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ADVANCEMENT OF FLASH HYDROGASIFICATION
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By
A. Y. Falk
Project Engineer, Coal Hydrogasification

Approved by:

D. R. Kahn
Program Manager, Synthetic Fuels Programs

Rockwell International

Environmental & Energy Systems Division
Energy Systems Group
8900 De Soto Avenue
Canoga Park, California 91304

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FOREWORD

This document is the first quarterly technical progress report summarizing work performed by Rockwell International for the U.S. Department of Energy, Morgantown Energy Technology Center (METC), under Contract DE-AC21-78ET10328* titled "Advancement of Flash Hydrogasification." The period of performance for the technical tasks of this contract work is from 23 December 1983 through 30 May 1985 (17 months). This report covers the period from the start of the Advancement of Flash Hydrogasification effort through 30 March 1984.

The Rockwell program manager is Dr. Daniel R. Kahn. Technical administration of the program for DOE-METC is being provided by Mr. Gary R. Friggens.

*This basic contract has been modified several times since its inception in 1978. Formerly, it was referred to as "Coal Gasification Process Development" under DOE Contracts ET-78-C-01-3125 and DE-AC01-78ET10328.

ABSTRACT

This first quarterly report documents technical progress during the period 31 December 1983 through 30 March 1984 on Rockwell International's Advancement of Flash Hydrogasification Program for the U.S. Department of Energy. The technical effort is 17 months in duration and is divided into two major technical tasks: Task VII, Hardware Fabrication and PDU Modifications, and Task VIII, Performance Testing. The design of test hardware and process development unit modifications had been previously completed as part of Task VI of the current contract.

Task VII involves the fabrication of test hardware and modification of an existing 1-ton/h hydroliquefaction PDU at Rockwell's facilities for use as a hydrogasifier test facility. During this report period, fabrication of the test hardware and modifications to the PDU were initiated. Test hardware fabrication is now approximately 80% complete and should be completed by the end of May 1984. PDU modifications are progressing well and should be completed by the end of June 1984.

The completed test hardware fabrication and PDU modifications will allow the conduct of short duration (1 to 2 h) hydrogasification tests along with preburner assembly performance evaluation tests in order to fulfill the test program objectives. Separate supplies of hydrogen, oxygen, methane, carbon monoxide, and water (for steam generation) are provided for this purpose. The modified facility is designed to accommodate both 10- and 20-ft-long hydrogasifier reactors so that residence times will be in the range of 2 to 6 s when coal is fed at a nominal 1/2 ton/h into reactors at 1000 psia pressure. Provisions are being made for real-time analysis of the product gases using an on-line gas chromatograph system.

Test planning was the only Task VIII effort active during this report period. An initial (preliminary) test matrix has been defined, preparation of

a data analysis plan is underway, and data reduction programs are being programmed. The test matrix will be part of the test program plan and schedule to be submitted to DOE for review and approval in early May. Test system activation is planned for July 1984, and reactor performance testing will be conducted during the August-December 1984 time period.

ACKNOWLEDGMENTS

The technical work on the Advancement of Flash Hydrogasification Program, Contract DE-AC21-78ET10328, is being organized and conducted under the technical direction of a Rockwell Energy Systems Group (ESG) project engineer, Dr. Arthur Falk.

Major technical contributions to the conduct of the program during this report period were made by the following ESG personnel:

Task VII-A--Test Hardware Fabrication	M. Marko I. Burow
Task VII-B--Facility Modifications	N. Doshier F. Mulligan
Task VII-C--Process Systems Modifications	J. Lecce V. Dayan
Task VIII-C--Test Planning and Data Analysis	M. Schuman C. Erickson

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I. INTRODUCTION

Under U.S. Department of Energy (DOE) sponsorship, Rockwell International has been developing a process for coal flash hydrolysis (FHP) based on the reaction of pulverized solid carbonaceous feedstocks with hydrogen at elevated temperature (1400 to 1900°F) in a single-stage, short-residence-time, entrained-flow reactor. Gases and liquids, if desired, are produced in the reactor, and unreacted material forms a dry, free-flowing char. Following product gas quench, liquid products are separated and gases are processed for recycle and product recovery. By varying the reactor operating conditions, the makeup of the product slate can be controlled over a broad range--from predominantly a syncrude oil with substitute natural gas (SNG) byproduct, to SNG with a relatively pure (>98 wt. %) benzene byproduct, to SNG alone as the sole hydrocarbon product. The hydrogasification process operates in one of the latter two product regimes, such that a large fraction of the feedstock's carbon is converted directly to methane.

On 10 May 1983, Rockwell initiated work on Task VI (Design of PDU Modifications) of the Coal Hydrogasification Process Development Program under DOE Contract DE-AC21-78ET10328. The objective of this task was to design the test hardware and facility modifications necessary to convert an existing 1-ton/h coal hydroliquefaction process development unit (PDU) at Rockwell's facilities for use as a hydrogasification test facility. This would provide the capability for subsequent engineering-scale testing to address key questions on reactor chemistry associated with the advancement of flash hydrogasification.

The Task VI design effort was conducted over a 5-month period ending in September 1983. The test hardware and facility modifications were designed such that the PDU would be capable of accommodating hydrogasifier testing over a wide range of operating conditions. The modified facility will permit short-duration (1 to 2 h) hydrogasification testing and preburner performance evaluation testing to fulfill subsequent program objectives. Separate supplies of hydrogen, oxygen, methane, carbon monoxide, and water (for steam generation)

are provided for this purpose. The modified facility is designed to accommodate both 10- and 20-ft-long hydrogasifier reactors so that residence times will be in the range of 2 to 6 s when coal is fed at a nominal 1/2 ton/h into reactors at 1000-psia pressure. Provisions have been made for real-time analysis of the product gases using an on-line gas chromatograph system.

A. PROGRAM OBJECTIVES

On 23 December 1983, DOE and Rockwell International mutually agreed to further amend the contract statement of work to incorporate two additional tasks: Task VII - Hardware Fabrication and PDU Modifications, and Task VIII - Performance Testing. The overall objective of the modified program is to investigate and verify the reactor chemistry associated with the Rockwell Advanced Flash Hydropyrolysis (FHP) concept for the production of SNG from coal. Specific objectives involve:

- Fabricating test hardware and modifying an existing 1-ton/h hydroliquefaction PDU to allow it to be operated in a mode that simulates the preferred reactor operating conditions.
- Operating the modified facility to develop the data base required to verify the reactor chemistry associated with the advancement of flash hydrogasification.

Further reflecting this modification of the scope of work, the contract title has been changed to "Advancement of Flash Hydrogasification."

B. BACKGROUND

Rockwell International has been developing flash hydropyrolysis (FHP) of carbonaceous feedstocks under several DOE contracts since 1975. The basic Rockwell process concept and its flexibility in control of the product slate were noted earlier.

A key element of the FHP reactor concept is the injector/reactor technology pioneered and developed by Rockwell. This technology, first used in liquid-fueled rocket engines, has several distinctive features that make it superior to alternative means of achieving hydrolysis of carbonaceous feedstocks. These include extremely rapid mixing and heating of reactants (10 to 20 ms), precise and uniform control of temperature and reaction time, rapid reactor startup and shutdown, unrestricted choice of feedstock, and simple scaleup. In addition, because the reactions occur in a short-residence-time (20 ms to 6 s), high-pressure (500 to 1500 psig), entrained-flow mode, the reactor is inherently compact. This permits shop fabrication of even commercial-scale hardware, with cost and reliability advantages over field fabrication. Reliability also benefits from the mechanically simple design and because it is economically possible to use high-quality materials in a more compact unit. The small reactor size contributes to rapid startup, stable operation, and facile process control. The small reactor size and modularity also make the process more readily maintainable than competitive processes and make incorporating a spare reactor cost effective. These factors lead to increased plant availability and benefit overall plant economics.

Rockwell International's first coal hydrogasification project began in March 1977 as a joint effort with Cities Service Company under DOE Contract EX-77-C-01-2518, and involved a 20-month bench- and engineering-scale study.¹ Subsequently, Rockwell undertook the current development program under DOE Contract DE-AC-01-78ET10328.

The initial engineering-scale hydrogasification tests conducted at Rockwell were performed in a 1/4-ton/h facility originally developed for hydroliquefaction testing. Forty-three successful tests were conducted, consisting of 30 tests with three types of coal (Montana Rosebud subbituminous, Kentucky hvAb, and Illinois hvCb) and 13 tests with Minnesota peat. Reactor pressures ranged from 500 to 1500 psig, temperatures from 1000 to 2000°F, and residence times from 500 to 5500 ms. Carbon conversions to gases as high as 71% with coal and 84% with peat were achieved. Tests were also run with hydrogen feed

gases containing 15 to 20% CH₄, simulating a recycle process gas. After 1 year, testing was moved to a 1-ton/h process development unit (PDU) facility to allow running longer tests (1 to 2 h). Tests were conducted with the bituminous coals used in the smaller scale facility experiments.

At the conclusion of these test activities, the second contract²⁻⁴ was initiated. This effort consisted of testing in a 3/4-ton/h engineering-scale test unit (ETU), together with the design and construction of an 18-ton/day integrated process development unit (IPDU).

In the first year of testing under the latter program, both Montana Rosebud subbituminous and Kentucky hvAb bituminous coals were used in the ETU. During the second year, 32 additional tests were completed, primarily with Kentucky No. 9 hvAb bituminous coal at a reactor pressure of 1000 psia. Injectors demonstrated stable operation with high carbon conversion to gas at low hydrogen-to-coal ratios (0.2 to 0.4 by weight). A hydrogen-cooled recuperator was constructed and tested. Significantly, three replicate runs (Tests 318-36, -37, and -50) were made under nearly identical conditions and produced results showing high reproducibility. This demonstrated the advance of the hydrogasification process to the stage of reliable test operations. These runs also demonstrated a high carbon conversion to BTX (~11%) and subsequently served as the design point for a commercial plant design performed by The Lummus Company under subcontract to Rockwell. The BTX (benzene-toluene-xylene) produced from the reactor is 98 to 99% benzene and can be readily upgraded to chemical-grade purity using existing commercial technology.

Design of the IPDU facility was completed in May 1980, and construction at Rockwell's Santa Susana Field Laboratory near Los Angeles proceeded until being halted by DOE at about 60% completion. The facility is designed for continuous operation for periods up to 30 days and has provisions for on-site coal storage, coal pulverization and drying, pneumatic lock hopper pressurization of the pulverized coal feed, lock hopper letdown of the char produced, and work-around (versus remote) facility operation.

Rockwell has also performed extensive modeling studies and has developed a sophisticated coal FHP reactor model for correlation and prediction of FHP hydrogasification and hydroliquefaction test results. The method used involved defining a set of phenomenological reactions representing the processes occurring during FHP and then applying a nonlinear multiple regression technique to a set of equations derived according to the kinetics of those reactions.⁴

As an integral part of Rockwell's Coal Hydrogasification Process Development Program, The Lummus Company (now Lummus Crest Inc.), with the concurrence of DOE, prepared under subcontract to Rockwell a preliminary design of a complete 250 billion Btu/day commercial-scale SNG-from-coal plant based upon the Rockwell Coal Hydrogasification Process. This study was conducted over an 18-month period, culminating in the issuance of a Predesign Report submitted to Rockwell in May 1981, entitled "Coal Hydrogasification Plant--Preliminary Design Study for a 250 Billion Btu-Per-Day SNG Plant."⁵ In addition to the 250 billion Btu/day of SNG, the baseline plant was designed to produce 5310 bbl/day of chemical-grade benzene as a coproduct. The plant showed excellent economic potential, with a cost of gas estimated by Lummus at \$3.68 per million Btu in mid-1979 dollars, using the economic methodology developed by C. F. Braun⁶ for DOE. Total plant investment was estimated at \$1331 million, including a 15% project contingency.

Beyond the DOE-sponsored programs, Rockwell has continued its efforts to advance FHP technology and has studied process alternatives with potential for improving the process, increasing its efficiency, reducing capital requirements and operating costs, etc. These in-house Rockwell studies were aimed primarily at alleviating the expensive burden of makeup hydrogen production, freeing the process from dependence on other developmental technologies, and simplifying downstream product processing. These efforts yielded a new Rockwell process concept--termed Advanced FHP.

A Rockwell coal FHP reactor model was used extensively in developing the Advanced FHP process. This model enabled coal conversion levels and reactor effluent compositions to be predicted as functions of reactor operating conditions. Combining these results with parallel analyses of downstream processing led to the establishment of desired process operating points. Various process designs were subsequently developed and their economics evaluated. This Rockwell process design effort provided the basis for further proposed experimental testing and led to the present modification of Contract DE-AC21-78ET10328 to investigate and verify the reactor chemistry associated with the Advanced FHP concept.

II. SUMMARY OF PROGRESS DURING THE QUARTER

A schedule for the Advancement of Flash Hydrogasification program is presented in Figure 1. The technical effort is 17 months in duration and is divided into two major tasks: Task VII - Hardware Fabrication and PDU Modifications, and Task VIII - Performance Testing.

Task VII is divided into three subtasks: Task VII-A, Test Hardware Fabrication; Task VII-B, Facility Modifications; and Task VII-C, Process Systems Modifications.

During the current report period, effort on Task VII-A was initiated. The test hardware drawings prepared during Task VI were reviewed, materials were procured, and fabrication of the test hardware component parts was begun. Fabrication of the hardware is progressing well and is ahead of schedule. Most of the component parts have been machined, and assembly of the injectors and preburner is the primary task remaining. Completion of hardware fabrication is expected by the middle of May.

A subcontract to complete the facility modifications (Task VII-B) was awarded to Southern Mechanical, Inc., on January 24, 1984. The construction specifications for this work had been prepared as part of Task VI of the contract. This effort consists primarily of modifications to the PDU facility structure necessary to (1) accommodate longer residence time reactors (nominal 10- and 20-ft-long reactor trains) and (2) incorporate provisions for locating an on-line gas chromatograph (GC) adjacent to the PDU in a separate existing structure. Onsite contractor work was initiated on February 27, 1984. The work is progressing according to schedule, and Southern Mechanical plans to complete this subtask effort by the end of April 1984.

The process systems modifications (Task VII-C) include modification of existing H_2 and O_2 systems, installation of new CO , CH_4 , water coolant, and water for steam generation systems, and installation of an on-line GC

MAJOR MILESTONE LOG

▲ FACILITY MODIFICATIONS COMPLETED

▲ TEST HARDWARE FABRICATION COMPLETED

▲ PROCESS SYSTEMS MODIFICATIONS COMPLETED

▲ FACILITY ACTIVATION INITIATED

▲ FACILITY ACTIVATION COMPLETED

▲ REACTOR TESTING INITIATED

▲ REACTOR TESTING COMPLETED

▲ DATA ANALYSIS COMPLETED

▲ DRAFT TOPICAL (TASK VIII) AND FINAL REPORTS SUBMITTED

▲ APPROVED TOPICAL (TASK VIII) AND FINAL REPORTS SUBMITTED

◆ DRAFT

□ FINAL

Figure 1. Advancement of Flash Hydrogasification Program Schedule

sampling system for monitoring of preburner and reactor exit gases. Procurement of new, and servicing of existing, process systems components is in progress. The majority of the effort on this subtask will be accomplished in the April-June time period, once the subcontractor has completed most of the facility modifications. The process systems modification work is being performed by Rockwell Operations personnel. Installation and/or modification of several of the gaseous feed systems has been initiated. Progress on this subtask is slightly behind schedule; however, activity will be increased starting in April to meet the program schedule.

Task VIII is divided into three subtasks: Task VIII-A, System Activation; Task VIII-B, Testing; and Task VIII-C, Test Planning and Data Analysis.

Test planning was the only Task VIII activity occurring during this report period. Work is in progress on preparation of the planned test matrix and a data analysis plan. The test matrix will be part of the detailed test program plan and schedule to be submitted to DOE for review and approval in early May. A preliminary test matrix is presented later in this report. The data analysis plan will describe planned sampling, the chemical analyses to be performed, system measurements to be made, and how this information will be utilized to characterize reactor system performance. This document will be completed later in May.

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III. DISCUSSION OF PROGRESS DURING QUARTER

The technical effort on DOE Contract DE-AC21-78ET10328, Advancement of Flash Hydrogasification, was initiated on 23 December 1983. The program is divided into two major tasks, each containing three subtasks, as noted below:

Task VII--Hardware Fabrication and PDU Modifications

- Task VII-A--Test Hardware Fabrication
- Task VII-B--Facility Modifications
- Task VII-C--Process Systems Modifications

Task VIII - Performance Testing

- Task VIII-A--System Activation
- Task VIII-B--Testing
- Task VIII-C--Test Planning and Data Analysis

During this report period, work was initiated on Subtasks VII-A, VII-B, VII-C, and VIII-C. Progress on these various tasks/subtasks is discussed in detail in the following sections.

A. TASK VII--HARDWARE FABRICATION AND PDU MODIFICATIONS

1. Task VII-A--Test Hardware Fabrication

The progress on test hardware fabrication is summarized in Figure 2. The fabrication status (i.e., % complete) of the various specific work packages is noted by the shaded bars. The test hardware drawings prepared in Task VI have been reviewed, materials have been procured, and the majority of the hardware component parts have been machined. Assembly of the service lines, service ring, injectors, and preburner is the primary task remaining on the hardware fabrication effort. Overall, this subtask is approximately 80% complete.

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Figure 2 . Test Hardware Fabrication Status (Task VII-A)

a. Test Hardware Assembly

Test hardware has been designed to accommodate hydrogasifier testing over the range of operating conditions listed in Table 1 and permit short-duration (1 to 2 h) hydrogasification tests to be performed. The specific test hardware components designed in Task VI include: (1) a preburner assembly; (2) an injector; (3) 10- and 20-ft-long reactor tubes (one each); (4) a reactor pressure vessel cap, spool, and service ring; (5) hardware service lines and supports for use within the reactor pressure vessel; and (6) a gas chromatograph (GC) sample line assembly. Where possible, maximum use was made of existing proven PDU components and associated test hardware. For example, the injector is a modification of an existing coal hydroliquefaction injector described in Ref. 7. Several components, including a cap, spool and service ring, were designed to be added to an existing reactor pressure vessel to accommodate the longer reactors and new preburner assembly. Reactor flanges from existing FHP reactors are being employed. The preburner assembly, GC sample line assembly, and hardware service lines and supports, however, were newly designed components.

The test hardware assembly shown in Figure 3 comprises the major process components required to fulfill the program objectives. High-temperature hardware components such as the preburner assembly, injector, and reactor tube are contained within a reactor pressure vessel to provide a pressure-balanced environment. The reactor tube is pressure balanced at the reactor operating pressure, while the upstream components are designed to withstand the differential pressure load due to the fluid flow induced head losses. The new reactor pressure vessel components (a cap, spool, and service ring) required to permit testing with 10- and 20-ft-long reactors are also shown in Figure 3. A simplified sketch of the test hardware configuration is presented as Figure 4.

The pressure vessel cap is designed to provide ready access to the preburner assembly and service lines for inspection and/or maintenance. The service ring provides fluid service ports, pressure measurement taps, and thermocouple line penetrations. Because the test program requires two different

TABLE 1
TEST HARDWARE DESIGN CONDITIONS

Component	Parameter	Design Range
Preburner ^a	Pressure	1500 psia (operating pressure 1100 to 1300 psia)
	H ₂ /CH ₄ mixture (at 1100°F) flow rate	150 to 460 lbm/h
	Ambient temperature O ₂ flow rate	250 to 730 lbm/h
	Hot water (steam) flow rate	180 to 1250 lbm/h
	Ambient temperature H ₂ flow rate	40 to 250 lbm/h
	Coolant water flow rate	10 gpm (ambient temperature at 1000 psig)
Injector ^a	Pressure	1500 psia (operating pressure 1050 to 1250 psia)
	Coal flow rate	1000 lbm/h
	Hot gas inlet flow rate	0.20 to 0.25 moles/lbm coal
	Hot gas inlet temperature	1500°F
	Ambient temperature O ₂ flow rate	55 to 300 lbm/h
	Hot gas exit flow rate	0.20 to 0.25 moles/lbm coal
Reactor tubes ^a	Hot gas exit temperature	Up to 2600°F (2000 to 2600°F)
	Pressure	1000 psia (balanced pressure operation)
	Temperature	1800 to 2000°F
Reactor pressure vessel	Residence time	2.5 to 5.5 s
	Pressure	1400 psia (operating pressure 1000 psia)
GC sample line assembly	Temperature	650°F (operating temperature 300°F)
		Designed to cool gas sample from 2000°F to 1000°F (essentially balanced pressure operation)

^aContained within 1000-psia reactor pressure vessel.

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Figure 3. Test Hardware Assembly

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Figure 4. Simplified Sketch of Test Hardware Configuration

reactor lengths, the length of the pressure shell can be adjusted by adding a 10-ft spool section.

As shown in Figure 3, a manifold for a nitrogen gas purge is provided to keep hydrogen gas from collecting around the oxygen lines and to minimize migration of char throughout the test hardware internals.

The reactor internals assembly will be instrumented with pressure tap sensor lines and thermocouples (T/Cs) as required to provide key operational and performance data. Pressure taps are located at the injector face, in the preburner exit line, and on the reactor pressure vessel wall. Thermocouples will be located on the reactor tube wall, in the reactor near its outlet, in the injector body, at various positions in water coolant for the preburner assembly, and in or on the preburner hot gas inlet and exit lines. Critical T/Cs located on the preburner assembly exit gas line, preburner assembly throat coolant, and reactor exit streams are redundant. Stream pressures and temperatures will also be measured entering and leaving the reactor pressure vessel.

A minimum 0.5-in.-thick Fiberfrax 6 lb/ft³ thermal insulation blanket will be wrapped around the reactor tube, injector body, and the approximately 1500°F and 1100°F expansion lines to limit process heat losses and pressure shell temperatures to acceptable levels.

The test hardware assembly is designed to facilitate component assembly, the checkout of internal pressure connections for leak tightness, and installation into the pressure shell. The preburner assembly, injector, reactor tube, and service ring will be assembled, instrumented, insulated, and pressure-checked in the test area workshop prior to its installation into the reactor pressure vessel on the test stand.

The overall assembly is 321-1/4 in. long by 34-3/4 in. wide and weighs approximately 1200 lb with the nominal 20-ft-long reactor tube attached. It

is 205-1/4 in. long and weighs approximately 1100 lb with the 10-ft reactor tube.

The reactor internals assembly is comprised of the following components:

- Preburner assembly
- Hot gas/coal, 4-on-1 impingement, water-cooled injector
- Reactor tube, 10 ft or 20 ft
- Service ring
- Preburner hot gas (approximately 1100°F) inlet line
- Injector hot gas (approximately 1500°F) line
- Preburner support
- Injector support
- Service lines (O_2 , H_2 , H_2O for steam, H_2O coolant, CO, N_2)
- GC sample line assembly
- Pressure and temperature instrumentation
- Fiberfrax thermal insulation.

b. Test Hardware Components

(1) Preburner Assembly

The preburner assembly, which is approximately 24 in. long by 4 in. in diameter and weighs about 50 lb, is comprised of the $H_2/CH_4/O_2$ preburner and the steam generator. Design of the preburner assembly was based on supplying the required hot feed gases to the injector at $\sim 1500^\circ F$. This unit provides for the partial oxidation of methane and the generation of steam in situ by evaporating hot water in the presence of the other reactants. Provisions have also been made to add ambient temperature CO to the preburner assembly exit gas upstream from the injector, as required.

The preburner (P/B) assembly is composed of these functional parts:

● H ₂ /CH ₄ /O ₂ coaxial injector	}	H ₂ /CH ₄ /O ₂ preburner (P/B)
● P/B combustion chamber		
● Water injector		
● H ₂ injector	}	Steam generator (S/G)
● S/G chamber		
● P/B exit flange		
● Thermal sleeve		

The unit, designed with a removable injector and flared tube service line connections, is flanged to the exit line to facilitate assembly, disassembly, and modification or repairs.

Each major component function is described below.

H₂/CH₄/O₂ Coaxial Injector--The preburner injector design provides for the efficient mixing of a preheated H₂/CH₄ mixture with ambient temperature O₂ prior to subsequent injection of hot water and ambient temperature H₂ streams into the steam generator section of the preburner assembly. Established mixing criteria obtained from Refs. 8 and 9 were used in developing these designs. A coaxial element injector scheme was selected for the H₂/CH₄/O₂ injector because of its good performance capability over a wide fuel/oxidizer flow range. The key factor for establishing good mixing effectiveness was determined to be the fuel/oxidizer velocity ratio. Also, a multi-element (7-hexagonal array) approach was selected to minimize chamber length (and thus heat losses) and to assure ignition by surrounding the central element with barrier elements to prevent quenching by the water-cooled wall of the chamber during ignition. Uniform fuel and oxidizer flow to each element is assured by high pressure drop design combined with low plenum inlet and distribution velocities.

P/B Combustion Chamber--Design of the combustion chamber is based on the data from Refs. 8 and 9, which determined the effect of combustion interference on shear element cold-flow mixing. These results clearly demonstrated a significant increase in mixing length required because of combustion interference. Based on these data, a chamber length was selected to assure complete combustion.

Water Injector and S/G Chamber--The water injector and S/G chamber extends from the water injection orifice plane to the end of the preburner assembly. The water necessary to provide the desired steam concentration and, combined with the downstream hydrogen, cool the process gases from approximately 3500°F to approximately 1500°F is injected at about 200°F through 16 orifices at a nozzle throat. The throat is provided to increase the process gas velocity and thus generate 20- μ m water droplets, which rapidly evaporate to minimize the S/G heat losses.

The drop size analysis was based on the experience of Rockwell's Rocketdyne Division with gas/liquid cross-flow atomization and the determination of the stream/drop penetration and mixing.¹⁰⁻¹²

H₂ Injector--The H₂ injector consists of two sets of orifices: one with four equally spaced 1/8-in.-diameter holes, the second with four equally spaced 1/16-in.-diameter holes. Both sets are located downstream of the S/G water injection. The design of this injector is based on Refs. 13, 14, and 15. Two sets of orifices are required because a wide range of hydrogen flows must be handled and because good mixing and penetration could not be obtained with a single set. Good mixing is required to assure meeting the 1500°F outlet temperature. Only one set of orifices will be employed for any test.

P/B Assembly Exit Flange--A water-cooled flange is provided to facilitate assembly and disassembly of the P/B assembly from the 1500°F exit line.

Thermal Sleeve--An Incoloy 800 thermal sleeve is provided in the P/B assembly exit flange to minimize heat and pressure losses.

With the exception of the Incoloy 800 internal thermal sleeve of the preburner assembly body, all of the preburner assembly component parts have been machined. Photographs of the major component parts of the preburner injector and preburner combustion chamber and steam generator components are presented in Figures 5 and 6, respectively.

(2) Injector

The injector assembly, a hot gas/coal 4-on-1 impingement, water-cooled unit is a modification of a successful design used previously in Rockwell's coal hydroliquefaction program to provide up to 2600°F hot reaction gases.⁷ A photograph of one of the injector assemblies used during that program is presented in Figure 7. Figure 8 is a photograph of the modified injector components that will be employed during this program.

Two liquefaction injector assemblies (one primary plus a backup unit) have been modified for use on this program. All of the required injector components have been machined, and only assembly of these units remains to be completed.

The injector consists of three basic elements:

- 4-on-1 impingement configuration
- Main burner, recessed coaxial elements
- Water-cooled face and coal tube.

Hot Gas/Coal Mixing--The hot gas/coal mixing performance of the 4-on-1 impingement injector is based on the cold flow test results cited in Ref. 1. These results are shown in Figure 9 with the range of performance for this program superimposed. The diameter of the coal tube has been chosen to provide the optimum mixing at the nominal hot gas flow rate for the required gas

Figure 5. Photograph of Preburner Injector Components

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Figure 7. Photograph of Liquefaction Injector To Be Modified
for Use on Advancement of Flash Hydrogasification Program

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Figure 8. Photograph of Modified Injector Components

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Figure 9. 4-on-1 Injector Mixing Efficiency Correlation

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port diameter. The spread in mixing efficiencies is due to the planned hot gas flow rate and density variations throughout the test matrix at a nominal 1000-lb/h coal flow rate.

Main Burner--The main burner elements are designed to provide optimal mixing at a hot gas outlet temperature of 2100°F. The fuel annulus of the existing hydroliquefaction injector body was increased by reboring the post hole to provide good mixing at an acceptable fuel side pressure drop (i.e., approximately 100 psid). The O₂ post has also been redesigned to provide for operation at approximately 1500°F.

Water-Cooled Coal Tube--Water cooling of the injector face and coal tube is provided to assure a coal tube temperature less than 350°F, thus preventing coal sticking and blockage of the coal exit port and keeping the copper sealing gasket and reactor tube mating flange and bolts at acceptable temperatures.

The water coolant for the coal tube is extended through the 1500°F expansion coil, as shown in Figure 3, to provide a low-temperature, cooled support for the injector and reactor tube. These components are supported from the service ring by a bolted cantilever.

(3) Reactor Tubes

Two reactor tubes (Figure 10), nominally 10 and 20 ft in length, were designed and fabricated to meet the process gas residence time requirements (2 to 6 s) of the program. An 8-in.-diameter pipe is the maximum permissible for the ~12-in.-ID pressure vessel shell while still having enough room for thermal insulation and installation clearance. The upper (inlet) end is flanged and copper-gasket sealed to the water-cooled face of the 4-on-1 injector body for ease of changeout. This same seal arrangement has been successfully employed during hydroliquefaction testing.

a. NOMINAL 20-ft-LONG REACTOR TUBE

b. NOMINAL 10-ft-LONG REACTOR TUBE

Figure 10. Sketch of Reactor Tubes

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The reactor tubes were fabricated from 304 stainless steel which, based on Rockwell's previous coal hydrogasification test experience, has sufficient strength and corrosion resistance under the temperature and char/gasification environment for the proposed test durations. The inlet and outlet flanges of both reactor tubes were obtained from existing hydroliquefaction reactors.

A simplified sketch and a photograph of the reactor tubes are presented in Figures 10 and 11, respectively. The diverging/converging sections in the reactors were required to obtain the desired residence time within the physical constraints of the reactor test system.

(4) Reactor Pressure Vessel Components

Three new reactor pressure vessel components--a cap, a spool, and a service ring--were designed for use during the planned test program. The cap is designed to provide ready access to the preburner and its service lines for inspection and/or maintenance as needed. This will be accomplished by removing only the pressure vessel cap, since all service lines penetrate the service ring. Because the test program requires two different reactor tube volumes, the length of the pressure shell can be adjusted by adding the 10-ft spool assembly. The service ring was made from a 14-in., 1500-lb blind flange with a 13.5-in.-diameter bore and an R-63 ring groove seal in its top surface. The service ring provides for all the fluid service, pressure measurement tap, and thermocouple line penetrations, as well as providing locations for attachment of injector and preburner supports.

The pressure vessel components are designed for conditions of 1400 psig and 650°F. This provides a substantial margin above the planned 985 psig at approximately 300°F operational levels, thereby minimizing the need to activate the pressure relief system during operation.

Figure 11. Photograph of Nominal 10- and 20-ft-long Reactor Tubes

Fabrication of the reactor pressure vessel spool and cap has been completed. Although the service ring component parts have been machined, assembly of this unit remains to be completed. A sketch and photograph of the reactor pressure vessel spool and cap are presented in Figures 12 and 13, respectively.

(5) Injector and Preburner Supports

Two brackets have been designed to support the hardware within the reactor pressure vessel. One of these is a carbon steel cantilever support for the injector and reactor tube assembly. This support is designed to limit the lateral deflection of the reactor tube at its exit flange to avoid hard contact between the tube and the pressure shell during heatup of the reactor. It is bolted to both the service ring and coal coolant tube to facilitate assembly and disassembly. The second bracket, also a carbon steel cantilever, is bolted between the water-cooled exit flange on the preburner assembly and the service ring. This support is designed to remove the 1500°F and 1100°F expansion line loads generated during operation due to the support anchor to line temperature differences. Fabrication of both of these supports has been completed and they are shown in Figures 5 and 8.

(6) Service Lines

Nearly all of the fluid service lines were manufactured from 0.5-in. OD x 0.035-in. wall Type 304 stainless tubing with 37° flared fittings for ease of assembly and disassembly. Because of small thermal expansion motion at the preburner and injector end (top) and the need for some flexibility during assembly to avoid tight fabrication tolerances, only minor thermal expansion loops are provided in the cool lines.

Two hot service lines are required. These are the preburner H_2/CH_4 mixture inlet line (1100°F) and the main burner hot gas inlet line (approximately 1500°F).

a. 10-ft-LONG REACTOR PRESSURE VESSEL SPOOL

b. REACTOR PRESSURE VESSEL CAP

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Figure 12. Sketch of Reactor Pressure Vessel Cap and Spool

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Figure 13. Photograph of Reactor Pressure Vessel Cap and Spool

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The 1100°F preburner inlet line is a three-loop helically coiled, 8.5-in.-mean-diameter, 3/4-in. Schedule 40, Type 304 stainless steel expansion line designed to limit thermal expansion loads to 200 lb. This line is attached between the service ring thermal standoff and preburner injector with standard 37° flared fittings for ease of assembly and disassembly. Experience on the coal hydroliquefaction program^{7,16} with 37° flared lines operated at 1100°F to 1200°F under low primary stress levels was excellent with regard to seal retention. An insulated, welded thermal sleeve is used at the service ring penetration to avoid bulk heatup of the ring by the 1100°F gases.

The 1500°F main burner inlet hot gas line from the preburner assembly to the injector main burner is a helical coil expansion loop made from Incoloy 800 material. The salient design features are that it minimizes the fuel flow pressure drop, limits the thermal expansion load into the injector to 200 lb, and is flanged at the steam generator exit to facilitate assembly and disassembly of the preburner assembly. Incoloy 800 was selected as the line material because of its good high-temperature strength, corrosion (steam) resistance, and hydrogen compatibility.

Fabrication of the service lines was contracted to an outside machine shop and is expected to be completed during April.

(7) GC Sample Line Assembly

The GC sample line assembly was designed to permit sampling the reactor gases near the injector face. It consists of a 1/8-in.-OD sample tube line attached to the reactor tube (Figure 3); a 7-in.-long N₂/hot gas tube-tube heat exchanger to cool the sampled gas from about 2000°F to about 1000°F before exiting the reactor pressure vessel shell; and the 1/4-in.-OD N₂ coolant line. Both lines contain expansion loops necessary to limit the thermal expansion stresses to allowable levels.

The sample line assembly will be employed during the planned preburner assembly evaluation tests, and prior to coal flow during the hydrogasification tests, in order to evaluate the performance of the preburner assembly and injector main burner.

Fabrication of this assembly has been completed.

2. Task VII-B--Facility Modifications

Rockwell's 1-ton/h coal hydroliquefaction PDU is being modified to accommodate reactor testing under operating conditions characteristic of flash hydrogasification. This PDU modification effort is divided into two work packages: facility modifications and process systems modifications. Facility modifications consist primarily of modifications to the facility structure necessary to accommodate testing with the nominal 10- and 20-ft-long reactor trains and to incorporate provisions for locating an on-line GC adjacent to the PDU in a separate existing structure.

Design of the facility modifications was completed as part of Task VI of this contract. The facility modifications work is being performed by a subcontractor to Rockwell, namely, Southern Mechanical, Inc., of Long Beach, California.

This section of the report contains a description of the 1-ton/h liquefaction PDU followed by a discussion of the facility modifications being performed to convert the PDU for use on the current program.

a. Description of 1-ton/h Liquefaction PDU

Figure 14 is a simplified schematic of the 1-ton/h hydroliquefaction PDU prior to its modification as part of this contract work. Pulverized feedstock

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Figure 14. Simplified Schematic of 1-ton/h Coal Hydroliquefaction PDU

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(coal, peat, oil shale, etc.) is fed from the high-pressure feeder in dense-phase flow using nitrogen as the pressurant/carrier gas. Hydrogen for the reactor is heated initially by a gas-fired heat exchanger to about 1200°F and then by partial combustion with oxygen, as necessary, to supply the required process heat. The feedstock is thoroughly mixed with hydrogen using a rocket-engine-type injector and is then permitted to react for the desired reactor residence time. The reactions are quenched at the reactor exit by cooling the reaction products to the desired temperature (600 to 1000°F) using a direct water spray quench. During earlier coal liquefaction testing,^{7,16} Rockwell also successfully employed an indirect heat exchange quench unit.

Three stages of char/vapor separation are included: an initial gravity-settling separation within the main char receiver, followed by two cyclone separators in series.

In the heavy-oil condenser, a high-pressure shell-and-tube heat exchanger, the reaction products are cooled from the reactor exit quench temperature to about 425°F by heat exchange against 400°F boiling water (which is maintained by a pressurized circulating hot-water system). Steam generated in the hot-water system is condensed by heat exchange with reclaimed facility water. The heavy-oil condensate is recovered by means of a downstream vapor/liquid separator, and liquid product is flowed periodically through a pressure letdown valve into a flash drum. The product (heavy oil) is removed from the system after each test.

The vapor/phase material is further cooled and condensed in a second shell-and-tube condenser to about 70°F by heat exchange against ambient temperature water. The condensate is recovered in a vapor/liquid separator and flows through an accumulator, a pressure letdown valve, a flash drum, and into a light-oil/water decanter. The water in the product gases also is condensed in this light-oil condenser. The light-oil/water mixture is removed from the decanter after each test.

The product gases from the light-oil vapor/liquid separator pass through a pressure letdown valve, a carbon bed benzene-toluene-xylene (BTX) adsorber, and a venturi flowmeter and then past a gas-sampling station into a scrubber system before being vented to a flare stack. The BTX adsorption, product gas flowmeter, and gas-sampling portions of the system are operated at ~60 psig.

The intermediate temperature between the two condensers (~425°F) was chosen to: (1) permit a sufficiently high wall temperature in the heavy-oil unit to keep tarry materials fluid and (2) avoid water condensation with the heavy oil because it would flash off when the pressure is reduced.

The activated carbon-bed adsorber recovery system was installed between the two pressure letdown valves to recover vapor phase BTX and other light hydrocarbon oils in the product gas stream leaving the light-oil condenser. A significant fraction of BTX formed in the reactor can remain in the vapor phase downstream from the light-oil condenser because its molar concentration is so low that it cannot be recovered by further cooling the gas stream. The loss would be significant even if the gases were cooled to the freezing temperature of benzene (42°F).

Alternative means of BTX recovery include absorption in a liquid solvent or adsorption on a solids adsorbent bed. Adsorption was selected because it could be implemented more easily in moderately short runs of the 1-ton/h PDU system with fewer potential complications and uncertainties. During a reactor test, product gas is passed through the carbon bed to adsorb the BTX. After the test, steam is passed through the bed to desorb the BTX, whereupon the BTX and steam are condensed and separated by decantation. A conventional propane-fired packaged boiler is used to generate the steam required for stripping.

Incomplete char recovery was determined to be a problem in early PDU testing; however, this problem has been resolved and excellent material balance

closure has been obtained on subsequent programs.^{7,17} Char is collected primarily in the main char receiver. In order to obtain good material balances in the existing PDU, special emphasis is placed on complete recovery of the char from the system. Great care is taken in its removal and precautions include the use of wind barriers to prevent air currents from blowing the char and postponement of char removal under windy conditions. A grating under the main char receiver is covered with aluminum sheets to catch and recover any material dropped.

b. Modified PDU System

The status of the facility modifications effort is presented in Figure 15. A facility modifications subcontract was awarded to Southern Mechanical, Inc., on January 24, 1984. Offsite material procurement, preparation of detailed structural and piping modification drawings, and fabrication of the structural steel and piping component parts were initiated during the first week of February. Onsite work was initiated on February 27, 1984. This task (VII-B) is currently approximately 80% complete, slightly ahead of schedule, and is expected to be completed by the end of April.

The PDU is located at Rockwell's Santa Susana Field Laboratory (SSFL) at the Energy Bowl Area on the VTS-II test stand. The facility modifications include:

- Structurally modifying the test stand to accommodate the test hardware assemblies.
- Installing two new work platforms and ladders for use in servicing the 10- and 20-ft-long reactor trains.
- Installing a new 2-ton jib crane required for installation of the reactor trains.
- Modifying the Firex (fire-fighting) system to protect the top end of the reactor trains.

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Figure 15. Facility Modifications Status (TASK VII-B)

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- Piping modifications to bypass the heavy-oil condenser and upgrade the BTX recovery system.
- Installing an existing flue (vent) stack and associated product gas line to the vent stack.
- Relocation of a 600-gal water tank for a hot water (steam) system.
- Upgrading a building to house the on-line GC.
- Installing the on-line GC system.
- Installing an on-stand instrumentation and/or breathing air system.
- Installing additional electrical power outlets and lighting.
- Installing a communications system between the Control Center and the building housing the on-line GC.

An existing 8-in.-thick concrete structure located adjacent to the VTS-II test stand is being used to house the GC. The GC unit to be used was procured on the subject contract under Task II and was located at the Control Center in the Energy Bowl Area. The subcontractor has relocated the GC unit within the concrete structure, installed most of the support equipment, and prepared the building to house personnel during the tests. This has included relocation of an air compressor system, two GC analyzer units, GC ovens, and the GC controller console. A new air conditioner (heat pump) and communications system between the GC building and the test area Control Center are in the process of being installed. Two personnel will be located inside the GC building to operate the GC during each test. The air compressor will provide both breathing/instrument air and shop air.

Two trace-heated GC sample lines with associated sample boxes containing filters and valves are being installed. Autotransformers and temperature indicators (to read thermocouple outputs) are being installed in the GC building to allow the GC operator to remotely control and monitor the temperature of the GC sample line filters, valves, and lines. The on-line GC system has

been designed to permit sampling at two locations. Sample point 1 will be used to sample the feed gas mixture shortly after it is injected into the reactor tube. Sample point 2, located just upstream from the light oil condenser, will be used to sample the process effluent gases. Details of the on-line GC system are described later in the Task VII-C discussion.

The planned test program is expected to yield results showing high concentrations of water vapor in the reactor exit gas stream. Since the existing heavy oil condenser on the PDU operates at an outlet temperature of $\sim 425^{\circ}\text{F}$ at 1000 psia, substantial amounts of water vapor would be condensed and collected in the 400°F traced heated heavy oil separation and collection system. Because the capacity of the high-pressure heavy oil separation vessel is small (~ 15 gal), the vessel would need to be emptied periodically during a test to the low-pressure (~ 60 psig) heavy oil flash drum. This would result in substantial flashing off of the water and its subsequent recondensation in the BTX adsorber system. This can be avoided by bypassing the heavy oil condenser. Consequently, the heavy oil condenser, shown in the schematic of the existing liquefaction PDU (Figure 14), will not be used during the upcoming test program. Instead, the reactor product stream will be routed from the second cyclone directly to the light oil condenser. The light oil condenser will therefore be operated with an inlet stream temperature of about 800°F and an outlet product temperature between 60 and 80°F .

Modifications are also being made to the BTX recovery system to improve its operation. This includes removal of a nonfunctional adsorber vessel, rerouting of piping, and installation of two valves. The piping changes and valve additions should simplify the post-test BTX stream stripping procedure and improve the physical recovery of liquid BTX by eliminating locations where BTX may be lost due to condensation within system piping.

A 600-gal water tank is being relocated from near the test area Control Center to an area just adjacent to the test stand to provide a hot water supply to be used for steam generation. An existing pump and electrical heater, which were part of the heavy oil condenser system, will be employed to

preheat the water prior to each test. This system will be insulated to minimize heat losses.

Modification of the PDU to accommodate testing with the 10- and 20-ft-long hydrogasifier reactor trains actually began with removal of a large (45,000 gal) liquid hydrogen (LH₂) tank from the PDU facility during the summer of 1983. This nonfunctional tank remained from Rockwell's previous rocket engine testing on VTS-II. Its removal from the facility and disposal as inoperable/obsolete excess government property was accomplished as part of the close-out of DOE Contract DE-AC22-80PC30018. The location of this tank had previously limited the use of the VTS-II test stand; its removal facilitated conversion of the liquefaction PDU to a hydrogasifier PDU by allowing for longer residence time reactors.

A photograph of the liquefaction PDU taken prior to removal of the LH₂ run tank is presented in the upper portion of Figure 16. This photograph was taken from the test area control center. The bottom portion of Figure 16 presents a more recent view of the PDU facility. This photograph was taken just prior to the start of the Task VII-B facility modifications effort. Removal of the LH₂ run tank, Colby gantry crane, and the flare stack from the PDU is readily evident from these photographs.

The upper deck of the test stand, which was attached to the LH₂ tank, was removed with the tank. The flare stack, which was attached to the upper deck, has been reinstalled on the PDU as part of the Task VII-B effort. Gas supply lines to the coal feeder, flare stack, and various valve control/actuation systems were also removed as part of the LH₂ tank removal. These gas supply systems are being replaced as part of the Task VII-C effort.

Several photographs of the PDU taken just prior to initiation of Task VII-B in January and in early April are presented in Figures 17 and 18 to highlight progress on the facility modifications effort.

a. PDU PRIOR TO LH₂ TANK REMOVAL

b. PDU AFTER LH₂ TANK REMOVAL

Figure 16. Photographs of PDU Prior to and after
LH₂ Tank Removal

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Figure 17. Photographs of PDU Illustrating Facility Modification Progress - South View

b. PDU STATUS IN EARLY APRIL

a. PDU PRIOR TO START OF FACILITY MODIFICATIONS (TASK VII-B)

a. PDU PRIOR TO START OF FACILITY MODIFICATIONS (TASK VII-B)

b. PDU STATUS IN EARLY APRIL

Figure 18. Photographs of PDU Illustrating Facility Modification Progress - West View

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Figure 17a/b shows the installation of the flare stack, the two new work platforms for servicing the 10- and 20-ft-long reactor trains, the modifications made to the BTX recovery system plumbing, and the new tubing installed to supply gases to the upper portion of the test stand. Note that the old hoist system for servicing the reactor system has been removed. Figure 18a/b shows the installation of the hot water (steam) system tank, flare stack, the location for the new jib crane, the trace-heated GC sample lines, GC sample boxes, the GC building, and the roof over the air compressor system. Note that grating has been installed on the upper deck level of the test stand. The new, larger jib crane will be installed in the same general location as the previous unit.

A simplified schematic of the newly modified coal hydrogasification PDU is presented in Figure 19. This figure depicts the system configuration once all of the facility and process system modifications have been completed.

3. Task VII-C--Process Systems Modifications

The design of the process systems modifications required to convert the hydroliquefaction PDU for use in flash hydrogasification was completed as part of Task VI of this contract. The status of the process systems modification effort is summarized in Figure 20. This task includes: (1) preparation of the PDU for subsequent subcontractor facility modifications (Task VII-B), (2) system component procurement and servicing, (3) detailed layout and installation of the instrumentation, controls, and data acquisition systems (DAS), (4) updating of system test procedures, (5) installation and/or modification of various feed systems, (6) installation of an on-line gas chromatograph (GC) sampling system, and (7) assembly and installation of the reactor into the PDU.

During this report period, the PDU was prepared for facility modifications, new system components were procured and servicing of existing system components initiated, and detailed layout of the instrumentation, controls,

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Figure 19. Simplified Schematic of Coal Hydrogasification PDU

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Figure 20. Process Systems Modification Status (Task VII-C)

and DAS systems were completed. Several of the new system components (e.g., venturi meters, ΔP transducers, etc.) were ordered early in the program in order to be available for the subsequent activation and test efforts. Servicing of system components is being performed by the instrumentation and equipment laboratories of Rockwell's Rocketdyne Division. As noted in Figure 20, work has been initiated on installation and/or modification of several of the feed systems (O_2 , CH_4 , and CO), updating of the test procedures, and installation of the instrumentation, controls, and DAS systems. The process systems modifications effort is slightly behind schedule; however, this work will be accelerated during April and May to meet the program schedule. The majority of the work on the process systems modifications is scheduled to be accomplished in the April-June time period after the subcontractor has completed most of the facility modifications.

a. Data Acquisition System

The data acquisition system (DAS) consists of a Hewlett-Packard (HP) 1000 Model F central computer processing unit, a 20 M byte hard disc, two magnetic tape drives, three HP Model 2250 measurement and control units (MCU), a 400-line/min printer, a printing terminal, four CRT terminals, and a TV display monitor.

One of the MCUs is capable of accepting up to 352 analog signal inputs. The planned testing will use approximately 125 channels. All channels are scanned five times per second with the average value stored on disc and tape. The disc can store up to 3.6 h of data and each reel of tape about 8 h.

Data acquired during a test may be displayed in several forms. The typical configuration is as follows:

- 40 parameters in tabular form at a CRT terminal
- 4 trend graphs at a CRT terminal
- Call-up of any parameter at a CRT terminal
- 12 parameters in tabular form at the TV monitor.

All displays are concurrent and are updated every 3 s. Values calculated from one or more measured parameters may also be displayed.

Alarm levels (both low and high) may be set for up to 40 parameters (including calculated values). If an alarm level is exceeded, a message appears on the printing terminal indicating the time, the parameter name, and whether it was a low or high alarm. If the parameter is one of the 40 in the tabular display, the displayed value will be in inverse video.

Up to 19 measured parameters may be monitored for test shutdown ("CUT") conditions. These parameters are scanned by a second MCU at a rate of once every 40 ms. If a CUT level is exceeded, an additional scan is made to verify the reading. If both scans are above the CUT level, a relay output from the MCU will initiate test shutdown. Some interlock checks are also controlled by this MCU.

Measured and calculated values may be retrieved following a test from either disc or tape. Data may be printed out for any or all channels in tabular form as a function of time. Data plots may also be obtained for up to five channels plotted as a function of time or another chosen parameter.

b. System Plumbing and Instrumentation

A simplified plumbing and instrumentation schematic of the modified PDU system is presented in Figure 21. Systems that are being added or modified are highlighted. Table 2 is a listing of the parameters to be monitored (recorded) by subsystem, keyed to Figure 21.

Additionally, the following injector/reactor assembly parameters will also be monitored during a test but are not shown on the flow diagram:

- Preburner assembly coolant-water temperature (seven locations)
- Redundant (two) steam generator exit-gas temperatures
- Steam generator exit-pipe wall temperature (four locations)

Figure 21. Plumbing and Instrumentation Schematic of Hydrogasification PDU

TABLE 2
INSTRUMENTATION LIST FOR COAL HYDROGASIFICATION PDU TESTING
(Sheet 1 of 3)

Flow Diagram Instrument Number	Measured Parameter
<u>Steam Generator Hydrogen System</u>	
H-3	Venturi meter upstream pressure
H-4	Venturi meter differential pressure
H-5	Venturi meter upstream temperature
H-9	Injection pressure
<u>Main Hydrogen System (to H₂/CH₄/O₂ Preburner)</u>	
138	Supply pressure
139	Venturi meter upstream pressure
169	Venturi meter differential pressure (No. 1)
170	Venturi meter differential pressure (No. 2)
210	Venturi meter upstream temperature
187	Hydrogen/methane heater exit gas temperature
134	H ₂ /CH ₄ injection pressure
186	H ₂ /CH ₄ injection temperature
<u>Hydrogen/Methane Heater System</u>	
143	Heater burner supply pressure
144	Heater burner main valve pressure
201	Heater stack gas temperature
202	Heater burner igniter temperature
214	Heater tube wall temperature
216	Heater housing outside wall temperature
<u>Methane System</u>	
M-5	Supply pressure
M-6	Supply temperature
M-10	Venturi meter upstream pressure
M-11	Venturi meter upstream temperature
M-12	Venturi meter differential pressure
M-16	H ₂ /CH ₄ heater upstream pressure
<u>Carbon Monoxide System</u>	
C-5	Supply pressure
C-9	Venturi meter upstream pressure
C-10	Venturi meter differential pressure
C-11	Venturi meter upstream temperature
C-15	Injection pressure

TABLE 2
INSTRUMENTATION LIST FOR COAL HYDROGASIFICATION PDU TESTING
(Sheet 2 of 3)

Flow Diagram Instrument Number	Measured Parameter
<u>Oxygen System (Preburner)</u>	
140	Venturi meter upstream pressure
165	Venturi meter differential pressure
211	Venturi meter upstream temperature
148	Injection pressure
<u>Oxygen System (Main Burner)</u>	
OX-3	Venturi meter upstream pressure
OX-4	Venturi meter differential pressure
OX-5	Venturi meter upstream temperature
OX-13	Injection pressure
<u>Coal Feeder System</u>	
129	Feeder top pressurizing supply pressure
130	Feeder bottom pressurizing supply pressure
132	Feeder tank pressure
<u>Water System (Hardware Coolant/Quench)</u>	
145	Tank pressure
178	Injector coolant flow rate
206	Injector coolant inlet temperature
207	Injector coolant outlet temperature
208	Injector coolant outlet pressure
CW-3	Preburner coolant flow rate
CW-4	Preburner coolant inlet pressure
CW-5	Preburner coolant inlet temperature
CW-6	Preburner coolant outlet temperature
CW-7	Preburner coolant outlet pressure
180	Water spray quench flow rate
<u>Hot Water (Steam) System</u>	
S-5	Tank pressure
S-14	Water flow rate
S-15	Water temperature
S-19	Injection pressure

TABLE 2
INSTRUMENTATION LIST FOR COAL HYDROGASIFICATION PDU TESTING
(Sheet 3 of 3)

Flow Diagram Instrument Number	Measured Parameter
<u>Reactor and Product Recovery System</u>	
136	Reactor pressure
204	Reactor pressure vessel external wall temperature (-1)
215	Reactor pressure vessel external wall temperature (-2)
212	Quench vessel external wall temperature
217	Quench vessel exit gas temperature (-1)
226	Quench vessel exit gas temperature (-2)
149	Quench vessel exit gas pressure
213	Char receiver external wall temperature
133	Char receiver exit gas pressure
198	Char receiver exit gas temperature
224	Cyclone No. 1 inlet gas temperature
131	Cyclone No. 2 inlet gas pressure
152	Condenser inlet gas pressure
226	Condenser inlet gas temperature
208	Condenser coolant water outlet temperature
153	Condenser exit gas pressure
218	Condenser exit gas temperature
155	Flash drum pressure
175	Oil/water decanter weight
151	BTX adsorber inlet gas pressure
205	Product gas venturi meter upstream temperature
147	Product gas venturi meter upstream pressure
166	Product gas venturi meter differential pressure (-1)
167	Product gas venturi meter differential pressure (-2)

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- Preburner assembly exit pressure
- Reactor pressure
- Reactor tube-wall temperature (eight locations)
- Redundant (two) reactor exit-gas temperatures.

c. Subsystem Descriptions

The major systems being added or modified as part of the process systems modifications package are summarized below:

- Hydrogen--System modified to provide for dual but separate injection of hydrogen to two stages of the preburner assembly.
- Oxygen--System modified to provide for dual but separate injection of oxygen to the preburner and the main injector burner.
- Methane--Existing system upgraded to provide the methane to be mixed with the main hydrogen stream (and fed to the preburner injector).
- Carbon monoxide--New system installed to provide the CO to be injected.
- Water--Existing hardware coolant and/or quench water system modified to provide coolant water for the preburner.
- Water (Steam)--New system installed to provide heated water to be injected into the preburner assembly to generate the required process steam.
- On-line gas chromatograph--System installed to provide real time analysis of the injected gas mixture and/or product gases.

Design of the process system modifications was based on many factors, including the reactants to be used, planned test matrix flow rates, reactor operating conditions, data reliability, system operability, and system integrity and safety. Accordingly, process system components and lines were sized to handle the wide range of reactant and product flow rates expected in the planned test program. Gas flow rate measurements will be made using calibrated venturi or orifice meters in conjunction with calibrated pressure and differential pressure transducers. These components have been sized to cover as

wide a flow rate range as possible consistent with obtaining reliable data. Water flow rate measurements will be made using calibrated turbine-type flowmeters.

The process systems modifications are being performed by Rockwell personnel. The new or modified systems are described below.

(1) Hydrogen System

The existing hydrogen feed system will be modified by the addition of a parallel system that will allow ambient temperature hydrogen to be injected into the steam generator portion of the preburner assembly. The existing or main hydrogen system will supply hydrogen (sometimes blended with methane) through the gas-fired heater (Figure 19) to the top of the preburner assembly. The new parallel system will be smaller (1/2-in. tubing and components) than the existing system (1-in. tubing and components) but will contain all essential components, such as a pressure regulator, venturi flowmeter, pressure and differential pressure transducers, thermocouples, shutoff valve, and check valve. Several venturi flowmeters with different throat diameters will be used to span the expected flow rate range. This system has been designed so that it may be operated at pressures up to the supply system maximum pressure (~2750 psig).

Hydrogen will be delivered via pipeline to the test area from Rockwell's adjacent Rocketdyne Division storage area.

(2) Oxygen System

Oxygen is required for both the preburner assembly and injector coaxial elements. An existing system is currently being modified to provide the two oxygen injection systems from a common supply source. An oxygen feed system existed for the preburner, but this system has been completely disassembled and is being reinstalled at the new preburner location. A new parallel feed system is being installed to supply oxygen to the injector coaxial elements.

This 1/2-in. system, which is being installed complete with essential components (Figure 21 and Table 2), is designed to operate at pressures up to the maximum supply pressure (~2500 psig).

Commercially available oxygen demisters will be used for the oxygen supply.

(3) Methane System

An existing methane feed system is being upgraded and reactivated to supply the methane to be blended with the main hydrogen stream. This stream will then pass through the gas-fired heater and to the preburner (Figure 21). A methane feed system was used previously on this facility during a prior coal hydroliquefaction program⁷ but was removed to allow testing on a recent oil shale flash hydroretorting program.¹⁷

Most of the components for this system (valves, pressure regulator, heat exchangers, heaters, etc.) are still available to be reinstalled; however, some system changes are necessary to satisfy the requirements of the planned hydrogasifier testing. Two heat exchangers will be used to maintain the methane temperature at ~80°F during its cooling due to expansion. Past experience has shown that flow variations can occur unless the methane temperature is maintained above 50 to 60°F during its expansion. In addition to the heaters and heat exchangers, other major feed-system components are a pressure regulator, filter, venturi meter, pressure and differential pressure transducers, thermocouples, a main shut-off valve, and a check valve (Figure 21). The system has been designed for operation at pressures up to 2500 psig.

Methane from a 110,000-scf tube trailer will be used for the testing.

(4) Carbon Monoxide System

A new carbon monoxide feed system is currently being installed on the PDU. The ambient temperature carbon monoxide will be injected immediately

downstream from the preburner assembly. The feed system for carbon monoxide is shown schematically in Figure 21. The CO will be procured and stored in a 45,000-scf tube trailer. From here, it will be conveyed through 1/2-in. OD tubing and components to its injection point immediately downstream from the preburner. This system, designed with a pressure regulation and flow measurement system similar to the other gaseous feed systems, will operate at pressures up to 2000 psig, the CO supply pressure.

(5) Hardware Coolant/Quench Water System

A system has been designed to supply ambient-temperature water to the preburner assembly cooling jacket. This system will be installed in parallel with existing systems used for injector coolant and reaction spray quench (Figure 21). Soft water for this system will be supplied from a 2500-gal, 3000-psi tank. The water flow rate will be regulated by selecting the proper tank pressure and orifice size for the desired coolant water flow rate. Since heat losses in the preburner are of interest, the system has been designed with a turbine flowmeter and thermocouples appropriately positioned along the preburner coolant water path. Because the preburner water jacket coolant passage dimensions are small, a filter has been included in the system to prevent plugging. This system is designed to operate at pressures up to 2000 psig.

(6) Hot Water (Steam) System

Process steam will be generated in situ by the vaporization in the preburner assembly of water injected at $\sim 200^{\circ}\text{F}$. An entirely new water feed system has been designed and is currently being installed for this purpose (Figure 21). It includes a 600-gal, 2000-psi tank; nitrogen pressurizing and venting systems; water flow measurements and control systems; and a water heating system. The flow measurement and control system includes a turbine meter, shutoff valve, and throttling valve. To keep from plugging the water injection orifices in the preburner, a filter unit has been included in the system.

The water will be heated by an electric heater and recirculation pump previously used on the coal hydroliquefaction programs to control the operating temperature of the heavy oil condenser. New insulated piping has been installed to connect the pump and heater to the insulated 600-gal tank. Two isolation valves have been installed to separate the tank (high-pressure system) from the pump and heater (low-pressure system). Before each test run, the water will be preheated to 200 to 225°F; then the heater and pump will be turned off and the isolation valves closed. The tank can then be pressurized to the desired test operating conditions. This system has been designed to operate at pressures up to 2000 psig.

(7) On-Line GC System

An on-line GC system has been designed to provide for real-time analysis of the gas mixtures injected into and exiting from the reactor. The basic GC units to be used were procured to support ETU hydrogasifier testing under Task II of this contract.³ Since the measurements made previously are similar to those desired for the planned hydrogasification testing, Rockwell had the majority of the components required for the on-line GC sampling system. The on-line GC system has been relocated from the test area Control Center to a structure adjacent to the PDU.

The on-line GC system has been designed to permit sampling at two locations (Figure 19). Sample point 1 will be used to sample the feed gas mixture shortly after it is injected into the reactor tube. Sample point 2, located just upstream from the light oil condenser, will be used to sample the process effluent gases.

On-line gas sampling just downstream from the injector face (sample point 1) during preburner evaluation tests will allow direct analysis for H_2 , CO_2 , C_2H_4 , C_2H_6 , O_2 , N_2 , CH_4 , and CO in the gas phase every 4 min. During the full-scale coal hydrogasifier tests, this sample point will be used to analyze the injected gas mixture from startup until just before the coal

feed commences. A sample line assembly has been designed to rapidly cool samples taken from sample point 1. The samples will be cooled from about 2000°F to 1000°F in approximately 10 ms to freeze the stream composition.

On-line gas sampling upstream from the light oil condenser (sample point 2) will allow direct analysis for the same components listed above plus the determination of benzene and toluene. The benzene-toluene (BT) analysis at this point will permit a direct measurement of the total BT produced. These analyses can be repeated every 4 min.

A flow diagram of the GC (Daniel Industries custom-built H-1000) and associated sampling systems are shown in Figure 22. The flow diagram for the carrier and calibration gases is shown in Figure 23. Included on this figure is the compressed air plumbing, which is used for valve actuation and heating of the GC and sample system ovens. The chromatographic columns and associated parameters are listed in Table 3. Unit A contains two separate chromatograph systems. The first uses nitrogen as the carrier gas and a two-column arrangement that only determines hydrogen. The second system uses helium as the carrier gas and a three-column arrangement that determines CO_2 , C_2H_4 , C_2H_6 , O_2 , N_2 , CH_4 , and CO . Water and hydrocarbons higher than C_2 's are back-flushed off of the first column. Unit B uses helium as the carrier gas and a two-column arrangement to determine the benzene and toluene content of the stream.

For analysis from either sample point, a small portion of the process stream ($\sim 0.1\%$) will be diverted through one of two parallel Balston solid-particle filters. An orifice will drop the pressure to approximately 100 psi at a flow of approximately 50 liters/min (dry at STP). The sample flow will be maintained at this level to minimize lag time to the analyzer(s). Should the flow rate drop significantly due to increased resistance through the filter, flow will be diverted through the second Balston filter. The sample lines from the sample points on the PDU to the GC units are trace heated (350°F) to keep the water vapor from condensing. The sample will flow through these heated stainless steel 1/4-in.-OD tubes into the first sample system

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Figure 22. Sample Systems for the H-1000 Gas Chromatographs

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Figure 23. Calibration Gases, Carrier Gases, and Compressed Air Plumbing for Gas Chromatographs

TABLE 3
DETAIL OF GAS CHROMATOGRAPH UNITS

	GC Unit A		GC Unit B
Instrument	Daniel Ind. H-1000		Daniel Ind. H-1000
Measured output	Peak height		Peak height
Analysis frequency	4 min		4 min
	<u>H₂ analyzer</u>	<u>Light gas analyzer</u>	<u>Benzene-toluene analyzer</u>
Carrier gas	H ₂	He	He
Detector	Thermal conductivity	Thermal conductivity	Thermal conductivity
Automatic valving	Gas sampling with backflush	Gas sampling with backflush	Gas sampling with backflush
Columns used	Porapak T (backflush H ₂ O) Porapak Q: H ₂	Porapak T (backflush H ₂ O, C ₃ , and up) Porapak Q: CO ₂ , C ₂ H ₄ , C ₂ H ₆ Molecular sieve 5A: O ₂ , N ₂ , CO, & CH ₄	Carbowax 20M (backflush H ₂ O and heavies) ² TCEP: benzene, toluene

oven, located under GC unit B. Before this oven, the line tees off to a back-pressure regulator that maintains the line pressure below 100 psig. The oven, maintained at 300°F, contains two parallel Balston coalescing filters. After passing through the coalescing filter, the gas sample will enter the second sample system oven (300°F) under GC unit A where a small portion (~1 liter/min) will be filtered through a Balston bypass filter and regulated down to 10 psig. The bulk of the sample gas will bypass the filter, exit through the bypass line, and vent to outside the building. The small 10-psig sample will be split into several streams and routed to the GC columns.

For the preburner evaluation tests, one stream will enter GC unit A and be analyzed for H₂ only. A second stream will also enter GC unit A and be analyzed for the remainder of the light gases listed above. For coal tests (sample point 2), a third portion of the stream will also pass through a heat-traced line into GC unit B, where it will be analyzed for benzene and toluene.

Hydrogen sulfide (H₂S) and ammonia (NH₃) will be determined in the gas sample stream on selected tests. Aqueous scrubber samples will be taken from the bypass line through valve 32 (Figure 22). The sample line will be split into two streams, one for each analysis. Each stream will consist of two 250-ml glass scrubbers, a front and back, and a wet test meter. The samples will be flowed at 1 to 2 liters/min for the duration of the test. The H₂S scrubbers will use 0.1N sodium hydroxide solution, while the NH₃ scrubbers will use 0.1N sulfuric acid solution. After the test, appropriate measures to preserve the H₂S and NH₃ will be taken, and the solutions will be transported to the laboratory for chemical analysis.

VIII--PERFORMANCE TESTING

B. TASK VIII--PERFORMANCE TESTING

Subtasks VIII-A, System Activation, and VIII-B, Testing, were not active during this report period. The test planning portion of Subtask VIII-C is in progress. The status of test planning activities is summarized in Figure 24.

▽ TEST PROGRAM PLAN AND SCHEDULE TO BE SUBMITTED TO DOE

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Figure 24. Test Planning Status (Task VIII-C)

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An initial, preliminary test matrix has been defined, preparation of a data analysis plan is underway, and data reduction programs are being programmed. The test matrix will be part of the test program plan and schedule to be submitted to DOE for review and approval. Rockwell plans to submit this document to DOE in early May. The data analysis plan will describe planned sampling, the chemical analyses to be performed, system measurements to be made, and how this information will be utilized to characterize reactor system performance. The request for test is a Rockwell internal document that defines for Test Operations personnel the test requirements needed to accomplish the program objectives.

1. Preliminary Test Matrix

A preliminary test matrix, designed to yield quantitative data addressing the key questions concerning reactor chemistry and to support subsequent preliminary process studies, is outlined in Table 4. All tests, except where noted, will be carried out at a reactor pressure of 1000 psia and 0.2 lb-moles of reactor gas fed per pound of coal. Specific objectives of the test program are to:

- Check out the operation and performance of the preburner and steam generator subsystems;
- Determine baseline product slates under hydrogasification conditions at high reactor exit temperatures and long residence times;
- Determine the extent to which steam in the reactor reacts with char to produce carbon oxides. Similarly, to determine whether steam affects the hydrocracking of vapor-phase liquid hydrocarbons;
- Determine the effects of temperature and reactor gas composition on the product slate;
- Determine the effects of carbon oxides generated in the preburner system by partial oxidation of methane on the reactor chemistry and product slate;

TABLE 4
PRELIMINARY TEST MATRIX FOR ADVANCEMENT OF FLASH HYDROGASIFICATION
(Sheet 1 of 2)

Test Series	Test No.	Reactor Length	Reactor Exit Temperature	Steam Mole Percent	Methane Addition	CO Addition
A	<u>Preburner Performance Characterization/Checkout</u>					No Variable
	PB-1 PB-2	20 ft 20 ft	Variable Variable	Variable Variable	No Variable	
A	<u>Facility Checkout at Baseline Conditions</u>					No No
	R-1 R-2	20 ft 20 ft	1800°F 1950°F	Minimum Minimum	No No	
A	<u>Investigate Steam Enhancement</u>					No No
	R-3	20 ft	1950°F	~45%	No	
Based on this test, select test series B or C						
Test Series B - Significant steam/char reaction in Test R-3						
Test Series C - Minimal steam/char reaction in Test R-3						
B	<u>Significant Steam Enhancement in Test R-3</u>					No No No No No Selected No No No No No No No Selected
	R-4	20 ft	1950°F	~30%	No	
	R-5	20 ft	1800°F	~45%	No	
	R-6	20 ft	1800°F	~30%	No	
	R-7	20 ft	Selected	Selected	Low	
	R-8	20 ft	Selected	Selected	High	
	R-9	20 ft	Selected	Selected	Selected	
	R-10	10 ft	1950°F	~45%	No	
	R-11	10 ft	1950°F	~30%	No	
	R-12	10 ft	1800°F	~45%	No	
	R-13	10 ft	1800°F	~30%	No	
	R-14	10 ft	Selected	Selected	Low	
	R-15	10 ft	Selected	Selected	High	
	R-16	10 ft	Selected	Selected	Selected	

TABLE 4
PRELIMINARY TEST MATRIX FOR ADVANCEMENT OF FLASH HYDROGASIFICATION
(Sheet 2 of 2)

Test Series	Test No.	Reactor Length	Reactor Exit Temperature	Steam Mole Percent	Methane Addition	CO Addition
C		Minimal Steam Enhancement in Test R-3				
	R-4	20 ft	Maximum	Maximum	No	No
	R-5	20 ft	Maximum	Maximum	No	No
			(Maximum Reactor Pressure)			
	R-6*	20 ft	Maximum	Maximum	No	No
			(Maximum Pressure/Maximum Main Burner Temperature/Minimum Gas Flow)			
	R-7	20 ft	1950°F	Maximum	Low	No
	R-8	20 ft	1950°F	Maximum	High	No
	R-9	20 ft	1800°F	Maximum	Low	No
	R-10	20 ft	1800°F	Maximum	High	No
	R-11	20 ft	Selected	Selected	Selected	No
	R-12	20 ft	Selected	Selected	Selected	Selected
	R-13	10 ft	1950°F	Maximum	Low	No
	R-14	10 ft	1950°F	Maximum	High	No
	R-15	10 ft	1800°F	Maximum	Selected	No
	R-16	10 ft	Selected	Selected	Selected	Selected

*If significant steam/char reaction is not obtained, modified/alternate hardware may be employed to increase the maximum reactor exit temperature.

- Acquire credible material balances and obtain quantitative data on the coal elemental conversions and the slate of hydrocarbon products obtained under suitable FHP reactor conditions; and
- Acquire data to increase METC's coal gasification data bank.

The preliminary test matrix comprises several series of tests, each of which addresses one or more of the above objectives. The test series are organized so that preburner and steam generator performance and reactor operability can be assessed early in the test program during system shakedown and checkout before full-scale tests are made with mixed gases fed to the reactor. Table 5 describes the objective and approach for each series in the preliminary test matrix.

2. Planned Sampling and Chemical Analyses

A preliminary sampling and chemical analysis plan, designed to yield quantitative data on the burner system performance and reactor product slate, is outlined in Table 6. The sample numbers referenced in Table 6 are shown in the facility schematic in Figure 25. The plan includes chemical analysis of all gas, liquid, and solid feeds and products. Provisions are made to determine burner system exit gas composition and coal characterization in order to accurately quantify reactor input feeds. Sampling and analysis of all gases, liquids, and solids downstream of the reactor will yield detailed material balances and reactor product slate along with coal carbon conversions and coal elemental conversions.

TABLE 5
 ADVANCEMENT OF FLASH HYDROGASIFICATION
 DESCRIPTION OF PRELIMINARY TEST MATRIX
 (Sheet 1 of 3)

Test	Objective and Approach
A/PB-1 A/PB-2	<p>The main objective of these tests is to check out the operation and performance of the preburner and steam generator subsystems. These tests will also serve as facility checkouts for the gas feed systems and gas systems downstream of the reactor. No coal will be fed to the reactor in these tests.</p> <p>Test A/PB-1 (no methane) will involve varying the preburner temperature, steam generator temperature, main burner temperature, and steam concentration by varying the hydrogen, oxygen, and water flow rates. This test will yield experience in controlling critical temperatures, check out the performance of the steam generator, and provide data on system heat losses.</p> <p>The A/PB-2 will involve methane addition to the preburner, and conditions will be varied over the range required for subsequent coal testing with methane addition. This test will check out the preburner performance and define the percent methane conversion in both the preburner and main burner together with the gas compositions under various conditions. Checkout of the CO feed system will also be conducted during Test A/PB-2.</p>
A/R-1 A/R-2	<p>The objectives of these tests are to check out facilities involving the coal feed system and to establish baseline carbon conversions at long residence times and high gas temperatures. For these tests, no methane will be added to the preburner, and the steam concentration will be maintained at the system's minimum value. Test A/R-1 has a reactor exit temperature similar to past FHP test conditions but with a longer residence time (i.e., about 6 s). Test A/R-2 will be conducted at a reactor exit temperature higher than past test conditions, and corresponds to the reactor exit temperature to be utilized for investigation of steam enhancement.</p>
A/R-3	<p>In this test, steam enhancement will be investigated. The planned reactor exit temperature will be approximately 1950°F, and the steam mole fraction at the reactor inlet will be approximately 45%. These conditions were selected such that (1) equilibrium conditions will not restrict the CO production in the reactor, and (2) the hydrogen partial pressure is high enough to insure liquid decomposition.</p>

TABLE 5
ADVANCEMENT OF FLASH HYDROGASIFICATION
DESCRIPTION OF PRELIMINARY TEST MATRIX
(Sheet 2 of 3)

Test	Objective and Approach
A/R-3 (cont.)	Depending on the results of this test, either test series B or test series C will be conducted. If results show enhanced overall carbon conversion in comparison with Test A/R-2, test series B will be conducted. If there is little evidence of reactivity of steam with char in this test, test series C will be conducted.
B/R-4 B/R-5 B/R-6	The objective of these tests is to determine the effect of reactor exit temperature and steam concentration on the steam/char/hydrocarbon reactions.
B/R-7 B/R-8	In these tests, the effect of methane on the steam enhancement reactions will be determined. The methane addition rate for Test B/R-8 will be such that hydrogen consumption is approximately balanced by CO production from methane combustion and steam/char/hydrocarbon reactions. Test B/R-7 will have approximately one-half that methane addition rate.
B/R-9	At present, this is an open test condition. This test could be a repeat of a previous test condition or any other selected condition based on the previous test results.
B/R-10 through B/R-16	The objective of these tests is to determine the effects of residence time. The conditions are similar to tests B/R-3 through B/R-9 but with a residence time of about one-half that of the previous test conditions.
C/R-4 C/R-5 C/R-6	Test series C will be conducted if there is little evidence of steam/char reaction in Test A/R-3. The objective of these tests will be to investigate the possibility of steam enhancement at more severe reactor conditions subject to test facility and hardware limitations. Test C/R-4 will be conducted at 1000 psia, maximum reactor exit temperature, and maximum steam concentration. Test C/R-5 is similar to C/R-4 but at maximum reactor operating pressure. Test C/R-6 conditions are the most severe, with pressure, residence time, and all temperatures at their maximum permissible levels. If significant steam/char reaction is not obtained in these three tests, modified/alternate hardware will be considered to increase the maximum reactor exit temperature.

TABLE 5
ADVANCEMENT OF FLASH HYDROGASIFICATION
DESCRIPTION OF PRELIMINARY TEST MATRIX
(Sheet 3 of 3)

Test	Objective and Approach
C/R-7 through C/R-10	The objective of these tests is to investigate the effects of methane addition and reactor exit temperature. The amount of steam will be maximized while maintaining enough hydrogen partial pressure to insure liquid decomposition. The higher amount of methane addition will correspond to that at which hydrogen consumption is approximately balanced by CO production from methane combustion.
C/R-11 C/R-12	At present, these are open test conditions. These tests could be repeats of previous test conditions or any other selected conditions based on the previous test results.
C/R-13 through C/R-16	The objective of these tests is to determine the effects of residence time. The conditions are similar to Tests C/R-7 through C/R-12 but with a residence time of about one-half that of the previous test conditions.

TABLE 6
ADVANCED FHP PROGRAM PLANNED SAMPLING AND CHEMICAL ANALYSES
(Sheet 1 of 3)

Sample	Frequency	Analyses
1. <u>Coal</u> - Kentucky No. 9 hvAb (90% <200 mesh)	- Material when received. - Each test.	- Moisture, C, H, and ash by ESG--each test. - Ultimate and proximate analysis by CTE*--each test. - Sieve analysis on <u>two</u> selected tests by ESG. - Free swelling index on <u>three</u> selected tests by CTE. - One each of above plus true and bulk density and ash fusion temperature on sample of material when received at ESG.
2. <u>Main Char Receiver</u> <u>(V-240) Char</u>	- Each test.	- Moisture, C, H, and ash by ESG--each test. - Ultimate and proximate analysis by CTE--each test. - Sieve analysis, true and bulk density on <u>two</u> selected tests by ESG.
3. <u>Cyclone Chars (No. 1</u> <u>and No. 2)</u>	- Each test.	- Moisture, C, H, and ash by ESG--each test--each cyclone - Sieve analysis, true and bulk density on <u>one</u> selected test by ESG--each cyclone

*CTE - Commercial Testing & Engineering (Chicago, Illinois)

TABLE 6
ADVANCED FHP PROGRAM PLANNED SAMPLING AND CHEMICAL ANALYSES
(Sheet 2 of 3)

Sample	Frequency	Analyses
4. <u>Injected Gas</u> (GC Sample Pt. No. 1-- in reactor just down- stream of injector)	<ul style="list-style-type: none"> - On preburner tests-- every ~4 min. - Pre-coal on reactor tests --every ~4 min. 	<ul style="list-style-type: none"> - For H₂, O₂, N₂, CO, CO₂, CH₄, C₂H₄, and C₂H₆ plus H₂O (by difference). - <u>On-Line GC Analysis</u>
5. <u>Product Gas</u> - Just upstream of condenser (GC Sample Pt. No. 2)	<ul style="list-style-type: none"> - Periodically during pre- burner tests. - Reactor tests ~ every 4 min. 	<ul style="list-style-type: none"> - Light gases as in (4) above plus BT (ben- zene and toluene) during reactor tests. - <u>On-Line GC Analysis</u> - GC BT analysis to be employed in calcula- tion of carbon conversion to BT.
6. <u>Product Gas</u> - Between pressure letdown valves (downstream of BTX adsorber). <u>Gas Sample Bottles.</u>	<ul style="list-style-type: none"> - Total of ~12 bottles dur- ing P.B. tests. - 6 to 8 bottles per test for each reactor test. 	<ul style="list-style-type: none"> - Light gases as in (4) above--each test. - C₃ and C₄ hydrocarbons--selected tests.
7. <u>Light Oil</u> (BTX)	<ul style="list-style-type: none"> - Each test. 	<ul style="list-style-type: none"> - Moisture, density, C, H, N, and ash--each test (C, H, N together). - S--selected tests. - B, T, X, thiophene and naphthalene-- selected tests. - Calorific value and distillations--selected tests.

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TABLE 6
ADVANCED FHP PROGRAM PLANNED SAMPLING AND CHEMICAL ANALYSES
(Sheet 3 of 3)

Sample	Frequency	Analyses
8. <u>Stripped BTX</u>	- Each test.	- Same as (7) above.
9. <u>Decanted L.O. Water</u>	- Each test.	- TOC and benzene--selected tests. - Phenols--selected tests.
10. <u>Decanted BTX Water</u>	- Each test.	- Same as (9) above.
11. <u>Product Gas Stream</u> at GC Sample Pt. No. 2	- <u>Two</u> selected tests.	- For NH ₃ and H ₂ S (for conversion of N and S to NH ₃ /H ₂ S).
12. <u>Product Gas Stream</u> - Just downstream of BTX adsorber.	- Selected tests.	- BT (to verify adsorption of BT in BTX adsorber).

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Figure 25. Schematic of Hydrogasification PDU with Sampling Location Points Noted

REFERENCES

1. Rockwell International, Development of a Single-Stage, Entrained-Flow, Short-Residence-Time Hydrogasifier, Final Report FE-2518-24, National Technical Information Service, Springfield, Virginia (July 1979)
2. Advanced Development of a Short-Residence-Time Hydrogasifier, Annual Technical Progress Report ET-3125-12, Government Fiscal Year 1979, Rockwell International Corporation, Environmental & Energy Systems Division, Canoga Park, California (January 1980)
3. L. P. Combs et al., Coal Hydrogasification Process Development: Volume I: Coal Studies, Second Annual Technical Progress Report ET-3125-24, Government Fiscal Year 1980, Rockwell International Corporation, Environmental & Energy Systems Division, Canoga Park, California (October 1980)
4. L. P. Combs et al., Coal Hydrogasification Process Development, Third Annual Technical Progress Report ET-10328-36, Government Fiscal Year 1981, Rockwell International Corporation, Environmental & Energy Systems Division, Canoga Park, California (October 1981)
5. The Lummus Company, Coal Hydrogasification Plant, Preliminary Design Study for a 250-MMM Btu per Day SNG Plant, Predesign Report, Rockwell International Corporation Contract N006-0004AX, Lummus Job No. 9087, Bloomfield, New Jersey (May 31, 1981)
6. Coal Gasification Concepts Gas Cost Guidelines, Revision 1, C. F. Braun Company, FE-2240-100 (August 1978)
7. L. P. Combs, Partial Liquefaction of Coal by Flash Hydropyrolysis, Phase IV Final Technical Report PETC-30018-6, Rockwell International Corporation, Environmental & Energy Systems Division, Canoga Park, California (November 1982)
8. D. L. Kors and D. F. Calhoon, Gaseous Oxygen/Gaseous Hydrogen Injector Element Modeling, AIAA Paper 71-674, Aerojet Liquid Rocket Company, Sacramento, California (June 1971)
9. D. F. Calhoon et al., Investigation of Gaseous Propellant Combustion and Associated Injector/Chamber Design Guidelines, N73-29799, Aerojet Liquid Rocket Company, Sacramento, California (July 1973)
10. H. A. Arbit et al., Lithium-Fluorine-Hydrogen Propellant Study, NASA CR-72325, Rocketdyne, A Division of North American Rockwell Corporation, Canoga Park, California (October 1967)
11. J. A. Schetz and A. Padhye, Penetration and Breakup of Liquids in Subsonic Airstreams, AIAA Journal, Volume 15, No. 10, p 1385 (October 1977)
12. B. J. Clark, Breakup of a Liquid Jet in a Transverse Flow of Gas, NASA-TN-D-2424, NASA-Lewis Research Center, Cleveland, Ohio (August 1964)

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13. A. J. Sistino, Kinetics-Mixing NO_x Model for Secondary Combustion in an Open-Cycle MDH-Steam System, 19th Symposium, Engineering Aspects of Magnetohydrodynamics, University of Tennessee Space Institute, Tullahoma, Tennessee (June 1981)
14. J. D. Holdeman and R. E. Walker, Mixing of a Row of Jets with a Confined Crossflow, AIAA Journal, Volume 15, No. 2, p 243 (February 1977)
15. R. E. Walker and R. G. Eberhardt, Multiple Jet Study Data Correlations, NASA CR-134795, Aerojet Liquid Rocket Company, Sacramento, California (April 1975)
16. C. L. Oberg et al., Partial Liquefaction of Coal by Flash Hydrolysis, Final Technical Report FE-2044-52, Rockwell International Corporation, Environmental & Energy Systems Division, Canoga Park, California (September 1982)
17. A. Y. Falk et al., Application of Hydrolysis to the Hydroconversion of Eastern Oil Shale, Final Technical Report ESG-DOE-13406, Rockwell International Corporation, Environmental & Energy Systems Division, Canoga Park, California (November 1983)

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