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# COAL LIQUEFACTION PILOT PLANT

# CRESAP, WEST VIRGINIA

# MONTHLY TECHNICAL PROGRESS REPORT

# DECEMBER, 1977

Fluor Engineers and Constructors, Inc.

3333 Michelson Drive

Irvine, California 92730

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PREPARED FOR THE UNITED STATES

DEPARTMENT OF ENERGY

Under Contract No. EX-76-C-01-1517

REA

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## NOTIFICATION OF TRIPS

| Name | Duration | Destination | Purpose |
|---|---|---|---|
| G. O. Fredrickson | 11/27-12/23 | Cresap, West Va. | ERDA Meeting & LCDC Support |
| L. Gasendo | 11/27-12/23 | Cresap, West Va. | Plant Startup |
| R. V. Cates | 12/7-12/16 | Cresap, West Va. | Plant Startup |
| F. U. Leonard | 12/5-12/16 | Cresap, West Va. | ERDA Meeting |

## I. SUMMARY

An abbreviated 55-hour extraction run was completed in December; 15 tons of coal were processed as a 15 percent slurry. The depth of extraction was 62 percent. The revised extract disposal system operated satisfactorily. Coal processing was abruptly terminated when the lower extractor agitator shaft seal failed, bringing an end to Run 004.

Runs during which extraction operations were conducted are summarized below:

Run 001 lasted less than one hour on October 11, 1977. It was terminated when the slurry preheater coked due to a leak-induced flow interruption.

Run 002 extended for a 24-hour period ending on October 19, 1977. The run was terminated when all means for the removal of concentrated extract failed.

Run 003 was a 140-hour effort which began on November 15, 1977 and was terminated because of the inoperability of the extract granulation system. Thirty-two tons of coal were processed in an 11.5 percent slurry. Conversion averaged 62 percent; more than one hundred 55-gallon drums of extract-rich intermediate were produced.

Run 004 was abruptly halted after 55 hours of operation when the lower agitator shaft seal failed on December 18, 1977. Satisfactory operation of the revised extract granulation system was demonstrated.

Run 005 is now scheduled for January 1978. Baseline operating data of the solvent deashing section of the pilot plant will be developed first at 15 percent coal, then at 25 percent coal. The latter will be at a coal feed rate of 12 tpd.

Warm solvent has been circulated through most of the hydrotreating section of the pilot plant under a 2700 psig nitrogen blanket. Leaks at the heads of the absorber and the entrainment separator persisted beyond the end of the month. Much of January is scheduled for maintenance and modification in the hydrotreating section. In addition to the repair of leaky vessels and Conval valves, the attritor loops will be removed from the two hydrogenation reactors as requested by the licensor.

An apparent high phenol level of the treated industrial waste water discharge to the Ohio River exceeded EPA limits during December. An extensive effort to define and resolve the problem has begun.

The National Labor Relations Board has certified the results of the September election, during which Liquefied Coal employees chose not to be represented by the International Chemical Workers Union.

## II. MAJOR PROBLEMS AND SOLUTIONS

### A. Elevated Phenol Level in the Industrial Waste Water Effluent

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

Industrial Waste Water Phenol Content, ppm

| | Highest Single Reading | Monthly Average |
|---|---|---|
| Maximum Allowable (NPDES Permit) | 1.00 | 0.05 |
| December 1977 | 2.00 | 1.50 |
| November 1977 | 0.09 | 0.02 |
| October 1977 | 0.03 | 0.01 |
| September 1977 | 0.05 | 0.02 |

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

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

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

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

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

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

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

### B. Recurring Extractor Seal Failures, Section 200

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

## II. MAJOR PROBLEMS AND SOLUTIONS

### B. Recurring Extractor Seal Failures, Section 200 (Continued)

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

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

### C. Failure of the Pump Coating Materials

The PTI-54 pump linings continue to fail. In December one failure occurred in a service where there had been no previous problems; a second failure confirmed that any chips or cracks in the ceramic coating will ultimately lead to spalling and total removal of the lining. To date PTI-54 coatings have failed in five of the 16 pumps so equipped. There also have been some problems with tungsten carbide and Stellite linings. Other details are given under Test 1100.

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

### D. Pump Seal Problems

Five mechanical seal failures were experienced in December; there were 10 in November. The immediate causes of the failures include debris in the seal cavity, damaged springs, tar buildup on the seal faces and packing leaks. The packing leaks may have resulted from the relatively-low resiliency of the Durafite packing. Viton-A O-rings were substituted for the Durafite packing in two seals. There have been no O-ring failures to date. Further details are provided under Test 1100.

## II. MAJOR PROBLEMS AND SOLUTIONS

### E. Leaky High-Pressure Valves in Hydrogenation, Section 600

Sixteen Conval valves were repaired and/or repacked during December. One Conval valve packing blew out at 2700 psig nitrogen pressure. Fortunately, there were no injuries. The stem packing of all Conval valves will be replaced next month with an assembly which includes braided graphite filament rings in the first and fifth positions and die-formed Grafoil rings in the middle three positions.

### F. Condensate System Problems

Steam generating capacity has been limited, in part, by the inability to recover condensate for re-use as boiler feed water because of excessive pressures and temperatures in the condensate return system. The high pressures in the condensate system appear to arise mainly from malfunctioning steam traps. Impulse traps are used almost exclusively on tracing systems. These traps are relatively intolerant of the dirt and scale associated with carbon steel tubing which was used for most of the refurbished steam tracing. At any given time a high proportion of the traps are blowing through.

The problem is being addressed by the replacement of failed tracing with stainless steel and by a vigorous program to maintain trap operability. As many as eight craftsmen daily are assigned to this problem.

### G. Slow Heatup Rate and Low Temperature In Solids Separation, Section 300

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

## III. PROGRESS BY PLANT SECTIONS

### Section 100 - Coal Preparation

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

The fiberglass suspension springs in the L-A117 hammermill vibrating feeder were replaced early in December.

## III. PROGRESS BY PLANT SECTIONS

### Section 200 - Coal Extraction

During the 55-hour run of Run 004 the extraction depth was about 62 percent. A 15 percent coal slurry was fed at 6.5 gpm to the B-A201 slurry preheater. The run was discontinued when the bottom seal of the L-B205 extractor mixer failed. The seal problem is discussed further in Sections II and IV of this report.

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

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

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

### Section 300 - Solids Separation

The limited duration of Run 004 prevented the attainment of the steady-state conditions that would allow a detailed process analysis to be made. Samples taken for process control purposes showed that the settler overflow stream contained less than 0.5 percent ash on extract for most of the run.

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

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

## III. PROGRESS BY PLANT SECTIONS

### Section 300 - Solids Separation (Continued)

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

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

### Section 500 - Solvent Recovery

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

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

### Section 600 - Extract Hydrogenation

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

This loop was then expanded to include the F-A609 residue separator, the F-A612 residue letdown tank, the B-A704 vacuum column reboiler and the J-A704A/B vacuum column bottoms pumps. A temporary line was run from the J-A704A/B discharge to the F-A608A extract feed tanks to complete the expanded solvent loop.

## III. PROGRESS BY PLANT SECTIONS

### Section 600 - Extract Hydrogenation (Continued)

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

Level, flow and pressure instruments were checked out in each loop. Freeze-damage was not uncommon. The reactor nuclear level and density detectors were also checked out. Although the J-A614A/B instrument purge oil pumps were commissioned, the purge oil froze at the lower ambient temperatures. Alternate purge fluids which freeze at -20°F or lower are being sought.

The B-A601 hydrogen heater coil was ultrasonically inspected and found to be free from defects. Plans were developed to install the missing burner tiles in B-A601 and B-A602A/B fired heaters. Attempts to seal leaks at the closures of the F-A605 entrainment separator and the E-A601 absorber were unsuccessful. Solid Teflon seal rings will be tried next month. Sixteen Conval valves were repaired and/or repacked as described in Section II of this report. Rockwell-Edwards-type valves, suitable for most of the Conval services, were located in stock. These may be installed at a later date if problems with Conval valves continue.

The detailed plan and schedule for completing the remaining prestart maintenance activities was updated and issued separately.

### Section 700 - Fractionation

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

A solvent spill occurred when a backflow from the E-A702 stabilizer column into the F-A710 economizer surge drum overfilled F-A710. A PMR to install a check valve between the columns and thus prevent a recurrence was prepared.

### Section 800 - Carbonization

The D-A801 carbonizer was operated for two days during Run 004. When the lower spray nozzle plugged, feed was switched to the upper spray nozzle. The upper nozzle discharges directly into the fluidized char bed; the lower nozzle empties into a higher-velocity riser pipe. The upper spray

## III. PROGRESS BY PLANT SECTIONS

### Section 800 - Carbonization (Continued)

nozzle had no plugging problems; the in-line strainer upstream from the nozzle was cleaned once. After the run a new upper feed nozzle with an enlarged orifice was installed. The lower nozzle will be changed when the bed level is below the nozzle entry point.

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

### Section 900C - Hydrogen Compression

Insulation of JC-A900A/ B makeup hydrogen compressors was completed by the subcontractor. JC-A900B was used to supply high-pressure nitrogen to support Section 600 solvent circulation operations. One of the oil lubricator pumps on JC-A900B was replaced.

### Section 900G - Hydrogen Generation

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

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

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

### Section 1000 - Utilities

1010: A boiler outage was taken to replace the butterfly suction valves with gate valves and to clean the suction strainers on the J-A1011 boiler feed water pumps. The ceramic packing from the deaerator J-A1010 was found in the suction strainers. Investigation showed that the support screen had failed in the deaerator. Repairs were made and the boilers were returned to normal service.

1030: Excessive vibration of the plant air compressor discharge line continues to be a problem. Besides the noise, noticeable wear is occurring at a pipe support.

## III. PROGRESS BY PLANT SECTIONS

### Section 1100 - Offsites

1110: The bottoms of the F-A1101A/B recycle solvent tanks were found to contain 12 percent water in an emulsion. This material will be recycled through Section 500 to eliminate the moisture.

An additional 10,400 gallon shipment of Sure-Sol 180 was received and stored in the F-A1106A/B donor solvent tanks.

1130: The Seal Oil System operates routinely except when a pump or agitator seal starts leaking. Until the leak can be found and isolated, the full-time attention of two operators is required to maintain levels in the seal oil tanks. A PMR has been submitted to provide an improved seal oil (solvent) makeup system.

### Section 1200 - Environmentals

1210: Repairs to the C-A1213 Sour Water Stripper column reboiler were completed. This section is now in normal operation treating process waste waters.

1230: The Waste Water Treating Section remains on manual operation during regeneration due to problems with the automatic controller. A PMR is being prepared to improve the control system. A major problem with phenol removal is discussed in Section II of this report.

1240: Industrial and sanitary effluent analyses for December are attached. Solids occasionally pass through the Sanitary Waste Treating Section to escape into the outfall. Assistance on troubleshooting the system is being provided by personnel from the West Virginia Department of Health.

## IV. TEST PROGRAMS

### General

A meeting between SCD and LCDC personnel was held on December 13 to discuss the test programs in light of the current resource limitations. The results of this meeting are summarized in Conference Notes No. 148.

### Test 1100 - Pumps

Modifications to the J-A202A/B and J-B202 extractor charge pumps were completed. These changes included the replacement of reduced port ball valves in the suction lines with full-ported valves, the substitution of quarter-turn bypass valves for the original gate valves, the elimination of deadlegs for standby slurry lines, the addition of several flush connections and the installation of added piping to permit slurry recirculation through the J-A202 suction header. Following the revamp, the J-A202A pump functioned without incident during the entire 55 hours of Run 005.

IV. TEST PROGRAMS

Test 1100 - Pumps (Continued)

Centrifugal

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

| | |
|---|---|
| J-A302 | Slurry Recycle Pump |
| J-B303A | Underflow Pump |
| J-A313A | Primary Feed Pump |
| J-A806A | Tar Quench Pump |
| J-A806B -- J-A314D | Pump in Tar Quench Pump Service |

A PMR was prepared and the materials were purchased to install valved connections on the bearing housings to facilitate their draining.

Five seal leaks were experienced in the following services:

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

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

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

While repairing the J-A806A pump it was found that the PTI-54 coating had failed around the wear ring area resulting in erosion of the case and back cover. Similarly, the PTI-54 coating on the case of the J-A313B primary feed pump had flaked from large portions of the coated area. The Triballoy coating used on J-A314D, the pump which is substituted in the J-A806B tar quench service, showed no signs of deterioration. The coefficient of thermal expansion for Triballoy is closer to the base metal than are the expansion coefficients of either PTI-54 or tungsten carbide. A comparison of coefficients of expansion follows:

IV. TEST PROGRAMS

Test 1100 - Pumps (Continued)

Centrifugal (Continued)

Selected Thermal Expansion Coefficients, in/in/F

| | |
|---|---|
| Carbon steel | $6.5 \times 20^{-6}$ |
| 11-13 stainless steel casting | $6.5 \times 10^{-6}$ |
| Triballoy 800 | $7.0 \times 10^{-6}$ |
| Tungsten carbide | $4.4 \times 10^{-6}$ |
| PTI-54 | $4.2 \times 10^{-6}$ |

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

Test 1300 - Fired Heaters

An evaluation of the inside heat transfer coefficient, $h_i$, for coal-free solvent in the B-A201 slurry preheater was begun. The initial work was limited to solvent because its physical properties are better known. Solvent operations before and after Run 003 were evaluated. A preliminary review of the data shows that an order-of-magnitude drop in $h_i$ occurs in the second zone after slurry has been processed. The second zone corresponds to that part of the coil where the process temperatures are increasing from 450°F to 750°F. A report is in preparation.

Test 1800 - Extractor

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

Test 1900 - Corrosion/Erosion

A laboratory study of the potential causes of crevice corrosion on the tungsten carbide balls in J-B202 was initiated. Because of the apparent low corrosion rate, the test will be of long duration. Early data may be available in three months.

## V. LABORATORY

A sample bomb retaining device was completed and set up to provide a reasonably safe method for removing process samples from Type A flow-through sample containers. A survey of laboration ventilation was conducted by Kewaunee Scientific Equipment Company; their recommendations were discussed with Maintenance and Engineering personnel. A final recommendation is being prepared.

## VI. SAFETY AND HEALTH

The annual employee physical examinations continue; quarterly skin examinations were begun. The clothing security trailer was installed and connected to the locker room. The "Line and Flange Breaking" Job Safety Instruction was approved and distributed.

## VII. DEPARTMENTS

### Administrative

Staffing: Job offers have been accepted by candidates for the positions of mechanical engineer, instrument engineer and shift engineer. One chemical engineering position remains open. A data clerk resigned; candidates are being interviewed.

Personnel: The National Labor Relations Board has certified the results of recent employee election rejecting union representation. Meetings were held with all employees to discuss current status of the project and to review any subjects brought up for discussion.

### Maintenance and Engineering

Amendments to Liquefied Coal's nuclear license were submitted to the Nuclear Regulatory Commission to reflect the appointment of A. C. Nippert as radiation officer and to improve monitoring specifications for the nuclear density meters. A meeting was held with the contractor to review construction on pressurizing the lunch room; completion is scheduled for February 7, 1978. A PMR was issued to modify the sanitary facilities in the north warehouse to provide better accessibility for both men and women.

### Operations

Section 600 prestart-up work has received top priority. Operating personnel are working closely with Maintenance and Engineering to conduct operations according to the facility start-up plan of November 9, 1977, Revision 3.

## VII. DEPARTMENTS

### Technical

The source programs for laboratory and process data were received from SCD. These are being reviewed for program modifications to include the current sampling and analytical efforts. The first of three steps to expand the laboratory sample point file from 150 to 300 points was completed. Updating of the warehouse data file continues. A new "tape read" head was installed to correct the recurring system preserve problems. The Run 005 Plan was prepared and distributed. Analyses and evaluation of the operating conditions and sample results for Runs 003 and 004 have begun.