Table 2.8-2. Diesel fuel produced at Chevron Corp. from H-Coal synthetic crude from Illinois No. 6 (Burning Star) coal.

Property	Diesel fuel specification ASTM D 975-78		Properties of the 250°F product from severely hydrofined Illinois H-Coal oil
	No. 1D	No. 2D	
cetane number	> 40	> 40	42.5
cloud point	varies	varies	50
sulfur, weight percent	<0.5	<0.5	0.001
flash point (Pensky-Martin), °F	>100	>125	116
ash, weight percent	<0.01	<0.01	< 0.002
distillation 90%, °F	<550	540-640	524
viscosity at 40°C, centistokes	1.3-2.4	1.9-4.1	1.678
viscosity at 100°F, Saybolt universal seconds	< 34.4	32.6-40.1	32.1
Cu-strip corrosion	< No. 3	< No. 3	No. 1
aromatics	-	-	2.7

chemicals can be recovered from various product streams. The heterocyclic nature of numerous compounds makes coal liquids suitable for extractive separations. Very high volumes of BTX can be extracted from the reformate streams. Phenolics are extractable from raw naphtha. Numerous other opportunities exist and await identification. Research will contribute to energy-efficient separation processing for a broad range of chemicals. A second need exists in the areas of health and industrial hygiene effects relating to coal-derived liquids. Broad-based evaluations of raw and upgraded (hydrotreated/extracted) streams are needed to assess short-term and long-term health issues associated with use as chemicals. A major concern is the carcinogenic nature of many high molecular weight polynuclear (polycyclic) aromatics (PNAs). Procedures need to be developed to insure the health of the workers in the industry and limits need to be defined for PNAs in coal-derived products.

The foregoing comments relate to liquids derived from coals by any of several direct hydroliquefaction routes. Indirect routes through gasification and Fischer-Tropsch synthesis (Sasol) or methanol to gasoline conversion (Mobil process) produce very different products. Fischer-Tropsch synthesis products are highly paraffinic and may contain appreciable amounts of chemically combined oxygen but they are essentially free of nitrogen or sulfur. These liquids should present no particular refining problems, other than those associated with high paraffinicity (i.e., high pour point, high cloud point, low octane number, etc.). The products produced in the Mobil M process are reported to be mainly highly aromatic, high-octane naphtha and C_4 and lighter gases. These products should be directly usable in any transportation fuel refinery.

Mention should also be made of other energy uses for coal-derived liquids, especially electric power generation. As substitute fuels for coals in boiler plants, raw coal feeds such as SRC-I should be satisfactory with proper burner design and configuration. The use of lighter, coal-derived distillates as fuels for combined cycle power plants may require changes in turbine design because of the high aromaticity of these fuels. Also, NO_x emissions could prove to be a problem that will require the use of remedial measures.

A. Research recommendations

While refining processes capable of producing conventional fuels from coal-derived oils are available, high hydrogen consumption, poor selectivity and, in some cases, low catalyst activity and life leave room for major improvement. The large industrial capability for development of petroleum-refining processes is a major national resource for the development of synthetic fuel refining processes. While there is a considerable amount of current exploratory research aimed at synthetic fuel refining, the availability of first generation technology, together with the long-term requirements for extensive synthetic fuel refining facilities, has delayed industrial commitment to a major effort in this field. Duplicating or supplanting these existing industrial research and development capabilities is undesirable. However, DOE support of basic and exploratory research leading to better understanding and improvements in synthetic fuel refining processes could result in earlier development of second generation processes.

A list of research areas is given in Table 2.8-3.

Table 2.8-3. Recommended research areas on the down-stream refining of coal-derived liquids.

a. Basic and Exploratory Research

1. Determinations should be made of the molecular structure of O, N, and S compounds and of polynuclear aromatics in coal oils and these data should be compared with results for typical petroleum fractions.

2. Mechanism for the removal of heteroatoms from coal oils by conventional catalytic hydroprocessing should be studied.

3. Alternate ways to remove heteroatoms from coal oils should be sought.

4. More active and more selective hydroprocessing catalysts are needed for heteroatom removal. The mechanisms and rates of these processes should be investigated.

5. The mechanism of those retrograde reactions should be studied that cause viscosity and solubility changes in some coal oils during storage.

b. Applied Research

1. The kinetics, selectivity, and catalyst deactivation steps in the hydroprocessing of coal oils for heteroatom removal should be studied.

2. Studies should be performed of the hydroprocessing of coal oils alone and in mixtures with petroleum stocks. Synergisms or antagonisms should be identified.

3. Investigations are needed to define the compatibility of coal oils with petroleum fractions after varying degrees of hydroprocessing.

4. Investigations are required of the potential health hazards (potential mutagenicity and oncogenicity) of coal oils after varying degrees of hydroprocessing.

5. Means must be found to optimize selectivity and catalyst life in the hydroprocessing of coal oils. These investigations should include a search for and evaluation of improved hydroprocessing catalysts.

Talbe 2.8-3. (Continued)

c. Process Development

1. Additional economic studies are needed on the downstream refining of coal oils, including coal-oil and petroleum mixtures.

2. A systems approach should be employed in assessing the costs of refining and using these fuels. When are basically distinct coal-oil refineries economically desirable ?

3. A systems approach should be applied to determine when modified engines to handle coal-oil products should be developed for selected end uses.

4. The development of advanced coal-liquefaction technologies should be pursued with proper allowance for downstream refining objectives.

d. Pilot Plants

1. It may be desirable to acquire or lease facilities for the experimental refining of coal-derived oils to produce standard products for engine development and fleet testing.

2. These facilities should be used for scale-up demonstration of optimal refining schemes.

3. Pilot-plant scale demonstrations should be initiated for the manufacture of hydrogen from coal-liquefaction bottoms streams.

e. Combustion Emissions and Health Problems

1. Emission from combustion of coal-derived liquids in existing and developing equipment should be assessed and control approaches defined where needed. Attention should be paid not only to SO_x, NO_x, particulates and smoke, but research should also address problems created by the presence of polynuclear atomatics, heterocyclics, and trace metals.

2. Threshold limits for exposure to polynuclear aromatics, polycyclics and trace metals should be defined.

3. Medical hazards associated with lighter aromatics (benzene, toluene) should be reexamined in view of the high concentrations of these molecules in coal-derived liquid streams.

CHAPTER 3:

COAL PYROLYSIS

3.1 Introduction

Pyrolysis occurs in virtually all coal-conversion and utilization processes and is an established coal-liquefaction route. Oil produced by hydroliquefaction requires three times as much hydrogen per barrel of product as oil produced by pyrolysis. Pyrolysis has greater thermal efficiency than hydroliquefaction because of lower hydrogenation requirements; about half of the coal is converted to char. Coal-fired boilers are an incremental source of liquid fuels if the coal is first pyrolyzed to recover the hydrogen-rich volatiles and the carbon-rich char is then used for power generation.

3.2 Comments on Coal-Pyrolysis Technologies

Pyrolysis involves a primary thermal decomposition step generating volatile species, which undergo secondary degradation reactions until quenched. The yields and compositions of the primary products are determined by coal type and heating schedule. Higher rank coals and fast heating favor high primary liquid yields. Prolonged pyrolysis reduces the liquid yields and increases char and gas yields through secondary cracking and polymerization reactions. Stabilization of liquid-range precursors may occur by internal hydrogen transfer and by direct hydrogenation, which require high hydrogen pressure, catalysis, or both. Temperature, residence time, pressure, reactive gaseous and solid species, and catalysts are important parameters. In low-temperature carbonization in the absence of air (final char temperatures of 850-1400°F), at atmospheric pressure and in dense beds (fixed-bed retorts, continuously-moving bed retorts, and fluidized beds), prolonged exposure of volatiles leads to secondary cracking reactions and high char yields.

Rapid pyrolysis has been used near atmospheric pressure in an entrained-flow reactor. The small coal particles are heated very quickly and a high primary liquid yield results. Rapid breakup of the coal structure and fast vaporization minimize coke formation. Short vapor residence times and rapid quenching retard secondary cracking and polymerization reactions and maximize the net liquid yield.

In hydropyrolysis, dry hydrogenation is performed at hydrogen pressures of 500-2000 psi. In a fluidized bed, this process is referred to as hydrocarbonization. Rapid hydropyrolysis in an entrained reactor at short residence times is a recent development. The objective is to improve the yield and quality of the raw liquids.

Selected pyrolysis technologies are listed in Table 3.2-1. The liquid yields obtained by rapid pyrolysis and hydropyrolysis are seen to be nearly twice those achieved by carbonization with slow heating. The liquid yields from bituminous coal are generally 50% higher than from subbituminous coal. The maximum liquid yield is about 38%. The char yield is typically greater than 50%. Rapid pyrolysis and hydropyrolysis liquid yields are roughly equivalent.

The direct products of pyrolysis generally require further processing. The liquids require hydrogenation to reduce the molecular weight and remove heteroatoms. However, sufficient H_2 for these purposes is generally available from reforming of the pyrolysis gases. The sulfur content of the raw char is about the same as of the parent coal. Utilization of the char as fuel therefore requires desulfurization and gasification, followed by clean-up or post-combustion control. Although hydropyrolysis liquids are preferred over the products derived from rapid low-pressure pyrolysis, the maximum yield is about the same and hydrofining is still required.

A. The Lurgi Ruhrgas Process

This low-pressure pyrolysis process was developed for the liquefaction of European brown coals and is the only pyrolysis process presently in commerical use. A 1,600 tpd Lurgi-Ruhrgas plant was built in 1963 in Yugoslavia for lignite and operated successfully. The char is fired in an adjacent power plant. Coal is crushed to one-quarter inch size and is then fed into a mechanical mixer, where it is rapidly heated to 850 to 1100°F by direct contact with hot, recirculated char that was previously heated by partial oxidation with air in an entrained-flow heater. Net product char is withdrawn. The gases from the mixer are passed through a cyclone for particulate removal and then through condensers to collect liquid that is further hydrotreated to form a range of products.

B. The COED Process

The COED (Char-Oil-Energy-Development) process of the FMC Corporation has been under development since 1962 and produces a synthetic crude oil by fluidized bed pyrolysis and separate oil hydrotreating. Following testing in a 100 lb/hr PDU, a 36 tpd pilot plant was built (1970). Tests with durations in excess of 30 days were completed before the plant was dismantled. Another pilot plant to evaluate gasification (COGAS) began operation in 1974. A conceptual commerical COED design (Ralph M. Parsons Co., 1975) produces 28,000 bpsd of 25° API syncrude from 27,400 tpsd (21,500 tpsd, MAF basis) HVCB bituminous coal. Minus 6 mesh crushed coal is processed at atmospheric pressure in fluidized beds in which agglomeration is prevented by operating at successively higher temperatures. Three pyrolysis stages

Table 3.2-1 Summary of pyrolysis technologies; liquid yields refer to maximum reported amounts.

Process/ Developer	Reactor Type	Residence Time of		Temperature °F	Pressure, psia	Coal type	Liquid yield in wt.% (MAF)	Status and comments
		coal	gas					
Fixed-bed retorts	fixed-bed	>4 hr.		850-1400	~15		6-12	Obsolete; extensive commerical operations in Europe.
Lurgi-Ruhrgas	mechanical mixer/conveyor	> 20 sec.		1110	~15	Brown	24	A 1600 TPD plant has operated in Yugoslavia since 1963.
COED, FMC	multiply-staged, fluidized beds	1-4hr.		575, 815, 1050, 1600	~25	HVCB Sub. C	22 12	A 100 lb./hr. PDU operated 1976-70; a 36 TPD pilot plant operated 1970-75.
Flash pyrolysis, Occidental	entrained flow	1-3sec.	1-3sec.	1200	~60	HVCB Sub. C	35 22	A 3 TPD PDU operated 1976-78.
Hydrocarbonization, Union Carbide	fluidized bed	5-12min.		1050 1040	940 310	Sub. C Sub. C	29 21	1, 10, and 1500 lb./hr. units operated in the 1960s.
Flash hydropyrolysis, Rockwell	entrained flow	>100msec.	>100msec.	1570-1770	500-1500	HVCB	38	A 1 TPH reactor operated 1976-78.
BNL	entrained flow	1-10sec.		1380	2000	Lignite	20	A 10 lb./hr. bench scale unit has operated since 1975.

operate at 575, 815, and 1050°F, respectively; generally, the temperatures and the number of stages depend on the agglomerating properties of the coal. Representative performance data are listed in Table 3.2-2.

Hydrotreating of the raw oil in an ebullating bed reactor yields 25% product oil of -250°F, 40° API, 0.07% naphtha and 75% of +250°F, 4.6° API, 0.5% fuel oil. Hydrogen consumption is 1700 SCF/bbl. The high boiling fraction of the oil product from the hydrotreater contains char fines entrained from the pyrolysis section and is recycled to the pyrolysis reactors, where the oil is converted to char and lighter oil products. Agglomeration problems were experienced with both Kentucky and Illinois coals, which have free swelling indices of about 4-5. The char yield from bituminous coal was about 60%, with a sulfur content approximately equal to that of the parent coal. Several char-utilization schemes have been investigated at FMC, including gasification, desulfurization, and direct combustion in a utility boiler. Partial gasification was successfully demonstrated in the fourth-stage reactor of the pilot plant.

C. Rapid Pyrolysis

Studies on rapid pyrolysis have been performed, especially at the Rockwell Corporation and at the Occidental Petroleum Corporation. These studies are discussed in connection with the site-visit reports summarized in Sections AC-9.1 and AC-9.2.

D. Hydropyrolysis

In hydropyrolysis, coal is pyrolyzed under elevated hydrogen pressure to effect direct, dry, gas-phase hydrogenation of the primary volatiles as they are evolved in the pyrolysis reactor. Since the primary volatiles are unstable and tend to undergo secondary cracking and free radical condensations, the presence of molecular hydrogen can alter the reaction paths and lead to improved liquid yield and quality.

Hydrocarbonization is a fluidized bed process that is conducted under relatively moderate conditions (300-1000 psi hydrogen pressure, temperatures of 950-1100°F, and coal and vapor residence times up to 25 seconds). The liquid yield generally increases with hydrogen pressure. In flash hydropyrolysis (FHP) using entrained-flow, hydrogenation occurs at short residence times. The influence of severe conditions is being investigated (temperatures to 2000°F, pressures of 4000 psi, and contact times as low as 0.03 second). One objective of this approach is to achieve high primary oil yields with rapid pyrolysis while effecting gas-phase hydrogenation at short contact times. For details, reference should be made to the site visit reports in Section AC-9.1.

Table 3.2-2 COED liquid yield and characterization data.

Coal			
Rank	Bituminous C	Bituminous C	Subbituminous C
Source	Illinois No. 6	W. Ky. Nos. 9 & 14	Big Horn, Wy.
Yield (MAF basis)			
Wt. %	21.7	19.6	12.2
Bbl/ton	1.1	1.0	0.63
Analysis			
Ultimate, Wt. %			
C	81.5	81.9	82.7
H	7.8	7.5	8.0
N	1.0	1.2	1.0
S	2.4	1.6	0.6
O	7.1	7.7	7.5
Atomic H/C	1.15	1.10	1.15
Gravity, °API	-4.0	-4.8	-4.0

E. The Hyderabad Process

At India's Regional Research Laboratory (RRL), Hyderabad, an improved version has been developed of the Lurgi Spuelgas Process for the low-temperature carbonization of coal. The program involves carbonizing 20 to 100 mm lumps of subbituminous coals from Indian mines; the semi-coke product is a substitute fuel for wood charcoal. The Lurgi Spuelgas Process employed brown coals and lignites. In the Hyderabad process, better feedstock (subbituminous coal) is used with improved thermal efficiency. A pilot plant carbonizing 25 tpd was operated for a number of years. A commercial plant for 1,000 tpd has been operational since late 1978. In mid-1979, capacity expansion to 3,000 tpd (2050 tpd of semi-coke product) was authorized; tar output will be about 180 tpd and the plant will also produce about 500,000 Nm^3 per day of low-Btu gas.

3.3 Summary of Research Needs in Pyrolysis

We list here identified research needs relating to the development of improved methods of coal pyrolysis.

(i) A comprehensive model of pyrolysis is needed to relate yields and product distributions to coal type and process conditions. This model will require elucidation of the controlling chemistry and transport phenomena and should permit optimization of yields and product distributions, as well as process scale-up.

(ii) Reliable thermochemical data are needed for the polynuclear aromatics, radicals, and coal fragments which may exist as intermediates in coal pyrolysis.

(iii) Additional information is required on the primary coal-decomposition reactions (e.g., the mechanisms, rates, and primary products as functions of coal type and reaction conditions).

(iv) Further work is needed on the fluid mechanics and dynamics of molten particles. The mechanisms of bubble formation, movement of volatile species out of the decomposing particles, and pressure developments within coal particles should be studied, with emphasis on the porosity and reactivity of the residual char.

(v) Control of free radicals during pyrolysis is required to optimize liquid yields. Recombination of free radicals, formed in the primary pyrolysis, with the parent coal or with other large free radicals, will lead to the loss of liquids. Control of coal-surface reactivity and the introductions of chain-transfer catalysts (e.g., HCl or H_2S) and gas-phase Lewis acids (e.g., BF_3) should be investigated.

(vi) The use of oxygen during pyrolysis to stimulate oxidative degradation has been suggested and may be worth examining.

(vii) The impacts of disposable catalysts and of native mineral matter must be better understood. The effects of clays, alkalis and pyrites on liquid yields should be studied.

(viii) The impact of yield on product quality should be defined. Does higher liquid yield necessarily produce liquids of lower quality (i.e., liquids more deficient in hydrogen) and stability? The selectivity for hydrodesulfurization and hydrodenitrogenation under hydropyrolysis conditions should be determined.

(x) The reactivity of char formed from caking and non-caking coals should be investigated. We need to know how these chars behave as boiler fuels.

(xi) Uninterrupted, long-term support is required if rapid coal pyrolysis is to be developed successfully for commercial applications.

(xii) Supporting research is needed to assure better instrumentation and diagnostic measurements than are currently made. Model flow studies may be useful, especially for successful scaling.

(xiii) It would be desirable to compare pyrolysis yields for oxygen-free coals with those obtained under presently used conditions of feed preparation and injection.

CHAPTER 4:

COSTING OF COAL LIQUIDS

Presentations were made on coal liquefaction economics by representatives of four of the active groups working on coal liquefaction (see Appendix AC-10 for details). The purpose of the discussions was not a comparison of relative process economics but rather the identification of specific process areas where improvements might lead to significant economic benefits.

Economic data presented on the four processes referred to different bases (i.e., form of financing, project life, tax credits, etc.). In Table 4-1, all costs were adjusted to 1979 dollars at 7%/yr. and then distributed to major process cost areas. No attempt was made to place all of the processes on the same economic basis. Costs of coal liquids will vary widely because of the economic bases chosen. In addition, the levels of investment will also vary, based on the amount of engineering detail involved and the project/process contingencies included. The plant capacity factor that is used also plays a significant role in determining product cost. Reference to Table 4-1 shows that the capital costs, either on a per ton or per barrel of liquid product basis, are high. As the result, the capital charges are the major component in the final product costs, independent of the form of financing and investment credits used. The distribution of capital costs shows that the cost of hydrogen manufacture approaches that of the basic liquefaction process facilities.

We now proceed to identify those areas where additional research and development may lead to improved technologies and better economics, especially reductions of capital costs. In general, little can be done about the coal feed cost itself. Improvements in that area require increased mining or transportation efficiencies. We note especially the following desirable goals: process simplification, new catalysts that have improved selectivity for producing liquid products and thus lower hydrogen consumption, and process improvements of all types, for example, the development of processes for operations at lower pressures. The high relative cost of hydrogen manufacture has led to the generally accepted view that improvements in gasification processes are as important to the nation in achieving direct coal liquefaction at acceptable costs as they are for SNG production, power generation, and indirect coal liquefaction. An important area for improvement in the overall economics for direct coal liquefaction relates to improvements in the quality, as well as in the quantity, of the liquid product output. The costs of refining coal-derived synthetic crudes to marketable products are not insignificant. Hence, better processes and catalysts for the primary coal-liquefaction step will reduce downstream refining costs and thereby reduce the overall costs for producing specification products for the market. Higher coal conversions and reduced gas-make will also increase the total number of barrels of distillate products for a given plant investment, thus reducing the costs per barrel. However, an important consideration to bear in mind is the "balanced plant" concept of a coal liquefaction facility. All streams must have an ultimate disposition and there is a need to assure that all hydrogen and fuel requirements can be met. For example, increased conversion would reduce the amount of bottoms produced. If there are insufficient bottoms to produce the needed hydrogen, additional hydrogen must be brought into the system, usually by bringing in some coal directly for hydrogen production.

Table 4-1. Costs and cost distributions in coal liquefaction.

	Range for EDS, SRC-II, Dow and Zinc Chloride Processes
Capital cost, \$/T/CD	54,900 - 96,300
Liquid products, Bbl/T	1.9 - 3.5
Capital cost, \$/Bbl	15,700 - 47,000
<u>Capital cost distribution, %</u>	
Liquefaction	18.0 - 35.7
Hydrogen manufacture and cleanup	15.2 - 41.2
Other (power, utilities, tankage, offsites)	40.1 - 63.5
<u>Total costs, % (feed, capital charges, operation</u>	
Coal	11 - 35
Capital charges	38 - 62
Operating costs	26 - 27

Note: All costs are adjusted to 1979 dollars at 7%/yr. No attempt was made to place all of the processes on the same economic basis, except that 1979 dollars are used.

APPENDIX A:

FERWG-II STATEMENT OF WORK

The objective of this project is to conduct an independent assessment providing for identification of the long-range research needs associated with coal liquefaction. This work is expected to include recommendations to DOE for research programs that can best contribute to the successful long-term development of new coal-liquefaction technologies.

The contractor shall provide the necessary personnel, facilities, services, materials and documentation required to perform all of the required tasks.

In fulfillment of the project objectives, the contractor will be expected to work with both the academic community and industry. The assessment will consider all of the basic disciplines involved in the development of coal-liquefaction technologies. The researchers will be expected to gain first-hand familiarity with operational aspects of the gasification technologies through site visits, interviews, examination of development studies and reports, and other means.

Typical of the kinds of long-range issues that will be addressed are the following:

1. How much cheaper or more efficient may we expect coal-liquefaction technologies to be in the future compared with those that are now in use or under development?

2. Can we identify the scientific and engineering directions that will be useful in making these technological improvements?

3. What scientific and technical areas that are key to the success of ongoing coal-liquefaction R&D are still "open" areas for research, i.e. areas likely to profit from a broader or deeper look?

4. What disciplinary or interdisciplinary fields or research ideas should be supported because they hold long-range potential for generating innovative and useful technologies?

In complying with the objectives, the contractor shall perform the following tasks.

Task 1: Prepare a Detailed Work Plan

The contractor will prepare a work plan for DOE review and approval that will define the execution of the remaining two (2) tasks. This detailed work plan will include documentation of the following activities:

Description of a methodology for obtaining independent assessments representing a wide diversity of views concerning coal-liquefaction technologies.

Site-visits as required.

Technical approach for evaluating research needs for each project. The work plan will be submitted for approval by the DOE technical representative. DOE comments shall be furnished to the contractor within 5 working days after receipt.

Task 2: Conduct Assessment

In accordance with the detailed work plan (Task 1), the contractor shall conduct the necessary research and assessment activity. It is expected that a credible assessment will require use of a variety of information sources, including:

Site-visits to liquefaction-related projects to gain first-hand familiarity with currently available or developing liquefaction, technologies, including operational aspects; technical discussions with government, industrial, and research community specialists in coal liquefaction; review of relevant technical literature; other means as deemed appropriate, including the use of consultants from industry and the academic research communities.

The contractor will submit to DOE an interim letter report describing each site visit and significant technical meeting during this assessment.

Task 3: Prepare Final Report

The contractor will prepare a final report of this assessment for DOE. This report will include the following:

Purpose of study; study approach; list of consultants, if any, and other information sources; brief description of sites/projects visited; research needs identified, according to technology area, urgency, and expected benefit time-frame; recommendations.

SCHEDULE

Item 1: The Detailed Work Plan in accordance with Task 1: June 1979.

Item 2: A draft report on each site visit in accordance with Task 2: three (3) weeks after each site visit.

Item 3: Final Report, including Identification of Research Needs for Coal-Liquefaction Technologies in accordance with Task 3: March 1980.

APPENDIX B:

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APPENDIX C

SELECTED REPORTS OF SITE VISITS, CONFERENCES AND DISCUSSIONS

This section contains edited copies of site-visit and other reports prepared by FERWG members. Some of the hand-out materials prepared by DOE contractors and others are included (without explication) to permit readers the construction of a coherent picture of work in progress.

AC-1.1 REPORT OF SITE VISIT TO THE SRC FACILITIES AT FT. LEWIS, WASHINGTON

(MAY 11, 1979; VISITORS: F. LEDER, S. S. PENNER, E. REICHL, J. ROSS, R. SIEG, A. SQUIRES, L. TOPPER)

A. Research Needs

Discussions were held with John Ward (plant manager) and other SRC staff members, including J. Naylor, D. Canfield, and J. A. Segerson. R. Hamilton (Section Chief, Coal Liquefaction, DOE in Washington) also participated. The current status of the SRC-I and SRC-II programs was reviewed, including the authorized scale-up to a 6000 ton per day SRC-II demonstration plant.

Only about one half of the active volume of the dissolver is used in SRC-I. The residence time of the reactants in the dissolver is about 1 hour in SRC-II. The detailed flow behavior in this critical unit is not now well understood. For this reason, radioactive tracer studies of "backmixing" have been proposed in support of numerical model development for scaling by Norman Carr (Gulf R and D, Harmarville, Pa.). These studies are now in progress.

A visit to the facilities and dialogue with plant personnel (Duane Konen, David Williams, Fred Von Bargen) provided informative insight into plant operational problems.

Although SRC-II has operated successfully for 160 days, FERWG members attempted to identify hardware and operational problems which might profit from R and D activities that are performed concurrently with the scale-up of the SRC-II reactor to the demonstration plant at Morgantown, W. Va. The following listing is indicative of the topics that received attention in this connection:

1. Continued development activities are needed for let-down valves, especially for the handling of coal slurries. Let-down valves now last about six to twelve weeks. However, scale-up may lessen the trim breakage problem and lead to longer useful life.

2. The development of rheological models for coal slurries should be supported.
3. The design of a practical and inexpensive method for the preparation and blending of the coal feed should be supported in order to maintain uniform coal-feed properties. A computer-controlled procedure, of the type that has been used in Japan for the preparation of coke-oven feed, was suggested.
4. Methods for the identification and elimination of plugging need to be developed. Plugging has been the principal cause of shut downs: it has been associated with start-ups, shut-down or operator error and may be the result of local failures of electrical heating (plugging by chilling) or by repolymerization in the absence of adequate amounts of hydrogen (especially at elevated temperatures). Critical concentration ranges for operation without plugging must be defined.
5. The development of an on-line viscometer for measurements on slurry-filled pipes would provide a useful operational tool.
6. Reliable pumps must be identified or developed for handling vacuum-tower bottoms.
7. Corrosion problems can probably be handled by selecting the right type of steel. The relation of corrosion rate to chloride concentrations should be studied. Corrosion rates have been high in the middle fraction distillation column. There is a substantial temperature difference between two adjacent distillation columns. Observed chloride concentrations range from 6 to 200 ppm and chlorides in the oil may be corrosive.
8. A better solution than hastalloy needs to be found for the control of chloride corrosion.

9. Coking occurs on the pipes over long periods of time. The coke falls off periodically (during temperature cycling, start-up or shut-down). A device for measuring the rate of coke build-up on interior pipe walls and better knowledge of the mechanism of coking in the dissolver environment would be useful.

10. The nature of regressive reactions should be studied. We require definitions of allowable windows for hydrogen, hydrocarbons, stable coke build-up, etc. The early mesophase material is probably very sticky but formation may perhaps be reversed through introduction of appropriate hydrogen concentrations. Perhaps a "dirty" reactor can be run that will effectively isolate small (i.e., micron-size) mesophase material as it is formed and prevent its agglomeration to large domains.

11. Microscopic examinations of early residue formations may be useful.

12. A "run-away" reaction in the dissolver is probably not possible. Temperature spikes to ~930°F have been produced when the preheater outlet temperature was not used adequately for control of the reactor temperature; the normal reactor temperature is ~850°F. It might be useful to attempt identification of operational conditions needed for run-away reactions to occur.

13. The development of reliable control units, tied to a few important measurements, should be supported.

14. The relation between variability in coal-feed input and unit output requires study. Much useful information has been assembled empirically and will presumably be obtained also during operation of the demonstration plant.

15. Scale-up of preheater designs to commercial scale is uncertain and requires study.

B. Process Data

A schematic diagram of the Fort Lewis SRC-II facility is shown in Fig. AC-1.1-1. Eighty percent of the coal is fed in at a size range that will pass a 200-mesh screen. The total slurry feed rate is about 6000 lbs/hr of which about 2000 lbs/hr are coal. After drying, the moisture content is less than $\frac{1}{2}$%. The slurry blend tank is maintained near 350°F. The dissolver is at about 2000 psi and the slurry preheater at a somewhat higher pressure while a temperature of 750-800°F is reached. The inlet H_2-gas velocity (which may enter from one or more of three possible injection-port locations near the top, middle, and bottom of the dissolver) is fairly high. The normal entrance temperature to the dissolver is 750-800°F while the exit temperature is 860°F. The residence time in the dissolver is about 1 hour at a pressure of 2000 psi. The letdown system takes the material in two stages to about 150 psi.

Figure AC-1.1-2 shows a chronological summary of pilot-plant experience at Fort Lewis. Material balance and yield data for SRC-II are shown in Table AC-1.1-1 for controlled, equivalent feed material. The data show that the total output of distillates varies by about 15%. SRC-II product yields are shown in Table AC-1.1-2. The data summarized in Table AC-1.1-3 refer to results obtained with coals from three different mines. The SRC yields are seen to vary by more than 50% while the total distillate yields vary by about 40%. Comparison of the results shown in Tables AC-1.1-1 and AC-1.1-2 indicates that changes in coal feed produce substantially larger output variations than correspond to normal process variability. Results obtained in a materials balance test are given in Table AC-1.1-3.

C. Comments on Increasing the Probability of Successful Scale-Up of SRC-II

The following comments are offered in the hope that they will increase the chances of success in extrapolating the current 24 ton/day pilot plant results to a 3000 ton/day reactor system. This 125:1 scale-up ratio is proposed for the 6000 ton/day, 2 train, SRC-II demonstration plant at Morgantown, West Virginia.

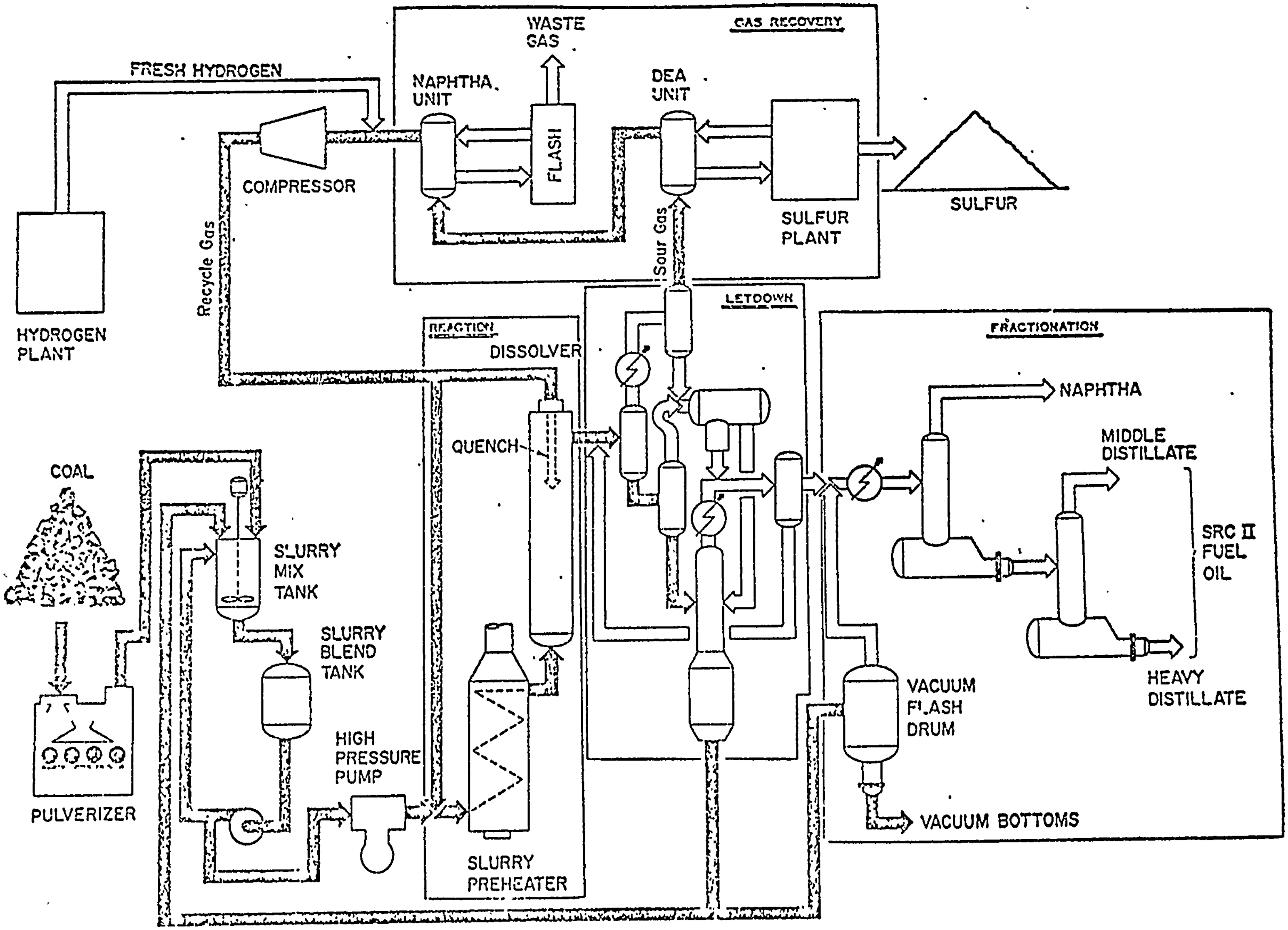


Fig. AC-1.1-1 Schematic diagram of the Fort Lewis SRC-II pilot plant.

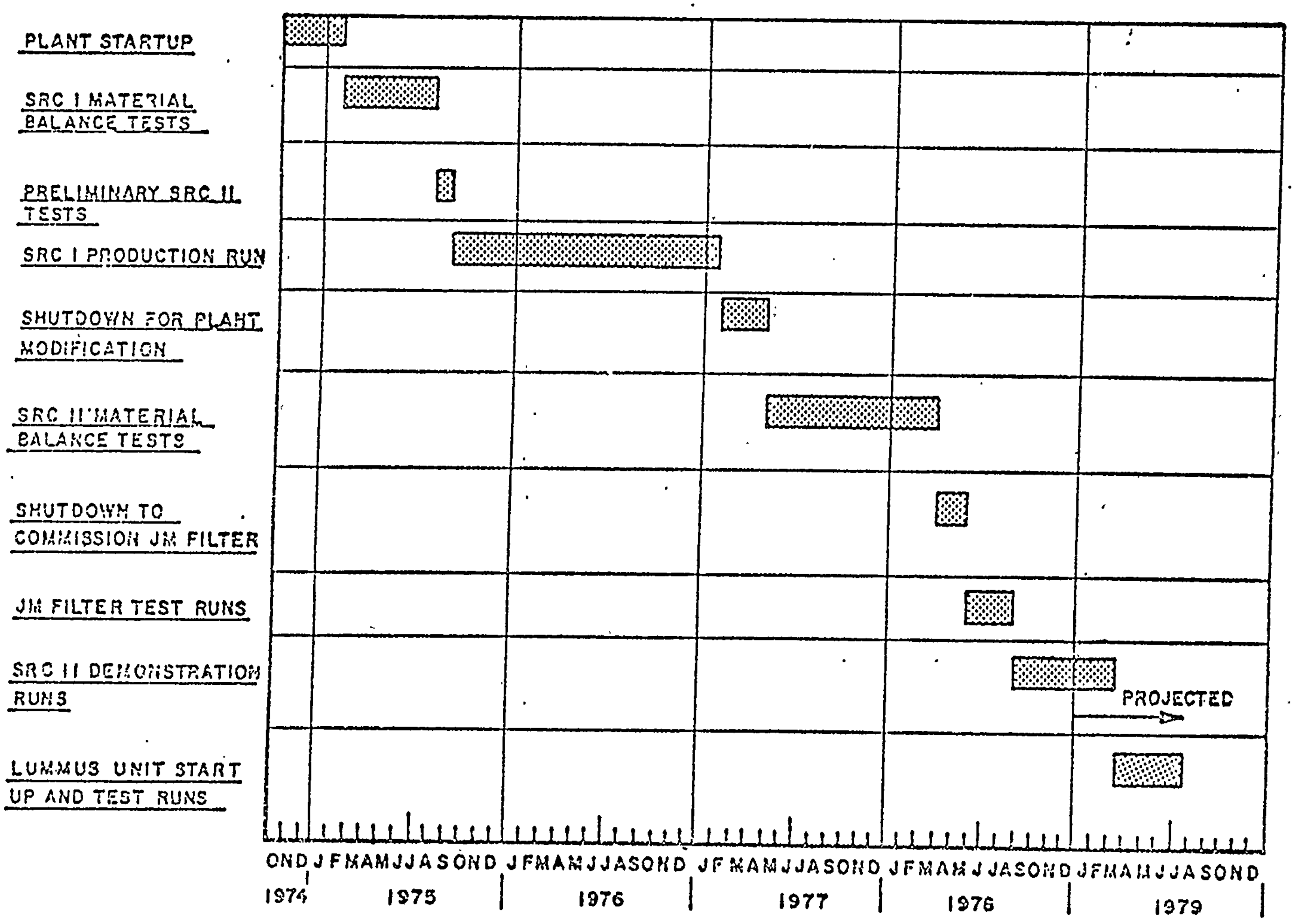


Fig. AC-1.1-2 Chronology of operating experience at the Fort Lewis SRC pilot plant.

Table AC-1.1-1 Material balance and yield data for the SRC-II pilot plant.

MATERIAL BALANCE RESULTS - ELEMENTALLY BALANCED YIELDS - DISTILLATE YIELDS BASED ON P&M DISTILLATION PROCEDURE

Conditions	Run 78SR-22	Run 78SR-23	Run 78SR-24	Run 78SR-25	Run 78SR-26	Run 78SR-27	Run 78SR-28	Run 78SR-29	Run 79SR-30
Dehumidified Coal Feed, lb/hr	2108	2072	2089	2104	2038	2089	2072	2136	2099
Feed Slurry Composition, wt%									
Coal	29.2	27.1	27.9	30.9	30.3	30.2	30.6	31.3	29.6
Mid. Dist (350°-450°F)	1.9	3.2	8.3	6.7	6.6	1.5	1.6	2.3	2.3
Hvy. Dist (450°-850°F)	28.2	26.9	21.8	21.1	25.2	27.6	29.5	25.5	27.3
SRC	25.5	26.6	25.1	24.7	23.3	27.0	25.1	27.0	26.9
Ash (due to recycle slurry)	10.2	11.0	11.3	10.7	9.6	9.1	9.2	9.4	9.6
IOM (due to recycle slurry)	5.0	5.2	5.6	5.9	5.0	4.6	4.0	4.5	4.2
Iron (total in feed slurry)	1.97	2.25	2.25	2.25	2.05	1.99	2.22	2.36	
Hour Space Rates, lb/hr ft^3									
Coal	22.9	22.5	22.6	22.9	22.7	22.7	22.5	23.2	22.8
Total Slurry	78.5	83.2	81.4	73.6	74.8	75.1	73.6	74.1	76.8
Nominal Dissolver Res. Time, hrs	0.91	0.86	0.88	0.97	0.96	0.95	0.97	0.97	0.93
Hydrogen Purity, Mole %	85.6	85.6	86.0	84.7	85.7	85.9	85.5	86.0	93.2
H_2/Slurry Ratio, lb H_2/100 lb slurry	4.35	4.66	4.96	5.06	5.35	5.56	7.13	6.70	5.18
Dissolver Pressure, psig	2005	2007	2003	2005	2004	2003	2003	2008	2002
Average Dissolver Temp., °F	853	852	853	860	857	852	852	851	851
Feed Coal	Powhatan #5	Powhatan #5	Powhatan #5	Powhatan #5	Powhatan #5	Powhatan #5	Powhatan #5	-1/8" Powhatan #5	Ireland Mine #5
%Ash in Moisture Free Feed Coal	11.24	11.76	11.47	11.24	11.79	10.53	9.99	12.32	11.51

ELEMENTALLY BALANCED YIELDS, %maf COAL FEED

Component	Run 78SR-22	Run 78SR-23	Run 78SR-24	Run 78SR-25	Run 78SR-26	Run 78SR-27	Run 78SR-28	Run 78SR-29	Run 79SR-30
H_2	-4.5	-4.6	-5.5	-5.2	-4.7	-3.9	-4.5	-4.5	-4.0
C_1	5.5	7.4	8.8	8.5	6.8	6.0	6.6	6.3	6.8
C_2	4.0 } 14.9	4.3 } 17.5	5.2 } 21.5	5.1 } 21.7	4.1 } 17.0	3.8 } 15.6	4.2 } 17.6	4.1 } 16.9	4.0 } 17.3
C_3	3.2	3.7	4.6	4.8	3.7	3.6	4.1	4.0	4.0
C_4	2.2	2.1	2.9	3.3	2.4	2.2	2.7	2.5	2.5
CO	-1.7	-0.1	0.2	-0.7	-0.5	-0.6	0.0	-0.6	-0.6
CO_2	2.0	1.9	1.4	2.3	1.3	1.6	1.3	1.5	1.2
H_2S	2.3	2.3	2.6	2.6	2.5	2.7	2.7	2.4	2.6
NH_3	0.5	0.5	0.6	0.5	0.5	0.4	0.6	0.6	0.6
Water	6.3	7.4	6.8	6.8	7.5	5.0	5.8	6.4	5.7
IOM	6.2	6.4	6.1	7.1	6.3	5.7	4.8	5.0	6.0
Naphtha + C_5^+	10.4	7.0	11.5	9.7	12.8	9.0	5.6	7.7	7.1
Mid. Dist.	30.7 } 43.3	21.6 } 41.0	22.7 } 40.7	21.7 } 37.2	28.4 } 42.0	28.1 } 43.1	22.3 } 45.5	19.3 } 44.1	23.3 } 40.0
Hvy Dist.	2.2	12.4	6.5	5.8	0.8	6.0	17.6	17.1	9.6
SRC	30.7	27.7	25.6	27.7	28.1	30.4	26.2	28.2	31.6
Fusion Point, °F	363	354	333	349	351	364	315	333	312
% Solids in Vac. Btms.	38.0	41.6	42.8	41.6	41.1	36.5	37.9	40.3	38.2

Table AC-1.1-2 SRC-II product yields.

Coal	K. Ky.	Ill. #6	Pitt. Seam
Dehumidified Coal Feed Rate, lb/hr	1984	2008	1998
Kg/hr	900	911	906
Feed Slurry Composition, wt %			
Deh. Coal	29.5	29.5	30.3
Solvent	33.4	35.6	29.8
SRC	23.9	20.0	24.1
Ash (due to recycle slurry)	8.2	11.0	8.3
IOM (due to recycle slurry)	5.0	3.9	7.5
Nominal Dissolver Residence Time, hrs	0.98	0.97	1.00
Hydrogen Purity in Feed Gas, mole %	89.8	93.7	91.6
Average Dissolver Temperature, °C	461	457	456
°F	861	854	855
Dissolver Pressure, MPa	13.34	13.44	14.09
psig	1920	1934	2029
Yields, wt % M.A.F. Coal			
Hydrogen Consumption	-4.8	-4.7	-3.5
Hydrocarbon Gas (C_1 to C_4)	18.4	15.8	15.5
Naphtha	14.2	17.0	11.9
Middle Distillate + Heavy Distillate*	28.2	30.3	22.3
SRC*	26.1	23.0	36.8
IOM (unreacted coal)	6.6	5.0	11.9
Total Distillate Yield** (bbl/ton M.F. coal)	2.27	2.46	1.89

*Adjusted to produce a vacuum bottoms product with a 177°C (350°F) fusion point.

**Naphtha, Middle Distillate, and Heavy Distillate

Table AC-1.1-3 Results obtained in a material balance test.

Operating Conditions

Coal	Western Kentucky #9/#14	
Moisture Free (M.F.) Coal Feed Rate	1837.0 Kg/hr	(4050 lbs/hr)
Solvent Feed Rate	2860.4 Kg/hr	(6306 lbs/hr)
Hydrogen Purity in Feed Gas	94.1 mol %	
Hydrogen Feed Ratio	529 cu m/ M.T. moisture free coal feed	
Dissolver Outlet Temperature	451°C	(846°F)
Dissolver Pressure	10.33 MPa	(1484 psig)

Product Yield	% M.F. Coal
Hydrogen	-2.4
Methane	1.4
Ethane	1.0
Propane	0.9
Butane	0.4
Carbon Monoxide	0.0
Carbon Dioxide	0.8
Hydrogen Sulfide	1.4
Water	5.0
Light Oil (Ambient - 193°C)	5.1
Wash Solvent (193°C-249°C)	4.0
Process Solvent (249°C-454°C)	4.4
SRC	63.0
Ash	9.6
Unreacted Coal (IOM)	5.3

No advice is offered with regard to the following critical questions:

(a) Is the demonstration at a 6000 ton/day scale to be recommended?

(b) Is the processing of the vacuum-tower bottoms ready for larger scale application by the Texaco process?

Comments on the SRC-II mode of operation:

(1) The slurry preheater is one of the critical items of equipment. The present 3" tube heater, or a new 2" tube heater, will be further studied to yield data for plant design. A detailed description of heater design should be included in Phase O - Scope of Work to assure that the extrapolation is as safe as possible.

(2a) Dissolver

The projected extrapolation in handling capacity is 125:1 for the unit at Ft. Lewis. However, extrapolation from German commercial units is 10 to 1 or less. Although German operating conditions were at 50 to 100% higher pressures, the basic operations of the slurry phase reactor (using gas circulation as a source of agitation) are quite similar. An attempt should be made to find German engineering data for sump-phase hydrogenation reactors in the current review of German documents conducted at Texas A&M; DOE should arrange access to this project for the SRC-II managers.

(2b) Dissolver Model

It is recommended to include (in the scope of Phase I of the demonstration-plant project) a cold model for the proposed full-size reactor to study the 3-phase system for channeling and settling techniques. A program using radioactive tracer particles is now scheduled at the Tacoma plant.

This study will be useful to assure better understanding of the behavior of solid, liquid and gas flows under process conditions.

(3a) Vacuum Tower

The low-pressure, hydrogen-free section of the process is apparently the more difficult part of the system to use, from the point of view of operating reliability. This difficulty results from the high tendency for coking of the heavy primary product. The SRC conditions are much milder than those used in German commercial experience and the resulting product is far less stable.

To obtain better operating reliability, improved knowledge must be gained of the condition of the vacuum-tower inventory. Measurements of viscosity and/or solids concentration would be helpful. It is suggested that on-line instrumentation be explored for this purpose (e.g., a Stormer viscosimeter).

(3b) Vacuum-Tower-Bottoms Pump

Development of a reliable positive displacement pump for handling the vacuum-tower-bottoms stream should prove helpful. We learned that this work is already in progress. Availability of such a pump may obviate the need for the costly high elevation of the vacuum flash unit that is now used.

(4) Slurry Handling

It is evident that a great deal of very valuable detailed mechanical and operational know-how has been accumulated at Tacoma. Much of this now resides in the experienced operating crews, at all levels.

It is particularly important for the scale-up project that this know-how is applied, to the maximum extent possible, in the detailed mechanical design of the demonstration plant. The plant operators should be given specific time to review the mechanical layouts in great detail (particularly piping details) and the plan of work should include specific time and money for changes requested by this review. There is no way to replace this operational know-how by "science".

(5) Philosophy in Designing the Demonstration Plant

The SRC processes (I and II) operate under conditions of low severity, at least insofar as complete degradation and conversion of coal to liquid are concerned. As a result, the processes are not very "forgiving" to errors, particularly in the downstream sections (deashing, vacuum distillation).

It would be best to design the demonstration plant with some leeway as far as the severity of processing conditions is concerned. Thus, plant reliability may require some increase in both maximum H_2 pressure and maximum H_2 circulation rate. It is recommended that this extra capacity (above the base-case) be provided in the demonstration plant, in spite of the obviously higher cost.

AC-1.2 REPORT OF SITE VISIT (OCTOBER 22, 1979) TO THE GULF OIL RESEARCH LABORATORY, HARMARVILLE, PA.

The primary purposes of the site visit to the Gulf Oil Research Laboratory at Harmarville, Pa., were further discussions of the SRC-II experience and program plans, with special reference to completed studies on the P-99 PDU, modeling and scaling. The following FERWG members participated: Clardy, Furlong, Leder, Penner, Ross, Sieg, and Squires. The group was joined by I. Wender (Fossil Energy) and R. H. Kropschot (Office of Basic Energy Sciences) of DOE and by R. F. Bauman of the Exxon Baytown Research Laboratory. Discussions were held with the following staff members of the Gulf Oil Research Laboratory: N.L. Carr (Gulf Research and Development Center, GR&DC, C&MD), H. G. McIlvried (GR&DC - C & MD), K. Parimi (GR&DC - C&MD), J. L. Stephenson (GR&DC), R. G. Sperhac (GMRC), and others.

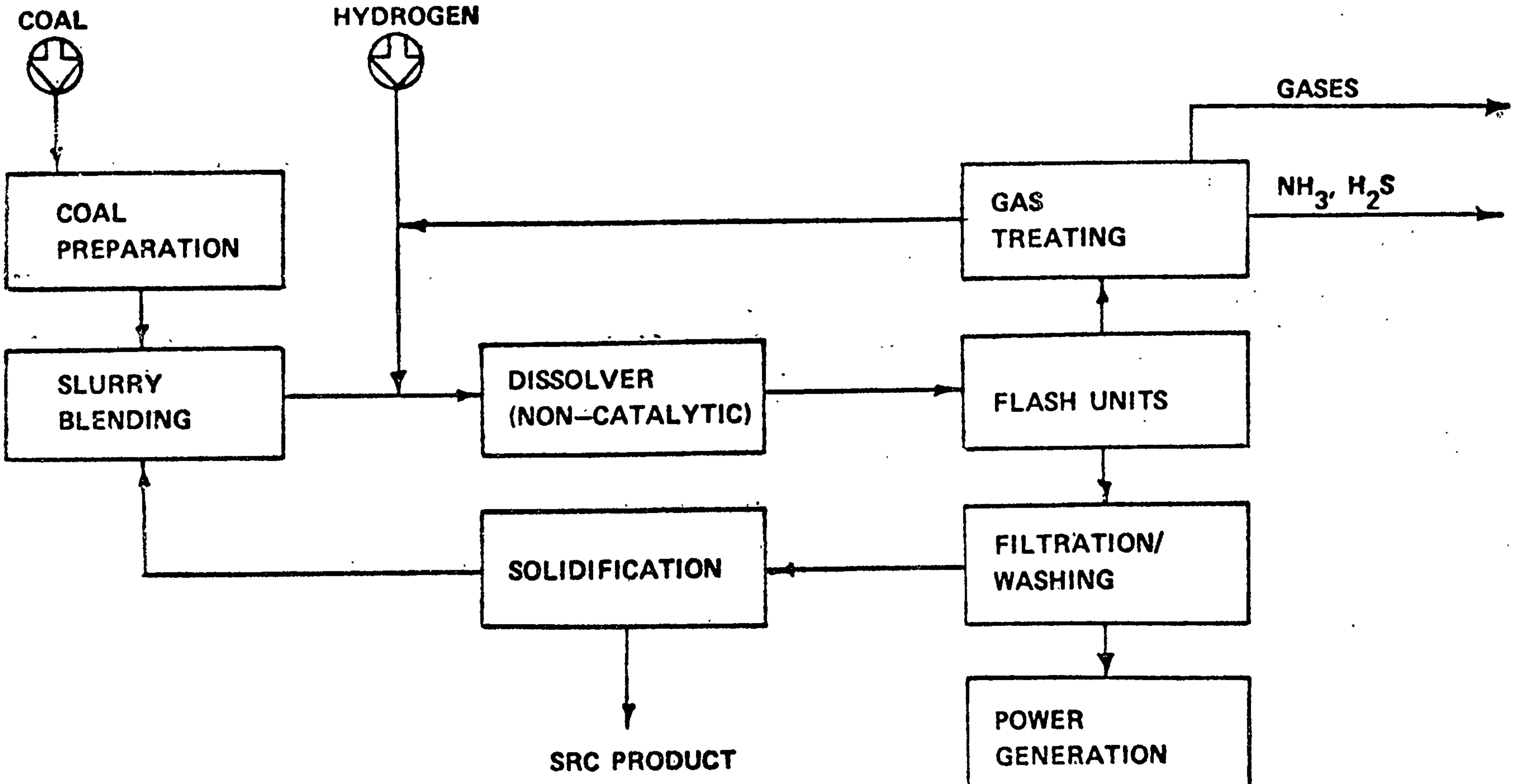
The following formal presentations were made:

McIlvried, P-99 Operations;
Carr, Mathematical Modeling and Correlation Work;
Stephenson, Physical Properties;
Parimi, Correlation and Equipment Design.

A. The SRC-I Process

A flow diagram showing the SRC-I process is reproduced in Fig. AC-1.2-1.

Fig. AC-1.2-1 Schematic diagram showing the SRC-I process.



B. P-99 PDU Studies on the SRC-II Process

A schematic diagram of the SRC-II process is shown in Fig. AC-1.2-2. A simplified flow diagram for the P-99 PDU is reproduced in Fig. AC-1.2-3. The P-99 PDU is designed to utilize 1 ton of coal per day but has usually been operated at a one half ton per day utilization rate. Program objectives are summarized in Table AC-1.2-1. Operating schedules are indicated in Fig. AC-1.2-4. The coals that have been tested in the P-99 PDU are listed in Table AC-1.2-2 while Table AC-1.2-3 shows the ranges of operating conditions used (these include anticipated process conditions for each of the listed variables in the demonstration plant). The P-99 feed and product streams and the corresponding methods of measurements are summarized in Table AC-1.2-4.

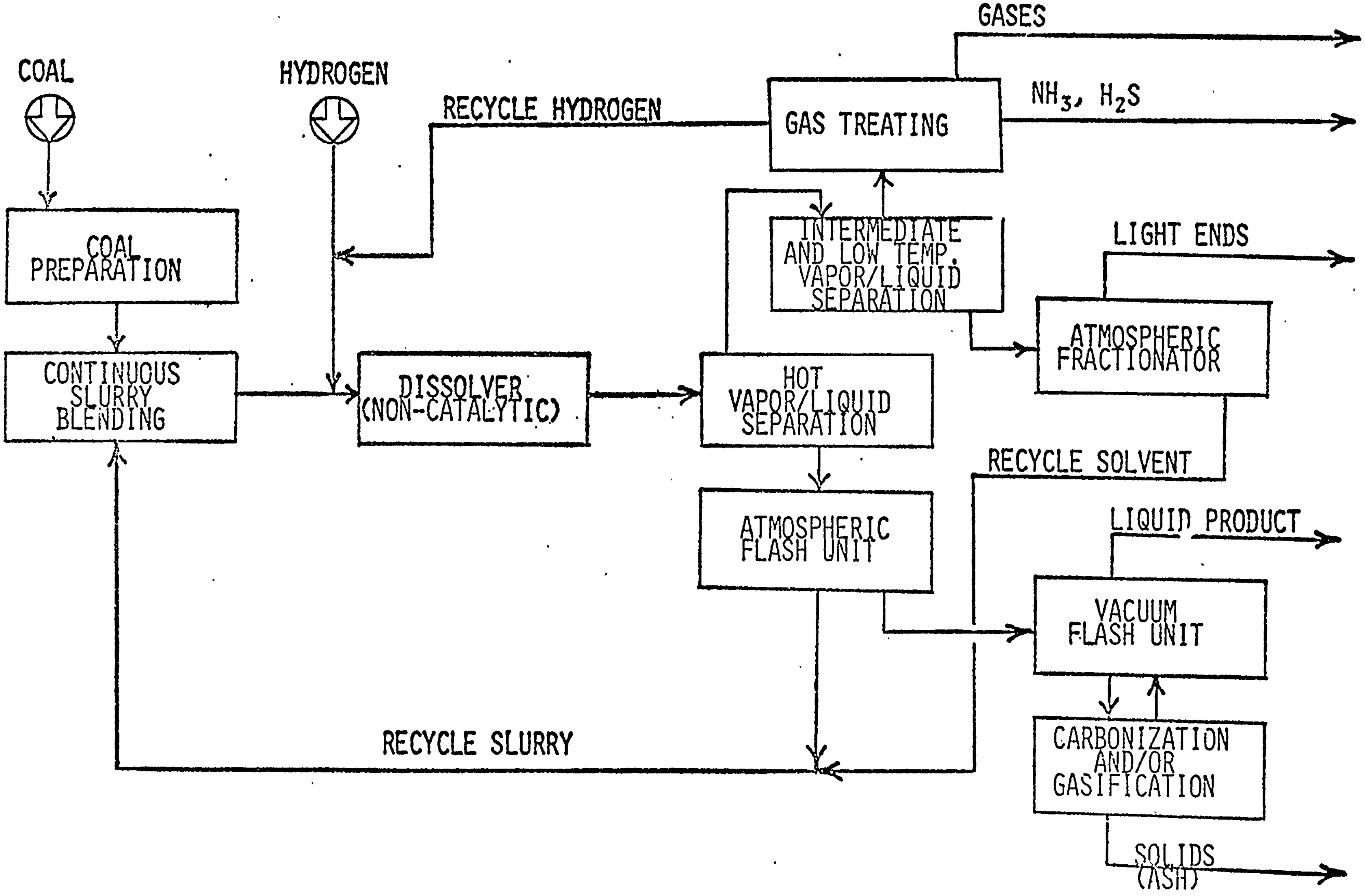
A listing of needed or measured physical and thermal properties data appears in Table AC-1.2-5.

We refer to our earlier site visit report (dated May 11, 1979) for detailed comments by FERWG members on the Ft. Lewis SRC-II studies and for recommendations concerning the large demonstration plant which is currently under construction. Here we confine further detailed assessments and recommendations to remarks on modeling and scaling.

C. Modeling the SRC-II Dissolver; Management Problems

Workers at Gulf claim to have a good model for the hydrodynamics and chemistry of the SRC-II dissolver. However, only broad general statements about the model were made to FERWG. Modeling was funded by Gulf until January 1, 1979; the contract for DOE support of the effort from this date onward is not yet complete; until Gulf has been paid retroactively for work performed this year under the new contract, details of the model will apparently not be divulged.

Fig. AC-1.2-2 Schematic diagram showing the SRC-II process.



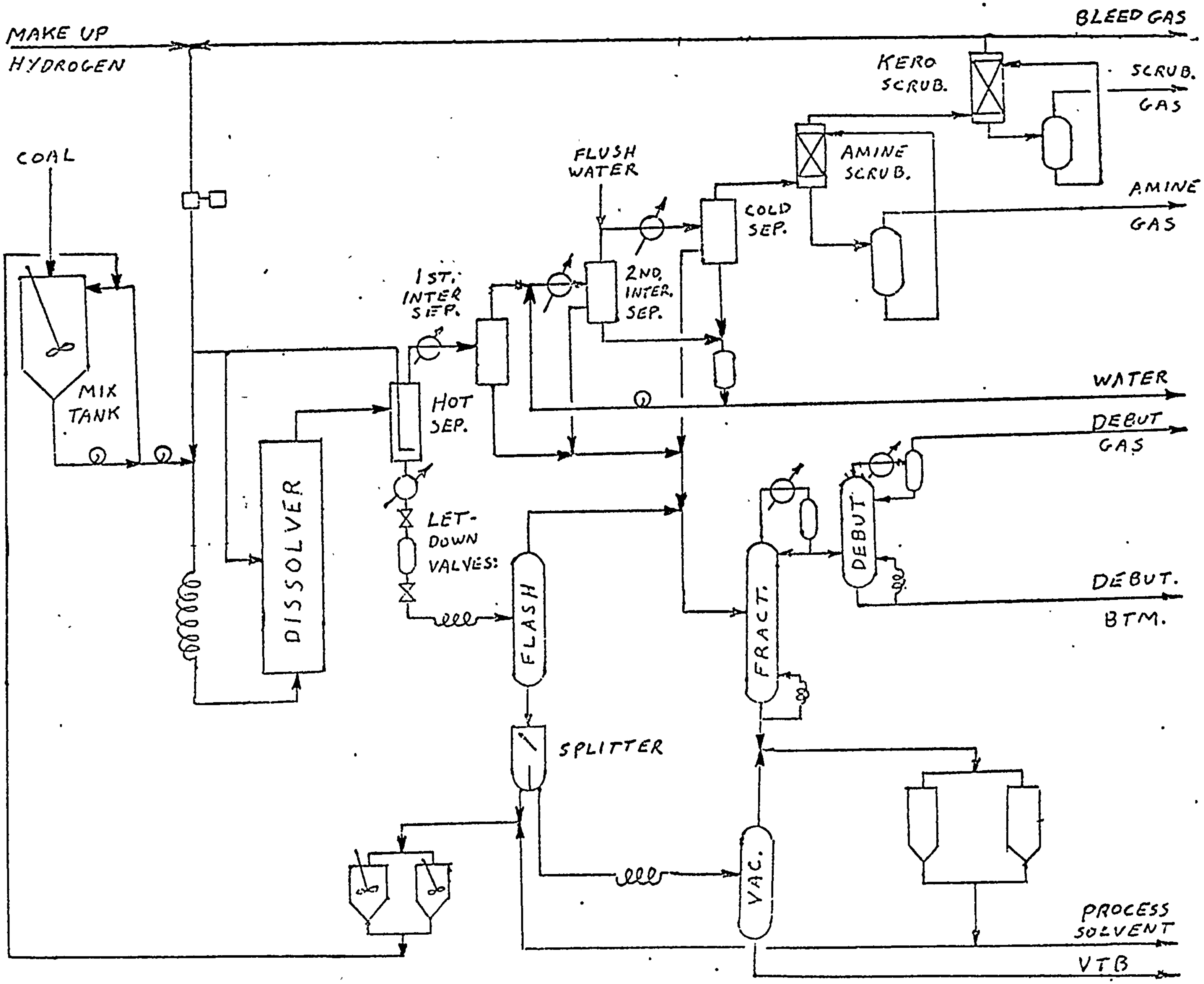


Fig. AC-1.2-3 Simplified flow diagram of the P-99 facility.

Table AC-1.2-1 Program objectives of the SRC-II pilot-plant studies.

DEMONSTRATE PROCESS OPERABILITY

Accomplished; stability has been verified.

Use bituminous coals: Kentucky, Illinois and Pittsburgh

Seam coals have been run; the ash is satisfactory.

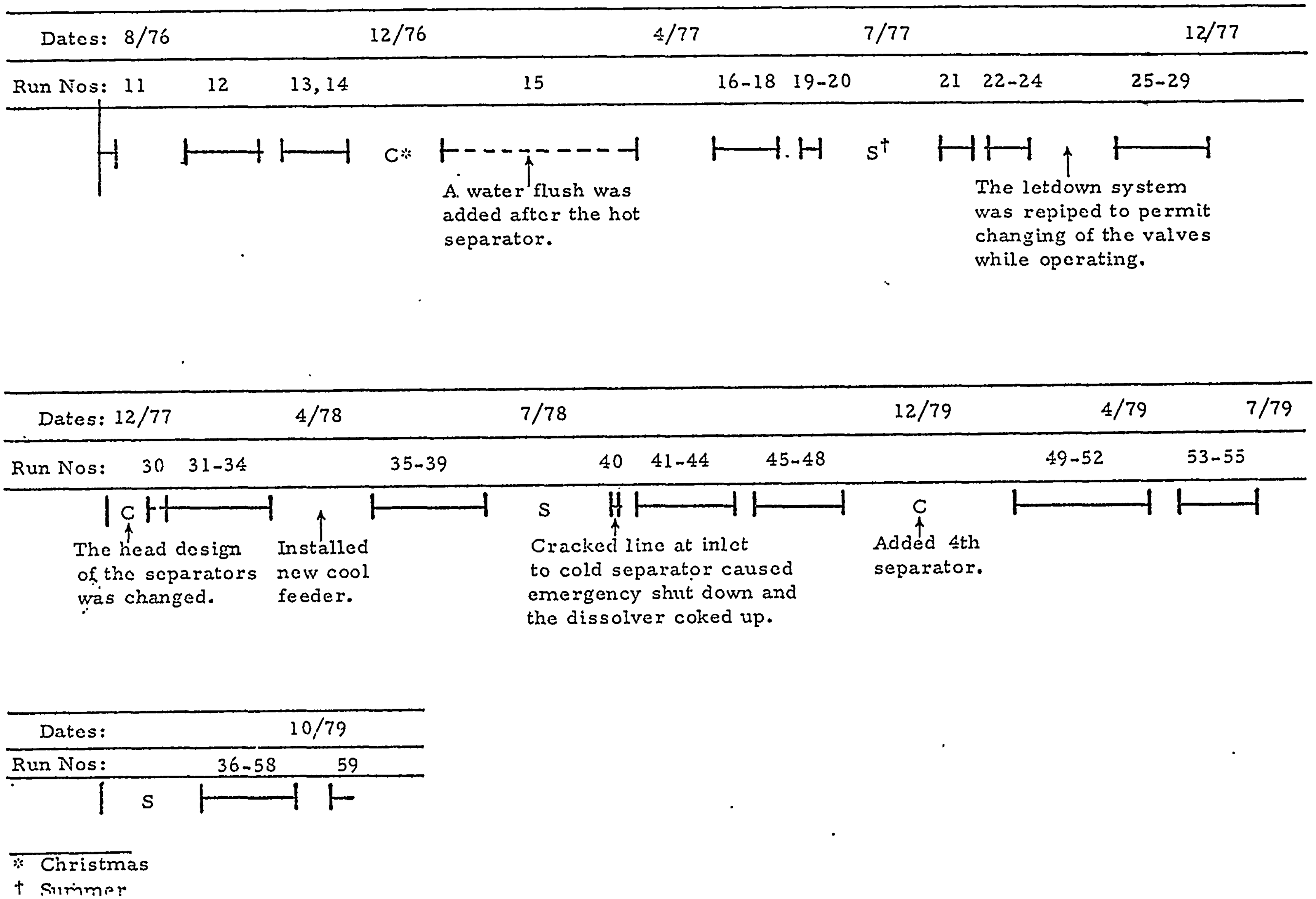
Beneficiated coal has been tried.

OBTAIN YIELDS AS A FUNCTION OF OPERATING CONDITIONS AND COAL SOURCE

TEST EQUIPMENT CONFIGURATIONS

PROVIDE DESIGN DATA

Fig. AC-1.2-4 Operating schedules followed for Kentucky coal; the lengths of the lines measure the test durations.



A water flush was added after the hot separator.

The letdown system was repiped to permit changing of the valves while operating.

The head design of the separators was changed.

Installed new cool feeder.

Cracked line at inlet to cold separator caused emergency shut down and the dissolver coked up.

Added 4th separator.

* Christmas
† Summer

Table AC-1.2-2 Coals run in the P-99 program.

A steady-state point is defined in 5-7 days; a materials balance is done once per day. All coals are deep-mined. There is very little difference in process solvents for different coals.

KENTUCKY COAL	11 RUNS
PITTSBURGH SEAM COALS	
BLACKSVILLE	6 RUNS
IRELAND	2 RUNS
POWHATAN (I)	3 RUNS
VALLEY CAMP	2 RUNS
ROBINSON RUN	4 RUNS
POWHATAN (II)	3 RUNS
POWHATAN (III)	8 RUNS
POWHATAN (IV)	2 RUNS

Table AC-1.2-3 Range of operating conditions used in the P-99 program.

PRESSURE	2000 - 2500 PSIG
TEMPERATURE	851 - 869°F
SLURRY RESIDENCE TIME	0.7 - 1.3 HR.
COAL CONCENTRATION	25 - 40 WT %
GAS CIRCULATION RATE	48000 - 89000 SCF/T
GAS PURITY	80 - 94 VOL % H_2
TOTAL FEED SOLIDS	40 - 49 WT %

Table AC-1.2-4 P-99 feed and product streams.

Feed	How Measured
Coal	Weigh Belt
Make-up Hydrogen	McAfee Gauge
Flush Water	Calibrated Tank
Flush Oil	Calibrated Tanks Minus Weight of Flush Oil Leakage
Product	
Excess Process Solvent	Change in Inventory of all Tanks
Debutanizer Bottoms	Weigh Cells
Vacuum Tower Bottoms	Scales
Process Water	Weigh Cells
Bleed-Off Gas	Gas Meter + Chromatographic Analysis
Naphtha Scrubber Reactivator Gas	McAfee Gauge + Chromatographic Analysis
Amine Scrubber Reactivator Gas	McAfee Gauge + Chromatographic Analysis
Debutanizer Overhead Gas	Gas Meter + Chromatographic Analysis
Chromatograph Purge Gas	Gas Meter + Chromatographic Analysis
Compressor Leak Gas	Nitrogen Content

Weight and elemental balances are performed; elemental analyses are done by adjusting H_2O, etc., so that elemental balances are good to a couple of percent for 5-7 days; larger variations may be observed on individual days. "Settling" in the lines had been eliminated by not using tees; all tank contents are stirred.

Table AC-1.2-5 Physical and thermal property data for the SRC-II project.

Coal Liquid Characterization - 50°F Cuts

density

molecular weight

elemental analysis

viscosity

specific heat

heat of vaporization

thermal coefficient of expansion

Heat of Reaction in Dissolver

- data from adiabatic dissolvers in pilot plants
- function of coal type and hydrogen consumption
- consistent method for calculating quench requirements for demonstration plant design conditions

Table AC-1.2-5 (continued)

Vapor/Liquid Equilibrium Correlations

Basis: modified Grayson-Streed correlation

- Standard correlation for critical temperatures, critical pressures modified to suit data on coal liquid vapor pressures.
- Experimental data on light gas solubilities (hydrogen and methane), at high P and T, have been incorporated.
- Experimental program for verification of K-value correlations, using representative composite streams from the SRC-II pilot plant (P-99).
  - High-pressure separator conditions:
    2000 psia @ 400 to 800°F.
  - Intermediate pressures:
    50 to 200 psia @ 500 to 800°F.
  - Vacuum system studies:
    15 to 30 mm Hg @ 600 to 750°F.

It is claimed that only small performance differences have been encountered among the three operating SRC-II dissolvers (Merriam, Harmarville, and Ft. Lewis) that cannot be explained by Gulf's reactor model. The gas flow is plug flow, while the liquid-solid slurry is essentially fully back-mixed. The conception of the reaction path is that the first, fast reaction regime must be handled by empirical correlations, with no real handle on the reaction kinetics, whereas good kinetic correlations can be developed for the slower reaction regime that follows. Hold-up relationships have been developed for the gas and slurry phases. There is no evidence of solids settling at the gas velocities used and no detectable change in particle-size distributions in and out of the dissolvers; the demonstration-plant dissolver will use higher velocities and therefore settling is not expected to occur. The particles display a 3 μm median diameter, with a range of 1 to 20 μm. Large inert particles may settle, but means for dealing with this problem are available.

There is a bench-scale, continuous, stirred-tank dissolver, and results from this unit and the three SRC-II dissolvers mentioned above are consistent. These comparisons have served to verify the reactor model to some extent.

There are minimum values for the liquid and gas velocities that must be maintained to preserve thermal stability. More research could be used on this problem.

The Ft. Lewis dissolver is 24 inches in diameter. There are no plans for a full-scale mockup of the demonstration plant dissolver; instead, the reactor model (based, in part, on experiments with mockups at a smaller scale) is being used for this purpose.

A dialog developed over Gulf's desire to use its reactor model for a harder look at operating transients, such as start-up or upsets. Workers at Gulf want to use the model to develop control philosophies: e.g., is it best to turn down the gas and slurry flows in step with one another or to turn down one of these flows at a faster rate? Gulf would like to develop a simulator that could address such questions and could eventually be used to train SRC-II operating personnel.

D. Modeling of the Preheater

Workers at Gulf believe that they have some feeling for the preheater from using kinetics for the fast reaction regime in this unit. However, research is clearly needed to obtain a tighter handle on preheater design and operation. The demo plant will use a short-residence-time preheater. Ft. Lewis personnel will operate such a preheater at some time in the future. Ft. Lewis is not the proper place to study the fundamentals of the preheater: it will give an overview, but other work should be done on a smaller scale.

There are plans to study flow regimes in the preheater and in the reactor effluent line. DOE has agreed to fund this work at the technical level, and the matter is now in DOE procurement. It is hoped that "homogeneous" flow can be achieved in the preheater, i.e., the so-called bubble/froth regime will obtain with a well-dispersed liquid phase.

E. Comments on the SRC-II Development Plan

It appears that some of the Gulf engineers are not totally satisfied with current SRC-II development plans. Some of the Gulf people would modify Ft. Lewis to conform to the flowsheet now contemplated for the demo plant and would obtain data from Ft. Lewis operating in this mode in time to modify the demo plant design. [Scoping studies of the recommended Fort Lewis modification became later (February 1980) a part of the SRC-II demonstration project.]

The "design coal" for the demo plant is a "Panhandle Coal", meaning a range of coals from the West Virginia Panhandle, with enough flexibility in the demo plant design to handle a range of these coals. A group of FERWG members, who visited Ft. Lewis last May, was struck by the variability in performance on a number of Pittsburgh Seam coals. When asked about the extreme difference in behavior, for example, between Blacksville and Powhatan coals during the Harmarville visit, Gulf personnel stated that the Blacksville coal "has an ingredient missing", but further information was withheld because Gulf has filed a patent application on this. It is likely that Gulf personnel know how to "treat" the Blacksville coal or to add something to make it perform better; however, there does not yet appear to be a handle on the economics of "upgrading" Blacksville coal in this way. Blacksville coal can be made to perform as well as Powhatan by operating at higher reaction severity (higher pressure and temperature). The tenor of the discussion of the poor performance of Blacksville coal, in comparison with Powhatan, suggested that Gulf workers are still deciding how to deal with the Blacksville coal most economically. Extensive reserves of the Powhatan type coal exist that are sufficient to support the demonstration plant and several commercial plants using Pittsburgh Seam coal.

Another element of uncertainty is the gasification of vacuum bottoms by the Texaco process. Only limited test data are available for operation of the Texaco process on liquefaction bottoms. It might fairly be questioned whether the data base for this process is sufficient for confident design of the demo plant at this time.

F. Process Research

Most of the needed process studies are in progress, either at Gulf or at PETC. The following additional investigations may be useful:

a. Coal Effects -- Most of the research to date has been done on Kentucky and Pittsburgh Seam coals. Identification of effects produced by coal-ash constituents, added pyrites and catalysts on a broader range of coals would be helpful to a future industry.

b. Solvent Effects -- The observation that the solvent has the same composition in and out of the reactor does not rule out significant solvent effects in the process but could reflect on a particular steady-state that is achieved during processing.

c. Control Strategies -- Modeling and simulation to plan control strategies for transients and system perturbations should be done as soon as possible and were suggested by Gulf personnel.

d. Reactor Design -- The proposed modeling and tracer studies were well thought out.

e. The PDU P-99 is a well designed, small unit which, based on a record of over 40 runs since 1976, has supplied useful process data with only modest operational problems. Problems with letdown valve life, preheater designs scale-up, etc., have been common to all of the coal-liquefaction units. Mention of an actual failure due to chloride stress-corrosion cracking of a line is a warning on possible materials problems that must be understood and considered in any scale-up design. A great deal of additional information on the SRC-II process could be obtained with the PDU P-99, particularly evaluations of a broader range of coals or even coals from the same seam but different mines or locations in a mine. An adequate range of operating variables has been covered to support the demonstration plant design.

The PDU P-99 dissolver has an L/D of about 20, the plant at Ft. Lewis of about 15, and the demonstration-plant design about 10. If PDU P-99 is substantially backmixed, the demonstration plant will be even more strongly backmixed.

The ability to obtain good material balances and sustained steady-state operations indicates good design practices. The data base on the coals tested (Kentucky and Pittsburgh Seam coal) should be sound.

G. Basic Research on Modeling and Scaling

For reasons that are unclear to us, we have encountered educated opinion to the effect that "scale-up of coal liquefaction plants represents no problem and research is not needed in this area." This judgement is further emphasized by the existence of systematic studies at university laboratories on modeling and scaling of coal-gasification systems (largely supported by EPRI), whereas comparable basic studies on modeling and scaling of coal liquefaction units do not exist. While coal-gasification plants are being scaled by about a factor of 10, coal-liquefaction plants are being scaled by more than a factor of 100 in proceeding toward commercial plant sizes.

FERWG and Gulf Oil Co. participants at the discussions of the SRC-II pilot-plant and demonstration-plant program on October 21 concurred that a substantial, well integrated, and sound program on modeling and scaling of coal-liquefaction processes is of the utmost importance and should be viewed as a small incremental charge for increasing the probability of success of the SRC-II demonstration plant. Studies of this type will furthermore assist in the development of logical process-modification procedures in the event that unexpected operating difficulties are encountered.

The motivation for the recommendation made in the preceding paragraph will now be clarified.

The normal progression of complex process technology, performed in industrial laboratories and funded by discretionary company resources, involves a meticulous progression of size from bench scale to the small pilot plant and then to progressively larger process development units before a demonstration module is constructed. There are no fixed rules for the measures of scale up. Instead, depending on the process under development and the operating difficulties encountered at each scale, intelligent judgements are made on how to proceed in view of acceptable risks at the needed levels of investment. This widely accepted procedure is being short-circuited because of (a) economic and political pressures for early demonstration of successful operation and (b) the infusion of very large amounts of government money that reduce or eliminate the risk of failure for the operating company. Both the SRC-II and H-coal demonstration plants are being scaled up more rapidly than they would be scaled up without government support.

Accepting the decisions on funding and scale-up that have already been taken, it clearly falls within the FERWG charter to seek supporting R and D activities that will reduce the risks of failure in these costly programs. While our fundamental knowledge of procedures for modeling and scaling is adequate, it is clear that the available methods cannot be applied without learning about process performance and variables that must be defined empirically. What is the set of dimensionless groups that must remain invariant for "exact" scaling? Which of these dimensionless groups (Peclet, Reynolds, Damköhler, etc. numbers) are of primary and which of secondary importance? What are the proper constitutive equations for the non-Newtonian fluids that represent coal slurries? What kinds of physical measurements are needed on properties for a detailed modeling study?

We were gratified to find at the Gulf Research and Development Corporation a group of dedicated people who were asking questions of the type specified in the preceding paragraph. We strongly support their proposals for an augmented effort on modeling and scaling within the active research group that is responsible for the development of the SRC-II demonstration plant. In addition, we recommend the development of a longer-term, university-based program on the general problems of modeling and scaling of coal-liquefaction components and processes, coupled with systematic measurements of those special physical and chemical properties that must be known to improve the process descriptions provided by models and the probabilities of successful scale-up.

Studies on modeling and scaling are not right or wrong. They are either useful or useless. We expect that they may prove to be useful in increasing the successful and cost-effective development of SRC-II demonstration plants for coal liquefaction.

AC-2. REPORT OF FERWG SITE VISIT TO
EXXON RESEARCH AND ENGINEERING COMPANY,
BAYTOWN RESEARCH AND DEVELOPMENT DIVISION
BAYTOWN, TEXAS
(JULY 13, 1979)

FERWG Members Who Participated: John Clardy, L. E. Furlong (host), S. S. Penner, A. M. Squires, and J. R. Thomas.

EXXON Participants: R. F. Bauman (Director, EDS Process Research Laboratory), L. E. Furlong (Director, Coal Research Laboratory), W. N. Mitchell (Section Head, EDS Process Research Laboratory), R. P. Souther (Process Superintendent, ECLP-Carter Oil), S. Zaczepinski (Section Head, EDS Process Research Laboratory).

R. C. Neavel (Scientific Advisor) joined the group for discussions during the afternoon and L. L. Ansell (Project Leader for CLPP) served as a guide at CLPP.

A. Facilities and Status of the Exxon Donor Solvent (EDS) Process

The following facilities are currently available or under consideration at Baytown:

(a) A tubing bomb reactor, which is a batch autoclave for the liquefaction of samples amounting to several grams of coal in a donor solvent and under an atmosphere of hydrogen; also a stirred autoclave for liquefaction of larger coal samples (30 g) in a donor solvent under H_2 pressure.

(b) RCLU (for Recycle Coal Liquefaction Unit), which is a flow system for isothermal reaction of coal at a rate of 50 and 100 pounds per day. The solvent is recycled while the hydrogen flow passes once through the RCLU; there are two RCLUs at Baytown.

(c) CLPP, pronounced clip (for Coal Liquefaction Pilot Plant), which is a complete unit for the liquefaction of one ton per day of coal. Both hydrogen gas and solvent are recycled. The reactor is approximately adiabatic. There is a vacuum pipe still giving a 1000° F cut point.

(d) ECLP, pronounced eeclip (for EDS Coal Liquefaction Plant). This unit processes 250 tons per day and is sufficiently large to test furnaces, let-down and other valves, pumps, compressors, and other components that may then be readily adapted or scaled for commercial applications. The flow scheme is the same as for CLPP. The ECLP facilities cost $\$110 \times 10^6$ while the program is funded for $\$240 \times 10^6$. A Flexicoker pilot plant is available for use in the Baytown refinery. Modifications are required to run coal liquefaction bottoms. The decision to modify and test this unit is currently being evaluated.

The ECLP is about 1% of full scale insofar as coal-handling capacity is concerned. It is a salutary experience to view the enormity of this operation and to contemplate the prospect that production of 2×10^6 B/D at 60-70% efficiency will require 36 plants, each processing 100 times the amount of coal that will be used in ECLP.

(e) A design basis for a commercial EDS plant processing 25,000 to 30,000 tons per day of coal is expected to be ready for licensing by 1982. A developed bottoms process would also be required.

A CLPP One unit operated at 0.5 ton/day between about 1965 and 1969. CLPP One had centrifuges and filters, as well as a vacuum tower to recover residue-free product. The primary purpose in the operation of CLPP One was to establish the process scheme for the EDS Process. CLPP One was dismantled when Exxon's interest in coal liquefaction waned because of development of Alaskan North Slope oil. CLPP Two, the present unit, was built in 1975. ECLP was designed in 1974-75, but incorporates results of CLPP operations obtained during 1975-78.

The EDS development since September 1977 has been funded through a cooperative agreement. Participants include EPRI, DOE, ARCO, JCLD, Ruhrkohle, Phillips, and Carter Oil. Essentially all of the major pieces of equipment for ECLP are now in place, but piping is not yet complete. ECLP is scheduled to start operation in February 1980.

With the currently envisioned cooperative agreement and assuming sponsor approval of the Flexicoking Part II program, the design basis for a pioneer commercial EDS plant could be established in 1982. Without the Flexicoking component, an acceptable alternative such as partial oxidation of bottoms would require definition. If a large pilot plant program were required for partial oxidation, a design basis might not be available until 1984 or later. Given a design basis in 1982, a commercial plant could be on-stream in 1988 or 1989 at the earliest. This projection assumes that the commercial plant owner would have favorable financial incentives and expeditious assistance on permitting, particularly with respect to environmental and health issues. It would be very difficult to accelerate this schedule at acceptable levels of risk.

As we have already noted, the ECLP (which is one percent of full scale) project is funded at $\$240 \times 10^6$. Exxon had $\$32 \times 10^6$ in the EDS Process before this project was funded jointly. Contributions are coming into the $\$240 \times 10^6$ budget, reducing Exxon's and EPRI's shares. A $\$20 \times 10^6$ contribution earns the right to "bring in a coal" for testing in CLPP and to become a candidate for testing in ECLP as well. The Japan Coal Liquefaction Development Co. has made such a contribution. A $\$5 \times 10^6$ contribution (obtained so far from Phillips, Atlantic Richfield, and Ruhrkohle) earns limited voting rights, access to certain data, and the right to attend all meetings.

B. Process Considerations

The question why German coal liquefaction people persist in using much higher pressures than are used for EDS was discussed. Use of a good donor solvent reduces the effect of increasing H_2 pressure. German coals are apparently harder to liquefy than U.S. coals.

Brief mention was made of the utility of a proprietary "solvent quality index" in processing coal liquids.

CLPP is now operating in a series of tests with recycle of some of the bottoms from the vacuum tower. This procedure returns some heavy liquefied matter to the reactor along with some mineral matter and unreacted carbonaceous residue. Typically, the vacuum bottoms might contain about 25% mineral matter. CLPP was running on July 13 on Monterey Illinois No. 6 coal and will be shifted to another coal in about a week. The next coal will be Pittsburgh Seam coal from the Ireland mine.

There was no opportunity for an extended discussion of the effects seen in the EDS process performance as the result of the recycle of heavy matter. Apparently, R. Bauman (Exxon) believes that the role of the recycle is simply that of shortening the residence time in the liquefaction reactor for the "easy" parts of the coal to liquefy; these yield lighter products that are not recycled except as donor solvent species after hydrotreating. At the same time, the residence time is lengthened for the "hard" parts of the coal, i.e., for the heavy material being recycled. Bauman stated that Exxon has experimented with additions of mineral matter and found little effect if a "good" solvent is used. However, these additions were seen to have beneficial effects when a "poor" solvent was used.**

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*The Germans frequently added sodium sulfide to their liquefaction systems (primarily to control chlorine, but incidentally also adding H_2S). Some investigators believe that the addition of the sulfide improved liquefaction performance.

**Results presented at the EPRI Coal Liquefaction Contractors meeting in Palo Alto in early May tend to indicate that some unidentified chemical species in the recycled heavy solubilized matter may serve as excellent "hydrogen shuttles", transferring hydrogen from the gas phase onto coaly matter. Workers on the SRC II Process believe that the buildup of mineral matter in the liquefaction reactor can produce important catalytic effects; this view may not be inconsistent with the Exxon experience reported by Bauman, if Exxon's solvent is of better "quality". A personal communication from Douglas Montgomery to suggests that one result of the buildup of mineral matter and of unreacted residue in a liquefaction reactor is to coat tiny spherules of mesophase with such matter soon after such spherules are formed. The coatings tend to prevent spherules from agglomerating into larger masses leading to harmful accumulations of solid reactor residues and also provide catalytic matter in close proximity to the mesophase to promote its hydrogenation and its "reversal" to solubilized matter.

C. Comments on Equipment

RCLU has a preheater with tubing 1/18 inch in inside diameter. Experience has been good with this preheater and with the 3/8" I.D. tubing used in the preheater of CLPP. The later consists of a coil in a fluidized-bed sand "bath". ECLP will use a fired heater, and the ECLP program includes extensive work on the preheater.*

CLPP has four reactor segments in series, each 25 feet tall and 2.62 inches in inside diameter. The flow is upward in each segment. ECLP will also have four reactors in a similar configuration, each 60 feet tall and 2 feet in inside diameter. These are open reactors without internals. The velocity of flow is about 3 times greater in ECLP than in CLPP; it is about 3 to 4 feet per minute in ECLP.

Exxon has conducted extensive tests of full-scale cold models of the ECLP reactor. Bubble sizes of 2 inches and smaller are contemplated; workers at Exxon believe that the presence of solids in the reactor limits the growth of gas bubbles. Backmixing caused by bubble tracks and eddies is not believed to be a problem.**

Letdown valves have shown service life times of a few days in early CLPP operations. The valves now last for 15 to 30 days. A special tungsten carbide with a minimum of binder is used in these valves: Kennemetal 703 with 2% Co-Cr matrix. EPRI is currently sponsoring development work in this area.

ECLP will have block valves so that letdown valves can be changed during the run.

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*A long-range possibility is a coal-burning fluidized-bed preheater. The bed would be at atmospheric pressure, and preheater tubes would be housed within the combustion bed.

**It has been noted by several FERWG members that the hydrogen gas flows in the EDS and H-Coal processes are on the order of one third of the hydrogen flow in SRC II, which may lead to easier scale-up for these processes. The larger hydrogen flow in the SRC II reactor, achieved through a larger recycle of gas, is probably necessary to provide a larger hydrogen gas inventory in this reactor.

Vacuum bottoms from the vacuum tower have a residence time at 700° F of about 20 minutes in CLPP. It was noted that some of the viscosity problems experienced in handling vacuum bottoms may possibly be associated with long residence times although Exxon experience, based on tests in which the material was held at 700° F for much longer times, did not lead to problems.

The holdup time of vacuum bottoms at 700° F in ECLP will be 30 to 60 seconds. The bottom of the vacuum tower in ECLP is at an elevation of about 50 feet, and the pump can be situated close to the bottom of the tower. The top of the tower is 130 feet from the ground. The elevation of the tower provides a barometric leg to maintain vacuum and flow in the event of a pump failure.

Vacuum bottoms pumps in CLPP had service lives of a few hours at first. Current experience is a life of about 1 month. The pumps are off-the-shelf models modified by Exxon. Longer pump life is expected in ECLP because it will be possible to purge bearings and seals. Purges cannot be afforded at the scale of CLPP because of the dilution of process material; purges would affect material balances adversely.

Corrosion during fractionation, of the type reported to FERWG at Fort Lewis, has not occurred, presumably because "acids" do not survive the EDS hydrogenation process.

Exxon experience confirms the advice heard from the SRC II process crews: do not allow coal liquids to sit at high temperatures; keep them moving!

D. Facilities Visited

Ansell served as tour leader for the FERWG group to the RCLU and CLPP facilities. Noteworthy is a nitrogen-blanketed set-up for processing coal samples to produce comminuted material at the sizes needed by Exxon's various coal-conversion pilot plants.*

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*One of the FERWG members commented on discussions with Whitehurst and others concerning the difficulties encountered when a coal researcher tries to obtain good coal samples that have been protected from oxidation. It would be useful if arrangements could be made for Exxon to distribute samples routinely to a number of qualified "subscribers" whenever a new coal is processed in its coal-preparation system.

Furlong reviewed some of Exxon's gasification research. Exxon uses tiny glass U-tubes for fluidized-bed tests, as well as tiny fixed beds. Exxon's one ton per day pilot plant for catalytic gasification is now in shakedown: the reactor is 10 inches in inside diameter and 80 to 100 feet tall.

Souther showed FERWG members the ECLP facilities. Major equipment is in place, whereas much of the piping still needs to be installed. Coal receiving and preparation occupies a large area but represents only about 10% of the $\$110 \times 10^6$ cost for ECLP. As is usually the case for coal-liquefaction plants, the liquefaction reactors themselves are very small in comparison with the remainder of the equipment. The plant was engineered from a model, and a view of the model provides an excellent impression of the plant to be.

E. The Exxon Flexicoking* Pilot Unit

A Flexicoking pilot unit for 2 barrels per day has been run on coal liquefaction bottoms. ECLP will not have a Flexicoking unit. Plans have been made, but were not discussed on July 13, for larger tests of the Flexicoking of liquefaction bottoms. The "ash" purge from a Flexicoking unit is expected to contain about 5% of the carbon in the liquefaction bottoms fed to the Flexicoking unit. Disposal of this "ash" will require procedures related to those used by boiler operators burning large quantities of coal.

Flexicoking is not viewed as a mandatory component of the EDS project.

F. Public Health Issues

Since coal-derived gasoline components have comparable cut points to those of petroleum-derived gasolines, the concentrations of polynuclear aromatics (PNAs) are also expected to be similar and will thus not introduce new environmental hazards. Heavier fuel oils made from coal are, however, higher

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* Flexicoking is an Exxon service mark.

in PNAs than oils made from petroleum; therefore, appropriate testing and perhaps special precautions must be considered, at least for the heavier fuel oils made from coal liquids. The development of proper disposal systems for the "irrevocable" bottoms requires study; perhaps permanent storage in clay-enclosed areas will be acceptable.

G. Research Recommendations

The principal research recommendations are summarized below:

1. Coal-characterization procedures must be developed that define the suitability of coals for liquefaction by a specified conversion process.*

2. Work should be done on the hydrodynamic configurations used in the liquefaction unit, which may well show recirculation and bubbling at full commercial scale. Fluid flows have been studied in hot, three-phase systems; the observed bubble-size limits were about 2 inches.

3. Homogeneous catalytic processes in coal liquefaction should be studied. In this connection, a deeper understanding must be acquired of the chemical processes that occur during coal liquefaction when a recycle is used of a heavy, solubilized substance containing mineral matter (e.g., a vacuum tower bottoms).

4. An urgent practical problem deals with the development of materials and/or designs for better valves, pumps and compressors for use with coal-liquid or residue-liquid slurries. The proper design and construction of long-lived letdown valves has appeared as an especially troublesome problem. Such materials as LTα (developed for heat shields on reentry vehicles), resilient polymers or rubbers should be screened systematically to assess their potential utility in withstanding erosion and corrosion during liquefaction.

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*A discrepancy was observed between the performance of Burning Star and Monterey Illinois No. 6 coals. Furlong expressed the view that Exxon's work with Burning Star may not have been sufficiently extensive to define the best processing conditions for the primary coal liquefaction step. Nevertheless, the striking difference in behavior observed for these two coals is a paradox needing study.

5. There is an urgent need to develop a number of instruments for measurements of important process features and controls. These instruments are the same as those identified by workers at the SRC II facility in Fort Lewis. The following important and specialized measurements are not currently being performed successfully or the needed techniques are not available:

(a) on-line characterizations of slurry concentrations, especially with small density differences between coals and slurries;

(b) flow measurements for slurries;

(c) slurry levels and heterogeneous matter distributions in flow channels;

(d) accurate measurements to define water-oil interfaces, which are now estimated from density differences;

(e) convenient techniques to define the oxidation states of coals and coal liquids, which are probably not adequately defined by sulfate levels and may change drastically with very small oxygen uptakes;

(f) development of on-line viscometers to characterize the flows of the non-Newtonian fluids arising in coal-liquid handling;

(g) design of accurate sensors for line openings and closing.

6. Handling and viscosity estimations for vacuum bottoms represent special problems because of their non-Newtonian rheological properties. What are allowable hold-up times for the bottoms? In CLPP, vacuum-tower separation was accomplished in minutes at $\sim 700^{\circ}F$ and 25 mm Hg. The "repolymerization" processes which occur are complex, poorly understood, and justify careful chemical research to elucidate the nature of the phenomena.

7. Identification of important problems in scale-up (e.g., proper design of the flushing system or predictions of pump failure), if any. Slurry pumping will become easier for larger units. Flow studies have involved pressure measurements on heptane-nitrogen-coal mixtures (heptane was chosen because it has nearly the same density and viscosity as the liquefaction solvent). Vortex flows have not been observed in the prototype studies whereas solids precipitation ($CaCO_3$ forming on the walls) is known to occur. Scale-up to commercial plant

size is based on use of the same chemical conversion times that have been found to be acceptable in pilot-plant studies.

8. Are there special problems associated with solvent fractionation and hydrogenation, as was suggested by workers at the SRC II facility? Although the evidence is inconclusive, the Fort Lewis experience may be the result of selection of a "special, highly corrosive cut." In the Exxon experience, fractionation and hydrogenation were directly adapted from petroleum practice.

9. What types of product specifications (e.g., contents of sulfur, nitrogen, etc.) must be met by coal-derived liquids in order to meet current and anticipated future environmental standards?

10. Effective procedures for separating ash from coal liquids should be developed.

H. Coal-Characterization Research

Richard Neavel described a long overdue program "to bring coal science into modern times." This program encompasses several stages: (a) the careful collection and preservation of a large (66) number of U.S. coals that span the usual rank classification; (b) the measurement of a large (unspecified and some proprietary) number of physical and chemical properties of vitrinite-rich samples; (c) rating the performance of these coals in a variety of processes; (d) correlating the measured chemical and physical properties with performance in coal processing. The output is thus a matrix of coal "properties" that will predict performance in liquefaction, pyrolysis, gasification, combustion, or other coal-conversion application. There are no restrictions concerning the "properties" that are not yet recognized to be important. In particular, Neavel concurs with the value of John Larsen's suggestion that the level of some obscure chemical functionality in a coal, ordinarily present in only small amounts, may turn out to be crucial in determining behavior in an application such as coal liquefaction.

An obvious problem in the development of a useful matrix is the failure to measure an important property. An example of this type arose in the discussions which showed that pore-size measurements (which are known to be important in defining combustion behavior) are not currently included in the properties table. Omissions of this type will probably be corrected through the participation of a significant segment of the coal-science community.

A more significant problem is a methodological one. What would a successful outcome of this program be? One outcome stressed by Neavel is the matching of coal to process. It is known in a general way what coals do well in what processes but in detail there are troublesome problems. For example, the relative merits of Monterey and Burning Star (both Illinois No. 6 coals) in the SRC II and EDS processes are probably reversed. Will this type of behavior be predicted with the proposed correlation matrix? If the answer is yes, then a great deal will have been achieved. It is, however, doubtful that the coal-characterization matrix will eliminate the need for fine tuning of coals in process applications.*

An important goal of coal research is the development of leads to new chemistry for processing coal. Speculations and science on the molecular level will be required to create new processes. The connection of the correlation matrix with molecular structure remains as a challenging problem. In a sense, the proposed coal-characterization program is an effort to augment quantitative understanding (which will require decades of detailed work) by well defined empirical tests and empirical knowledge.

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*This type of problem is being addressed by focusing research on a close examination and comparison of "coal pairs", one of which is known to perform well in coal liquefaction and one of which is known to be unsatisfactory; Burning Star and Monterey coals provide an example of such a pair. Similar pairs (or trios or quartets) can be identified among other Pittsburgh Seam coals.

AC-3. REPORT ON FERWG SITE VISIT TO HYDROCARBON RESEARCH, INC., LAWRENCEVILLE, NEW JERSEY

(AUGUST 16, 1979)

The following FERWG members participated in the site visit to Hydrocarbon Research, Inc.: Clardy, Furlong, Leder, Penner, Ross, Sieg, and Squires. The group was joined by Irving Wender of DOE in all of the discussions; he provided valuable comments and advice.

AC-3.1 The H-Coal Process Developed by Hydrocarbon Research, Inc. (HRI), a Subsidiary of Dynalectron Corp.

The following HRI personnel participated in the discussions:

Dr. P. H. Kydd, Vice President-Technology;
Mr. A. G. Comolli, Director of the Liquefaction Program;
Mr. M. Merdinger, Manager of the Trenton PDU;
Mr. E. Johnson, inventor of the ebullating bed and analyst of H-Coal process development studies and data;
Mr. M. Chervanak, Assistant Director of the HRI Lawrenceville Research Laboratory.

We are greatly indebted to these experts for technical advice and information. Visual displays used in the presentations have been freely incorporated in this trip report.

The H-Coal process was started in 1964 and has been supported by DOE, EPRI, the Ashland Oil Company, Conoco Coal Development Co., Commonwealth of Kentucky, Mobil Oil Corporation, and the Standard Oil Company of Indiana.

It is patterned after the commercial H-Oil process. Both the H-Coal and H-Oil processes employ ebullating beds (see below) and conventional hydrodesulfurization catalysts (typically, 3% CoO-15% MoO_3 on alumina extrudates). Variants in catalyst formulation have been explored to some extent. The formulation, life, and management of the catalysts are important problem areas for research.

The flows in the 3.5 ton per day process development unit (PDU) are shown schematically in Fig. AC-3.1-1. Test-runs were terminated after 30 days. The H-Coal process contains a two-stage pressure let down from 2750 to 1200 psi and then from 1200 psi to atmospheric pressure, through tungsten carbide let-down valves with service lives of 30-60 days. Improved designs and materials for the construction of let-down valves are important problem areas for research.

Yields of three barrels of oil per ton of coal were reported in the boiler fuel mode. Hydrogen consumption was 4000 SCF/barrel. The products consisted of C_1 - C_4 gases, naphtha, boiler fuel, residue, and ash. The liquids produced are about half in the fuel oil range. Distillates typically contained 0.1-0.2% and the boiler fuel less than 0.5% S. It was reported that 80-90% of the organic sulfur was removed.

Yields from PDU runs in the syncrude mode were reported as follows: C_1-C_4, 8-11%; C_4-400°, 17-19%; 400-650°, 10+%; 650-975°, 8.5+%; 400-975°, up to 29%; 975°+, 20-30%; unconverted coal, 5-7%; ash, 11%. The vacuum bottoms were about 45% solids. Hydrogen additions to liquids are 10% based on liquids or 5% based on coal. The management, supply, and cost of hydrogen for H-Coal plants are primary factors in determining product costs.

Representative process operating conditions and product properties are given as follows: system pressure, 2750 psig; H_2 partial pressure, 1800 psig; reactor temperature, 850°F (intentional temperature excursions up to 880°F were used); space rate or equivalent, 2 wet tons/hr/inventory wet ton; sulfur reduction, 80% of organic and 50% of pyritic sulfur; catalyst make-up rate, 0.3-6 lb/ton

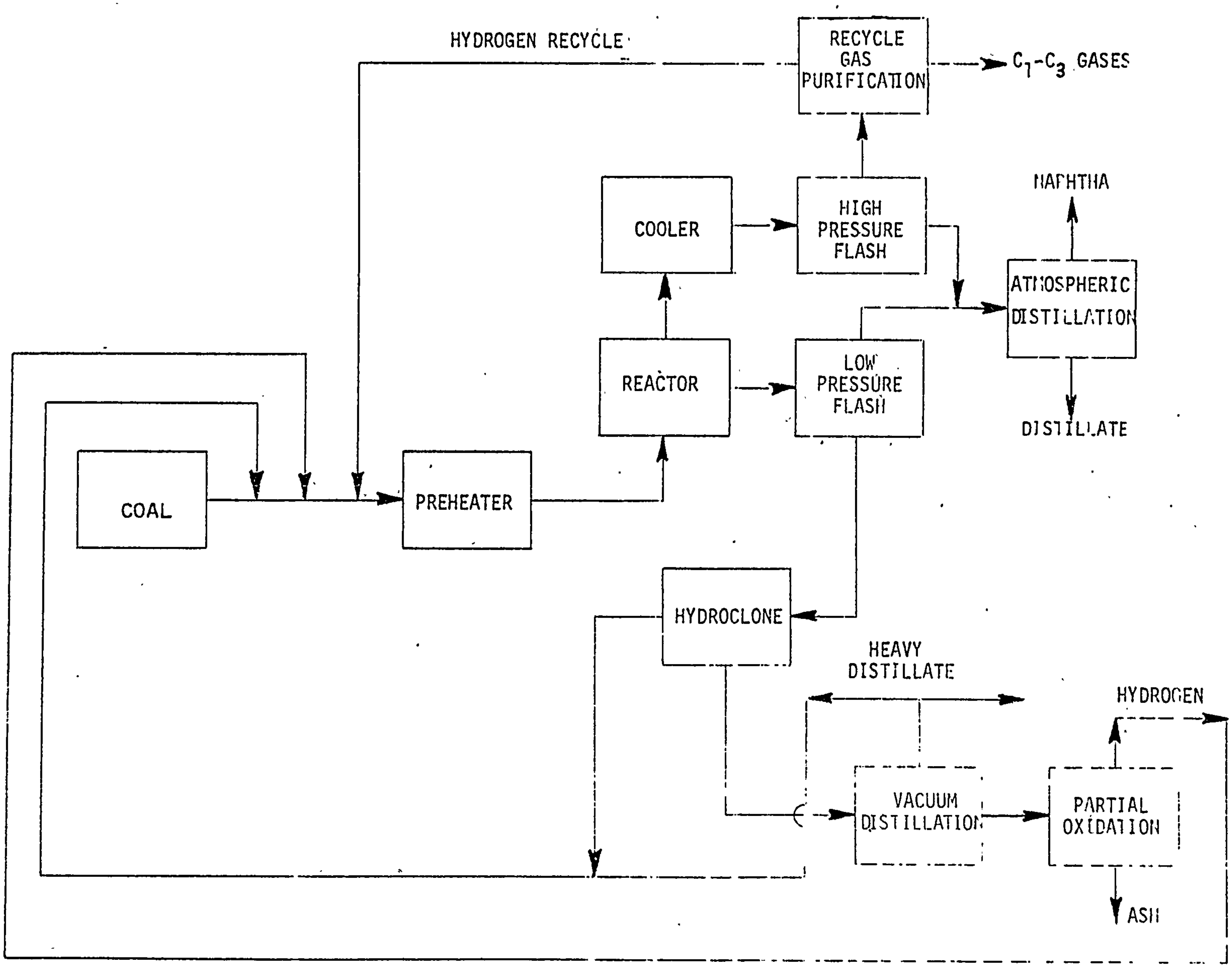


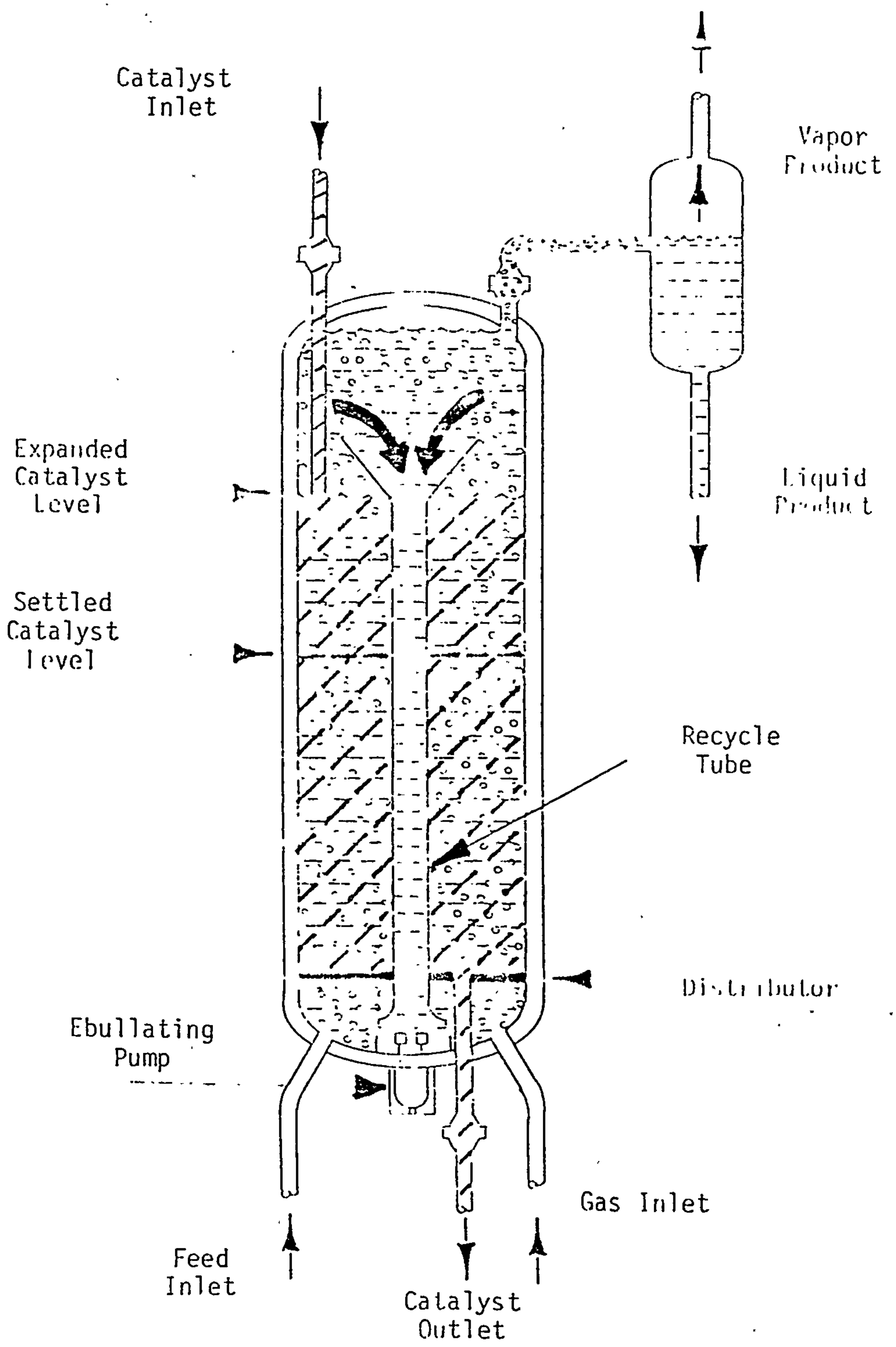
Fig. AC-3.1-1 Schematic diagram showing the PDU flows.

and typically 1 lb/ton; equilibrium catalyst activity, low relative to that of the fresh catalyst; carbon content of the equilibrium catalyst, ~ 20%; solids content of vacuum bottoms, 45-50%; hydrogen consumption, 4000 SCF/bbl when operating in the Illinois No. 6 boiler fuel mode; the PDU reactor temperature control was good; extensive external oil recycle was employed.

FERWG members generally concur with the view that the H-Coal process can be commercialized and that at least the distillate portion of the product may be further processed to transportation fuels using available petroleum-processing technology.

i. The Ebullating-Bed Reactor

The H-Coal process uses a catalytic, ebullating-bed reactor (see Fig. AC-3.1-2) for the initial coal-conversion step. In the ebullating-bed reactor, catalyst pellets are supported by rising flows of hydrogen gas and coal-solvent slurry. The rate of upward flow of the slurry is adjusted by means of a pump that is used to recycle material withdrawn from the top of the vessel and reinjects this same material into the bottom of the reactor. In a commercial-scale H-Coal reactor, the recycle pump will be housed within the bottom of the reactor structure. The recycled material flows downward toward the pump through a down-comer that is situated along the vertical, central axis of the vessel. The primary purpose of the recycle is to maintain proper ebullation of the catalyst bed. Within a well-defined liquid-flow range, efficient mixing occurs and the temperature is relatively uniform throughout the ebullated bed. At the same time, the upper catalyst bed level remains quite distinct. The H-Coal reactor represents a good approximation to the stirred reactor.



EBULLATED BED REACTOR

Fig. AC-3.1-2 Schematic diagram of the ebullated bed reactor.

A key function of the ebullating bed is distribution of the heat load across the reactor. Available operational experience indicates temperature control within ± 5° F at any given vertical position. The slurry enters the preheater at 250-300° F and is then raised to a design maximum temperature of about 850° F at the reactor exit.

The ebullated bed reactor was developed by HRI and has been used commercially in the H-Oil process. Two commercial H-Oil units have been in operation for 11 years in a refinery in Kuwait; each of these processes 27, 500 bbl of oil per day. Another H-Oil unit has been used in a Pemex refinery in Mexico for 6 years and processes 20, 000 bbl of oil per day. Thus, the development of the H-Coal process may be viewed as an adaptation of commercial H-Oil technology to coal liquefaction. In Table AC-3.1-1, we summarize the reactor dimensions that have been used or will be used in the development of commerical scale, ebullated-bed reactors.

Table AC-3.1-1 Inside diameters for ebullated-bed reactors at various scales.

Process Scale	Reactor diameter for	
	H-Oil	H-Coal
Bench scale	0.75 inch	0.75 inch
Process development unit (PDU)	8.5 inch.	6 and 8.5 inch.
Pilot plant	4.5 ft	5 ft (at Catlettsburg, Ky.)
Commercial plant	13.5 ft	13.5 ft (projected)

An advantage of the ebullated bed for processing coal, that has been observed by HRI, is absence of formation of reactor deposits of the type observed, for example, by Exxon in its EDS Process. For a properly operating, ebullated bed, incipient deposits that may form on catalyst particles appear to be promptly removed from the particles by turbulent shear flow.

HRI personnel have built a full-scale cold model of the 5-ft. ebullated bed reactor for the Catlettsburg pilot plant (see Table AC-3.1-1) and have used this model to develop proper designs for internal components that will hopefully ensure successful operation of the internal slurry recycle pump. The original design of the inlet at the top of the slurry downcomer was modified to reduce the quantity of hydrogen gas that is entrained by the down-flow of the recycled slurry liquid. Workers at Amoco are conducting analytical studies of cold-flow modeling on the ebullated bed under a DOE contract at the Naperville research laboratories; this work is closely coordinated with HRI activities.

ii. The Slurry Heater

Workers at HRI, as elsewhere, consider the design of a slurry heater to be a critical problem in scale-up. Alternate designs have been developed for Catlettsburg, with the hope that all will work and with the expectation that the multiplicity of designs will provide safety and flexibility in the operation. The H-Coal process uses a preheater temperature well below the reactor temperature. The SRC II and EDS processes require preheating of the slurry stream to temperatures about 100°F higher than are used in the H-Coal process.

iii. The Catalyst

It appears that practically all of the H-Coal work to date has been carried out with a conventional Co/Mo catalyst. Recently, some work has been initiated with Ni/Mo catalysts and molybdenum-supported catalysts. The available results indicate superior performance for these catalysts. Bench-scale development tests have been made on a number of catalysts. These data suggest that significant improvements may be expected in catalyst performance in the H-Coal process, both with respect to selectivity of the desired products and especially with respect to catalyst life.

Catalyst make-up rates fall in the range 0.3 to 6 lb. of catalyst per ton of coal processed, depending on the coal used and the desired product mix. A limited amount of work has been done on regeneration of catalyst activity: 50-60% recovery of catalyst activity was achieved by combustion and, with new materials, there is the promise of doing better.

HRI personnel made a strong recommendation that catalyst research is an area that will yield significant pay-off.

The results were described on two tests (one on Illinois No. 6 coal and one on Wyodak coal) that were intended to illustrate H-Coal performance with an equilibrium catalyst, i.e. with a level of catalytic activity that remains at a time-independent, steady value when catalyst additions are made on a regular schedule from the beginning of the run. Both tests had durations of only 30 days. The short duration of these runs was set by DOE contract monitors in the light of budgetary restraints. All of the participants at the HRI discussions concurred with the view that considerably longer runs would be helpful to prove that the data reflect the performance of an equilibrium catalyst. Since theoretical calculations show that the equilibrium catalyst level will be achieved only shortly before the expiration of 30 days, catalyst studies should be extended to durations of 60-90 days.

iv. Studies of Upsets

The ebullating bed liquid recycle pump has been shut-off abruptly, without the immediate occurrence of serious consequences. In fact, the bed remained operational for at least 12 minutes, thus allowing time for appropriate operator response.

The ebullated bed has been subjected, on a number of occasions, to abrupt cut-off of flows. Each time the bed recovered easily.

On the other hand, sudden removal of the preheater led to serious consequences because a corresponding, sudden increase occurred in viscosity. This result was not anticipated and its discovery amply justifies a program on the study of upsets. The operating answer to a sudden loss of the preheater is rapid introduction of light oil into the system in order to maintain low slurry viscosity.

Coke formation was not observed after a brief excursion in temperature to ~ 900° F.

v. Letdown Valves

The letdown valves are viewed as critical components by workers at HRI and elsewhere. On the whole, HRI personnel appear to be content with the letdown valves now in use in their bench and PDU units. Sparing is provided so that valves can be changed without interrupting a run. Tungsten carbide valves usually last from 30 to 60 days, which is viewed as satisfactory in process development operations by HRI workers.

Recently, two stages of letdown have been installed in the PDU: the first stage covers the range from about 2750 psig to 1200 psig and the second leads to atmospheric pressure. HRI personnel believe that the performance of this two-stage letdown system represents an important operational improvement.

vi. Carbon Dioxide vs. Water as a Product from Low-Rank Coals

The fact was noted that German researchers in coal liquefaction find oxygen from brown coal to be released primarily as carbon dioxide, whereas HRI H-Coal data for Wyodak subbituminous coal show that the oxygen is mainly in the output water. Johanson stated that the initial oxygen-containing product from Wyodak coal is indeed carbon dioxide, but that this is shifted to water under the conditions of the H-Coal reactor.

vii. Effect of Deep Cleaning

HRI workers showed results indicating better performance with deep-cleaned coal (at 6.4% mineral matter) than with run-of-mine coal (at 18% mineral matter). The inference is that the presence of "ash" in the H-Coal reactor is detrimental.

However, this conclusion cannot be drawn with certainty without careful tests, in which the level of mineral matter of relatively constant composition is systematically varied. The run-of-mine coal in the test probably contained a great deal of slate.

iix. Instrumentation of Ebullating Beds

A temperature reading is taken every foot of height in the bed. There is a density monitoring device on liquid recycle to the ebullating pump; this measurement is used to show if hydrogen is entrained in the liquid. A density reading is also taken in the ebullating bed itself.

ix. Progress in the Construction at Catlettsburg

The construction at Catlettsburg has been delayed. Reorganization of the construction-management team has been an essential step in the redirection of this activity. FERWG members have not performed an independent evaluation of past and current activities relating to construction delays, program management and overview, cost overruns, and related potential problem areas.

x. H-Coal Costing

An independent and definitive cost evaluation for H-Coal has not been made by FERWG members.

Replacement of cobalt-molybdenum oxide catalyst at a rate of 0.3 lb per ton of coal processed was estimated by HRI workers to imply a catalyst-replacement cost of about $0.30 per bbl of oil produced. At the highest quoted catalyst replacement rate of 6.0 lb/per ton of coal processed, catalyst costs with regeneration are substantially less than $6.00 per bbl of oil produced.

Currently preferred catalyst choices are Amocat Co/Mo and Ni/Mo; the Ni/Mo has performed well in nitrogen reduction while the Co/Mo appears to be the preferred catalyst for lowering viscosity and reducing resin and distillate yields. Neither of these catalysts is totally satisfactory for heteroatom removal and for maintenance of hydrogenation activity.

We were told that the cost of a 70,000 bbl/day plant would be about 1 billion dollars (1978), and that this information was available through the DOE. A report was prepared by the Fluor Corporation for ERDA in March 1976, which has been widely circulated but never officially issued. R. Fischer (DOE) has stated that financial parts of the report are not proprietary but the report was not released. At any rate, figures comparable to those cited to us were available in 1976. If the cost estimates refer to 1975 instead of 1978 financial data, they are out of date. In 1975, projected prices of oil were around $17-18, based on optimistic assumptions about the process and about the cost of capital. It is likely, based on current costs of capital, coal, construction, etc., that oil from H-Coal will cost $30-45/bbl.

AC-3.2 Research Recommendations

The near-term and long-term directions of supporting research that are required for efficient implementation of commercial H-Coal processes are clearly defined by the remarks contained in the preceding sections. These recommendations are consistent with the view that the ebullating-bed concept used in the H-Coal process represents an attractive alternative for contacting coal, recycled oil, hydrogen, and catalyst, with low preheat requirements. Our research recommendations are also consistent with the views elaborated in joint discussions involving HRI project personnel.

i. Catalyst Development

Improved results in catalyst performance have been associated with improving the bimodal distribution in the size of pores within the catalyst pellets. Experimental findings clearly indicate that research at much greater depth might pay off.

Optimization of the product slate is difficult, because the economic rules that will be in effect 5, 10, or 15 years from now are simply not known. For this reason, catalyst work should be directed toward finding a number of formulations that lead to slates of preferred products, so that ultimately there will be greater flexibility in the choice of catalysts to allow optimization of products under a variety of given sets of economic rules. Thus, ranges of catalysts are needed with improved hydrogen selectivity.

Improved catalysts are needed with better stability against coking and deactivation. Catalysts are required that function efficiently at significantly lower pressures than are now used in the H-Coal process. The many types of process alternatives and controls, which we seek through optimal catalyst selection, clearly justify long-term, fundamental studies in this field. These studies should not deal exclusively with near-term process improvements. Research areas should include mechanisms of catalyst deactivation and activation, as well as kinetic control through catalysts.

ii. Research to Reduce Hydrogen Consumption

The cost of hydrogen is an important cost component in liquefaction and research to reduce hydrogen consumption is clearly justified. The oxygen contents of the processed coals require hydrogen during liquefaction. Therefore, pretreatment of the coal to remove oxygen may become an attractive step. Ideas for pretreating coal to reduce oxygen content should be explored.

In a broader context, we conclude that coal chemistry directed at understanding mechanisms and rates of bond rupture clearly bears on the development of efficient processes for oxygen removal during coal pretreatment, on hydrogen requirements in the H-Coal process, and hence on a dominant cost item in H-Coal technology.

iii. Process Optimization: Instrumentation, Analysis, Process Control, and Modeling

Greatly improved instrumentation is needed for efficient process control and for proper analysis of operating conditions. Tight material balances are an essential component of efficient process technologies and clearly require careful and adequate instrumentation.

The current choice of operating conditions is probably non-optimal and may be responsible, in part, for rapid catalyst deactivation and high hydrogen consumption through cracking and ring-opening reactions. The search for improved operating conditions will require very close coupling between carefully instrumentated operating units and modeling of hot-flow reactors, using interactive and iterative techniques in a search for optimization of process variables.

The goal of optimization of process variables leads to the requirement of hot-flow modeling research with adequate consideration of the fluid mechanics in multiphase, reacting, partially or fully stirred, turbulent flow reactors. The numerical aspects of this work will not be meaningful unless they are tied to a measurement program on small- or large-scale units using proper instrumentation to measure those process variables which are shown to be of dominant importance in the modeling studies.

iv. Materials Problems

Pressure let-down valves have been found to have short service lives in the H-Coal, as well as in other liquefaction processes. Short service lives are the result of erosion and of chemical attacks under the operating conditions that are used in practice. The best available let-down valves are Hammel-Dahl angle valves with tungsten carbide seats. These have been found to have service lives of 1-2 months in the PDU.

Material problems with let-down valves will be encountered whenever slurries of coal products are handled in flow systems, especially at elevated temperatures. We recommend a broadly-based program that is designed to define the relative roles of erosion and of chemical attack in these valves, coupled with materials-development research designed to handle inlet and outlet slurries for much longer periods of time than is now possible. In this connection, a careful look at the performance of reentry heat-shield materials (e.g., LTα) may be justified.

In the H-Coal process, the problem of hydroclone erosion appears to have been solved by the use of ceramic hydroclones and proper flow control. This observation indicates the importance of defining the relative roles played by physical and by chemical processes during erosion.

v. Instrumentation for Process Operation

As for other coal-liquefaction processes, instrumentation development to measure slurry flows is needed.

vi. Studies on Reactant Rheology

The extreme temperature sensitivity of viscosity observed during preheat failure in the PDU has been noted. The operational solution of rapid oil injection may well be acceptable. Systematic studies on process upsets should be continued. At the same time, a broadly based research program should be defined in order to minimize the number of surprises that may be encountered during designed and unintentional process upsets.

In connection with reports on earlier site visits, we noted both the complexity and lack of knowledge concerning the rheological properties of coal-slurry flows, which behave as non-Newtonian fluids. It is worthwhile to extend these studies to examination of rheological properties of the reactant, multi-phase mixtures under the conditions encountered in ebullating beds. In particular, viscosity measurements as functions of temperature and pressure should be made for wide ranges of hydrogen flows, a variety of catalysts, and a representative range of coal types.

AC-4. SITE VISIT (OCTOBER 21, 1979) TO THE CONOCO PROCESS DEVELOPMENT UNIT (PDU) FOR COAL LIQUEFACTION USING $ZnCl_2$

The following FERWG members participated in the site visit to the Conoco Coal Development Company (CCDC) at Library, Pa.: Clardy, Furlong, Penner, Reichl, Ross, Sieg, and Squires. The group was joined by I. Wender (Fossil Energy) and R. H. Kropschot (Office of Basic Energy Sciences) of DOE and by R. F. Bauman (Exxon, Baytown). Personnel from CCDC included J. D. Sudbury, R. T. Struck, M. Pell, C. W. Zielke, and others.

A. Process Description

Figures AC-4-1a and AC-4-1b show simplified flow diagrams for the $ZnCl_2$ PDU. Table AC-4-1 contains a summary of the development process; Table AC-4-2 shows the project goals.

B. FERWG Observations on the Conoco Program

The difficulties that have been encountered with the $ZnCl_2$ process are readily traced to the following features of the process:

1. It is an innovative process using methods and techniques that are different from those applied in other coal-liquefaction technologies. The presence of molten and gaseous $ZnCl_2$ and of its derivative compounds dominates every step of the operating procedure and demands the utilization of components that are suitable for handling materials of this type.

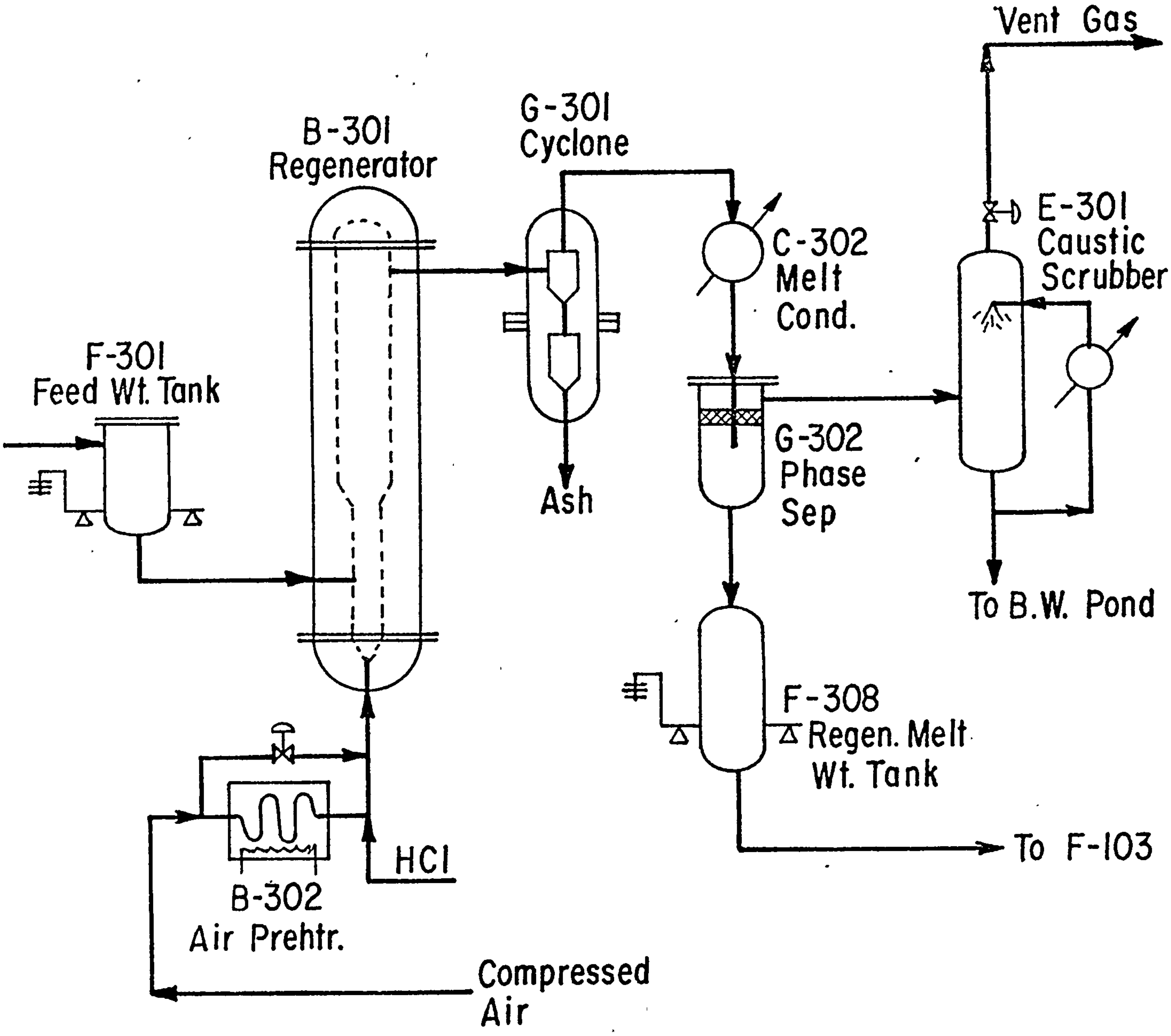


Fig. AC-4-1a Simplified flow diagram of the zinc chloride PDU.

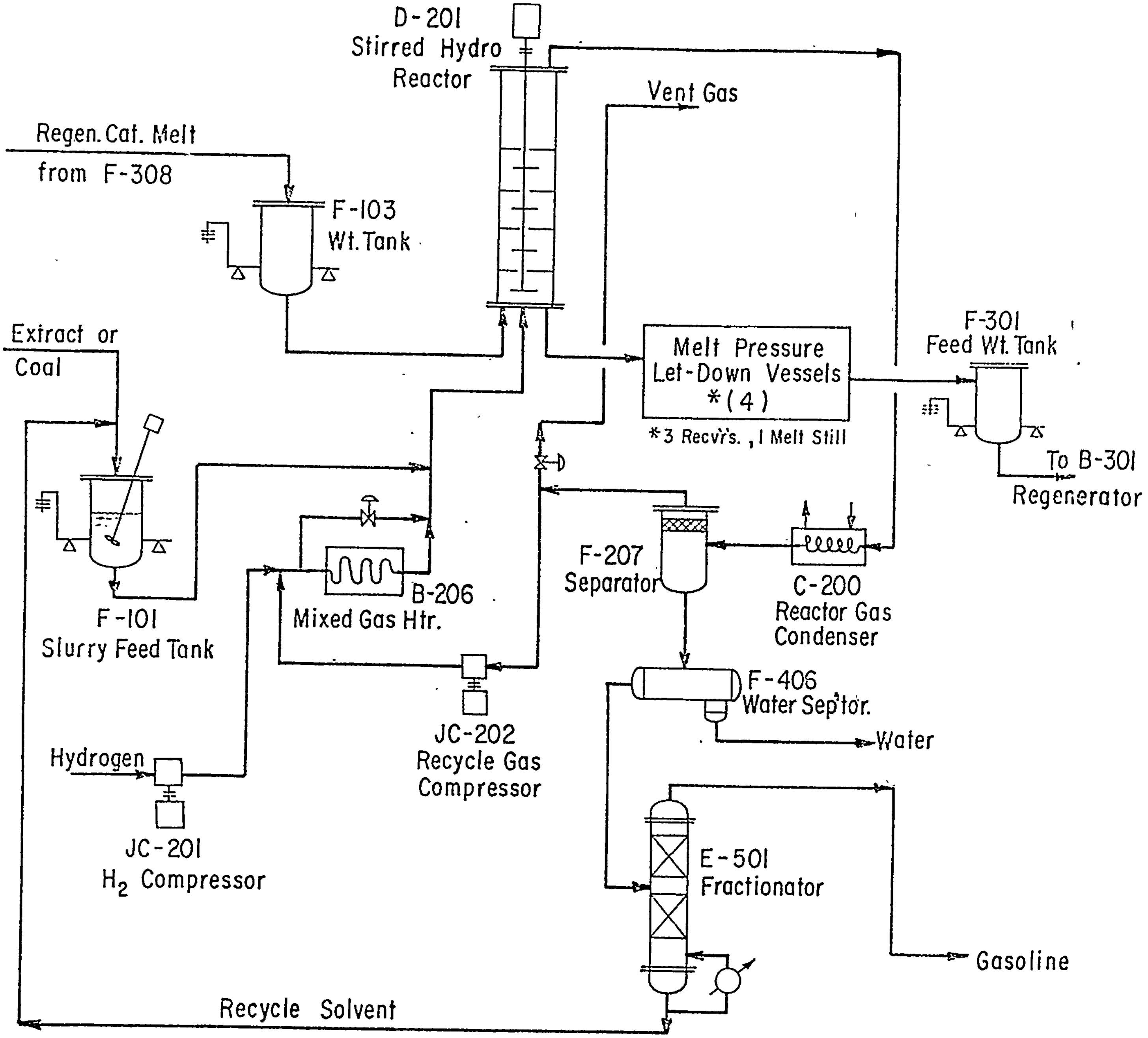


Fig. AC-4-1b Simplified flow diagram of the zinc chloride PDU.

Table AC-4-1 Highlights of the zinc chloride process development.

1963-1967 CONSOL COAL (CONOCO COAL) UNDER OCR CONTRACT (1.4 MM $)

- DEVELOPED CONCEPT OF MOLTEN ZINC HALIDE AS A CATALYST FOR LIQUEFACTION OF COAL AND COAL EXTRACTS.
- DEMONSTRATED CONTINUOUS HYDROCRACKING WITH COAL EXTRACT AND $ZnCl_2$ IN A 2 LB/HR UNIT.
- BATCH TESTS WITH COAL FEED.
- TESTED SOLVENT SEPARATION TECHNIQUES FOR CATALYST REGENERATION. DEVELOPED A UNIQUE COMBUSTION PROCESS FOR CATALYST REGENERATION.
- DEMONSTRATED CONTINUOUS CATALYST REGENERATION IN 5 LB/HR CONTINUOUS COMBUSTION UNIT.

1968-1974 CONOCO COAL CONTINUED WORK ON OWN (0.4 MM $)

- SHOWED PROCESS APPLICABLE TO ALL TYPES OF COAL.
- CONTINUOUSLY REGENERATED SIMULATED SPENT CATALYST FROM COAL LIQUEFACTION AND SHOWED THAT HYDROLYSIS OF $ZnCl_2$ COULD BE CONTROLLED BY ADDING HCl TO THE FEED AIR.

1975-1979 DoE CONTRACT WITH CONOCO AND SHELL

- CONTRACT PERIOD: JANUARY 1975 TO MAY 31, 1980
- TOTAL FUNDING: $11,410,413

DoE	90%
CONOCO	5%
SHELL	5%

Table AC-4-2 Goals of the zinc chloride project for development of the molten zinc chloride process for the production of gasoline from coal.

I. IN CONTINUOUS BENCH UNITS

- Demonstrate feeding of coal to the molten bed
- Demonstrate high yields of distillate fuels
- Define reaction kinetics and develop reaction model
- In catalyst regeneration, demonstrate high recovery of $ZnCl_2$ with high removal of N,O,S, and coal ash
- Test materials of construction

II. DESIGN, CONSTRUCT, AND OPERATE A 100 LB/H PDU TO

- Demonstrate operability and equipment reliability with recycle of the catalyst and hydrocarbons (An integrated run of at least 7 days)
- Demonstrate high recovery of $ZnCl_2$ in regeneration
- Define suitable materials of construction
- Test alternate reactor designs
- Test methods of achieving low-C spent melts
- Test novel methods for absorbing HCl from gases
- Define all effluent streams from environmental viewpoint

III. CONDUCT ECONOMIC STUDIES TO GUIDE DEVELOPMENT AND EVALUATE COMMERCIAL POTENTIAL

IV. SUPPORTING LABORATORY AND PATENT ACTIVITIES AS NEEDED

The products formed have very desirable properties (they correspond to high yields of unleaded, high-octane gasoline and smaller amounts of LNG or LPG) and are usable with little further processing. The $ZnCl_2$- procedure may well be applicable to a wide variety of coals. Heteroatoms are preferentially removed. Cost projections by Conoco (~ $0.76 per gallon of gasoline) are favorable but may also be optimistic.

2. The difficulties of the process were apparently not fully appreciated, and the PDU was underfunded and/or underdesigned.

3. The lack of adequate financial support, in turn, has led to deficiencies in each of the following categories: (i) process design with reasonable redundancies; (ii) component developments suitable for handling the fluids at all stages of the process; (iii) adequate research to address fundamental problems, especially in the materials and chemical sciences; (iv) development and installation of adequate instrumentation and diagnostic procedures for process monitoring and control.

C. Observations on Process Experience

Bench-scale runs at 2-5 lbs/hr have produced a high quality naphtha, comparable to unleaded gasoline, in reasonable yields.

The process is based on the fact that zinc chloride will "catalyze" the hydrocracking of coal to single ring aromatics. The used melt, with zinc chloride partly converted to zinc oxide and sulfide and containing also residual hydrocarbons, can be treated with air and HCl at high temperatures (e.g., 1600-1900° F) to allow distillation of pure zinc chloride from the regenerator.