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MASTER

EVALUATION OF THE ROCKWELL INTERNATIONAL FLASH HYDROLIQUEFACTION PROCESS (FINAL SUMMARY REPORT)

PREPARED FOR

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EVALUATION OF THE ROCKWELL INTERNATIONAL
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(FINAL SUMMARY REPORT)

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FOREWORD

This report describes work performed on Task Order No. 18, Subtask No. 10 sponsored by the United States Department of Energy under Contract No. DEAC01-78 ET10159 (formerly Contract No. ET-78-C-01-3117). The DOE program manager is Dr. Theodore Simpson. The UOP/SDC program manager of liquefaction projects is Mr. James Gantt. The UOP/SDC task manager is Dr. Viren Sirohi.

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EXECUTIVE SUMMARY

At the request of the Department of Energy, UOP/SDC has evaluated the Rockwell Hydroliquefaction Process to determine the adequacy of the existing PDU data base and to assess the practicability and operability of the process. To meet these objectives, UOP/SDC conducted nine studies. Their findings follow.

1. Evaluation of Data from Flash Hydroliquefaction

A mathematical correlation of the data was first prepared by statistical analysis since, for data to be useable, it must be correlated. This was the first attempt to fully correlate these data. The recommended models for carbon conversion and liquid production are:

$$\%C = 8.1 + 0.025T + 0.015t - 1.59D - 0.19G$$

$$\%L = 66.0 - 0.059t - 0.153G - 0.013T + 6.1 \text{ H/C}$$

These models are useful for parameters with predominant ranges tested in the 1 tph test program (residence times 50-150 msec, reactor temperatures 1530-1930°F, H₂/C ratio 0.4 to 0.6 lb/lb, etc.) and have been found to predict test results to within 2 to 3%. However, only a low-accuracy correlation could be developed for the yield of individual cuts of liquids. The existence of these multivariable correlations points up the fallacy of reaching definitive conclusions based on plotting yield vs. a single variable as reported by Rockwell. The effect of H/C shown in the above %L model was not detected by Rockwell, who specified an H/C of 0.2 for the conceptual design (although essentially all of their PDU tests had been at 0.4 and above) and assumed that this would have no effect on yields. Also, analysis did not confirm the significance of the mixing variable, DPR, that was reported by Rockwell. This was because DPR proved to be highly cross-correlated with other variables so that its effect could not be distinguished. This cross correlation of various operating variables proved to be a very general problem, caused by poor design of the PDU experiments. It made it impossible to determine what the effect was of several of the other operating variables.

There were also problems with the quality of the PDU data. For example, some carbon balances were as low as 80% even after forcing a 50% ash balance up to 100%. The missing carbon was then presumed to be accounted for as liquid product. This certainly requires confirmation. Another problem is that some reported test data up to about a year old were modified after recalibration of instrumentation at the completion of the design test program. Also, there are insufficient data on liquid product quality and volatility, since these data were collected essentially only for the last 29 PDU tests. The overriding limitation, however, was the inadequate design of experiments, which limited the usefulness of the resultant data. When added to the fact that all the PDU runs were very short, it is concluded that the PDU data are inadequate to characterize the RI process fully or to form a basis for designing a scaled-up pilot plant.

2. Pilot Plant Design Feasibility

Design of a pilot plant is not now feasible. This is true because of the poor quality and incompleteness of the data on the reactor chemistry, as noted above. It is also true because data are required to support the design of specific process units and components. The more important of these needs are (1) data on whether the fine feed coal can be handled in a lockhopper system; (2) long runs to provide materials data, erosion data, and fouling factors in the pre-heater, reactor, quench, and condensers; (3) tests with multiple feed injectors; (4) successful tests of the "hot hydrogen" precombustion system; and (5) tests to show that the cryogenic hydrogen separation process will not be plugged by trace components.

3. Commercial Reactor Feasibility

The commercial reactor is both highly complex and also operates under some extreme conditions. This results in a list of problems, for some of which no resolution can be foreseen. The use of exotic alloys and the very cramped

spacing complicates the problem. The following are the more important limitations that have been encountered in this brief study. It is concluded that it would be unwise to attempt to prepare a commercial reactor design at this time.

- 1) The accuracy of the RI dense phase coal feeding system and its ability to split this coal into 64 equal streams have not been determined.
- 2) The data base - both chemical and mechanical - is incomplete. Long runs and scale-up data are needed. Long runs are needed for materials, erosion, reliability, and fouling data.
- 3) The injector coal lines are exposed to high temperature (about 1500°F) hydrogen. This could cause plugging problems over a sustained period of operation. If cooling or insulation of these lines is required, it will add to both the complexity and the cost of the reactor.
- 4) The H₂ injector line where the H₂ combustion occurs within the reactor assembly is subjected to a temperature of 2500°F.
- 5) The geometrical spacing for the injectors does not permit maintaining the desired configuration of the four-on-one injector design (hot burner). This problem is accentuated as injectors approach the center of the reactor. It appears that this concept might lose a number of injectors because of this problem. Thus, the reactor's capacity and the reactor's length (which is related to residence time) are in question.
- 6) Balancing and closely controlling the flows of both oxygen and hydrogen, as is required, may prove to be a very difficult problem, particularly since the various oxygen and hydrogen annuli are handling different amounts of these gases.
- 7) The reliability of the hydrogen and oxygen orifices is an area of grave technical concern. The oxygen orifice diameter is only 0.08 inch. Because of the method of fabricating this design, it is virtually impossible to

service or maintain the orifices once the reactor head components are assembled and welded together. Entrained scale or carbon or other deposits could quickly throw off the required close control.

- 8) The reactor head is essentially unserviceable except by replacement.
- 9) There is doubt that the interior face of the injector can withstand or survive the reactor's severe environment, even with provision of water cooling in the face plate. It is even conceivable that cooling the face can give rise to severe thermal gradients. Use of insulation for the injector face plate would present a host of additional problems, since it would impact on the injector design and mixing efficiency and on reactor fabrication.
- 10) As presented, the RI design shows cooling water entering the outermost annulus in the face of the head; however, no provision for bringing this water into the inner cooling water annuli is given. Interconnecting these annuli will present additional stress problems. Also, the fabrications will become more complicated and undoubtedly result in a higher reactor cost.
- 11) A 3.5 tph coal injector capacity is necessary to attain the desired velocity in a commercial reactor. However, such a high feed rate through a 1-inch injector nozzle has never been demonstrated. Neither has the use of multiple injectors been verified.
- 12) The use of a refractory with a metal liner (e.g., stainless steel) backed with insulation is seriously questioned for the commercially sized reactor. Determining the cost, fabricability, and reliability of this configuration would depend on finding answers to many unknowns.
- 13) The small clearance (1/2") between the vertical coal lines and the walls of the hydrogen annuli will make the required welding impossible. The oxygen lines (feed to the oxygen manifolds) are quite close to some of the coal lines. This will present a fabrication problem.

4. Feasibility of Presently Used H₂ Throughput and Recycle Requirement

In this evaluation, a process scheme was established, and estimates were made of the importance of (1) causes of H₂ losses, (2) economics, (3) down stream processing, (4) precombustion method, and (5) how diluents such as CO and CH₄ react in the reactor and precombustor. Numbers (3) through (5) proved most important. Downstream processing is costly because the large excess of feed hydrogen required makes at least some purification, separation, and recycle necessary. Consequently, raw gas recycle was called for. It was found that up to 60% of the product gas could be recycled as raw gas. The resultant buildup of impurities concentration is only 10-15%. Even without raw gas recycle, the impurity concentration will be approximately 7%.

It was necessary to assume how the diluents would act in the reactor, since there are no data at all from the PDU on the effects of using impure H₂. Two assumptions were tested: (1) the diluents are inert chemically and simply reduce the H₂ partial pressure; and (2) the diluents react and reach chemical equilibrium. If the latter proves true, significant benefits result. In the reactor, some methanation of the recycled CO occurs that provides exothermic heat of reaction, reduces the precombustion requirement, and eliminates the need for a methanator in the purification train.

Raw gas recycle itself offers another important advantage. It eliminates the disadvantage of the precombustion mode compared with the hot hydrogen injector. The precombustor mode requires an H₂/coal ratio of 0.4 (wt) vs. 0.2 for the hot hydrogen mode. With raw recycle, the extra hydrogen can be provided cheaply. Accordingly, the precombustion mode appears to have two advantages over the hot hydrogen mode: (1) a lower combustion temperature that eases the materials problem, and (2) an indicated higher total and liquid yield, according to UOP/SDC's analysis of the data. (Rockwell's analysis does not agree with this.)

A major unknown is whether the diluents in the recycle H₂ will break down and deposit carbon and thus create plugging. Chemical equilibrium data indicate they are likely to do so, especially at the higher hot hydrogen temperatures.

As a result of all these unknowns and required assumptions, it is not possible to say whether the presently planned use of hydrogen is feasible. Also, UOP/SDC's estimate of the recycle gas quantity and quality must be considered very tentative.

5. Effectiveness of a Heat Exchange Quench

Though calculations indicate that a direct water quench is about ten times faster, economics and thermal efficiency call for the use of a heat exchange quench instead. To estimate the effect of a heat exchange quench on the liquid product yield, a conceptual exchange quench was designed; its size was based on a calculated overall heat transfer coefficient of 200.

The effect of the resultant added residence time on liquid yield was based on a derived relationship, since no yield data for added residence time at independent conditions have been measured by Rockwell. The result was an estimated 7.9% decline in the liquid yield, from 38% to 35%. Rockwell's report from initial tests of an exchange quench is considered in agreement with UOP/SDC's findings within the limited accuracy of the Rockwell data.

But there are other problems. There is no way to say whether erosion, corrosion, fouling, or plugging will cause the premature failure of an exchange quench without multiple tests in PDU runs of 7 to 30 days. The reactor effluent stream before entering the quench unit contains particulate matter in the amount of about 50,000 grains/scf. The amount of the particulate matter in the effluent gas is extremely high in comparison with the range experienced in other commercial processes. The gas velocity in the exchanger is about 200-250 ft/sec, which is much higher than the velocity (80 ft/sec) normally used in exchanger design. The particulate laden gas will impinge directly on the tube sheet. The question of whether there will be extreme erosion of this exchanger can be answered only by taking data from extended operation of the PDU.

There are too little data to determine such important things as whether the quality of the liquid product suffers from use of the exchange quench. Several other data gaps were found.

The cost of an exchange quench is estimated at 1% of the cost of a commercial plant.

6. Evaluation of Supplemental Coal Feed Systems

A comparison was made of three alternative supplemental coal feed systems, namely lockhoppers, slurry feed, or the J.P.L. extrusion feeder. In a cost estimate, the lockhopper was the most attractive. This is the system Rockwell specified for its conceptual design/cost estimate of a commercial plant.

However, because the coal is (-) 200 mesh, the lockhopper can be expected to have problems with high dust losses and poor solids flowability. Available valves are too small; and lockhoppers have a reputation for poor reliability even when feeding coarser coal. Although the slurry feeder might appear to be more dependable, experience with slurry feeders is not much, if any, better than with lockhopper systems.

If a slurry feeder were selected, a greater cost would be imposed on the process. Tests would be required before a final selection could be made.

7. Need for Refining and Upgrading of Rockwell Liquid Products

There is some question as to the reliability of the Rockwell data on the quantity and quality of their liquid product. However, based on present data, the Rockwell liquids are clearly not marketable as commercial fuels without upgrading. They are seriously deficient with respect to the ASTM specifications for both light and heavy fuels and are believed to be unstable in storage. The heavy oil probably would not be usable even as a plant fuel, because it is likely to plug burners, to have high emissions, and to prove unstable in storage (where it would have to be held above 200°F to be stored as a liquid).

Refining is therefore necessary. However, refining is likely to be difficult and costly. The cost of refining coal liquids has been correlated with feed-stock H/C ratio by Mobil, with the general result that costs go up as H/C ratio goes down. In this respect, Rockwell liquids are markedly inferior; the heavy oil has a lower H/C than anthracite coal and SRC-I, and the light oil a lower H/C than COED tar or 400°F(+) H-Coal fuel oil. Removal of the relatively high S and N content of Rockwell liquids would also be difficult, and yields low, because the low H/C indicates a predominance of fused-ring compounds. The indicated instability of the Rockwell liquids would not only limit surge storage time but also make it necessary to limit fractionator temperatures to prevent possible coking. To permit further processing, hydrotreating would have to be an early step. To upgrade the Rockwell light oil to an acceptable catalytic cracker feed of an 11% hydrogen content, 5 lbs of hydrogen per 100 lbs of oil would have to be added. This compares with the 2 to 3 lbs required for the H-Coal, EDS, and SRC-II liquids.

Even production of aromatics from this highly aromatic oil may not be economical, since removal of S and N would seriously reduce yield as a result of simultaneous ring saturation and coke formation.

Hence, the cost of attempted refining would be high. UOP estimated \$11/Bbl to upgrade H-Coal Heavy Distillate to transportation fuels. Rockwell light oil with a lower H/C would cost more. Application of a Mobil correlation of the cost of refining coal liquids with H/C indicates that, if the Rockwell oil were as good as SRC-II, refining cost would be \$3.80/MM Btu. This makes the total cost so high in comparison to the total cost of producing coal liquids by other processes that it calls into question the advisability of further work on this process, which is still in the exploratory phases.

8. Determination of Need for Heat Exchange and for Additional Data for Future Commercial Design

Heat exchange is required at almost every step of the process, primarily for H₂ preheat, raw product quench, liquid product condensation, and cryogenic gas

separation. The total heat exchange exclusive of that for the cryogenic unit equals 55% of the heating value of the coal. With so large a total, effective heat exchange is vital to process thermal efficiency. So large a need will hurt process economics. This is especially true of the preheater and the quench.

Heat exchange data needs have not yet been filled at this stage of the process development; difficult problems exist. These include fabrication of the quench exchanger, possible plugging of the preheater by carbon deposition, erosion, fouling, corrosion, loss of liquid product in the exchange quench, plugging with char or fouling with byproduct of the liquid condensers, mist formation in the condensor, plugging of the cryogenic unit, and others.

The data gaps are large. What is needed includes long runs using a hydrogen feed that contains the diluents, materials data, better planned tests, downstream particulates measurement, more complete exchange data, and data from a test cryogenic separator.

9. Unusual Materials Requirements

A materials evaluation study has been performed for a conceptual design of a commercial plant using Rockwell International hydroliquefaction technology. In it, properties of candidate materials for major equipment, information gaps, and research needs are discussed. There are unusual materials requirements throughout this process that result either from the severe conditions the process requires or from the chemical composition of the process stream. The hydrogen preheater coil is one of the more critical materials problems because economics calls for pushing its temperature as high as possible. The reactor injector head assembly has a combination of several severe materials problems - high temperature corrosion by H_2 and oxygen at 2500°F, erosion by flowing coal, creep and differential expansion distortion of nozzles and orifices as small as 0.08 inches, and thermal stress and thermal stress corrosion as a result of being adjacent to areas with temperature differences as high as 2300°F. The quench exchange has erosion, thermal stress, and the possibility of unusual corrosion problems. Similar situations exist in the

char collection and liquid product collection areas. The process also entails special char gasification materials problems because transfer of the finely divided residual char at 1000°F to a high temperature disperse phase gasifier is called for. These problems and others are itemized, with comments, in this report.

The most troublesome categories that these problems fall into are (1) erosion - the char loading of the high velocity gaseous product is very high; (2) H₂ embrittlement - the relatively high purity H₂ is at as high a temperature as 2500°F; (3) high thermal stress - some high- and low-temperature streams are mixed, and others are in close proximity; and (4) component availability - uncertainty of availability of large lockhopper valves operable with (-) 200 mesh char.

A comprehensive program for identification of materials that can be used under the conditions encountered in the Rockwell process is required. Essentially no such work has been done yet in the Rockwell International Flash liquefaction process. The data gaps are serious: materials could not be specified with any confidence at this time for a pilot or commercial plant. For each case where a suitable material must be found, generally a 30-day cumulative exposure test with an acceptable loss rate is required.

10. Overall Conclusions and Recommendations

1. A complete designed set of experiments must be run on the present PDU to make possible satisfactory analysis of the data for the effects of variables, especially the effect of diluents in the H₂ feed and the possibility of carbon deposition problems.
2. Basic improvements in the equipment and operation of the PDU should first be made, including:
 - a. Improving the material balances on ash and on carbon.
 - b. Making possible at least a few proof runs in which ash recovery approaching 100% is achieved.

- c. Establishing the nature of the product that accounts for the chronically deficient carbon balance.
 - d. Feeding of recycle gas or a synthetic mix.
 - e. A hydrogen preheater and combustor of the same nature as that which would be considered in the pilot plant.
 - f. Apparatus to provide scaling data for multiple injectors.
 - g. A means of checking the reliability of dense phase solids feeding accuracy for each run.
 - h. A cylindrical, larger char receiver for long duration runs that will permit testing of its dense phase transfer to a gasifier.
 - i. Reconciliation of analysis of uncondensed liquid product by BTX Absorber vs. gas chromatographic analysis of the gas.
 - j. Apparatus to see if a cryogenic gas separator will operate without plugging.
3. A reactor design concept must be developed that looks feasible for design, fabrication, and operation.
 4. A conceptual commercial design and economics should be prepared based on a realistic set of design bases and criteria.
 5. If the above are accomplished successfully with attractive results, then and only then:
 - a. A study should be made of the refining requirements of the product.
 - b. The PDU should be modified for continuous runs of up to one month. It should be operated to:
 - (1) Confirm the data correlations.
 - (2) Estimate the reliability of the reactor and process.
 - (3) Adhere to the requirements stated above.
 - (4) Test cryogenic gas separation on a slip stream.
 - (5) Study fouling and erosion of the quench exchanger and product condenser.

- (6) Study lockhopper feeding of the ground feed coal.
- (7) Establish initial reliability of the coal injector and precombustion assembly head.
- (8) Collect scale-up and design data.
- (9) Conduct a materials study and confirm materials choices for each section.

c. Determine particle size of the char and of the solids in the oil product, the degree to which they may be separated, and how the ash should be removed from the product oil.

6. After the above requirements have been met, a decision could be made on the feasibility of and the justification for a pilot plant for the Rockwell Flash Hydroliquefaction process.

SECTION 1 - INTRODUCTION

In response to DOE Task Order 018, UOP/SDC is providing technical and engineering support to the Department of Energy in evaluating the Rockwell Flash Hydroliquefaction process, presently in the Integrated Process Development Unit (IPDU) step of development. A principal purpose of the evaluation is to determine whether there is a need for DOE to initiate and fund a pilot plant program for the process. As part of the UOP/SDC effort, nine studies have been undertaken:

- 1) Evaluation of the Rockwell data.
- 2) Evaluation of the adequacy of the data to provide a design basis for a pilot plant.
- 3) Evaluation of the practicability of building a commercial scale reactor.
- 4) Evaluation of the use of hydrogen in the process.
- 5) Evaluation of the effectiveness of the quench system.
- 6) Evaluation of supplemental coal feed systems.
- 7) Evaluation of the need for product refining and upgrading.
- 8) Evaluation of needs for heat exchange in the system.
- 9) Evaluation of unusual material requirements.

These studies are interrelated to the extent that sometimes the results or findings of one are used in another; basically, however, they stand alone. In each, gaps in the available data are discussed in reference to the subject of the study.

SECTION 2 - BACKGROUND

2.1 INTRODUCTION

For the past five years, Rockwell International has been engaged in the development of a coal hydroliquefaction process that reacts finely ground coal with high-temperature hydrogen injected under high pressure into a reaction chamber at high velocity flow. To bring the reactants to reaction temperature, a large excess of preheated and partially precombusted hydrogen must be used. After a short residence time (about 100 msec), the product is rapidly quenched from 1700°F to 1000°F, the gases and vaporized condensables are separated from the char, and, after a series of condensations, heavy and light oils are obtained that can be further refined for use as a synthetic fuel.

The process development proceeded in two steps, with a Bench Model Unit operating at a nominal 1/4 tph followed by a Process Development Unit (PDU) operating at 1 tph.

Before operation of the 1/4 tph unit, a number of cold flow tests were conducted at company expense to develop the components necessary for system operation. These included: a) dense phase feeding and transport of pulverized coal, and b) cold flow chamber mixing for optimum injector design. In addition, a method of combustion of a small amount of oxygen with the injected hydrogen to increase its temperature to about 2000°F was also developed.

2.2 DESCRIPTION OF PDU

A simplified flow diagram of the current 1 tph PDU is presented in Figure 2.1. Earlier versions of the unit were smaller or less comprehensive. The reactor tube and injectors are located within the pressure vessel, but insulated from it. Coal is fed to the reactor in dense phase, using nitrogen (O₂ impurity 2 mole %) as the pressurant/carrier gas. Hydrogen for the reactor is heated in a fired exchanger, then by partial combustion with oxygen as necessary to raise the coal to the reactor temperature. In most of the 1 tph PDU tests,

Figure 2.1 Simplified Flow Diagram of 1 TPH PDU.

this oxygen was added in a small preburner attached to the feed injector. During a limited number of tests, the oxygen was added within the hydrogen passages on the main injector (hot-hydrogen mode).

The hydrogen and the coal streams then run to the injector. Both the injector configurations used a single-element four-on-one configuration. Four hydrogen streams impinge at a 30-degree angle onto a central coal stream. This rocket-engine-type injector is believed to thoroughly mix the hydrogen with the coal. A second injector configuration was built and briefly tested to allow oxygen injection directly into the reactor.

In the beginning, a direct water spray quench of the reactor product was employed. However, because heat recovery is important to the economics of a commercial coal liquefaction plant, a water-cooled heat-recovery quench unit was designed by Rockwell and installed in the 1 tph PDU. It was employed from approximately November 1979 through February 1980. This unit has been found to cool the products from a reactor temperature of 1700°F to about 1250°F. Several water-spray nozzles were installed near the outlet of the heat recovery quench to supply supplemental cooling to about 1000°F, the minimum quench temperature at which the product char has no solubility.

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

The Rockwell 1 tph reactor unit was originally constructed with a two-stage low-pressure liquid product recovery system. This recovery system had two deficiencies - incomplete condensation because of low pressure, and a tendency to foul the condenser with tarry material. Because of these deficiencies, the low-pressure recovery system was replaced by a high-pressure system having two condensers. The cooling surfaces of the first stage upstream heavy oil condenser are maintained at a temperature of about 500°F so that the tarry materials will remain fluid on the surface.

The vapor phase material is further cooled in a second stage shell-and-tube condenser to about 100°F. This condensate is recovered in a vapor-liquid separator and passes through an accumulator, a pressure letdown valve, and a flash drum, then into a light oil storage tank. The water in the system from reaction plus the water spray is condensed in the light oil condenser. Light oil (and water) are removed from the light oil storage tank after each test. The remaining product gas from the light oil vapor/liquid separator passes through a pressure letdown valve, through a flow metering orifice, and past a gas sampling station into a scrubber system before being vented to a flare stack.

A benzene-toluene-xylene (BTX) recovery system was installed in December 1979 to improve the material recovering the vapor-phase BTX product in the gas stream leaving the light oil condenser. The vapor-phase BTX cannot be recovered by further cooling of the gas stream, because the loss is still significant at the freezing temperature of benzene, 42°F. An activated carbon adsorber system with steam stripping was selected. The BTX product is stripped from the carbon with steam.

2.2.1 Spent Char Handling

Test durations on the present 1 tph system are limited by the capacity (360 gallons) of the char-vapor separator (main char receiver). Consequently, a maximum of approximately 3/4 tons of coal can be fed per test. The capacity of the high-pressure coal feeder is 3-1/2 tons.

2.2.2 Description of PDU Runs

A series of tests was made to assess the effects of reactor temperature, location of oxygen addition, residence time, and velocity. To avoid reactor plugging due to wall adhesion, reactor temperatures were held above 1400°F.

Table 2.1 shows a summary of the typical 1 tph test conditions and results. Included are pressures, temperatures, residence times, duration, flow rates, coal consumption, and carbon conversion. All tests shown used the preburner mode of operation.

Table 2.1 Summary of 1 TPH PDU Test Conditions and Preliminary Results*

Test No.	Date	Reactor Pressure psig	Reactor Temperature °F	Residence Time msec	Duration minutes	Flow Rates lbm/sec			Total Coal lbm	Carbon Conversion		
						Coal	Hydrogen	Oxygen		Overall	To Gas	To Liquid By Difference
79 -1	2/12/79	990	1325	67	3.6	0.432	0.210	0.087	92	45.2	8.0	37.2
3	3/08/79	990	1625	67	26.5	0.441	0.190	0.104	701	56.4	20.7	35.7
4	3/12/79	999	1725	61	18.3	0.429	0.200	0.122	472	56.1	23.2	32.9
5	3/14/79	498	1550	46	25.0	0.377	0.146	0.082	566	52.9	16.0	36.9
6	3/19/79	502	1650	48	4.8	0.502	0.145	0.088	143	52.1	11.3	40.8
7	3/29/79	999	1630	63	9.7	0.424	0.210	0.117	247	57.7	18.7	39.0
8	4/06/79	987	1600	66	10.4	0.451	0.198	0.104	280	55.6	16.8	38.8
9	4/12/79	498	1750	53	11.2	0.227	0.118	0.078	157	55.8	26.7	29.1
10	4/19/79	497	1685	59	18.2	0.243	0.112	0.064	266	55.5	16.2	39.3
12	5/03/79	999	1660	65	60.2	0.402	0.202	0.099	1447	59.7	21.7	38.1
13	5/10/79	495	1710	61	2.9	0.245	0.102	0.063	43	56.6	16.5	40.1
15	5/23/79	991	1767	61	58.1	0.402	0.208	0.106	1400	66.8	26.9	39.9

*Reactor for all tests was 2.37-inch diameter by 5 feet long. Preburner mode of operation.

Coal for all tests was Kentucky No. 9 (70% <200 mesh for Tests 1-10; 90% <200 mesh for Tests 11-15).

High-volatile bituminous Kentucky No. 9 coal was used for all tests. The nominal particle size was about (-) 200 mesh.

The test conditions (reactor pressure, temperature, and residence time) used for all of the testing were in the range known from previous testing to produce high liquid product yields. The nominal test condition ranges used were as follows: temperatures from approximately 1550 to 1800°F, pressures of 500, 1000, and 1500 psig, and residence times from 20 to 250 msec.

2.3 PROPOSED DESIGN BASIS

The following are the process operating conditions that were used in most of the process studies in this UOP/SDC task.

- Coal Type - Average PDU analysis (Bituminous Coal)
- Reactor Temperature - 1700°F
- Reactor Pressure - 500 psig
- Reactor Residence Time - 150 msecs
- H₂ Preheat Temperature - 1500°F
- Preburner Maximum Temperature - 2000°F
- Heavy Oil Condenser - 500°F outlet
- Light Oil Condenser - 100°F outlet
- AGR Absorber Solvent - 100°F
- Methanation Inlet Temperature - 550°F
- Cryogenic Partial Condensation Temp. - -260°F
- Plant Capacity (coal requirement) - 250TD

The following are the design conditions for the heat exchanger systems.

- BFW Temperature - 250°F
- Deaerator Feed Temperature - 120°F
- Cooling Water Supply Temp. - 75°F
- Cooling Water Return Temp. - 110°F
- Air Cooler Outlet Temp. - 140°F

Table 2.2 shows the reactor material balance estimated from the given conditions.

2.4 PROPOSED BASELINE PROCESS DESIGN

The baseline process design is comprised of the following major sections: Coal Receiving, Pulverizing, and Drying; Coal Feeding; Preheating, Reactor, and Quench System; Char Cooling and Storage; Liquid Product Recovery System; Acid Gas Removal; Methanation; and Hydrogen Separation. These units are the major units required to test the system in recycle operation. Hydrogen, oxygen, and nitrogen supply systems will also be necessary. Figure 2.2 is a block flow diagram for the pilot plant conceptual design. As noted in the figure, the following units are assumed to be provided at the plant site: Sour Water Stripping, Waste Water Treatment, Cooling Water, Process Water, Steam Generation, Power Generation, Flue Gas Treatment, Solid Waste Disposal, Air Systems, Blowdown and Flare Systems, Fuel System, and Unloading and Loading Systems.

The main process units detailed in the block flow diagram are described below.

2.4.1 Coal Receiving, Pulverizing, and Drying

Coal is withdrawn from the pile either by means of front end loaders (batch feed) and or bucket elevators (continuous feed) and discharged into a raw coal feeder (most probably a hammer mill). From here it is charged into a pulverizer (an air swept ball mill, for example). Hot air (or flue gas) about 350°F sweeps through the mill and carries the powdered coal to a cyclone. The bottoms from the cyclone, which is dry coal powder, is conveyed to the dry coal storage. The cyclone vent is partially mixed with fresh air and reheated and recycled. The other part and the various conveyor hoods are vented to the atmosphere via a dust removal system (baghouse or wet scrubbers). The schematic drawings for this section are shown in Figures 2.3 and 2.4.

Table 2.2 Reactor Material Balance
(page 1 of 2)

Assumptions & Bases

- Coal Analysis: Average PDU Test Analysis
- Char Analysis: PDU Data, UOP Analysis, and Conversion Data
- Oil Analysis: UOP Analysis and PDU Data
- "Separated" Gas Analysis: (PDU Data)
 - 78.5 Vol. % CH₄
 - 14.0 % CO
 - 1.0 % CO₂
 - 0.5 % C₂H₄
 - 6.0 % C₂H₆
- Sulfur and Nitrogen form H₂S & NH₃, respectively
- Oxygen Consumed forms CO, CO₂, and H₂O (by difference)
- Hydrogen Feed estimated by Energy Balance
- Carbon Conversion to Oil = 39%
- Carbon Conversion to Gases = 19.1%

Table 2.2 Reactor Material Balance
(page 2 of 2)

In (lb/lb coal)

Coal	1.000
Coal Moisture	0.0204
Hydrogen	0.4923
Oxygen	0.1372

Out (lb/lb coal)

Char	0.4199
Oil	0.3263
H ₂ O	0.2271
H ₂	0.4502
CH ₄	0.1379
CO	0.0430
CO ₂	0.0048
C ₂ H ₄	0.0015
C ₂ H ₆	0.0198
CH ₄	0.1379
H ₂ S	0.0176
CO	0.0430
NH ₃	0.0018

Coal Analysis

C	73.46 wt %
H	5.02
O	8.78
N	1.13
S	2.64
Ash	8.97
HHV	13,182 Btu/lb (dry)

Char Analysis

C	73.30 wt %
H	2.00
O	0.76
N	1.05
S	1.53
Ash	21.36
HHV	11,757 Btu/lb

Oil Analysis

C	87.80 wt %
H	6.41
O	3.10
N	1.65
S	1.04
HHV	16,435 Btu/lb
sp gravity	= 1.15 (avg)

Figure 2.2 Block Flow Diagram of Pilot Plant Conceptual Design.

2-11

SH-1101
BULL DOZER OR
FRONT-END LOADER

SH-1102
RAW COAL
RECEIVING
HOPPER

SH-1103
RAW COAL
FEEDER

SH-1104
RAW COAL CONVEYOR

SH-1105
RAW COAL
WEIGH SCALE

LEGEND

- PRESSURE, PSIG
- TEMPERATURE, °F
- DUTY, MM BTU/HR
- STREAM NUMBER

Figure 2.3 Process Flow Diagram, Coal Storage and Handling

Figure 2.3 Process Flow Diagram, Coal Storage and Handling

2.4.2 Coal Feeding System

Dry powdered coal is charged into two low-pressure lockhoppers operating at about 300 psig. While one of the hoppers is being charged, the other is discharging into one of two high-pressure lockhoppers. These latter also alternate in their operation; one discharges while the other is charging. From the high-pressure hoppers, the dry coal goes to the coal feeder vessel. Both high-pressure hoppers and the feeder vessel operate at 650 psig.

2.4.3 Reactor and Quench System

The recycle hydrogen used as a reactor feed is combined with make-up hydrogen, and the mixture is heated in an externally fired heater to 1500°F. This hot hydrogen gas is heated to 2000°F by burning some of it with oxygen in the pre-burner. Coal from the dense phase feeding system is fed into the reactor and mixed instantaneously with the 2000°F hydrogen. The mixing of the coal and hydrogen is accomplished by multiple injectors similar to those used in liquid combustion rocket engines. The reactor effluent is quenched after a short residence time (150 milliseconds) in the reactor vessel to avoid deterioration of the liquid product. This quench is accomplished in a recuperative tube and shell heat exchanger in which product flows downward inside parallel tubes and water flows countercurrent in the shell. The effluent gas from the quench is then separated from the solid char in the char/vapor separator and two cyclones and goes to liquid product recovery. The process flow diagram is shown in Figure 2.5.

2.4.4 Char Cooler and Char Storage

Hot char is present in the char separator at about 1000°F and 500 psi. It could be used directly in hydrogen production, in which case it would not need to be cooled. However, for the pilot plant, it is withdrawn from the separator and cooled for storage. Withdrawal of hot char is done through a system of lockhoppers, and the cooling is accomplished by a water cooler before the char is taken to storage. The process flow diagram is shown in Figure 2.5.

2.4.5 Liquid Product Recovery System

Liquids produced in the reactor are recovered in a system designed to yield three major products: heavy oil, light oil, and BTX. The heavy oil product includes all liquids condensed at 500°F. The BTX stream consists mainly of benzene and small quantities of toluene, xylene, and other light oils. The remainder of the liquids produced are included in the light oil product.

The process flow diagram is shown in Figure 2.6.

2.4.6 Heavy Oil Condenser

The first unit in the system is the heavy oil condenser. The 1000°F gas from the cyclone separators is cooled to 500°F in a shell-and-tube heat exchanger. Saturated boiler feed water is pumped to the shell side of the exchanger to provide the necessary cooling for condensation of the heavy oil. Steam produced in the exchanger is sent through a steam drum to the steam header. Condensate from the steam drum and makeup boiler feed water are recycled to the heat exchanger.

Liquids condensed in the heavy oil condenser are separated from the remaining gases in the heavy oil accumulator. After reducing the pressure on the heavy oil stream and separating flashed gases in the heavy oil flash drum, the heavy oil is piped to the product storage system. The heavy oil pipeline will be steam-traced to maintain liquid phase flow.

2.4.7 Light Oil Condenser

The second stage in the liquid recovery section is designed to cool the gases from the heavy oil accumulator to 100°F and collect the condensed liquids. This cooling is accomplished in the light oil condenser, in which water is the cooling medium. Liquids condensed in the light oil condenser are separated from the gas stream in the light oil accumulator. After a reduction in pressure, the light oil stream passes through the light oil flash drum.

Figure 2.6 Process Flow Diagram, Liquid Products Recovery

Liquids in the light oil stream include water and a variety of hydrocarbon oils. Free water in the light oil stream is separated from the light oil in the light oil decanter and sent to the oily/sour water treatment system.

2.4.8 BTX Recovery

The gas leaving the light oil condenser still contains a significant quantity of benzene. This material is recovered by a typical lean oil absorption system designated as the BTX recovery system.

The principal components of the BTX recovery system are the BTX absorber and the BTX fractionator. Gases from the light oil accumulator are contacted with the lean oil in the BTX absorber. The stripped gases from the absorber are then sent to the amine scrubber for further processing.

The lean oil used in the BTX absorber consists of the oils recovered in the light oil recovery section after stripping. Before being used in the BTX absorber, this oil and the rich oil from the absorber are stripped in the BTX fractionator. The BTX fractionator system, which operates at 15 psig, consists of the fractionator tower, a reboiler, a water-cooled overhead condenser, an overhead accumulator/separator, and required reflux and reboiler pumps.

The fractionator bottoms stream is cooled and recycled to the BTX absorber. Part of this stream is also withdrawn and sent to the light oil product storage tank.

The liquid products produced are stored in tankage. The sources of the products are as follows: heavy oil from the heavy oil flash drum, light oil from the BTX fractionator bottoms, and BTX from the BTX fractionator separator.

2.4.9 Acid Gas Removal

The hydrogen sulfide and carbon dioxide are removed from the gas stream leaving the BTX removal section in an acid gas removal system, such as MEA. In the gas

removal unit (see Figure 2.7), the sour gas first passes into the base of an absorber and flows upward, countercurrent to an amine-water solution that removes the acid constituents. This solution is next pumped from the bottom of the absorber through heat exchangers, and the temperature is raised to approximately 195°F. The sour amine solution is then sent to the upper section of the stripper and flows downward, contacting the hot vapors from the reboiler, and the acid gases are removed. The acid gases liberated from the amine solution contain a portion of the vaporized amine and water. This is removed by passing the hot vapor mixture through condensers, and the condensate is recycled to the stripper tower. The acid gases are burned in a flare or sent to sulfur recovery.

The hot reconcentrated or lean amine solution flows from the reboiler to heat exchangers and is cooled by the incoming cold rich solution. The cool lean amine solution is then piped back to the absorber, thus completing the cycle.

2.4.10 Methanation

The feed gas to the methanation unit, previously processed through the AGR, is first heated to about 500°F through a feed-effluent heat exchange and a fired heater, if necessary. This heated gas is first passed through a desulfurizer (ZnO bed) to remove trace quantities of H₂S, COS, and mercaptans to protect the methanation catalyst from sulfur poisoning. The CO, CO₂, C₂H₄, and C₂H₆ are then converted in the methanation reactor to CH₄. The product gas from the methanator, approximately 100°F higher in temperature than the inlet gas because of the exothermic nature of the methanation reactions, is cooled in the feed-effluent exchange and a water cooler before condensate is separated from the gas in a knock-out drum. The process flow diagram is shown in Figure 2.8.

2.4.11 Hydrogen Separation

The purpose of the cryogenic separation is to remove from the recycled hydrogen stream most of the non-hydrogen product gas components while concentrating the hydrogen portion of the stream for recycle to the reactor.

R-2501 A/B
SULFUR GUARD BEDS

E-2501
METHANATION FEED
EFFLUENT EXCHANGER

H-2501
FEED GAS
HEATER
(START-UP)

R-2502
METHANATION
REACTOR

E-2502
METHANATION EFFLUENT
CONDENSER

D-2501
METHANATION
UNDER-OVER DRUM

2-20

Figure 2.8 Process Flow Diagram, Methanation

The feed gas, previously processed through the AGR and methanation systems, enters the cryogenic system. It is then dried by a molecular sieve to clean up traces of water, H_2S , CO_2 , and other impurities. The gas then enters the cold box. In this unit, the feed is cooled through heat exchange with returning product streams. This cooling occurs when hydrogen-rich vapor, obtained by partial condensation, is separated and warmed by heat exchange with the feed gas. At the same time, the hydrocarbon (methane) rich liquid is expanded to low pressure to provide Joule-Thomson refrigeration for the process and is then vaporized and warmed against the feed.

The feed gas is cooled to approximately $-260^{\circ}F$, then the liquid and vapor are separated into the methane and hydrogen-rich products. The methane must be compressed to pipeline pressure, and the hydrogen must be compressed enough to overcome the system pressure drop so that it can be recycled to the reactor.

SECTION 3 - RESULTS OF ROCKWELL DATA EVALUATION (SUBTASK 1)

3.1 INTRODUCTION

UOP/SDC performed comprehensive evaluations of test data from the RI one tph flash hydroliquefaction PDU, since, for the data to be usable, they must be correlated. This was the first attempt to fully correlate the operating conditions affecting process yields.

3.2 CORRELABILITY OF DATA

The principal statistical analysis method used in the data evaluations was a multiple regression analysis. This determined a functional relationship between a dependent variable Y (product yield) and several independent variables $X_1, X_2, \dots, X_k$ (operating conditions).

The multiple regression analyses were performed in a stepwise manner, sequentially adding independent variables to the model. If they reduced the estimated residual error, the result is not only a predictive model but also an assessment of the significance of operating conditions in influencing the product yield.

3.2.1 Total Carbon Conversion and Conversion to Total Liquid

The initial analyses produced linear mathematical models to predict the amount of carbon converted and the amount of liquid produced in the Rockwell one tph hydroliquefaction PDU. Predictions based on these models predict experimental results to within 2-3 percent of the actual yield.

A similar correlation was attempted to calculate the yield of individual fractions of the hydroliquefaction liquid product in the three boiling point ranges of C_5 - 400°F, 400-100°F, and greater than 1000°F. Useful initial predictive models were developed as a function of reactor temperature, residence time, and hydrogen to coal ratio.

The best models for Carbon Conversion and Liquid Production are:

$$C = 8.1 + 0.025T + 0.015t - 1.59D - 0.19G$$

$$L = 66.0 - .059t - .153G - .013T + 6.1 H/C$$

where

C = percent total carbon converted, range 50-60%

L = percent carbon converted to liquid, range 30-40%

T = reactor temperature (deg R)

G = coal mass flux (lb/ft² - sec)

t = reactor residence time, range 50-150 msec

D = reactor diameter (in)

H/C = hydrogen to coal ratio (lb/lb) (0.4 to 0.6)

3.2.2 Quality of Liquid Product

The data evaluation also examined the effects of variables on liquid product quality. There were not enough data to develop a correlation like that above. However, quality was found to be affected by three variables, at varying levels of confidence. Temperature appeared to be a major parameter, as shown in Figure 3.1. The hydrogen to coal ratio may also significantly affect liquid product distribution, with a higher H/C ratio appearing to produce more heavy liquids.

3.2.3 Effect on %C and %L of Various Specific Parameters

3.2.3.1 Residence Time

No measurable effect of residence time on the quality of the liquid was possible. Additional data are required to determine preferred residence time.

Figure 3.1 Temperature Versus Liquid Yields.

3.2.3.2 Hydrogen to Coal Ratio (H/C)

A knowledge of the hydrogen requirements is essential in assessing process economics. Most of the test data were taken for H/C values in the 0.4 to 0.6 range, which are necessary to produce desired reactor temperatures. The design basis specified by Rockwell for its latest commercial plant conceptual design and economics has included a much lower H/C ratio of 0.2 and use of the unproven "hot hydrogen" injection mode to achieve optimal levels of conversion of 40% to liquids and 35% to gas. Analysis of the data suggests that such a reduction in H/C may reduce both the carbon conversion and the conversion to liquid. Nothing more definite can be said, both since the testing to date was not run at this low level of H/C (0.2), which is outside the range of PDU runs, and since the H/C became cross-correlated with other variables by the design of the RI tests. Additional test runs to establish the effect of low H/C on product yield warrant a high priority.

3.2.4 Reactor Mixing Efficiency

The dynamic pressure ratio (DPR) is used by Rockwell as a measure of mixing efficiency in rocket engines. DPR is defined as the ratio of the density times velocity squared of the hydrogen gas to the density times velocity squared of the coal input. Simple RI plots have indicated a correlation between DPR and liquefaction product yields. Variations in yields have been attributed to non-optimum dynamic pressure ratio conditions. Analyses do not confirm the significance of DPR, and demonstrate high cross correlation between DPR and other parameters in the tests run to date. Thus, it is not evident that process design with "optimal mixing" will significantly improve yield, unless other conditions are also optimized.

3.2.5 Other Variables

The effects of reactor diameter (and length), velocity, and coal mass flux were difficult to isolate because of intercorrelations of these parameters in the tests run. A major result of the analysis was identification of intercorrelations among the operating parameters during PDU testing and corresponding

quantitative indications of low confidence levels for correlations between these parameters and product yields. This result demonstrates the fallacy of reaching definitive conclusions based on plotting a single variable versus yields, as reported by Rockwell. The full multivariable model is necessary to determine interrelationships appropriately. PDU tests were not run to identify the effect of variations in coal type, oxygen purity, and hydrogen dilution.

The assessment of the effects of individual parameters clearly indicates that the use of simple variable plots to correlate variables may lead to wrong conclusions as to the effect of changes in variables. The PDU data to date are inadequate to fully characterize the RI process or form a basis for designing a scaled up pilot plant.

3.3 QUALITY OF DATA

The RI test data were in many cases of questionable quality, with carbon closures as low as 80 percent reported. Rockwell assumed that unaccounted for carbon was liquid product. This assumption must be justified.

In January 1980, because of an instrument recalibration, data for more than a year back were changed by as much as 30 percent. Reported operating conditions were sometimes modified by instrument recalibrations. In the future tests, all instruments should be well calibrated prior to taking any data.

Ash and carbon accountability were as low as 50 percent. Insufficient data have been obtained on liquid quality; such data were obtained only in the last six months upon the insistence of DOE. The overriding limitations in the available data were caused by inadequate experimental design, which did not permit fully characterizing the process from the test data. However, even with the best quality data possible from the present PDU, it will not be possible to get really meaningful results from such short runs. Before the question of whether a pilot plant should be funded - or even considered - can be answered, 7- to 30-day runs must be made on a continuously operated PDU.

3.4 RECOMMENDATIONS

On the basis of this analysis, DOE has recommended to RI an additional test program to obtain the necessary PDU data. This program consists of a statistically designed fractional factorial set of tests and that number of test runs that will provide a reasonable level of confidence in the results. This test program should be conducted as soon as possible.

In the future, DOE should monitor selected tests and attend selected test reviews from this program to assess data validity and identify data discrepancies. Test results should then be incorporated into upgraded predictive models, with emphasis on improving the liquid product characterization (boiling point cuts). With such more complete results, inclusion of nonlinear characteristics of the independent variables may be called for by the correlation. The models should then be verified against selected experimental data and refined if necessary. They may then be fully used for economic and technical assessments and evaluations of the RI flash liquefaction process, as well as to support scale-up of pilot and commercial plant designs.

SECTION 4 - RESULTS OF EVALUATION OF ADEQUACY OF THE DATA AS BASIS FOR PILOT PLANT DESIGN (SUBTASK 2)

The objective of this subtask was to determine what gaps exist in the present PDU test data if they are to serve as the basis for the design of a pilot plant. This objective was met by: 1) defining a conceptual pilot plant, 2) determining what data are required to design this conceptual plant, and 3) determining the data gaps by comparing these PDU data with those gained from the present PDU testing.

The conceptual pilot plant design selected has already been described in Section 2, Background, Subsection 2.5, Proposed Pilot Plant Baseline Process Design. In this description, the utilities, auxiliary, and offsite units were assumed to be supplied by an outside source, such as a nearby petrochemical plant or refinery complex.

Design of a pilot plant without a better correlation of reactor variables would be dangerous indeed. Data on equipment performance in long runs (seven days minimum) would be essential. As shown in Section 3 of this report, the present data do not permit determination of the effects of a number of variables; there are problems with the quality of the data; and there are other data that cannot be obtained in the short runs (one hour or less) that are all of which the present PDU is capable.

UOP/SDC's review showed that there are gaps in the required design data for almost every section of the pilot plant. The following are some of the more significant of these:

- Data are lacking on the operability of the proposed lockhopper feed system with 90% (-) 200 mesh coal. It could prove inoperative because of high dusting losses or failure of the coal to flow smoothly.
- There are no data for a multiple-injector system in the reactor. Results from cold flow testing of a multiple injector system and from

hot testing to show the controllability of the small flows are required for the design of the pilot plant.

- Demonstration of the hot hydrogen mode of operation must precede its inclusion in the design.
- Data are needed to show whether the missing product carbon is liquid.
- Vital materials data are almost totally missing.
- For proper design of the reactor quench and particulate removal systems, the char must be characterized with respect to size distribution, densities, and flowability.
- Data on fouling characteristics, erosion characteristics, heat transfer coefficients, and possible mist formation must be determined for design of the quench and product condenser heat exchange systems.
- Data required to characterize H_2S , COS , mercaptans, oil, and BTX have not been gathered.
- Test data on the efficiency of BTX absorption and of the cryogenic H_2 separation are needed for design.
- Oil properties must be measured.
- No data have been gathered to show the effects of operation with impurities in the hydrogen feed. Commercial and pilot plant operation would use a hydrogen feed recycle, which means that impurities would also be recycled. Their effect on the reactor must be determined.

A major design difficulty is that the PDU has consistently produced ash and carbon balances averaging only 70-80% and char balances of only about 80-90%. The reason must be found before any confidence can be placed in the balances as the pilot plant design base.

It should be noted that the hot hydrogen mode of operation, recommended by Rockwell, has not worked successfully in the PDU.

The more significant of the data gaps standing in the way of designing a pilot plant are summarized in Table 4.1.

In addition, several other aspects need to be covered in the present PDU testing. First, testing with impurities in the hydrogen feed is a necessity. Commercial and pilot plant operation will use a hydrogen feed recycle, and consequently impurities will also be recycled. Finally, material testing is needed for the preburner area to determine how this high temperature can be contained.

Table 4.1 Present PDU Information Gaps

<u>Unit</u>	<u>Information Gap</u>
Reactor	Testing with impurities in H ₂ feed, cold flow testing with multiple injectors, systematic testing for determination of key variables.
Preburner	Testing with impurities in H ₂ feed. Design durability, material testing for hot hydrogen and preburner modes.
Quench	Erosion characteristics, fouling characteristics, particle size distribution, particulate loading, better ash and carbon balances in PDU.
Particulate Removal	Better ash balances, particle size distribution, particle density, bulk density, particulate loadings.
Oil Recovery	Oil characterization, fouling characteristics, better ash balance, separability of oil/water.
BTX Recovery	BTX analysis improvement, light oil characterization, tests of recovery data.
AGR	H ₂ S analysis.
Methanation	COS, mercaptan analysis of the feed gas.

SECTION 5 - RESULTS OF EVALUATION OF ROCKWELL'S CONCEPTUAL DESIGN OF A COMMERCIAL HYDROLIQUEFACTION REACTOR (SUBTASK 3)

5.1 INTRODUCTION

The prime objective of this subtask was to determine the practicability of building and operating a commercial scale reactor designed to Rockwell's specifications. The study involved the follow major areas: a general description; a detailed description and evaluation of the design, fabricability, operability, and reliability of the reactor; the economic feasibility of such a unit; and data gaps and required information.

5.2 REACTOR DESIGN CONCERNS

5.2.1 Design Description - Reactor Head

The conceptual design of the commercial reactor head proposed by Rockwell is shown in Figure 5.1. This design involves a reactor whose injector plate contains internal manifolds for hydrogen, oxygen, a cooling water network, and an appropriate system for transferring and metering the hydrogen and oxygen from their respective manifolds to achieve the proper gas flows. This design also involves 64 coal injectors (each with a capacity of 3.5 tph), yielding an overall reactor capacity of 224 tph. Each coal injector is surrounded by four jets that feed hot hydrogen into the reaction zone; these four jets intersect the coal stream approximately one inch away from the injector plate. It is claimed that this configuration results in excellent mixing within the reaction zone, which in turn promotes extremely fast reaction times with concomitantly high conversion of the coal. The hydrogen and a proper amount of oxygen for precombustion are metered by orifices separately to the jets from annular passages. This precombustion reaction boosts the temperature of the hydrogen to approximately 2500°F just before it enters the reactor.

Figure 5.1 Conceptual View of Two Quadrants of R.I. Flash Liquefaction Reactor Head Showing Complicated Reactor Injection System

5.2.1 Injector Spacing and Symmetry

The injectors are spaced equidistantly along the radii of the reactor head. However, it is not possible to make the spacing between adjacent injectors uniform.

The pattern chosen for laying out the injectors is efficient from an overall point of view, since it gives rise to a reactor with a relatively small diameter (about 4 ft). However, certain inconsistencies exist in this pattern. As one proceeds radially toward the center of the injector plate, it becomes physically impossible to achieve the 90° angle between adjacent jets; the included angle between the two jets being fed by the central oxygen annulus approaches 45°. Another anomaly noted is the overlapping of oxygen inlets in the annular passageways as one proceeds towards the center of the reactor. This is totally unsatisfactory, since it could lead to interference problems during fabrication and also to flow problems. Yet, there is no alternative; increasing the reactor diameter reduces the reactor velocity to an unacceptably low value. Accordingly, the feasibility of the entire reactor concept is questionable.

5.2.2 Coal Flow

Another design problem is the fact that the coal feeding lines (in every case) pass through the hot hydrogen manifolds, which are at 1500-1600°F. It can be visualized that some of the fine coal will gradually become plastic and form a layer on the tubes that will build up with time and result in plugging of the coal inlets. This is quite serious: it would cause a plant shutdown, and repairs would be difficult if not impossible. The question of whether or not these coal lines will have to be cooled will have to be addressed.

5.2.3 Fabricability

Fabrication of the Rockwell reactor will be extremely difficult because of its complexity, relative smallness, need for exotic material, and the relatively severe conditions.

The reactor head is the principal problem. Its face contains 320 separate injection points in a four foot round area; their size and orientation must be precise. Extensive welding of small members, typically one inch in diameter, in very close quarters is required.

The spacing is extraordinarily close at the places where the sixty-four coal feed lines run into the head through the hydrogen distribution annuli. Here the welder would be required to work with 1/2" clearance between the line and the wall of the well into which it runs that is perhaps a few inches deep. In all probability, such welding is impossible.

Another major problem is the need to assemble and join the plates of which the reactor head would be composed. Annuli would have been machined into one or both of these plates to provide separate passages for oxygen, hydrogen, and cooling water. It would then be necessary to seal these to prevent leakage from one to another. Other specific problems are noted above and below.

5.2.4 Internal Flow - Hydrogen

The question of flow through the internal hydrogen manifold must be given very careful consideration. This reactor must handle a very large amount of hydrogen, and each internal hydrogen annular passageway must handle different flowrates (see Table 5.1).

Table 5.1 Hydrogen Flow Rates

<u>Annulus No.</u>	<u>No. Coal Injectors Fed</u>	<u>lb/hr H₂</u>	<u>ACFM</u>
1 (smallest)	4	5,562	1,256
2	8	11,125	2,511
3	16	22,250	5,023
4	16	22,250	5,023
5 (largest)	<u>20</u>	<u>27,813</u>	<u>6,279</u>
TOTAL	64	89,000	20,092

Insuring that each coal injector (and its associated jets) receives the same amount of hydrogen will require careful pressure and flow balance and freedom from buildup during use. RI will have to tailor the size of internal manifolds to accommodate the actual flow rates of hydrogen. Also, the possibility of pressure shocks and dynamic instability will require careful consideration; cold flow testing will be warranted to establish the critical parameters. Whether these passageways will remain clean as impure H_2 is recycled is highly uncertain. Similar comments can be made about the oxygen distribution system manifolds.

5.2.5 Stress Analysis of Reactor

A cursory thermal stress analysis of this concept was conducted previously by UOP/SDC. Although this analysis was very preliminary, it did indicate that material selection would be quite important. It pointed up extremely stressed areas where cooling water and 1500 or 2500°F H_2 were in close proximity. This study concluded that the proposed design was not feasible.

5.2.6 Reactor Instability - Orifices

The orifices for providing the vital close control flow to the oxygen and hydrogen jets are essentially buried within the injector plate. Once installed, there is no way to calibrate them. Depending on service, commercial orifices are generally recalibrated at least once a year. The ability of these hydrogen and oxygen orifices to survive for a long period of operation is essential to the success of this design. This is particularly true of the oxygen system, since its flow rate determines the temperature of the hydrogen entering the reactor. Failure to control this flow could result in reactor instability (excessive temperatures leading to component and material problems and ultimately a catastrophic failure). Yet, the oxygen orifice is only 0.08 inch in diameter. There is also concern that use of recycled hydrogen (which contains some methane) may foul the orifices through carbon depositing because of the high temperature in this area.

5.2.7 Injector Flow Rate

The capability of the coal injector to handle 3.5 tph is questioned. Prior experience has only demonstrated flow up to 1 tph. Of course, if the rate were set at 2 tph, for example, the reactor would grow both in size and complexity. Also, reactor velocity would decrease and could become unacceptable because of the reactor L/D that would result.

5.2.8 Multiple Injector Interaction

Other unknown factors, at this time, are the interaction of multiple injectors and whether the mixing achieved with a single injector will be maintained. Some cold flow testing is required here before a commercial design can be carried out with a degree of confidence.

5.2.9 Injector Malfunction Detection

A problem common to all multiple injector designs is the detection of potential malfunctions, such as plugging of coal feed lines. Even after the fact, there appears to be no way of determining whether a specific line inside the head has plugged.

5.2.10 Layout

Although this concept yields a relatively small reactor overall, the external piping arrangements for coal, hydrogen, and oxygen become extremely congested and difficult to assemble and maintain.

5.2.11 Cooling Water Analysis

Another area that will require study will be the cooling water system. Analysis of this system will have to be closely coordinated with the thermal stress analysis of the design that finally is evolved.

Here, one extreme of the potential problems is that keeping sensitive parts of the reactor head cool enough will cause so great a heat loss to the cooling water that the amounts of oxygen and hydrogen required for precombustion will increase significantly. The other extreme is that it will not be possible to keep the sensitive coal and oxygen lines cool enough.

5.2.12 Material - High Temperature

The combustion of hydrogen and oxygen imposes a severe material problem because of the high temperature (2500°F). It must take place inside the small diameter (about 1") individual line to a hydrogen jet in the hot hydrogen mode of operation.

5.2.13 Nozzle Integrity

The coal and hydrogen inlets (which really are orifices) will be subjected to a very severe environment; most likely they will suffer significant degradation unless provided with some means of cooling. Whether the proposed method of circulating cooling water is adequate is problematical. Cooling the tip of the coal inlet nozzle (if it is required) will be an added design and fabrication complication. Use of insulation for the injector plate face might be required to promote the reactor's reliability, but this would cause other problems.

5.2.14 Reactor Internal Insulation

Rockwell has shown an internal reactor liner consisting of "refractory metal." Just what this material might be is not known at this time; use of other types of refractories should also be considered for this application.

5.2.15 Safety Considerations

Consideration must be given to providing a means of pressure relief for the reactor. Also, instrumentation (i.e., strain gages) for determining the status of the metal response may be required.

5.2.16 Reactor Quench

The heat recovery quench surface exchanger is another area where further testing is indicated. Long term runs are needed to evaluate the fouling effects, erosion data, heat transfer coefficients, etc., of these units. A multi-tube assembly would be preferred in order to assess the unit from a fabrication and operational point of view.

5.2.17 Instrumentation

There is insufficient information on the control and measurement of the rate of dense phase flow of coal where an initial stream must be split into sixty-four precisely equal streams. The difficulty of metering with suitable precision some 250 flow streams of hydrogen and oxygen has already been mentioned.

5.3 ECONOMIC FEASIBILITY

Assessment of the economic factors involved indicates that the RI reactor will cost just as much as reactor systems in other liquefaction processes, despite its smaller size. The heat recovery quench surface exchanger is unusually costly because of an unproven design, the use of high alloy materials, and a large thermal gradient to overcome. In addition, because of anticipated erosion, maintenance and operating costs should be higher than normal.

The reactor injector system probably imposes the greatest economic burden on the reactor of any of the elements. The solution to the design and fabrication problems will be costly. Malfunctions of the feed system, such as plugged coal lines and burned-out injector systems on the gas feeds, will force complete reactor disassembly. These unusual maintenance problems result from vital elements' being sealed inside and the close proximity of the individual elements.

For all coal conversion processes that have been cost estimated in the past, the reactor section of the plant has accounted for about 10-15% of the total plant investment. The RI hydrogasifier is smaller than most reactors, but it is internally more complicated in the feed and quench sections than others.

The cost estimate (\$930,000 per reactor/quencher) provided by RI seems extremely optimistic. For another project, RI estimated the cost of a single hydrogasification reactor at \$2,600,000. The hydrogen recuperator (used instead of a quencher) was estimated to cost \$5,700,000. Although the latter is a different concept than the heat exchanger intended for the hydroliquefaction program, certainly the latter would probably be in the range of \$500,000-\$1,000,000 per unit. The RI estimate looks low.

5.4 SUMMARY

The following are the more important design problems that have been noted in this study.

- 1) The accuracy of the RI dense phase coal feeding system and its ability to split this coal into 64 equal streams has not been determined.
- 2) The data base -- both chemical and mechanical -- is incomplete. Long runs and scale-up data are needed. Long runs are needed for materials, erosion, reliability, and fouling data.
- 3) The injector coal lines are exposed to high temperature (about 1500°F) hydrogen. This could cause plugging problems over a sustained period of operation. If cooling or insulation of these lines is required, it will add both to the complexity and cost of the reactor.
- 4) The H₂ injector line where the H₂ combustion occurs is subjected to a temperature of 2500°F.
- 5) The geometrical spacing for the injectors does not permit maintaining the desired configuration of the four-on-one injector design. This problem is accentuated as injectors approach the center of the reactor. It appears that this concept might lose a number of injectors because of this problem. Thus, the reactor's capacity, as well as the reactor's length (which is related to residence time) are in question.

- 6) Balancing and closely controlling the flows of both oxygen and hydrogen, as is required, may prove to be a very difficult problem, particularly since the various oxygen and hydrogen annuli are handling different amounts of these gases.
- 7) The reliability of the hydrogen and oxygen orifices is an area of grave technical concern. The oxygen orifice diameter is only 0.08 inch. Because of the method of fabricating this design, it is virtually impossible to service or maintain the orifices once the reactor head components are assembled and welded together. Entrained scale or carbon or other deposits could quickly throw off the required close control.
- 8) The reactor head is essentially unserviceable except by replacement.
- 9) There is doubt that the interior face of the injector can withstand or survive the reactor's severe environment, even with provision of water cooling in the face plate. It is even conceivable that cooling the face can give rise to severe thermal gradients. Use of insulation for the injector face plate would present a host of additional problems, since it would impact on the injector design and mixing efficiency as well as reactor fabrication.
- 10) As presented, the RI design shows cooling water entering the outermost annulus in the face of the head; however, no provision for bringing this water into the inner cooling water annuli is given. Interconnecting these annuli will present additional stress problems. Also, fabrication will become more complicated and undoubtedly result in a higher reactor cost.
- 11) A 3.5 tph coal injector capacity is necessary to attain the desired velocity in a commercial reactor. However, such a high feed rate through a 1-inch injector nozzle has never been demonstrated. Neither has the use of multiple injectors been verified.
- 12) The use of a refractory with a metal liner (e.g., stainless steel) backed with insulation is seriously questioned for the commercially sized reactor.

Determining the cost, fabricability, and reliability of this configuration would depend on finding answers to many unknowns.

- 13) The small clearance (1/2") between the vertical coal lines and the walls of the hydrogen annuli will make the required welding impossible. The oxygen lines (feed to the oxygen manifolds) are quite close to some of the coal lines. This will present a fabrication problem.

5.5 CONCLUSIONS

The major overall conclusion drawn from the results of this study is that it would be undesirable to attempt design of a commercial reactor at this time. A durable hydrogen precombustor must be demonstrated in the PDU prior to design work.

SECTION 6 - RESULTS OF EVALUATION OF FEASIBILITY OF THE PRESENTLY USED H₂ THROUGHPUT AND THE RECYCLE REQUIREMENTS (SUBTASK 4)

The hydrogen utilization subtask evaluated the feasibility of using the present through-put of hydrogen, including the use of hot hydrogen injection, and the hydrogen recycle requirement. This objective was achieved through studies of the data base, of sensitivity of H₂ consumption, of hydrogen preheat, of economics, of hydrogen losses, and finally of downstream optimization. The additional PDU testing data required were identified.

The hydrogen consumption and hydrogen circulation were found sensitive to (1) gas analysis and (2) carbon conversion to gas because of the high hydrogen content of components of the gas. Emphasis should be placed on the accuracy of these parameters in future PDU testing.

The hydrogen preheat study evaluated the potential methods for providing heat to the reactor hydrogen feed. Through recovery of waste heat and use of a fired heater, the hydrogen should be preheated to the maximum temperature permitted by allowable stress of the equipment. At 500 psig, this temperature is 1500-1600°F. The remainder of the heat should be provided by a hydrogen precombustor encased in a pressure vessel to relieve much of the stress due to pressure. The precombustor mode of hydrogen combustion (2000°F) is preferred over hot hydrogen injection (2500+°F) because of its better testing success in the PDU and lower operating temperature.

The economic study determined that no one unit or section contributes a disproportionate share of the total capital cost.

The hydrogen consumption study determined areas where H₂ disappears in the process, estimated the relative importance of these losses, and evaluated alternatives for hydrogen use reduction. Ninety-seven percent of the hydrogen disappearance is caused by reactions in the reactor, the methanator, and the preburner. The remaining 3% represents hydrogen dissolved in oil products, benzene, absorber solvent, AGR solvent, and SNG product.

The final study, the hydrogen separation and raw gas recycle study, evaluated the applicability of various hydrogen separation options and of raw gas recycle to the Rockwell process. This study is believed to be important in the overall scope of this hydrogen utilization subtask because of the potential of cost reduction from raw gas recycle and because the hydrogen separation unit and a raw gas recycle result in impurities' being recycled with the hydrogen to the reactor.

A partial condensation cryogenic unit was chosen as the best means of hydrogen separation for the RI process. Although it produces a separated hydrogen with approximately 5% methane content, it offers the lowest operating and capital costs of any of the cryogenic options.

Raw gas recycle is recycle of gas before methanation, or possibly before AGR, directly back to the reactor to reduce equipment size downstream of the bypass. This is a potentially significant improvement in the processing scheme because otherwise all the processing equipment must be capable of handling the great quantities of hydrogen circulated to heat up the reactants. Therefore, any reduction will affect the overall cost significantly. The results of this analysis indicate that about 60% of the reactor effluent gas can be recycled after oil recovery directly to the reactor, thus making it necessary to process only 40% of the total gas in the AGR, methanation, cryogenic hydrogen separation, and recycle gas compression. The impurities were estimated to build up to only 10-15%, depending on whether the impurities are assumed to react in the reactor to equilibrium or merely serve as diluents. The raw materials to the process did not significantly change until bypasses were in excess of 60%. If, as in the first assumption, the impurities recycled with the hydrogen come to thermal equilibrium in the reactor, the resultant exothermic formation of CH_4 not only adds to yield but also reduces the H_2 preheat required and eliminates the need for a methanator. This would be very valuable if confirmed.

Also, this "equilibrium" case, if it proves to be the way the reactor behaves, eliminates the advantage that the hot hydrogen mode of precombustion has - i.e., the lower H/coal ratio requirement. This is because most of the hydrogen

required for the precombustor mode can be supplied by this recycle of raw gas. This is an advantage because of the very severe concern over materials problems for the hot hydrogen burner. That tested in the PDU burned out.

Raw gas recycle at the rate of 60% appears to have the potential for reducing downstream processing equipment by about 40%. Impurities are estimated to build up in the recycle to only 10-15%, depending on whether they simply reduce the partial pressure of the H_2 or alternatively come to thermal equilibrium in the reactor. The latter case provides a valuable increment of exothermic reaction heat to the reactor that reduces its total gas requirements.

In either case, gas will be recycled, and, whether it is "raw" or "separated," it will contain some CH_4 and CO . As a result, there is danger that these diluents will decompose in the preheater, preburner, or reactor and cause carbon deposition that would shut the process down. The hot hydrogen mode represents the greatest danger because of its higher precombustion temperature. This is another reason for preferring the preburner mode. Other factors, such as the net amount of downstream processing, are nearly a standoff.

The conclusions of the study are given below.

The precombustor mode of H_2 precombustion looks better than the hot hydrogen mode because of the lessened danger of plugging through carbon deposition and less severe materials problems.

However, it still is not certain whether use of the present H_2 throughput is feasible because of the many important unknowns. These include:

- Nothing is known about the long term operability of the RI equipment.
- RI has not tested H_2 with diluents. Nothing is known of possible carbon deposition or effects on product slate.
- Nothing is known about scale-up of the reactor.

- There is the danger that trace components from recycle will plug a cryogenic separator and require a different process.
- UOP/SDC's correlation indicates that use of hot H₂ injector conditions will change the product slate to less liquid and lower total conversion.

The study had the following recommendations:

- The calculations that have been used in this study depend on some unproven assumptions. Hence, it is necessary to confirm feasibility of recycle of impure H₂ and estimate the recycle requirements.
- Material testing in the PDU should be undertaken to determine the maximum allowable temperatures in the preburner and in a direct hydrogen fired combustor at 500 psig.
- Cost reduction should be emphasized in the hydrogen plant and reactor systems, as they represent the largest individual contributors to the total capital cost.
- The reactor, methanator, and preburner should be evaluated for possible alternatives for reduction of hydrogen consumption.
- PDU testing is needed immediately to determine the effects of impurities, primarily CO and CH₄, on the preburner and reactor systems. Emphasis should be placed on the effect of impurities on potential formation of carbon in the preburner, on the deterioration of liquid quality and quantity, and on gas quality and yield. The impurity concentration that should be tested is 0-15% for methane and 0 to 5% for carbon monoxide.
- A partial condensation cryogenic unit appears to be the best cryogenic hydrogen separation alternative.

SECTION 7 - RESULTS OF EVALUATION OF THE EFFECTIVENESS OF THE QUENCH SYSTEM IN RECOVERING LIQUIDS (SUBTASK 5)

7.1 INTRODUCTION

The hydroliquefaction reactor product must be quenched rapidly from about 1700°F to 1000°F to recover the liquid product by freezing the reaction and avoiding decomposition of the liquid produced. Rockwell has used two techniques, (1) water spray and (2) a water cooled heat exchanger-quench exchanger as the coolant.

7.2 EXCHANGER QUENCH

7.2.1 Effect on Liquid Yield

To address this topic, the following activities were performed:

- (a) The reactor effluent stream characteristics for an 18,000 tpd reactor were computed by scaling from the 250 tpd Pilot Plant.
- (b) The overall heat transfer coefficient U was calculated for a commercial single-pass counterflow shell and tube exchanger using the hydrogen recycle stream as the coolant. The overall heat transfer coefficient was estimated to be about 200 Btu/hr-ft²-°F. The exchanger surface area was computed as 15,600 ft².
- (c) The resultant exchanger had 1 inch BWG tubes with a 1.25" triangular pitch in a shell diameter $D_S = 82$ inches and a tube count $N = 3,740$ tubes.
- (d) The pressure drop for the single-pass counterflow exchange was computed as 5.5 psia tube side and 27.5 psia shell side.
- (e) The temperature vs. time history of the product stream in the exchange-quench was calculated.

Rockwell reported its liquid yield in terms of total and the percent converted to each of the three liquid cuts - C₅ - 400°F to 1000°F, and >1000°F.

7.2.2 Liquid Yield Versus Quench

The linear regression model developed in Subtask 1 was used to relate liquid yield in the reactor to residence time, t_r :

$$L = -(.00059)t_r - (.00142) G - (.00015) T + (.1501) H/C + .6766 \quad (1)$$

For the heat exchange quench analysis, it was assumed that the average values H/C and G remain constant in both the reactor and heat exchanger at $G = 21.9$ and $H/C = .39$. This reduces equation (1) to:

$$L = -.00059t - .00015T + .704 \quad (2)$$

Care should be taken in extrapolation of the above outside of the range of experimental data. It should be noted that the relationship is good only for total time, not increments of time. The equation is consistent with the fact that, in the reactor, when T is constant L decreases with time. The effect of the quench on liquid yield is calculated using equation (3) below, in which:

T_i and t_i are the average temperature and the residence time in the i^{th} segment of the exchange quench

L_{T_i, t_i} is the liquid yield out of the reactor plus the exchanger to that point from equation (2) above at T_i and t_i .

i ranges from 0 to N, where N is the number of exchange quench segments. L_i is the liquid yield at the end of the i^{th} segment.

$$L_i = 1 - \left[\frac{L_{Ti,ti-1} - L_{Ti,ti}}{L_{Ti,ti-1}} \right] L_{i-1} \quad (3)$$

L_1 thru L_N are calculated in turn, and L_N is the liquid yield out of the combination of reactor plus exchange quench.

Table 7.1 gives the product, temperature, time, and overall liquid yield at 4-ft intervals for two reactor temperatures. The value of the liquid selectivity L is computed from Eq. (3) at 4-ft intervals.

The results are plotted in Figure 7.1. The liquid selectivity decreases by about 10% in the quench. A more detailed analyses is given in Reference 5. While the Rockwell tests in which the heat exchange quench was substituted for the faster direct water quench indicated little change in liquid yield, their experimental accuracy is such that replicate tests would be required to have confidence in this conclusion.

7.3 OPERABILITY AND COST OF EXCHANGE QUENCH

The combination of high grain loading (about 5×10^4 gr/scf), high velocity (about 200 ft/sec), high temperature, and reactive components will produce a severe erosive environment on the tube side. Data are needed from long continuous runs at the PDU to evaluate materials for the exchange quench on the shell side. The recycle hydrogen or water coolant operates at a low velocity and has virtually no grain loading, therefore erosion should not be a problem there.

The cost of a single exchanger made of Incoloy 671 having approximately $16,000/6 = 2,677$ ft² of heat transfer surface is estimated at \$1.88 million (\$700/ft²). Since six quench sections are needed for the whole plant, their total cost is \$3.6/MM Btu/day, which compares with \$700/MM Btu/day for the whole liquefaction process. Thus, the quench section above represents less than 1% of the plant cost.

Table 7.1 % L - Selectivity Calculations

At Reactor Temperature of 1700°F (2160°R)

<u>X*</u> (ft)	<u>T</u> (°R)	<u>t</u> (msec)	<u>% L Conversion</u> <u>to Liquid in</u> <u>Reactor and Quench</u>	<u>% Decrease</u> <u>in Liquid Yield</u>
0	2160	0	0.38	0
4	1975	14.8	0.37	2.6
8	1688	42.6	0.36	5.3
12	1577	69.2	0.35	7.9
16	1460	98.4	0.345	9.2

At Reactor Temperature of 1750°F (2210°R)

0	2210	0	0.37	0
4	2025	14.8	0.36	2.7
8	1738	42.6	0.35	5.4
12	1627	67.2	0.34	8.1
16	1510	98.4	0.33	10.8

* X = Distance into quench from reactor inlet

Figure 7.1 Liquid Selectivity Versus Quench

7.4 DATA GAPS

The following additional test data are necessary to provide the basis for a satisfactory quench unit commercial design:

- Composition of product with position and time in the tube.
- Temperature distribution of product in the tube.
- Reaction rates of product in the tube (to predict liquid selectivity and quality).
- Effect of char on heat transfer rates.
- Extent of tube side fouling, corrosion, erosion, or plugging resulting from long term operation.
- Extent of shell side fouling, corrosion, erosion, or plugging resulting from long term operation.
- Data on properties and yields of individual liquid cuts vs conditions.

7.5 DIRECT WATER QUENCH

A model was postulated based on the evaporating of water droplets injected into the product stream. It was calculated that a spray of 200 micron (6.56×10^{-4} ft diameter) droplets injected at a high velocity relative to the gas stream would quench the product stream almost ten time faster than a surface type shell and tube exchanger (about 10 msec vs. 100 msec) for the same heat load. The high velocity of the droplets relative to the product gas stream is important in obtaining a high heat transfer rate between the product stream and the droplets.

7.6 CONCLUSIONS

It is estimated that the carbon converted to liquid product decreases in the heat exchange quench by 7.9%.

Above all, more and better data are needed. The above analysis of the effect of quench on liquid yield does not consider the effect of the quench on the yield of the individual liquid cuts: there are not adequate data to predict this effect.

Moreover, the fact that the data and correlation of total liquid yield vs operating variables are weak and hopelessly cross correlated makes the conclusion about the effect of the quench on total liquid yield uncertain.

Finally, there are serious grounds for concern that erosion, fouling, or corrosion may render a heat exchange quench inoperative, or that thermal stress may cause failure. This last is likely with the product at 1000° F and the coolant at 100° F. The only way to develop an answer to this concern is through operation of a PDU for an extended period for at least 7 to 30 days.

SECTION 8 - RESULTS OF EVALUATION OF SUPPLEMENTAL COAL FEED SYSTEMS (SUBTASK 6)

8.1 INTRODUCTION

In this study, the high-pressure lockhopper coal feed system proposed by Rockwell, a slurry feed system, and the Jet Propulsion Laboratory (JPL) extrusion feeder were evaluated. The lockhopper and slurry feed systems would be integrated with the dense phase system developed by Rockwell International. The data on a lockhopper system were taken from Rockwell's hydrogasification study, the data on the slurry feed system were taken from the information provided by Cities Service company, and the data on the JPL extrusion feeder were developed from JPL reports.

8.2 LOCKHOPPER WITH DENSE PHASE SYSTEM

Low-and high-pressure lockhoppers are used in series to supply ground coal to a high-pressure storage vessel supplying the Rockwell dense phase feed system.

In the Rockwell dense-phase system, the coal feed rate is adjusted by adjusting the pressure drop between the feeder and reactor. A complete lockhopper system along with a dense phase feeder is shown in Figure 8.1.

8.3 SLURRY FEEDING SYSTEM

In the slurry feed system, pulverized coal is mixed with a mixture of benzene, toluene, and xylene (BTX) produced in the hydroliquefaction process. Coal slurry is pumped through a coal slurry heater at a temperature above 550°F. The coal slurry is directed to a flash drum, where BTX is stripped from the coal by a hot hydrogen stream. The dry coal then flows by gravity into the pressurized coal hoppers and then to the dense-phase coal feed system. The slurry feed system is shown in Figure 8.2.

Figure 8.1 Lockhopper System

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Figure 8.2 Slurry Feed System

8.4 EXTRUSION COAL FEEDING SYSTEM

In the Jet Propulsion Laboratory (JPL) extrusion feeder system, coal becomes semi-fluid (plastic) under the action of heat and pressure. The resultant viscous fluid coal at elevated pressure is sprayed at 400°F into the flash liquefaction reactor.

In a commercial plant, a array of extrusion feeders is used for each reactor. Each extrusion feeder has its individual feed hopper. The extruders are equipped with downstream venting devices to discharge volatile matters produced in the extruder.

8.5 ECONOMIC COMPARISON

A summary of total estimated installed and operating costs of the three coal feed systems is shown in Table 8.1. From the table, it appears that the lock-hopper system is cheaper than the other two systems.

8.6 OPERABILITY

Information on satisfactory coal feeding systems for high-pressure, high-temperature coal conversion reactors is limited. Problems that are likely to show up with the lockhopper system are (1) excessive dust losses of the 90% (-) 200 mesh coal feed in the lockhoppers, (2) unavailability of valves large enough for the lockhoppers, and (3) failure of the 90% (-) 200 mesh coal to flow where required through the system because of its talc-like consistency. Experience with lockhoppers, even feeding coarser coal, has not been good, but there has been more experience than with the other two systems.

Hands on experience with slurry feed systems has been somewhat better in other processes. Based on experience with HyGas, slurry feed appears to be more reliable and easier to maintain than a lockhopper system. Use of a slurry feed system would, however, impose a greater cost on the process and would have to be proven before it could be adopted for the pilot or commercial plant.

Table 8.1 Installed Capital Costs Comparison

	<u>Lockhopper with Dense Phase Feed</u>	<u>Slurry Feed with Dense Phase System</u>	<u>Extrusion Feeders</u>
Equipment	\$ 4,600,000	\$ 5,040,000	\$ 4,500,000
Installation	12,800,000	11,200,000	17,500,000
Contingency	3,000,000	3,000,000	3,000,000
	<hr/>	<hr/>	<hr/>
	\$ 20,400,000	\$ 19,240,000	\$ 25,000,000
Operating Cost	1,300,000	\$ 5,600,000	\$ 2,500,000

Commercial operability of the JPL process cannot be evaluated because the technique is in its infancy.

SECTION 9 - RESULTS OF EVALUATION OF UPGRADING OF ROCKWELL COAL LIQUIDS
(SUBTASK 7)

9.1 EVALUATION OF UPGRADING OF ROCKWELL COAL LIQUIDS

The need for and possibility of refining the liquid product from the Rockwell International Coal Flash Hydropyrolysis process was evaluated based on the data that are available on its properties. Rockwell has only recently made available information on the properties of its liquid products. It is not certain how reliable the Rockwell data are. This can be concluded from the fact that Rockwell had difficulty providing UOP with representative samples of its liquid product for the three analytical crosschecks UOP made. Rockwell's problems appeared to be: 1) loss of light ends from the first light oil sample; 2) failure to include a gas sample in the first sample set; 3) lengthy delays in dispatching sets of samples; 4) emulsified water in a light oil sample; 5) leakage of air into a gas sample; and 6) some evidence of non-homogeneity of the heavy oil sample. Consequently, only one of three runs checked (79-40) was used as a basis for evaluation of the commercial utility of these liquid products.

The as-is crude Rockwell products are clearly not marketable as any industrial fuel. This is made evident in Tables 9.1 and 9.2, in which ASTM specifications for liquid fuels are compared with the crude Rockwell liquids. The light oil fails to meet the gravity, boiling range, and sulfur specifications for No. 2 fuel. The heavy oil fails to meet the specifications for residual fuel in that it is solid below 190°F. It is also too high in sulfur and nitrogen content.

The crude Rockwell heavy oil probably would not even be suitable for use in the coal conversion plant as plant fuel. This is because, in burning this heavy oil, the high viscosity and heptane insolubles levels could cause feed control, atomization, and burner tip plugging problems, particularly if burning were attempted without blending with a conventional cutter stock. Both Rockwell coal oils would present environmental problems in terms of SO_x and NO_x emissions unless a flue gas treating operation is included.

Table 9.1 Comparison of Rockwell Light Oil with ASTM Standards for No. 2, No. 4, and No. 5 Fuel Oils

	<u>Light Oil</u>	<u>No. 2</u>	<u>ASTM Standards</u>	
			<u>No. 4</u>	<u>No. 5 (Light)</u>
Gravity, API	4.3	30 (min)	--	--
Distillation, °F				
10%	199		--	--
90%	713	540-640	--	--
Viscosity				
Furol at 250°F, SFS		--	--	--
Kinematic at 100°F, CST	2.2	2.0-3.6	5.8-26.4	>26.4-65
at 122°F, CST	1.7	--	--	--
Ash, wt-%	0.001	0	0.10	0.10
Sulfur, wt-%	1.0	0.5	--	--

Table 9.2 Comparison of Rockwell Heavy Oil with ASTM Standards for No. 6 Fuel Oil

	<u>Heavy Oil</u>	<u>ASTM No. 6</u>
Gravity, API	-20.5	--
Distillation, °F		
10%	739	--
90%	--	--
Viscosity		
Furol at 250°F, SFS	87.3	>45-300 (at 122°F)
Kinematic at 100°F, CST	Solid	--
at 122°F, CST	Solid	>92-638
Ash, wt-%	1.82	--
Sulfur, wt-%	0.9	--*

* No ASTM standard, but EPA standard applies.

Also, the storage stability of the RI crude products was questioned because it was found that UOP's distillation data could be interpreted to indicate that the product light oil is highly unstable. A sample sent by Rockwell to UOP over a month after it had been drawn from a run was found to have a much lower distillate content than that of a fresh sample, possibly because the former had polymerized. This would indicate not only that storage life of the light oil would be short but also that the bottoms temperature in commercial fractionation would have to be severely limited to prevent coking. (Although other coal liquids - H-Coal, EDS, and SRC-II - also show a tendency to polymerize, they do so to a lesser extent.) Consequently, proper hydrogen pretreatment and handling of the Rockwell liquid seems to be essential. This processing step would stabilize the material and minimize degradation but add to its cost.

Hence, it is apparent that refining of the Rockwell liquids is necessary. To show the extent of this need, the H/C atomic ratio and hydrogen content of the Rockwell light oil is compared to those of other coal-derived liquids in Table 9.3, and the H/C ratio of the Rockwell heavy oil is compared to those of other fuels in Table 9.4. The naphtha fraction of the liquid distillates from the H-Coal, EDS, and SRC-II processes has been removed in a prior fractionation, and consequently the samples represented in Table 9.3 are heavier than the total coal liquid distillate actually produced from each of the processes. Even so, the Rockwell light oil is in comparison extremely hydrogen deficient, which may explain its suspected instability.

The Rockwell heavy oil is lower in H/C than even anthracite coal (Table 9.4). This extremely low hydrogen content will render upgrading both difficult and expensive.

Comparisons of detailed analyses of Rockwell Light Oil vs EDS Gas Oil and SRC-II Process Solvent and of Rockwell Heavy Oil vs H-Coal Bottoms are shown in Tables 9.5 and 9.6.

Mobil Oil finds that refining cost of such liquids "is a function of hydroprocessing severity (i.e. hydrogen input) which in turn is a function of feedstock quality."¹⁰

Table 9.3 Comparison of Rockwell Light Oil with Other Coal Liquids

<u>Fuel</u>	<u>%H(wt)</u>	<u>%C(wt)</u>	<u>H/C Atomic Ratio</u>
H-Coal Distillate	10.1	88.8	1.3
EDS Distillate	8.7	86.0	1.2
SRC-II Distillate	8.4	88.0	1.2
SRC Recycle Solvent	7.7	87.5	1.1
Rockwell Light Oil	6.6	90.7	0.9

Table 9.4 Comparison of Rockwell Heavy Oil with Other Fuels

<u>Fuel</u>	<u>%H(wt)</u>	<u>%C(wt)</u>	<u>H/C Atomic Ratio</u>
Texas Crude Oil	11.5	86.3	1.6
H-Coal 400+ Fuel Oil	9.4	89.3	1.3
Coal Tar	8.7	87.0	1.2
Lignite Coal	5.4	65.1	1.0
Sub-Bituminous Coal	5.1	67.7	0.9
Bituminous Coal	4.8	71.5	0.8
SRC	5.7	88.5	0.8
Anthracite Coal	4.3	73.8	0.7
Rockwell Heavy Oil	4.6	89.6	0.6

Table 9.5 Comparison of Analyses of Rockwell Light Oil with EDS and SRC-II

	<u>Rockwell</u> Light Oil Run 79-40	<u>EDS</u> Gas Oil	<u>SRC-II</u> Process Solvent
API at 60°F	4.3	7.6	7.0
Sp. Gr. at 60°F	1.0420	1.0173	1.0217
Distillation, °F			
IBP	158	278	343
5, vol-%	185	428	
EP	879	978	862
<u>10</u>	<u>199</u>	<u>435</u>	<u>478</u>
20	212	452	507
30	358	469	529
<u>40</u>	<u>434</u>	<u>511</u>	<u>558</u>
50	475	561	583
60	521	619	612
<u>70</u>	<u>571</u>	<u>720</u>	<u>633</u>
80	632	825	678
90	713	908	752
<u>95</u>	<u>822</u>	<u>953</u>	
ED	879	978	862
% Over	96.0	96.0	
% Bottoms	4.0	4.0	
Hydrogen, wt-%	6.62	8.73	8.44
Carbon, wt-%	90.71	86.00	88.04
Sulfur, wt-%	1.00	0.65	0.25
Nitrogen, wt-%	1.08	0.46	0.98
Oxygen, wt-%	0.73	0.91	2.30
Conradson Carbon, wt-%		4.02	
Heptane Insolubles, wt-%	1.0	8.11	

Table 9.6 Comparison of Rockwell Heavy Oil with H-Coal Bottoms

	Rockwell Heavy Oil Run 79-40	H-Coal <u>Atmos. Btms</u> After Flash Distillation
Sp. Gr. at 60°F	1.28	1.04
Distillation		
IBP, °F	557	435
% Overhead	30.0	50.0
at Temp., °F	858	571
% Overhead	50.0	99.0
at Temp., °F	970	850
Hydrogen, wt-%	4.58	9.36
Carbon, wt-%	89.65	89.32
Sulfur, wt-%	0.90	0.07
Oxygen, wt-%	2.11	0.64
Nitrogen, wt-%	1.76	0.36
Ash, wt-%	1.82	0.25
Heptane Insolubles, wt-%	71.9	0.6
Toluene Insolubles, wt-%	21.61	10.8
DMF/Xylene Insolubles, wt-%	14.02	-
Water, wt-%	0.2	<0.1

The nitrogen and sulfur contents of the Rockwell light oil are somewhat higher than those of the other coal liquids. Although this could be due to the coal feedstock quality, the higher contaminant levels are not unexpected, since the hydroprocessing conditions of the Rockwell process are thought to be less severe than those of the other coal liquid processes. These contaminants would have to be removed or reduced in the upgrading to transportation fuels. Contaminant removal from the Rockwell liquid would probably be at least as difficult as that for the other coal liquids, and could prove to be considerably more so. Because of the hydrogen deficiency of the Rockwell liquid, much of the sulfur and nitrogen compounds could be part of condensed ring structures. If so, high hydrogen consumption and high processing severity may be required to saturate and crack the rings in converting the nitrogen and sulfur to ammonia and hydrogen sulfide, respectively.

The low H/C atomic ratio of the Rockwell liquids indicates the presence of a high concentration of hydrogen-deficient compounds, such as polynuclear aromatics and diolefins. Because these compounds are not found in such concentrations in crude oil, conventional processing techniques would not be possible without prior upgrading. Central to any prior upgrading scheme would be hydrotreatment to obtain a stable, pumpable liquid.

The heavy oil as such would be extremely difficult to process catalytically. Therefore, noncatalytic methods of upgrading, such as solvent extraction or thermal processing, might be more economical. If solvent extraction is to be considered, pilot plant tests would be required to determine what percentage of the heavy oil might be recoverable for further processing.

In processing the light oil, a first step is likely to be fractionation. In this case, the bottoms temperatures of the fractionators are limited to 450°F maximum. Higher temperatures would probably cause the diolefins to form deposits in the tubes of the heaters and heat exchangers. The effluent streams from the fractionators must be hydrotreated prior to subsequent processing steps or, if the coal liquid exhibits extreme instability, it may have to be stabilized by hydrotreating prior to this low temperature fractionation.

A heavy distillate fraction from the Rockwell liquid must be severely hydro-treated to be considered as an acceptable fluid catalytic cracking (FCC) charge stock. Laboratory studies on H-Coal heavy distillate have shown that hydrogen saturation to about 11 wt-% produces a material suitable for use as FCC charge stock. (Sixty-five vol-% conversion was obtained on such an upgraded FCC feed versus 30 vol-% for the raw H-Coal distillate.) Obviously, the raw Rockwell liquid having hydrogen content 2.7 wt-% less than the H-Coal liquid (see Table 9.6) will require a correspondingly higher process hydrogen consumption to make it an acceptable FCC feedstock.

To upgrade the Rockwell light oil into acceptable FCC feedstock containing 11 wt-% hydrogen, more than 5 lbs of hydrogen will be consumed for every 100 lbs of liquid processed. On the same basis, processing of H-Coal, EDS, and SRC-II coal liquids will consume 2 to 3 lbs of hydrogen for every 100 lbs of liquid.

The low hydrogen content of the Rockwell light oil might indicate a high concentration of desirable aromatics, making it a possible candidate for conversion to petrochemical feedstock. UOP analyses⁽⁷⁾ show that more than 99% of the Rockwell material consists of aromatic compounds as compared to about 86% for the H-Coal. There are 23% benzenes in the Rockwell light oil versus 10% for the H-Coal. The large difference is probably caused by the high initial cut point selected for the H-Coal. However, the Rockwell light oil has a large quantity of heteroaromatic molecules containing oxygen, nitrogen, and sulfur atoms. The concentration of these compounds is greater than 17%, compared to 11% for the H-Coal. These aromatic-bound oxygen, nitrogen, and sulfur atoms will be difficult to remove, and hydroprocessing conditions required to effect their removal will most probably result in significant ring opening and/or saturation. This will degrade the yield.

Coke formation is also a potential problem and cause of loss. The extreme hydrogen deficiency of the heavier Rockwell liquid suggests the presence of polynuclear aromatics. These are potential coke precursors and are not considered to be attractive sources of BTX or other light aromatics. These potential coke precursors would render catalytic processing unattractive and refining yields low.

9.2 ECONOMIC CONSIDERATIONS

An earlier UOP study concluded that the cost of upgrading H-Coal heavy distillate into a slate of transportation fuels, with primary emphasis on the production of gasoline, would be \$9 to \$11 per barrel.⁽¹¹⁾ In comparison with the H-Coal liquid, Rockwell light oil has been shown to contain 2.7 wt-% less hydrogen and significantly higher levels of sulfur, nitrogen, and oxygen contaminants. These factors should render the Rockwell light oil more expensive to upgrade than H-Coal heavy distillate. It should, therefore, cost more than \$9 to \$11 per barrel to upgrade the Rockwell light oil.

The approximate cost of upgrading the Rockwell liquid was obtained by reasonable extrapolation of a study by Mobil Oil Company for EPRI.¹⁰ This study estimated the cost of refining H-Coal with an H/C by wt of 7.4/88.2 and SRC with an H/C 5.7/88.5. Rough extrapolation to the Rockwell heavy liquid (H/C = 4.63/90.2) results in a refining cost of \$2.00/MM Btu, based on 100% equity financing and inflation, and of \$3.80/MM Btu based on constant dollar and equity financing. When this is added to the cost of producing the raw Rockwell liquids, the total cost will look high. In consequence, the justification for continuing development of this process that is still in the exploratory phases should be closely examined.

SECTION 10 - RESULTS OF EVALUATION OF HEAT EXCHANGE REQUIREMENTS (SUBTASK 8)

The purpose of this subtask was to evaluate the needs for heat exchange in the Rockwell coal liquefaction plant, to point out the design and operating problems, and to establish data needed to support the commercial heat exchanger design. The task was also concerned with determining the need for future information from the PDU program.

A preliminary evaluation of heat exchange needs of the process was made by employing general economic heat exchanger design guidelines. Table 10.1 summarizes the results of this evaluation. Approximately 80% of the exchanger duty, excluding the cryogenic, is associated with heating the reactants, the subsequent cooling of the reactor effluent, and light and heavy oil recovery. The total heat duty of the exchangers totaled 54.8% of the HHV of the feed coal even without the cryogenic gas separation. This clearly is of great importance to the process thermal efficiencies. It is much higher than would be expected for other coal conversion processes.

The problem areas in the design and fabrication, erosion, fouling, and limitations of the heat exchange system were evaluated. The heat recovery quench exchanger may be fabricated from such a special alloy that special techniques may be required. However, at present, no major fabrication problems are foreseen for most of the rest of the heat exchangers contemplated for the commercial plant design. These exchangers are large in size but well within present day manufacturing capabilities.

In the evaluation of heat exchange systems, an attempt was made to determine if heat exchange contributes a disproportionate share of the cost and, therefore, places an unreasonable economic burden on the process. The largest contributor to the overall capital cost was the hydrogen preheater, \$10 million for one of three, followed by the reactor quench system, \$700/sq ft. These costs were great enough to consider these two exchangers to be unusual economic burdens on the process in comparison to the cost of heat exchange in other processes.

Table 10.1 Major Heat Exchanges

<u>Name</u>	<u>Duty(1)</u> <u>(MMBtu/hr)</u>	<u>Duty/HHV Coal</u>
Hydrogen Heater	1466	0.204
Reactor Quench	477.9	0.063
Heavy Oil Condenser	595.5	0.083
Light Oil Condenser	577.9	0.080
Feed/Bottoms Benzene Absorber Exchange	56.0	0.008
Absorber Feed Cooler	59.7	0.008
BTX Fractionator Reboiler	76.4	0.011
BTX Fractionator Condenser	17.0	0.002
AGR Feed/Bottoms Exchanger	35.7	0.005
Stripper Condenser	39.6	0.006
Stripper Reboiler	69.6	0.010
Absorber Feed Cooler	18.1	0.003
Methanator Feed/Effluent Exchange	379.2	0.053
Methanator Effluent Condenser	86.1	0.012
Methanator Feed Gas Heater	Start up only	-
Cryogenic H ₂ Separation (Package Unit)	-	-
		<hr/> .548

(1) Estimated for 1 train of 3.

A number of possible critical heat exchange operating problems are foreseen, namely:

1. Plugging of the preheater by carbon from decomposition of impurities in the hydrogen feed.
2. Erosion, corrosion, fouling, thermal stress or stress corrosion of the quench exchange. Furthermore, the heat exchange quench velocity of about 200 fps and high solids loading at 1700-1000°F are extreme conditions.
3. Occasional or persistent particulate carry over from the char cyclones. This excessive solid particle carry over will cause the following problems:
 - Reduce the life expectancy of the heavy and light oil condenser tubes through erosion.
 - Increase the dew point of these gaseous streams.
 - Increase the viscosity of the heavy oil, restricting its free flow and ultimately clogging the tubes.
 - Result in solids in the liquid product that must be filtered out.
4. Transient operating conditions at which the liquid products condense as a stable mist that can not be collected. This is likely because of the high partial pressure of non-condensables in the condensers.
5. Possible plugging of the cryogenic unit exchangers by a trace component that can not be economically removed.
6. Possible plugging/fouling of the heavy oil condenser from coking of the heavy oil. This possible condition has not been detected as yet; however, the PDU runs have been very short.

The data gaps that were identified are significant.

1. Most of the data were gained from Rockwell's PDU testing, which has not been conducted in a systematic test matrix, thus making it impossible to calculate the key parameters for the design of the heat exchange system.
2. PDU runs have been on the order of one hour each because of system limitations, and the total run time up to the present has been about 25 hours. It would be extremely risky to scale up from this short an experience to a full scale pilot or commercial plant. It is necessary to modify the PDU so that long continuous runs are possible. Steady state conditions must be reached with respect to the heat transfer units. A 24 to 48 hour period is suggested for steady state heat transfer operations to take place, and a continuous run of at least one week should be made to provide information on any fouling characteristics. A continuous run of one month's length should provide sufficient erosion and corrosion information to allow design of larger units.
3. To determine cyclone efficiency, data on percent solids carry over and their quantity and particle size should be measured on a continuous basis. Alternatives to the cyclones should be considered to remove char and ash particles.
4. Materials should be tested in the PDU to determine the maximum allowable temperatures in the hydrogen preheater at 500 psig and the erosion resistance characteristics for reactor quench and heavy oil exchangers.
5. PDU testing is necessary with CH_4 and CO in the hydrogen feed to the reactor. This testing is needed to confirm reactor operability when impurities are recycled from the raw gas recycle. The effects of these impurities on the size of the downstream heat exchangers, on the deterioration of liquid quality and quantity, and on the gas quality and yield should be determined for design of the heat exchange system.

6. PDU testing should emphasize greater accuracy in the measurement of flow temperature to obtain heat transfer coefficient data for special units, such as quench systems and heavy and light oil condensers. More accurate balances of ash and carbon are necessary for the design of heat exchange systems.
7. Exchange-quench data, including fouling factors, heat exchange coefficients vs condition, and rate of liquid product decomposition vs time=temperature, are needed.
8. Test data from a cryogenic gas separator exchanger are needed.

SECTION 11 - RESULTS OF EVALUATION OF UNUSUAL MATERIALS REQUIREMENTS (SUBTASK 9)

11.1 INTRODUCTION

The objectives of Subtask 9 were to identify unusual material requirements of the Rockwell process for each location and service condition, determine the best experience with candidate materials and their probable service life, risks, and reliability, and identify any information gaps and testing that would be required in the Rockwell PDU.

The results of UOP/SDC's examination of potential materials problems are presented in this section in tabular form. The critical components and candidate materials for Section 100 (coal receiving, handling, and feeding) are shown in Table 11.1; for Section 200 (reactor, quench, and char recovery) in Table 11.2; for Section 300 (BTX recovery, acid gas removal, methanation, and cryogenic separation) in Table 11.3; and for Section 500 (char gasification and hydrogen production) in Table 11.4.

11.2 INFORMATION AND DATA GAPS

The existing information on materials for use in coal conversion plants is very limited and narrow in scope. Most of the data are concerned with materials for coal gasification processes. Little materials testing has been done for liquefaction processes - essentially none for the Rockwell liquefaction process. The lack of data on materials for hydroliquefaction reactors and components constitutes a serious information gap. To fill it, a comprehensive program of materials testing should be implemented as part of the Flash Hydroliquefaction PDU program.

Further, methodology should be developed to permit application of these results to the design of commercial plant components. This last recommendation also applies to the data presented for gasification programs.

Table 11.1 Critical Components and
Candidate Material for Section 100

Component	Service	Material	History (of Material & Component)
1. Coal conveyors (belts).	Capacity 1000 t/hr; ambient pressure and temperature.	C-Steel.	No problems except high maintenance.
2. Dust collectors (bag filters or wet scrubbers).	Ambient pressure/ (under pressure) and temperature.	Felt in case of bag house or C-Steel for scrubbers.	Maintenance is a problem. Corrosion resistant coating rubber lined pumps needed for scrubbers.
3. Primary and secondary crushers (hammer mill and bowl mill).	Capacity 1000 t/hr ambient pressure. Slightly elevated temperature, 350°F.	Carbon steel	Regular items. No major material problems.
4. Coal feeding system. Lockhoppers, valves, pressurizing and depressurizing system, controls, and safeguards.	Ambient temperature but the pressures are high: 200 psig, 1200 psig.	Valves must be highly erosion resistant. Alloy steels.	Experience with these materials is inadequate. Unproven as a valve material. General erosion problem.
5. Dust collectors (from lock-hoppers).	Ambient: But the valves before the collectors must provide for 200 and 1200 psi.	Valve erosion problems are similar to those above.	Same as above.

Table 11.2. Critical Components and Candidate Materials for Section 200
(page 1 of 4)

COMPONENT	SERVICE	POTENTIAL PROBLEM	CANDIDATE MATERIAL	EXPERIENCE HISTORY OF MATERIAL AND COMPONENT
1. Hydrogen Preburner	2400°F in oxidizing and reducing atmosphere. Pressure 1200 psig.	Degradation due to high temperature. Hydrogen embrittlement. Dimensional instability of injection orifices.	Ceramic Nozzles.	Experience inadequate for high temperature hydrogen heating (greater than 1550°F).
2. Dense Coal Feed System	Pulverized Coal Multi-injector System.	Plugging of injector nozzles and distribution system.	304 S.S.	Further experimental work needed.
Coal Injector	Coal particles; H ₂ injection system; O ₂ injection system (Temp. H ₂ and O ₂ nozzle - 2000°F to 2500°F).	High temperature and press., plugging, erosion and hot spots, difficult to build and extremely difficult to repair, thermal stress, containment of H ₂ + O ₂ reaction, dimensional stability of inlet and metering orifices.	Incoloy 800	More experimental data needed.
3. Reactor System	Pulverized coal, H ₂ , H ₂ S, CH ₄ , CO ₂ , CO, H ₂ O, hydrocarbon gases and liquids.	High temperature, corrosion, abrasion problem.	Refractory lined 304 S.S. C-1/2 Mo Steel	More experimental data needed. Experience limited.
Internals	Same as in Reactor System.	Same as in Reactor System.	Alloy 800	Same as in Reactor System.

Table 11.2. Critical Components and Candidate Materials for Section 200
(page 2 of 4)

COMPONENT	SERVICE	POTENTIAL PROBLEM	CANDIDATE MATERIAL	EXPERIENCE HISTORY OF MATERIAL AND COMPONENT
Quench	Tubeside - char, H ₂ , H ₂ S, CO, CO ₂ , H ₂ O, CH ₄ , hydrocarbon liquids and gases	Erosion, corrosion, high grain loading.	Alloy 800	Same as in Internals.
	Shellside - steam or water.		C.S.	Experience adequate.
Char Receiver	Char, H ₂ , H ₂ S, CO ₂ , CH ₄ , H ₂ O, CO liquids, gases	Abrasion, erosion.	Refractory line, 304 S.S. clad C-1/2 Mo	More data needed.
4. Char Vapor Separator 202	Separates solids and gases. The stream contains char, tar, H ₂ , CO ₂ , CO, H ₂ S, etc. Maximum temperature 1600°F, pressure 1200 psig.	Erosion due to char and ash particles. Corrosion by the gases.	Incoloy 800H	Ref. Huntington alloys bulletin for Incoloy 800H. Metal Property Council reports.
5. Cyclones	Same as in Component 4.	Erosion by high velocity. Char and ash particles coupled with corrosion by H ₂ , CO ₂ , H ₂ S, H ₂ O, etc.	Same as in Component 4. or S.S. with refractory lining.	Same as in Component 4.

Table 11.2. Critical Components and Candidate Materials for Section 200
(page 3 of 4)

COMPONENT	SERVICE	POTENTIAL PROBLEM	CANDIDATE MATERIAL	EXPERIENCE HISTORY OF MATERIAL AND COMPONENT
6. Char Lockhoppers	Separate solids from the main. The stream max. temp. = 800°F, max. pressure - 1200 psig.	Erosion due to char and ash particles and H ₂ embrittlement.	310 S.S.	Data needed at 1000°F.
7. Char Valves	Max. temp. - 1000°F Max. pressure - 1200 psig.	Erosions from char lockhoppers.		Experience inadequate, further work desired.
8. Waste heat recovery	Cooling hot gases below the dew point and generating high pressure steam. Max. temp. - 1600°F Max. pressure - 1200 psig.	Corrosion by H ₂ , CO, CO ₂ , NH ₃ , H ₂ S, H ₂ O, and hydrocarbon gases. Erosion due to char and ash particles.	Incoloy 800H or In 671 for tubes, C.S. for shell.	Experience inadequate, more data on erosion needed.
9. Heavy and light oil separation drums.	Separate liquid and vapor phases. Max. temp. 600°F. Pressure 1100 psig.	Corrosion by acidic condensate with char slurry.	.5 Mo Steel with SS clad	No problem but care must be taken to provide impingement protection.
10. L.P. waste heat recovery system (light oil condenser).	Cooling hot gases Maximum temp.=600°F pressure = 1100 psig.	Same as in 8.	.5 Mo Steel with SS clad	Experience adequate.

Table 11.2. Critical Components and Candidate Materials for Section 200
(page 4 of 4)

COMPONENT	SERVICE	POTENTIAL PROBLEM	CANDIDATE MATERIAL	EXPERIENCE HISTORY OF MATERIAL AND COMPONENT
11. Char Valve to remove char from Char Separator.	Valve to operate at 1600°F and 1200 psig and transport heavy solids.	Extremely erosive condition due to high pressure drop. Corrosion due to H ₂ , CO, CO ₂ , H ₂ S, etc. at high temp. and pressure.	Alloy Steel	Similar problem as in lockhoppers, only more severe because of high temperature.
12. Decanter to separate oil and water layers.	Separate two liquid phases at T = 100°F and P = 1100 psig.	No special problems.	Carbon Steel	Experience adequate.
13. Chars return from cyclone.	Returns solids from cyclone bottom at 1000°F (design at 1600°F) and 1100 psig.	If a gravity return is contemplated, erosion of the pipes could be a problem.	Stainless steel with refractory lining.	Experience adequate.
14. Char Cooler	Cools hot char from 1000°F to ambient.	Potential erosion problem.	Carbon steel with refractory lining.	Experience adequate.
15. Hydrogen Heater	To heat hydrogen to 1500°F.	High temperature corrosion and hydrogen diffusion.	IN 800H.	Experience inadequate with specific gas mixture.

Table 11.3. Critical Components and Candidate Materials for Section 300
(page 1 of 2)

COMPONENT	SERVICE	POTENTIAL PROBLEM	CANDIDATE MATERIAL	EXPERIENCE HISTORY OF MATERIAL AND COMPONENT
1. BTX Absorber and Fractionator System	BTX is absorbed and separated from the gaseous stream containing H ₂ , CO, CO ₂ , H ₂ S, and organic gases. The temperature ranges from min. 100°F to max. 300°F, pressure from 1050 psig to atmosphere.	H ₂ S	Stainless Steel	Standard Manufacturing practice.
2. AGR System (includes absorber, fractionator, heat exchanger, etc.)	H ₂ S and CO ₂ are removed from the gaseous stream. Temp. range = 100°F to 300°F. Pressure range = 1030 psig to atmosphere.	None Expected	Carbon Steel - heavy gauge preferred	No problems - proven technology.
3. Primary Exchanger	H ₂ rich gaseous environment Temp: Shell 350°F Tube 550°F Pressure: Shell 965 psig Tube 1010 psig.	Hydrogen Embrittlement	Shell = Carbon Steel Tube = 1/2 Mo Steel	This selection of materials as per Nelson's Chart, which is commonly used in industry for H ₂ rich environment.
4. Secondary Exchanger	H ₂ rich gaseous environment. Shell Temp. = 650°F pressure = 1005 psig Tube Temp. = 800°F pressure = 970 psig.	Hydrogen Embrittlement	Shell = 1/2 Mo Steel Tube = 1 Cr, 1/2 Mo Steel	Same as above.

Table 11.3. Critical Components and Candidate Materials for Section 300
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COMPONENT	SERVICE	POTENTIAL PROBLEM	CANDIDATE MATERIAL	EXPERIENCE HISTORY OF MATERIAL AND COMPONENT
5. Sulfur Guard	Remove traces of S present in the gases Temp. 650°F Pressure 1000 psig	Hydrogen Embrittlement	1/2 Mo Steel	Same as above.
6. Methanation reactor	Convert CO into CH ₄ and water. Temp. 800°F Pressure 990 psig.	Hydrogen Embrittlement	1 Cr 1/2 Mo Steel	Same as above.
7. Crude H ₂ Cooler and K.O. Drum	Cools the gases and separate water Max. Temp. = 300 Max. Pressure = 990 psig.	None	C.S.	Same as above.
8. Dryers and Cryogenic Hydrogen Separation System	Removes traces of H ₂ O and separate CH ₄ from H ₂ .	None	Per manufacturer's recommendation.	Unit Package

Table 11.4. Critical Components and Candidate Materials for Section 500
(page 1 of 2)

COMPONENT	SERVICE	POTENTIAL PROBLEM	CANDIDATE MATERIAL	EXPERIENCE HISTORY OF MATERIAL AND COMPONENT
1. Char Storage and Feeding	Similar to coal feeding	Erosion, high temp. galling	S.S.	PDU needed
2. Gasifier (slagging types)	Char, oxygen, and steam reacting at 2000-2500°F and 1000 psig.	High temperature corrosion, H ₂ embrittlement and thermal stress and removal of slag.	0.5 Mo steel with refractory lining.	Experience inadequate, more data needed.
3. Gasifier Internals	Same as above.	Same as above.	Incoloy 800 for internals.	Experience inadequate, more data needed.
4. Cyclones	Up to 1500°F, 1500 psig.	High temperature corrosion, H ₂ embrittlement and thermal stress.	0.5 Mo steel for cyclones.	Experience inadequate, more data needed.
5. Waste Heat Recovery	Cooling hot gases and generating steam.	Corrosion by H ₂ , CO, CO ₂ , NH ₃ , and H ₂ S gases. Erosion due to char and ash.	Incoloy 800 or Thermalloy 63W for tubes and refractory lined C.S. for shell.	Experience inadequate, more data needed.
6. Raw Gas Quench Tower and Internals	Quenching the gases to dew point. Temperature 100°F and pressure 1500 psig.	Same as above but less severe.	S.S. clad C.S. for tower and 304 S.S. for internals.	Experience adequate.

Table 11.4. Critical Components and Candidate Materials for Section 500
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COMPONENT	SERVICE	POTENTIAL PROBLEM	CANDIDATE MATERIAL	EXPERIENCE HISTORY OF MATERIAL AND COMPONENT
7. Shift Converter	Maximum temperature of 950°F and 1200 psig.	Corrosion by sulfidation, and H ₂ embrittlement.	0.5 Mo steel with 304 cladding.	Experience adequate.
8. Waste Heat Recovery		No problems.		Experience adequate.
9. AGR System (includes absorber, stripper, exchangers, etc.)	H ₂ S and CO ₂ are removed from the gaseous stream. Temperature 100°F and pressure 1150 psig.	None expected.	Carbon steel.	Proven technology.
10. Sulfur Guard	Remove traces of Sulfur.	Hydrogen embrittlement.	0.5 Mo steel.	Same as above.
11. Methanator	Convert CO to CH ₄ and H ₂ O. Temp. = 800°F and pressure 1100 psig.	Hydrogen embrittlement.	1 Cr, 0.5 Mo steel.	Same as above.
12. Drying and Compression		None		Standard items.

11.3 RESEARCH NEEDS

As a result of the foregoing study and analysis, the following research needs can be identified and incorporated in the Rockwell PDU test program.

1. The PDU can be used to set up coupons and test sections at suitable places; also, ultrasonic thickness measurements, corrosion meters, and component performance analysis should be used.
2. The reactor heat exchanger design and the candidate material must be given special attention. More data on material strength, erosion, and corrosion can be obtained in PDU operation. Similarly, the reactor injector head assembly must be intensively studied.
3. Refractory linings and data on their resistance to erosion in all the two-phase flow situations must be studied in the PDU operation, especially the effects of impingement velocities at various angles.
4. A data base should be designed to collect and collate all failures of suitable components and materials. This data base must be continuously updated.

SECTION 12 - NOMENCLATURE

C	-	% Total Carbon Converted
L	-	% Carbon Converted to Liquid
T	-	Reactor Temperature ($^{\circ}\text{R}$)
G	-	Coal Mass Flux ($\text{lb}/\text{ft}^2 - \text{sec}$)
t	-	Reactor Residence Time (msec)
D	-	Reactor Diameter (inches)
M/C	-	Hydrogen to Coal Ratio (lb/lb)
dp	-	Particle Size (micron)
T _i	-	Average Temperature in i th segment
t _i	-	Average residence time in i th segment
LT _i t _i	-	Liquid yield at T _i and t _i
i	-	Ranges from zero to N
N	-	Number of exchange - quench segments
L _i	-	Liquid yield at the end of i th segment
U	-	Overall heat transfer coefficient ($\text{Btu}/\text{hr ft}^2 \text{ } ^{\circ}\text{F}$)
h	-	Individual heat transfer coefficient ($\text{Btu}/\text{hr ft}^2 \text{ } ^{\circ}\text{F}$)
A	-	Area of heat transfer surface (ft^2)
D _d	-	Droplet diameter (ft)
q	-	Rate of heat transfer (Btu/hr)
n	-	Number of tubes
D _s	-	Shell diameter (ft)

SECTION 13 - REFERENCES

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