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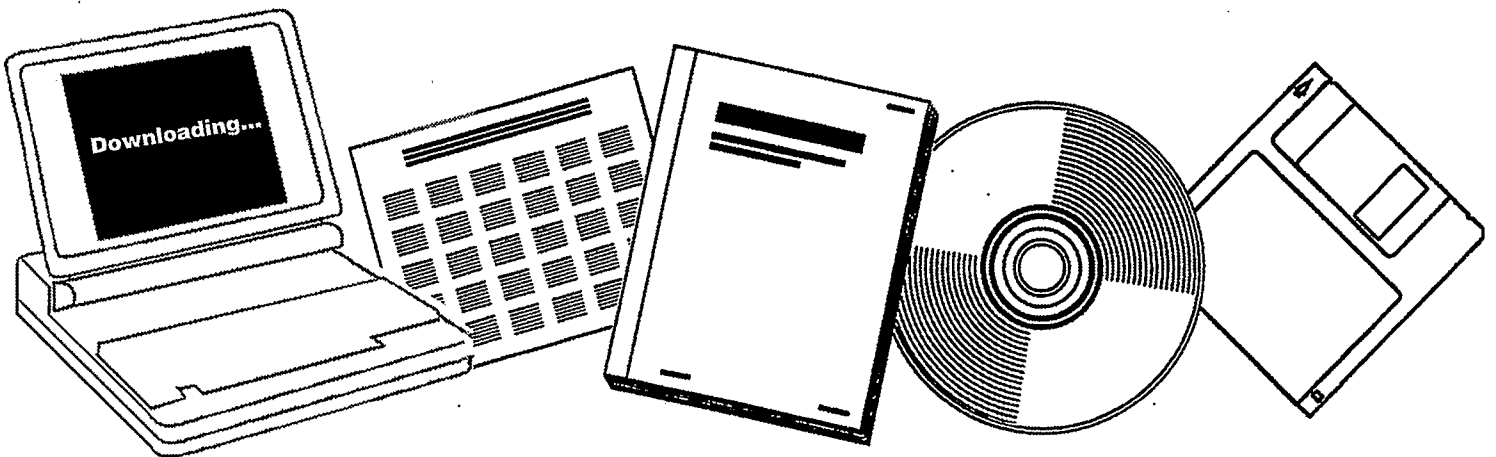
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**HYGAS: 1964 TO 1972. PIPELINE GAS FROM  
COAL--HYDROGENATION (IGT HYDROGASIFICATION  
PROCESS). VOLUME 2**

INSTITUTE OF GAS TECHNOLOGY, CHICAGO,  
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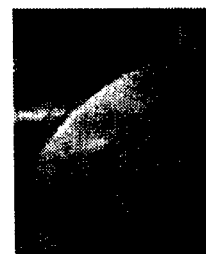
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HYGAS<sup>TM</sup> : 1964 TO 1972  
PIPELINE GAS FROM COAL — HYDROGENATION  
( IGT HYDROGASIFICATION PROCESS )

R&D REPORT NO. 22 FINAL REPORT

VOLUME 2

PART III : PILOT PLANT DEVELOPMENT  
PART IV : HYDROGEN GENERATION

OCR Contract No.

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Institute of Gas Technology  
IIT Center  
Chicago, Illinois 60616

HYGAS<sup>(T.M.)</sup> : 1964 TO 1972  
PIPELINE GAS FROM COAL — HYDROGENATION  
( IGT HYDROGASIFICATION PROCESS )

Final Report

by

**IGT Process Research Division**

Prepared for

**ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION**

Washington, D.C. 20545

PART III  
PILOT PLANT DEVELOPMENT

3-i-d

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### 3.0 Summary of Pilot Plant Development

The preliminary engineering design of the HYGAS pilot plant was performed by Bechtel Corporation in association with IGT.

The final design and construction of the HYGAS pilot plant was performed by Procon, Inc., a division of UOP, in association with IGT.

Detailed discussion in Part III covers process chemistry, preliminary plant design, design modifications and construction, and start-up operations. Problems are delineated as well as their solutions, and findings and recommendations applicable to other coal hydrogasification plant designers are informally presented.

The first seven start-up test runs are reported in detail; however, the limited amount of operational data obtained during the contract period made it inadvisable to present that material here. Instead, with the consent of OCR, operational data from the period reported here will be presented in more meaningful perspective in reports of the subsequent HYGAS contract sponsored by OCR with the support of the A. G. A. (OCR Contract No. 14-32-0001-1221, now ERDA Contract No. E(49-18)-1221.)

The HYGAS process for the hydrogasification of coal is tailored to maximize the direct production of methane in the hydrogasification reactor by the use of high temperature (1200°-1850°F) and high pressure (1000 psig). The process can use as feed caking bituminous coals as well as noncaking lignite and subbituminous coals.

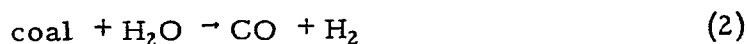
The HYGAS process is based on gasification studies that started in 1944. By the mid-1950's, two processes to convert coal to synthetic pipeline gas were being developed: 1) gasification of powdered coal in suspension with oxygen and steam to produce a mixture of carbon monoxide and hydrogen (synthesis gas), which was then methanated, and 2) direct hydrogenation of the coal at elevated temperature and pressure in a fluidized bed. The present HYGAS process incorporates the principles developed in both of these concepts.

Of the total methane formed in the HYGAS process, 65-70% is directly formed in the hydrogasifier. This is a key feature of the process, and contributes significantly to the high overall thermal efficiency of more than 70%. If the process were operated at a lower pressure, more methane would need to be made indirectly, and losses in efficiency would result.

The second key feature of the process is the use of hydrogen and steam in hydrogasification. The coal-hydrogen reaction,



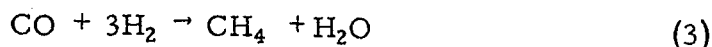
is strongly exothermic, while the coal-steam reaction,



is strongly endothermic. By using a mixture of hydrogen and steam instead of hydrogen alone, the heat released by Reaction 1 is absorbed in situ by Reaction 2, resulting in

- Built-in temperature control, and
- Internal hydrogen generation.

The raw gas from the hydrogasifier contains carbon monoxide and hydrogen. These are converted to methane through Reaction 3,



in the catalytic clean-up methanation unit, where the carbon monoxide content is reduced to the pipeline standard of less than 0.1%, and the heating value of the product gas is raised to satisfactory SNG levels.

In the pilot plant, the hydrogasifier reactor vessel is 135 feet high; the outer pressure shell has a 5.5-foot inside diameter. The slurry is sprayed into the dryer, a 2.5-foot-diameter, 10-foot-high fluidized drying bed. The sensible heat in the gaseous reaction products from the later stages vaporizes the oil. At this point, the dry coal is heated to about 600°F.

The coal flows by gravity from the drying bed into a 3-inch-diameter, vertical-lift-line reactor in which the hot gases (1700°F) from the reaction section below provide the lifting force, the heat to raise the solids temperature to 1200°F, and hydrogen that reacts with about 20% of the coal to produce methane. This is the first stage of hydrogasification — the low-temperature reactor. At the top of the lift line, the gas and coal disengage. The gas moves up to vaporize the oil in the slurry-drying bed. The partially reacted coal can be split into two streams; part of it can be transferred to the base of the lift line to be mixed with the incoming fresh coal. By this means, IGT believes, raw caking coal can be fed directly to the reactor without pretreatment. Eliminating pretreatment can reduce gas cost significantly.

The remainder (or all) of the partially reacted coal flows by gravity to the second-stage hydrogasifier — the high-temperature reactor. The second-stage bed is 2.5 feet in diameter, is lined with refractory, and is 15 feet deep. Here the solids are heated in a fluidized bed to about 1700°F and further gasified by the steam and hydrogen-rich gas rising from the steam-oxygen gasification\* below. (Or, alternatively, the hydrogen-rich gas may flow from the steam-iron reactor or the electrothermal generator.)

In this second stage of the reactor, the exothermic hydrogen reaction produces methane, and the endothermic steam reaction produces carbon monoxide and hydrogen. If the temperature rises, the steam-char reaction speeds up and prevents the temperature from rising any further. If the temperature drops, the steam-char reaction slows down and thus provides an automatic temperature control. About 25% more of the coal is converted in this reaction stage, making the total about 45% in the two stages.

---

\* An electrothermal gasifier was built in the period reported; a steam-oxygen gasifier was installed after this contract period; a steam-iron gasifier is under construction in mid-1975.

From this reaction stage, the char goes to the hydrogen-producer gasifier, where, depending on the process being used, the char undergoes different degrees of additional gasification. The steam-oxygen gasifier\* being used as the hydrogen producer in the pilot plant is directly below the high-temperature stage hydrogasifier. The steam and high-purity oxygen introduced into the gasifier convert char into hydrogen and carbon oxides at 1850°F in a fluidized bed 2 feet in diameter and 12 feet deep. Ash is discharged from this stage without being slagged. The ash is discharged into a tank where water is added to make a slurry, which is then depressurized. The ash is recovered by filtering and the water is recycled.

The composition of gas synthesized in the two principal reactor stages, and passed upward through the slurry dryer at the top of the hydrogasification reactor, depends on the type of hydrogen producer, as shown in the following table. In addition to these major components, the gas contains the slurry oil, coal dust, and trace constituents such as ammonia and hydrogen cyanide.

#### GAS COMPOSITION LEAVING HYGAS REACTOR

<u>Component</u>	<u>Steam-Oxygen System</u>	<u>Steam-Iron System</u>	<u>Electrothermal System</u>
CO	18.0	7.4	21.3
CO <sub>2</sub>	18.5	7.1	14.4
H <sub>2</sub>	22.8	22.5	24.2
H <sub>2</sub> O	24.4	32.9	17.1
CH <sub>4</sub>	14.1	26.2	19.9
C <sub>2</sub> H <sub>6</sub>	0.5	1.0	0.8
H <sub>2</sub> S	0.9	1.5	1.3
Other	<u>0.8</u>	<u>1.4</u>	<u>1.0</u>
Total	100.0	100.0	100.0

The gas mixture delineated is at 600°F; it passes to a baffle tower in which it is quenched and washed with water. This removes the dust and water-soluble trace components and condenses the water and light-oil vapors. The gas then flows to a conventional, packed-tower acid-gas removal system in which the carbon dioxide and the hydrogen sulfide are absorbed in a diglycolamine-water solution. Upon regenerating this solution, the carbon dioxide and hydrogen sulfide are released and flow to a Claus plant for sulfur recovery. The amine purification system used in the pilot plant is not intended as a commercial design because it does not provide for separate collection of the various constituents. It was selected because it can handle the wide range of acid-gas concentrations arising from the various coals to be tested.

\* Installed in the base of the existing HYGAS reactor vessel after the close of this contract period.



Calculations indicate that the purified gas entering the methanation section of a commercial plant would typically have the composition tabulated below. The methanation step has two purposes: One is to raise the heating value of the gas to near that of methane; the other is to reduce the carbon monoxide concentration to the requisite 0.1% or less. This is accomplished by carrying out Reaction 3, given earlier.

#### GAS COMPOSITION ENTERING THE HYGAS METHANATOR

<u>Component</u>	<u>Steam-Oxygen System</u>	<u>Steam-Iron System</u>	<u>Electrothermal System</u>
CO	18.0	12.8	16.8
CO <sub>2</sub>	2.0	2.0	2.0
H <sub>2</sub>	54.0	38.5	50.5
CH <sub>4</sub>	25.0	45.0	29.5
C <sub>2</sub> H <sub>6</sub>	<u>1.0</u>	<u>1.7</u>	<u>1.2</u>
Total	100.0	100.0	100.0

To obtain nearly complete elimination of carbon monoxide and low residual hydrogen (pipeline-quality gas) in the methanation section, the ratio of hydrogen to carbon monoxide is adjusted to slightly above 3\*. High pressure and low temperature favor completion of the methanation reaction i. e. — in the methanator. Very reactive, high-nickel-content catalysts are generally preferred to make the reaction proceed rapidly at the low temperatures employed. The temperature of the catalyst must be above 450° F to avoid formation of nickel carbonyl, which causes depletion of the nickel content of the catalyst, and below about 950° F to avoid carbon deposition and catalyst sintering. The reaction is very exothermic; therefore, to avoid excessive temperature rises, the HYGAS process uses a cold-gas recycle system.

### 3.1 Introduction

Early laboratory and bench-scale research on the hydrogasification of coal presented in Part II of this report was performed at IGT under sponsorship of the American Gas Association. By the early 1960's, the IGT work had reached a stage where expanded facilities were required. Beginning on July 29, 1964, the program gained momentum under sponsorship of the Office of Coal Research, U. S. Department of Interior, through OCR Contract No. 14-001-0001-381, with sponsorship participation by A. G. A.

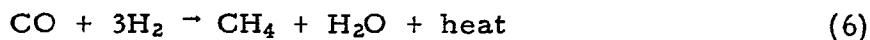
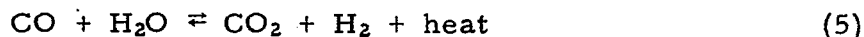
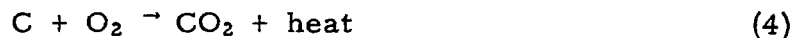
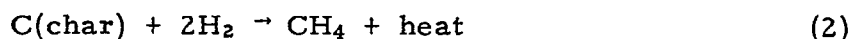
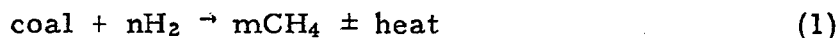
A principal focus of the expanded work was the design, construction, and start-up of the HYGAS pilot plant at an IGT facility in Chicago. Only that portion of funds provided by OCR was used in construction; therefore, the plant is totally U. S. property. The sections that follow describe the

\* A water-gas shift section would be installed for this purpose in a commercial operation.

preliminary engineering design, final design, and construction of the HYGAS pilot plant by IGT and its principal subcontractors, Bechtel Corporation (preliminary engineering design), and Procon Inc., a subsidiary of UOP (final design and construction). The section ends with a description of start-up operations and problems. All process flow diagrams and tables of stream data in this Part III of the Final Report HYGAS 381 represent Bechtel preliminary design configurations and data. Figures 3-1 through 3-6 represent the plant configuration at the time that start-up tests began.

### 3.2. HYGAS Process Considerations

The HYGAS process takes into account the advantages and disadvantages of the six basic coal gasification reactions, and applies contacting methods best suited for each process situation. The reactions are -



The process and plant design attempted to maximize reactions 1 and 2 in such a manner that the heat generated from these reactions can be used to promote reaction 3. Thus, the HYGAS process design incorporates reaction concepts that enhance reactivity, improve direct methane yield, conserve energy with built-in thermal controls and, at the same time, provide optimum system efficiency.

The high methane concentrations in typical HYGAS gas compositions are a result of the high-pressure operation, in excess of 1000 psi, and of the staging of gasification steps for the system. For example, because reaction 1 proceeds rapidly, this operation is carried out in a suspension, or transport contactor. Because reaction 2 is somewhat slower, and produces considerable heat, it is carried out in a fluidized-bed contactor that provides 1) longer residence times for the char - sufficient to undergo gasification, and 2) ample contacting time between the char and the steam present in the gas; thus enabling reaction 3 to absorb much of the heat produced by reaction 2.

The plant is designed to produce about 1.5 million standard cubic feet per day of pipeline-specification Substitute Natural Gas (SNG) when the coal feed rate to the hydrogasification section is 3 tons per hour of bituminous coal, or about 1.0 million standard cubic feet per day of SNG when the coal feed rate to the hydrogasification section is 3 tons per hour of lignite.

The design output of by-product char, and of oil and gas streams to disposal are shown on the material balance and physical properties tables accompanying various process flow diagrams that appear in this section.

The design is predicated on coal feed consisting of bituminous coal, such as Pittsburgh No. 8 from the Ireland mine, or of lignite, such as that from the Savage mine in North Dakota, having the following typical ultimate analyses:

<u>Components</u>	<u>Bituminous</u> (dry wt/%)	<u>Lignite</u> (dry wt/%)
Carbon	71.50	65.60
Hydrogen	5.02	4.38
Sulfur	4.42	0.78
Nitrogen	1.23	0.92
Oxygen	6.53	19.87
Ash	11.30	8.55

The design is also predicated on the use of natural gas both for fuel and for hydrogen plant feed. The natural gas is presumed to have the following typical analysis:

<u>Components</u>	<u>Vol/%</u>
Carbon Dioxide	0.72
Helium	0.10
Nitrogen	3.30
Methane	87.9
Ethane	5.6
Propane	1.7
n-Butane	0.31
iso-Butane	0.24
C-5's	0.11
C-6's	0.02
C-7's	0.02
Gross heating value	1040 Btu/SCF
Specific gravity	0.630
Pressure, psig	30 psig
Temperature	Ambient

(The odorant level was established as 0.75 lb of "BP Captans" per million cubic feet of gas)

In the initial HYGAS pilot plant design, the gases required to support the second stage of hydrogasification were introduced to the hydrogasification section at the base of the main reactor vessel. These gases were brought up to system temperature by direct-fired superheaters and through contact with a heat-exchange bed of partially gasified char at the bottom of the main reactor vessel. Gases introduced were hydrogen obtained

by reforming methane and steam. By mid-1974, modifications and additions available or under construction at the pilot plant included provision at the site to provide hydrogen by any of three hydrogen-rich gas producing systems, reported in Part IV: Hydrogen Generation, of this Final Report HYGAS 381. More detailed information on these innovations is available in Interim Report No. 1 HYGAS 1221, covering operations between September 1972 and 1974, which was submitted to OCR August 15, 1974.

The preliminary design of the HYGAS pilot plant, described here, was intended to introduce an optimum selection of the various major design options available in 1968, when the design work was begun. Note, especially, that the use of separate reactor stages permits the sequential employment of different sets of reactions to optimum advantage.

### 3.3 Preliminary Design: General

The block flow diagram in Figure 3-1 shows the principal original sections of the HYGAS pilot plant in Chicago.

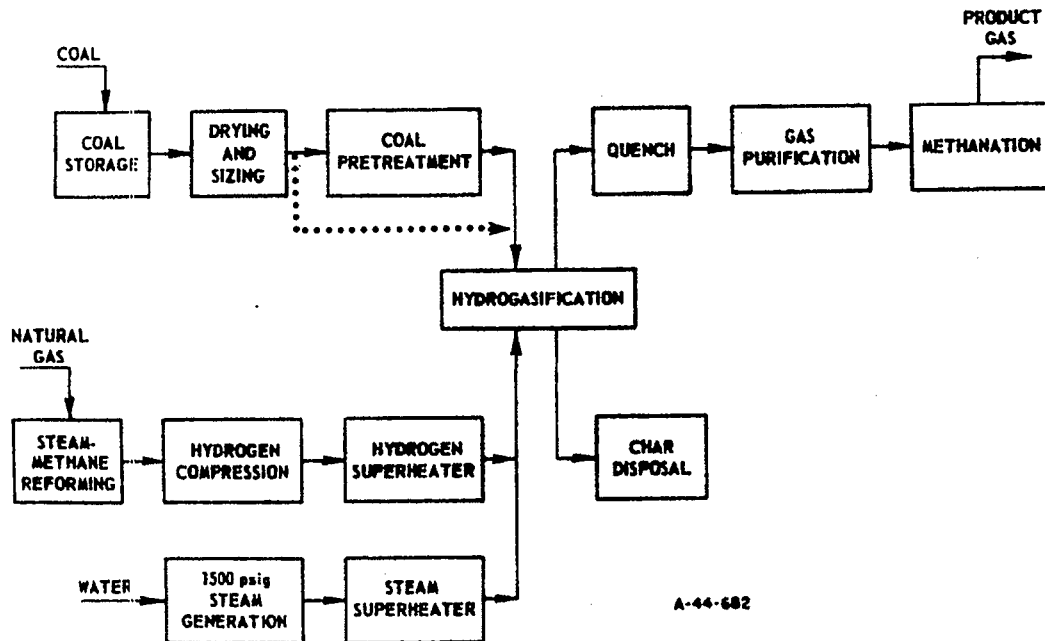


Figure 3-1. BLOCK FLOW DIAGRAM OF HYGAS PILOT PLANT AT START-UP

Brief descriptions of major pilot plant sections follow.

### 3.3.1. Raw Coal Handling

Coal cars deliver feed to the raw coal handling section (Figure 3-2) and the coal is dumped and conveyed to storage.

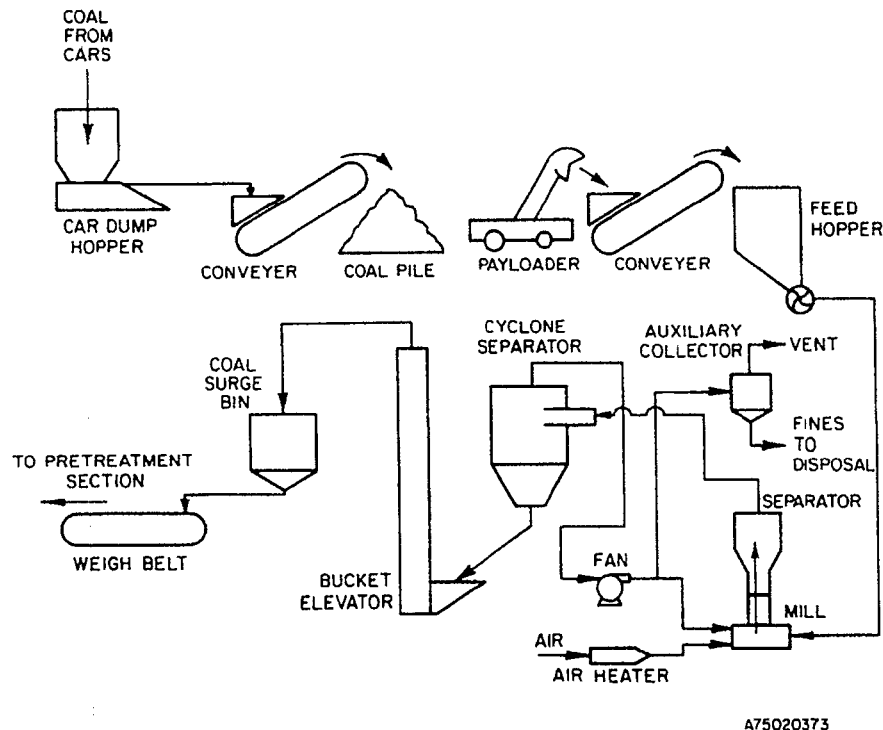


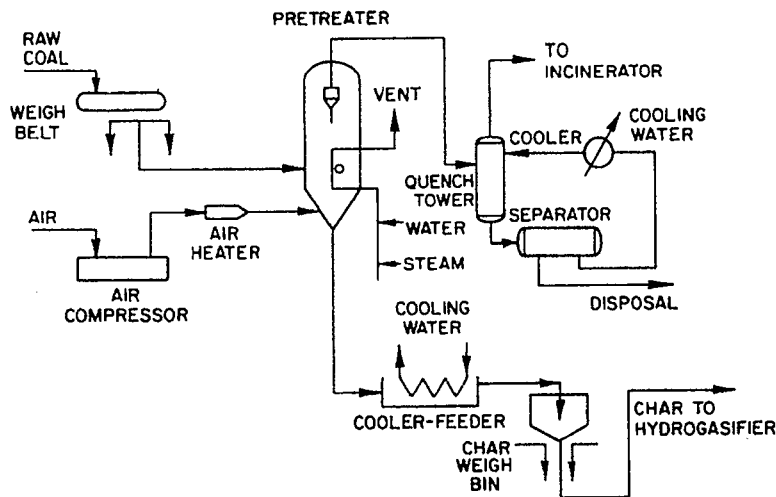
Figure 3-2. RAW COAL HANDLING IN HYGAS PILOT PLANT AT START-UP

The coal is dried and crushed to the proper size in the mill. Hot combustion gases and recycle gases are used to dry the coal as received before it enters the mill system. Air, crushed coal of the required size, and fines, exit via the separator. The crushed coal is separated from the air and fines in the cyclone separator. The air and fines leave the system through the fan and auxiliary collector. If pretreatment is required to eliminate the tendency of some coals to agglomerate, the coal is conveyed to a coal surge bin, weighed, and fed into the pretreatment section.

### 3.3.2. Coal and Char Pretreatment

The pretreatment section (Figure 3-3) is required only if an agglomerating coal is being used as feed. Here, crushed, raw coal is weighed and charged to the pretreater vessel, where it is dried and slightly oxidized by contact with air. The pretreater is a fluidized-bed reaction vessel that utilizes an in-line air heater for start-up. In operation, excess heat is removed from the fluid bed by steam generation coils in the bed. The pretreated coal or char is discharged into a quench tower, cooled, and sent to storage. The pretreater off-gas is cooled in the first

stage quench system to remove tars, heavy oils, and fines. The off-gas and oily water go to disposal.



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Figure 3-3. PRETREATMENT SECTION OF HYGAS PILOT PLANT AT START-UP

### 3.3.3 Hydrogasification Section

The principal process units of the hydrogasification section are shown in Figure 3-4. Here, the sized and dried coal (or in the case of agglomerating coal, the pretreated char) is weighed and charged into a slurry mix tank. The feed is mixed with a light aromatic oil that is a process by-product, to form a slurry, and is circulated through the intake manifold of a positive displacement pump. The positive displacement pump feeds it at process pressure (above 1000 psi) through a slurry heater, to the top of the 132-foot-high hydrogasifier vessel. Upon entering the top of the vessel, the slurry is dried by the action of the rising hot gases leaving the product gas separator using a fluidized-bed contactor. The pretreated, heated, dried solids then enter the first of two continuous-flow reactors located beneath the slurry drying bed.

Solids flowing from the drier into the first-stage reactor enter a stream of fast-moving, rising gas from the lower, second-stage reactor. The gas from the second stage heats, lifts, and reacts with the dried feed. The mixture passes upward to the product gas separator where a rapid expansion of reactor diameter causes the stream velocity to diminish, and solids drop out and collect for transfer to the second-stage reactor bed. The rising mixture of solids and gases in the first-stage cocurrent flow undergoes reactions that directly form approximately one-third of the methane produced in this process using reaction 1. A recycle stream

of solids also can be returned to the first-stage reactor for process temperature control. The product then rises to the drying section.

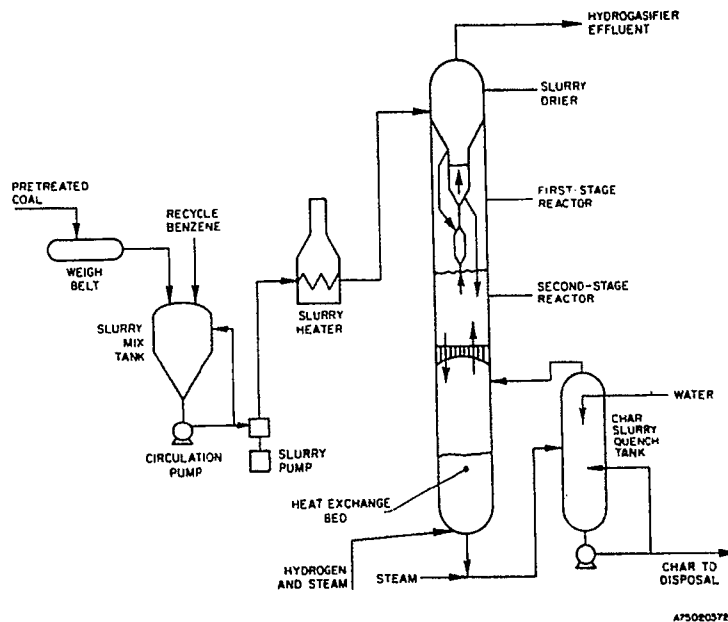
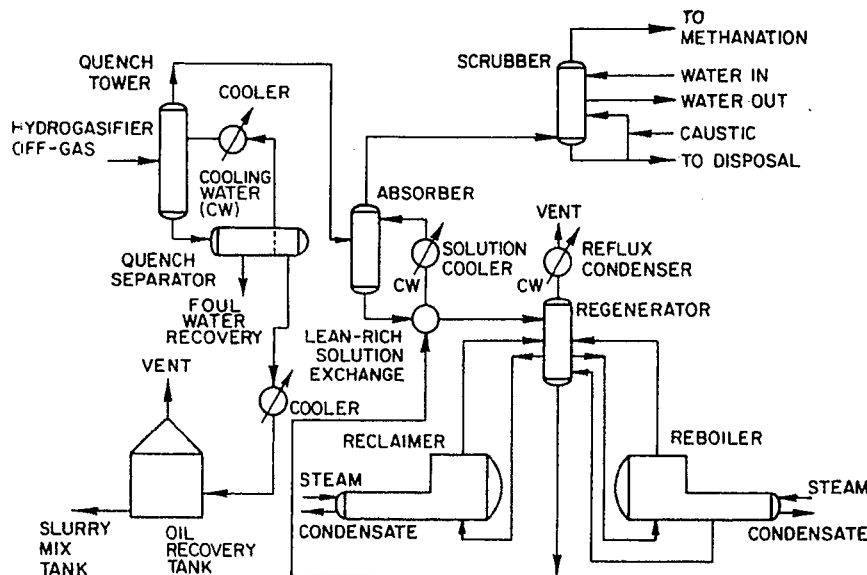


Figure 3-4. HYDROGASIFICATION SECTION OF HYGAS PILOT PLANT AT START-UP

From the first-stage reactor bed, solids flow downward, assisted by gravity, into the second-stage reactor where they are reacted with a mixture of hydrogen and steam, directly producing an additional one-third of the methane produced in the process, mostly by reaction 2. Additional hydrogen is generated in this fluidized-bed stage by reaction 3. Char from the second-stage reactor enters the fluidized-bed heat-exchange char cooler, where it is cooled by the incoming steam-hydrogen mixture. High-pressure steam is then used to convey the char to the quench tank. Water injected into the quench tank cools the char and forms a slurry. The water-slurry is reduced to atmospheric pressure through a valve, and the slurry passes to a vacuum filter, or alternatively to a char-settling pond.

### 3.3.4 Purification Section

The purification section of the HYGAS pilot plant is shown in Figure 3-5. Here, the hydrogasifier effluent gases are cooled by direct contact with water to remove water, light oils, and fines. The light oils and fines are then separated from the circulating quench water. Carbon dioxide, hydrogen sulfide, some COS, and CS<sub>2</sub> are removed from



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Figure 3-5. HYGAS PILOT PLANT PURIFICATION AT START-UP

the gas using a diglycolamine scrubbing system. The process gas leaving the absorber is scrubbed with dilute caustic, followed by a water wash to remove traces of caustic from the gas stream. The gas from this column is fed to the methanation section.

### 3.3.5. Methanation

Major elements of the methanation section are sketched in Figure 3-6. Gas from the purification section is blended, when necessary, with hydrogen to the degree required in order to provide the approximately 3:1 hydrogen-to-carbon monoxide ratio needed in methanation. This technique is used to replace a shift reactor which would achieve the 3:1 ratio through reaction 5. A portion of this gas is then further blended with recycle gas to limit the concentration of carbon monoxide. The blended gas is heated by exchange with effluent gas and fed into the first-stage methanator where reaction 6 occurs. A heater is provided for start-up.

The balance of the feed gas is also mixed with recycle gas to limit the concentrations of carbon oxides. These gases are then blended under temperature control with the hot effluent gas from the first stage to provide preheated feed gas for the second-stage methanator. Both fixed-bed methanation reactors contain a nickel catalyst. Reactor effluent is cooled to remove water from the product gas, and to supply cool gas to the recycle compressor.



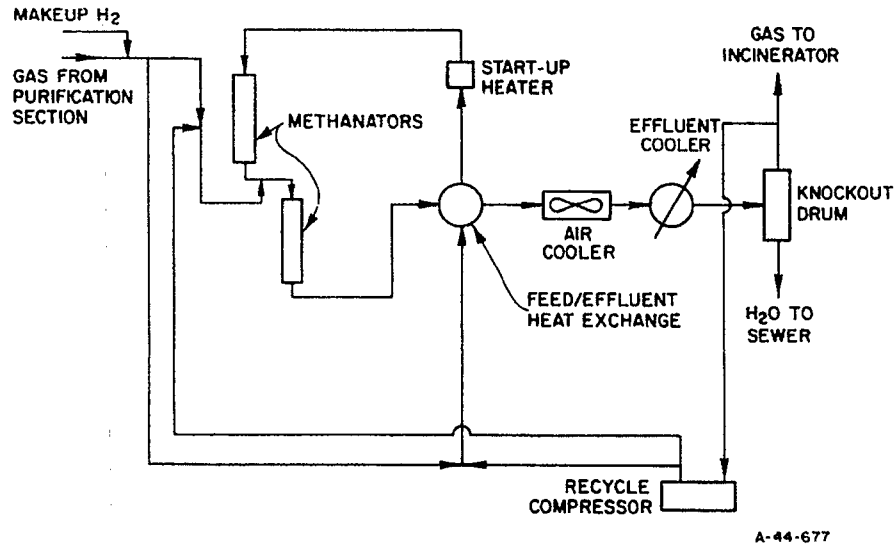


Figure 3-6. METHANATION SECTION OF HYGAS PILOT PLANT AT START-UP

### 3.4. Design Elaboration

This section elaborates on design philosophy, and discusses general practices and safety aspects of the HYGAS pilot plant. Included are brief explanations of 1) the function of each major piece of equipment or service, 2) selection criteria, 3) safety procedures where warranted, and 4) brief discussion of alternatives (if any).

The design specified by IGT and Bechtel was subject to modifications during detailed engineering by IGT and Procon because many preliminary engineering design areas were of necessity conceptual. Available equipment and fabrication techniques necessitated redesign or modification of certain concepts and equipment. These changes are described in later portions of this discussion.

The major sections of the detailed preliminary design that are discussed here are —

- Coal preparation, which includes coal storage, particle size, size reduction, and drying.
- Coal pretreatment, which includes the pretreater reactor, quench, and pretreated coal handling sections.
- Hydrogasification, which includes the coal slurry feed system, hydrogasification reaction section, hydrogen compressor, steam and hydrogen superheaters, and the spent char discharge section.

- Purification, which includes raw product gas quench, acid gas removal, caustic and water wash treatment sections.
- Methanation, which includes the methanation reactor, gas recycle, and product gas coolers.

### 3.4.1. Coal Pretreatment Section

#### 3.4.1.1. Basis for Design

The coal pretreatment section consists of a pretreater reactor with support facilities to feed the coal into the reactor, provide air for fluidization and oxidation, cool and condense the off-gas, and cool and transport the char to storage.

This section has been designed to supply 3 tons/hr of pretreated bituminous coal to the hydrogasification section. A process flow diagram is presented in Figure 3-7, together with the material balance and stream physical properties in Table 3-1.

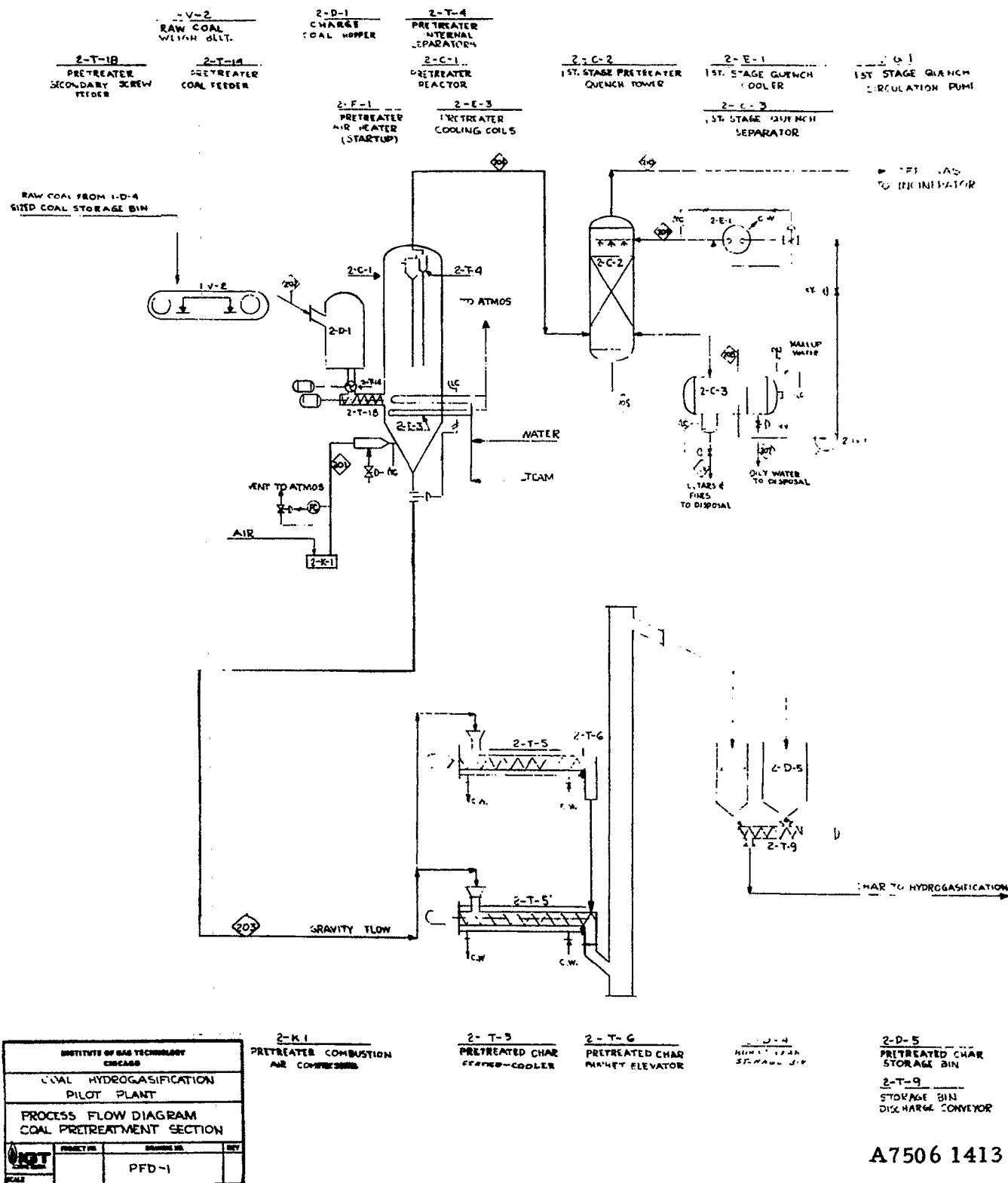
HYGAS-associated laboratory and bench-scale work had determined that some raw, bituminous coals tend to agglomerate during heating if they are charged directly into a fluidized-bed hydrogasifier reactor. Where not needed, however, as with lignite, pretreatment is undesirable because approximately 10 percent of the coal charged to the pretreater is lost as pretreater off-gas, oils, and entrained fines.

#### 3.4.1.2. Pretreater Reactor

The pretreater is a fluidized-bed type reaction vessel whose major dimensions are 98-inch ID by 16 feet 8 inches seam-to-seam. Air is compressed to provide the fluidizing medium, and this air is also used as an in-process oxidant. Raw crushed and sized coal is weighed and charged into a small surge hopper. The coal then enters the reactor through a star-and-screw feeder that is capable of feeding against a 10-psig back pressure. The off-gas from the pretreater enters an internal cyclone where entrained fines are returned to the bed.

The pretreater bed operates at about 800°F and from 1.0 to 10.0 psig. Internal water-cooled coils remove excess heat from the fluidized bed. Cooling water is vaporized under controlled temperature, and the steam exhausts to the atmosphere. The direct injection of water or steam for temperature control is impractical because it would change the superficial fluidizing velocity and increase the load on the quench system.

The pretreated coal is discharged through a fluidized standpipe and a slide valve. Air is distributed through a metallic deck with uniformly-spaced holes, achieving good fluidization. The holes, about 0.25 inch in diameter, are designed to provide about 1-psi drop across the deck at maximum flow conditions. The deck is curved to provide strength, yet permit expansion. A drain valve provided in the plenum below the deck permits the periodic removal of solids that have "wept" through the holes in the deck.



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Figure 3-7. PRELIMINARY DESIGN PFD FOR COAL PRETREATMENT SECTION OF HYGAS PILOT PLANT

Table 3-1, Part I. PRETREATER DESIGN CASE MATERIAL  
BALANCE AND STREAM PHYSICAL PROPERTIES  
(Bechtel Preliminary Design Data)

Stream No. Revision No.	201	202	203	204	205	206	207	208
Stream Description	Air to Pretreater	Coal to Pretreater	Char from Preheater	Pretreater Off-Gas	1st Stage Tower Bottoms	1st Stage Tar, Oils & Fines to Dispose.	1st Stage Oily Water To Disposal	Make Up Water to 1st Stage Separator
Temperature, °F	170	60	800	800	175	175	175	100
Pressure, psig	10	--	--	0.8	0.8	0.8	0.8	0.8
Components								
N <sub>2</sub>	--	--	--	129.2	--	--	NORMALLY NO FLOW	--
CO <sub>2</sub>	--	--	--	10.5	--	--	--	--
CO	--	--	--	12.5	--	--	--	--
H <sub>2</sub> O	--	--	--	35.0	19494 lb/hr	--	--	18
SO <sub>2</sub>	--	--	--	1.8	--	--	--	--
CH <sub>4</sub>	--	--	--	0.3	--	--	--	--
C <sub>2</sub> H <sub>6</sub>	--	--	--	0.3	--	--	--	--
C <sub>3</sub> H <sub>8</sub>	--	--	--	1.4	--	--	--	--
Char	--	6667 lb/hr	5000 lb/hr	--	--	53 lb/hr	--	--
Air	176	--	--	53 lb/hr	53 lb/hr	53 lb/hr	--	--
Tar & Oil	--	--	--	135 lb/hr	135 lb/hr	135 lb/hr	--	--
Fines	--	--	--	--	--	--	--	--
Total moles/hr	176	--	--	202.0 (1)	19680	186	--	18
Total lbs/hr	5104	6667	6000	5748	--	--	--	324
Molecular Weight	29.00	--	--	27.55 (1)	--	--	--	18.02
Vapor	0.106	--	--	0.32 (1)	--	--	--	--
lbs/ft <sup>3</sup> at Oper. Cond.	803	--	--	2897 (1)	--	--	--	--
acfm at Oper. Cond.	--	--	--	--	--	DESIGN 2.0	DESIGN 2.0	--
Liquid	--	--	--	--	40.6 (2)	--	--	0.65
gpm at Oper. Cond.	--	--	--	--	.35 (2)	--	--	0.68
Viscosity, cp at Oper. Cond.	--	--	--	--	--	--	--	--
Critical Properties								
PC - Pseudo Critical Pressure	--	--	--	1001 (1)	--	--	--	--
TC - Pseudo Critical Temp.	--	--	--	416 (1)	--	--	--	--
Z, Compressibility	--	--	--	1.0 (1)	--	--	--	--

Note: (1) Calculated on an oil free basis.  
(2) Oily water solution. Physical properties based on water.

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Table 3-1, Part 2. PRETREATER DESIGN CASE MATERIAL BALANCE AND STREAM PHYSICAL PROPERTIES

Stream Number Revision No.	209	210
Stream Description	1st Stage Tower Quench Water	1st Stage Tower Off-Gas
Temperature, °F	140	150
Pressure, psig	C.7	0.7
Components		
N <sub>2</sub>	--	139.2
CO <sub>2</sub>	--	10.5
CC	--	13.5
H <sub>2</sub> O	132.8 lb/hr	53.0
SiO <sub>2</sub>	--	1.8
CH <sub>4</sub>	--	0.3
C <sub>2</sub> H <sub>6</sub>	--	0.3
C <sub>3</sub> H <sub>8</sub>	--	1.4
Par <sup>n</sup>	--	--
Oil	--	--
Fines	--	--
Total Moles/Hour	--	220.0
Total Lbs/Hour	195.8	386.6
Molecular Weight	--	26.75
Super	--	0.064
Lbs/Ft <sup>3</sup> at Oper. Cond.	--	1523
Aer. at Oper. Cond.	--	--
Liquid	40.4(2)	--
CP at Oper. Cond.	0.47(2)	--
Viscosity, CP at Oper. Cond.	--	1.81
Critical Properties	--	477
PC - Pseudo Critical Press.	--	0.99
TC - Pseudo Critical Temp.	--	--
Z - Compressibility	--	--

Note: (2) 20% water solution. Physical properties based on water.

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#### 3.4.1.3. Pretreater Internal Solids Separators (2-T-4)

Internal cyclones, rather than external cyclones, are used in the design to remove the bulk of fines from the off-gas. Heavy oils and tars might condense and solidify in an external separator. While a two-stage cyclone is shown on the PFD (Figure 3-7), a single-stage cyclone might prove acceptable.

#### 3.4.1.4. Pretreater Cooling Coils (2-E-3)

The reaction of coal with air during pretreatment is exothermic; part of the energy released heats the incoming coal and air to reaction temperature, and the balance is removed in the cooling coil. A design duty of 3 million Btu per hour satisfies requirements of the various coals to be treated over a range of pretreatment severities and operating temperatures.

#### 3.4.1.5. Pretreater Air Heater (2-F-1)

The air heater unit is intended to heat air by direct combustion with fuel gas. The unit is fired during start-up only when bituminous coal is pretreated. The exothermic heat of reaction provides sufficient heat to more than maintain the pretreatment reaction at more than steady-state. Design specifications for the unit actually installed are tabulated below:

Manufacturer: John Zink & Co.

Air Flow Rate: Minimum: 1200 SCF/min  
Maximum: 1500 SCF/min

Air Inlet Temperature: 230°F

Air Outlet Temperature: 1200°F

Heat Release: 1,750,000 Btu/hr

Working Pressure: Minimum: 10 psig  
Maximum: 20 psig

Fuel: Natural gas or SNG

#### 3.4.1.6. Pretreater Combustion Air Compressor (2-K-1)

This compressor was conceived to move 1500 actual cubic feet per minute (ACFM) of air at a discharge pressure of 20 psig. Normal process requirements would be about 1100 ACFM at an operating pressure of about 1 psig in the reactor. The pressure in the reactor could be as high as 10 psig, depending upon the actual pressure drop in the overhead system. The discharge pressure of 20 psig would accommodate about a 10 psig drop through the air heater. In Figure 3-7, air is shown venting to the atmosphere downstream of the compressor to control flow. Design specifications for the unit actually installed are tabulated below:

Ingersoll Rand Type "L" AXI Compressor

Inlet: 1590 CF/min

Inlet Pressure: 14.5 psia

Discharge Pressure: 30 psia

ABS CR: 2.069

Inlet Temperature: 60°F

Discharge Temperature: 245°F

#### 3.4.1.7. Pretreated Char Cooler (2-T-5)

Unit 2-T-5 was conceived to cool pretreated char from 800° down to 200°F in a closed vessel. Vapors from this cooling are mixed with the off-gas from the pretreater-reactor and handled with that train. Cooled char is discharged into the char storage hopper and later sent to the reactor. The unit actually installed is a direct water-spray quench chamber of 3 feet ID and is 15 feet 10 inches long.

#### 3.4.1.8. Pretreater Quench System

This quench system consists of a pretreater quench tower with separator, cooler, and circulation pump. The off-gas is cooled by counter-current contact with water in the vertical packed tower. Operating experience on similar type units indicates — when the off-gas is cooled from 800°F to a minimum of about 150°F — practically all of the heavy oils and tars are condensed, and that most of the oil can be removed in the separator. Cooling of the heavy oils and tars to below 150°F tends to induce high viscosity, greatly increasing the risk of plugging the system.

The separator has an internal overflow weir; the heavy oil layer is removed through a boot on the bottom of the separator, and oils lighter than water flow over the weir for removal. This system constantly requires water make-up. The incoming gas vaporizes some of the quench water so that gas leaving the top of the tower is saturated. In addition, the overhead stream contains entrained water.

In order to maintain temperature in the tower, the incoming quench water is under temperature control with a bypass around the cooler.

Water is used as the quench medium rather than oil. The use of water permits more accurate qualitative and quantitative analyses of the tars, oils, and other components in the pretreater overhead effluent.

#### 3.4.1.9. Pretreater Quench Tower (2-C-2)

The vessel is 3 feet 6 inches ID by 15 feet 10 inches long with a 5-foot-long section with 2-inch metal pall rings. In actual practice the packing material in the tower plugged and was replaced by a venturi scrubber and side-to-side baffles.

### 3.4.1.10. Quench Separators (2-C-3)

The quench separator is of 3 feet 0 inches ID by 10 feet long, a horizontal vessel with a bottom draw-off boot. The elevation of the tower and separator are fixed to give a 12-inch liquid holdup in the bottom of the tower, to protect the bottom of the tower in case of a total circulating-quench-water failure.

In preparation of material balance tables, it is assumed that all of the oil, tars, and fines, etc., leave via the boot on 2-C-3. For design purposes, a design rate equal to the flow volume shown is assumed for the overflow.

The pump suction nozzle is raised 18 inches into the vessel in order to obtain oil-free quench water.

### 3.4.1.11 Quench Cooler (2-E-1)

The tube side of the exchanger is designed to permit quick cleaning because the quench water will contain tars, fines, and oils. Design specifications of the unit installed are tabulated below:

	<u>Shell Side</u>	<u>Tube Side</u>
Fluid Circulated	Cooling H <sub>2</sub> O	Quench H <sub>2</sub> O
Total Fluid Entering	17,800 lb/hr	24,240 lb/hr
Gravity-Liquid (in/out)	0.995/0.989	0.971/0.977
Viscosity CP (in/out)	0.90/0.56	0.36/0.47
Temperature In	75 <sup>0</sup> F	175 <sup>0</sup> F
Temperature Out	120 <sup>0</sup> F	140 <sup>0</sup> F
Operating Pressure (Inlet)	90 psig	25 psig
Pressure Drop (Allowable)	10 psig	10 psig
Fouling Factor	0.006	0.006
Heat Exchanged	800,000 Btu/hr	

Manufacturer: Basco Multi-Tube heat exchanger.

### 3.4.1.12. Quench Circulation Pump (2-G-1)

Vendors were advised that the oily water solution in the pump might contain heavy fines and viscous tars. Design specifications of the unit installed are tabulated below:

Manufacturer: Ingersoll-Rand

Type: In-line centrifugal

Horsepower: 5

Flow Rate, gpm: 40

Temperature: 173<sup>0</sup>F

Inlet Pressure: 4.5 psig

Outlet Pressure: 50.5 psig



### 3.4.2. Hydrogasification Section

#### 3.4.2.1. Basis for Design

The gasification section utilizes four separate high-pressure fluidized-bed contacting steps to --

1. Dry the slurry.
2. Afford an initial reaction stage between coal and partly-reacted gas.
3. Provide a final reaction stage between the partly reacted coal (char) and preheated treating gas.
4. Provide heat-exchange contact between partially gasified char and feed gas to conserve heat and cool the product solids.

This section consists of a two-stage continuous-flow hydrogasification reaction section with char cooling, an aromatic-oil-based slurry system to elevate the pretreated char to operating pressure, a slurry drier to remove the aromatic carrier and dry feed for processing, and a water slurry system to return the spent char to atmospheric pressure. The preliminary design process flow diagram (PFD) for this section is presented in Figure 3-8, together with the material balance and stream physical properties in Table 3-2.

The hydrogasification steps operate at a nominal pressure of about 1150 psig; the basic equipment design pressure is 1650 psig. Temperatures through the hydrogasification stages range from 1400<sup>o</sup> to 1800<sup>o</sup>F. Design criteria presumed a nominal bulk density of pretreated coal of about 25 pounds per cubic foot, and that of the spent char of about 15 pounds per cubic foot.

The high temperature and pressures of this section require special consideration for insulation. For a pilot unit, heat loss must be held to a minimum. Bechtel recommended that the refractory insulation material be selected making use of the combined experience of vendors as well as of IGT.

#### 3.4.2.2. Hydrogasifier Process Flow

In the preliminary hydrogasifier PFD, a coal-benzene slurry is shown being pumped to high pressure and introduced into a fluidized slurry drier. Any of the several aromatic oils are suitable for this, but IGT chose to use toluene, a process by-product. In the drier, at the top of the main reactor tower, hot gases from the first stage hydrogasifier evaporate the aromatic oil. A perforated metallic deck is used as a gas distributor. The dried coal flows into a small mixing chamber where it contacts hot gases rising from the second-stage hydrogasifier. The gas

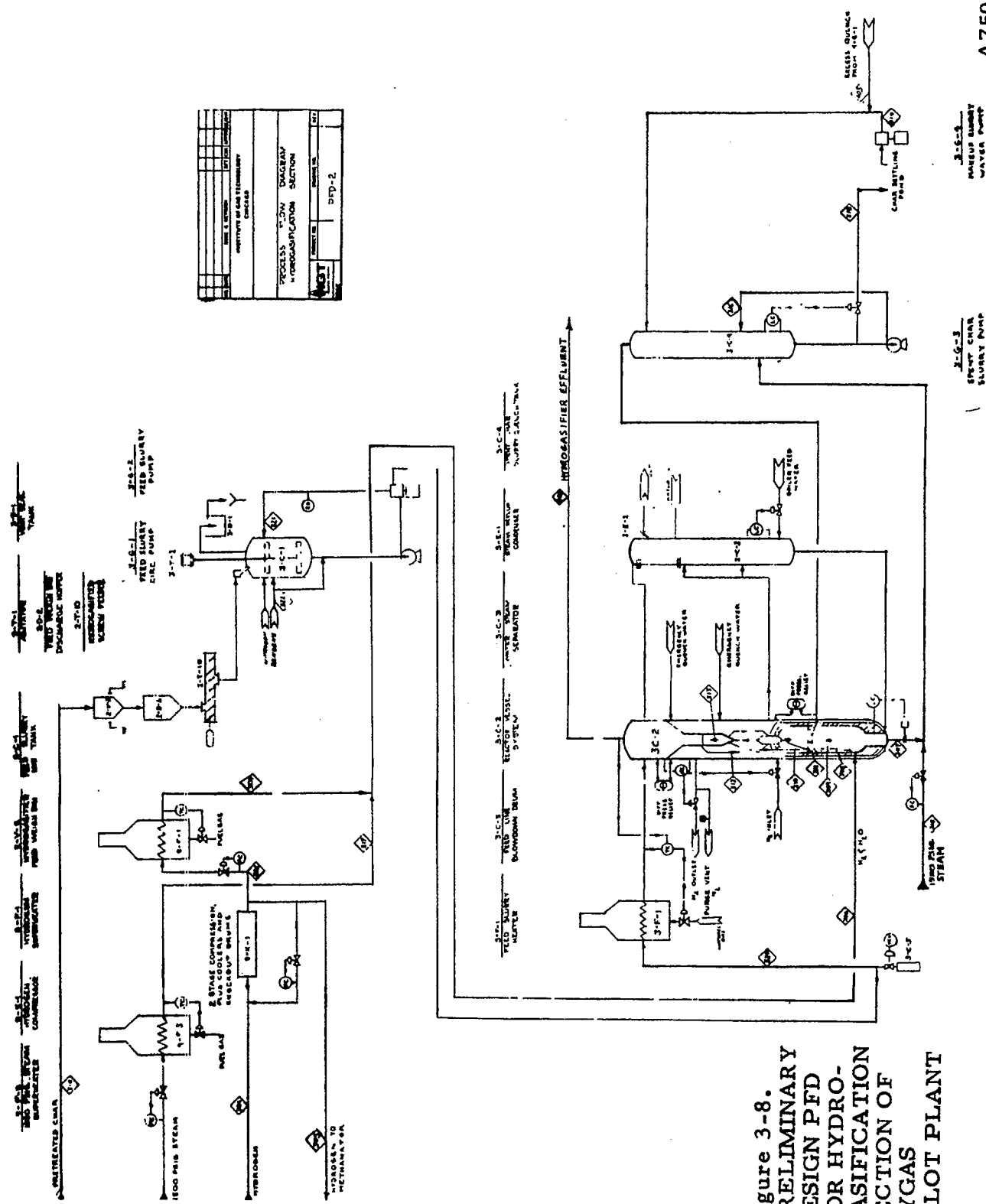


Figure 3-8.  
PRELIMINARY  
DESIGN PFD  
FOR HYDRO-  
GASIFICATION  
SECTION OF  
HYGAS  
PILOT PLANT

REVISIONS	
NO.	DESCRIPTION
1	INITIAL DESIGN
2	DESIGN CHANGES
3	DESIGN CHANGES
4	DESIGN CHANGES
5	DESIGN CHANGES
6	DESIGN CHANGES
7	DESIGN CHANGES
8	DESIGN CHANGES
9	DESIGN CHANGES
10	DESIGN CHANGES

PROJECT NO.	57D-2
PROCESS	LOW GRADE HYDROGASIFICATION SECTION
DATE	
BY	
CHECKED BY	
APPROVED BY	

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Table 3-2, Part 1. STEAM AND HYDROGEN SYSTEM DESIGN  
CASE MATERIAL BALANCE AND STREAM PHYSICAL PROPERTIES  
(Bechtel Preliminary Design Data)

Stream Number Revision No.	301	302	303	304	315	317
Stream Description	Hydrogen Compressor To	Hydrogen To Superheater	Hydrogen To Methanator	Hydrogen From Superheater	Steam To Char Lift	Superheated Steam To Hydrogasification
Temperature, °F	100	100	100	1200	596	1200
Pressure, psig	100	1500	1500	1500	1500	1500
Components	220.3	168.2	52.1	168.2	166.6	222.0
H <sub>2</sub>	--	--	--	--	--	--
H <sub>2</sub> O (18.02)	--	--	--	--	--	--
Total Moles/Hour	220.3	168.2	52.1	168.2	166.6	222.0
Total Lbs/Hour	444	337	105	337	330	440
Molecular Weight	2.016	2.016	2.016	2.016	18.016	18.016
Temp at Oper. Cond.	5.07	0.50	0.05	0.17	3.40	1.61
Temp at Oper. Cond.	230.0	11.3	3.3	23.2	14.7	41.4
Temp at Oper. Cond.	--	--	--	--	--	--
Viscosity, CP at Oper. Cond.	--	--	--	--	--	--
Critical Properties	188	188	188	168	3206	3206
PC - Pseudo Critical Press.	60	60	60	1165	1165	1165
TC - Pseudo Critical Temp.	1.02	1.01	1.01	1.0	0.71	0.95
Compressibility						

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Table 3-2, Part 2.a. HYDROGASIFIER CONTROLLING CASE  
FOR BITUMINOUS COAL 50:50 WATER-TO-HYDROGEN:  
MATERIAL BALANCE AND STREAM PHYSICAL PROPERTIES

Stream Number: Revision No.	306	307	308	309	310	311	312	313
Stream Description	Steam & Hydrogen To Char Cooler	Char From Char Cooler	Char From 2nd Stage Reactor	Gas To 2nd Stage Reactor	Char To 2nd Stage Reactor	Gas From 2nd Stage Reactor	Char 1st Stage Reactor	Gas From Product Separator
Temperature, or Pressure, psia	1200 1160	134C --	170C --	134C 1159	1500 --	170C 1156	600 --	1500 1150
Components	CO {28.01} {44.01}	--	--	--	--	12	--	43.8
H <sub>2</sub>	{2.02}	--	--	173.8	--	10.5	--	21.6
CH <sub>4</sub>	{18.02}	--	--	200.0	--	109.2	--	145.9
C <sub>2</sub> H <sub>6</sub>	{16.04}	--	--	--	--	166.2	--	143.5
C <sub>3</sub> H <sub>8</sub>	{30.07}	--	--	--	--	72.0	--	79.8
C <sub>4</sub> H <sub>10</sub>	{44.09}	--	--	--	--	--	--	1.9
C <sub>5</sub> H <sub>12</sub>	{78.11}	--	--	--	--	--	--	0.7
C <sub>6</sub> H <sub>14</sub>	{54.09}	--	--	--	--	2.6	--	1.2
H <sub>2</sub> S	{28.01}	--	--	--	--	1.0	--	4.8
N <sub>2</sub>	--	3084 lb/hr	3084 lb/hr	--	4421 lb/hr	--	6000 lb/hr	2.0
Char	--	--	--	--	--	--	--	138 lb/hr
Oil	--	--	--	--	--	--	--	--
Total Moles/Hour	373.8	3084	3084	373.8	4421	373.8	6000	445.2(1)
Total Lbs/Hour	3953	3084	3084	3953	4421	5297	6000	6876
Molecular Weight	10.58	--	--	10.58	--	14.17	--	15.13(1)
Vapor	0.70	--	--	0.64	--	0.71	--	0.83(1)
ACFM at Oper. Cond.	94.1	--	--	102.9	--	124.3	--	135.3(1)
1st St. Oper. Cond.	--	--	--	--	--	--	--	--
Viscosity, CP at Oper. Cond.	--	--	--	--	--	--	--	--
Critical Properties	1803	--	--	1803	--	1668	--	1340
TC - Pseudo Critical Press.	651	--	--	651	00	631	--	521
TC - Pseudo Critical Temp.	1.0	--	--	1.0	00	1.01	--	1.01
Z - Compressibility	--	--	--	--	--	--	--	--

Note: (1) Calculated on an oil free basis.

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Table 3-2, Part 2.b. HYDROGASIFIER CONTROLLING CASE FOR  
 BITUMINOUS COAL 50:50 WATER-TO-HYDROGEN:  
 MATERIAL BALANCE AND STREAM PHYSICAL PROPERTIES

Stream Number Revision No.	314	316	318	319	320	321	322
Coal Feed To Slurry Tank	200						
Char Slurry Circulated	458 1160	458 1160	458 1160	100 1200	100 1200	100 ---	100 ---
Char Slurry Discharge			9284 lb/hr	4934 lb/hr			
Char Quench Meter							
Feed To Slurry Dryer					12000 lb/hr	30000 lb/hr	12000 lb/hr
Slurry Recirculated						15000 lb/hr	
Benzene To Slurry Tank							
Temperature, °F							
Pressure, psig							
Components							
CO	(28.01)						
CO <sub>2</sub>	(44.01)						
H <sub>2</sub>	(2.02)						
H <sub>2</sub> O	(18.02)	18100 lb/hr					
CH <sub>4</sub>	(16.04)						
C <sub>2</sub> H <sub>6</sub>	(30.07)						
C <sub>3</sub> H <sub>8</sub>	(44.09)						
C <sub>4</sub> H <sub>10</sub>	(58.12)						
C <sub>5</sub> H <sub>12</sub>	(72.15)						
C <sub>6</sub> H <sub>14</sub>	(86.18)						
N <sub>2</sub>	(28.01)	6000 lb/hr	3084 lb/hr		6000 lb/hr		
O <sub>2</sub>							
Oil							
Total Moles/Hour	6000	24100	12368	4934	18000	45000	12000
Total Lbs/Hour				18			78
Molecular Weight							
Vapor							
Lb/ft <sup>3</sup> at Oper. Cond.							
ACTW at Oper. Cond.							
Liquid							
CPK at Oper. Cond.							
Viscosity, CP at Oper. Cond.				10			27
Critical Properties				0.7			0.5
Pc - Pseudo Critical Press.		60 (slurry)	32 (slurry)		40 (slurry)	60 (slurry)	
Tc - Pseudo Critical Temp.							
Z - Compressibility							

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Table 3-2, Part 3.a. HYDROGASIFIER CONTROLLING CASE  
 FOR BITUMINOUS COAL 30:70 WATER-TO-HYDROGEN:  
 MATERIAL BALANCE AND STREAM PHYSICAL PROPERTIES

Stream Number Revision No.	306	307	308	309	310	311	312	313
Stream Description	Steam & Hydrogen To Char Cooler	Char From Char Cooler	Char From 2nd Stage Reactor	Gas To 2nd Stage Reactor	Char To 2nd Stage Reactor	Gas From 2nd Stage Reactor	Char To 1st Stage Reactor	Gas From Product Separator
Temperature, °F	1200	1420	1800	1420	1500	1600	600	1500
Pressure, PSIG	1160	--	--	1159	--	1156	--	1150
Components	(28.01)	--	--	--	--	13.8	--	53.7
CO <sub>2</sub>	(44.01)	--	--	--	--	4.2	--	10.6
H <sub>2</sub> O	(2.02)	--	--	168.2	--	134.5	--	119.6
CH <sub>4</sub>	(16.04)	--	--	91.0	--	53.0	--	54.6
C <sub>2</sub> H <sub>6</sub>	(30.07)	--	--	--	--	0.1	--	77.3
C <sub>3</sub> H <sub>8</sub>	(44.09)	--	--	--	--	--	--	0.3
C <sub>4</sub> H <sub>10</sub>	(78.11)	--	--	--	--	--	--	0.1
H <sub>2</sub> S	(34.08)	--	--	--	--	2.4	--	4.9
N <sub>2</sub>	(28.01)	--	--	--	--	1.1	--	2.2
Char	--	3195 lb/hr	3195 lb/hr	--	4354 lb/hr	--	6000	--
Oil	--	--	--	--	--	--	--	114 lb/hr
Total Moles/Hour	259.2	3195	3195	259.2	4354	258.0	6000	323.4(1)
Total lbs/Hour	1978	3195	3195	1978	4354	5148	6000	4802
Molecular Weight	7.63	--	--	7.63	--	12.20	--	14.49(1)
Vapor	0.49	--	--	0.45	--	0.60	--	0.79(1)
ACFM at Oper. Cond.	87.3	--	--	73.5	--	87.4	--	98.9(1)
Liquid	--	--	--	--	--	--	--	--
GM at Oper. Cond.	--	--	--	--	--	--	--	--
Viscosity, CP at Oper. Cond.	--	--	--	--	--	--	--	--
Critical Properties	1248	--	--	1248	--	1158	--	915(1)
Pc - Pseudo Critical Press.	448	--	--	448	--	449	--	372(1)
Tc - Pseudo Critical Temp.	1.02	--	--	1.02	--	1.02	--	1.02(1)
Z - Compressibility	--	--	--	--	--	--	--	--

Note: (1) Calculated on an oil free basis.

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Table 3-2, Part 3.b. HYDROGASIFIER CONTROLLING CASE  
 FOR BITUMINOUS COAL 30:70 WATER-TO-HYDROGEN:  
 MATERIAL BALANCE AND STREAM PHYSICAL PROPERTIES

Stream Number Revision No.	314	316	318	319	320	321	322
Stream Description	Coal Feed To Slurry Tank	Char Slurry Circulated	Char Slurry Discharge	Char Quench Water	Feed To Slurry Dryer	Slurry Recirculated	Benzene To Slurry Tank
Temperature, °F	200	513	513	100	100	100	100
Pressure, psig	--	1160	1160	1200	1200	--	--
Components							
CO	--	--	--	--	--	--	--
CO <sub>2</sub>	--	--	--	--	--	--	--
H <sub>2</sub>	--	--	--	--	--	--	--
H <sub>2</sub> O	--	18100 lb/hr	7860 lb/hr	5112 lb/hr	--	--	--
CH <sub>4</sub>	--	--	--	--	--	--	--
C <sub>2</sub> H <sub>6</sub>	--	--	--	--	--	--	--
C <sub>3</sub> H <sub>8</sub>	--	--	--	--	--	--	--
C <sub>4</sub> H <sub>10</sub>	--	--	--	--	--	--	--
C <sub>5</sub> H <sub>12</sub>	--	--	--	--	--	--	--
C <sub>6</sub> H <sub>14</sub>	--	--	--	--	--	--	--
C <sub>7</sub> H <sub>16</sub>	--	--	--	--	--	--	--
C <sub>8</sub> H <sub>18</sub>	--	--	--	--	--	--	--
C <sub>9</sub> H <sub>20</sub>	--	--	--	--	--	--	--
C <sub>10</sub> H <sub>22</sub>	--	--	--	--	--	--	--
N <sub>2</sub>	6000 lb/hr	6000 lb/hr	3195 lb/hr	--	6000 lb/hr	15000 lb/hr	--
Char	--	--	--	--	--	--	--
Oil	--	--	--	--	--	--	--
Total Moles/Hour	6000	24100	11055	5112	18000	45000	12000
Total Lb/Hour	--	--	--	5112	--	--	78
Molecular Weight	--	--	--	--	--	--	--
Vapor	--	--	--	--	--	--	--
Lb/fts at Oper. Cond.	--	--	--	--	--	--	--
Lb/fts at Oper. Cond.	--	--	--	--	--	--	--
Liquid	--	60(slushy)	30(slushy)	11	40(slushy)	60(slushy)	27
Vis at Oper. Cond.	--	--	--	0.7	--	--	0.5
Vis at Oper. Cond.	--	--	--	--	--	--	--
Critical Properties	--	--	--	--	--	--	--
Pc - Pseudo Critical Press.	--	--	--	--	--	--	--
Tc - Pseudo Critical Temp.	--	--	--	--	--	--	--
Z - Compressibility	--	--	--	--	--	--	--

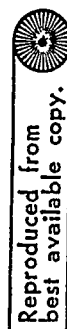
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Table 3-2, Part 4. a. HYDROGASIFIER CONTROLLING CASE FOR LIGNITE: MATERIAL BALANCE AND STREAM PHYSICAL PROPERTIES

Stream No. Revision No.	306	307	309	310	311	312	313
Stream Description	Heat Exchanger Cooler	Char Feed Reactor	Gas From 2nd Stage Reactor	Char To 2nd Stage Reactor	Gas From 2nd Stage Reactor	Char To 1st Stage Reactor	Gas From Product Separator
Temperature, °F	1200	1400(3)	1700	1500	1700	600	1500
Pressure, psig	1160	--	--	--	1150	--	1150
Components							
CO	(28.01)	--	--	--	15.0	--	25.2
CO <sub>2</sub>	(44.01)	--	--	--	18.2	--	35.4
H <sub>2</sub>	(2.02)	--	--	--	90.6	--	92.6
H <sub>2</sub> O	(18.02)	--	--	--	94.7	--	102.5
CH <sub>4</sub>	(16.04)	--	--	--	66.0	--	76.4
C <sub>2</sub> H <sub>6</sub>	(30.07)	--	--	--	--	--	2.7
C <sub>3</sub> H <sub>8</sub>	(44.09)	--	--	--	--	--	1.2
C <sub>4</sub> H <sub>10</sub>	(78.11)	--	--	--	0.3	--	0.6
C <sub>5</sub> H <sub>12</sub>	(94.12)	--	--	--	0.3	--	0.6
N <sub>2</sub>	(28.01)	--	--	--	--	--	1.7
Char	--	2400 lb/hr	2400 lb/hr	3666 lb/hr	--	6000 lb/hr	325 lb/hr
Oil	--	--	--	--	--	--	--
Total Moles/Hour	253.8	2400	2400	3666	277.8	6000	344.4(1)
Total Lbs/Hour	2590	2400	2590	3666	3856	--	6198
Molecular Weight	10.20	--	10.20	--	13.88	--	17.06(1)
Vapor	0.67	--	0.51	--	0.70	--	0.99(1)
ACFM at Oper. Cond.	64.4	--	70.8	--	91.8	--	104.2(1)
Liquid	--	--	--	--	--	--	--
GFW at Oper. Cond.	--	--	--	--	--	--	--
Viscosity CP at Oper. Cond.	--	--	--	--	--	--	--
Critical Properties							
Pc - Pseudo Critical Press.	1733	--	1733	--	1985	--	1347(1)
Tc - Pseudo Critical Temp.	254	--	254	--	523	--	527(1)
Z - Compressibility	1.01	--	1.01	--	1.01	--	1.01(1)

Note: {1} Calculated on an oil free basis.  
{3} Temperature Assumed.

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Table 3-2, Part 4. b. HYDROGASIFIER CONTROLLING CASE FOR LIGNITE: MATERIAL BALANCE AND STREAM PHYSICAL PROPERTIES

Stream Number Revision No.	314	316	318	319	320	321	322
Coal Feed To Slurry Tank	200						
Char Slurry Circulated		505 1160					
Char Slurry Discharge			505 1160				
Char Quench Water				100 1200			
Feed To Slurry Dryer					100 1200		
Slurry Recirculated						100	
Benzene To Slurry Tank							100
Temperature, °F							
Pressure, psig							
Components							
CO	(28.01)						
CO <sub>2</sub>	(44.01)						
H <sub>2</sub>	(2.02)						
H <sub>2</sub> O	(18.02)						
CH <sub>4</sub>	(16.04)						
C <sub>2</sub> H <sub>6</sub>	(30.07)						
C <sub>3</sub> H <sub>8</sub>	(44.09)						
C <sub>4</sub> H <sub>10</sub>	(58.12)						
H <sub>2</sub> S	(34.08)						
N <sub>2</sub>	(28.01)						
Char							
Oil							
Total Moles/Hour	6000	24100	9905	2840	18000	45000	12000
Total Lbs/Hour				18.02			78
Molecular Weight							
Vapor							
Lbs/Ft <sup>3</sup> at Oper. Cond.							
ACFM at Oper. Cond.							
Liquid							
GFW at Oper. Cond.							
Viscosity, CP at Oper. Cond.							
Critical Properties							
Pc - Pseudo Critical Press.							
Tc - Pseudo Critical Temp.							
Z - Compressibility							

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lifts the solids through a vertical transport section into a spouting-bed separator; off-gas from the separator enters the slurry drier above. A recycle stream of solids may be returned to the mixing chamber to help control temperature. The balance of the solids enters the second stage hydrogasifier. The principal reactions and direct methane production of the first-stage hydrogasifier occur during the vertical transport when solids and gases are in contact while freely rising; see the description of Stream 313 in Table 3-2 for an indication of off-gas content at this point in the process.

In the second-stage hydrogasifier, the partially gasified coal is reacted with a hydrogen and steam feed gas mixture during a solids holding time of several minutes at a fluidized-bed temperature of about 1800°F. The spent char is discharged to a heat recovery fluidized bed in the preliminary design reactor configuration. The gases from the second stage rise into the first-stage reactor.

Spent char from the 1800°F second-stage reactor enters the fluidized bed heat exchanger where it is cooled to about 1400°F. The heated gas flows into the second stage reactor. The char is moved from the heat-exchange bed via a short fluidized standpipe and throttling valve to enter a riser; here, it is lifted by a flow of high-pressure steam to the spent char slurry tank. Water is injected into the slurry tank in order to quench the char and condense the carrier steam. The spent char-slurry is then let down to atmospheric pressure to facilitate spent char removal.

The fluidized bed of the second-stage reactor is supported on a ceramic deck. The pressure drop available for gas distribution here is small to avoid excessive seal leg height. Too low a differential could cause unsatisfactory bed fluidization.

#### 3.4.2.3. Coal Feed

A lock-hopper system to introduce coal into the high-pressure reactor was deemed expensive and bulky and — in 1968 when the design effort began — valves that must operate hot and seal against high differential pressure with a solids system were not proved commercial items. For this reason, a coal slurry feed system was selected.

The size range for coal feed has been specified as 10 to 100 mesh U.S. sieve. Coal of this range can be mixed with a light aromatic oil to form a slurry which can be charged to the high-pressure reaction system by a slurry pump. The slurry liquid must be evaporated before the dried coal enters the actual reaction zone. This drying is done in a fluidized-bed using effluent gases from the first-stage reactor to provide the heat.

Feed coal is charged to the plant at a constant rate by means of a weigh-belt conveyor. Feed is dumped into a hopper, flows down a gravity chute, and enters a slurry feed mix tank which has about 1 hour surge capacity. The tank is operated at a few inches of water head; i.e., slightly above atmospheric pressure. Normally, coal is received at about 200°F,

the hot coal from the feed preparation section being cooled to that temperature level before flowing to the weight scale. The slurry oil is received from storage at about 100°F; the slurry mix is at about 115°F. A "trickle valve" on the coal inlet chute and a "seal" nitrogen combine to exclude air from the slurry mix tank, preventing the occurrence of aromatic-oil and air mixtures at this point.

An agitator in the tank and external circulation pumps serve to mix the slurry of coal and aromatic oil, and to maintain even consistency through the system. A centrifugal pump is arranged to circulate directly through the suction manifold of a mud-pump type reciprocating pump, thus avoiding pockets where solids can settle from the slurry. (A duplicate pumping system is provided for standby.) All the slurry lines are arranged so that there are no dead ends.

The plunger pump runs at constant-volume discharge per stroke. The discharge rate can be manually adjusted through a variable-speed drive. Makeup aromatic oil is pumped into the mix tank as required to maintain this slurry surge volume by means of a level control. As a result of this arrangement, coal feed is deposited in the slurry at the weight rate set by the weigh belt; the oil-to-coal ratio is determined by the plunger pump discharge rate.

The plunger pump discharges slurry at high pressure through a high-pressure steam heat exchanger\* and hence to the fluidized drying bed at the top of the reaction system. In the fluidized bed, hot gases from the first stage reactor are cooled from 1400° to 600°F. The oil is vaporized and leaves with the product gases. Dried feed coal at 600°F flows down to the first-stage reactor through a fluidized standpipe and flow control valve. The slurry exit temperature from the steam heat exchanger is controlled to maintain the desired temperature in the drier bed.

Aromatic oil and water in the overhead stream are condensed in a quench tower. The condensed mixture flows into a horizontal settling vessel. A weir extends above the oil-water interface level so that the oil phase overflows. The oil is removed under level control and flows through an oil cooler to atmospheric pressure storage; there, dissolved gases are flashed and vented to a flare system. Water is circulated through a cooler to the top of the quench tower. Excess water is removed under interface level control and routed to the spent char-water slurry mix system. Any excess oil from the storage tank is removed periodically to waste disposal.

A slurry composed of two parts by weight of oil and one part of coal can be readily pumped. By adjusting the slurry outlet temperature from the slurry heat exchanger to between ambient and 500°F, sufficient heat is available from the first stage hydrogasifier off-gases to maintain the slurry drier bed at 600°F.

---

\* This is a specially designed double pipe heat exchanger, not a direct-fired heater as shown in Figure 3-8.

Water is not a practical choice for the coal feed slurry in this HYGAS process system because its heat of vaporization is about five times that of the usual aromatic oil at the drying bed operating conditions, and heat balance could not be achieved with a slurry of pumpable consistency. Water is used for the emergency control of drying bed temperatures, and a quench line with an automatic temperature-control station was installed.

#### 3.4.2.4. Reaction System Vessel

The combination of high pressures and temperatures with hydrogen and steam in the reaction system vessels and piping calls for careful design. The experience and recommendations of the engineering contractor proved particularly important in these decisions.

Observations at the time the preliminary design was performed included -

##### 3.4.2.4.1. Refractory Considerations

Cold wall designs have internal refractory and insulation layers, and usually operate with vessel walls at 250<sup>0</sup> to 350<sup>0</sup>F. These temperatures are below the dew point of the process fluids in the system and would cause condensation of vapor on the inside of the shell. Refractory producers, in fact, were uncertain how "wet" operation would affect the refractory. In addition to steam and hydrogen, the process gases contain carbon oxides and sulfur compounds. There appeared to be no operating experience under comparable conditions to draw on that would permit selection or rejection of a cold-wall system.

Hot wall designs with the vessel shell metal in direct contact with the process fluids require alloys adequate for the operating temperatures. For this application with metal temperatures of 1400<sup>0</sup> to 1800<sup>0</sup>F and 1650 psig design pressure, especially considering the corrosive effect of the steam- and hydrogen-rich environment, as well as the erosive effect of solids under these conditions, wall thicknesses would be too great to be practical or economical without refractories.

In addition, a combination of both internal and external insulation appears to be unattractive. Metal temperature of the vessel could vary widely from the desired level, depending on the accuracy of heat flow calculations, weather conditions, the physical condition of the insulation, and on how hydrogen affects the conductivity of the internal insulation. These variables would dictate a costly system of metal temperature measurement. Even with such a measurement system, hot spots - once detected - could be corrected only by plant shutdown and repair. Hot spots not located, on the other hand, would constitute a hazard. The principal advantage promised by internal/external insulation in the preliminary design is avoidance of condensation at the inside surface of the metal shell.

#### 3.4.2.4.2 Reactor Vessel Design

A water-jacketed vessel with both internal and external insulation, operated with water at reaction system pressure, offers advantages for the ranges of 1400° to 1800°F, and 1000 to 1500 psig operating conditions. The jacket water absorbs heat transmitted through the internal insulation, and maintains both inner and outer metal walls of the vessel at substantially the same temperature. Steam generation occurs and the steam enters the reactor steam separator drum where the steam is condensed and the water returned to the bottom of the jacket. This drum is connected to the process zone of the vessel through a pressure-balance line. In this design, then, the jacket water temperature is the equilibrium temperature of steam, or about 550°F for 1000 psig operation, to 600°F for 1500 psig. These temperatures are above the dew points of the fluids in the reaction system, so that no condensation of vapor occurs in the internal insulation next to the metal vessel shell. These metal temperatures, too, are such that an inner jacket shell of carbon one-half molybdenum steel should provide reasonable protection against hydrogen attack. The outer strength shell can be carbon steel, because the two metals have similar coefficients of expansion and present no particular problems for fabrication as a double-shell assembly. This water-jacketed vessel contains the high-temperature second-stage hydrogasifier and the heat-exchange bed.

The first-stage reaction of the process includes a small mixing zone where 1800°F second-stage off-gas is mixed with 600°F coal feed solids. There is a coal feed standpipe, a lift-line from the mixing zone to an upper reaction and separation vessel, and a solids recycle standpipe from the separator to the mixing zone for temperature control. The feed line is a fluidized standpipe with a throttling valve, as is the solids recycle line. Lines, mixing zone, and separator are relatively small, but have high operating temperatures and pressures. A hot-box design concept was used for this system in order to provide flexibility in maintenance and modifications, as well as in cost savings.

Alloy piping and pressure equipment components, capable of withstanding both the anticipated 1400°F operating temperature, and possible transient temperatures to 1800°F during operating upsets, are enclosed in a pressure vessel adequate for the required operating pressure. The hot internal piping and parts are wrapped with insulation. The void volume between internals and the pressure enclosure vessel is filled with nitrogen to balance the pressure inside the process zones. With this design, the metal of the enclosing vessel is at relatively low temperature and presents no special design problems. The internal process components operate at quite high temperatures, but have very low operating pressure differentials.

#### 3.4.2.4.3 Reaction System Vessel Layout

A "stacked assembly" was considered a probable arrangement of the various high-pressure sections of the hydrogasification process from the

beginning of design. In this assembly, the main envelope is a carbon-steel shell of 5 feet 6 inches inside diameter and 120 feet 0 inch from tangent line to tangent line. The vessel is supported from grade by an integral skirt. The lower section accommodates the second-stage reactor and the product char cooling fluid bed, and utilizes a water jacket to control metal temperatures, as discussed earlier. Although internal changes have been incorporated since September 1972, the initial design is described here.

The upper section of the vessel incorporates the hot-box system. The upper section contains the slurry feed drying bed, the first-stage reactor and lift pipe, the recycle pipe, the gas-solids separator, and related piping. These components are of alloyed Incoloy 800 that will provide reasonable service life under the severe operating conditions described. The alloy parts are externally insulated and surrounded by a nitrogen atmosphere maintained in balance with the reaction system pressure. The pressure enclosure of the hot box is the main vessel shell with reasonable access for maintenance work on the alloy internals and insulation. Two manholes are located on the hot box shell to provide ventilation for maintenance work during shutdown. The top of the slurry drier vessel operates at reaction pressure and relatively lower temperature of about 600°F. The slurry drier vessel does not require internal insulation, but external insulation is required for upper and lower sections of the vessel.

The operating pressure of the reaction system is a little over 1000 psi and the design pressure is 1650 psi. The pressure in the nitrogen-filled shell, is held as close to those in the reaction zone as possible. This is accomplished by a control system in the case of the nitrogen atmosphere of the hot box, and by means of an equalizing line to the reaction system from the reaction steam separator drum, in the case of the water-jacketed vessel.

Only small pressure differentials exist during normal operation, but these differentials can become appreciable during operating upsets. Four percent of the 1500 psig design pressure — i.e., 60 psig in either direction — was suggested as a design value for allowable pressure differential between hot box and upper and lower vessel sections. The inner shell of the water jacketed lower shell must also withstand this differential as an external pressure. In connection with the alloy internals of the hot box, the 60 psig differential is only a transient condition that would result from an operations upset. The creep stress limits that apply for continuous loading at elevated temperatures are not applicable, and design was based on the appropriate elastic modulus.

The 60 psig differential noted above brings the design pressure inside the hot box to 1560 psig, corresponding to the requirement of 1500 psig in the actual reaction zones of the equipment.

Pressure relief protection is required between the different sections of the reaction system vessel. Two-way safety heads or "rupture disks", with rupture pressures of 40 to 60 psi, are used. Safety heads are incorporated in this rupture range that utilize combination metal and Teflon elements.

#### 3.4.2.5 Product Char Removal System

Char from the reaction system vessel is discharged through the bottom solids control valve, and is transferred by carrier steam flow into a char slurry quench tank. The tank is at system pressure, being connected by an equalizing line to the reaction vessel. Excess water from the overhead quench system plus recycled makeup water from the char settling pond is pumped into the quench tank, cooling the hot char and condensing the carrier steam. A circulating pump takes suction from the bottom of the tank and discharges back into the vessel, providing char-water slurry mixing and maintaining an even slurry consistency. Slurry concentrations of up to 30 weight percent solids can be readily handled.

#### 3.4.2.6 Reactor System Control

Each of the four fluidized beds of the reaction system is operated under level control. Feed solids enter at the top and are discharged from the bottom of the respective bed via a fluidized standpipe in combination with a throttling valve. The following five standpipes require connections for high-pressure nitrogen, providing blowback to clear the lines as required:

1. From slurry drier to first-stage mixing chamber.
2. From spouting bed separator to first-stage mixing chamber.
3. From spouting bed separator to second-stage reactor.
4. From second-stage reactor to the heat exchange bed.
5. From the heat-exchange bed to spent-char removal.

All blowback connections must be made directly above the respective solids valves involved. At the time start-up testing began, the primary element of the level controller for the char slurry drying bed was of the nuclear radiation and adsorption type. A pressure differential controller later came to be relied on exclusively for level-sensing. The remaining bed levels are controlled by a pressure differential controller operating on the pressure drop caused by solids level in the fluidized bed.

The recycle stream of solids that is returned to the first-stage mixing chamber serves as a means to control first-stage reaction conditions. This recycle stream is controlled by the temperature of the stream leaving the mixing chamber. Water sprays are used if needed to control reaction temperature in the spouting bed separator.

Instrument tubing connections to all parts of the reaction system require blowback gas lines to prevent plugging by the entry of solids.

With the hot box design, instrumentation is required to maintain a closely balanced pressure between the hot box and the process side of the reaction system vessel. Balance is maintained by admitting make-up nitrogen into the hot box to compensate for pressure increases on the process side, and by venting nitrogen if process pressure falls. A split-range differential pressure controller is connected between the hot box and the upper section of the reaction system vessel. Because there are no valves in the gas piping between the lower and upper fluid beds of the reaction system, the lower reaction also is held in pressure balance with the hot box.

The pressure balance between the hot box and process side can be upset by —

1. Severe surges in process operating pressure.
2. Faulty action of the nitrogen injection or vent controls.
3. Blockage of process gas lines in the reaction system vessel.

The internal piping and reaction zones of the vessel sections are adequate for pressure differential transients up to 60 psi. Protection against higher pressure differentials is provided by pressure relief devices such as the Black Sivalls and Bryson "two-way"-type that combines a Teflon diaphragm with perforated metal rupture discs. The use of Teflon limits maximum temperatures that can be tolerated for gases in contact with the devices. A satisfactory arrangement would be to locate each relief device in a separate external pressure container connected to the reactor system by pipes arranged to drain any condensation back into the reaction system.

Protection of the reaction system vessel against pressures in excess of the 1500 psig design value is provided by downstream pressure relief valves at the top of the gasifier. Valves for this service are code approved and pilot operated types, that close accurately after venting when the system pressure is not reduced much below the relief pressure setting. This precaution is needed to avoid excessive surges in the balanced pressure sections of the reactor, thus keeping rupture discs intact and limiting the effects of process upsets.

Balanced pressure is inherent in the water-jacketed vessel and piping designs that are discussed here, because the jackets are connected to the process side of the reaction system by an equalizing pipe. The process gas is pushed out when the jacket water system reaches normal operating temperature. The steam separator and all piping of the jacket water circulation system are sized to allow for process pressure surges that can occur in plant operation under emergency conditions.



### 3.4.2.7. Solids Flow Control Valves

Solids flow valves that are suitable for the service conditions of the process have not yet become commercially available, although progress has been made in this area. Existing valve types must be modified to meet HYGAS pilot plant duty requirements.

One coal feed valve handles solids at about 600°F; an existing standard commercial design was considered adequate for this valve body, but seals proved to be a problem. Three other solids valves operate at a 1400°F solids temperature in the pilot plant; special valve bodies are required for this high-temperature, high-pressure service. Although one of these valves is located in a normally 1400°F environment, it must operate at 1800°F solids temperature. All of the solids valves are in throttling service, and all handle fluidized solids. Operating differential pressures are small, and tight shut-off is not required.

Special attention is required to seal valve stems against the solids content of the reaction system streams. The final valve design for the hot-box section utilized butterfly pattern valves with high-temperature alloy bodies, stems, and valve disks.

In the hot box system, master and slave hydraulic operators provide a solution for the valve operation problem. The slave valve operator cylinder is located inside the hot box; hydraulic lines lead to the external master cylinder that is positioned by a control instrument signal. Differential pressures move the slaved valve to the same relative position taken by the master valve. Hydraulic operating pressures on both sides of the cylinders could be set above the reaction system pressures, so that any leakage across valve seals are outward. This design avoids the problem of running valve operating shafts through vessel shells, and all the attendant problems relative to process pressure, temperature, and sealing.

The valve that controls solids flow from the second-stage hydrogasifier to the heat-exchange stage is shown in Figure 3-9. The valve that controls the spent char outlet line below the reaction system vessel is also shown in Figure 3-10. Because no commercial designs were found to be adequate, both these valves were designed by HYGAS project engineers; final design was performed by T. D. Itil and Associates, Toledo, Ohio, in consultation with HYGAS personnel.

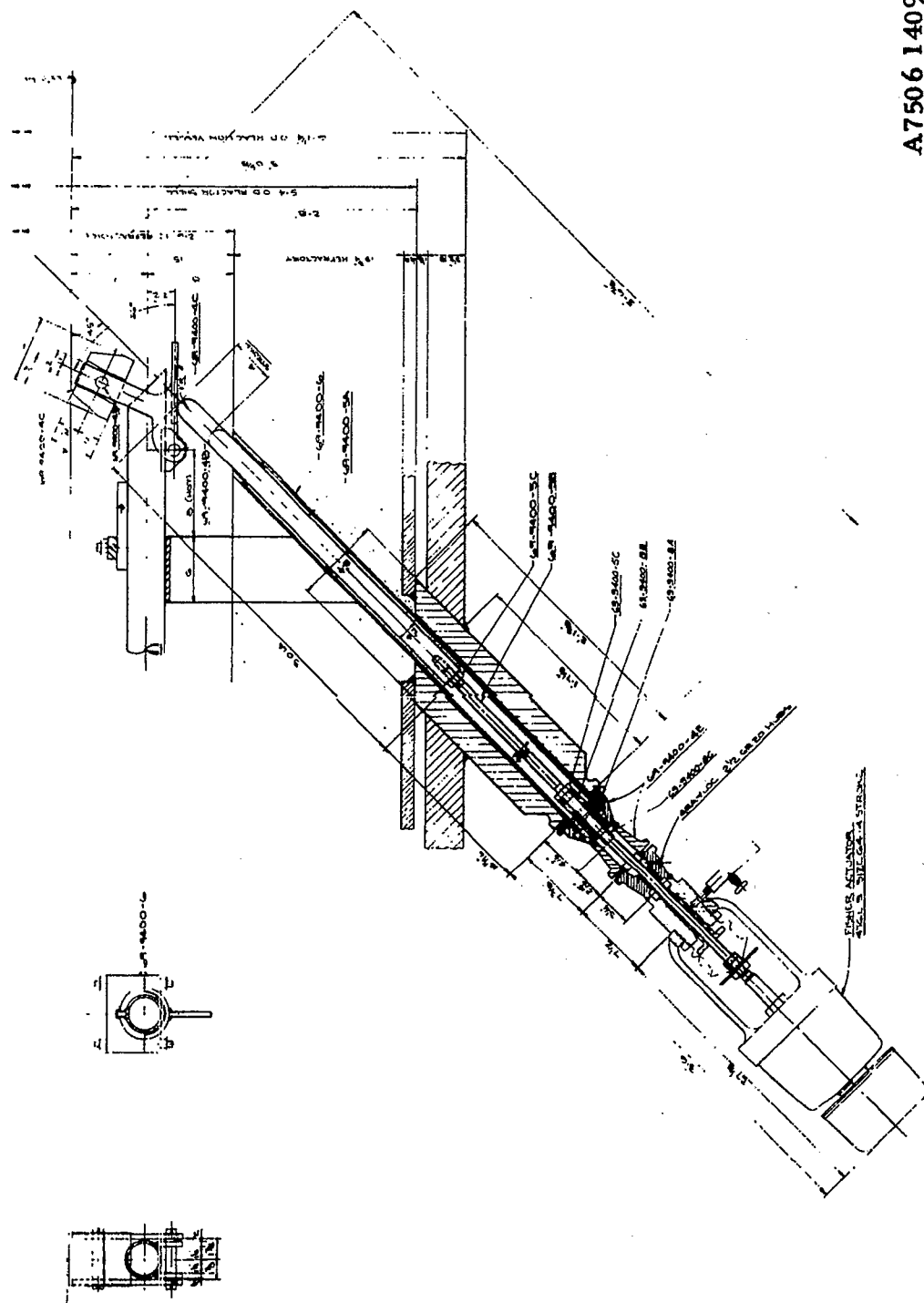
Generally, in start-up and subsequent operations, HYGAS engineers have found that —

- The design used in the Willis\* choke flow-control valve, which has contrarotating tungsten-carbide coated elements for valve trim, has proven to be satisfactory under high-pressure slurry letdown service for liquids.

- Kel-F, Viton, or Teflon elastomer seal materials are required for the HYGAS pilot plant aromatic light oil stream valves.

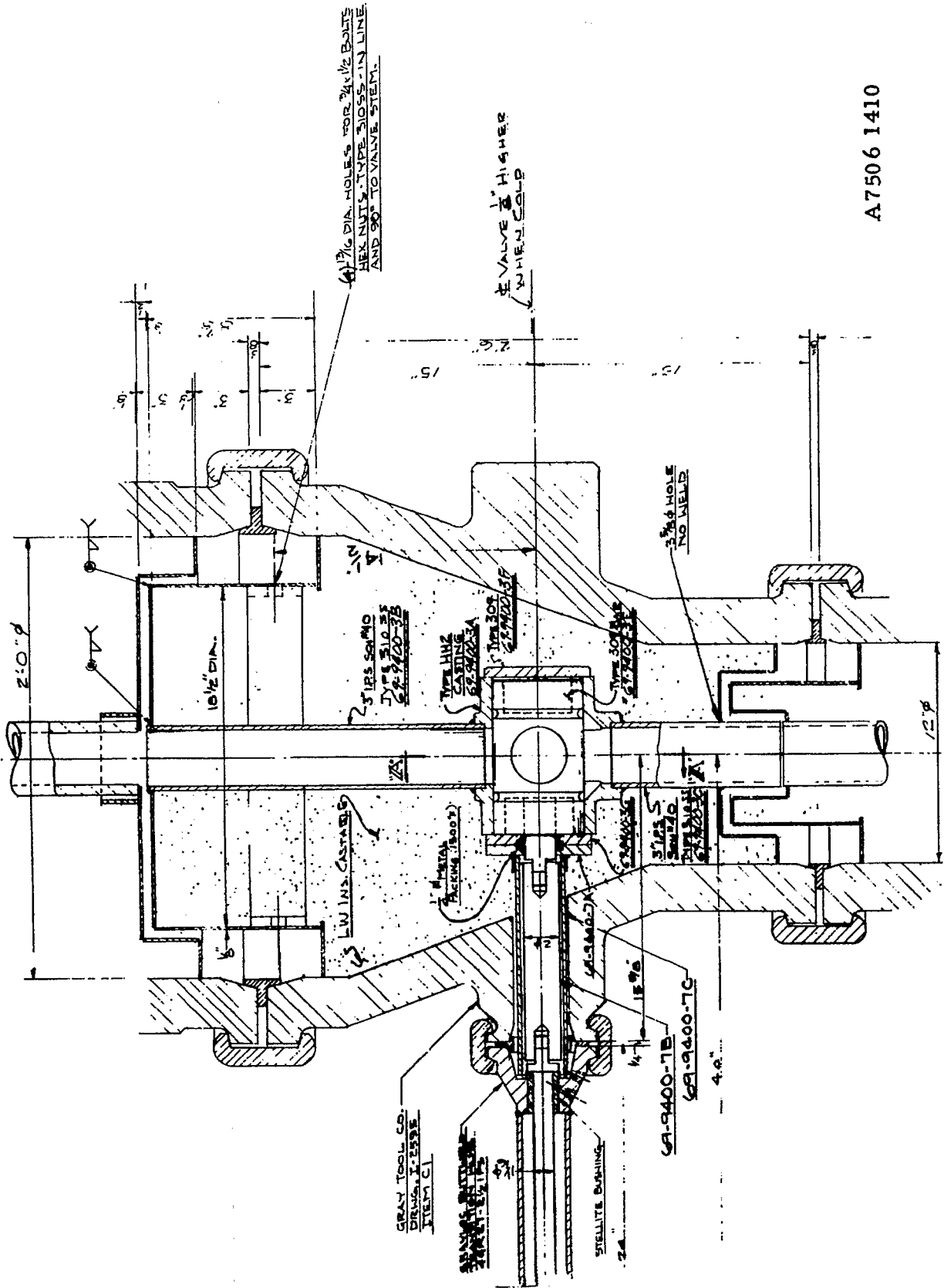
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\* Willis Oil Tool Corporation, Long Beach, California.



A7506 1409

Figure 3-9. VALVE TO CONTROL HYGAS PLANT SOLIDS FLOW FROM THE SECOND-STAGE HYDROGASIFIER TO THE HEAT-EXCHANGE STAGE



A7506 1410

Figure 3-10. VALVE TO CONTROL HYGAS PILOT PLANT SPENT CHAR  
OUTLET LINE BELOW THE REACTION SYSTEM VESSEL

- Centrifugal char-slurry pumps must run at speeds lower than 1800 rpm, and the casing and impeller should be hard-faced with Stellite or stainless steel overlays in order to withstand the erosive conditions of pumping even low concentrations of char slurry.

- The initial design of the HYGAS reactor incorporated Type 446 high-chrome, stainless steel for piping and certain components. This material was chosen for its excellent resistance to a hydrogen sulfide attack under reducing conditions. However, its lack of high-temperature tensile strength caused that material to be rejected after initial trial runs on the HYGAS reactor. Subsequently, HYGAS engineers have used Incoloy 800 or Type 330 stainless steel exclusively for internal hot transfer piping and reactor wall materials.

A report on materials and corrosion considerations in the HYGAS Process is included in Appendix 3-A.

### 3.4.3 Purification Section

#### 3.4.3.1 Scope

This section is divided into three functional units, namely, 3.4.3.2 Hydrogasifier Effluent Quench System, 3.4.3.3 CO<sub>2</sub> and H<sub>2</sub>S Removal System, and 3.4.3.4 Caustic Water Wash Scrubber System. Material balances and physical properties of streams for the three controlling cases are presented in Table 3-3. Please note that each of the three functional units within the purification section is designed for the requirements of its individual controlling case. For example, the design case for carbon dioxide removal is based on higher carbon dioxide concentrations prevalent in the lignite syngas from the electrothermal unit. On the other hand, the design case for the sulfur removal capacity of the system is based on a bituminous coal that is higher in sulfur content than are the other cases. Figure 3-11 presents the preliminary design PFD for this section.

The effluent quench system is designed for bituminous coal with a 50:50 steam-to-hydrogen ratio. To allow for variations in the heat load and off-gas rate, the system is designed for 120 to 125 percent of the calculated maximum rate. A controlling case material balance is shown in Table 3-4.

The carbon dioxide and hydrogen sulfide removal system is designed to clean up product gas from the gasification of lignite coal with synthesis gas generated in the electrothermal gasifier. (See Part IV: Hydrogen Generation of this report for details.) A design case material balance is provided in Table 3-5. The material balance and stream data for the controlling case of the MEA reclaimer is shown in Table 3-6.

The caustic and water wash section is designed for bituminous coal with a 50:50 steam-to-hydrogen ratio. The design caustic rate is based on the assumption that 0.3 mole per hour of carbon dioxide and 0.2 mole per hour of hydrogen sulfide must be removed from the gas. The material balance and stream physical properties for the caustic and water wash section is shown for a controlling case in Table 3-7.

**Table 3-3, Part 1. PURIFICATION SYSTEM CONTROLLING  
CASE FOR BITUMINOUS COAL 50:50 WATER-TO-HYDROGEN:  
MATERIAL BALANCE AND STREAM PHYSICAL PROPERTIES  
(Bechtel Preliminary Design Data)**

Stream Number Revision No.	401	403	404	406	407	413	414	427	
Stream Description	Quench Tower Feed	Quench Separator Water Disposal	Quench Separator Oil/Benzene Recycle	Oil To Disposal	Quench Tower Off-Gas	Absorber Off-Gas	Stripper Overhead Vent	Makeup Water To Stripper	Caustic & Wash Water Effluent
Temperature, °F	600	200	200	100	110	110	140	100	110
Pressure, Psig	1130	1200	1130	--	1125	1115	2	2	1105
Components	43.8 (44.01)	--	--	--	43.8	43.8	--	--	43.8
CO	21.6	--	--	--	21.6	21.6	--	--	--
H <sub>2</sub>	145.9	--	--	--	145.9	145.9	--	--	145.9
C <sub>12</sub> H <sub>10</sub>	79.8	--	--	--	79.8	79.8	--	--	79.8
C <sub>16</sub> H <sub>14</sub>	1.9	--	--	--	1.9	1.9	--	--	1.9
H <sub>2</sub> O	4.8	--	--	--	4.8	4.8	--	--	--
N <sub>2</sub>	2.0	--	--	--	2.0	2.0	--	--	2.0
HCN	143.5	143.2	--	--	0.3	0.3	6.3	6.3	0.3
Cells	154.8	--	12000 lb/hr	--	1.0	1.0	--	--	1.0
Oil	0.7	--	154 lb/hr	154 lb/hr	0.7	0.7	--	--	0.7
Oil	138 lb/hr	--	154 lb/hr	154 lb/hr	--	--	--	--	--
Total Moles/Hour	595.8(1)	2650	12154	154	20.8	20.8	32.7	6.3	275.4
Total lbs/Hour	1887.6	18.02	12154	154	137.2	137.2	1226	114	3028
Molecular Weight	31.3(1)	18.02	--	--	--	16.3	37.55	18.02	10.99
Vapor	3.77(1)	--	--	--	2.61	2.61	0.37	--	2.02
LCFM at Oper. Cond.	3.55(1)	--	--	--	26.4	26.4	211	--	25.0
Liquid	--	5.3(3)	0.2	--	--	--	--	0.2	--
GPM at Oper. Cond.	--	0.3(3)	--	--	--	--	--	0.68	--
Viscosity, CP at Oper. Cond.	--	--	--	--	--	--	--	--	--
Critical Properties	--	--	--	--	--	--	--	--	--
Tc - Pseudo Critical Press.	--	--	--	--	455	455	--	--	391
Tc - Pseudo Critical Temp.	--	--	--	--	212	212	--	--	182
Z - Compressibility	0.96(1)	--	--	--	0.95	0.95	--	--	1.00

Note: (1) Calculated on an oil free basis.  
(2) Only water solution. Physical properties based on water.

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Table 3-3, Part 2. PURIFICATION SYSTEM CONTROLLING CASE FOR BITUMINOUS COAL 30:70 WATER-TO-HYDROGEN; MATERIAL BALANCE AND STREAM PHYSICAL PROPERTIES

Stream Number Revision No.	401	403	404	460	407	413	414	427
Stream Description:	Quench Tower Feed	Quench Separator Water Disposal	Quench Separator Oil & Benzene Recycle	Oil to Disposal	Absorber Off-Gas	Stripper Overhead Sent	Makeup Water To Stripper	Caustic & Wash Water Effluent
Temperature, °F	600	200	200	100	110	110	100	110
Pressure, psi±	1150	1200	1150	--	1115	1115	2	1105
Component	53.7	--	--	--	53.7	--	--	53.7
CO	10.6	--	--	--	10.6	10.6	--	--
CO <sub>2</sub>	119.6	--	--	--	119.6	--	--	119.6
H <sub>2</sub>	77.3	--	--	--	77.3	--	--	77.3
CH <sub>4</sub>	0.1	--	--	--	0.1	--	--	0.1
C <sub>2</sub> H <sub>6</sub>	4.9	--	--	--	4.9	4.9	--	--
H <sub>2</sub> S	2.2	--	--	--	2.2	--	--	2.2
H <sub>2</sub> O	54.0	54.0	--	--	0.3	3.7	3.7	0.3
Cells	153.7	--	12000 lb/hr	--	0.9	--	--	0.9
C <sub>2</sub> H <sub>4</sub>	0.3	--	52 lb/hr	--	0.3	--	--	0.3
O <sub>2</sub>	114 lb/hr	--	52 lb/hr	52 lb/hr	--	--	--	--
Total Moles/Hour	477.0(1)	--	12052	52	254.4	19.2	3.7	254.4
Total Lbs/Hour	16802	978	--	--	3128	599	67	3138
Molecular Weight	35.0(1)	18.02	--	--	12.33	36.41	18.02	12.33
Vapor	4.4(1)	--	--	--	2.28	0.094	--	2.26
lbs./ft <sup>3</sup> at Oper. Cond.	63.6(1)	--	--	--	24.1	124	--	23.1
ACFM at Oper. Cond.	--	2(2)	28	--	--	--	0.1	--
Liquid	--	0.3(2)	0.2	--	--	--	0.68	--
GPM at Oper. Cond.	--	--	--	--	--	--	--	--
Viscosity, CP at Oper. Cond.	--	--	--	--	--	--	--	--
Critical Properties	--	--	--	--	--	--	--	--
Pc - Pseudo Critical Press.	--	--	--	--	411	--	--	411
Tc - Pseudo Critical Temp.	--	--	--	--	189	--	--	189
Z - Compressibility	0.82(1)	--	--	--	1.0	--	--	1.0

Note: (1) Calculated on an oil free basis.  
(2) Only water solution. Physical properties based on water.

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Table 3-3, Part 3. PURIFICATION SYSTEM CONTROLLING CASE  
FOR LIGNITE: MATERIAL BALANCE AND STREAM PHYSICAL PROPERTIES

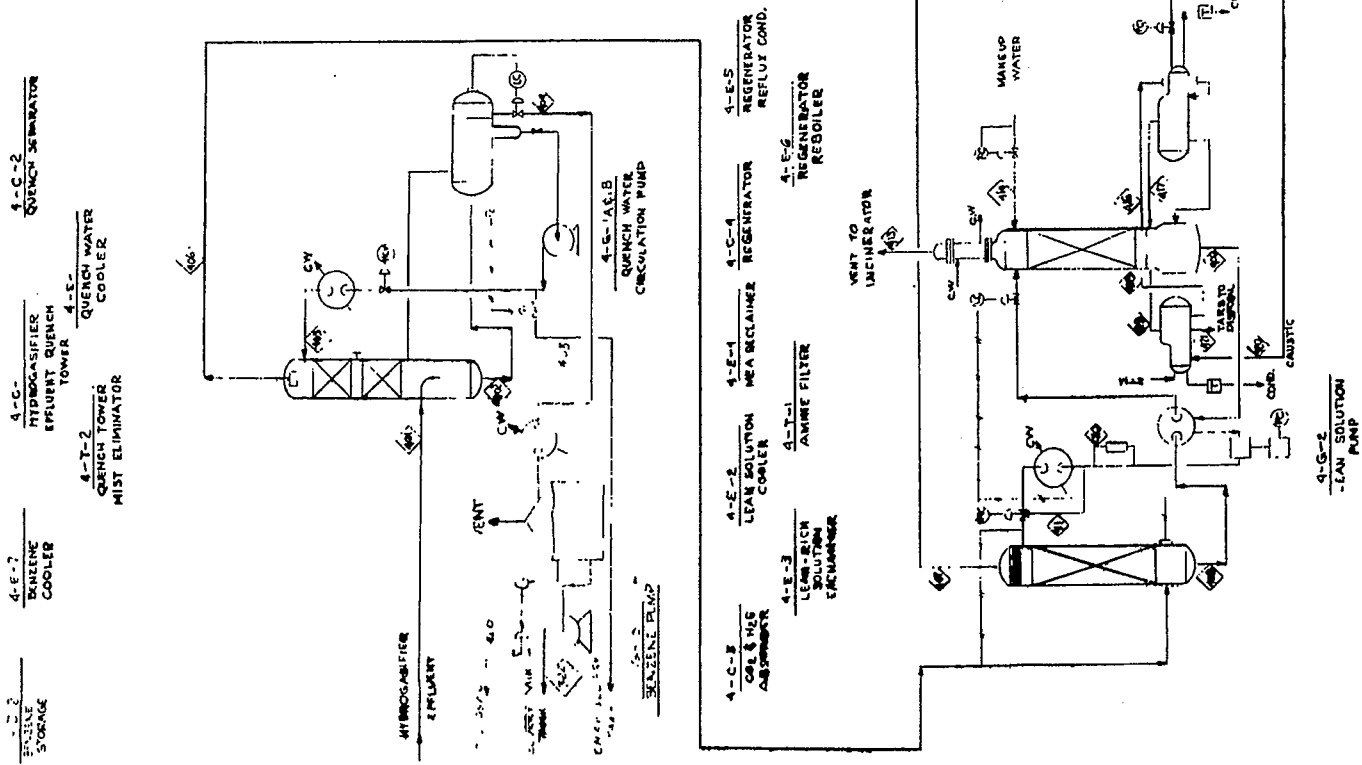
Stream Number Revision No.	401	403	404	460	407	413	414	427	
Stream Description	Quench Tower Feed	Quench Separator Water To Disposal	Quench Separator Oil & Benzene Recycle	Oil To Disposal	Absorber Off-Gas	Stripper Overhead Vent	Makeup Water To Stripper	Quench & Wash Water Effluent	
Temperature, °F	600	200	200	100	110	140	100	110	
Pressure, psig	1130	1200	1130	--	1125	2	2	1105	
Components	25.2 35.4 25.2 76.4 30.07 24.08 28.01 18.02 78.11 44.09	-- -- -- -- -- -- 105.2 -- -- --	-- -- -- -- -- -- -- 12000 lb/hr 307 lb/hr	-- -- -- -- -- -- -- 307 lb/hr	110 1115 25.2 93.6 76.4 3.7 1.1 1.7 0.3 0.8 1.2	140 2 35.4 -- -- -- 1.1 7.7 -- -- --	-- -- -- -- -- -- 7.7 -- -- --	25.2 -- 93.6 76.4 3.7 -- 1.7 0.3 0.8 1.2	110 1105 25.2 -- 93.6 76.4 3.7 -- 1.7 0.3 0.8 1.2
Total Moles/Hour	488.0(1)	1895	12307	307	232.4	44.2	7.7	202.9	
Total Lbs/Hour	18198	1802	12307	307	1796	1735	138	2401	
Molecular Weight	35.9(1)	1802	--	--	16.69	39.26	18.02	11.83	
Vapor	4.5(1)	--	--	--	3.24	0.162	--	2.18	
Lbs/Ft <sup>3</sup> at Oper. Cond.	58.0(1)	--	--	--	21.5	283	--	18.4	
ACFM at Oper. Cond.	--	--	--	--	--	--	--	--	
Liquid	--	4(2)	28	--	--	--	0.3	--	
GM at Oper. Cond.	--	0.3(2)	0.2	--	--	--	0.68	--	
Viscosity, Cp at Oper. Cond.	--	--	--	--	--	--	--	--	
Critical Properties	--	--	--	--	530	--	--	431	
Tc - Pseudo Critical Press.	--	--	--	--	260	--	--	208	
Tc - Pseudo Critical Temp.	0.83(1)	--	--	--	0.96	--	--	1.0	
Z - Compressibility	--	--	--	--	--	--	--	--	

Note: (1) Calculated on an oil free basis.  
(2) Only water solution. Physical properties based on water.

A7506 1546B

INSTITUTE OF GAS TECHNOLOGY CHICAGO	
COAL HYDROGASIFICATION PILOT PLANT	
PROCESS FLOW DIAGRAM PURIFICATION SECTION	
PROJECT NO.	DRAWING NO.
IGT	PFD-3
SCALE	REV.

Figure 3-11. PRELIMINARY DESIGN PFD FOR PURIFICATION SECTION OF HYGAS PILOT PLANT



A7506 1415



**Table 3-4. EFFLUENT QUENCH SYSTEM CONTROLLING  
CASE FOR BITUMINOUS COAL 50:50 WATER-TO-HYDROGEN:  
MATERIAL BALANCE AND STREAM PHYSICAL PROPERTIES  
(Bechtel Preliminary Design Data)**

Stream Number Revision No.		401	402	403	404	405	406
Stream Description		Quench Tower Feed	Quench Tower Bottoms	Quench Separator Water Disposal	Quench Separator Oil & Benzene Recycle	Quench Water Inlet	Quench Tower Off-Gas
Temperature, °F		600	200	200	200	100	110
Pressure, psig		1130	1130	1200	1130	1125	1125
Components							
CO	(28.01)	43.8	--	--	--	--	43.8
CO <sub>2</sub>	(44.01)	21.6	--	--	--	--	21.6
H <sub>2</sub>	(2.02)	145.9	--	--	--	--	145.9
CH <sub>4</sub>	(16.04)	79.8	--	--	--	--	79.8
C <sub>2</sub> H <sub>6</sub>	(30.07)	1.9	--	--	--	--	1.9
H <sub>2</sub> S	(34.08)	4.8	--	--	--	--	4.8
H <sub>2</sub> O	(28.01)	2.0	--	--	--	--	2.0
C <sub>2</sub> H <sub>4</sub>	(28.02)	143.5	3862.1	143.2	--	3718.9	1.2
C <sub>2</sub> H <sub>2</sub>	(78.11)	154.8	153.6	--	12000 lb/hr	--	1.2
C <sub>2</sub> H <sub>6</sub>	(44.09)	7	--	--	--	--	0.7
Oil	--	10/hr	154 lb/hr	--	154 lb/hr	--	--
Total Moles/Hour		526.8(1)	--	--	12154	--	301.8
Total Lbs/Hour		18724	2560	18.02	--	67000	4142
Molecular Weight		31.3(2)	--	--	--	--	13.72
Vapor		2.5(1)	--	--	--	--	2.61
Liquid		2.5(1)	--	--	--	--	26.4
SPV at Oper. Cond.		--	170.0(2)	5.2	28	135.4	--
Viscosity, CP at Oper. Cond.		--	0.23(2)	0.3	0.2	0.68	--
Pc - Pseudo Critical Press.		--	--	--	--	--	455
Tc - Pseudo Critical Temp.		--	--	--	--	--	216
Z - Compressibility		0.86(1)	--	--	--	--	0.98

Note: (1) Calculated on an oil-free basis.  
(2) Only water solution. Physical properties based on water.

A7506 1547

Table 3-5. CARBON DIOXIDE AND HYDROGEN SULFIDE  
 ABSORPTION SYSTEM DESIGN CASE FOR SYNTHESIS GAS AND  
 LIGNITE: MATERIAL BALANCE AND STREAM PHYSICAL PROPERTIES  
 (Bechtel Preliminary Design Data)

Stream Number Revision No.	406	407	408	409	410	411	413	414	416	417	
Stream Description	Quench Tower Off-Gas	Absorber Effluent Off-Gas	Rich DEA From Absorber	Lean DEA From Stripper	Lean Sol. To Filter	Lean Sol. Exchanger By-Pass	Stripper Overhead Vent	Make-Up Water To Stripper	Regenerator Reboiler Inlet	Regenerator Reboiler Vapor Outlet	
Temperature, °F	110	110	139	238	197	197	140	100	238	238	
Pressure, psig	1125	1115	1125	8	1200	1175	2	2	8	8	
Components	CO (8.01) CO <sub>2</sub> (4.01) H <sub>2</sub> (2.02) CH <sub>4</sub> (16.04) C <sub>2</sub> H <sub>6</sub> (2.07) H <sub>2</sub> S (28.01) H <sub>2</sub> (1.1) H <sub>2</sub> O (28.92) C <sub>2</sub> H <sub>6</sub> (4.09) DEA (105.14)	29.2 87.3 87.7 76.4 3.7 1.7 0.3 1.2	90.3 -- -- -- 1.1 6082 -- 261	-- -- -- -- -- 6082 -- 261	Basis for Basis for Design Design 10% Lean DEA Lean DEA From Stripper Reg 608	10% Design 20% Lean DEA Lean DEA From Stripper Reg 608	90.3 -- -- 1.1 19.2	-- -- -- -- 19.2	-- -- -- -- 19.2	-- -- -- -- 6987.7	-- -- -- -- 905.7
Total Moles/Hour	292.6	201.2	6434.4	6343	634	1268	110.6	19.2	7248.7	905.7	
Total lbs./hour	6527	2516	141012	137000	13700	27400	4358	346	153320	16320	
Molecular Weight	22.31	12.5	21.91	21.6	21.6	21.6	39.40	18.02	21.15	18.02	
Vapor	434	2.31	--	--	--	--	0.102	--	--	0.58	
lb./ft. <sup>3</sup> at Oper. Cond.	25.1	18.2	--	--	--	--	7.12	--	--	4700	
ACFM at Oper. Cond.	--	--	--	--	--	--	--	--	--	--	
Liquid	--	--	272	272	27	54	--	0.7	314	--	
GF <sub>M</sub> at Oper. Cond.	--	--	0.94	0.35	0.54	0.54	--	0.68	0.35	--	
Viscosity, CP at Oper. Cond.	--	--	--	--	--	--	--	--	--	--	
Critical Properties	--	--	--	--	--	--	--	--	--	--	
Pc - Pseudo Critical Press.	--	--	--	--	--	--	--	--	--	--	
Tc - Pseudo Critical Temp.	--	--	--	--	--	--	--	--	--	--	
Z - Compressibility	--	--	--	--	--	--	--	--	--	--	

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Table 3-6. MEA RECLAIMER SYSTEM DESIGN CASE: MATERIAL BALANCE AND STREAM PHYSICAL PROPERTIES (Bechtel Preliminary Design Data)

Stream Number	418	419	420	421
Revision No.				
Stream Description	Reclaimer Inlet	Reclaimer Outlet	Caustic to Reclaimer	Tars From Reclaimer
Temperature, °F	278	300	300	300
Pressure, psig	28	8	0	0
Component	Estimated Maximum Values Conditions Vary greatly over regeneration period		Normally No Flow Properties based on 10% NaOH Solution	Normally No Flow
CO				
CO <sub>2</sub>				
H <sub>2</sub>				
CH <sub>4</sub>				
C <sub>2</sub> H <sub>6</sub>				
H <sub>2</sub> S				
H <sub>2</sub> O				
CaH <sub>2</sub>				
CaS				
MEA				
Total Moles/Hour	12.8	14.9 <sup>5</sup>		
Total Lbs/Hour	288	268		
Molecular Weight	20.9	18-61		
Vapor		0.051 <sup>5</sup>		
Lbs/Ft <sup>3</sup> at Oper. Cond.		57.2		
Liquid				
GPM at Oper. Cond.	0.74			
Viscosity, CP at Oper. Cond.	0.474		5.0	5.0
Critical Properties			1.0	
Pc - Pseudo Critical Press.				
Tc - Pseudo Critical Temp.				
Z - Compressibility				

Note: (4) Physical properties based on 30 wt % MEA  
 (5) Physical properties based on steam

A7506 1549

Table 3-7. CAUSTIC AND WATER WASH SYSTEM CONTROLLING  
CASE FOR BITUMINOUS COAL 50:50 WATER-TO-HYDROGEN:  
MATERIAL BALANCE AND STREAM PHYSICAL PROPERTIES  
(Bechtel Preliminary Design Data)

Stream Number Revision No.	407	422	423	424	425	426	427	
Stream Description	Absorber Off-Gas	Spent Caustic To Disposal	Recirculated Caustic Solution	Make-up Caustic	Wash Water Inlet	Wash Water Outlet	Caustic & Wash Water OVRD Vapor	
Temperature, °F	110	110	110	110	110	110	110	
Pressure, psig	1115	1110	1110	1108	1105	1108	1105	
Components	43.8 0.3 145.9 79.8 1.9 2.0 0.3 1.0 0.7	Caustic CO <sub>2</sub> & 0.2 mol/hr H <sub>2</sub> S in feed gas. Note that there is no change in the Benzene & water content of the gas (from mench to methanator feed - benzene (based on partial pressure) will be condensed in amine or caustic systems unless there is an increase in the temperature of the gas	Scrubber	Design	Based on	0.3 mol/hr		43.8 145.9 79.8 1.9 2.0 0.3 1.0 0.7
Total Moles/Hour	275.9	340	5065	320	2500	2500	275.9	
Total Lbs/Hour	3048						3028	
Molecular Weight	11.05						10.99	
Vapor	2.05						2.02	
Lbs/Ft. <sup>3</sup> at Oper. Cond.	24.9						25.0	
Acry at Oper. Cond.								
Liquid		0.67	10	0.62	0.61	0.61		
GPM at Oper. Cond.								
Viscosity, CP at Oper. Cond.								
Critical Properties								
Tc - Pseudo Critical Press.	393						391	
Tc - Pseudo Critical Temp.	183						182	
Z - Compressibility	1.0						1.00	

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### 3.4.3.2. Hydrogasifier Effluent Quench System

#### 3.4.3.2.1. Basis for Design

The hydrogasifier effluent quench system consists of a hydrogasifier effluent quench tower, a quench separator, quench water cooler, and quench water circulation pump.

The cooling medium is water. The primary functions of this section are to cool the effluent gases from the hydrogasifier from about 600<sup>0</sup> down to about 100<sup>0</sup>F, and to remove water, light aromatic slurry oil, tars, oils, and fines that might be in the gas stream. The off-gas from this tower, which is the feed to the CO<sub>2</sub> and H<sub>2</sub>S removal system, is saturated with water and light aromatic oil. It is important, therefore, to keep the overhead temperature as low as possible, in order to minimize the possibility of having light aromatic oil condense in the carbon dioxide and hydrogen sulfide absorber and/or in the caustic and water wash scrubber.

An interface controller determines the level of water in the settler and discharges, from the boot in the bottom of the settler, excess water, oils, and tars that are heavier than water. Light oils and benzene overflow the internal weir and are drawn off under level control.

Water is condensed in this system; therefore, under normal operating conditions, no makeup water is required. A makeup line is included on the pump suction line, however, in the event that it becomes necessary to add water while the system is at operating pressure.

Temperature of the quench water being returned to the tower is controlled. As mentioned, under normal operating conditions the temperature of the overhead should be kept as low as possible to minimize the possibility of benzene condensation.

A control valve on the off-gas line from the quench tower is instrumented to shut at high temperature if the cooling or quench water systems should fail. Simultaneously, the following flows also are stopped:

1. Hydrogen into the hydrogasifier.
2. Steam into the hydrogasifier.
3. Pretreated char into the hydrogasifier.

Steps 1 and 2 may be accomplished by diverting the normal flow of hydrogen and steam upstream of the superheaters. If normal hydrogen and steam flows are diverted, the superheater burners must be shut down. Further, if it is necessary to prevent local overheating in the firebox of these superheaters, snuffing steam might have to be used to protect the tubes from local overheating.

#### 3.4.3.2.2. Hydrogasifier Effluent Quench Tower (4-C-1)

This effluent quench tower is 2 feet 0 inch ID by 34 feet 10 inches long. A Centrifix-type separator is located in the top to reduce solid and liquid entrainment in the overhead gas. After overhead gas enters the tower, the inlet gas nozzle is turned downward to prevent hot gas impingement on the tower wall.

The original design of this section included a packed section containing 1.5-inch pall rings; however, the section proved to be inoperative following a few runs because coal fines carried overhead with effluent gases from the hydrogasifier plugged the small spaces available for gas flow in the packed section. The packed section was taken out and a section of side-by-side baffle trays was installed in place of the quench-tower packing.

#### 3.4.3.2.3. Quench Water Circulation Pumps (4-C-1, 1B)

The quench water circulation pumps provide one of the more critical pump services in the HYGAS pilot plant. All equipment downstream of this system had to be protected against exposure to the hot hydrogasifier gases. A spare pump is provided and is instrumented to start automatically on low quench water circulation flow. In order to avoid thermal shock or possible freeze-up in the pump that was on standby, provisions were made to keep the standby warm at all times. Specifications of the pumps actually installed are tabulated below:

Manufacturer: Ingersoll-Rand  
Model Number and Type: 1-1/2 X 8 MHH

<u>Operating Conditions</u>	<u>Main</u>	<u>Spare</u>
	Motor	Turbine
Horsepower	20	16.1
GPM	175	175
Temperature, °F	200	200
Inlet Pressure	1465 psig	1465 psig
Outlet Pressure	1525 psig	1525 psig

#### 3.4.3.2.4. Quench Separator (4-C-2)

The quench separator is a horizontal vessel 4 feet 0 inch ID by 19 feet 8 inches long. The elevation of the quench tower and separator are such that a 24-inch liquid holdup is provided in the bottom of the quench tower. This liquid level provides protection to the bottom of the tower in case the entire circulating quench water flow fails.

For materials balance calculations, all of the oil is assumed to leave via the overflow, with the aromatic slurry oils.

### 3.4.3.2.5. Quench Water Cooler (4-E-1)

The quench water cooler is designed to remove 8 million Btu per hour, based on cooling the quench water from 200° down to 100°F. For the three design cases specified, the maximum required cooling duty is slightly above 6.5 million Btu per hour. Upset conditions in the hydrogasification section may put an increased burden on the quench system. Specifications of the cooler installed are:

	<u>Shell</u>	<u>Tube</u>
Fluid	Cooling Water	Quench Water
Total Fluid Entering	148,890 lb/hr	67,000 lb/hr
Gravity (in/out)	0.995/0.989	0.965/0.995
Temperature (in/out)	75°/120°F	200°/100°F
Operating Pressure	90 psig	1200 psig
No. Passes	1	4
Velocity	--	2.07 ft/s
Pressure Drop	10.0	4.0
Fouling Factor	0.006	0.006
Heat Exchanged	6,700,000 Btu/hr (8,000,000 designed)	

### 3.4.3.3. CO<sub>2</sub> and H<sub>2</sub>S Removal System

#### 3.4.3.3.1. Basis for Design

The system described is designed to remove essentially all of the CO<sub>2</sub> and H<sub>2</sub>S in the gas, using either 20 weight percent MEA or DEA. As testing progressed, the HYGAS plant switched from an MEA/DEA solution mix to diglycolamine as the absorbing medium, and initially experienced a heavy foaming of the diglycolamine. After adding an antifoaming agent, the foaming problem was resolved. Normally, amine systems are not used to remove organic sulfur compounds such as mercaptans, COS, etc.; however, diglycolamine can be used to remove COS by direct chemical reaction with amine.

The design case for this section occurs when lignite is used with synthesis gas (CO and H<sub>2</sub> mixtures), instead of with hydrogen. The synthesis gas would be generated in the electrothermal gasifier, an item that was later built and tested.

#### 3.4.3.3.2. CO<sub>2</sub> and H<sub>2</sub>S Absorber (4-C-3)

The absorber is a 30-inch I.D. by 69 feet 0 inch long column. The feed gas entering the absorber is saturated with light aromatic oil which, if permitted to condense, would cause foaming and maloperation of the column. To prevent aromatic oil condensation, the lean solution enters the column under differential temperature control with the incoming gas. The lean solution, therefore, must always be a minimum of 10°F higher than the incoming gas.

The column is such that the following packings were considered: 1-inch metal pall rings, 1-inch Intalox saddles, or 1.5-inch carbon Raschig rings; 1-inch polypropylene rings were installed. Subsequently, the pilot plant standardized on 1.5 or 2.0-inch rings.

The diameter of the column was calculated using the United States Stoneware Company "generalized pressure drop correlation". Column loading data sheets for this and other packed columns have been compiled. Packing manufacturers such as Stoneware and Koch were asked to verify the column diameters, because the pressure drop correlations might not be applicable at these high pressures.

The absorber has been specified for two 25-foot packed sections, with a liquid redistributor between the two beds. A 6-inch-thick stainless steel demister pad is specified to minimize the liquid entrainment in the overhead vapor.

#### 3.4.3.3.3. Regenerator (4-C-4)

The regenerator is a 60-inch by 48-inch I.D. by 73-foot long column. The diameter of this column is selected so that the following packings might be used: 1.5-inch pall rings, 1-inch pall rings, 1.5-inch Intalox saddles, or 2-inch carbon Raschig rings. In practice, 1.5-inch 304 stainless steel and 1.5-inch polypropylene pall rings were installed.

The column diameter and packing listed was verified with the packing manufacturers.

The bottom section of the column is enlarged to allow for 20 minutes holdup, in order to provide surge for the system. The reboiler must be so located that liquid from the draw-off pan flows into it by gravity; overflow from the reboiler flows into the column.

#### 3.4.3.3.4. Lean Solution Pump (4-G-2)

If a reciprocating pump were to be used in this service, extreme care would have to be taken in the design of the circuit to allow for pulsations caused by the pump. The pump should be at least triplex, and supplied with pulsation dampeners, etc. Specifications were originally made for a reciprocating pump (Bechtel Drawing Number A-4G2-1), and for a multistage centrifugal pump (the alternative shown in Bechtel Drawing A-4G2).\*

A centrifugal pump was selected for this service. A 9-stage centrifugal pump with a control system was installed. NPSH requirements for this pump determined the required height of the regenerator. Operating characteristics of the pump installed are tabulated below.

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\* These drawings are not included here.



Manufacturer: Ingersoll Rand  
 Model Number and Type: 3HMTA-9  
 S/N: 056936  
 Duty: DGA Solution ( 50% )  
 Operating Conditions:  
     HP 50  
     GPM 300  
     Temperature 194° F  
     Inlet Pressure 65 psig  
     Outlet Pressure 1600 psig

3.4.3.3.5. Amine Filter (4-T-1)

This filter has a design throughput of 300 gpm with a 20 psi drop allowed. For the specified flow rate with a clean filter the pressure drop is very low. As the filter becomes fouled, the pressure drop increases to a maximum of 20 psi for the specified flow rate. It will be necessary to readjust the bypass valve to maintain the specified flow as the filter fouls. When the filter fouling causes a drop of 20 psi, the filter internals must be replaced. Extra filter elements are stocked for this purpose.

3.4.3.3.6. Lean Solution Cooler (4-E-2)

This exchanger is designed with a hot bypass on the process side for temperature control of the lean solution entering the absorber. The cooling water side of the exchanger is protected against the high-pressure process side with a safety valve. Specifications of the shell and tube heat exchanger installed are:

	<u>Shell Side</u>	<u>Tube Side</u>
Fluid Circulated	Cooling Water	20% DEA solution
Total Fluid (lb/hr)	214,000	137,000
Gravity (in/out)	0.995/0.989	0.989/1.008
Viscosity CP (in/out)	0.90/0.56	0.54/1.30
Temperature in	75° F	194° F
Temperature out	120° F	110° F
Operating Pressure -Inlet (Max)	90 psig	1600 psig
Fouling Factor	0.006	0.006
Heat Exchanged	9,650,000 Btu/hr	

3.4.3.3.7 Lean-Rich Solution Exchanger (4-E-3)

The low-pressure shell side of this exchanger is located on the suction side of the lean-solution pump. The allowable shell-side pressure drop was specified as 2 psi. Specifications of the shell and tube heat exchanger actually installed are tabulated below.

	<u>Shell Side</u>	<u>Tube Side</u>
Fluid Circulated	Lean 20% DEA Solution	Rich 20% DEA Solution
Total Fluid Entering	137,000 lb/hr	141,012 lb/hr
Gravity (in/out)	0.977/0.989	1.001/0.989
Viscosity (in/out)	0.35/0.54	0.94/0.64
Temperature in	238°F	139°F
Temperature out	194°F	180°F
Operating Pressure-Inlet (Max)	12 psig	1125 psig
Pressure Drop Allowable	2 psi	10 psi
Fouling Factor	0.002	0.002
Heat Exchanged	5,510,000 Btu/hr	

#### 3.4.3.3.8. Regenerator Reflux Condenser (4-E-5)

This unit was originally specified as a vertical reflux type condenser, flanged to the top of the regenerator. The exchanger data sheet specified the cooling water on the shell side. In design refinement, a U-tube condenser was used with water on the tube side. Operating characteristics of the U-tube heat exchanger installed are tabulated below.

	<u>Shell Side</u>	<u>Tube Side</u>
Fluid Circulated	Regenerator Overhead	Cooling Water
Fluid Entering (Vapor 4358; Steam, 6065)	10,423 lb/hr	133,000 lb/hr
Gravity (in/out)	0.99	0.945/0.995
Viscosity (in/out)	0.56	0.90/0.56
Temperature in	215	75
Temperature out	213 to 140	120
Operating Pressure (Inlet)	2 psig	50 psig
Pressure Drop Maximum Allowable	Min.	10 psi
Fouling Factor	0.001	0.006
Heat Exchanged	6,000,000 Btu/hr	

#### 3.4.3.3.9. Regenerator Reboiler (4-E-6)

Specifications for this unit require a kettle-type regeneration reboiler with the liquid overflow being returned to the regenerator. Forty psig steam at 287°F is the specified heating medium. Higher tube side temperatures will increase the corrosion rate on the amine side of the reboiler.

#### 3.4.3.3.10 MEA-DEA Reclaimer (4-E-4)

MEA-DEA degradation products and contaminants are removed from this system in the MEA-DEA reclaimer. The reclaimer may be operated continuously or intermittently, depending on the condition of the solution and the results obtained. The purpose of the reclaimer is to boil water and

MEA-DEA out of the amine solution, thus concentrating the impurities in the reclaimer. Amine solutions tend to be more corrosive when they contain degradation products and contaminants. The concentrated sludge is withdrawn from the system.

#### 3.4.3.4. Caustic and Water Wash Scrubber System

##### 3.4.3.4.1. Basis for Design

The scrubber includes a 10-foot-high packed caustic wash section, followed by a 5-foot-high water wash section. A trapout tray keeps water from entering the caustic section. A demister pad is specified to minimize liquid entrainment in the overhead vapor.

This column is designed to remove CO<sub>2</sub> and H<sub>2</sub>S that have not been removed from the gas stream in the absorber. Inlet wash water, which enters at 110°F, is taken from the low-pressure condensate return through a high-pressure pump.

The caustic injection pump specified is a small, single acting reciprocating pump with a built-in flow regulator. The caustic circulation pump specified is a low-head centrifugal type.

##### 3.4.3.4.2. Caustic and Water Wash Column (4-C-5)

This column is 18 inches I.D. by 31 feet 10 inches long and includes a 5-foot packed section for water wash and a 10-foot packed section for caustic wash.

The column diameter accommodates the following packings (although any suitable 3/4-inch to 1-inch packing may be used):

0.75-inch and 1.0-inch Carbon Raschig Rings

In practice, 1-inch polypropylene rings were installed. The column diameter and packing shown above were verified with the packing manufacturer.

##### 3.4.3.4.3. Caustic Circulation Pump (4-G-3)

The specified design flow of this pump is 12 gpm, but it operates at lower flow rates. Characteristics of the pump installed are tabulated below.

Manufacturer:	Ingersoll Rand
Model Number and Type:	1-1/2 inch x 8 MHH
S/N:	066965
Operating Conditions:	
HP	10
GPM	12
Temperature	110°F
Inlet Pressure	1450 psig
Outlet Pressure	1500 psig

#### 3.4.3.4.4 Caustic Injection Pump (4-G-4)

The caustic injection pump operates continuously at the flow rate shown. The pump rate specified was calculated based on an assumed CO<sub>2</sub> and H<sub>2</sub>S concentration in the feed gas; variations in this concentration would change the caustic make-up requirements. Operational characteristics of the pump installed are tabulated below.

Manufacturer:	Hills-McCanna
Model Number and Type:	P/D - II - 1F
S/N:	32314
Duty:	10% NaOH Solution
Operating Conditions:	
HP	2
GPH	35
Temperature	110°F
Inlet Pressure	Gravity Feed
Outlet Pressure	1550 psig

#### 3.4.4. Methanation Section

##### 3.4.4.1. Basis for Design

Hydrogen gas from the hydrogen compressor is blended as required with the purified gas to give the proper hydrogen-to-carbon monoxide ratio.

This system consists of two fixed-bed reactors, in series. The gas normally enters each reactor at about 550°F and exits at about 900°F. The temperature of gas from the first methanation stage is reduced by a cold gas quench to achieve the desired inlet temperature for the second methanation stage. The recycle compressor is oversized to guarantee that there will be sufficient gas for quench. Surplus gas is by-passed around the reactors under differential pressure control. Materials balances and streams data for three cases are presented in Table 3-8.

Figure 3-12 is the methanation section PFD.

The composition of the feed gas to the first stage and the quench is monitored by infrared analyzers and adjusted as required to control the carbon monoxide concentration.

The hot effluent from the second-stage methanator is partially cooled in the methanator feed-effluent exchanger. A by-pass on the hot side of the exchanger facilitates temperature regulation. The gas is then further cooled prior to entering a knockout drum.

It may be desirable to locate the trim cooler and knockout drum as close as possible to the recycle compressor; relocation would tend to minimize the liquid water in the gas to the compressor. To minimize condensate carryover into the compressor, the last 10 or 20 feet of inlet piping should be steam traced.

A high-pressure steam heater was specified for start-up.

Table 3-8, Part 1.a. METHANATION SYSTEM CONTROLLING CASE  
 FOR BITUMINOUS COAL 55:50 WATER-TO-HYDROGEN:  
 MATERIAL BALANCE AND STREAM PHYSICAL PROPERTIES  
 (Bechtel Preliminary Design Data)

Stream Number Revision No.	427	428	429	430	431	432	433	435	436	437	438
Stream Description	Effluent From Scrubber	Methanator Feed Gas To Vent	Hydrogen Addition	Total Feed To Methanator	Feed Gas To Quench	Feed Gas To 1st Stage	Total Gas To 1st Stage	Effluent Gas From 1st Stage	1st Stage Quench Gas	Total Gas To 2nd Stage	Effluent Gas From 2nd Stage
Temperature, °F	100	100	--	100	100	100	550	880	100	550	880
Pressure, psia	1100	1100	--	1100	1100	1100	1072	1070	1084	1070	1068
Component											
CO	43.8	32.9	NORMALLY	19.9	7.0	3.9	3.9	7.1	7.0	7.0	--
H <sub>2</sub>	145.9	109.3	NO	36.6	23.6	13.0	19.2	7.1	27.0	34.1	12.6
CH <sub>4</sub>	79.8	59.8	FLOW	20.0	12.8	7.2	74.1	76.7	49.7	128.4	136.3
H <sub>2</sub> O	--	--	--	--	--	--	--	3.9	--	3.9	10.9
C <sub>2</sub> H <sub>6</sub>	1.9	1.4	--	0.5	0.3	0.2	0.2	--	--	0.2	--
C <sub>3</sub> H <sub>8</sub>	0.7	0.5	--	0.2	0.1	0.1	0.1	--	0.1	0.1	--
N <sub>2</sub>	3.0	2.2	--	0.8	0.5	0.3	1.9	1.9	1.4	3.3	3.3
C <sub>6</sub> H <sub>6</sub> (44.70) <sup>o</sup>											
Total Moles/Hour	275.1	206.1	--	69.0	44.3	24.7	99.4	91.6	85.5	177.1	163.1
Total lbs/Hour	3023	2261	--	762	487	275	1432	1432	1124	2550	2550
Molecular Weight	11.00	11.00	--	11.00	11.00	11.00	14.41	15.63	13.15	14.41	15.66
Vapor	2.04	2.04	--	2.04	2.05	2.04	1.45	1.16	2.46	2.46	1.16
lb/Ft <sup>3</sup> at Oper. Cond.	24.7	18.5	--	6.2	4.0	2.2	16.50	20.4	7.61	23.43	38.7
AGPM at Oper. Cond.											
Liquid	--	--	--	--	--	--	--	--	--	--	--
GPM at Oper. Cond.	--	--	--	--	--	--	--	--	--	--	--
Viscosity, CP at Oper. Cond.	--	--	--	--	--	--	--	--	--	--	--
Critical Properties											
PC = Pseudo Critical Press.	388	388	--	388	388	388	571	748	509	627	656
TC = Pseudo Critical Temp.	178	178	--	178	178	178	285	359	249	303	323
Z = Compressibility	1.0	1.0	--	1.0	1.0	1.0	1.0	1.02	0.98	1.02	1.02

Note: (6) Average Molecular Weight.

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Table 3-8, Part 1. b. METHANATION SYSTEM CONTROLLING  
CASE FOR BITUMINOUS COAL 50:50 WATER-TO-HYDROGEN:  
MATERIAL BALANCE AND STREAM PHYSICAL PROPERTIES

Stream Number Revision No.	445	44E	447	44E	450	451
Stream Description	Water From Separator	Total Gas From Separator	Final Product Gas	Recycle Compressor Suction	Recycle Gas To 1st Stage	Recycle Gas To Quench
Temperature, °F	100	100	100	100	100	100
Pressure, psig	1041	1041	1041	1041	1100	1100
Components						
CO	--	--	--	--	--	--
H <sub>2</sub>	(28.01)	--	--	--	--	--
CH <sub>4</sub>	(2.02)	12.6	3.0	9.6	6.2	3.4
H <sub>2</sub> O	(16.04)	136.3	32.5	103.8	66.9	36.9
C <sub>2</sub> H <sub>6</sub>	(18.02)	--	--	--	--	--
C <sub>3</sub> H <sub>8</sub>	(30.07)	--	--	--	--	--
C <sub>4</sub> H <sub>10</sub>	(44.09)	--	--	--	--	--
N <sub>2</sub> & C <sub>2</sub> H <sub>6</sub>	--	3.3	0.8	2.5	1.6	0.9
(44.70) <sup>a</sup>	--					
Total Moles/Hour	10.9	152.2	36.3	115.9	74.7	41.2
Total lbs./hour	192	2360	564	1796	1157	639
Molecular Weight	18.02	15.51	15.51	15.51	15.51	15.51
Vapor	--	2.90	2.90	2.90	3.07	3.07
lbs./ft <sup>3</sup> at Oper. Cond.	--	13.6	3.3	10.3	6.29	3.47
ACFM at Oper. Cond.	--	--	--	--	--	--
Liquid	0.39	--	--	--	--	--
GM at Oper. Cond.	0.68	--	--	--	--	--
Viscosity, CP at Oper. Cond.	--	--	--	--	--	--
Critical Properties	--	--	--	--	--	--
Tc - Pseudo Critical Press.	--	631	631	631	631	631
Tc - Pseudo Critical Temp.	--	319	319	319	319	319
Z - Compressibility	--	0.94	0.94	0.94	0.94	0.94

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Note: (6) Average Molecular Weight.

Table 3-8, Part 2.a. METHANATION SYSTEM CONTROLLING CASE FOR BITUMINOUS COAL  
30:70 WATER-TO-HYDROGEN: MATERIAL BALANCE AND STREAM PHYSICAL PROPERTIES

Stream Number Revision No.	427	428	422	430	431	432	433	435	436	437	438
Stream Description	Effluent From Scrubber	Methanator Feed Gas To Vent	Hydrogen Addition	Total Feed To Methanator	Feed Gas To Quench	Feed Gas To 1st Stage	Total Gas To 1st Stage	Effluent Gas From 1st Stage	1st Stage Quench Gas	Total Gas To 2nd Stage	Effluent Gas From 2nd Stage
Temperature, °F	100	1100	100	100	100	100	550	872	100	550	861
Pressure, psig	1100	1100	1100	1100	1100	1100	1072	1070	1084	1070	1068
Components											
CO	53.7	29.9	--	13.8	9.0	4.8	4.8	--	9.0	9.0	--
H <sub>2</sub>	119.6	88.7	13.4	44.3	28.9	12.4	22.8	8.4	33.7	42.1	15.1
CH <sub>4</sub>	77.3	57.5	--	19.8	12.9	6.9	92.2	97.8	68.1	165.1	174.1
H <sub>2</sub> O	--	--	--	--	--	--	--	4.8	--	4.8	13.8
C <sub>2</sub> H <sub>6</sub>	0.1	0.1	--	--	--	--	--	--	--	--	--
C <sub>3</sub> H <sub>8</sub>	0.3	0.3	--	--	--	--	--	--	--	--	--
H <sub>2</sub> & C <sub>2</sub> H <sub>4</sub>	3.1	2.2	--	0.9	0.6	0.3	2.7	2.7	2.1	4.8	4.8
(42.54) °											
Total Moles/Hour	254.1	188.7	13.1	78.8	51.4	27.4	122.5	112.9	112.9	225.8	207.8
Total lbs./Hour	3134	2332	2	829	540	289	1774	1774	1500	3276	3276
Molecular Weight	13.33	12.33	2.02	10.51	10.51	10.51	14.48	15.71	13.29	14.50	15.76
lb./ft <sup>3</sup> at Oper. Cond.	2.3	2.3	0.37	1.96	1.96	1.96	1.45	1.17	2.48	1.43	1.18
lb./ft <sup>3</sup> at Oper. Cond.	22.7	16.3	1.2	7.05	4.59	2.45	23.39	25.3	10.08	36.1	46.2
Specific Gravity at Oper. Cond.	--	--	--	--	--	--	--	--	--	--	--
Viscosity, Cp at Oper. Cond.	--	--	--	--	--	--	--	--	--	--	--
Diffusivity, ft <sup>2</sup> /hr at Oper. Press.	408	408	188	370	370	370	58	763	515	636	635
Thermal Conductivity, Btu/hr-ft <sup>2</sup> -°F at Oper. Press.	187	187	60	166	165	165	293	367	291	368	368
Compressibility	1.0	1.0	1.01	1.0	1.0	1.0	1.0	1.02	0.98	1.02	1.02

Note: (°) Average Molecular Weight.

A7506 1551B

Table 3-8, Part 2. b. METHANATION SYSTEM CONTROLLING  
CASE FOR BITUMINOUS COAL 33:70 WATER-TO-HYDROGEN:  
MATERIAL BALANCE AND STREAM PHYSICAL PROPERTIES

Stream Number Revision No.	445	446	447	448	450	451
Stream Description	445	446	447	448	450	451
Temperature, of	100	100	100	100	100	100
Pressure, psig	1041	1041	1041	1041	1100	1100
Components						
CO	--	--	--	--	--	--
H <sub>2</sub>	15.1	15.1	2.9	12.2	7.4	4.8
CH <sub>4</sub>	16.04	174.1	35.6	140.5	85.3	55.2
H <sub>2</sub> O	13.8	--	--	--	--	--
C <sub>2</sub> H <sub>6</sub>	--	--	--	--	--	--
C <sub>3</sub> H <sub>8</sub>	--	--	--	--	--	--
N <sub>2</sub>	--	4.8	0.9	3.9	2.4	1.5
C <sub>2</sub> H <sub>4</sub>	--	--	--	--	--	--
C <sub>2</sub> H <sub>2</sub>	--	--	--	--	--	--
Total Moles/Hour	13.8	194.0	37.4	156.6	95.1	61.5
Total lbs/Hour	249	3027	583	2444	1485	959
Molecular Weight	18.02	15.61	15.61	15.61	15.61	15.61
Vapor lbs/Hr <sup>3</sup> at Oper. Cond.	--	2.95	2.95	2.95	3.14	3.14
Liquid lbs/Hr <sup>3</sup> at Oper. Cond.	--	17.1	3.3	13.8	7.85	5.09
Wt% at Oper. Cond.	0.50	--	--	--	--	--
Viscosity, CP at Oper. Cond.	0.88	--	--	--	--	--
Critical Properties						
T <sub>c</sub> - Pseudo Critical Press.	--	639	634	634	634	634
T <sub>c</sub> - Pseudo Critical Temp.	--	323	321	321	321	321
Z - Compressibility	--	0.93	0.93	0.93	0.92	0.92
Recycle Gas To 1st Stage						
Recycle Compressor Suction						
Recycle Gas To Quench						
Product Gas						
Total Gas From Separator						
Meter From Separator						

Note: (6) Average Molecular Weight.

A7506 15510



Table 3-8, Part 3.a. METHANATION SYSTEM CONTROLLING  
CASE FOR LIGNITE: MATERIAL BALANCE  
AND STREAM PHYSICAL PROPERTIES

Stream Number Revision No.	427	428	429	430	431	432	433	435	436	437
Stream Description										
Temperature, °F	100	100	--	100	100	100	550	903	100	550
Pressure, psig	1100	1100	--	1100	1100	1100	1072	1070	1084	1070
Components										
CO	25.2	18.7	NORMALLY	6.5	4.2	3.3	2.3	--	4.2	4.2
H <sub>2</sub>	92.6	89.9	NO	23.7	15.3	8.4	11.9	4.5	17.4	21.9
CH <sub>4</sub>	76.4	58.9	FROM	19.5	12.2	6.9	42.0	45.2	33.2	78.4
H <sub>2</sub> O	--	--	--	--	--	--	--	2.3	--	2.3
CH <sub>3</sub> OH	1.7	2.9	--	0.8	0.5	0.3	0.3	--	0.5	0.5
Gas	1.2	0.9	--	0.3	0.2	0.1	0.1	--	0.2	0.2
Slurry										
Me A										
Me B										
Calc	2.5	1.9	--	0.6	0.4	0.2	0.9	0.9	0.8	1.7
DESIGN FOR										
Total Moles/Hour	202.6	151.2	--	51.4	33.2	18.2	57.5	52.9	56.3	109.2
Total Lbs/Hour	2995	1789	--	606	392	214	815	815	744	1559
Molecular Weight	11.80	11.80	--	11.80	11.80	11.80	14.17	15.40	13.22	14.28
Vapor	2.20	2.20	--	2.20	2.20	2.20	1.42	1.12	2.44	1.40
Lbs/Hr <sup>3</sup> at Oper. Cond.	18.16	13.57	--	4.59	2.97	1.62	9.56	12.1	5.08	18.6
ACW at Oper. Cond.										
Liquid										
GM at Oper. Cond.										
Viscosity, CP at Oper. Cond.										
Critical Properties										
Pc - Pseudo Critical Press.	426	426	--	426	426	426	547	722	497	606
Tc - Pseudo Critical Temp.	204	204	--	204	204	204	271	343	285	292
Z - Compressibility	1.0	1.0	--	1.0	1.0	1.0	1.0	1.02	0.99	1.02

Note: (5) Average Molecular Weight.

A7506 1551D

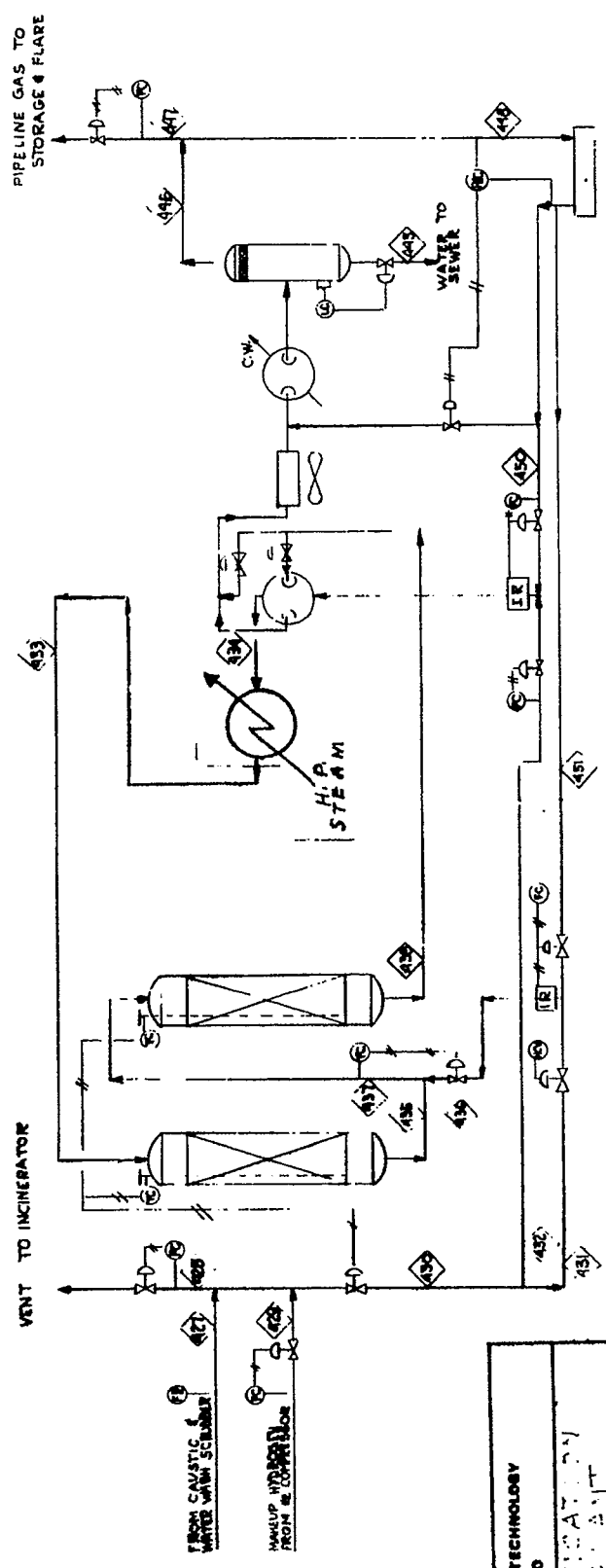
Table 3-8, Part 3. b. METHANATION SYSTEM CONTROLLING CASE FOR LIGNITE: MATERIAL BALANCE AND STREAM PHYSICAL PROPERTIES

Stream Number Revision No.	438	445	446	447	448	450	451
Stream Description	Effluent Gas From 2nd Stage	Water From Separator	Total Gas From Separator	Product Gas	Recycle Compressor Suction	Recycle Gas To 1st Stage	Recycle Gas To 'Jench
Temperature, °F	823	100	100	100	100	100	100
Pressure, psig	1068	1041	1041	1041	1041	1100	1100
Components							
CO	--	--	--	--	--	--	--
H <sub>2</sub>	8.4	--	8.4	2.8	5.6	3.5	--
CH <sub>4</sub>	84.2	--	84.2	28.5	55.7	35.1	2.1
H <sub>2</sub> O	6.5	6.5	--	--	--	--	20.6
C <sub>2</sub> H <sub>6</sub>	--	--	--	--	--	--	--
C <sub>3</sub> H <sub>8</sub>	--	--	--	--	--	--	--
N <sub>2</sub> & C <sub>4</sub> H <sub>10</sub>	1.7	--	1.7	0.6	1.1	0.7	0.4
Total Moles/Hour	100.8	6.5	94.3	31.9	62.4	39.3	23.1
Total Lbs/Hour	1559	117	1442	489	953	601	352
Molecular Weight	15.47	18.02	15.29	15.29	15.29	15.29	15.29
Vapor							
Lbs/ft <sup>3</sup> at Oper. Cond.	1.13	--	2.86	2.86	2.86	3.02	3.02
ACFM at Oper. Cond.	23.0	--	8.40	2.85	5.55	3.32	1.94
Liquid							
GPM at Oper. Cond.	--	0.23	--	--	--	--	--
Viscosity, CP at Oper. Cond.	--	0.68	--	--	--	--	--
Critical Properties							
Pc - Pseudo Critical Press.	773	--	603	603	603	603	603
Tc - Pseudo Critical Temp.	363	--	303	303	303	303	303
Z - Compressibility	1.02	--	0.94	0.94	0.94	0.94	0.94

Note: (6) Average Molecular Weight.

A7506 1551E

- 5-C-1 FIRST STAGE METHANATOR
- 5-C-2 SECOND STAGE METHANATOR
- 5-F-1 METHANATOR GAS FIBED STARTUP HEATER
- 5-E-3 METHANATOR EFFLUENT AIR COOLER
- 5-C-5 METHANATOR KNOCK OUT DRUM
- 5-E-2 METHANATOR FEED EFFLUENT EXCHANGER
- 5-E-4 METHANATOR EFFLUENT COOLER
- 5-K-1 METHANATOR RECYCLE COMPRESSOR



A7506 1416


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COAL GASIFICATION PILOT PLANT	
PROCESS FLOW DIAGRAM METHANATION SECTION	
PROJECT NO.	DRAWING NO.
	PFD-4
	
SCALE	REV.

Figure 3-12. PRELIMINARY DESIGN PFD FOR METHANATION SECTION OF HYGAS PILOT PLANT

The temperature of each quench stream to the reactors is controlled. A high temperature shutdown station shown on the total feed is used only to protect the unit against high temperature runaway. Under normal operations, the shutdown station will remain 100 percent open and will not close as long as the compressor and analyzers are working properly. The knock-out drum includes a high-level device that will shut down the compressor if there is a danger of water carryover, and will also stop the flow of feed gas to the unit.

#### 3.4.4.2. Methanators (5-C-1 and 5-C-2)

The two fixed-bed catalytic methanation reactor vessels have the same diameter, but different tangent-to-tangent lengths. Dimensions are 24-inches I. D. ; one unit is 9 feet 8 inches long and the other is 14 feet 8 inches long. A deflector plate on the inlet nozzle prevents direct impingement of incoming gas on the catalyst bed. A 6-inch layer of alumina balls is included on the top of each catalyst bed to hold the catalyst down and further distribute the gas over the total catalyst bed. The catalyst is supported by graded alumina balls of gradual increasing size. The gas exits via the bottom nozzle; an internal slotted pipe sleeve covered by a mesh screen keeps catalyst and fines out of the lines. A handhole has been provided near the top and bottom of each vessel to insert and remove catalyst.

#### 3.4.4.3. Methanator Feed-Effluent Exchanger (5-E-2)

During normal operation, the methanator feed-effluent exchanger conceived would heat to 550°F. The exchanger was to be designed with a by-pass line on the hot side. Flow through the exchanger would vary widely from operation to operation. Operating characteristics of the exchanger installed are tabulated below.

	<u>Shell Side</u>	<u>Tube Side</u>
Fluid Circulated	Methanator Feed	Methanator Effluent
Total Fluid Entering (Vapor)	1774 lb/hr	3276 lb/hr
Viscosity - Gas CP (in/out)	0.0144/0.0133	0.0260/0.0224
M. W. (Vapors)	14.48	15.76
Temperature in	100°F	880°F
Temperature out	550°F	630°F
Operating Pressures (Inlet - max)	1082 psig	1068 psig
Pressure Drop (Allowable)	5 psi	5 psi
Fouling Factor	0.001	0.001
Heat Exchanged	600,000 Btu/hr	

#### 3.4.4.4. Methanator Effluent Air Cooler (5-E-3)

The methanator effluent air cooler removes the bulk of the heat from the system, and it is followed by a trim cooler. This unit is designed for

6.4 million Btu per hour heat removal with an inlet temperature of 900°F and an outlet temperature of 150°F. Under normal operating conditions, the inlet temperature would be around 650°F, which is 250°F below the intended design maximum. During upset conditions, however, this air cooler would provide the only means to remove the bulk of the heat from the system. Specifications are presented below.

Tube Side Data

Total Fluid Entering	9954 lb/hr
(Vapor - 8981)	
(Steam - 973)	
Steam Condensed	937
Fouling Factor	0.001
Temperature in	900°F
Temperature out	150°F
Operating Pressure	1068 psig
Gravity (Liquid)	1.0
Viscosity in	0.0224
Viscosity out	0.0136
Allowed Pressure Drop	5 psi

Air Side Data

Temperature in	90°F
Temperature out	180°F
Air Quantity	297,000 lb/hr
Air Quantity/Fan	34,600 ACFM
Static Pressure	0.625

3.4.4.5. Methanator Start-up Heater (5-F-1)

This heater is designed for use only during start-up. In the normal bootstrapping start-up, the load on this unit would gradually decrease from the specified maximum to zero. Operating characteristics of the heater installed after final design are presented below.

	<u>Tube Side</u>	<u>Shell Side</u>
Fluid	Steam	Methanator Feed
Total Fluid Entering	1100 lb/hr	1774 lb/hr
Steam Condensed	1100 lb/hr	
Viscosity Gas		0.0144-0.0133
M.W. - Vapors		14.48
Temperature in	570°F	100-550°F
Temperature out	570°F	550°F
Operating Pressure		
(Inlet - max)	1200 psig	1077 psig
Pressure Drop		
(Allowable)	10 psi	5 psi
Fouling Factor	0.0005	0.001
Heat Exchanged	600,000 Btu/hr	

#### 3.4.4.6. Methanator Recycle Compressor (5-K-1)

This compressor is designed to handle 55 ACFM at suction conditions. The actual material balance suction rate is less than the rating. The 40 ACFM of extra capacity has been added to permit increase of throughput, and to provide a reserve for quench cooling in an emergency. Operating characteristics of the compressor installed are:

Gas Handled	Product Gas
M. W.	15.80
CP/CV	1.29
MMCFD at 14.7 psia, 60°F	5.184
Suction Temperature	100°F
Suction Pressure	1027 psia
Discharge	1132 psia
Compression Ratio	1.1
H. P.	40.7
Design Pressure	1700 psig
Test	3400 psig

#### 3.4.5. General

Additional process flow diagrams presented here include Figure 3-13, for the waste and blowdown section; Figure 3-14, the steam material balance; and Figure 3-15, the fuel gas material balance for initial operations.

Figure 3-16 is the plot plan of the HYGAS pilot plant, and Figure 3-17 is the site plan of the HYGAS pilot plant. Figures 3-18 through 3-22 are photographs of the early plant from various views.

The balance of this section is devoted to a discussion of the start-up operations following completion of the initial HYGAS pilot plant.

### 3.5 Start-up and Initial Test of the HYGAS Pilot Plant

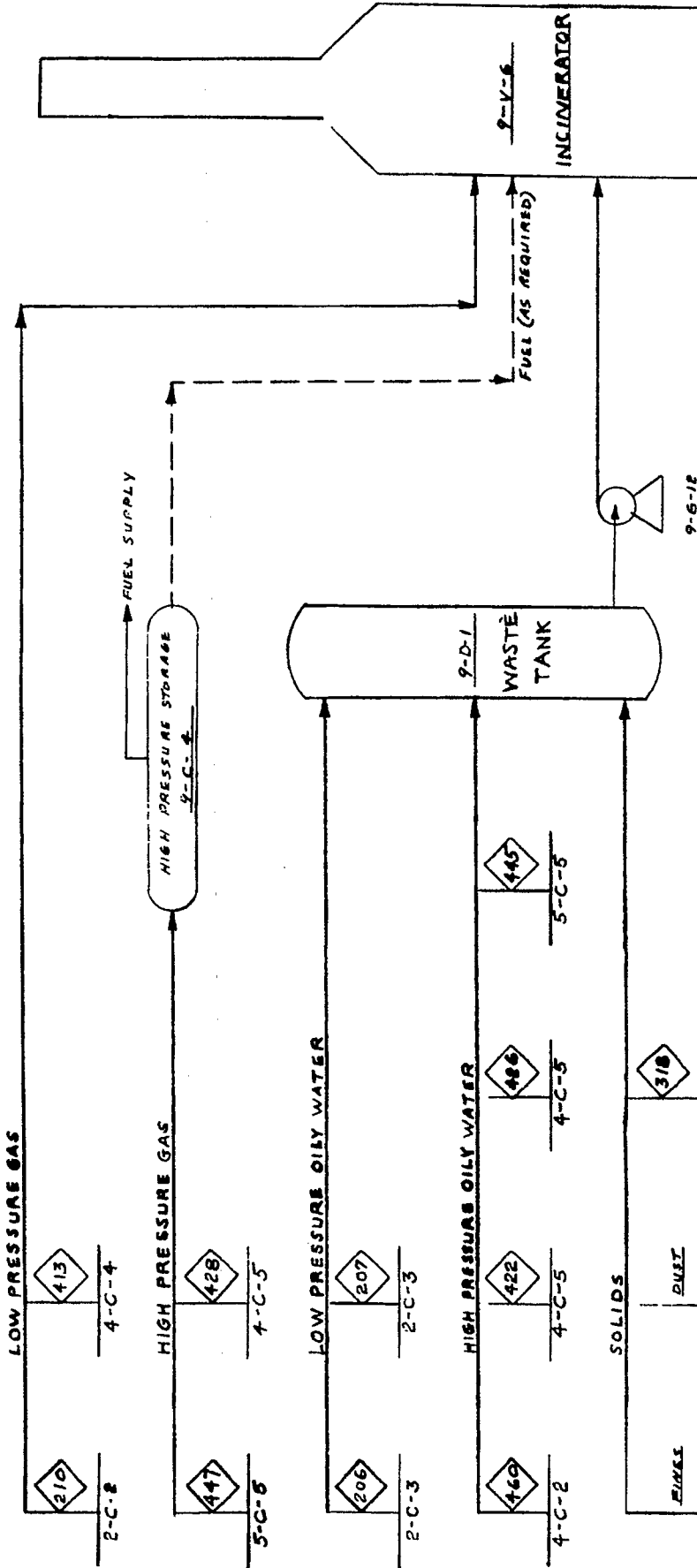
#### 3.5.1 Introduction

Beginning August 1970, when the HYGAS pilot plant was in its final stages of construction, engineers and technicians were put on full-time training. A number of equipment items were shaken down and commissioned. By December 1970, Procon Inc. closed out its field office and left the site. The remaining work consisted of insulation, instrument piping, mostly in the reactor structure, and some electrical work.

Morrison Construction Company was brought in to complete the remaining work and to make changes, corrections, and improvements in the plant as shakedown proceeded. During the first winter in 1970-71, most of the time was devoted to repairing steam-tracing problems. Construction of the plant was considered complete in May 1971.

#### 3.5.2 Equipment Commissioning

The plant air and instrument air compressors, the low-pressure boiler, and the water demineralizer system were commissioned in October 1970. In



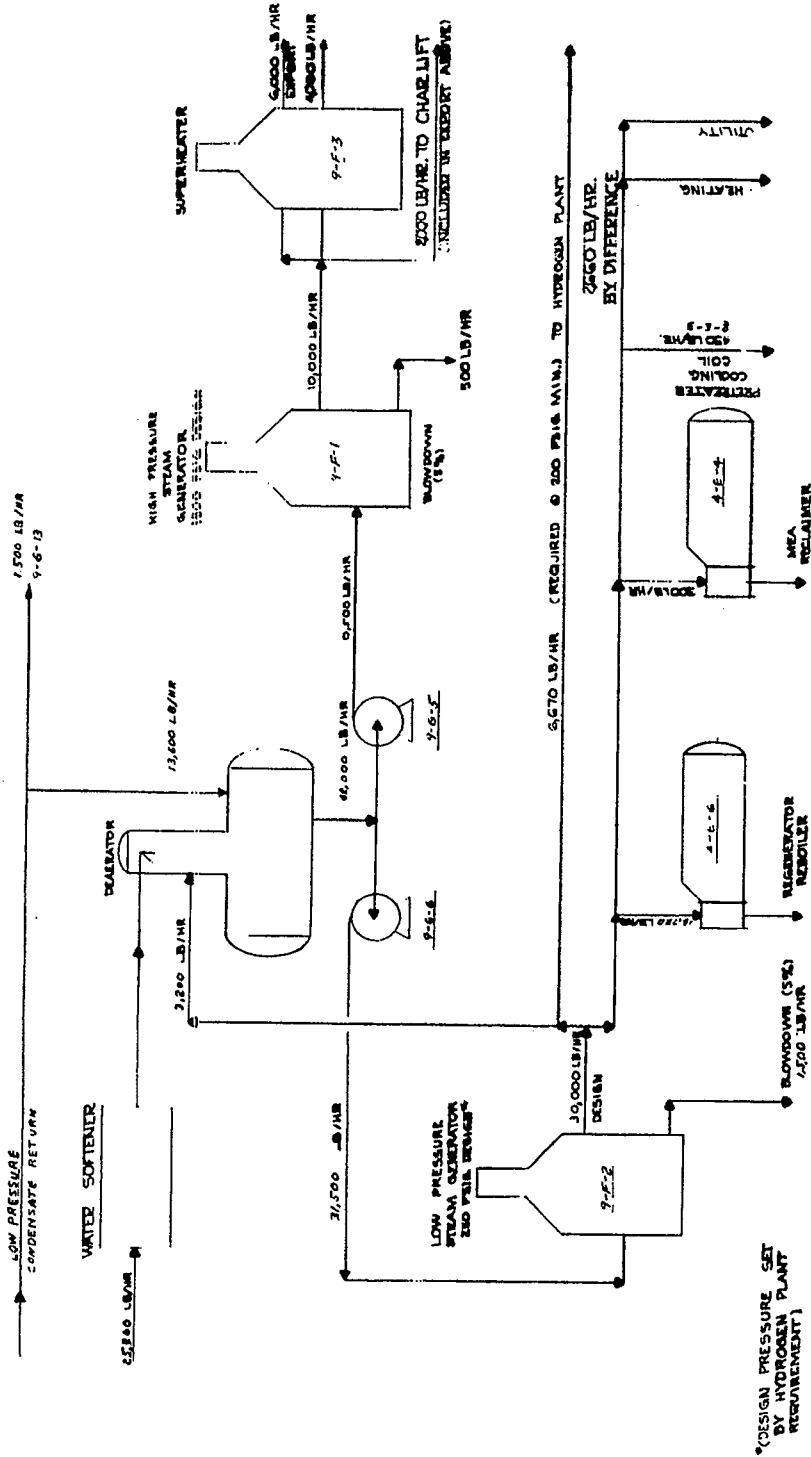
WASTE PUMP

PROJECT NO.		DRAWING NO.		REV
		PFD-5		
SCALE				

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COAL HYDRGASIFICATION

A7506 1417

Figure 3-13. PRELIMINARY DESIGN PFD FOR WASTE AND BLOWDOWN SECTION OF HYGAS PILOT PLANT



A7506 1418

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COAL HYDROGASIFICATION PILOT PLANT	
STEAM MATERIAL BALANCE	
PROJECT NO.	DESIGN NO.
SCALE	REV
	PFD-6
	0

Figure 3-14. PRELIMINARY DESIGN PFD FOR STEAM MATERIAL BALANCE FOR HYGAS PILOT PLANT



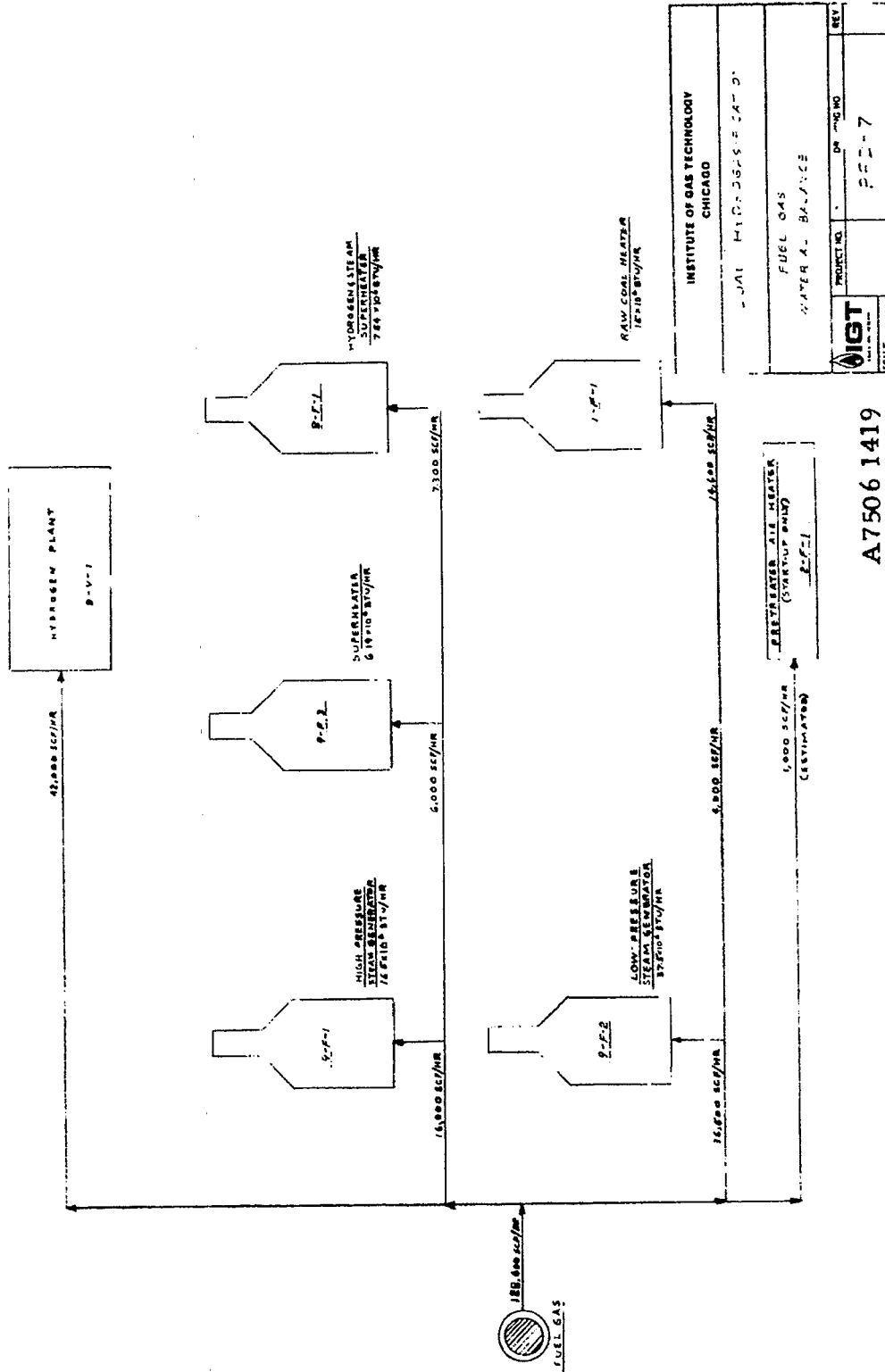
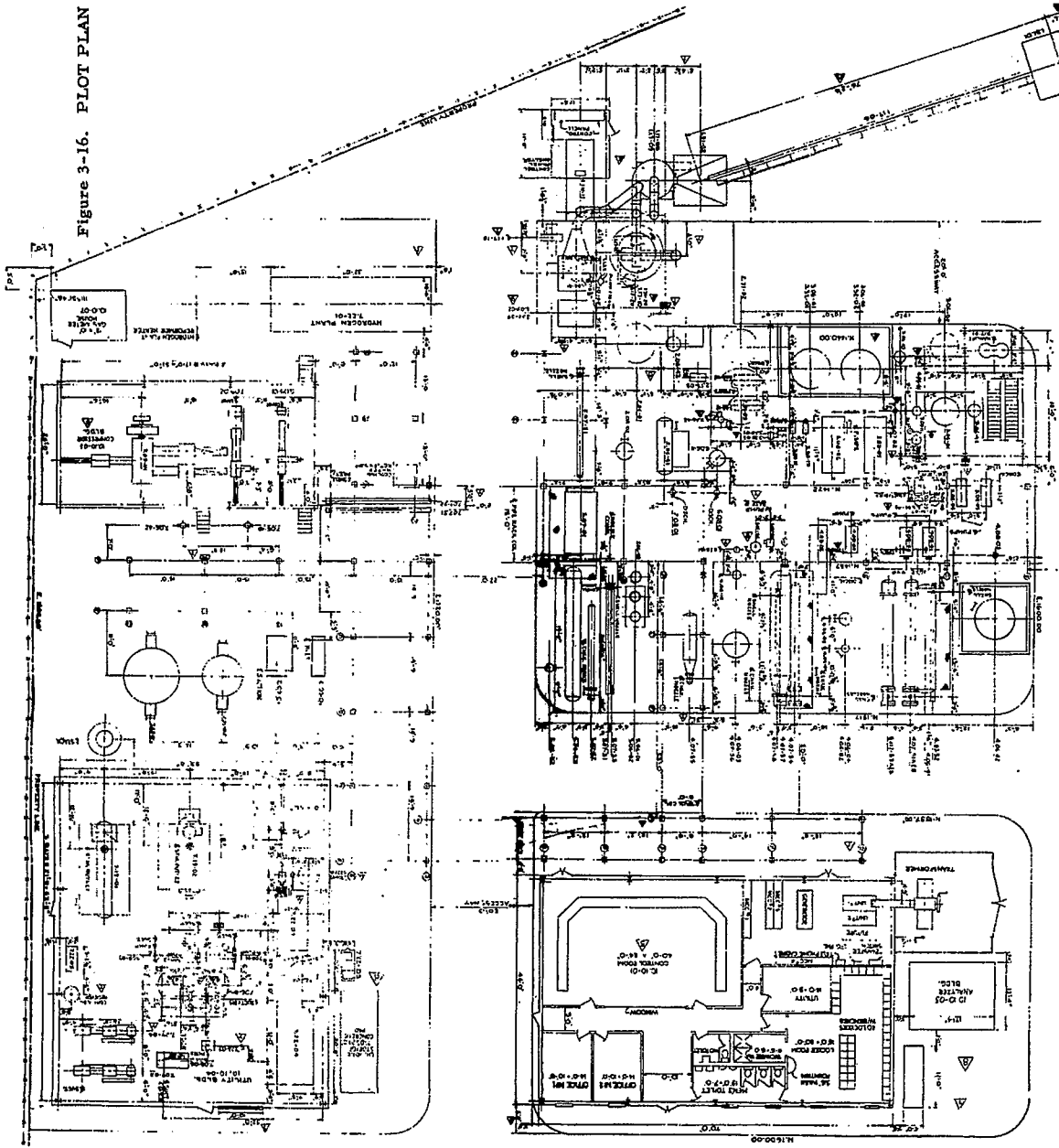


Figure 3-15. PRELIMINARY DESIGN PFD FOR FUEL GAS MATERIAL BALANCE OF THE HYGAS PILOT PLANT

Figure 3-16. PLOT PLAN OF THE HYGAS PILOT PLANT



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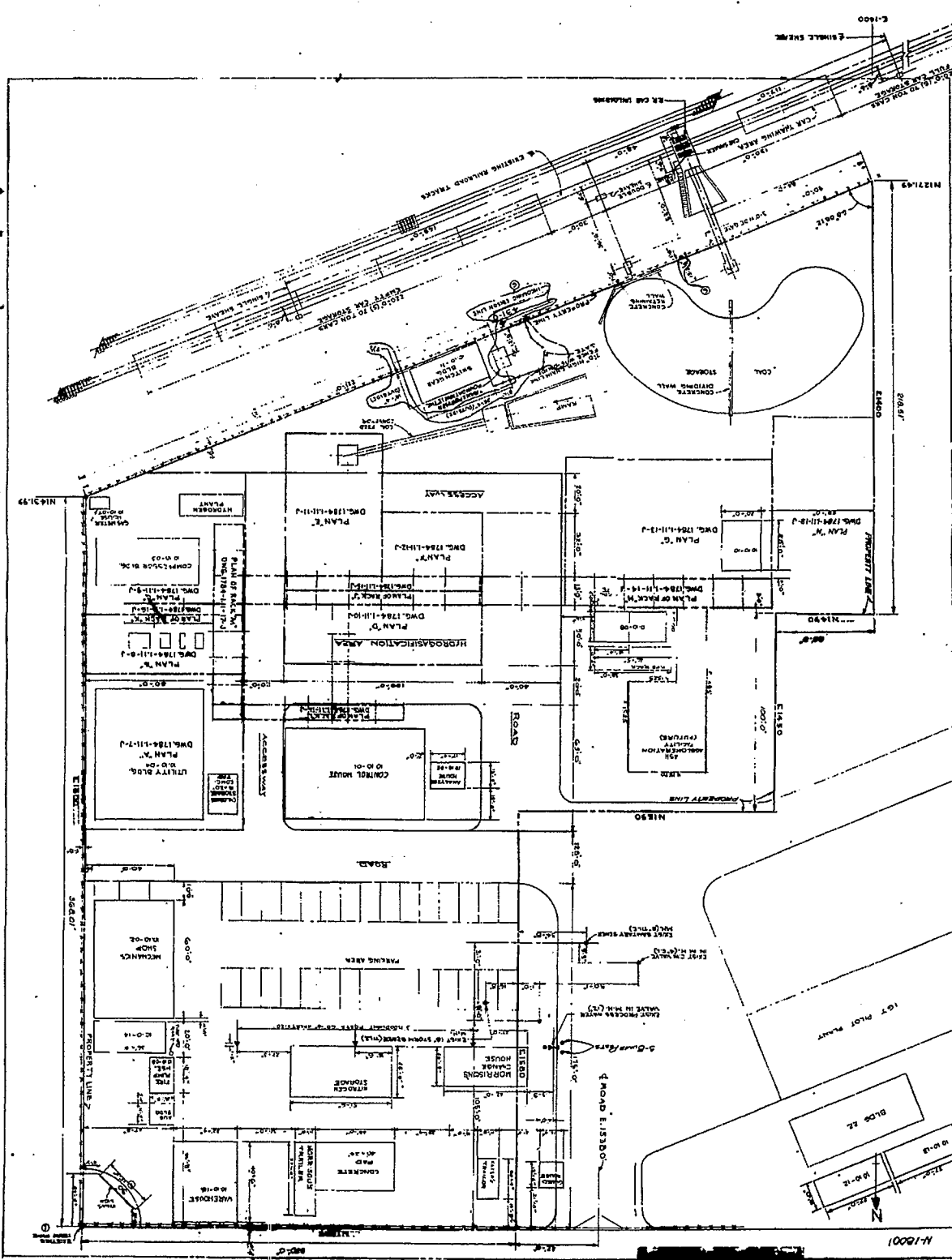


Figure 3-17. SITE PLAN OF THE HYCAS PILOT PLANT

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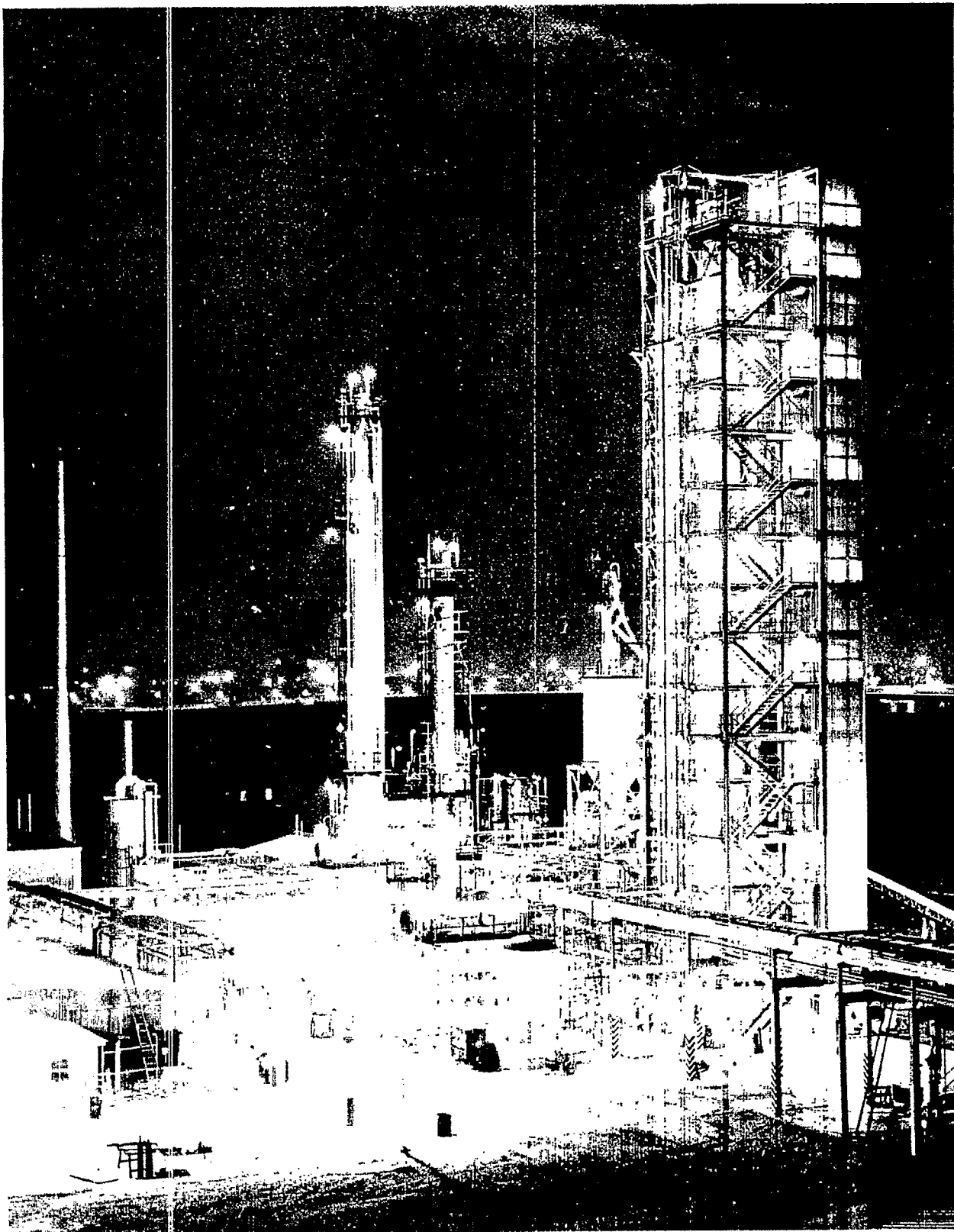


Figure 3-18. THE HYGAS PILOT PLANT: BARREL-SHAPED STRUCTURE AT LEFT NEAR TALL STACKS IS THE STEAM AND HYDROGEN PREHEATER SECTION. THE FOUR TALL STRUCTURES STARTING AT CENTER, AND READING FROM LEFT TO RIGHT ARE THE PURIFICATION SYSTEMS REGENERATOR, ACID GAS ABSORBER, PRETREATED CHAR STORAGE TANKS, AND THE HYDROGASIFICATION REACTOR

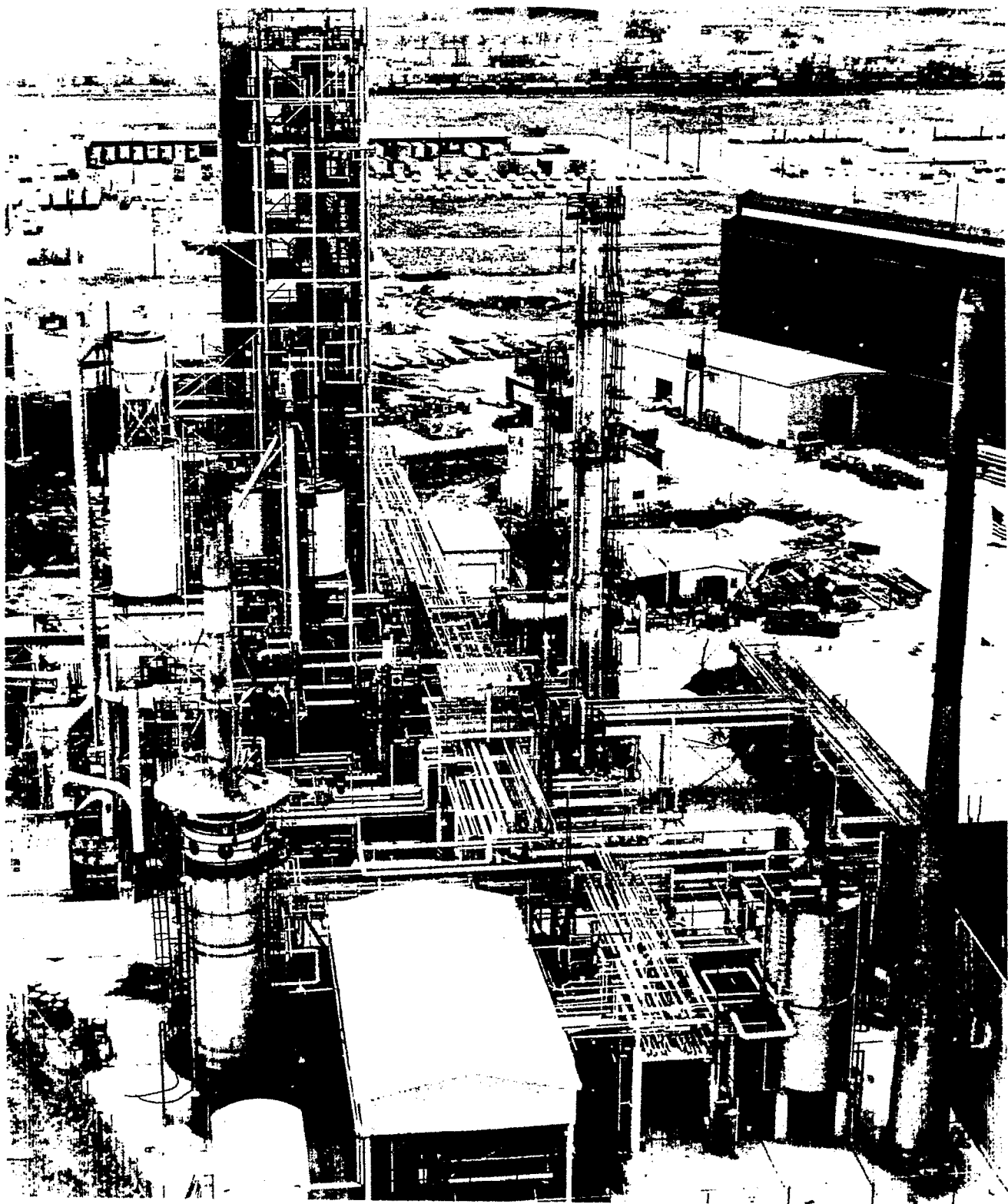


Figure 3-19. THE HYGAS PILOT PLANT: 1972

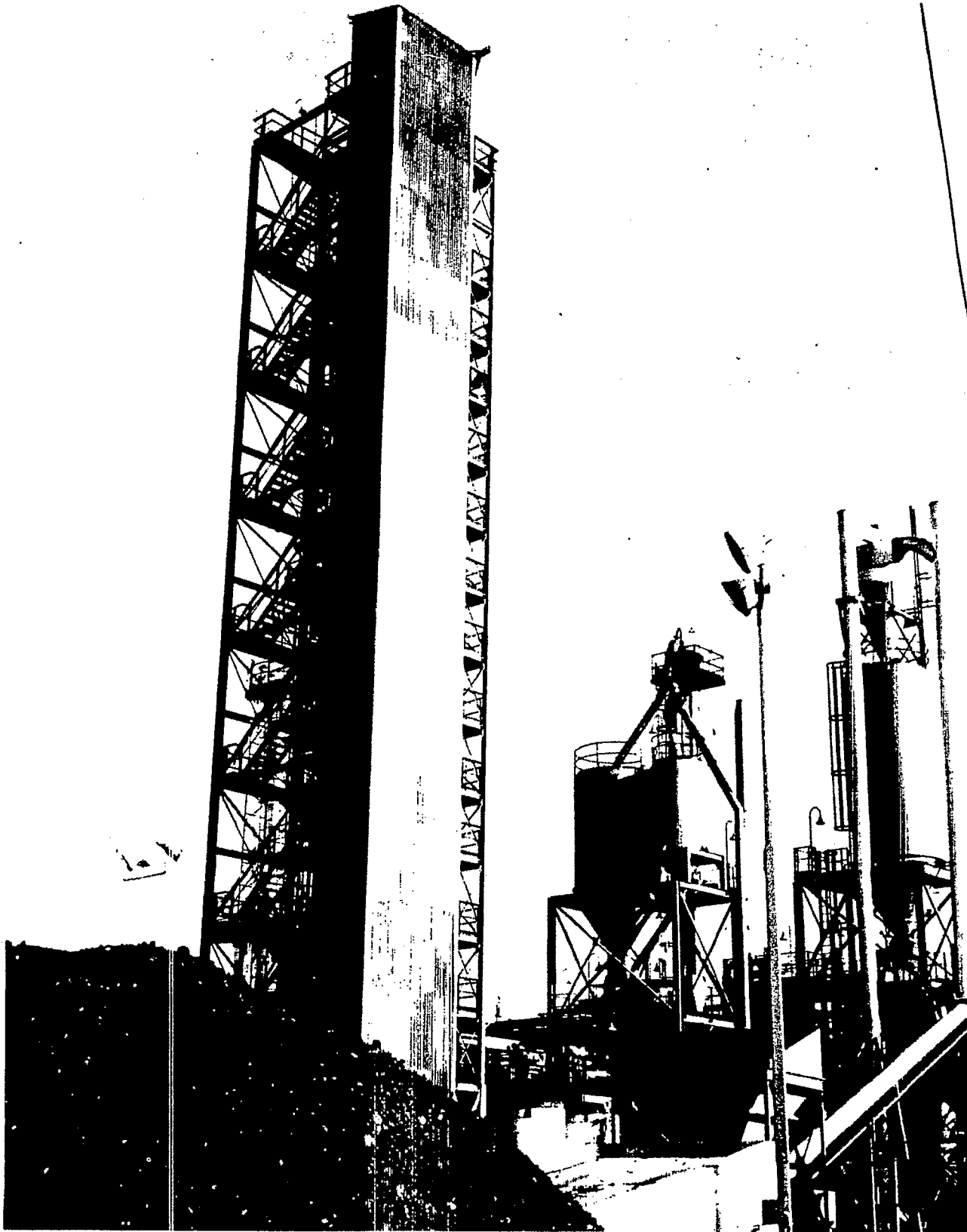


Figure 3-20. THE HYGAS PILOT PLANT: HYDROGASIFICATION UNIT  
SHOWING ELEVATOR ENCLOSURE IS AT LEFT BEHIND PILE OF  
FEED. TALL STRUCTURE AT RIGHT INCLUDES A CYCLONE  
ABOVE THE RAW CRUSHED-COAL HOLD TANK. TALL OB-  
JECT IN CENTER INCLUDES BUCKET ELEVATOR TO TOP  
OF PRETREATED COAL/CHAR HOLD TANK. P7506 1360

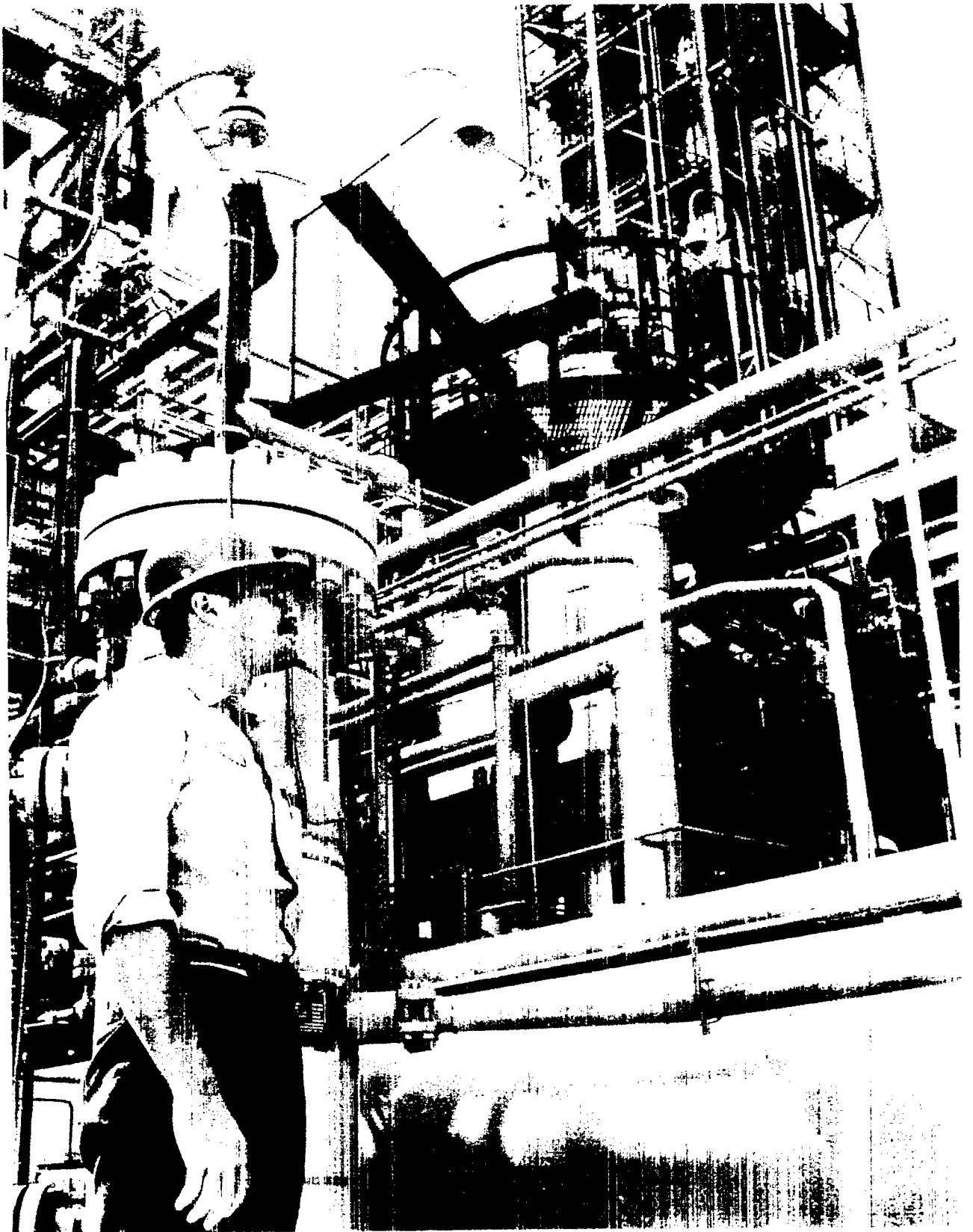


Figure 3-21. THE HYGAS PILOT PLANT: METHANATION SECTION VIEW INCLUDING METHANATION WATER KNOCKOUT DRUM (BEHIND STANDING FIGURE), METHANATION TOWERS (TO RIGHT OF KNOCKOUT DRUM), AND HORIZONTAL TANK FOR STORAGE OF PIPELINE-QUALITY LNG.



Figure 3-22. HYGAS PILOT PLANT: TALLEST TOWER IS THE ABSORBENT REGENERATOR UNIT. AT RIGHT ARE CARBON DIOXIDE/HYDROGEN SULFIDE SCRUBBING TOWER, AND RE-BOLLER TO REMOVE GASES FROM ABSORBENT.



November 1970, the low-pressure boiler was put on-stream around the clock to supply tracing steam to the plant. The coal receiving and unloading facilities were commissioned in December, with the receipt of 700 tons of Montana lignite and 700 tons of Illinois No. 6 bituminous coal. In January 1971, the package hydrogen plant, based on steam reforming of natural gas, was commissioned. After 2 weeks of debugging, the plant underwent a successful 48-hour performance test at full capacity and design conditions, producing 2 million standard cubic feet per day of 95% hydrogen product gas.

In February, the hydrogasifier reactor water jacket was commissioned. This allowed a slow drying of the refractory lining and also kept the shell warm during the winter. The pretreater refractory was dried and cured. The initial charge of diglycolamine (DGA) was charged to the gas purification section. In March, the three gas compressors in the plant were put in service and each showed some problems. The natural gas booster compressor had a vibration problem which was solved when an aluminum piston was installed. The methanation recycle compressor had to have its piston rods polished because of mild rusting. The hydrogen compressor showed severe rusting on the piston rods, and this condition was repaired.

The methanation catalyst was charged in April, 1971. The high-pressure boiler and superheaters were commissioned in May, 1971.

### 3.5.3 Early Coal Preparation Tests

The pretreatment section was tested in April 1971. Because of tar-acid corrosion in the pretreater quench section, ammonia was injected to control the pH of the quench liquor. In May, the pretreatment section was operated for 36 hours. The main operating problem was excessive dust carryover which plugged the quench tower. In June, the packing was removed from this quench tower, and the unit converted to a venturi scrubber. The original feeder to the pretreater was not satisfactory in preventing hot gases leaking from the pretreater back to the upstream weighbelt; it was later replaced with a Gemco airlock valve system.

In the coal mill, different sets of dust collector bags were tried to reduce pressure drop across the bags from dust collecting on the bags. In July 1971, the coal mill was adapted to dry and crush lignite. A set of idler pulleys was installed on the motor to reduce the mill speed. Because of the high moisture content in lignite, the temperature of the hot flue gas from the furnace to the mill had to be increased. This, combined with the extremely reactive nature of lignite — particularly in the form of fine particles — caused the dust collector bags to catch fire easily. The mill shutdown logic was modified to stop all air flow in the mill system when the furnace went down. The original carbon steel hot duct from the furnace to the mill was changed to stainless steel.

### 3.5.4 Hydrogasifier Heat-up

A natural gas-air burner was used to cure the refractory in the hydrogasifier and to bring the temperature up to 1600°F. Before start-up, a bed of sand was charged to the second-stage gasifier and to the slurry dryer. Several kinds of coarse material, such as alumina spheres and grit, were used to prevent weeping of the fine sand through the holes in the grid during start-up. Once the reactor was heated up, natural gas was

cut off, and the system was purged with high-pressure nitrogen several times by pressurization and depressurization until the oxygen content was below 1 percent. Hydrogen was then introduced, and if the temperature in the second-stage sand bed was above 1300°F at this time, high-pressure air was introduced to burn with hydrogen on contact in the fluidized sand bed. Combustion under reducing conditions continued until the temperature became high enough to begin the flow of light oil followed by slurry.

The refractory curing of the hydrogasifier was completed in July 1971. The first heat-up of the reactor occurred in August. After two attempts, operators were able to successfully switch from an oxidizing condition to a reducing condition and then initiate air-hydrogen combustion in the second stage at high pressure. During the heat-up in September, however, cracks were detected in the 3-inch diameter coal-feed lines from the slurry dryer bed to the first-stage mixing pot. Dissimilar metals and poor weld penetration combined to cause this. The defective pipe was made of Type 446 stainless steel, a material chosen for its high sulfur resistance, but which

tended to become embrittled in hydrogen service. A number of skin thermocouples, which had been improperly installed, were relocated. Four additional aeration taps were installed on the various transfer lines to improve solids movement. During the initial heat-up tests, the quench water circulation pumps, immediately after the hydrogasifier, were found to be badly corroded by the dissolved carbon dioxide in the circulating water. This acidic condition would disappear once natural gas was cut off and combustion between air and hydrogen commenced. Nevertheless, ammonia injection was used to control the pH of the water during the heat-up period.

Before getting ready for the first gasification test, the six centrifugal pumps involved in slurry or dirty water service were converted to double-mechanical seals. Clean seal flush fluid was provided to each pump. The original design of using fluid from the discharge end of the pump through a hydroclone to serve as seal flush was found to be inadequate. At the same time, three vertical pumps handling dirty water service were converted from mechanical seals to packing.

#### 3.5.5 Gasification Test No. 1, October 1971

In October, a used high-pressure air compressor was obtained and installed to provide additional air to bring the second-stage hydrogasifier to the necessary temperature prior to starting the coal feed. With this additional air input, the reactor temperature could be raised to 1700°F.

The first coal to be tested in the HYGAS plant was a Montana lignite from the Savage mine. The typical analysis is given in Table 3-9. Lignite, being a noncaking coal, does not require a pretreatment step. After the lignite tests are completed, HYGAS engineers plan to operate next with pretreated bituminous coal.

Table 3-9. THE MONTANA LIGNITE ANALYSES

Proximate Analysis (Net)

% Moisture	9.32
% Ash	34.44
% Volatile	42.96
% Fixed Carbon	12.44
% Sulfur	0.84

Ultimate Analysis

% Carbon	62.58
% Hydrogen	4.00
% Nitrogen	1.02
% Chlorine	0.06
% Sulfur	0.94
% Ash	13.85
% Oxygen (by difference)	17.55

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Heating Values, Btu/lb 9200

The first batch of lignite-toluene slurry was prepared on October 13. The slurry circulation could not be maintained and the flow stopped, resulting in solids plugging up the lines in the circulation loop. The circulation loss was traced to the following combination of factors:

- 1) The coal particle size was coarser than originally planned because of the difficulty in screening lignite,
- 2) The suction head of the centrifugal circulating pumps was limited.
- 3) The mixing system in the slurry preparation tank required modification.

A size-8 mesh screen was installed in the lignite feed section which had maximum supplemental vibration to get the lignite through the smaller screen. An extension to the shaft of the slurry agitator was machined and installed, because the original shaft was found to be 16 inches shorter than the supplier's drawings indicated. This extension was designed to eliminate any dead pockets at the bottom of the slurry-mixing tank. A nitrogen sparger ring was installed at the bottom of the tank to provide supplemental mixing if required. A choke used commonly in oil field drilling was installed on the discharge side of the slurry circulating pumps to create sufficient back-pressure on the centrifugal pumps to avoid pump cavitation and to control flow rate. The high-pressure mud pumps were then activated, and pumped toluene into the hydrogasifier. The slurry preheat exchanger and the quench system, with its oil-water separator, and their associated controls, were then activated. Operation was smooth after a faulty instrumentation element was corrected.

The feeding of coal to the hydrogasifier was first tested on October 16, 1971. More than 8 tons of coal was fed to the slurry system during this 5-hour test; approximately 4 tons of this coal went into the hydrogasifier. About 2000 cubic feet of methane was made in the test. The reactor pressure was at 600 psig. The slurry system performed well, pumping 44% by weight of lignite slurry without any difficulties. The test was stopped when solids could not be discharged from the slurry dryer bed.

The results of this test were analyzed and it was concluded that the heat loss in the nitrogen jacket section of the reactor was much higher than anticipated. With the dense gas at high pressure, free convection became the dominant heat transfer mechanism. Also noticed was an increasing pressure drop across the quench tower, indicating the possibility of coal dust collecting in the 1-inch Pall ring packing. This situation is similar to that encountered in the pretreatment quench tower.

When the reactor was brought up in pressure, the hydrogen compressor was not able to deliver the full design output. This problem was traced to leaky discharge valves on the first stage of the compressor, as evidenced by the discharge temperature being higher than design. The discharge pressure from the first stage dropped off gradually until the compressor was shut off for 8 hours; all the valves were replaced with a spare set. The gasifier was then restarted and heating was resumed with high-pressure air. The problem was partially solved; a 1200-psi discharge pressure was obtained, compared with the earlier 1000-psi. However, the compressor still did not achieve the design flow. Because of the compressor problem, the first gasification test was run at 600 psig. After the test, the hydrogen compressor was shut down and disassembled; piston rod seals were worn and valve channels were broken.

### 3.5.6 Gasification Test No. 2, December 1971

Before this test, contractors completely revamped the insulation in the nitrogen jacket portion of the gasifier. The original insulation was not adequate to limit the heat losses caused by free convection under the high-pressure conditions. All of the pipes and vessels inside the nitrogen jacket were insulated, first with a 1/2-inch layer of high-temperature Kaowool blanket, followed by two staggered layers of 1.5-inch pipe insulation, another 0.5 inch of blanket, and then with a complete stainless steel covering.

Calculations showed that the gasifier off-gas quench tower, filled with 1-inch pall ring packing, had begun to plug because of the coal fines in the gas. It was then converted from a packed tower to one with side-to-side baffle trays. The slurry quench drum was also modified from packing to baffles.

In the hydrogen compressor, the valve seats on the second stage were replaced. Ingersoll-Rand informed engineers that this compressor, designed for high-pressure hydrogen service, could not be used with nitrogen. Originally, the use of nitrogen for initial idling had been suggested by a contractor's service engineer. In accordance with the supplier's new recommendation, the operating procedure and piping were modified. From that time on, the hydrogen compressor would handle only hydrogen from the hydrogen plant.

After running the hydrogen compressor for 72 hours, the unit was shut down for a check. Everything appeared to be in excellent condition, with no noticeable wear on the valves. All the tolerances on the new rings on the piston rods held within designated limits. There was no sign of any overheating on the discharge of the three stages, and the pressure ratio on each stage was within design limits. A suction strainer was added to the compressor's first stage to provide further protection.

Additional testing on the coal mill showed that greater drying could be achieved by adjusting the damper on the primary fan discharge. Additional insulation on the ductwork to conserve heat, and modified operating procedures permitted the achievement of sustained crushing runs without interruption, and with little pressure-drop buildup in the dust collector. After installing a 4-mesh screen on the discharge of the 60-ton storage hopper, a much less oversized recycle was achieved, and plugging of the bag was stopped. Anticipating winter operation, the 60-ton storage bin was steam traced and then insulated to prevent the condensation problem that had given some difficulty the first winter of operation.

The hydrogasifier was heated up and successfully switched to hydrogen in preparation to feeding coal. However, a number of Grayloc closures, which should have been exposed but were completely insulated by Procon, leaked hydrogen, and the heat-up operation had to be discontinued in order to repair them. During the heat-up, the improved insulation on the internals of the upper half of the main reactor caused the temperatures on the lift lines and internal vessels to rise dramatically, to be very close to the design values. Because of the improved insulation, the temperature in the nitrogen jacket decreased from more than 300° F to less than 100° F.

After the Grayloc closures had been repaired preparations were made to heat up the reactor; however, the natural gas burner could not be lighted and the reactor had to be opened. When the inside of the reactor was checked, inspectors found that, during the previous heat-up, when a partially plugged control valve opened up, a surge in the hydrogen flow caused a pressure surge which partially lifted the refractory grid. Although the bricks on this arch grid were displaced, the grid was still serviceable and able to support the sand bed above.

The contractor who originally installed the refractory was brought in to try to realign and reposition the bricks. The bricks were tightly wedged against each other, however, and could not be loosened sufficiently to put them back in their correct positions. Forcing a correct alignment would probably have cracked some of the bricks. A certain number of the 12-inch-thick bricks already had 2 to 3 inches cracked off. In consultation with the refractory installer, it was decided to close up the reactor and use the grid as it was for the next test.

The hydrogasifier reactor was heated up and successfully transferred over to operation with hydrogen. The procedure was followed with a smooth introduction of toluene into the system.

On December 8, 1971, coal was introduced to the gasifier for the second test. Toluene and a toluene-lignite slurry were pumped in for more than

9 hours. During this time, the operation of the slurry dryer was checked and found to be satisfactory. Some difficulty was encountered in initiating solids flow in the transfer line, which had been standing stagnant for several days; however, by using the aeration taps engineers were able to clear this line and start the solids flow. Once the solids flow started, the control was steady. Lignite was successfully introduced into the first-stage lift reactor for the first time. The operation was good, and observers measured the bed level with the radiation gage and by the pressure differential measurements. Some problem was encountered in discharging solids from the top of the lift line to the second stage because the solids in the line, charged before the test, were stagnant for several days. The line was cleared with aeration and established good controlled flow of solids into the second stage. The temperature in the lift line was maintained at 1200° to 1300°F, as planned.

All three differential-pressure transmitters indicated a good bed measurement in the second stage; however, it was difficult to discharge the solids from it. The discharge valve at the bottom was open, but the line could not be cleared, even by the use of aeration. One of the two aeration taps installed before this test was working, and the other was plugged. Because the transfer valve is counterweighted to close, excessive purging would simply have blown the valve open. There was no way to close the valve positively and to use aeration to blow the solids loose in an upward direction. Before shut down, the operation of the carrier steam line with solids had been checked out for the first time. Some of the starting bed of sand was injected into the final heat-exchange bed into the carrier steam. The solids were carried into the quench drum and because the flow of solids into the carrier steam was not properly regulated, the discharge slurry loop was plugged.

Overall, this test showed that the slurry-drying bed and the lift line were operable in a controllable fashion, with none of the gas short-circuiting of the type encountered in the first test. About 1600 standard cubic feet of methane per ton of lignite fed was produced, thus demonstrating that gasification had occurred in the lift line.

While the hydrogasifier reactor was being cooled down, after a run at a temperature of approximately 200°F, a sudden pressure equalization was observed between the nitrogen jacket and the reactor side, indicating that a pipe had ruptured. Upon opening up the reactor, the char recycle line from the top of the lift pipe to the mixing pot below broke because of tension at the mitered joint directly below the recycle control valve. This line was to have been fully extended in the cold position without any cold spring in the pipe. A 1-inch gap resulted at the rupture point, however, indicating that the pipe had been put under a 1-inch cold spring.

The second-stage gasifier was examined to determine why solids transfer had been impossible during the last test. A large number of refractory slabs, about 1 inch thick and averaging from 4 to 8 inches square, were found to have spalled from the refractory lining. Some of these slabs knocked off the 1/2-inch screen on top of the transfer line, and refractory chips filled the transfer line and bridged the entire cross section, thus preventing solids from being discharged. A flanged screen was installed on top of the pipe to keep the refractory chips from entering the transfer line. The valve at the end of the transfer line was cut and re-

positioned to counteract the bow in the line created by the heating and cooling. A flow test was run at ambient conditions to check the ability to start and stop flow in this transfer line with Calamo, a coarser refractory material, to be used in place of sand. The results indicated that the flow of solids could be controlled.

### 3.5.7 Gasification Test No. 3, January 1972

Hydrogen was switched into the system on December 22, 1971 and high-pressure air was injected to combust with the hydrogen for the final heat-up before starting coal feed. However, the slurry high-pressure air compressor broke down when the head gasket to the third stage failed. This allowed water to enter the cylinder which, on compression, broke the piston rod. Without air, the temperature dropped so that the reactor had to be shut down.

While the system was shut down, the slurry-dryer stage was examined through the top flange to check how much of the starting bed remained. During the heat-up, observers saw some abnormally high differential pressures across the slurry-dryer bed at the start; these later dropped to zero. Inspection showed that the removable, metallic slurry-dryer grid had been lifted by the high-pressure drop across the grid caused by wet sand plugging some of the distributor holes. Then on a subsequent flow change, during the combustion of the natural gas with air, the grid had dropped down, pivoted about its axis on a retainer ring, and had come to rest in a vertical position. This dumped all of the starting bed into the lift pipe.

The top closure was removed and the grid and the original hold-down weights were brought out of the reactor. All of the transfer lines were cleaned through the top and bottom. The hold-down device was modified by installing four jack-bolts to replace a weight formerly placed on top of the metal grid. Two additional oil-field chokes were received; one was installed in the discharge slurry circulation loop for flow control, and the other was installed as a letdown device in the filter house in place of the fixed orifice.

The original packing of the Wilson-Snyder mud pumps was replaced with Teflon. The suction and discharge valve seats made of polyurethane showed signs of initial wear. Investigations indicated that polyurethane was not compatible with toluene and had to be replaced. Similarly, the check valve sleeves on these pumps, also made of polyurethane, had become swollen from contact with the toluene and had to be replaced. Ball check-valves were installed to replace the sleeve check-valves, and Buna-N rubber valve seats were inserted to replace the polyurethane seats. Conflicting data exist on the extent of the effect of toluene on the properties of rubber, Buna-N rubber is considered to be the best material to use with toluene, although it is not ideal.

The hydrogen compressor operated satisfactorily on a total recycle of hydrogen; about 10 to 20 grams of fine black powder was collected every 5 days. The hydrogen compressor was examined while the plant was down;

the first and second stages were in excellent condition. The third-stage valves were also in fine condition; however, the piston rods and the packing rings showed wear. The wear on the packing rings may have been caused by the use of a bronze-filled Teflon instead of a carbon-filled Teflon on the first and second stages. The bronze-filled material was originally specified by Ingersoll-Rand when the unit was purchased. The company later recommended the carbon-filled material, which was to be installed at the next opportunity.

The reactor was heated up over the weekend of December 31 and was successfully switched over to hydrogen. Toluene was introduced, followed by the injection of coal slurry on January 3, 1972, for the third gasification test. The level in the slurry dryer bed was built up, but the transfer of solids could not be made from this bed into the first-stage gasifier. Two of the three nitrogen purge taps on the transfer line were partially plugged and were unable to deliver a blast of nitrogen to initiate the solids flow. Various means were attempted to induce solids flow. For example, the reactor pressure was changed to cause a surge of gas flow that might dislodge and move the solids. None of the attempts was successful.

A sub-zero cold spell occurred while a constant temperature was being maintained at 400-psi pressure with hydrogen and steam being fed into the reactor. Although all the steam tracing was turned on during this time, a number of the flanges on the level transmitters, which were not insulated to provide access to the bolts, froze in the cold. The level transmitter was indicating a false level, while the level in the quench water separator continued to rise, and eventually backed up into the quench water tower. When this occurred, water was carried over with the product gas from the reactor and accumulated in the flare. The resultant 30-psi increase in back pressure on the flare stopped the steam flow to the hydrogen plant, which in turn shut down the hydrogen plant. The run was terminated at this point.

In addition to the above weather-related problems, a number of instruments froze during the cold spell as a result of the water carry-over into the gas line; the frozen instruments made it difficult to determine pressures and flows. However, technicians were able to drain the water from the quench separator, the quench tower, and the flare, in order to clear the system of liquid and to prevent it from backing into the main gasifier vessel.

During the winter of 1970-1971, HYGAS personnel worked hard to repair the tracings that were installed but were not in service. During the second winter, problems concerned with inadequate amounts of tracing were encountered; the inadequacy was not apparent during the 1970-71 winter because most of these units were not on-stream.

After the reactor had cooled sufficiently, water was found in the top of the slurry dryer. This water had accumulated from the quench-water spray, which had been turned on during the test in an effort to control the off-gas temperature.

The top head was removed and the wetted solids were cleaned out. After the lines had been cleared, the internals of the slurry dryer bed were re-assembled; a new distributor grid with larger holes was included.



Upon entry into the manhole on the second-stage gasifier, HYGAS technicians found that the Calamo, which was used for the starting bed, had fused in many areas, forming lumps of various sizes. The Calamo is much coarser than sand and, therefore, does not fluidize as well. It was used, in the HYGAS plant, instead of sand, in an effort to minimize the weeping of sand through the grid and the transfer lines. Apparently poor fluidization caused inadequate mixing of the high-pressure air and hydrogen and resulted in local hot spots and fused some of the Calamo. All of the starting bed was removed, and sand was once again used as a starting material for the next run.

While the unit was shut down, the refractory grid was removed and a new grid of better design — including larger holes — was installed. The size of the aeration taps on all of the transfer lines was also increased, from 0.25 to 0.5 inch.

A number of repairs were also performed during the plant shut down. The natural gas-steam mixer, located before the reformer inlet, had been plugged during the previous 2 weeks, either with rust or with carbon dust from the carbon drum; this had resulted in a high-pressure drop across it. The mixer was disassembled, cleaned, and reinstalled. Parallel strainers were added ahead of the mixer, to prevent similar occurrences in the future. The stack damper on the reformer furnace presented new problems because it could not be moved to adjust the draft on the stack. A hole was cut in the stack and the damper was solidly welded to the shaft. A superheated steam connection to the carbon drum was also installed to provide better regeneration than had been obtained. The lower yields were caused by the use of dimethyl sulfide as an odorant in the gas, a component which had not been used until a year earlier, when it was introduced by Peoples Gas Company.

Members of the Ingersoll-Rand staff visited the HYGAS site to study the hydrogen compressor. They had no reasonable explanation for the scoring of the third-stage rod. During the last test, 55,000 standard cubic feet per hour of hydrogen was withdrawn from the plant, and the hydrogen compressor did not appear to be able to deliver the design capacity.

The valves on the hydrogen compressor were examined; two discharge valves on the first stage and one discharge valve on the second stage were found to be badly worn. This was caused by broken retainer clips on the valve guides. Checking with Ingersoll-Rand, it was learned that this problem had plagued other hydrogen compressors as well because of the brittle material used for the valve guides. Ingersoll-Rand ordered replacement guides made out of heat-treated, higher nickel content steel. The new type of nickel-steel valve guides were not received in time for the next test.

### 3.5.8 Gasification Test No. 4, February, 1972

The reactor was heated in preparation for the fourth gasification test in February. After switching over to hydrogen, a sudden equalization of pressure between the reactor side and the nitrogen jacket indicated that something had ruptured. The heat-up was terminated and the reactor was cooled. In the nitrogen jacket, workers found that a mitered elbow on the

solids-transfer line, between the first and second-stage gasifiers, had ruptured. The rupture was caused by a combination of the hard Type-446 stainless-steel metal and the inability of the expansion joint to move after it became filled with solids. The expansion joint and the pipe were replaced.

The initial bed of sand in the second-stage gasifier was also lost. When the reactor was opened, it was found that the sand had leaked through the openings in the new grid. A number of gaps existed between the bricks, and between the bricks and the wall; this increased the opening on the grid so much that gas flow was not maintained adequately to prevent weeping. The extraneous openings were filled with mineral wool and mortar. The transfer pipe was extended above the grid to ensure that a bed of sand would be present in the second-stage gasifier at all times.

While replacing the 0.25-inch blast connection with the 0.5-inch connection on the transfer line from the second-stage gasifier, workmen found that 14 inches of the transfer line had been burned out in an area directly above the start-up gas ring burner; this section was replaced. The gas ring burner had been completely filled with a mixture of coke and sand. The coke came from the cracking of natural gas. The blockage caused most of the gas to flow out near the burned-out transfer pipe. High local gas concentrations led to high temperatures, which were directly responsible for the failure of the pipe. The gas ring burner was cleaned out and reinstalled.

Before the reactor was depressurized, the slurry discharge system was activated. The flow of solids was not properly controlled, and consequently the line was plugged.

After replacing the expansion joint and a new section of pipe, which had a smooth bend, a low-pressure system test was attempted, and another ruptured pipe was found — the solids recycle line from the top of the lift line to the bottom. This rupture was smaller than the other described, which made detection impossible until the other rupture had been repaired. The rupture point was at the juncture of the recycle line to the mixing pot at the beginning of the lift line. Because the recycle line was not needed for lignite, both ends of the line were blanked off for the upcoming test.

On another pressure test on the hydrogasifier vessel, yet another crack was found on the solids-transfer line from the slurry dryer bed to the first-stage lift line. The break, at another mitered joint below the control valve, was also caused by the inability of the expansion joint to move freely when it was filled with solids. This particular expansion joint is always filled with solids because the slurry dryer bed is full of solids at the beginning of each test. However, no trouble had been encountered with this line except for one previous break, which was attributed to dissimilar metals. A new replacement expansion joint was installed at a new position below the control valve, which was raised to a point where sufficient seal leg remained above the control valve.

The hydrogen plant, which had been operating at one-third capacity on a standby basis, developed a leak in the quench desuperheater immediately downstream of the reformer tubes. This is the unit in which the hot effluent from the reformer is quenched with condensate to bring the temperature down to approximately 700 °F. While workmen were grinding out the crack

on the stainless steel vessel, more subsurface cracks were detected; these extended into the parent metal away from the main longitudinal weld. Radiographs showed that additional subsurface cracks were present in an area well away from the first crack. Upon consultation with Girdler, the manufacturer, HYGAS engineers decided to cut out the damaged section of the vessel and replace it in the field. It was then found that the inner Incoloy liner was severely cracked and also required replacement. The failure of the inner liner caused the failure on the outer pressure vessel. After cutting off the inner liner, no damage was found on this quench vessel. However, the spray connection for distributing condensate into the hot gas was not located properly, according to Girdler's drawings. This factor may have been responsible for improper distribution, and the resultant splash of liquid on the hot Incoloy plate may have led to its failure. Once the Incoloy liner had failed, hot gases leaked through the crack and directly contacted the outer pressure shell, causing it to crack. The distributor was relocated - again in consultation with Girdler - to prevent a recurrence of the cracking. Special alloy pieces were rolled and installed. All of the welds were 100 percent radiographed and found to be satisfactory.

After these repairs, a pressure test on the gasifier showed that the unit was tight, and the heat-up of the system was begun. The switchover to hydrogen was successful.

The reactor was brought up to 400 psig pressure, and engineers discovered that a differential pressure could not be maintained between the reactor side and the nitrogen jacket side, indicating another crack on the vessel. The reactor temperature was lowered and the pressure was let down.

The walls of the slurry dryer bed, immediately above the distributor grid, were eroded through at two spots. The grid was removed and found to be plugged with wet sand. At the two eroded spots there were clear indications that gas had flowed around the grid, through the gaskets between the grid and its support ring. This stream of gas flowing into a bed of fine sand had created a sandblasting effect that eroded the metal in that area. The plugged nozzles were cleaned and the nozzle configuration was modified slightly to minimize plugging. A 0.25-inch-thick Incoloy reinforcing pad was welded onto the eroded area. The reactor was reassembled and pressure-tested to 50 psig without finding any additional leaks. Heat-up of the reactor was initiated.

The hydrogasifier system was successfully switched over to hydrogen and brought up to pressure. A defective block valve was detected in the slurry system, requiring a pressure letdown in the system to make the change and then to raise system pressure again. Slurry was introduced to the system on February 21 when the fourth gasification test was begun. After feeding for a period, a high-pressure surge in the slurry system caused the slurry pump to be shut down. Slurry feed was started again on February 22 and continued for 24 hours before another high-pressure surge in the slurry system stopped the flow. During this 24-hour period, coal was fed through all four stages of the main gasifier. Solids were discharged out of the bottom of the reactor through the slurry letdown system. For the first time, solids were successfully put through from the top to the bottom of the reactor.

Solids flow was controlled by the solids-control valves on each stage. The highest methane yield, through the first four shakedown tests, was 8 percent, on a dry nitrogen-free basis, with a heating value of approximately 400 Btu per cubic foot. Increasing knowledge gained in these operations permitted constant improvement in control of bed levels and the solids flow rate to make operations smoother. The reactor pressure was approximately 450 psig.

A few 0.0625- to 0.125-inch particles in the -4 mesh coal feed had lodged between the valve and the seat on the Wilson-Snyder slurry pumps. This resulted in leakage through the valve and inability to build up pressure. The 4 mesh screen was then replaced with an 8 mesh screen on the feed vibrator to eliminate the large particles. The 4 mesh screen had been used earlier to resolve difficulty in screening.

During this period, the screening operation was improved by substituting a more powerful vibrator, and a steam-traced and heated feed hopper. In addition, the coal-toluene mixture, which contains the large particles, was flushed out.

The recurring problem of steam-tracing failures was analyzed and a basic flaw was detected in the manner in which the tracing system was set up. According to the published criteria used by construction companies, with a 40-psig steam supply the condensate should be discharged directly to the atmosphere. For a system in which condensate is collected, the steam supply pressure should be much higher, preferably over 100 psig, to push the condensate back to the deaerator. The HYGAS pilot plant steam-tracing system operates at 40-psig and collects condensate for return. A high back-pressure had been experienced consistently on the condensate return line which, of course, cut down the differential pressure available to push the condensate out of the tracers. The problem was analyzed from the standpoint of available water supply, steam balance, and tracing needs, to determine what fundamental modifications might be required. In the meantime, temporary drain lines were set up to discharge the condensate to the atmosphere during the remainder of the second winter of operation. The temporary drain lines eliminated the tracing problems. All of the frozen tracing lines were repaired.

The fourth gasification test continued until February 29, when it was terminated. Throughout the test, more than 30 tons of coal were fed to the reactor and more than 100,000 standard cubic feet of methane was produced. The highest concentration of methane achieved during this period was 20.4% on a dry, nitrogen-free basis.

The gasifier vessel was cooled down and opened; and was found to be in excellent condition. The slurry dryer bed was full of very fine coal, contrary to the indications of the radiation bed-level detector and of the differential pressure gage. The slurry dryer grid was also found to be in good condition; there were no signs of erosion in the grid holes.

The piping in the first-stage gasifier remained in good condition. The expansion joint on the solids transfer line between the first- and second-stages was now modified in the same manner as the expansion joint in the solids line feeding the riser reactor had been modified; the control valve was raised, and the expansion joint was moved to a position below the valve. External pressure from the