	9
mol/hr			
365	86	30	0

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 73. HYGAS REACTOR DATA FOR TEST 63 FOR STEADY PERIOD FROM 6/23/77 (1800 Hours) TO 6/25/77 (1700 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
Carbon (net) = total carbon in – carbon in overhead

lb Oxygen/lb Carbon (net) = 0.46
lb Steam/lb Carbon (net) = 4.0
lb Oxygen/lb Coal Fed = 0.25
lb Steam/lb Coal Fed = 2.1
lb Hydrogen/lb Coal Fed = 0
lb Coal Fed/1000 SCF Product Gas = 87

By Ash Balance

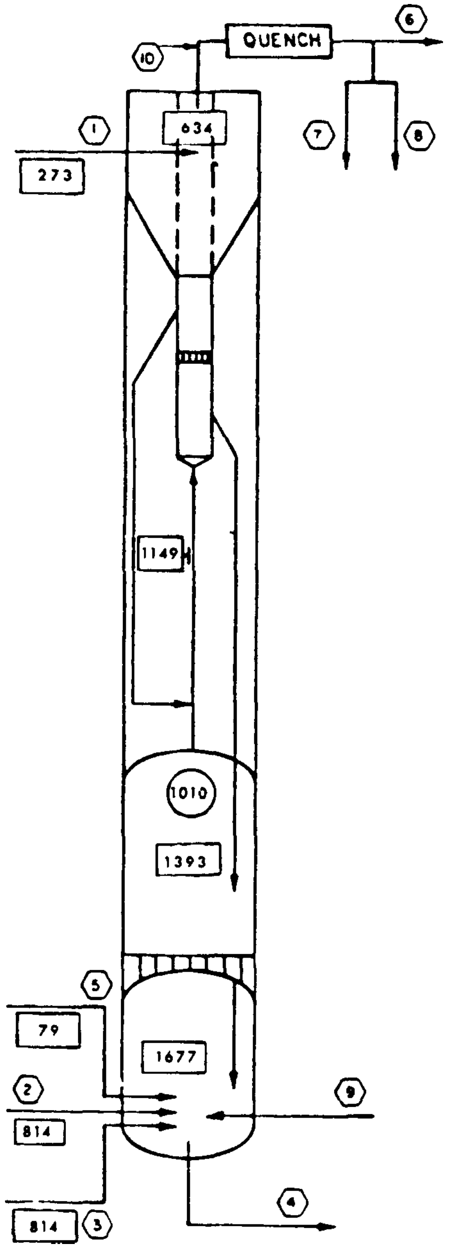
Coal Gasified, % = 66
Carbon Gasified, % = 62

Methane Yield SCF/lb coal fed = 4.2
Equivalent Methane Yield, SCF/lb coal fed = 6.3

Bed Height, ft

Slurry Dryer = *
HTR = 16
SOG = 23

Figure 74. HYGAS REACTOR ENGINEERING DATA FOR TEST 63 FOR STEADY PERIOD FROM 6/23/77 (1800 Hours) TO 6/25/77 (1700 Hours)



MATERIAL BALANCE, %

C

H

O

ASH

	GAS STREAMS	
	6	10
mol % (dry)		
H_2	30.06	26.87
CO_2	30.74	26.87
C_2H_6	0.58	0.64
Ar	—	—
N_2	8.68	8.18
H_2S	1.37	1.50
CH_4	20.49	19.74
CO	8.08	7.16
mol/hr (dry)	186	215

	SOLIDS STREAMS		
	1	10	4
wt % (dry)			
C	68.36	75.20	56.10
H	3.51	2.62	0.96
N	1.37	1.14	0.34
Cl	—	—	—
S	4.53	2.82	1.00
ASH	14.78	14.60	41.35
O	7.45	3.62	0.25
lb/hr (dry)	3834		
MOISTURE, wt %	2.1		
SLURRY CONCN, wt %	27		

	LIQUID STREAMS		
	7	8	1
	lb/hr		
H_2O	7300	—	—
LIGHT OIL	—	—	10,688

GAS FEED STREAMS			
2	3	5	9
mol/hr			
369	86	30	0

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

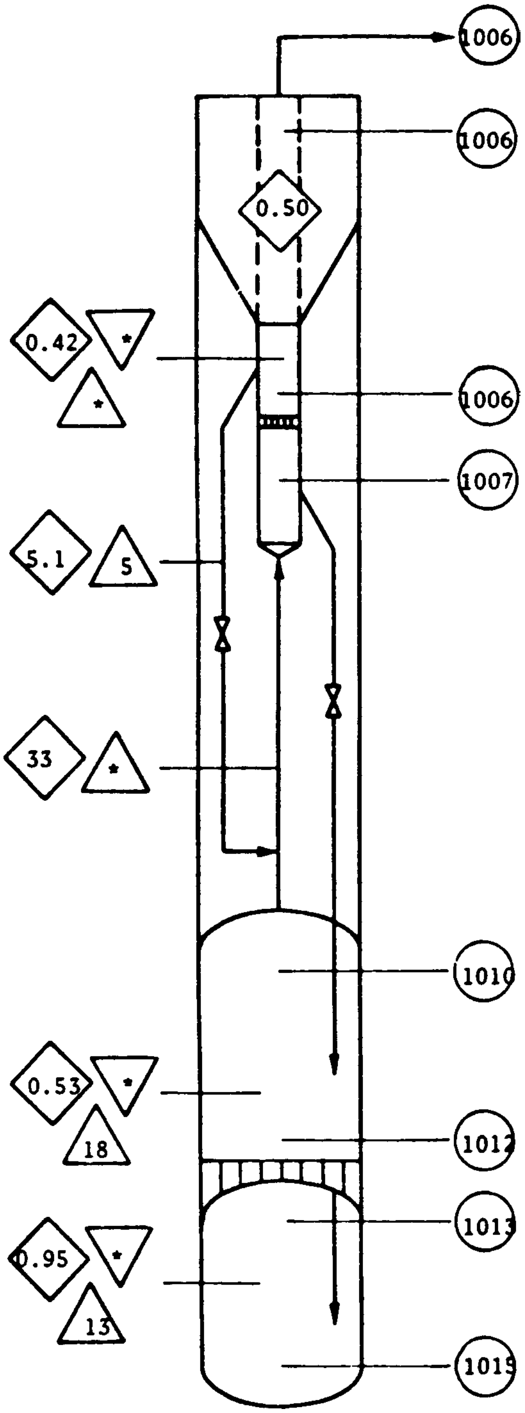
⑦ WATER MADE

⑧ LIGHT OIL TO RECYCLE

⑨ GASES FROM EXTERNAL HEATER

⑩ REACTOR OVERHEAD

Figure 75. HYGAS REACTOR DATA FOR TEST 63 FOR STEADY PERIOD FROM 6/24/77 (1400 Hours) TO 6/25/77 (1000 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
Carbon (net) = total carbon in – carbon in overhead

lb Oxygen/lb Carbon (net) = 0.49
lb Steam/lb Carbon (net) = 4.1
lb Oxygen/lb Coal Fed = 0.25
lb Steam/lb Coal Fed = 2.1
lb Hydrogen/lb Coal Fed = 0
lb Coal Fed/1000 SCF Product Gas = 86

By Ash Balance

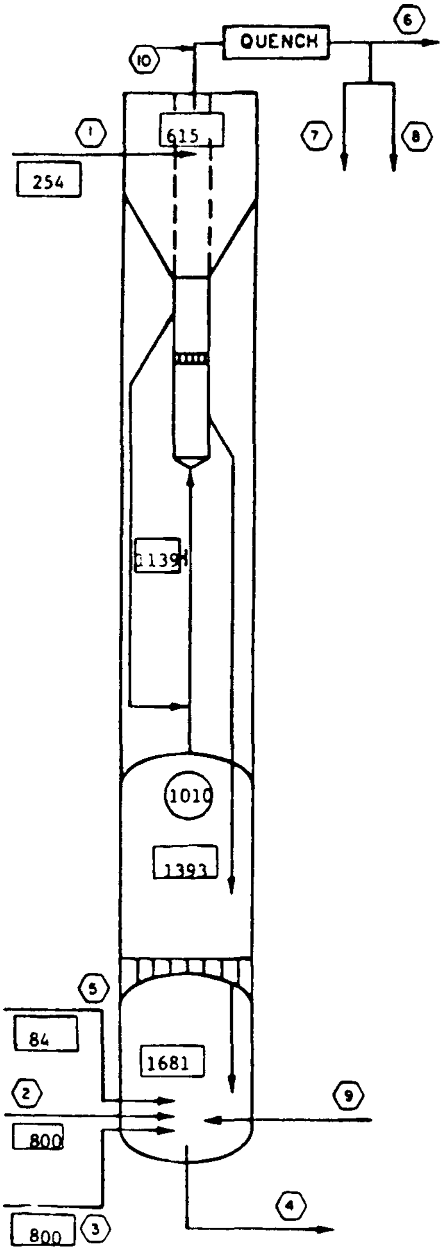
Coal Gasified, % = 75
Carbon Gasified, % = 71

Methane Yield SCF/lb coal fed = 4.2
Equivalent Methane Yield, SCF/lb coal fed = 6.2

Bed Height, ft

Slurry Dryer = *
HTR = 16
SOG = 22

Figure 76. HYGAS REACTOR ENGINEERING DATA FOR TEST 63 FOR STEADY PERIOD FROM 6/24/77 (1400 Hours) TO 6/25/77 (1000 Hours)



○ STREAM No.
○ PRESSURE, psig
□ TEMPERATURE, °F

MATERIAL BALANCE, %
C
H
O
ASH

	GAS STREAMS	
	6	10
mol % (dry)		
H_2	30.63	26.91
CO_2	30.16	36.11
C_2H_6	0.61	0.66
Ar	—	—
N_2	8.39	7.87
H_2S	1.22	1.38
CH_4	20.68	19.79
CO	8.31	7.28
mol/hr (dry)	182	215

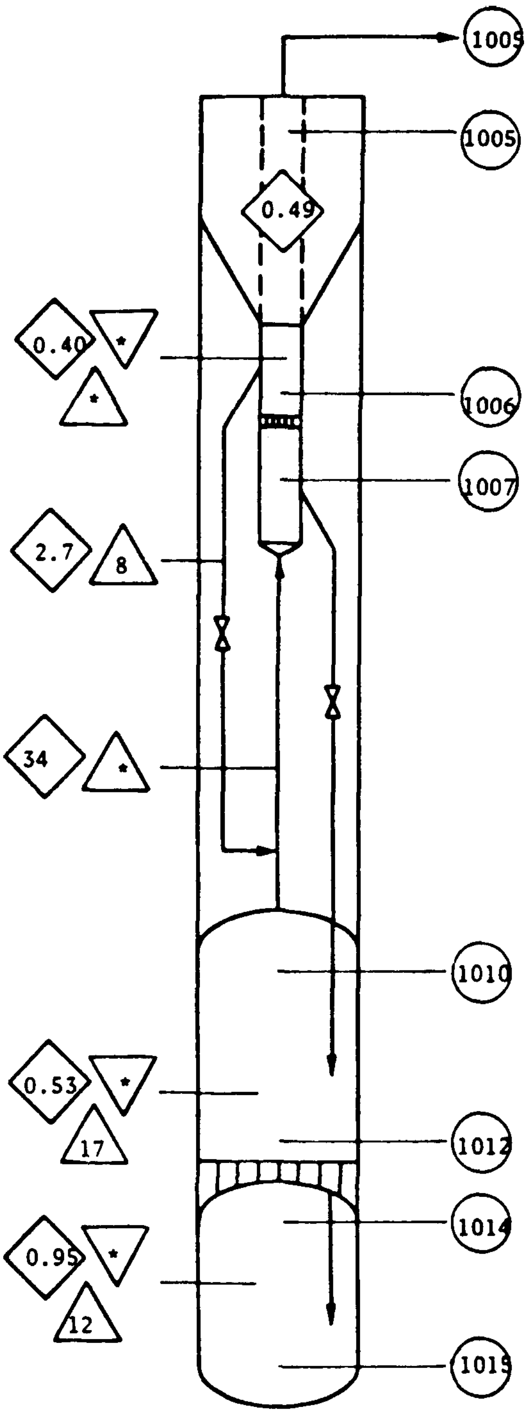
	SOLIDS STREAMS		
	1	10	4
wt % (dry)			
C	68.70	74.80	57.85
H	3.47	2.70	0.93
N	1.30	1.14	0.31
Cl	—	—	—
S	4.69	2.83	0.75
ASH	14.35	14.79	39.57
O	7.49	3.74	0.59
lb/hr (dry)	3702		
MOISTURE, wt %	2.4		
SLURRY CONCN, wt %	24		

	LIQUID STREAMS		
	7	8	1
	lb/hr		
H_2O	7042	--	--
LIGHT OIL	—	—	11,828

GAS FEED STREAMS			
2	3	5	9
mol/hr			
367	86	31	0

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 77. HYGAS REACTOR DATA FOR TEST 63 FOR STEADY PERIOD FROM 6/25/77 (1200 Hours) TO 6/25/77 (1700 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
Carbon (net) = total carbon in – carbon in overhead

lb Oxygen/lb Carbon (net) = 0.50
lb Steam/lb Carbon (net) = 4.1
lb Oxygen/lb Coal Fed = 0.27
lb Steam/lb Coal Fed = 2.2
lb Hydrogen/lb Coal Fed = 0
lb Coal Fed/1000 SCF Product Gas = 83

By Ash Balance

Coal Gasified, % = 74
Carbon Gasified, % = 69

Methane Yield SCF/lb coal fed = 4.4
Equivalent Methane Yield, SCF/lb coal fed = 6.5

Bed Height, ft

Slurry Dryer = *
HTR = 17
SOG = 23

Figure 78. HYGAS REACTOR ENGINEERING DATA FOR TEST 63 FOR STEADY PERIOD FROM 6/25/77 (1200 Hours) TO 6/25/77 (1700 Hours)

EFFLUENT TREATMENT AND WATER REUSE

Task 2 under this contract involved work in the effluent treatment section of the HYGAS plant. The performance of this equipment during individual hydrogasification tests is reported along with the other details of each run, in the previous section of this report. A summary of equipment performance and modifications in the effluent treatment section is given below.

During August of 1976, the Edens solids separator was modified: A top skimmer was added to the equipment to skim the floating solids that had been noted on top of the water layer during the previous tests with Illinois coal (which requires pretreatment). This top skimmer removes floating solids the same way that settled solids are removed from the Edens equipment (using a continuous chain-driven skimmer bar). It materially aids in preparing the liquid effluent from the Edens separator for filtration in the Alar filter.

Also during August 1976, a chemical sewer system was installed in the plant to replace the storm sewer system. This new system collects all effluents from drips and drains throughout the plant and conducts them to a common sump where a pump transports these liquid streams to the Edens separator for recovery of any solids and/or oil. This chemical sewer completes the cycle for identifying and recovering all of the effluent streams from the HYGAS Process.

During Test 58 (November, 1976) the light oil stripper (LOS) did not operate properly due to solids carryover from the stripping column into the condensor and separator. These solids plugged the separator, causing LOS shutdown. A probable reason for this solids carryover could be excessive gas velocity in the stripper caused by intermittent flows of raw product gas from the reactor cyclone slurry dust collection vessel. Improper detection of the liquid level in this vessel sometimes allowed the slurry liquid discharge valve to hang open, allowing process gas to bypass the normal purification train and enter the LOS section directly through the liquid discharge valve. An improved level detecting system in the cyclone slurry vessel was installed, and it was also decided to install a large surge vessel ahead of the LOS stripper tower, to smooth out fluctuations in both slurry concentration (liquid flow rate) and gas rate, to obtain better control of steady feed to the stripper section. The surge vessel was designed and ordered, and was received late in February. The concrete pads for the vessel and the circulation pump were poured during March, the vessels and equipment were installed during April, and instrumentation was completed during May. The new surge system was first operated during Test 63, when over-pressurization of the vessel blew a pump seal and the surge system was bypassed for the rest of the test. It was repaired and readied for Test 64.

Other work in the effluent treatment section included carrying out a LOS flow test during January, when flow meters and level controls were calibrated and checked for proper operation.

METHANATION TESTS

Task 3 under this contract involved methanation testing. The performance of the methanation section of the HYGAS pilot plant during individual hydrogasification tests is reported along with the other details of each run, in the previous section of this report. Specific work done on the methanation section of the plant, the results of tests of various catalysts, and details of work with Chem Systems Liquid-Phase Methanation (LPM) unit are given below.

IGT Fixed-Bed Methanation Unit

At the start of this contract period, the catalyst in the IGT fixed-bed methanation unit had become deactivated due to sulfur poisoning. In order to replace the catalyst, it was first necessary to passivate the old catalyst and remove it from the methanation reactor.

On June 25, 1976, a passivation procedure was started on the catalyst in the first- and second-stage methanation reactors. Passivation converts the reduced nickel in the catalyst to nickel oxide, by mild oxidation with gaseous O_2. This is necessary prior to handling the catalyst in air. The first-stage methanation reactor was isolated from the second-stage reactor, and a 1-inch vent line was attached to the first-stage discharge piping to permit independent venting of the spent oxidation gases. After heating the methanation reactor to $300^{o}F$, an air-nitrogen mixture containing 1/2% oxygen was admitted. The oxygen concentration was increased in steps over a 3-day period until 100% air (6500 SCF/hr) was used. The temperature rise associated with this procedure was very small (10^{o} to $20^{o}F$) and easily controlled. The flow of air was terminated, and the vessel was purged with nitrogen in preparation for making the switch to the second-stage vessel. Because no difficulties were encountered in the first-stage passivation, the entire procedure for the second stage was conducted in 42 hours. The second stage was purged with nitrogen, both stages were opened at the bottom handholes, and the MC-100 catalyst was dumped into drums. Several 2-inch, stainless-steel mesh cubes containing tagged samples of catalyst and ZnO (at particular depths) were recovered, and a catalyst sample was shipped to the manufacturer for analysis.

Union Carbide reported that a sulfur concentration of 700 ppm was found in the spent catalyst. Some nickel crystallite growth was observed, but no evidence of pore blockage was found as measured by surface area determinations. Union Carbide conducted microreactor tests at 575°F, which showed insignificant methane production, and therefore almost complete deactivation. They reported that the sulfur concentrations could explain the deactivation observed in the pilot plant reactor.

IGT decided at this time that an additional sulfur-guard system should be used with the methanation section in the HYGAS pilot plant to ensure an extended catalyst life and good operating data. Therefore, a series of tests were made in a laboratory reactor to determine the effects of a ZnO sulfur-guard catalyst used with the MC-100 catalyst. Earlier work at IGT had indicated a tendency for ZnO to promote the formation of alcohols under operatng conditions similar to those required for methanation systems in the HYGAS Process; however, quantitative data were lacking.

The first tests were performed with the ZnO sulfur-guard catalyst designated Zingard 1000 (New Jersey Zinc Company). Table 26 shows the results of this test series. At space velocities ranging from 500 to 5000 SCF/CF-hr, alcohol production over the Zingard catalyst was very low (≤ 375 ppm) and was restricted to principally methanol, which is not harmful to MC-100 (or other methanation catalysts) and is normally converted to methane over the catalyst. The Zingard catalyst also had negligible activity for methane formation as indicated by the low consumption of carbon monoxide and the very low increase or change in methane concentration in the feed and product gases.

Next, a test series was initiated in which a combination of Zingard 1000 and MC-100 methanation catalyst was tested in series bed operations. Figure 79 indicates the placement of the Zingard and methanation catalysts in the laboratory reactor. Gases typical of those used as feed to the HYGAS methanation section were passed over this combination of Zingard and MC-100. The results are shown in Table 27. No decrease in catalyst activity was noted at space velocities ranging from 5000 to 30,000 SCF/CF-hr. Good carbon monoxide conversion was maintained at all space velocities, and only traces of methanol and acetone were found in the product gas.

Table 26. OPERATING RESULTS FROM SULFUR STUDY OF ZINC OXIDE (Zingard 1000, 1/8-in. Spheres) AS A GUARD FOR THE METHANATION CATALYST

Run No.	463-a	463-b	463-c	464-d	464	465	466	467	468
Time, hr	0	4	95	120	146	168	193	217	242
Pressure, psig	7	7	1000	1010	1007	1002	1002	1002	1002
Temperature, top furnace, °F	75	460	460	475	470	469	431	431	430
Temperature, bottom furnace, °F	75	480	475	500	465	467	426	431	393
Temperature 1, reactor entrance, °F	75	503	519	523	502	408	401	457	428
Temperature 2, middle bed, °F	75	520	525	529	501	493	458	450	401
Temperature 3, 3/4 bed, °F	75	520	525	539	501	491	458	450	401
Temperature 4, exit, °F	75	462	522	520	482	490	450	434	373
Feed Flow Rate, SCF/hr	2.2480	2.2480	7.2406	7.9007	7.8689	18.2439	17.8914	8.0129	2.2269
Space Velocity, SCF/hr-cu ft	562	562	1811	1977	1969	4565	4477	1973	557
Feed Composition, mol %									
CO	0	0	7.3	6.2	6.1	4.2	4.6	6.5	15.0
CO_2	0	0	0.2	0.2	0.1	0.1	0.2	0.1	0.1
H_2	0	0	32.6	33.5	34.5	31.5	29.2	29.0	26.9
CH_4	0	0	29.8	29.3	29.0	34.8	32.9	37.7	33.5
C_2H_6	0	0	0.8	0.9	0.9	1.2	0.9	0.36	0.3
C_3H_8	0	0	0.1	0.3	0.3	0.53	0.44	0.24	0.15
N_2	100	100	29.1	29.57	28.5	27.17	31.75	26.1	22.85
He	0	0	0.1	0.03	0.6	0.5	0.01	--	1.2
Alcohols (C_1-C_6)	0	0	0	0	0	0	0	0	0
Ketones (C_1-C_7)	0	0	0	0	0	(	)	0	0
Acids (C_1-C_5)	0	0	0	0	0	0	0	0	0
Aldehydes (C_2-C_5)	0	0	0	0	0	0	0	0	0
Total	100	100	100.0	100.00	100.00	100.00	100.00	100.00	100.00
Product Flow Rate, SCF/hr	2.2480	2.2480	7.1212	7.8769	7.8203	18.2046	17.9802	7.8269	2.2076
Product Composition, mol %									
CO	0	0	7.3	5.9	6.2	3.8	4.5	5.9	14.9
CO_2	0	0	0.2	0.2	0.1	0.1	0.2	0.1	0.1
H_2	0	0	32.5	33.2	34.7	30.5	28.5	27.0	27.0
CH_4	0	0	29.9	29.7	29.1	35.5	34.5	39.7	33.3
C_2H_6	0	0	0.8	0.9	0.9	1.2	0.4	0.34	0.3
C_3H_8	0	0	0.1	0.3	0.3	0.53	0.32	0.23	0.26
N_2	100	100	29.1	29.77	28.5	27.87	31.57	26.73	23.04
He	0	0	0.1	0.03	0.2	0.5	0.01	--	1.1
Methanol	0	0	280ppm[a]	375ppm[a]	40ppm[b]	159ppm[c]	108ppm[c]	161ppm[d]	371ppm[d]
Ketones (C_1-C_7)	0	0	7ppm	--	--	--	--	--	--
Acids (C_1-C_5)	0	0	15ppm	--	--	--	--	--	--
Aldehydes (C_2-C_5)	0	0	15ppm	--	--	--	--	--	--
Total	100	100	100.00	100.00	100.00	100.00	100.00	100.00	100.00
CO Consumed, lb-mol/hr	0	0	0	0	0.000012	0.000190	0.000035	0.000145	0.000013
H_2 Consumed, lb-mol/hr	0	0	0.000017	0.000081	0.000003	0.00051	0.000256	0.000556	0.0000076
CH_4 Changed, lb-mol/hr	0	0	+0.000020	+0.000064	+0.000017	+0.00029	+0.000824	+0.000223	+0.000028

[a] Higher alcohols at concentrations less than 50 ppm cannot be detected because of the interference of methanol.
[b] About 550 ppm acids (acetic and propanoic acids) were detected.
[c] About 50 ppm dimethoxymethane was found.
[d] About 0.05 wt % hydrocarbons, which were not alcohols, aldehydes, or acids, were found in the liquid product. They were unidentified.

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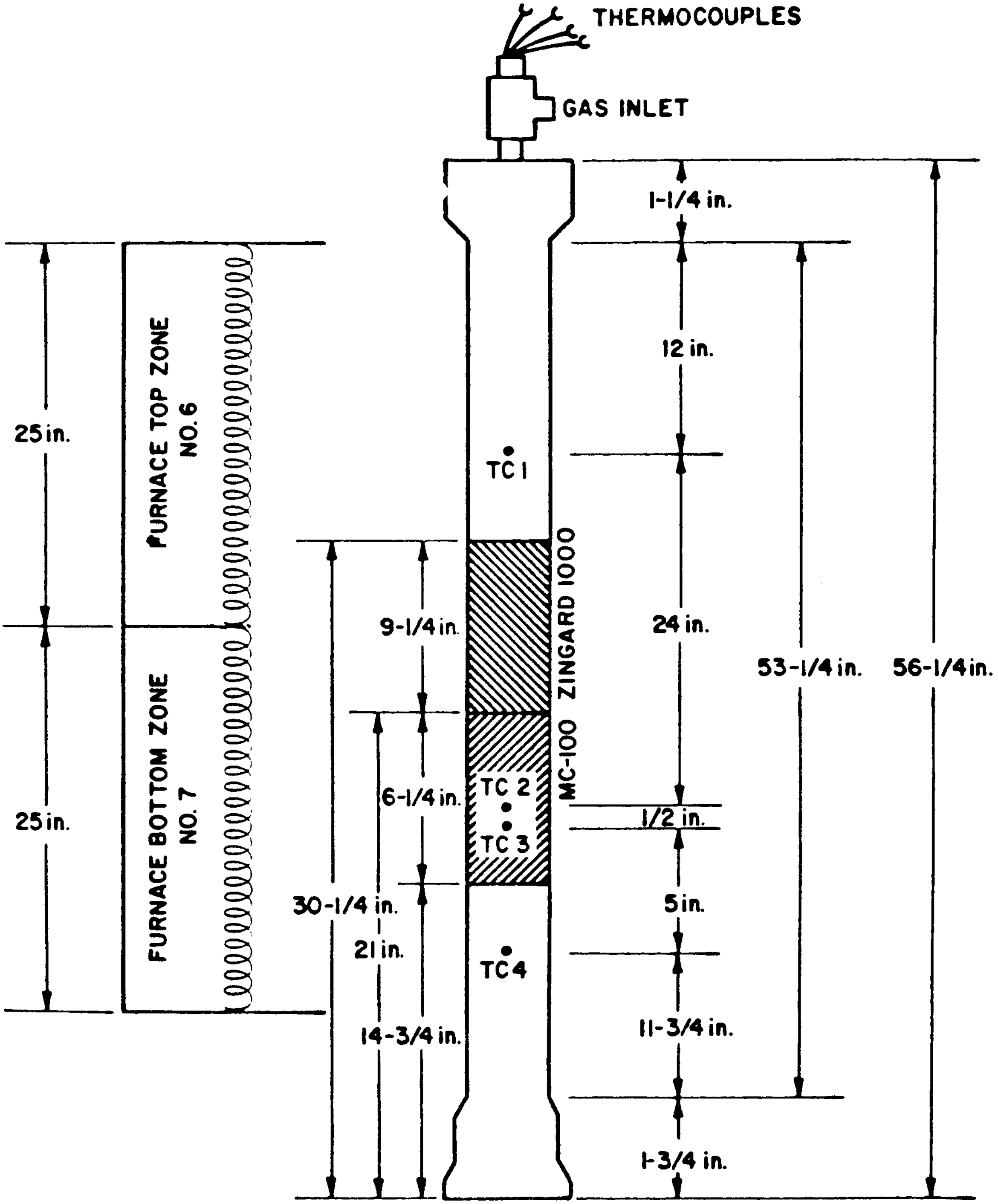

Figure 79. LABORATORY REACTOR CHARGING DIAGRAM FOR COMBINED ZnO (Zingard 1000)-MC-100 METHANATION TESTS

Table 27. OPERATING RESULTS OF COMBINED ZnO (Zingard 1000)-MC-100 METHANATION TESTS

Run No.	469	470	471	472	473
Time, hr	7	7	5	6	6
Pressure, psig	1000	1000	1000	1000	1000
Temperature, furnace top zone, °F	465	470	470	638	800
Temperature, furnace bottom zone, °F	500	502	503	500	501
Temperature 1, reactor entrance, °F*	478	390	328	360	440
Temperature 2, middle bed, °F*	793	915	872	890	855
Temperature 3, lower bed, °F*	805	920	873	900	875
Temperature 4, exit, °F	595	715	753	885	930
Feed Flow Rate,+ SCF/hr	15.24	30.73	46.73	68.01	95.74
Space Velocity, SCF/hr-cu ft	4761	9605	14603	21253	29918
Feed Composition, mol %					
CO	4.8	4.50	4.40	3.6	3.9
CO_2	--	0.06	--	--	0.09
H_2	35.4	31.90	34.1	46.8	33.5
CH_4	32.4	34.60	35.0	28.2	35.3
C_2H_6	0.3	0.33	0.2	0.1	0.53
C_3H_8	0.2	0.15	0.1	0.1	0.13
n-C_4H_{10}	--	0.02	--	--	0.02
i-C_4H_{10}	--	0.02	--	--	0.02
C_5's	--	0.01	--	--	--
N_2	26.90	28.40	26.10	21.2	26.5
He	--	0.01	0.10	--	0.01
Total	100.00	100.00	100.00	100.00	100.00
Product Flow Rate, SCF/hr		27.18	42.28	63.15	81.8
Product Composition, mol %					
CO	--	--	--	--	--
CO_2	--	--	--	--	--
H_2	22.0	17.70	22.9	37.8	23.3
CH_4	44.8	48.10	46.2	37.9	43.4
N_2	33.2	34.10	30.9	24.2	32.7
He	--	0.10	--	0.1	0.6
Total	100.00	100.00	100.00	100.00	100.00
Liquids Collected in H_2O (Dry-Ice Trap), Flow Rate, g/min	0.2	0.5	0.8	1.3	1.6
Acetone, wt %	1.0+0.2	0.05+0.01	0.01+0.005	0.02+0.005	--
Methanol, ppm (wt)	26 ± 8	12 ± 4	5 ± 2	< 10	--
Trace Product-Gas Analysis, ppm (vol)					
Methanol	< 10	6 ± 3	18± 6	--	12 ± 4
Acetone	< 5	< 3	< 5	--	45 ± 5
Benzene	0.7	< 0.5	1.5 ± 5	--	2.5 ± 5
CO Converted, %	100.0	100.0	100.0	100.0	100.0

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* See Figure 6 for thermocouple location.

\+ Based on MC-100 catalyst volume.

Because no evidence of catalyst deactivation was observed when the Zingard was used with the MC-100 catalyst, it was decided to use this material as a sulfur-guard catalyst in the HYGAS pilot plant. The Zingard 1000 catalyst was charged to the 5-cubic-foot depth normally occupied by the first-stage methanation catalyst, and 10 cubic feet of the MC-100 catalyst was charged to the second-stage methanation reactor, the normal catalyst depth for that reactor. In operation, the product gases were passed a) through the heater ahead of the first-stage reactor and preheated to approximately 500°F, b) through the Zingard 1000 catalyst, and c) then through the MC-100 methanation catalyst. Only single-stage operation was permitted using this arrangement, but it was sufficient to establish space velocity and conversion efficiencies for this particular catalyst with subsequent pilot plant runs.

Estimates were made of the lifetime of the Zingard 1000 catalyst under the normal operating conditions of temperature, pressure, feed-gas flow rate, and at various inlet sulfur concentrations. Table 28 shows that at sulfur concentrations presumed to be normal (between 0.5 and 5 ppm), the 1200 pounds of Zingard 1000 sulfur-guard catalyst should have a lifetime between 10,000 to 100,000 hours. This is sufficient to operate the methanation section in the HYGAS plant for at least 1 year.

Table 28. ESTIMATED ZINGARD 1000 LIFETIME AT VARIOUS INLET SULFUR CONCENTRATIONS (Feed Rate = 150 lb mol/hr, T = 525°F, P = 1000 psig ZnO Weight = 1200 lb, ZnO Average Absorption at Breakthrough = 20% by Weight)

Sulfur Concentration in Feed, ppm	Lifetime, hr
0.5	100,000
5	10,000
50	1,000
500	100

The reduction procedure to activate the MC-100 catalyst in the second-stage pilot plant reactor was conducted from September 6, 1976 (0000 hrs), to September 8, 1976 (2400 hrs), during Test 55. The vessels were first heated to 500°F with natural gas (the usual procedure) prior to exposure to hydrogen. Hydrogen was admitted at a continuous flow rate of 4200 SCF/hr and recycle product gas at a flow rate of 149,000 SCF/hr. The space velocity through the reactor was 4900 hr^{-1} based on a catalyst volume of 31.4 cu ft.

The average temperature of the vessel was slightly over 500°F. The pressure was held at 700 psig. The composition of the hydrogen plant product during a typical period while conducting this procedure is reported in Table 29, the average product gas composition, which is the same as the recycle gas composition for the same period, is shown in Table 30

Table 29. PRODUCT GAS FROM HYDROGEN PLANT

Component	Mol %
H_2	97.50
N_2	.16
CH_4	.56
CO	1.78
Total	100.00

Table 30. PRODUCT GAS FROM METHANATION (Recycle)

Component	Mol %
H_2	97.31
N_2	.32
CH_4	2.37
CO	.00
Total	100.00

CO was completely converted to CH_4, which indicated the catalyst was activated, although the extent of activation could not be determined at this low CO concentration. The length of the activation procedure was considered sufficient for the volume of catalyst in the system.

After the installation of the ZnO sulfur-guard system, the IGT fixed-bed methanation section was brought on line during HYGAS Tests 57 and 58. The performance of this section, and of the sulfur-guard system in particular, was entirely satisfactory. The IGT fixed-bed methanation section was on standby during the rest of this reporting period.

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

During the summer of 1976, the Institute of Gas Technology's HYGAS pilot plant was selected by ERDA as the test site for the Chem Systems, Inc. Liquid-Phase Methanation (LPM) pilot unit. The LPM system components were received in October, 1976 from the fabricator in Houston, Texas. IGT met with Chem Systems and ERDA to review the physical arrangement, the installation of the system, and utility requirements.

After the LPM unit parts were received, personnel from Chem Systems and IGT completed an inventory of all materials. The reactors were also inspected, and Chem Systems' representatives were satisfied as to the condition of the reactor components. IGT received the engineering data books for the equipment and several meetings were held to clarify final details on the installation of the LPM pilot unit.

Installation work started in November, after the completion of Test 57. It was necessary to rearrange some equipment in IGT's methanation section to optimize the installation of the LPM unit. The water knockout pot and some associated piping were relocated.

The LPM skid sections were rolled into place, leveled, and anchored, and all loose equipment was installed. All the utility piping from HYGAS sources to the LPM and all interconnecting piping on the skids was completed. The feed gas connection from HYGAS to the LPM unit and a bypass line around the hydrogen plant (natural gas reformer) shift converter were shop fabricated and installed after the completion of HYGAS Test 58. Instrument connections from the control trailer to the individual field units were installed. The controllers, recorders, and transmitters were all checked and calibrated. The main electric power was connected from the HYGAS motor control center to the LPM control trailer. Electric conduit and wiring from various junction boxes on the LPM unit to the control trailer were completed.

The nuclear level gage lifting device was assembled and found to be too difficult to operate manually and a new motorized device was ordered and received. The nuclear gage was checked for proper mounting and background radiation by IGT's consultants, Health Physics Associates, to make certain the device conformed to all Nuclear Regulatory Commission requirements. (This device was delivered and installed during February and technicians from Texas Nuclear Corporation completed calibration of the device during March.)

All the pumps on the pilot unit were energized to check the rotation of the motors. Improper wiring was corrected. Also, the wiring of the local start/stop pushbutton stations for the pumps had to be connected. Other electrical systems were debugged.

Pressure testing and final checking of all instruments and electrical systems were completed, and major work in steam tracing and insulation to properly winterize the plant was required. 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.

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 misaligned. Calibration of the flow system 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 repair. After repairs, the oil heater was checked out completely, and hot oil at $200^{\circ}F$ was successfully circulated. Gas flow was also initiated. The gas heater was used to raise the reactor temperature to $500^{\circ}F$ and a hot, pressurized oil and gas circulation test was successfully completed.

During February, all analytical equipment was installed. 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 LPM unit.

The LPM pilot unit was operated during Test 60 for 2 hours on April 4, and catalyst activity was established. After 2 hours of operation, a process upset upstream of the LPM unit resulted in a slug of diglycolamine solution entering the LPM pilot unit, and the test was terminated. Following Test 60, a high hydrogen-to-carbon monoxide ratio gas from the hydrogen plant reformer was fed to the LPM pilot unit, to determine the activity of the catalyst. These tests indicated that the LPM catalyst had lost two-thirds of its original activity, presumably during the process upset noted above.

Chem Systems then decided to replace the old catalyst, and a new catalyst charge was activated. The IGT Zingard sulfur-guard catalyst was also replaced with a commercial (CCI-C7-2) zinc-oxide sulfur-guard catalyst due to the probable fouling of the Zinguard.

The LPM unit was not put on-line for Test 61 because a fire occurred during operations on May 22 while the hot-oil filters were being changed. Discussions were held with Chem Systems and C. F. Braun personnel to determine the necessary modifications to either equipment or operating procedures that would eliminate this source of fire hazard in the operation of the pilot unit.

During the week of June 5, 1977, Chem Systems personnel met with IGT to finalize the details of modifications of the unit's hot-oil filter system and modification work began soon after.

Methanation Catalyst Evaluation Studies

The evaluation of methanation catalysts in the bench-scale reactor was continued during this year. 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. In the temperature range (550° to 680°F) studied in a packed-bed reactor, equilibrium conversion was achieved at 40,000 SCF/hr-cu ft and 1000 psig, and 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, however, the conversion decreased rapidly and nonlinearly (with space velocity) at pressures below 200 psig. The dependence of conversion on the pressure and space velocity is illustrated in Figure 80.

The catalyst was supplied in its oxidized form, making it necessary to reduce it to in order activate it. 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 the pilot plant, the hydrogen is recycled, and the optimum space velocity can

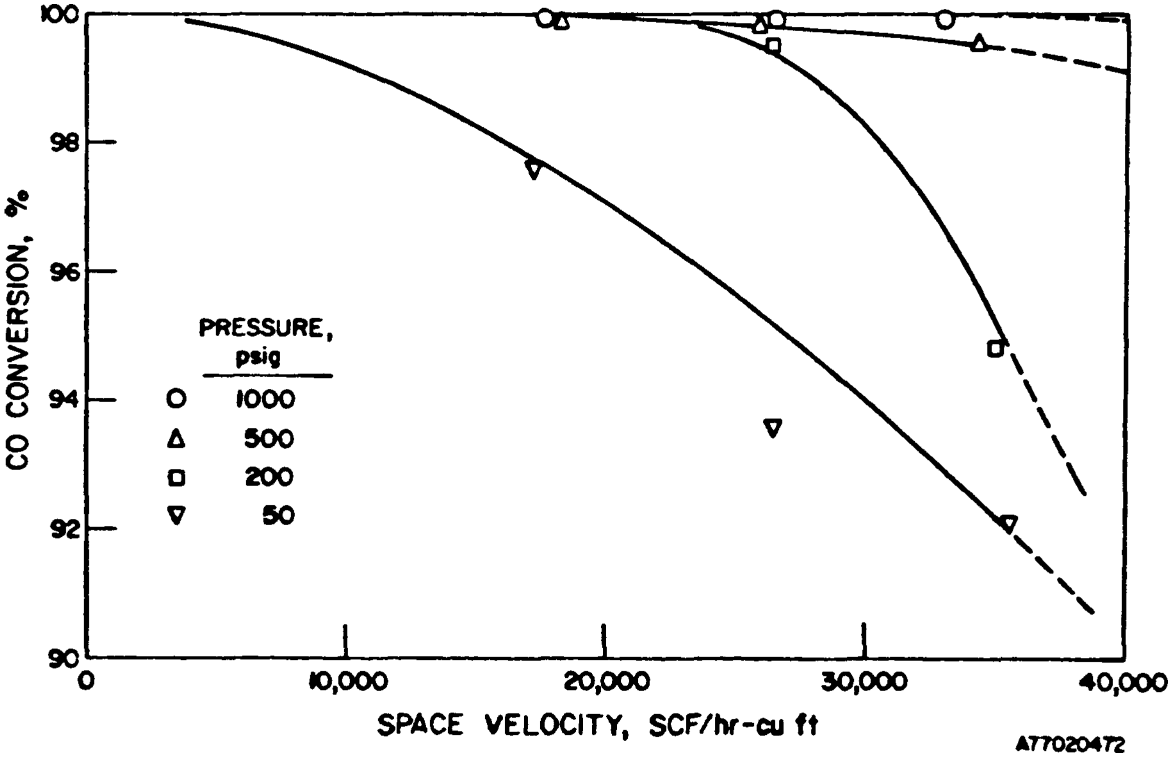

Figure 80. EFFECT OF SPACE VELOCITY ON THE CONVERSION OF CARBON MONOXIDE AT TEMPERATURES FROM 550° to 680°F (LDI X-826 Catalyst, 1/8-Inch Cylinders)

be easily achieved. 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. Also, in the pilot plant, the methanator can not be heated to an initial temperature of 750°F for start-up. The temperature that can be achieved is approximately 500°F, or less; thus the catalyst may not be reduced properly (i.e., achieve maximum activity). Because of these limitations, a new catalyst activation (i.e., reduction) method was developed. It consisted of the following:

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 completely 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 the 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 continue to increase the bed temperature.

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.

It is recommended 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 the laboratory test of the above method was a synthetic mixture of high-purity components, which was sulfur-free. Both the feed and product compositions as well as the operating conditions are presented in Table 31. 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.

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 Ni0104T, G-87p, and MC-100 catalysts.

Table 31, 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.

Table 31, 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.

Table 31, 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 31, 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	--

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[*] 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.

MATERIALS TESTING

Task 4 under this contract concerned testing the materials used in the HYGAS process. The Metal Properties Council (MPC) has an ERDA contract to conduct erosion and corrosion testing of materials (metals and refractories) in coal gasification atmospheres. The HYGAS pilot plant was selected for specific metal coupon exposures for evaluation of the resistances of certain alloys to the coal gasification atmospheres. Exposures for MPC corrosion and erosion test coupons were carried out during all tests. Specific work under this task was done during the July 1976 plant turnaround, and also throughout the year concerning testing various refractories. Details of pertinent work done during the turnaround, and the results of refractory tests, are given below.

Plant Turnaround

The annual HYGAS plant inspection and maintenance period began on July 16, 1976. The plant was shut down for maintenance work, extensive nondestructive testing and analysis of all operating sections, and necessary repairs. The reactor was opened for a complete inspection. The utility sections, including the high-and low-pressure boilers, all boiler feedwater pumps, boiler feedwater treatment equipment, and the hydrogen plant were opened up for inspection. The solids discharge valve LV-340, at the base of the reactor, was inspected and repaired.

The high-pressure cyclone at the raw-product-gas discharge from the reactor was also removed and sent to Argonne National Laboratory for a wall thickness inspection (by ultrasonics) during the shutdown period. Argonne National Laboratory completed its investigation and reported on these measurements. This group has been monitoring the operation of this cyclone since its installation and has complete information on over 1268 hours of operation with coal feed. A previous inspection was conducted after 518 hours of total operation.

The high-pressure cyclone is a unique design developed by IGT engineers under the HYGAS program. Its performance and wear rates are of primary importance in the development of satisfactory equipment for coal gasification. Figure 81 shows the construction details of the cyclone. The area labeled "A" has a stellite overlay; area "B" is the solids outlet neck.

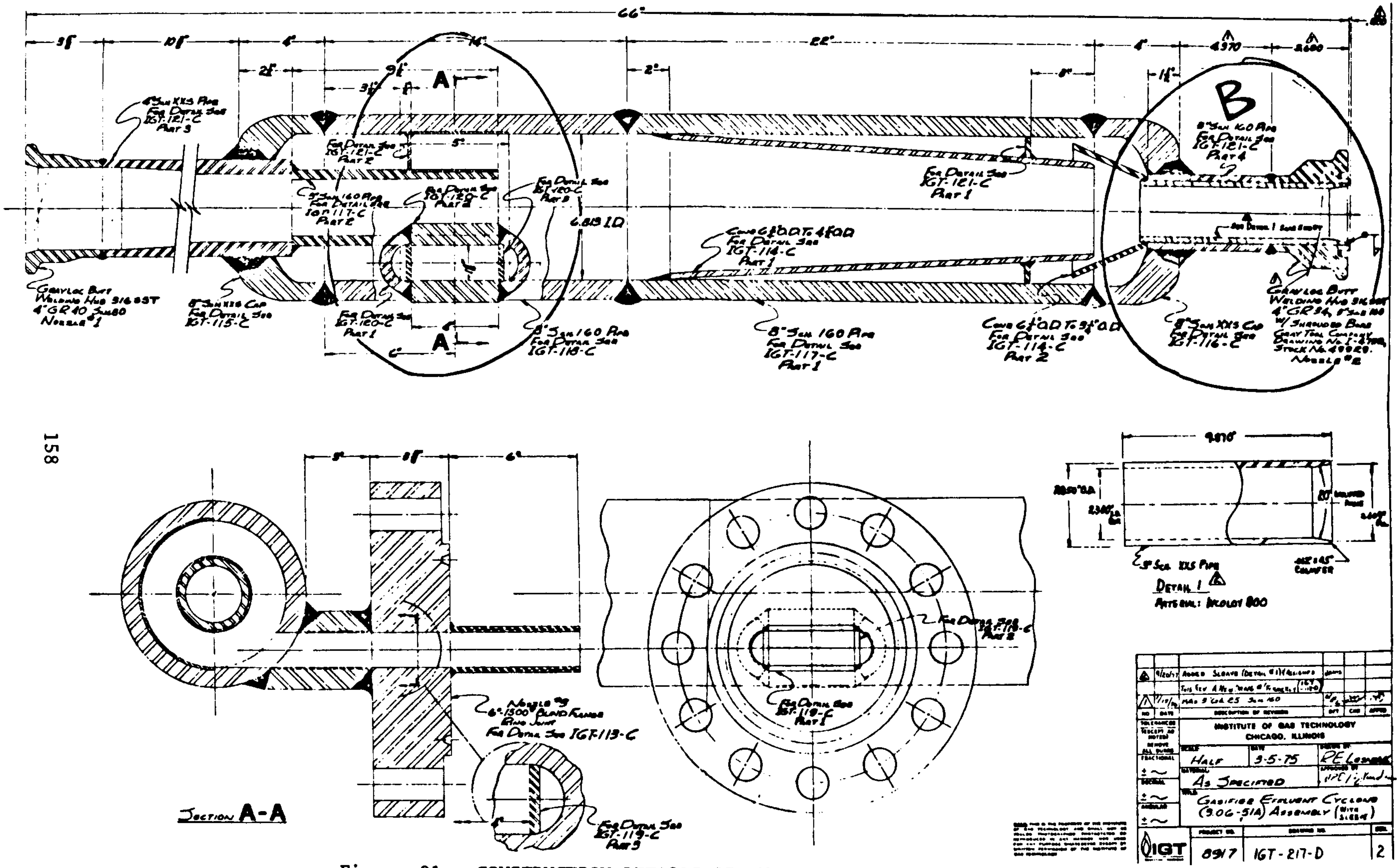

Figure 81. CONSTRUCTION DETAILS OF HYGAS HIGH-PRESSURE CYCLONE

The significant findings from the July 1976 inspection were: a) the maximum erosion in the stellite reinforced area in the top of the cyclone was approximately 0.017 inches, or about 2% of the total wall thickness at that point, b) slightly more erosion occurred in the solids outlet neck at the bottom of the cyclone of about 0.080 inches, or approximately 17-1/2% of the original wall thickness. This wear was probably caused by the high-velocity vortex that forms in the lower area of the cyclone, as shallow spiral-shaped gouges have been observed in this area in the past. A replaceable wear sleeve was installed in this area of the cyclone which would absorb most of the wear from the vortex and which could be replaced without affecting the base metal of the cyclone itself (see Figure 81). Based on measurements taken to date, the wear rate is linear with operating time. Figure 82 shows the average cumulative erosion of the hard-faced (stellite) area of the high-pressure, raw gas cyclone. The average wear rate of 15 mills per 1000 hours of operation indicates that the stellite-covered area (originally 125 mills thick) will last approximately 8300 hours. Wear rates to date have not been excessive and have been within original design tolerances. Once data such as this is developed, components can be designed for any required life by varying the wall thickness in critical areas or by using special abrasion resistant materials or coatings.

Nondestructive testing was conducted during the annual plant maintenance and shutdown period on slurry piping loops and other parts subjected to high erosion and high stress.

An X-ray inspection, made of the 1-inch slurry line between the slurry mix tank and the slurry letdown valve (Willis Chokes, LV-342-A & B), revealed that 12 crosses in this line showed excessive wear and there was excessive wear on portions of the 1-inch main pipe section. All crosses and pipe sections that showed excessive wear were replaced.

Dye-penetrant tests of the cone section in the slurry dryer area of the HYGAS gasifier indicated numerous cracks requiring repair. This section is in the non-pressure-retaining area of the reactor internals and does not present any safety problem during operations. The leaks were repaired to prevent migration of process gases into the dry-nitrogen shell area of the HYGAS reactor, where all of the instrumentation, solids transfer valve operators, and some other mechanical parts of the HYGAS reactor are located.

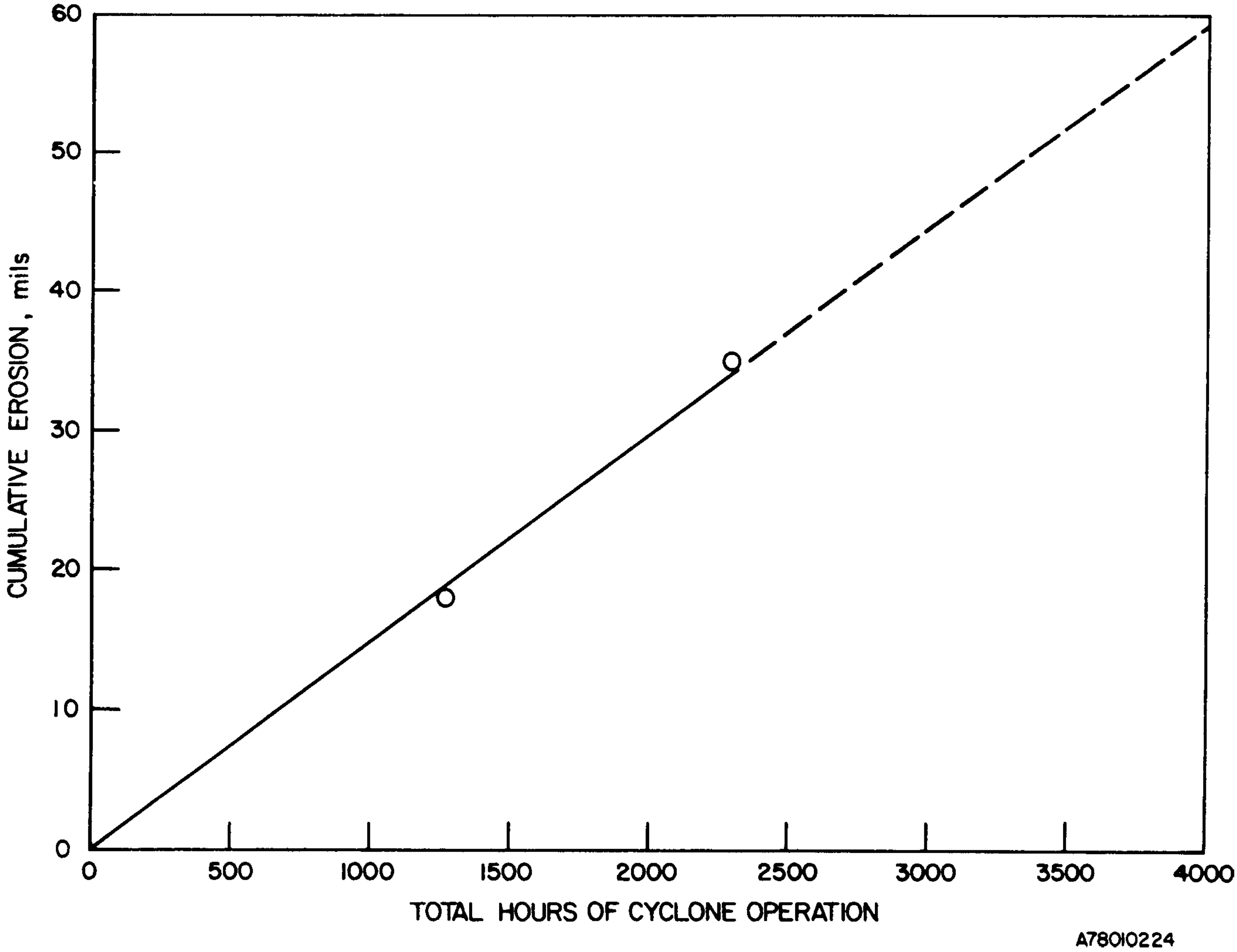

Figure 82. AVERAGE CUMULATIVE EROSION OF HARD-FACED (STELLITE) AREA OF HIGH-PRESSURE CYCLONE

One of the major projects during the plant maintenance period (July, 1976) was the conversion of the cooling-water loop from direct once-through service with water from the Chicago Sanitary and Ship Canal, to a recirculating cooling-water service supplied from a cooling tower located in the Steam-Iron pilot plant, immediately adjacent to the HYGAS Plant. The Steam-Iron cooling tower has a capacity equivalent to the full-cooling duty for both the HYGAS and the Steam-Iron plants. The use of cooling tower cooling water should greatly reduce corrosion and fouling that has been experienced in the past several years in the HYGAS heat exchangers when using the canal water. This change will promote longer heat exchanger life and better cooling efficiency for all of the HYGAS plant heat exchangers.

Refractory Testing

Objective

The refractory testing was conducted to test the performance of some of the commonly used castable refractory materials in a condensing acid-gas environment, which is 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 refractory 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 83 and 84.

By virtue of the thermal gradients existing in reactor walls of the type shown in Figure 84, 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 83, 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.

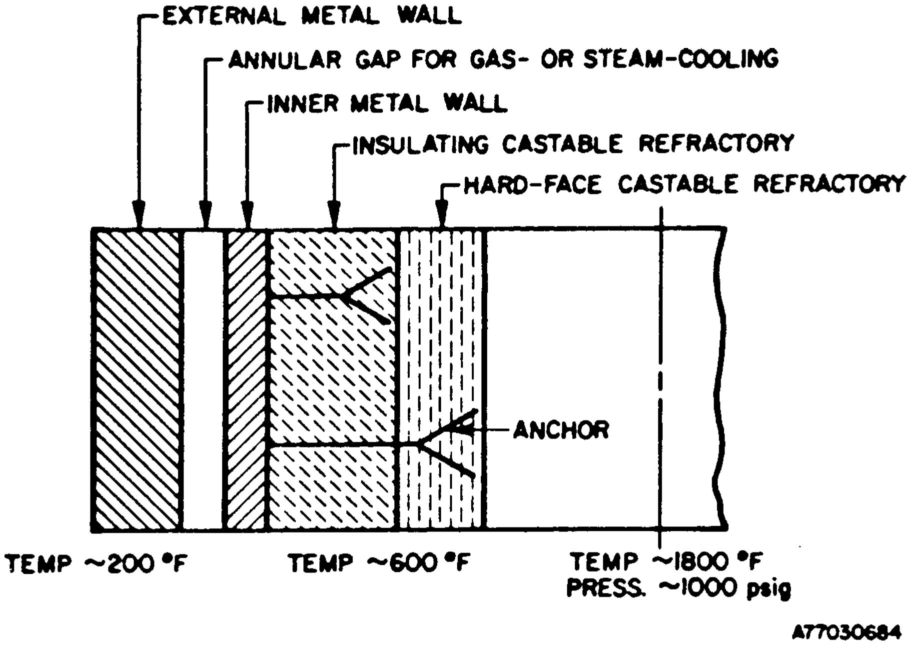

Figure 83. EXAMPLE OF A GAS-COOLED REACTOR WALL CONSTRUCTION

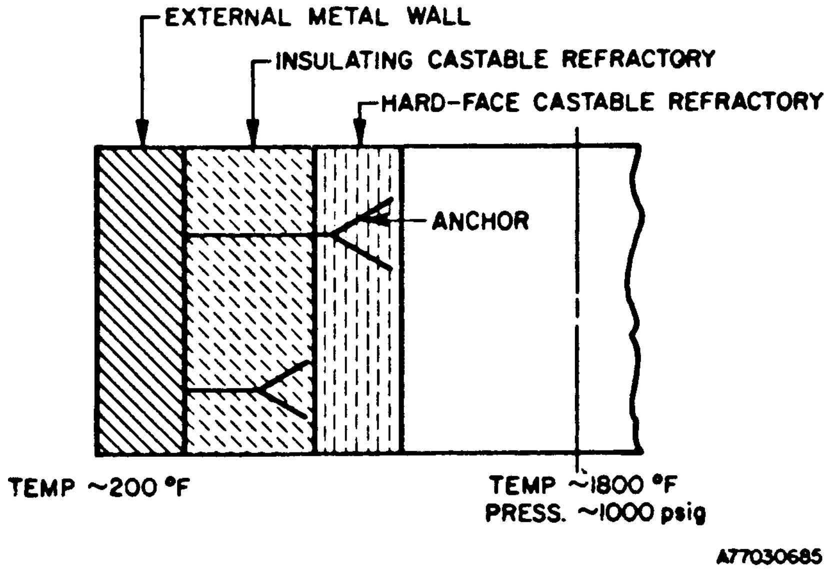

Figure 84. EXAMPLE OF AMBIENT AIR-COOLED REACTOR WALL CONSTRUCTION

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.

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 85. 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 86. The equipment assemblies employed in this investigation are shown in Figures 86 through 88. 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 type 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 32 . 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 84 through 86. 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:

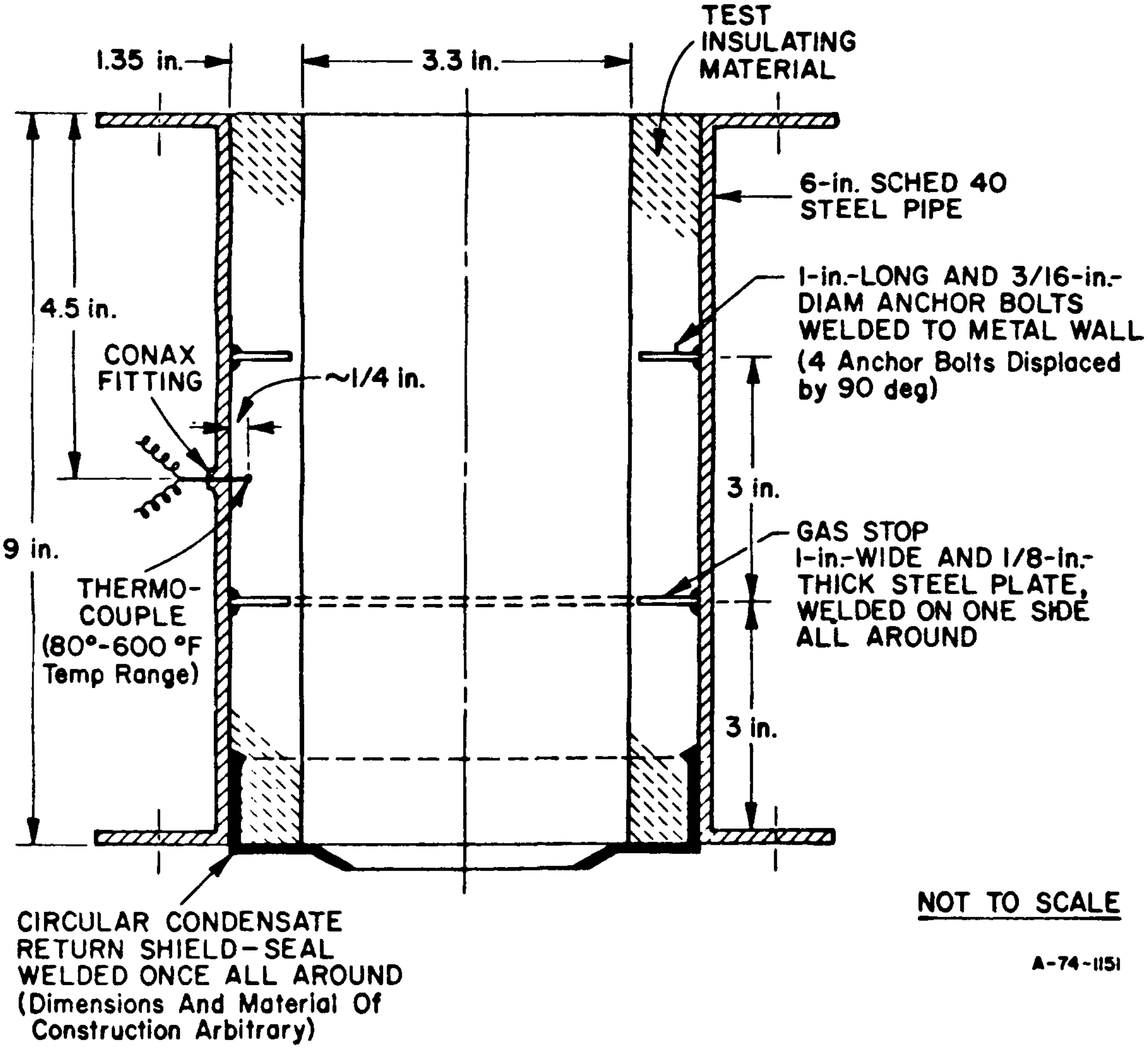

Figure 85. CONSTRUCTION DETAILS OF THE FLANGED INSULATING MATERIAL TEST SECTION

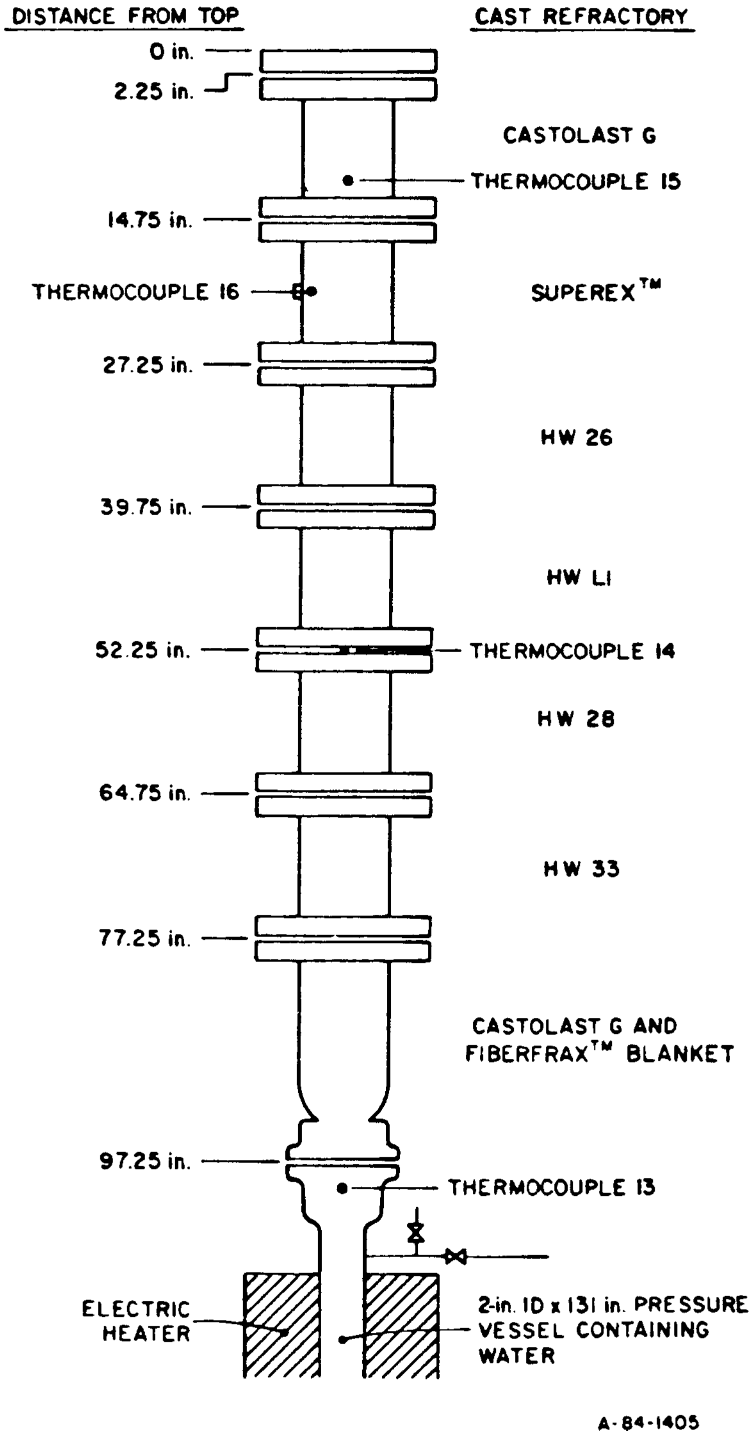

Figure 86. EQUIPMENT SETUP FOR TESTING CASTABLE REFRACTORIES

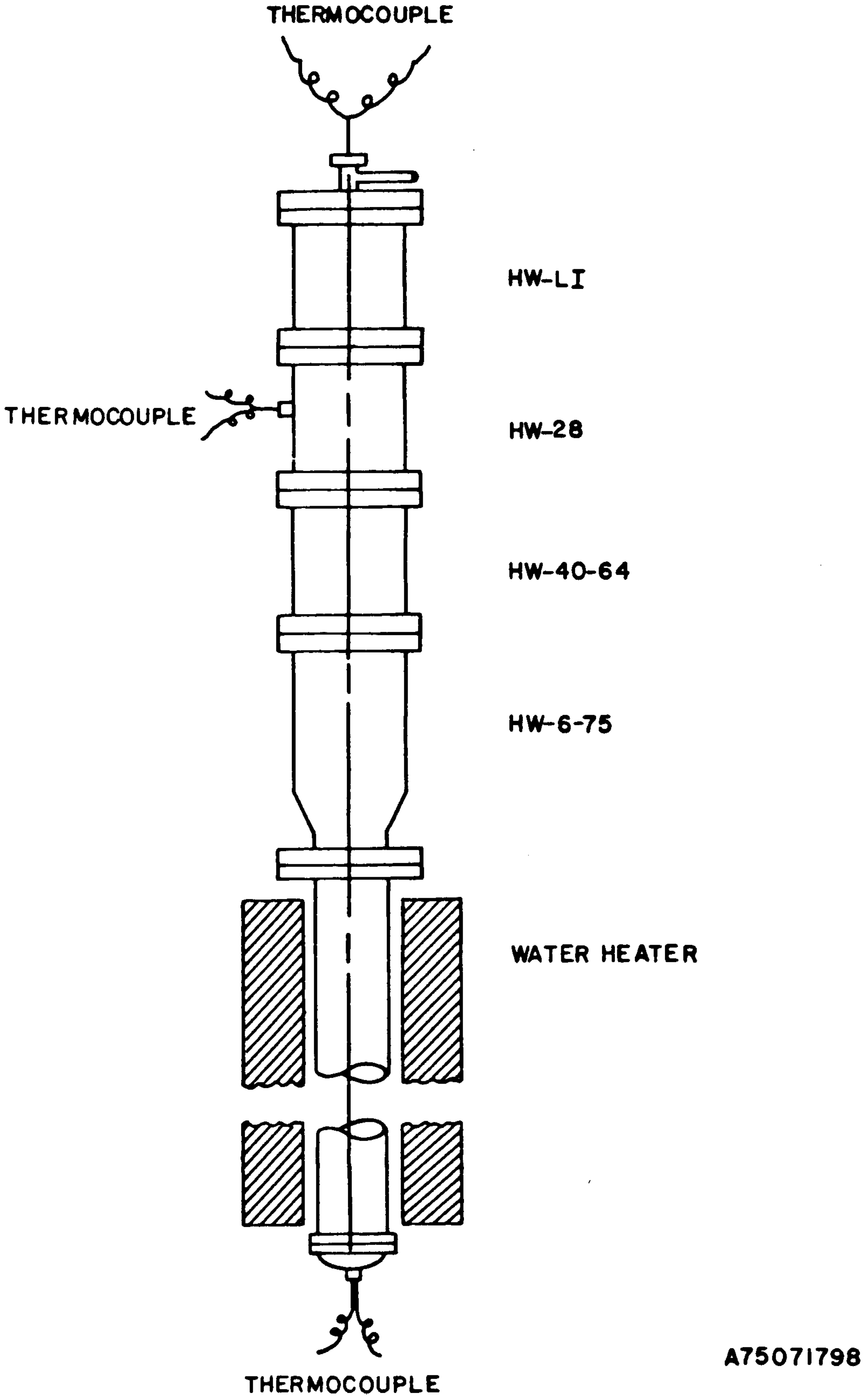

Figure 87. ASSEMBLY OF CASTABLE REFRACTORY MATERIALS

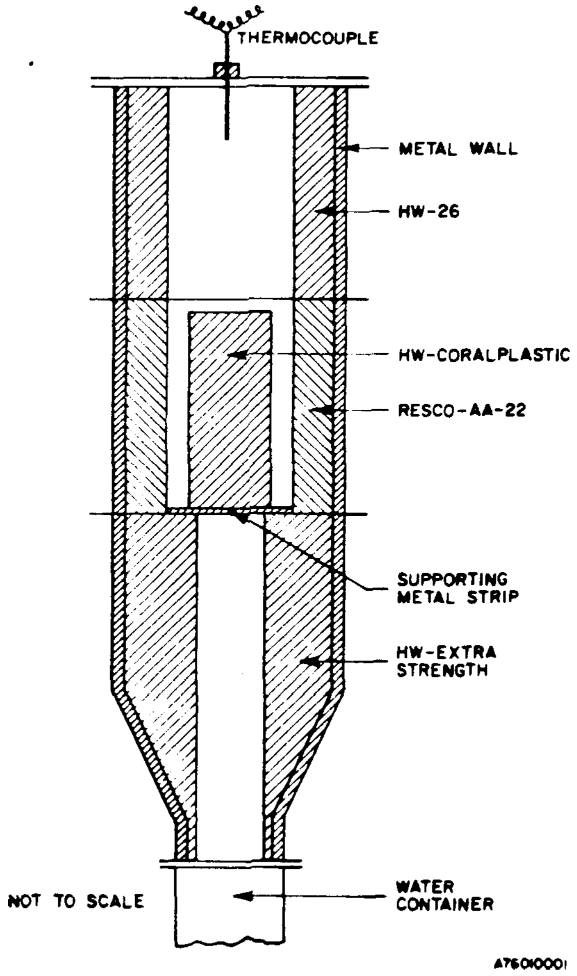

Figure 88. ASSEMBLY OF CERAMIC REFRACTORY MATERIALS FOR TESTING IN AN ACID-GAS ENVIRONMENT

Table 32. 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 33 through 35. 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 89 and 90. 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 33. PHYSICAL AND CHEMICAL ANALYSIS OF CASTABLE MATERIALS EXPOSED TO CONDENSING STEAM AND CONDENSING ACID-GAS IN TEST I

Castable Material	HW-33		HW-28		HW-26		HW-LI		HW-Castolast G	
Physical Properties	Used[a]	Reference[b]	Used	Reference	Used	Reference	Used	Reference	Used	Reference
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 34. PHYSICAL AND CHEMICAL ANALYSIS OF CASTABLE MATERIALS EXPOSED TO CONDENSING STEAM AND CONDENSING ACID-GAS IN TEST II

Castable Material	HW 40-64		HW 6-75		HW-LI		HW-28	
Physical Properties	Used[a]	Reference[b]	Used	Reference	Used	Reference	Used	Reference
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 35. 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	
Physical Properties	Used[a]	Reference[b]	Used	Reference	Used	Reference
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.

BEFORE

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AFTER

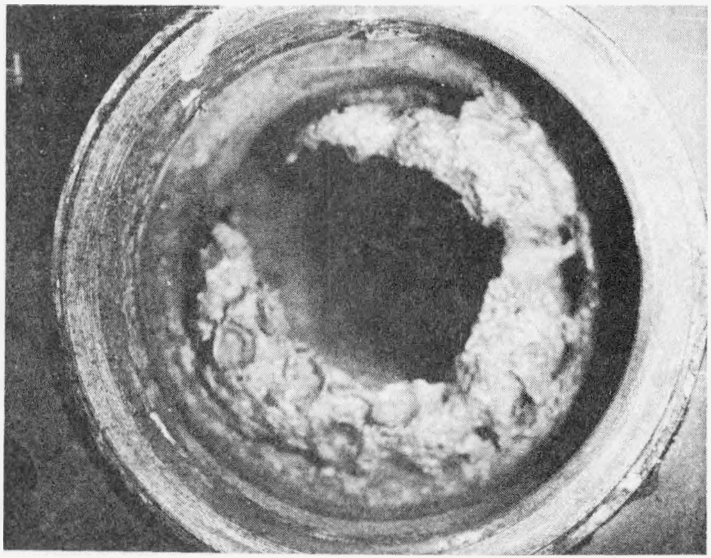

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Figure 89. PERFORMANCE OF SUPEREX™ INSULATING MATERIAL IN A CONDENSING ACID-GAS (1000 psig and 540°F)

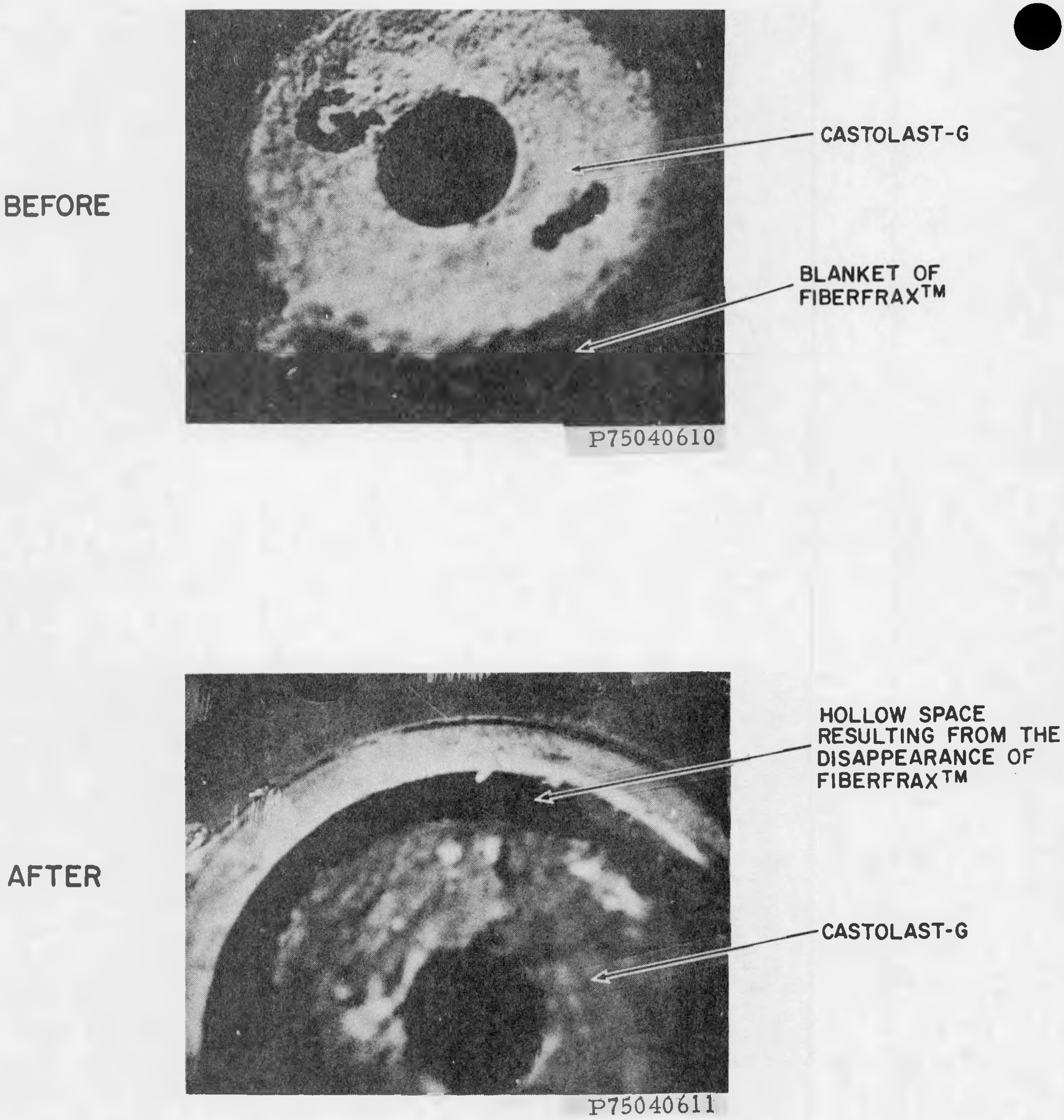

Figure 90. PERFORMANCE OF CASTOLAST-G AND FIBERFRAX™ INSULATING MATERIAL IN A CONDENSING ACID-GAS (1000 psig and 540°F)

Refractories, Pittsburgh, Pa. The effects of the condensing acid-gas on the refractory properties can be evaluated from the results shown in Table 33. 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 33 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 34) 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 35 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 35). 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 36). 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 36, 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 36. ANALYSIS OF FEED WATER 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	--		Feed water
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		Feed water (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		Feed Water
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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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 37. 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.

Table 37. 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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Conclusions

- 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.

Acknowledgment

Detailed physical, chemical, and mineralogical analyses were conducted by the Garber Research Laboratories, a subsidiary of Harbison-Walker Refractories, Pittsburgh, Pennsylvania.

PROGRAM ASSESSMENT

Task 5 under this contract involved a critical review of the progress made in the HYGAS program.

In October 1976, the HYGAS program was reviewed by an ERDA team headed by personnel from the Division of Planning and Program Analysis and their consultants. The review covered the entire history of the HYGAS program under the support of the U. S. Government, which began in 1964. A special publication was prepared for the review committee, highlighting the achievements of the HYGAS program.

In addition, during this reporting period, ERDA received weekly letter reports and detailed monthly work reports. Four quarterly reports were prepared. These reports (FE-2434-4, FE-2434-8, FE-2434-12, and FE-2434-16) are available from the government printing office. This interim report was also prepared.

After each HYGAS test conducted during this reporting period, a debriefing session was held. These sessions were generally attended by representatives of IGT, ERDA, and C. F. Braun. Their purpose was to provide an informal review of the hydrogasification test just conducted.

The A.G.A. Project Advisory Committee meetings were held on November 18, 1976, February 10, 1977, and May 5, 1977 at IGT.

ENGINEERING SERVICES

Task 6 under this contract involved the various engineering services required to acquire data to improve pilot plant operation. Notable achievements included an evaluation of the pretreater prior to Test 54, which resulted in recommendations which significantly improved pretreater operation. Tests were conducted on the fines generated by the Rosebud subbituminous coal used during the fall of 1976, and fluidization tests were run on this coal. Tests were conducted in early January to select a bituminous coal to be used in a series of HYGAS tests. Runs were made in a bench-scale unit to help determine optimum conditions for tests with this coal. Work was done to determine optimum bed heights in the HYGAS reactor, and detailed study of the quench system was made. Details of all these studies follow.

Pretreater Operation

Prior to HYGAS Test 54, pretreatment operations with the pilot-plant, 7-1/2 foot ID pretreater using a –10 mesh Illinois No. 6 bituminous coal had resulted in some periods of unsatisfactory operation. During some of the tests with bituminous coal, the pretreater reactor developed clinkers and hot spots, showed an accumulation of agglomerate material in the pretreater reactor itself, and failed to produce completely nonagglomerating feeds for all test conditions. Accordingly, a laboratory test series investigating various pretreatment conditions required to produce nonagglomerating feed without attendant clinker and/or agglomerate buildup in the pilot-plant reactor was initiated. Results of these tests are presented in Table 38. The major result from this test series was an indication that the coarser particle-size material was more difficult to pretreat and resulted in agglomerating material when compared with results of tests with identical operating conditions with the finer particle-size material. Using the results of the tests shown in Table 38, it was established that the optimum size for pretreatment is a –14 mesh particle size. The pilot plant screen section ahead of the pretreater was changed to a 14 mesh screen, which eliminated all of the +14 mesh material from the raw-coal feed to the pretreater reactor. The +14 mesh material was sent to an oversize bin for storage and subsequent recycling to the coal preparation section of the plant for ultimate consumption. Operation of the pretreater using the –14 mesh feed was excellent compared with earlier test results, and the pretreater

Table 38. AIR PRETREATMENT TESTS WITH ILLINOIS NO. 6 BITUMINOUS COAL

Test No.*	Superficial Velocity, ft/s	Mean Residence Time, min	Temperature, °F	SCF O_2/ lb Coal	IGT† Boat Test	Remarks
BR0	0.6	60	750	2		Sintered in the 10-inch reactor. Two attempts made.
BR1	1	60	825-850	2	Lightly	
BR2	1	60	750	1	Some	
BR4	1	30	750	1	Agglomerated	Batch
BR5	1	60	750	2	Caked	Batch
BR6	1	60	750	2.5	Agglomerated	Batch
BR7	1	60	750	3	Agglomerated	Batch
	1		825	2.5		Caked in bed twice.
BR8	1	30	750	1	Free flow	–20+80 Mesh
	1		750			Caked in bed, +20 mesh
BR9	1.25	30	750	1	Stick	+20 Mesh
BR10	1	30	750	1	Free flow	–80 Mesh
BR11	1	30	750	1	Free flow	–20 Mesh
BR12	0.875	30	750	1	Free flow	–14 Mesh

* Tests BR0 through BR2 were conducted in the 10-inch diameter continous-flow unit. Tests BR4 through BR12 were conducted in the 1-inch diameter batch unit.

† Refers to the IGT boat test for a visual measure of the agglomeration characteristics of the pretreated material. Free flow means that the product is completely nonagglomerating.

reactor showed no tendency for hot spots or poor temperature control. When subsequently inspected, the pretreater reactor and char cooler were entirely free of clinkers or agglomerate formations.

Fines Generated With Rosebud Subbituminous Coal

The Rosebud subbituminous coal used in HYGAS Tests 55 through 58 had the appearance of being a very friable material. This led to some concern that excessive fines generation would occur when operating the coal mill and also there might be a high attrition rate with this coal in the HYGAS fluidized bed gasification section, leading to large carryover losses from the reactor. IGT has developed a comparative attrition test and it was decided to test the Rosebud coal by this method. The attrition test is conducted with air in an atmospheric pressure Plexiglas column 1.5 inches inside diameter. A single, small orifice (0.097-inch-diameter) is situated in the middle of the base of the column. Air is passed through this orifice at critical flow to give a gas velocity in the column of 0.31 ft/s. Tests for coal attrition are normally run for 6 hours, and the sample degradation is measured as a weight fraction of the –100 mesh and –200 mesh materials that were generated during the test.

The data in Table 39 indicate that the Rosebud coal has greater attrition resistance than Montana lignite or pretreated bituminous coal. Raw bituminous coal is given an arbitrary attrition resistance of 1. This relatively good attrition resistance was surprising in respect to the appearance of the coal. Operations in the HYGAS reactor on Test 55 indicated that there was no tendency to have more carryover of fines from the reactor than with previous coals tested. The operation of the coal crushing equipment itself was excellent, with no increase in the amount of –200 mesh material generated during the crushing operations.

Fluidization Tests

A series of fluidization tests were run during October on various size consists of the Rosebud subbituminous coal to determine whether some basic fluidization problem could be the cause of solids flow interruptions in the upper part of the HYGAS reactor during Tests 55 through 57.

Velocities for complete fluidization (V_{cf}) were experimentally determined for the –8 mesh feed material used in Tests 55, 56, and 57 and for –12, –14, and –20 mesh fractions. Results are shown in Table 40.

Table 39. SUMMARY OF ATTRITION TESTS OF COAL FEEDS TO THE HYGAS PILOT PLANT

Material*	Bituminous Coal	Pretreated Bituminous Coal	Montana Lignite	Pretreated Bituminous Coal	Rosebud Subbituminous Coal
Lab No.	46-377	46-498	37-252	46-498	Sample No. 4
Bulk Density, g/ml	0.77	0.312	0.72	0.311	0.748
Feed Wt, g	46	45.2	44.3	19.1	46.0
Bed L/D	1.38	3.34	1.42	1.42	1.42
Run Time, hr	6	6	6	6	6
−100 mesh material generated					
Total Wt, g	4.83	25.43	16.36	11.98	8.8
Wt % of feed, R_I	10.5	56.3	36.9	62.7	19.1
−200 mesh material generated					
Total Wt, g	3.12	20.93	13.62	9.17	6.3
Wt % of feed, R_I	6.8	46.3	30.7	48.0	13.7
Attrition tendency relative to bituminous coal					
Based on −100 mesh, R_w†	1.0	5.27	3.38	2.48	1.82
Based on −100 mesh, R_v‡	1.0	13.0	3.61	6.14	1.87
Based on −200 mesh, R_w†	1.0	6.69	4.34	2.93	2.01
Based on −200 mesh, R_v‡	1.0	16.5	4.64	7.25	2.07

* Particle size range −10+50 mesh; average particle size 693-705 microns.
† Attrition tendency based on relative weights of fines generated.
‡ Attrition tendency based on the relative volume of fines generated.

Table 40. VELOCITIES REQUIRED FOR COMPLETE FLUIDIZATION OF SUBBITUMINOUS COAL FEED MATERIALS

Mesh Size	Experimental V_{cf} ft/sec†	Calculated V_{cf} for Slurry Dryer Conditions, ft/sec††
–12*	1.42	0.45
–14	1.12	0.39
–20	0.44	0.24

*–8 mesh and –12 mesh results are similar.

†Atmospheric pressure, room temperature air used as the fluidizing gas.

††Calculated for conditions in the slurry dryer at 1015 psia and 600°F.

The results indicate that the coarser feed material used in the initial tests is more difficult to completely fluidize than the finer material. Poor fluidization could cause solids flow problems and lead to plugging. Based on these tests, a finer particle size feed (–14 mesh) was used in Test 58.

Selection of a Bituminous Coal

When the ERDA/A.G.A. Operating Committee directed IGT to return to an Illinois Basin bituminous coal for the next series of HYGAS pilot plant test operations, samples of candidate coals were obtained from several sources in the selected area. Two likely candidate mines were found that would be able to supply a steady source of coal for a long period. Coal from both sources were subjected to crushing tests, batch pretreatment tests, to and fluidization tests to determine processing conditions for pretreatment operations and to form a basis for selection of one of the coals. The results of these evaluation tests were reported under Task 1. A washed coal from the Peabody No. 10 Mine was selected as the feed material for HYGAS test operations.

Bench-Scale Unit Tests

The bench-scale PDU steam/oxygen gasifier was used to help establish the operating conditions for high carbon conversion (90% or more) of the Peabody No. 10 mine coal used in Test 59 onward. The volatile matter content in the raw coal was reduced (by extensive high temperature pretreatment) to about 5% to simulate steam/oxygen gasifier char feed material. The six-cone steam/oxygen gas distributor assembly was used in these tests.

The standard test procedure is described here. Preheated nitrogen is passed through the reactor for initial heat-up and feeding of the char is begun when the unit reaches 850°F. Oxygen and steam are then gradually introduced into the unit as the nitrogen flow is reduced to maintain a constant superficial velocity in the reactor. The reactor temperature is raised to 1800°F, and the pressure is maintained at ≃400 psig. Conditions are held constant and gas samples are obtained for the steady-state period.

In the initial PDU test with Peabody No. 10 Mine char, three hours after the initial introduction of oxygen into the unit, there was a pressure upset in the reactor when the nitrogen flow was removed. This resulted in a jammed solids screw feeder which 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, more successful PDU test was then carried out and results from these two tests are presented in Table 41. 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 slightly above 1800°F. Results of both tests show that Peabody No. 10 mine coal was gasified to yield carbon conversions of about 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 showed that complete fluidization is observed at 0.45 ft/s. As a result, pretreater char cooler fluidization velocities were kept above 0.45 ft/s at all times to ensure good fluidization and even temperature distribution.

Bed Heights

After a review of the results of Test 60, it was concluded that the process upset resulting in flooding of both the quench system and the gas purification system was caused by a) the high bed level in the slurry dryer area that caused excessive solids carryover, b) inefficient operation of

Table 41. 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, °F	1820	1820
Pressure, psig	399	398

the cyclone, and c) improper level control in the vessels because of plugged instrument sensing lines. It was decided that for Test 61 the slurry-dryer bed level would be controlled at a maximum of 5 feet, allowing a 15-foot freeboard, which should help to minimize fines elutriation. It was also decided to increase the surveillance of the operation of the cyclone system to ensure that the diplegs and slurry pot operated properly. With minimum fines elutriation from the reactor, the operation of the cyclone and slurry pot should be trouble-free. The same applies to the level control instrumentation on the quench vessels, which were cleaned, calibrated, and put back in service.

During Test 60, considerable difficulties were encountered in transferring solids from the second-stage reactor to the steam/oxygen gasifier. Data from Test 60 showed that the steam/oxygen gasifier bed was kept too deep because of low bed densities with this coal. With the additional pressure drop on the refractory grid caused by the refractory brick that had fallen from the Manway 3 insulating bricks, the seal capacity available on Standpipe 339 was marginal. This pressure imbalance caused the stoppage of solids flow to the steam-oxygen gasifier region. At times there were indications also that the second-stage gasifier was filled up, which might have caused problems in solids downflow from the slurry dryer bed to the lift-line gasifier. Therefore, it was decided that, for Test 61, the steam/oxygen gasifier bed level would be kept at 12 feet and the second-stage gasifier bed level would not exceed 15 feet.

After Test 61, it was decided that because of the importance of bed heights and pressure balance to the smooth operation of the HYGAS reactor, instantaneous bed-height and pressure-balance data would be printed out by computer in addition to the online material balance and engineering data already available to the operating personnel.

Quench System Study

Due to the quench system upset experienced during Test 62, a study was conducted to evaluate the behavior of the system on the following points:

a. Flooding in the towers

b. Pressure drop in the transfer lines and vessels

c. Vapor-liquid equilibrium in the towers

The results are discussed below according to the above points. First, the conditions applying during the past are tabulated.

Stream Composition and Flow Rates

A base pressure of 950 psia was assumed at the inlet to the prequench tower. All other pressures were determined from pressure drop calculations. The temperatures are averages from pertinent recorders from 0700 to 0900 hours on June 2 (Test 62).

Unless otherwise noted, all flow rates and compositions were obtained from the on-line material balance for 0800 hours on June 2 (Test 62). (Refer to Figure 91 for stream identification.)

Stream A — Quench Product Gas

P = 949.45 psia

T = 90°F

G = 5300 lb/hr; mol wt = 22.8 lb/lb-mol

Composition*	mol %
H_2	29.3
CH_4	21.3
CO_2	29.1
CO	11.1
N_2	7.7
C_2H_6	0.5
H_2S	1.1

* Average analysis 0500 to 0600 hours, June 2.

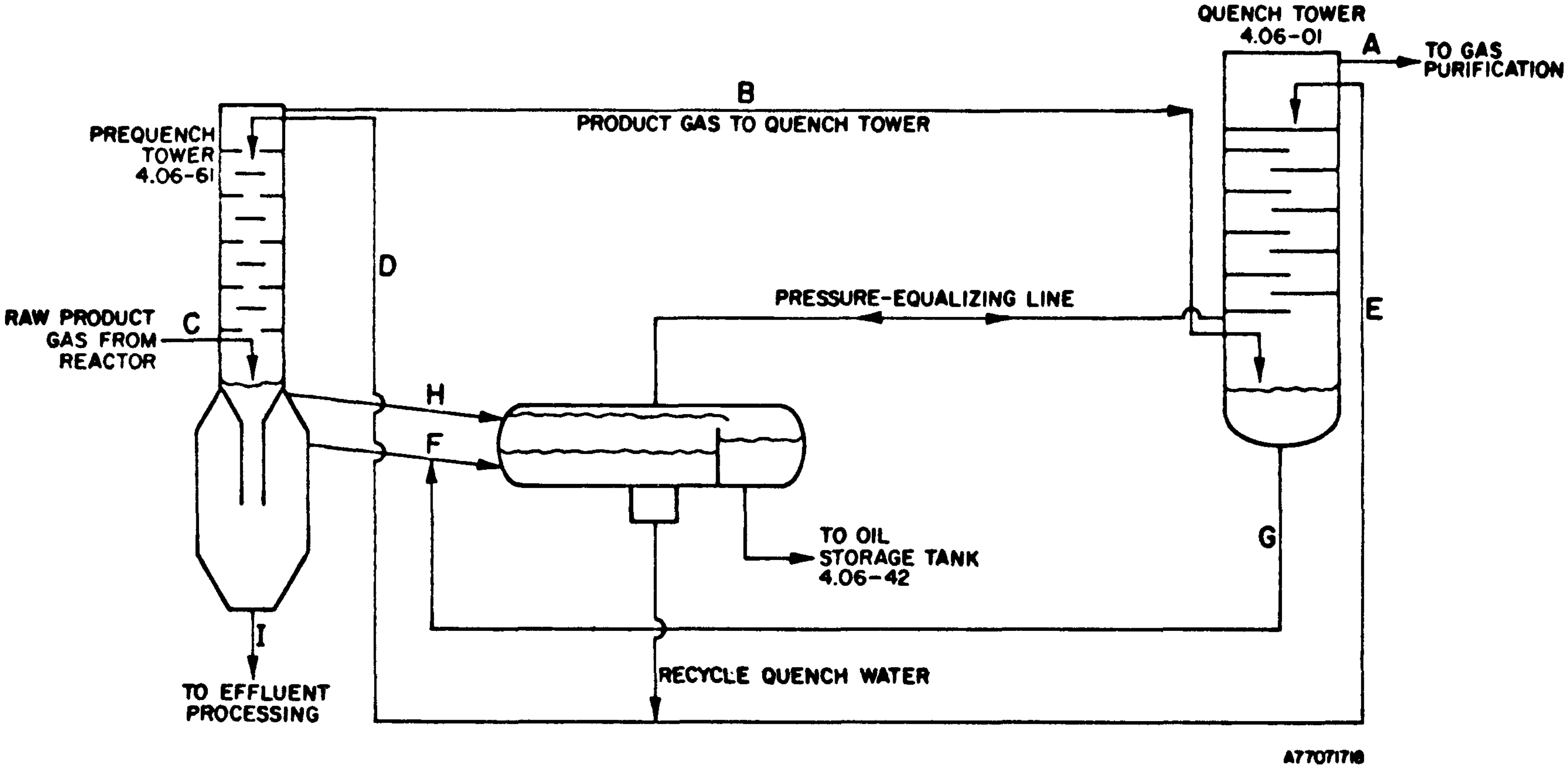

Figure 91. DIAGRAM OF THE HYGAS PILOT PLANT QUENCH AND OIL-WATER SEPARATION SYSTEM

Stream B

T = 100°F

Composition: Same as Stream A (i.e., only sensible heat removed in quench tower).

Stream C

P = 950 psia

T = 580°F

G = 800 lb-mol/hr

Composition*	mol %
H_2	8.73
CH_4	6.84
H_2S	0.38
C_2H_6	0.18
N_2	2.39 (mol wt = 33.8 lb/lb-mol)
CO_2	10.77
CO	3.30
H_2O	48.29
C_7H_8	16.87
C_6H_6	2.26

* From material balance on system.

Stream D

H_2O 150 gpm

Stream E

H_2O	125 gpm		

Stream F

H_2O	150 gpm;	3942.9 lb-mol/hr	T = 170°F
CO_2		70.97 lb-mol/hr	

Stream G

H_2O	125 gpm;	3470.8 lb-mol/hr	
CO_2		71.70 lb-mol/hr	

Stream H

Oil	25 gpm		

Stream I

H_2O	14 gpm	386.3 lb-mol/hr	T = 170°F
CO_2		6.95 lb-mol/hr	

Flooding

Flooding in Quench Tower (4.06-01)

The formulas[2] used here were –

$$V_c, \max = 1.15 \left(\frac{\rho_{\ell d} - \rho_g}{\rho_g}\right)^{1/2} = q/A_c$$

$$V_w, \max = 0.5\, V_c, \max = q/A_w$$

where –

q = volumetric flow rate, cu ft/s

A = sq ft

c = curtain area

w = window area

It was found that V_c, max = 4.5 ft/s and V_w, max = 2.3 ft/s. These are the maximum recommended velocities in the towers to prevent flooding.

The actual velocities found in the tower were –

$$V_c = 0.196 \text{ ft/s}$$

$$V_w = 0.118 \text{ ft/s}$$

The actual velocities are much less than the maximum velocities so that it can be safely assumed that there should be no problem of flooding in the tower.

Flooding in Prequench Tower (4.06-61)

The formula[1] used in this calculation was the ESSO correlation for disk and doughnut columns –

$$V_c = 4.05 \left(\frac{ST}{MP}\right)^{1/2}$$

where –

V_c = critical velocity, ft/s

S = specific gravity of liquid

T = temperature, R

M = molecular weight, vapor

P = pressure, psia

V_c is used here as the critical dimension for the column diameter. A value 3 times this determines the critical velocity in the open area between the trays.

The results were –

V_c at top of column = 0.654 ft/s; 3 X V_c = 1.962 ft/s

corresponding to –

V actual at 100°F = 0.062 ft/s

V_c at bottom of column = 0.72; 3 X V_c = 2.17 ft/s

V actual at 580°F = 0.410 ft/s

The conclusion is that the column is operating with an adequate safety margin to prevent flooding.

Pressure Drop Considerations (See the worksheets for calculations.)

The pressure drop for fluid flow in the quench system was calculated. A 5% occlusion of transfer pipes, caused by scale, was observed on these lines and taken into account in these calculations. Figure 91 shows relative liquid levels in the quench system to maintain proper fluid flow. It is apparent that these levels would not contribute to flooding of the quench or prequench towers.

Pressure Drop in the Vent Line From the Light-Oil Storage Tank (4.06-42)

A ΔP of 4.25 psi is expected (2.9 psi with swing-type check valve) for a gas (molecular weight = 38.7) flow of 1130 lb/hr through the light-oil storage vent line. With waste-gas header pressure at 10 psi, the light-oil storage tank (4.06-42) working pressure will be approximately 15 psi. A recording chart for Run 62 shows (for the most part) that this pressure remained under 12 psi. The vent line is, therefore, properly sized as long as it remains clean.

Flare

Flare Tip: 6 inches

Mass Flow Rate:

Product Gas: 5500 lb/hr; mol wt 23 = 240 lb-mol/hr

Vent Gas: 1000 lb/hr; mol wt 36 = 28 lb-mol/hr

H_2 (30% production): 24,000 SCF/hr = 63 lb-mol/hr

Total Flow 331 lb-mol/hr

P = 5 psig

T = 100°F

$$Q = 331\left[\frac{14.7}{(14.7+5)}\right]\left[\frac{(460+100)}{520}\right] 379 = 100,810 \text{ actual CF/hr}$$

$$A_F = \frac{\pi D^2}{4} = \frac{\pi(0.5)^2}{4} = 0.196 \text{ sq ft}$$

$$V = \frac{100,810}{(0.196)(3600)} = 143 \text{ ft/s}$$

$$V_{sonic} = \sqrt{g\,kRT/M} \approx \sqrt{(32.17)(1.2)(1546)(560)/20.1} = 1290 \text{ ft/s}$$

To prevent a blowout, V should be no more than 1/5 (1290) or 258 ft/s (rule of thumb).

John Zink Co. designed the flare for 2832 lb/hr of gas that has a molecular weight of approximately 25 with a safety factor of 4.0. We are currently running at a rate of 6650 lb/hr with gas that has a molecular weight of approximately 20. This rate is 55% of capacity.

Pressure Control Valves 401 and/or 502

$$Q = \sqrt{\frac{520}{G\cdot T}}\; C_g P_1 \left[\sin\left(\frac{3417}{C_1}\sqrt{\frac{\Delta P}{P_1}}\right)_{deg}\right] \text{ for each valve}$$

$Cg = 180$

$C_1 = 34.7$; mol wt = ~22

G (specific gravity of gas) = mol wt/28.9

$$Q = \sqrt{\frac{520}{(22/28.9)(89 + 520)}} \quad 180 \cdot P_1 \; [\sin (\frac{3417}{34.7} \sqrt{\frac{\Delta P}{P_1}})]$$

$$= 190.6 \; P_1 \; [\sin (98.47 \; \frac{\Delta P}{P_1})]$$

P in line to the flare, waste-gas header = ~5 psig.

P_1 = ~1000 psig

sin term = ≃ 1.00

Q = 190.6 (P_1) = 190,000 SCF/hr.

This is multiplied by 2 (pressure control valves PV 401 and PV 502). Q ~380,000 SCF/hr as limited by the control valve, when reactor pressure is 1000 psig, or ~ 22,000 lb/hr. This is the approximate maximum flow to flare (from product-gas line) as limited by plant pressure control valves.

Vapor-Liquid Equilibrium

K-values for light oil-product gas equilibrium were obtained using the Chem Share DISTIL program, which employs Chao-Seader thermodynamics for the calculation of K-values. These values were then used to predict the light-oil storage tank vent-gas rate and composition. The approximation of two-phase equilibrium (really three phase) was employed. The following results were obtained.

Light-Oil Storage Tank (4.06-42)

T = 90°F

	Predicted				Observed	
	P = 40 psia		P = 18 psia		Avg P	35 psia
Composition	lb-mol/hr	mol %	lb-mol/hr	mol %	lb-mol/hr	mol %
H_2	1.71	6.5	1.72	5.89	1.28	5.5
CH_4	4.61	17.6	4.79	16.43	4.15	17.80
H_2S	0.64	2.4	0.84	2.89	0.33	1.4
C_2H_6	0.25	1.0	0.29	1.00	0.20	0.84
N_2	0.57	2.2	0.56	1.99	1.00	4.3
CO_2	16.74	63.7	17.77	60.94	15.07	64.6
CO	1.08	4.1	1.10	3.76	0.45	1.9
C_7H_8	0.48	1.8	1.49	5.12	0.51	2.2
C_6H_6	0.18	0.8	0.58	1.99	0.35	1.5
Total	26.26(9950 SCF/hr)		29.14(11,050 SCF/hr)		23.34(8850 SCF/hr)	

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These predictions were made on the basis that equilibrium is reached in all quench system vessels. This is reasonably accurate in the condensing conditions of the prequench and quench towers. In the quench separator, if equilibrium is reached between the oil and water phases, an exchange of 6 lb-mol of CO_2 from the water to the oil phase will result. The assumption of equilibrium, however, is poor in this case.

Predicted results are very close to results obtained during normal operation. It is, therefore, concluded that flow through the 4.06-42 vent will range between 8000 and 12,000 SCF/hr under normal operating conditions. The associated pressure drop in the vent line will be approximately 5.0 psi at maximum flow rates.

K-Values Obtained From Chem Share DISTIL Program

Component	P=18 psia T=90°F	P=40 psia T=90°F	P=949.4 psia T=85°F	P=949.4 psia T=132°F	P=950 psia T=170°F
H_2	2276.9	1016.5	37.58	31.83	27.28
CH_4	286.32	129.03	6.37	6.93	7.27
H_2S	32.22	14.612	0.83	1.18	1.52
C_2H_6	67.75	30.721	1.82	2.19	2.50
N_2	1317.7	591.42	26.28	24.26	22.18
CO_2	147.1	66.488	3.57	4.07	4.44
CO	852.7	383.24	17.63	16.97	16.06
C_7H_8	0.058	0.027	0.0036	0.0075	0.014
C_6H_6	0.175	0.081	0.0091	0.018	0.032

Summary of Observations

The following abnormalities were found in the quench system:

1. Small amount of solids on the quench tower baffles (4.06-01)
2. Lower six baffles in quench tower corroded (4.06-01)
3. Oil line between prequench tower and quench separator completely plugged (Stream H)
4. Gas line between prequench and quench tower partially blocked (Stream B).

Observations 1 and 2 have no effect on the operation of the quench system. Item 3 would cause an increase in flow through the water line between the prequench tower and the quench separator. The increase in pressure drop would be approximately 4 inches H_2O. This would raise the liquid level in the prequench tower by 4 inches. It should not cause the flooding problems observed.

In item 4, a short plug occluding 92% of the gas line, or a 12-foot-plug, occluding 42% of the gas line, would cause a 3-psi pressure drop in the line. This can lead to gas flow through the liquid lines from the prequench tower to the quench separator and stop water return from the quench tower. This, in turn, may lead to entrainment of liquid from the quench tower into the raw product-gas line.

Worksheets

Entrainment Calculations[2] for Quench Tower 4.06-01

$$V_c, \text{ max} = 1.15 \left(\frac{\rho l - \rho g}{\rho g}\right)^{1/2} = \frac{q}{A_c}$$

V_w, max = 0.5 V_c, max = q/A_w

q = volumetric flow rate cu ft/s, $A_{c,w}$ are window and curtain areas, sq ft

V_c, V_w = maximum velocities

q $\cong$ 5500 lb/hr of gases (mol wt = 23)

$$\rho g = \frac{PM}{RT} = \frac{(1000)(23)}{(10.73)(90 + 460)} = 3.9 \text{ lb/cu ft} \quad \rho l = 64 \text{ lb/cu ft}$$

$$M = \rho VA; \quad \frac{M}{\rho A} = V$$

$$A_c = \frac{12 \text{ in. X } 24 \text{ in.}}{144} = 2.0 \text{ sq ft} \qquad A_w = \frac{\pi (2)^2}{4} + 2.\frac{1}{12} = 3.31 \text{ sq ft}$$

$$V_c, \text{ max} = 1.15 \left(\frac{64-3.9}{3.9}\right)^{1/2} = 4.51 \text{ ft/s} \quad V_w, \text{ max} = 2.25 \text{ ft/s}$$

$$V_c = \frac{5500}{(3.9)(2.0)(3600)} = 0.196 \text{ ft/s}$$

$$V_w = \frac{5500}{(3.9)(3.31)(3600)} = 0.118 \text{ ft/s}$$

Therefore, the velocities are only 1/20th of the maximum allowable velocities and the tower cannot be considered to be flooded.

Flooding in Prequench Tower[1]

$$V_c = 4.05 \left(\frac{ST}{MP}\right)^{1/2}$$

V_c = critical velocity in ft/s for tower diameter

S = specific gravity of liquid

T = temperature, ^{o}F

M = molecular weight of vapor

P = pressure, psia.

The column has a 3.0 foot inside diameter.

5500 pph, mol wt = 23 $\quad \rho_g = 3.9$ lb/cu ft

$$\text{in} = \rho_g VA \qquad A = \frac{\pi(3)^2}{4} = 7.07 \text{ sq ft}$$

$$\left(\frac{5500}{3600}\right)\left(\frac{1}{7.07}\right)\left(\frac{1}{3.9}\right) = \text{ ft/s in tower at } 100^{o}\text{F exit temperature}$$

$$= 0.055 \text{ ft/s actual velocity in } 4.06\text{-}61$$

$$V_c = 4.05\left(\frac{1.0.600}{23\cdot 1000}\right)^{1/2} = 0.654 \text{ ft/s}$$

Free-flow area = 1 $\pi \cdot 2$ = 6.28 sq ft

Here we use V_c X 3.00 = 1.962 max

$$V \text{ actual} = (0.055)\left(\frac{7.07}{6.28}\right) = 0.062 \text{ ft/s}$$

In conclusion, the tower was not at flooding conditions at the exit. At the inlet –

mol wt = 33.8

~ 800 lb-mol/hr

in = 27,040 lb/hr = 7.51 lb/s

$$\rho = \frac{(965)(338)}{(10.73)(460 + 580)} = 2.92 \text{ lb/cu ft}$$

$$V = \frac{M}{\rho A} = \frac{7.51}{2.92\cdot 6.78} = 0.4095 \text{ actual ft/s}$$

$$V_c = 4.05\left(\frac{ST}{MP}\right)^{1/2} = 4.05\left[\frac{(1.0)(580 + 460)}{(33.8)(965)}\right]^{1/2} = 0.723 \text{ ft/s}$$

or 2.17 ft/s max

Therefore, conditions are good at the gas inlet, too.

Pressure Drop Calculations

Storage tank vent line –

Straight 24-foot pipe	L/D = 150
6 to 90-degree elbows	L/D = 180
3 gate valves	L/D = 40

1 check valve	L/D = 450
Expansion and Contraction	L/D = 130
(Entrance, Exit, Flame Arrestor, and Orifice)	$(L/D)_t = 950$

$Q = 9498.2$ actual CF/hr

$\rho = 0.1189$ lb/sq ft

$\mu = 0.01358$ cp

$V = 128.7\ \beta_{ps}$

$$Re = \frac{DV\rho}{\mu} = \left(\frac{1.939}{12}\right)\left[\frac{(128.7)(0.1189)}{(0.01358)(6.72 \times 10^{-4})}\right] = 270,900$$

$$f = 0.021 = \frac{h_l}{(L/D)\frac{v^2}{2g}} \qquad h_l = \frac{(0.021)(950)(128.7)^2}{(2)(32.2)} = 5131$$

$$\Delta P = \frac{(5131)(0.1189)}{144} = 4.25 \text{ psi}$$

Pressure Drop in Gas Line Off of Prequench Tower 4.06-61

Stream B

Line Size: 4-inch Schedule 120 (72-ft pipe + 7 elbows)

L/D: 238 + 350

Gas

5500 pph; 1000 psi = 100°F; mol wt = 23.

30%	CO_2
30%	H_2
20%	CH_4
10%	CO
10%	N_2

di = 3.624 in.

= 0.033 lbm/ft-hr

Ax= 0.07163 sq ft

$$W = \frac{5500}{0.07163} = 146,471 \text{ lb/sq ft-hr}$$

V = 12.5 ft/s

$$\rho = \frac{PM}{RT} = \frac{(1000)(23)}{(10.73)(560)} = 3.90$$

$$Re = \frac{(\frac{3.624}{12})\ 146,471}{0.033} = 3,340,000$$

$$f = 0.017 = (\frac{L}{D})(\frac{V^2}{2g})$$

$$588(0.017)\frac{(12.5)^2}{2(37.2)} = h_L = 24.25\ \frac{ft\text{-}lb_f}{lb_m}$$

$$\frac{(24.25)3.90}{144} = 0.65 \text{ psi or} \approx 18 \text{ in. } H_2O$$

Stream G (125 gpm H_2O)

Pipe: 6-inch Schedule 120, 5% occlusion of pipe from observation

Straight: 40 ft D = 5.501 − 0.275 = 5.226

Four 90-degree bends L/D = 200

1 Tee L/D = 60

$$Leq = (206)(\frac{5.501}{12}) = 120 \text{ ft}$$

Leq (entrance and exit) = 40 ft

Total Leq = 200 ft

$$\Delta P = 0.055\ (\frac{psi}{100\ ft})(\frac{6.065}{5.226})^5 \times \frac{200\ ft}{100\ ft}\ (2.77 \text{ in./psi}) = 6.4 \text{ in. } H_2O$$

Stream F (150 gpm)

Pipe: 6-inch Schedule 120, 5% occlusion of pipe (observation)

Straight Run: 35 ft

Four 90-degree bends, 1 Tee (L/D = 260) Leq ≐ 120 ft

Leq (entrance and exit) = 40 ft

Total Leq = 195 ft

$$\Delta P = 0.077\ (\frac{psi}{100\ ft})(\frac{6.065}{5.226})^5\ (1.95)(27.7) = 8.8 \text{ in.}$$

Streams F + G (275 gpm)

Straight Run: 2 ft

Exit: 15 ft

$0.275 \left(\frac{6.065}{5.226}\right)^5 (0.017)(27.7) = 2.3$ in.

Stream H (25 gpm oil)

Pipe: 4-inch Schedule 120

Straight pipe: 40 ft

Four 90-degree turns (L/D = 200)

$Leq = 200 \text{ X } \frac{3.624}{12} - 60$ ft

$Leq = 60 + 40 = 100$ ft

$\Delta P = (0.020) \left(\frac{4.027}{3.624}\right)^5 (1)(27.7) = 0.9$ in. H_2O

ΔP between prequench and separator because of fluid flow = 11.1 in. H_2O

ΔP between quench and separator because of fluid flow = 8.7 in. H_2O

ΔP between prequench and quench because of gas flow = 18 in. H_2O

References Cited

1. Esso correlation.

2. Fair, J. R., "Designing Direct-Contact Coolers and Condensers," Chem. Eng. (1972) June 12.

FUTURE WORK

As of July 1977, ERDA contract No. EF-77-C-01-2434 was continued through September 1978. The objective of work conducted after July 1977 is to perform the necessary pilot plant operations and related support studies required to acquire data for a commercial/demonstration plant design based on the HYGAS process.

Tasks 1 through 6 were completed during the period covered by this interim report. After July 1977, work began on Tasks 7 through 9: Pilot Plant Experimental Operation, Demonstration Plant Support, and Support Studies. The work conducted under Tasks 7 through 9 will be summarized in a subsequent report.

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