

8/21/79

3053

FE-1517-72  
Distribution Category UC-90d

COAL LIQUEFACTION TEST CENTER

MASTER

Quarterly Technical Progress Report  
for the period  
July - September 1978

Fluor Engineers and Constructors, Inc.  
3333 Michelson Drive  
Irvine, California 92730

Date Published - February, 1979

Prepared for the U.S. Department of Energy  
Division of Fossil Fuel Processing

Under Contract EX-76-C-01-1517

NOTICE  
This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Department of Energy, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

DISTRIBUTION STATEMENT A THIS DOCUMENT IS UNCLASSIFIED

## **DISCLAIMER**

**This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.**

---

## **DISCLAIMER**

**Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.**

### NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Department of Energy, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, mark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

**Available from:**

National Technical Information Service (NTIS)  
U.S. Department of Commerce  
5285 Port Royal Road  
Springfield, Virginia 22161

**Price:** Printed copy: \$6.50  
Microfiche: \$3.00

## QUARTERLY REPORT

### 1.0 SUMMARY

During the third quarter this year there were three periods of integrated operation. Run 011 was conducted from June 29 to July 2 and consisted of 44 hours of coal extraction with 3 hours of integrated operations. Run 012 covered the period of July 7 to July 14 which achieved 30 hours of coal extraction with 21 hours of integrated operations. Run 013 covered the period of July 25 to August 11 and consisted of 142 hours of coal extraction and integrated operations. The hydrogenation portion of the plant processed various mixtures of fresh and stored extract material for 3, 56 and 339 hours during these runs. A Run Summary Log of operations to date is presented in the Appendix - Table I.

Run 011 was terminated when level instrument malfunctions allowed the extract/solvent mixture to overfill the first reactor. The mixture congealed in the overhead cooler and plugged almost half of the tubes. Runs 012 and 013 were stopped when process fluids leaked through the internal pressure liner of a reactor recycle pump. In both cases the leak resulted in a fire. In Run 012 the incident occurred as the plant was being shut down due to operating problems with the carbonizer. The carbonizer problems resulted in the destruction of the internal lift-leg. The Run 013 incident happened during normal operations.

## 1.0 (Continued)

The duration of integrated operations during Run 013 permits an establishment of base conditions with which to compare future results. At an average hydrogenation temperature of 805°F, disappearance of the +880°F fraction in the hydrogenation feed (conversion) was 53 percent. The corresponding desulfurization was 90 percent. An average temperature of 735°F was achieved in the extractor; the observed depth of extraction was typically 77 percent. The 560°F temperature of the solvent deashing settling vessel was well below the preferred operating temperatures of 625° to 650°F. The lower temperature is due to heat loss and will be compensated for in future runs by utilization of an idle fired heater to reheat the settler feed.

The planned schedule of activities for the fourth quarter of the year is presented in the Appendix - Figure 1. The planned work addresses three major objectives. These are: 1) determining the maximum slurry concentration that the extraction section can reasonably be expected to handle, 2) determining the relationship between upflow velocity in the settler and solids/ash content in the solvent deashing settler overflow at this maximum slurry concentration, and 3) extending the duration of the integrated operations periods. Minor objectives in support of the above include a revision in the solids separation section to achieve the desired processing temperature in the settler.

## 2.0 MAJOR PROBLEM AREAS AND SOLUTIONS

During this quarter, eight major problem areas were addressed. Three of these were resolved; five are still active.

### 2.1 Active Problem Areas

#### 2.1.1 Hydrogenation Reactor Recycle Pumps

During this period, there were two separate incidents (on July 14 and August 11) involving failure of a J-A608 reactor recycle pump. Both failures were accompanied by release to atmosphere of hot oil and hydrogen. The resulting fires terminated operations and damaged adjacent equipment.

The failure mechanism was similar in the two incidents. Loss of the front bearings allowed the rotor to contact the stator liner, resulting in abrasion and perforation of the liner. This allowed hot process material to enter the stator band housing at full pressure. The high pressure and temperature of the process fluid caused a rupture of the stator band electrical feedthrough seal which then allowed the process to vent to atmosphere.

2.1.1 (Continued)

Several factors were involved in the two bearing failures:

- a. In the first case involving the J-A608C pump, large internal clearances developed as a result of heavy metal loss from the bearing balls and races. An independent laboratory (Metcut Research Associates) confirmed that the loss was due to flaking fatigue. The latter normally results from excessive contact stresses (overloading) and/or inadequate lubrication.
- b. In the first incident, there was further evidence that overloading occurred. The wet-end components (impeller, wear plates, and associated fasteners) showed heavy damage from mechanical contact. Such contact is apt to occur during erratic operation (e.g., feed interruption). This will generate large and unpredictable bearing loads.
- c. Poor lubrication and overheating were evident in both cases. It was far more obvious in the second incident, which involved the J-A608D pump. (The latter operates roughly 150°F hotter than the J-A608C pump.)

#### 2.1.1 (Continued)

- d. The bearings in the J-A608D pump did not exhibit flaking fatigue. Instead, the front bearing had turned in the housing, severely abrading the outside diameter of the outer race. Metal loss was approximately 0.1 inch on the diameter. Rotation of the outer race is attributable to improper housing fit and lack of proper bearing retention.

Both pumps (and a warehouse spare) are now being rebuilt, with the following modifications to prevent recurring failures:

1. Electrical feed-throughs are being provided that are capable of withstanding system pressure and temperature. A relief connection is being provided that will safely discharge process material to a blowdown header in the event of a liner failure.
2. An external heat exchanger is being provided to cool lubricating oil adequately. The heat transfer area is being increased by a factor of five, since detailed calculations have shown the existing exchangers to be undersized.



2.1.1 d. (Continued)

3. Bearing housings are being modified to ensure proper bearing retention at operating temperature.

4. Wet-end wear plates and fasteners are being strengthened to reduce vulnerability to damage.

These changes should result in reliable machines that are capable of extended periods of operation. In addition, a number of external monitoring and safety systems are being provided. (See Engineering Tests and Projects below for further details.)

2.1.2 Makeup Hydrogen Compressors

Following the shutdown on August 11, it was decided to overhaul the JC-A900A makeup hydrogen compressor after approximately 2,500 hours of running time. This decision was based on the recommendation of the manufacturer to rebuild after 3,000 hours of service on dry gas (or more frequently on wet or dirty feed). Further, there was a recurring problem with high pressure between the first and second stages, resulting in lifting of the interstage relief valve. This was considered indicative of ring or

### 2.1.2 (Continued)

cylinder wear in one of the adjacent (higher pressure) cylinders.

Major inspection findings and repairs were as follows:

- a. The pulsation bottles and interconnecting piping were removed and inspected. The bottles were generally contaminated with a reddish brown, abrasive material (probably iron oxide) mixed with oil. This deposit was removed by flushing with kerosene. A proposal is under review to install a filter and moisture separator in the suction line.
- b. The first, second and third stage cylinders and rings showed no unusual wear. The cylinders were honed to remove minor scratches.
- c. A groove which was found in the fourth stage cylinder liner was determined to be too great for field repair. The damaged cylinder was returned to the manufacturer. It was rebored enough to remove the score, thus increasing the bore by 0.010 inch. This change was small enough to allow reuse of the existing piston with standard rings.

2.1.2 (Continued)

- d. In the fourth stage, the end of the No. 1 (head end) piston ring had a broken segment at the ring gap and the broken part was missing. The broken part is believed to have caused the scoring. Aside from this problem, the bronze rings showed normal wear. The machine was rebuilt using new bronze rings.
- e. In the fifth stage cylinder, a small ferrous metal particle was found during disassembly, apparently part of a failed fourth or fifth stage valve. The end of the piston and cylinder itself were not damaged.
- f. The cause of the high interstage pressure between the first and second stages was not fully defined. It is speculated that this was due to an abnormal pressure distribution caused by leakage from the damaged fourth stage cylinder.

The JC-A900B makeup hydrogen compressor has been disassembled since April awaiting a new third stage cylinder.

Five attempts were required to produce a cylinder that passed hydrotest. It has been received and the machine is being assembled with the new cylinder and all bronze

### 2.1.2 (Continued)

piston rings. The pulsation bottles and interconnecting piping are currently being cleaned and reinstalled. The machine will be run in around mid-December.

### 2.1.3 Hydrogen Generation Desuperheater

In late June, the F-B902 desuperheater developed a crack in a weld on the discharge nozzle. The weld was repaired, the nozzle shortened and a flange installed to eliminate another crack. The process line from the desuperheater to the D-A903 converter was modified to accommodate the change in nozzle configuration.

During the above repairs, the vessel welds were completely radiographed. Weld samples taken from the repaired sections also were sent to an outside laboratory for analysis. The analysis showed that the correct welding materials were used during vessel fabrication. However, the radiographs indicated there were weld deficiencies (lack of penetration) around the bottom head. After removal of the bottom head for repairs, a complete visual and dye penetrant inspection was made on the entire vessel. This revealed other weld deficiencies and numerous cracks on the four inch outlet

### 2.1.3 (Continued)

nozzle. The poor weld quality probably was due to poor workmanship and lack of quality control during original vessel fabrication. The stress cracking on the outlet nozzle is believed to be the result of thermal shock caused by liquid carryover due to poor gas/liquid contact in the vessel. Excessive piping stresses apparently were not a major contributing factor.

The repair work included:

- a. Replacement of the discharge nozzle and rewelding of all deficient welds.
- b. Modification of the process line from the desuperheater to the converter to provide proper flange alignment and eliminate any piping stresses.
- c. Installation of packing (Pall rings) inside the vessel to increase the liquid/vapor contact surface, thereby reducing the chances of liquid carryover and resulting thermal shock (PMR 273).
- d. Replacement of the vessel head as noted above.

#### 2.1.4 Section 600 Instruments and Controls

##### a. Instrument Purges

Pluggage of instrument impulse lines in Section 600 has been a major problem since start-up of the Hydrogenation Section, especially on the D-A601A reactor. This problem was caused by an inadequate liquid purge system which did not effectively prevent extract from plugging instrument lead lines. Inadequacies of the present purge system have been defined as follows:

1. Insufficient flow to prevent diffusion of process material into impulse lines.
2. Lack of compatibility between the lubricating oil purge fluid and the process material.
3. Poor mechanical reliability of the J-A614A/B purge pumps which resulted in frequent loss of purge.
4. Excessive hydraulic pulsations on transmitters resulting from use of individual Simplex piston pumps without damping.

2.1.4 a. (Continueud)

For Run 011, a short-term solution to the above mentioned problems was implemented. A temporary high pressure pump was installed with pulsation dampener, filter and flow-controlling capillary tubes to provide purging fluid to the "A" reactor instrumentation. To avoid incompatibility problems, Sure-Sol 180 was used as the purge liquid. Purge rates were increased to ten times the rates previously provided by the J-A614A/B pumps. These changes substantially improved performance of the "A" reactor instrumentation. Results also showed that capillary tubes, in conjunction with pulsation dampener, were effective in reducing pulsations on the transmitters.

During July, the design was completed for a permanent installation of this type, covering all liquid instrument purges in Section 600 (PMR 223). Fabrication is presently underway and will be completed before the next run.

#### 2.1.4 (Continued)

##### b. Nuclear Density Detectors

The existing Robertshaw nuclear density detectors on the D-A601D reactor were replaced with more reliable Kay-Ray detectors prior to Run 010. The Kay-Ray circuitry was modified to give a five second full scale response time versus 18 seconds for the Robertshaw detectors. This modification permitted more responsive control of catalyst bed ebullation. As a result of these changes, no catalyst carryover problems were experienced during this quarter.

##### c. Carryover from D-A601A

Carryover of liquid from the D-A601A reactor into the overhead cooler C-A601 has been associated with anomalous indications on the LI-6002A/B nuclear level detectors. During carryover incidents, both upper and lower detectors have shown similar readings. These were intermediate in value between those normally associated with gas (top detector) and liquid (bottom detector). Currently, the liquid level is lowered when the meter readings come together; this has minimized the problem.



2.1.4 c. (Continued)

In addition, the LI-6002A/B and LI-6003A/B nuclear level detectors were recalibrated to indicate level at a lower process density than was originally specified.

Further addressing the carryover problem, XFR-1204 and XFR-1211 reactor overhead flow recorders were moved from the test panel to the main control room panel (PMR 210). This enables the operators to respond more rapidly to abnormal overhead flows.

d. F-A602 Interface Problems

Adequate oil/sour water separation from the F-A602 high pressure separator tank finally was achieved during Run 013. The displacer for the LC-6007 level controller was found to be improperly sized and was replaced with a lighter one. The loading pressure on the actuators for the LCV-6007 and LCV-6009 control valves for the F-A602 underflow was increased to provide for tight shutoff of these valves. A capacitance type of level interface detector is being investigated to provide even better control for oil/water separation in this tank.

#### 2.1.5 Solids Handling in Sections 300 and 800

During Run 013, solids accumulated in the F-B331 settler and the F-B330 underflow receiver which resulted in a sheared drive pin on the rake of F-B331 and plugging of the underflow line from F-B330. The solids concentration in the return stream from Section 800 to Section 300 was twice as great as anticipated, probably due to partially restricted dip legs from the carbonizer cyclones. The sampling and analytical efforts during the next run will be intensified for the above three slurry streams. We believe that this was an isolated incident, and that the analytical results will show normal conditions for the next run.

### 2.2 Resolved Problem Areas

#### 2.2.1 Carbonizer Internal Damage

Inspection of the D-A801 carbonizer following Run 012 revealed that the lift-leg had been extensively damaged by high temperature. Portions of the lift-leg gas line were burned and melted above the grid plate. Tips of the thermowells in the general area were similarly damaged.

### 2.2.1 (Continued)

It was determined that the fluidized bed level transmitter had malfunctioned which caused the carbonizer to be improperly operated. A false low level was indicated. If the level drops below the cyclone dip legs, the remainder of the bed is almost immediately lost. To prevent this from happening, feed from Section 300 was started at lower than normal bed temperatures. This resulted in bed agglomeration and a gross maldistribution of fluidizing gas.

### 2.2.2 Reactor Effluent Cooler Leaks

Run 011 was terminated because of extensive tube pluggage in the C-A601 reactor effluent cooler. This was caused when extract-containing liquids overflowed the D-A601A/D hydrogenation reactors due to instrument malfunctions. Differential temperature distribution caused some of the plugged tubes to be pulled out of the tube header. When Run 012 was terminated because of a reactor recycle pump failure, the rapid system depressurization caused process fluids to again carry over into the effluent cooler.

After the plugged tubes were cleaned or replaced, the cooler was hydrotested in each incidence. Leaks were

### 2.2.2 (Continued)

found on hydrotest which required several tubes to be backwelded and several header plugs to be seal welded. The cooler has been returned to service.

### 2.2.3 Liquid Carryover from the Hydrogenation Reactors

Extract carryover from the D-A601A hydrogenation reactor (which contains no catalyst) also occurred during Run 013. A high liquid level in D-A601A was caused by the sudden failure of letdown valve LCV-6006B. The reactor overfilled before the spare letdown valve could be placed into service.

The reliability of the reactor level indicator/recorder has been improved with a modified instrument purge system. In addition, high and low level alarms have been installed on the nuclear level indicators to alert the operators to changes in liquid level.

## 3.0 OPERATIONS BY PLANT SECTION

### 3.1 Section 100 - Coal Processing

This section has operated reliably to replenish the F-A104 product storage bin and remains fully operable.

### 3.2 Section 200 - Coal Extraction

Routine extraction operations took place for 44 hours on July 1 and July 2 (Run 011), and again for 30 hours on July 11 and July 12 (Run 012), with the latter run being terminated because of problems in Section 800. Routine coal extraction totaled 142 hours during Run 013. Coal feed was interrupted several times because of problems downstream from the extraction unit.

During the emergency shutdown on August 11, loss of level control on the D-A201 extractor caused by low nitrogen pressure because of heavy nitrogen usage during the fire resulted in overfilling and overpressuring of the vessel. This caused a dislocation of an O-ring in the upper seal on the L-B205 extractor mixer shaft which resulted in a leak. This is currently being repaired.

Closed loop recirculation began September 22 to test solvent/product fuel oil mixtures for coking tendencies and/or other operating complications. Operations on mixtures of this kind will have the dual advantages of demonstrating the ability to operate on broader boiling range material to the system and will provide additional material for use as solvent during future operations. No unusual operating problems have been experienced on these tests.

### 3.2 (Continued)

Sprinkler fire protection was installed in this section and Sections 300 and 600. They were successfully tested on September 22.

### 3.3 Section 300 - Solids Separation

This section was on hot internal circulation during July with the exception of the two intervals of operation with Section 200.

There were no problems. The termination of Run 012 on July 12 left a higher solids concentration in the system than desired for standby status (fifteen percent instead of ten). No problems were experienced due to these solids.

After a relatively smooth start-up and considerable operating time, major problems developed during Run 013 with pluggages in the settling vessels starting with the F-B331 settler. F-B331 had to be taken off line and cleaned by hydroblasting. After restoring the system to service, the underflow from the F-B330 underflow receiver began to plug on the morning of August 11. An analysis of the material removed from the bottom of the settler vessels showed it to be largely char solids which recycled into Section 300 due to malfunctions in Section 800.

### 3.3 (Continued)

The B-B304 secondary slurry heater has been tied into the circulation loops to provide its 610,000 Btu/hour of additional heating capacity to maintain temperatures within the system necessary for proper settler operation.

### 3.4 Section 500 - Solvent Recovery

This section has operated without problems to concentrate extract from stored material and to provide fresh extract material for feed to Section 600. There has been one brief period of extract granulation, and two occasions when material was drummed to prevent solids carried over from Section 300 from reaching Section 600. The section also has processed materials from various slop and holding tanks to recover solvent.

### 3.5 Section 600 - Extract Hydrogenation

Run 011 was begun on June 29 with the introduction of fresh extract occurring on July 1, and was terminated the same day when instrument problems caused the D-A601A reactor to overflow, immediately plugging 20 of the 49 tubes in the C-A601 reactor effluent cooler.

Following cleanout and repairs to the cooler, stepwise start-up for Run 012 began with initiation of stored extract feed to Section

### 3.5 (Continued)

600 on July 10. This feedstock was replaced with fresh extract on July 11. Stored extract feed began again on July 13 following shutdown of the front end because of the carbonizer problem in Section 800. Shortly after introduction of stored extract feed to 600, pump J-A608C experienced an apparent "locked rotor" failure which then forced the shutdown of the 600 unit.

During the shutdown, a fire was experienced at the base of the D-A601A reactor caused by failure of the J-A608C reactor recycle pump. This is described in Section 2.0.

On July 23, repairs, purging, and pressure testing were completed and the system was readied for Run 013. This run logged 416 hours of continuous operation on various feedstocks under hydrogenation conditions. Operations continued under good control until 1200 hours on August 11 when the recycle pump on the D-A601D reactor failed and released hot oil from the system. This failure occurred at a time when the total hydrogenation system was in normal operation. The resulting fire was similar to the July 14 incident.

Repair to fire damage and overall plant operability improvement projects have occupied the balance of the reporting period. Several design improvements will be made to the reactor recycle pumps before they are returned to service.



### 3.6 Section 700 - Product Fractionation

This section has operated as required during extract hydrogenation operations.

There have been some performance problems related to water coming into the section with the Section 600 product, but it has been possible to bleed water from the system at the reflux drums when necessary. The water intrusion was due to the "F-A602 Interface Problem" reported in Section 2.0.

### 3.7 Section 800 - Low Temperature Carbonization

This section was brought on-line and operated well during the extraction run of July 1 and July 2. However, shortly after start-up in support of Run 012 on July 13, the D-A801 carbonizer bed was overfed with slurry during conditions of low bed temperature and insufficient fluidization. This caused the termination of Run 012 in order to clean out the solvent-soaked char. A decision was made to heat the carbonizer in an attempt to drive off excess solvent in order to attempt a restart or, at worst, to make the cleanout job easier, quicker, and safer. During this drying out period, combustion air flowed preferentially through and around the open lift-leg and, as a result, this riser was overheated and damaged beyond repair. The carbonizer was opened, cleaned out,

### 3.7 (Continued)

and a new lift-leg fabricated. Aside from the lift-leg damage and six ruined thermocouples, there was no other damage during this incident. Recurrence of this problem will be prevented in the future by assuring adequate fluidizing gas flow and bed temperatures before initiating slurry feed.

During Run 013, coal feed to the extraction section was stopped on two occasions while the carbonizer was out of service to clean plugged spray nozzles or to replenish char level. Pluggages in the Section 300 settler bottoms probably were the result of plug-gage in the internal cyclone separators in the carbonizer vessel which permitted char carryover into the tar quench system. Excessive solids in the tar quench are routinely returned to Section 300.

During the present maintenance period, quick-change spray nozzles are being fabricated and installed in the carbonizer vessel which will minimize interruptions of slurry feed flow and will result in more stable operation of this unit.

### 3.8 Section 900C - Hydrogen Compression

The JC-A900A makeup hydrogen compressor was operated as required throughout this period with regular changeout of the high pressure (fourth and fifth stage) suction and discharge valves. To prevent the first and second interstage relief valves from frequently lifting, it was necessary to continuously bleed gas from the first stage discharge. Repeated shutdowns were taken to inspect the second stage valves. Nothing out of the ordinary was found during these inspections. The machine was routinely operated in this manner, until it was found that by lowering the suction pressure, it could be operated without the continuous bleed.

### 3.9 Section 900G - Hydrogen Generation

This section was taken off line on June 27 because of a Hope Natural Gas Company outage. During this shutdown, inspection of the F-B902 desuperheater and associated piping resulted in the work described under "Hydrogen Generation Desuperheater" in Section 2.0.

### 3.10 Section 1000 - Utilities

The F-A1010 boiler feedwater tank and the L-A1010 deaerator vessel were inspected due to evidence of scaling. The inspection showed

### 3.10 (Continued)

corrosion in the F-A1010 tank. Repairs included application of a metallic aluminum coating.

The L-A1030A/B instrument air dryers continue to be a problem in that the maximum temperature which can be reached on regeneration cycle is well below the manufacturer's recommended temperature. Plant Engineering is reviewing this problem for correction during the next quarter.

### 3.11 Section 1200 - Environmental

Section 1220 Stretford Unit removed hydrogen sulfide routinely from off-gas streams during periods of operation. However, the sulfur solids concentration has not yet reached the level required to operate the sulfur removal filter. Operability of the filtration system has been previously demonstrated.

Section 1230 Waste Water Treatment continues to operate with primary and secondary adsorbers charged with activated carbon. The Tricellulator Unit, a rental device which removes oil and solids from the waste water ahead of waste water treatment, continues under test to optimize chemical addition rates. Excessive pressure drop across the L-A1230A primary adsorber late this

### 3.11 (Continued)

quarter was caused by a heavy contamination of oily sludge and silt on the carbon. Cleaning and recharging has been completed. Each of the two carbon beds has been changed twice during this period. The inlet water of the F-A1234 final holding pond was diverted directly to the Ohio River on July 11. This was done because the accumulation of sludge in the pond was beginning to contaminate the outfall water. After removal of sludge, it was returned to service on July 31.

## 4.0 TECHNICAL ANALYSIS OF PLANT OPERATIONS

Runs 011, 012 and 013 resulted in 216 hours of coal extraction operation and 398 hours of extract hydrogenation. Typical operating conditions for 011 and 012 (which total 24 hours of integrated operation) are shown in the Appendix - Tables II through VII. Run 013 (142 hours of integrated operation) is discussed in a separate report in the Appendix.

The coal concentration in the feed slurry was 15 percent at the start of Run 011 but was increased to 20 percent during the run. Slurry concentration was 20 percent for the other two runs. At a feed concentration of 20 percent, the concentration in the extractor was 18.5 percent when seal flush solvent was included. The coal feed rate at this concentration was 7.7 tons per day in Run 011 and 11 tons per day in the other two

#### 4.0 (Continued)

runs. The donor solvent to coal ratio was 0.4:1.0. The remainder to obtain the 20 percent concentration was recycle solvent.

The average extractor temperature ranged from 725° to 735°F and the corresponding depths of extraction ranged from 70 to 80 percent. Available information is insufficient to relate extraction depth to temperature as the donor solvent quality was also changing from run to run.

There was no attempt made during these runs to vary the process conditions in the Solids Separation Section. The primary settler vessel temperature varied from 540° to 560°F, well below the desired 625° to 650°F. Upflow velocities ranged from 0.34 to 0.51 inch per minute due to the different coal feed rates. The solids content of the overflow stream varied from 0.2 to 0.6 percent. Ash content of the solids has typically been around three percent. Overall extract recovery was typically 91 percent while solids rejection was about 95 percent.

The Solvent Recovery Section was operated to provide a 40 to 45 percent extract of +880°F material as feedstock for hydrogenation. Fresh extract was concentrated in all three runs; stored material also was processed in the latter two runs. Solvent recovery was typically 70 percent. This is below the design expectations; however, it is reasonable in that lower coal slurry concentration was used and extract was concentrated to only 45 percent.

#### 4.0 (Continued)

The fresh extract hydrogenation portion of Run 011 lasted only three hours. Typical operating conditions are presented in Table V; however, no attempt at analysis has been undertaken due to the brevity of the run. In Run 012, fresh extract was hydrogenated for 21 hours. Typical conditions are also shown in Table V. Subsequent to Runs 011 and 012, the TI-5-A temperature indicator was found to be out of calibration relative to the TR-3 temperature recorder by 10°F. The reported reactor exotherm values have been corrected. Due to insufficient sampling, the conversion of +880°F material was not determined. Desulfurization of the fresh extract approximated 75 percent.

In Run 013, the average hydrogenation reactor temperature was varied through a range of 720° to 835°F. The average temperature during the reported material balance period was 805°F. Conversion of extract (defined as the disappearance of the material having an atmospheric boiling point above 880°F) at this condition was 53 percent. As discussed later, this is a conservative assessment of conversion. The corresponding desulfurization was 90 percent and oxygen/nitrogen removed was about 70 percent. Hydrogen consumption during this period was 1,250 standard cubic feet per barrel of feedstock or 2.75 pounds per 100 pounds of coal processed. The above results compare favorably with the research data developed by the process licensor and with other published data on hydrogenation of coal derived liquids.

#### 4.0 (Continued)

The heavy fuel oil product made during this period contained 0.2 percent sulfur and 0.03 percent ash. The target maximums were 0.5 and 0.2 percent, respectively. The pour point of 33°F indicates that donor solvent recovery was not as complete as expected even though the product contained 50 percent +880°F material. The target pour point was 50°F.

During the last portion of Run 013, fresh extract feed to the hydrogenation reactor was replaced with heavy fuel oil product having a 40 to 45 percent +880°F fraction. The results of this operation (which are discussed in the appended report) were felt to be indicative of what might be achieved if two catalytic reactors were used in series for hydrogenation of extract. Conversion of the +880°F fraction in this operation was not precisely determined but appeared to be in the neighborhood of 67 percent.

Run 013 has provided a base for future run comparisons. In Run 014, this base will be expanded by characterizing the effect of variations in temperature on yield structure. In addition, modifications to the Solids Separation Section will provide a temperature increase in the settler, bringing it closer to the preferred operating temperature.

Conversion in the hydrogenation section can be related to the decrease of +880°F material from feed to product. The +880°F material is determined by distillation. The distillation proved to be troublesome to run



#### 4.0 (Continued)

because of the characteristics of the material. Analyses of the respective +880°F fractions by LCDC give typical compositions listed in the tabulation below. A significant upgrading of the fraction remaining as +880°F material also occurs. Characteristic data are shown below.

##### Composition of +880°F Fractions, wt%

	Fresh	Single	Double
	Extract	Hydrogenation	Hydrogenation
<u>Component</u>	<u>%</u>	<u>%</u>	<u>%</u>
*Benzene Insolubles	32	5	1
**Asphaltenes	22	18	10
Oil	46	77	89
Total +880°F (100# basis) 100-LB		47-LB	15-LB

\*Pre-Asphaltenes or benzene - insolubles are that fraction of the extract that is insoluble in benzene at its atmospheric boiling point.

\*\*Asphaltenes - the fraction of the benzene-soluble extract insoluble at room temperature in a mixture of 100 parts of cyclohexane and 9 parts of benzene when the ratio of the liquid mixture to the weight of benzene solubles is 109.

#### 4.0 (Continued)

Oil - the benzene-soluble, cyclohexane-soluble fraction of the extract.

Considering this information along with the raw conversion of the +880°F fractions shows that one pass through the hydrogenation reactor not only reduced the +880°F fraction by 53 percent, it also converted 80 percent of the benzene insolubles and asphaltenes to oil and solvent. Two passes resulted in an 85 percent overall conversion of the +880°F fraction and a 97 percent conversion to oil and solvent.

Component analysis of recycle and donor solvents is presented in Table VIII. Solvents used during Run 012 are compared with those being made at the end of Run 013. The composition of the donor solvent shows the inclusion of higher boiling components. A comparison of current solvents and the original start-up solvent is shown below:

##### Typical Solvent Boiling Curve Data, vol%, °F

Distillate	Start-up	Recycle	Donor
<u>Recovered</u>	<u>Solvent</u>	<u>Solvent</u>	<u>Solvent</u>
IBP	418	395	362
5	430	440	457
10	446	450	458
30	468	467	488

#### 4.0 (Continued)

50	480	475	513
70	492	490	592
90	514	543	722
EP	524	584	730

It appears that additional runs will produce broad boiling range solvents uniformly covering the middle distillate to extract boiling range.

Indeed, post Run 013 solvent recovery from the heavy fuel oil product has resulted in a donor solvent covering the range of 419° to 871°F.

#### 5.0 ENGINEERING, TESTS AND PROJECTS

##### 5.1 Tests

##### 5.1.1 Pump Seals (Test 1100)

##### a. Double Mechanical Seals

During the third quarter, there were three seal failures on the 15 critical process pumps equipped with Dura-metallic double mechanical seals. This compares with previous experience as follows:

5.1.1 a. (Continued)

<u>Period</u>	<u>Failures</u>	<u>Hours in Coal Service*</u>	
Pre-coal	8	-	
4thQ-77	17	Extraction-220	Hydrogenation- 0
1stQ-78	13	Extraction-137	Hydrogenation- 40
2ndQ-78	2	Extraction- 13	Hydrogenation-130
3rdQ-78	3	Extraction-216	Hydrogenation-398

\* Excludes operation on solvent and DOX oil. However, solvent circulated between runs usually contains some solids (1 to 5 percent).

The relatively favorable performance of these seals (as compared with earlier operations on coal during the fourth quarter of 1977 and the first quarter of 1978) can be attributed to three things:

1. Six failures during the earlier period were due to the melting of polypropylene filter elements in the seal oil system. These were replaced with Type 304 stainless steel elements. In addition, the seal oil system was adjusted to give proper flow of ~2 gpm per seal.

5.1.1 a. (Continued)

2. Several failures during Run 003 were due (at least in part) to pump starvation and cavitation caused by malfunctioning level transmitters in Section 300. This was corrected by upgrading the instrument purge system (PMR 42).
3. Many early failures showed fretting of the shaft sleeve beneath the rotary seal member, leading to binding of the seal. Often, there was also fretting and fatigue of the springs and drive pins. To cushion the relative movement of these parts, the compressed graphite (Durafite or Grafoil) secondary seals were replaced with Viton O-rings. The sleeves were hard chrome plated for improved wear resistance. These changes were effective; however, further operation has indicated that Viton is not fully adequate for sealing the inboard stationary member in hot services. Therefore, Kalrez O-rings are being installed in this location when a seal is repaired. The three seals that failed during this reporting period included two still equipped with Durafite (J-A313A/B Primary Hydroclone Feed Pump) and one overheated inboard Viton O-ring

5.1.1 a. 3. .(Continued)

(J-A314B Wash Hydroclone Feed Pump). The former was replaced with Viton and the latter with Kalrez.

b. Single Mechanical Seals

Fourteen failures occurred during this quarter, all involving Chempro-Sealol bellows seals. Ten of these were associated with the hydrogenated products, including seven failures in the heavy fuel oil product pumps (J-A704A/B and J-A1109A/B) and three in the donor solvent pumps (J-A706A/B). Four actions are being taken to improve single seal reliability, including improving the quality of the seal flushing oil.

1. On the four heavy product oil pumps, steam tracing and insulation will be provided to keep the hydroclone separator and stuffing boxes hot (PMR 198).
2. On a number of pumps, the seal flush hydroclone will be relocated directly above the pump so that the underflow can return to the pump suction via a short vertical line (PMR 198). These lines are now long and pocketed. When pluggage occurs, the

5.1.1 b. 2. (Continued)

hydroclone discharges solids into the seal, resulting in failure.

3. External hydroclones will be added to improve the quality of seal flush on certain Sundyne pumps that have experienced repetitive seal failures (PMR 100).

4. Other single seal designs that may have greater inherent reliability are being investigated. A limited evaluation will be recommended via PMR.

5.1.2 Pump Coatings (Test 1100)

Three PTI-54 coated pumps that were removed for inspection showed coating failure and extensive erosive damage.

These were the J-A205 and J-A206 slurry pumps (10 - 25 percent coal, 100°F) and the J-A806A tar quench pump (2 - 20 percent char, 250°F). Erosive damage was evidenced in high turbulence areas (e.g., the cutwater, the wear rings, wear ring screws, and adjacent parts of the head and case). The impellers showed erosion at the vanes, balance holes and shrouds. (The shrouds on the J-A206 impeller

### 5.1.2 (Continued)

were worn through in places.) This reflects the fact that the plasma spray process cannot coat the internals of the impeller. As a temporary measure, the eroded areas and wear rings of the head and case were weld repaired with an overlay of "Hardalloy 32" (a low carbon martensitic material). The remnant PTI-54 coating (which was in good condition) was left in place. A new nitrided 12 percent chrome impeller was installed in J-A206.

Actual coating erosion (as opposed to flaking) was seen in the J-A806B tar quench pump. The Tribaloy 800 coating was eroded down to the base metal on the case, on part of the head wear ring inside diameter and around the balancing holes on the impeller. This is due to the lower hardness of Tribaloy 800 (RC-44) compared to PTI-54 (RC-68). A new Stellite No. 6 pump was installed.

In the higher temperature ranges, complete flaking of the tungsten carbide coating was noted on the J-B303A slurry underflow pump which sees 10 percent solids at 500°F. However, coatings on the J-A711A (PTI-54) and J-A711B (Stellite No. 6) vacuum column feed pumps were in excellent condition. During the operating period, the latter pumps



### 5.1.2 (Continued)

see an average of 1 percent solids at temperatures up to 650°F. Good performance under these conditions is surprising in view of repeated coating failures in other hot services. However, one possible explanation is the absence of shock loading from solids. Several alternatives are being pursued and evaluated for better wear resistance of pump parts. The following was accomplished during this period:

- a. The Pump Reliability Program was issued. It outlines the plan of action that will be followed to do mechanical work and hard coat the pumps which have lining and erosion/corrosion problems.
- b. The new J-C303 slurry underflow pump is being activated. This is a semi-open impeller type with PTI-54 coating and a replaceable casing wear plate.
- c. Alternate high integrity coatings that will not suffer from flaking and which can reach all areas to be coated are being investigated. A pump is being prepared for coating with TMT-5 (boron halide) applied by the chemical vapor diffusion process.

### 5.1.2 (Continued)

- d. Thought is being given to the use of solid impellers made out of 28 percent chromium iron. Since an impeller will continue to function despite sizable dimensional changes, it appears longer service life can be achieved by using a through-hardened material. Certain design modifications to provide greater protection to high wear areas are also under review.
- e. Because of its versatility and economics, a modified plasma spray process along with a better coating is being looked into. This will involve rounding off corners (for good bonding) and using extension guns (for reaching internals). The new material, Metco 136F (95 percent  $\text{Cr}_2\text{O}_3$ , 5 percent  $\text{SiO}_2$  and 3 percent  $\text{TiO}_2$ ), has better hardness and bonding properties than the previous materials used.
- f. Two pumps already coated with electroless nickel are being evaluated in the J-A804 solvent quench service. (Uncoated pumps in this service formerly suffered from severe erosion/corrosion attack.) Two more pumps, one coated with electroless nickel and another with Nye-Carb (30 percent silicon carbide in an electroless nickel

5.1.2 f. (Continued)

matrix) will be tested in the high temperature J-A313 primary feed pumps.

5.1.3 Valves (Test 1600)

The Test 1604 Marlin check valve was removed for inspection during repairs to the carbonizer after Run 012. One of the Rene-41 springs showed wear at the ends from rubbing against the internal metal parts. The other had yielded and deformed. Some amount of corrosion was noted on the plates and seating surfaces. It is believed that this failure is associated with the high temperature upset in the carbonizer. The valve was rebuilt using two new Rene-41 springs. The performance of the valve will be monitored in the coming runs. The valve had been in service at carbonizing conditions for about 2000 hours.

A new Wheatley ceramic gate valve was installed on the discharge line of the J-B202 extractor charge pump and successfully pressure tested (PMR 224). The gate and seats of this valve are made of 99 percent alumina ceramic. The gate slides between the two lapped seating surfaces, opening and closing with a self-wiping action. It should have good resistance to solids binding as well as scoring

### 5.1.3 (Continued)

and erosion. Acceptable performance of this valve in slurry service will demonstrate the usefulness of valves of this type in coal liquefaction services.

### 5.1.4 Control Valves (Test 1700)

#### a. DPCV-301

The tungsten carbide plug for DPCV-301 broke twice in separate incidents during the third quarter reporting period. This differential pressure control valve controls the product stream from the F-B332 primary feed tank. It normally sees 5 to 10 weight percent solids. Fractures of the plug were attributed to shock loading, which exceeded the impact resistance of the tungsten carbide. (This is a brittle grade of carbide with only 1.5 percent cobalt binder.) Shock loading is believed to have occurred in trying to stroke the valve to clear pluggage. Recommendations are under review to fabricate a plug from a less brittle grade of carbide and to change the valve trim to a top guided design to avoid buildup of product between the plug and the bonnet.

#### 5.1.4 (Continued)

b. FV-331

Control valve FV-331 was inspected to determine cause of leakage across the seat. The valve is in the feed line to solvent heater B-B305. Significant erosion was found on the stellite plug, causing excessive leakage. The trim was replaced with a smaller stellite trim to correct an oversizing error. A different material is being investigated for a replacement trim.

c. LCV-6006A/B

Control valve LCV-6006B was removed to check for pluggage. This is a letdown valve from the F-A609 residue separator tank. Inspection revealed that the tungsten carbide tip had separated from the stainless steel stem at the silver soldered junction. A higher melting point material was used to rebraze the tip. The plug separated again after two days of operation. It was then pinned to the shaft. The valve will be installed and tested during the next run.

5.1.4 c. (Continued)

Two alternate designs for LCV-6006B trim are currently being considered. One is a shrink fit for attaching the tip to the stem, while the other is a screw thread and locking pin.

Control valve LCV-6006A, which is a backup for LCV-6006B, was removed to check for causes of excessive leakage through the valve. Inspection revealed severe erosion between the tungsten carbide seat insert and the stainless seat retainer. The seat was replaced with an identical spare.

d. PCV-802

Control valve PCV-802, which controls sour gas to the Stretford Unit, was found to be undersized. (To obtain adequate flow rates, the bypass valve had to be opened.) The valve was replaced, changing the trim from 1 inch to a 2 inch size.

e. XLV-1781

This test valve is the high pressure product letdown valve backup for LCV-6006A/B. During July and August,

#### 5.1.4 e. (Continued)

it was operated for 169 hours using the "A" trim (tungsten carbide grade K-701). The valve performed without mechanical problems. However, it does not provide a tight shutoff, having a minimum flow of 1.7 gpm. Thus, control is poor at low flow rates (less than 2.5 gpm). This makes the valve somewhat oversized when the unit is operated in the single-reactor mode, especially if the concentration of +880°F material in the feed is low. This is not expected to be a problem when the section is operated closer to design conditions.

#### 5.1.5 L-B205 Extractor Mixer Seals (Test 1800)

After the conclusion of Run 013, the top and bottom mechanical seals on the L-B205 mixer were opened for inspection and repair. At that time, both seals had operated for 1,025 hours (229 hours in integrated coal operation and the balance in various modes of recirculation). The top seal had developed a substantial leak, while the bottom seal was operating satisfactorily.

Inspection of the bottom seal revealed only trace amounts of solids in the seal cavity. The internal spring-loaded

#### 5.1.5 (Continued)

packing assembly and the seal faces were both in good condition. The shaft sleeve was slightly worn in the external auxiliary packing area. It was apparent that the seal could have run substantially longer.

The Durametallic double seal used at the top of the vessel was leaking due to a failure of the "Viton" O-ring on the inboard stationary face. The rotary member had one spring broken, apparently due to fatigue. The seal may have failed because of overfilling the vessel. However, subsequent problems with the O-rings cast some doubt on this hypothesis.

The bottom seal represents a state-of-the-art application, in terms of temperature exposure, size and submerged operation in a high solids environment. It is therefore appropriate to summarize the major problems and resulting modifications that have led to the present configuration. (Please refer to Figure 2 showing a half section through the seal.)

- a. During early operations, a spring-loaded packing assembly was added to improve solids exclusion while



5.1.5 a. (Continued)

controlling the flow of seal flushing oil into the process. A limited improvement resulted. After Run 007, it was observed that the carbon spacer ring used to compress the packing had shattered, flooding the seal cavity with solids. It was replaced with a 304 stainless steel ring, which allowed the packing to function effectively.

- b. Prior to this, the double shroud was installed. Its purpose was to concentrate flushing oil onto the seal faces and exclude coal fines from the bellows assembly. A second flushing port was added to give better distribution and allow solids to be bled from the seal cavity.
- c. After some initial success, numerous failures (cracking) were encountered with the silicon carbide stationary face supplied by Chempro-Sealol. Attempts to solve this by adjusting face loading were unsuccessful. A tungsten carbide stationary face was then installed and the seal gland was modified to accommodate and secure it against rotation (a recurring problem with the previous design). The bellows assembly was shifted

5.1.5 c. (Continued)

to its present location, which corrected initial excessive wear by reducing face loading.

- d. A recurring problem in the past has been fatigue failure of the seal bellows. This was fundamentally due to the fact that the bellows must flex each revolution to accommodate any misalignment between the shaft and the vessel head. It was greatly aggravated when the convolutions of the bellows pack with solids since this restricted flexibility and increased stress levels. Effective solids exclusion may solve this problem, but greater operating time is needed to prove this. A more fundamental solution would be to use a seal design which places the flexible member in the stationary, thereby eliminating fatigue as a limiting factor in seal life.

5.1.6 Erosion/Corrosion (Test 1900)

a. Piping

A 1-1/2 inch socket weld tee and a 2 inch socket weld elbow on the slurry recirculating line above the

5.1.6 a. (Continued)

F-A203B slurry mix tank were replaced due to through-wall leakage. Erosion occurred on these fittings (both carbon steel) at the joint between the fitting and the pipe on the downstream side. (The upstream side showed no significant material loss in either case.) Erosive attack was initiated by localized turbulence. Causes of the latter include:

1. The use of Schedule 80 pipe with 3,000# fittings created an internal shoulder because of the smaller pipe internal diameter. There was also the usual small crevice between the end of the pipe and the socket weld fitting.
2. Secondary flow (internal recirculation) typically occurs when there is a change in flow direction.

Once erosion began, the resulting irregular surface caused additional turbulence and accelerated attack.

The two failed fittings were replaced with new ones of the same type. Ultrasonic thickness readings were taken on other fittings in the line. These showed

5.1.6 a. (Continued)

some metal loss, but none were worn to the point of failure.

A DOE failure report on this problem is being prepared. It is recommended that all or a portion of this line be replaced with long-radius butt weld fittings to see if this will reduce or eliminate erosive failures.

b. Nozzles

Type 316 stainless steel spray nozzles experienced severe erosion in both the D-A801 carbonizer and the E-A801 spray tower. An average of 57 hours operation was observed in three different upper feed nozzles in the carbonizer. A new design (Spraco "ramp bottom") made of hardened 400 series stainless steel is being tested in the carbonizer. One of these nozzles was recently installed and shows little wear after 30 hours of operation. New nozzles, also made of 400 series hardened stainless, are being tested in the spray tower. These are of the original tangential whirl design that were unsuccessful in the 316 nozzles. Consideration is also being given to improved work materials or hardcoatings.

#### 5.1.6 (Continued)

##### c. Reactor Bubble Caps

Brittle failure of the carbon steel bubble caps on the bottom grid plate of the D-A601D hydrogenation reactor is believed to be the result of hydrogen attack. A 2-1/4 Cr - 1 Mo steel has sufficient resistance to hydrogen attack, but the effect of other corrodents needs to be considered. A failure report is in preparation.

#### 5.2 Projects

The following projects received major emphasis during the third quarter of 1978. Some are complete and operational, while the rest should complete prior to the resumption of plant operation in November. In addition, a number of minor PMR's was also completed during this period.

##### 5.2.1 PMR 142 - J-A204A/B Minimum Flow Cooler

The heat exchanger was installed to cool the minimum flow return from the J-A204A/B spray solvent pumps. This will prevent overheating of the F-A204 storage tank which reduces the NPSHA for the pumps, thus causing cavitation.

#### 5.2.2 PMR 222.- Quick Change Nozzle for Carbonizer

The upper feed nozzle assembly on the D-A801 carbonizer was modified to permit quick changeout without cooling or depressurizing the carbonizer. This included a slight relocation of the feed nozzle to avoid interference with the dip legs and a new valve and packing gland assembly to permit withdrawal of the nozzle without allowing process material to escape.

#### 5.2.3 PMR 223 - High Pressure Purge System

Installation of the new J-A614A/B pumps and associated equipment (pulsation dampeners, filters and capillary tubes) is approximately 90 percent complete. (See the Major Problem section for further details.)

#### 5.2.4 PMR 230 - Instrument Air Dryers

The piping modifications around the instrument air dryers include new check valves, bleedline and rotameter. These will provide correct air flow and desiccant regeneration to achieve the design dew point of -10°F. Block valves will be added to permit dryer isolation for maintenance. Also, a new heavy duty timer, thermocouples and controller

#### 5.2.4 (Continued)

will be installed to improve overall system reliability and provide a high temperature shutoff and alarm.

#### 5.2.5 PMR 245 - Activate B-B304

Jumper lines were installed to connect the B-B304 secondary slurry heater (not previously used) into the recycle line from the J-B303A/B underflow pumps to the F-A301 feed tank. This will provide additional heating capacity (610,000 Btu/hr) to attain higher deashing temperatures in the F-B331 primary settler.

#### 5.2.6 PMR 246 - Remove Cold H<sub>2</sub> Lines

The cold hydrogen lines associated with the TCV-6010 and 6011 control loops were removed because the injection of cold hydrogen to the reactors is no longer required.

#### 5.2.7 PMR 252 - J-A608C/D Heat Exchanger/Filter

An external heat exchanger and filter system will be installed on the J-A608C/D reactor recycle pumps. This will provide clean and cool lubricating oil for the Chem-pumps, thereby eliminating a major cause of bearing failure.

5.2.8 PMR 254 - Lab Ventilation

The sample unloading area in the laboratory will be enclosed and a larger exhaust fan installed to provide adequate venting of hydrocarbon vapors. Performance of the hood in the distillation lab will also be upgraded.

5.2.9 PMR 267 - J-A608C/D Vibration Monitoring

Bently Nevada velocity transducer systems will be installed on the J-A608C/D reactor recycle pumps to provide continuous remote monitoring of bearing vibration levels. This system will give warning of deteriorating equipment condition so that action can be taken to prevent catastrophic bearing and pump failure.

5.2.10 PMR 268 - J-A608C/D Relief System

Board mounted high pressure alarms and a safety relief system will be installed on the motor stator band of the J-A608C/D reactor recycle pumps. The high pressure alarm will indicate failure of the internal pressure liner, and the associated rupture disc and blowdown line will safely reduce system pressure in the event of such failure.



#### 5.2.11 PMR 270 - J-A608C/D Current Recorders

Board mounted recorders will be installed to record motor currents on each of the J-A608C/D reactor recycle pumps. These will provide immediate indication of motor current abnormalities, which in the past have given an early indication of pump problems. The recorded data will also be used to analyze and interpret motor performance.

#### 5.2.12 PMR 271 - J-A608C/D Circuit Breakers

Circuit breakers for the J-A608C/D pumps will be modified to trip the motors instantaneously in case the line current exceeds a predetermined set point. Previous experience indicates that internal mechanical failure of these pumps is accompanied by high and erratic motor currents. Should this occur, the modified circuit breakers will safely remove power from the motors in less than 0.2 seconds, while permitting normal high starting currents.

### 6.0 MAINTENANCE

This quarter, the annual inspection of the two boilers was performed and a satisfactory rating was received. During the boiler inspection shut-down, other work associated with this system was accomplished. This included cleaning and coating the boiler feedwater tank, repairing the

## 6.0 (Continued)

boiler blowdown pipe, sight glasses, and various steam leaks on the main headers. Elimination of these leaks is part of an ongoing program initiated during this report period in an effort to repair all plant steam leaks prior to the winter months.

The remaining work in this area includes the installation of pumps, insulation and instrumentation.

The JC-A900A makeup hydrogen compressor has been dismantled and is undergoing a complete inspection. The JC-A900B also will undergo a complete inspection and cleaning before being returned to service.

Various heat exchangers and coolers were cleaned during the shutdown and all items on the maintenance work order backlog are being addressed at this time. Items completed to date include reworking fire water system post indicator valves and vessels, valves, pumps, associated piping and insulation in Sections 200, 300 and 800.

The installation of the Grinnell deluge fire protection system for Sections 200, 300 and 600 was completed. Subcontracts for the purchase and installation of the emergency generator have been let; installation of the generator foundation was completed.

## 7.0 ENVIRONMENTAL

### 7.1 Incidents and Permit Compliance

The large amounts of water used to fight the fires in Section 600 caused slight amounts of oil to escape the final holding pond. Both incidents were reported to the National Response Center (U.S. Coast Guard).

During this quarter, we received an EPA Administrative Order-Findings of Violations and Order for Compliance as a result of our previously filed noncompliance reports. The reply to this order was prepared with the assistance of legal and environmental personnel from SCD.

The state of West Virginia has modified our water pollution control permit as a result of our request last quarter. The pH limits on the sewage outfall have been changed from 6.0-8.5 to 6.0-9.0 standard units. Now both the industrial and sewage outfalls have the same pH limits on both State and Federal permits. In addition, the EPA is proceeding on our request to modify the NPDES permit limit on phenols. We are asking that the maximum average concentration limit be increased from 0.05 ppm to 0.5 ppm. The current value is excessively restrictive and can only be met during periods of nonoperation. Based on our current knowledge, the requested limit is more practical for operating periods.

## 7.2 Process Modifications

At the closing of the last quarter, an induced air flotation device (Tricellerator) was leased and placed in trial service. The objective is to pretreat the industrial waste water being fed to the activated carbon beds. The results to date indicate satisfactory performance and that air flotation should be a processing step in our water treatment operations.

Even with air flotation, however, a small amount (~20 ppm) of oil and grease along with a very fine, silty particulate passes through to the carbon beds. Accumulation of these materials causes the pressure drop across the beds to increase. Finally, the carbon must be replaced. This appears to be the same type of material which fouled the adsorption resins initially used in this operation. A laboratory evaluation program has begun to determine if chemical regeneration of the carbon can effect removal of these foulants. Filtration is also being considered.

## 8.0 SAFETY AND HEALTH

### 8.1 Accident Analysis

At the end of this quarter, the accident frequency rating is 6.0 and the severity rating is 90.5 per 200,000 manhours. The previous quarter ended with ratings of frequency of 6.5 and severity of

## 8.1 (Continued)

86.1 per 200,000 manhours. No injuries were sustained in the fires on July 14 and August 11.

## 8.2 Indicators

8.2.1 First aid cases - 145

8.2.2 OSHA recordables, lost time - 2

8.2.3 OSHA recordables, no lost time - 2

8.2.4 Number of days lost - 65

## 8.3 Fire Experience and Protection

On August 30, the main fire water pump was inspected. Sprinkler systems were tested on September 22. Gravel in the system caused partial blockage of the nozzles. Since flushing has not successfully removed remaining gravel, a PMR requesting installation of strainers for these systems has been submitted.

#### 8.4 Training

The following employee training sessions were conducted: new employee orientation, advanced first aid, cardiopulmonary resuscitation, lift truck drivers safety training, instrumentation, respirator and weekly fire brigade training.

#### 8.5 Industrial Hygiene

On August 1, hydrogen sulfide was detected above 500 ppm in the waste water treatment equalization basin. Acid, used to adjust pH, had reacted with char to cause this problem. Corrective measures taken: the acid is now put into the waste water system after it leaves the treatment facility.

#### 8.6 Medical

Fourth quarter skin examinations started in August. Preemployment and annual physicals continued.

#### 8.7 Other

Representatives for NIOSH and one of their advisory subcontractors, JRB Associates, conducted an audit of the plant's operation. The information obtained will be used along with that from other coal

## 8.7 (Continued)

liquefaction pilot plants in preparing a criteria document of recommended safety and health guidelines for such plants.

As a result of the OSHA inspection last quarter, Liquefied Coal received one serious and six nonserious citations with penalties. On August 29, a hearing was held at OSHA's office in Charleston, WV concerning the citations and penalties. The serious citation and one nonserious citation were dropped along with all fines and penalties.

## 9.0 DEPARTMENTS

### 9.1 Operations

Areas of major emphasis during this period have included effort at all levels toward revision of fire brigade structure and development of specific section-by-section fire fighting procedures. Safety, safety training and training in the use of protective and respiratory equipment also have been addressed.

Progress also has been made toward clarification of the Operations/Maintenance interface and the areas involving the respective disciplines more clearly defined. A part of this effort is being

## 9.1 (Continued)

directed toward a reduction in the number of high priority work orders written by Operations.

Mechanical Flow Diagram checkouts of all systems have been done, as well as identifying, marking, and listing of steam and other utility leaks.

## 9.2 Technical and Engineering

Prior to the termination of Run 013, engineering efforts were focused on: a) modifications to improve plant reliability and operability, with special emphasis on Section 600 instrumentation; and b) direct process and mechanical support for the achievement of sustained integrated operation.

Since the end of Run 013, efforts have shifted to: a) technical analysis of plant operation during the successful run, b) reliability improvements to the reactor recycle pumps and installation of related monitoring and safety systems, and c) engineering support for fire damage repairs in Section 600.



## 9.2 (Continued)

A major organizational realignment in the Process Engineering group has been effected. This was required by the net reduction of three people. As a consequence, the process engineering activities were reprioritized to more realistically match the workload to the available personnel. Emphasis will be placed on the process analysis of Sections 300 and 600 with "as available" attention given to the other sections. Efforts have been intensified to computerize the gathering and reporting of data to maximize our effectiveness.

Programs have been written to correct the automatically logged flow values for actual process conditions. These corrected values will be used in the Daily Summary Report and the Operating Conditions Report. These reports are being formatted and programmed. They will be put into use during the next quarter.

Procedures are being developed in the laboratory to fractionate and characterize feed to, and the total liquid product (TL) from Section 600. This has been especially troublesome due to the tendency of the still pot contents to superheat and violently boil over. Good progress is being made toward solutions here and a trip to the licensor's laboratory to review their procedures is scheduled.

## 9.2 (Continued)

As part of the restructuring of the Technical and Engineering organization, the Test Engineering group was merged into Plant Engineering. It is anticipated that this will prevent duplication of effort and provide a more effective utilization of limited resources. (The two groups together are short of authorized strength by four people.)

In conjunction with this merger, Test Program activities were reviewed and prioritized. A plan was issued for the fourth quarter of 1978, reflecting three necessary constraints:

- 9.2.1 Development work will be focused on things which directly contribute to operability and reliability.
- 9.2.2 Equipment evaluation will be in terms of overall performance, service life and qualitative assessment of wear.
- 9.2.3 Report output will be limited, with emphasis on DOE Failure Reports.

## 9.3 Maintenance

During the latter part of the quarter, a program was initiated to reorganize the plant maintenance activities. The objective is to

### 9.3 (Continued)

consolidate all maintenance related work into a closely knit group, and to better define the key responsibilities of maintenance and plant engineering groups.

Initial organizational activities have been previously directed toward the refinement of existing, and development of new, work-management control systems. Work order priorities have been redefined along functional lines. The goal is to yield job groupings which provide a firm basis for scheduling the maximum amount of work a week in advance.

The computerized active work order file has been reprogrammed to list work orders by priority and by date within priority with the oldest listed first.

Work has also been initiated on a computerized Preventive Maintenance Schedule program. The program is similar to and complements the existing computerized weekly plant lubrication schedule.

A Materials Tracking System has been formulated and has been put into use. Materials are now being prepackaged by the warehouse for each work order and all materials disbursement is tied directly to individual work orders.

### 9.3 (Continued)

Two new supervisors, with extensive experience, have been hired by Catalytic, the craft maintenance subcontractor. Both are now also on the job. The craft labor force has been restructured in an attempt to better fulfill the requirements of the current work backlog.

During the next quarter, it is planned to strengthen the planning activity and to integrate electrical and instrumentation work into the new departmentalized organization.

## APPENDIX

### TABLES

- I. Run Summary Log
- II. Typical Operating Conditions, Section 200 - Extraction
- III. Typical Operating Conditions, Section 300 - Solids Separation
- IV. Typical Operating Conditions, Section 500 - Solvent Recovery
- V. Typical Operating Conditions, Section 600 - Extract Hydrogenation
- VI. Typical Operating Conditions, Section 700 - Product Fractionation
- VII. Typical Operating Conditions, Section 800 - Carbonization
- VIII. Capillary G. C. Analysis Report

### FIGURES

- 1. Fourth Quarter Plan, 1978
- 2. Extractor Mixer Seal

### REPORTS

Run 013 Process Report

**TABLE I**  
**RUN SUMMARY LOG**  
**Hours of Operation**

Run No. Date	001 10/11-77	002 10/17/77	003 11/15- 21/77	004 12/16- 18/77	005 1/25- 2/7/78	006 3/19- 20/78	007 3/20- 24/78	008 5/2- 17/78	009 5/24- 6/1/78	010 6/15- 26/78	011 6/29- 7/2/78	012 7/7- 14/78	013 7/25- 8/11/78	Time to Date
EXTRACTION (200)					(1)*	(2)	(1)	(1)	(2)	(2)				
Slurry Concentrations														
10%	1	20												21
15%			144	55	73						27			299
20%							64	13			17	30	142	266
														<u>586</u>
Temperature Rate, tpd	<700 5	640 5	700± 5±	700± 5±	700± 6±		700± 11	700± 15			725± 7-11	725± 11	730 11.3	
HYDROGENATION (600)														
Solvent/DOX Oil						33	11	130	67	104	53	95	77	570
Stored Extract								15		109		35	185	344
Fresh Extract							40	6			3	21	154	244
														<u>1,138</u>

\* (1) Intermittent  
(2) Section 600 only

TABLE II  
TYPICAL OPERATING CONDITIONS  
SECTION 200 - EXTRACTION

Run Number		011	012
Coal Feed Rate, tpd		7.7	11
Raw Coal Concentration, wt% in B-A201 Feed		15	20
Solvent Feed Rate, gpm		7.6	7.1
Extraction Depth, wt% MAF Coal		69	86
Flow Rates, scfh or gpm			
Slurry to Preheater	FR-223	7.8	8.0
Spray Solvent to Extractor		0.3	0.3
<u>B-A201 Slurry Preheater</u>			
Process Temperatures, °F			
Inlet	XTI-1311-1	115	106
First Section	XTI-1311-20	366	403
Second Section	XTI-1311-21	441	431
Outlet	TRC-206	740	755
Pressures, psig			
Inlet	PR-208-1	490	470
1st Pass Drop	XPDR-1311-1		
2nd Pass Drop	XPDR-1311-2	25	25
3rd Pass Drop	XPDR-1311-3	28	28
Outlet	PR-208-2	405	400
<u>D-A201 Extractor</u>			
*Agitator Speed, rpm	XSI-1800	54	54
<u>Residence Time, Minutes</u>			
Above 700°F		49	58
Above 725°F		33	43
Above 750°F		0	0
Above 775°F		0	0

\*See Figure 4 for Extractor Temperature Profile.

TABLE III  
TYPICAL OPERATING CONDITIONS  
SECTION 300 - SOLIDS SEPARATION

Run Number		011	012
Flow Rates, gpm			
F-B331 Primary Settler Feed	FR-325	10.9	10.7
F-B331 Primary Settler Overflow	FRC-327	7.9	7.5
F-B331 Primary Settler Underflow	FRC-326	3.0	3.2
Wash Solvent to Secondary Settler	FRC-328	2.7	2.2
F-B330 Secondary Settler Overflow	FRC-331	2.8	2.7
F-B330 Secondary Settler Underflow	FRC-323	2.9	2.7
Emulsion to Section 800		1.0	1.0
Temperatures, °F			
F-B332 Primary Feed Tank	TI-1-16	651	646
F-A301 Wash Feed Tank	TI-1-20	580	573
F-B331 Primary Settler	TI-1-46-9	552	544
F-B330 Secondary Settler	TI-1-36	509	506
F-B308 Overflow Surge Tank	TI-1-38	535	524
Solids Concentration, wt%			
F-B331 Primary Settler Feed			6.54
F-B331 Primary Settler Overflow		0.60	0.31
F-B331 Primary Settler Underflow			23.43
F-B330 Secondary Settler Overflow			0.66
F-B330 Secondary Settler Underflow		19.87	22.68
Characteristic Data			
Primary Overflow/Underflow Ratio		2.63	2.34
Secondary Overflow/Underflow Ratio		0.97	1.00
Primary Upflow Velocity, in./min		0.36	0.34
Secondary Upflow Velocity, in./min		0.45	0.44



TABLE IV  
TYPICAL OPERATING CONDITIONS  
SECTION 500 - SOLVENT RECOVERY

Run Number		011	012
Flow Rates, gpm			
Flash Still Recirculation	FRC-502	37.5	39.3
Light Distillate Product	FR-506	0.2	0.0
Recycle Solvent Product	FR-509	6.8	5.4
Extract Product	FRC-507	3.6	
Temperatures, °F			
B-A501 Heater Outlet	TRC-501	520	520
E-A501 Flash Still Vapor	TI-3-7	480	474
E-A501 Flash Still Liquid	TR-4-1	490	480
E-A502 Vacuum Column Overhead Vapor	TI-3-9	418	407
B-A502 Vacuum Column Reboiler Vapor	TR-4-9	447	457
B-A502 Vacuum Column Reboiler Liq.	TI-3-15	465	463
Pressures			
E-A501 Flash Still, psia	PR-502	12	12
E-A502 Column Overhead, psia	PRC-503	12	12
E-A502 Column Differential, in. HG	PDR-508	2	5
Extract Concentration, wt%		50	36
THFI wt%		1.9	0.4

TABLE V  
TYPICAL OPERATING CONDITIONS  
SECTION 600 - EXTRACT HYDROGENATION

Run Number		011	012
Feed Material		Nominal 35% Fresh Extract	25%-45% Fresh Extract
Liquid Flow Rates, gpm @ 60°F			
Feed to D-A601A Hydrogenation Reactor	FR-6037	2.1	4.5
D-A601A Reactor Recirculation	FRC-6005	19.8	19.0
Feed to D-A601D Hydrogenation Reactor	FRC-6013	2.0	2.9
D-A601D Reactor Recirculation	FRC-6006	11.6	21.1
D-A601D Reactor Product	FRC-6014	2.0	2.9
Hydroresidue Product	FRC-712	1.85	3.2
Gas Flow Rates, scfh			
Makeup Hydrogen	FRC-6036	3500	5100
D-A601A Reactor Treat Gas (1)	FRC-6001	14000	14100
D-A601D Reactor Treat Gas (1)	FRC-6002	14000	16000
Sections 600 and 700 Combined Off-Gas	FR-6052	620	1100
Process Temperatures, °F			
B-A602A Reactor Recycle Heater Outlet	TRC-6005	560	655
D-A601A Reactor Inlet	TI-5A-20	552	622
D-A601A Reactor, 5 ft above grid	TR-3-18	558	625
D-A601A Reactor, 17 ft above grid	TR-3-17	558	625
D-A601A Reactor, Vapor Space	TR-3-13	480	591
D-A601A Reactor Exotherm		6	3
B-A602B Reactor Recycle Heater Outlet	TRC-6001	718	770
D-A601D Reactor Inlet	TI-5A-23	690	746
D-A601D Reactor, 5 ft above grid	TR-3-20	725	757
D-A601D Reactor, 17 ft above grid	TR-3-19	730	765
D-A601D Reactor Vapor Space	TR-3-14	653	714
D-A601D Reactor Exotherm		38	15
B-A601 Gas Heater Outlet	TRC-6004	790	785
Pressures, psia (2)			
D-A601A/D Reactor Pressure	PR-6012	1.20RP	1.14RP
Hydrogen Partial Pressure		1.32RH	1.28RH
JC-A601A/B Recycle Compressor			
Differential	DPRC-6019	98	98
Characteristic Data (2)			
Treat Gas Hydrogen Content vol%		88	90
Space Velocity, lb extract/lb catalyst per hr		0.56SV	0.74SV

\*Notes: Numbers in parentheses refer to these comments:

- (1) Includes approximately 4,000 scfh added to the recycle heater.
- (2) Proprietary information is shown as follows: for pressures, read "1.20 RP" as "120% of the reference pressure" and "1.32 RH" as "132% of the reference hydrogen partial pressure." For space velocities, read "1.05 SV" as "105% of the reference space velocity."

TABLE VI  
TYPICAL OPERATING CONDITIONS  
SECTION 700 - PRODUCT FRACTIONATION

Run Number		011	012
Liquid Flow Rates, gpm			
B-A704 Vacuum Column Feed from F-A612*	FRC-712	1.7	2.1
B-A704 Vacuum Column Bottoms	FR-718	1.5	1.5
B-A704 Vacuum Column Overheads	FR-717	2.8	1.7
E-A702 Stabilizer Feed	FRC-701	1.75	2.7
E-A703 Fractionator Feed	FRC-703	1.0	2.6
Naphtha Make	FR-709	0.0	0.2
Cutter Stock Make	FRC-705	0.0	0.01
Donor Solvent Make	FR-708	1.25	2.2
Fuel Oil Product	FR-720	1.5	1.5
Gas Flow Rates, scfh			
F-A710 Economizer Off-Gas	FR-710		344
E-A702 Stabilizer Off-Gas	FR-702		200
Temperatures, °F			
B-A704 Vacuum Column Vapor	TI-3-5	542	425
B-A704 Vacuum Column Liquid	TI-3-35	586	520
E-A702 Stabilizer Overhead	TR-4-10		
B-A702 Stabilizer Reboiler Vapor	TR-4-16	348	389
B-A702 Stabilizer Reboiler Liquid	TIC-702	500	555
E-A703 Fractionator Tray 18	TI-3-24	420	460
B-A703 Fractionator Reboiler Vapor	TIC-704	470	530
Pressures			
B-A704 Vacuum Column Overhead, psia	PR-707	7.5	8.5
F-A710 Economizer Overhead, psig	PRC-705	20	20
E-A702 Stabilizer Overhead, psig	PRC-702	80	81
E-A703 Fractionator Overhead, psig	PRC-703	2.5	2.5
Characteristic Data-			
ASTM D-86 5% Boiling Point, °F			
Donor Solvent			450
Fuel Oil		465	600
API Gravity, degrees			
Donor Solvent			15.9
Fuel Oil		8.6	7.4 to -3.5
Fuel Oil Analyses			
Pour Point, °F		<-30	45 to 122
Viscosity, centipoise at 122°F		5.3	300 to 3800
Solids Content, wt%			0.4
Sulfur Content, wt%		0.55	0.5

\*Does not include unmetered liquid feed from F-A617.

TABLE VII  
TYPICAL OPERATION CONDITIONS  
SECTION 800 - CARBONIZATION

Run Number		011	012
Gas Flow Rates, scfh			
Recycle Gas	FRC-809	8,300	10,200
Combustion Air	FRC-801	4,500	3,900
Fluidizing Gas	FR-830	11,800	14,100
Liquid Flow Rates, gpm			
Feed Rate	FRC-323	3.0	2.5
Return to Section 300*	FR-833	3.3	3.3
Temperatures, °F			
D-A801 Carbonizer Top	TI-1-26	660	570
D-A801 Carbonizer Upper Bed	TR-2-8	840	720
D-A801 Carbonizer Lower Bed	TI-1-23	840	720
D-A801 Carbonizer Grid Plate	TR-1-21	840	760
D-A801 Carbonizer Pressure, psig		3.0	4.0
Characteristic Data for this Run			
Feed Concentration, wt% solids		20	25
Tar Char Slurry, wt% solids		5	10

\*Additional solvent added to Tar Quench System for ease of solids handling.

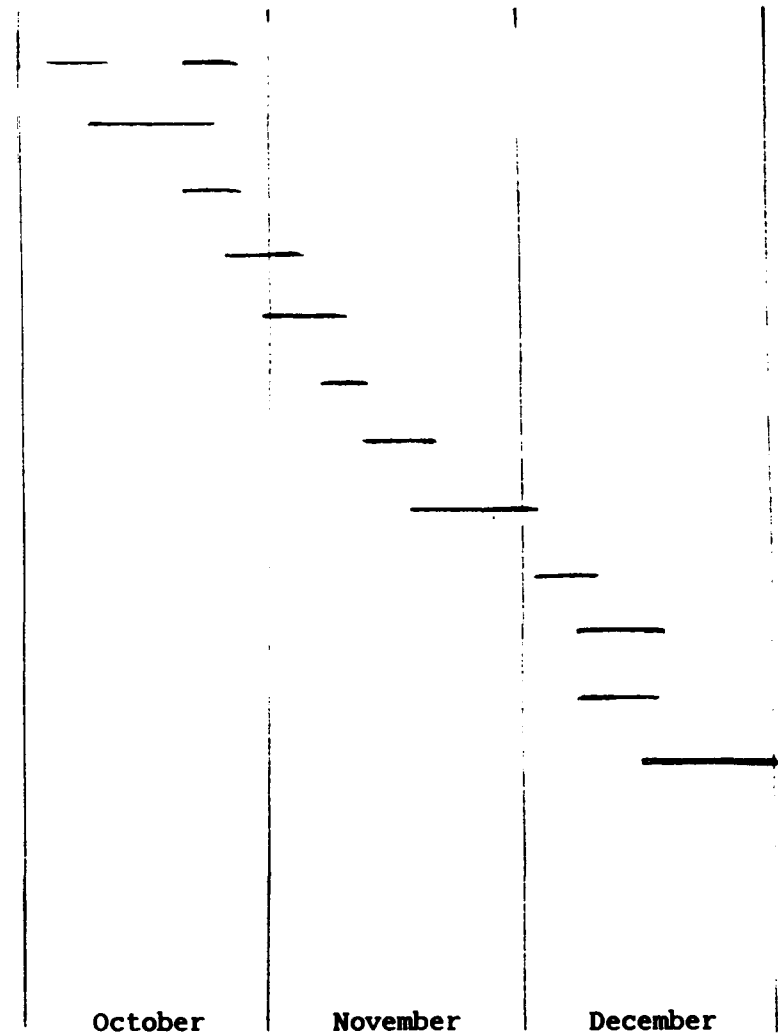
**TABLE VIII**  
**CAPILLARY G.C. ANALYSIS REPORT**

Sample I.D.		Recycle Solvent			Recycle Solvent		Donor Solvent		Donor Solvent	
Sample Point		36			36		58		58	
Section		500			500		700		700	
Sample Number		11059			12158		11066		12488	
Date Sampled		Run 012			Run 013		Run 012		Run 013	
#	Compound	B.P. (F)	Wt. %	Cum.Wt. %	Wt. %	Cum.Wt. %	Wt. %	Cum.Wt. %	Wt. %	Cum.Wt. %
	Unknowns				0.06					
1	(2,3)-Benzofuran	345			-					
	Unknowns				-					
2	Indane	349			-					
	Unknowns				0.26	0.32				
3	Trans-Decalin				-					
	Unknowns		1.08		-		0.02			
4	Cis-Decalin	381	0.76	1.84	-		0.10	0.12		
	Unknowns		0.74	2.58	3.74	4.06	3.08	3.20		
5	(1,2)-Dihydronaphthalene	405			-					
	and/or(1,2,3,4)-Tetralin	405			-					
	Unknowns		2.93	5.51	1.55	5.61	0.64	3.84	0.23	
6	Naphthalene	424	4.45	9.96	3.19	8.80	1.61	5.45	1.50	1.73
	Unknowns		9.72	19.68	23.04	31.84	25.17	30.62	6.53	8.26
7	2-MN	466	18.72	38.40	15.79	47.63	8.58	39.20	3.09	11.35
8	1-MN	469	15.35	53.75	13.62	61.25	11.61	50.81	8.26	19.61
	Unknowns		3.45	57.20	5.37	66.62	6.82	57.63	5.37	24.98
9	Biphenyl	493			0.45	67.07	0.67	58.30	0.64	25.62
	Unknowns				-				0.74	26.36
10	(1+2)-EN	496	5.71	60.91	5.93	73.00	7.39	65.69	8.23	34.59
11	(2,6)-DMN	504	4.25	67.16	3.63	76.63	2.79	68.48	3.32	37.91
12	(1,7 + 2,7)-DMN	504	3.27	70.43	2.83	79.46	2.77	71.25	3.35	41.26
13	(1,3 + 1,6)-DMN	505	7.25	77.68	6.01	85.47	6.86	78.11	7.67	48.93
14	(1,4 + 1,5 + 2,3)-DMN	516	3.30	80.78	2.47	87.94	3.00	81.11	2.96	51.89
15	(1,2 + 1,8)-DMN		2.15	83.13	1.12	89.06	1.52	82.63	1.64	53.53
	Unknowns		1.21	84.34	1.24	90.30	1.71	84.34	2.89	56.42
16	Acenaphthene	534	1.96	86.30	-				-	
	Unknowns				1.95	92.25	2.34	86.68	5.19	61.61
17	Dibenzofuran	547	1.30	87.60	1.01	93.26			2.21	63.82
	Unknowns		12.40	100	1.36	94.62	3.24	89.92	3.41	67.23
18	(2,3,5)-TMN				1.09	95.71	1.57	91.49	2.74	69.97
	Unknowns				1.16	96.87	1.76	93.25	2.90	72.87
19	Fluorene	568			-		0.55	93.80	1.97	74.84
	Unknowns				2.21	99.08	6.20	100	12.76	87.60
20	(9,10)-Dihydroanthracene	594			-				-	
	Unknowns				-				-	
21	(9,10)-Dihydrophenanthrene				0.14	99.22			-	
	Unknowns				0.78	100			11.07	98.67
22	Dibenzothiophene	631								
	Unknowns									
23	Anthracene	644							1.33	100
	Unknowns									

FIGURE 1

FOURTH QUARTER PLAN, 1978

Heavy Fuel Oil Heating Test  
Solvents Recovery  
B-B304 Heat Improvement Test  
Slurry Concentration Test  
Settler Upflow Velocity Test  
Solvents Recovery  
Section 600 Shakedown  
Demonstrate Integrated Operation  
Instrument and Mechanical Check  
Hydrogenate Stored Extract  
Solvents Recovery  
Integrated Operation



CHEMPRO - SEALOL  
TYPE 602

SEAL APPLICATION

L-B 205 MIXER

SK. # 93  
FULL SCALE  
DATE : 10-24-78

- ① SPRING LOADED PACKING ASSEMBLY
- ② S.S. SPACER
- ③ SHROUD ASSEMBLY
- ④ BELLOWS

- ⑤ ROTARY FACE
- ⑥ STATIONARY FACE
- ⑦ AUXILIARY PACKING

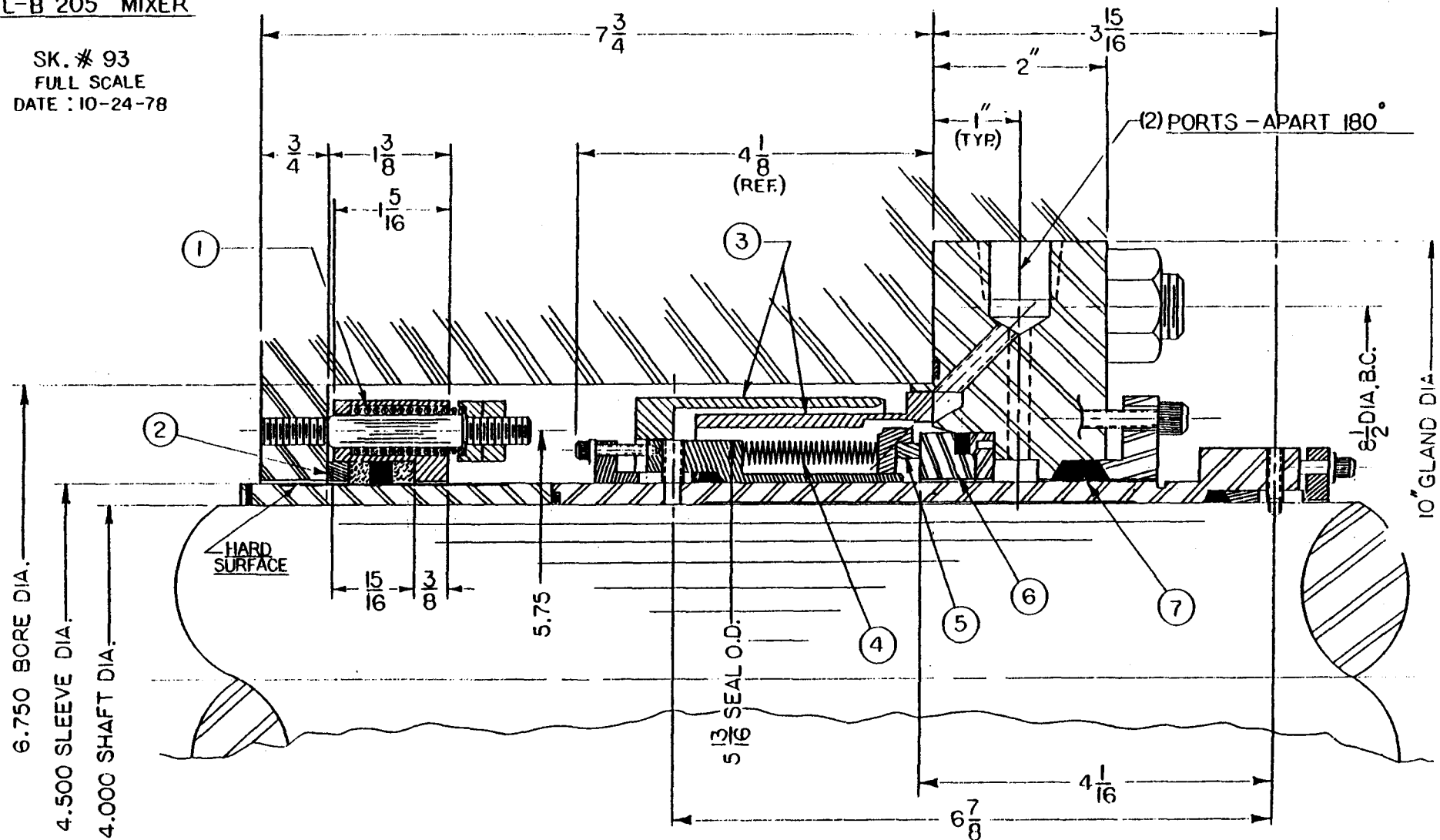


FIGURE 2

RUN 013

PROCESS REPORT

Covering the period of  
July 25 to August 11, 1978



## INTRODUCTION

Run 013, covering the period of July 29 to August 14, 1978, was the fifth fully integrated operation and the most successful run to date. Fresh extract was hydrogenated for 142 continuous hours, more than twice as much as had been logged in all the previous runs combined.

The goals for Run 013 were:

1. Develop the ability to switch from stored to fresh extract feed in the Hydrogenation Section, making this section somewhat independent of the Extraction Section.
2. Run the integrated plant as long as possible.
3. Develop a data base from which operating parameters could be varied during later runs to improve extract conversion and desulfurization.

Attention was focused on the performance of the Hydrogenation Section, so conditions were held steady in the Extraction Section.

Catalyst was charged in only the second hydrogenation reactor. The first reactor was used as a feed preheater and stabilizer. The average temperature of the second reactor was varied from 720°F to 833°F. Extract feed rates were 2.2 to 3.5 gpm. Extract concentration in the feed ranged from 20 to 58 weight percent.

At an average reactor temperature of 805°F, 53 percent conversion was achieved. (That is, 53 percent of the material boiling over 880°F was converted to fractions boiling below 880°F.) The amount of benzene insolubles in the +880°F material also fell sharply between feed and product.

Plans for future runs include varying the feedrate and extract concentration to the Hydrogenation Section to study the effect on conversion and desulfurization.

The effect of temperature on conversion and product yield structure will also be investigated. The ability of the Extraction Section to handle a wider boiling range donor solvent will also be investigated. A piping modification will be made in Section 300 to increase the settler temperature and to improve settler performance.

## DISCUSSION OF EXTRACTION PERFORMANCE

### Operating Data

Coal was fed at the rate of 11 tons per day in a 20 percent slurry. About 80 percent of the organic material was removed from the coal at an average extractor temperature of 730°F and extractor residence time of 39 minutes. Operations were targeted for a 0.4:1.0 donor solvent-to-MAF coal ratio, but this ratio was not achieved because of a recycle solvent shortage at the beginning of the run which led to a juggling of solvent feeds and a widely varying donor solvent-to-coal ratio. The primary settler temperature stayed between 540°F and 570°F. Upflow velocity in the primary settler was 0.51 inches per minute. The overflow contained from 0.24 to 0.4 weight percent solids, representing a 95 percent solids rejection. Extract recovery was 91 percent.

3.4 tons of char per day was produced in the Carbonization Section.

### Material Balancing

An elemental balance for the Extraction Section is given in Figure 1. Flow rates are based on weigh tank readings in Section 200 and flow meter readings in Section 300. Char production was determined by ash content of the char. The overflow to Section 500 was fixed by the Section 500 material balance. Elemental compositions are based on laboratory analyses of the coal, solvent, char, off-gases, emulsions, and some of the intermediate slurry streams. The laboratory technique determines oxygen by difference. The balance was closed by forcing the compositions of the underflow from Section 300 to Section 800 and the off-gases from Section 800.

### Slurry Preheater

A typical temperature profile for the B-A201 slurry preheater is shown in Figure 3. It is similar to those of previous runs. The temperature profile of the process fluid flattens in the second pass of the preheater after several hours of slurry operation and maintains this appearance until Section 200 is returned to solvent feed. Pressure drop across the preheater varied from 80 psi (at 8 gpm) to 120 psi (at 9.5 gpm), similar to previous runs. No significant increase in pressure drop was experienced at any given flow rate during the run. There was no evidence of coking, even though the outlet temperature got as high as 765°F and one of the tubeskin thermocouples reached 875°F.

### Extractor

The average extractor temperature went from 700°F at the beginning of Run 013 to 750°F toward the end. The temperature was increased by raising the set point of the extractor strip heaters and by raising the slurry preheater outlet temperature. A typical temperature profile for the extractor is shown in the Appendix, Figure 4.

The depth of extraction, based on ash on solids in the extractor effluent, varied from 67 percent to 91 percent. The average of 11 samples was 80 percent. Figure 2 in the Appendix is a plot of the extraction parameters (extractor residence time, average temperature, and donor solvent-to-coal ratio) with the depth of extraction for the entire run. No cause-effect relationship is apparent from these data. High depths of extraction were obtained every time the donor solvent-to-coal ratio was high, but high depths of extraction were also observed when donor solvent feed was low or nil. It should be pointed out that donor solvent is differentiated from recycle solvent by the fact that the donor solvent has just been through 600 hydrogenation, while recycle solvent has made at least one pass through extraction. Recycle solvent does retain hydrogen donor capability for several cycles, however.

### Settler

The temperature of the F-B331 primary settler has continually been 30°F to 60°F below the design value of 600°F. Although the settler has performed well, we plan to improve its performance by relocating the B-B304 secondary slurry heater to the recirculation line at the F-A301 wash stage feed tank.

### Carbonizer

A plugged cyclone dip leg caused high solids carryover from the D-A801 carbonizer, resulting in plugging in the bottom of the F-B331 primary settler. The dip leg was probably plugged at start-up, but the reason for the plug is not known. More attention will be given to the carbonizer operating parameters in the future to determine the cause of this recurring problem.

The depth of extraction, based on ash on solids in the extractor effluent, varied from 67 percent to 91 percent. The average of 11 samples was 80 percent. Figure 2 in the Appendix is a plot of the extraction parameters (extractor residence time, average temperature, and donor solvent-to-coal ratio) with the depth of extraction for the entire run. No cause-effect relationship is apparent from these data. High depths of extraction were obtained every time the donor solvent-to-coal ratio was high, but high depths of extraction were also observed when donor solvent feed was low or nil. It should be pointed out that donor solvent is differentiated from recycle solvent by the fact that the donor solvent has just been through 600 hydrogenation, while recycle solvent has made at least one pass through extraction. Recycle solvent does retain hydrogen donor capability for several cycles, however.

### Settler

The temperature of the F-B331 primary settler has continually been 30°F to 60°F below the design value of 600°F. Although the settler has performed well, we plan to improve its performance by relocating the B-B304 secondary slurry heater to the recirculation line at the F-A301 wash stage feed tank.

### Carbonizer

A plugged cyclone dip leg caused high solids carryover from the D-A801 carbonizer, resulting in plugging in the bottom of the F-B331 primary settler. The dip leg was probably plugged at start-up, but the reason for the plug is not known. More attention will be given to the carbonizer operating parameters in the future to determine the cause of this recurring problem.

## DISCUSSION OF HYDROGENATION PERFORMANCE

During Run 013, feedstock to the Hydrogenation Sections was smoothly switched from stored extract to fresh extract and back to stored extract twice. Operation of the Solvent Recovery Section was routine during these periods of adjustment, as flash still bottoms extract concentration was varied from 20 percent to 58 percent. Control of the flash still bottoms concentration was generally  $\pm 10$  percent of the desired value. Tables IV, V and VI present typical operating conditions in the Solvent Recovery Section, Hydrogenation Section, and Product Fractionation Section, respectively. The first column of data is for fresh extract; the second column is for previously hydrogenated material.

The average reactor temperature was adjusted during the run from 720°F to 833°F to determine the operating stability of the reactor at various temperatures. A graph of reactor average operating temperatures is shown in Figure 5. Fresh extract feed was hydrogenated at reactor average temperatures ranging from 780°F to 820°F. Previously hydrogenated material, however, was hydrogenated at a reactor average temperature ranging up to 833°F because of its reduced susceptibility to hydrogenation. Typical reactor exotherms observed while hydrotreating fresh extract and previously hydrogenated material were 35°F and 18°F, respectively. The higher sulfur content of fresh extract feed accounted for the higher reaction exotherm during the processing of fresh extract. The feasibility of preheating nonhydrogenated extract was tested using the first reactor's recycle heater. A mixture of fresh extract and hydrogen was heated as high as 680°F during the run with no indication of heater coking. Figure 5 shows that preheated extract feed temperatures ranged from 610°F to 665°F in the first reactor, D-A601A.

Figure 6 tracks the sulfur content of feed and heavy fuel oil product throughout the run. Sulfur content of extract feed ranged from 0.21 percent to 1.22 percent. The heavy fuel oil product sulfur content ranged from 0.07 percent to 0.61 percent. The wide variations in sulfur content of the feed and product streams are due to variations in extract concentration and the processing of previously hydrogenated material whose sulfur content averaged 0.28 percent. The observed level of denitrogenation was 68.6 percent.

### Material Balancing

Material balancing in the Hydrogenation Section is based on flow meter readings of the feed and product streams. The procedure also balances the elements carbon, hydrogen, nitrogen, sulfur and oxygen (by difference). Figure 7 provides the Hydrogenation Section elemental and mass balance during the material period. The elemental balance is based on elemental analyses of feed and product streams and was closed by balancing the unmetered sour water (containing hydrogen sulfide and ammonia) production against the disappearance of oxygen, nitrogen, and sulfur. Carbon and hydrogen were balanced against the production of light hydrocarbon gases during hydrotreating.

### Nitrogen, Oxygen, Ash Removal

At an average reactor temperature of 805°F and pressure of 1.20 RP\*, 41.7 percent fresh extract at a feedrate of 2.52 gpm (1.62 SV\*) was hydrogenated with the following levels of denitrogenation and deoxygenation.

#### Nitrogen Removal, % of Feed

Total Liquid Product	68.6
----------------------	------

#### Oxygen Removal, % of Feed

Total Liquid Product	73.9
----------------------	------

#### Ash Removal, (%)

Total Liquid Product	80.0
----------------------	------

The observed nitrogen and oxygen removal approximates that reported by licensor when processing solvent refined coal (SRC) materials. The licensor also observed ash removal during a catalyst aging study. No catalyst deactivation was reported relating to the ash removal.

Future runs will determine the effects of reactor space velocity, reactor temperature and extract feed concentration on the levels of nitrogen, oxygen and ash removal.

### Conversion and Desulfurization

At an average temperature of 805°F, the disappearance of +880°F material on fresh feed during the material balance period was 52.9 percent and the sulfur removal from the total feed was 90.3 percent. Desulfurization of the +880°F fraction of the fresh feed was 88 percent on the first pass. Because the operation used only one reactor charged with catalyst, two stage hydrogenation was simulated by reprocessing the heavy fuel oil product. At an average reactor temperature of 833°F, the approximate levels of conversion and desulfurization of the previously hydrogenated material were 67 percent and 88 percent, respectively. This produced a final heavy fuel oil product with 0.07 percent sulfur content. Run 013 has provided a set of base operating conditions on fresh extract feed. The effects of changes in operating severity on the levels of conversion and desulfurization will be studied in future runs.

### Yield Structure

A typical product yield as a percentage of feed is shown on Table VII. This table is based on analyses of composite feed and total liquid product (TLP) samples taken during the material balance period. Since total liquid product

\*RP and SV refer to operating system reference pressure and reference space velocity.

samples are not available directly from the Hydrogenation Section, a composite total liquid product sample was prepared by mixing naphtha, middle distillate, donor solvent, and heavy fuel oil product in the material balance ratios. For the purposes of analyses, the licensors considers extract to be +850°F material instead of +880°F material. The yield data are, therefore, presented in a manner consistent with the lummus analytical procedures. The production of C<sub>1</sub>-300°F naphtha was 5.5 weight percent. Conversion of +850°F material into 650°F to 850°F heavy distillate was 18.6 weight percent. The yield structure compares closely to the licensors's results from hydrotreating 37 weight percent SRC material at 805°F. Hydrogen consumption was 1,250 scf/bbl of extract feed. This corresponds to a hydrogen consumption of 2.75 lbs/100 lbs coal feed.

Table VIII presents comparative data between the extract feed and heavy fuel oil product. Elemental analyses are presented on both the -880°F and +880°F fractions of the feed and product streams. Distillation information on the -880°F fractions of the feed and product streams is also shown. The product analyses in Table VIII apply only to the heavy fuel oil product and should not be construed as representative of the total liquid product from the hydrogenation section. Heavy fuel oil product from fresh extract had a sulfur content 0.2 percent, an ash content below 0.05 percent, and pour point equal to or lower than 50°F. Target specifications were less than 0.5 percent sulfur, less than 0.2 percent ash and a pour point of 60°F maximum. Heavy fuel oil product yield was three barrels per ton of coal processed.

Table IX presents elemental analyses and distillation data on the recycle solvent and donor solvent products. Progress has been made toward widening the boiling range of donor solvent by refractionating the heavy fuel oil product after completion of Run 013. A wider boiling range solvent will improve the controllability of the Solvent Recovery Section and reduce the fluctuations of extract concentration in the feed to the Hydrogenation Section. During the run, a considerable quantity of donor solvent was sent to tankage with product fuel oil because it could not be flashed in the vacuum column reboiler. Donor solvent recovery in the product fractionation section is expected to improve during the next run because of a duty reduction on the vacuum column reboiler. A light distillate stream from the hydrogenation section has been diverted from the vacuum column reboiler to the economizer to effect this.

#### Future Plans

The next run will replicate the extract feed concentration and reactor space velocity of this run. Material balance periods will be taken at three different average reactor temperatures. The goal is to determine a relationship between reactor temperature, extract conversion and product yield structure. The ultimate objective of future work will be to determine the range of operating conditions which will provide solvent balance to the process.

## APPENDIX

### TABLES

- I. Typical Operating Conditions, Section 200 - Coal Extraction
- II. Typical Operating Conditions, Section 300 - Solids Separation
- III. Typical Operating Conditions, Section 800 - Carbonization
- IV. Typical Operating Conditions, Section 500 - Solvent Recovery
- V. Typical Operating Conditions, Section 600 - Extract Hydrogenation
- VI. Typical Operating Conditions, Section 700 - Product Fractionation
- VII. Yield Data as Percentage of Feed
- VIII. Feed and Product Analyses
- IX. Recycle Solvent and Donor Solvent Analyses
- X. Typical Coal Analysis
- XI. D-1160 Distillation Total Liquid Feed to Hydrogenation
- XII. D-1160 Distillation Total Liquid Product

### FIGURES

- 1. Material Balance, Extraction Sections
- 2. Extraction Process Parameters vs. Time
- 3. Typical Temperature Profile, Slurry Preheater
- 4. Typical Temperature Profile, Extractor
- 5. Reactor Average Temperature
- 6. Feed/Product Sulfur Content
- 7. Material Balance, Hydrogenation Sections



TABLE I  
TYPICAL OPERATING CONDITIONS  
SECTION 200 - COAL EXTRACTION

Run Number		013
Coal Feed Rate, tpd		11.4
Coal Concentration, wt% in B-A201 Feed		20.4
Donor Solvent to MAF Coal Ratio		0.0
Extraction Depth, wt% MAF Coal		76
Flow Rates, scfh or gpm @ 60°F		
Slurry to Preheater		8.9
Spray Solvent to Extractor		0.5
Overhead Condensate	FR-202	0.3
Overhead Gas	FR-201	650
<u>B-A201 Slurry Preheater</u>		
Process Temperatures, °F		
Inlet		135
First Section		398
Second Section		462
Outlet		759
Pressures, psig		
Inlet		518
Outlet		419
<u>D-A201 Extractor</u>		
Agitator Speed, rpm		54
Average Slurry Temperature in D-A201, °F		734
Residence Time, Minutes		35

TABLE II  
TYPICAL OPERATING CONDITIONS  
SECTION 300 - SOLIDS SEPARATION

Run Number		013
Flow Rates, gpm @ 60°F		
F-B331 Primary Settler Feed	FRC-325	15.6
F-B331 Primary Settler Overflow	FRC-327	11.1
F-B331 Primary Settler Underflow	FRC-326	4.5
Wash Solvent to Secondary Settler	FRC-328	2.4
F-B330 Secondary Settler Underflow	FRC-323	2.7
Temperatures, °F		
F-B332 Primary Feed Tank	TI-1-16	652
F-B331 Primary Settler	TI-1-46-9	566
F-B330 Secondary Settler	TI-1-36	558
Pressure in F-B331 Primary Settler, psig	PRC-330	165
Solids Concentration, wt%		
F-B331 Primary Settler Overflow		0.24
F-B330 Secondary Settler Underflow		36.3
Characteristic Data		
Primary Overflow/Underflow Ratio		2.47
Primary Upflow Velocity, in./min		0.51
Extract Recovery, wt%		91

TABLE III  
TYPICAL OPERATING CONDITIONS  
SECTION 800 - CARBONIZATION

Run Number		013
Gas Flow Rates, scfh		
Gas to Lift Leg	FRC-831	2000
Combustion Air from JC-A801	FRC-801	4400
Plant Air	FI-828	2600
Fluidizing Gas (1)	FR-830	10200
Liquid Flow Rates, gpm @ 60°F		
Feed Rate	FRC-323	2.7
Solvent Make Rate	FR-833	3.5
Sour Water Make Rate	FR-843	<0.1
Temperatures, °F		
D-A801 Carbonizer Top	TI-1-26	662
D-A801 Carbonizer Upper Bed	TR-2-8	885
D-A801 Carbonizer Lower Bed	TI-1-23	885
D-A801 Carbonizer Grid Plate, West	TR-1-19	785
South	TR-1-20	610
East	TR-1-21	885
North	TR-1-22	850
Northwest	TR-1-23	665
D-A801 Carbonizer Upper Lift Leg	TI-1-48-20	880
D-A801 Carbonizer Lower Lift Leg	TI-1-48-19	880
Fluidizing Gas	TR-2-7	120
E-A801 Spray Tower	TI-1-30	290
F-A811 Solvent Separator	TI-48-3	230
D-A801 Carbonizer Pressure, psig		1.8
Characteristic Data for this Run		
Average Bed Particle Diameter, in.		.008
Minus 325 Mesh Content, wt%		1.0
Feed Concentration, wt% solids		36
Tar Char Slurry, wt% solids		15

Notes: (1) Fluidizing gas flow includes combustion air from JC-A801, plant air and recycle gas.

TABLE IV  
TYPICAL OPERATING CONDITIONS  
SECTION 500 - SOLVENT RECOVERY

Run Number		013	013
Feed Material		Fresh Extract	Rerun Product
Flow Rates, gpm @ 60°F			
Feed From Section 300	FRC-501	11.0	2.7
Flash Still Recirculation	FRC-502	44	42
Light Distillate Product	FR-506	0.2	0
Recycle Solvent Product	FR-509	8.4	0
Extract Product	FRC-507	2.5	2.6
Temperatures, °F			
B-A501 Heater Outlet	FRC-501	540	560
E-A501 Flash Still Vapor	TI-3-7	472	490
E-A501 Flash Still Liquid	TR-4-1	490	550
E-A502 Vacuum Column Overhead Vapor	TI-3-9	412	94*
B-A502 Vacuum Column Reboiler Vapor	TIC-502	440	430
B-A502 Vacuum Column Reboiler Liquid	TI-3-15	454	452
Pressures			
E-A501 Flash Still Pressure, psia	PR-502		
E-A502 Vacuum Column Overhead, psia	PRC-503	12.2	12.0
E-A502 Vacuum Column Differential, in. HG	PDR-508	1	**
880°F+ Concentration, wt%		41.7	50.9
THF Insoluble, wt%		0.37	0.15

\*No overhead cut was taken during the reheating of rerun product.

\*\*Instrument inoperative at this time.

**TABLE V**  
**TYPICAL OPERATING CONDITIONS**  
**SECTION 600 - EXTRACT HYDROGENATION**

Run Number		013	013
Feed Material		40% Fresh Extract	45% Rerun Product
Liquid Flow Rates, gpm @ 60°F			
Feed to D-A601A Hydrogenation Reactor	FR-6037	2.6	2.6
D-A601A Reactor Recirculation	FRC-6005	19	20
Feed to D-A601D Hydrogenation Reactor	FRC-6013	2.2	2.2
D-A601D Reactor Recirculation	FRC-6006	24	23.5
D-A601D Reactor Product	FRC-6014	2.0	1.2
Hydroresidue Product	FRC-712	1.1	0.9
Gas Flow Rates, scfh			
Makeup Hydrogen	FRC-6036	8000	3500
D-A601A Reactor Treat Gas (1)	FRC-6001	16000	12500
D-A601D Reactor Treat Gas (1)	FRC-6002	19750	14750
Sections 600 and 700 Combined Off-Gas	FR-6052	4000	3200
Process Temperatures, °F			
B-A602A Reactor Recycle Heater Outlet	TRC-6005	680	700
D-A601A Reactor Inlet	TR-3-15	635	650
D-A601A Reactor, 5 ft above grid	TR-3-18	630	640
D-A601A Reactor, 17 ft above grid	TR-3-17	630	640
D-A601A Reactor Vapor Space	TR-3-13	604	630
D-A601A Reactor Exotherm		-5	-7
B-A602B Reactor Recycle Heater Outlet	TRC-6001	810	850
D-A601D Reactor Inlet	TR-3-16	770	810
D-A601D Reactor, 5 ft above grid	TR-3-20	795	825
D-A601D Reactor, 17 ft above grid	TR-3-19	815	830
D-A601D Reactor Vapor Space	TR-3-14	815	800
D-A601D Reactor Exotherm		35	18
B-A601 Gas Heater Outlet	TRC-6004	860	850
Pressures, psia (2)			
D-A601A/D Reactor Pressure	PR-6012	1.20RP	1.20RP
Hydrogen Partial Pressure		1.28RH	1.14RH
JC-A601A/B Recycle Compressor Differential	DPRC-6019	100	100
Characteristic Data (2)			
Feed Gravity, API		-2.0	5.8
Feed Sulfur Content, wt%		1.11	0.24
Treat Gas Hydrogen Content, vol%		85.0	76.0
Space Velocity, lb feed/lb catalyst per hr		1.62SV	1.65SV

\*Notes: Numbers in parentheses refer to these comments:

- (1) Includes approximately 4,000 scfh added to the process inlet of recycle heater.
- (2) Proprietary information RD, RH, and SV should be read as multiplier factors.

**TABLE VI**  
**TYPICAL OPERATING CONDITIONS**  
**SECTION 700 - PRODUCT FRACTIONATION**

Run Number		013	013
Feed Material		Fresh	Rerun
<b>Liquid Flow Rates, gpm @ 60°F</b>			
B-A704 Vacuum Column Feed from F-A612	FRC-712	1.1	0.9
B-A704 Vacuum Column Bottoms	FR-718	1.0	1.3
B-A704 Vacuum Column Overheads	FR-717	1.6	1.1
E-A702 Stabilizer Feed	FRC-701	1.8	1.1
E-A703 Fractionator Feed	FRC-703	1.9	1.4
Naphtha Make	FR-709	0.5	0.2
Middle Distillate Make	FRC-705	0.1	0.4
Donor Solvent Make	FR-708	1.2	0.7
Fuel Oil Product	FR-720	1.0	1.3
<b>Gas Flow Rates, scfh</b>			
F-A710 Economizer Off-Gas	FR-710	300	300
E-A702 Stabilizer Off-Gas	FR-702	184	123
<b>Temperatures, °F</b>			
B-A704 Vacuum Column Vapor	TI-3-5	484	600
E-A702 Stabilizer Overhead	TR-3-20	276	256
B-A704 Vacuum Column Liquid	TI-3-35	538	620
E-A702 Stabilizer Tray 9	TI-3-18	532	332
B-A702 Stabilizer Bottoms	TI-3-19	580	440
B-A702 Stabilizer Reboiler Vapor	TR-4-16	600	560
B-A702 Stabilizer Reboiler Liquid	TIC-702	630	560
E-A703 Fractionator Overhead	TI-3-27	290	260
E-A703 Fractionator Tray 18	TI-3-24	450	460
B-A703 Fractionator Reboiler Vapor	TIC-704	530	570
<b>Pressures</b>			
B-A704 Vacuum Column Overhead, psia	PR-707	9.8	9.8
F-A710 Economizer Overhead, psig	PRC-705	20	20
E-A702 Stabilizer Overhead, psig	PRC-702	82	82
E-A703 Fractionator Overhead, psig	PRC-703	3	1.5
<b>Characteristic Data</b>			
ASTM D-86 5% Boiling Point, °F			
Naphtha		228	208
Middle Distillate		235	360
Donor Solvent		451	511
Fuel Oil		490	422
<b>API Gravity, degrees</b>			
Naphtha		44.9	48.2
Middle Distillate		23.3	21.0
Donor Solvent		15.3	15.1
Fuel Oil		2.7	6.6
<b>Fuel Oil Analyses</b>			
Pour Point, °F		33	30
Viscosity, centipoise at 122°F		222	
THF Insoluble, wt%		0.08	0.38
Ash Content of Stream, wt%		0.03	0.35
Sulfur Content, wt%		0.02	0.07

TABLE VII  
YIELD DATA AS PERCENTAGE OF FEED

Run Number	013
Feed	Fresh Extract
wt% -850°F	69.1
wt% +850°F	30.9
Average Reactor Temperature, °F	805
Space Velocity (times SV)	1.62
Conversion, of +850°F	56.7
Yield, wt% of Feed Based on Total Liquid	
H <sub>2</sub> O	2.1
H <sub>2</sub> S	1.0
NH <sub>3</sub>	0.4
C <sub>1</sub> - 300°F	5.5
300 - 500°F	45.6
500 - 650°F	13.5
650 - 850°F	18.6
+850°F	13.4
Total	100.5
H <sub>2</sub> Consumption (scf/bbl)	1250

Note: Yield data are calculated from licensor's analyses of composite feed and total liquid product samples taken during material balance period. Licensor uses 850°F as the solvent end-point instead of 880°F.

TABLE VIII  
FEED AND PRODUCT ANALYSES

<u>Extract Feed</u>		<u>Heavy Fuel Oil Product</u>	
Run 013		Run 013	
Wt% -880°F	58.0	Wt% -880°F	50.0
Wt% +880°F	42.7	Wt% +880°F	49.0
Wt% THFI	0.3	Wt% THFI	0.08
Gravity Sp 60/60°	1.093	Gravity Sp 60/60°F	1.054
		Pour Point, °F	33
		Viscosity @ 122°F cps	222
<u>-880°F</u>		<u>-880°F</u>	
Elemental Content wt%		Elemental Content wt%	
Carbon	91.23	Carbon	89.54
Hydrogen	8.63	Hydrogen	8.43
Nitrogen	0.05	Nitrogen	0.18
Oxygen*	-	Oxygen*	1.74
Sulfur	0.09	Sulfur	0.12
<u>Distillation, °F (D-1160)**</u>		<u>Distillation, °F (D-1160)**</u>	
IBP	255	IBP	329
5	460	5	490
10	468	10	500
30	479	30	548
50	493	50	636
70	506	70	691
90	560	86	750
Final Temperature, °F	589	Final Temperature, °F	760
Distillate Recovered, vol%	97	Distillate Recovered, vol%	94
Loss, vol%	3	Loss, vol%	6
<u>+880°F</u>		<u>+880°F</u>	
Elemental Content wt%		Elemental Content wt%	
Carbon	83.90	Carbon	90.08
Hydrogen	6.10	Hydrogen	7.24
Nitrogen	1.16	Nitrogen	0.75
Oxygen*	6.22	Oxygen*	1.60
Sulfur	2.53	Sulfur	0.30
Ash	0.09	Ash	0.04
		Btu/lb	17209

\*All oxygen calculated by difference.

\*\*D-1160 corrected to 1 ATM BP.

Note: See Tables XI and XII for Feed and Product Distillation data.



**TABLE IX**  
**RECYCLE SOLVENT AND DONOR SOLVENT ANALYSES**

	<u>Recycle Solvent</u>	<u>Donor Solvent</u>
Run 013		
Gravity Sp 60/60°F	14.6	15.3
Elemental Content wt%		
Carbon	90.94	90.64
Hydrogen	8.99	9.34
Nitrogen	0.07	-
Oxygen	-	-
Sulfur	-	-
Distillation, °F (D-1160)		
IBP	435	410
5%	448	451
10%	451	454
30%	457	464
50%	468	475
70%	484	495
90%	503	610
95%	538	661
Final Temperature, °F	569	661
Distillate Recovered, vol%	99	98
Loss, vol%	1	2

TABLE X  
TYPICAL COAL ANALYSIS

Proximate, wt%, as received

Volatile Matter	41.71
Fixed Carbon	47.37
Ash	9.52
Moisture	1.40

Ultimate, wt%, dry

Carbon	71.74
Hydrogen	5.09
Nitrogen	1.13
Oxygen (by difference)	7.64
Sulfur	4.88
Ash	9.52

Sieve Analysis, Cumulative wt% Retained

8 Tyler Mesh	0.0
14	0.0
28	0.5
48	6.5
100	21.5
200	51.5
325	74.0
-325, wt%	26.0

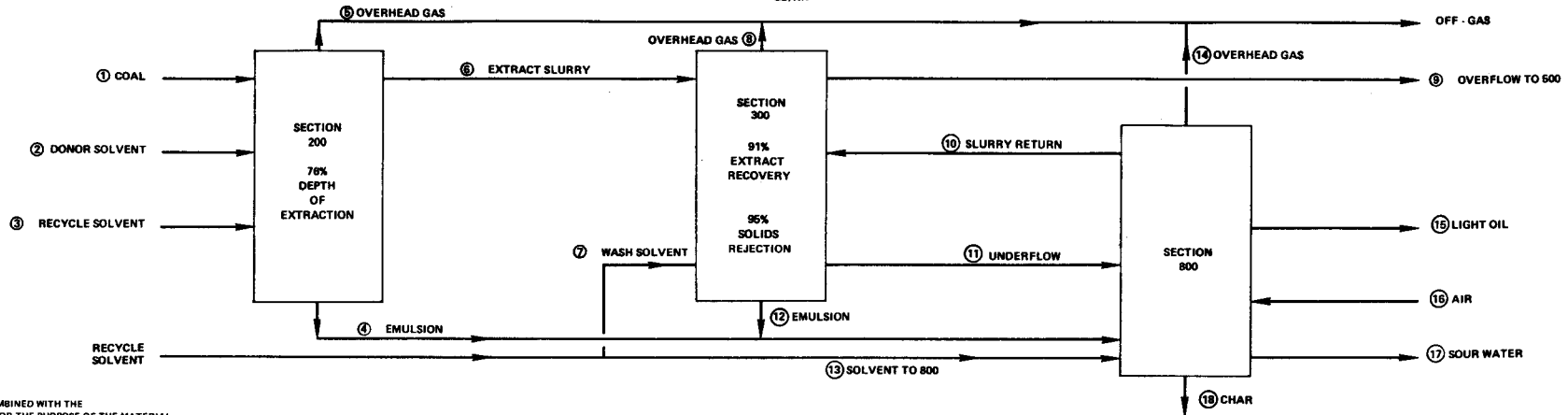
TABLE XI  
D-1160 DISTILLATION  
TOTAL LIQUID FEED TO HYDROGENATION

<u>Volume %</u>	<u>Overhead Vapor Temperature °F Corrected to 760 mm Hg</u>
1BP	455
1	458
2	461
5	466
7	469
10	471
15	474
20	480
25	484
30	490
35	496
37.2	500
45	512
50	523
55	543
60	570
65	622
67.2	650
70.8	699
78.8	850

TABLE XII  
D-1160 DISTILLATION  
TOTAL LIQUID PRODUCT

<u>Volume %</u>	<u>Overhead Vapor Temperature °F</u> <u>Corrected to 760 mm Hg</u>
1BP	250
0.5	-
1	290
2	341
5	415
7	425
10	432
15	443
20	450
25	451
30	455
35	463
40	471
45	479
50	486
54.4	500
60	529
65	577
69.2	650
75	713
80	756
85	810
88.8	850

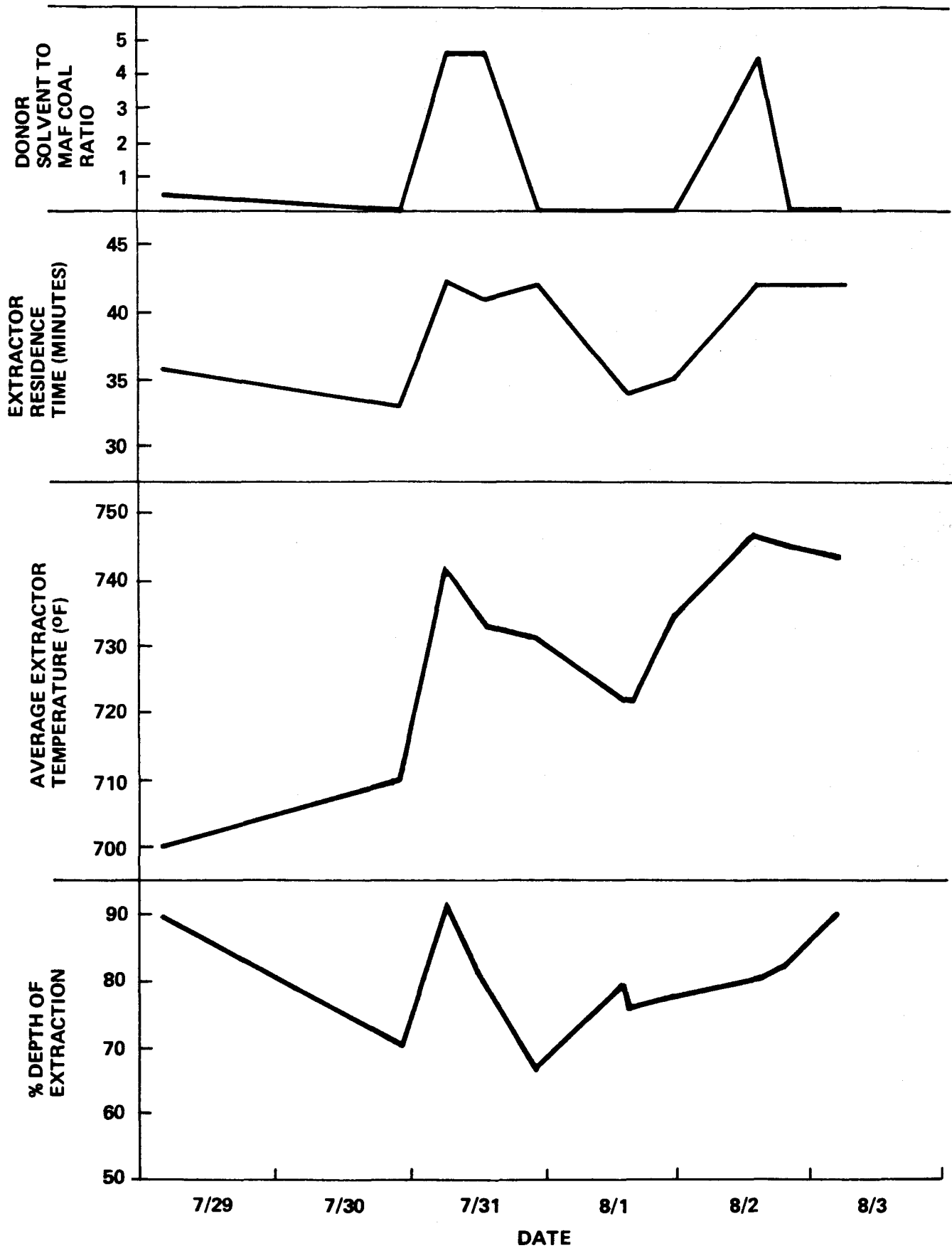
**FIGURE 1**  
**RUN 013 ELEMENTAL BALANCE SECTION 200,300,800**  
**LB/HR**



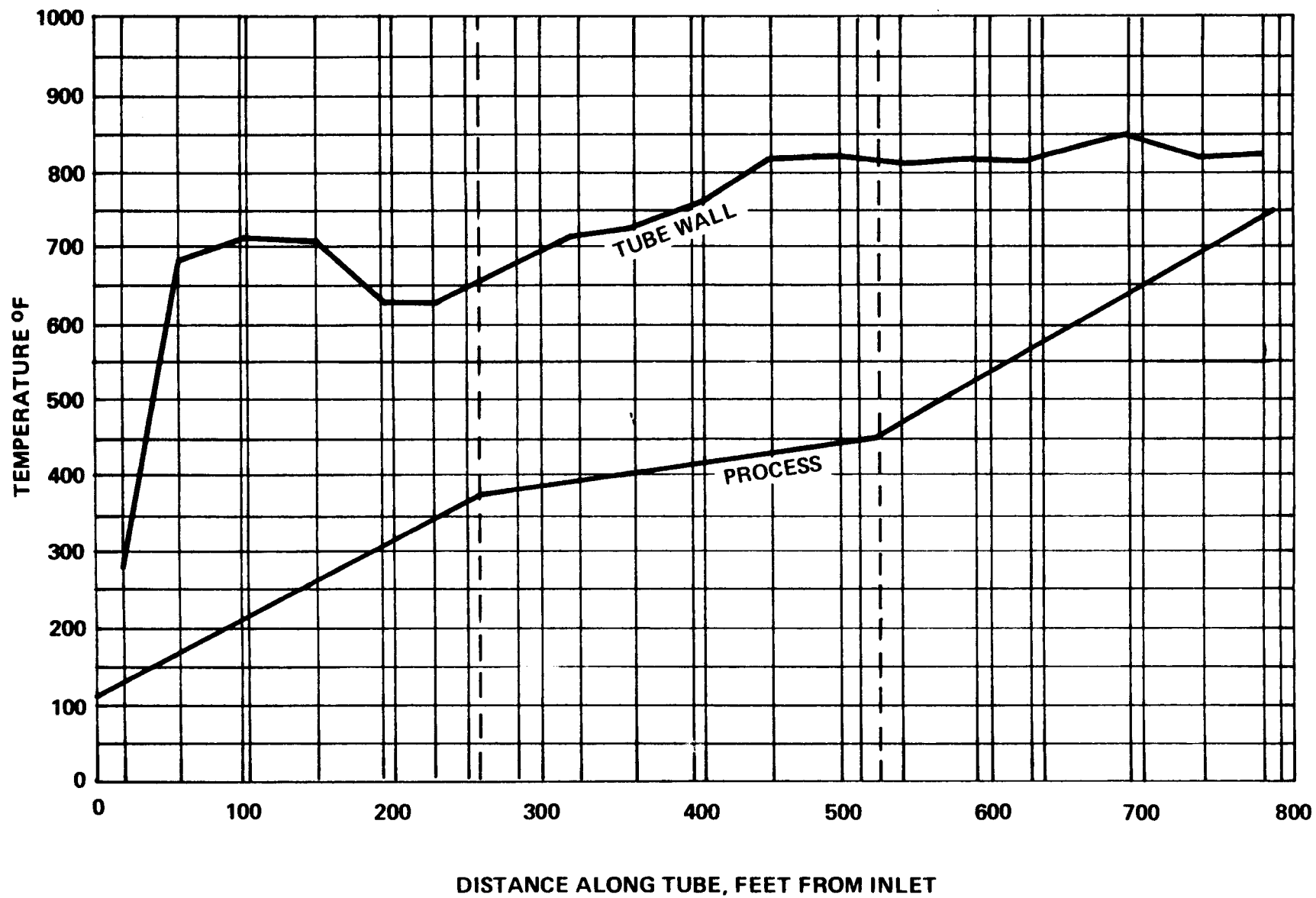
NOTE:  
 DONOR SOLVENT COMBINED WITH THE  
 RECYCLE SOLVENT FOR THE PURPOSE OF THE MATERIAL  
 BALANCE.

① C 695 H 50 N 13 O 70 S 30 ASH 88 TOTAL 946	② 0	③ C 3584 H 352 N 2 O 0 S 2 TOTAL 3940	④ WATER 6 ORGANIC 132 C 132 H 12 N 0 O 0 S 0 TOTAL 150	⑤ C 13 H 4 N 0 O 3 S 0 TOTAL 20	⑥ LIQUID 6 C 3985 H 378 N 11 O 34 S 14 TOTAL 4422 SOLIDS C 149 H 7 N 4 O 28 S 18 ASH 88 TOTAL 294 880 % : 613 TOTAL 4716	⑦ C 1089 H 106 N 0 O 0 S 0 TOTAL 1196	⑧ C 72 H 10 N 0 O 1 S 0 TOTAL 83	⑨ LIQUID 9 C 5192.5 H 498.2 N 10.0 O 31.2 S 14.1 SUBTOTAL 5746 SOLIDS C 7.3 H 0.5 N 0.1 O 4.4 S 1.2 ASH 0.5 SUBTOTAL 14 880 % : 559 TOTAL 5760	⑩ LIQUID 10 C 1477 H 146 N 0 O 0 S 1 SUBTOTAL 1624 SOLIDS C 159 H 6 N 3 O 16 S 14 ASH 86 SUBTOTAL 283 880 % : 0 TOTAL 1907	⑪ LIQUID 11 C 896.5 H 84.8 N 2 O 2.8 S 0.9 TOTAL 987 SOLIDS C 304.7 H 14.5 N 5.9 O 34.6 S 30.8 ASH 172.5 TOTAL 563 880 % : 54 TOTAL 1560	⑫ WATER 4 ORGANIC 62 C 386 H 36 N 0 O 0 S 0 TOTAL 425	⑬ C 629 H 62 N 0 O 0 S 0 TOTAL 691	⑭ C 37.7 H 6.3 N 412.9 O 128.4 S 1.7 TOTAL 587	⑮ C 518 H 47 N 0 O 0 S 0 TOTAL 565	⑯ N 411 O 124 TOTAL 535	⑰ H 1 O 9 TOTAL 10	⑱ C 156.5 H 3 N 3 O 17 S 16 ASH 87.5 TOTAL 282
--	-----	--	---	--	--	--	---	--	--	--	--	---	---	---	-------------------------------	--------------------------	--

**FIGURE 2**  
**EXTRACTION PROCESS PARAMETERS**  
**RUN 013**

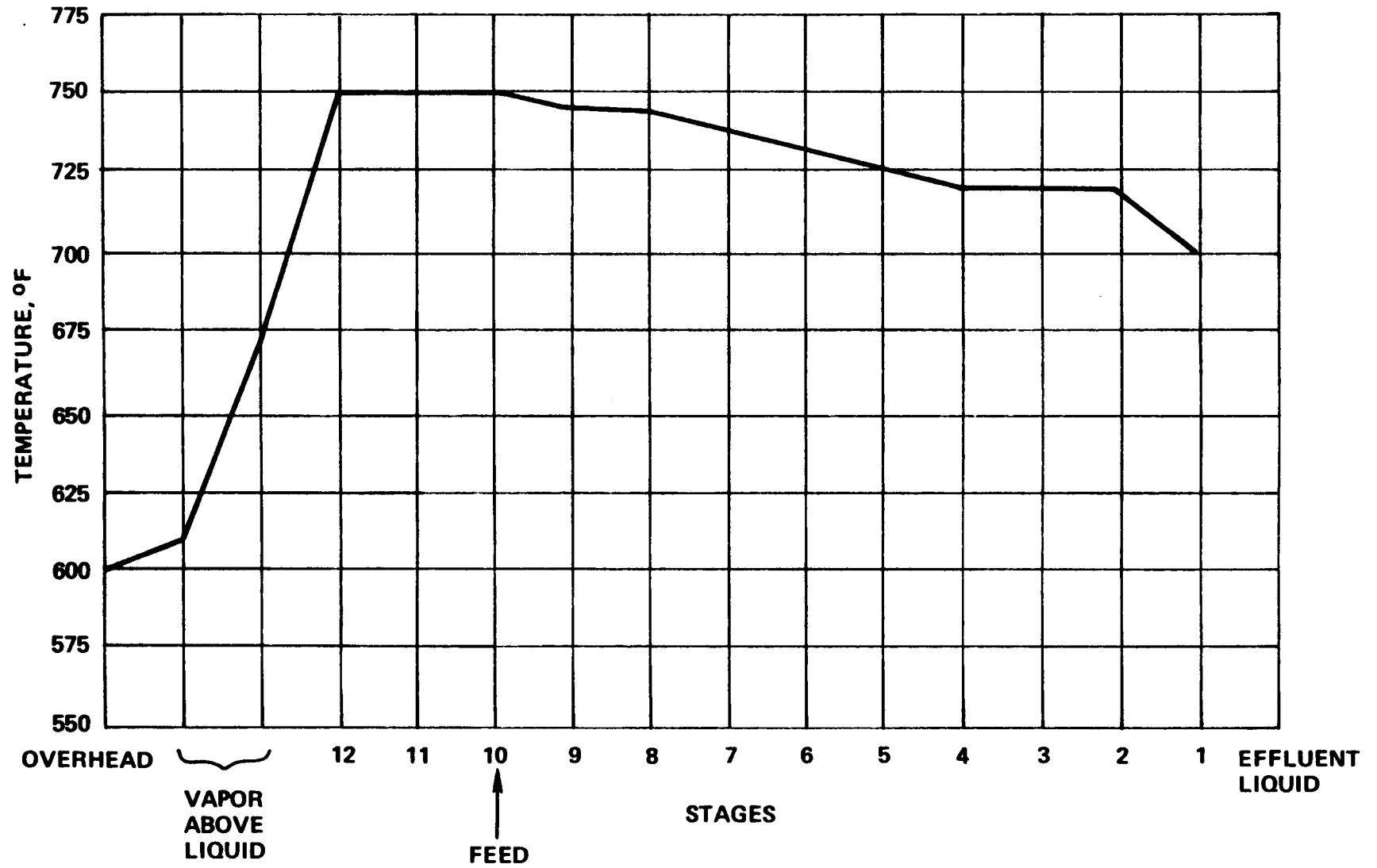


**FIGURE 3**  
**TYPICAL TEMPERATURE PROFILE, SLURRY PREHEATER**  
**RUN 013**



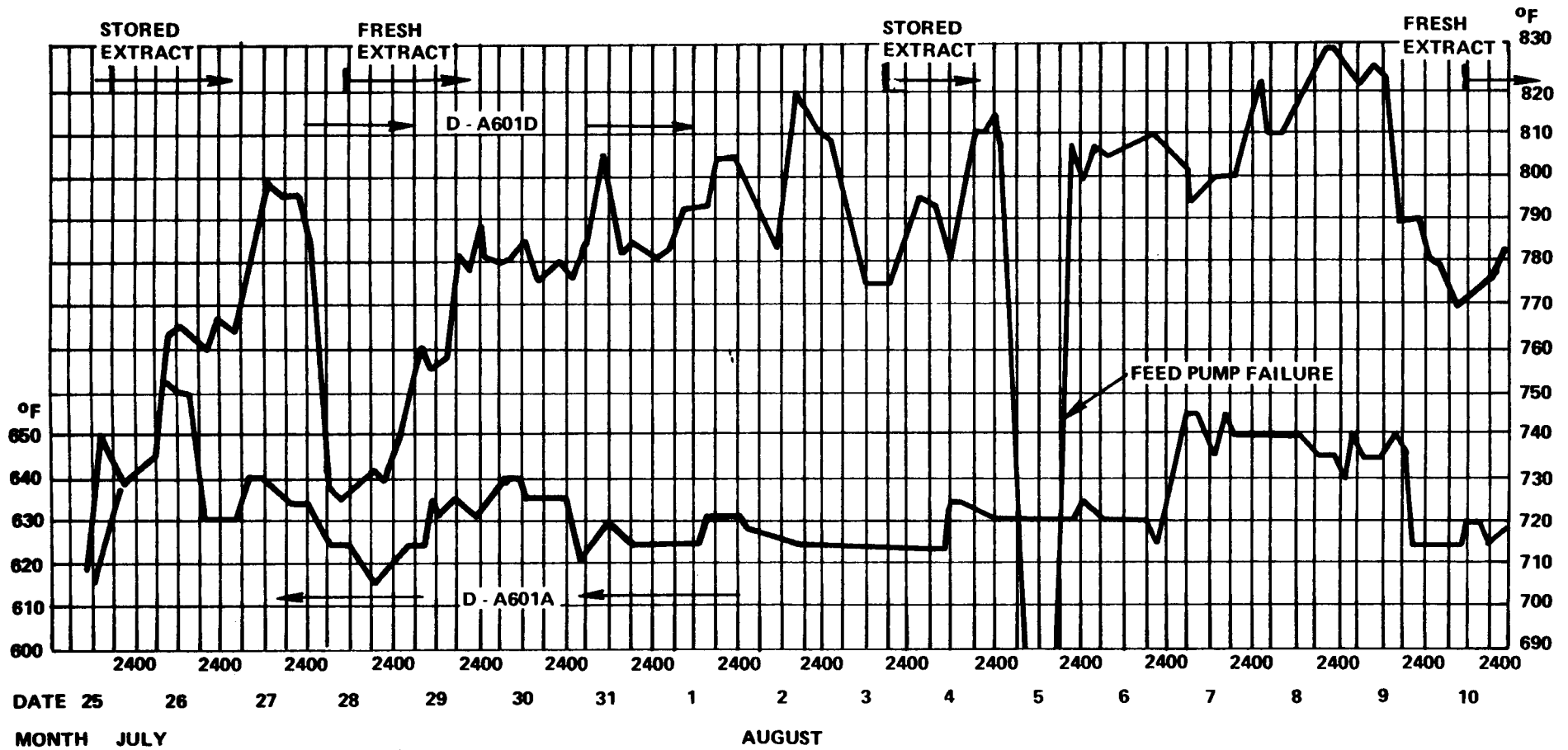
**FIGURE 4**  
**TYPICAL TEMPERATURE PROFILE, ETRACTOR**

**RUN 013**

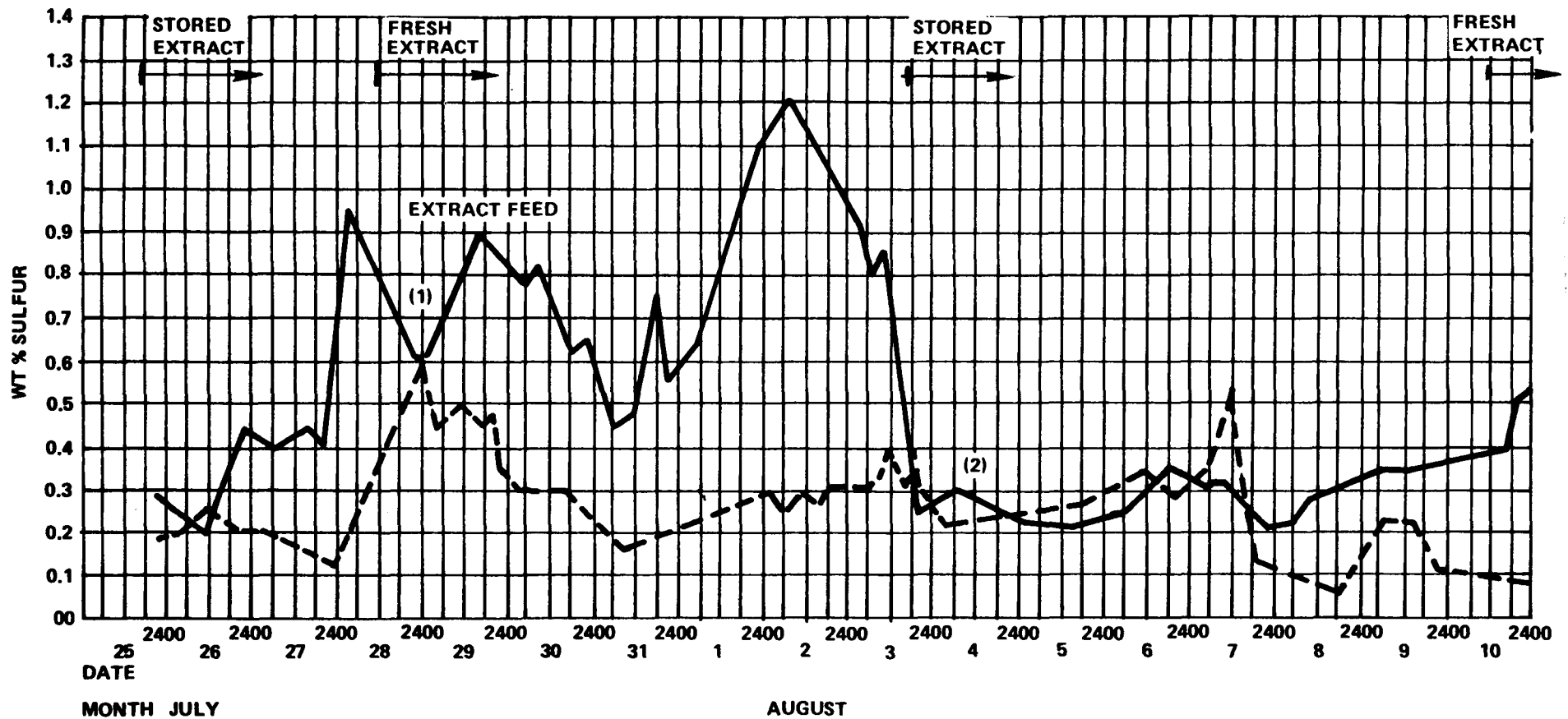




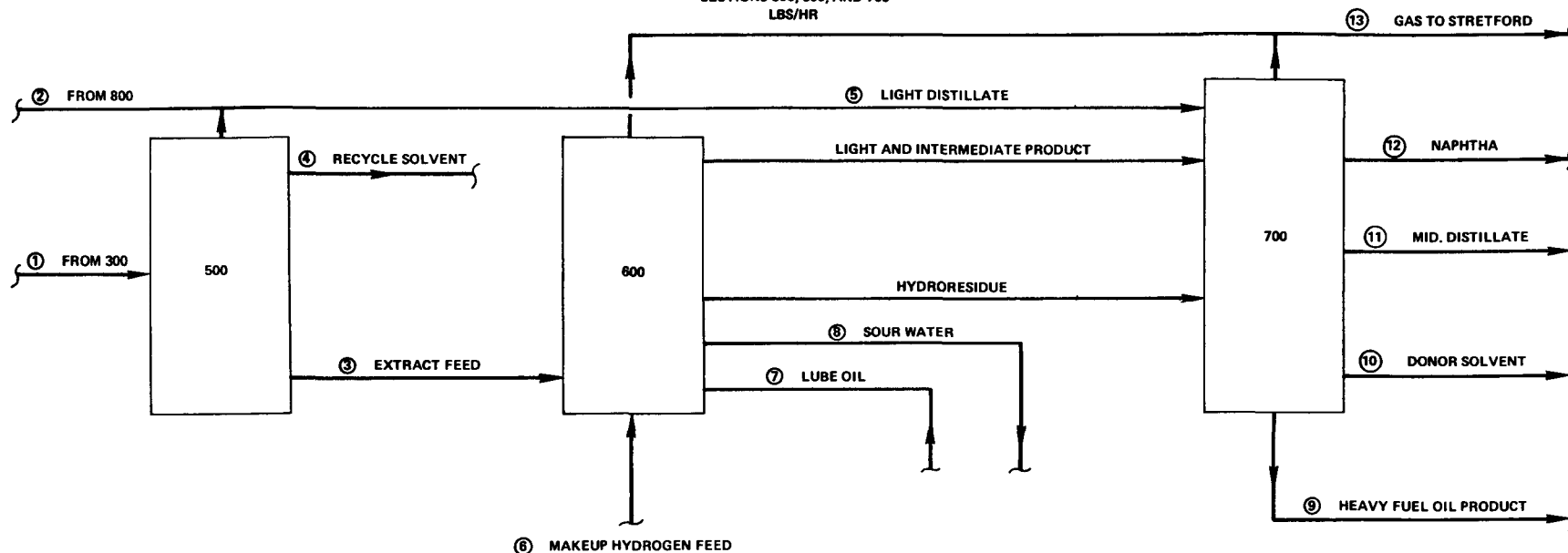
RUN 13 FIGURE 5 REACTOR AVERAGE TEMPERATURE



**RUN 13 FIGURE 6 FEED/PRODUCT SULFUR CONTENT**



- (1) DECREASE IN REACTOR TEMPERATURE FOR INTRODUCTION OF EXTRACT.
- (2) FEED DURING PERIOD AUG., 3 TO AUG. 7 DILUTED WITH PREVIOUSLY DESULFURIZED SOLVENT WHICH LOWERS THE OVERALL CONCENTRATION OF SULFUR IN THE FEED. THE OPERATING CONDITIONS IN PRODUCT FRACTIONATION WERE ALSO BEING ADJUSTED TO INCREASE RECOVERY OF DONOR SOLVENT FROM THE PRODUCT.

[illegible]