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COAL LIQUEFACTION TEST CENTER

Annual Technical Progress Report
for the period
January 1, 1977 - December 31, 1977
including
Quarterly Technical Progress Report
for the period
October 1, 1977 - December 31, 1977

Fluor Engineers and Constructors, Inc.
3333 Michelson Drive
Irvine, California 92715

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ANNUAL REPORT

1.0 OBJECTIVES

The objectives of this contract are:

a. Reactivate, renovate, and convert the Cresap Pilot Plant for production of low-sulfur fuel oil. Demonstrate by integrated steady state operations a coal liquefaction process consisting of proven reliable components to achieve coal liquefaction and desulfurization.

b. Provide the plant capability for component and equipment testing. Develop and/or test equipment components which may be required for use in any or all processes for converting coal into a clean liquid fuel suitable for electric power generation and other industries.

c. Develop operating parameters and techniques to achieve improved onstream efficiency of the facility and provide equipment and component test data in conjunction with normal plant operating activities.

This contract provides for the redesign, refurbishment, operation, maintenance and security of the facility to accomplish these objectives.

2.0 SUMMARY

All of the Offsites, Utilities, and Process Units, with the exception of the Filter Systems (Unit 300) and the Compressor Systems (Unit 900), were turned over to LCDC for inspection and preliminary checkout by the middle of January.

It was planned to complete the final construction activity by the middle of April. However, the severe winter weather of January through March greatly affected labor productivity, caused a natural gas curtailment affecting the operation of the fired heaters, and resulted in a delay in the schedule. In addition, the LCDC checkouts turned up certain items affecting plant operability. These items needed correction, and caused a delay in the final completion of the refurbishing effort until the end of August.

This additional construction activity resulted in a rescheduling of the planned date for the start of integrated operations from July 11 to the spring of 1978.

There was a redefinition of the emphasis of the program by DOE. The new emphasis will concentrate on plant operations, with test data to be obtained and reported within the framework of LCDC normal operating and

2.0 (Continued)

maintenance activities. This is discussed in greater detail in the Quarterly Report section.

2.0 (Continued)

Four runs involving the front end of the plant (Units 100, 200, 300, 500, and 800) were attempted during the last quarter of 1977. They are summarized in Table I and are discussed in detail in the Quarterly Report section.

TABLE I
SUMMARY OF OPERATING CONDITIONS

	Run No. 001	Run No. 002	Run No. 003	Run No. 004
Start Date	Oct. 11	Oct. 18	Nov. 15	Dec. 16
Duration, hours	1	20	144	55
Slurry Concentration, %	10	10	11.5	15
Temperature, °F	<700	640	700±	700±
Coal Feed Rate, tpd	5	5	5.3	6.3

3.0 DETAILED DESCRIPTION OF TECHNICAL PROGRESS

3.1 REFURBISHING

During June, July, and August, Fluor Construction completed the following list of activities associated with preparing the plant for operations:

- Holding Pond - reline with concrete
- Control room air conditioner installation
- Rework all nuclear detector installations
- Work orders left over from original refurbishment
- Modify piping to relieve stress problems and process problems
- Rewire heaters with larger wire; replace and install electric tracing
- Install monitoring stations for EPA measurements
- Rework deteriorated carbon steel steam tracing
- Rework sample stations
- Rework heaters for proper operation
- Modify piping to eliminate process problems
- Unit 600/900C punch lists
- Replace and rework instrumentation items to achieve proper plant operation
- Install items on MFD but missing in plant
- OSHA piping changes

3.1 (Continued)

- OSHA structural changes
- Parson's punch list items
- Rework reactor charge pump installation for start-up access
- Rework various pump installations for start-up access and disassembly
- Start and run in compressors 600C/900C
- Install test items
- Install Cities Service recommended Unit 600 instrument and piping changes
- Reorient valves for clearance
- Complete Unit 600 electrical and instrumentation construction
- Relocate and install utilities to trailers
- Review of NDE records to assure compliance with appropriate codes

3.2 OPERATIONS

3.2.1 Progress by Plant Sections

3.2.1.1 Section 100 - Coal Preparation

The Coal Preparation Section was successfully commissioned and subsequently produced 110 tons of product coal this year.

An extensive program of leak checking was carried out during the prestart-up. Leak repairs included installation of new closures on the maintenance ports of the L-A103B roller mill and the L-A103C spinner separator. Regasketing and weld repairs were required on the L-A103D air heater. A cap was installed at the top of the start-up stack to prevent air from leaking back into the system. The L-A103DM combustion air fan was wired into the heater shutdown network, and a butterfly valve with an automatic actuator was installed in the fan suction line. Valve V-106 was changed to fail closed instead of open on instrument air failure. A 15-second time delay was put into the PSH-106 high pressure switch at the heater outlet.

The fuel-to-air ratio on the heater was adjusted to give 3 percent O_2 in the combustion gas on low fire and 0.75 percent O_2 on high fire. After leak repairs, the oxygen level was controllable at 5 percent by volume, well below the maximum permissible level of 15 percent.

3.2.1.1 (Continued)

Coal grinding was begun on February 23. Throughput rates were low (2.4 tons per hour versus the 4.0 tons per hour design rate), but product quality was good. Moisture content was below the 1 percent design specification. The product coal contained more fines than had been expected. During later operations, coal was ground with the L-A103 spinner separator motor turned off, and the resulting coal size distribution was acceptable. Total product inventory at the end of March was 40 tons.

Modifications were made to the L-A103A rotary feeder to increase the coal feed rate and the L-A103B roller mill was altered to permit external refilling of the lubrication system.

During the month of May, raw coal was received from the Ireland Mine that was determined to be high in ash content (over 15 percent). This coal was removed from the F-A101A/B unloading and storage hoppers with the use of a vacuum tank and transferred to the Mitchell Power Plant. The Ireland Mine coal processing facilities were afterwards inspected and a program of sampling and analysis was established to provide closer control over the quality of coal received.

All outstanding construction items, predominantly related to OSHA regulations, were completed in July. On August 2, 1977, Liquefied Coal accepted care, custody, and control of Section 100.

In September, the total product coal inventory was increased to 60 tons during a smooth extended run of 5 hours.

At the conclusion of Run 003 in November, the product coal inventory was built back up to 50 tons. After Run 004 in December, coal was ground to bring the product inventory up to 70 tons.

Coal bridging and freezing problems in the feed chute to the roller mill hampered operation during December. The chute will be modified to relieve the bridging.

3.2.1.2 Section 200 - Coal Extraction

Four coal extraction runs were completed in the last quarter of 1977. Coal slurry concentrations were 11-15 percent MAF coal. The depth of extraction was typically 62 percent. The major areas of concern in Section 200 operations were:

a. Extractor mixer seal reliability.

b. C-A204 spray solvent heater reliability.

c. Plugging of the preheater coil caused by settling or coking.

d. Inadequate purge solvent supply pressure to critical instrumentation.

e. Excessive pressure drop between the slurry preheater and the extractor.

Line flushing and leak checking were completed in February and mechanical checkout of equipment and instrument loops was completed in March.

Process data log sheets were prepared. Computer programs were written to provide slurry batch recipes for the operators and to compile data from the slurry batch log sheets.

In early May, No. 2 fuel oil was introduced and recirculated through the entire unit. Afterwards, the unit was drained. During fuel oil circulation, temporary repairs were made to a leak in the thermocouple connection on the C-A204 spray solvent heater. Subsequent repairs on this heater included a baking-out of the electrically shorted heating elements at low voltage.

The recirculation of start-up solvent Sure-Sol 180 was begun while fixing the slurry preheater, B-A201. Recirculation was terminated on May 13 when the L-B205 reactor mixer mechanical shaft seals developed leakage after six hours' operation at 40 rpm. Internal inspection of the seals revealed a number of problems which are discussed in detail under Section 4.

On May 16, the section was made available to Construction for completion of critical work

3.2.1.2 (Continued)

items that included rework of the welds on the bottom section of the extractor vessel. During this time, efforts continued to repair the L-B205 mechanical seals (see Section 4).

This seal repair work was completed on July 8. At the same time, the internal and external vibration probes for the extractor tests were installed. Galling problems with the internal probes were resolved by installation of a grease fitting for lubricant injection into the probe's threaded section. However, friction and excessive vibration between the probe tip and the agitator shaft were experienced and, subsequently, the internal probes were removed from the extractor. Scoring of the probe button tips was observed.

On August 2, 1977, Liquefied Coal accepted care, custody, and control of Section 200.

After flushing and leak checking, the system was charged with solvent for closed loop recirculation. Before normal operating temperatures were reached, failure of the bottom extractor agitator seal again interrupted start-up activities. Inspection of the seal revealed the following:

a. Both seal faces were slightly scored.

b. The metal seal bellows was found to contain a small rupture-type crack.

To minimize the chances of these problems recurring, the following steps were taken:

a. The complete seal-flush piping system was chemically cleaned with inhibited citric acid and then neutralized.

b. New 10 to 20-micron screens were installed in the solvent strainers, L-A210A and B.

c. A new 10-micron filter system was installed locally for the bottom seal flush solvent supply system.

3.2.1.2 (Continued)

d. A new rotary head seal assembly and a new stationary seal mating ring were installed. The auxiliary Grafoil packing was also replaced.

Hot solvent was circulated for a total of fifteen days during mid-September, as integrated circulation was accomplished with Section 300 at 575°F. The section was shut down to correct a high-pressure drop (160 psi at 8 gpm) across the slurry line from the preheater to the extractor, and various piping modifications were made. During subsequent operation, the pressure drop across this line was observed to be 30 psi at 8 gpm.

Failure of the top seal on L-B205 occurred September 27 and interrupted integrated hot circulation of solvent from Section 200 to Section 300. Additional repairs were made as detailed in Section 4.

Coal extraction Run No. 001 began on October 11 at a slurry concentration of 10 percent MAF coal and a slurry feed rate of 4,000 lbs/hr. One hour after coal feed was initiated, a gasket failure on the pressure transmitter, PT-208-1, caused an emergency shutdown that resulted in coking of the middle section of the coil in the B-A201 slurry preheater. Efforts to dislodge the blockage at hydrotest pressures were unsuccessful. The plugged coil section was replaced; the Garlock gasket that blew out of PT-208-1 was replaced with a Flexitallic gasket.

Inspection of the J-A202A/B extractor charge pumps showed excessive wear on the Delrin disc valves. The discs were replaced with ball valves.

The subsequent coal extraction Run No. 002 began on October 18 at the same conditions as Run No. 001, and continued for 20 hours.

On October 21, internal solvent circulation in Section 200 was stopped because of a failure on the bottom seal on the L-B205 extractor mixer. The seal was inspected; the primary cause of leakage was found to be a ruptured bellows.

3.2.1.2 (Continued)

Also, a roll pin on the stationary seal member was sheared. Repair work included replacement of the bellows, both seal faces, packing, and installation of hardened shear pins. Also, the rotary member was lowered 1/4 inch on the shaft to compensate for the fact that thermal growth was less than had been expected. Further, a new bottom bearing cartridge was installed because of the damage resulting from solids contamination.

The on-off controller for the C-A204 spray solvent heater failed after 18 hours of operation and was replaced with an SCR proportional controller.

Run No. 003 began on November 15 and ended six days later on November 21. Virgin Sure-Sol 180 was used as a donor solvent, while solvent recovered in Section 500 was used as recycle solvent. The slurry concentration for this run was 11.5 percent MAF coal. Extraction depth averaged 62 percent. The B-A201 slurry preheater outlet temperature was initially 750°F. However, this was raised during the run to 770-780°F. At these conditions, the maximum tube wall temperature observed was 845°F. Over a three day period, the pressure drop on the third preheater coil increased from 15 to 35 psi, which indicated coking inside the tubes. Following the end of the run, the preheater was decoked. The extractor agitator was operated at 50 rpm at the beginning of Run 003. During the second day, the speed was smoothly increased to 100 rpm. The seals performed without incident during this six-day run.

Piping modifications were made around J-A202A/B and J-B202 to eliminate plugging in standby slurry lines and to improve pump operability.

The leakage problems on the C-A204 spray solvent heater were resolved by installing Swagelok fittings to provide a seal around the thermocouple leads.

Run No. 004 began on December 16 and ended on December 18 as a result of bottom extractor seal failure. Length of the run was 55 hours.

3.2.1.2 (Continued)

During this time, a 15 percent coal slurry was fed to the B-A201 slurry preheater at 615 gpm. The depth of extraction was about 62 percent.

High pressure drops were again observed between the preheater and the extractor. Subsequent review suggests that solvent or coal which contains moisture may result in two phase flow through this line causing abnormally high pressure drops.

Disassembly of the L-B205 bottom seal revealed that the lower seal cavity contained a large amount of coal fines and extract. The spring-loaded inner packing apparently had lifted, allowing process material to pack into the bellows. Cracks in the bellows were diagnosed as having been caused by metal fatigue. Repair work continued through the end of December.

3.2.1.3 <u>Section 300 - Solids Separation</u>

Following receipt of the Solids Separation Section from Construction, line and vessel flushing was carried out in February. Leak checking, first with air and soap, then with Genetron (a halocarbon), was completed in March. Curing of the refractory in two of the three heaters (B-A301, 304) and initial water circulation took place in April. Efforts in May were limited to curing of the refractory in the third heater (B-A302) and instrument tagging.

As part of mechanical preparation, all seventeen lined pumps were disassembled for complete internal inspection by a team comprised of representatives from Liquefied Coal and SCD, as well as the pump manufacturer and lining vendor. It was decided that all pumps exhibiting defects (chipped coating) in low velocity areas would be placed in service. Three pumps were sent to the vendor, Pacific Pumps, for repairs as follows:

a. J-A313C - Repair back cover plate.

b. J-A314D - Repair pump case.

c. J-A342A - Repair impeller.

3.2.1.3 (Continued)

Three other pumps had extensive coating defects and were sent to a Pittsburgh area shop for repair. These included:

a. J-A313B - Repair case.

b. J-A342B - Repair impeller.

c. J-A208 - Repair (warehouse spare).

Some repairs on other pumps were accomplished by the use of spare parts and/or swapping, so that one pump was provided for each service as required for plant start-up.

Cold, then hot, solvent circulation was started and maintained through June. Emphasis was on flow meter and equipment checkout in the hydroclone mode of operation. Circulation was primarily in a closed loop within the section; however, some integrated flow with Section 500 was maintained.

Instrument checks identified numerous instruments that required recalibration to meet revised specifications. These repairs were generally made in the field, and related design problems were resolved with SCD.

Proper installation of dip-tube type level instruments was verified for all vessel locations. All PSV's and relief valves were inspected and reset; instrument transmission lines were sloped to meet specification; and various piping was relocated to meet OSHA standards.

In early June, internal circulation of hot start-up solvent was established. Seal leaks appeared at the L-B330 and L-B332 agitators due to the Grafoil packing being installed incorrectly by the manufacturer. The installation of Teflon rings corrected the problem. Later in June, this section was turned back over to Construction for NDE review of piping welds. Vessel and instrument changes were made during this period to permit operation in the solvent deashing (SDA) mode upon the return of the unit to LCDC care, custody and control early in August.

3.2.1.3 (Continued)

Closed-loop circulation was established in early September. Unit temperatures were elevated to near design levels, and both closed loop and integrated circulating modes were maintained. During this period of hot circulation, more than sixty flange and valve bonnet leaks were identified. The bonnet gaskets and stem packing were replaced on the valves and new Teflon packing rings were installed on the two Strahman valves located under F-B308 surge tank and F-B332 feed tank.

The initial coal runs (001 and 002) took place in October. The total coal feed time was 21 hours. However, during this period solids separation efficiency was observed to be satisfactory. To improve the heat-up rate and temperature profiles, insulation was added to the bare manway flanges of vessels throughout the section.

Run 003, which included 144 hours of coal feed, took place in November, and provided an opportunity for extensive evaluation of Section 300 performance. The details are covered in Section 4 of this report under Test 1000. While solids separation was generally acceptable, the operation was continually plagued by pluggage in control valves and level dip-tubes. Operator attention to these items was constant, of necessity, in order to maintain operability. The larger control valves installed in the slurry line to 800 operated satisfactorily after minor modifications. Equipment failures and repairs during this time are as follows:

a. The mechanical seals on the feed pumps J-A313B/C were clogged with polypropylene following the disintegration of the filter cartridges in the seal oil system (Section 1130). The drive pins and springs were broken and the sleeves fretted around the packing area. New seal components were installed. A similar failure occurred on J-A314B pump; repairs included a new shaft sleeve.

3.2.1.3 (Continued)

b. The ceramic coating (PTI-54) on J-A313B was chipped on the outer edge of the case wear ring. The tungsten carbide coating on J-A313C was peeling off around the head wear ring and gasket area.

c. Pump repairs included the following:

J-A302 Slurry Recycle Pump - On both pumps, cracked bearing housings were experienced due to freezing weather. To prevent this from reoccurring, valved connections on the bearing housing were installed to facilitate draining.

J-A313A Primary Feed Pump - A cracked bearing housing was experienced due to the near-zero weather. The pump coatings on the case (tungsten carbide) and impeller (Stellite) were found to be damaged. Repairs were made by installing the case, head, and bearing housing from the J-A503A extract disposal pump which is coated with PTI-54. A new Stellite-coated impeller was also installed.

J-A313 Primary Feed Pump - A mechanical seal failure was experienced due to severely damaged springs and drive pins. Repairs were made by replacing the Durafite packing with Viton O-rings on both the stationary and rotating seal members. The PTI-54 coating on the pump case was also noted as being severely damaged (flaked off).

J-A314C Feed Pump - A mechanical seal leak was repaired.

J-B303B Underflow Pump - A seal leak was caused by debris lodged on springs and failure of Durafite packing. The shaft sleeve was worn; there was no wear on seal faces. New Viton O-rings were installed to replace the Durafite packing.

Run 004 marked the first use of wash solvent in the separation scheme. Its use may have contributed to a marked reduction in line and control

3.2.1.3 (Continued)

valve plugging during the run. The mixer on F-B332 primary feed tank failed immediately prior to the start of Run 004, and was inoperative during the run. No adverse effects on the operation were observed.

Hot forward feed, via the hot flush system, is being maintained at year's end as repairs to the F-B332 mixer are being made.

3.2.1.4 <u>Section 500 - Solvent Recovery</u>

Electrical, instrumentation, and mechanical checkouts were performed following the taking of care, custody, and control in January. Electrical and instrument deficiencies involving improper valve trim or position, absent or incorrect wiring, and improper control logic were identified and repaired. Mechanical difficulties were found with the J-A501 flash still feed pump (cracked wet-end casing and an out of round wear ring), the J-A504A recycle solvent pump (improperly factory machined mechanical seal), and the J-A503A extract disposal pump (extensive ceramic (PTI-54) coating defects). The J-A501 and J-A504A pumps were repaired with spare or swapped parts, and the J-A503A pump was returned to the factory.

After checkout of all pumping systems on water/glycol and the curing and decoking of the B-A501 flash still heater in March, preoperation on No. 2 fuel oil was carried out in April. Internal circulation at near normal process conditions was performed through the use of temporary jumper lines to simulate forward feed.

Early in May, the fuel oil operation was terminated and the section drained and flushed for maintenance.

During the maintenance period, a preoperation report of the fuel oil preparation was prepared and issued. The as-built locations of the skin thermocouples on the B-A501 flash still heater coil were measured and documented.

3.2.1.4 (Continued)

Following the maintenance period, Sure-Sol 180 start-up solvent was charged and standby circulation conditions were established. This start-up phase was halted to allow NDE weld inspection and repair work by Construction.

After release by Construction, hot closed loop circulation on solvent was resumed in August and maintained through September. During this period, excess solvent from 300 was recovered in Section 500 and returned to the tank farm. Extensive instrument checkouts were made to prepare for material balance calculations. All the electrical tracing elements were activated and checked for operability. In late September it was discovered that the J-A503 extract disposal pumps were capable of supplying less than the design value of 30 gpm of water flow to the extract disposal pond. In an effort to increase this flow to a rate capable of transporting granulated extract, the 10-5/8 inch diameter impeller in the J-A503B pump was replaced with an available 12-7/8 inch diameter impeller.

Runs 001 and 002 in October provided the first opportunities to operate the section on extract. The shortness of Run 001 only allowed the concentration of a small quantity of heavy ends and solids in the flash still; these were removed by draining. During Run 002, sufficient extract was produced to attempt normal operation. Initial conditions, as predicted using the computer physical properties package, were 21 inches Hg vacuum and 475°F at the E-A501 flash still overhead. At these conditions, the J-A502A flash still bottoms pump "froze up" early in the run due to overly concentrated extract. Operation was continued using the J-A502B pump at reduced conditions of 19 inches Hg vacuum and 440°F. Within 24 hours, however, the drum-out station and the granulation system were both plugged with extract. Operations were terminated when the LT-501 flash still ball float level indicator hung up, causing overfilling of E-A501. The extract was removed from the system through the flash still bottoms sample station. Following the shutdown, the bottoms piping was cleaned by a hydroblasting subcontractor.

3.2.1.4 (Continued)

Evaluation of the problems encountered during Run 002 resulted in the following changes:

a. The piping system to feed extract to the granulation syphon was modified to allow extract to enter below the water level.

b. A new extract recirculation line around the granulator, with adequate hot flush connections and no dead legs, was installed and equipped with extra tracing and insulation.

c. Extra insulation was added to the drum-out station control valve and exit line.

d. Procedures were modified to hot flush all extract from the drum-out station after each use.

e. Modified processing conditions were established to allow higher concentrated extract product temperatures.

During this same time, leakage from the B-A501 flash still heater coil was observed. Ultrasonic inspection indicated a large number of cracks in the tubes which apparently were related to polythionic acid corrosion. The coil was judged to be irreparable. Temporary jumper lines were installed to bypass the B-A501 heater and use the B-A603 start-up oil heater in this service. A new stainless steel (Type 321) coil was purchased and plans were made to restore B-A603 and B-A501 to their normal service upon receipt of the coil.

Following completion of the repair work, Run 003 began in early November at the new processing conditions: four to five inches Hg vacuum and 505°F at the flash still. These conditions enabled Section 500 to operate at design extract concentrations (75 to 80 percent) for the duration of Run 003 with no extract pluggage.

The F-A501 granulation tank, the disposal pumps, and the transport line became plugged with granulated extract during each attempt, and drumming of the extract product at a rate of 25 drums/day was required.

3.2.1.4 (Continued)

Examination of the granulated extract material indicated a good product was being formed by the relocated syphon, but that slurry velocity to the pond was too low to prevent pluggages from occurring in the transport line. To correct this problem, the J-A503A pump was replaced with a larger capacity, higher head pump. This included modifications to associated piping and electrical systems as well as the pump foundation.

Run 004, which took place in December, provided an opportunity to test the new pump. The granulation system operated effectively for 36 out of a possible 40 hours.

At year's end, the section was on standby, tanking solvent feed from either Section 300 or the F-A1107 slop tanks.

3.2.1.5 Section 600 - Hydrogenation

Care, custody, and control of Section 600 was taken on March 4, 1977.

Leak testing identified 19 Conval valves with seat or packing leaks. Following inspection of the valves by representatives from SCD, LCDC, and Conval, Inc., it was determined that required repairs would be accomplished on site by Construction. The section was returned to Construction from April 25 until the middle of August to complete the valve repairs and resolve a variety of instrumentation problems identified during the checkout of the instrument loops. Construction work in Section 600 was discontinued from May 16 until early June as full attention was directed to NDE review of piping welds in the Front End Sections of the plant.

Construction returned to Section 600 in early June to carry out NDE review and repair of piping welds, which were completed by August 19. Following a thorough steam flushing, a 600 psig nitrogen leak test revealed additional Conval valve leaks; inspection of the valves indicated the presence of sandblasting grit in the valve seats. Extended condensate flushing was required to remove the residual grit from the system.

3.2.1.5 (Continued)

Extensive repairs were made to correct damaged valve seats, defective packing, and damaged machined parts. All valves were systematically tested on nitrogen at 600 psig, 2000 psig, and 3000 psig. Valve seat refinishing, packing replacement, and valve parts replacement were required on 94 Conval valves during the year. Approximately 45 of the repairs were required on valves which had been previously repaired after leaking at a lower pressure. All Conval valves will eventually be repacked with Garfite 100 in accordance with the vendor's recommendation.

Inlet air damper modifications were required on the B-A602A/B reactor recycle heaters, B-A601 hydrogen heater, and B-A603 start-up oil heater before the refractory could be cured. Inspection of the heaters also revealed missing burner tiles in all heaters and these were replaced.

The reactor nuclear density detectors were switched to the emergency power supply, and all nuclear detectors were calibrated.

Below is a summary of major mechanical changes made to the hydrotreating section and the reasons for the changes:

- Installation of Grayloc flanges on reactor overhead lines to permit the heads to be removed.
- Steam tracing and insulation of F-A616 and associated piping to J-A608 BX/DX because of winter time transport problems with high viscosity lube oil.
- Installation of DOX oil and solvent jumpover lines around F-A608A/B to permit a safer and more orderly unit start-up and shutdown.

Operating highlights of the year are summarized below:

- All pumps and compressors were test run.
- The heaters were steam/air decoked, and NDE tested, and underwent refractory cure.

3.2.1.5 (Continued)

- The unit was leak checked up to 3000 psig with nitrogen at normal operating pressures.

- Warm solvent (approximately 300°F) circulation was established through the reactor loops, the liquid letdown vessels, into Section 700 and back to the Section 600 feed tank at a system operating pressure of 2800 psig.

- The gas recovery system in the section was operated under 2800 psig nitrogen pressure and circulated with recycle compressors JC-A601A/B via E-A601 absorber while oil circulation was maintained through the absorber system.

Late in December, a maintenance shutdown was started to make repairs to items identified during operation. Planned maintenance activities included a repacking of all of the unworked Conval valves, removal of the reactor attritor loops, installation of Grayloc connectors on the reactor bottom heads, and repairs to the leaking seals on E-A601 absorber and F-A605 entrainment separator.

3.2.1.6 <u>Section 700 - Fractionation</u>

Care, custody, and control of the section was taken on January 31. Line breaking, flushing, and leak checking proceeded in February with progress being hampered by extremely cold weather.

The preoperational checks for electrical, instrument, and mechanical equipment, including checking of all piping systems using a water/glycol mixture, resulted in the following:

a. Instrumentation deficiencies similar to those found in Sections 200 and 500 were identified and repaired.

b. The wiring for the B-A704 vacuum reboiler was found incorrect and incomplete. Repairs included redistribution of the heater loads to prevent premature fuse blowout.

3.2.1.6 (Continued)

c. All pressure relief and safety valves were bench tested and reset as required to prevent premature relief.

d. Inspection revealed that the ceramic coating (PTI-54) on the feed pumps J-A711A/B was satisfactory.

Following the necessary repairs, No. 2 fuel oil and gasoline were charged to the unit in April. The fractionation system circulated at near process conditions on a closed loop. Electrical problems continued to impede heat-up with the B-A704 reboiler.

The section was shut down late in April to accommodate construction work taking place in the area, as well as to make additional repairs to B-A704.

Upon restart of the unit in May, the vacuum system was fully activated with the B-A704 reboiler performing satisfactorily. The fractionation system was circulated cold due to defective wiring in the B-A703 fractionator reboiler.

This section was then shut down in mid-May to permit Construction to complete work on critical punch-list items. During this time, instruments were recalibrated to meet revised specifications. Investigation into the B-A703 reboiler problem revealed that incorrect, low temperature rated wiring had been used in the B-A701 preheater, and B-A702, -703, and -704 reboilers. This caused a deterioration of wire insulation due to overheating, resulting in a shortout. High-temperature wiring was installed in all the systems.

Late in June, the fractionation system was restarted and the rewired heaters were evaluated during hot closed loop circulation. All fuel oil and gasoline in the unit was transferred to the fuel oil tanks for disposal in the boilers, and the section was steamed and dried and turned over to maintenance at the beginning of July for weld evaluation and repairs. Care, custody, and

3.2.1.6 (Continued)

control was re-accepted on August 4. The SCD/LCDC instrument loop checks following the construction activities were completed August 12. The section was left on standby and no further activities took place until mid-October, when solvent was charged to the unit. Cold closed-loop circulation was maintained in all systems to prevent freezing and to continue monitoring equipment reliability. During November and December, the temperature of the circulating solvent was elevated to protect against freeze-up. The warm circulation was hampered in November due to burned out heating elements in the B-A702 and 703 reboilers. The main terminal boxes were cleared and the broken terminal studs repaired as required.

In December, the vacuum distillation section was integrated with Section 600 to simulate process conditions. Circulation was routine and continued until month's end, when Section 600 was shut down for repairs.

At year's end, Section 700 remained on standby, prepared for full plant integrated operation.

3.2.1.7 Section 800 - Carbonization

The beginning of the year was spent on chemical cleaning, line flushing, and leak testing, followed by glycol-water circulation, then fuel oil circulation. During this time, the B-A801 start-up heater could not be properly operated.

In April, the start-up heater became reliable enough to run for days with outlet oxygen levels consistently maintained around 2 percent. Recycle gas inlet piping had to be modified, mismatched fuel gas controls were replaced, the pilot gas flow was reduced, and the flame detector had to be modified.

Char-loading methods were developed. A previously spare connection on the carbonizer was used to load char while the carbonizer was fluidized at operating temperature.

3.2.1.7 (Continued)

Solvent spills inside the carbonizer caused plugs which prevented initial efforts to fluidize the carbonizer bed. A new 3/4 inch nozzle was added above the inlet check valve to free the check valve for start-up. The carbonizer overhead line was sloped away from the carbonizer to prevent backflow from the L-A807 No. 1 venturi injector. Inert gas spargers were added to the bottom of the dip legs.

After difficulty in September in igniting start-up char purchased from Consol, more volatile Cresap I char was successfully ignited and used for integrated operation. Operating ranges and optimums were established for the carbonization unit.

Among the repairs required, the following items were significant: On the spray tower, E-A801, a refurbished globe valve had to be replaced when it eroded through. When solids concentrations exceeded 30 percent during start-up attempts, the J-A806 tar quench pumps suffered erosion damage to the impellers, cases, and one seal flush cyclone. Nondestructive examination of other tar quench piping and instruments showed only slight erosion. The steel J-A804 solvent quench pumps were pitted by erosion/corrosion. These were temporarily replaced by 13 percent chrome alloy J-A701 pumps. The carbonizer refractory required repair above the grid plate. The G-A801 cyclone dip log deflectors had to be modified to eliminate plugging during operation.

Changes were made in the unit as originally constructed: Solvent lines were run to the recycle gas compressors so that recycle solvent could be used as seal oil instead of fuel oil. Motor control logic on the JC-A804 recycle gas compressors required correction and a new adjustable trip breaker was installed for reliable starting. The unit vent line was increased from 1 inch to 2 inches in size. The trim size on the spray tower level control valve, LCV-803, was increased from 1/4 inch to 1/2 inch. The recycle gas control valve, FCV-804, was replaced with a larger valve. Steam tracing was added to a few lines and numerous instruments for winter

3.2.1.7 (Continued)

protection. New carbonizer spray nozzles will be installed with less internal restriction to fix plugging problems, which occurred frequently during Runs 003 and 004. Modifications are also planned to prevent further pluggage of the closed-impeller J-A803 char slurry pumps.

3.2.1.8 <u>Section 900G - Hydrogen Generation</u>

Initial start-up and operation of Section 900G took place during March and April, 1977. The unit was operated successfully at design capacity (21,000 scfh), meeting the product purity specification (98 percent H_2). After two weeks of operation, the run was ended to accommodate a planned electrical outage.

Hydrogen production is scheduled to begin again in late January 1978 in preparation for total plant integrated operation.

Line flushing began in January and was hampered by extremely cold weather. All catalysts were charged into the reactors. Before the activated carbon was changed, repairs to the support screens in the D-A901 carbon drums were required. Following charging of the reformer catalyst, pressure drop tests proved the loading to be acceptable.

A natural gas curtailment because of extremely cold weather and lack of progress insulation delayed start-up progress. Leak checking of this unit was completed in early March. Several of the pressure safety valves were found to relieve prematurely and were reset.

Chemical cleaning of the monoethanolamine (MEA) carbon dioxide absorption system was accomplished by circulating a weak MEA solution. After cleaning, the solution was replaced by the normal 20 percent MEA operating solution.

The carbon beds of D-A901 were steam regenerated in readiness for service.

By March 31, the reformer refractory had been cured and nitrogen was carrying heat from the

3.2.1.8 (Continued)

reformer to the D-A903 shift converter in preparation for initial hydrogen generation. A Girdler representative was on site for consultation during start-up. Hydrogen generation began on April 1 without incident at 10 percent of design production capacity. Over the next two weeks, hydrogen generation rates were increased gradually until hydrogen was being generated at 100 percent of design rate. Purity of the hydrogen was 98 percent. The D-A903 shift converter and the D-B904 methanator were slow to reach design conditions because of heat losses through uninsulated manway covers. MEA level control in the E-A901 absorber required the constant attention of the operators.

Following modifications to the level control system, MEA circulation was restarted again in June to check the operability of the new controls on the E-A901 absorber. The new controller was a definite improvement over the previous method of control. An absorber level indicator is planned for the utility building control board to improve operability. Additional process insulation was completed in July. Process instrument impulse lines were all sloped to permit free draining.

The entire Hydrogen Generation unit was leak tested during August. Process leaks on the D-A903 shift converter inlet, the C-A903 hydrogen intercooler, and the C-A906 MEA reboiler required repairs.

A new vessel (F-B902) was installed in place of the F-A902 desuperheater. Records from Cresap I operations indicated that the old vessel had been operated above its design temperature several times, greatly reducing its expected life.

In November, a blue color in the circulating MEA solution was detected. Analysis indicated a copper concentration in excess of 1000 ppm. Review of the heat exchanger materials revealed that the C-A904 MEA solution cooler contained copper bearing alloys. It was decided that the solution could still be used, since the copper

3.2.1.8 (Continued)

would not interfere with the process chemistry. A new exchanger will be ordered; the existing cooler can be used until the replacement is obtained.

In December, MEA circulation was continued for freeze protection. The nitrogen purge was maintained to protect the catalysts.

3.2.1.9 <u>Section 900C - H_2 Compression</u>

Care, custody, and control of refurbished compressor JC-A900B was taken February 7.

Internal inspection of JC-A900B revealed rust spots on the second stage cylinder, slight pitting on the valve bodies, and interstage piping rust. All internal components were cleaned and the compressor was reassembled following replacement of the cast iron piston rings on the first, second, and third stages with filled TFE (Teflon) rings. The fourth stage piston rings were replaced with bronze rings. Suction piping to JC-A900A/B and the interstage piping on JC-A900B were acid cleaned. JC-A900B was subsequently operated on nitrogen with its bronze rings and nitrogen valves at pressures in excess of 3000 psig.

Attempts to operate JC-A900A were plagued by interstage gasket leaks until the neoprene gaskets were replaced with Garlock material. The JC-A900A makeup hydrogen compressor was then operated on nitrogen at pressures exceeding 3300 psig.

Both compressors were utilized as required to nitrogen leak test Section 600.

Several months' effort was devoted to subcontractor insulation of the compressor.

3.2.1.10 <u>Section 1000 - Utilities</u>

<u>January</u> - The steam generating unit had been fully commissioned on natural gas in late 1976. On January 7, the boilers were switched to No. 2

3.2.1.10 (Continued)

fuel oil with assistance provided by the Cleaver-Brooks representative. Electrical control problems with the fuel oil pump logic were identified and corrected. Due to extremely cold weather, extensive freeze damage occurred to steam tracing and condensate return lines.

As a result of the higher steam demand due to cold weather, the deaerator was identified as being undersized. The deaerator was only partially debottlenecked by repiping the boiler feed water pump relief to enter the supply drum, instead of recycling through the deaerator as originally piped. Compensation for improper deaeration was provided by adding more sodium sulfite, an oxygen scavenger.

Plant and Instrument Air utilities were fully commissioned in 1976. A solenoid control valve on the instrument air dryer required replacement.

The fuel oil supply system to the boilers was fully commissioned after receipt of fuel oil. An electrical control problem relating to the control logic of the fuel oil pumps was identified and resolved. The boilers were fired with fuel oil following the natural gas curtailment imposed in January.

Commissioning activities for the inert gas generator continued. The carbon monoxide safety monitor and alarm was installed in the generating room. Installation of code-conforming electrical disconnects for the two compressors was started.

During natural gas curtailments, nitrogen was used to satisfy inert gas demands.

Care, custody, and control of the emergency electrical generator was accepted in late 1976. The generator was checked and tested for operability. Minor instrument problems were identified and corrected.

February - Processing fuel gas was 100 percent curtailed from January 17 to February 9, at which date the curtailment was reduced to 30

3.2.1.10 (Continued)

percent. On February 25, the curtailment was further reduced to 20 percent. The boilers continued to operate on fuel oil to conserve allotted fuel gas for other users.

Warm weather in February was exploited by taking a steam outage to allow several tie-ins and repairs to major steam leaks.

The water softening system experienced control problems that were identified as the result of low water supply pressure. Hoses were used temporarily to debottleneck this problem.

A load study of the L-A1090 emergency generator to identify priority users was completed. As a result, revised operating procedures and load switching priorities were issued for critical users.

April - The plant air compressor, JC-A1030B, was inspected and repaired after the intake filter disintegrated. The compressor cylinder walls were undamaged. New valves were installed and the suction piping was cleaned. A redesigned filter was installed. A change request was issued to modify the suction and discharge piping to provide better vibration isolation.

May - All the utilities except the cooling tower, the plant air compressor, the water softener, and the steam generator were shut down to permit construction crews to complete acceptance exceptions items.

All the PSV's in the utility units were removed, calibrated, and reinstalled.

June - Repairs to the instrument air and the inert gas dryers were completed, resulting in improved temperature control during the regeneration cycle.

The nitrogen unloading pump failed and was removed for repairs. While the pump was off site, refilling of the storage tank required depressurization to 200 psi.

3.2.1.10 (Continued)

July - The steam boilers, B-A101A/B, were inspected and recertified by the National Board inspector on July 28. Work to maintain the boilers included:

a. Fireside tube and tube sheet cleaning.

b. Cleanout of filter systems.

c. Refractory mortar repair.

d. Electrical and instrumentation checkout.

August - The injection of concentrated industrial wastewater into the fuel oil burner was demonstrated.

Treated water was supplied to the cooling tower to provide better quality cooling water.

Modification to the air compressor suction and discharge piping was completed by Construction.

November - A steam outage was scheduled to repair several leaking steam and condensate valves and to install branch line isolation valves. Additionally, new check valves were installed on the discharge lines of the boiler feed pumps, J-A1011A/B.

December - Due to colder winter weather, steaming rates increased to 15,000 to 17,000 pph, at which conditions the boiler feed water pumps could not maintain design discharge pressures. These pumps, J-A1101A/B, will be inspected internally and repaired early next year.

Extensive and repeated steam trap surveys and repairs have not eliminated live steam from the condensate system. Investigation into this problem will continue next year. After repairs to the boiler feed pumps are completed early next year, each boiler will be given a capacity test.

3.2.1.11 Section 1100 - Offsites

January - Care, custody, and control of the tank farm was accepted on January 3. Fuel oil inventories were accumulated to maintain a 30-day supply of fuel oil. Sure-Sol 180 was received in preparation for start-up activities in process units.

Care, custody, and control of the chemical sewer system was accepted January 17; of process ponds, January 19; and of the process relief and flare system, January 28.

March - All transfer and distribution lines in the 1110 tank farm were flushed and leak tested. Tank interiors were inspected and swept clean, and the tanks were closed in preparation for normal use.

Flushing and leak testing of the entire 1130 seal oil system was completed.

The 1140 flare systems downstream of all the knockout pots were leak tested and inerted, and the flare pilots were commissioned. Activation of the flare relief lines upstream of the knockout pots proceeded as plant commissioning progressed.

April - After completion of chemical cleaning and sandblasting of the seal oil tanks, jumpers were installed at each individual user, and oil was recirculated through the system and the system filters to ensure a clean system. The seal oil system was placed into normal service on April 16.

August - During start-up of the 1130 seal oil system, the Buna-N filter gaskets failed in the system filters. Viton gaskets were specified to correct this problem.

October - The F-A1106 donor solvent tanks were set up to hold virgin Sure-Sol 180 for use as donor solvent. The F-A1101 recycle solvent tanks were set up to hold recycle solvent recovered in Section 500.

3.2.1.11 (Continued)

The 1120 char and extract ponds were fully commissioned. Since the extract liner has better chemical resistance, the initial char product was sent to the extract pond. This protected the char pond liner in the event that the char contained any solvent.

November - The 1130 seal oil system filters failed when the polypropylene supports melted. Replacement filters made with stainless steel supports were installed. Several mechanical seals were fouled with polypropylene deposits.

December - The recycle solvent in the tank farm was found to contain excessive moisture. This solvent was dried by recycling through the 300 hot flush system and the 500 vacuum distillation system.

3.2.1.12 Section 1200 - Environmentals

January - Care, custody, and control of the environmental units was completed in late 1976.

During January, a soda ash solution was circulated through the Stretford Unit to provide chemical cleaning and treatment. Circulation was maintained throughout the month for freeze protection.

Start-up and operating procedures for the Industrial Water Treatment facility were completed, as were flushing and leak checking activities. Minor instrument and control problems were identified and resolved, and the dirty methanol basin closures were sealed for personnel safety. Facet (the supplier) was consulted concerning affects of glycol, used in plant start-up, upon the resins and carbon absorbants. The aerator and oil retention boom were installed in the final holding pond to improve control of effluent water quality.

The sanitary waste treatment facility was affected by flow stoppage due to freezing of recirculation lines. Additionally, freeze damage occurred to the chlorine bleach addition pump. This upset was brought back into control

3.2.1.12 (Continued)

manually, and repairs to the pump subsequently returned this unit to normal service.

February - The Unit 1210 sour water stripper was commissioned using distilled water obtained from the Mitchell Power Plant.

Chemical cleaning of the 1220 Stretford Unit was completed. The soda ash solution was sewered and the system refilled with treated water.

March - The 1210 sour water stripper operated extensively on distilled water.

The 1220 Stretford Unit was fully activated using treated water in the filtration system. Process chemicals were charged.

The 1230 Unit was in process start-up at the end of the month with the assistance of a Facet start-up engineer.

April - In the 1230 Unit Waste Water Treating System, the top screen in the secondary resin column and the bottom screen on the primary resin column failed. Extensive repairs and the replacement of the primary resin followed. The vendor was consulted concerning design modifications to strengthen the screen supports.

May - The 1220 Stretford Unit was prepared for an operability test. The Sour Water Stripper was prepared to receive hydrogen sulfide at simulated design conditions. In the 1230 Unit, repairs to the screens were completed and new resin was added to the primary tank. The methanol thermo syphon reboiler (L-A1230m), was found severely fouled and was chemically cleaned.

June - The hydrogen sulfide test of Sections 1210 and 1220 was completed.

A review of the 1220 system by a Peabody representative identified several problems which were subsequently corrected:

3.2.1.12 (Continued)

a. The filter belt required replacement.

b. Filter cake spray nozzles were oversized.

c. The oxidizer liquor weir assembly required modification.

The support screens in the 1230 Unit resin columns failed again. Reinforcements were installed and the unit was returned to service.

July - The hydrogen sulfide test showed that the 1210 Unit had satisfactory boilup rates and the 1220 Unit removed the hydrogen sulfide satisfactorily. Filtration problems in the Stretford system were only partially solved by addition of a filter aid.

Consultation with Facet, the manufacturer of the Waste Water Treating System (Unit 1230), resulted in agreement that the resin screens failed due to static and dynamic loads experienced during normal system operation. Modifications agreed to were:

a. Installation of screen reinforcement.

b. Replacement of screens.

c. Better fastening of screen assemblies.

August - Filtration tests in the 1220 Unit showed that use of 4 percent filter aid as a body feed produced good cake.

The liner in the Final Holding Pond, which previously failed, was replaced by a sprayed concrete lining and was returned to normal service.

September - The 1210 and 1220 Units were placed on standby until required for normal operation.

The 1230 Unit required close monitoring during regeneration to control methanol losses and to ensure proper regeneration sequencing.

3.2.1.12 (Continued)

The sanitary treatment bleach pump was replaced by a more reliable pump.

December - During initial coal operations, the 1220 Unit processed all sour gas streams routinely. The 1210 Unit was in hot standby for normal service during the entire month.

The 1230 Unit appeared to have lost phenol absorptivity. Subsequent investigation indicates that the analytical results for phenols is being confused by some unidentified substance in the chemical waste stream. The investigation of phenols identification and control problems continues.

3.2.2 Laboratory

The laboratory received 4,900 samples and performed approximately 6,000 analyses in 1977. Approximately 60 percent of the samples were received during the last four months of 1977 during plant operations.

The samples include extensive evaluation of water samples from wells, river and effluent.

A master file of analytical procedures has been compiled and is presently being revised into a formal format. The procedures are being reviewed and approved by the Laboratory Supervisor and Technical Superintendent.

A master log system has been installed for all samples submitted to the laboratory.

A supplementary system to the computer program has been developed to record the report analytical data by Unit and Sample Point on Data Summary Forms.

An additional system has been developed to record and report special or occasional samples of a nonroutine nature. The system provides analytical data in a systematic manner for both reporting and retrieval.

The laboratory staff includes 1 Laboratory Supervisor, 1 Chemist, 4 Lead Analysts, 4 A Analysts, and 7 B Analysts. The staff has completed training to perform analyses required for plant operation. A continuing training

3.2.2 (Continued)

program is utilized to institute new or revised procedures. Monthly meetings including the Technical Superintendent, Laboratory Supervisor, Chemist, and Lead Analyst were utilized to discuss the problems, procedures, plans, and operation of the laboratory facilities.

For 1978, the ventilation system will be improved in the laboratory to remove fumes from samples and in-process analyses. The Engineering Department is providing a study and recommendations for the needed improvements.

Continued training of laboratory personnel to provide a wide base of experience and expertise with instruments and procedures is planned as well.

The precision of each type of analysis will be developed for an individual analyst and various analysts on each shift. This program was begun in November 1977 for two types of analyses. The results of the study will be published.

Storage area for retained samples and laboratory supplies will be necessary as the space available at the present time is inadequate.

There will be a continuing emphasis on performance by the laboratory staff to provide analytical services for plant operations.

3.2.3 Administration

3.2.3.1 Staffing

During 1977 there were 26 resignations from Liquefied Coal broken down among the departments as follows: Operations Department - 7, Maintenance and Engineering Department - 6, Technical Department - 10, Administrative Department - 3. All of these vacancies have been filled with the exception of one chemical engineer for the Technical Department. Vacancies exist in the Maintenance and Engineering Department for some recently created positions: mechanical engineer and draftsman.

3.2.3.2 Personnel

The major item in the personnel area during 1977 was an attempt by International Chemical Workers' Union to unionize the plant. This effort was defeated by a vote of 47 to 31. The union filed five objections to the election but withdrew them and the results of the election were certified by the National Labor Relations Board in early December.

3.2.4 Data Processing

3.2.4.1 Computer

All Digital Equipment Corporation (DEC) Engineering Change Orders (ECO) required for acceptance of DEC maintenance coverage were installed. Additional ECO's were installed at DEC expense to reflect improvements in system hardware since LCDC began maintenance coverage. Effective May 15th, the hours of maintenance coverage were increased from nine to thirteen per day, five days per week.

The installation of an adequately-sized HVAC System in May prevented further environmentally caused computer shutdowns.

A smoke detector and a high/low temperature alarm were installed. The computer lease from Fluor was extended through January 30, 1979. DEC maintenance coverage has been extended through December 15, 1978.

3.2.4.2 Laboratory and Process Systems

The Scani-valve automatic data logger is operating normally. A troubleshooting session in May with a representative from Recording Devices, Inc., and Fluor Special Systems isolated and corrected three separate Scani-valve problems. Subsequent calibration and checkout with the Instrument Department corrected several specific data points which gave erroneous readings and verified acceptability of remaining points.

Modifications and additions to Fluor process and laboratory programs were made as required to fill LCDC needs. Laboratory and process source

3.2.4.2 (Continued)

programs were received from Fluor in December. Additional modifications to laboratory programs are in progress. Laboratory and process data were entered as received.

3.2.4.3 Source Documents

The cataloging and storage of logsheets and instrument charts has continued throughout the year.

3.2.4.4 Time Distribution

Technical Department Time Distribution Reports have been run monthly since April.

3.2.4.5 Maintenance and Engineering Systems

The Lubrication Scheduler was modified to display the date on all pages and data files were changed as requested.

The Work Order System has been expanded and modified as need dictated. Two reports and a data file for closed work orders were added.

Installation of the Warehouse Inventory System was begun in July. Several follow-up trips were required to debug and modify the system. The major emphasis has been to correct, modify, and expand the original warehouse file, which was keypunched in Irvine from copies of existing warehouse data cards. The bulk of the work has been completed. Initialization of on-hand quantities is scheduled for early January, after which Receiver/Disbursement and accompanying reports for this part of the system can begin.

3.3 TEST PROGRAM SUPPORT

3.3.1 Test Program Status

In November, budget restrictions necessitated an evaluation of the test program content. In subsequent discussions and meetings between DOE, SCD, AND LCDC the scope of work was modified. Cresap is still regarded as a test facility; however, it is imperative to first demonstrate integrated steady state operation. This type of operation

3.3.1 (Continued)

is a prerequisite to a formal component and equipment testing program. In this interim start-up/operability period, there will be valuable equipment information available. Recognizing both the need for this information and limited resources, it was decided to use the following guidelines:

a. Expand the equipment under observation to include all items in liquefaction service under a given category; e.g., pumps, valves, etc.

b. Eliminate the highly sophisticated data gathering and reporting program.

c. Obtain data within the framework of the normal operating and maintenance activities.

d. Supply the bulk of the information on equipment performance from the maintenance records and observations of LCDC personnel.

3.3.2 Data Collection and Reporting

The detailed test procedures developed by SCD call for prerun and post run inspections coupled with a compilation of operating data. Procedures were developed for the inspection which facilitates collecting the necessary inspection data. It was decided to use the computer and data processing group to provide the detailed reports outlined in the test procedures. The log sheets were formatted for direct entry to computer files. Three programs were designed to enter, display, and report the test data. The first two programs were made operational, but programming of the third was discontinued until the formal testing is resumed.

To facilitate reporting, the plant was divided into thirteen data zones defined by the homogeneous nature of the process. Within each zone all of the data sources were identified as called for in the test procedures. These sources are shown in Appendix No. 1. The final computer reports were developed for review by SCD before final programming. Samples of these reports are illustrated in Appendix No. 2.

Currently the programming for this system is on hold, but programs could be activated within two weeks should the need arise.

3.3.3 Progress by Test Element

3.3.3.1 Test 1000 - Solvent Deashing

a. Cresap Testing

Settling, without the use of deashing solvent, was the sole solid-liquid separation technique employed during coal processing operations in 1977. As plant operability was the principal objective of the runs that were made, steady-state operating conditions were not generally attained. Process performance data resulting from these runs is, therefore, more qualitative than quantitative. In addition, coal slurries processed were more dilute than will be the case once normal operation has been attained, 10 to 15 wt.% versus 25 wt.%.

In spite of the qualifications listed above, several useful generalizations can be drawn from this operating experience:

1. THF insoluble concentrations in the solvent-extract product normally ranged between 0.3 and 0.5 wt.%.

2. Ash content of the THF insolubles in the overflow typically ranged between 1.0 and 7.0 wt.%.

3. Extract in the underflow to the Solids Separation Unit ranged between 2 to 6 wt.%.

Settler operating conditions during these runs were normally within the limits listed below:

Temperature, °F	500-530
Upflow Velocity, in./min.	0.25-0.45
Overflow to Underflow Ratio, O/U	1.3-3.6
Solids Flux in Underflow, lb/hr ft^2	9.0-17.0
Solids in Overflow	0.3-0.5 wt.%

More detailed summaries describing representative process conditions can be found in Appendix No. 3.

3.3.3.1 (Continued)

b. Laboratory Testing

A solvent deashing study was conducted by Conoco Coal Development Company under a subcontract with Fluor. This work was completed this past year. The study included three experimental phases:

1. Simulate start-up and steady-state operation of the Cresap pilot plant in the Consol Synthetic Fuel (CSF) mode of operation using continuous bench-scale extraction, settling and catalytic hydrocracking units on Pittsburgh Seam 8 Coal.

2. Screen various antisolvents and study solvent deashing parameters for the SRC process using batch autoclaves with filter feed obtained from the Ft. Lewis SRC Pilot Plant.

3. Conduct continuous bench-scale extraction and solvent deashing studies in the SRC mode of operation using Kentucky 9 and 14 coal and steady-state liquefaction solvent from the Ft. Lewis Pilot Plant.

All continuous extraction tests were performed on a 10 lb/hr coal unit. The batch program used a 1 gallon autoclave equipped with a movable sample probe. Highlights of these experiments are summarized below.

CSF Program Results

1. The lower boiling (Sure-Sol 180) of two petroleum-derived aromatic liquids was selected as start-up solvent for the Cresap Pilot Plant because of superior solids separation.

2. At the low settler upflow velocity of 0.3 inch/minute, the extract from steady-state operations contained 0.22 wt.% ash, slightly exceeding the target ash specification on fuel oil of 0.2 wt.%.

3.3.3.1 b. (Continued)

3. Addition of a paraffinic antisolvent (Soltrol 130) to precipitate part of the coal extract to improve settling gave little improvement in solids removal. Operational problems were encountered, resulting in massive agglomeration occurring at a moderate antisolvent rate, forcing shutdown of the rake equipped settler.

4. Settler performance improved as the liquefaction solvent approached steady-state composition.

5. About 85 percent of the extract was recovered in this single stage settling system.

6. Mass spectral and nuclear magnetic resonance analyses indicated that the liquefaction solvent approached steady-state with three cycles of integrated operation. The predominant hydrogen donors were tetralins, hydrophenanthrenes and hydropyrenes.

Batch SRC Program Results

Using a Conoco technique for initial settling rate determinations, it was shown that Soltrol 130, a 185 x 207°C paraffinic naphtha produced by the Phillips Petroleum Company, was much more efficient than SRC pilot plant light oils and various treated light oils used in the batch program. It was also demonstrated that antisolvent screening using Kauri-butanol value was a simpler and as effective a method as the batch settling tests.

Continuous SRC Program Results

1. At the low settler upflow velocity of 0.4 inch/minute, SRC product with 0.1 wt.% ash was obtained.

2. Addition of paraffinic antisolvent was required to produce low ash SRC. An

3.3.3.1 b. 2. (Continued)

antisolvent ratio of 0.3 lb/lb liquefaction solvent gave specification product (<0.18 wt.% ash) at a 0.6 ratio. Massive deposits formed, forcing shutdown of the rake-equipped gravity settler.

3. The hydroliquefaction results closely matched Ft. Lewis data. Mass spectral analyses showed that the liquefaction solvent recovered from these tests was essentially the same as the starting Ft. Lewis solvent, indicating steady-state operation.

4. SRC product recovery in the solvent deashing unit approximated 86 percent.

5. Moderate coal liquid contamination of recycled antisolvent significantly decreased antisolvent efficiency.

6. Severe mixing of the liquefaction effluent and antisolvent is not necessary. A static Koch type mixer may be adequate for a large facility.

7. It is preferable to plan an integrated bench-scale operation of extractor and settler using coal and pilot plant liquefaction solvent than just a settler operation using a pilot plant filter feed.

Process Engineering Study

In addition to providing the design basis of the solvent deashing section for the Cresap Pilot Plant, Conoco's contract also required that the experimental work be evaluated in terms of potential commercial plant design and should discuss the optimum method and region of operation.

Conoco provided a conceptual process flow sheet of a two-stage settler system applied to a commercial scale hydroextraction plant.

3.3.3.2 Test 1100 - Pumps

In conjunction with SCD, a set of shop instructions was developed for the inspection of pumps specified in the test program. The prerun inspections were completed for all but one pump (J-C303) which was returned to the vendor. Future pump inspections, however, will be limited to only a few critical measurements coupled with extensive photographic documentation.

a. Reciprocating Pumps

The reciprocating pumps have been very successful in pumping slurry. The J-A202A extractor charge pump supplied slurry for the entire 144 hours of Run 003. By the end of the run, chronic packing leaks required daily attention but did not affect operation. Also, a lubricator failure caused extreme wear and scoring of a PTI-54 ceramic coating on one plunger. Otherwise, the general success of these pumps has precipitated few occasions to inspect the plunger coatings and no relative wear data has been compiled to date.

Run 001 lasted only one hour but was sufficiently long enough to demonstrate the inadequacies of disc valves, especially those made of polymers (Delrin). The disc valves were replaced with ball check assemblies using high chrome balls (410SS) and champhered tungsten carbide seating inserts. No pumping problems were experienced in Run 002 with a 10 percent coal slurry for 20 hours and later for 144 hours in Run 003.

A number of minor operability problems required piping modifications. Modifications to the J-A202A/B and J-B202 extractor charge pumps' piping were completed in December. These changes included the replacement of reduced port ball valves in the suction lines with full-ported valves, the substitution of quarter-turn bypass valves for the original gate valves, the elimination of deadlegs for standby slurry lines, the addition of several flush connections and the installation of added piping to

3.3.3.2 a. (Continued)

permit slurry recirculation through the J-A202 suction header. Following the revamp, the J-A202A pump functioned without incident during the entire 55 hours of Run 005.

b. Centrifugal Pumps

Four major problems developed during the year and are summarized below:

1. Freeze Damage

The extreme cold of the first quarter caused cooling water in the bearing housing to freeze and crack numerous housings. Near-zero weather in December caused five additional failures. These cracks have been successfully repaired with a Devron sealing compound and work is underway to install a valved drain connection on the bearing housings.

2. Prerun Pump Coating Problems

As part of mechanical preparation, all (17) lined pumps were disassembled for complete internal inspection by LCDC and SCD, as well as the pump manufacturer and lining vendor. It was decided that all pumps exhibiting defects (chipped coating) in low velocity areas would be placed in service, and all others would be repaired. As a result, three pumps were sent to the vendor, Pacific Pumps, for repairs as follows:

(a) J-A313C - Repair back cover plate.

(b) J-A314D - Repair pump case.

(c) J-A342A - Repair impeller.

Three other pumps were noted to have extensive coating defects and were sent to a Pittsburgh area shop (Daman Industries) for repair. These included:

3.3.3.2 b. 2. (Continued)

(a) J-A313B - Repair case.

(b) J-A342B - Repair impeller.

(c) J-A208 - Repair (warehouse spare).

Some repairs on other pumps were accomplished by the use of spare parts and/or swapping, and at least one pump was provided for each service as required for plant start-up.

3. Pump Coating Failures

As longer duration runs were accomplished in November and December, it became apparent that there were serious problems with the coating-to-base material bonding of the plasma-sprayed coatings.

During seal repairs in November, it was noted that the PTI-54 coatings in three pumps and the tungsten carbide coating in a fourth pump had failed. Sixteen pumps were originally installed with PTI-54 linings and two with tungsten carbide linings. The failures took the form of chipping, cracking, and spalling of large areas of coating. These failures are thought to have resulted from poor bonding or thermal stresses due to differences between the coefficients of thermal expansion of the substrate and liner. A comparison of coefficients of expansion follows:

Selected Thermal Expansion Coefficients, in./in./F

Carbon Steel	6.5×10^{-6}
11-13 Stainless Steel Casting	6.5×10^{-6}
Triballoy 800	7.0×10^{-6}
Tungsten Carbide	4.4×10^{-6}
PTI-54	4.2×10^{-6}

3.3.3.2 b. 3. (Continued)

In December, one failure occurred in a service where there had been no previous problems; a second failure confirmed that any chips or cracks in the ceramic coating will ultimately lead to spalling and total removal of the lining. To date, PIT-54 coatings have failed in five of the 16 pumps so equipped. There have also been some problemh tungsten carbide and Stellite linings.

A decrease in coating thickness, improved surface preparation, and alternate coating methods such as the detonation gun application of tungsten carbide may result in a better bond between the liner and the base metal. Hard-chrome coating remains a potential candidate for lining pump cases. Consideration is being given to gas nitriding or chemical vapor deposition as methods to protect the more complex geometrics of impellers. Pumps specifically designed for slurry services, where coated off-the-shelf pumps are now being used, also need consideration.

4. <u>Pump Seal Failures</u>

The deleterious effects of coal processing on mechanical seals became apparent during the last few months of the year. Ten mechanical seal failures were experienced in November; six were double mechanical seals and four were single-type seals, primarily due to fouling with extract and solids. Five mechanical seal failures were experienced in December. The immediate causes of the failures include debris in the seal cavity, damaged springs, tar buildup on the seal faces and packing leaks. To some extent, the seal performance may have been affected by temperature-imposed piping stresses or changes in alignment. Improvements to date include changes in seal component materials, increased seal flush flow rates, the

3.3.3.2 b. 4. (Continued)

replacement of polypropylene with stainless steel filter components in the seal oil system and instrument improvements to prevent the undetected loss of level in the suction vessels.

The packing leaks may have resulted from the relatively low resiliency of the Durafite packing. Viton-A O-rings were substituted for the Durafite packing in two seals. There have been no O-ring failures to date.

3.3.3.3 1200 - Heat Exchangers

The only liquefaction heat exchanger to have had problems was the C-A204 Spray Solvent Heater. Chronic electrical problems resulted from leaks around the thermocouples. This leakage damaged the electrical insulation around the heating elements and limited operations to 600°F.

The C-A802 Tar Cooler was designated as a test heat exchanger in the test program. Cursory process analyses indicate good performance of the exchanger. Detailed analysis will commence as more time becomes available; however, there are some concerns that the exchanger is over-designed and it may be necessary to plug some tubes in order to analyze the heat transfer coefficient.

A second designated test exchanger is the C-A602 reactor effluent cooler. Commissioning of the hydrotreating section was started in late December and it is expected that heat exchange data will become available as operation commences in early 1978.

3.3.3.4 Test 1300 - Fired Heaters

Test program activities concentrated on the performance of B-A201 and B-A501.

a. B-A201 Slurry Preheater

The major testing activity has been to monitor the operation and development estimates of the

3.3.3.4 a. (Continued)

inside heat transfer coefficient. The heater has operated well at rates equivalent to 12 tons of coal per day and a 3:1 solvent-to-coal ratio.

A major failure was experienced during Run 001 on October 11. One hour after coal feed was initiated, a loss in the flow rate from charge pump J-A202B and a gasket failure on pressure transmitter PT-208-2 caused an emergency shutdown that resulted in coking of the middle coil in the heater, thereby terminating Run 001. Efforts to dislodge the blockage at hydrotest pressures were unsuccessful. The coil was replaced, followed by hydrotesting and x-ray examination. The Garlock gasket that blew out of PT-208-2 was replaced with a Flexitallic gasket.

The extended operations which were achieved in Run 003 facilitated evaluation of the heat transfer data. The initial work was limited to solvent because its physical properties are better known. Solvent operations before and after Run 003 were evaluated.

Calculations of the inside heat transfer coefficient, h_i, for the B-A201 slurry preheater were completed for solids-free solvent heating before and after coal processing. For clean, decoked tubes, h_i is typically in the range of 60 to 100 Btu/hr-ft^2-°F. The coefficient drops to a range of 5 to 20 Btu/hr-ft^2-°F after coal processing.

b. <u>B-A501 Flash Still Heater</u>

Beginning in late October, annual temperature profiles were noted. There was an abnormally high temperature peak approximately 50 feet (20 percent of coil length) downstream from the inlet. This problem was traced to radial cracks in the heater tube. An NDE inspection indicated that the cracks were numerous, widespread and impractical to repair. These cracks were indicative of polythionic acid corrosion which had been

3.3.3.4 b. (Continued)

accelerated after sensitization of the Type 316 stainless steel at decoking temperatures. This coil was in service during Cresap I. Temporary jumper lines to the B-A603 start-up oil heater were made prior to Run 003. A new Type 321 stainless steel coil for B-A501 was received in December and installation was scheduled for early 1978.

3.3.3.5 Test 1400 - Vessels and HP/HT End Closures

This test primarily concerns itself with the performance of the reactors and the reactor closures in Section 600. Test data will be available in 1978 after this section is brought on line.

3.3.3.6 Test 1500 - Filtration

Construction activities were completed late in the first quarter. Subsequently, punch listing, checklists and work orders for the preoperational checkout were completed. A detailed review of the L-B341 Rotary Drum Filter mechanical checkout procedure was conducted. Other than winterization, little preoperational checkout work was completed due to limited manpower available for the main-stream start-up activities.

During the year, representatives from both Krauss-Maffei and Funda visited the plant and toured the filtration area.

It has been decided by mutual agreement between DOE and Fluor to indefinitely postpone any operational activity with the filters. They will be mothballed.

3.3.3.7 Test 1600 - Valves and Piping

Throughout the year the Test 1604 Marlin check valve has been underway. This valve is located on the recycle gas line to the D-A801 carbonizer and is an integral part of all operations. Three inspections have been performed; the first was a preoperations inspection and the others were as a result of operating difficulties. The first post operations inspection occurred when

3.3.3.7 (Continued)

the valve became caked with coked fuel oil and the second was after the introduction of char. The first time one of the springs was bent such that it was only connected on one side. This was probably the result of attempts to clear the blockage in the valve. Both springs were replaced and it was returned to service.

During initial operations with char, the process engineer observed that the valve was flapping, creating pressure pulsations in the carbonizer. Therefore, when the valve was taken out for cleaning char blockage on September 7, 1977, one of the springs was disconnected. Disconnecting the one spring has resulted in more stable performance of the carbonizer. Minor problems to clear fines which have settled on the flappers have occurred after each major shutdown.

All other valves specifically identified in the test procedures will require a prerun inspection before being placed in service.

On a plant-wide basis, the hand valves have generally functioned well with only two major erosion failures. A more than usual number of minor problems were experienced with refurbished Cresap I valves. The most serious problems with new valves were experienced in Section 600 with the high-pressure valves furnished by Conval. During March, leak checking began with a 95 psig pressure test. A number of high-pressure Conval valves were identified as leaking at the packing and/or through the valve trim. Following inspection of the valves by SCD, LCDC and Conval representatives, it was determined that the required repairs would be accomplished on site. A change request was issued for construction to lap the valve seats or tighten the packing on approximately 19 valves. By mid-September, leak checking had progressed to 600 psig and approximately 21 Conval valves were found leaking. Weld spatter and blasting grit were found in the bottom areas of many valves. The problems were characterized as follows:

3.3.3.7 (Continued)

a. Damaged seats;

b. Defective packing;

c. Damaged machined parts.

Only four valves out of the original 21 were repaired by late September. To determine the total scope of work, further leak testing was progressed by loop sections. The repair work was defined as follows:

a. Seat refinishing - 18 valves;

b. Repacking - 28 valves;

c. Replacement of parts - 7 valves.

The general repair procedures were reviewed with Conval representatives. By late October, the bulk of work consisted of repacking the valve stems with Garfite 100 and seat lapping. Repairs of this type continued throughout November and December.

3.3.3.8 <u>Test 1650 - Piping Hydraulics</u>

Work on this test was limited to MFD checkout and preparation of prerun inspection work orders. Some flushing and pressure testing was included as part of the initial start-up. All prerun inspection and pressure testing would be required to commission the test.

3.3.3.9 <u>Test 1700 - Instrumentation</u>

Other than MFD checkout and inventory inspection, work on the flow instruments and letdown valves specified in the test program was limited. The Fisher high pressure letdown valve will be used in Unit 600 and a detailed prerun inspection will be made when the valve is received from Fisher. The Section 200 letdown valves will have second priority with more extensive work to be initiated if that service demonstrates chronic problems.

3.3.3.10 Test 1800 - Extractor

Throughout 1977, the L-B205 extractor agitator was undergoing development aimed at improving the potential onstream time. The best performance was achieved between October 25 and December 18. During this time the extractor operated continuously on slurry for 144 hours during Run 003 and for 55 hours in Run 004. In addition, this period included 9 days of cold solvent circulation, 7 days of hot solvent circulation and three start-ups.

In arriving at these extended operations, it was necessary to make several modifications:

a. Carbon throttle bushings were replaced with a flushing cup on the top seal and an auxiliary packing on the bottom.

b. The seal flush system was instrumented more effectively to help insure cleanliness of the seal area.

c. Both seals were repositioned on the shaft to correct for less than expected shaft growth and thereby exert sufficient force on the seal faces.

d. Roll pins used to prevent rotation of the stationary seal face were replaced with hardened shear pins to prevent failure.

The most chronic problem centers on keeping coal fines, extract and debris out of the seal area. These solids pack into the bellows, restrict movement and lead to failures at the seal faces or fatigue of the bellows metal. Several improvements in the seal flush system have been made and others are under engineering evaluation.

The machine surveillance system (MSS) was commissioned and remains operational. The elaborate data collection program was deleted when scoring, and vibration problems were experienced with the internal probes. The internal probes were removed but the external probes have continued to be used to monitor for bearing vibration.

3.3.3.10 (Continued)

In the next year, it is anticipated that some further development of the single mechanical seals will evolve from operating experience. The use of double seals will be considered for the top of the extractor if extensive problems develop. No further formal vibration studies are planned and the agitator speed will be set to minimize seal deterioration yet provide enough mixing for extraction.

3.3.3.11 <u>Section 1900 - Corrosion/Erosion</u>

An early start of corrosion/erosion testing was planned and in June, all of the corrosion coupons, probes, and erosion sticks were installed. As start-up activities commenced, sealing problems developed with all of the installations. Normal maintenance procedures were unsuccessful in sealing the leaks. Therefore, special attention was given to the Test 1931 installation in Section 300 and all other loops were isolated from their respective units so as not to interfere with the start-up. In the 1931 loop, careful attention permitted sealing at ambient conditions. After heating with process fluid, however, further tightening was required to seal the elements. The second tightening caused the corrosion coupons to become misaligned in a manner that would give erroneous results. The sealing problem was reviewed with SCD and discussed with the vendor, who has prepared a recommendation and cost information. The possibility of using an elastomeric seal was investigated. A material called Vespel (a polymide resin) was tested in Sure-Sol 180 for two days at 400°F. No degradation was observed; dimension, elasticity, and strength appeared to have been retained. However, ultimately the vendor recommended an aluminum seal ring and special seal caps. A proposal to concentrate and solve the mechanical problems on one loop in Section 300 was under development and scheduled for review in early 1978.

Several special projects not directly associated with the formal test program gave some interesting results:

3.3.3.11 (Continued)

a. A bench test was conducted to evaluate the relative abrasivity of the coal and char under consideration for slurry testing in Sections 200 and 300. Against carbon steel, the char slurry exhibited the higher erosion rate of 1.1 mpy at 7-1/2 fps velocity.

b. A laboratory study on the effects of moisture in Sure-Sol 180 on carbon steel indicated that the corrosion rate with moisture present was three times greater than the control sample, i.e., 1.74 mpy versus 0.45 mpy. Clearly visible evidence of pitting and visible corrosion residue was found only in the sample with 1 percent water added.

Appendix No. 1

Data Collecting and Reporting

Test Zones and Test Data Sources

TEST ZONES

Test Zone	Unit	Location/Process Stream	Tests
1	200	J-A206 and F-A203 B Loop/Slurry Preparation	1132 1607 1608 1660 1911
2	200	From F-A203A to Extractor	1101 1103 1105 1110 1115 1120 1311 1921
3	200	Between D-A201 and F-B332/Extractor Effluent	1751 1800 1922 1923
4A	300 HYD	F-B332, B-A302 and J-A313 Loop/Primary Hydroclone Feed -or-	1140 1145 1150 1155
4B	300 SDA	F-B332, B-A301 and J-A313 Loop/Unit 300 Feed	1702 1931 1932
5A	300 HYD	F-A301, B-B304, and J-A314 Loop/Wash Hydroclone Feed -or-	1160 1165
5B	300 SDA	F-B330 and J-A314 Loop/Settler Underflow	1170 1175
6A	300 HYD	F-B330 and J-B303 Loop/Slurry to 800 Area -or-	1130 1135 1180 1185
6B	300 SDA	F-B301 and J-B303 Loop/Settler Feed	1190 1191 1606 1670
7	500	E-A501, B-A501 and J-A502 Loop/B-A501 Feed	1316 1601 1603 1703 1941

Test Zone	Area	Location/Process Stream	Tests
8	600	Between D-A601 and F-A612/Hydrotreated Residue	1621 1780 1790
9	600	Between D-A601 and F-A602/Hydrotreater Overhead	1204 1211
10	700	Between E-A703 and E-A708/Donor Solvent	1961
11	800	Between JC-A803 and D-A801/Carbonizer Fluidizing Gas	1604
12	800	Between E-A801 and C-A802/Tar and Char Cooler	1208 1971
13	800	Between F-A802 and Char Pond/Char-Water Slurry	1602 1605

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TEST DATA SOURCES

Test Zone 1

Tests Equipment No.	1132 J-A206	1607	1608	1660	1911
Flow Rate	XFR-1607	XFR-1607	XFR-1607	XFR-1660	XFR-1607
Temperature	THI-254	XTI-200-6	XTI-200-6	XTI-200-6	XTI-200-6
Pressure	PI-231/PI-206	PI-206	PI-206	XPI-221	PI-206
Sample Point	SC-1	SC-1	SC-1	SC-1	SC-1
Special:					
Seal Oil Pressure	PI-224	—	—	—	—
Seal Oil Flow	FI-260	—	—	—	—
Corrosometer	—	—	—	—	XCP-1911-1

Analysis:

Analysis	Sample Point
Solids Content	7
Particle Size	6 (-325 needed)
H_2O	7
H_2S	Spot Check Overhead (2/run)
S (liquid only)	8 (needed)
CO_2	Spot Check Overhead
NH_3	" " "
H_2	" " "
Neutralization Number	8 (in place of pH)
Viscosity	Physical Properties Data

Test Zone 2

Tests	1101 1115	1103 1110	1105 1120	1311	1921
Equipment No.	J-A202A	J-A202B	J-B202	B-A201	
Flow Rate	FR-223	FR-223	FR-223	FR-223	FR-223
Temperature	THI-253	THI-253	XTI-200-9	XTR-1311-1/22	TRC-206
Pressure	PI225/PI-226	PI-227/PI-228	PI-227/XPI-214	PR-208-1	PCR-201

Special:

Varidrive Speed	Scale	Scale	Scale	---	---
Pressure Drops	---	---	---	XPDR-1311-1,2,3	---
Intermediate Temp	---	---	---	XTR-1311-20,21	---
Tube Wall Temp	---	---	---	XTR-1311-2 thru 19 TRC-259,TIC-204 TIC-205	---
Fuel Flow	---	---	---	FR-254	---
Fire Box Temp	---	---	---	XTI-1311-25,26,27	---
Stack Exit Temp	---	---	---	XTI-1311-28	---
Flue Gas Oxygen	---	---	---	XAW-1311	---
Flush Pressure	---	---	XPI-216	---	---
Flush Flow	---	---	XFI-1115	---	---
Corrosometer	---	---	---	---	XCP-1920-1,2,3,5

Analysis	Sample Point
Solids Content	8
Particle Size	6 (-325 needed)
H_2O	7
S (-liquid only)	8 **needed**
H_2S (overhead)	9
CO_2 (overhead)	9
NH_3 (overhead)	9
H_2 (overhead)	9
Neutralization Number	8
Viscosity	Physical Properties Data 8 Spot Check (2/week)
Stack Gas Composition	130

Test Zone 3

Tests	1751	1800 D-A201	1922	1923
Flow Rate	FR-256	FR-256	FR-256	FR-256
Temperature	TIC-202	LS-201	TI-1-45	TI-1-11
Pressure	XPI-218/XPI-219	PRC-201	PI-268	PRC-201

Special:

Level	---	LRC-20/LI-203	---	---
Spray Temp	---	TIC-223	---	---
Spray Filter ΔP	---	PDI-201	---	---
Varispeed Setting	---	Scale	---	---
Bearing Temp	---	XTI-1800-1/2	---	---
L-A205M Current	---	XAI-1800	---	---
Shaft Growth	---	Dial Indicator	---	---
Machine Protection System	---	XSR-1800	---	---
Valve Stem Indication	Scale	---	---	---

Analysis	Sample Point
Solids Content	11
Particle Size	11
S (liquid only)	11 (needed)
H_2O (overhead)	9
H_2S "	9
CO_2 "	9
NH_3 "	9
H_2 "	9
Neutralization Number	11 (needed)
Viscosity	11
Specific Gravity	11

Test Data Sources (cont'd)

Test Zone 4 A-Hydroclone

Tests	1140 J-A313A	1145 J-A313B	1150 J-A313c	1155 J-A313D*	1702	1931 1932
Flow Rate	FR-315+FR-317	FR-315+FR-317	FR-315+FR-317	FR-315+FR-317	FR-315	FR-315
Temperature	TI-1-16	TI-1-16	TI-1-16	TI-1-16	TI-1-46-3	TI-1-46-3
Pressure	PRC-306/ PI-378	PRC-306/ PI-380	PRC-306/ PI-382		XPI-310/ XPI-311	PI-350

Special:

Seal Oil Pressure	PI-379	PI-381	PI-383		---	---
Seal Oil Flow	FI-372A	FI-372B	FI-372C		---	---
Corrosometer	---	---	---	---	---	XCP-1930- 1,2,3,5
Test Flow Meters	---	---	---	---	XFR-1700-1, 2,3,4	---
Test Valve Stem Indication	---	---	---	---	Scale	---

Analysis	Sample Point
Solids Content	13
Particle Size	11
S	13
H_2O (overhead)	Spot Check F-A318 (1/week)
H_2S "	126
CO_2 "	126
NH_3 "	126
H_2 "	126
Neutralization Number	13 (in place of pH)
Viscosity	13
Specific Gravity	13

Test Data Sources (cont'd)

Test Zone 4 B-SDA

Tests	1140 J-A313A	1145 J-A313B	1150 J-A313c	1155 J-A313D*	1702	1931 1932
Flow Rate	FR-315+FR-317	FR-315+FR-317	FR-315+FR-317	FR-315+FR-317	FR-315	FR-315
Temperature	TI-1-16	TI-1-16	TI-1-16	TI-1-16	TI-1-46-3	TI-1-46-3
Pressure	PRC-306/ PI-378	PRC-306/ PI-380	PRC-306/ PI-382		XPI-310/ XPI-311	PI-350

Special:

Seal Oil Pressure	PI-379	PI-381	PI-383		---	---
Seal Oil Flow	FI-372A	FI-372B	FI-372C		---	---
Corrosometer	---	---	---	---	---	XCP-1930-1,2,3,5
Test Flow Meters	---	---	---	---	XFR-1700-1,2,3,4	---
Test Valve Stem Indication	---	---	---	---	Scale	---

Analysis	Sample Point
Solids Content	11
Particle Size	11
S	11
H_2O (overhead)	9
H_2S "	9
CO_2 "	9
NH_3 "	9
H_2 "	9
Neutralization Number	11 (needed)
Viscosity	11
Specific Gravity	11

Test Zone 5 A- Hydroclone

Tests	1160 J-A314A	1165 J-A314B	1170 J-A314C	1175 J-A314D*
Flow Rate	FR-316+FR-311	FR-316+FR-311	FR-316+FR-311	FR-316+FR-311
Temperature	TI-1-20	TI-1-20	TI-1-20	TI-1-20
Pressure	PRC-305/PI-367	PRC-305/PI-369	PRC-305/PI-371	

Special:

Seal Oil Pressure	PI-368	PI-370	PI-372
Seal Oil Flow	FI-373A	FI-373B	FI-373C

Analysis	Sample Point
Solids Content	13
Particle Size	13 (desired)

*Shop Spare

Test Zone 5 B-SDA

Tests	1160 J-A314A	1165 J-A314B	1170 J-A314C	1175 J-A314D*
Flow Rate	FRC-331	FRC-331	FRC-331	FRC-331
Temperature	TI-1-36	TI-1-36	TI-1-36	TI-1-36
Pressure	PRC-322/PI-367	PRC-322/PI-369	PRC-322/PI-371	
Sample Point:	300 SC-A-12	300 SC-A-12	300 SC-A-12	300 SC-A-12

Special:

Seal Oil Pressure	PI-368	PI-370	PI-372
Seal Oil Flow	FI-373A	FI-373B	FI-373C

Analysis	Sample Point
Solids Content	22
Particle Size	22

*Shop Spare

Test Zone 6 A - Hydroclone

Tests	1130 1185 1191 J-B303A	1135 1190 J-B303B	1180 J-C303*	1606	1670
Flow Rate	XFR-1130	XFR-1130	XFR-1130	XFR-1130	XFR-1670
Temperature	TI-1-36	TI-1-36	TI-1-36	TI-1-46-2	TI-1-46-2
Pressure	PRC-322/PI-373	PRC-322/PI-375	PRC-322/PI-377	PI-392	XPI-314

Special:

Seal Oil Pressure	PI-374	PI-376	PI-376
Seal Oil Flow	FI-371A	FI-371B	FI-371B

Analysis	Sample Point
Percent Solids	16
Particle Size	16 (needed)
Specific Gravity	16
Viscosity	16

*Second Spare will Replace J-B303B.

Test Zone 6 B-SDA

Tests	1130 1185 1191 J-B303A	1135 1190 J-B303B	1180 J-C303*	1606	1670
Flow Rate	XFR-1130	XFR-1130	XFR-1130	XFR-1130	XFR-1670
Temperature	TI-1-20	TI-1-20	TI-1-20	TI-1-46-2	TI-1-46-2
Pressure	PRC-305/PI-373	PRC-305/PI-375	PRC-305/PI-377	PI-392	XPI-314
Sample Point	300 SC-A-8	300 SC-A-8	300 SC-A-8	300 SC-A-8	300 SC-A-8

Special:

Seal Oil Pressure	PI-374	PI-376	PI-376
Seal Oil Flow	FI-371A	FI-371B	FI-371B

Analysis	Sample Point
Percent Solids	18
Particle Size	18
Specific Gravity	18
Viscosity	Physical Properties Data

*Second Spare will Replace J-B303B.

Test Zone 7

Tests	1316 B-A501	1601	1603	1703	1941
Flow Rate	FRC-501+FRC-502	FRC-502	FRC-502	XFR-1700-5,6,7,8	FRC-501+ FRC-502
Temperature	XTR-1316-1/17	TI-5-C-15	TI-5-C-15	TI-5-C-15	XTR-1316-17
Pressure	PI-5041	XPI-5046	PI-5003	PI-5003 or 5002	PI-5045

Special

Pressure Drops	XPDR-1316-1,2,3	---	---	---	---
Intermediate Temp	XTR-1316-2,16	---	---	---	---
Tube Wall Temp	XTR-1316-3 thur 15	---	---	---	---
Fuel Flow	XFI-1316	---	---	---	---
Fire Box Temp	XTI-1316-25,26,27	---	---	---	---
Stack Temp	XTI-1316-28	---	---	---	---
Flue Gas Oxygen	XAW-1316	---	---	---	---
Draft	XPI-1302	---	---	---	---

Analysis	Sample Point
Stack Gas	
Composition	? (needed)
Solids Content	19, 34
Particle Size	19
H_2O	Not Available
H_2S	"
CO_2	"
NH_3	"
H_2	"
S (liquid only)	19 (need), 34 (need)
Neutralization Number	19 (need), 34 (need)
Specific Gravity	19, 34
Viscosity	19, 34

Test Zone 8

Tests	1621	1780 1790
Flow Rate	FRC-6013+FRC-6014	FI-6043+FRC-712
Temperature	XTI 750-6	TR-3,4
Pressure	XPI-639	PI-633/PRC-6013
Sample Point:	600 SC-B-12 & 600 SC-5	600 SC-B-12 & 600 SC-5
Special:	Leak Observation	----
Stem Indication	---	Scale

Analysis	Sample Point
Solids Content	128
Particle Size	128 (need-325)
Specific Gravity	128, 42 (need)
Viscosity	Physical Properties Data
ASTM D-86	42 (need)
Vapor Analysis	42

Test Zone 9

Tests	1204 C-A601	1211 C-A602
Flow Rate	XFR-1204	XFR-1211 + FQI-6053
Temperature	TI-2-8/TRC-6040	TI-2-5/TI-2-9
Pressure	XPI-635	PRC-6015
Sample Point		
Special		
Wall Temperature	XTE-1204 1 through 7	-----
Pressure Drop	-----	XPDI-1211
Cooling Water Flow	---	XFI-1211
Cooling Water Temp	---	XTE-1211-3/-4
Ambient Temp		---

Analysis	Sample Point
None Requested	None Available

Test Zone 10

Test	1961
Flow Rate	FR-708 + FO-711
Temperature	TI-3-26
Pressure	PRC-703

Special:

Analysis	Sample Point
Solids Content	58 (needed)
H_2O	Not Available
H_2S	"
CO_2	"
NH_3	"
H_2	58
S	58
Neutralization Number	58 (in place of pH)

Test Zone 11

Test	1604
Flow Rate	FR-830
Temperature	TI-1-25
Pressure	PI-8102
Sample Point	None
Special:	None
Analysis	None Requested

Test Zone 12

Tests	1208 C-A802	1971
Flow Rate	FR-829	FR-829+FR-833 + FO-812
Temperature	XTE-1208-1/-2	TI-811
Pressure	PI-8014	PI-8023 or PI-8024
Sample Point:	800 SC-A-4	800 SC-A-4
Special:		
Condensate Flow	XFI-1208-1	---
" Blowdown	XFI-1208-2	---
" Temp	XTE-1208-3	---
" Blowdown Temp	XTE-1208-4	---
Steam Pressure	PRC-816	---
Process Pressure Drop	XPDI-1208	---

Analysis	Sample Point
Solid Content	67
Particle Size	67 (-325 needed)
H_2O	Spot Check Overhead
H_2S	" (2/run)
CO_2	"
NH_3	"
S	67
Neutralization Number	67 (in place of pH)

Test Zone 13

Tests	1602	1605
Flow Rate	XFR-1602	XFR-1602
Temperature	XTI-200-6	XTI-200-6
Pressure	PI-8002	PI-8002
Sample Point	800-SC-1	800-SC-1

Analysis

Percent Solids	59
Particle Size	59 (-325 needed)
Specific Gravity	59
Viscosity	Calculate

Appendix No. 2

Data Collecting and Reporting

Computer Format for Test Data

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Computer Format for Test Data

The attached typical formats have been developed to allow computer compiling and reporting of test data. Most of the data can be taken from the log sheets which are already formatted for computer input. For the remainder, specifically the corrosion probe readings, computer formatted data sheets have been designed. Once in the computer, the data can be manipulated in a variety of useful ways. Raw numbers such as "roots" and probe resistances can be converted into engineering units such as pound per hour and mils per year. Running and daily averages can be calculated. Preliminary data reduction can be performed; for example, calculating the slurry velocity in the erosion test sections. This test can then be printed out in report form on the line printer.

The heading of the attached report format provides the report data, test zone number and description, and a listing of the test numbers for which the data on that page are applicable. The first two columns provide the time and data for each reading shown. The next three columns provide general process information. The remaining columns contain information specific to a given test.

TEST ZONE 2

SLURRY FEED — TEST 1100 1103 1105 1110 1115 1120

****

TIME	DATE	PROCESS INFO			J=A202A		J=A202B		J=B202			
		FLOW LB/HR FR-223	TEMP DEGF THI-253	PRESS PSIG PI-227	VARIDRIVE SPEED RPM	DISCHARGE PRESS PSIG PI-226	VARIDRIVE SPEED RPM	DISCHARGE PRESS PSIG PI-228	VARIDRIVE SPEED RPM	DISCHARGE PRESS PSIG XPI-214	FLUSH PRESSURE PSIG XPI-216	FLUSH FLOW GPH XFI-1115

TEST ZONE 2

SLURRY FEED TEST 1311
****

TIME DATE	PROCESS INFO FLOW LB/HR FR-223	TEMP DEGF XTR-1311	PRESS PSIG PR-208-1	B-A201 TUBE VEL FT/SEC	OUTLET PRESS FR-208 PSIG	OUTLET TEMP DEGF TRC-206	FUEL FLOW SCFM FR-254	FLUE GAS OXYGEN% XAW-1311	STACK TEMP DEGF TR-2-16	STACK VEL T/C DEGF XTE-28	STACK DRAFT IN H2O XPI-1301

DATE:

LIQUEFIED COAL DEVELOPMENT CORP.
CRESAP, WV
PROCESS AND TEST ENGINEERING

TEST ZONE 2

SLURRY FEED TEST 1921
****

TIME DATE	PROCESS INFO			TEST=1921				
	FLOW LB/HR FR-223	TEMP DEGF TRC-206	PRESS PSIG PRC-201	CORSMTR CS MIL/YR XCP-1	CORSMTR 5CR MIL/YR XCP-2	CORMSTR 9CR MIL/YR XCP-3	CORSMTR 316SS MIL/YR XCP-5	SLURRY VELOCITY FT/SEC 0.75 PIPE

TEST ZONE 3

EXTRACTOR EFFLUENT

TEST 1751 1800 1922 1923

****

TIME DATE	PROCESS INFO			TEST 1751				TEST 1922		TEST 1923
	FLOW LB/HR FR-256	TEMP DEGF TI-1 -24	PRESS PSIG PRC-201	TEMP DEGF TIC-202	INLET PSIG PI-218	OUTLET PSIG PI-219	STEM POSITION SCALE	TEMP DEGF TI-1 -45	PRESS PSIG PT-268	SLURRY VELOCITY 0.75 PIPE

TEST ZONE 3

EXTRACTOR EFFLUENT TEST 1800
****

TIME DATE	TEST 1800 D=2012L=A2558			TEST 1800 UPPER SEAL					TEST 1800 LOWER SEAL				
	LEVEL FEET LRC-201	MIXER RPM XSE-1802	MIXER CURRENT AMPS XAI-1800	BEARING DEGF XTI-1	FLUSH GPH FI-203	FLUSH PSIG PI-271	FLUSH DEGF TI-260	QUENCH PSIG PI-274	BEARING DEGF XTI-2	FLUSH GPH FI-204	FLUSH PSIG PI-272	FLUSH DEGF TI-261	QUENCH PSIG PI-275

Appendix No. 3

Test 1000 - Solvent Deashing

Material Balance and Performance Information

Solids/Liquid Separation
Material Balance & Performance
Date 11/16/77 Time 1200

(Values in Parens are calculated)

(A) Feed to Primary Settler

	lbs/Hr	%
Total	7011	
Extract	(407)	(5.8)
Solvent	(5875)	(83.8)
Solids	(729)	(10.4)
Ash	(147)	(2.1)
Ash on Extract		26.6

F-8331

Temp	510 °F
°/u	1.4
V_0	0.37 in/min
G_{us}	16.8 lb/hr ft²

Overflow to 500

	lbs/Hr	%
Total	3912	
Extract	(266)	(6.8)
Solvent	(3615)	(92.4)
Solids	32	0.83
Ash	0.45	0.0115
Ash on Extract		0.17

(B) Wash Solvent

	lbs/Hr	%
Total		
Extract		
Solvent		
Solids		
Ash		
Ash on Extract		

Feed to F-8330

	lbs/Hr	%
Total	2796	
Extract	(140)	(5.0)
Solvent	(1915)	(68.5)
Solids	741	26.5
Ash	218	7.8
Ash on Extract		61

F-8330

Temp.	500 °F
°/u	1.5
V_0	0.62 in/min
G_{us}	17.3 lb/hr ft²
W/F	0

Sec. Overflow to F-A301

	lbs/Hr	%
Total	1932	
Extract	(106)	(5.5)
Solvent	(1457)	(75.4)
Solids	369	19.1
Ash	85	4.4
Ash on Extract		44

Underflow to 800

	lbs/Hr	%
Total	1280	
Extract	(72)	(5.6)
Solvent	(991)	(77.4)
Solids	218	17
Ash	47	3.7
Ash on Extract		40

Performance - Recoveries

		F-8331	F-8330	Overall
Extract,	%F	65	76	91
	Theo.	52	660	
Solids,	%F	102	29	30
	Theo.	95	<0	
Ash	%F	148	22	32
	Theo.	100	<0	

Material Balance

	F-8331	F-8330	Overall
Total	96	115	102
Extract	100	127	109
Solvent	94	128	103
Solids	106	79	85
Ash	149	61	90

(Values in Parens are calculated)

Solids/Liquid Separation
Material Balance & Performance
Date 11/20/77 Time 2100

Feed to Primary Settler

	lbs/Hr	%
Total	4640	
Extract	264	(5.7)
Solvent	3944	(85.0)
Solids	432	(9.3)
Ash	96	(2.16)
Ash on Extract	27	

F-B331

Temp 530 °F
δ/μ 1.35
V_0 0.26 in/min
G_{us} 9.2 lb/hr ft²

Overflow to 500

	lbs/Hr	%
Total	2915	
Extract	246	8.44
Solvent	2650	90.9
Solids	19	0.64
Ash	0.64	0.0221
Ash on Extract	0.26	

Wash Solvent

	lbs/Hr	%
Total	0	
Extract		
Solvent		
Solids		
Ash		
Ash on Extract		

F-B331 Underflow

	lbs/Hr	%
Total	2159	
Extract	149	6.9
Solvent	1604	74.3
Solids	406	18.8
Ash	101	4.7
Ash on Extract	40	

F-B330

Temp. 498 °F
δ/μ 0.64
V_0 0.29 in/min
G_{us} 33.9 lb/hr ft²
W/F 0

Sec. Overflow to F-1301

	lbs/Hr	%
Total	940	
Extract	39	4.2
Solvent	699	74.4
Solids	201	21.4
Ash	44	4.64
Ash on Extract	47.5	

Underflow to 800

	lbs/Hr	%
Total	1470	
Extract	37	2.54
Solvent	1007	68.5
Solids	426	29.0
Ash	139	9.43
Ash on Extract	78.8	

Performance – Recoveries

		F-B331	F-B330	Overall
Extract,	%F	93	26	97
	Theo.			
Solids,	U/F	94	105	157
	Theo.			
Ash,	U/F	105	137	210
	Theo.			

Material Balance

	F-B331	F-B330	Overall
Total	109	112	115
Extract	150	51	122
Solvent	108	106	110
Solids	98	154	150
Ash	106	181	191

QUARTERLY REPORT

1.0 OBJECTIVES AND SCOPE OF WORK

1.1 OBJECTIVES

1.1.1 The objectives of this contract are:

a. Reactivate, renovate, and convert the Cresap Pilot Plant for production of low-sulfur fuel oil. Demonstrate by integrated steady state operations a coal liquefaction process consisting of proven reliable components to achieve coal liquefaction and desulfurization.

b. Provide the plant capability for component and equipment testing. Develop and/or test equipment components which may be required for use in any or all processes for converting coal into a clean liquid fuel suitable for electric power generation and other industries.

c. Develop operating parameters and techniques to achieve improved onstream efficiency of the facility, and provide equipment and component test data in conjunction with normal plant operating activities.

1.1.2 This contract provides for the redesign, refurbishment, operation, maintenance and security of the facility to accomplish these objectives.

1.2 SCOPE OF WORK

The scope of work consists of three major tasks: refurbishment of the original facility, operation of the refurbished facility, and development of a test program to be performed in conjunction with plant operations.

1.2.1 Refurbishment

The pilot plant has been refurbished and redesigned as necessary to accomplish the program objectives.

a. Plant Evaluation - An evaluation of the original facilities was carried out and recommendations were made as to the improvements required to bring the plant into a reliable operating condition.

b. Engineering Design - Engineering design recommendations were made for modifying the pilot plant to: 1) produce

1.2.1 b. (Continued)

low-sulfur fuel oil, and 2) include the capability to test equipment which may be required for use in a coal liquefaction plant. In order to obtain maximum utilization of onsite equipment, the process basis for the design was essentially the same as that for which the plant was originally designed, i.e., low pressure donor liquefaction (CSF) process.

c. Construction - Design or specify, procure and install all new machinery and equipment, instrumentation, electrical, piping and structural work, and specify and implement all refurbishing requirements of existing machinery, equipment, etc., as necessary to implement the design developed.

1.2.2 Operations

This task requires Fluor to maintain, and use its best efforts to operate the pilot plant in an integrated, steady state mode to 1) provide a coal liquefaction process as a base for equipment and process testing; 2) demonstrate low-sulfur, low-ash fuel oil production with the installed equipment and acquire the necessary process data for characterization of the CSF process; and 3) produce sufficient products for testing.

Correlations will be developed by Fluor to provide direction for the selection of operating conditions for a commercial plant design.

1.2.3 Test Program

This task involves the development of a test program to establish the reliability of components which are critical to coal liquefaction processes. The testing is to be done in an environment representative of coal liquefaction processes. The testing program will be run concurrently with the operation of the integrated CSF process. The components found to be reliable will define equipment which can be used in other coal liquefaction pilot and demonstration plants supported by the Federal Government at other sites. The size of components developed and tested at Cresap will be made to match (where feasible) the size of existing equipment and facilities at Cresap in the interest of minimizing costs and the time required for modifications.

1.2.3 (Continued)

The test program will include the following tasks:

1.2.3.1 Heaters and Heat Exchangers

Monitor and characterize the performance of plant heat exchange equipment, including:

a. Heat transfer coefficients as a function of process parameters, such as temperature and flow rate.

b. Observation of tube fouling and coke formation as related to equipment operating conditions.

c. Monitoring of erosion and corrosion of tube material.

1.2.3.2 Corrosion and Erosion

Evaluate the corrosion and erosion effects of Unit 300 process streams on alternative materials of construction in coal liquefaction service through the use of test coupons and corrisometer probes. Of particular interest will be slurry streams. Stream variables will include: temperature, pressure, stream linear velocity, solids content, particle characterization.

1.2.3.3 Valves and Piping

a. Evaluate hand valve configurations (including plug tips, and seats) and trim materials that will maintain their dimensional integrity and design performance characteristics in coal liquefaction slurry service.

b. Evaluate dependability and maintainability of seals, joints, flanges, rings and welds at process temperatures and pressures.

c. Evaluate piping configurations to handle coal slurries, including pipe and fitting life as a function of pipe wall thickness and slurry velocity and other stream properties.

1.2.3.4 Pumps

Evaluate pump and seal design configurations and materials for use in coal liquefaction processes, with special emphasis on performance with coal slurries.

a. Reciprocating Pumps - Evaluate types, design, lubrication, and cooling techniques of packing. Candidates for consideration are: Teflon-filled braided asbestos with Teflon fillers between rings; and metallic, non-adjustable lip-type packing. Evaluate plunger coatings, such as tungsten carbide, ceramics and nickel-chrome alloys. Evaluate alternative pump valve designs (ball and disc types) and materials (such as delrin, tungsten carbide, and heat treated stainless steel).

b. Centrifugal Pumps - Evaluate hard surface coatings for cases and impellers to improve wear resistance when handling coal slurries. Coatings to be evaluated include plasma-sprayed alumina-titania, tungsten carbide, stellite, and cobalt-based alloy. Evaluations shall also include solid stellite and solid martensitic white iron impellers. Evaluate mechanical seal configurations. Modified designs including the use of internal wear plates shall be considered.

1.2.3.5 Instrumentation

Evaluate flow metering techniques for use in hot coal slurries. Types to be evaluated include orifice plates, venturi tubes, target flow meters.

1.2.3.6 Control and Letdown Valves

Evaluate control and letdown valve configurations and trim materials that will maintain their dimensional integrity and design performance in coal liquefaction service.

a. Control Valves - Evaluate performance of angle and globe types, and the eccentric rotating plug type, with various plug and seat materials, including tungsten carbide, hardened stainless steel, and stellite.

1.2.3.6 (Continued)

b. High Pressure Letdown Valves - Evaluate performance of an angle valve in Unit 600 with various trim such as tungsten carbide, boron carbide and alumina-ceramic. Evaluate impact of expansion chambers located downstream of the letdown valve for energy absorption during coal slurry discharge from high to low pressure.

1.2.3.7 High Pressure/High Temperature Pressure Vessel End Closures and Flanged Connections

Monitor the operational capability of the high pressure/high temperature hydrogenation reactor closures and flange assemblies.

1.2.3.8 Vessel Design

Monitor performance of vessels with regard to possible design changes for slurry service. Of interest are the erosive effects of coal liquefaction process streams, particularly at vessel inlets, outlets, and nozzles.

1.2.3.9 Liquid/Solids Separation - Nonmechanical

Evaluate the applicability of solvent deashing as a viable liquid/solid separation technique, including both laboratory studies and field testing. The field testing will include a solvent deashing unit to be designed, built, and tested at the Cresap Test Facility on coal liquefaction products to determine its commercial feasibility as a liquid/solid separation technique.

1.2.3.10 Liquid/Solids Separation Mechanical

A program will be developed including selection and installation of equipment for the future evaluation of improved filters and filter techniques for removing ash and unreacted coal from liquefaction reactor effluent at conditions up to 650°F and 150 psig. Filters to be considered are: 1) rotary drum, 2) horizontal leaf, and 3) crossflow tube.

1.2.3.11 Bearing Design and Metallurgy in Motive Equipment

Evaluate improved bearing designs and modifications for rotating equipment, with special emphasis on the existing high temperature extractor/mixer. A major emphasis will be on the development and proper use of mechanical seals to permit the use of external bearings.

1.2.3.12 Process Definition and Development

Process monitoring, definition and development will be carried on concurrently with the operation/testing phase of the project. Specific tasks are:

a. Characterization of the CSF process in an integrated steady state operation. Included will be the development of information required for a commercial design. Correlations will be required to describe the various process units. Of particular interest are:

   1. Coal extraction

   2. Liquid/solids separation

   3. Hydrogenation of extract

   4. Low temperature carbonization of hydroclone or solvent deashing underflow.

b. Process improvements will be implemented as required to maintain reliable operations. Included will be:

   1. Improvements in component and equipment design and/or arrangement.

   2. Modifications to minimize equipment problems by avoiding components that are unreliable.

2.0 SUMMARY OF PROGRESS TO DATE

2.1 INTRODUCTION

This Quarterly Progress Report covers work performed during October, November, and December, 1977.

2.1 (Continued)

As of the beginning of this reporting period, all work associated with the task of refurbishing was completed, and care, custody, and control of the plant was turned over to the Liquefied Coal Development Corporation (LCDC).

The principal thrust of the project is now directed towards operations, with emphasis on achieving fully integrated plant operations.

The test program is being reassessed in light of the DOE directive to emphasize operations, and revision of the Statement of Work is in preparation.

2.2 TEST PROGRAM

A meeting with the DOE Program Manager was held on November 14 and 15 at the Fluor offices in Irvine, California. He noted that the highest priorities have to be given to the tasks of starting up and operating the plant and thus the major efforts have to be concentrated on those tasks. However, Cresap is still regarded as a test facility. With the time and money that went into researching, designing, and installing the test equipment DOE believes that a substantial amount of data will be obtained from the test program. However, instead of a highly sophisticated data gathering and reporting program, the bulk of the information will have to come from the maintenance records as well as the trained observations of LCDC personnel. Now that the resources required to start up and operate the plant are known it is necessary to review the remainder of the program to maximize the amount of test program data that is attainable within the funds now available to the project.

This meeting was followed up with one in December between LCDC and Fluor E&C personnel, to discuss the impact of the new philosophy on each individual test element. This discussion is presented in detail under Section 3.0.

2.3 OPERATIONS

The initial attempt to process coal (Run 001) on October 11, 1977, was interrupted one hour into the run by a leak at the inlet to the slurry preheater. During the subsequent emergency shutdown, the preheater coil plugged with coke. The middle section of the preheater was replaced with the spare coil. Other operating delays resulted from two failures of the extractor agitator shaft seals prior to the introduction of coal.

On October 19, 1977, a run of 20 hours involving all units except Units 600 and 700 was achieved (Run 002). Approximately four tons

2.3 (Continued)

of coal were processed at 9:1 solvent-to-coal ratio. Extraction depth was limited by low temperatures in the extractor. Line blockages in the Solvent Recovery Section forced a halt to the run. All major process equipment, including the new settlers, performed satisfactorily and the front-end of the plant demonstrated a good degree of operability for a first full run. Detailed data on this run is presented in Appendix A.

Run 003 during which coal was fed for 144 hours was completed over the period of November 15-21. Thirty-two tons of coal were processed. The average coal feed concentration was 11.5 percent MAF; the average extraction depth was 62 percent. The overflow from the solids separation section typically contained less than 0.5 percent solids; the ash content in extract produced was approximately 0.3 percent. Extract recovery was above 80 percent; solvent recovery was 97 percent. The revised extract granulation loop functioned well. Intermittent blockages in the granulated extract slurry system made it necessary to use the extract drumming facility which functioned quite well during this run. Detailed data on this run is presented in Appendix A.

Run 004 began on December 16 with an 11.5 percent MAF coal slurry batch which remained from Run 003. The slurry concentration was increased to 15 percent MAF by the third feed batch. Installation of an open impeller, high head pump in the granulator water loop appeared to have solved the difficulties previously encountered. Run 004 had to be terminated after 55 hours of operation on December 18, when the Extractor (D-A201) agitator shaft seals began leaking. This is discussed in more detail in Section 3.0. Solvent and nitrogen recirculation was maintained through the Hydrogenation Section (Unit 600) at 2700 psig and 225°F.

Pressure testing of the block valves in the hydrotreating section continued in November. All of the high pressure relief valves were checked and adjusted; they are now operational. The makeup and recycle hydrogen compressors were operated as required to supply nitrogen for testing the high pressure sections of the plant.

3.0 DETAILED DESCRIPTION OF TECHNICAL PROGRESS

3.1 TEST PROGRAM

The following are the status of the Test Program and the results of the November 14-15 Fluor/DOE discussion and the December 6-7 Fluor/CCDC discussion on the test program.

3.1.1 Solvent Deashing, Test 1000

Plant operation will be planned around solvent deashing as the only mode of liquid/solids separation. All equipment is installed and checked out.

3.1.2 Pumps, Test 1100

All test pumps are installed and all pretest base line measurements have been made. Further detailed physical measurement of pumps will be discontinued. Operation of all pumps will be reported on as a function of their operational and maintenance activity, using standard maintenance reports, ERDA Failure Reporting Forms, etc. Where alternate lining materials warrant evaluation, Fluor will be consulted by LCDC before any action is taken.

3.1.3 Heat Exchangers and Heaters, Tests 1200 and 1300

With the exception of the B-A501 Vacuum Distillation Preheater, all other heater and heat exchanger test installations are complete. Continue with heat transfer work where the data is gathered and processed by the PDP 11/45 computer. Cancel the Physical Properties S/C. Instrument readings on heater and heat exchanger performance are to be gathered only if a necessary function of plant operation or if automatically gathered by the computer.

3.1.4 Vessels, Test 1400

No further activity required. Vessels performance will be reported as part of normal operations and maintenance reports only.

3.1.5 Filtration, Test 1500

Defer activity. Filters to be mothballed.

3.1.6 Valves, Test 1600

a. Test valves installed, but no prestart-up measurements of valve geometry taken.

b. No work to be done with test valves until after integrated operation has been achieved.

c. In the interim, LCDC to define a revised test program.

3.1.6 (Continued)

d. The revised test program will be reviewed by Fluor. If the revised test program is not approved, then pending available resources, the test valves will be utilized in their current system configuration.

e. Until a decision is made with regard to the LCDC-proposed test program, the use of the test valves may be considered in areas where necessary to support operations.

3.1.7 Erosion/Hydraulics, Test 1650

The test loop is installed. However, LCDC recommended against the performance of this test. This is to be discussed further with DOE in January.

3.1.8 Flow Meters and Letdown Valves, Test 1700

a. Flow meters in Units 300 and 500, and globe-type hand valve in Unit 300 - No work to be done until after integrated operation has been achieved. At that time, test program to be carried out per Fluor recommendations, using "Priority - 1" trim.

b. Letdown Valve in Unit 200 - Valve 1751 V1 currently installed. This to be left in place until a need to change it is perceived. At that time it will be changed to Valve 1751 V4.

c. Letdown Valve in Unit 600 - Will install the Fisher valve upon its arrival in late December. It will be used in conjunction with the 3" x 72" expansion chamber. Backup will be with Valve 6006B (angle body). Valve 6006A (globe body) will be used only as a last resort.

3.1.9 Extractor Seals and Bearings, Test 1800

All additional test work to be cancelled. The Extractor is to be run with the installed Sealol single mechanical seals. The Durametallic double mechanical seals are available in the event they are needed as spares or in test activity, as LCDC sees fit. The Machine Surveillance System will be utilized without the internal mechanical vibration probe rod assemblies. The replacement eddy current probes (currently on order) will be installed upon arrival at the site.

3.1.10 Corrosion/Erosion, Test 1900

Proposed to limit efforts to Unit 300 only (most severe environment). Conduct full range of tests, including use of corrosion coupons, erosion sticks, and corrosometer probes. It will be necessary for LCDC to define piping modifications in Unit 300 to permit safe and timely access to test elements. The above program assumes that Grant Oil Tool Company will provide workable specimen holders, and that LCDC will prove their reliability. DOE approval will be requested after completion of LCDC recommendations.

3.2 OPERATIONS

3.2.1 Progress by Plant Sections

3.2.1.1 Section 100 - Coal Preparation

This section saw limited activity during October and November. At the conclusion of Run 003, sufficient coal was crushed and dried to bring inventory of prepared coal back to 50 tons in the F-A104 product storage bin.

In December, fifteen tons of coal were fed to Section 200 during Run 004. Coal was ground afterwards to bring the inventory in the F-A104 product storage bin to 70 tons. The stockpile contained an additional 500 tons of coal at the end of the year.

3.2.1.2 Section 200 - Coal Extraction

Coal slurry was processed for the first time on October 11 (Run 001). After one hour of operation at a slurry feed rate of 4,000 lb/hr, a gasket failed on the pressure transmitter, PT-208-1, requiring an immediate shutdown. Upon attempting to restart, the slurry preheater, B-A201, was found to be plugged. Attempts to dislodge the blockage failed; the middle coil in B-A201 was replaced. During the shutdown, inspection of the J-A202A and B slurry feed pumps revealed that the disc valves were pitted and did not seat properly. Ball checks with tungsten carbide seats have, therefore, been installed in place of the discs in both pumps. This will be discussed in detail in a forthcoming report covering pump performance.

3.2.1.2 (Continued)

A second run (Run 002) was begun on October 18 at the same conditions as Run 001. Twenty-one hours later coal feed was discontinued because of plugged lines in Section 500.

Repairs to the extractor agitator, L-B205, shaft seals were made twice this month. The details are discussed under Test 1800, below.

The on-off controller for the solvent heater, C-A204, failed after 18 hours of operation and was replaced with a silicon controlled rectifier proportional controller. Thermocouple leaks on C-A204 prevented the heater's use during the 20 hour run.

The tip of the plug in LCV-201-1, slurry letdown valve, broke off during operation, apparently because of a misalignment. The trim in the valve has been replaced.

Run 003 coal slurry feed began at 1600 hours on November 15 and ended November 21 at 1600 hours. Fresh Sure-Sol 180 was used as the donor solvent; Sure-Sol 180 returned from Section 500 was the recycle solvent. Total solvent feed including extractor seal flush solvent was 53,000 gallons during the run, and 32 tons of coal were processed.

The target feed slurry concentration was 13.6 percent coal (12.4 percent MAF coal). Including solvent added to the extractor for seal flushing, the slurry concentration was about 11.5 percent MAF coal. A feed rate of 6.8 gpm was maintained throughout most of the operation. The first batch of slurry was low in coal (8.8 percent); the second was high (14.5 percent). Disregarding these two, the average for the first day was 13.6 percent coal in the feed stream. For the first five days, most of the batch mixes were very close to the target value.

Extraction depth averaged 62 percent. Temperature profiles for the extractor at the time samples were taken were compared with depth of extraction analysis. No correlation between the temperatures and the extent of reaction is apparent from the limited data available.

3.2.1.2 (Continued)

The temperature at the B-A201 slurry preheater outlet initially was about 750°F. This was increased to 770-780°F to raise the extractor temperature; tube wall temperatures also increased. The highest tube wall temperature observed was 845°F. The same point had been 800°F when the outlet was 740°F. The pressure drop of the third tube pass rose during a three-day period from 15 psi to 35 psi indicating that coking was occurring in the preheater. This was further confirmed when the outlet temperature was reduced to 740°F and the tube skin remained at 820°F, 20°F higher than it had been at the beginning of the run. After shutdown, the preheater was decoked.

Throughout the run, four of the burners in B-A201 remained off as a temporary solution to prevent peaks in the tube wall temperature profile. Selas No. 72 burner tips were installed in B-A201 before Run 003 was begun and all of the leaking fuel gas ball valves were repaired.

At the feed point (Stage 10) of the extractor, D-A201, the average temperature was 775°F during Run 003. The average extractor temperature was 740°F; the outlet temperature was 725°F.

The L-B205 agitator was started at 50 rpm. During the second day of slurry operation, the speed was increased to 100 rpm where it remained for the duration of the run. The seals performed without incident.

The C-A204 spray solvent heater was out of service due to a leak; no spray solvent was used in the extractor during the run. Seal fittings around the thermocouple connections have since been installed in the tubesheet and backwelded to prevent process leakage. Previous leakage damaged electrical insulation around the heater elements.

During the 55-hour run of Run 004, the extraction depth was about 62 percent. A 15 percent coal slurry was fed at 6.5 gpm to the B-A201 slurry preheater. The run was discontinued when the bottom seal of the L-B205 extractor mixer

3.2.1.2 (Continued)

failed. Detailed data will be included in the next Quarterly Report (January-March 1978).

During Run 004, the process outlet temperature averaged 740°F at B-A201. The process inlet temperature at D-A201 was 725°F; the average extractor temperature was 718°F. The agitator, L-B205, was driven at 125 rpm.

Prior to Run 004, the piping around the J-A202A/B and J-B202 extractor charge pumps was modified to eliminate plugging in standby slurry lines and to improve pump operability.

During the solvent circulation period before the start of the run, a high-pressure drop was noted in the processing section between the charge pumps and the D-A201 extractor. The piston-type check valve at the extractor inlet was removed and the pressure drop returned to normal. No foreign material was found in the check valve. Plugging was experienced in VS-226, the three-way dump valve at the outlet from the extractor. After opening and closing the valve several times, normal flow was restored.

The maximum operating temperature for the C-A204 spray solvent heater was 500°F during Run 004. Above this temperature, solvent seeps into the terminal box causing electrical shorts. A new heating element is on order.

3.2.1.3 Section 300 - Solids Separation

Both integrated and closed loop circulating modes, at normal operating conditions, were conducted routinely during October. Operation on extract did not present any abnormal problems. During the period when extract slurry was being processed, solids concentrations in the settler overflow varied between 2.62 percent and 0.26 percent with a median value of 0.31 percent. Bottoms from the underflow receiver (F-B330) were disposed of routinely in the carbonizer.

During Run 003, separation efficiency generally was good. F-B331 settler overflow solids concentration consistently was below 0.5 percent and

3.2.1.3 (Continued)

ash content based on extract was about 0.3 percent. The solids content of underflow from the F-B330 underflow receiver (carbonizer feed) ranged from 20-30 percent. Overall extract recovery in the section was in excess of 80 percent.

Periods of near steady-state operation frequently were interrupted by blockages in the lines transferring the solids-rich stream from the bottom of the F-B330 underflow receiver to the carbonizer D-A801. When this occurred, it was necessary to recycle this stream to the vessels feeding the primary settler to keep the line out of F-B330 open. During this time, the feed to Section 300 increased in solids concentration.

The pluggage of lines and valves was a continual problem. Frequent hot solvent flushing of slurry valves was required to maintain flow. The original F-B330 underflow valves FCV-322A/B were replaced with valves having a larger 1/2 inch trim. Initially, plugging was experienced with the new valves due to solids buildup around the stem. This was corrected by cutting a groove in each valve stem guide to relieve the packing area as recommended by the manufacturer. After this modification, their performance in heavy slurry service was satisfactory.

Pluggage also was experienced in the lines returning the overflow from F-B330 to the primary settler. This stream passes through B-B305, the primary antisolvent heater. Flow was lost when the level in F-B330 dropped below the overflow weir due to an interruption of feed which, in turn, was caused by a plugged FCV-326, the primary settler underflow valve. Coking in B-B305 is suspected to have been the result.

The line used for the addition of wash solvent to the F-B331 settler underflow stream also plugged; no wash solvent was added during Run 003.

Typical of the many instrument-related problems are those associated with LT-306, the level transmitter in the primary feed tank F-B332. It

3.2.1.3 (Continued)

became inoperable fairly early in the run. On several occasions, this resulted in starving the J-A313 pumps.

Brief flow outages were taken to isolate the B-B305 primary antisolvent heater in order to clear a blockage which occurred during Run 003. When these efforts failed, temporary piping was run to tie in the B-B306 heater as a substitute. Continued use of these heaters will be necessary to help maintain temperatures in this section.

The addition of insulation reduced heat losses and allowed a modest increase in settler temperature to 530°F. CONOCO Coal Development Co. (CCDC) data indicate that 600°F is required to obtain the 0.2 percent solids concentration desired in the feed to the solvent recovery system.

During Run 004, samples taken for process control purposes showed that the settler overflow stream contained less than 0.5 percent ash on extract for most of the run.

Prior to and following the termination of Run 004, system temperatures were maintained by introducing hot flush solvent through the F-A301 wash stage feed tank and transferred the excess from the F-B308 overflow surge tank to Section 500. This allowed system repairs, including the seal at the L-B332 primary feed tank mixer, to be made without cooling the entire section. Gasket and bonnet leaks on lines and valves related to the F-B332 primary feed tank were addressed. The ram shaft of the bottom outlet Strahman valve showed damage similar to that found on the agitator shaft. Both shafts were machined and then coated with hard chrome.

High-pressure nitrogen purges were installed on level transmitters serving the following vessels: F-B332 primary feed tank, F-A301 wash stage feed tank, F-B331 settler and the F-B330 underflow receiver. Interruptions in the high-pressure inert gas supply formerly used to purge these transmitters resulted in false level indications due to the pluggage of the impulse lines with process solids.

3.2.1.3 (Continued)

Low ambient temperatures resulted in minor freeze-damage to several Section 300 pumps. Freezing has occurred in the vapor lines from all process vessels upstream of the C-A305 off-gas cooler. Insulation is being added to these vent lines.

Several mechanical seal failures occurred in November, notably in all three of the J-A313 primary hydroclone feed pumps and in the J-A314A wash hydroclone pump. Some of the failures were caused by the debris from the breakdown of the polypropylene components of the seal oil system filters. Damaged pump linings, both PTI-54 and tungsten carbide, also were found in the J-A313 pumps.

3.2.1.4 <u>Section 500 - Solvent Recovery</u>

The first coal addition (Run 001), which lasted one hour, resulted in the concentration of a heavy material in the E-A501 flash still loop. This material was drummed out when laboratory analysis indicated it was not a true extract, and the section was returned to the hot standby mode.

The second coal addition (Run 002) allowed unsteady state operation on extract for approximately 30 hours before plugging of lines and equipment forced a shutdown. The first pluggage occurred about ten hours into the run when the J-A502 flash still bottoms pump was lost due to having less than 15 percent solvent in the extract material being processed. The conditions of 4 psia and 475°F at the E-A501 overheads were too extreme for the boiling range material being produced in Section 200. Operations were continued at conditions of 4.7 psia and a maximum temperature of 443°F. The heavy ends were drummed out through the E-A501 bottoms sample station, due to plugging of the drum-out station, until granulation was attempted. Efforts to granulate the extract stream were unsuccessful, and were aborted after the granulator feed line and the granulator system plugged. A failure of the LT-501 ball float flash still level indicator occurred about the same time as the granulator

3.2.1.4 (Continued)

pluggage, allowing overfilling of E-A501, forcing the pilot plant to be shut down for cleanup of Section 500.

Thirty-one drums of flash still bottoms were generated during the coal operation, and a sufficient number of bottoms samples was taken to allow calibration of the laboratory liquid chromatograph.

Throughout Run 003, the solvent recovery section operated routinely at 505°F and 4 - 5 inches Hg vacuum. Ninety-seven percent of the solvent fed was recovered as product; the balance was drummed out with the extract.

None of the flow interruptions and plugged lines encountered during Run 002 were experienced. Although the modified granulation system worked well, the J-A503 pumps were unable to transport the extract slurry satisfactorily to the disposal pond due to an insufficient flow rate. In each of three different attempts only 3-1/2 hours of granulation time was logged before the F-A501 disposal tank, the transfer lines and the pumps plugged with granulated extract. This situation required the drumming of about 10 gallons of concentrated extract product every 20 to 30 minutes in order to maintain the level in the E-A501 flash still. Approximately 25 drums of extract plus the hot flush solvent used to clean the drum-out station after each use were accumulated daily.

Prior to the start of Run 003, the solvent recovery section was brought on-line to rerun the contents of the slop tanks F-A1107A/B and to control the inventories in Section 300. Similar operations followed the conclusion of the run.

Earlier, a nondestructive examination (NDE) of the B-A501 flash still heater coil identified widespread defects to an extent which precluded repairs. A new Type 321 stainless steel coil was installed. Temporary jumper lines were installed to allow the use of the B-A603 start-up oil heater as the flash still heater until the new coil installation was complete.

3.2.1.4 (Continued)

The increase in the extract slurry velocity provided by the larger granulator pump installed at J-A503A was sufficient to transport granulated extract to the storage pond as required. Although further improvements to the control of extract flow to the extract granulator syphon G-A501 are needed, Run 004 was of sufficient duration to demonstrate the operability of the extract disposal system.

Solvent fed from Section 300 was distilled as required. The accumulated material in the F-A1107A/B slop tanks was also worked off in order to recover the solvent fraction. The slop tanks are used as receivers for the organic phase from the API separator. Hot, closed-loop circulation was maintained in order to provide freeze protection. In spite of all efforts, however, freezing of transmitters and process lines was a problem on the colder days.

3.2.1.5 Section 600 - Extract Hydrogenation

Leak testing at 600 psig and repair of the Conval valves continued. The Conval valve leaks were caused by damaged seats, defective packing, and damaged machined parts. Initial repair work was defined as:

1. Seat refinishing: 18 valves

2. Repacking: 28 valves and

3. Replacement of parts: 7 valves

General repair procedures were reviewed with the Conval representative. Repair work consisted of repacking the stems with Garfite 100 (top and bottom rings) plus valve seat lapping.

Operation of the JC-A601A hydrogen recycle compressor, using nitrogen, was carried out by all shifts at 3000 psig. The JC-A601B hydrogen recycle compressor experienced an internal knock during a test run. It required replacement of the rod needle bearings.

3.2.1.5 (Continued)

The section nuclear level devices were checked out by the service representative and made operational.

During November, all high pressure piping and vessels were flushed with condensate to remove extraneous gritty material. Following the flush, which appears to have been effective, nitrogen leak checking at 2000 psig and 3000 psig was conducted on all Conval and Rockwell-Edwards valves in critical services. Of the 42 Conval valves tested, 23 leaked; only three of the 40 Rockwell-Edwards valves which were tested leaked. Repairs to the more critical valves were made. A number of surplus Rockwell-Edwards valves suitable for some of the Conval services were located in stock. These may be installed at a later date if problems with the Conval valves continue.

The B-A602A/B reactor recycle heaters were decoked prior to NDE tests. No tube defects were identified during the visual and ultrasonic testing. The E-A601 absorber was hydroblasted, coated with a preservative and reassembled. A Viton weir seal was installed in the F-A602 high pressure separator and tested satisfactorily.

Shop testing of all Section 600 relief valves was completed and the relief valves were reinstalled.

Several subsystems were commissioned and placed into closed-loop nitrogen/solvent operation during December. The J-A607A/B extract feed pumps, the J-A611 seal oil pump, the J-A608C/D reactor recycle pumps and the J-A608B/X-DX lube oil pumps were operated to circulate warm solvent through the D-A601A/D reactors and through the B-A602A/B recycle heaters. The pilot burners were fired to maintain solvent temperature above freezing. The system pressure was maintained at 2700 psig with nitrogen supplied from Section 900C.

This loop was then expanded to include the F-A609 residue separator, the F-A612 residue letdown tank, the B-A704 vacuum column reboiler,

3.2.1.5 (Continued)

and the J-A704A/B vacuum column bottoms pumps. A temporary line was run from the J-A704A/B discharge to the F-A608A extract feed tanks to complete the expanded solvent loop.

Solvent flow through the absorber loop, including the J-A605 absorber oil recycle pump, the E-A601 absorber, the F-A606 absorber first stage flash tank and the F-A607 absorber second stage flash tank was also maintained against 2700 psig nitrogen pressure in the absorber.

Level, flow and pressure instruments were checked out in each loop. The reactor nuclear level and density detectors were also checked out. Although the J-A614A/B instrument purge oil pumps were commissioned, the purge oil froze at the lower ambient temperatures. Alternate purge fluids which freeze at -20°F or lower are being sought.

The B-A601 hydrogen heater coil was ultrasonically inspected and found to be free from defects. Plans were developed to install the missing burner tiles in B-A601 and B-A602A/B fired heaters. Attempts to seal leaks at the closures of the F-A605 entrainment separator and the E-A601 absorber were unsuccessful. Solid Teflon seal rings will be tried in January.

3.2.1.6 <u>Section 700 - Fractionation</u>

The fractionation system was maintained on warm, closed-loop circulation throughout the period. Low ambient temperatures resulted in minor operational difficulties when solvent froze in some of the impulse lines. The vacuum system was connected to Section 600 by temporary jumper lines to facilitate start-up efforts in the hydrotreating section. The B-A704 vacuum column reboiler provided some heat input to the recirculating solvent.

3.2.1.7 <u>Section 800 - Low Temperature Carbonization</u>

During the earlier part of Run 003, 5300 pounds of char was collected in drums for subsequent restarts of the carbonizer. The char make rate measured during this period was typically 125

3.2.1.7 (Continued)

lb/hr indicating 30 percent of the coal feed was being recovered as char. On an MAF basis, the char represented 25 percent of the feed coal.

Once the start-up inventory was established, further char make was slurried to the char pond. Operation was routine except for frequent feed nozzle pluggages which occurred 10 times during the 10 days of carbonizer operation. The pluggage generally was due to scaly, coke-like material.

The L-A807 primary venturi in the tar quench system was found to be severely eroded; it was replaced. The J-A804B solvent quench pump which had been running all the time the section was operational was found to have severe erosion damage to the case and impeller. The J-A701A stabilizer feed pump was moved in a temporary replacement until repairs are complete.
The closed-type impellers of the J-A803A/B char slurry pumps plugged with debris on initial start-up. After cleaning, the pumps worked well while handling a low concentration char slurry. At the higher char concentration occasioned by the intermittent removal of char from the carbonizer, both pumps plugged again. Subsequent char transport flow was marginally adequate.

The D-A801 carbonizer was operated for two days during Run 004. When the lower spray nozzle plugged, feed was switched to the upper spray nozzle. The upper nozzle discharges directly into the fluidized char bed; the lower nozzle empties into a higher-velocity riser pipe. The upper spray nozzle had no plugging problems; the in-line strainer upstream from the nozzle was cleaned once. After the run, a new upper feed nozzle with an enlarged orifice was installed. The lower nozzle will be changed when the bed level is below the nozzle entry point.

Operation of the char disposal system was hampered by plugging of the J-A803A/B closed-impeller char quench pumps with char agglomerates and pieces of refractory from the carbonizer. Temporary cone strainers in the pump suctions plugged rapidly and were removed. Parallel

3.2.1.7 (Continued)

basket-type strainers will be installed at the next opportunity.

3.2.1.8 Section 900C - Hydrogen Compression

The JC-A900A/B compressors were operated on nitrogen at 3300 psig. Insulation of the compressor drain systems was completed by the subcontractor. JC-A900B was used to supply high-pressure nitrogen to support Section 600 solvent circulation operations. One of the oil lubricator pumps on JC-A900B was replaced.

3.2.1.9 Section 900G - Hydrogen Generation

The F-B902 desuperheater was installed in place of F-A902, the old desuperheater. Leak-checking revealed that an incorrectly sized seal ring was installed by the supplier, Alpha Tank. This was corrected.

Observation of a color change in the amine solution led to the discovery that the process side of the C-A904 solution cooler was refurbished with baffles and tie-rods of a copper-bearing alloy. Plans to repipe the exchanger were abandoned when the copper and zinc content of the amine revealed that approximately 30 percent of the alloy had been dissolved. A replacement exchanger is on order.

The cast iron impeller in the J-A901A MEA solution pump was found to be badly corroded; a new impeller has been ordered.

The Performance Summary Report for the initial start-up and operation of Section 900G was issued.

3.2.1.10 Section 1000, 1100, and 1200 - Utilities, Offsites, and Environmental

These units continued to operate generally satisfactorily in support of operations. A major problem with phenol removal is discussed in Section 4.

3.3 TEST PROGRAM SUPPORT

3.3.1 Test 1100 - Pumps

3.3.1.1 Reciprocating

Modifications to the J-A202A/B and J-B202 extractor charge pumps were made during this period. These changes included the replacement of reduced port ball valves in the suction lines with full-ported valves, the substitution of quarter-turn bypass valves for the original gate valves, the elimination of deadlegs for standby slurry lines, the addition of several flush connections and the installation of added piping to permit slurry recirculation through the J-A202 suction header. The J-A202A pump functioned without incident during the entire 144 hours of Run 003.

3.3.1.2 Centrifugal Pumps

During November, 12 pump failures were experienced. Ten of these failures started from seal leaks. The failure of three PTI-54 coatings and one tungsten carbide coating were observed during subsequent seal repairs. These seal failures generally resulted from running the pumps dry (fallout of plugged level instruments in the suction vessels as discussed under Section 300 above), extract and/or solids fouling or the carryover of decomposed polypropylene from the filter failures in Section 1130. Six of the pump failures, primarily in the J-A313, J-A502, and J-A503 services, occurred while substantial quantities of coal derived materials were present in the process stream.

A list of failures chronologically by sections is noted below:

Date	Pump	Description
		(Section 300)
10/26/77	J-A313A	During disassembly for a seal failure, it was observed that the PTI-54 ceramic coating had experienced extensive chipping and cracking. The coating failure appeared to

3.3.1.2 (Continued)

Date	Pump	Description
		be due to a lack of bonding and/or thermal stress. The pump was replaced with the J-A313D pump. Consideration is being given to recoating J-A313 with hard chrome.
11/4/77	J-A313B	The mechanical seal failed and was replaced. Inspection revealed broken springs and a cracked carbon insert. As of this inspection, the PTI-54 ceramic coating is in satisfactory condition.
11/15/77	J-A313B	The mechanical seal failed. The drivepin and springs on the inboard seal were broken and the drive collar was slipping on the shaft sleeve. Some additional chipping of the PTI-54 ceramic coating occurred on the outer edge of the case wear ring. New seal components were installed, set screws were added to prevent the drive collar from slipping and the pump was returned to service.
		(Section 500)
11/17/77	J-A502B	The mechanical seal failed. This was handled by a routine repair and returned to service.
11/18/77	J-A503B	The mechanical seal failed due to fouling with extract. The entire pump was plugged with extract and during hydroblasting, the PTI-54 coating spalled off. Due to the pump's critical need, it was returned to service without making repairs to the liner.

3.3.1.2 (Continued)

Date	Pump	Description
11/18/77	J-A503B	The entire pump became plugged with extract. A recommendation for modifications to this service was prepared.
		(Section 800)
10/25/77	J-A503B	The mechanical seal failed. Inspection revealed that the uncoated pump had been severely eroded on the impeller and casing. The pump was sent out for repairs which included buildup with weld metal, remachining and coating with tungsten carbide. As a temporary measure, the spare J-A314D pump was installed in the service with a modified single seal.
11/15/77	J-A804B	Loss of capacity was caused by severe corrosion/erosion of the casing, impeller and stuffing box. This service was temporarily spared with the J-A701A pump while J-A804B is being rebuilt.
11/17/77	J-A806B	The J-A314D pump which was converted to a single seal configuration and substituted for J-A806B experienced a seal failure. The seal was rebuilt and returned to service.

During December, near zero weather caused the cooling water to freeze in the bearing housings of five pumps; the housings cracked. These failures occurred in the following services:

J-A302	Slurry Recycle Pump
J-B303A	Underflow Pump

3.3.1.2 (Continued)

J-A313A	Primary Feed Pump
J-A806A	Tar Quench Pump
J-A806B -- J-A314D	Pump in Tar Quench Pump Service

The materials were purchased to install valved connections on the bearing housings to facilitate their draining.

Five seal leaks were experienced in the following services:

J-A204A Spray Solvent Pump	Internals dirty
J-A313B Primary Feed Pump	Damaged springs and drive pin
J-A314C Feed Pump	Durafite packing leak; packing replaced with Viton-A O-rings
J-B303B Underflow Pump	Debris in seal area. Durafite packing leak; packing replaced with Viton-A O-rings
J-A806B Tar Quench Pump	Tar buildup separated seal faces

Problems with the Durafite packing are attributed to its lack of elasticity; once deformed by forces acting on the seal assembly, it will not recover adequately to maintain its seal. It is anticipated that the elastomeric properties of Viton-A will allow some relative movement without the loss of sealing integrity.

During its repair, the J-A313A primary feed pump was found to have damaged coatings on both the case (tungsten carbide) and the impeller (Stellite). The case and head were replaced with parts from J-A503A which were coated with PTI-54. A new Stellite-coated impeller was installed.

While repairing the J-A806A pump, it was found that the PTI-54 coating had failed around the

3.3.1.2 (Continued)

wear ring area resulting in erosion of the case and back cover. Similarly, the PTI-54 coating on the case of the J-A313B primary feed pump had flaked from large portions of the coated area. The Triballoy coating used on J-A314D, the pump which is substituted in the J-A806B tar quench service, showed no signs of deterioration. The coefficient of thermal expansion for Triballoy is closer to the base metal than are the expansion coefficients of either PTI-54 or tungsten carbide. A comparison of coefficients of expansion follows:

Selected Thermal Expansion Coefficients, in/in/F

Carbon steel	6.5×10^{-6}
11-13 stainless steel casting	6.5×10^{-6}
Triballoy 800	7.0×10^{-6}
Tungsten carbide	4.4×10^{-6}
PTI-54	4.2×10^{-6}

The new J-A503A extract disposal pump was used during Run 004. This larger capacity, higher head, open impeller pump successfully transferred the granulated extract and water slurry to the disposal pond.

3.3.2 Test 1300 - Fired Heaters

Monitoring of the B-A201 slurry preheater continued through all runs. The differential pressure transmitter for each pass was calibrated just prior to the start of Run 003. Normal pressure drops were 15 to 20 psi across each of the three zones. In Run 003, coking of the third section was suspected on the fifth day onstream when the zone pressure differential increased to 35 psi.

The evaluation of the inside heat transfer coefficient, h_i, for coal-free solvent in the B-A201 slurry preheater was begun in December. The initial work was limited to solvent because its physical properties are better known. Solvent operations before and after Run 003 were evaluated. A preliminary review of the data shows an order-of-magnitude drop in h_i occurs in the second zone after slurry has been

3.3.2 (Continued)

processed. The second zone corresponds to that part of the coil where the process temperatures are increasing from 450°F to 750°F. A report will be issued detailing the heater performance.

Temperature profiles on the B-A501 flash still heater at the beginning of the month indicated an abnormally high temperature peak approximately 50 feet (20 percent of coil length) downstream from the inlet. This problem was traced to radial cracks in the heater tube. An NDE inspection indicated that the cracks were numerous, widespread and impractical to repair. This coil was in service during Cresap I. Temporary jumper lines to the B-A603 start-up oil heater were made prior to Run 003. A new Type 321 stainless steel coil for B-A501 was received and installed in December, and B-A501 is back in operation.

3.3.3 <u>Test 1800 - Extractor</u>

The top seal of the extractor agitator, L-B205, failed on September 27. Inspection revealed that the 3/32-inch roll pin holding the stationary member sheared and lifted the stationary seat allowing leakage past the secondary packing. The bellows, seal faces, and packing (except the secondary) were in satisfactory condition. The flushing cup was slightly scored due to misalignment between the shaft and the seal gland. This resulted from a lack of perpendicularity between the seal gland and the shaft. The seal gland was machined to compensate for the misalignment and a hardened tool steel pin was installed to retain the stationary seat. These repairs did not stop the leakage. During the second disassembly, the carbon seal face was lapped to correct out-of-flatness, but this repair was also unsuccessful. Effective results were achieved by raising the rotary seal member 1/2 inch to increase the pressure on the seal faces. This corrected a design error by Sealol, who had assumed that thermal expansion would be sufficient to compress the seat against the stationary face. The top shaft seal on L-B205 performed satisfactorily during both runs with coal.

On October 21, while Section 200 was on standby circulating hot solvent, the bottom seal failed. Inspection revealed that the bellows had ruptured and that the 3/32-inch roll pin on the stationary face was sheared. In addition, the bottom bearing was damaged by solids contamination. The bellows, seal faces, packing, and bearing were replaced, and the rotary member was lowered 1/4 inch

3.3.3 (Continued)

to provide more pressure at the seal face, compensating for the fact that thermal growth was less than predicted.

Run 004 was terminated due to leakage at the L-B205 extractor agitator shaft seal. Disassembly revealed that the lower seal cavity contained a large amount of coal fines and extract. The spring-loaded inner packing apparently had lifted, allowing process material to pack into the bellows. Cracks in the bellows were diagnosed as having been caused by metal fatigue.

The failure of the lower shaft seal terminated Ron 004 at 1555 hours, December 18. A smaller leak was also observed at the top seal. Inspection showed the lower bearing to be bound up with coal fines and extract. These appear to have accumulated over a period of time as the lower seal began to leak. The seal itself was clogged with process materials and the bellows had numerous fatigue cracks. The cracks were probably due to abnormal forces generated as the solids became packed between the convolutions. Repairs to the top and bottom were completed as follows:

1. The shaft sleeves for both top and bottom seals, including the bottom packing sleeve, were refinished and coated with tungsten carbide.

2. At the top seal, a new carbon seal face was installed on the rotary member and the stationary seal face was replaced. Location of the rotary member was adjusted to reduce bellows compresssion by 0.125 inches. Previously, the seal had been raised 0.500 inches on Sealol's recommendation. This resulted in excessive face wear.

3. At the bottom seal, the seal face on the previously rebuilt rotary member was relapped and the stationary seal face was replaced.

3.3.4 <u>Test 1900 - Corrosion/Erosion</u>

A laboratory study on the effects of moisture in Sure-Sol 180 on carbon steel indicated that the corrosion rate with moisture present was three times greater than the control sample; i.e., 1.74 mpy versus 0.45 mpy. Clearly visible evidence of pitting and visible corrosion residue was found only in the sample with 1 percent water added.

3.3.4 (Continued)

Further evidence of an unknown corrosion problem related to Sure-Sol was found in the check valves to J-B202. Pitting of the tungsten carbide balls was indicative of crevice corrosion. Since Sure-Sol and perhaps some moisture were the only known materials in the pump, a laboratory study is being planned to diagnose the problem. In addition, the vendor will be contacted to discuss any treatment or processing which might have left trace quantities of materials which could have led to the corrosion problems observed.

The L-A807 primary venturi nozzle was photographed to document erosion characteristics of Type 316 stainless steel.

4.0 PROBLEMS AND SOLUTIONS

4.1 LEAKY HIGH-PRESSURE VALVES IN SECTION 600 - HYDROGENATION

Valve leaks with the Conval valves have been a continuing problem. The leaks have occurred in the packing, bonnet, and/or seat. The stem packing of all Conval valves will be replaced in January with an assembly which includes braided graphite filament rings in the first and fifth positions and die-formed Grafoil rings in the middle three positions.

4.2 SLOW HEAT-UP RATE AND LOW TEMPERATURE IN SOLIDS SEPARATION, SECTION 300

A continuing problem has been the slow heat-up rate and low temperature profiles in this section. The addition of insulation to the exposed manways, vessel heads, and flanges increased the overall temperature profile in the Solids Separation Section by only 10-15°F. The primary settler, F-B331, reached only 515°F, more than 100°F below design during October operation.

Continental Coal Development Corporation's recent bench-scale experience strongly suggests that settling temperatures must be increased toward 600°F as coal-derived liquids replace the start-up solvent.

Further insulation of bare line segments, flanges, and valves was carried out in November. A temperature of 530°F was attained in the F-B331 settler; this was an increase of 15°F over the highest temperature achieved during Run 002 despite the lower ambient temperatures.

4.2 (Continued)

Solids separation during Run 003 met objectives; however, higher temperatures may be required to effect the separation needed to meet product specifications.

Although some additional bare line segments, flanges, and valves were insulated prior to Run 004, the F-B331 settler temperature remained between 500°F and 515°F, 15°F lower than during Run 003. This reflects the lower ambient temperatures during December. A review is being made of the fired heater temperature limits to determine whether, from a process and a metallurgical standpoint, higher process temperatures are feasible at the front end of Section 300.

4.3 <u>RECURRING EXTRACTOR SEAL FAILURES, SECTION 200</u>

Both the top and bottom extractor agitator shaft seals failed during October. In each case, the roll pin which positions the stationary seal member sheared. The subsequent rotational movement damaged the bellows. Hardened pins were installed with the replacement seals. Also, the differential thermal expansion of the shaft and extractor shell was less than expected. The seal components were repositioned closer together to increase the loading across the seal faces at operating temperatures.

The failure of the lower shaft seal on the extractor agitator L-B205 terminated Run 004 at 1555 hours, December 18. A smaller leak was also observed at the top seal. The seals had seen a total of 10 days operation with coal in the extractor and a substantially longer period of solvent circulation.

Inspection showed the lower bearing to be bound up with coal fines and extract. These appear to have accumulated over a period of time as the lower seal began to leak. The seal itself was clogged with process materials and the bellows had numerous fatigue cracks. The cracks were probably due to abnormal forces generated as the solids became packed between the convolutions.

Whether this failure is attributable to design weaknesses as well as to a temporary loss of positive purge across the seal face, could not be determined. The seal is being rebuilt in its existing configuration for the next run. A pressurized backup supply of seal oil is planned. Improvements to the seal design are under consideration, and will be described in the next Quarterly Report (January-March 1978).

4.4 PUMP SEAL PROBLEMS

Five mechanical seal failures were experienced in December; there were 10 in November. The immediate causes of the failures include debris in the seal cavity, damaged springs, tar buildup on the seal faces, and packing leaks. The packing leaks may have resulted from the relatively low resiliency of the Durafite packing. Viton-A O-rings were substituted for the Durafite packing in two seals. There have been no O-ring failures to date. Further details are provided under Section 3.3.1.2.

4.5 FAILURE OF THE PUMP COATING MATERIALS

To date, PTI-54 coatings have failed in five of the 16 pumps so equipped. The failures took the form of chipping, cracking and eventual spalling of large areas of coating. These failures are thought to have resulted from poor bonding or thermal stresses due to differences between the coefficients of thermal expansion of the substrate and liner.

There have also been some problems with tungsten carbide and Stellite linings. See Section 3.3.1.2 for additional details.

A decrease in coating thickness, improved surface preparation and alternate coating methods such as the detonation gun application of tungsten carbide may result in a better bond between the liner and the base metal. Hard-chrome coating remains a potential candidate for lining pump cases. Consideration is currently being given to gas nitriding or chemical vapor deposition as methods to protect the more complex geometries of impellers. Pumps specifically designed for the services where coated off-the-shelf pumps are now being used also need to be considered.

4.6 EXTRACT HANDLING DIFFICULTIES IN SOLVENT RECOVERY, SECTION 500

The extract granulator proved inoperable during the first attempt to slurry extract to the storage pond. All other means for removing the bottoms product from Section 500 plugged with extract in less than a day. Part of the problem resulted from the extract-to-solvent ratio rising above 8:1. The extract-rich streams solidified at points where metal temperatures were not maintained due to deficiencies in insulation and/or tracing.

Insulation and tracing were supplemented where problems occurred. Some points of hot-flush solvent addition were modified to permit more efficient preuse heat-up and post-use purging of lines and equipment which process extract-rich streams. Operating conditions were modified, reflecting the limited information collected to date, to maintain the initial extract-to-solvent ratio at 8:1, or less, in the extract-rich streams.

4.6 (Continued)

The higher operating temperature plus piping modifications resulted in the routine circulation of extract to the granulation syphon and/or back to the flash still. No difficulties in preheating the lines with hot flush solvent were encountered and no line pluggages occurred. The granulation syphon, running with 150 psig steam and discharging beneath the water surface, appears to do an excellent job of granulating the extract material.

The difficulties encountered were the result of insufficient flow to allow the transporting of granulated material to the disposal pond. This low flow caused plugging of the disposal tank, the pumps and/or the transport line on each of three granulation attempts. Greater velocity to the pond was provided through the use of a larger pump and motor to increase the discharge pressure and flow rate. Also, the routing of the disposal lines was simplified to reduce flow resistance. See additional discussion in Section 3.2.1.4.

4.7 CARBONIZER FEED NOZZLE PLUGGING

During Run 003, plugging of feed spray nozzles occurred as often as twice a day, resulting in the carbonizer shutdown while the spray nozzle was removed, cleaned and replaced. Each of the existing feed spray nozzles, which are the same kind used during Consol operation, contains a tungsten carbide insert with dual 0.184-inch restrictions. The frequency of plugging was greatly reduced when new spray nozzles were installed with a 0.297-inch restriction. These were not available with carbide inserts so they were hard coated before installation. Some of the pluggage found (Teflon tape, gasket material, insulation) would plug even the new nozzles, so parallel coarse strainers were installed. A quick-removal nozzle assembly was designed.

4.8 SLURRY PIPING CONFIGURATION, SECTION 200

Coal accumulated and plugged the standby slurry lines on the inlets and outlets of the extractor charge pumps when they were not in service. The slurry piping around J-B202 and J-A202A/B was altered to reduce the potential for plugging. A recirculation line was tied into the end of the J-B202 suction manifold to keep it swept clear of settling solids. Also, the discharge lines from J-A202A/B which presently enter the B-A201 slurry preheater feed line from the bottom were rerun to enter the feed line from the top.

4.9 CONDENSATE SYSTEM PROBLEMS

Steam generating capacity has been limited, in part, by the inability to fully recover condensate for reuse as boiler feed water because of excessive pressures and temperatures in the condensate return system.

The problem is being addressed by the replacement of failed tracing with stainless steel and by a vigorous program to maintain trap operability.

4.10 ELEVATED PHENOL LEVEL IN THE INDUSTRIAL WASTE WATER EFFLUENT

From December 7, 1977 to the present, the apparent phenol content of industrial effluent has been above allowable limits.

Inspection of the three absorption columns in Section 1230 revealed no mechanical problems. Preliminary laboratory studies indicate a positive analytical interference produced by nonphenolic material which is not removed by the absorption resins. The following steps are being taken to combat the problem:

1. To reduce the apparent phenol content of the outfall, an oxidizer, such as hydrogen peroxide, will be added to the effluent from Section 1230.

2. With the advice of the vendors and SCD, every aspect of adsorbent regeneration will be reviewed; new resins will be ordered.

3. The sorptive capacity of the resins will be checked in the laboratory.

4. All plant materials will be screened for specific interference with the phenol analytical procedures.

5. The specific identity of the interfering specie will be sought.

EPA, Region III, will be kept informed of the steps being taken and the progress made to control the problem.

5.0 WORK FORECAST

5.1 TEST PROGRAM

The test program activities will be carried out in conjunction with plant operations and maintenance activities and in accordance with the discussions noted in Section 3.1.

5.2 OPERATIONS

5.2.1 Start of Integrated Operations

The start of integrated operations is currently scheduled for February 28. Much of January is scheduled for maintenance and modification of Section 600 - Extract Hydrogenation. Repairs will be made to the leaks in the heads of the absorber and the entrainment separator as well as the leaky Conval valves. Also, the attritor loops will be removed from the two hydrogenation reactors as requested by the licensor.

5.2.2 Run 005

Run 005 is scheduled to start in January. Baseline operating data of the solvent deashing section of the plant will be developed first at 15 percent coal, then at 25 percent coal. The latter will be at a coal feed rate of 12 tpd.

APPENDIX A

DETAILED DESCRIPTIONS OF RUNS 001, 002, AND 003

INTRODUCTION

This report covers the first three runs processing coal slurry in the first stage liquefaction - solids separation section of the Cresap Pilot Plant. The report covers the three-month period October 1, 1977 through December 31, 1977. During this time, the front-end process units - Extraction, Solids Separation and Low Temperature Carbonization - were commissioned and operated. Coal was crushed and dried in the Coal Feed Preparation Unit. Solvent and extract, suitable for feed to the second stage Extract Hydrogenation Unit, was processed in the Solvent Recovery Unit. All offsites and utility systems were operated during this period.

The project objective during this period was to conduct mechanical and operational shakedown of the first stage liquefaction - solids separation units. Operations were initiated at a 5-ton/day coal feed rate. The strategy was to start at 10 wt.% MAF coal and to increase the concentration in steps of 5 wt.% as experience was gained.

A considerable amount of operating time on hot solvent circulation in preparation for the addition of coal was achieved. A total of 165 hours on coal was accomplished during the three runs. The first two runs were halted due to mechanical problems. The third run lasted 144 hours before an orderly shutdown was ordered because of problems with the extract slurry disposal system.

FIRST STAGE LIQUEFACTION - SOLIDS SEPARATION UNIT OPERATION OF THE CRESAP PILOT PLANT

1. Operations During the Initial Test Runs

The first run processing coal slurry started at 9:00 a.m. on October 11, 1977. A gasket failure on the process side of a pressure transmitter near the inlet of the slurry preheater required an immediate shutdown after one hour on coal. During the shutdown and/or the ensuing repair period, the center section of the slurry preheater coil coked. Flow through the coil was not achieved on the restart. The plugged middle coil section was replaced with the spare.

The second run began on October 18, 1977, at 4:30 p.m. and lasted for 20 hours. Heavy extract material, concentrated in the solvent recovery section, was withdrawn into drums prior to achieving an extract concentration suitable for granulation. Heat losses in the withdrawal station resulted in extract solidification and pluggage. The drumming operation was then shifted to the flash still bottoms sampling station. Later, when granulation was attempted, the granulator system plugged with the extract material. This was caused by solidification occurring between the granulating steam syphon and the extract disposal tank. The extract material was also found to be of a heavy tar nature instead of a brittle, friable solid. This material was difficult, or impossible, to granulate under the best of conditions.

Run 003 began at 4:00 p.m. on November 15, 1977 and lasted 144 hours. Experience gained during the previous two runs provided the basis for some slight changes in operating strategy. The major emphasis was given to achieving better operability conditions including an increase in coal concentration to 11.5 wt.% MAF.

Swings in operating conditions prevented the accumulation of comprehensive sampling data. However, analysis of the available data does provide a basis for the general characterization of the operations. These are presented in the discussions of the various units.

The extract granulating system, which was modified after the second run, functioned satisfactorily during this run. Disposal of the granulated extract became the next problem as the extract/water slurry disposal piping plugged each time granulation was attempted. The accumulating extract was drummed out while attempts were made to solve this problem. Finally it became obvious that the solution would require replacement of the granulation pump. Combined with this were indications that line pluggages were developing in the drum out loop, so the run was terminated.

2. Extractor Unit Operations

The Extractor Unit was operated for 165 hours using Ireland Mine Coal. Feed slurry concentrations of nearly 14 wt.% coal have been pumped to approximately 800 psig in reciprocating pumps J-A202A, B. Coal slurries of 13 to 14 wt.% have been heated to 770 to 780°F in the direct fired B-A201 with pressure drop less than design value for the length of run. Operations with the Extractor D-A201 gave no evidence of rapid fouling or coke like deposits forming at the top head. Most of the operations to date have been conducted without the extractor top sprays in service. The discussions that follow focus on Run 003 during which the most experience and data were observed.

Average values of proximate and ultimate coal analysis representative of Run 003 are given in Table 1. The ash and sulfur content are 9.18 and 4.3 wt.%, respectively. The screen analysis of the coal is presented in Table 2.

During Run 003, 32 tons of ground coal were processed in 53,000 gallons of solvent. The initial feed batches were made up with 100 percent fresh Sure-Sol 180 solvent. As recycle solvent from the Solvent Recovery Section became available, fresh Sure-Sol 180 was used only for the donor solvent (0.4 donor solvent to coal ratio). Tables 3 and 4 present the analysis of Sure-Sol 180 and recycle solvent. The solvents generally ranged 450-550°F boiling range. The UOPK is 9.9 and 10 for the Sure-Sol 180 and recycle solvent, respectively. The low characterization factor is evidenced by the high percentage of naphthene compounds given in the gas chromatograph analysis of Table 4.

The target feed slurry concentration was 13.6 percent slurry (12.4 percent MAF coal). This mixture was pumped using the reciprocating feed pumps and passed through the slurry preheater before dilution in the extractor. Solvent added to the extractor for seal flushing lowers the overall slurry concentration to 11.5 percent MAF coal. A slurry feedrate of 6.8 gpm was maintained throughout most of the run. As shown in Tables 5 and 6, the target feed concentration was achieved except at the start and finish of the run. Toward the end of the run ground coal began to hang up in the coal feed chute to F-A203B, causing some inconsistencies in batch weights.

Depth of extraction, as a percent of extractable material, is calculated by the following relationship:

$$ED = \frac{(1 - Ac/AS)\ (100)}{1 - Ac}$$

ED = Extraction Depth, wt.% of the MAF coal.

Ac = Weight fraction of ash in the feed coal.

TABLE 1

TYPICAL COAL ANALYSIS - RUN 003
Ireland Mine - Moundsville, West Virginia

	Averages
Proximate Wt.%	
Moisture	1.1
Volatile Matter	41.7
Fixed Carbon	48.0
Ash	9.2
Ultimate Wt.%	
Hydrogen	5.0
Carbon	72.9
Nitrogen	1.2
Oxygen (Difference)	7.4
Sulfur	4.3
Ash	9.2
Moisture as Received Wt.%	2.1
Metals (in Ash), Wt.%, except as noted	
Sodium	0.24
Potassium	1.17
Cobalt	66 ppm
Nickel	0.04
Titanium	0.31
Aluminum	10.00
Calcium	1.41
Magnesium	0.49
Molybdenum	62 ppm
Iron	18.6
Silicon	18.3

TABLE 2

TYPICAL COAL SIEVE ANALYSIS

CUMULATIVE WT.% RETAINED ON TYLER SCREEN NUMBER

Mesh	
28	0.3 wt.%
48	4.5
100	15.8
200	47.1
325	90.7
-325	9.3
	100.0

TABLE 3

COMPARATIVE ANALYTICAL DATA

SURE-SOL 180 AND TYPICAL RECYCLE SOLVENT

Test	Sure-Sol 180	Typical Recycle Solvent
ASTM D-86		
Distillation, °F:		
Initial Boiling Point (Volume)	450	446
10%	470	472
20%	475	-
30%	478	481
40%	481	-
50%	486	490
60%	488	-
70%	494	502
80%	504	-
90%	520	529
End Point	548	559
Recovery %	99	92
Specific Gravity @ 77°F	0.991	0.979
Kinetic Viscosity @ 77°F	2.32 CST	-
Elemental Analysis, Wt.%		
Carbon	92.1	91.5
Hydrogen	7.9	7.8
Nitrogen	0.0	0.6
Oxygen (Difference)	0.0	0.0
Sulfur	0.0	0.1
Ash	0.0	0.0

TABLE 4

ANALYSES OF SURE-SOL 180 AND RECYCLE SOLVENT

Compounds*	Composition, Wt.% Sure-Sol 180	Recycle Solvent
(2,3) Benzofuran	0.03	0.01
Indane	0.04	0.02
Cis-Decalin	0.38	0.33
(1,2) Dihydronaphthalene	0.06	0.04
(1,2,3,4) Tetralin	0.09	0.09
Naphthalene	5.53	4.32
Quinoline	0.05	0.04
2 MN	24.30	23.02
1 MN	13.77	13.94
Biphenyl & (1+2) EN	3.53	4.13
(2,6) DMN	4.94	6.56
(1,7-2,7) OMN	2.43	3.10
(1,3+1,6) DHM	5.98	8.44
(1,4+1,5+2,3) DMN	3.28	4.11
(1,2+1,3) DMN	1.95	2.26
Acenaphthene	0.76	0.73
Dibenzofuran	1.25	1.61
(2,3,5) TMN	0.96	1.08
Fluorene	0.77	0.73
(9,10) Dihydroanthracene	0.42	0.24
(9,10) Dihydrophenanthrene	0.27	0.13
Anthracene	0.28	0.11
Carbazole	0.57	N/D
(9,10) Benzophenanthrene	0.25	0.24
Unknowns	28.11	24.72

*D - Di
E - Ethyl
M - Methyl
N - Naphthalene
O - Ortho
T - Tri
N/D - None Detected

TABLE 5

RUN 003 - TYPICAL OPERATING DATA FOR SECTION 200 - EXTRACTION

	11/15/77	11/16/77	11/17/77	11/18/77	11/19/77	11/20/77	11/21/77
Batch Tank, F-A203B							
Donor Solvent, lb.	1,480	3,940	3,620	3,990	4,200	4,240	2,050
Total Solvent, lb.	25,900	66,400	62,800	70,100	73,200	73,300	38,000
Total Coal, lb.	3,500	10,300	9,900	11,000	11,500	11,700	4,700
Feed Slurry Concentration							
By Weight, %	11.9	13.4	13.6	13.6	13.6	13.8	11.0
By Analysis (SP-8), %	13.8	14.2	10.5	11.9	12.3	16.7	7.3
Slurry Preheater Feed							
FR-223, gpm	7.0	6.2	6.8	6.7	6.8	6.8	6.2
Slurry Preheater, B-A201							
Inlet Temperature, F (TI-1-7)	106	78	73	62	58	62	74
Inlet Pressure, psig (PR-208-1)	490	470	475	470	480	490	490
Outlet Temperature, F (TRC-206)	660	770	765	780	770	760	735
Calculated Absorbed, Duty MM Btu/hr	0.9	1.13	1.06	1.23	1.27	1.23	0.90
Extractor, D-A201							
Outlet Temperature, F (TR-1-24)	625	680	685	693	710	710	690
Pressure, psig (PRC-201)	400	400	400	405	400	400	405
Agitator Speed, rpm	50	100	100	100	100	100	100
Extractor Effluent							
Flowrate, gpm	11.3	6.6-11.0	7.6-11.0	6.9-11.7	9.6	9.9	4.1-8.9
Extract (SP-11), %		8.0	4.8	3.0		10.6	7.9
Solids (SP-11), %	3.5	6.4	5.0	9.7	6.5	4.4	5.2

TABLE 6

SAMPLE POINT 8

ANALYSIS OF SLURRY REED TO B-A201 PREHEATER

Date	Time	THF Insolubles, %	Ash on THFI, %
11-15-77	2200	13.8	8.8
11-16-77	0230	18.6	8.8
11-16-77	0630	14.2	9.7
11-16-77	1730	11.0	7.1
11-16-77	2130	11.4	8.3
11-17-77	0545	10.5	8.8
11-17-77	1822	13.5	9.5
11-17-77	2130	13.1	13.0
11-18-77	0200	11.9	10.1
11-18-77	1755	14.6	8.8
11-18-77	2130	13.6	8.4
11-19-77	1400	12.3	8.3
11-19-77	1810	12.7	8.3
11-19-77	2230	12.7	8.6
11-20-77	0515	16.7	8.9
11-20-77	1350	12.1	10.6
11-20-77	2100	13.0	8.5
11-21-77	0130	14.7	7.4
11-21-77	0800	7.3	8.0
11-21-77	1325	12.2	7.6
Average		13	8.9

2. (Continued)

As = Weight fraction of ash in the THF insolubles of the extractor effluent (Table 5).

During Run 003, the average extraction depth was approximately 62 percent. Table 7 shows the variability of these values. Most of this variability is attributed to technique of sample gathering and sample preparation.

The average absorbed duty of the B-A201 slurry preheater was 1.1 to 1.25 MMBtu/hr during Run 003. Overall pressure drops of 70 to 100 psi were observed. The temperature of the B-A201 slurry preheater outlet initially was about 750°F. This was increased to 770 to 780°F to raise the extractor temperature, thereby compensating for heat losses in the system. The highest tube wall temperature observed was 845°F. This same tube wall temperature had been 800°F when the heater outlet was 740°F. Typical tube skin and process fluid temperature are shown in Figure 1.

The pressure drop of the third tube pass rose during the last three days from 15 psi to 35 psi indicating that some coking was occurring in the preheater. This was further confirmed when the outlet temperature was reduced to 740°F and the tube skin remained at 820°F, 20°F higher than it had been at the beginning of the run. The design allowable overall pressure drop of the preheater is 250 psi at 4,234 lb/hr slurry flow, corresponding to 160 psi at the Run 003 flow rate. The actual overall pressure drop was 70 psi at the beginning of the run and rose to 100 psi at the end, still well below the design value. After shutdown, the preheater was decoked.

Heat losses from the D-A201 Extractor were greater than anticipated, resulting in a temperature drop of 50 to 80°F across the Extractor as shown in Table 8. Insulation of the exposed nozzles and flanges has been recommended.

Due to the C-A204 spray solvent heater being out of service, no spray solvent was used at the extractor top. There was no indication of solids buildup in the vapor space due to the lack of spray solvent. The Extractor overhead system functioned very well during this run. The seal flush solvent, which is also heated by C-A204, was added cold. This also contributed to the lower extractor temperatures.

The L-B205 agitator was started at 50 rpm. During the second day of slurry operation, the speed was increased to 100 rpm where it remained for the duration of the run. No apparent difference in extraction was noted. The seals performed without incident. Other operating data summarized in Table 5 were used to calculate the overall extractor material balances shown in Figure 2. These values are averaged to represent typical 200 unit operations during this time frame.

TABLE 7

ANALYSIS OF D-A201 EXTRACTOR EFFLUENT

SAMPLE POINT 11

Date	Time	THF Insolubles, %	Ash on THFI, %	Calculated Depth of Extraction*	Solvent, %	Extract, %
11-15-77	2200	3.5	15.6	47.9		
11-16-77	0230	3.7	18.5	56.9		
11-16-77	0630	6.4	24.0	66.0	85.6	8.0
11-16-77	1130	0.3	25.8	73.2	98.6	1.1
11-16-77	2130	3.7	20.0	63.8		
11-17-77	0530	5.0	16.4	50.8	90.2	4.8
11-17-77	1400	2.1	15.9	51.0	>85.0	
11-18-77	1755	6.2	21.4	63.8		
11-18-77	1145	3.4	35.3	83.0		
11-19-77	1400	6.5	19.9	63.7		
11-19-77	2230	4.4	22.8	68.0		
11-20-77	0515	4.4	16.5	50.9	85.1	10.6
11-20-77	1350	4.9	24.5	50.9	89.5	5.6
11-20-77	2100	3.8	18.9	60.0	89.9	6.3
11-21-77	1500	5.2	21.0	65.1	86.9	7.9
11-21-77	1800	2.4	24.1	70.8	97.1	0.5

*Based on the equation, Depth of Extraction = $\frac{(1-A_c/A_s)100}{1-A_c}$

Where A_c = Weight fraction ash in coal feed
A_s = Weight fraction ash in THF insolubles of the extractor effluent

FIGURE 1
TYPICAL TEMPERATURE PROFILE FOR
B - A201 SLURRY PREHEATER

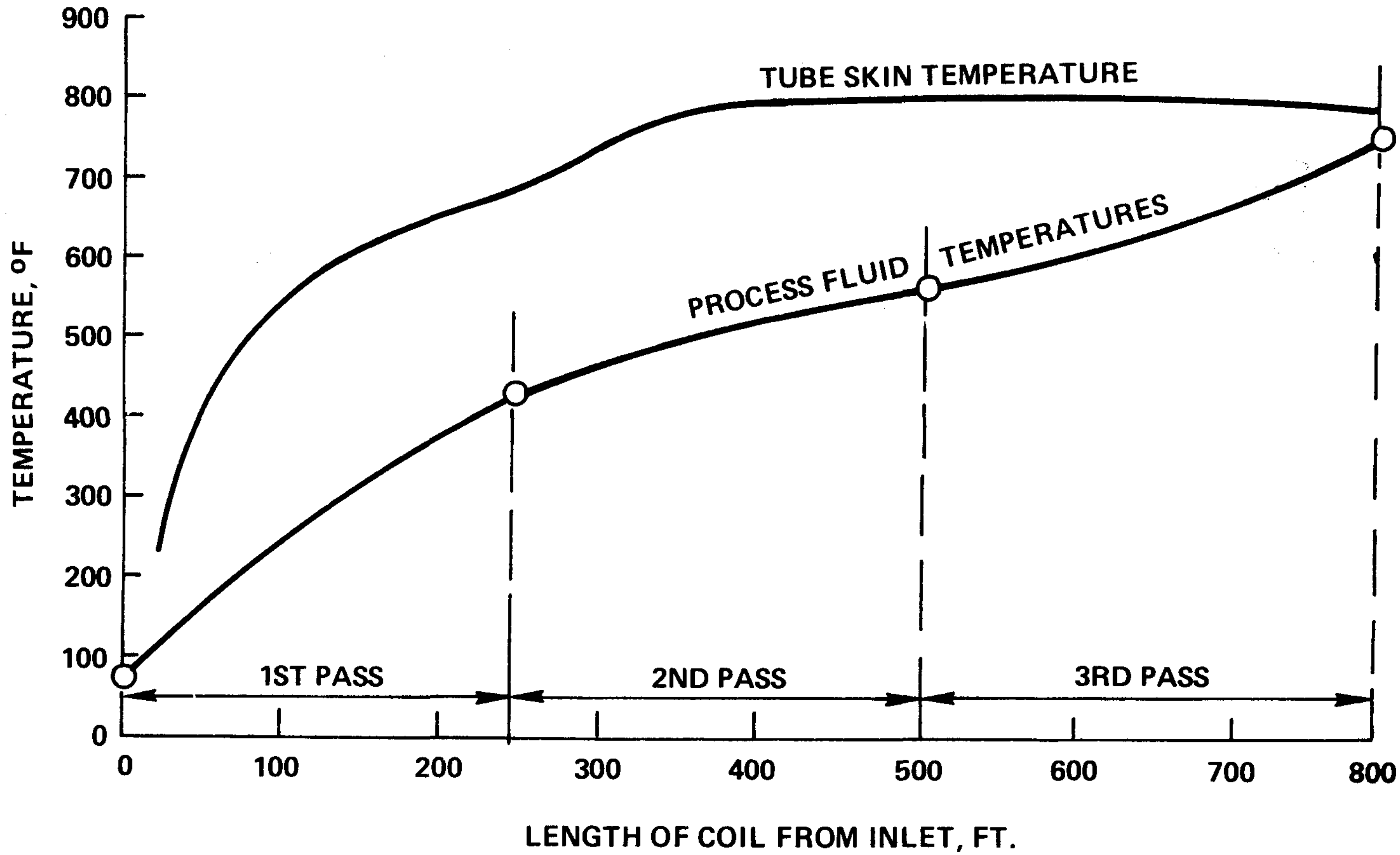

TABLE 8

D-A201 EXTRACTOR TEMPERATURES

		Temperature, F									
		TRC-206	TR-1-9	TR-1-8	TR-1-7	TR-1-6	TR-1-5	TR-1-4	TR-1-3	TR-1-2	TR-1-24
Date	Time	B-A201 Outlet	10th (Feed) Stage	8th Stage	7th Stage	5th Stage	4th Stage	3rd Stage	2nd Stage	Bottom Stage (1)	D-A201 Outlet
11-15-77	2200	740	720	715	715	710	705	705	620	615	670
11-16-77	0230	740	720	715	715	710	710	705	685	675	665
	0630	740	720	715	715	710	705	700	680	670	665
	1130	750	705	705	700	700	695	690	680	680	670
	2130	770	720	720	715	710	705	705	700	700	690
11-17-77	0530	745	700	700	700	695	695	680	675	675	670
	1400	780	735	730	725	720	715	715	705	700	710
	2130	755	730	730	725	735	720	715	710	705	700
11-18-77	0200	775	730	730	725	725	720	715	710	705	700
	1145	775	730	725	725	720	715	710	705	705	690
	1430	775	735	725	730	725	720	715	705	705	695
	1755	785	740	740	735	730	725	725	715	710	700
11-19-77	1400	775	750	745	740	735	735	730	725	720	710
	2230	765	745	745	740	735	730	730	720	720	705
11-20-77	0515	765	745	745	740	740	735	735	725	720	710
	1350	770	750	750	745	740	735	730	735	725	710
	2100	760	745		740	735	730	730	720	720	705
11-21-77	1500	730	715		710	710	705	705	695	695	[illegible]75
	1800	725	700		695	695	690	685	680	680	660

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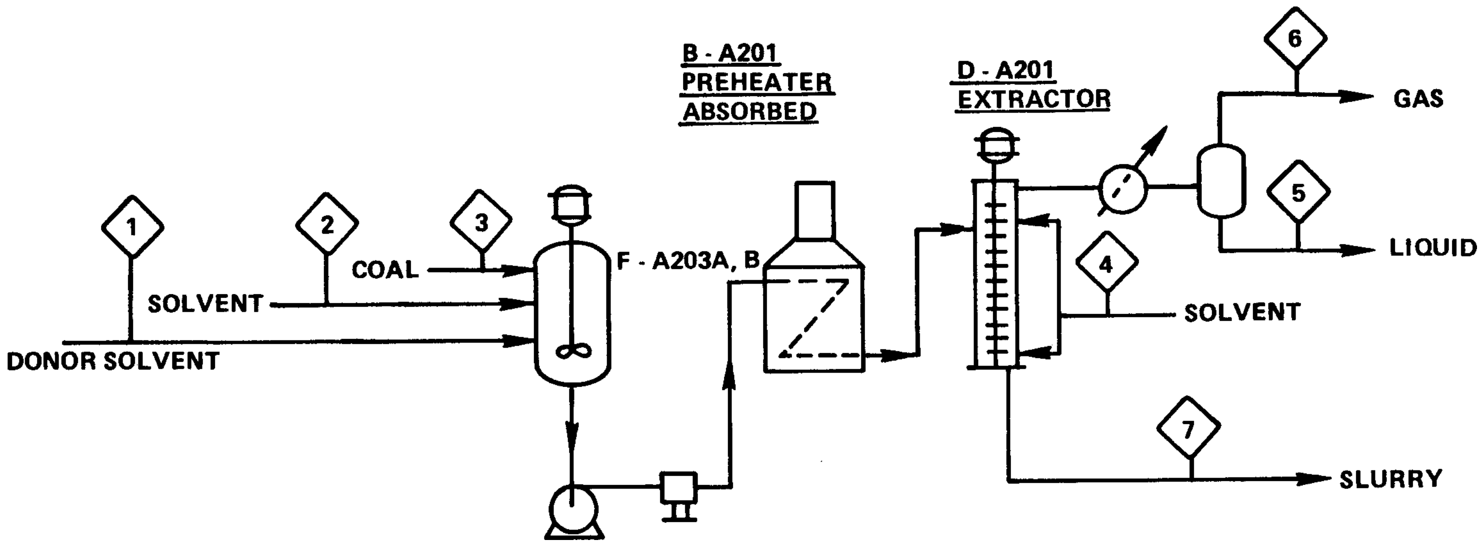
FIGURE 2
UNIT 200
COAL EXTRACTION SECTION



EXTRACTOR UNIT MATERIAL BALANCE

	1	2	3	4	COMBINED FEED	5	6	7
LIGHT GASES	—	—	—	—	—	—	3	—
WATER	—	—	—	—	—	20	—	—
DONOR SOLVENT	173		—	—	173	—	—	—
SOLVENT	—	2796	—	210	3006	72	—	3084
EXTRACT	—	—	—	—	—	—	—	270
COAL	—	—	467	—	467	—	—	—
SOLIDS	—	—	—	—	—	—	—	197
TOTAL, LBS/HR.	173	2796	467	210	3646	92	3	3551

2. (Continued)

Plans for Future Operations

- Increase the solids content in the feed to the Extractor up to 25 wt.% coal.
- Study Extractor effluent sampling procedure and revise as appropriate.
- To obtain additional data under steady operating conditions, thereby providing information for additional technical evaluations.

3. Solids Separation Unit

This report presents the first Cresap Pilot Plant data obtained using the CCDC Solvent Deashing process. The CCDC process was developed under Fluor Subcontract No. 448434-9-0002 as part of ERDA Contract No. EX-76-C-01-1517.

Based on operations at 6.3 T/D coal feed rate and 11.8 wt.% MAF coal, the solids separation unit conditions were less severe than design. The combined feed to the unit, presented in Table 9, was 5 to 7 wt.% solids for most of Run 003. Ash on solids ranged from 19 to 27 wt.%. The extract concentration ranged between 5-7 wt.%, with solvent at 87-90 percent.

Heat losses from the piping system dropped temperatures below the settler design range of 600 to 625°F. However, the basic operability to separate solids from the system was demonstrated. Analytical results of the settler overflow stream, Table 10, show that the average ash in the settler overflow feed to the solvent recovery section was 0.017 wt.%. Most of the time, this concentration was within the range of 0.002 to 0.016 wt.%. An ash concentration of less than 0.013 wt.% is required in this stream in order to meet the fuel oil target specification of 0.2 wt.%.

Insulation added to the settler system after Run 002 increased the settler operating temperature to 530°F. The analysis of the extract/solvent stream given in Table 10 indicated extract concentrations in the range of 5 to 8 wt.%. Several values above and below this range are due to system upsets.

The analysis of the settler underflow stream, Table 11, shows that solids were concentrated to 20 to 35 percent during most of the run. Ash content of solids generally was 22 to 30 percent. The extract in the underflow ranged from 2 to 6 wt.%. Design extract concentration in the primary settler was approximately 14 wt.%.

TABLE 9

ANALYSIS OF COMBINED FEED TO SOLIDS SEPARATION

Sample Point 13

Date	Time	THF Insolubles, %	Ash on THFI, %	Solvent, %	Extract %
11-16-77	1420	6.7	19.1	86.9	6.4
11-17-77	2300	6.2	27.5	87.2	6.6
11-18-77	2100	5.0	25.0	89.8	5.2
11-19-77	1930	10.3	22.2	83.0	6.7
11-20-77	2250	6.5	24.4	87.4	6.1

TABLE 10

ANALYSIS OF EXTRACT/SOLVENT PRODUCT

Sample Point 19

Date	Time	THF Insolubles, %	Ash on THFI, %	Total Ash, %	Extract %
11-15-77	2000	0.53	26.7		
11-16-77	0400	0.26	5.7	0.02	5.0
11-16-77	1420	0.83	1.4	0.01	
11-16-77	2200	0.42	6.5	0.03	
11-17-77	0530	0.48			
11-17-77	1354	0.54	7.2	0.04	7.1
11-17-77	2300	0.54	2.0	0.01	6.7
11-18-77	0530	0.28			
11-18-77	1140	0.31			
11-18-77	2030	0.37	1.6	0.01	7.1
11-19-77	1200	0.45			6.9
11-19-77	1930	0.86	1.9	0.02	7.5
11-20-77	0400	0.33	1.2	<0.01	
11-20-77	1000	0.29	3.5	0.01	
11-20-77	2130	0.64	3.5	<0.01	8.3
11-22-77	2050	0.37	4.8	0.02	
11-23-77	0430	0.56	10.5	0.03	1.8
11-23-77	0830	0.65	2.2	0.01	2.7
11-24-77	0845	0.65			

(Continued)

Additional solids separation was achieved in secondary settling in Underflow Receiver, F-B330. Overflow from F-B330, Table 12, shows that 2-4 wt.% extract was concentrated at this stage. The final extract concentration in the slurry feed to the carbonizer ranged from 1 to 4 percent which compares favorably with the design figure of 6.5 wt.%. This stream, given in Table 13, shows solids ranging from 20 to 30 wt.% during most of the run. The ash content of the solids varied from 25 to 35 wt.% during much of Run 003.

The overflow from the secondary settler, Table 12, shows extract concentrations from 2 to 4 wt.%. No wash solvent was used during this run. Solids content of the overflow from the Underflow Receiver was 19 to 27 wt.%. This stream was analyzed for a relatively constant 21 to 24 wt.% ash.

The performance data characterizing this operation are summarized in Table 14. Overall extract recovery was in excess of 90 percent. Solids rejection to the underflow was approximately 95 percent. The settler upflow velocity averaged approximately 0.3 in./min. which is close to the CCDC laboratory values. Design upflow velocity is 0.7 in./min. with an overall extract recovery of 97 percent.

Typical operating data representative of the solids separation section operation are presented in Table 15. These data have been averaged to present a composite overall material balance as given in Figure 3. This balance, showing 0.5 wt.% solids in the settler overflow and approximately 25 wt.% solids in the feed to the carbonization unit, is representative of Run 003 performance. These data demonstrate the overall operability of the CCDC technology although many upsets occurred which prevented the attainment of steady operations. The vessels in this section are very large in relation to the flow rates. Upsets in plant operations require considerable time to line out compositions to steady conditions. Frequent flush with hot solvent was required to clear plugs in lines and valves. Considerable experience was gained in freeing and preventing pluggage with slurry streams of 20 to 40 wt.% solids. The transfer line to the carbonizer was frequently plugged with solids. This caused an internal recycle within the unit which resulted in non-steady-state conditions. During the latter part of the run, the overflow line from the secondary settler was plugged, thereby reducing the overall separation efficiency. A pluggage in the wash solvent line prevented the use of wash solvent during the run.

Plans for Future Operations

Future activities in the solids separation unit include:

TABLE 11

ANALYSIS OF THE SETTLER UNDERFLOW

Sample Point 20

Date	Time	THF Insolubles, %	Ash on THFI, %	Solvent, %	Extract %
11-16-77	1420	26.5	30.5	70.4	3.2
11-17-77	2250	22.8	26.8	74.9	2.3
11-18-77	2100	23.9	24.7		
11-19-77	1200	30.9	22.4	66.4	2.7
11-19-77	1930	35.6	24.3		
11-20-77	2100	6.5	24.4	87.4	6.1
11-20-77	2250	18.8	25.0		

TABLE 12

ANALYSIS OF THE UNDERFLOW FROM THE UNDERFLOW RECEIVER

Sample Point 22

Date	Time	THF Insolubles, %	Ash on THFI, %	Solvent, %	Extract %
11-16-77	1420	19.1	23.0	80.9	<0.1
11-17-77	2250	21.1	24.1	75.7	3.3
11-18-77	2030	20.4	24.0	75.5	4.1
11-19-77	1930	27.4	20.7	72.6	2.1
11-20-77	2100	21.4	21.7	74.4	4.2

TABLE 13

ANALYSIS OF SLURRY TO THE CARBONIZER

Sample Point 16

Date	Time	THF Insolubles, %	Ash on THFI, %	Solvent, %	Extract %
11-15-77	2000	18.2	33.8		
11-16-77	0400	19.6	22.9		
11-16-77	1420	17.0	21.7		
11-16-77	2200	27.5	39.7		
11-17-77	0530	18.6	24.8	80.4	1.0
11-17-77	1354	24.0	27.7	72.0	4.1
11-17-77	2250	24.7	29.8	71.0	4.4
11-18-77	0530	12.9	36.9		
11-18-77	1140	29.8	42.9		
11-18-77	2030	25.7	30.9	72.2	2.1
11-19-77	1630	33.0	24.6	66.7	0.3
11-20-77	0400	27.2	27.6		
11-20-77	1000	29.2	42.5	71.8	0.0
11-20-77	2130	29.0	32.5	68.5	2.5
11-22-77	1500	22.8	35.9	72.0	3.2
11-22-77	2030	27.0	33.4	73.0	0.0
11-23-77	0430	25.0	21.8	75.0	0.0
11-23-77	0820	24.8	38.8	75.2	0.0
11-23-77	1130	26.6	28.7	73.4	0.0
11-23-77	1800	23.8	23.5	75.4	0.9

TABLE 14

SOLIDS SEPARATION PERFORMANCE SUMMARY

Settler:	
Temperature	510-530°F
Overflow/Underflow Ratio	1.3 - 1.4
Upflow Velocity	0.25-0.40 in./min
Solids Flux	9-17 lb/ft^2-hr
Extract Recovery	65-90%
Solids Rejection	93-102%
Overall:	
Extract Recovery	91-97%

TABLE 15

TYPICAL OPERATING DATA FOR SECTION 300 - SOLIDS SEPARATION

Date:	11/16/77	11/17/77	11/18/77	11/19/77	11/20/77
Primary Feed Tank, F-B332					
Temperature, F (TI-1-16)	632	643	644	650	644
Primary Slurry Heater, B-A302					
Outlet Temperature, F (TRC-302)	660	665	675	680	675
Wash Feed Tank, F-A301					
Temperature, F (TI-1-20)	544	552	576	580	571
Settler, F-B331					
Feed					
Flowrate					
FRC-325, gpm	17.1	14.5	12.9	11.4	11.1
Temperature, F (TI-1-42)	538	546	572	576	565
Solids (SP-18), %	10.4	11.6	7.6	13.5	9.3
Overflow					
Flowrate					
FRC-327, gpm	9.4	8.7	8.9	9.0	6.9
Solids (SP-19), %	0.8	0.5	0.4	0.9	0.6
Underflow					
Flowrate					
FRC-326, gpm	6.2	6.1	4.6	3.8	4.7
Temperature, F (TI-1-44)	519	517	508	530	542
Solids (SP-20), %	26.5	22.8	23.9	35.6	18.8
Underflow Receiver, F-B330					
Temperature, F (TI-1-36)	500	481	470	480	498
Flowrate to D-A801					
FRC-323, gpm	2.9	2.5	3.0	2.9	3.1
Solids (SP-16), %	17.0	24.6	25.7	33.0	29.0
Overflow Surge Tank, F-B308					
Temperature, F (TI-1-38)	500	499	496	516	518
Flowrate to B-A501					
FRC-309, gpm	10.1	10.7	10.5	10.1	10.4

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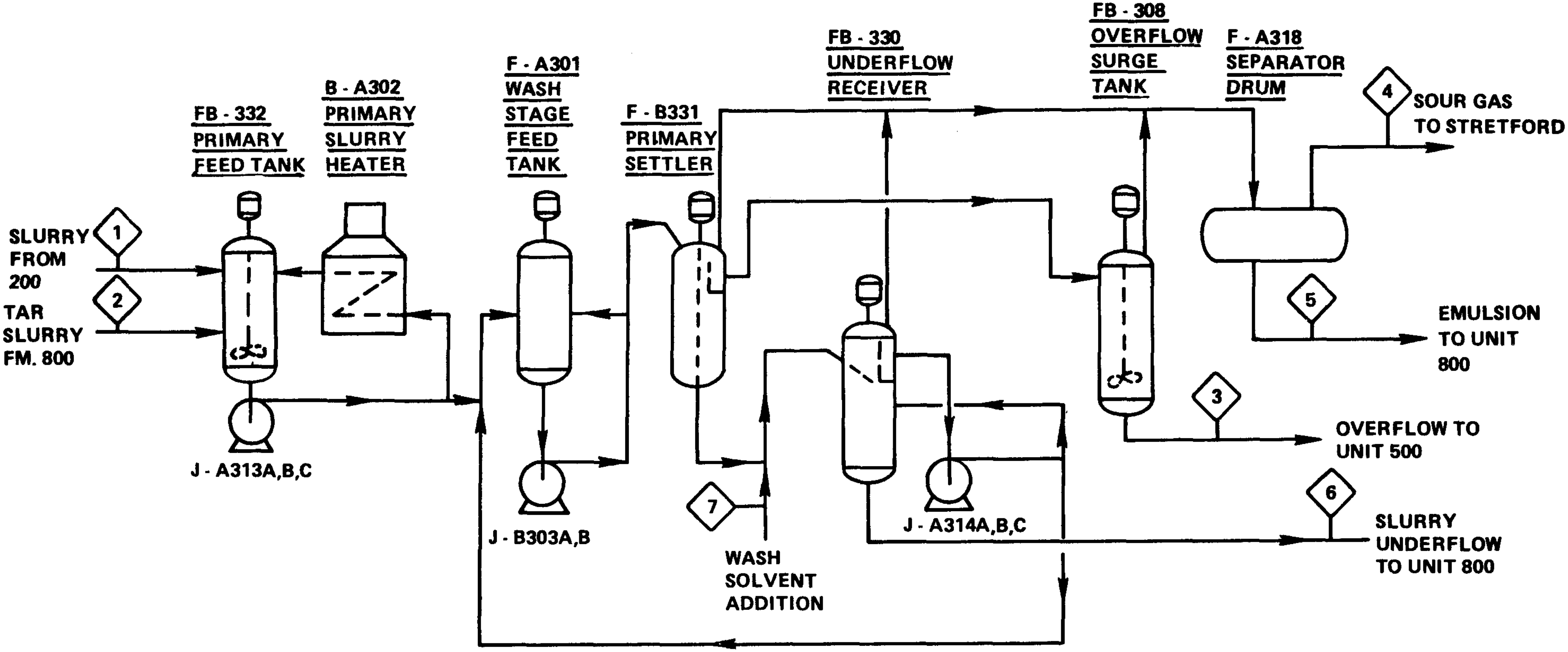
FIGURE 3
UNIT 300 - SOLIDS SEPARATION



SOLIDS SEPARATION UNIT MATERIAL BALANCE

	SLURRY FM. 200 (1)	TAR SLURRY FM. 800 (2)	COMBINED FEEDS	OVERFLOW TO 500 (3)	LIGHT GASES (4)	EMULSION TO 800 (5)	UNDERFLOW TO 800 (6)	WASH SOLVENT (7)
LIGHT GASES	—	—	—	—	10	—	—	—
WATER	—	—	—	—	—	—	—	—
SOLVENT	3084	1022	4106	3140	—	75	881	—
EXTRACT	270	41	311	243	—	—	68	—
SOLIDS	197	171	368	17	—	—	351	—
TOTAL, LBS/HR.	3551	1234	4785	3400	10	75	1300	—

3. (Continued)

- o Operation with wash solvent to the secondary settler.
- o Elimination of several piping restrictions, thereby providing more successful slurry transfer operations.
- o Attainment of higher settler temperatures.

4. Solvent Recovery Section

A continuous extract/solvent feed from the Solids Separations Section was processed during Run 003 without process line pluggages occurring. The feed rate was generally constant at about 3,400 lb/hr, with variable composition. This variation was caused by the upsets experienced in solids separation. These changes, as well as those caused by attempts to granulate extract, did not allow a true steady state operation to be reached. In spite of the non-steady conditions, it was determined that this process section performed well. As shown by the data in Table 16, 97 to 98 percent of the solvent fed was recovered as recycle solvent during the last five days of the run.

The extract concentration in the flash still bottoms stream was monitored frequently to prevent excess extract concentration. At the beginning of the run, material with a high solids level which had been accumulated from the previous runs was worked off as the extract from Unit 300 was concentrated. During the run, the quality of the concentrated extract stream was according to plan. The average extract concentration was 76 percent and the solids level was 0.4 percent. Toward the end of the run the solids level began to increase due to operational upsets in Unit 300.

Three attempts were made to operate the granulated extract disposal system. The portion of the system which granulates the extract performed well as a result of modifications made after the second run. The slurry disposal pump, however, did not provide a sufficient flow of water to convey the granulated extract to the disposal pond. Indications are that with the addition of a larger pump, this portion of the system will perform as desired.

Figure 4 shows the relationship between the solvent content in the concentrated extract and the overhead temperature of the flash still at operating conditions. There is only a 5°F range of temperature to maintain the desired 20 to 25 percent concentration in this stream. Other operating data are summarized in Table 17. These data define the key operating conditions and flows.

The performance data for the solvent recovery section have been averaged to obtain a representative material balance across the unit. This balance is presented in Figure 5 Solvent recovery was typically 97 to 98 wt.%.

TABLE 16

SOLVENT RECOVERY PERFORMANCE SUMMARY

11-17-77 to 11-21-77

Total Feed (FRC-501):	3200 to 3400 lb/hr; avg. ~3280 lb/hr
Extract in Feed:	6.8 to 8.4%; avg. 7.4%
Solids in Feed:	~0.5%
Lb/hr Extract & Solids:	~259 lb/hr
Total Solvent Feed:	3021 lb/hr
Avg. Conc. % Extract & Solids Drummed Out:	77%
Solvent Drummed Out:	77 lb/hr
Solvent Recovered:	2944 lb/hr
Make Solvent by FR-509:	3200 lb/hr
Allowance for Hot Flush & Purges:	150 lb/hr
Difference:	3050 lb/hr net
Solvent Closure:	104%
Avg. Recycle Solvent on 11/18/77:	IBP 452F EP 546F API 10.5
Avg. Column Reflux on 11/17/77:	IBP 331 EP 430 API 24.9

FIGURE 4
BOTTOMS COMPOSITION VS. FLASH
STILL OVERHEAD TEMPERATURE

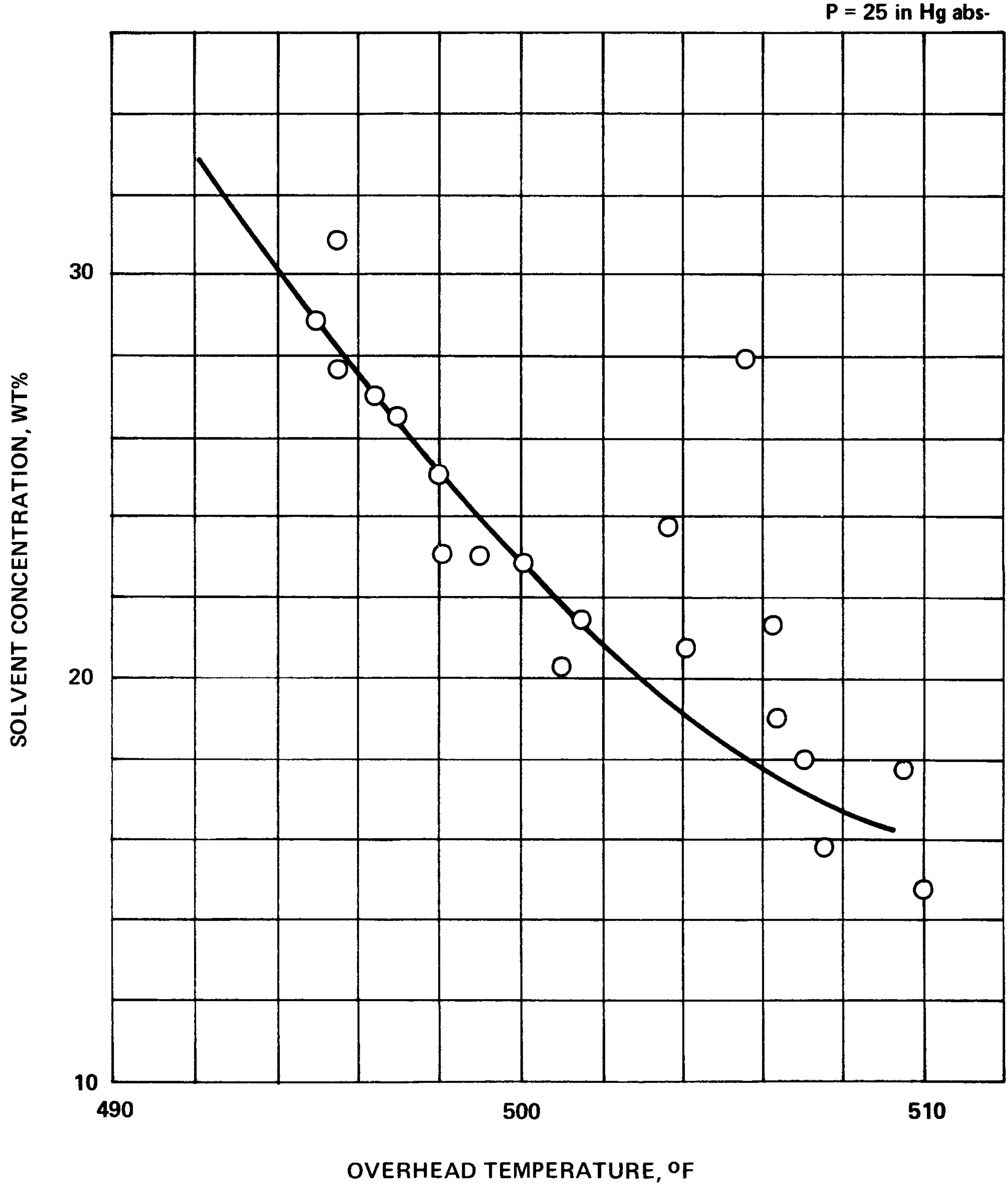


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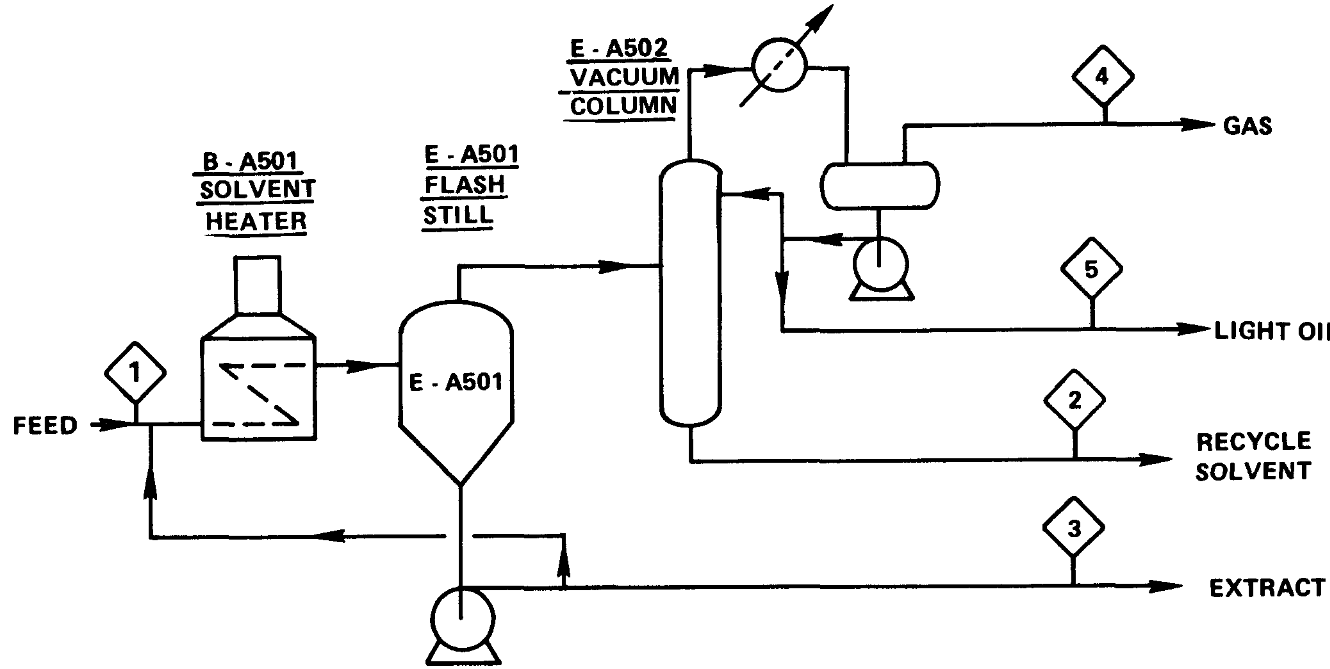
FIGURE 5
UNIT 500
SOLVENT RECOVERY



SOLVENT RECOVERY UNIT MATERIAL BALANCE

	1 300 UNIT OVERFLOW	2 RECYCLE SOLVENT	3 EXTRACT	4 LIGHT GASES	5 LIGHT OIL
LIGHT GASES	—	—	—	7	
WATER	—	—	—	—	—
SOLVENT	3140	3050	77	—	6
EXTRACT	243	—	258	—	—
SOLIDS	17	—	2	—	—
TOTAL, LBS/HR.	3400	3050	337	7	6

TABLE 17

TYPICAL OPERATING DATA FOR SECTION 500 - SOLVENT RECOVERY

Date, 1977	11/16	11/17	11/18	11/19	11/20	11/21
Flash Still Heater, B-A501						
Fresh Feed Flow Rate						
FRC-501, gpm	8.0	7.8	7.6	7.5	7.5	7.9
Outlet Temperature, F (TRC-501)	589	570	570	580	580	585
Recycle Flow Rate						
FRC-502, gpm	37.5	38.8	38.2	38.3	38.6	38.9
Flash Still, E-A501						
Inlet Pressure, psig (PRC-501)	30	32	32	32	32	32
Overhead Temperature, F (TI-3-7)	504	496	497	505	506	505
Overhead Pressure, in. Hg Abs (PR-502)	25.0	25.0	25.0	25.3	25.6	25.5
Bottoms						
Temperature, F (TI-56-15)	513	496	498	509	510	510
Extract & Solids (SP-34), %	82.0	81.3		81.0	76.9	79.7
Vacuum Column & Reboiler, E/B-A502						
Reboiler Temperature, F (TIC-502)	375	373	380	377	380	380
Overhead Temperature, F (TI-3-9)	359	356	350	348	344	344
Overhead Pressure, in. Hg Abs (PRC-503)	24	24	24	24	24	23
Column Pressure Drop, in. H_2O (PDR-508)	9	8	8	9	10	10
Recycle Solvent to F-A507						
Flow Rate						
FR-509, gpm	6.8	6.8	6.6	6.4	6.5	6.9

4. (Continued)

The analysis of this solvent is presented in Table 3. The extract analysis given in Table 18 shows that the average extract, 70 to 80 wt.%, contains a wide range of solids. It is expected that when steady operations are achieved, less than 1 wt.% solids can be achieved. Table 19 presents representative elemental analysis of the total extract stream.

Plans for Future Activities

- o Change out the slurry disposal pump to provide a higher discharge pressure.

5. Low Temperature Carbonization

The basic operability of the low temperature carbonization unit was demonstrated for an 8-day period during Run 003. The fluid bed carbonizer was operated on feed containing 24 to 33 wt.% solids and 2 to 6 wt.% extract. The feed from the 300 unit underflow varied between 2.4 to 4.6 gpm. There was no evidence of bed bogging. Stable bed dynamics were achieved at fluidizing gas velocities of 0.8 to 0.9 ft/sec. Bed temperatures were in the range of 830 to 870°F. The product tar slurry, generally 75 to 95 percent solvent, was returned to Unit 300.

The carbonizer feed control valve plugged frequently, requiring hot flush solvent to clear it. Additional pluggage occurred in the carbonizer feed spray nozzles. During periods of pluggage typically lasting for 10 to 30 minutes, the 300 unit was placed on total recycle to bypass the 800 unit. Interruptions of flow and surges of hot solvent did not cause problems with carbonizer operation, but did create variances in the solids concentration leaving the 300 unit.

After the 300 unit was no longer receiving slurry from the extractor, the carbonizer was operated until the underflow solids concentration dropped to below 10 percent, at which time the carbonizer was no longer receiving enough solids to maintain the bed level.

Typical operating data for Run 003 are presented in Table 20. These data were used in preparing the composite material balance presented in Figure 6. Carbonizer tar slurry, containing a wide range of solids varying from 10 to 30 wt.% as shown in Table 21, was returned to the solids separation unit. Char production of 125 lbs/hr was approximately 25 wt.% of the feed coal. Char production was approximately 3.0 times the feed coal ash content. The composition of the char is presented in Table 20. Char production rate was calculated over a one-day period at the beginning of the run. Emulsion and sour water streams were processed in batches, so flows were estimated from vessel level changes. Although flow rates of the slurry streams were unsteady due to control valve problems (especially toward the end of the run), the data logger was able to time-average a large number of readings to smooth out fluctuations.

TABLE 18

ANALYSIS OF EXTRACT PRODUCT - FLASH STILL BOTTOMS

Sample Point 34

Date	Time	THF Insolubles, %	Solvent, %	Extract, %
11-15-77	1800	18.6	14.8	66.6
11-16-77	0200	14.0	15.9	70.1
11-16-77	0530	7.4	20.3	72.3
11-17-77	0245	1.6	22.9	75.5
11-17-77	0645	0.3	26.5	73.2
11-17-77	1430	0.4	30.8	68.8
11-17-77	1715	0.4	18.7	80.8
11-18-77	0530	0.2	23.0	76.8
11-18-77	2015	0.3	25.0	74.7
11-18-77	2400	0.3	28.9	70.8
11-19-77	0830	1.0	20.8	78.2
11-19-77	1530	0.3	18.1	81.6
11-20-77	0325	0.7	21.4	77.9
11-20-77	1000	0.2	23.8	76.0
11-20-77	1300	0.2	19.0	80.8
11-20-77	1540	0.2	28.0	71.8
11-21-77	0245	0.9	21.5	77.7
11-21-77	0800	3.5	17.8	78.7
11-21-77	1430	5.4	22.8	71.8
11-22-77	0230	0.9	26.9	72.2
11-22-77	1100	3.5	38.3	58.2
11-22-77	1400	4.5	25.8	69.7
11-22-77	1610	1.3	23.0	75.7

TABLE 19

ELEMENTAL ANALYSIS OF EXTRACT PRODUCT FROM SECTION 500

H	6.4%	
C	87.2	
N	1.8	
O	2.9	(by difference)
S	1.7	
	100.0	

TABLE 20

TYPICAL OPERATING DATA FOR SECTION 800 - LOW TEMPERATURE CARBONIZATION

Date, 1977	11/16	11/17	11/18	11/19	11/20	11/21	11/22	11/23
Carbonizer, D-A801								
Temperature, F								
Above Bed (TR-2-10)	610	635	680	670	690	610	655	610
Top of Bed (TR-2-8)	830	835	870	865	845	765	805	740
Mid Bed (TR-2-5)	830	830	870	865	845	765	805	740
Above Grid (TR-1-21)	850	850	910	870	845	765	810	745
*Bed Level, % (LRC-802)	60	60	60	65	68	-	50	54
Bed Particle Size (SP-65)								
d_{50}		150	200					
-325 mesh		9%	9%					
Fluidizing Gas								
Flowrate, scfm	190	170	170	170	170	90	180	170
Velocity, ft/sec	0.8	0.8	0.85	0.85	0.85	0.5	0.9	0.8
Feed from F-B330								
FRC-323, gpm	~3	2.4	2.4	2.8	2.5	2.4	3.7	4.6
Solids (SP-16), %	27	24	24	25	33	2**	25	27
Tar and Solvent to F-B332								
Flowrate, FR-834, gpm	~2	2.2	2.5	2.6	2.2	3.8	2.0	-
Solids (SP-67), %		9.3	26	20	13	7	14	27
Light Oil to F-A710								
Flowrate, FR-804, gpm	0	0	0	0	0	0	0	0

*A 60% bed level corresponds to 5200 lbs of char.
**Diluted due to hot flush solvent.

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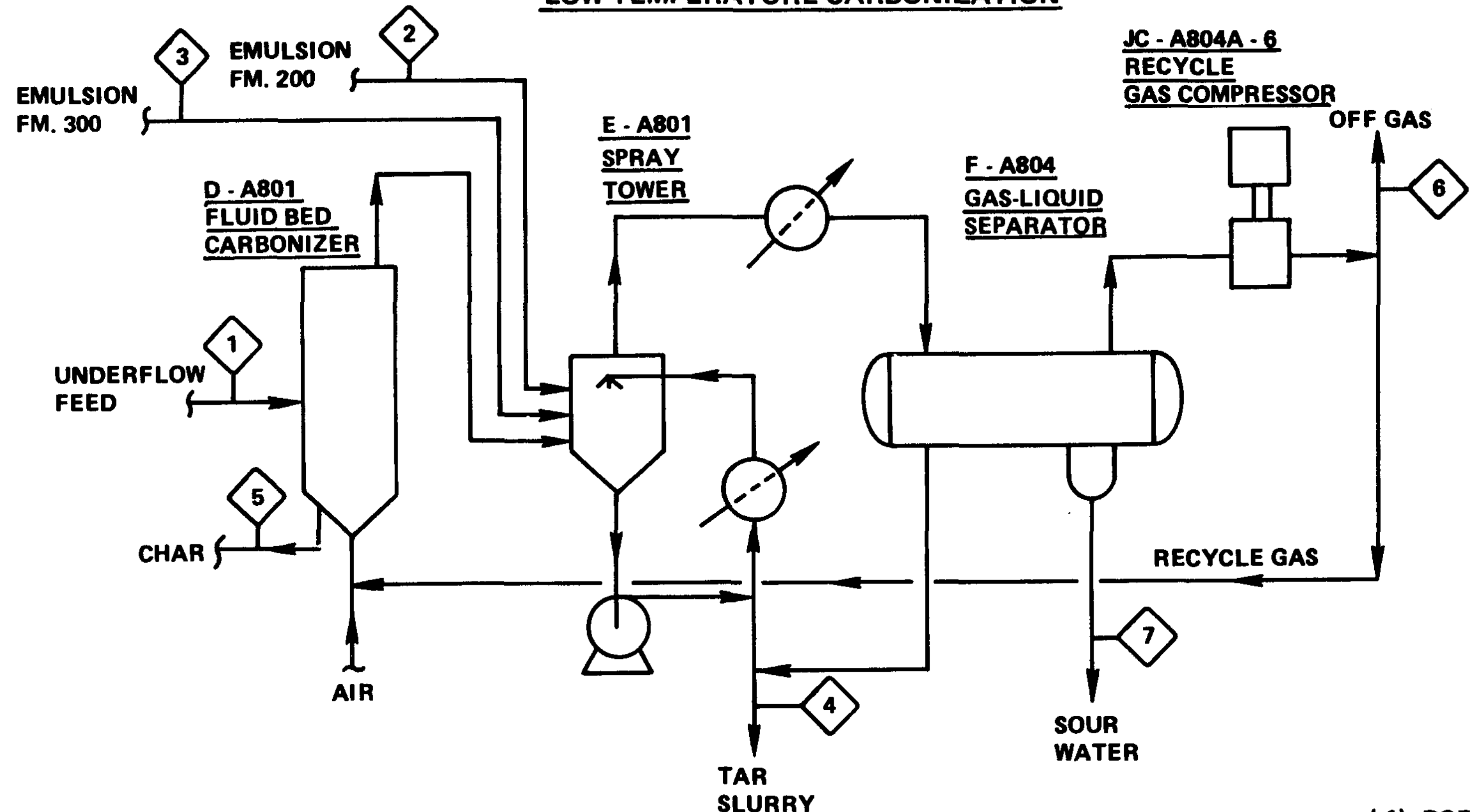
FIGURE 6
SECTION 800
LOW TEMPERATURE CARBONIZATION



LOW TEMPERATURE CARBONIZATION UNIT MATERIAL BALANCE

	① UNDERFLOW FROM 300	② EMULSION FROM 200	③ EMULSION FROM 300	COMBINED FEEDS	④ TAR SLURRY	⑤ CHAR	⑥ (1) OFF GAS	⑦ SOUR WATER
LIGHT GASES	—	—	—	—	—	—	103	—
WATER	—	20	—	20	—	—	—	5
SOLVENT	881	72	75	1028	1022	—	—	—
EXTRACT	68	—	—	68	41	—	—	—
SOLIDS	351	—	—	351	171	125	—	—
TOTAL, LBS/HR.	1300	92	75	1467	1234	125	103	5

(1) DOES NOT INCLUDE N_2 & O_2 IN THE COMBUSTION AIR.

TABLE 21

ANALYSIS OF CARBONIZER TAR SUMMARY

Sample Point 67

Date	Time	THF Insolubles, wt.%	Ash on THFI, wt.%	Solvent, wt.%	Extract, wt.%
11-17-77	0245	9.2	20.5		
11-17-77	0800	9.3		95.2	0.0
11-18-77	0435	14.2	42.1		
11-19-77	0510	28.3	22.3		
11-19-77	1045	19.7	22.1	79.4	1.0
11-20-77	0435	15.2	22.0		
11-20-77	1030	13.0	18.1		
11-21-77	1025	6.6	16.5	91.4	2.1
11-22-77	2110	22.5	22.0	77.6	0.0
11-22-77	1220	5.5	15.1		
11-23-77	0315	26.0	14.5		
11-23-77	0810	27.5	22.5		
11-23-77	1750	29.5	23.8		
11-24-77	0610	5.9	17.0		
11-24-77	0915	9.4	21.0		
11-24-77	1800	3.1			

5. (Continued)

Future Plans

Future plans call for carbonizer feed spray nozzles with simpler internal construction and a 0.25-inch minimum flow restriction as opposed to the previous 0.184-inch restriction.

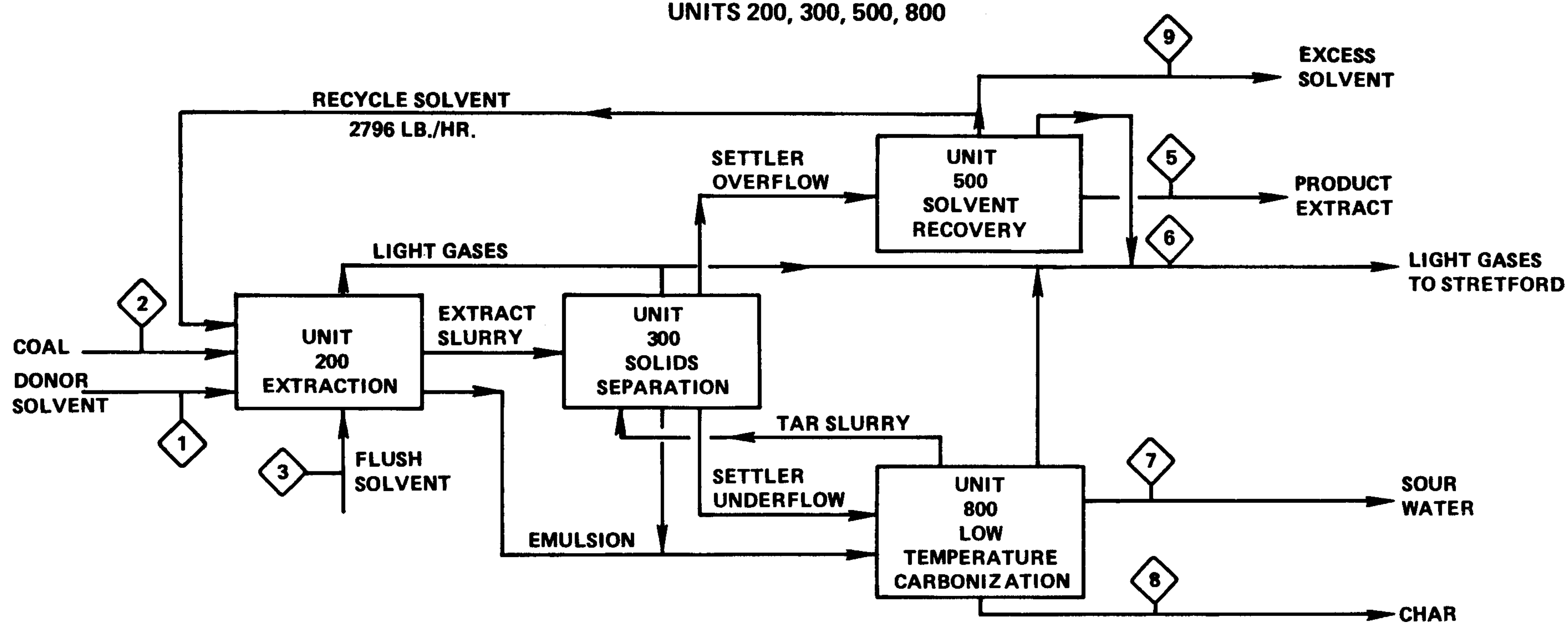
6. OVERALL MATERIAL BALANCE

The overall block flow diagram, Figure 7, shows the summary of feed and product flows representative of average Run 003 conditions. These results reflect usage of Sure Sol 180 as the Donor Solvent medium. A considerable amount of recycle solvent, 210#/hr, was used in the extractor to flush the seals. Concentrated extract, 73 wt.%, yielded about 55 wt.% on coal. Carbonizer solids at 127#/hr average rate was 27 wt.% on coal feed. The ash content of the char, as shown in Table 22, was 24 to 28 wt.%. The carbon content was 57-58 wt.%. The remainder of the products was distributed between light gases and compounds boiling in the solvent range. Additional operating time with corresponding analytical data is required to improve upon the accuracy of the overall material flows and compositions.

Future Plans

- o Initiate operations in the Extract Hydrogenation Unit to produce a coal derived donor solvent and hydrotreated extract product.
- o Operate in an integrated mode recycling donor solvent to the Extraction Unit instead of using the purchased Sure Sol 180.

FIGURE 7
OVERALL BLOCK FLOW DIAGRAM
RUN 003
UNITS 200, 300, 500, 800



OVERALL MATERIAL BALANCE

	1	2	3	TOTAL INPUT	5	6	7	8	9	TOTAL
LIGHT GASES	—	—	—	—	—	123	—	—	—	123
WATER	—	—	—	—	—	—	5	—	—	5
DONOR SOLVENT	173	—	—	173	—	—	—	—	—	—
SOLVENT	—	—	210	210	77	—	—	—	260	337
EXTRACT	—	—	—	—	258	—	—	—	—	258
COAL	—	467	—	467	—	—	—	—	—	—
SOLIDS	—	—	—	—	2	—	—	125	—	127
TOTAL, LBS/HR.	173	467	210	850	337	123	5	125	260	850

TABLE 22

CHAR ANALYSIS

Run 003 - November, 1977
Section 800 Sample Point 65

Date	11-15-78	11-17-78	11-17-78
Time	1100	1140	2130
Sample Number	3412	3490	3512
Moisture, wt.% as received	24.3	11.9	3.9
Proximate Analysis, wt.%, as received			
Volatile Matter	22.5	21.8	38.4
Fixed Carbon	29.5	38.1	29.5
Ash	23.7	28.2	28.2
Ultimate Analysis, wt.% dry			
Carbon	58.6	57.8	58.4
Hydrogen	1.4	1.5	1.6
Nitrogen	1.6	1.7	1.0
Oxygen (Diff)	12.2	5.2	4.5
Ash	23.7	28.2	28.2
Sulfur	2.5	5.6	6.3
Sieve Analysis, wt.%, Tyler			
+8	0.0	0.0	0.0
8 x 14	0.3	0.0	0.0
14 x 28	0.4	0.3	0.2
28 x 48	7.0	7.9	6.1
48 x 100	14.7	26.4	23.3
100 x 200	21.9	41.4	42.2
200 x 325	19.3	15.2	17.3
-325	36.4	8.8	10.9
Bulk Density, lb/ft^3	58.1		

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