MASTER

A DATA REVIEW AND OPERATING EXPERIENCE

EVALUATION OF THE SRC II PROCESS

by

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ABSTRACT

Scientific Design Company (SD) has reviewed the Ft. Lewis pilot plant operation and data gathered in the SRC II Solvent Refined Coal (liquid fuel production) program, which tested Kentucky, Illinois, and Pittsburgh Seam coals. The aim of this work is to further the understanding of the process, help to evaluate its potential and assist the Department of Energy (DOE) in future development decisions. Ft. Lewis information was assembled and organized to aid review and interpretation. SD carried out the tasks of: description of the SRC II operation, assessment of the expected data uncertainty from an evaluation of equipment and procedures, assessment of data reliability and usefulness for design of larger facilities, and investigation of the apparent discrepancies in Heavy Distillate make and ash balance. Process flowsheets, data tables and other exhibits are presented.

The plant was not specifically designed for obtaining material balance information and its ability to generate reliable data was limited because it was revamped from SRC I (solid fuel production) mode and for other reasons detailed in this report.

The major conclusions that SD has reached are:

1. Extensive and varied operating experience gained in the program has demonstrated the basic feasibility and stability of the

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process. A high on-stream factor was achieved throughout the program and the reaction section showed no tendency to plug or coke.

2. The total data base, including all of the data which Gulf obtained at its Harmarville laboratory in conjunction with published information obtained at Ft. Lewis, could be used to design a commercial plant with reasonable confidence, within the range of variables explored.
3. The equipment, procedures and programs of Ft. Lewis to date have not been appropriate to the gathering of complete process development data. This multi-functional plant is better suited to its other tasks of demonstrating the basic operability of the process, testing components, and accumulating large product and residue samples for combustion and gasification tests.
4. The experience and data obtained at Ft. Lewis should be used to develop a continuing pilot plant program to improve the process further.
5. Most of the Heavy Distillate loss is not real but the result of using an empirical yield prediction model outside the range of its applicability; however, real loss factors have been identified and are susceptible to a significant reduction using straightforward loss prevention methods and process design modifications.
6. The apparent "ash disappearance" problem has not been satisfactorily resolved at present. Inaccurate weighing of the Vacuum Bottoms residue and variation of the coal feed rate are likely contributing factors.

Gulf's Harmarville (P-99) pilot plant has completed an augmentative SRC II program with Kentucky coal, a summary report of which was recently made available to DOE. Gulf has not yet made public other important data obtained from P-99. Data acquired at Ft. Lewis alone were insufficient and unacceptable to reliably correlate the reaction yield and kinetics, which would be required for a commercial process design and optimization. Only some trends could be perceived from the best data to characterize the general effect of the particular parameters investigated at Ft. Lewis. It should be realized, however, that industrial process commercializations have often proceeded on less data and experience than has been gained at Ft. Lewis alone.

This report points out the types of necessary process development data, obtainable in pilot plants, which would be needed for a commercial plant design. It is essential that all future data analysis and pilot plant operation in the SRC II development effort should be directed toward what is needed for a commercial plant design.

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I. INTRODUCTION

A. Task Definition

The DOE gave SD the task of analyzing the operation and data taken in the SRC II mode from the coal liquefaction pilot plant operated (under DOE sponsorship) by P & M (Gulf Oil Co.) in Ft. Lewis, Washington to determine if the data base in the public domain is sufficiently complete and reliable to be used for the design of a demonstration plant.

With one important exception, SD approached this task as it would any of the private process commercialization studies it has undertaken in the past. The major steps followed by SD were:

- familiarization and information gathering
- facilities and procedures evaluation
- data analysis

The exception involves a commercial plant design usually evolved in parallel with pilot plant programs. When SD is called upon to analyze its own pilot plant research or the research of others for potential commercialization, a proposed commercial plant design is usually available for comparison with the operation of the pilot plant and to ascertain the relevance of data gathered. Such a design is normally developed from the first exploratory research

in the laboratory and refined as the data become more extensive.

The Gulf organization has undoubtedly worked out such a commercial plant design, but this was not available to SD and constraints of time and funds did not permit SD to evolve its own commercial plant design.

B. Process Development History and Status

1. Early Work

The early work on the solvent refining of coal in solution with pressurized hydrogen was carried out by A. Pott and H. Broche in Germany in the 1920's.

In their process, the coal was extracted in a solvent (either extrinsic or process-derived) in a high temperature (780 - 806°F) and high pressure (1460-2200 psig) reactor. The crude extractor effluent was filtered to remove insolubles and the filtrate was subjected to vacuum distillation to recover solvent for hydrogenation and recycle and to recover net Solvent Refined Coal (SRC) product. The extracted SRC product was hydrogenated under pressure to yield lighter products. Hydrogenated product was separated into a liquid (fuels) stream, which could be further separated by distillation, and a sludge, which was recycled to combine with the crude extractor effluent. Distillation of the liquid product stream produced benzene, heating oil and other liquid fuels. This is a two-step process (extraction of coal first), also known as "indirect liquefaction", with separate hydrogenation of a donor solvent. Another approach is a direct, one-step hydrogenation of the raw coal in solution, to extract the coal and make some liquid products.

For a short time, during World War II, The Pott-Broche process was utilized in Germany for the production of liquid fuels. Due to difficulties in hydrogenation, the facility was eventually converted to the production of high pour point extract as raw material for carbon electrodes.

The Uhde-Pfarrman process, patented in the late 1930's, used hydrogen directly during the extraction step, thereby conducting in situ hydrogenation and reducing the donor solvent requirements. (Donor solvents are those coal extract fractions which are chemically active in donating or transferring molecular hydrogen in the coal extraction step.) The process operated at about 770°F and about 4400 psig.

2. Gulf's Involvement

During the 1950's, research and development work on further improvement of the Pott-Broche and Uhde-Pfarrman processes was carried out by Spencer Chemical Company. In 1962, the Office of Coal Research (OCR) awarded a research contract to Spencer Chemical to evaluate the technical feasibility of the SRC process. This contract was concluded in 1965 upon the successful completion of the demonstration of the process in a 50 pound per hour continuous flow process development unit. During the period of this contract, Gulf Oil Corporation acquired Spencer Chemical Company and reassigned the SRC project to the Research and Development Department of the Pittsburgh and Midway Coal Mining Company (P & M), a subsidiary of Gulf Mineral Resources Company (GMRC), which is itself a division of Gulf Oil Corporation.

3. Ft. Lewis Pilot Plant

In late 1968, OCR awarded a contract to P & M to continue research and development of the SRC process. This provided for a study of the commercial feasibility of the process through design, construction and operation of a pilot plant to process 50 tons a day of coal. The objectives of the pilot plant program were:

- to further develop the SRC process.
- to provide design data for future commercial plants.
- to provide bulk samples of Solvent Refined Coal and process by-products for market development studies.

The Stearns-Roger Corporation completed the design of the pilot plant located in Ft. Lewis, Washington, in 1969. A shortage of funds delayed the start of construction until early 1972 when a contract was awarded to Rust Engineering Co. of Birmingham, Ala. Completion and preliminary start-up took place in mid-October 1974 on the SRC I (solid fuel) process. The plant ran in SRC I mode through early 1977, except for a short period in 1975 when the SRC II (liquid products) mode was explored by simple adaptation of existing equipment. On the basis of information gained, the plant was revamped for SRC II operation with the aid of Hallanger Engineers and began a series of runs in SRC II mode from mid-May, 1977, through March, 1978. During this running period, the plant was again being revamped for testing SRC I ash separation steps (Lummus anti-solvent and EPRI filters). In March 1978 the SRC II program was suspended and in April 1978 SRC I operation was resumed.

The pilot plant is a nearly fully integrated facility including all the major steps in the conceptual SRC I and II processes, except that for the production of hydrogen (or synthesis gas) from the process residue. The hydrogen production step is under study in another DOE sponsored project. The make-up hydrogen for the dissolving section is generated in the pilot plant by steam-reforming natural gas in a packaged unit. The reactor capacities are respectively 50 and 30 tons per day of raw coal in the SRC I and SRC II modes.

The original SRC program allowed only three months operation on western Kentucky coal (No.9 Colonial), but P & M was granted several extensions through 1978 by the DOE, to test Illinois (No. 6, River King) and Pittsburgh Seam (Blacksville) coals. Major emphasis was placed on demonstrating operability (on-stream factors of over 80% were achieved) and on producing liquid fuel for combustion tests. In addition to the production effort, a series of test runs (generally lasting three days each) were conducted in an attempt to measure the effect of certain reaction parameters (primarily coal concentration and residence time) on coal conversion, yields and operability. A total of twenty-one such tests called "material balance runs" (MBR) were completed (seven with each type of coal). Section V of this report discusses the test program and results.

4. Merriam Laboratory

Bench-scale tests at P & M's Merriam, Kansas, research laboratory have supported the Ft. Lewis effort and are continuing to provide back-up data for the operation of the pilot plant. All of the data and information gathered at these two locations have been in the public domain since the inception of the program.

5. Harmarville (P-99) Pilot Plant

In a more recent development, Gulf has released to the DOE and to SD a report on some aspects of the operation with Kentucky coal (August 1976 to September 1977) of its Harmarville, Pa., 1 TPD (nominal) SRC II pilot plant. This plant and program, called P-99, was operated by Gulf and privately funded. The P-99 program was intended to cover deficiencies in the SRC II program, mainly due to Ft. Lewis' dual program of running SRC I and SRC II. Harmarville,

being a much smaller scale facility, is much more flexible and easier to revamp and repair. Nonetheless, the plant did take many months to get operating properly. There was apparently little interaction or data exchange between Harmarville and Ft. Lewis (except indirectly, through guidance offered by GMRC), until late in the Ft. Lewis program, when firm results were available from Harmarville.

Some process conditions in P-99 (such as H_2 partial pressure) can be varied over a wider range than the Ft. Lewis plant and a somewhat different approach from Ft. Lewis to material balance data collection is taken. The data obtained is expected to fill some important gaps in the available SRC II process data base. SD has received indications that in addition to yield data, the P-99 program may have produced some critically needed, but as yet unpublished, physical/chemical data for the design of SRC II process unit operations.

6. Proposed Demonstration Plant

A 6,000 TPD SRC II demonstration plant is being proposed by Gulf, to be jointly funded by Gulf and the DOE. This program has received preliminary government approval and the early engineering work is proceeding towards a project cost estimate. The design of this plant will not necessarily closely correspond to the Ft. Lewis process flowsheet and equipment. Gasification of the ash-containing residue by the Texaco process will be incorporated to provide the hydrogen requirement of the liquefaction and to produce excess syn-gas for fuel.

C. Comparison of SRC I with SRC II

The SRC II process, which aims at converting coal mainly to hydrocarbon-like liquids for use as fuel oil or other products, is an outgrowth and extension of the development of the SRC I process.

In SRC I, the main product is a solid at room temperature with much lower ash and sulfur content than the feed coal, and is usable as a boiler fuel. SRC I and II should not be considered as technologies independent of each other, but as extremes on a continuous spectrum of technical and economic possibilities. They differ from some other coal liquefaction technologies in that neither extrinsic catalysis nor separate solvent hydrogenation is used. Instead, coal is slurried in a recycled liquid and contacted with hydrogen in heating and reaction steps which break down the coal and generate solvents and hydrogenate them, creating various "donor solvent" fractions in situ. Any catalytic effects are created by the indigenous coal mineral residues.

The SRC II mode differs from SRC I in that:

- 1) The coal and high-boiling coal-derived fractions are subjected to reaction conditions of greater severity (longer residence time and higher hydrogen partial pressure), thereby producing a larger quantity of liquids, which are the major product of the SRC II process.
- 2) The mineral separation step of SRC I (by filtration, centrifugation, or anti-solvent de-ashing) is eliminated because the mineral residue can be purged as Vacuum Flash Bottoms along with the smaller amount of SRC and insoluble

organic residue which remain in this stream after a higher degree of conversion to liquids and light hydrocarbons.

- 3) Recycle slurry separated from the reactor effluent, rather than process generated distillate solvent, is used to slurry the raw coal feed. This feature:
- a. Reduces the size of the required backend solvent handling and distillation equipment,
 - b. Relieves the process from "solvent imbalance" conditions which can develop in SRC I when the process derives less solvent from the coal than is lost by side reactions and physical losses,
 - c. Provides greater capability for hydrocracking to distillate products by recycle of SRC and by increased concentration of catalytic mineral residue in the Dissolver.

D. Summary Description of SRC II

Raw coal is pulverized and mixed with a coal-derived solvent or slurry. (In SRC I, the slurry medium is a process-generated distillate with a general boiling range of 480-850°F). In SRC II, the raw coal is slurried with a recycle slurry containing partially hydrogenated and hydrocracked coal fractions and mineral residue. There is substantial evidence that coal ash catalyzes the conversion of coal to liquid products. Hydrogen, or a hydrogen-rich synthesis gas, is added to the coal-solvent slurry and passed through a fired Preheater to a Dissolver vessel. The Ft. Lewis Dissolver is operated at 850-860°F and 1900 psi and with a large excess of hydrogen. Under these conditions, over 90% of the (moisture and ash-free) coal is dissolved. The actual degree of dissolution of the coal and the distribution of products depends greatly on the "reactivity" of the particular coal feed. In addition to solution of the coal, several other major types of reactions occur. These are:

- 1) Depolymerization of the coal, necessarily accompanied by hydrogenation of the coal;
- 2) Hydrocracking of the solvent liquids to lower molecular weight hydrocarbons, ranging from light oil to methane;
- 3) Removal of part of the organic and mineral sulfur by hydrogenation to hydrogen sulfide; and

- 4) Partial removal of the molecular "hetero-atoms", oxygen and nitrogen, by hydrogenation to water and ammonia, respectively.

The product stream from the dissolution-hydrogenation step consists of excess hydrogen, light hydrocarbon gases (methane-rich), liquids boiling in the range between 100-850°F, SRC (defined as pyridine soluble higher boiling organics), unreacted coal plus IOM (pyridine insoluble organic matter) and undissolved mineral matter (ash). The gas stream and light oil are separated by flashing and stripping from the recycle slurry. The acid gases (CO_2 and H_2S) and light hydrocarbon gases are scrubbed from the gas stream and an appropriate gas bleed is taken, if necessary. The scrubbed gas is compressed and recycled with fresh hydrogen make-up to the Preheater and Dissolver reactor. The recovered light hydrocarbons can be separated cryogenically and sold or utilized within the process.

The net, unrecycled slurry is flashed under vacuum to recover heavy oil, leaving a high pour point product stream of SRC and organic and mineral residue. This residue can be used to generate the required fresh hydrogen make-up in a commercially available steam oxygen gasification and shift. Excess residue could conceivably be used to generate synthesis gas to provide fuel for the plant. Alternatively, the required hydrogen can be generated by steam reforming other by-product streams such as methane or Naphtha and then shifting the resulting synthesis gas. By-product fuels such as light hydrocarbons or Naphtha could also be burned to provide process heat and motive energy.

The mixed light oils from recycle slurry stripping are separated from water and fractionated, along with recovered heavy oil, to produce streams of Naphtha, fuel oil (the net major product, which is to be a mixture of Middle and Heavy Distillate) and heavy oil for flushing slurry pump seals. The process will generally yield weights of about 35-45% total liquid distillates, 12-20% hydrocarbon gas ($C_1 - C_4$), 25-35% SRC and 5-10% insoluble organic residue, while consuming hydrogen in quantities of 4-5% by weight of the (moisture and ash-free) coal fed. Reported yields for the Ft. Lewis SRC II operation are summarized in Table 1.

Process flowsheets for the SRC II mode of operation have been drawn up by SD and are shown in Section III of this report. These process flowsheets describe the Ft. Lewis pilot plant and thus reflect a particular embodiment of the basic process concept outlined above. It is expected that a commercial design will differ from these flowsheets in a number of important unit operations, flow patterns and ancillary facilities.

TABLE 1

SRC II PRODUCT YIELDS
Elementally Balanced
 (wt% MAF Coal)

<u>Run No.</u>	<u>C₁-C₄ GASES</u>	<u>NAPHTHA</u>	<u>FUEL OIL</u>	<u>VACUUM BOTTOMS</u>
<u>KENTUCKY COAL</u>				
77SR-1	18.3	9.1	33.4	31.4
77SR-2	18.3	10.5	37.1	26.7
77SR-3	16.4	15.4	30.2	29.3
77SR-4	15.4	6.9	30.5	29.3
77SR-5	12.7	9.1	30.1	39.7
77SR-6	14.3	13.0	18.7	46.3
77SR-7	18.4	14.2	28.2	32.7
<u>ILLINOIS COAL</u>				
77SR-8	16.7	17.2	31.9	26.3
77SR-9	15.8	17.0	30.3	28.0
77SR-10	10.9	19.4	27.9	33.3
77SR-11	12.4	8.8	32.4	37.8
78SR-19	12.9	6.7	32.9	38.2
78SR-20	13.2	5.0	36.2	36.9
78SR-21	14.4	11.2	35.0	29.8
<u>PITTSEAM COAL</u>				
77SR-12	19.0	5.9	22.2	48.1
77SR-13	18.4	3.7	30.1	42.2
77SR-14	18.3	11.2	30.0	36.4
77SR-15	13.8	3.9	35.6	42.3
78SR-16	15.9	13.3	24.2	42.3
78SR-17	13.5	11.9	22.3	48.7
78SR-18	11.6	7.4	28.1	48.0

E. Basis of Study

During an initial two-day inspection visit to Ft. Lewis by SD and DOE personnel on February 20 and 21, 1978, operations and data acquisition procedures were outlined by the P & M staff and discussed. An SD process engineer and an operations specialist remained in the plant for over three weeks to gather data and equipment specifications, interview process and operating staffs, prepare detailed working process and engineering flowsheets for SRC II (which did not previously exist) and generally learn about the Ft. Lewis plant and SRC II technology. Following this, the study was expanded and continued at SDNY. Information and data gathered at Ft. Lewis were reviewed along with a great volume of published reports and previous studies. During this time SD was continually in communication with the Ft. Lewis process department. On April 17 and 18, 1978, a P & M process engineer visited SDNY for further discussion and elucidation of the Ft. Lewis operation and material balance procedures. Reasonable efforts have been made by SD within the time available, to check and verify salient Ft. Lewis data and qualitative information in P & M's published reports and to give P & M an opportunity to comment before publication on the facts and opinions expressed in this report.

On April 7, 1978, SD and DOE personnel inspected Gulf Science and Technology Co's. P-99 (1 TPD) pilot plant at Harmarville, Pa., and held discussions with Gulf research staff. (On April 28, 1978, SD received a copy of the report "SRC II Processing of Kentucky Coal in Pilot Plant P99" by McIlvried, Gall and Tsai, dated April 15, 1978. This report summarizes P-99's SRC II operations and data gathered during one year of running (1976-1977) and contains a great deal of information which augments Ft. Lewis data. SD

has studied and has accepted this report at face value, since SD's exposure to the program included only a brief tour of the plant and review of the limited information in the published report and time and funds were not available for an in-depth analysis and exchange of views with Harmarville on specifics.

During the week of June 19, 1978, an analytical chemist from SD visited Ft. Lewis to discuss analytical techniques and material balance procedures with the P & M process engineering and laboratory staffs.

During the week of September 4, 1978, the authors visited Ft. Lewis to discuss and resolve comments by P & M and GMRC personnel on a draft of this report.

An assessment of the data base developed by Ft. Lewis and others on occupational and environmental safety and health aspects of this technology were not a part of SD's task.

II. OBSERVATIONS AND CONCLUSIONS

A. SRC II Distinct but Not Separate from SRC I

From the point of view of the coal liquefaction reaction, SRC II should not be viewed as a process development independent of SRC I. Rather SRC I and II are opposite ends of a continuum of possible process operating conditions. Clearly, SRC I experience was valuable in developing SRC II so rapidly and successfully. For example, the Preheater at Ft. Lewis has never experienced significant coke deposits in either mode, although it has been operated at conditions in SRC II that some analysts and critics have identified as "very near coking". Also, the physical/chemical and corrosion data developed for SRC I materials can be extrapolated to SRC II designs, in some cases.

B. Elimination of Mineral Separation is an Important Improvement

In SD's view, the elimination in the SRC II process of the mineral separation step, which is an important feature of SRC I (and some other liquefaction processes), and may consist of either filtration, centrifugation, or anti-solvent de-ashing, is a clear process improvement and advantage. A commercial development in the chemical or petrochemical industry would explore all possible avenues to eliminate such unit operations, not only because of their reportedly high capital costs,

but also because of their obviously high expected maintenance costs. Such a step appears undesirable in an energy process with a low product unit value.

C. SRC II Operability

The SRC II process appears to be stable, robust, capable of long term operation and not susceptible to frequent upsets, shutdowns, emergencies, etc. Such long-term operation was achieved in a process involving three phase flow (with one containing polymerizable material), relatively high temperatures and pressures, and while handling corrosive liquids, erosive solids and large recycles. The process seems sufficiently flexible to tolerate factors such as apparent non-uniformity of feeds and variations, due to equipment problems, in operating conditions, flows and product distributions. This was reflected in the plant's remaining in operation despite a constant cycling of certain important stream compositions. Ft. Lewis is an excellent plant for equipment testing, though for many program-related reasons and because of some design choices, it is extremely cumbersome for process design data generation. It is to the credit of the basic process concept as well as of the staff at Ft. Lewis that the plant has achieved long running times and essentially met oil production objectives.

D. Suitability of Ft. Lewis for Data Gathering

The Ft. Lewis plant has some crucial limitations in reaction and yield data gathering and analysis. As a result of this, closure of material balances is difficult and usually unreliable. The plant was unable to achieve and hold non-cycling operation suitable for data gathering experiments, partially because of limitations in the Slurry Blending and

Vacuum Flash systems, which were exacerbated by simultaneous conditions of high ash coal, high coal feed concentration and high liquid residence times.

Since no provisions were made for feed coal homogenization or for organized retrieval from storage, coal feed compositions (especially ash content*) varied measurably during and between most runs, to which some of the problems in the blending and vacuum flash systems can be attributed. Aside from more readily analyzable differences in coal compositions, random feeding of coal from storage undoubtedly introduced, at various times, coal of widely different ages within single test periods. It is known that oxidative aging can effect coal's performance in liquefaction processes. This was not accounted for in Ft. Lewis' operations, procedures, or data analyses.

Hydrogen partial pressure was limited to about 1800 psig, which is at the lower end of the range of possible interest (1700 to 2200 psig). In contrast, the P-99 unit at Harmarville is a much more flexible and accurate tool for basic reaction data gathering because of its capability to operate at higher pressure and more reliable material balance procedures. The coal feed to P-99 is not subjected to exposure aging during storage since it is received pre-ground and is stored in containers.

*Variation of (catalytic) ash content within the range of that experienced at Ft. Lewis has been found by some workers to significantly effect the conversion and yield of liquefaction processes. (Granoff, B. and Traeger, R., Sandia Labs, paper presented at 85th Annual Meeting of the AIChE, Phila. PA, June 8, 1978).

E. Usefulness of Ft. Lewis Data

Ft. Lewis has had an extensive program of liquids production and of testing various coals in a short time frame. Development goals appear to have been somewhat sidetracked in order to produce large quantities of fuel oil for a number of related test programs, demonstrate process operability and prepare for further SRC I testing. In an attempt to develop a sufficient number of correlatable points, operating parameters were varied according to plan among some runs, but often these conditions had to be modified as required to maintain the operation. Duplicate runs were made in some cases to establish reproducibility. Ft. Lewis data were primarily useful in verifying the operability of the process at various conditions using three different coals. To a lesser extent, the Ft. Lewis plant can be used to observe scale effects between its own and the Harmarville and Merriam reactors. Ft. Lewis has also helped identify and provide information on critical and troublesome unit operations such as Slurry Blending, the Stripper and Vacuum Flash.

Though some trends can be perceived for most of the important design variables, reliable "correlations" of Ft. Lewis data alone are essentially impossible. Some runs had several key parameters simultaneously varied among them. As a result, effects of a single variable could not be distinguished. Also, the data adjustment procedure used by P & M to achieve an elemental balance distorted some of the raw data, particularly the Naphtha and Fuel Oil yields. Only a few reliable data points with corresponding operating parameters were found between Ft. Lewis and Harmarville data on Kentucky coal. These points seem to offer some limited evidence that the two plants generate similar product distributions.

Although GMRC has stated in a recent paper* that there are no major differences between Merriam, Harmarville and Ft. Lewis results, the detailed statistical study of all the data supporting this claim remains unpublished. Indications from this same paper are that Gulf's proposed demonstration plant design will more resemble Harmarville than Ft. Lewis in the arrangement and design of the separations downstream of the Dissolver. (SD has not had access to other than this limited, published exposition of the proposed design.) Thus, it would seem important to make further runs at Ft. Lewis at as close to demonstration plant conditions as Ft. Lewis can operate and with demonstration plant coal, if possible. It would also be highly advisable, for further testing in support of this project in the long range, to revamp the Ft. Lewis plant to reflect the ultimate demonstration plant configuration and operating conditions.

F. Heavy Distillate Loss

Heavy Distillate accumulation during the program appeared to be less than was originally expected. P & M was requested by the DOE to characterize distillate yields in non-material balance periods when distillate production is not closely followed as it is during material balance runs. To do this, P & M modeled distillate production based on process performance during material balance periods. When P & M found less Heavy Distillate in plant tankage than had been predicted, the difference was designated as "loss". SD has concluded that this "loss" is mainly optical and a result of P & M's having used a totally empirical model, which is founded on an insecure data base. On the other hand, there probably is a

* Jackson, D.M., and Schmid, B.R. - "Commercial Scale Development of the SRC II Process" - University of Pittsburgh, August 1-3, 1978.

small true loss of the HD actually made in the reaction through a combination of effects such as:

1. variations in the amount of degradation (to lighter products and residue) of the HD introduced back to the reactor in the seal flush and recycle slurry during non-material balance run periods,
2. inaccuracies in another P & M correlation to correct for HD lost with the Vacuum Bottoms, based on the measured fusion point of the residue,
3. a large random error in determining HD make from measurements during material balance runs,
4. evaporation and loss to atmosphere of HD in the residue solidification area,
5. physical losses such as spillage and leakage and
6. loss as an emulsion with purged waste water.

The last two effects have been well defined and largely resolved through investigative and control efforts by the Ft. Lewis staff during the latter part of the SRC II program.

G. Ash Balance

The apparent "loss" of ash between the feeding of the coal and the collection of residue remains unresolved to SD's satisfaction. This apparent loss averaged 17% of the ash in the feed coal over the 21 runs and was as high as 27% for one run (77SR-13). In another example, 77SR-16, with an apparent ash recovery of 90%, the rate of ash loss appeared to be about 24 lbs/hr. The coal feed rate varies widely, but appears to be measured with reasonably good accuracy (see IV. D1.a), and the variable measurements were probably not always high as they would have had to be if

such bias were the major source of the error. It appears virtually impossible that coal ash could build up in the equipment and be hidden in ton quantities as the apparent "loss" would require. Nor can coal ash leak from the system as readily as gases and liquids do. Furthermore, a comparison of a few coal and residue ash analyses shows they are nearly identical, so that differential dissolution of some ash component into the waste water would seem to be precluded. It can only be assumed that analysis of ash in either the coal or residue is inaccurate and/or that the weighing of the residue is inaccurate, and always low, so as to lead to most of this apparent loss. The forcing of the material balance to make ash in the vacuum bottoms equal to that in the feed coal is one of the expediciencies contributing to the generally poor reliability of the Ft. Lewis data base.

H. Commercial Plant Design

SD usually has available a speculative commercial plant design, based on exploratory information from the laboratory, when it is asked to participate in a pilot plant data evaluation aimed at developing a commercial design basis. A commercial design must be made available to guide a more detailed and definitive investigation of the complete data base for SRC II and for the DOE to monitor the pilot plant program in a more effective manner in support of a demonstration plant design effort.

I. Hydrogen Donor Solvent Effects

Evidence was developed in the research efforts of others - such as by workers at Stoke Orchard in Britain, fundamental work done by Mobil (under EPRI sponsorship), background work by Exxon on their EDS process

and observations from the Wilsonville SRC pilot plant - which indicates the common importance of hydrogen donor properties of the liquefaction solvent in determining coal liquefaction conversion and yield, with and without catalysts or molecular hydrogen present. An examination of all of this fundamental work shows that hydrogen donor compounds can be found in various boiling ranges across the entire spectrum of coal liquefaction liquid products, but appear to be most concentrated in the higher boiling fractions. Donor solvents appear to be major factors effecting mechanism in all other liquefaction processes. Ft. Lewis did not test for the donor solvent properties of the Stripper Bottoms and of the substantial quantities of seal flush used to slurry the coal feed. From the cycling of solvent and SRC content analyzed in the Stripper Bottoms (See Table 2 following, and Figure 13 in V.C.4.), and from the variation in quantity and composition of the seal flush, it can be assumed that there was a substantial and continual variation in the hydrogen donor solvent content of the reactor feed during various runs at Ft. Lewis. We strongly suspect that the hydrogen donor quality of the reaction feed mixture may be as important in SRC II as in other liquefaction processes and that its probable variability may be a hidden parameter in the Ft. Lewis operation. We believe it is at least partially responsible for the irregular yield results, including some of the "loss" of Heavy Distillate.

SD recognizes that no universally accepted method exists for determining hydrogen donor properties of coal solvents. However, attempts should be made to evaluate the quantitative effects of hydrogen donor solvent. Even if exact yield effects remain ultimately undefinable, enough of an understanding should be gained to isolate the role of the donor solvent in the

operation of the reaction section and to direct the evolution of the demonstration plant design basis in the solvent separation and recycle steps.

TABLE 2

COMPOSITION OF STRIPPER BOTTOMS, WT. %

<u>Coal Type</u>	<u>Month</u>	<u>Run No's</u>	<u>MD</u>	<u>HD</u>	<u>SRC</u>	<u>IOM</u>	<u>ASH</u>
<u>AVERAGE MONTHLY COMPOSITION (1)</u>							
Kentucky	June	77SR-1,2	2.6	43.1	33.8	7.1	13.1
"	July	77SR-3	6.3	34.6	37.3	7.9	13.5
"	August	77SR-4,5,6,7	12.6	33.1	34.7	7.0	12.2
Illinois	September	77SR-8,9	8.0	40.1	29.9	6.1	15.6
"	October	77SR-10,11	11.7	37.7	30.8	5.1	14.3
Pitt Seam	November	77SR-12,13,14	6.5	32.3	36.7	10.3	13.9
"	December	77SR-15	4.8	30.4	39.1	10.8	14.7
"	January	78SR-16,17,18	8.3	31.2	35.2	11.1	14.1
Illinois	February	78SR-19,20,21	12.5	35.6	31.9	5.2	14.5
<u>AVERAGE COMPOSITION DURING MBR'S (2)</u>							
Illinois		77SR-9	8.9	37.3	30.6	6.0	16.9
"		77SR-10	15.0	36.1	28.5	5.2	14.9
"		77SR-11	15.6	36.0	31.4	4.7	12.0
"		78SR-19	12.4	37.3	30.2	5.7	14.2
"		78SR-20	14.5	36.6	28.8	5.7	14.3

(1) From Monthly Fort Lewis Plant Laboratory Progress Reports.

(2) From Raw Data supplied by Fort Lewis.

III. PRESENTATION OF BACKGROUND MATERIALS

A. Ft. Lewis SRC II Process Flow Description

1. PFS 2002-X-101 - Coal Preparation, Reaction and Slurry Recycle

a. Coal Preparation

In this section, coal is first dried and pulverized and then is blended with recycle process slurry (containing catalytic coal minerals and process solvent fractions). The slurry is diluted by pump seal flush solvent as it is recirculated and pumped to a high pressure for feeding to the Reaction System.

Coal (Pittsburgh Seam, Kentucky or Illinois) is reclaimed from one of several separate storage piles by a front-end loader, dumped into a hopper and, if the received or stock-piled coal is too wet for handling in the elevators or rotary valves, it is moved by conveyor to two steam-pan coal dryers where it is partially dried to facilitate handling and storage. The dryers discharge the coal through a grating into a conveyor which takes it to the Primary Coal Crusher, which reduces the coal to minus 1/8 inch. A bucket elevator lifts the coal to one of two bins which alternately discharge to another elevator carrying the coal into the Crushed Coal Surge Bin. At times, coal can alternatively be loaded directly from rail cars to the

storage bins, and if sufficiently dry and easily handled, can bypass the dryers. The preceding steps are not detailed on the flowsheets.

All of the following equipment is shown on PFS-2002-X-101.

A Gravimetric Feeder controls flow to a (ball-race) Dry Pulverizer which is swept with dried heated inert gas. Here the coal is dried and reduced in size to approximately 70% minus 200 mesh. Coal in the desired size range is classified out of the mill by the inert gas and recovered in the Pulverized Dust Collector (bag house) from which the coal is removed to blending by a Vibra-Screw Feeder. Wet inert off-gas at 150°F from the Collector is dried by direct adiabatic contact with cooled circulating water in the Water Scrubber and the dry gas is returned by a blower to the natural-gas fired Gas Preheater.

b. Slurry Recycle

The pulverized coal is contacted and mixed with recycled slurry (Stripper Bottoms which has been cooled to approximately 350°F) in the highly agitated Slurry Mix Tank. Temperature must be controlled to avoid a high viscosity gel state which occurs above 400°F with slurries of high solids concentration and with the long holding time of the tanks in this system. The maximum allowable temperature seems to be specific for various coal types. Gases venting from the Mix Tank, which is held under a slight vacuum, contact incoming slurry in a small Scrubber. Mixed coal slurry discharges to the agitated Slurry Blend Tank. The Slurry Blend Pumps maintain a high rate of circulation to the Slurry Mix and Slurry Blend Tanks.

The Circulation Pump seals are supplied with a flush fluid from the Vacuum Flash Condensate Drum (or, alternatively, with solvent from the Process Solvent Accumulator.)

A slight vacuum is maintained on the Mix Tank with a steam jet ejector system to prevent Mix Tank vapors from entering and condensing in the Coal Feed system. The Slurry Blend Tank Vent Condenser and the Slurry Blend Condensate Tank recover water with some organics vented from the Blend system. Collected condensate is intermittently pumped to drums, which are periodically emptied with a steam-driven circulating pump to the Widing Tank (an atmospheric drum for slops collection) together with other miscellaneous streams containing organics. The Widing Tank is steam-heated and vented to atmosphere to allow boiling-off of water contained in the slops.

(An alternative blending device, the Coal Dust Scrubber, which is a solids eductor, was used in SRC I where the viscosity of the process solvent is much lower than that of the recirculated slurry of SRC II. This system is available for use during upsets in the Mix Tank. However, it requires careful addition of seal flush to the circulating slurry and close monitoring because liquids tend to splash into the feed hopper and cause plugging.)

c. Preheater and Dissolver System

The reciprocating Preheater Charge Pump is used to raise the pressure of the slurry discharging from the Slurry Blend Pumps to a pressure of over 2000 psig.

In this system, coal/solvent slurry is reacted with recycled hydrogen gas under pressure in a fired Preheater and in an adiabatic high residence time Reactor called the "Dissolver". Physical and chemical processes break down the coal structure and convert most of mineral sulfur by the reduction of FeS_2 to FeS and H_2S , convert some of the organic sulfur to H_2S , some of the coal oxygen to water and some of the coal nitrogen to ammonia, and hydrocrack the coal-derived liquids and high-boiling SRC to lighter products. The reactions yield a net production of coal liquids and SRC, gaseous hydrocarbon byproducts, and mineral and organic insoluble residues.

High pressure coal feed slurry from the Preheater Charge Pumps is joined with a stream of compressed, preheated recycle hydrogen. The gas and slurry enter the coil of the gas-fired Preheater in three phase flow. The Preheater contains 50 coil turns, but it is oversized for this service, so the lower 23 coils have been insulated with a refractory coating to render them inoperative. The coal structure is thermally cracked and dissolved into a plastic mass. Repolymerization is prevented by the transfer of hydrogen to the cracking products, presumably via the donor solvents present. The process stream reaches a high viscosity peak (and exhibits apparent, non-Newtonian flow characteristics) within the coil. The viscosity then decreases as the temperature increases to the outlet condition of approximately 800°F . The hydrogen recycle to the Preheater induces the proper velocity in the Preheater coil to provide a good heat transfer coefficient and provides hydrogen for dissolution and partial hydrogenation reactions which take place

in the Preheater. Coking is believed to be prevented by the proper movement of the slurry through the coil and by the presence of hydrogen which stabilizes free radicals.

The Preheater effluent and recycle hydrogen flow to the Dissolver, a vertical up-flow, gas bubbling reaction chamber with no internals. The bulk of the exothermic hydrogen reactions with coal or coal-derived liquids take place in this unit. Reaction temperature is moderated by the hydrogen quench. Three hydrogen injection points are installed at different levels. The top and middle injection points are preferred, as they give a more isothermal profile, while use of the bottom sparger results in a substantial temperature gradient. Though all the spargers have plugged at various times due to pressure accidents, and were cleared with heat tracing, the upper two eventually collapsed and so the bottom sparger is the only one that could be maintained in continuous operation throughout the program. Because of appreciable back-mixing of liquids and solids due to the bubbling action of the gas, the Dissolver departs substantially from plug flow behavior.

Coal minerals (especially FeS released from the coal structure by dissolution and hydrogenation of FeS_2) are believed to catalyze the hydrogenation reactions, particularly hydrocracking to form liquids and gases. Other hydrogenation reactions are: hydrogenation of sulfur to H_2S and the conversion of coal oxygen to water and coal nitrogen to ammonia. To maintain a reasonably constant partial pressure of hydrogen throughout the Dissolver, a

large recycle of hydrogen is used so that hydrogen consumption per pass in the two-step reaction system is restricted to about 20-25%.

Water from the Recycle Process Water Tank (RPWT) is used to quench the Dissolver effluent to about 700°F. (This quench is required at Ft. Lewis since the design temperature of the downstream vessel is 750°F.)

The Dissolver effluent is processed in the following manner. It is initially separated at reactor pressure in the HP Flash Drum. The vapor is air cooled, water cooled and then separated in the Recycle Condensate Separator. The non-condensed gas - comprised of unreacted hydrogen, methane, and light hydrocarbons, H₂S and CO₂ - is sent to the Gas Treatment Section (PFS 2002-X-104).

The slurry from the HP Flash Drum is flashed into the Intermediate Pressure Flash Drum, operating at approximately 800 psig and 700°F. The separated slurry is sent to the lower section of the Stripper and becomes the bulk of the Bottoms Slurry product of that column. The hot vapor from the Drum is combined in the No. 3 Separator with the liquid from the Recycle Condensate Separator. Non-condensed gases are flared. Condensed water is returned to the Recycle Process Water Tank (RPWT, on PFS 2002-X-102). The condensed oil, consisting mainly of Naphtha and Middle Distillate is sent to the top of the Stripper. (It is possible, for control purposes, to divert part or all of the oil flow from the No. 3 Separator directly to the RPWT, where the Stripper overhead is eventually sent in any case.)

d. Stripper

The Stripper produces an overhead containing the bulk of the Naphtha and Middle Distillate. The overhead from the Stripper is partially condensed in an Air Cooler. The condensate is pumped and the vapor flows from the Reflux Drum to the LP Flash Condenser (PFS-2002-X-102). The Stripper Bottoms is split into a slurry stream recycled to the Slurry Blend system, and a stream which flows without pumping to a Vacuum Flash System for separation.

The Stripper does not have a reboiler and therefore depends on the sensible heat of the Intermediate Pressure (IP) Flash bottoms and on Dowtherm heating of the oil portion of the Recycle Condensate Separator Bottoms from the No. 3 Separator (upper feed) to provide heat for vaporization. Some stripping gas is also provided by a flow of inert gas to the Column bottom.

The Stripper Bottoms composition is controlled by adjusting the total inert and flash vapor provided to the column and the amount of oil fed from the No. 3 Separator.

2. PFS 2002-X-102 - Vacuum Flash and Wet Solvent Accumulation

This equipment and that on PFS 2002-X-103 has been adapted and rearranged, largely ad hoc, from SRC I, which requires filtration for minerals separation, to SRC II, which purges the minerals along with organic solid residue from the Vacuum Flash and requires no filtration steps.

In SRC I, recovered process solvent (see PFS 2002-X-102 and 103) is used to mix with feed coal in slurry preparation, whereas recycled slurry from the Stripper Bottoms is the feed coal slurrying medium in SRC II. These sections, therefore, entail somewhat different processing schemes and conditions in SRC II compared to SRC I, but many vessels maintain names relevant to SRC I operation. The arrangement of flows in the Ft. Lewis plant has evolved into a rather complex and confusing scheme, but one of considerable flexibility.

This flexibility is required, for example, to use alternatively either of the Vacuum Flash Systems - both have long gravity-flow bottoms discharge legs, complex piping, and other mechanical features which make them troublesome. The Old System was designed for low mineral content SRC I and has difficulty handling the higher mineral content SRC II residue. Plugging with coke or polymerized material is frequent. Coking of stagnant material is induced by the Dowtherm tracing used throughout both systems to maintain fluidity, by overheating of pumps in the Old System, and by coking in the Preheaters. Consequently, both systems must be kept under intensive circulation during start-up.

a. Stripper Overhead Processing

The condensate and vapor from the Stripper Reflux Drum are cooled in the Feed Flash Vapor Condenser and then pass through the No. 1 and No. 2 Separators to the Recycle Process Water Tank (RPWT). These separators are simply in the SRC I piping route and serve no real purpose in SRC II. Gas is flared from the No. 1 Separator that could otherwise be vented from the

RPWT. The RPWT itself serves as the collector of mixed solvents and recycle water in the back end of the plant. This tank develops three layers: light oil on top, a water layer in the middle and heavy oil in the bottom. The bottom layer forms only during start-up or upsets in operation.

The water is pumped to high pressure for use as quench water for the Dissolver effluent, and the net water make (containing some dissolved and suspended organics) is pumped to the surge reservoir in the Waste Treatment Section. Heavy oil (essentially Heavy Distillate), if it accumulates, is pumped to the Light Ends Column Feed Tank.

During non-material balance run (non-MBR) operation, the light oil (essentially Naphtha and Middle Distillate) flows to the Wash Solvent Accumulator. During MBR operation, the Wash Solvent Accumulator normally collects and holds most of the net solvent produced in the plant.

b. Stripper Bottoms Processing

The Bottoms from the Stripper (the total minus slurry recycle to the Slurry Blend system) is flashed in either the "New" or "Old" Vacuum Flash (VF) system into a recovered solvent overhead and a bottoms (containing undissolved organic matter, net mineral residue plus the distillation residue part of the dissolved coal) which will become the solidified by-product of SRC II. Either system may be used during MBR mode operation. The New System is normally fed directly, without pumping, from the Stripper. The Old System is fed through the Filter Feed Flash Vessel (which can alternatively

be used to feed the New System.) (During non-MBR times or normal production running, the Widing Tank contents are pumped through the bottom of the Flare KO Drum and into the Filter Feed Surge Vessel, each containing miscellaneous liquids. This mix is intermittently emptied to the Filter Feed Flash Vessel and can then be pumped to the operating Vacuum Flash System).

The Old Vacuum Flash System consists of a Feed Accumulator Drum and a gas-fired Vacuum Flash Preheater through which the feed is pumped to the Vacuum Flash Drum. This is a simple Flash Drum which separates the hot feed slurry at about 1.5 psi pressure into an overhead vapor and a bottoms residue slurry. This slurry is discharged through a long, barometric downleg to the water cooled (with under- and over-sprays) Sandvik steel belt solidifier. The solidified SRC II residue is conveyed to trucks and weighed. The cooling water effluent, containing organics, flows by sewer to the Surge Reservoir.

The New Vacuum Flash System is designed for SRC II (higher solids and smaller flows) so it operates without a feed accumulator or pump and uses a downflow, Dowtherm heated coil (rather than an up-flow fired heater) to provide vaporization heat for the flash. The bottoms are discharged to the same Sandvik belt.

Overhead vapor from either system is partially condensed in an air cooler and further condensed and cooled in a water cooled exchanger. The condensate is accumulated in the Vacuum Flash

Drum and pumped to the Wash Solvent Accumulator.

During an MBR, a side stream of Vacuum Flash condensate oil can also provide flow of seal flush or diluent for the Filter Feed Flash Vessel contents.

The vapor overhead from the Vacuum Flash Condensate Drum is kept under vacuum by a two stage steam jet ejector with water circulation-cooled barometric condensers. The organics-containing Hotwell effluent from the system is pumped to the Wash Solvent Accumulator, or alternatively, to the Light Ends Column Feed Tank in the distillation area. The contents of the Wash Solvent Accumulator (light oil, Vacuum Flash overheads and Hotwell effluent) are pumped to intermittent (campaign-continuous) distillation. The water phase from the Hotwell is sent on level control to the RPWT for oil-water separation.

Various vents flow to the flare header and Flare Knock-Out Drum, which accumulates knocked-out liquid.

3. PFS 2002-X-103 - Distillation and Seal Flush Systems

Solvent is fractionated (during non-MBR mode) into Naphtha (C_5 and heavier, boiling up to $350^{\circ}F$), Middle Distillate ($350-550^{\circ}F$), and Heavy Distillate ($550-850^{\circ}F$). These boiling ranges vary somewhat from Light Oil, Wash Solvent, and Process Solvent, respectively, of SRC I. The Middle Distillate is the major product and has the lowest sulfur content of the three.

The distillation consists of a Light Ends Removal Column with ancillaries and a Wash Solvent Fractionator with ancillaries. Solvent feed is split in the first Column into a Naphtha and water overhead stream and a Bottoms consisting of the Middle and Heavy Distillates. Condensed overhead Naphtha is further cooled and separated from water. The water is returned to the RPWT and the oil goes to the Naphtha storage in the tank farm.

The Light Ends Column Bottoms are split in the Wash Solvent Fractionator into Middle Distillate overhead, which is cooled and pumped to the Middle Distillate Storage Tanks, and Heavy Distillate bottoms, which are cooled and flow to the Process Solvent Accumulator. From there, net Heavy Distillate can be pumped to the Heavy Distillate Storage Tank, to the Flush Solvent Accumulator which feeds a closed-loop plant flushing system, or primarily to the Seal Flush Accumulator to provide open flow seal flush for the Slurry Blend and Preheater Charge Pumps and for other miscellaneous users. Heavy Distillate can also provide open pump seal flush to the Filter Feed Flash Vessel feeding either Vacuum Flash System, or can be used directly to dilute the circulating Vacuum Flash System feed when it is going to the Old Vacuum Flash System. A high pressure pump can also provide emergency solvent flush to the Preheater, but this is seldom used.

4. PFS 2002-X-104 - Gas Treatment and Recompression

The gaseous overhead of the Recycle Condensate Separator, the last stage of the Dissolver effluent quench and flash steps, passes to the High Pressure Desulfurization Unit, a DEA acid gas absorber, which removes CO_2 and H_2S . These acid gases are stripped to

regenerate DEA and are sent to the low-pressure Stretford Desulfurization unit - which consists of a scrubber column, reactor tanks and a melter - for recovery of solid sulfur. The desulfurized off-gas stream (mostly CO_2) goes to the Flare.

Desulfurized hydrogen gas flows to the Naphtha Absorber for the removal of light hydrocarbons, the net make of which must be purged from the gas recycle loop. The Naphtha Absorber is now actually run with less selective Middle Distillate as the absorption oil, since the Naphtha cut from this plant was found to foam excessively. The light hydrocarbons (methane through pentane) as well as absorbed (and lost) hydrogen, are removed from the absorption oil via a simple flash in the Naphtha Flash Drum.

The overhead of the Naphtha Absorber (still containing some impurities such as CO and methane) is joined by hydrogen from the discharge of the Fresh Hydrogen Compressor and flows to the Recycle Hydrogen Compressor Scrubber (actually a KO pot) and then enters the hydrogen recycle compression loop. (Recycle gas may also bypass the Naphtha Absorber and flow directly to the gas loop for pressure control and other reasons.) This "Scrubber" sends knocked-out condensate to the Flare KO Drum.

Fresh hydrogen enters the system from the H_2 Plant compressor. Hydrogen is generated in the H_2 /Syngas Process by reforming natural gas with steam in a furnace, shifting to H_2 on a catalyst, removing CO_2 in an MEA absorber and compressing to 75 psig. The

Fresh Hydrogen Compressor is a three-stage inter-cooled unit which raises the fresh hydrogen pressure to 1200 psig.

The total hydrogen stream is raised to the system supply pressure of about 2300 psig in one of the two simultaneously used Hydrogen Recycle Compressors ("A" and "B") which differ somewhat in design and capacity. Both are required in SRC II, while only the smaller one is required in SRC I. (Substantial hydrogen seal leakage can occur in the compression system, estimated to comprise up to 25% of the make-up.) The compressed hydrogen stream is cooled and flows to the Recycle Hydrogen Scrubber (KO Pot) and then to the Preheater and Dissolver or to storage. Hydrogen may be stored in two pressure tanks for use in maintaining system pressure and flushing the system during emergency shut-downs.

The major portion of the 100-200^oF total(cooled) hydrogen stream is preheated in an Exchanger with steam to prevent what is believed to be overcooling of the slurry and high viscosity/pressure drop effects at the inlet of the Slurry Preheater. The remaining portion is sent cold to the Dissolver as quench gas, whose flow is controlled on Dissolver temperature. Hydrogen consumption is about 20-30% per pass in the overall reaction system.

5. Off-site Waste Treatment

With the exception of the Sandvik belt and RPWT water, plant area drains, runoff, and various process water purges pass through an oil-water separator (where emulsified solvent can be reclaimed) to the Surge Reservoir, which can also be periodically skimmed or

dredged to return organics streams to the process. The aqueous effluent from this reservoir is treated in an aerated bio-digester to remove organics, which are mostly phenolics.

B. SRC II Process Flowsheets

Simplified process flowsheets were prepared by SD and are included here. These are as follows:

<u>Dwg. No.</u>	<u>Description</u>
2002-X-101	Coal Preparation, Reaction and Slurry Recycle
2002-X-102	Vacuum Flash and Wet Solvents Accumulation
2002-X-103	Distillation and Seal Flush Systems
2002-X-104	Gas Treatment and Recompression

The flowsheets show the basic features of the Ft. Lewis pilot plant as adapted to SRC II operation. As noted in the text, the pilot plant was originally designed for SRC I operation and was subsequently adapted to SRC II. Hence, equipment sizing and some of the stream routings reflect SRC I requirements. Equipment which is used only in SRC I operation (e.g. mineral separation facilities) is not shown.

C. P & I Engineering Diagrams

Prints of SRC I piping and Instrumentation (P & I) Engineering Diagrams have been marked up to reflect SRC II operation by eliminating lines and equipment not used in SRC II and also to indicate liquid sample points and gas chromatograph taps. It has been decided not to redraft these documents or reproduce them at present. They are available for reference at SD, New York. An index of the diagrams is included in Appendix G.

D. Physical/Chemical Data

At P & M there is no such document as a "technical data book" which is generally compiled by SD for processes which reach the commercial stage. Physical/chemical data which is apparently available for process analysis or for a demonstration plant design falls into the following general categories of decreasing availability:

- 1) Empirical data, used by P & M for process analysis or for designing revamps, which have been obtained from the P & M labs or from Gulf.
- 2) Literature correlations from the petroleum industry (mostly vapor/liquid and thermodynamic data) which were spot-checked by the P & M process department and were thought sufficiently applicable for their present uses. For example, the Grayson-Streed correlations for V-L equilibrium, complicated by the presence of water, have been used to calculate flash and distillation separations.
- 3) Data, which may be applicable to SRC II materials, being produced by other organizations under DOE or other sponsorship (e.g. enthalpy data from the Colorado School of Mines, relative volatility data from Oklahoma State University and from Purdue University by K.C. Chao, the Lummus Co.'s correlation of Wilsonville data, etc.). Most of this data will probably not be available and correlated for several years.
- 4) Data which likely exists in lab and process department records at Ft. Lewis, but which are still uncorrelated. It was estimated that six man weeks would be required to complete a data base from

information on hand in lab reports, etc. Limited manpower has held up this effort. These data would include: specific gravities, viscosities, boiling points from distillations, elemental analyses and flash points (there would be limited data on vapor pressure or V-L equilibrium).

- 5) Potential data to be obtained by lab analysis and tests on stored samples of process materials. P & M has stated that samples of liquid products are stored from nearly all MBR's which would give almost complete vapor pressure data.
- 6) Data which would be available only from further operation of the SRC II mode under specified conditions, e.g. during a demonstration plant design phase, if the pilot plant is revamped to allow an operation closely corresponding to demonstration plant conditions.
- 7) It has been indicated that some critically needed, but as yet unpublished, vapor-liquid equilibrium data was privately obtained by Gulf, through an outside laboratory from samples generated at Harmarville. Raw data from this work was said to have been reduced and correlated by Gulf, using a corporate computer program. It was further indicated that Gulf's contract with the DOE allows this data to remain "unpublished" until a later stage of development occurs.

The Ft. Lewis and Merriam programs have not included an adequate effort to obtain, for the public domain, this type of physical/chemical data needed for unit operations evaluations and designs, particularly thermodynamic and vapor-liquid equilibrium data.

E. Analytical Procedures

1. General

A list of analytical procedures obtained from DOE publications was reviewed with the Chief Analytical Chemist of the Ft. Lewis lab. Copies were obtained of relevant ASTM and other standard procedures which form the basis of most of the P & M tests. Many of the tests listed in Table 13, FE 496-T13 (Interim Report No. 22, June 1977) are not used or are seldom used and some others are not relevant to the process material balance (such as certain waste and cooling water tests). Finalized procedures were obtained for the many relevant tests which are P & M adaptations of well-established standards, such as ASTM. For others, only unfinalized, "working" procedures were available. SD agreed not to publish these without giving P & M an opportunity to finalize them.

2. Comments

a. Gas Analysis

P & M determines the average molecular weight of gas streams via gas chromatograph (G.C.) analysis. The mass flow rates are then calculated from orifice plate readings.

Analyses with 95-100% accountability are considered acceptable. These are normalized to 100%.

Hydrogen, nitrogen, C₁-C₄ hydrocarbons, CO, CO₂ and hydrogen sulfide are presently measured by peak height measurement rather than by integration of the area below each peak.

Significant errors may be introduced by measuring peak heights, as peaks become broader and less symmetrical. C_5+ is presently measured by area integration, but the readings are not compared with standards, so these results are limited in meaningfulness.

P & M is adding a G.C./microprocessor system which would give analyses for eight gas streams via integrated area measurement for H_2 and NH_3 . Other constituents of these streams will continue to be derived from the existing gas chromatographs. This should increase the precision and reproducibility of gas analyses. It was suggested by SD that NH_3 analyses be periodically checked by a wet method available for measurement of basic nitrogen in gases by low temperature acid scrubbing (UOP Method 315-59). See Appendix F for details. Considerable NH_3 may also be dissolved in the water phase pumped from the plant, and this purge should also be identified.

b. Hydrogen Balance

A correction for non-linear hydrogen response was not applied to the chromatographic data for runs 77SR-1 through 77SR-11. This would clearly cause some error in the gas analyses for these runs. Response factors for hydrogen were obtained over the entire range after 77SR-11, thereby compensating for non-linear H_2 response and improving H_2 balance and the overall confidence level in subsequent runs.

c. Sulfur Balance

Hydrogen sulfide is measured in the gas streams.

Measurement of sulfur and other impurities in the water purge, however, is not included in the material balance data. Analysis and flow measurement of this stream is therefore needed to help close the sulfur balance (as well as to account for losses of other material).

d. C₅-C₆ Losses

Presently, the calculated Naphtha production at Ft. Lewis is forced to balance total carbon in the products with carbon in coal feed (partly to account for C₅-C₆ losses). The C₅ + fraction of the off-gases is now calculated on the basis of total peak area integration. The four columns used in the process chromatographs were selected four years ago by Rust Engineering and Beckman Instruments. It is suggested that P & M contact Beckman Instruments and inquire about advances in separate analyses of components of the C₅+ segment of the lights. This would be an important step toward eliminating a forced Naphtha balance.

e. H₂O Analysis

Many on-line H₂O monitoring devices are commercially available. The choice would depend on a rough estimate of the percent H₂O in the stream at the sampling point. If the percent H₂O in the gas stream is determined, the percent H₂O in the liquid could be calculated from vapor-liquid equilibrium data. This improvement in analytical procedure would certainly increase the

reliability of the reported "water plus losses". It should be understood, however, that the high ratio of water circulation to throughput makes a water balance very difficult at Ft. Lewis, which operates without a Dissolver quench cooler, instead using a water quench.

f. Ash Analysis

The sources of the differences in activity among the coals tested (Illinois #5, Kentucky #9 and 14, Pittseam Blacksville #2) should be identified. A possible means would be low temperature ash determination (LTA, see note below) and subsequent accurate mineral determination. This may give a clue as to what is catalyzing the coal liquefaction reaction. It is widely held that iron sulfide is a catalyst. However, this does not preclude other minerals from exhibiting catalytic properties.

P & M performs a "quick ash" analysis on coal samples taken at two or four hour intervals during runs. This analysis is performed by P & M on SRC by placing the sample (contained in a platinum crucible) into a quartz tube which is then heated in a furnace to a temperature between incandescent and white hot. The actual temperature is not measured but is probably about 1000°C. First an N₂ flow, then an air flow, and finally an O₂ flow are passed through the tube to carry off volatiles and provide oxygen to burn organics in the sample.

SD has pointed out that reactions might occur under the P & M "quick ash" conditions which would not occur under ASTM ashing conditions, e.g.:

These might possibly explain part of the "loss of ash" phenomenon observed in certain runs. However, coal analyses have also been done by the "quick ash" method for many of the runs and this would tend to cancel differences between analyzed ash in the coal and SRC in those runs. Only daily composite samples of coal are ashed by the ASTM method (750°C). Due to time limitations, P & M does not routinely cross-check the many in-run "quick ash" analyses against the ASTM method. P & M has compared the two methods and reports that "quick ash" gives a slightly lower ash content than the ASTM method, but that the difference between methods is less than one percent.

Note on Low Temperature Ash (LTA) Determination

LTA is produced by an O₂ apparatus. The sample temperature remains low (100-200°C). Therefore, losses are avoided which are incurred in the conventional high temperature (750°C)

ASTM ashing procedure due to vaporization, diffusion, and thermal decomposition. The gaseous products of oxidation, CO_2 and H_2O , are continuously exhausted from the system. Ashing time required for bituminous coals is 24 hours and for lignite 70-130 hours.

After the production of low temperature ash by the O_2 plasma system, minerals are principally characterized by two methods:

- 1) Scanning electron microscope (SEM) used for particle shape, size, and crystal structure identification.
- 2) Energy Dispersive X-ray Analysis System (EDXRA) used to give complementary semi-quantitative information on the main elements present in the particle.

IV. EVALUATION OF FACILITIES AND PROCEDURES

To view in perspective the function of the Ft. Lewis, Merriam and Harmarville facilities in the SRC process development and commercialization effort, it is important to enumerate some of the purposes pilot plants can serve. Depending on the scale of their equipment, their degree of correspondence to a fully integrated commercial design, their programming and other factors, pilot plants can be suited to a variety of purposes or emphasize only one.

A. General Objectives of Pilot Plants

1. Demonstration of Operational Reliability

- a. On stream time, identifying sources of possible upsets-
does the process have problems which would prevent sustained operation?
- b. Materials of construction testing.
- c. Definition of mechanical equipment requirements.
- d. Equipment maintenance factors.
- e. Build-up of deposits, identification of by-products.
- f. Controllability of recycle loops.
- g. Operator training.
- h. "Political" demonstration; hands-on facility available to sponsors and prospective users.
- i. Definition of control instrumentation and analytical procedures.

2. Feedstock Testing, Product Samples

- a. Applicability or sensitivity of operation to various feedstocks.
- b. Variation of product quality and yield with different feedstocks.
- c. Production of samples for end-user tests.

3. Process Development Data Gathering

- a. Physical/chemical data development.
- b. Kinetic and yield studies.
- c. Exploratory research on a full range of process variables aimed at selecting and optimizing process conditions for a commercial design basis.
- d. Development of analytical procedures.
- e. Determination of unit operations data such as adsorption factors, multi-phase flow correlations, heat transfer coefficients; etc.

4. Developing Factors for Equipment Scaling

Identification of scaling relationships and problems, especially for reactors which do not fit simple models like liquid well-stirred tanks, nor are composites of modular elements like plug flow tubular reactors.

The Ft. Lewis plant--because of its size, complexity, certain equipment limitations, program, and production requirements - was not particularly suited to some process development data gathering objectives such as items 3.b and 3.c above. Because of manpower constraints, item 3.c above has also received limited attention.

B. Data Required for a Commercial Plant Design Basis

For the SRC II process, the following variables and factors have been identified as being of prime importance in evolving a design basis for a demonstration plant (which would be a module or single train of a commercial scale design). This list is concerned mainly with the "reaction" section of the plant. The other ("downstream" and ancillary) systems will generate numerous questions which are alluded to by category, but are not detailed below:

1. Independent Parameters

These factors can be independently selected to achieve an optimum design.

- a. Selection of coal type
- b. Feed coal particle size (as might effect hydraulics and chemical reactivity in the Slurry Mix system and the Preheater and Dissolver)
- c. Recycle Slurry/coal feed ratio
- d. Solvent concentration (and properties) in the Preheater feed
- e. Ash concentration and composition in Preheater feed
- f. Design of Dissolver with respect to:
 - (i) Recycle hydrogen ratio to feed, and hydrogen partial pressure,
 - (ii) Flow regime with respect to plug flow vs. back mixing,
 - (iii) Slurry average residence time,
 - (iv) Operating temperature and
 - (v) Degree of potential buildup of solids residue concentration (perhaps as a function of feed coal particle size), and the necessity to consider this in the design or internals

- g. Design of Preheater with respect to:
- (i) Recycle hydrogen ratio to feed,
 - (ii) Slurry/gas flow regime (i.e. maximum and minimum allowable liquid and gas velocities),
 - (iii) Residence time requirements,
 - (iv) Heat of reaction/solution,
 - (v) Outlet temperature, temperature "profile" requirements, physical properties vs. temperature and
 - (vi) Rate of heat transfer, and avoidance of coking.

2. Dependent Parameters

This information should be empirically determined by pilot plant, bench scale equipment or laboratory experiments.

- a. Expected overall yield structure: gases, liquids and residue composition
- b. Qualities of all products
- c. Performance of liquid products in burn tests
- d. Overall hydrogen consumption
- e. Operational flexibility and stability in coal preparation, reaction and all other individual (downstream) units
- f. Response of process (a-e above) to coal selected for the commercial plant
- g. Use of SRC II residue in gasifiers
- h. Chemical/physical and thermodynamic properties for design of unit processes and all process equipment
- i. Corrosivity and erosivity of process fluids at the conditions anticipated in the commercial design
- j. Generation of atmospheric and water pollution effluents.
- k. Health effects of SRC II materials

Some of these conditions and questions were addressable in the Ft. Lewis plant. Obviously, questions 2.c and g and to the complete extent, 2. h could not be directly answered by Ft. Lewis alone, but products from Ft. Lewis were made available for testing by others. Conceivably, the rest of the conditions could have been tested and the required data generated by the pilot plant but the program developed little information on a number of these specifics because of time and other constraints. The need to develop these data should be examined in light of the additional information now available to DOE from the Harmorville facility.

C. Factors Limiting Ft. Lewis Performance as a Process Development Data Gathering Facility

P & M has kept excellent logs and records of data and events at Ft. Lewis and has made a thorough reporting to DOE, and therefore to the public. However, because of the complexity of the operation, the large amount of information generated, and the subtlety of the technology, it has apparently been difficult for outside observers to appreciate the problems which might lead to exceptions and qualifications in data interpretation. The following problems could be expected to lead to shortcomings in the quality and usefulness of the data gathered at Ft. Lewis.

1. Program-related Factors

a. Tested Three Different Eastern Coals (TABLE 3)

Coals arrived in various shipments, and Ft. Lewis has had no deliberate homogenization system. This resulted in variability in the properties of the coal feed (as suggested by the record of variation in coal ash content), to which the cycling of stream compositions and yields is probably related.

b. Short Program

The program, in retrospect, appears to have been quite short considering the difficulties in testing three different coals and in dealing with a system which had just been revamped to SRC II. This, along with changes in the GMRC demonstration design basis, led to an evolutionary (boot-strap) approach to the experimental program planning.

TABLE 3

E L E M E N T A L C O A L A N A L Y S E S

Based on Averages of Daily Composites

(Weight % MF Coal)

<u>Run No.</u>	<u>C</u>	<u>H</u>	<u>N</u>	<u>S</u>	<u>O</u>	<u>ASH</u>
<u>KENTUCKY COAL</u>						
77SR-1	72.55	5.20	1.34	3.68	8.10	9.13
77SR-2	70.52	5.23	1.39	3.58	8.26	11.02
77SR-3	71.84	5.14	1.56	3.37	8.34	9.75
77SR-4	70.49	5.07	1.47	2.85	10.50	9.62
77SR-5	71.54	5.20	1.46	3.26	9.13	9.41
77SR-6	72.63	5.26	1.51	3.37	7.67	9.56
77SR-7	72.23	5.19	1.47	3.31	7.96	9.84
<u>ILLINOIS COAL</u>						
77SR-8	69.67	5.15	1.23	3.91	8.49	11.55
77SR-9	68.31	5.15	1.28	4.07	8.79	12.40
77SR-10	67.44	4.87	1.20	3.86	8.83	13.80
77SR-11	68.44	5.04	1.36	3.78	8.52	12.46
78SR-19	67.90	5.09	1.19	3.46	8.41	13.95
78SR-20	67.42	5.16	1.24	4.07	8.21	13.90
78SR-21	67.84	4.95	1.40	3.84	9.22	12.75
<u>PITT SEAM COAL</u>						
77SR-12	72.62	4.91	1.38	3.01	5.80	12.28
77SR-13	72.32	4.84	1.34	2.68	6.28	12.54
77SR-14	72.81	5.09	1.36	2.97	5.60	12.17
77SR-15	72.59	4.93	1.31	2.90	6.39	11.88
78SR-16	73.08	4.89	1.35	2.83	5.74	12.11
78SR-17	73.55	5.01	1.35	2.66	5.81	11.62
78SR-18	73.44	4.97	1.36	2.81	5.64	11.78

The program had originally been scheduled for a much shorter duration and less extensive testing. It was continued through several extensions requested by P & M. This month-to-month existence was a factor, in retrospect, contributing to some weaknesses in the program.

c. Original Design for SRC I

- (i) The Slurry/Coal Mixing system, which was adequate for SRC I where coal is mixed with solvent only, was a serious limitation in achieving high slurry concentration without continual operating problems in SRC II. Dilution with flush solvent was often necessary to maintain fluidity as well as to keep pumps operating. These facts led to uncertainties in relating the reaction mixture to the yield structure obtained. Coal slurry gel formation in the high-residence time blending unit limited the temperature of the slurry feed to an uneconomically low level for a commercial design. A pug mill system (as used at the Grand Forks ERC) is now being tested as a replacement, which will probably be a better blending option.
- (ii) The short-fall in hydrogen recycle capacity in the hydrogen system designed for SRC I (which uses far less hydrogen and operates with lower hydrogen partial pressure) led to the installation of the initially troublesome Naphtha Absorber. This Absorber is required to remove light hydrocarbons and thus insure an adequate hydrogen concentration in the recycle. The unit experienced foaming problems until the Naphtha absorption oil was replaced with

Middle Distillate.

- (iii) Vessel designs also limit the hydrogen partial pressure that can be tolerated (maximum, 1700 psig) so that runs at higher pressure could not be carried out in any case without complete re-design and replacement of these vessels.
- (iv) Many other problems stem simply from the fact that the facility and its design are old and intended for a process with a quite different arrangement of many internal flows.
- (v) It appears that the DOE had minimal active technical or expediting roles in the revamp to SRC II or in helping P & M to solve the early problems in SRC II operation.

d. Simultaneous Effort to Revamp for SRC I Filter Tests

The effort to install and prepare to run the Lummus de-ashing system and the EPRI filters tied down technical manpower and thwarted engineering record-keeping efforts (updating SRC II drawings and specifications, etc).

e. Limited Access to Harmarville Data

The P-99 program was carried out solely by Gulf under private sponsorship and was kept company confidential. The information was not made available to Ft. Lewis personnel until the results were firm, i.e., not until late in the Ft. Lewis program. This late transfer of information seems to have limited the access of the on-site Ft. Lewis staff to techniques and insights that might have helped design a stronger Ft. Lewis program and perform a more

acute data analysis. The (public) Ft. Lewis data base is, in hindsight, augmented by the (private) Harmarville data. GMRC (Denver), and not the Ft. Lewis staff or the DOE, had essential control of the direction of the Ft. Lewis program. They assumably carried out their planning with full knowledge of the Harmarville results.

f. No On-Line Distillation

The oversized Distillation system, designed for high rates of solvent recycle in SRC I, forced a campaign-continuous operation in Ft. Lewis to separate solvent fractions. Instead of measuring and being able to follow distillates accumulation in separate tanks, the operators had to rely for control and material balancing on level measurements of mixed solvents primarily in the Wash Solvent Accumulator. Significant accumulations or losses of mixed solvents in other vessels were also often involved in making a material balance. This could introduce more error than would on-line distillation with accumulation of pure solvent cuts.

g. Excessively Complex Routing in the Back End

The adapted piping and tankage system was eventually mastered by the operators, but since it was engineered for expediency, it did greatly increase the time and energy required to keep the plant on stream and under control.

h. Two Troublesome Vacuum Flash Systems

The Old Vacuum Flash System was designed for a low IOM and ash concentration residue and for higher SRC I flows. Though

usable when the New System was down, it did cause different process conditions and some apparent differences in yield structure. The New System was added to the plant, but had to be positioned offset on the structure, so consequently its gravity discharge pipe had a dog-leg configuration. Both systems have long down-legs that are subject to frequent blocking problems which cause system upsets.

Low Vacuum Bottoms fusion points must often be maintained (by diluting the Bottoms with Heavy Distillate) to keep these systems in operation. This leads to uncertainties in the Heavy Distillate make because a final reported material balance involves a "normalization" of fusion points to 350°F via a re-apportionment of Vacuum Bottoms and Heavy Distillate yields, in an attempt to put Heavy Distillate make on a comparable basis for different runs. The normalization formulas for this were developed from empirical data, using data for a single coal to characterize each Vacuum Flash system. Since the corrections are sometimes applied to coal/VF system combinations different from those of the formulas derivations, their application can be expected to introduce additional error. The magnitude of error is also probably proportional to the deviation of the fusion point from the 350°F normalization temperature. (see TABLE 4)

There is no doubt that a much better Vacuum Flash System could be designed. Such a system could employ a suitable pump for conveying the Bottoms out of the Flash Vessel instead of relying on a barometric leg. (One feasible technique is now being used

TABLE 4

VACUUM BOTTOMS YIELDS

Elementally Balanced
(wt % MAF Coal)

<u>RUN NO.</u>	<u>AVERAGE FUSION POINT, °F</u>	<u>VACUUM FLASH SYSTEM</u>	<u>YIELD (1) (at actual fusion pt.)</u>	<u>ADJUSTED (2) YIELD (@ 350°F)</u>
77SR-1	345	Old	31.8	31.4
77SR-2	310	Old	29.9	26.7
77SR-3	345	New	29.7	29.3
77SR-4	384	New	33.0	36.0
77SR-5	336	New	41.1	39.7
77SR-6	394	New	41.6	46.3
77SR-7	362	New	31.7	32.7
77SR-8	276	New	28.2	26.3
77SR-9	267	New	30.5	28.0
77SR-10	224	Old	37.7	33.3
77SR-11	307	New	39.3	37.8
77SR-12	387	New	46.6	48.1
77SR-13	329	New	43.0	42.2
77SR-14	316	New	37.5	36.4
77SR-15	330	New	43.0	42.3
78SR-16	329	New	44.6	42.3
78SR-17	317	New	50.1	48.7
78SR-18	295	New	50.2	48.0
78SR-19	246	New	42.1	38.2
78SR-20	240	New	40.9	36.9
78SR-21	291	Old	35.4	29.8

(1) Yields listed in this table are the organic content of the Vacuum Flash Drum Bottoms stream divided by the organic content of the coal, times 100.

(2) Adjustment of the VB yield to a 350°F fusion point is based on correlations developed at Ft. Lewis.

by Texaco to feed melted Vacuum Bottoms to their test gasifier.) Since the bottoms would have to be pumped to a gasifier in any case, the development and testing of techniques for this service should be given high priority.

i. Stretford System

Attention paid by the operators and process staff to the troublesome Stretford off-gas desulfurization unit, with its problems of corrosion and molten sulfur handling, seems to have significantly distracted manpower from the general effort. The unit's problems stem partly from application to a gas stream with a relatively high H_2S concentration, whereas the Stretford process is generally most useful for treating dilute H_2S streams. This was compounded by lack of technical support and in some cases, obstruction of (P & M's attempts to use the services of other experts) by the particular vendor of the Ft. Lewis Stretford unit.

The plant is geographically remote from the vendors of much of the plant's equipment, and from their service organizations. Thus, expediting problems could generally have been expected in obtaining parts and service. The DOE seems to have lent little support in these matters. Nonetheless, SD believes a trouble-free sulfur removal unit could be designed using Stretford or one of several alternatives for this plant, or for the demonstration plant.

j. Slurry Flow Meters

The lack of reliable flow meters for use with concentrated and viscous slurries is a general problem of coal liquefaction plants. This led to ad hoc indirect techniques for metering and controlling slurry flows (such as following Slurry Charge Pump speed and Mix Tank level to calibrate the wavering and inaccurate readings of the recycle slurry flow meter). This continued until the end of the program when some success was achieved with the use of a quadrant-edged orifice meter. Continued operation of such a large scale plant could probably be justified simply to develop a better slurry flow meter and other specialized instrumentation required for the services encountered in this process.

k. Hydrogen Compressor

A substantial leak in the New Recycle Hydrogen Compressor led to measurement of five separate gas streams to obtain a gas material balance.

2. Other Factors

a. Water Quench

A heat exchanger cooler was originally installed at Ft. Lewis, but was discarded when it failed in initial operation. Since this system was revamped from SRC I operation, there were temperature limitations which prevented the use of a hot Dissolver effluent separation. The amount of circulating water resulting from the Water Quench (now used to reduce the high pressure dissolver effluent temperature by 100^oF) makes accounting for the reaction-generated water almost impossible in the material balance.

The key problem is interface control of the large RPWT and metering erratic water flow to waste treatment. Losses in the material balance are lumped into "water plus losses", which clearly also includes C_5 and C_6 losses in the off gases and vents.

In P-99 (which operates with a hot Dissolver effluent quench), a small metered quantity of water is also introduced, but only to dissolve ammonium chloride in the Dissolver flash overheads to prevent plugging of heat exchangers, etc. It does not lead to the inaccuracies which the Water Quench introduces at Ft. Lewis.

b. No Analysis for C_5 and C_6

P & M staff maintains they have made a systematic search for an automatic on-line chromatograph that will analyze for C_5 and C_6 in the gas stream, either combined with the other gas analyses or separate, but have been unsuccessful in finding a suitable device. Also, they claim that little or no condensate (presumably containing C_5 - C_6) is found in the cold traps ahead of the existing chromatograph. Forcing of the Naphtha production to obtain the carbon balance (the discrepancy is partly attributed to C_5 - C_6 losses) is a source of great inaccuracy and unreliability in a number of runs (See TABLE 9). No correlation or characterization factor was developed by P & M to account for these products directly in a material balance.

c. HD Losses from the Sandvik Belt

Organics losses by evaporation from the Sandvik Belt were long recognized to occur. This received some attention, but no reliable collection or metering of these losses were achieved. One measurement was made of organics in the "whistle tube" (vent stack) from which some of the losses may vent. This measurement could not be considered definitive. These losses can be estimated by a simple vapor pressure calculation, which indicates that substantial amounts of the Heavy Distillate in the Vacuum Bottoms could be lost under normal conditions.

d. Analyses of Coal Feed

Proximate and ultimate analyses of coal feed are scant in the record of runs. Ash content, which is regularly measured, may not be a sufficient parameter to alone correlate the variability of coal behavior in the process. Stockpiling of coal due to the miner's strike and the subsequent problems of managing the inventory probably led to a significant variation in the state of oxidative aging of the coal feed.

e. Coal Feed Rate Measurement

Coal feed rate is measured in a time-consuming and essentially manual mode (See IV.D.1.a.). The measurements are then used to calibrate the continuous reading of the Weigh Belt Feeder. It is possible that this technique may introduce some error, as contrasted with, say, measuring the total feed in a properly designed weigh cell. Strain gauges are in place on the support legs of the Crushed Coal Surge Bin (5 tons) which is refilled every several hours, but these have had many problems and are considered inaccurate.

f. Engineering Records at Ft. Lewis

Because of manpower limitations, engineering record-keeping at the Ft. Lewis plant has been generally poor. Engineering standards introduced by Stearns-Roger make the situation more difficult. The equipment numbering convention is cumbersome and poorly systematized. SRC I process flowsheets did not show equipment specifications and were rather random as to degree of detail. SRC II process flowsheets, P & I diagrams, equipment specifications and drawings were either non-existent or not kept up to date. Piping isometrics were often without legends, orientations or location references. There were no equipment arrangement or overall elevation drawings, as one normally would expect for a chemical plant or refinery, showing the location (outline) of each major item of equipment. The "plot plan" available only outlines major process blocks. All of this makes process analysis and conceptualization of future revamps much more difficult.

g. Stripper Bottoms Hydrogen Donor Properties

The average solvent composition (MD/HD content and ratio) seems to vary substantially among material balance runs, but is more even, through cycling, during runs. Harmarville reconstitutes a recycle stream from a concentrated recycle slurry plus recycle solvent from fractionation, which gives P-99 more independent control of the level and regularity of the solvent content of the feed slurry.

Hydrogen donor properties of the recycle stripper bottoms are not measured and reported. Some techniques are available, but

none of these are universally accepted measures of solvent donor capability and would probably be suspect as applied to the complex mixtures involved at Ft. Lewis. Analyses of distillate fractions by boiling point ranges alone will not necessarily describe the behavior of the solvent. However, donor solvent characterizations, had any at all been made, might have been a clue to the apparent varying performance of the reaction system.

h. Flush Solvent Variation and Degradation

The Ft. Lewis Slurry Preparation System and Slurry Charge pumps require a varying, but usually quite high, rate of seal flush injection. The rate generally varies (among the runs) between 40% and 50% of total liquid distillate production and it can cycle continually during and between runs. The volume and boiling range of solvent in the Stripper Bottoms recycled to slurry preparation also varies significantly. Nonetheless, it appears that the effect of this stream property is not closely followed nor seriously considered by P & M as having an impact on the material balance or reaction yield structure. P & M contends that conversion of seal flush to lighter products was probably negligible, using as evidence their study which followed gas make only during process idling (no net coal feed, process temperature lowered and full recycles continued). This idling test is probably not relevant to answering the question of what proportion per reactor pass of the two substantial solvent streams (seal flush and recycle solvent) is converted to lighter products and residue, because it was conducted at a lower temperature than normal and without "fresh" coal ash (catalyst) addition.

Work (which was not completely defined in Gulf's report) was done at Harmarville with autoclave holding of flush solvent at SRC II reaction conditions. However, the flush solvent for Harmarville is said to be petroleum derived and is probably highly paraffinic. This work indicated that over two-thirds of flush solvent per pass can convert to lighter materials and residue. Harmarville uses this data to make an estimated correction for flush solvent degradation in their material balance, but it probably cannot be directly applied to Ft. Lewis due to the different nature of the solvent.

In any case, the proportions of these circulating streams are likely to be different in a commercial design than at Ft. Lewis and thus one is left with the question of how such a difference might effect the observed yield. The conversion of recycle streams at process conditions should receive greater study in any future work on SRC II.

D. Adequacy and Reliability of Equipment, Instrumentation and
Procedures of Process Development Data Aquisition

1. Individual Plant Systems

Many of the individual plant systems, though operable, appear to be not fully equal to the task of obtaining process design data. Though SD has not made a complete review of plant instrumentation, the study did focus on the functioning of some critical instruments such as the coal feeder, gas chromatograph and certain slurry flow meters.

a. Coal Preparation and Feeding System

Coal is not deliberately homogenized, either by means of silo homogenization or by organized stacking and reclaiming techniques. This has often led to swings of over 25% in ash concentration of daily averaged feed coal samples in as little as two days of running. If these large variations occur in analyses of daily composites, one could imagine much larger hourly variations. Similarly high weekly variations could be seen in some "forms of sulfur" analyses (organic sulfur levels especially). Though organic sulfur analyses are not entirely reliable, this does indicate the possibility that the coal composition and ash quality is also allowed to swing widely during, between, and among material balance runs. All of these factors related to coal quality could be effecting yield structure and operability to a greater extent than has been recognized. Of course, a commercial plant design would also have to face variability in coal quality, but SD presumes that if this is determined to be important, better homogenization could be achieved.

The Gravimetric Feeder (weigh belt) is oversized for this service and though it runs at a steady rate, it has a consistently unreliable reading, which leads P & M to perform bi-hourly bucket and scale measurement of the crushed coal feed to the pulverizer. This technique is believed by P & M to present no more than a few percent error plus or minus. The coal immediately enters the blending system with little holdup in the pulverizer and collector, thus the procedure seems basically adequate for metering the feed. The gravimetric feeding is common in coal conversion pilot plants; however, a more accurate and reliable method would be a properly designed weigh-cell charging of the coal.

SD has not had time nor appropriate personnel available during this period to definitively ascertain the reliability of the "quick ash" analysis used by the P & M lab.

b. Slurry Blend System

P & M wanted to run coal blending at 450°F. It is presumed that this condition would more closely reflect thermodynamically efficient conditions for commercial plant design than the 350°F at which they were forced to operate. The two-tank system, which was evolved to replace the SRC I Solids Eductor, itself limits the plant in the feed temperature/coal concentration range that can be explored. It has a rather high residence time, relies on tank agitation to maintain the slurry, and presents opportunities for solids from heavy slurries to hang up on the vessel walls. At high slurry concentrations and at temperatures of over 400°F and high residence time, there is a tendency to form an extremely

high viscosity "gel" state in the slurry, which makes it difficult to operate the agitators and keep the material fluid. Besides operating at low temperature, it was often necessary to introduce additional seal flush to the circulation pumps to reduce slurry viscosity to an operable level.

The total flush solvent consumption in both circulation and preheater is normally 300-500 lb/hr and occasionally has been increased to 800 lb/hr. The charge pumps generally require 200-500 lb/hr of this flow. This flow has been varied widely when problems arose in the pumps or blend system.

A slight vacuum is drawn on the system to keep organic vapors out of the coal introduction equipment. Resulting organics losses are probably small, but are not accounted for.

Recycle slurry feed to the Blend System was not measured directly in the SRC II operation because of the inaccuracy of slurry flow meters available, a general problem in coal conversion pilot plants. The flow meter on the Stripper Bottoms (recycle slurry to blending) is consistently inaccurate. P & M instituted a procedure of keeping the Blend Tank level constant by adjusting the speed of the Charge Pumps which remove material from the Blend System. This procedure is used to obtain the "true" rate of slurry flow, which is then used to reset the flow controller which operates on the signal from the slurry flow meter. Although this appears to be an expeditious procedure under the given conditions, it may be

inadequate to accurately follow the feed composition and feed rate to the reaction, since it only measures the slurry after blending, and the slurry make-up obtained by difference. The widely used approach of measuring the concentration of a "component" of a slurry (such as ash) which may appear in different concentration in the two streams is not applicable here because the slurry and coal have nearly the same ash concentrations, and other components are also indistinguishable by available analyses.

The introduction of a quadrant-edged orifice meter, which P & M recently tested with some success, and/or of a pug mill mixing system could eliminate much of the inherent error in this area of the plant.

c. Preheater/Dissolver

i. Preheater

The Preheater has never coked, even under conditions that would be expected to lead to severe coking, but the unit is over-designed for this service. It is not known whether a unit properly sized for the heat transfer service or one with a smaller tube diameter, such as P & M is planning to install, would be as immune to coking. Few samples have been taken of the Preheater effluent since this is a hazardous procedure at high temperature and pressure. Thus, the separation of conversion and hydrogenation effects in this unit is difficult; and conclusions as to its functioning are merged with the operation of the Dissolver.

ii. Dissolver

The usable volume of slurry plus gas bubbles of the Dissolver is measured only during shutdown, by filling the vessel to the normal operating half-level with water, and measuring the volume added. Only small changes in this volume are observed over time, indicating little coke build-up during SRC II operation. However, as another matter, the concentration of suspended settled mineral residue solids is not reliably measured and this may effect the degree of catalysis in the unit. Solids build-up could conceivably be estimated by measuring pressure drop across the unit at low gas flow while on-line and hot, or during shut-down by isolating the unit and collecting solids, or by utilizing nuclear devices on-line. P & M has attempted to estimate solids hold-up by measuring the Dissolver pressure drop while hot and on-line at normal operating gas flowrates, but with little success. However, high gas flow rates lead to uncertainty by introducing the complicating factor of an unknown degree of gas bubble bed expansion.

The two upper hydrogen quench injection points (which give isothermal operation) were used during the earlier runs. P & M switched to the lowest sparger only during the latter runs, with a significant Dissolver temperature gradient resulting for these runs. This inconsistency between the runs makes the outlet Dissolver temperature a possibly unreliable single parameter for comparing the runs.

The exit hydrogen partial pressure, which can vary with the gas production as well as with inlet hydrogen concentration and rate and overall system pressure, is only indirectly measured via readings of the vapor composition from the HP Flash Drum. This primary variable is not reported, discussed or correlated in P & M publications. Instead, they use inlet-partial pressure as a correlating parameter, which introduces a degree of uncertainty in comparing run results.

d. Dissolver Quench

The Air Cooled Heat Exchanger, which was the original Dissolver effluent quench device, failed in early operation and has never been replaced. The subsequent use of a circulating water quench introduces a high degree of data unreliability in making a water balance and overall material balance, as discussed in several other sections of this report.

e. Flash Separations

SD has not ascertained whether the sequence of flash steps is optimum for the condensation, separation and removal of water so that it will not carry out emulsified or dissolved organics. A temperature of 750°C was selected for the High Pressure Flash Drum operation because of limitations on the design temperature of vessels revamped from SRC I.

There is no measure of C₅-C₆ sent to gas treating from this area, nor from the No. 3 Separator vent to Flare. This introduces uncertainty in the carbon elemental balance.

f. Stripper

This unit seems to be a quite critical item in the overall process, yet there is little independent control of the Bottoms composition. The feed to the Stripper is poorly measured, in general, and this is one of the reasons the unit operation would be difficult to control. The nature of the Stripper itself seems to exacerbate variation and even to impose cycling in the concentration and makeup of the solvents recycled to the slurry preparation. Since it also feeds Bottoms directly to Vacuum Flash, these swings impose a fluctuation in the amount and composition of the overheads from the Vacuum Flash, an otherwise troublesome unit.

To the credit of the operation, however, P & M does make frequent (bi-hourly) measurements of SRC, IOM and ash content of the Stripper Bottoms, mostly for control of the mechanics of coal/slurry mixing. Frequent measurements are also made of the Distillate content of the Stripper Bottoms. This information can be useful in interpreting MBR and production running results.

In general, this unit should be re-examined for any future program of SRC II running at Ft. Lewis.

g. Vacuum Flash Units/Solidification

The nature of these systems forces the operators to run at low and variable Vacuum Bottoms fusion points, which leaves a large and variable amount of Heavy Distillate in with the residue. There is then an opportunity for the solvent to be lost by evaporation in the front end of the subsequent solidification step. The

degree to which coking in the Flash units effects product distribution is not well known, but it could be a factor in the variability of run results.

Variations in the feed to these units, identified above as a partial cause of their uneven operation, can then be reflected as cycling of the composition of the flush solvent recycled to the Coal/Slurry Blending system. It should be noted that the rate of Heavy Distillate (mostly Vacuum Flash overhead) recycled to the seal flushes alone is usually several times the rate of Heavy Distillate production. A dynamic analysis of this system is probably required to verify the potential for kinetic and yield variation, especially as a function of residence time.

Most of the "ash balance" problems of the plant probably originate in the operations of the Vacuum Flash and Solidifier. Clearly, the two units operate quite differently from each other, and their alternate use makes data interpretation, especially comparisons, of the production of Vacuum Bottoms much more difficult.

h. Mixed and Wet Solvents Collection

It was concluded from a preliminary error bounds analysis by P & M that about 90% of the solvent is generally collected in the Wash Solvent Accumulator and that, therefore, about the same proportion of the error in determining yields is attributable to level and concentration measurements of Distillates collected in this unit in most runs.

The vents, albeit small, from the Vacuum Flash Condensate System can be sources of organics loss.

The purge of waste water from the RPWT is a potential source of major organics loss as an emulsion in the waste water.

P & M claims that emulsion formation is not significant.

This contention, in SD's view, is not proven. A large volume of Heavy Distillate has been recovered from the Surge Reservoir. Although this has gone far to rationalize the "loss of Heavy Distillate" it clouds the results of those MBR's that might have contributed to this loss.

i. Distillation and Distilled Solvents Collection

The intermittent campaign-continuous operation of the existing, oversized Distillation system, made necessary by the lack of an on-line Distillation system, is undoubtedly a large source of error during material balance runs. It forces (previously mentioned) indirect measure, by simultaneous level and composition readings, of Distillate production. This presents another set of level and analytical errors which are multiplied and do not necessarily cancel as would level readings of different distilled fractions in separate tanks.

Somewhat different combinations of solvent fractions are used as seal flush during various times of operation because of the flexible back-end operational choices available, especially in MBR vs. non MBR mode. The variation in solvent donor properties and reaction decomposition of these solvents may be a significant factor in variation of results.

The overall back end system has quite complex routing with many available options. Though the system is flexible, it seems to invite errors in routing of fluids, which may be a hidden source of some variations in results.

j. Hydrogen Recycle System

The Naphtha Absorption system, though it meets the objective of making higher purity hydrogen recycle available in SRC II, does not present a means of separately collecting and recording the production of C_5 and C_6 , which are not well analyzed by the chromatograph.

2. General Comments on Operation

a. Operational Stability

During the period of SRC II operations, it was demonstrated that the process as utilized is extremely stable and had little or no tendency to foul, coke or present uncontrollable reaction conditions. Occurrences that have caused major upsets without subsequent catastrophe include loss of Preheater hydrogen, total loss of hydrogen to the Preheater and Dissolver, loss of liquid flow to the Preheater and total loss of water quench. Additionally, the firing to the Preheater has been lost on a number of occasions without serious recovery problems. Chronic Coal Feed and/or Slurry Mix problems led to repeated interruptions of the reaction section and other front end equipment and downstream units up to and including the Vacuum Flash System. Aside from plugging in the Vacuum Flash equipment, no adverse effects were noted during plant recovery.

Faulty check valve operation allowed slurry to back up into the hydrogen quench lines to the Dissolver when both Recycle Compressors were lost and resulted in rapid plugging of these lines. These lines were rather quickly and easily cleared. Installation of improved check valves was carried out and a high pressure surge bottle was connected to the header to avoid recurrence.

The Slurry Preheater would be expected to be the unit most sensitive to major operating upsets and particularly to total loss of liquid flow. In the event of major upset or interruption of front end operations, operating practices include rapid flushing of the heater coil with 23-30 gpm of solvent to avoid coil fouling or coking. However, during a major power failure in 1977, the emergency generator could not be started so that the Preheater coil was left full of slurry mixture for more than an hour before solvent flushing could be initiated. During this time, although the Preheater firing had immediately ceased with the loss of power, the unit remained hot since cooling down was only by air convection. The coil was cleared without difficulty and apparently without coke formation. This clearly demonstrates the excellent recovery ability of the SRC II system and provides an answer to fears that the system operates "near coking" conditions.

Frequent problems were experienced with the Vacuum Flash Equipment. Most of these problems related to plugging, particularly during start-up operations. Interruption of Vacuum Flash operations also necessitated stopping coal feed to the front end, usually without serious recovery or re-start problems.

The excellent on-stream factor achieved in this pilot plant operation offers good assurance that the mechanical availability of a demonstration plant would be acceptable. Advances through studies now proceeding on higher temperature (450°F) slurry mix technology and on continuous Vacuum Flash operations, could result in a further significant gain in on-stream factor.

b. Equipment Flexibility

The SRC II system as operated at Ft. Lewis is the result of imposition specific SRC II process and mechanical requirements on an existing SRC I plant. Thus, certain equipment such as the Slurry Air Cooled Exchanger, Filter Feed Flash Vessel and Heater, Old Vacuum Flash Drum and the Distillation System are oversized for SRC II operations. Nonetheless, the Ft. Lewis team has demonstrated good industry and ingenuity in arranging and using flow alternatives throughout the SRC II operations to achieve an excellent on-stream factor. It was learned quite early in the SRC II operations that establishment of suitable pressure levels in the No. 3 Condensate Separator and the Stripper permitted running the SRC II processing scheme with a minimum number of pumps. This greatly simplified the process route and resulted in a major reduction in seal flush solvent requirements. It was further discovered that the New Vacuum Flash System required a Circulating Pump only during start-up.

Once start-up is achieved, the basic SRC II system from the coal preparation area through the Vacuum Flash System is remarkably simple with minimal rotating equipment - a highly desirable feature in any coal liquefaction design.

c. Process Oscillation

Reasonably constant stream composition of the principal process streams of the SRC II operation up to and including the Vacuum Flash system was not achieved. Variations in stream compositions occurred even though principal operating parameters were maintained constant. The process as installed at Ft. Lewis appears to be susceptible to constant cycling of stream compositions, which may be strongly aggravated by the heavy recycle of Stripper Bottoms to the Slurry Preparation area where coal and a small quantity of solvent are mixed, and by the marked variation in the ash content of the feed coal. (See Figure 11.)

The use of an additional surge vessel on the recycle slurry stream, with controlled dilution by a fractionated solvent (Heavy Distillate) to establish a fairly constant feed to the Preheater (as in P-99) may be necessary at the expense of more complexity. The Stripper is a key item of equipment in this process, furnishing a majority of the pasting medium to Coal Mixing and feeding the Vacuum Flash System. Any variations in the composition of the Stripper Bottoms are quickly reflected in the slurry preparation area and in the Vacuum Flash equipment.

The fusion points of the Vacuum Bottoms product have not been controllable. Even under steady operating conditions, wide variations in fusion point occur on a daily basis resulting in continuously uneven removal of Heavy Distillate from the bottoms.

d. Physical Properties of Distillates

The physical property data on the SRC II distillates reflect wide variations in specific gravity, viscosity, flash point and pour point for each of the coals studied. These variations are particularly obvious for the Heavy Distillate fraction where the viscosity range exceeded 100% of the minimum value measured on each of the coals tested (see TABLE 5). Although these swings in physical properties could not be correlated with the operating parameters maintained for the given test runs, it is believed that they relate to other process oscillations as described above. Further, in the case where viscosity of Heavy Distillate is doubled in value from one run to another on the same coal, it suggests that selected operating parameters may strongly influence chemical composition and molecular weight.

TABLE 5A

PROPERTIES OF NAPHTHA

<u>COAL FEED</u>	<u>RUN NO.</u>	<u>VISCOSITY (100°F) cST</u>	<u>SPECIFIC GRAVITY</u>	<u>FLASH POINT °F</u>
KENTUCKY	77SR-3	--	0.840	--
"	77SR-4	0.922	0.843	< 82
"	77SR-5	1.016	0.852	< 74
"	77SR-6	0.933	0.850	4
"	77SR-7	0.901	0.841	< 30
ILLINOIS	77SR-8	0.944	--	2
"	77SR-9	--	--	--
"	77SR-10	0.718	0.775	< 50
"	77SR-11	0.737	0.800	< 58
"	78SR-19	0.755	0.978	--
"	78SR-20	0.722	0.800	--
"	78SR-21	--	--	--
PITTSEAM	77SR-12	0.674	0.799	47
"	77SR-13	0.695	0.797	50
"	77SR-14	0.722	0.811	45
"	77SR-15	0.717	0.803	46
"	78SR-16	0.768	0.819	-35
"	78SR-17	0.751	0.820	75
"	78SR-18	0.716	0.811	30

TABLE 5B

PROPERTIES OF MIDDLE DISTILLATE

<u>COAL FEED</u>	<u>RUN NO.</u>	<u>VISCOSITY (100°F) cST</u>	<u>SPECIFIC GRAVITY</u>	<u>POUR POINT °F</u>
KENTUCKY	77SR-3	--	0.977	--
"	77SR-4	3.63	0.980	-25
"	77SR-5	3.60	0.979	-25
"	77SR-6	3.64	0.982	-29
"	77SR-7	3.26	0.976	-35
ILLINOIS	77SR-8	3.40	--	-48
"	77SR-9	--	--	--
"	77SR-10	3.38	0.982	-40
"	77SR-11	3.09	0.974	-50
"	78SR-19	3.207	0.809	-55
"	78SR-20	2.915	0.972	-55
"	78SR-21	--	--	--
PITTSEAM	77SR-12	2.73	0.971	-45
"	77SR-13	2.72	0.968	-55
"	77SR-14	3.12	0.980	-40
"	77SR-15	3.60	0.980	-30
"	78SR-16	2.56	0.964	-55
"	78SR-17	2.79	0.967	-54
"	78SR-18	2.74	0.965	-60

TABLE 5C

PROPERTIES OF HEAVY DISTILLATE

<u>COAL FEED</u>	<u>RUN NO.</u>	<u>VISCOSITY</u> <u>(100°F) cST</u>	<u>SPECIFIC</u> <u>GRAVITY</u>	<u>POUR POINT</u> <u>°F</u>
KENTUCKY	77SR-1	--	--	--
"	77SR-2	--	--	--
"	77SR-3	--	1.075	--
"	77SR-4	74.6	1.077	45
"	77SR-5	120.6	1.092	35
"	77SR-6	124.8	1.094	30
"	77SR-7	150.8	1.064	40
ILLINOIS	77SR-8	41.5	--	14
"	77SR-9	--	--	--
"	77SR-10	64.7	1.087	-3
"	77SR-11	85.0	1.087	20
"	78SR-19	26.8	1.075	25
"	78SR-20	27.6	1.071	32
"	78SR-21	27.4	1.076	--
PITTSEAM	77SR-12	26.8	1.075	5
"	77SR-13	27.6	1.071	10
"	77SR-14	27.4	1.076	25
"	77SR-15	32.8	1.069	35
"	78SR-16	27.0	1.052	13
"	78SR-17	83.9	1.092	35
"	78SR-18	33.2	1.089	5

E. Material Balance Procedures

The procedures used at Ft. Lewis to measure and calculate consumptions and yields are outlined in this section. A step-by-step illustration of these calculations is included in Appendix C.

1. General Description

Each material balance run (MBR) was scheduled to continue for seventy two hours at targeted reaction conditions of pressure, temperature, coal concentration, and total feed rate. In most of the MBRs hydrogen purity in the recycle gas was controlled to provide a hydrogen partial pressure in the range of 1700-1800 psig. Recycle gas rate was targeted to provide approximately 5.5 lbs. of hydrogen per 100 lbs. of total slurry feed to reaction. Recycle slurry composition and flow rate were adjusted as well as could be done within the limitations of the plant equipment and instrumentation.

2. Raw Yield Data

Yields based on actual measurements are reported as "Raw Yield Data" in Tables 16, 17 and 18 in Appendix D. The yields were obtained as described below:

- a. Coal feed rate was measured once every two hours by an operator who would briefly divert the entire feed to a drum and measure the weight collected over a three minute time interval. (The gravimetric feeder proved adequate to maintain a reasonably constant coal feed rate, but the readings from this device were not sufficiently accurate since feed rates ranged from only 20% to 32% of the feeder capacity of 8,000 lbs./hr.) Composition of the coal was based on laboratory analyses of daily composite samples.

- b. Gas Yield and Hydrogen Consumption were determined by material balance around the reaction and flash section of the plant. Since there was no flowmeter reading the total hydrogen recycle gas to the reaction section, it was necessary to add readings for each of the five branches of the recycle gas: gas to the Preheater, gas to each of the three Dissolver Quench Spargers, and gas purge to instruments (a very minor stream). There were only two gas streams leaving this section of the plant: the gas from the Recycle Condensate Separator, and the vent from the No. 3 Separator. Compositions of all streams were determined by on-line gas chromatographs (G.C.'s). Flow rates and composition were computed, logged and averaged automatically by the computerized data acquisition system.
- c. Liquid Products were collected as one mixed stream in the Wash Solvent Accumulator. Tank level and composition (according to fractions characterized by boiling point ranges) were measured at the start and end of the material balance run. To correct for in-plant inventory changes these same measurements were made on a number of other vessels as illustrated in Table 12 in Appendix C.
- d. Vacuum Bottoms (VB) production was measured by weighing the solidified product removed from the Cooling Belt. Samples were analyzed for C, H, N, S and ash. Oxygen was obtained as the difference between other components and the total. IOM content was calculated based on the measured ratio of IOM to ash in the Stripper Bottoms (feed to the Vacuum Flash). SRC

content (actually SRC plus any Heavy Distillate remaining in the VB) was calculated as the difference between the total and the ash plus IOM. A correction is made later to adjust the Heavy Distillate (HD) content of the Vacuum Bottoms to correspond to a 350°F VB fusion point. This is done by transferring a portion of the reported VB to HD production if the fusion point is below 350°F, or by transferring a portion of the reported HD to the VB if the VB fusion point is above 350°F. This procedure is described in paragraph 5 below.

- e. Unaccounted losses - The difference between the net coal and hydrogen feeds and the measured gas, liquid and solid products is termed "water plus losses" (see Raw Yield Data Tables in Appendix D). This figure includes products which are simply not measured (water, impurities contained in the water, C₅ and C₆ hydrocarbons in the gaseous products, ammonia in the gaseous products), weighing errors, and any real losses. The total of "water plus losses" is estimated at 7-9% of the moisture and ash free coal feed.

3. Changes in Procedures - Changes made during the test program effected the values of certain Raw Yield data. These changes include the following:

- a. Measurements of hydrogen concentration by the on-line GC's were corrected for the hydrogen non-linear behavior beginning with MBR 77SR-12. (In the first eleven runs, a linear response had been assumed.)
- b. Vacuum Bottoms Weighings - Beginning with the thirteenth run, solidified Vacuum Bottoms were collected in trucks and weighed. Prior to this VB was conveyed into drums which were individually weighed.

4. Elementally Balanced Yields

Forcing techniques were developed by the Ft. Lewis staff to obtain material balance closure and elemental balance between products and feeds. Significant adjustments were made - using these somewhat arbitrary procedures - to the measured hydrogen consumptions and to the measured yields of Vacuum Bottoms (i.e., SRC and IOM) and of Naphtha. In fact, the elementally balanced values of hydrogen consumption and yields of Naphtha, water, ammonia and hydrogen sulfide are simply numbers calculated to achieve closure of the elemental balances for hydrogen, carbon, oxygen, nitrogen and sulfur, respectively. The forcing steps are described below and are illustrated in the tables immediately following:

- a. Calculate the ash recovery in the Vacuum Bottoms. (It is assumed that all ash in the coal feed leaves with the Vacuum Bottoms.) Ash content in the Vacuum Bottoms is simply the average ash content, as determined by the Ft. Lewis "quick ash" analysis, multiplied by the average production rate of Vacuum Bottoms. Ash content in the coal feed is simply the average ash concentration in the coal feed, as determined by the ASTM method, multiplied by the average coal feed rate.
- b. Force the VB production to obtain an ash balance. (The adjusted values are equal to measured VB divided by fractional ash recovery.) Refer to the following Table, Step 1.
- c. Force the Naphtha production to obtain a carbon balance. (The Naphtha production is simply set in this step to provide sufficient carbon such that carbon contained in all products equals carbon contained in the coal feed.) Refer to the following Table, Step 2.

- d. Calculate ammonia production to obtain a nitrogen balance.
(Ammonia production was not measured.) Refer to the following Table, Step 3.
- e. Force hydrogen sulfide production to obtain a sulfur balance. (Considerable sulfur may actually leave with the water purge, and be reported in the "water plus losses" term. This is not considered in this procedure.) Refer to the following Table, Step 3.
- f. Calculate a water make to obtain an oxygen balance. (As explained previously, water production is not measured.) Refer to the following Table, Step 4.
- g. Calculate hydrogen consumption to obtain a hydrogen balance. (Hydrogen consumption is set equal to total hydrogen content of all products less hydrogen content of the coal feed.) Refer to the following Table, Step 5.

The final step, adjustment of Vacuum Bottoms yield to 350°F fusion point, is described and illustrated in paragraph 5 below.

5. Heavy Distillate Content of Vacuum Bottoms

Vacuum Flash temperatures varied considerably throughout the Ft. Lewis SRC-II program. Equations were developed relating Heavy Distillate retention to fusion point of the Vacuum Bottoms. These equations were then used to estimate the HD and VB yields which would have been obtained had the Vacuum Flash systems consistently produced a 350°F fusion point bottoms product.

MATERIAL BALANCE RUN 78SR-19

Units: lb/hr

Raw Yield Data (Refer to Appendix C)

	<u>C</u>	<u>H</u>	<u>N</u>	<u>S</u>	<u>O</u>	<u>ASH</u>	<u>TOTAL</u>
MF COAL	1666.3	124.9	29.2	84.9	206.4	342.3	2454.0
H ₂		-93.7					-93.7
C ₁	89.8	30.0					119.8
C ₂	58.6	14.7					73.3
C ₃	47.7	10.6					58.3
C ₄	16.2	3.4					19.6
CO	-2.9				-3.9		-6.8
CO ₂	11.8				31.6		43.4
H ₂ S		5.8		93.5			99.3
NH ₃ ⁽¹⁾							
H ₂ O ⁽¹⁾							
Naphtha	123.2	18.2	0.8	0.0	5.2		147.4
MD	308.7	33.1	3.1	0.4	18.9		364.2
HD	222.0	18.7	3.1	1.3	2.4		247.5
VB	<u>713.2</u>	<u>46.4</u>	<u>14.3</u>	<u>30.9</u>	<u>-7.7</u> ⁽²⁾	<u>306.9</u>	<u>1104.0</u>
TOTAL	1588.3	87.2	21.3	126.1	46.5	306.9	2176.3

(1) Not measured.

(2) Negative value of O in VB indicates sampling/analysis inaccuracy.

MATERIAL BALANCE RUN 78SR-19

Units: lb/hr

Step 1: Force VB to balance ash

	C	H	N	S	O	ASH	TOTAL
MF COAL	1666.3	124.9	29.2	84.9	206.4	342.3	2454.0
H ₂		-93.7					-93.7
C ₁	89.8	30.0					119.8
C ₂	58.6	14.7					73.3
C ₃	47.7	10.6					58.3
C ₄	16.2	3.4					19.6
CO	-2.9				-3.9		-6.8
CO ₂	11.8				31.6		43.4
H ₂ S		5.8		93.5			99.3
NH ₃							
H ₂ O							
Naphtha	123.2	18.2	0.8	0.0	5.2		147.4
MD	308.7	33.1	3.1	0.4	18.9		364.2
HD	222.0	18.7	3.1	1.3	2.4		247.5
	795.4	51.7	16.0	34.5	-8.6	342.3	1231.3
VB	<u>713.2</u>	<u>46.4</u>	<u>14.3</u>	<u>30.9</u>	<u>-7.7</u>	<u>306.9</u>	<u>1104.0</u>
	1670.5	92.5	23.0	129.7	45.6	342.3	2303.6
TOTAL	1588.3	87.2	21.3	126.1	46.5	306.9	2176.3

MATERIAL BALANCE RUN 78SR-19

Units: lb/hr

Step 2: Force Naphtha to balance carbon

	C	H	N	S	O	ASH	TOTAL
MF COAL	1666.3	124.9	29.2	84.9	206.4	342.3	2454.0
H ₂		-93.7					-93.7
C ₁	89.8	30.0					119.8
C ₂	58.6	14.7					73.3
C ₃	47.7	10.6					58.3
C ₄	16.2	3.4					19.6
CO	-2.9				-3.9		-6.8
CO ₂	11.8				31.6		43.4
H ₂ S		5.8		93.5			99.3
NH ₃							
H ₂ O							
	118.9	17.6	0.7		5.1		142.3
Naphtha	123.2	18.2	0.8	0.0	5.2		147.4
MD	308.7	33.1	3.1	0.4	18.9		364.2
HD	222.0	18.7	3.1	1.3	2.4		247.5
VB	795.4	51.7	16.0	34.5	-8.6	342.3	1231.3
	<u>1666.2</u>	<u>91.9</u>	<u>22.9</u>		<u>45.5</u>		<u>2298.5</u>
TOTAL	1670.5	92.5	23.0	129.7	47.6	342.3	2303.6

MATERIAL BALANCE RUN 78SR-19

Units: lb/hr

Step 3: Force NH₃ and H₂S to balance nitrogen and sulfur

	C	H	N	S	O	ASH	TOTAL
MF COAL	1666.3	124.9	29.2	84.9	206.4	342.3	2454.0
H ₂		-93.7					-93.7
C ₁	89.8	30.0					119.8
C ₂	58.6	14.7					73.3
C ₃	47.7	10.6					58.3
C ₄	16.2	3.4					19.6
CO	-2.9				-3.9		-6.8
CO ₂	11.8				31.6		43.4
		3.0		48.7			51.7
H ₂ S		5.8		93.5			99.3
NH ₃		1.3	6.3				7.6
H ₂ O							
Naphtha	118.9	17.6	0.7	0.0	5.1		142.3
MD	308.7	33.1	3.1	0.4	18.9		364.2
HD	222.0	18.7	3.1	1.3	2.4		247.5
VB	<u>795.4</u>	<u>51.7</u>	<u>16.0</u>	<u>34.5</u>	<u>-8.6</u>	<u>342.3</u>	<u>1231.3</u>
		90.4	29.2	84.9			2258.5
TOTAL	1666.2	91.9	22.9	129.7	45.5	342.3	2298.5

MATERIAL BALANCE RUN 78SR-19

Units: lb/hr

Step 4: Force H₂O to balance total mass

	C	H	N	S	O	ASH	TOTAL
MF COAL	1666.3	124.9	29.2	84.9	206.4	342.3	2454.0
H ₂		-93.7					-93.7
C ₁	89.8	30.0					119.8
C ₂	58.6	14.7					73.3
C ₃	47.7	10.6					58.3
C ₄	16.2	3.4					19.6
CO	-2.9				-3.9		-6.8
CO ₂	11.8				31.6		43.4
H ₂ S		3.0		48.7			51.7
NH ₃		1.3	6.3				7.6
H ₂ O		20.2			173.8		195.5
Naphtha	118.9	17.6	0.7	0.0	5.1		142.3
MD	308.7	33.1	3.1	0.4	18.9		364.2
HD	222.0	18.7	3.1	1.3	2.4		247.5
VB	<u>795.4</u>	<u>51.7</u>	<u>16.0</u>	<u>34.5</u>	<u>-8.6</u>	<u>342.3</u>	<u>1231.3</u>
		112.1			219.3		2454.0
TOTAL	1666.2	90.4	29.2	84.9	45.5	342.3	2258.5

MATERIAL BALANCE RUN 78SR-19

Units: lb/hr

Step 5: Adjust H₂ gas rate and H₂O rate to force H and O balances to closure

	C	H	N	S	O	ASH	TOTAL
MF COAL	1666.3	124.9	29.2	84.9	206.4	342.3	2454.0
		-79.3					-79.3
H ₂		-93.7					-93.7
C ₁	89.8	30.0					119.8
C ₂	58.6	14.7					73.3
C ₃	47.7	10.6					58.3
C ₄	16.2	3.4					19.6
CO	-2.9				-3.9		-6.8
CO ₂	11.8				31.6		43.4
H ₂ S		3.0		48.7			51.7
NH ₃		1.3	6.3				7.6
		21.7			161.0		181.0
H ₂ O		20.2			173.8		195.5
Naphtha	118.9	17.6	0.7	0.0	5.1		142.3
MD	308.8	33.1	3.1	0.4	18.9		364.2
HD	222.0	18.7	3.1	1.3	2.4		247.5
VB	<u>795.4</u>	<u>51.7</u>	<u>16.0</u>	<u>34.5</u>	<u>-8.6</u>	<u>342.3</u>	<u>1231.3</u>
		124.9			206.4		
TOTAL	1666.2	112.1	29.2	84.9	219.3	342.3	<u>2454.0</u>

The first of the two correlations was based on test data obtained using the Old Vacuum Flash system while feeding Kentucky coal.

The equations is:

$$\% \text{ HD retained in VB} = 0.189 (\Delta T)$$

T is the difference between 350°F and the actual average fusion point temperature of the Vacuum Bottoms.

A second equation was developed somewhat later in the program based on data obtained from the New Vacuum Flash system while feeding Illinois coal. This equation is:

$$\% \text{ HD retained in VB} = 0.0634 (\Delta T)$$

ΔT is defined as above.

P & M judged that the equations developed were more dependent on the Vacuum Flash System used rather than on the type of coal fed. On this basis, the first operation was applied in all MBRs in which the Old Vacuum Flash was operated - regardless of coal type. Similarly, the second equation was applied in all MBRs in which the New Vacuum Flash system was in operation. Data on all possible combinations of coal feed and Vacuum Flash System are required to establish more reliable relationships.

The fusion point corrections to the VB yields in each MBR are listed in Table 4. This adjustment is illustrated in Step 6.

MATERIAL BALANCE RUN 78SR-19

Step 6: Correction of Vacuum Bottoms to 350°F Fusion Point

Temperature variation in the Vacuum Flash Drum effects the HD content and the fusion point of the Vacuum Bottoms - at lower temperature, the HD retention in the Vacuum Bottoms is large and the fusion point is low.

VACUUM BOTTOMS COMPOSITION

<u>Component</u>	<u>lb/hr.</u>	<u>Wt. %</u>	<u>Wt. % MF Coal</u>
SRC (1)	753.6	61.2	30.7
IOM	135.4	11.0	5.5
ASH	<u>342.3</u>	<u>27.8</u>	<u>13.9</u>
Total	1231.3	100.0	50.1

Fusion Point = 246°F

$$\begin{aligned}\% \text{ HD retained in Vacuum Bottoms } (1) &= 0.0634 (\Delta T) = \\ &= 0.0634 (350-246) = 6.6\%\end{aligned}$$

$$\begin{aligned}\text{lbs of Heavy Distillate retained} &= (0.066)(1231.3) = \\ &= 81.3\end{aligned}$$

Thus, 81.3 lb of Heavy Distillate should be subtracted from the total Vacuum Bottoms and added to the HD yield. The corrected HD and SRC yields then become:

$$\begin{aligned}\text{HD} : 247.5 + 81.3 &= 328.8 \text{ lb/hr} \\ \text{SRC} : 753.6 - 81.3 &= 672.3 \text{ lb/hr}\end{aligned}$$

This correction completes the adjustment of the Raw Yield Data. The Elementally Balanced Yield Data thus developed have been tabulated on the next page.

(1) Includes some Heavy Distillate.

Elementally Balanced Yields for MBR 78SR-19
(Yields Adjusted to 350°F Fusion Point)

<u>Component</u>	<u>lb/hr</u>	<u>Wt% MF Coal</u>	<u>Wt% MAF Coal</u>
H ₂	-79.3	-3.3	-3.8
C ₁	119.8	4.9	5.7
C ₂	73.3	3.0	3.5
C ₃	58.3	2.4	2.8
C ₄	19.6	0.8	0.9
CO	-6.8	-0.3	-0.3
CO ₂	43.4	1.8	2.1
H ₂ S	51.7	2.1	2.4
NH ₃	7.6	0.3	0.3
H ₂ O	181.1	7.4	8.6
Naphtha	142.3	5.8	6.7
MD	364.2	14.8	17.3
HD	328.8	13.4	15.6
SRC	672.3	27.4	31.8
VB IOM	1150.0 135.4	5.5	6.4
ASH	<u>342.3</u>	<u>13.9</u>	<u>-</u>
Total	2454.0	100.0	100.0

6. Comparison with Harmarville

The elemental balances for the Harmarville P-99 pilot plant were based on the measured quantities and compositions of the gaseous, liquid and solid hydrocarbon products. In contrast to the Ft. Lewis procedure, none of these measured values of hydrocarbons were changed in the forcing procedure. Instead, at Harmarville the coal feed rate is calculated to obtain a carbon balance. (Coal feed rate was set equal to the weight of carbon contained in all products and by-products divided by the carbon concentration in the coal.) Forcing of the coal feed rate was adopted as the most reasonable approach since no systematic error or omission could be found in the measurements of product streams. Moreover, considerable difficulty was experienced in maintaining an accurate calibration of the weigh belt in P-99.

Using the adjusted coal feed rate, together with the average coal composition for the entire Harmarville program, elemental balances of oxygen, nitrogen, sulfur, and hydrogen were obtained by forcing the yields of water, ammonia, hydrogen sulfide and, as a final step, the consumption of hydrogen. Ash in Vacuum Bottoms was simply set equal to the ash contained in the coal.

More consistent elemental balances were achieved by using an overall average coal analysis rather than the analysis for each particular run. It was concluded that analytical errors were greater than any true variation in coal composition between runs.

Aside from the forcing of coal feed rate, other adjustments were relatively minor in the Harmarville forcing procedure. For example, in MBR number 22, the measured hydrogen consumption and water production were each reduced by approximately only five per cent.

V. DATA EVALUATION

A. Summary

This section deals with the data obtained during the twenty-one material balance runs (MBRs). As noted earlier, primary emphasis in the overall SRC II program was placed on production of liquid products for combustion tests. Consequently the MBRs comprised only a minor portion of the total on-stream time. It was only during the MBRs, however, that an attempt was made to measure the effects of reaction conditions on process performance.

The limitations of the Ft. Lewis plant and program discussed in the preceding section resulted in an apparent difficulty in changing only one variable at a time and holding all other conditions constant. Shortcomings in measurement of flows and compositions led to very poor material balance closure. The adjustments made to the measured yields via the forcing procedure may have distorted some results - particularly the Naphtha and Fuel Oil yields.

Despite these limitations, a fairly systematic - though short - program was completed with Kentucky coal. The effect of coal concentration on product yields was measured. Effects of residence time and of recycle slurry composition were also observed.

During the testing of the Illinois and Pittseam (Blacksville Mine) coals, however, it was necessary to forego a systematic experimental program in order to avoid plant shutdowns and to meet requirements for producing fairly large quantities of liquid fuel for combustion tests. Dissolver temperature and pressure effects were investigated to a minor extent with the Pittseam coal.

Product yields are summarized in Table 1 (Section I). More detailed operating and yield data are given in Appendix D. Average composition and properties of the three liquid distillate fractions for each type of coal are listed in Table 6. Variations in these values have been discussed in Section IV, Paragraph D.

Figures 1 through 10 show the yields of the four main products: C - C₄ Gases, Naphtha (IBP-350°F, BP), Fuel Oil (Middle plus Heavy Distillates, 350° - 850°F BP), and Vacuum Bottoms. Also shown are Total Distillate and SRC yields. Coal concentration was chosen as the independent variable in these graphs since this parameter was most thoroughly investigated at Ft. Lewis.

The Ft. Lewis program confirmed the Merriam Laboratory's findings on the relative reactivity of the three coals tested. The Pittseam Blacksville coal was by far the least reactive of the three coals tested - producing relatively less Distillate and more Vacuum Bottoms than the other coals. Illinois coal was the most reactive. (The combination of high reactivity and relatively higher ash content in the Illinois coal, however, led to operating difficulties. In order to avoid plugging of

TABLE 6

COMPOSITION AND PROPERTIES OF SRC II LIQUID PRODUCTS

<u>COAL TYPE</u>	<u>KENTUCKY</u>	<u>ILLINOIS</u>	<u>PITT SEAM</u>
<u>NAPHTHA</u>			
% C	83.84	84.70	84.49
% H	11.58	12.40	12.23
% N	0.74	0.68	0.59
% S	0.19	0.32	0.36
% O (by difference)	3.65	2.09	2.32
Viscosity (cST @ 100°F)	0.9	0.8	0.7
Specific Gravity	0.845	0.797	0.809
<u>MIDDLE DISTILLATE (MD)</u>			
% C	84.37	84.08	85.74
% H	9.16	9.09	8.99
% N	0.98	0.94	0.96
% S	0.23	0.18	0.23
% O (by difference)	4.26	4.71	4.08
Viscosity (cST @ 100°F)	3.5	3.3	2.9
Specific Gravity	0.979	0.976	0.971
<u>HEAVY DISTILLATE (HD)</u>			
% C	89.08	89.49	89.23
% H	7.46	7.50	7.55
% N	1.42	1.29	1.27
% S	0.37	0.40	0.39
% O (by difference)	1.67	1.32	1.56
Viscosity (cST @ 100°F)	117.7	36.9	51.8
Specific Gravity	1.080	1.073	1.075

the Vacuum Bottoms with solids, the Vacuum Flash was operated at lower than normal temperature-leaving more Heavy Distillate in the bottoms.) Western Kentucky coal was somewhat less reactive than the Illinois coal. Typical product yields at 30% coal concentration and one hour nominal slurry residence time in the Dissolver were:

Typical Yields, wt % MAF Coal

<u>Coal</u>	<u>Kentucky 9/14</u>	<u>Illinois 6</u>	<u>Pittseam</u>
C ₁ -C ₄ Gases	18	16	14
Naphtha	11	9	8
Fuel Oil	30	37	25
Vacuum Bottoms	33	28	48

B. Experimental Program and Test Results

Three different coals were tested during the eleven month period from May 11, 1977 through March 31, 1978. These coals were:

- Kentucky No. 9 and 14 - a mixture from the No. 9 and No. 14 seams of P & M's Colonial Mine (Hopkins County).
- Illinois No. 6 from Peabody's River King Mine (St. Clair County).
- Pittsburgh (Pittseam) from Consolidation's Blacksville No. 2 Mine (Monongalia County, W. Va.).

During the program, twenty-one material balance runs (MBRs) were completed - seven runs with each type of coal feed. These MBRs usually lasted seventy two hours. Table 7 lists the starting date of each MBR, its duration and the type of coal fed. Table 3 (Section IV) gives the average elemental analysis of the coal feed during each MBR. A summary

TABLE 7

LISTING OF MATERIAL BALANCE RUNS

<u>Run No.</u>	<u>Starting Date</u>	<u>Length of Run, hrs.</u>	<u>Coal Type</u>
77SR-1	06/03/77	81	Kentucky Nos. 9 & 14
77SR-2	06/12/77	116	"
77SR-3	07/21/77	89	"
77SR-4	08/04/77	66	"
77SR-5	08/11/77	67	"
77SR-6	08/17/77	79	"
77SR-7	08/24/77	95	"
77SR-8	09/06/77	82	Illinois No. 6
77SR-9	09/14/77	60	"
77SR-10	10/22/77	81	"
77SR-11	10/28/77	69	"
77SR-12	11/14/77	66	Pittseam (Blacksville No. 2)
77SR-13	11/24/77	71	"
77SR-14	11/28/77	84	"
77SR-15	12/06/77	38	"
78SR-16	01/12/78	95	"
78SR-17	01/20/78	69	"
78SR-18	01/24/78	46	"
78SR-19	02/09/78	72	Illinois No. 6
78SR-20	02/12/78	69	"
78SR-21	02/23/78	48	"

of operating conditions for each MBR is given in Table 8. Table 1 (Section I) summarizes the adjusted product yields. More detailed operating conditions and yield data are given in Tables 13-21 in Appendix D.

During the first fourteen runs, Gulf was attempting to establish operating conditions which would produce an SRC yield equivalent to 20% of the MAF coal fed. It had been estimated that this yield would provide a Vacuum Bottoms rate which - upon gasification of this material - would produce hydrogen sufficient to balance the feed requirement. Beginning with runs 77SR-15 this criterion was relaxed; a yield of SRC plus IOM equivalent to 35% of the MAF coal was set.

1. Kentucky Coal

a. Program

In June, July and August of 1977, seven tests with Western Kentucky coal were conducted. These tests were designed to measure the effects of coal concentration and slurry feed rate on conversion of coal, product yields, and operability. Five (5) of the tests from a 2² factorial experiment, as indicated below:

<u>Run Number</u>	<u>77SR-1&2</u>	<u>77SR-3</u>	<u>77SR-4</u>	<u>77SR-7</u>
% Coal in Feed Slurry	25	25	30	30
Dissolver Residence Time Hrs.	1.0	0.8	0.8	1.0

(The values given above were the target conditions. Actual operating conditions deviated slightly from these values and are reported in Tables 18 and 13.)

TABLE 8
SUMMARY OF OPERATING CONDITIONS

<u>RUN NO.</u>	<u>COAL CONCENTRATION wt%</u>	<u>RESIDENCE TIME hrs. (1)</u>	<u>DISSOLVER TEMPERATURE °F</u>	<u>DISSOLVER PRESSURE psig</u>
<u>KENTUCKY COAL</u>				
77SR-1	26.1	1.00	861	1912
77SR-2	24.2	0.98	858	1913
77SR-3	24.6	0.82	859	1896
77SR-4	29.9	0.80	862	1899
77SR-5	30.6	0.78	857	1911
77SR-6	33.5	0.96	859	1905
77SR-7	29.5	0.98	861	1920
<u>ILLINOIS COAL</u>				
77SR-8	24.6	0.99	852	1928
77SR-9	29.5	0.97	854	1934
77SR-10	34.4	0.98	852	1895
77SR-11	34.6	0.87	852	1883
78SR-19	32.6	0.87	853	1902
78SR-20	33.4	0.87	854	1903
78SR-21	33.5	0.87	852	1916
<u>PITTSEAM COAL</u>				
77SR-12	21.1	0.83	862	1909
77SR-13	19.8	0.78	862	2012
77SR-14	19.9	0.78	870	2006
77SR-15	20.6	0.81	837	2007
78SR-16	29.9	0.99	869	2022
78SR-17	30.3	1.00	853	2029
78SR-18	29.6	0.98	852	2004

(1) Dissolver volume divided by volumetric slurry feed rate. This is a nominal slurry residence time since no allowance is made for gas holdup.

In Run 77SR-5, the solvent recycle was purposely increased (and the Stripper Bottoms correspondingly decreased) to observe the effects of higher solvent and lower solids composition in the feed slurry. (All other conditions were nearly the same as in Run 77SR-5).

In run 77SR-6, coal concentration was increased to 33.5% (at one hour residence time), thus providing yield data at three different levels of coal concentration (Runs 77SR-1&2, 77SR-7, 77SR-6), all at a nominal slurry residence time of one hour.

During these seven tests, Dissolver conditions were very nearly constant at 860°F and 1900 psig.

b. Results

Yields of the major products are discussed below. Results are compared with the Harmarville data later in this Section.

(i) C₁-C₄ Gases (Figure 1)

A fairly clear effect of residence time is seen in runs 77SR-1, 2, 3, 4 and 7. At one hour nominal residence the yield is approximately 18% of the MAF coal. At 0.8 hours, the gas yield decreased to approximately 16% of the MAF coal. Yields decrease slightly as coal concentration increases from 25 wt% to 30 wt%.

The lower gas yields in Runs 77SR-5 and 77SR-6 correspond to lower ash content of the feed slurry (8.0 and 9.2% for 77SR-5 and 77SR-6 vs. 11.0-13.0% in the other five runs). Hence ash concentration does appear to be a significant variable in the SRC II process.

SRC concentration in the recycle was also lower in these runs, and this could have been a contributing factor to the lower gas yields.

(ii) Naphtha (Figure 1)

The adjusted yield data are widely scattered. The Raw Yields (Table 16) are considered to be more reliable (see discussion in Paragraph C below). Except for Run 77SR-3 and 77SR-5, these raw yields are approximately 11 wt% of the MAF coal. The very low reported value for 77SR-3 is probably related to the poor overall product recovery in that run. (See Table 9). The slightly low yields in 77SR-5 may be partially attributable to the low concentration of ash. Effects of either coal concentration or of residence time cannot be discerned.

(iii) Fuel Oil (Figure 2)

Adjusted yields decreased with increasing coal concentration (from approximately 35% of the MAF coal at 25% coal concentration in the feed slurry to approximately 30% of the MAF coal at 30% coal concentration). No clear effect of residence time can be seen. The raw yield data show a much flatter response to coal concentration (from approximately 31% to 28% while coal concentration increased from 25% to 30%).

FIGURE 1

GAS AND NAPHTHA YIELDS VS. COAL CONCENTRATION

KENTUCKY COAL

▲ Approximately 0.80 hrs. residence time.

△ Approximately 0.98 hrs. residence time.

Notes:

1. Yields shown are adjusted "elementally balanced" values.
2. Ft. Lewis test run numbers (series 77SR) are indicated next to symbols.

FIGURE 2
(MD+HD) AND SRC YIELDS VS. COAL CONCENTRATION

(iv) Vacuum Bottoms (Figure 3)

As expected, the organic residue increased with increasing coal concentration. At one hour residence time, yields ranged from 28% to 33% of the MAF coal, as coal concentration increased from 25% to 30%. At 0.8 hours, yields were approximately 2 - 3% higher. The low ash concentration in Runs 77SR-5 and 77SR-6 contributed to the high yields in these tests.

(v) Hydrogen Consumption (Figure 4)

Both raw yield data and adjusted yields are shown. The adjusted yields seem to cluster around 4.5 wt% of the MAF coal for Runs 77SR-1, 2, 3, 4 and 7. The raw data are more widely scattered. Once again, the combined effects of lower ash and SRC in the feed slurry on the extent of hydrogenation is evidenced by the lower hydrogen consumption in Runs 77SR-5 and 77SR-6.

As an internal check of these values, hydrogen consumption can be estimated based on the yield data suggested in paragraphs (i) through (iv) above. Using average values for hydrogen content in the coal feed and in the various products, consumptions of 5.3 wt% and 5.1 wt% at 25% and 30% coal concentrations can be calculated (MAF coal basis, one hour Dissolver residence time). These estimated values are consistent with the adjusted yields for Runs 77SR-3 and

FIGURE 3 TOTAL DISTILLATE AND (SRC+IOM) YIELDS VS. COAL CONCENTRATION

KENTUCKY COAL

▲ 0.8 hrs Res. Time
 △ 0.98 hrs Res. Time

COAL CONCENTRATION IN FEED SLURRY, Wt. %

FIGURE 4

HYDROGEN CONSUMPTION

KENTUCKY COAL

■ □ : Raw Yields
 ▲ △ : Elementally Balanced Yields

Open symbols : 0.80 hrs. Nominal Residence Time in Dissolver
 Shaded symbols : 0.98 hrs. Nominal Residence Time in Dissolver

Ft. Lewis Test Run Nos. are indicated next to symbols.

77SR-4 at 0.8 hour nominal slurry residence time
(4.5% at 25% coal conc., 4.3% at 30% coal conc.)

Thus it appears that hydrogen consumption at an average slurry residence time will be higher than indicated by the Ft. Lewis adjusted reporting data - probably about 5% of the MAF coal at 30% coal concentration and one hour residence time. Average feed slurry composition for these runs was approximately 30 wt% coal, 30 wt% solvent, 25 wt% SRC, 6 wt% IOM, 9 wt% recycled ash.

2. Illinois Coal

a. Program

A test program - identical to the one followed for testing the Kentucky Coal - had been planned to measure the effects of coal concentration and Dissolver residence time on yields, conversion and plant operability. Conditions in 77SR-7 and 77SR-9 correspond to those in 77SR-1&2 and 77SR-7 with Western Kentucky coal. Target conditions were:

<u>Run Number</u>	<u>77SR-8</u>	<u>77SR-9</u>
% Coal in Feed Slurry	25	30
Dissolver Residence Time, Hrs.	1.0	1.0

The plan to complete a 2^2 factorial experiment (i.e. by conducting complementary runs at 0.8 hour residence time and 25% and 30% coal concentrations) was abandoned after 77SR-9. A significant increase in coal feed ash content after completion

of that run led to plugging of the New Vacuum Flash System. The Old Vacuum Flash System was used in 77SR-10. Conditions for that run (35% coal concentration, 1 hour Dissolver residence time) were selected to produce more SRC, thereby insuring a high organic content in the Vacuum Bottoms to thereby avoid plugging. Run 77SR-11 was also conducted at 35% coal concentration, but at a slightly higher slurry feedrate (lower residence time).

The final three Illinois test runs (77SR-19, 77SR-20 and 77SR-21) were conducted in February, 1978 when there was considerable impetus to maximize the production rate of liquid products for combustion tests. Conditions were chosen to maintain a high rate of liquids production (33% coal concentration and 0.9 hour residence time). Runs 77SR-19 and 77SR-20 were run under identical operating conditions.

The Dissolver conditions were approximately 852°F and 1900 psig in all seven runs. In the last three runs, however, the purity of the recycle gas was low (83% hydrogen), resulting in low hydrogen partial pressure at the inlet to the Dissolver.

b. Results

Yields of gases and Naphtha were lower than with Western Kentucky coal. Vacuum Bottoms production (at 350°F fusion point) are projected to be substantially lower. Fuel Oil production (at 350°F Vacuum Bottoms fusion point) appears to be substantially higher.

As noted above, it was necessary to leave extra Heavy Distillate in the Bottoms to prevent solids plugging of the gravity flow downleg. Consequently, the measured yields of Vacuum Bottoms (at the actual fusion point) were only slightly lower than those with Kentucky coal at similar operating conditions.

(i) Gases (Figure 5)

Yields decrease slightly with increasing coal concentration from approximately 17% of the MAF coal at 25% coal concentration to 16% of the MAF coal at 30% coal concentration in the feed slurry. It appears that yields are slightly lower at 0.87 hours nominal residence time. However, since all the lower residence time runs were at a higher coal concentration, no firm conclusion can be drawn. (Note that one run at high coal concentration and one hour residence time - Run 77SR-10 - was reported by Ft. Lewis to have unreliably low gas yield.)

(ii) Naphtha (Figure 5)

The adjusted ("Elementally Balanced") values scatter widely. The raw yield data are generally more consistent (9.5% of the MAF coal at 25% coal concentration to 7.8% at 25% coal concentration - both at one hour nominal slurry residence time).

FIGURE 5 GAS AND NAPHTHA YIELDS VS COAL CONCENTRATION ILLINOIS COAL

▲ Approximately 0.87 hrs. residence time.

△ Approximately 0.98 hrs. residence time.

(See notes on Figure 1)

COAL CONCENTRATION IN FEED SLURRY, Wt. %

FIGURE 6
(MD + HD) AND SRC YIELDS
VS COAL CONCENTRATION
ILLINOIS COAL

(See notes on Figure 1.)

▲ 0.87 hrs Res. Time
 △ 0.98 hrs Res. Time

COAL CONCENTRATION IN FEED SLURRY, Wt. %

FIGURE 7 TOTAL DISTILLATE AND (SRC + IOM) YIELDS VS COAL CONCENTRATION ILLINOIS COAL

(See notes on Figure 1)

▲ 0.87hrs Res. Time
 △ 0.98 hrs Res. Time

(iii) Fuel Oil (Figure 6)

Surprisingly, the adjusted yields are higher at lower residence times. This anomaly can probably be explained by the poor product recoveries in the higher residence time runs (Runs 77SR-8, 77SR-9 and 77SR-10). A major portion of the carbon imbalance may be attributable to faulty measurements of Fuel Oil yields. It is estimated that the actual yields might be 7% higher (MAF coal basis) giving true yields of perhaps 39% of the MAF coal at 25% coal concentration and 37% of the MAF coal at 30% coal concentration. (See discussion of the Forcing Procedures in Paragraph C below.)

(iv) Vacuum Bottoms (Figure 7)

The Vacuum Bottoms yields (adjusted to 350°F) follow a more expected pattern. Yields are higher at the lower residence time and increase with increasing coal concentration.

3. Pittseam Coal

a. Program

During a three month period (from November, 1977 through January, 1978) seven material balance runs were completed.

The first four runs (77SR-12, 77SR-13, 77SR-14 and 77SR-15) were conducted at 20% coal concentration in the feed slurry and at a slurry feed rate corresponding to a nominal 0.8 hours residence time in the Dissolver. (Target conditions had been 25% coal concentration and one hour residence time. The recycle slurry flowmeter however, gave a false low reading.

After completion of these four runs, the meter was re-calibrated.) The first run gave a low yield of total liquid distillates and a high yield of SRC plus IOM. In the subsequent three runs, Dissolver pressure was raised to 2000 psig to improve the extent of hydrogenation. Dissolver temperature was varied in these runs to measure its effect on SRC yield and distribution of the liquid fractions.

The last three runs (77SR-16, 77SR-17, 77SR-18) were conditions chosen to maximize the rate of Distillate production (30% coal concentration - one hour residence time, 2000 psig Dissolver pressure.) Run 77SR-16 was made at an average Dissolver temperature of 869°F. Runs 77SR-17 and 77SR-18 were run at average Dissolver temperatures of approximately 852°F.

b. Results (Figures 8-10)

This coal is clearly not a suitable feed for the SRC II process. Vacuum Bottoms yields were nearly 50% (MAF coal basis); liquid yields were quite low - typically 25% (MAF coal basis). Effects of coal concentration and Dissolver residence time were not established.

Increased hydrogen partial pressure in Run 77SR-13 appears to have resulted in an increase in Fuel Oil production and a decrease in Vacuum Bottoms yields. Ash and SRC concentrations were also higher, however, in Run 77SR-13 (as compared to 77SR-12); the deeper hydrogenation in that run may therefore be partially attributable to the change in total recycle slurry composition.

FIGURE 8
GAS AND NAPHTHA YIELDS
VS. COAL CONCENTRATION
PITT-SEAM COAL

▲ Approximately 0.80 hrs. residence time.

△ Approximately 1.00 hr. residence time.

(See notes on Figure 1)

COAL CONCENTRATION IN FEED SLURRY, Wt. %

FIGURE 9.

(MD + HD) AND SRC YIELDS VS. COAL CONCENTRATION

PITT-SEAM COAL

(See notes on Figure 1)

▲ 0.8 hrs Res. Time
△ 1.00 hrs Res. Time

FIGURE 10 TOTAL DISTILLATE AND (SRC+IOM) YIELDS VS. COAL CONCENTRATION PITT-SEAM COAL

(See notes on Figure 1)

▲ 0.8 hrs Res. Time
△ 1.00 hrs Res. Time

COAL CONCENTRATION IN FEED SLURRY, Wt. %

Some effects of Dissolver temperature may be seen in Runs 77SR-13, 77SR-14 and 77SR-15. Vacuum Bottoms yields were lowest at the highest temperature. Gas yields were lowest at the lowest temperature.

C. Accuracy and Variability of Data

Table 9 attempts to highlight inaccuracies; such as poor recoveries, large differences in corrected vs. measured values of Naphtha yields and known difficulties during the runs. Appendix E, "Notes on the Reliability of Raw Yield Data", explains some of the problems encountered in each of the runs.

1. Product Recovery

Overall product recoveries ranged from 72.4% to 93.5% of the MAF coal (Table 9). The lack of closure is partially attributable to the fact that certain products are not measured specifically:

- net water produced (with contained impurities such as solvents, NH_3 and H_2S)
- ammonia contained in gas streams
- C_5 and C_6 hydrocarbons contained in gas streams

Allowing 7-9% total yield debits for these losses (based on typical calculated water makes of 6% for Kentucky coal, 7% for Illinois coal, and 4.5% for Pittseam coal, and allowing 2-2.5% for other unmeasured by-products) one might expect a 91-93% recovery at Ft. Lewis (assuming that all other reported products and the coal feed are measured accurately.) On this basis, Runs 4, 12, 18 and 20 show excellent recoveries and Runs 6, 17, and 21 are nearly as good. On the other hand, Runs 8, 9, 10, and 14 are clearly of low

TABLE 9

PRODUCT RECOVERIES, ASH RECOVERIES,
AND NAPHTHA ADJUSTMENTS

RUN NO.	OVERALL (1) RECOVERY (% MAF Coal)	ASH (2) RECOVERY (Wt%)	NAPHTHA (3) ADJUSTMENT (% MAF Coal)	REMARKS (4) (Note 4)
77SR-1	86.5	86	-1.5	Low methane yield
77SR-2	82.1	90	+0.5	Low methane yield
77SR-3	83.0	95	+9.1	
77SR-4	93.5	97	-4.4	Low C and O in coal
77SR-5	86.3	87	-0.1	Low methane yield
77SR-6	89.3	90	+2.5	Chromatograph difficulty
77SR-7	85.8	88	+2.7	Low H ₂ S reported
77SR-8	80.4	82	+7.7	
77SR-9	79.9	89	+8.3	
77SR-10	79.4	87	+7.0	Low gas yield
77SR-11	89.7	89	+1.0	Chromatograph difficulty
77SR-12	92.6	89	-1.5	
77SR-13	84.1	73	-1.4	
77SR-14	79.4	78	+5.3	
77SR-15	85.8	81	-0.1	Short run
78SR-16	83.4	90	+3.6	Low hydrogen purity
78SR-17	89.5	88	+5.2	
78SR-18	90.4	98	-0.1	
78SR-19	87.4	87	-0.2	Low hydrogen purity
78SR-20	92.9	93	-2.0	Low hydrogen purity
78SR-21	89.8	99	+2.3	Low hydrogen purity

- (1) Net products are total gases, liquid, and solids produced, less hydrogen consumed. Production of water and C₅/C₆ hydrocarbons are not included.
- (2) Ash recovery is weight of ash in vacuum bottoms divided by the weight of ash in the coal.
- (3) Naphtha adjustment is the difference between the Elementally Balanced and the Raw Yields.
- (4) For further details, see Appendix D.

quality, and Runs 2, 3, 13, and 16 are also not satisfactory with regards to recovery.

2. Ash Imbalance

Ash recovered in the Vacuum Flash Bottoms (VB) varied from a low of 73% in Run 13 to 99% in Run 21 -- based on the ash content in the coal feed. These recoveries are also listed in Table 9. The poor recoveries in Runs 13 and 14 might be due to faulty VB weighings. Prior to Run 13, VB was loaded into drums. Starting with Run 13, VB was loaded into trucks and weighed. Perhaps there were spills or weighing errors in this transition period. However, one would not expect systematically low reported weights of the Vacuum Bottoms as Ft. Lewis has suggested to SD.

3. Inaccuracies in Measurements

a. Flow Rates

(i) Gases: Apparently, there was enough concern about inaccuracy in the gas meter readings to motivate P & M to install a number of new meter runs during the April, 1978 shutdown, just after the end of the SRC II program and before the return to SRC I operation. A total recycle gas flowmeter is now in service, which verifies the total flow measured by the combination of the five hydrogen meters.

(ii) Liquids: Random errors in measurements of liquid accumulators at the start and end of each MBR were estimated by P & M to be 10.8% of liquids production. (See b. (ii) below for further discussion.)

(iii) Vacuum Bottoms: The practice of forcing the Vacuum Bottoms to obtain an ash balance follows from Ft. Lewis' explanation that the reported VB weights were consistently low.

(iv) Coal: A more accurate weight could be obtained by weighing the entire coal feed, such as by use of a weigh cell.

b. Sampling

(i) Gases: Many samples are taken at frequent intervals on each stream, which tends to reduce random error.

(ii) Liquids: P & M undertook an analysis to estimate the maximum random errors in liquid yields. Based on data obtained from MBR 78SR-20, estimated errors - at a 95% confidence level - were 10.8% for total liquid products, 20.1% for Naphtha, 22.0% for Middle Distillate and 51.4% for Heavy Distillate. P & M's study concluded that the sampling and analysis of contents in the Wash Solvent Accumulator contributed over 90% of the estimated errors. Some details are reported in Ft. Lewis' April, 1978 report.

The 10.8% estimated error for total liquids production seems unusually large since the determination of this yield should depend only on tank level measurements and analysis of any small quantities of foreign (i.e. non-Distillate) material that might be present in the accumulators. The estimated errors for individual liquid fractions include inaccuracies in sampling and

laboratory analysis. Nonetheless, these errors also appear to be unusually large. MBR 78SR-20 may have had some particular circumstances related to measurement of liquid inventory which led to atypically high errors. Further clarification of the basis for these large estimated errors is needed.

- (iii) Vacuum Bottoms: Sample composition - which vary considerably - may be a source of significant errors.
- (iv) Coal: Daily composites were analyzed. Normally, this would appear to be adequate. However, in the case of MBR 77SR-19, and perhaps in other runs, two 24 hour composite samples were discarded, leaving the coal analysis based on 24 out of 72 hours of operation.
- (v) Analytical Errors: These have already been discussed in Section III. E. That discussion concentrates on errors in gas analyses. Possible inaccuracies in the ash measurements are also discussed.

4. Variability

a. Within Test Runs

Figures 11, 12 and 13 show the oscillations in various parameters during MBR 78SR-19. This run was chosen for detailed investigation by SD since P & M maintained that this run and 78SR-20 were well controlled.

C_1-C_4 gas make varies approximately $\pm 15\%$ from the mean value.

This large variation probably arises from variations in the coal feed ash and in the recycle slurry composition.

461510

K&E 10 X 10 TO THE CENTIMETER 18 X 25 CM.
KEUFFEL & ESSER CO. MADE IN U.S.A.

FIGURE 11
MBR 785R-19

COAL FEEDRATE AND ASH CONCENTRATION

FIGURE 12
MBR 78SR-19

HYDROGEN CONSUMPTION,
HYDROCARBON GAS PRODUCTION
& RECYCLE GAS FLOWRATE

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K&E 10 X 10 TO THE CENTIMETER 18 X 25 CM.
KEUFFEL & ESSER CO. MADE IN U.S.A.

FIGURE 13
MBR 785R-19

STRIPPER BOTTOMS COMPOSITION

b. Among Test Runs

A lack of total correspondence of operating conditions among any related set of runs makes it difficult to assess the effect of a given variable. This can be seen, for example, from an examination of the runs with Kentucky coal. Four of the runs were conducted at approximately one hour residence time (77SR-1, -2, -6, and -7). Coal concentration was varied from 24.2% to 33.5% in these runs. Runs 1 and 7 had similar Dissolver temperature profiles and ash concentrations in the feed slurry. Run 77SR-2, however, had a slightly lower average temperature in the Dissolver and a considerable temperature gradient due to introduction of hydrogen quench gas via the bottom sparger. Moreover, ash concentration in the feed slurry was considerably higher than in Runs 77SR-1 and 77SR-7. 77SR-6 had a lower ash concentration in the feed slurry than Runs 77SR-1 and 77SR-7.

5. Evaluation of Material Balance Forcing Procedures

Procedures were developed by P & M to try to standardize results between runs. In some cases, these procedures may actually have distorted the comparability of the results.

a. Forcing of Vacuum Bottoms

The forcing of Vacuum Bottoms to effect an ash balance is justified if the ash imbalance is caused by systematically low errors in weights of the VB solids as Ft. Lewis has reported to SD.

b. Forcing the Naphtha to Balance Carbon

By this procedure, which follows the forcing of VB, a carbon balance is achieved. The adjustments to Naphtha yields are listed in Table 9. For certain runs (77SR-4, -12, -13, and -20) large negative corrections were made. This is somewhat surprising since the rationale for this procedure is mainly to account for unmeasured C_5 - C_6 hydrocarbons. In other runs (77SR-3, -8, -9, -10, -14, -16 and -17) large positive corrections are made. This is questionable since the amount of C_5 and C_6 product is probably small. Comparison of the Naphtha yields for each set of runs with a particular coal indicated that the corrected results seem more dispersed than the measured data.

It is not likely that all of the carbon imbalance in the various runs can be attributed to undetected C_5 's and C_6 's. It would have been more logical to distribute some of the imbalance to the yield of Fuel Oil, since some of the error probably arises from transfers and level measurements of mixed solvents. Distributing the carbon imbalance would have reduced the Naphtha adjustments, increased Fuel Oil adjustments, and resulted in more consistent yields of both products (as these relate to changes in reaction parameters).

D. Comparison of Harmarville and Ft. Lewis Data

Hydrogen consumption and product yields from corresponding material balance runs at the Ft. Lewis and Harmarville (P-99) pilot plants are compared in Figures 14 through 18. Yields are plotted as a function of slurry residence time in the Dissolver - the most widely studied parameter in the Harmarville progra. The yields shown in the Figures are elementally balanced values, expressed as weight percent of moisture free (MF) coal feed, in accordance with the convention used in the Harmarville report.

The MBR's selected (Runs 77SR-4, -5, -6 and -7 from the Ft. Lewis program and run numbers 15, 20, 23 and 24 from the Harmarville program) were conducted with mixtures of coals from the same seams (Kentucky No. 9 and 14). Similar reaction conditions existed in these runs (e.g., Dissolver temperature and pressure, and coal concentration in the slurry feed to the Preheater). However, the slurry feed at Harmarville generally was richer in solvent and leaner in SRC and ash.

The product yields and hydrogen consumption for the Ft. Lewis Runs 77SR-4 and 77SR-5 generally agree quite well with the lines fitted to the Harmarville data at 2000 psig. Naphtha yields at Ft. Lewis, however, were higher.⁽¹⁾ This higher Naphtha yield might reflect the higher levels of SRC and of ash in the Ft. Lewis feed.

Hence, although the yields obtained at Ft. Lewis and Harmarville show a similar relation to Dissolver residence time, the levels of hydrogenation at these two facilities is inconsistent with the relative levels of SRC and ash concentrations in the feed slurries.

FIGURE 14

COMPARISON OF FT. LEWIS AND HARMARVILLE DATA

HYDROGEN CONSUMPTION

○ Harmarville Data
△ Ft. Lewis Data

Solid Symbols: Approximately
33 wt.% Coal in Feed Slurry
Empty Symbols: Approximately
30 wt.% Coal in Feed Slurry

FIGURE 15

COMPARISON OF FT. LEWIS AND HARMARVILLE DATA

HYDROCARBON GAS YIELD

○ Harmarville Data
△ Ft. Lewis Data

Solid Symbols: Approximately
33 wt. % Coal in Feed Slurry
Empty Symbols: Approximately
30 wt.% Coal in Feed Slurry

FIGURE 16

COMPARISON OF FT. LEWIS AND HARMARVILLE DATA

NAPHTHA YIELD

○ Harmarville Data
△ Ft. Lewis Data

Solid Symbols: Approximately
33 wt. % Coal in Feed Slurry
Empty Symbols: Approximately
30 wt. % Coal in Feed Slurry

FIGURE 17

COMPARISON OF FT. LEWIS AND HARMARVILLE DATA

FUEL OIL YIELD

○ Harmarville Data
△ Ft. Lewis Data

Solid Symbols: Approximately
33 wt. % Coal in Feed Slurry
Empty Symbols: Approximately
30 wt. % Coal in Feed Slurry

FIGURE 18

COMPARISON OF FT. LEWIS AND HARMARVILLE DATA

VACUUM BOTTOMS YIELD

○ Harmarville Data
△ Ft. Lewis Data

Solid Symbols: Approximately
33 wt. % Coal in Feed Slurry
Empty Symbols: Approximately
30 wt. % Coal in Feed Slurry

APPENDIX A

CHRONOLOGICAL SUMMARY - OPERATIONAL EVENTS THAT MAY IMPACT MASS BALANCE RUN PERFORMANCE

The plant operations in the SRC II mode commenced on May 11, 1977 and continued through February, 1978. A total of 21 material balance runs were performed utilizing three separate feed coals during this period.

Several equipment revisions were necessary during the development runs for mechanical or process reasons. Those changes, events, and equipment malfunctions considered pertinent to plant performance and/or having impact on reported results are presented chronologically as follows:

June, 1977 MBRs 77SR-1 and 77SR-2

1. Coal Mix System: new mix system inoperable due to plugging; down for modifications. Original coal dust mixing system in service all month.
2. Slurry Circulating Pump: one of the Morris pumps was replaced with a Durco high capacity, high head pump.
3. Coal Feeder: occasionally used to establish coal feed rate; calibration is generally unstable.
4. Wallace and Tiernan gravimetric weigh belt not considered reliable as a weight rate device. Coal feed rates are spot checked via platform scale on bi-hourly basis.
5. FE-2482 Target meter for recycle slurry flow rate to coal blending failed calibration in June; meter was re-calibrated.
6. Fresh Hydrogen Compressor failure accounted for major plant upset (plugged Dissolver Quench lines).
7. Ammonium Salts caused pressure control problems and contamination of BFW with sour water; hydrogen purity below spec. for a week.
8. Side draw seal flush from Stripper caused pumps to vapor lock; resumed use of liquid from Vacuum Flash Condensate Drum as seal flush and supplemented solvent supply to Old Vacuum Flash System.
9. No. 3 Separator Light Oil: installed new line from Light Oil Pump through Heavy Oil Meter to RPWT; This new line allows only desired portion of light oil to flow to Stripper for control of Stripper Bottoms solids concentration, with the remainder directed to RPWT.

10. New Vacuum Flash System was tested in June and found inoperable; old Vacuum Flash System in service all month.
11. Naphtha foaming noted in gas recovery.
12. Dowtherm Heater operating erratically due to low loading.
13. New Vacuum Flash Drum: downleg piping to Sandvik belt totally jacketed with Dowtherm. Installed temperature indicator downstream of control valve in coal product line. Provided a Durco pump in Bottoms service.
14. No. 3 Separator overhead gas not analyzed by chromatograph because of line plugging and condensation in line; daily samples collected and analyzed by laboratory.
15. Plant chromatographs noted to have calibration errors; corrections were made during runs.
16. C₅-C₆ gases leaving Reaction area are not analyzed and must be included in losses reported.
17. Water yield not measured; it is therefore included in net losses.
18. Ammonia make is not analyzed; it is also included in net losses.

July, 1977 MBR 77SR-3

19. Coal Slurry Preparation accounted for numerous interruptions in coal feed to process; a Steam jet was installed on the Slurry Mix Tank to vent to prevent vapors from backing into the Coal Chute.
20. No. 3 Separator operation caused two major process upsets; the Separator Oil Pumps failed on July 22 causing loss of oil flow to the Stripper.
21. The No. 3 Separator pressure was raised to pressure oil to the Stripper; pumps no longer utilized.
22. Both Quench Water Pumps failed resulting in plant shutdown.
23. The New Vacuum Flash System was used successfully during July; a new centrifugal Recirculation Pump was installed to replace one of gear pumps.
24. The Naphtha gas recovery unit was severely upset by DEA contamination on July 20, 21 and 22; recycle hydrogen purity was adversely effected by the upset.
25. Light Oil flow to stripper was reduced to 900 lbs/hr; remainder of the light oil was transferred out of the reaction loop.
26. Chromatograph problem led to using only final six gas analyses of 77SR-3 to establish gas yields.

27. Vacuum Flash System: during a test at constant flash conditions (temperature, pressure, feed rate) fusion point of product noted to vary significantly (395-440°F).

August, 1977 MBRs 77SR-4, 5, 6, 7

28. Coal Slurrying system problems continued to cause some coal outages
29. Faulty slurry recycle meter (FT-2482) signal resulted in high coal concentrations which led to Preheater upset and high pressure drop.
30. The Slurry Recycle Cooler is still oversized (could not maintain 350°F Slurry Blend Tank temperature); insulated top of cooler and plugged all holes top and bottom; this is adequate.
31. 77SR-5: on-line gas chromatograph problems caused hydrogen gas consumption and hydrocarbon gas yields to be suspected of errors.
32. 77SR-6: continued on-line gas chromatograph problems; hydrogen gas yields considered good.
33. 77SR-7: gas chromatograph modifications made to achieve reliability for both hydrogen consumption and hydrocarbon gas yields.

September, 1977 MBRs 77SR-8, 9

34. Coal delivery problems (chute plugging) to the mix equipment were acute in September; New Coal Slurry Mix System down most of month for revisions; Mix Tank overflow line diameter increased from 3" to 5".
35. A Hydrogen Preheater (steam heated) was installed in the hydrogen feed line to the slurry Preheater.
36. Rockwell Piston check valves were installed in hydrogen and water quench lines to the "A" Dissolver.
37. A new demister mat for solids carryover control was installed in the Vacuum Flash Drum after MBR 77SR-9 was completed; ΔP on the old mat had increased to 50" H₂O.
38. A demister mat was installed in the Old Vacuum Flash Drum to eliminate solids carryover.
39. Loss of both Recycle Hydrogen Compressors on September 17 resulted in plugging of Dissolver Quench lines and recycle hydrogen header.
40. The RPWT pressure relief valve was relocated upstream of the flow orifice and the thermowell removed.
41. A Naphtha flow control system for the gas recovery unit was installed.
42. SRC product weighing to be accomplished by truck scales (accuracy of 0.2% total span) on future runs.

43. High liquid yield and resulting low SRC yield caused Vacuum Bottoms ash and solids level to run high during both MBRs. Vacuum Flash conditions were adjusted to lower fusion points to achieve operability.

October, 1977 MBRs 77SR-10, 11

44. Continued coal delivery problems (inlet chute plugging) to the Coal Mix System and Coal Dust Scrubber plugging accounted for numerous outages; modifications made to new Coal Mix System during month.
45. The new quadrant edged orifice meter (FT-1152) was placed in slurry recycle service; installed in vertical line and purged with Heavy Distillate; minimum purge requirement is 100 lb/hr.
46. Slurry metering was difficult all month; the target meter gave false output signals and the quadrant-edged meter drifted because of unsteady supply of clean purge; new meter not flow-calibrated prior to placing in service for run data; used charge pumps RPM and volumetric calibration for flow control.
47. New Vacuum Flash System inoperable first 27 days of month; installed demister mat and cleaned; on-line October 28.
48. Heavy foaming in Naphtha unit led to liquid carry-over to Hydrogen Recycle Compressors.
49. Chromatographic analysis problems occurred on both runs this month; it is expected that hydrogen consumption should have been lower in 77SR-10 and that gas yields should have been higher in both 77SR-10 and 77SR-11; principal problem is related to non-linear hydrogen response in the range of hydrogen purity being recirculated in the plant.

November, 1977 MBRs 77SR-12, 13, 14

50. Coal delivery problems (chute plugging) to mix system encountered on Pittseam Coal; installed salvaged Coal Dryer to reduce moisture level of incoming coal and recommissioned the Primary Crusher to break larger coal pieces.
51. Preheater Charge Pump valves and seats repeatedly fouled with SRC - type deposits when handling Pittseam Coal.
52. Recycle slurry flow measurement not known; target meter is unreliable; quadrant-edged orifice meter now stable but no calibration exists; actual flow much less than indicated.
53. Oil-Water separation system experienced control problems due to plugging of pressure control valve with ammonium salts.
54. Stripper Bottoms slurry stream diluted by flush solvent leak to process at end of MBR 77SR-12.

55. Middle Distillate replaced Naphtha in the gas recovery section to eliminate foaming; however, liquid circulation rate had to be nearly double to control gas purity.
56. Gas Chromatograph modifications were performed in early November prior to beginning MBR 77SR-12 to improve accuracy; this included the addition of new equations to the chromatograph data operation programs to correct for non-linearity of hydrogen response; it was decided not to correct the non-linearity which existed in the first eleven (11) runs.
57. Slurry flow rate error resulted in:
 - (a) All MBRs being performed at 20% coal concentration as opposed to target of 25%,
 - (b) Reactor residence time of 0.8 hr. versus targeted 1 hr., and
 - (c) Hydrogen gas to slurry ratio in Dissolver feed of much less than target of 5.50 lb/100 lb. slurry.

December, 1977 MBR 77SR-15

58. Coal outages resulted from gravimetric feed inlet chute plugs, airlock and chute plug and switches to new mix system.
59. Charge pump packing failures (five) led to one 200 gallon spill to pad as well as other lesser spills.
60. Numerous Hydrogen Compressor outages resulted in erratic plant operation.
61. Volume measurement of Dissolver "A" after MBR 77SR-15 completed shows no accumulation of solids not removable by flushing since September, 1977; volume measurement reflects 89.2 cu. ft. total Dissolver volume, or 97% of "clean" dissolver volume.
62. MBR 77SR-15 not considered very accurate because:
 - a. Run lasted only 38.42 hrs.,
 - b. Slurry solids concentration higher than previous runs and
 - c. Large amounts of flushing and solvent transferred during runs.
63. All slurry feed meters and area 01 solvent meters flow calibrated after 77SR-15 completed; this included the FT-1152 quadrant-edged orifice.
64. The distillation section was tested with a finite quantity of feed stock to study losses; the tests confirm no losses of liquid products are occurring in the distillation process.

January, 1978 MBRs 78SR-16, 17, 18

65. Numerous coal outages were caused by the Coal Feed and Mixing System.

66. Dissolver temperature excursion resulting from lower purity hydrogen (high in CO₂) occurred on January 18.
67. New Vacuum Flash Heater coil out of service beginning January 13, 1978 because of Dowtherm leak; Vacuum Bottoms fusion point had to be lowered about 70°F to 290°F.
68. Demister mats are required in the Flash Drums to prevent solids carryover into the Vacuum Flash Condensate Drum. Fouling of demister mats with solid deposits is normal; however, rapid fouling and ultimate failure of the demister mat can result from an upset (high liquid level or overflow) which may be caused by erratic or false level indication or downleg plugs; downleg plugs have resulted from temperature control problems, foreign matter (broken j-bolts from mats) and coke deposits spalled from the Heater coil.
69. Flow meter in top quench to Dissolver failed after 78SR-16 was completed; had to switch to bottom quench since middle quench was plugged; bottom quench operation normally produces a temperature gradient of 25°F bottom to top; original plan was to conduct all runs at identical conditions to study experimental error.

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70. Coal outages resulted from feed system problems and plugs in the mixing system.
71. Seal flush solvent was switched from Heavy to Middle Distillate on February 4 prior to beginning MBRs.
72. On Feb. 2, the first transfer of 2800 gallons of recovered mixed distillate was made to process. Further, 22,050 gallons of recovered emulsion being dewatered and 35,900 gallons are awaiting processing.
73. High pressure drop across the Slurry Preheater occurred several times causing recycle hydrogen flow to be backed out.
74. February goals: accomplish duplicative runs to obtain data on MBR accuracy and reproducibility. These goals were essentially attained.

APPENDIX B

SRC II EQUIPMENT ADDITIONS/REVISIONS

The required new items of equipment for SRC II operations were installed during the February - May period, 1977. These changes are outlined in detail in the Quarterly Technical Report for the period April 1, 1977 - June 30, 1977. Principal revisions to the plant including the following:

Area 01; Coal Receiving & Preparation

1. New equipment installed.
 - a. Durco 4 x 3-10 slurry circulation pump
 - b. Pulverized coal-slurry Mix Tank
 - c. Slurry Blend Tank vent condensate system
2. Modification to Existing Equipment
 - a. Installed cooling jackets on Wilson-Snyder high pressure Charge Pump plunger packing glands.
 - b. Reduced the surface area of the slurry Recycle Cooler by two thirds.

Area 02; Preheating and Dissolving

1. Preheater - no changes
2. Dissolver
 - a. Additional thermocouples were provided
 - b. The Dissolver Quench system was revised to enable quench to be added at top, middle or bottom locations.
3. Slurry Recycle System
 - a. Installed new No. 3 Separator.
 - b. Modified Stripper with installation of a new packed section between the two existing sections.

Area 03; Mineral Separation and Drying

1. Installed a skimmer funnel inside the RPWT for better oil separation.

2. Added a level control pot to the RPWT to enable automatic transfer of separated oil to Area 04.
3. Installed Drexelbrook capacitance-type oil-water interface transmitters on the RPWT, No. 1 Separator and No. 3 Separator.

Area 04; Solvent Recovery

1. New Vacuum Flash Drum was installed.
2. New Vacuum Flash Preheater was installed.
3. Vacuum Flash Drum Recirculation Pumps were provided for start-up.
4. Installed a new feed Preheater for the Light Ends Column.
5. Installed pressure control system on the Wash Solvent Column.

Area 09.5 and 05; Hydrogen Generation, Recovery and Recompression

1. Revised the hydrogen plant Worthington Compressor suction pressure control to conserve hydrogen.
2. Increased capacity of both Fresh Hydrogen Compressors by 20%.
3. Added a second, larger Recycle Gas Compressor.
4. Installed a Naphtha Gas Scrubbing system.

APPENDIX C
CALCULATION OF RAW YIELDS

Material Balance Run (MBR) 78SR-19 has been selected to present the procedure for calculating "Raw Yields" from the measured data. The forcing procedure used by P&M to obtain "Elementally Balanced Yields" is described in Para. IV E.

MBR 78SR-19 was run from noon on February 9 to noon on February 12, 1978.

During this seventy-two hour period the following data were collected:

A. Coal Rate and Composition

1. Feed Rate

Raw Coal (average of 34 weighings)		2841 lb/hr
Moisture content of Raw Coal (average of daily composites)		10.91 wt%
Moisture content of Dehumidified Coal (average of daily composites)		0.55 wt%
Total Dehumidified Coal Feed = $2841 \times \frac{(100-10.91)}{(100-0.55)}$ =		
		2545 lb/hr
Dehumidified Coal loss (1)	$\frac{5550 \text{ lb}}{72 \text{ hrs}}$	= 77 lb/hr
Net Dehumidified Coal rate		2468 lb/hr
Net MF Coal rate	= 2468 (1-0.0055)	= 2454 lb/hr

(1) This loss is simply the total coal diverted during weighings.

2. Composition

Four elemental analyses of daily composites were performed on coal during MBR 78SR-19. These analyses are presented in Table 10 for reference. Note that the weight percents of carbon and hydrogen in analyses II and III were considered inaccurate by P&M and were ignored when calculating the average coal composition.

B. Gas Yields

Gas yields are determined by material balance around the reaction section.

<u>Inlet Streams</u>	<u>Outlet Streams</u>
- Hydrogen to Slurry Preheater	- Recycle Condensate Separator Overhead
- Hydrogen Purge to Instruments	- No. 3 Separator Gas
- Hydrogen Quench to Dissolver ⁽¹⁾	

Rates and compositions of these streams are presented in Table 11. Net yields were calculated by difference between inlet and outlet streams. The values reported in Table 11 are average values of many frequent measurements and recordings of gas chromatographs and flow transmitters.

The net reported yield of $C_5 + (7.80 \text{ lb/hr})^{(2)}$ has been added to the liquid yield of Naphtha in the final presentation of the Raw Yield Data.

Other streams analyzed by chromatographs are as follows:

-
- (1) Flow recordings are obtained for each of the three quench lines. Normally, flow is only through one or two of these lines.
 - (2) The result of a single combined peak on the GC reading.

TABLE 10

Coal Analysis - Daily CompositesMaterial Balance Run 78SR-19

<u>Date</u> <u>Time Period for composite</u>	<u>2-9-78</u> <u>12 hours</u>	<u>2-10-78</u> <u>24 hours</u>	<u>2-11-78</u> <u>24 hours</u>	<u>2-12-78</u> <u>12 hours</u>	<u>Weighted</u> <u>Avg Coal</u> <u>Analysis</u>	<u>MF Coal</u>	<u>lb/hr</u> <u>MF Coal</u>
Composition, Wt%							
C	67.73	(61.59)	(65.26)	67.30	67.52	67.90	1666.3
H	4.91	(3.88)	(4.02)	5.20	5.06	5.09	124.9
N	1.22	1.21	1.13	1.20	1.18	1.19	29.2
S	3.92	2.56	3.76	4.10	3.44	3.46	84.9
O	7.95	16.69	11.07	7.63	11.85	8.41	206.4
Ash	13.76	13.61	14.14	13.96	13.87	13.95	342.3
Moisture	0.51	0.46	0.62	0.61	0.55	-	-

Notes: Analyses shown above were performed on composite samples.

Parentheses indicate that those values were not used in calculating the average elemental coal composition.

TABLE 11
Gas Yields

Material Balance Run 78SR-19
(lbs/hr)

Component	Input				Output			Net Yield
	Hydrogen To Slurry Preheater	Hydrogen Purge	Hydrogen Quench To Dissolver	Total Input	Recycle Condensate Separator Overhead	No. 3 Separator Gas	Total Output	Output Minus Input
H ₂	236.50	10.43	125.15	372.08	275.91	2.47	278.37	-93.71
N ₂	17.34	0.76	9.18	27.28	27.43	0.00	27.43	0.15*
C ₁	267.89	11.81	141.76	421.45	530.45	10.84	541.29	119.84
C ₂	46.47	2.05	24.59	73.10	139.60	6.80	146.40	73.30
C ₃	30.76	1.36	16.28	48.40	100.65	6.05	106.70	58.31
C ₄	10.98	0.48	5.81	17.28	35.13	1.80	36.93	19.65
CO	93.28	4.11	49.36	146.75	138.92	1.00	139.92	-6.83
CO ₂	0.00	0.00	0.00	0.00	42.83	0.57	43.40	43.40
H ₂ S	0.00	0.00	0.00	0.00	96.64	2.69	99.34	99.34
C ₅ +	<u>0.00</u>	<u>0.00</u>	<u>0.00</u>	<u>0.00</u>	<u>0.78</u>	<u>7.02</u>	<u>7.80</u>	<u>7.80</u>
Total	703.22	31.00	373.13	1106.35	1388.34	39.24	1427.58	321.23

*The Nitrogen net yield (0.15 lb/hr) is neglected during the material balance forcing procedure.

TABLE 12
MATERIAL BALANCE RUN 77SR-19
Units: lbs

<u>Vessel</u>	<u>Start of Run Inventory</u>			<u>End of Run Inventory</u>			<u>Net Accumulation</u>		
	<u>Naphtha</u>	<u>MD</u>	<u>HD</u>	<u>Naphtha</u>	<u>MD</u>	<u>HD</u>	<u>Naphtha</u>	<u>MD</u>	<u>HD</u>
Slurry Blend Tank	3	231	1168	0	443	1401	-3	212	233
High Pressure Separator	2	139	537	0	205	485	-2	66	-52
Intermediate Pressure Separator	1	97	377	0	145	343	-1	40	-34
Recycle Condensate Separator	42	194	193	42	194	192	0	0	-1
Slurry Recycle Stripper	1	39	150	0	62	147	-1	23	-3
Stripper Reflux Drum	190	344	11	192	315	21	2	-29	10
Vacuum Flash Condensate Drum	3	869	2272	0	6808	10649	-3	5939	8377
Wash Solvent Accumulator	4664	7038	1009	14368	23078	9787	9704	16040	8770
Process Solvent Accumulator	0	166	32978	0	163	32435	0	-3	-543
Flush Solvent Accumulator	0	27	5295	0	16	3244	0	-11	-2051
Hotwell Oil Tank	27	63	35	27	20	11	-18	-93	-24
Widing Tank	248	1562	1198	248	3750	2876	348	2188	1678
Flare KO Drum	0	0	0	0	917	604	21	917	604
Slurry Blend Condensate Tank	0	0	0	0	242	11	4	242	11
Recycle Process Water Tank	<u>801</u>	<u>1305</u>	<u>120</u>	<u>881</u>	<u>1377</u>	<u>76</u>	<u>-51</u>	<u>72</u>	<u>-44</u>
Total (lb)	6062	12074	4534	16062	37735	622282	10000	25661	16939

- Fresh Hydrogen
- Recycle Gas
- Naphtha Absorber Feed
- Naphtha Flash Drum Overhead
- No. 1 Separator Vent Gas
- Gas Treatment and Recompression Area Vent Gas
- Inert Gas to Stripper
- Stripper Reflux Overhead

C. Liquid Yields

1. Liquid Accumulations

Laboratory analyses and tank level measurements provided the information for calculating liquid inventories of Naphtha, MD and HD at the start and end of each Material Balance Run. Results for 78SR-19 are shown in Table 12. Net yields are the difference between final and initial inventories over the 72 hour period of MBR 78SR-19 as shown below:

Naphtha	:	16062 - 6062 = 10000 lbs
MD	:	37736 - 12075 = 25661 lbs
HD	:	62282 - 4542 = 16939 lbs
Total	:	116080 - 63480 = 58600 lbs

In addition to the net accumulation of liquid measured in the vessels, drips occurring at various points within the plant are collected and analyzed for Naphtha, MD and HD. Over the 72 hour period the drips collected and measured amount to: Naphtha - 50 lbs., Middle Distillate - 560 lbs., Heavy Distillate - 884 lbs.

The net liquid yields (drips + accumulations) are as follows:

Naphtha	=	10,050 lbs/72 hrs	=	139.6 lb/hr
MD	=	26,221 lbs/72 hrs	=	364.2 lb/hr
HD	=	17,823 lbs/72 hrs	=	247.6 lb/hr
Total	=	54,094 lbs/72 hrs	=	751.4 lb/hr

2. Elemental Analysis of Liquids

The elemental analyses of the liquid fractions were determined from samples taken from the Wash Solvent Accumulator at the end of Run 78SR-19.

	<u>WT%</u>				
	<u>C</u>	<u>H</u>	<u>N</u>	<u>S</u>	<u>O</u> by diff.
Naphtha	83.55	12.36	0.53	0.03	3.53
MD	84.79	9.06	0.84	0.10	5.21
HD	89.69	7.54	1.24	0.53	1.00

D. Vacuum Bottoms Solid Product

1. Rate of Production

The following truck weighings were obtained during MBR 78SR-19:

<u>Date</u>	<u>Weight, lb</u>	<u>Period, hrs</u>
2/09/78	13680	12
2/10/78	23460	24
2/11/78	24950	24
2/12/78	<u>17400</u>	<u>12</u>
Total	79490	72

$$\text{Average Rate} = \frac{79490 \text{ lb}}{72 \text{ hrs}} = 1104 \text{ lb/hr}$$

2. Composition

(a) Elemental Analysis

The Elemental Analysis of the VB is obtained by averaging three out of four elemental analyses of the Vacuum Bottoms (VB) (namely, these of the following dates: 2/09/78, 2/10/78, and 2/11/78) obtained during the MBR period. The fourth analysis performed on VB on 2/12/78 was not taken into consideration

because the sample was taken after the end of the MBR. The analytical results were as follows:

<u>VB Elemental Analyses</u>					
<u>Date</u>	<u>2/09/78</u>	<u>2/10/78</u>	<u>2/11/78</u>	<u>2/12/78</u>	<u>Average</u>
<u>Composition, Wt%</u>					
C	65.35	67.20	61.22	63.84	64.59
H	3.93	4.87	3.84	4.02	4.21
N	1.43	1.29	1.32	1.37	1.35
S	2.15	4.12	2.27	1.46	2.85
Ash	<u>25.59</u>	<u>29.45</u>	<u>29.57</u>	<u>27.94</u>	<u>27.77</u>
Total	96.45	106.93	98.22	98.63	100.77

The oxygen content in the VB is found by difference. It should be noted that this oxygen content is calculated to be negative (-0.77 wt%).

(b) Composition By Component

The VB consists of ash, IOM, SRC and HD. IOM is the difference between pyridine insolubles (PI) and ash. PI is calculated by multiplying the ratio of PI/Ash concentrations in the Stripper Bottoms and the ash concentration in VB. The sum of SRC + HD is calculated by difference between VB and PI.

$$\begin{aligned} \% \text{ PI} &= \frac{(\% \text{ PI in Stripper Btm})}{(\% \text{ Ash in Stripper Btm})} \times (\% \text{ Ash in VB}) \\ &= \frac{19.88}{14.21} \times 0.2777 = 0.3881 \end{aligned}$$

$$\text{PI} = (0.3881) (1104) = 428.4 \text{ lb/hr}$$

$$\text{IOM} = \text{PI} - \text{Ash in VB} = 428.4 - 306.6 = 121.8 \text{ lb/hr}$$

$$\text{SRC} + \text{HD} = \text{VB} - \text{PI} = 1104 - 428.4 = 675.6 \text{ lb/hr}$$

(c) Composition By Component

VB Composition (by Component)

<u>Component</u>	<u>Rate, lb/hr</u>	<u>Wt%</u>
SRC + HD	675.6	61.2
IOM	121.8	11.0
Ash	306.6	27.8
Total	1104.0	100.0

APPENDIX D

Tabulation of Operating Conditions and Yields

The following nine tables list the operating conditions and yields reported by Ft. Lewis for the twenty-one SRC II material balance runs. These twenty-one runs consist of three sets of seven runs each, using Kentucky, Illinois or Pittseam coal, respectively. Three tables are included for each coal: Operating Conditions, Raw Measured Yield Data and Elementally Balanced Yields.

TABLE 13
OPERATING CONDITIONS

KENTUCKY COAL

Run No.	77SR-1	77SR-2	77SR-3	77SR-4	77SR-5	77SR-6	77SR-7
Dehumidified Coal Rate, lb/hr.	1730	1626	1973	2467	2591	2314	1984
Ash in Deh. Coal, wt. %	9.1	11.0	9.5	9.6	9.4	9.5	9.8
Ash in Feed Slurry, wt. %	11.0	13.0	12.9	12.1	8.0	9.2	11.1
Deh. Coal Space Rate, lb/hr/cu. ft.	18.8	17.7	21.4	26.8	28.2	25.2	21.6
Nominal Slurry Residence Time, hrs.	1.00	0.98	0.82	0.80	0.78	0.96	0.98
Outlet Dissolver Pressure, psig	1912	1913	1896	1899	1911	1905	1920
Inlet H ₂ Partial Pressure, psig	1796	1732	1748	1717	1754	1733	1724
Dissolver Temperature Profile, °F							
Inlet	850	804	806	805	804	802	803
4 ft. from Inlet	858	847	856	861	854	856	859
12 ft. from Inlet	859	857	858	861	855	857	859
20 ft. from Inlet	863	862	860	863	859	861	863
28 ft. from Inlet (outlet)	863	863	860	862	860	862	863
Avg. Dissolver Temperature	861	858	859	862	857	859	861
Feed Slurry Composition, wt. %							
Coal	26.1	24.2	24.6	29.9	30.6	33.5	29.5
Total Recycle	73.9	75.8	75.4	70.1	69.4	66.5	70.5
Solvent	37.8	38.4	31.1	28.4	41.6	35.9	33.4
SRC	22.4	22.3	27.8	26.3	20.2	19.8	23.9
IOM	5.1	4.8	5.9	6.2	2.5	4.8	5.0
Ash	8.6	10.3	10.6	9.2	5.1	6.0	8.2
Total Solids	39.8	39.3	41.1	45.3	38.2	44.3	42.7
Recycle Gas							
To Preheater, lb/hr.	374	561	611	583	628	541	509
To Dissolver, lb/hr.							
Top	0	0	101	102	42	201	66
Middle	219	0	106	277	169	40	133
Bottom	0	243	0	0	0	1	0
Total, lb/hr.	619	832	838	985	854	796	869
Total, SCFH	75660	81250	89940	88200	89210	81560	83510
Hydrogen Concentration, mole %	93.9	90.5	92.2	90.4	91.8	91.0	89.8
Hydrogen/Coal Ratio, SCF H ₂ /ton coal	82133	90446	84057	64638	63214	64150	75596
Hydrogen/Slurry Ratio, lb. H ₂ /100 lb. slurry	5.70	5.82	5.50	5.67	5.19	5.66	5.93

TABLE 14
OPERATING CONDITIONS

ILLINOIS COAL

Run No.	77SR-8	77SR-9	77SR-10	77SR-11	78SR-19	78SR-20	78SR-21
Dehumidified Coal Rate, lb/hr.	1642	2008	2321	2619	2468	2528	2530
Ash in Deh. Coal, wt. %	11.5	12.3	13.8	12.4	13.9	13.8	12.7
Ash in Feed Slurry, wt. %	14.0	14.6	13.1	11.0	12.5	12.5	12.1
Deh. Coal Space Rate, lb/hr / cu.ft.	17.9	21.8	25.2	28.5	26.8	27.5	27.5
Nominal Slurry Residence Time, hrs.	0.99	0.97	0.98	0.87	0.87	0.87	0.87
Dissolver Pressure, psig	1928	1934	1895	1883	1902	1903	1916
Inlet H ₂ Partial Pressure, psig	1803	1812	1751	1747	1588	1574	1652
Dissolver Temperature Profile, °F							
Inlet	803	803	808	805	807	804	805
4 ft. from Inlet	850	854	850	849	839	840	838
12 ft. from Inlet	851	853	853	853	852	853	851
20 ft. from Inlet	853	854	853	853	859	860	859
28 ft. from Inlet (outlet)	852	853	852	852	861	861	859
Avg. Dissolver Temperature	852	854	852	852	853	854	852
Feed Slurry Composition, wt. %							
Coal	24.6	29.5	34.4	34.6	32.4	33.4	33.5
Total Recycle	75.4	70.5	65.6	65.4	67.6	66.6	66.5
Solvent	38.3	35.6	38.0	38.6	39.2	39.5	37.4
SRC	21.6	20.0	16.2	17.5	17.0	16.0	18.2
IOM	4.3	3.9	3.0	2.6	3.2	3.2	3.0
Ash	11.2	11.0	8.4	6.7	8.0	7.9	7.9
Total Solids	40.1	44.4	45.8	43.9	43.6	44.5	44.4
Recycle Gas							
To Preheater, lb/hr.	508	422	423	359	703	716	599
To Dissolver, lb/hr.							
Top	66	128	287	288	0	0	0
Middle	133	166	0	0	0	0	0
Bottom	0	0	119	121	372	397	364
Total, lb/hr.	729	729	844	784	1106	1147	987
Total, SCFH	81000	83000	89680	85260	83680	84530	82330
Hydrogen Concentration, mole %	93.5	93.7	92.4	92.8	83.5	82.7	86.2
Hydrogen/Coal Ratio, SCF H ₂ /ton coal	92250	77460	71400	60420	56620	55310	56100
Hydrogen/Slurry Ratio	6.02	6.10	6.52	5.55	4.92	4.92	5.00
lb. H ₂ /100 lb. slurry

TABLE 15
OPERATING CONDITIONS

PITT SEAM COAL

Run No.	77SR-12	77SR-13	77SR-14	77SR-15	78SR-16	78SR-17	78SR-18
Dehumidified Coal Rate, lb/hr.	1673	1675	1677	1675	1987	1998	1998
Ash in Deh. Coal, wt.%	12.3	12.5	12.1	11.9	12.1	11.6	11.7
Ash in Feed Slurry, wt.%	12.4	14.7	15.4	15.4	13.7	11.8	11.8
Deh. Coal Space Rate, lb/hr / cu.ft.	18.2	18.2	18.2	18.2	21.6	21.7	21.7
Nominal Slurry Residence Time, hrs.	0.83	0.78	0.78	0.81	0.99	1.00	0.98
Dissolver Pressure, psig	1909	2012	2006	2007	2022	2029	2004
Inlet H ₂ Partial Pressure, psig	1703	1869	1868	1897	1737	1859	1826
Dissolver Temperature Profile, °F							
Inlet	813	808	810	811	802	809	811
4 ft. from Inlet	859	858	865	837	866	841	840
12 ft. from Inlet	863	863	871	838	871	850	850
20 ft. from Inlet	864	864	872	836	869	858	857
28 ft. from Inlet (outlet)	863	863	871	835	868	861	860
Avg. Dissolver Temperature	862	862	870	837	869	853	852
Feed Slurry Composition, wt.%							
Coal	21.1	19.8	19.9	20.6	29.9	30.3	29.6
Total Recycle	78.9	80.2	80.1	79.4	70.1	69.7	70.4
Solvent	34.2	30.7	30.7	25.1	30.0	29.8	31.7
SRC	26.4	28.7	26.9	32.9	21.8	24.1	23.4
ICM	8.5	8.6	9.5	8.4	8.1	7.5	7.0
Ash	9.8	12.2	13.0	13.0	10.2	8.3	8.3
Total Solids	39.4	40.6	42.4	42.0	48.2	46.1	44.9
Recycle Gas							
To Preheater, lb/hr.	553	452	452	458	437	526	549
To Dissolver, lb/hr.							
Top	184	204	190	236	296	0	0
Middle	0	0	0	0	0	0	0
Bottom	0	0	0	0	0	179	173
Total, lb/hr.	759	688	671	726	763	732	753
Total, SCFH	70990	76400	74180	88140	63500	77100	77870
Hydrogen Concentration, mole%	89.2	92.9	93.1	94.5	85.9	91.6	91.1
Hydrogen/Coal Ratio, SCF H ₂ /ton coal	75700	84750	82360	99450	54900	70690	71010
Hydrogen/Slurry Ratio ,	4.26	4.47	4.37	5.45	4.34	5.69	5.57
1b . H ₂ /100 lb. slurry							

TABLE 16
RAW MEASURED YIELD DATA
(Wt% MAF Coal)
KENTUCKY COAL

Run	77SR-1	77SR-2	77SR-3	77SR-4	77SR-5	77SR-6	77SR-7
H ₂	-3.6	-5.3	-3.4	-4.1	-2.3	-2.3	-4.0
C ₁	3.5	4.0	5.5	5.9	3.0	6.1	6.9
C ₂	5.1	5.0	4.5	4.1	3.5	4.3	5.3
C ₃	4.5	4.6	4.0	3.4	3.0	3.2	4.1
C ₄	2.2	2.3	2.4	2.0	1.5	1.7	2.1
Total C ₁ -C ₄ Gas	15.3	15.9	16.4	15.4	11.0	15.3	18.4
CO	0.2	1.7	2.5	2.0	0.7	1.4	0.4
CO ₂	1.3	1.7	1.5	1.9	1.2	1.5	1.8
H ₂ S	3.4	1.8	1.7	1.5	1.9	2.1	0.4
NH ₃	-	-	-	-	-	-	-
H ₂ O + losses	13.5	17.9	17.0	6.5	13.7	10.7	14.2
Naphtha	10.6	10.0	6.3	11.3	9.2	10.5	11.4
Middle Distillate	25.3	20.8	17.8	20.1	17.4	19.6	19.6
Heavy Distillate	6.4	9.2	12.0	13.4	11.2	4.0	9.6
Total Liquids	42.3	40.0	36.1	44.8	37.8	34.1	40.6
SRC	22.7	21.1	22.4	25.0	31.3	29.7	22.4
IOM	4.9	5.2	5.8	7.0	4.6	7.5	5.9
Vacuum Bottoms	27.6	26.3	28.2	32.0	35.9	37.2	28.3
TOTAL	100.0	100.0	100.0	100.0	100.0	100.0	100.0

Note: In the first nine runs, Naphtha was defined as recovered distillate with a boiling range of IBP - 380°F. Middle Distillate and Heavy Distillate boiling ranges were 380-550°F and 550-850°F respectively.

TABLE 17

RAW MEASURED YIELD DATA
(Wt% MAF Coal)
ILLINOIS COAL

Run	77SR-8	77SR-9	77SR-10	77SR-11	78SR-19	78SR-20	78SR-21
H ₂	-3.4	-3.8	-5.9	-3.5	-4.4	-4.3	-3.8
C ₁	5.4	5.0	3.6	4.6	5.7	5.8	6.5
C ₂	5.1	4.9	3.4	3.8	3.5	3.6	3.7
C ₃	4.2	4.0	2.8	2.8	2.8	2.8	3.0
C ₄	2.0	1.9	1.1	1.4	0.9	0.9	1.2
Total C ₁ -C ₄ Gas	16.7	15.8	10.9	12.6	12.9	13.1	14.4
CO	0.6	0.4	-0.6	0.2	-0.3	-0.3	0.1
CO ₂	1.8	2.2	2.1	2.2	2.1	2.1	2.0
H ₂ S	1.2	1.8	3.0	3.2	4.6	4.5	1.8
NH ₃	-	-	-	-	-	-	-
H ₂ O + losses	19.6	20.1	20.6	10.3	12.6	7.1	12.1
Naphtha	9.5	8.7	12.4	7.8	6.9	7.0	8.9
Middle Distillate	25.1	24.8	25.7	19.8	17.2	21.7	18.7
Heavy Distillate	4.7	3.0	-2.3	10.9	11.7	10.5	10.8
Total Liquid	39.3	37.4	35.8	35.8	35.9	39.2	38.4
SRC	19.8	22.6	29.0	31.5	31.1	32.1	29.5
IOM	4.4	4.4	5.1	5.0	5.6	6.1	5.5
Vacuum Bottoms	24.2	27.0	34.1	36.5	36.7	38.2	35.0
TOTAL	100.0	100.0	100.0	100.0	100.0	100.0	100.0

Note: Beginning with MBR 77SR-10 the oiling range of Naphtha was revised to IBP-350°F. Consequently, the MD boiling range became 350-550°F.

TABLE 18
RAW MEASURED YIELD DATA
(Wt% MAF Coal)
PITTSEAM COAL

Run	77SR-12	77SR-13	77SR-14	77SR-15	78SR-16	78SR-17	77SR-18
H ₂	-3.6	-5.7	-5.8	-4.1	-4.8	-5.8	-5.1
C ₁	9.1	7.8	7.7	5.8	6.7	5.4	4.8
C ₂	4.7	4.8	4.8	3.4	4.3	3.8	3.4
C ₃	3.6	4.0	4.0	3.0	3.8	3.0	2.5
C ₄	1.5	1.8	1.8	1.6	1.4	1.2	0.9
Total C ₁ -C ₄ Gas	18.9	18.4	18.3	13.8	16.2	13.4	11.6
CO	0.7	-0.1	-0.1	0.1	-0.1	-0.5	0.0
CO ₂	1.5	1.5	1.5	1.1	1.2	1.4	1.2
H ₂ S	1.4	1.6	1.4	1.2	2.1	1.8	0.7
NH ₃	-	-	-	-	-	-	-
H ₂ O plus losses	7.4	15.9	20.6	14.2	16.6	17.9	9.6
Naphtha	7.4	5.1	5.9	4.0	9.7	6.7	7.5
Middle Distillate	17.9	26.7	26.0	22.4	18.1	15.4	15.8
Heavy Distillate	5.8	2.6	3.0	12.5	3.8	5.5	10.1
Total Liquids	31.1	34.4	34.9	38.9	31.5	27.6	33.4
SRC	31.5	23.9	21.3	27.8	27.3	33.6	37.7
IOM	11.1	7.4	7.9	7.0	10.0	10.6	10.9
Vacuum Bottoms	42.6	31.3	29.2	34.8	37.3	44.2	48.6
TOTAL	100.0	100.0	100.0	100.0	100.0	100.0	100.0

Note: The Ft. Lewis Raw Yield Data starting with MBR 77SR-13 show ash-forced Vacuum Bottoms. The yields on this table are actual measured values, obtained from the ash-forced Vacuum Bottoms values using the ash recoveries obtained from Ft. Lewis. Back-calculation was required since the measured values of Vacuum Bottoms were not reported.

TABLE 19

ELEMENTALLY BALANCED YIELDS
(Wt% MAF Coal)
Kentucky No. 9 & 14 Coal

<u>Run</u>	<u>77SR-1</u>	<u>77SR-2</u>	<u>77SR-3</u>	<u>77SR-4</u>	<u>77SR-5</u>	<u>77SR-6</u>	<u>77SR-7</u>
H ₂	-4.5	-4.1	-4.5	-4.3	-3.6	-3.6	-4.8
C ₁	6.5	6.4	5.6	5.9	4.5	5.6	6.8
C ₂	5.1	5.0	4.5	4.1	3.6	4.0	5.4
C ₃	4.5	4.6	3.9	3.4	3.0	3.2	4.1
C ₄	2.2	2.3	2.4	2.0	1.6	1.5	2.1
Total C ₁ -C ₄ Gas	<u>18.3</u>	<u>18.3</u>	<u>16.4</u>	<u>15.4</u>	<u>12.7</u>	<u>14.3</u>	<u>18.4</u>
CO	1.7	1.5	2.5	2.0	0.7	1.4	0.4
CO ₂	1.4	1.7	1.6	1.9	1.2	1.5	1.7
H ₂ S	2.9	2.4	2.6	1.8	2.4	2.6	2.6
NH ₃	0.5	0.6	0.8	0.6	0.5	0.5	0.6
H ₂ O	5.8	5.3	5.7	9.2	7.2	5.3	6.0
Naphtha	9.1	10.5	15.4	6.9	9.1	13.0	14.2
Middle Distillate	22.3	20.2	17.8	20.1	17.4	18.7	19.6
Heavy Distillate	11.1	16.9	12.4	10.4	12.7	0.0	8.6
Total Liquids	<u>42.5</u>	<u>47.5</u>	<u>45.6</u>	<u>37.4</u>	<u>39.2</u>	<u>31.7</u>	<u>42.4</u>
SRC	25.7	20.7	23.3	28.8	34.5	37.9	26.1
IOM	5.7	6.0	6.0	7.2	5.2	8.4	6.6
Vacuum Bottoms	<u>31.4</u>	<u>26.7</u>	<u>29.3</u>	<u>36.0</u>	<u>39.7</u>	<u>46.3</u>	<u>32.7</u>
TOTAL	100.0	100.0	100.0	100.0	100.0	100.0	100.0

Vacuum bottoms yields are corrected to 350°F fusion point.

TABLE 20
ELEMENTALLY BALANCED YIELDS
(Wt% MAF Coal)
ILLINOIS COAL

Run	77SR-8	77SR-9	77SR-10	77SR-11	78SR-19	78SR-20	78SR-21
H ₂	-5.0	-4.7	-4.4	-3.8	-3.8	-3.6	-4.6
C ₁	5.4	5.0	3.6	4.5	5.7	5.8	6.6
C ₂	5.1	4.9	3.4	3.7	3.5	3.6	3.7
C ₃	4.1	3.9	2.7	2.9	2.8	2.8	3.0
C ₄	2.1	2.0	1.2	1.3	0.9	1.0	1.1
Total C ₁ -C ₄ Gas	16.7	15.8	10.9	12.4	12.9	13.2	14.4
CO	0.5	0.4	-0.5	0.2	-0.3	-0.3	0.1
CO ₂	1.8	2.2	2.1	2.2	2.1	2.1	1.9
H ₂ S	3.5	3.5	3.3	3.3	2.4	3.5	3.3
NH ₃	0.4	0.5	0.5	0.5	0.3	0.4	0.7
H ₂ O	6.7	7.0	7.5	6.2	8.6	6.6	8.2
Naphtha	17.2	17.0	19.4	8.8	6.7	5.0	11.2
Middle Distillate	25.2	24.9	25.8	19.9	17.3	21.7	18.6
Heavy Distillate	6.7	5.4	2.1	12.5	15.6	14.5	16.4
Total Liquids	49.1	47.3	47.3	41.2	39.6	41.2	46.2
SRC	21.2	23.0	27.6	32.3	31.8	30.4	24.2
IOM	5.1	5.0	5.7	5.5	6.4	6.5	5.6
Vacuum Bottoms	26.3	28.0	33.3	37.8	38.2	36.9	29.8
TOTAL	100.0	100.0	100.0	100.0	100.0	100.0	100.0

Vacuum bottoms yields are corrected to 350°F fusion point.

TABLE 21

ELEMENTALLY BALANCED YIELDS
(Wt% MAF Coal)

PITTSEAM COAL

Run	77SR-12	77SR-13	77SR-14	77SR-15	78SR-16	78SR-17	78SR-18
H ₂	-4.2	-4.7	-4.5	-3.6	-4.3	-3.5	-3.2
C ₁	9.2	7.8	7.7	5.7	6.6	5.5	4.8
C ₂	4.7	4.8	4.8	3.5	4.3	3.8	3.4
C ₃	3.6	4.0	4.0	3.0	3.7	3.0	2.5
C ₄	1.5	1.8	1.8	1.6	1.3	1.2	0.9
Total C ₁ -C ₄ Gas	19.0	18.4	18.3	13.8	15.9	13.5	11.6
CO	0.7	-0.1	-0.2	0.1	-0.1	-0.4	0.0
CO ₂	1.5	1.5	1.5	1.2	1.3	1.2	1.2
H ₂ S	2.2	1.7	2.2	2.0	2.0	1.7	1.8
NH ₃	0.5	0.5	0.6	0.4	0.6	0.4	0.6
H ₂ O	4.1	6.7	4.5	4.3	4.8	4.1	4.5
Naphtha	5.9	3.7	11.2	3.9	13.3	11.9	7.4
Middle Distillate	17.9	26.7	25.9	22.3	18.1	15.4	15.8
Heavy Distillate	4.3	3.4	4.1	13.3	6.1	6.9	12.3
Total Liquids	28.1	33.8	41.2	39.5	37.5	34.2	35.5
SRC	35.9	32.1	26.3	33.5	31.2	36.8	36.7
IOM	12.2	10.1	10.1	8.8	11.1	11.9	11.3
Vacuum Bottoms	48.1	42.2	36.4	42.3	42.3	48.7	48.0
TOTAL	100.0	100.0	100.0	100.0	100.0	100.0	100.0

Vacuum bottoms yields are corrected to 350°F fusion point.

APPENDIX E

NOTES ON THE RELIABILITY OF RAW YIELD DATA

Following are comments on conditions which influenced the reliability of data collected during the various runs. Most of the information included here was obtained from the monthly "Process Evaluation Progress Reports" prepared by J.D. Naylor and other members of the Ft. Lewis staff. (Reference should also be made to Table 9 in Section V which lists product and ash recoveries.)

Run 77SR-1

The Old Vacuum Flash System was used during this first run with Kentucky coal. (Consistent flow of Vacuum Bottoms to the cooling belt could not be maintained with the New Vacuum Flash System, and the Bottoms Recirculation Pumps proved to be unreliable.) Use of the Old System required that the Stripper Bottoms be routed through two intermediate surge tanks, Filter Feed Flash Vessel (FFFV) and Vacuum Flash Feed Accumulation Drum (VFFA). A third tank, the Seal Flash Accumulator (SFA), is needed to route seal flush to the pumps servicing the VFFA, FFV and the Vacuum Flash. Use of this additional tankage increases the possibility of errors in computing distillate yields, since these yields are based on changes in liquid inventory over the course of a run. Also, significant recycle of Vacuum Flash Condensate and Bottoms is required when using the oversized (for SRC II) Old Vacuum Flash System. It is possible that degradation of recycled material may occur, thereby altering the yield structure.

Chromatograph problems resulted in an unreliable value for methane yield (as reported in Table 16, "Raw Yield Data"). The adjusted yield for methane (as shown in Table 19, "Elementally Balanced Yields") was obtained by ratioing the methane yield to the measured ethane yield, based on the C_1/C_2 ratio reported for 77SR-7. Gas makes are calculated from a material balance around the reaction section based on recorded chromatograph and flow measurements. The gas yields obtained by this procedure are not normally changed at all in the forcing procedure used to obtain an elemental balance.

The reported yields of total distillates (Naphtha + MD + HD) were adjusted in order to obtain a carbon balance. This can be seen by comparison of the raw yields in Table 16 and the elementally balanced yields in Table 19. Normally, only the Naphtha yield is adjusted to obtain a carbon balance. The standard Ft. Lewis procedure could not be followed here since an elemental analysis of the Naphtha fraction alone was not performed. However, an elemental analysis of the mixed solvents in the Wash Solvent Accumulator was available and this analysis was used to obtain the elementally balanced yield.

Run 77SR-2

Errors in gas chromatograph calibrations were discovered and corrected during this run. Gas yields were based on the last thirty hours of the run after corrections were made. As in run 77SR-1, coal rate was determined by bi-hourly weighings of coal collected from the feed belt over short time intervals (rather than relying on the instrument readings from the gravimetric feeder). The Old Vacuum Flash System was continued in use. Reported yields of methane and total distillate were adjusted in the same manner as described for 77SR-1.

Hydrogen quench to the Dissolver was introduced through the bottom sparger. A significant temperature gradient across the Dissolver (647°F-863°F) resulted. (Normally, quench gas is introduced through the upper and/or middle spargers. This configuration leads to a more nearly isothermal operation.) The average MF coal analysis (Table 3) showed a higher ash content (11.0% vs. 9.1%) and a lower carbon content (70.5% vs. 72.6%) than for 77SR-1.

Run 77SR-3

Eleven brief interruptions of coal feed occurred during this run. These interruptions were judged, however, to have had negligible effects on the results of the run since slurry feed compositions did not change significantly during the coal outages.

The New Vacuum Flash System was used throughout this run. Net Stripper Bottoms flowed directly to the new Vacuum Flash Drum (FVD). Elimination of intermediate surge vessels (as are required when feeding to the old VFD) should have improved the accuracy of liquid yield measurements.

Ash content of the recycle slurry (Stripper Bottoms) was increased by bypassing a portion of the No. 3 Separator Oil around the Stripper (i.e., feeding some oil directly to the Recycle Process Water Tank). This was done to increase reactivity, thereby offsetting the effect of reduced Dissolver residence time. (77SR-3 was conducted at a higher slurry feed rate which corresponded to 0.82 hour nominal slurry residence time in the Dissolver versus approximately one hour residence time for Runs 77SR-1 and 77SR-2.)

Chromatograph problems occurred during the run. As a result gas yields were based on averages for the final six hours of the run.

Run 77SR-4

Overall recovery of products was very high on this run (93.3% of the MAF coal fed). Ash recovery was also very high (97%). Analyses of the coal (see Table 3) show an atypically low carbon and low oxygen content. Since the forcing procedure adjusts product rates to balance coal feed rates and composition, the elementally balanced yields (Table 19) show a high water make to balance reported coal oxygen and low Naphtha yield to balance reported coal carbon. By contrast, the raw yield data (Table 16) show a low water make and high Naphtha yield. It seems likely that the coal analysis is in error and that a typical coal analysis should be used for forcing the data for this run rather than the analyses made during the run.

Run 77SR-5

Chromatograph problems resulted in an uncharacteristically low methane yield. An adjusted methane yield based on the C_1/C_2 ratio for 77SR-7 and the reported ethane yield.

The average Dissolver temperature was slightly low in this run (857°F). Recycle solvent (Vacuum Flash Distillate) rate was increased to 750 lb/hr, from the 300 lbs/hr used for the previous runs, to achieve a low total solids concentration in the feed slurry. (P&M hoped to examine the effect of low solids concentration on yields.) Ash concentration in the feed slurry dropped to 8.0 wt% versus 12.1% for the previous run.

Run 77SR-6

Gas chromatograph problems continued during this run. P&M maintains however, that only the reported H_2 yield was effected, and not the C_1-C_4 gas yield.

Ash concentration in the feed slurry was again low (9.2 wt%) due to a combined effect of high coal concentration (33.5 wt%) and a slightly higher than average solvent recycle rate (479 lb/hr).

Run 77SR-7

No unusual conditions were reported for this run. Overall product recovery was 85.1% of the measured coal feed rate.

Run 77SR-8

The very high degree of liquefaction obtain in this first run with Illinois coal necessitated that Vacuum Flash temperature be lowered to permit gravity flow of bottoms to the cooling belt. Because SRC production was low more HD was taken with the Vacuum Bottoms to control solids content. Average fusion point of the Vacuum Bottoms was 276°F. Despite this low fusion point, ash content of the VB averaged 32% and total solids was close to 50 wt%. Some difficulties were encountered in maintaining Bottoms flow. Overall product recovery was quite poor (79% of MAF coal).

Run 77SR-9

The high liquids production and corresponding low yield of SRC necessitated that liquid product be intentionally left in the Vacuum Bottoms in order to maintain flow. Solids composition of VB was similar to the previous run. Overall recovery of products was again poor at 79% of MAF coal.

Run 77SR-10

Chromatograph problems led to unreliably low C_1 - C_4 hydrocarbon gas production. High coal ash content and use of the Old Vacuum Flash System combined to require a very low VB fusion point (average 224°F). Total recovery was low (78% of MAF coal). A negative HD yield was reported.

Run 77SR-11

Chromatograph problems apparently led to a slightly low reported gas yield. Higher SRC production and use of the New Vacuum Flash System combined to allow a much higher fusion point Vacuum Bottoms (307°F). Recovery of HD as liquid product was consequently much higher than in the previous three MBRs. Overall product recovery improved to 99% MAF coal.

Run 77SR-12 (Pittseam Coal)

Chromatograph errors caused by non-linear hydrogen response were corrected prior to this run.

Errors in measurement of the recycle slurry flow rate resulted in conducting Runs 77SR-12, 77SR-13, and 77SR-14. at approximately 20 wt% coal concentration and 0.8 hours residence time instead of at the targeted values of 25 wt% and one hour. Hydrogen circulation rate was also much lower than intended (4.26-4.46 lbs. per 100 lbs. slurry feed vs. the 5.5 target value).

Run 77SR-13

System pressure and H₂ purity were increased in this run to improve distillate yield.

Relatively high ash content in the recycle slurry resulted in an ash concentration of 14.7% in the slurry feed to the Preheater.

Starting with this run, the procedure for collecting and weighing VB was changed. Dump trucks were used rather than small drums. The poor ash recovery may reflect that the new procedure was probably not yet well established with the operators.

Run 77SR-14

Dissolver temperature was raised to an average of 870°F during this run. The very high losses (overall recovery of 79.4% of MAF coal) lead one to suspect erroneously low values of reported hydrocarbon gas and/or of liquid distillates. Ash recovery continued to be low at 78% and was presumably still due to difficulty in adjusting to truck weighings, according to P&M. Ash concentration in the feed slurry remained high at 15.4%.

Run 77SR-15

This run was considered by P&M to be inaccurate because of its short duration (38 hours, versus 72 hours typically). Also, several large transfers of distillate across the material balance envelope occurred during the run. Nonetheless, total product recovery improved to 85.8% of the MAF coal. Gas and Vacuum Bottoms yields were considered to be fairly accurate but the liquid yields were doubtful, according to P&M.

Note that Dissolver temperature was purposely lowered to 837°F (average). To increase reactivity, oil from the Recycle Condensate Separator was purposely diverted around the Stripper in order to increase ash content of the Stripper Bottoms. (The same procedure may have been used in 77SR-13 and 14, but this has not been reported).

Run 77SR-16

The average Dissolver temperature for this run was relatively high at 869°F. Hydrogen purity was low at 85.9%. (Hydrogen partial pressure is calculated to be 1737 psia in the recycle gas to reaction.) A leak in the Vacuum Flash Preheater coil required that the Dowtherm heating be shut off. This occurred during 77SR-16 and continued through 77SR-17 and 18.

Run 77SR-17

The hydrogen quench to the Dissolver was introduced via the lower sparger; this led to a 20°F temperature gradient (841°F to 861°F). The average temperature of 853°F is low (compared to 77SR-13 and 14). Overall recovery appears to be low.

Run 77SR-18

Conditions are quite similar to the previous run. Hydrogen quench gas was again introduced via the bottom sparger. A large gradient and low average temperature resulted. Average fusion point of VB was low at 295°F (due to lack of Vacuum Flash Preheater).

Run 77SR-19

Hydrogen purity in the recycle gas to reaction was quite low (83.5%). Since total system pressure was maintained at the standard (for Ft. Lewis) 1900 psig, the inlet hydrogen partial pressure was low (1588.8 psig). The overall organics recovery of % of MAF coal is acceptable. The lower hydrogen quench sparger was used. Dissolver temperature profile ranged from 839°F (at 4 ft. above the bottom) to 861°F (at 28 st.) A Ft. Lewis monthly report claims a high degree of accuracy for this run. Examination of the raw data does not seem to confirm this. Conditions during the run varied considerable for certain parameters (e.g. gas yield), during the run. Also, lab analyses of periodic samples varied. Coal carbon and hydrogen contents were based on composite samples for the first and last twelve hour periods of this 72 hour run. Please see Appendix C and Figures 8,9, and 10 for full details. It is SD's judgement, therefore, that accuracy of the run is reasonably good but not of high quality.

Run 77SR-20

This run was conducted at very similar conditions as 77SR-19 to measure reproducibility of the data. (This run started only two hours after 77SR-19 and terminated.) Recoveries of total products and of ash were high.

Run 77SR-21

The Old Vacuum Flash System was used. Losses were somewhat higher than 77SR-20 and 19, but still quite acceptable. This run lasted only 48 hours.

APPENDIX F

Measurement of Basic Nitrogen in Gases-UOP Method 315-59

The method involves passing the sample into an essentially non-aqueous solution of H_2SO_4 in acetone kept at dry ice-acetone temperature where the condensable hydrocarbons and basic compounds are retained. Quantitative retention of the basic compounds is ensured by the low temperature scrubbing through an acid solution and also by condensation of the bases along with the hydrocarbons.

Following sampling, the weight of condensables is determined and then they are evaporated. The remaining solution of H_2SO_4 in acetone is diluted with H_2O and analyzed for basic nitrogen by a conventional Kjeldahl distillation, i.e., strong alkali is added and the bases distilled into a solution of boric acid. The volatilized bases are titrated with standard acid to the methyl purple end point.

Appendix G
LIST OF P&I DIAGRAMS

<u>Title</u>	<u>P&I Dwg. No.</u>	<u>Major Pieces of Equipment</u>
Coal Receiving and Preparation	P3-3A	Storage Bin Bucket Elevator Crushed Coal Storage Bins A&B Surge Bin Bucket Elevator Gravimetric Feeder Coal Pulverizer Pulverizer Gas Preheater Dehumidifier
Coal Receiving and Preparation	P3-3B	Slurry Blend Tank Slurry Mix Tank Slurry Air Cooled Exchanger Slurry Blend Condensate Tank
Slurry Preheating and Dissolving	P3-4	Slurry Preheater
Slurry Preheating and Dissolving	P3-5	Slurry Dissolver HP Flash Drum IP Flash Drum Recycle Condensate Separator HP Flash Drum Vapor Air Cooled Exchanger
Slurry Recycle System	P3-26	No. 3 Separator Slurry Recycle Stripper Vapor Cooler Reflux Drum
Mineral Separation	P3-6	Recycle Process Water Tank Filter Feed Surge Vessel Filter Feed Flash Vessel Filter Feed Flash Recirculation Exchanger Feed Flash Vapor Condenser Flash Condensate Separator No. 1 Flash Condensate Separator No. 2
Vacuum Flashing	P3-11A	Vacuum Flash Preheater (Old) Vacuum Flash Feed Accumulator Drum
Vacuum Flashing	P3-11B	Old Vacuum Flash Drum Vacuum Flash Drum Preheater (New)

<u>Title</u>	<u>P&I Dwg. No.</u>	<u>Major Pieces of Equipment</u>
Vacuum Flashing	P3-11C	New Vacuum Flash Drum Vacuum Flash Condenser Vacuum Flash Condensate Drum Hotwell Tank Ejector System
Fractionation	P3-12	Light Ends Removal Column Light Oil Water Separator Wash Solvent Fractionator
Solvent Surge and Vent Disposal	P3-13	Process Solvent Accumulator Wash Solvent Accumulator Flare Knockout Drum Flare & Controls
Gas Recompression	P3-14A	Recycle Hydrogen Scrubber Recycle Hydrogen Compressor Scrubber Hydrogen Recycle Compressors A&B Fresh Hydrogen Storage A&B Fresh Hydrogen First Stage Scrubber Fresh Hydrogen Compressor
Gas Recovery	P3-14B	DEA Desulfurization Unit Naphtha Absorber Naphtha Flash Drum Naphtha Circulation Pump Naphtha Absorber Circulating Cooler
Product Solidification and Storage	P3-15	De-Ashed Coal Product Cooler De-Ashed Coal Product Conveyor off Cooling Belt
Utilities and Services - Inert gas and Hydrogen	P3-16	
Hydrogen/Syn-Gas Generation	P3-16A	
Inert Gas Generation	P3-16B	
Inert Gas Distribution	P3-16C	
Seal Flush and Solvent Flush	P-3-17	Flush Solvent Accumulator Seal Flush Accumulator Seal Flush Cooler Seal Flush Pumps Flush Solvent Cooler

1241125 03067047
WATER LEVEL CONTROL POT
12" DIA X 3' T
20 GAL
C.B.

NOTES:

1. FOR GENERAL NOTES SEE PFS 2002-X-1001
2. DURING MATERIAL RUNS, THERE IS NO FORWARD FLOW OF MIXED SOLVENTS TO DISTILLATION I.E. ALL LIQUID MATERIAL IS HELD IN THE WASH SOLVENT ACCUMULATOR.

DRAWING NO. 2002-X-102	
KEY	
	STREAM NO.
	TEMPERATURE
	PRESSURE (UNLESS OTHERWISE NOTED)
CONFIDENTIAL	

PRELIMINARY
9/29/78

04D56082
HOT WELL
OIL PUMP

04D67003
WASH SOLVENT
ACCUMULATOR
(WSA)
96" DIA X 85' T
C.S.

04D56083
WASH SOLVENT
PUMP
CENTRIFUGAL
15 GPM X 55 FT. TON
40 M.P.
C.I. INTERNALS

SCIENTIFIC DESIGN COMPANY INC.	
DESIGN	TWO PARK AVENUE NEW YORK
CONSTRUCTION	
SREX - F.L. WIS PILOT PLANT	
PROCESS FLOWSHEET	
FOR VACUUM FLASH AND WET SOLVENTS ACCUMULATOR	
DRAWN BY	DATE
CHECKED BY	DATE
APPROVED BY	DATE
No. 2002-X-102	

NO.	DESCRIPTION	DATE	BY	CHECKED

STREAM NO.		101	102	103	104	105	106	107	108
SERVICE		RAW COAL	WATER PURGE	DEHUMIDIFIED COAL	RECYCLED SLURRY	SEAL FLUSH	FEED SLURRY	HYDROGEN TO PREHEATER	PURGE HYDROGEN
COMPONENT	M.W.	LB/HR	WT%	LB/HR	WT%	LB/HR	WT%	LB/HR	WT%
HYDROGEN	2.0							256.5	118.2
NITROGEN	28.0							17.3	0.6
METHANE	16.0							267.9	16.7
ETHANE	30.1							46.6	1.5
PROPANE	44.1							30.8	0.7
BUTANE	58.1							11.0	0.2
CARBON MONOXIDE	28.0							93.3	3.3
CARBON DIOXIDE	44.0								
HYDROGEN SULFIDE	34.1								
AMMONIA	17.0								
WATER	18.0	267.7	9.8	253.7	100	14.0	0.6		
NAPHTHA						1.7	0.0	15.7	0.2
MIDDLE DISTILLATE						2.4	0.1	5.7	0.1
HEAVY DISTILLATE						829.9	12.0	276.6	32.9
SEC						1568.3	86.8	560.7	66.7
10M						1267.4	29.6		
ASH						291.3	6.8		
M.F. COAL		2464.0	90.2			2454.0	99.4		
						604.6	14.7		
								3454.0	32.5
TOTAL		2721.7		253.7		2468.0		4286.0	
								840.6	
								75646	
								703.3	141.2
									31.1
									6.1

RECYCLE
QUENCH WATER S
(PFS 2002-X-102)
HYDROGEN FROM RECYCLES
HYDROGEN COMPRESSORS
(PFS 2002-X-104)

CRUSHED COAL FROM
PRIMARY COAL CRUSHER

01033702
DRY GRINDING CIRCUIT
GRAVIMETRIC FEEDER
27,000 LB/HR
1/4 H.P.

01025501
DEHUMIDIFIER
SCRUBBER
3' DIA. X 15' 9" T-T
C.S.

0103
VIBRA SCREEN
4,000 LB/HR
1/2 HP
C.S.

C.W.R.
0103
DEHUMI
HEAT EXC
15675 SQ
2MM HTU
C.S.

01067035
HOT WELL
5' DIA. X 9' T-T
1500 GAL.
C.S.

WATER
PURGE

01044301
DRY PULVERIZER
70% MINUS 200 MESH
50 H.P.

01039801
DRY PULVERIZER
GAS PREHEATER
FIRED HEATER
2 MM BTU/HR
C.S. COIL

01013001
INERT GAS BLOWER
4000 IC FM
4.6 FT. WATER POLYTROPIC HEAD
60 HP

01066002
DEHUMIDIFIER DRAIN PUMP
CENTRIFUGAL
400GPM X 125 FT. TDH
25 HP
CI

1. FLOW AND COMPOSITION OF STREAMS
ARE PRESENTED HERE FOR ILLUSTRATIVE
PURPOSES ONLY.
VALUES WERE PROVIDED BY P&M BASED ON
SELECTED FLOWMETER AND ANALYTICAL
MEASUREMENTS OBTAINED DURING
MBR 78SR-12.

THERE VALUES WERE NOT REVIEWED FOR INTERNAL CONSISTENCY AND WERE NOT USED FOR DETERMINING THE REPORTED YIELDS.

2. TEMPERATURES AND PRESSURE SHOWN ARE
TYPICAL VALUES FOR SEC-II OPERATION.

5: - 146 - 4/49

PRELIMINARY
9/29/73

ANY PURCHASE OTHER THAN THAT FOR WHICH IT IS SPECIFICALLY FORWARDED WITHOUT THE PRIOR WRITTEN CONSENT OF

SCIENTIFIC DESIGN COMPANY INC.

DESIGN • TWO PARK AVENUE • CONSTRUCTION
NEW YORK

SRC II - FT. LEWIS PILOT PLANT

PROC-58 P. 010588T

FOR

COAL PREPARATION, REACTION, & SLURRY RECYCLE

DRAWN BY: _____ DATE: _____ SCALE: _____

CHECKED BY: _____ DATE: _____

APPROVED BY: _____ DATE: _____

No. 2002-X-101

05D75310
NAPHTHA ABSORBER
PACKED TOWER
1'-8" DIA. x 40' T-T
16' SS. PALL RINGS

NA
11
45

RECYCLE HYDROGEN

03D73230
NAPHTHA FLASH
DRUM
1100 GALS
45" DIA. X 12' T-T
C.S.

05D30722
NAPHTHA ABSORBER
CIRCULATION COOLER
352 SQ. FT. ; FTS
512,000 BTU/H2
C.S.

05D56121/122/123
NAPHTHA CIRCULATION PUMPS
SINGLE ACT. POT VALVE
RECIPROCATING
75 GPM X 2410 FT. TDH
75 H.P.
FORGED STEEL

05D
WATER CO
FRESH HYD
DOUBLE
87 SC
1.4 MM
C.

RECYCLE
OR "A"
ATING
STEEL

05018105
HYDROGEN RECYCLE
COMPRESSOR "B"
RECIPROCATING
25 CFM
125 HP
FORGED STEEL

05030705
HYDROGEN
RECYCLE EXCHANGER
DOUBLE PIPE
350 SQ. FT
162,000 BTU/HR
L.S.

05062109
RECYCLE HYDROGEN
SCRUBBER
KO POT
13" DIA. X 10' T-T
C.S.

PRELIMINARY
9/29/78

				SCIENTIFIC DESIGN COMPANY INC. CHIEF ENGINEER : TWO PARK AVENUE • CONSTRUCTION NEW YORK			
				SRC II - F-1 LEADS PILOT PLANT PROCESS FLOW SHEET FOR GAS TREATMENT AND RECOMPRESSION			
				DRAWN BY _____ DATE _____ SCALE _____ CHECKED BY _____ DATE _____ APPROVED BY _____ DATE _____			
NO. _____ DESCRIPTION _____ DATE _____ BY _____ CHECKED _____ REVISIONS _____				No. 2002-X-104			

MIXED SOLVENT
FROM WASH
SOLVENT
ACCUMULATOR
(PFS 2002-X-102)

04D75221
L.E. COLUMN
FEED TANK

84" DIA X 17'-0" T-T
5000 GAL
C.S.

04D560
LIGHT EN
FEED P

CENTRIF
70 GPM
1 1/2 HP

04018705
LIGHT OIL
CAVENDER CONDENSER

50 SQ. FT.; FT3
500,000 BTU/HR
C.S.

04075212
LIGHT OIL WATER
SEPARATOR

36" DIA X 8'-0" T-T
C.S.

04056002
LIGHT ENDS
PRODUCT PUMP

CENTRIFUGAL
3 GPM X 118 FT TDH
5 H.P.
C.S.

04030802
WASH SOLVENT
COLUMN REBOILER

695 SQ. FT.; U-BUNDLE TUBES
3.25 MM BTU/HR
C.S.

04056
WASH SOLV
BOTTOMS

CENTRIFU
65 GPM X
7 1/2 H.P.
C.S.

09030512
FLUSH SOLVENT
COOLER

52 SQ. FT. BARE SURFACE
1.76 MM BTU/HR
C.S. TUBES, ALUMINUM FINS

09067023
FLUSH SOLVENT
ACCUMULATOR

72" DIA X 25'-0" T
C.S.

09030610
FLUSH SOLVENT
HEATER

150 SQ. FT
1.1 MM BTU/HR
C.S.

TO MISC. FLUSH PTS AND SEAL FLUSH
ON OLD VACUUM FLASH BOTTOM'S
PUMPS

FLARE
HEADER

TO FLARE
STACK

04D76214
FLARE K.O. DR

96" DIA X 20'-6"
C.S.

FROM SLOPS
DRUMS

STEAM DRIVEN
RECIPROCATING PUMP

WATER DRAIN

2
ENT
IAL
10 FT TON

76214
K.O. DRUM
12'-0" T-T
C.S.

10 FLASH
11 EXCHANGER

						<div style="border: 1px solid black; padding: 5px;"> <p>_____</p> <p>_____</p> <p>_____</p> <p>DW</p> </div>
NO.	DESCRIPTION	DATE	BY	CHECKED	DRAWN BY _____ CHECKED BY _____ APPROVED BY _____	
REVISIONS						

DRAWING NO. 2002-X-103	
KEY
STREAM NO.
TEMPERATURE °
PRESSURE (UNLESS OTHERWISE NOTED)
CONFIDENTIAL	

PRELIMINARY
9/29/78

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SCIENTIFIC DESIGN COMPANY INC.			
	DESIGN • TWO PARK AVENUE • CONSTRUCTION NEW YORK			
	SRC II - FT. LEWIS PILOT PLANT			
	PROCESS FLOW SHEET			
	DISTILLATION AND SEAL FLUSH SYSTEM			
NO. _____		DRAWN BY _____	DATE _____	SCALE _____
REVISIONS		CHECKED BY _____	DATE _____	APPROVED BY _____
		No. 2002-X-103		

NO.	DESCRIPTION	DATE	BY	CHECKED