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SOLVENT REFINED COAL (SRC) PROCESS

Quarterly Technical Progress Report for Period April-June 1979

February 1980

Work Performed Under Contract No. AC01-76ET10104

The Pittsburg and Midway Coal Mining Company
Denver, Colorado

U. S. DEPARTMENT OF ENERGY

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QUARTERLY TECHNICAL PROGRESS REPORT
FOR THE PERIOD
APRIL 1979 THROUGH JUNE 1979

THE PITTSBURG & MIDWAY COAL MINING CO.
Denver, Colorado 80222

February 1980

PREPARED FOR THE
U. S. DEPARTMENT OF ENERGY
DIVISION OF COAL CONVERSION AND UTILIZATION
UNDER CONTRACT DEAC-0179-ET10104
(formerly EX-76-C-01-0496)

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ABSTRACT

This report summarizes the progress of the Solvent Refined Coal (SRC) Project by The Pittsburgh & Midway Coal Mining Co. for the Department of Energy for the period March 25, 1979, through June 24, 1979. SRC-I turnaround activities at the Fort Lewis Pilot Plant were complete March 26, but several problems developed which delayed successful startup until April 11. Slurry was injected to the Lummus Deashing Unit for the first time April 12. After approximately eight days, slurry feed was diverted from the Lummus system because of indications that solids were accumulating in the settler vessel. The Fort Lewis Pilot Plant was shut down from April 28 to May 12, 1979, while the Lummus Unit settler vessel was hydroblasted. Cleaning of the settler vessel was complete May 10. Modifications to the unit were nearly complete at the end of the reporting period. Slurry feed to the Lummus Unit was reestablished June 15. To date, continuous production of specification product at design rates has not been achieved. One material balance run, MBR 79-1, was completed using Kentucky No. 9/14 coal at proposed design conditions for the SRC-I Demonstration Plant. Process Development Unit P-99 was on-stream for all of the second quarter of 1979, with the exception of a two-week turn-around in May and the last week of June, when the unit was shut down for a scheduled turn-around. During this period, six different run conditions were tested feeding Pittsburgh Seam coal from the Powhatan No. 5 Mine.

I. SUMMARY OF OPERATIONS

The SRC Pilot Plant processed approximately 1657 tons of Kentucky No. 9/14 coal (Colonial Mine) and 155 tons of Pittsburgh Seam coal (Powhatan No. 5 Mine) in the second quarter of 1979. During this period the plant was in operation 54 days. All of the operations with Colonial coal were in the SRC-I mode and were primarily directed at providing feed for the Lummus Antisolvent Deashing Unit. Dissolver conditions were normally maintained at 1500 psig total pressure and 850°F outlet temperature, but a brief period of operation was made at 850°F and 2000 psig while conditioning raw solvent, and a six day material balance run was made at 825°F and 1700 psig. Operations were also carried out with Powhatan No. 5 coal at similar SRC-I conditions for two days and at SRC-II conditions of 850°F and 2000 psig for six days. Dissolver A was used at one-half volume throughout the reporting period.

Turnaround activities begun earlier in the first quarter were completed March 26, but a leak in the Wash Solvent Column reboiler flange, a broken trim in the High Pressure Flash Drum level control valve, and leaking Grayloc flanges in the high pressure area delayed successive startup attempts on March 29, 30 and 31. Repair of the Grayloc flanges curtailed coal feed until April 8. A brief period of operation was then required for conditioning the remaining raw solvent inventory.

The first attempt to introduce feed to the Lummus Unit was unsuccessful because of a construction error in the transfer line from the Recycle Slurry stripper to the topped feed surge drum. A shutdown was taken for repair of the line and coal feed was resumed April 11. Slurry was fed to the Lummus Unit for the first time April 12 at approximately one half of design rate.

Initial data showed specification ash content in overflow streams, but ash levels began to increase after three days of operation. Occasional flushing of the individual settler underflow lines was required to eliminate intermittent plugging. In addition, gas accumulated in the settler vessel, which was intended to run liquid-full, and this necessitated periodic manual venting. After the fourth day of operation, the ash balance on both the settler and the overall pilot plant began to indicate substantial buildup of ash in the system. An unsuccessful attempt was made to clear the settler by using underflow recirculation and by removing the taylor wedge flowmeters located in the individual settler underflow lines. On April 20, slurry feed to the Lummus Unit was stopped and the process of flushing, cooling, and securing the settler vessel for inspection was begun. The reaction and solvent recovery areas of the plant continued operation until April 28 to process accumulated slop material and recover process solvent.

It appears that this major upset of the Lummus system was the result of an inappropriate startup philosophy for the settler. The initial procedure selected by Lummus involved conditions at design underflow rate and antisolvent concentration with one-half of design feed rate. The resultant doubling of design settler

residence time likely led to an overprecipitated condition and subsequent accumulation of ash in the settler.

Between April 27 and May 10, a major maintenance effort was expended to remove the solids which had accumulated in the settler. Approximately 150 drums of material were removed. Several Lummus Unit modifications were also made including installation of a liquid level probe to allow automatic venting of the gas bubble formed in the settler vessel during operation and piping modifications to permit recirculation of antisolvent-free overflow from the fractionator bottoms to the overflow compartment of the settler vessel.

While the Lummus Unit was down for cleaning of the settler, the plant was operated using Colonial Mine coal in preparation for material balance run 79-1. This run was begun on May 15 for the purpose of providing yield data at conditions of one of the SRC-I Demonstration Plant design cases. After correcting some preliminary problems with plant chromatographs, the yield distribution appeared to be fairly typical of SRC-I operation with Colonial Mine coal. The material balance run was discontinued on May 21 due to delays in delivery of Colonial coal. On May 22, Powhatan No. 5 coal was introduced to provide vacuum bottoms necessary to process an accumulation of miscellaneous recoverable liquids needed to ensure an adequate inventory of SRC-I process solvent. On May 24, the plant was switched to SRC-II operation to provide additional reaction data at low coal concentration and high coal space rate in the dissolver while at the same time processing recoverable liquids in the solvent recovery area.

On June 9, Colonial coal was again fed in the SRC-I mode to produce feed for the Lummus Unit. A coal feed rate of 3300 lb/hr and recycle process solvent rate of 4580 lb/hr were used. Dissolver outlet temperature and pressure were 850°F and 1500 psig respectively. On June 11, an 18-hour coal outage was taken to replace four leaking bags in the coal pulverizer dust collector. On June 15, feed rates were raised (4505 lb/hr coal and 6250 lb/hr recycle process solvent) to satisfy the feed requirements of the Lummus Unit. These rates were maintained until the end of the reporting period.

Slurry was fed to the Lummus Deashing Unit on June 15 at design rates (instead of the substantially reduced rates of the initial startup) and at below design antisolvent-to-feed and overflow-to-underflow ratios. While the settler ash balance was continually monitored, the antisolvent-to-feed and overflow-to-underflow ratios were gradually increased by increments in order to achieve production of specification product at an acceptable SRC recovery rate. Slurry feed was removed on June 18 and returned on June 19. Sustained production of specification product had not been achieved at the end of the reporting period.

During April, the new vacuum flash drum was used to conduct tests to investigate vapor-liquid equilibrium and solids carryover. Results showed a direct dependence of condensate recovery per unit of feed on flash zone temperature. In addition, operation of the

system at vapor velocities up to 5.5 ft/sec with negligible carryover was demonstrated.

Beginning in May, the naphtha unit was utilized as an extra knockout pot between the DEA unit and the recycle hydrogen compressors. Also, during the period, the distillation columns were utilized to produce light oil, wash solvent and process solvent to SRC-I specifications.

In addition to material balance run 79-1 and tests on Vacuum Flash Drum B, a number of pilot plant special studies were made during both the first and second quarters of 1979. A study of SRC II plant startups showed that the gas stream from the dissolver reaches equilibrium much faster than does the slurry stream because of the shorter gas residence time within the dissolver. Relationships between hydrogen consumption and methane production versus temperature were studied to obtain data which will be useful for weight balance studies over relatively short operating periods. Another startup aspect studied was the buildup of heavy products in the vacuum flash feed. The study suggests that it may be possible to improve vacuum flash operations by limiting the amount of slurry fed to the vacuum flash system during the early stages of an SRC-II startup.

Because the pilot plant vacuum flash unit operates at less severe conditions than are presently envisioned for use in the demonstration plant, a new laboratory vacuum flash test stand was designed. This unit will be used to explore the effects of equilibrium flash conditions on vapor-liquid equilibria and product physical properties for use in SRC-II unit design.

New equipment was obtained and was being used to gather viscosity data on SRC-II feed slurry, recycle slurry, and vacuum bottoms over a wide range of conditions. The ultimate goal of the program is the development of a mathematical model of viscosity behavior as a function of the major intensive and extensive variables within the limits of commercial significance.

Work continued on the installation of the new slurry preheater. Structural steel was about 90% complete and shop fabrication of piping approximately 45% complete. Problems of stress in the piping system were considered to be resolvable by cold springing during construction. Because of late delivery of many critical items, it appeared a plant shutdown for new equipment installation would be deferred beyond August 1.

The Process Development Unit P-99 operating period that had been started in March was continued through May 4, during which time three additional runs, Runs 50, 51, and 52, were completed. After a two week turn-around, the unit returned to operation and completed three more runs, Runs 53, 54, and 55, before shutting down for a scheduled turn-around near the end of June. All runs were made feeding Pittsburgh Seam coal from the Powhatan No. 5 Mine.

II. PILOT PLANT OPERATIONS, ENGINEERING, AND MAINTENANCE

A. Coal Receiving and Preparation (Area 01)

In the second quarter of 1979, approximately 1657 tons of Kentucky No. 9/14 coal (Colonial Mine) and 155 tons of Pittsburgh Seam coal (Powhatan No. 5 Mine) were processed. The plant was shut down for various turnaround activities for three extended periods during the quarter (March 25 - April 8, April 28 - May 12, and May 30 - June 9) totaling almost 42 days. The hourly onstream factor, adjusted for downtime not directly caused by process problems or equipment failures, was 94.1%. A summary of coal processing in the second quarter of 1979 is given in Table A-1.

On April 8, the plant was brought on stream with Colonial Mine coal and through April 10 approximately 8000 gallons of raw solvent (coal tar distillate, 480°F IBP, less than 5% vacuum bottoms) were processed to increase the plant process solvent inventory in preparation for SRC-I operation. During this period, the coal and raw solvent feed rates were each 2000 lb/hr. In addition, a recycle slurry rate of 3000 lb/hr was used to serve as a diluent for the raw solvent because of a low process solvent inventory and because it was felt that operating with partial recycle would increase the process solvent yield.

From April 10 to April 28 the plant was operated in the SRC-I mode with Colonial Mine coal at a coal feed rate of 3500 lb/hr and a recycle process solvent feed rate of 5000 lb/hr. Operations during this period were primarily intended to furnish feed to the Lummus Antisolvent Deashing Unit which operated from April 12 to April 20 in its initial test. The entire plant was shut down from April 28 to May 12 to hydroblast the Lummus settler vessel of solids deposits. While work on the Lummus Unit was underway, several plant repairs were also completed.

On May 12, Colonial coal feed was resumed at 2675 lb/hr and a recycle process solvent rate of 4000 lb/hr to line out for Material Balance Run No. 79-1. This material balance run was conducted from May 15 to May 21 and its purpose was to measure the yield distribution of Colonial coal at conditions of a current SRC I demonstration plant design case.

On May 22, the feed was switched to Powhatan coal at similar SRC-I rates to provide vacuum bottoms necessary to process an accumulation of miscellaneous recoverable liquids through Area 04. Operations with Powhatan coal were shifted to the SRC-II mode on May 24 to evaluate short residence time SRC-II operation and to augment process solvent inventory. The flow rate of reacted coal slurry from Area 02 to the vacuum flash system was checked at this time by measuring level increases in the Filter Feed Flash Vessel. On May 30, the plant was shut down because the Lummus Unit was not ready for feed and

because the inventory of Colonial coal was low and delivery schedule uncertain.

On June 9, Colonial coal feed was established in the SRC-I mode at a feed rate of 3,300 lb/hr and a recycle process solvent feed rate of 4,580 lb/hr. On June 10, start up operations were initiated on the Lummus Unit, and on June 15, product slurry was fed to the unit. At this time, the coal feed rate was raised to 4,505 lb/hr and the recycle process solvent to 6,250 lb/hr. These rates were maintained through the end of the reporting period.

Coal surge bin plugging was a source of outages throughout the quarter. Gas activated Pulsonic bin activators were ordered and were scheduled to be shipped in July. They will be installed at the first opportunity and should alleviate this problem.

A major coal outage caused by the coal pulverizer dust collector occurred on June 11. Four bags were found to be torn and were replaced. In addition, nine out of the thirteen pulse jets were found to be defective. Most of the pulse jets were repaired, but not enough spare parts were available for all. As a result, subsequent operation of the baghouse was at higher than normal pressure differential. All bags in the baghouse will be replaced during the August turnaround and the remaining pulsors repaired.

The high pressure slurry charge pumps required a considerable amount of maintenance during the reporting period. Charge pump A required new plunger packing on May 14 and again on May 23. Charge pump B required new packing on May 20 and its pressure safety relief valve was repaired on May 23.

On May 15, Slurry Circulation Pump No. 3 was removed from service to repair a broken coupling. A routine inspection revealed the casing was extremely thin and a hole had almost eroded through the neck of the suction nozzle. The wear ring and impeller were also badly eroded. The pump was reinstalled with a new casing, but with the same impeller since a spare was unavailable.

On May 21, the slurry recycle flow control valve (FCV-1152) plugged during a routine attempt to establish circulation. When the valve was removed for repair, it was discovered that the tip of the plug had broken and lodged in the seat. It was also noted that the valve body was very badly worn and required replacement. A spare 1,500 psig, 316 stainless steel valve body for the Intermediate Pressure Flash Vessel level control valve (LCV-175) was fitted with a 6" extended tungsten carbide seat. New 1/2" Micro-Form tungsten carbide trim was installed and the valve was installed as FCV 1152 and returned to service.

During the plant shutdown of April 28 to May 12, the following area work was completed.

1. The dehumidifier demister pad was removed and a new pad installed. The old pad was partially plugged with coal fines and dust particles, which caused water carryover into the Inert Gas Blower.
2. The Inert Gas Blower shell was ultrasonically tested and found to be very thin and severely pitted. It was recommended that new plates be welded inside the shell on the next shutdown.
3. The Inert Gas Heater was inspected on May 3. The metal floor immediately around the burner was burned out and several other large cracks were noted. Repair was made by covering the entire floor with standard refractory and curing. The heater will be inspected again on the next shutdown.

Two new steam-heated coal predryers were installed and operated during the quarter. The new dryers were designed for 200 psig instead of 50 psig steam pressure and should facilitate operation and simplify maintenance. Initial operation has been very successful, with rates 3 to 4 times that of the old dryers. Very little maintenance has been required and one dryer has been able to handle the full plant rate.

Work was done on the redesign of the coal slurry recirculation loop. The primary objectives of redesigning the slurry recirculation system were to increase the capability of pumping higher viscosity slurries, to decrease the level that is required in the slurry blend tank to maintain net positive suction head to the recirculation pumps, and to improve the reliability of the system. The capability of pumping higher viscosity fluids would be increased by relocation of the circulation pump to directly below the slurry blend tank. The new pump location would eliminate several bends in the suction line and as a result improve the available net positive suction head. The slurry blend tank would also be raised approximately 4 feet in order to allow lower operating levels in the slurry blend tank during residence time studies.

The main improvement in the reliability of the system should result from the piping modifications. The major problem with the existing system has been the plugging of the suction and discharge piping to the two spare circulation pumps. The plugging was the result of pipe dead legs to and from the spare pumps. One of the two spare pumps could not be reinstalled without compromising the piping configuration. One existing circulation pump (a Morris pump) was not rated for the pressure and temperature needed for SRC-II operation and was not as reliable as the other two pumps (both Durco) for SRC-I operation so no significant reduction of process reliability was incurred by its removal. The new recirculation system will incorporate a Durco pump and partially lined Lawrence pump. Lawrence considers the partially lined pump

to be their "first" generation slurry pump. A fully lined "second" generation pump will be ordered, but the delivery of this pump is currently 30 weeks; therefore, it will not be available until after the 1979 shutdown.

In addition to the above modifications, two separate systems for measuring recirculation flow rate will be installed. One system will incorporate a quadrant edge orifice and the other a straight length of pipe.

The pressure drop across both the orifice plate and the length of pipe will be used to calculate flow rates. The above flow rates will be used to evaluate performance of the pumps. A schematic of the new system was prepared and reviewed by both Operations and Engineering. Once the final design changes are complete, the drawings will be sent to Hallanger Engineers for stress analysis of the piping design.

The coal hopper and related chutes were ultrasonically inspected. The coal hopper was found to be in satisfactory condition; however, the chute above the belt was found to be thin and required repair. The chute will be replaced during the turn-around.

B. Slurry Preheating and Dissolving (Area 02)

Area 02 was put on stream on April 8 with Colonial Mine coal and raw solvent at 2000 psig pressure and 850°F. Colonial coal was processed from April 10 to April 28 in the SRC I mode at 1500 psig pressure and 850°F outlet temperature. When operations were resumed with Colonial coal on May 12, the dissolver outlet temperature was 820°F and pressure was 1700 psig. Similar conditions were used during SRC-I operations with Powhatan coal starting on May 22. Powhatan coal SRC-II operations from May 24 to May 30 were at 850°F dissolver outlet temperature and 2000 psig pressure. Colonial coal processing in the SRC-I mode began again on June 9 with dissolver outlet temperature and pressure at 850°F and 1500 psig, respectively, and continued through the end of the quarter. Dissolver A was used at one-half volume throughout the reporting period.

Numerous problems in Area 02 were responsible for operating curtailments during this period. On March 30, with the area on circulation, both the Fisher and the Willis control valves on the High Pressure Flash Drum became at least partially restricted. Attempts to clear these valves were not successful, so each, in turn, was removed for inspection. The Fisher control valve was found to be plugged by the tungsten carbide trim insert which had come loose from its heat-shrunk stem fitting. A new trim was installed and arrangements were made with the manufacturer for testing of future trim assemblies to prevent this problem. At this time, the internals of the coke trap which had been installed upstream of the Fisher control valve, were removed. The perforated baffle plate serving as a

strainer had several plugged holes and it was feared that if many holes became plugged, high velocities through the remaining holes could lead to impingement erosion of the coke trap body and a potentially hazardous condition.

Inspection of the Willis control valve revealed nothing which would result in plugging, but the assembly was barely able to pass circulation flow. One week later, following repairs to other equipment, a second test on the Willis control valve showed it was still unable to control the High Pressure Flash Drum level. A new trim assembly, having two 1/4 inch holes rather than the original 1/8 inch holes, was installed. The valve was then able to operate at approximately 50% open with a coal feed rate of 3500 lb/hr and a solvent rate of 5000 lb/hr. During inspection of the Willis block valves, indications of corrosion attack were found on the Belleville washers, which are made of Inconel X-750. They will be replaced with Inconel A-286 washers at the earliest opportunity.

There were three separate instances of leaking Grayloc flanges during the reporting period. The two leaks (dissolver inlet and downstream of the erosion/corrosion loop) that affected the April plant start-up were the result of mechanical damage that occurred during the shutdown. The third leak, which occurred at the spool piece that replaced an obsolete valve, did not affect plant operation. The cause of this leak has not been determined. The first two leaks were repaired by Grayserv. In addition to repairing flanges, the Grayserv representative conducted a school for the Maintenance and Engineering staffs concerning the maintenance of Grayloc flanges.

The repair of the Grayloc flange on the inlet to the dissolver was complicated by the fact that the base metal of the vessel is 2-1/4 Cr. In order to minimize cost, lost time, and potential problems of field welding and stress relieving a 2-1/4 Cr pressure vessel, the flange was reworked without welding on the vessel. As shown in Figure A-1, the "A" dimension of a Grayloc flange has to be maintained within specification in order for the flanges to seat properly with the seal ring. Normally the "A" dimension is kept constant by weld overlaying the damaged flange and then remachining the sealing surface. In this particular case, the weld metal was added to the mating hub (347 SS), which could be easily welded. Approximately 0.060" of metal was machined from the face of the vessel nozzle and an equal amount added to the mating hub. Since it is undesirable and probably not possible to do further machining (without welding) on the vessel nozzle, an extra spool piece was added to the piping below the vessel inlet. This spool piece will not be removed so that the vessel nozzle will be protected from further damage.

On April 10, after an unsuccessful attempt to establish flow from Area 02 to the Lummus Unit, it was discovered that a sockolet had been installed in the line without drilling the

pipe. This was installed during the March shutdown when the eroded carbon steel Recycle Slurry Stripper bottoms piping was replaced with stainless steel. The problem was corrected and flow to the topped feed surge drum of the Lummus Unit was established.

On April 20, a small leak developed in a Grayloc flange in the line between the dissolver and the High Pressure Flash Vessel. The leak did not affect the operation materially, but on April 28 the plant was brought down for this and other repairs. The flange was repaired by Grayserv. During this period, two leaking block valves on the Recycle Slurry Stripper bottoms piping were replaced, and the existing steam/hydrogen preheater was relocated in compliance with plans for the new slurry preheater.

On May 12, while on solvent circulation prior to startup, the Willis control valve on the High Pressure Flash Vessel was found to be plugged. System circulation was stopped, pressures were adjusted, and the control valve was backflushed. This cleared the valve, which was then put on standby. Two hours after coal injection, as part of the startup procedure, the valve was put back in service and operated reliably through May 30.

During the May operations, the level control valve of the Recycle Slurry Stripper became unable to control process flow. The bypass valve was then used to throttle flow. Repairs were later made to this system during the May shutdown.

At the request of Process Evaluation, a tie-in was made on the line between oil/water Separator No. 3 and the Recycle Slurry Stripper to allow the direct addition of process solvent to the stripper during future SRC-II operation instead of adding it as extra seal flush in Area 01. Piping and instrumentation had not been completed at the end of the reporting period.

A routine inspection of the heat tracing on the dissolver revealed several faulty thermostats. In these particular cases, failure of heat tracing caused no damage to the vessel. However, in other types of failure there was the potential for severe damage to these critical high pressure vessels and it was, therefore, decided to disconnect the heat tracing on both the dissolver and the High Pressure Flash Vessel. The heat tracing is to be reconnected by an electrician only as required and then disconnected after that particular need is over. Heat tracing on the Intermediate Pressure Flash Vessel remained connected since it is necessary to keep this vessel warm when the vessel is pressurized due to the temper embrittlement that was incurred during its manufacture.

A computer program was written and necessary electronic hardware was added so that the valve positions (controller output) of

both the high pressure flash level control valves and the intermediate pressure flash level control valves could be monitored as a function of time. The approximate flow rate and pressure drop across each valve is also monitored and recorded. Examples of data are shown in Figure A-2. These data will be useful in predicting and evaluating the life of various abrasion resistant trim materials.

Since the erosion/corrosion test loop does not appear to be losing metal at the present velocities, half of the loop is going to be changed from 2 inch schedule XX (1.503" I.D.) to 1-1/2 inch XX (1.110" I.D.) as shown in Figure A-3. This should increase the velocities from 20-30 feet per second to 35-55 feet per second. All pipe and fittings should arrive in time to be installed during the August turnaround.

A meeting was held with Dr. Arthur Youngdahl of Argonne National Laboratory to discuss details of installing ultrasonic equipment to automatically monitor metal thickness in the erosion/corrosion test loop while the loop is operating (860°F). The system can automatically scan up to 30 points. The present schedule is for the system to be installed and operating before the completion of the August turnaround.

C. Mineral Separation (Area 03)

The new Lawrence pump which circulates the contents of the Filter Feed Surge Vessel was returned to service on March 27. It had been taken out of service on March 16 due to a seal failure after 300 hours of service. The pump casing was found to be worn when inspected. Upon the recommendation of the Lawrence Company, a smaller impeller was installed and the pump returned to service with the original worn casing. The small impeller will allow more clearance between the impeller and the case and lower the flow rate through the pump, reducing casing wear. The original impeller was sized for a higher discharge pressure than is normally encountered in the circulation of the Filter Feed Surge Vessel material.

During the plant shutdown in May, a new 1" line was installed from the Recycle Process Water Tank (RPWT) to the Light Ends Column Feed Tank to prevent the recurring problem of the RPWT oil pump shutting down on overload.

The Filter Feed Surge Vessel recirculation line, 2" SL-107, was ultrasonically tested and found to be very thin at one of the elbows (see Figure A-4). This particular elbow showed a 36% loss of nominal wall thickness. The thinned section of line will be replaced with 304L or 316L stainless steel during the annual shutdown.

Ultrasonic testing of the process (slurry) side of the Filter Feed Flash Vessel and Filter Feed Surge Vessel recirculation

exchanger tubes, removed in March, 1979, was completed in the shop (see Figures A-5 and A-6). Although the metal loss did not indicate an accelerated erosion/corrosion problem, removal and replacement of the tubes was justified by the fact that there had been two serious fires in this service during the past two years.

On May 24, during SRC-II operation with Powhatan coal, a special test was performed to check the flow rate of slurry leaving Area 02. This test is discussed in Section IV - Pilot Plant Special Studies.

During SRC-II operation from May 25 to May 30, coal slurry from Area 02 was diluted in the Filter Feed Flash Vessel with material from the Vacuum Flash Condensate Drum. The primary reason for this dilution was to permit operation of the old vacuum flash system at normal fresh feed to recirculation flow ratios and thus reduce the solids content of material circulating in the vacuum flash system. This was necessitated by the relatively low flow rates and high solids content of vacuum flash feed in SRC-II operation and the minimum flow requirements of the old vacuum flash preheater.

D. Solvent Recovery (Area 04)

Prior to resuming coal feed on April 8, the old vacuum flash system was run intermittently in a "stripping mode". Startup problems and resultant flushing and draining operations in Area 02 had created a large accumulation of miscellaneous solvent material in the Filter Feed Surge Vessel and the Vacuum Flash Feed Accumulator. Because there was essentially no vacuum bottoms in this material, it could not be processed in Vacuum Flash Drum A to a normal process solvent/vacuum bottoms cutpoint (the heavy ends of process solvent do not solidify on the Sandvik belt). Thus, the flash drum was operated at full vacuum and a drum temperature of 450°F (instead of the normal operating temperature of 600-630°F) to strip off the light oils and the lower portion of process solvent. This overhead fraction was then processed through the distillation columns to increase the process inventory.

The vacuum flash bottoms from this operation were returned to the surge vessels for the processing when coal feed could be established.

Area 04, operating with the old vacuum flash system, routinely processed coal slurry from Area 02 (or the combined anti-solvent-free overflow and underflow streams from the Lummus Unit when it was in operation) throughout the rest of April. In order to provide pump seal flush and flush solvent which would not contaminate the recovered antisolvent in the Lummus Unit, the target initial boiling point of the process solvent was set at 480°F.

During May operations, while the Lummus Unit was down, material from Area 02 was routinely processed with the old vacuum flash system. Some problems were experienced on May 5 with leak-through of the level control valve on Vacuum Flash Drum A which controls the flow of vacuum bottoms to the Sandvik cooling belt. When the block valve upstream of this control valve was closed to control the flow, the hand wheel broke. The system was drained and the block valve was repaired on May 7.

The old vacuum flash system processed slurry feed from Area 02 diluted with vacuum flash condensate to approximately 9% solids (pyridine insolubles), during SRC-II operations in late May. Plugging problems were encountered with the pressure control valves at the inlet to Vacuum Flash Drum A and with the bottoms level control valve. Repairs were made and on June 1, the vacuum flash system was returned to service to process all of the remaining slurry produced during the SRC II operating period.

During the early June shutdown, maintenance work was done on the old vacuum flash system. The unit was returned to service on June 9 and routinely processed SRC-I streams (from Area 02 or the combined effluent streams from the Lummus Unit) through the end of the reporting period.

The distillation columns were utilized to produce light oil, wash solvent and process solvent to SRC-I specifications throughout the reporting period. A process solvent initial boiling point at 480°F was required to provide pump seal flush and flush solvent which would not contaminate the recovered antisolvent in the Lummus Unit.

Numerous problems were encountered during this quarter. During the March-April shutdown, a new Wash Solvent Column reboiler was installed. On startup, the flange joining the reboiler to the column would not seal because the original double-jacketed asbestos gasket was damaged during installation. The leak was finally sealed by using two asbestos gaskets in conjunction with a spacer ring. Replacement double-jacketed gaskets were ordered and will be installed during the annual shutdown. New liquid level lines fabricated from 316L stainless steel were installed on the Wash Solvent and Light Ends Columns as a result of corrosion of the original carbon steel lines.

On March 30, the main Wash Solvent Column bottoms pump seal failed and on April 1, a leak developed at a weld on the suction line of this pump. Necessary repairs were made on both occasions. On April 20 the main vacuum flash feed accumulator pump coupling was replaced. Upon switching to the spare pump to work the coupling, it was discovered that the discharge line of the spare pump was plugged. The bonnet of the discharge check valve was removed, and the plug located and cleared.

On April 17, the new Dowtherm distribution system for the new Vacuum Flash Drum B was pressure tested. This distribution system consists of a remote valve manifold from which the flow of Dowtherm and thermal relief protection can be controlled to each of 12 separate Dowtherm flow circuits. The circuits supply Dowtherm to jacketed piping and valves in the new vacuum flash system. A similar distribution system had already been put in service on the Vacuum Flash Drum A system.

The Wash Solvent Column was ultrasonically tested on May 1. The upper section of the 316L column and, in particular, the area above the feed tray, showed significant corrosion (see Figure A-7). One reading was as low as 0.150 inches, a 40% loss of nominal wall thickness. Based on the installation date of April 1976, this is a minimum corrosion rate of 33 MPY of actual operating time. Additional ultrasonic testing will be done to determine if the problem is localized or general and if the high rate of corrosion is continuing.

On May 9, the demister pad was removed from the old vacuum flash drum in order to determine the amount of solids entrainment without a demister.

On May 22, the old vacuum flash system was shut down and the two stage steam eductor vacuum system was inspected. The steam nozzle on the secondary eductor was found to be loose, allowing steam to bypass the jet and cause poor performance. The nozzle was repaired and the system was returned to service.

On May 27, the level control valve of Vacuum Flash Drum A which controls flow to the Sandvik cooling belt again leaked through excessively with the valve fully closed. A block valve was used to control this flow. On May 30, the system was shut down, the bonnet of the control valve was removed, and a piece of what appeared to be demister mat (removed May 9 from the flash drum) was found.

On June 2, the heater coil of the old vacuum flash preheater was X-rayed to check for coke build-up. Only small deposits were found, but to ensure trouble-free operation during future Lummus tests, the coil was steam spalled. Both inlet pressure control valves were inspected and were found free of plugs. The upstream piping to the valves was removed and found to contain coke deposits. The deposits were removed and the piping reinstalled.

On June 23, a leak developed on the Wash Solvent Column feed line. The column was cooled and depressured and an asbestos patch was installed to allow a few hours of operation until a new section of line could be fabricated. The column was again cooled and depressured, the new section of line was installed and the column was returned to service.

The results from an ultrasonic survey of the corroded line are shown in Figure A-8. The wash solvent column was also inspected and there appeared to be no significant corrosion since the previous inspection (5-1-79).

A corrosion inhibitor injection system was installed to test several Betz filming and neutralizing amines in the wash solvent column. Results of these tests will be documented in future reports.

E. Gas Compression and Naphtha Unit (Area 05)

The naphtha recycle gas scrubbing unit was isolated from the rest of the plant on April 5, as it was not routinely used during SRC-I operations. In June the absorber vessel was put into use as a knockout pot between the DEA unit and the recycle hydrogen compressors in an attempt to relieve some compressor mechanical problems. The recycle hydrogen gas stream was routed through the naphtha absorber vessel with no naphtha circulation. No significant accumulation of liquid in the absorber was observed.

The pressure safety valve on the discharge of the fresh hydrogen compressor was reworked on March 31 after it was found to be leaking.

The valves in Recycle Hydrogen Compressor A were replaced on March 26. On April 6 and 7 the suction pressure safety valve on this compressor leaked through on two occasions. After the safety valve was reworked for the second time on April 7, no further problems were encountered.

Recycle Hydrogen Compressor B shut down twice on April 22 because of low oil pressure. These shutdowns were hard to explain because there was an adequate amount of oil in the machine. Recycle Hydrogen Compressor B was shut down on May 16 due to loss of capacity. A cracked valve disc on one of the discharge valves was repaired and the compressor was returned to service.

On May 30, the Fresh Hydrogen Compressor was shut down to repair the second stage suction valves and first stage pressure safety valve. When the compressor was returned to service, it was found that the third stage suction valves and second stage pressure safety valve also required maintenance. The third stage rings were also replaced.

During the April 28 shutdown, all the cooling water jackets on the area hydrogen compressors were acid cleaned. The operation was done by an outside contractor using an inhibited hydrochloric acid. Also two hot taps were made in the area cooling water supply for the future installation of a tempered cooling water

system. This system will supply 130-140°F jacket cooling water for all compressors in an effort to prevent condensation and the associated problems of excessive ring and valve wear.

During the June shutdown, the discharge piping check valve of Recycle Hydrogen Compressor A and a check valve on the common discharge line of both recycle compressors were replaced. A new cylinder liner and rings were installed in the Fresh Hydrogen Compressor. New cylinder liners, pistons, rods and packings were installed in both recycle hydrogen compressors.

F. Waste Treatment (Area 9.1)

Waste treatment operation was satisfactory during the second quarter of 1979 as contrasted to its generally substandard performance in the first quarter.

On March 29, an outside contractor finished the removal of 185,000 gallons of a heavy oil/water/solids emulsion from the bottom of the surge reservoir. This material was an accumulation of process derived heavy oils and solids which came from plant spills and plant runoff. The material was removed to off-site storage tanks to be held for future recovery of process solvent.

From May 14 to May 18, approximately 16,000 gallons of sour water were returned from offsite storage to the surge reservoir to be processed in the waste treatment area. This water had separated from the oil/water/solids emulsion removed from the surge reservoir in March.

A centrifuge was installed during April to process the sludge streams from the reactivator and flotation units. Its purpose was to concentrate the heavy oil and solids in these streams so they could be disposed of more economically. Due to severe vibration problems and resultant maintenance work, the centrifuge was run only intermittently. On May 10, the auger and bowl were sent out to be rebuilt, but this did not solve the vibration problem. On May 18, a new auger and bowl were installed, but the vibration persisted. The centrifuge was then sent out to be balanced, and it has remained out of service through the end of the reporting period.

On June 17, a new oil mop system was put in service to remove light oil from the surface of the surge reservoir. This system consists of a closed loop oleophilic rope which is pulled across the surface by a wringer which also squeezes the absorbed oil into drums. It is used intermittently whenever a significant amount of light oil has accumulated.

The charcoal filters were put in service from June 12 to June 15 to maintain plant effluent specifications.

G. Tank Farm (Area 9.2)

Approximately 111,000 gallons of SRC-II fuel oil were blended during April. This material was blended at a 2.9 middle distillate to heavy distillate ratio, as directed by the DOE.

From May 14 to 18, approximately 17,000 gallons of oil/water/solids emulsion from off-site storage were returned to the tank farm. The water was boiled off in steam-heated atmospheric storage tanks to prepare the remaining oil/solids phase for processing in Area 04 to recover process solvent.

H. Cooling Tower (Area 9.3)

The flowmeter for cooling tower makeup water remained out of service the entire reporting period because of difficulties in obtaining repair parts. As the meter controlled the addition of chemicals to the system, they were being added manually.

I. Gas Systems (Area 9.5)

Stretford Unit

Several instances of Stretford solution carry-over into the flare system occurred. On April 10, the sour gas absorber began venting solution to the flare. An inspection of the absorber showed that the demister pad in the overhead knockout had fallen from its supports and also that the drainback line from the knockout was plugged. After the overhead knockout was repaired and reinstalled, solution circulation was started, but venting due to excessive foaming occurred on April 13 and 19. On May 21, solution venting due to excessive foaming was again experienced. During June, a differential pressure transmitter was installed to monitor the pressure drop across the absorber and give a warning of excessive foaming of Stretford solution in this vessel.

Several lines in the Stretford unit were ultrasonically inspected. As shown in Figure A-9, there was up to 0.070" metal loss; however, the operating temperatures and pressures were low enough that replacement was deemed unnecessary.

Hydrogen Unit

On March 26, two valves were replaced on the Syngas Compressor. On April 7, all the valves were replaced. On April 8, new rings were installed. The compressor cooling water jackets were backflushed on April 18 to reduce the discharge temperature and increase the valve life. This proved ineffective, and on April 25 the Syngas Compressor was shut down for maintenance due to loss of capacity. Both compression rings were in very poor condition: one broken in half, the other very thin. The valves were covered with an emulsion-like grease, which was

analyzed as containing 12.71% ash, 52.2% heptane insolubles and 60% pyridine insolubles (some iron in ash). New rings, valves and a crosshead were installed and the compressor was returned to service. Subsequent discussions were held with the compressor manufacturer and the ring manufacturer to determine what action could be taken to improve compressor performance. Their recommendation was that in wet gas service, such as this, the cylinder should be run dry to prevent a "stripping" of lubricant which thins the rings and forms emulsions on the valves. It was also felt that the existing carbon filled Teflon rings would perform well in this service, if used without lubrication. Based on this recommendation, dry operation was begun on May 3 and has been successful to date.

DEA Unit

During the early May shutdown, the unit was drained and steamed. A hot detergent solution was circulated through the unit to remove a small accumulation of process oil.

The DEA reboiler was pressure tested at 100 psig inert gas pressure to check for a suspected leak. All tubes were satisfactory, including the sixteen plugged in March, 1979.

Several lines in the DEA unit were ultrasonically inspected. As shown in Figure A-10, the piping did not appear to have any appreciable metal loss.

J. Dowtherm Unit (Area 9.8)

Because of defective relays in the safety interlock system of the control panel, the Dowtherm heater repeatedly shut down during April. System repairs were made during the shutdown starting April 28 and included:

1. Repacking of leaking Dowtherm valves, including the minimum flow control valve.
2. Checking and testing of the Dowtherm heater burner control system.
3. Inspection and repair of a crack in the refractory of the Dowtherm heater burner throat.

From June 15 to June 21, the Dowtherm heater shut down unexpectedly on numerous occasions. The immediate problem was corrected by raising the fuel gas supply pressure to maintain the proper air/fuel gas ratio during operation at high heat loads.

Attempts to bring the Dowtherm reclaimers on line were unsuccessful. The problem is under study by Engineering.

K. Area 10 - Lummus Unit

In March, the overflow antisolvent fractionator and the underflow antisolvent stripper were operated to prove their ability to remove antisolvent from a 40% antisolvent/60% process solvent blend at design flow rates. This proved successful as only 1-2% antisolvent remained in the column bottoms.

Coal slurry was introduced to the unit on April 12. After 45 minutes, slurry feed was stopped to repair a leaking flange at a flow element. Approximately 6 hours later slurry feed was resumed and was maintained until April 20, at which time it was decided to bring the unit down and inspect the inside of the deashing settler vessel. This decision was based primarily on a highly unfavorable ash balance around the unit during the period of April 16 to April 19.

When the settler was opened, all three settling compartments and the bottom overflow compartment were at least 50% full of solids. These deposits presented an extremely difficult cleaning problem because of the complex internal geometry of the settler and the tendency of the solids to spontaneously burn on exposure to air.

Cleaning of the settler vessel was completed on May 10. A combination of hydroblasting, steaming and water flushing was used, and approximately 150 drums of solids were removed from the vessel. The consistency of this material ranged from 26 to 56% pyridine insolubles and 32 to 40% ash.

The remainder of May was used to clean and replace those parts that had been removed during the cleaning operation and to make several modifications. These activities are listed below:

1. Flush solvent connections were made upstream of the individual settler underflow meters to provide a means of flushing through the meters to downstream piping.
2. A liquid level probe was installed in an existing nozzle on the top of the settler vessel to allow the automatic venting of the gas bubble which forms here. The settler was originally designed for liquid-full operation.
3. Piping of the individual settler underflow sample points was revised to allow these samples to be taken safely.
4. A recirculation line with a rotometer and a hand flow control valve was installed to allow the circulation of antisolvent-free overflow from the fractionator to the bottom overflow compartment of the settler vessel. The original design maintained vessel pressure and temperature in this compartment with the combined settler overflow stream. It was felt that the antisolvent content of this

stream contributed to the solids deposition in this compartment and that this problem could be alleviated with the use of an antisolvent-free stream.

5. A pressure reducing control valve was installed on the Dowtherm supply header. Previously, thermal relief valves on the Dowtherm side of the heat exchangers failed to hold against plant Dowtherm supply pressure. They could not be set at a higher relief pressure without exceeding the design pressure ratings of the exchangers.
6. Inert gas purges were added to the vapor space of the antisolvent storage tanks to prevent oxygen from being drawn into the tank. Without the inert gas purges there was a good possibility that the explosive limits would be exceeded when the tanks were heated.
7. A system was installed to recirculate 1500-2000 lb/hr of fractionator bottoms through the settler overflow accumulator to purge any solids.
8. All Taylor wedge meters were removed, cleaned and reinstalled.
9. The settler feed distributors were removed and cleaned.
10. The settler pressure relief valves were removed and repaired.
11. All leaking Dowtherm valves were repaired.

A large amount of potentially explosive vapors were drawn into the Lummus Unit control room during the April test run. As this station was specified to be a purged room in a Division II area, the air intake had to be relocated.

Upon further investigation, it was discovered that the control room itself did not meet design requirements as specified by the National Fire Code (#496). The code required that the room be pressurized to 0.1" of water with the doors closed and that the velocity of the air be at least 60 feet per minute with all possible openings open. The original blower and heating system was only designed for about 10 fpm. The following actions were taken in order to bring the control room up to specification:

1. The air intake was relocated across the street near the main control room.
2. The fan size was increased from 600 cfm to 2,500 cfm.
3. One of the three doors was blocked closed.

Startup conditions were revised to avoid the solids deposition problems experienced during April. The general operating

philosophy for the second attempt was to feed the vessel at design slurry rates (instead of the substantially reduced rates of the initial startup) and below design antisolvent-to-feed and overflow-to-underflow ratios. While the settler ash balance was continually monitored the antisolvent-to-feed and overflow-to-underflow ratios were gradually increased by increments in order to achieve production of specification product at an acceptable SRC recovery rate.

On June 10, the unit was charged with process solvent and antisolvent and solvent circulation was established. Frequent plugging of the individual underflow flow meters prompted their removal. They were replaced with spool pieces prior to slurry injection. Control of settler underflows was maintained by manually setting each of the individual underflow control valves approximately 25% open and routinely checking the individual flows by observing the response of the total underflow flowmeter as each underflow valve was closed off and reopened. Slurry was fed to the unit on June 15 at design rates. Shortly thereafter, the individual underflow valves started to plug. It was decided to remove the antisolvent from the unit and continue to process slurry until plug-free operation could be established. Six hours later, antisolvent was reintroduced. During the next 36 hours, the antisolvent to feed ratio was gradually increased and the antisolvent-free overflow recirculation to the settler was reduced and finally stopped. On June 18, the bottom settler compartment of the settler was put on total recirculation with the antisolvent-free overflow stream. On June 19, feed was reintroduced to this compartment after ash balances indicated suitable performance. Small adjustments in antisolvent to feed and overflow to underflow ratios were then made and individual settler overflow and underflow samples were taken to evaluate individual compartment performance. Continuous production of specification product had not yet been achieved at the end of the quarter.

The bottom overflow compartment of the settler vessel was purged with a flow of antisolvent-free overflow throughout the run.

The newly installed level control probe on the top of the settler vessel was very unreliable. It frequently required recalibration, and the ceramic insulator on the high temperature Kasitrol probe broke several times. The venting of the gas bubble to the flare was often done manually.

III. PROCESS ANALYTICAL DATA

The coal used in the second quarter period was Kentucky No. 9 & 14 coal from the P&M Colonial Mine. Powhatan No. 5 coal was used for a period of two and half days, but those few data are not included in this report. The plant was operated in the SRC-I mode during the quarter.

The average analyses of the raw coal used during the reporting period are shown in Table 1.

TABLE 1

Average Raw Coal Analyses, Wt %

	<u>April</u>	<u>May</u>	<u>June</u>
Ash	8.13	10.92	9.55
Moisture	7.16	5.08	6.14

The average analyses of the dried, pulverized coal are reported in Tables 2, 3 and 4.

TABLE 2

Average Dried, Pulverized Coal Analyses, Wt %

	<u>April</u>	<u>May</u>	<u>June</u>
Carbon	71.62	70.71	70.76
Hydrogen	5.16	5.12	5.18
Nitrogen	1.57	1.44	1.53
Sulfur	3.23	4.17	3.57
Oxygen (by difference)	8.85	7.23	8.60
Ash	8.89	10.78	9.97
Moisture	0.68	0.55	0.39

TABLE 3

Average Analyses of Forms of Sulfur, Wt %

	<u>April</u>	<u>May</u>	<u>June</u>
Pyritic Sulfur	1.44	2.58	2.03
Sulfate Sulfur	0.40	0.23	0.27
Organic Sulfur	1.39	1.36	1.27
Total Sulfur	3.23	4.17	3.57

TABLE 4

Average Sieve Analyses of Dried, Pulverized Coal, Wt %

	<u>April</u>	<u>May</u>	<u>June</u>
- 40 mesh	99.37	98.80	98.83
-100 mesh	97.87	97.32	97.31
-140 mesh	91.52	90.88	90.80
-200 mesh	79.90	78.69	79.70

The average analyses of the stripper bottoms in this quarter are shown in Table 5.

TABLE 5

Average Analyses of Stripper Bottoms, Wt %

	<u>April</u>	<u>May</u>	<u>June*</u>
Water	0.12	1.15	-
Light Oil	0.01	0.03	-
Wash Solvent	2.42	0.00	-
Process Solvent	63.06	63.29	-
Vacuum Bottom (PI Included)	34.38	35.53	-
Pyridine Insolubles(as received)	6.90	7.00	7.34
Ash (as received)	3.98	4.82	5.41

* Routine distillation of stripper bottoms in the laboratory was discontinued in the month of June.

Laboratory determined coal conversion on a moisture-ash-free (MAF) coal basis are reported by month in Table 6.

TABLE 6

Average MAF Coal Conversion, Wt %

April	94.7
May	94.1
June	95.9

The average analyses of oil and water phases from the recycle process water tank (RPWT) are reported in Tables 7 and 8.

TABLE 7

Average Analyses of RPWT Oil Phase, Wt %

	<u>April</u>	<u>May</u>	<u>June</u>
Light Oil	29	28	Not
Analyzed			
Wash Solvent	12	12	
Process Solvent	59	59	
Specific Gravity @ 60/60°F	0.967	0.950	

TABLE 8

Average Analyses of RPWT Water Phase

	<u>April</u>	<u>May</u>	<u>June</u>
Phenol, Wt. %	0.45	0.57	0.79
Nitrogen, Wt. %	-	-	0.71
Sulfur, Wt. %	-	-	0.81
pH	-	-	8.7

Typical fractional analyses of liquid products based on ASTM D-86 distillations are reported in Table 9.

TABLE 9

Typical Fractional Analyses of Pilot Plant Liquid Products, Vol. %

<u>Laboratory Distillation Fraction</u>	<u>Light Oil</u>	<u>Wash Solvent</u>	<u>Process Solvent</u>
<u>April</u>			
IBP to 380°F	90	14	0
380 to 480°F	10	86	1
480 to 850°F	0	0	99
<u>May</u>			
IBP to 380°F	88	7	0
380 to 480°F	12	87	0
480 to 850°F	0	6	100
<u>June</u>			
IBP to 380°F	85	12	0
380 to 480°F	15	82	1
480 to 850°F	0	6	99

The average analyses of the pilot plant vacuum bottoms produced during this quarter are reported in Table 10.

TABLE 10

Average Analyses of Pilot Plant Vacuum Bottoms, Wt %

	<u>April</u>	<u>May</u>	<u>June</u>
Carbon	78.96	71.58	76.47
Hydrogen	4.99	4.86	5.18
Nitrogen	1.86	1.61	1.82
Sulfur*	1.80	3.03	2.31
Oxygen (by difference)	2.58	1.69	2.59
Ash*	9.81	17.23	11.63
Fusion Point °F	350	322	320

*Even though the plant was in the SRC-I mode of operation, specification mineral separation in the feed to the vacuum flash system had not been achieved. This is the reason for the high ash and high sulfur values obtained on the plant vacuum bottoms.

The analyses from the waste treatment units reflect acceptable control throughout the quarter. Typical analyses of waste water unit streams are shown in Table 11.

TABLE 11

Process Waste Treatment Analyses

	<u>Bio-Unit Feed</u>	<u>Bio-Unit Effluent</u>	<u>Plant Effluent (Composite)</u>
<u>April</u>			
pH	6.5	6.7	7.1
Phenol, ppm	53	0.44	0.09
Chemical Oxygen Demand, ppm	758	179	24
Biological Oxygen Demand, ppm	290	54.9	.09*
Total Suspended Solids, ppm	91	61	2.8
<u>May</u>			
pH	6.5	6.4	6.9
Phenol, ppm	17	0.42	0.06
Chemical Oxygen Demand, ppm	502	130	18.
Biological Oxygen Demand, ppm	161	11.4	1.9
Total Suspended Solids, ppm	54	49	4.7
<u>June</u>			
pH	6.3	6.4	6.9
Phenol, ppm	47	0.94	0.19
Chemical Oxygen Demand, ppm	617	164	27
Biological Oxygen Demand, ppm	159	11	4.7
Total Suspended Solids, ppm	69	44	3.9

*Analysis represents only one sample.

Typical analyses of slurry feed, antisolvent free underflow and antisolvent free overflow for the Lummus Deashing Unit are shown in Table 12.

TABLE 12

Typical Analyses of Lummus Deashing Unit Streams, Wt %

	<u>Feed</u>	<u>Underflow</u>	<u>Overflow</u>
<u>April</u>			
Toluene Insolubles	14.4	17.0	8.0
Pyridine Insolubles	5.3	5.8	0.5
Ash	3.4	3.1	0.4
I.B.P. to 500°F	3.4	2.4	4.5
500 to 650°F	31.4	28.7	36.8
650 to 850°F	31.1	30.7	31.4
Above 850°F	34.1	38.2	27.3
<u>May</u>	Unit Not Operated		
<u>June</u>			
Toluene Insolubles	17.1	-	-
Pyridine Insolubles	7.7	16.0	1.28
Ash	5.1	10.4	1.27
I.B.P. to 500°F	2.1	2.1	2.5
500 to 650°F	33.1	24.0	38.1
650 to 850°F	25.3	22.1	24.9
Above 850°F	39.4	51.8	34.5

IV. Pilot Plant Special Studies

A. Material Balance Run 79-1

During the period from 1400 hours May 15 through 1600 hours May 21, a material balance run (designated 79-1) was conducted in the SRC-I mode. The purpose of this run was to measure product yields and confirm operability at target conditions based on one of the current SRC I Demonstration Plant design cases. These conditions are tabulated below:

<u>INDEPENDENT VARIABLE</u>	<u>TARGET LEVEL</u>
Coal Space Rate (based on dissolver volume)	50 lb/hr/ft ³
Solvent/coal ratio	1.6 to 1 by wt.
Solvent boiling range	450°F plus
Preheater Inlet Gas	20 MSCF/ton coal 93% vol. H ₂
Dissolver Pressure	1700 psig
Dissolver Temperature	825°F outlet
Coal type	Kentucky No. 9

A list of operating conditions actually achieved during Material Balance Run 79-1 is presented in the attached Table A-2. A comparison of the measured levels of the parameters shown in Table A-2 with the design conditions shown above indicates several departures from target conditions, including a slightly different coal feed type, a somewhat higher boiling solvent cut, and a higher gas feedrate. These variations were the result of the following limitations: lack of the design coal feedstock at plantsite, insufficient time to refractionate plant solvents and limitations on minimum gas flowrates through the existing slurry preheater.

Plant operation during the run proved to be very stable, with no coal outages recorded during the entire 146 hour period. The only operating problem which occurred during the run was a loss of Recycle Hydrogen Compressor B which caused fluctuating gas flowrates during a 12 hour period.

Several problems interfered with the accuracy of MBR 79-1 material balance calculations, however. During the first part of the run period, the chromatographic analysis of the recycle condensate separator gas indicated an erroneously high hydrogen sulfide content. At the same time the chromatograph analyzing vapor from No. 1 & 2 oil/water separators was indicating an erroneously high level of C₅+. Both of these problems were

corrected by recalibration of the appropriate on-line gas chromatographs and gas yields for the run were based only on data taken following recalibration. (Gas yield data was obtained during the period from 1400 hours 5/17 through 2200 hours 5/20.)

Also, during the first part of the run, a seal flush line to the Filter Feed Surge Vessel recirculation pump was inadvertently activated, resulting in a diversion of nearly 2000 pounds of process solvent to this otherwise inactive vessel. This inventory was included in the calculation of material balance liquid yields.

Raw (directly measured) product yields from run 79-1 are shown in Table A-3. These raw results were derived from the following sources:

1. Gas yields and hydrogen consumption were obtained by mass component balances obtained around an envelope encompassing the reaction and product separation area. Instrumentation in use included four concentric orifice flowmeters and two on-line gas chromatographs.
2. Hydrocarbon liquid yields were calculated from overall plant inventory changes during the material balance period. All major process and tank farm vessels were gauged, sampled and analyzed (by Cushman and special D-86 distillations) to obtain the requisite data.
3. Vacuum bottoms yield was obtained directly from product weights measured on truck scales as produced.
4. An aggregate of water yield, unmeasured yields and losses was obtained by difference.

The raw product yields shown on Table A-3 were normalized using a standard technique designed to achieve primary elemental balances (C, H, N, O, and ash) as well as an overall material balance. This procedure consists of separating each measured input and output into primary elemental constituents based on a composite analysis obtained during the run (see Table A-4 for a listing of averaged component analyses). Elemental closure is then obtained in the following steps:

- a) Ash is forced to balance by adding the requisite amount of vacuum bottoms to the outlet stream. (This procedure assumes a certain amount of vacuum bottoms is lost in handling.)
- b) A carbon balance is forced by adding the requisite amount to the naphtha yield. (This assumes a loss of low boiling organic liquids throughout the plant.)

- c) Sulfur is forced to balance by adjusting the yield of hydrogen sulfide. (This assumes a loss of soluble H_2S to the reaction quench water stream.)
- d) Hydrogen, oxygen and total mass are simultaneously forced by assuming an appropriate yield of water.

Results of the forced elemental balance routine described above are shown on Table A-5 and the final normalized yield structure is presented on Table A-6. Significant findings include relatively low hydrocarbon gas and naphtha yields and a positive process solvent yield, presumably the result of low reactor temperature and long residence time. Further study of reaction yields at SRC I demonstration plant design conditions may be undertaken when the proper coal feedstock is received at the plantsite.

B. Vacuum Flash System Tests

Two sets of test runs designed to gather data around the plant vacuum flash system were completed. The primary objectives of the first series of runs were to characterize the vapor-liquid equilibrium at varying flash zone temperatures and to obtain representative samples of feed and products. This series has been designated the Vacuum Flash Drum mass balance runs (MBR). The second set of runs, designed to obtain data on carryover of solids at various vapor velocities, has been designated the Vacuum Flash Drum carryover tests. Each of these test runs is described in more detail in the sections which follow.

Prior to the test runs several modifications were made to the vacuum flash system to enhance the stability of the unit and to improve the quality of data obtained. These modifications included resizing of the feed control valve, calibration of the drum vacuum transmitter, and installation of a feed flowmeter. In addition, process flows were changed to allow the use of an intermediate feed surge vessel between the slurry recycle stripper and the vacuum flash preheater. This change permitted the accumulation of inventory ahead of the vacuum flash system, thus serving to dampen out fluctuations in flowrate to the vacuum system which had previously resulted in non-steady state drum operation.

1. Vacuum Flash Drum MBR

Four separate test run periods comprised the Vacuum Flash Drum MBR. In all tests, vacuum flash feedrate and drum vacuum were held constant, whereas feed temperature was different in each test. The four flash zone temperatures tested covered the range from 599°F to 634°F.

During each test, the Vacuum Flash Condensate Drum was blocked in and level change was used to calculate equilibrium vapor production rate. Feed and condensate samples were taken every hour and vacuum bottoms produced during

the tests were solidified, collected in drums, and weighed. A grab sample of the vacuum bottoms was taken every ten minutes and these samples were composited to make a representative hourly sample which was analyzed for ash, pyridine insolubles, and fusion point. Both feed and vacuum flash condensate samples were also composited and vacuum distilled every six hours.

The attached Table A-7 shows the operating conditions maintained during each of the four run periods. In each test, drum feedrate, vacuum, and flash zone temperatures were held constant for a six hour period. Feedstock was prepared in the SRC II mode using Powhatan No. 5 coal at a 30% feed coal concentration, thus approximating current demonstration plant design reaction conditions.

Calculated average compositions of the feed and products for each of the test runs are shown in Table A-8. Distillation data shown in Table A-8 were obtained using the "Cushman" vacuum distillation technique. Ash analyses were obtained by standard A.S.T.M. coal ashing procedures and pyridine insolubles levels were measured by standard P&M techniques.

The data shown on Table A-8 were used to calculate molar compositional distributions using estimated molecular weight values for each 50°F cut fraction. The resulting feed and condensate molar distributions, derived from a proprietary correlation of molecular weight and boiling point, are graphically presented in Figures A-11 through A-18.

The data in Table A-8 and Figure A-12 indicate an abnormally high amount of middle distillate in the condensate samples from Run 1. No satisfactory explanation for this anomaly has yet been advanced. Omitting the data from Run 1, the overall vapor-liquid equilibrium achieved in the Vacuum Flash Drum appears to vary directly with flash zone temperature, as shown in Figure A-19.

Material balances for hypothetical boiling range components were also calculated from the data presented above. These balances are shown on Table A-9. In all of the tests, the ash in the feed and bottoms balanced very well. However, most of the runs showed more IOM in the product than in the feed, thus indicating a slight production of IOM, perhaps due to repolymerization reactions occurring in the vacuum flashing operation. In addition, there generally was more light material (550°F) in the condensate than in the feed, thus indicating a net production of some lighter fractions perhaps as a result of cracking reactions.

A heat balance around the flash drum and heater was not made, since both the downleg and preheater use Dowtherm,

which, at present, is not metered. In addition, neither the drum overhead temperature nor the inlet temperature are measured at present and large convective heat losses occur in the system.

An aliquot of the composite condensate sample from Run 4 was analyzed at Gulf Research & Development Company laboratories by both a "Cushman" distillation and a Podbielniak Hyper-Cal simulation of a true boiling point distillation. Results are shown on Table A-10 and Figure A-20, wherein the results produced by the two procedures are compared. The indicated comparison shows reasonably close agreement between the two procedures, further confirming the validity of the "Cushman" technique used to develop the material balance data presented above.

2. Vacuum Flash Drum Carryover Tests

Six test runs were conducted on the Vacuum Flash Drum B to measure the effect of vapor velocity on the amount of solids entrained in the overhead vapor. During each test, the drum feedrate was held constant for a period of four hours, and two samples of vacuum flash condensate were taken each hour. The samples were stirred and a 50 ml aliquot was carefully withdrawn and ashed.

A listing of flash drum test conditions and results is presented in Table A-11. The vapor velocity and vapor density values shown assumed a condensate molecular weight of 200 (based on previous measurements of the molecular weight of similar boiling range coal liquids).

During the tests, the drum feedrate was higher than normal since slop material was being processed along with fresh feed. Despite this, the vapor velocity never exceeded 5.5 feet per second. In fact, the highest drum feedrate resulted in the lowest drum vapor velocity. This was apparently the result of a reduction in drum vacuum caused by the pressure drop in the overhead vapor line.

Throughout the tests, the carryover measured as ash in the vacuum flash condensate remained only slightly above the trace level. Figure A-21 shows a plot of the measured condensate ash values as a function of drum feedrate.

C. Vacuum Flash Test Stand

The pilot plant vacuum flash units operate at less severe conditions and lower vapor velocities than are presently envisioned for use in a demonstration plant. Because of the importance of the vacuum flash step in the SRC-II process and the lack of reliable data at the more extreme conditions, a vacuum flash test stand has been designed with construction expected to commence in the following quarter.

The primary objective of the vacuum flash test stand is to explore the effects of equilibrium flash conditions on overall and pseudocomponent vapor-liquid equilibrium and product physical properties. These properties include elemental composition, fusion point, differential solubility, molecular weight, density, boiling range and viscosity of both the overhead and bottom streams.

A secondary objective is the development of data concerning the influence of drum geometry and vapor velocity on inorganic solids carryover and heteroatom (primarily sulfur and nitrogen) contamination of the liquid product.

A schematic drawing of the vacuum flash test stand is shown in Figure A-22, and an elevation drawing of the unit is shown in Figure A-23. Unit feedstock consists of SRC-II vacuum flash chargestock which is preheated to 250°F and coarsely strained. This is pumped to the unit with a Milton-Roy positive displacement metering pump designed to maintain steady flow to the drum and heater. System design feedrate is approximately one pound per minute.

The flash drum heater closely simulates actual pilot plant units. The heater is designed to raise the feed material from 200°F to a maximum of 800°F. The heater will be fabricated in three equal length sections to facilitate easy cleaning or replacement as necessary.

A capillary pressure letdown will be installed just ahead of the vacuum flash drum. Several capillary sizes will be available to maintain various levels of back-pressure.

Several vacuum flash drums are planned with varying diameters and length/diameter ratios. Initially, two drums will be built: one 4" diameter and one 6" diameter. Each will be designed to operate at a vacuum of 2-30 mm Hg and at flash zone temperatures from 640°F to 720°F. Vapor velocity will range from 10 to 30 ft/second. Vacuum bottoms will be withdrawn from the drum through a lock-hopper arrangement. The drum itself will be fitted with a series of pipe reducers which will gradually taper the bottom diameter to the downleg size to minimize the chance for a buildup of solids. The downleg will connect directly to a Marpac Grafoil double-packed ball valve with discharge to a 5 minute holdup intermediate vessel. Vacuum bottoms will be withdrawn batchwise from the intermediate vessel through another Marpac ball valve to a receiver vessel. The temperature of the downleg system will be kept below that of the vacuum flash drum to prevent product degradation due to prolonged exposure to high temperature. A vacuum pump will be connected to the receiver vessel to assist the gravity flow of vacuum bottoms through the downleg system.

Vapor from the vacuum flash drum will be condensed in a vertical downflow water cooler. The transfer line to the cooler will

be designed to prevent cooling of the top of the vacuum flash drum and causing unwanted reflux. Water to the condenser will be tempered if necessary to ensure positive flow of viscous condensate. Vacuum flash condensate will collect in a receiver vessel and system vacuum will be maintained on the receiver by parallel positive displacement vacuum pumps.

The vacuum flash test stand will also be equipped with a flare and slop collection system. This will be used for disposal of initial startup solvent and for heater outlet safety release. The unit will also be provided with appropriate instrumentation and a remote control panel.

Present plans call for construction of major unit equipment to be completed early in July. All materials have been ordered and, pending delivery, final assembly will begin in July or early August. Unit startup is currently scheduled for late August.

D. Analysis of SRC II Plant Startup

Data from the 1/24/79 and 2/24/79 pilot plant startups in the SRC-II operating mode were analyzed to develop an understanding of the pilot plant's approach to steady state conditions. Both startups occurred after the reaction area had been thoroughly flushed with heavy distillate, and therefore the concentration of solids in the recycle slurry stream was initially very low (approximately 1 wt%).

Evaluation of the data obtained for each startup period leads to the following estimates of the lineout period:

ESTIMATED LINEOUT TIME REQUIRED

<u>STREAM</u>	<u>FIRST STARTUP - 1/24/79</u>	<u>SECOND STARTUP - 2/24/79</u>
Slurry	20 Hours	12 Hours
Gas	3 Hours -	5 Hours

Slurry lineout times were estimated from observation of the concentration of solids in the recycle slurry. The gas lineout period was estimated from analysis of the methane production rate. The substantial differences in the required lineout times of slurry and gas streams was probably due to the high superficial gas velocity in the reaction area (gas velocity is estimated to be nearly an order of magnitude greater than liquid velocity at Fort Lewis dissolver conditions). The estimated mean residence times of gas and slurry in the Fort Lewis reaction system (including coal-slurry mixing, slurry preheating) are approximately seven minutes and two and one half hours, respectively. The short gas residence time results in frequent turnover of the gas volume in the system, and hence a fast approach to equilibrium.

The rapid gas lineout experienced during both startups suggests that hydrocarbon gas production is not as heavily dependent on the composition of the recycle slurry as are the yields of distillate or organic vacuum bottoms.

E. Analysis of Hydrogen Consumption and Methane Production Versus Temperature

Hydrogen consumption and methane production data for February, 1979 are shown in Figures A-24 and A-25, respectively. Both curves show a very rapid change in product yields at temperatures above 800°F with both methane production and hydrogen consumption increasing substantially. Of particular interest in these graphs is the wide scatter in yields observed between 850-860°F, the temperature range for which the most data are available. The standard deviation for both hydrogen and methane yields in this temperature range was approximately 20-25 lb/hr, which is equivalent to approximately 1% of the moisture-free coal feed.

The widely dispersed yields of hydrogen and methane for the February, 1979 period are typical of results obtained at the pilot plant. The combination of inaccuracies in flow measurement and chromatographic analyses, along with changing compositions of major gas streams, result in individual hourly yield calculations that vary even though operating conditions remain constant. The standard deviation calculated from the February data set may prove useful in analyzing future gas balance data which are developed over relatively short operating periods.

F. Vacuum Flash Feed During Plant Startup

During the May 24 to 26 plant startup an attempt was made to monitor the flow rate of recycle slurry from the reaction area to the vacuum flash feed system. Since this stream is not metered, no previous data were available concerning the magnitude of variation in this flow during startup.

Recycle slurry exiting the reaction area represents the entire normal feed stream for the vacuum flash system. During startup, the recycle slurry initially consists primarily of heavy distillate. As a startup progresses, the heavy distillate is gradually displaced by vacuum bottoms. The ability to vacuum flash the slurry depends to a large extent on its composition. Surge capacity is required to accumulate the initial product slurry, which has a low vacuum bottoms concentration, until the vacuum bottoms concentration is high enough to permit stable operation of the vacuum flash system.

Since the pilot plant does not have built-in capability to measure the flowrate of recycle slurry to the vacuum flash system, a special test procedure was devised to estimate the flowrate from level changes in process vessels. The results of this test are presented in the bottom half of Figure A-26. During the test, it was necessary to introduce approximately

300 lb/hr of seal flush oil into the test vessels, therefore the estimated flowrate shown in Figure A-26 is 300 lb/hr higher than the actual flowrate of recycle slurry from the reaction area.

Figure A-26 indicates that the flowrate of recycle slurry to the vacuum flash system was initially very low, and possibly negligible, during the first several hours after coal feed began. During this period, the concentration of solids (pyridine insolubles) in the slurry increased rapidly (Figure A-26 top). Thereafter, the slurry flowrate increased to approach a lined-out value.

This study suggests that it may be possible to limit the amount of slurry feed to the vacuum flash system during the early stages of an SRC-II startup, when the concentration of vacuum bottoms in the slurry is low. By minimizing the slurry flowrate during the startup period it also should be possible to minimize the required surge capacity.

Further tests during pilot plant startups would be useful in identifying the conditions which result in the lowest slurry flowrates to the vacuum flash system before lineout is achieved.

G. Rheological Experimental Program

A rheological experimental program has been designed to gather viscosity data on SRC-II feed slurry, recycle slurry and vacuum bottoms over a wide range of conditions. The ultimate goal of the program is the development of a mathematical model of viscosity behavior as a function of the major intensive and extensive variables within the limits of commercial significance.

Initially, all viscosity data will be obtained in a controlled laboratory apparatus, although later work may also involve field measurement and confirmation of the laboratory data. The primary laboratory tool is a Haake Rotovisco RV-2 viscometer, which was selected following an extensive equipment search for an instrument capable of precision measurements at the elevated temperatures and pressures contemplated for the study.

The selected equipment consists of the following components: A model RV-2 operating console, drive motor and support stand; a DMK 50/500 dual range torque head; a D-40/300 viscosity sensor autoclave with constant temperature circulator; a programmer; an X-Y-T recorder; and associated accessory equipment. This system is designed to allow viscosity measurement at pressures to 580 psi and temperatures to 572°F.

A sample conditioning system (see Figure A-27) has been designed to prepare, contain and introduce samples to the Rotovisco. Samples will be maintained in an inert atmosphere and pressured from a feed preparation vessel to the viscosity sensing head. The feed vessel is a stirred, jacketed autoclave. Coal may be

added to the vessel by gravity feed from a coal hopper to ensure a freshly prepared sample. An overflow receiver vessel is also provided to maintain a constant level in the sensor autoclave vessel. A purge valve on the receiver vessel allows letdown of pressure as the sample is heated and at the end of each experiment.

The sample conditioning system has been designed for a 200 psig working pressure. Preliminary experiments have shown that this is sufficient pressure to prevent significant vaporization of the materials to be tested.

When the Rotovisco was first set up in the manufacturer's representative's presence on June 7, several problems were discovered. The external heat exchanger on the heater/circulator arrived improperly mounted, so that heat transfer fluid could not be pumped to the sample vessel. A set of high temperature thermometers was also missing and the cables designed to connect the motor, measuring head and controller were too short.

While the problems described above were being resolved, exploratory experimental work was begun with the high pressure measuring head. Initial measurements indicated a problem with the magnetic drive unit which introduced severe errors in torque readout at low shear rates (less than 90 per second). These errors are illustrated by curve A of Figure A-28, which displays torque output versus time with the measurement system completely assembled, but no sample introduced. (This curve should be a straight base line.)

In an effort to diagnose the cause of the fluctuating torque readings, various portions of the pressure probe were disassembled. The largest change in output occurred when the bolts securing the autoclave head were removed. (Curve B shows the torque response with the bolts removed.)

The bolts proved to be of carbon steel construction and thus subject to magnetic attraction. The arrangement of the nine bolts in two concentric circles of three and six bolts made three nodes of maximum attraction if the four magnets were unbalanced (twelve nodes if balanced) which appeared as three peaks per revolution (1.5 cm per revolution) in Curve A. Removal of only the bolts drastically decreased the amplitude of the oscillation as can be seen by comparing Curves A and B. Removal of the casing for the magnetic drive which was made from 316 SS (stainless steel) further reduced oscillation. Curve C was made with the drive spindle hanging freely. The spindle housing with cooling fins is visibly attracted to a magnet and may have caused the oscillation in Curve C. The results of these experiments indicate the necessity of replacing the autoclave bolts with material of low magnetic permeability. The drive spindle housing, drive casing, and drive spindle bearing also might require replacement as each has an effect

on torque output. The difference in amplitude among the peaks of oscillation during one revolution indicates imbalance in the strengths of the four magnets. The residual oscillation of Curve C may be due to imbalanced magnets attracting to the spindle housing which, though magnetically permeable, is evenly distributed about the spindle. The differences in strength among the magnets can be felt by hand as different attractions to a piece of steel.

The analysis outlined above has been reported to the manufacturer and lease payments have been suspended until all problems are resolved. Interim rheology work is continuing through the use of an ambient pressure probe supplied by Haake.

The primary parameters targeted for study when the equipment problems are solved are listed in Table A-12. Viscosity will be measured over a wide range of shear rates. Previous studies indicate that process solvents are Newtonian, but that slurries are non-Newtonian, pseudoplastic fluids for which viscosity varies inversely with shear rate.

Temperature will be varied from 250°F to 550°F in 50° increments. Previous studies indicated that recycle slurry viscosity always decreased with temperature. However, feed slurry viscosity varied inversely with temperature up to 350°F, above which coal swelling occurred and reactions took place which cause viscosity to increase with temperature.

The composition of recycle slurry will be varied by distilling or adding solvent in order to achieve the desired vacuum bottoms to solvent ratio. The composition of feed slurry will be varied by blending varying proportions of recycle slurry, solvent, and coal. Coal concentration will be varied from 25 to 40 weight percent in 5 weight percent increments. Two hundred mesh (74 micron) coal will be used initially and varied as other samples can be supplied and characterized.

The composition of vacuum bottoms will be varied indirectly by making measurements on a range of available samples obtained directly from the process. These samples will be characterized by standard plant laboratory analyses.

The time-temperature history of the sample will be found by extended constant temperature measurements at selected shear rates. Samples of feed slurry are expected to show increasing viscosity with time at temperatures above 350°F as the coal reacts and swells.

Recycle slurry and feed slurry will be studied first since data on these materials are needed for demonstration plant design of recycle slurry coolers, and feed slurry handling systems. Raw data, consisting of flow curves (shear stress versus shear rate) will initially be generated by programmed runs with the viscometer. When sufficient raw data are assembled,

viscosity will be plotted against two independent variables using families of contour lines for one independent variable. The final step will be development of a generalized correlation including each significant independent variable.

H. Stability of Vacuum Bottoms at Elevated Temperatures

Several experiments were conducted in the Fort Lewis plant laboratory to give an estimate of how long a sample of vacuum bottoms could be held at high temperatures under an inert atmosphere before its viscosity began to increase significantly.

Sample bombs, consisting of 1/2" diameter stainless steel pipe capped on both ends by Swagelok fittings were half filled with vacuum bottoms derived from Powhatan coal. The top nut was left unfastened and each bomb was placed in a large plastic bag along with the top nut and a wrench. The bag was evacuated by applying vacuum, then filled with argon. This procedure was repeated three times to assure an inert atmosphere. After the top nut was fastened to the bomb, the bomb was removed from the bag and inserted in the heating apparatus shown in Figure A-29.

Timing of sample heating began after temperature equilibration, which usually required 10 to 15 minutes. The bombs were removed at predetermined time intervals and allowed to cool to ambient temperature. Samples of the vacuum bottoms were then placed in a Brookfield Thermosel for viscosity measurements. Viscosities were measured with a Model HA Brookfield viscometer using a number 21 spindle. All viscosities of vacuum bottoms were run at 550°F. Shear rates used on the various samples are given in Figures A-30, A-31, and A-32.

As shown in these figures, the viscosities increased only slightly with time if the sample was held at moderate temperatures (675°F). However, after a threshold time, viscosities began to increase exponentially. The threshold time decreased significantly as the holding temperature of the sample exceeded 675°F.

I. Lummus Deashing Unit

The Lummus Antisolvent Deashing Unit first began operation April 12, 1979 at a slurry feed rate of 4900 pounds per hour. The run, which was designed as a shakedown at half of design feed rate, was terminated April 20, 1979 because of a continued increase in solids (ash) accumulation in the vessel.

Operation proceeded at first with only minor plugging on one of the underflow lines. This problem was corrected by replacing the underflow slurry flowmeter in this line with a spool piece. Eventually, however, all of the underflow meters had to be replaced with spool pieces as they were restricting flow from the settler.

Initial data showed specification ash content in overflow streams, but ash levels began to increase after three days of operation. Occasional flushing of the individual settler underflow lines was required to eliminate intermittent plugging. In addition, an accumulation of gas was experienced in the settler vessel, which was intended to run liquid-full, and this necessitated periodic manual venting to atmosphere. After the fourth day of operation the ash balance on both the Lummus system and the overall pilot plant began to indicate substantial buildup of ash in the system.

An unsuccessful attempt was made to unclog the settler by using underflow recirculation and by removing the Taylor wedge flowmeters located in the individual settler underflow lines. On April 20 slurry feed to the Lummus Unit was stopped and the process of flushing, cooling, and securing the settler vessel for inspection was begun.

The settler vessel was cooled by recirculating solvent, drained, inerted and finally opened April 27. Inspection of the top section through the manway revealed that it was essentially full of solids. Samples of the accumulated material indicated that some portions were hard and coke-like with a significantly lower fraction of ash than typical dissolver effluent solids while other portions were softer tar-like material with an ash-to-PI ratio closer to that of dissolver solids.

It appears that this major upset of the Lummus system was the result of the selection of incorrect startup conditions for the settler. The feed was introduced at 50% of design rate while the underflow rate was near design. This meant that overflow residence time was increased substantially above design which, coupled with the use of design antisolvent concentration, led to an overprecipitated condition and subsequent accumulation of ash in the settler. Based on this hypothesis, a new startup sequence was devised to avoid this situation in future startups.

The Lummus Antisolvent Deashing Unit was shut down during May for cleaning, maintenance and minor modification. Approximately 150 drums of high solids material were removed from the settler vessel.

The second Lummus Deashing Unit run extended from June 15 through the end of the reporting period. During this run the unit was fed at full design rate (8,300 pounds per hour) and the antisolvent concentration was increased gradually in order to achieve specification ash concentration in the overflow.

The unit was able to produce consistently low ash overflow for only short periods during the run. The lowest spot sample concentration of ash achieved in the cumulative antisolvent free overflow was 0.05 percent, and ash concentrations less than 0.1 percent were maintained for only short periods (the

longest period was two days from June 27 to June 29). However, analysis of individual overflow samples often showed significantly less ash, indicating contamination downstream of the settler. Assuming a typical SRC concentration in the overflow of 33 percent, overflow containing an ash concentration of 0.1 percent would yield, on the product belt, SRC product containing 0.3 percent ash.

Although antisolvent losses from the unit could not be precisely calculated during the run, there was some indication of depletion. Two main sources of antisolvent loss are retention in the stripper and fractionator bottoms streams and vapor losses from venting at the top of the settler.

J. Data Acquisition System

Several new programs were written during the second quarter. These include:

1. A program which calculates a series of flow meter material balances around the Lummus Unit to verify the accuracy of unit flow meters and identify instrumentation problems.
2. A program which retrieves selected data from magnetic tape storage and performs specific calculations on the data prior to printing.
3. A Lummus Unit flowmeter balance report which produces flowmeter checks on data previously recorded on magnetic tape.
4. A routine designed to calculate and report internal pipe stress.
5. A subroutine which performs octal to decimal conversions as a security check on various other programs.

Maintenance programming activities during the period include:

1. Modification of operator assistance routines to minimize extraneous messages.
2. Modification of the file utility programs to use a byte format and to include switch console references.
3. Modification of the high pressure letdown valve monitoring routine to record valve position data as well as hours of service, and to increase program efficiency and decrease program execution time.
4. Four programs (Current Loop Valve, Loop Index Cross Reference, Log, and Out of Range Report) were modified to use a new loop numbers file which increases the speed of program execution.

5. The data collection routine was modified to monitor and store data on the high pressure letdown valves.
6. The Gas Balance program was rewritten to account for the shutdown of No. 3 Oil-Water Separator.
7. One chromatograph servicing routine was recompiled as it was thought to be a source of computer shutdowns.
8. The program containing the inventory of laboratory data was modified to use either SRC I or SRC II cut points.
9. Modifications were made to the preheater plot program to permit more efficient execution.

The computer system was down for 56 hours during May because of damage to several printed circuit cards during the installation of a system ground by a Foxboro company service representative. In addition, three unexplained system shutdowns occurred during June. These were reported to Foxboro company service representatives. A list of computer system reliability for the second quarter is shown in Table A-13.

V. Special Projects

New Slurry Preheater

Early in the quarter, the preheater was set on its foundation, and the concrete slab and curbs were completed. Construction of structural steel members is about 90% completed. Shop fabrication of piping is approximately 45% completed. Field erection of piping has begun and the preheater has been painted.

Near the end of the quarter, the instrumentation and electrical drawings were completed by Hallanger Engineers and were issued to P&M for approval. Material take-offs for carbon steel piping were completed by Hallanger. Stainless steel piping take-offs were updated. The hydrogen purge panel piping was discussed in detail by P&M and submitted to Hallanger to incorporate in the drawings.

Hallanger completed the piping stress analysis. The results were submitted to Heat Research Corporation (HRC) to review the effect on the heater coil connections. HRC felt some of the forces and moments on the coil were slightly excessive. The Engineering Division of Gulf Science and Technology Company in Houston, Texas, was contacted to review the pipe stress analysis done by Hallanger for their assessment of the forces and moments exerted on the heater coil by the piping system. The Engineering Division staff felt that cold springing of the piping would be the easiest method of correcting excessive forces at this point in construction. Hallanger Engineers was to do the necessary engineering on this system.

A meeting was held at Hallanger offices with P&M to redefine limits of hazardous classified areas. This meeting was necessary to assure electrical equipment installed met the safety requirements of the respective areas.

Materials ordered during this quarter include a Hydrogen/Dowtherm heat exchanger, the differential pressure transmitters, lighting fixtures, a heat tracing panel board and most of the electrical and instrumentation items. Many critical items were quoted with deliveries too late to maintain the project schedule and shipment of many items previously ordered has been delayed. A vigorous expediting program was begun to minimize the impact on the project completion schedule.

At the end of this period, it was evident that the August 1 construction target would not be met. Feedback from the expediting program will be the basis for establishing a new completion date.

VI. SRC-II Operation on Process Development Unit P-99

The second quarter of 1979 began with a continuation of the operating period which had been started at the end of the first quarter. During this on-stream period, Runs 50, 51, and 52 were completed, all feeding Powhatan No. 5 coal from a Fort Lewis shipment. This on-stream period was completed on May 4 after 62 days of coal feed with only minor interruptions.

This operating period was relatively trouble-free. There were several pressure buildups in the dissolver due to the deposition of salts in the gas piping downstream of the hot high pressure separator, but when these occurred, they were controlled by flush water injection. Toward the end of the run, there also appeared to be some incidents of excessive pressure build-up across the preheater. The exact cause of the preheater pressure build-up was never determined, and after flushing the unit and shutting it down, nothing was found in the preheater to indicate a problem.

Inspection of the unit after shutdown showed it to be in generally good shape, with only the normal amount of coke in the dissolver. The only erosion found was in the high pressure letdown valve manifold. There was, however, a considerable quantity of coke found in the hot separator (Figure B-1). The coke filled most of the cross sectional area of the vessel from the bottom of the D/P cell tap upwards for about 8 inches. Within this coke plug were four corrosion coupons, and it is believed that these may have contributed to the coking problem by providing a large surface area for initial coke laydown. In spite of the coke build-up, the hot separator functioned properly during the entire operating period. The separator had operated at 780°F.

After a two week shutdown, Run 53 was started on May 20. The feed coal used after the shutdown was from a second batch of the Powhatan No. 5 coal from the Fort Lewis shipment. Analyses showed this batch to have slightly different properties from the first batch,

in particular, lower ash and sulfur levels. Analyses of the two batches of Powhatan coal from the Fort Lewis shipment are given in Table B-1. The first batch of this coal was used for Runs 48 through 52 and the second batch was used for Runs 53 through 55. (Run 53 appears to be a transition run, and better elemental balances are obtained for this run by using a coal analysis which is the average of the first and second batches.)

Run 53 was completed on May 31. Special unit samples were taken, and conditions were then changed to Run 54. There were a number of minor upsets during Run 54, so that several of the periods were omitted from the run averaging procedure. On June 15, a complete set of unit samples was taken, after which conditions were changed to Run 55. Run 55 was concluded on June 25, when the unit was shut down for a scheduled turn-around.

During the operating period for Runs 53 to 55, a series of tests were made with the vacuum flash column. This test series consisted of increasing the flash zone temperature in 9-18°F increments while sampling overhead and bottom products at each condition. Also, a complete material balance was calculated around the high pressure separator system for Run 55. Results of the vacuum flash tests and separator system material balance will be reported later.

Operating conditions for Runs 50 to 55 are given in Table B-2, and yields are given in Table B-3.

Runs 50 and 51, together with Run 49 completed last quarter, constitute a three-run series to investigate the effect of dissolver outlet hydrogen partial pressure on SRC-II yields. For Run 50, dissolver gas rate and purity were increased from the Run 49 values to raise dissolver outlet hydrogen partial pressure from 1285 to 1410 psia. For Run 51, gas rate and purity were returned to Run 49 values, but dissolver pressure was increased to raise outlet hydrogen partial pressure to 1485 psia. Other operating conditions were kept the same except that, due to a data logger programming error, the average dissolver temperature for Run 49 was a few degrees higher than for Runs 50 and 51. Also, the recycle solids level for Run 49 was slightly lower than for Runs 50 and 51. Run 52 was a repeat of Run 49, to see how closely yields could be duplicated.

Runs 53 and 54 were made to determine the effect on yields of increasing coal concentration in the feed slurry from 30 to 40 wt % while simultaneously increasing slurry residence time from 1.0 to 1.33 hr to keep the pounds of coal per hour per cubic foot of dissolver volume constant. Total feed solids level, pressure, and temperature for Run 53 were the same as for Run 50. Run 54 was made to determine the effect of solids recycle on yields, and the only difference between Runs 53 and 54 was the higher recycle solids level in Run 54.

Run 55 was made at conditions close to those being considered for the Demonstration Plant. The objective of this run was to provide additional data to support the Demonstration Plant design.

A P P E N D I X A

TABLE A-1
1979 COAL PROCESSING SUMMARY

	Coal Processed, Tons	On-Stream Time		Available Operating Time Adj. Hr. (a)	Average Feed Rate		On-Stream Factor		
		Days	Hours		Tons per On-Stream Day	Pounds per On Stream Hr	Days, %	Hours, %	Adjusted Hr., % ^(a)
April	592	17	346	350	34.8	3,422	54.8	46.5	98.9
May	467	17	331	347	27.5	2,322	56.7	46.0	95.4
June	753	20	461	512	37.7	3,267	64.5	62.0	90.0
Second Quarter	1,812	54	1,138	1,209	33.6	3,185	58.7	51.5	94.1
First Quarter	693	37	654	1,516	18.7	2,119	41.1	30.3	43.1
Year to Date	2,505	91	1,792	2,725	27.5	2,796	50.0	41.0	65.8

(a) Available operating time is adjusted for downtime not directly related to process problems or equipment failures.

TABLE A-2
MATERIAL BALANCE RUN 79-1
Reaction Conditions Summary

Raw Coal Feedrate, lb/hr	2686
Dehumidified Coal Feedrate, lb/hr	2511
Moisture Free Coal Feedrate, lb/hr	2501
Moisture Free Coal Space Rate, lb/hr/ft ³	50.0
Solvent Feedrate, lb/hr	4015
Slurry Feedrate, lb/hr	6526
Solvent/Dehumidified Coal Ratio, wt/wt	1.6
Hydrogen-Rich Gas Feedrate, lb/hr	360.7
Gas Feed Purity, Mole% H ₂	93.0
Hydrogen Feedrate, lb/hr	186.1
Hydrogen Feed Ratio/MF Coal Basis, SCF H ₂ /ton Coal	28200
Slurry Preheater Outlet Temperature, °F	741
Dissolver Pressure, psig	1705
Dissolver Outlet Temperature, °F	826
Dissolver Volume, ft ³	50
Coal Type	Kentucky #9 and #14 P&M Colonial Mine
Solvent Boiling Range	480°F plus

TABLE A-3
MATERIAL BALANCE RUN 79-1
RAW PRODUCT YIELDS

<u>COMPONENT</u>	<u>PERCENT M.F. COAL</u>
H ₂	-2.3
C ₁	1.6
C ₂	1.0
C ₃	0.9
C ₄	0.4
CO	-0.4
CO ₂	1.4
H ₂ S	2.3
Light oil (Ambient-380°F)	2.6
Wash solvent (380°F-480°F)	4.8
Process solvent (480°F plus)	1.8
Vacuum bottoms	70.6
Water, unmeasured yield, and loss	15.2

TABLE A-4

MATERIAL BALANCE RUN 79-1

AVERAGED ELEMENTAL FEED AND PRODUCT COMPOSITIONS

(All units wt %)

COMPONENT	COAL (As Received)	COAL M.F.	COAL M.A.F.	VACUUM BOTTOMS	LIGHT OIL	WASH SOLVENT	PROCESS SOLVENT
Moisture	6.90	-	-	-	-	-	-
Ash	10.09	10.84	-	13.87	-	-	-
Carbon	65.31	70.15	78.68	73.87	82.55	84.58	87.26
Hydrogen	4.71	5.06	5.68	5.19	11.01	9.40	8.37
Nitrogen	1.48	1.59	1.78	1.75	0.38	0.47	0.58
Sulfur	4.30	4.62	5.18	2.89	0.22	0.20	0.28
Oxygen	7.21	7.74	8.68	2.43	5.84	5.26	3.51

TABLE A-5
MATERIAL BALANCE RUN 79-1
FORCED ELEMENTAL BALANCES

<u>INPUTS, lb/hr</u>	<u>C</u>	<u>H</u>	<u>N</u>	<u>S</u>	<u>O</u>	<u>ASH</u>	<u>TOTAL</u>
M.F. Coal	1754	127	40	115	194	271	2501
Gas	<u>112</u>	<u>205</u>	<u>9</u>	<u>0</u>	<u>34</u>	<u>0</u>	<u>360</u>
Total Input	1866	332	49	115	228	271	2861
 <u>OUTPUTS, lb/hr</u>							
Gases	205	192	15	59	54	0	525
Water	0	13	0	0	114	0	127
Light Oil	81	11	0	0	5	0	97
Wash Solvent	103	11	0	0	6	0	120
Process Solvent	40	4	0	0	2	0	46
Vacuum Bottoms	<u>1437</u>	<u>101</u>	<u>34</u>	<u>56</u>	<u>47</u>	<u>271</u>	<u>1948</u>
Total Output	1866	332	49	115	228	271	2861

TABLE A-6
MATERIAL BALANCE RUN 79-1
NORMALIZED YIELDS

<u>COMPONENT</u>	<u>% M.F. COAL</u>	<u>% M.A.F. COAL</u>
H ₂	-1.6	-1.8
C ₁	1.6	1.8
C ₂	1.0	1.1
C ₃	0.9	1.0
C ₄	0.5	0.6
CO	-0.4	-0.5
CO ₂	1.4	1.6
H ₂ S	2.5	2.8
NH ₃	0.3	0.3
Water	5.0	5.7
Light oil (Ambient-380°F)	4.5	5.0
Wash solvent (380°F-480°F)	4.8	5.4
Process solvent (480°F plus)	1.8	2.0
Vacuum bottoms	67.0	75.0
Ash	10.8	-

TABLE A-7

VACUUM FLASH DRUM MBR OPERATING CONDITIONS

RUN NUMBER	DRUM FEEDRATE lb/hr	DRUM TEMPERATURE °F	DRUM PRESSURE mm Hg (Abs.)	CONDENSATE FLOWRATE lb/hr	BOTTOMS FLOWRATE lb/hr	CONDENSATE TO FEED RATIO	BOTTOMS FUSION POINT °F
1	1634	621	22	712	912	.44	346
2	1435	634	22	728	707	.51	341
3	1594	599	20	697	898	.44	295
4	1592	627	20	764	828	.48	342

TABLE A-8

FEED & PRODUCT INSPECTIONS*-VACUUM FLASH MASS BALANCE RUNS

RUN #	1			2			3			4		
TEMPERATURE	621°			634°			599°			627°		
COMPONENT	FEED	CONDENSATE	BOTTOMS	FEED	CONDENSATE	BOTTOMS	FEED	CONDENSATE	BOTTOMS	FEED	CONDENSATE	BOTTOMS
450°F	0.	7.	-	0.	0.	-	0.	0.	-	0.	0.	-
450-550°F	1.	24.	-	1.	7.	-	2.	4.	-	4.	7.	-
550-650°F	16.	21.	-	14.	36.	-	13.	28.	-	12.	30.	-
650-750°F	11.	18.	-	20.	31.	-	17.	35.	-	7.	34.	-
750-850°F	4.	13.	-	10.	15.	-	10.	22.	-	18.	17.	-
850°F	41.	17.	53.	38.	11.	63.	39.	11.	66.	40.	13.	60.
ASH	19.	-	33.	12.	-	25.	13.	-	22.	13.	-	25.
IOM	8.	-	14.	5.	-	12.	6.	-	12.	6.	-	15.

*All units are weight percent of total sample

TABLE A-9

VACUUM FLASH UNIT OVERALL MATERIAL BALANCE*BASIS: 100 Lb/Hour of Feed

COMPONENT	RUN 1			RUN 2			RUN 3			RUN 4		
	FEED	CONDENSATE	BOTTOMS	FEED	CONDENSATE	BOTTOMS	FEED	CONDENSATE	BOTTOMS	FEED	CONDENSATE	BOTTOMS
450°F	0	3	0	0	0	0	0	0	0	0	0	0
450-550°F	1	11	0	1	4	0	2	2	0	4	3	0
550-650°F	16	9	0	14	18	0	13	12	0	12	14	0
650-750°F	11	8	0	20	16	0	17	15	0	7	16	0
750-850°F	4	6	0	9	7	0	10	10	0	18	8	0
850°F	41	7	29.3	39.1	6	18.4	39.5	5	36.6	39.5	7	31.4
ASH	18.7	0	18.7	12.3	0	12.3	12.5	0	12.5	13	0	13
IOM	<u>8.3</u>	<u>0</u>	<u>8.0</u>	<u>4.6</u>	<u>0</u>	<u>6.1</u>	<u>6</u>	<u>0</u>	<u>6.9</u>	<u>6.5</u>	<u>0</u>	<u>7.6</u>
	100	44	56	100	51	49	100	44	56	100	48	52

*Units are Lb/Hour of component based on 100 Lb/Hour total feed

TABLE A-10

DISTILLATIONS OF VACUUM FLASH CONDENSATETrue Boiling Point Distillation*

<u>Observed Temp.: °F</u>		<u>Press</u> <u>Mm Hg</u>	<u>TBP Vapor</u> <u>Temperature: °F</u>	<u>Yield: Wt. %</u>	
<u>Still (Oil)</u>	<u>Vapor</u>			<u>Cut</u>	<u>Cum.</u>
463	109	80	230	-	-
474	350	60	526	4.91	4.91
482	378	55	556	4.99	9.90
482	378	40	576	5.21	15.11
484	384	35	588	5.02	20.13
496	400	35	606	5.23	25.36
499	404	30	622	5.14	30.50
507	409	26	637	5.22	35.72
500	398	15	657	5.20	40.92
508	398	11.7	669	5.30	46.22
517	413	11.5	684	5.21	51.43
523	419	10.0	701	5.41	56.84
519	400	5.0	716	5.24	62.08
539	416	5.1	733	5.40	67.48
418	330	250 Microns	768	5.34	72.82
416	360	290 Microns	800	5.38	78.20
428	359	134 Microns	834	5.31	83.51
469	404	134 Microns	870	5.61	89.12
545	477	170 Microns	973	5.38	94.50
646	552 ⁺	250 Microns	1045	2.18	96.68
646 ⁺	552 ⁺	-	1045	2.62	99.30
-	-	-	-	0.72	100.00

CUSHMAN DISTILLATION

<u>Cut</u> <u>No.</u>	<u>Observed Temp.: °F</u>		<u>Press</u> <u>Mm Hg</u>	<u>Atmospheric Equivalent</u> <u>Vapor Temperature: °F</u>	<u>Yield: Wt. %</u>	
	<u>Still (Oil)</u>	<u>Vapor</u>			<u>Cut</u>	<u>Cum.</u>
IBP	415	270	64	426	-	-
1	420	365	20	600	20.58	20.58
2	449	408	15	667	20.84	41.42
3	464	418	5	737	21.24	62.66
4	536	476	3	830	21.76	84.42
5	692 ⁺	583 ⁺	3	957 ⁺	12.56	96.98
Btms.	692 ⁺	583 ⁺	-	957 ⁺	1.80	98.78
Loss	-	-	-	-	1.22	100.00

*By Podbielniak Hyper-Cal Column

TABLE A-11

VACUUM FLASH DRUM CARRY-OVER TESTS

<u>Drum Feed Rate, lb/hr.</u>	<u>Drum Temp., °F</u>	<u>Drum Abs. Press. mm Hg</u>	<u>Condensate Rate, lb/hr.</u>	<u>Vapor Velocity, ft/sec.</u>	<u>Vapor Density, lb/ft³</u>	<u>Average %Ash in Condensate</u>	<u>Vacuum Bottoms M.P., °F</u>
1600	635	22.2	960	5.2	.0073	0.0095	357
2000	630	28.3	1040	4.4	.0093	0.0073	347
2400	622	29.4	1224	4.9	.0098	0.0060	335
2800	622	30.4	1372	5.3	.0102	0.0065	330
3000	620	34.5	1500	5.1	.0116	0.0085	310
4800	580	70.2	2208	3.6	.0241	0.0205	250

TABLE A-12

RHEOLOGICAL EXPERIMENTAL PROGRAM

PRIMARY INDEPENDENT VARIABLES

Recycle Slurry

Shear Rate

Temperature

Composition

Solvent

Vacuum Bottoms

Vacuum Bottoms

Shear Rate

Temperature

Composition

ASH

IOM

SRC

Time-temperature history

Feed Slurry

Shear Rate

Temperature

Composition

Recycle Slurry

Solvent

Coal

Particle concentration

Particle size distribution

Time-temperature history

TABLE A-13

COMPUTER SYSTEM RELIABILITY FOR THE

SECOND QUARTER OF 1979

<u>EQUIPMENT TYPE</u>	<u>% AVAILABILITY</u>		
	<u>April</u>	<u>May</u>	<u>June</u>
INPUT DEVICES:			
A.) Operator's Console CRT	100	100	100
B.) Tape Reader	100	100	100
C.) Decwriters (Avg. 3)	100	100	100
D.) Magnetic Tape	100	100	100
E.) Process/Computer Interface	100	100	100
OUTPUT DEVICES:			
A.) Tape Punch	89.72	100	100
B.) Decwriters (Avg. 3)	100	100	100
C.) Line Printer	100	100	100
D.) Magnetic Tape	100	100	100
E.) Operator's Console CRT	100	100	100
CENTRAL PROCESSOR	100	92.22	99.57
OVERALL SYSTEM	100	92.22	99.57

FIGURE A-1

"A" DISSOLVER NOZZLE REPAIR

FIGURE A-2

LETDOWN VALVE (166A) POSITION, FLOW RATE, PRESSURE DIFFERENTIAL VS TIME

FIGURE A-3

EROSION / CORROSION TEST LOOP

FIGURE A-4

PIPING INSPECTION ON
LINE 2" SL-107-EI

(ALL READINGS ARE IN INCHES)

INSPECTION DATE : 5/2/79

2" C.S. SCH 80 - NOMINAL
WALL THICKNESS = .218"

FIGURE A-5

FILTER FEED FLASH VESSEL RECIRCULATION EXCHANGER

(ALL READINGS ARE IN INCHES)

END
READING

INSPECTED BY : L.G.F.
DRAWN BY : R.I.H.

AREA 03
EAST BANK

INSPECTION DATE : 5/22/79
2-1/2" SCH 80 CS NOMINAL W/.276

FIGURE A-6

FILTER FEED SURGE SIDE/ARM EXCHANGER

(ALL READINGS ARE IN INCHES)

INSPECTED BY : L.G.F.
DRAWN BY: R.I.H.

AREA 03

INSPECTION DATE = 5/22/79

FIGURE A-7

WASH SOLVENT COLUMN **CORROSION-EROSION INSPECTION**

(ALL UNITS ARE IN INCHES)

THESE READINGS ARE SHOWN IN THE APPROXIMATE LOCATION FROM WHICH THEY WERE TAKEN ON THE INDICATED SECTION OF THE VESSEL WALL.

FIGURE A-8

INSPECTION OF THE WASH SOLVENT COLUMN
FEED LINE (HL-38-G1)

INSPECTED AND REPLACED - 6/23/79
3" SCH 40 C.S.
NOMINAL WALL THICKNESS = .216
ALL UNITS ARE IN INCHES

INSPECTED BY L.G.F.
DRAWN BY R.H.

FIGURE A-9

STRETFORD UNIT PIPING INSPECTION

6" SCH. 40 - NOMINAL WALL
THICKNESS = .280

INSPECTION DATE : 4/17/79

FIGURE A-10

DEA UNIT PIPING INSPECTION

(ALL UNITS ARE IN INCHES)

FIGURE A-11

FIGURE A-12

FIGURE A-13

MBR NO.2

FIGURE A-14

MBR NO.2

FIGURE A-15

MBR NO. 3

FIGURE A-16

MBR NO. 3

FIGURE A-17

FIGURE A-18

FIGURE A-19

VACUUM FLASH DRUM VAPOR-LIQUID EQUILIBRIUM

FIGURE A-20

DISTILLATION COMPARISON - VACUUM FLASH CONDENSATE

FIGURE A-21

PERCENT ASH IN CONDENSATE AS A FUNCTION OF NEW
VACUUM FLASH DRUM FEED RATE

FIGURE A-22

SCHEMATIC DIAGRAM - VACUUM FLASH TEST STAND

FIGURE A-23
ELEVATION LAYOUT-VACUUM FLASH TEST STAND

FIGURE A-24

HYDROGEN YIELD vs DISSOLVER TEMPERATURE

FIGURE A-25

METHANE PRODUCTION vs DISSOLVER TEMPERATURE

FIGURE A-26

VACUUM FLASH FEED DURING PLANT STARTUP

FIGURE A-27

SAMPLE CONDITIONING SYSTEM FOR HAAKE ROTOVISCO

FIGURE A-28

ROTOR SPEED VERSUS TORQUE OUTPUT

FIGURE A-29

APPARATUS FOR HEATING SAMPLE BOMBS

FIGURE A-30

**VISCOSITY OF VACUUM BOTTOMS VS. TIME HELD
AT HIGH TEMPERATURE. 1/**

FIGURE A-31

VISCOSITY OF VACUUM BOTTOMS VS. TIME HELD AT 700 °F.

FIGURE A-32

VISCOSITY OF VACUUM BOTTOMS VS. TIME HELD AT 600 °F.

A P P E N D I X B

TABLE B-1

ELEMENTAL ANALYSIS OF FIRST AND SECOND BATCHES
OF POWHATAN NO. 5 COAL FROM FORT LEWIS SHIPMENT

	<u>First Batch</u>	<u>Second Batch</u>
Elemental analysis* (moisture free), wt %		
Carbon	69.04	71.65
Hydrogen	4.96	5.24
Nitrogen	1.23	1.27
Sulfur	3.35	2.71
Oxygen	14.65	13.15
Chlorine	0.05	0.05
Metals**	<u>6.72</u>	<u>5.93</u>
	100.00	100.00
Ash	12.29	10.85

*Values shown are those used in material balance calculations.

**By difference.

T A B L E B-2

OPERATING CONDITIONS FOR SRC-II RUNS ON
PROCESS DEVELOPMENT UNIT P-99

Run No., P99-	50	51	52	53	54	55
Dissolver Pressure, psig	2000	2300	2000	2000	2000	2000
Average Dissolver Temperature, °F	851	851	855	851	851	851
Nominal Slurry Residence Time, hr	1.0	1.0	1.0	1.33	1.33	1.0
Feed Composition, wt %						
Coal	30	30	30	40	40	30
Recycle Solids	16	15	15	6	9	15
Inlet Gas Purity, %	94	90	90	90	90	90
Total Dissolver Gas Rate, MSCF/T Feed Coal	54.9	50.0	50.2	56.6	56.8	53.2

TABLE B-3

PRODUCT YIELDS FOR SRC-II RUNS ON

PROCESS DEVELOPMENT UNIT P99

Run No., P99	50	51	52	53	54	55
Yields, wt % of moisture free feed coal						
Hydrogen	-4.4	-4.5	-4.2	-3.8	-3.8	-4.0
Methane	5.3	5.1	5.5	5.5	5.4	5.5
Ethane	4.3	4.2	4.3	4.2	4.2	4.3
Propane	4.3	4.4	4.2	3.9	4.0	4.0
Isobutane	0.2	0.3	0.3	0.2	0.2	0.1
N-Butane	<u>2.2</u>	<u>2.2</u>	<u>2.0</u>	<u>1.8</u>	<u>1.9</u>	<u>2.0</u>
Total C ₁ -C ₄ Gas	16.3	16.2	16.3	15.6	15.7	15.9
Ammonia	0.4	0.4	0.4	0.4	0.4	0.4
Hydrogen Sulfide	2.1	2.1	2.0	2.1	1.8	1.8
Carbon Dioxide	0.8	0.8	0.9	0.8	0.9	0.9
Carbon Monoxide	0.1	0.1	0.1	0.1	0.1	0.1
Hydrogen Chloride	<u>0.1</u>	<u>0.1</u>	<u>0.1</u>	<u>0.0</u>	<u>0.0</u>	<u>0.0</u>
Total Other Gases	3.5	3.5	3.5	3.4	3.2	3.2
Water	8.0	7.7	7.6	7.4	7.1	7.2
C ₅ -380°F	7.8	8.1	7.4	7.0	7.5	6.9
380-550°F	14.9	15.6	15.0	14.5	13.0	14.5
550-900°F	<u>13.9</u>	<u>14.8</u>	<u>11.4</u>	<u>7.0</u>	<u>11.7</u>	<u>13.9</u>
Total C ₅ + Distillates	36.6	38.5	33.8	28.5	32.2	35.3
900°F+ Pyridine Solubles	22.8	21.7	24.7	30.7	27.1	24.1
Insoluble Organic Matter	4.9	4.5	6.0	6.6	7.6	7.4
Ash	<u>12.3</u>	<u>12.3</u>	<u>12.3</u>	<u>11.6</u>	<u>10.9</u>	<u>10.9</u>
Total 900°F+ Product	40.0	38.5	43.0	48.9	45.6	42.4

Figure B-1

HOT HIGH PRESSURE SEPARATOR - P99

Location of Coke Deposit Found at the End of Run 52