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SOLVENT REFINED COAL (SRC) PROCESS

QUARTERLY TECHNICAL PROGRESS REPORT
FOR THE PERIOD
APRIL 1, 1978, THROUGH JUNE 30, 1978

THE PITTSBURG & MIDWAY COAL MINING CO.
P. O. BOX 2900
SHAWNEE MISSION, KANSAS 66201

MAY 1979

PREPARED FOR THE
U. S. DEPARTMENT OF ENERGY
DIVISION OF COAL CONVERSION AND UTILIZATION
UNDER CONTRACT EX-76-C-01-0496

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ABSTRACT

This report summarizes the progress of the Solvent Refined Coal (SRC) project by The Pittsburg & Midway Coal Mining Co. for the Department of Energy for the period April 1, 1978 through June 30, 1978. Major activities at the pilot plant included a scheduled plant turnaround which began April 1 and continued through June 10 and the blending of 4,500 barrels of SRC II fuel oil for use in a large-scale combustion test. During the turnaround major efforts were directed towards tie-ins and modifications necessary for the startup of Filter C and an extensive examination of process piping, heat exchangers, and vessels. Construction of the Lummus anti-solvent deashing unit continued during the quarter. Activity at the Merriam Laboratory included a study of the process factors in SRC II that lead to increased oil yields relative to SRC I operation and exploratory short residence time SRC I operation. An upgraded and modernized continuous bench scale unit was started up during the quarter to replace the existing bench scale unit. The one-ton-per-day P-99 process development unit at Gulf's Harmarville facilities became part of the SRC program April 1. It ran SRC II experiments with Pittsburgh seam coals during the quarter.

I. SUMMARY OF FORT LEWIS PILOT PLANT ACTIVITY

At the end of the first quarter, the plant was operating in the SRC II mode processing Pittsburgh Seam coal from the Blacksville Mine No. 2. On March 31, operation was switched to SRC I mode with Kentucky Nos. 9 and 14 coal to condition raw solvent for use during startup after the scheduled turnaround.

Before the plant was shutdown, a brief test was conducted to determine the operating characteristics of the existing slurry blend system at higher temperatures. Operation was satisfactory at 375°F, but each time slurry temperatures were increased above 375°F (390°F maximum), the circulation pump began to cavitate and slurry flow could not be maintained.

The scheduled plant turnaround began April 1, 1978, and continued through June 10, 1978. Final inventory of SRC II liquids at shutdown showed 355 bbl of naphtha, 5059 bbl of middle distillate, and 905 bbl of heavy distillate.

During the turnaround, major efforts were directed toward the startup of Filter C and associated utilities. The flare, inert gas, seal flush, flush solvent and Dowtherm systems were recommissioned during the first week of May.

Also during the turnaround, an extensive examination of process piping, heat exchangers and vessels was made. Most of the utility vessels were also inspected. In most cases, the initial examination was made with an ultrasonic thickness tester. On the basis of these readings, suspicious areas were inspected more thoroughly. Other inspection techniques used included radiography, in-place metallography, visual, and analog ultrasonic (longitudinal and shear wave). Corrosion racks were changed and sent to both Gulf Science and Technology and Oak Ridge National Laboratory for analysis.

Coal feed was resumed on June 10, 1978 but was intermittent throughout the remainder of the quarter. Numerous startup problems, which included a series of control valve failures in Area 02 and a collapsed down leg on the old vacuum flash drum, limited total on-stream time to ninety-one hours through June 24.

Four thousand five hundred barrels of a blend of middle and heavy distillate were prepared for a large scale combustion test to be conducted by Consolidated Edison in New York. Approximately 37 tons of high-ash SRC II vacuum bottoms from Kentucky coal were shipped to the Texaco Development Research Laboratory in Montabello, California, for gasification tests.

Special studies were carried out to determine the heating values of SRC products, the particle size distribution in SRC II reactor effluent, and temperature-density relationships for recycle and preheater charge slurries.

Overall construction on the Lummus antisolvent deashing unit is approximately 72% complete. Seventeen of twenty-eight systems have been hydrotested.

II. PILOT PLANT OPERATIONS, ENGINEERING AND MAINTENANCE

A. Coal Receiving and Preparation - (Area 01)

1. Operation March 25 through April 1, 1978

Coal was processed on the first eight days of the period consuming 183 tons of coal. The plant operated in the SRC II mode for six days feeding Blacksville No. 2 coal at 2130 lb/hr and 30% concentration. The final two days were spent in SRC I operation to convert raw solvent (coal tar distillate) to suitable startup solvent for the SRC I operation scheduled to begin in June. The hourly on-stream factor adjusted for downtime not related to process requirements was 91.3% through April 1. A coal processing summary covering the second quarter and overall process operating histories is shown in Table 1.

2. Turnaround Maintenance

Coal feed was discontinued April 1, 1978 to begin the turnaround for SRC I. The major maintenance items completed during the turnaround were:

a. The pulverizer was completely rebuilt with new balls, rails, springs and classifier section cone.

b. A new impeller was installed in the pulverizer gas blower and cracked internal baffles were repaired. The blower housing will require replacement in the future because of corrosion by water carryover from the dehumidifier scrubber.

c. The demister pad in the dehumidifier scrubber was replaced.

d. The gravimetric feeder inlet chute, which had been abraded through in several places, was replaced.

e. A surface condenser was installed on the slurry blend tank eductor outlet.

f. A Sandpiper diaphragm pump was installed on the slurry blend tank vent condensate receiver to pump drips to Area 02.

g. The variable speed drive for charge pump B was rebuilt.

TABLE 1

COAL PROCESSING SUMMARY

	1974	1975	1976	1977	Mar. 25-Apr. 24 1978	Apr. 25-May 24 1978	May 25-June 24 1978	1978	Total
Coal Processed, Tons	678	8021	6559	4776	183	0	123	2357	22391
On-stream Days	30	241	234	216	8	0	11	97	818
On-stream Hours[a]	---	---	---	3990	168	0	91	1867	5857
Average Feed Rate Per On-stream Day, Tons/Day	22.3	33.3	28.0	22.1	22.9	NA	11.2	24.3	27.4
Average Feed Rate Per On-stream Hour, Lb/Hr[a]	---	---	---	2115	2179	NA	2703	2525	2246
On-stream Factor, Days, %	32.6	66.0	63.9	59.2	25.8	NA	33.5	55.0	60.0
On-stream Factor, Hours, %[a]	---	---	---	67.9	22.6	NA	12.2	44.4	59.9
On-stream Factor, Adjusted Hours, %[b]	---	---	---	---	91.3	NA	26.9	75.3	75.3

[a] Data accumulated since May 1, 1977.

[b] Data accumulated since January 1, 1978. Available operating time is adjusted for downtime not directly related to process problems or equipment failures.

3. Inspection of Piping and Vessels

The slurry blend tank and the dehumidifier were inspected and found to have some metal loss. The carbon steel dehumidifier scrubber revealed corrosion along the upper half of the vessel and around the stainless steel inlet nozzle. The demister mat was replaced. There was light erosion/corrosion in the area of the agitator on the slurry blend tank. The remainder of the vessel showed no noticeable wear.

The piping that was inspected included the preheater feed slurry circulation loop, recycle slurry piping, and the high pressure charge pump discharge piping. Only the piping down stream of the recycle slurry air cooled exchanger will be discussed in Section II-B. Most of the coal ducts were also inspected.

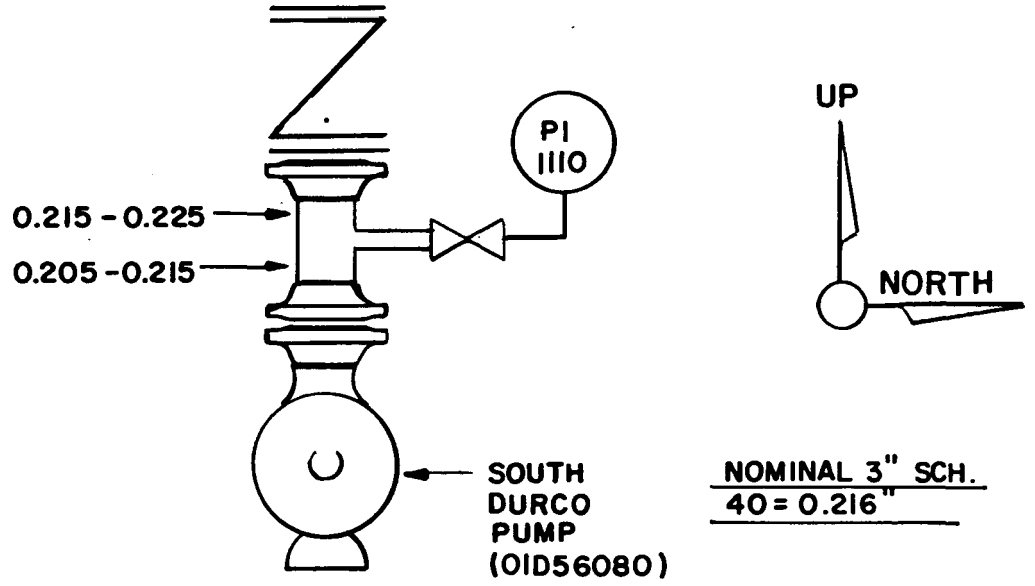
The preheater feed slurry circulation loop is mainly 3-inch schedule 40 carbon steel and runs from the circulation pumps to the mix tank or eductor and then back to the blend tank. The circulation pumps take suction from the blend tank. The high pressure charge pumps feeding the reaction area take suction from the higher pressure side of the circulation loop. Nominal operating temperature of this loop is 350°F. The normal liquid velocity is approximately 9 feet/second. Flow is generally laminar because of high slurry viscosities. During SRC I operation, the material flowing in this loop is a slurry consisting of recycle process solvent and pulverized coal. During SRC II operation, material in this loop is a slurry consisting of unfiltered recycle coal solution and coal.

Figure 1 shows the discharge piping of the Durco slurry circulation pumps. This piping was installed during the SRC II modifications turnaround in early 1977 and thus has seen one year of SRC II service. Approximately 0.075" wear occurred on the 90° bend on the discharge of the north Durco pump. Considerably less wear occurred on the straight discharge spool piece of the south Durco pump and the second 90° bend on the north pump. No significant general corrosion or erosion is seen on a typical five diameter bend of the loop shown in Figure 2. These bends are original plant construction.

The only piping that required any maintenance in the coal slurry preparation area was around the coal eductor. The bend directly downstream of the coal eductor had eroded through twice during the SRC II run. Originally, various piping restraints resulted in the placement of this bend in an undesirable location. However, to reduce the high rate of erosion in this area, the piping was changed and the troublesome bend eliminated.

FIGURE 1

A. DISCHARGE SOUTH DURCO CIRC. PUMP (3"-SL-124-GI)



B. DISCHARGE NORTH DURCO CIRC. PUMP (3"-SL-87-GI)

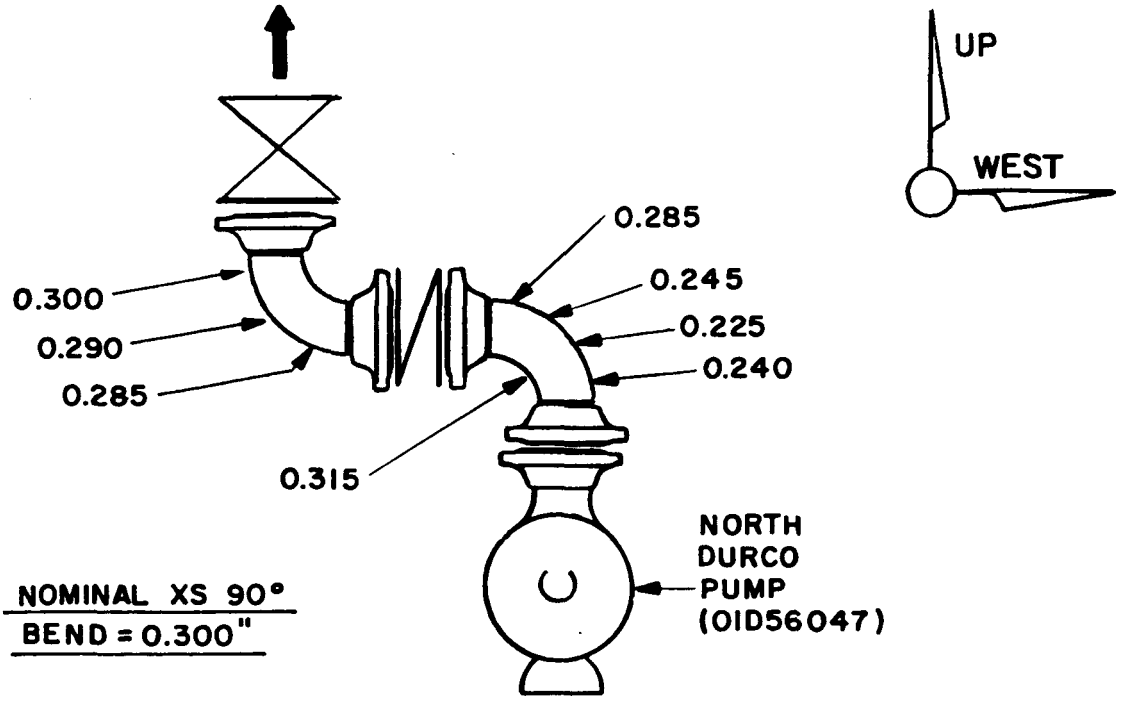
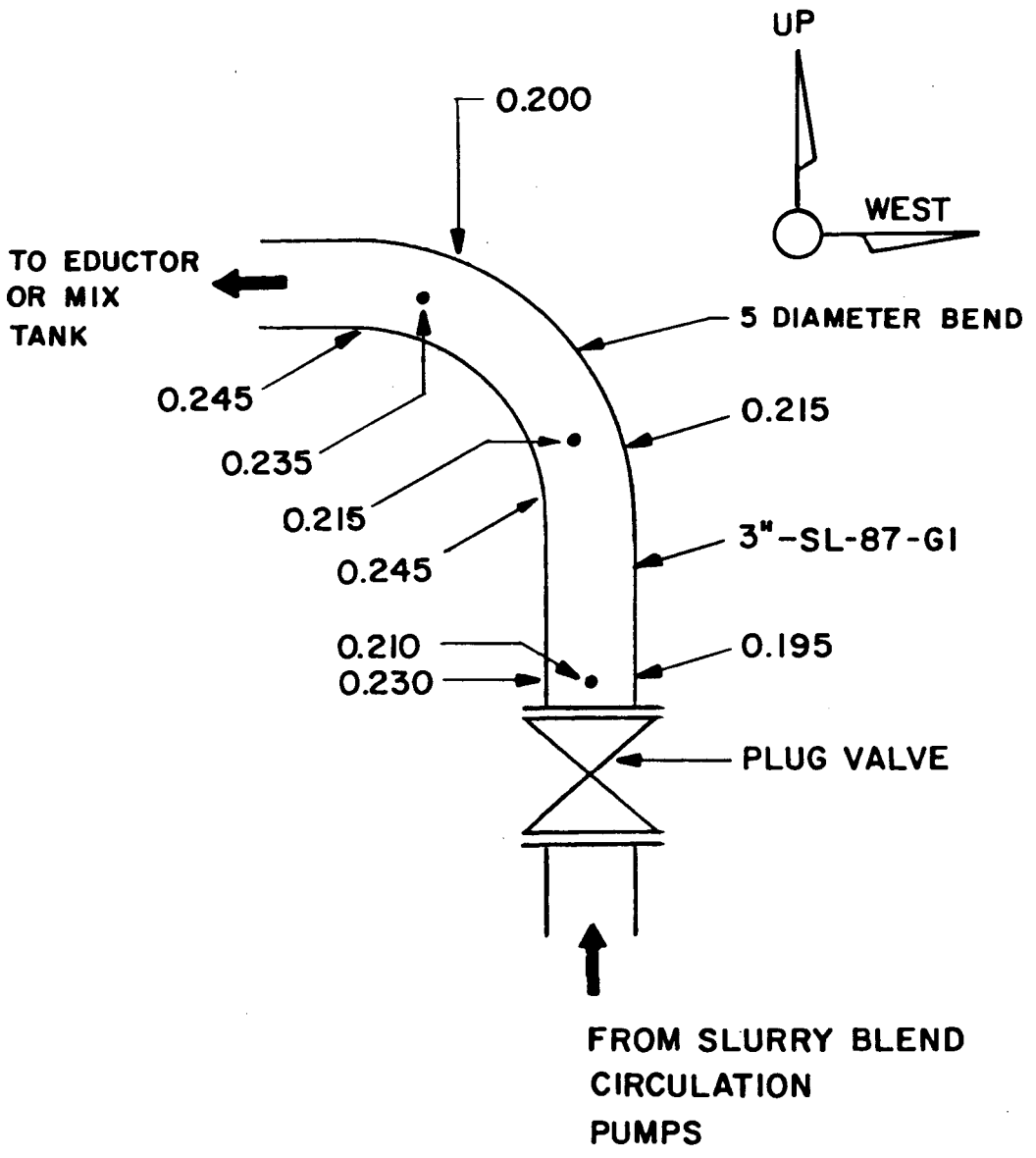


FIGURE 2

DISCHARGE SLURRY BLEND CIRCULATION PUMPS 3"- SL-87-GI

AT ON-LINE VISCOMETER DECK



NOMINAL 3" SCH. 40 WALL THICKNESS = 0.216"

As shown in Figure 3, the recycle slurry piping downstream of the slurry recycle cooler is not worn much. The maximum metal loss (0.021") is on the outside of the five diameter bend. However, this amount of loss is less than the originial ± 10% variation from the nominal thickness of 0.216".

The high pressure charge pump discharge piping, as shown in Figures 4 through 6, is schedule XX carbon steel pipe (ASTM A106, Gr. B). This piping transfers unreacted coal slurry from the high pressure charge pumps to the slurry preheater. This slurry is identical to the material described in the circulation loop. Nominal conditions in the two-inch transfer line are: temperature, 350°F; pressure, 1500 psig for SRC I operation and 2000 psig for SRC II operation; and liquid velocity, approximately 3-6 ft/second (laminar). No significant wear was detected in this piping.

The slurry recycle air cooled exchanger was inspected and found to have no detectable wear. The dehumidifier heat exchanger was not inspected since it was replaced with a 304 SS exchanger during the last turnaround (February 1977).

4. Operation June 10 through June 24, 1978

After the plant was restarted June 10, coal was processed intermittently through the remainder of the reporting period. Approximately 123 tons of Kentucky coal were processed during 11 days of SRC I operation. The adjusted hourly on-stream factor for this period was 26.9%. Target coal feed rates ranged from 2500 lb/hr to 3740 lb/hr. Coal Slurry concentration ranged from 33% to 40%.

5. Pump Performance

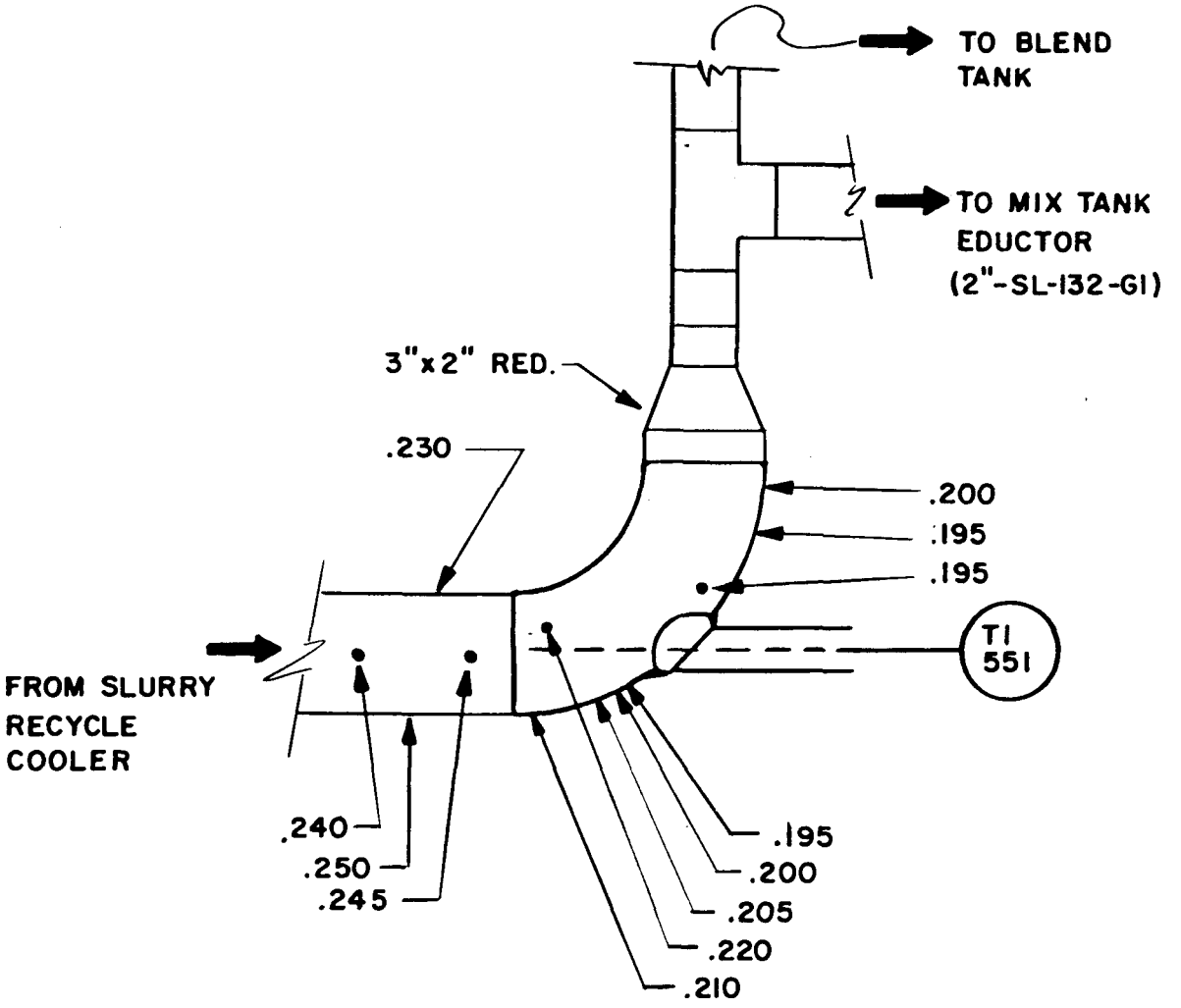
Slurry blend circulation pumps (01D56003, 01D56047, 01D56080) were virtually maintenance free during the reporting period.

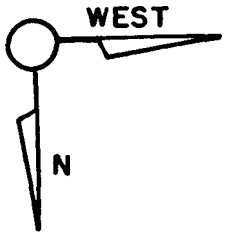
Preheater charge pumps (01D56101, 01D56102)--During the turnaround the magnetic variable speed drive on high pressure charge pump "B" was rebuilt by an outside electric company. The temperature control valve on the magnetic clutch cooling water failed when put into service. The unit operated without temperature control until new trim was obtained.

High pressure charge pump "A" was used extensively during June and plunger packing was replaced twice. The center suction valve was replaced on one occasion, and the center discharge valve fouled once with solids build-up. Glass filled Teflon packing was not available; hence, Teflon impregnated asbestos packing was used for the repair.

FIGURE 3

DOWNSTREAM SLURRY RECYCLE COOLER (3" SECTION-2" SL-117-GI)





3" SCH 40 PIPE NOMINAL WALL THICKNESS = 0.216"

FIGURE 4

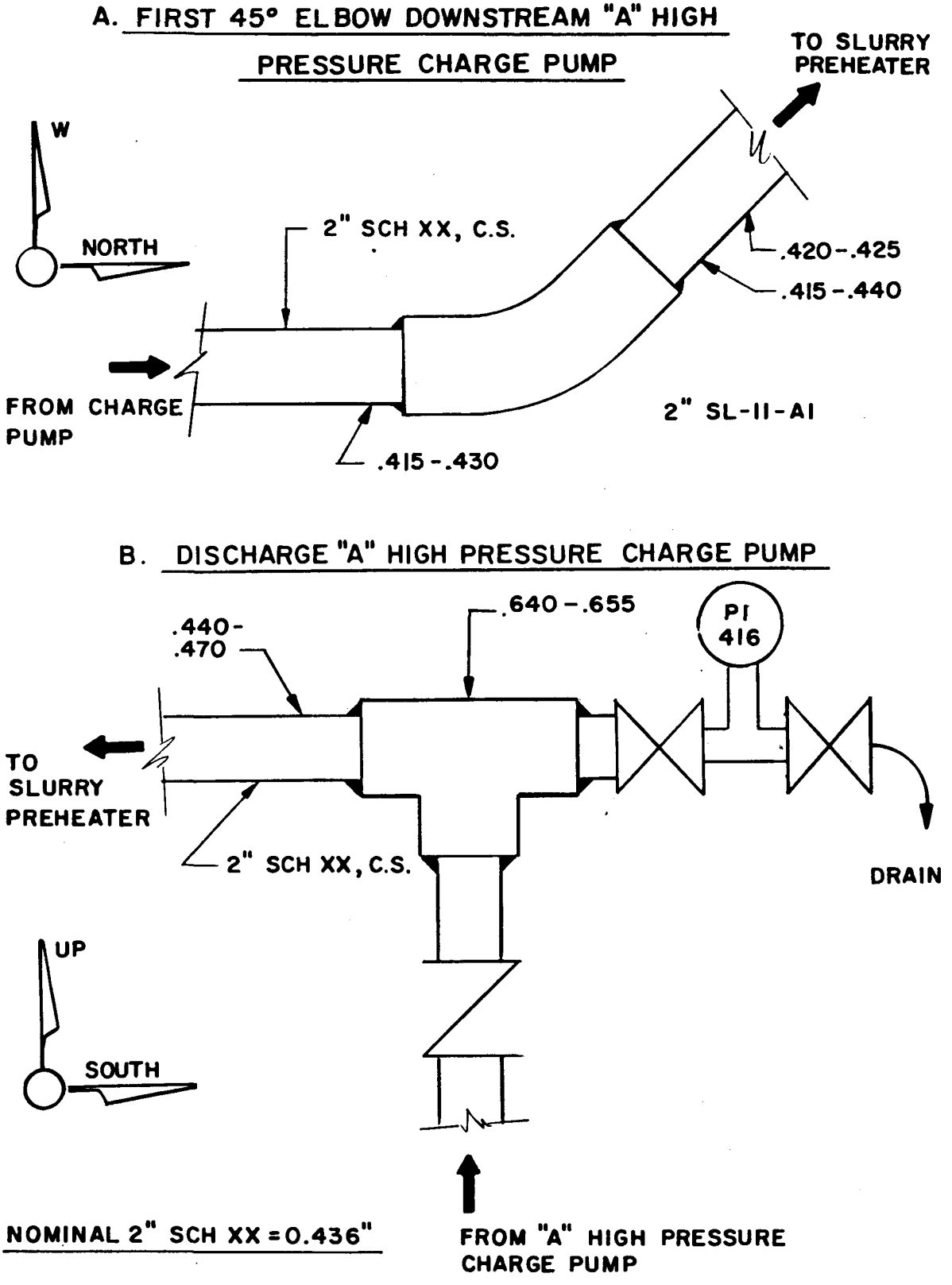

A. FIRST 45° ELBOW DOWNSTREAM "A" HIGH PRESSURE CHARGE PUMP
TO SLURRY PREHEATER
W
NORTH
2" SCH XX, C.S.
.420-.425
.415-.440
FROM CHARGE PUMP
2" SL-II-AI
.415-.430
B. DISCHARGE "A" HIGH PRESSURE CHARGE PUMP
.640-.655
PI 416
.440-.470
TO SLURRY PREHEATER
2" SCH XX, C.S.
DRAIN
UP
SOUTH
NOMINAL 2" SCH XX = 0.436"
FROM "A" HIGH PRESSURE CHARGE PUMP

FIGURE 5

DISCHARGE "B" HIGH PRESSURE CHARGE PUMP (2"-SL-12-AI)

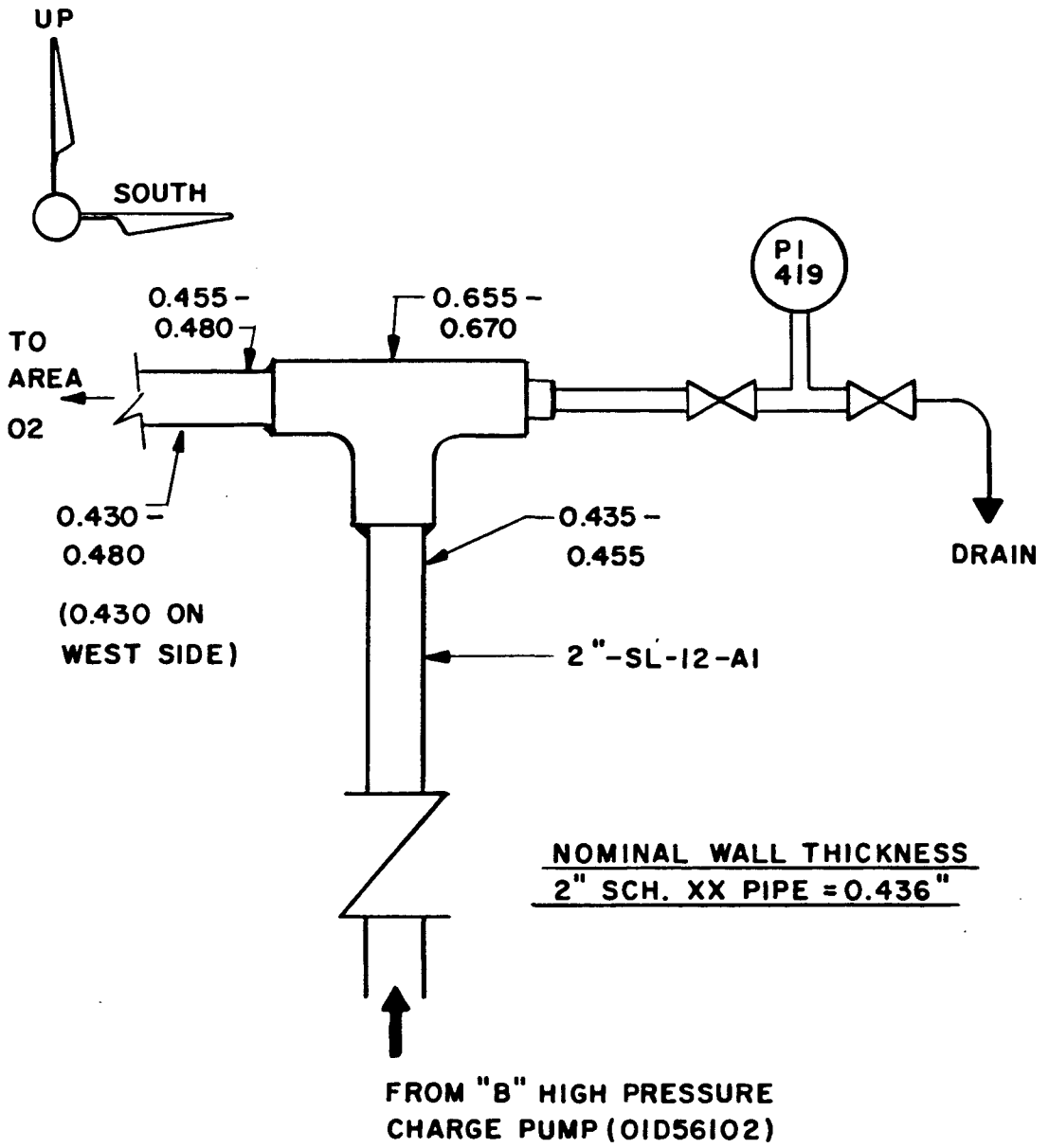
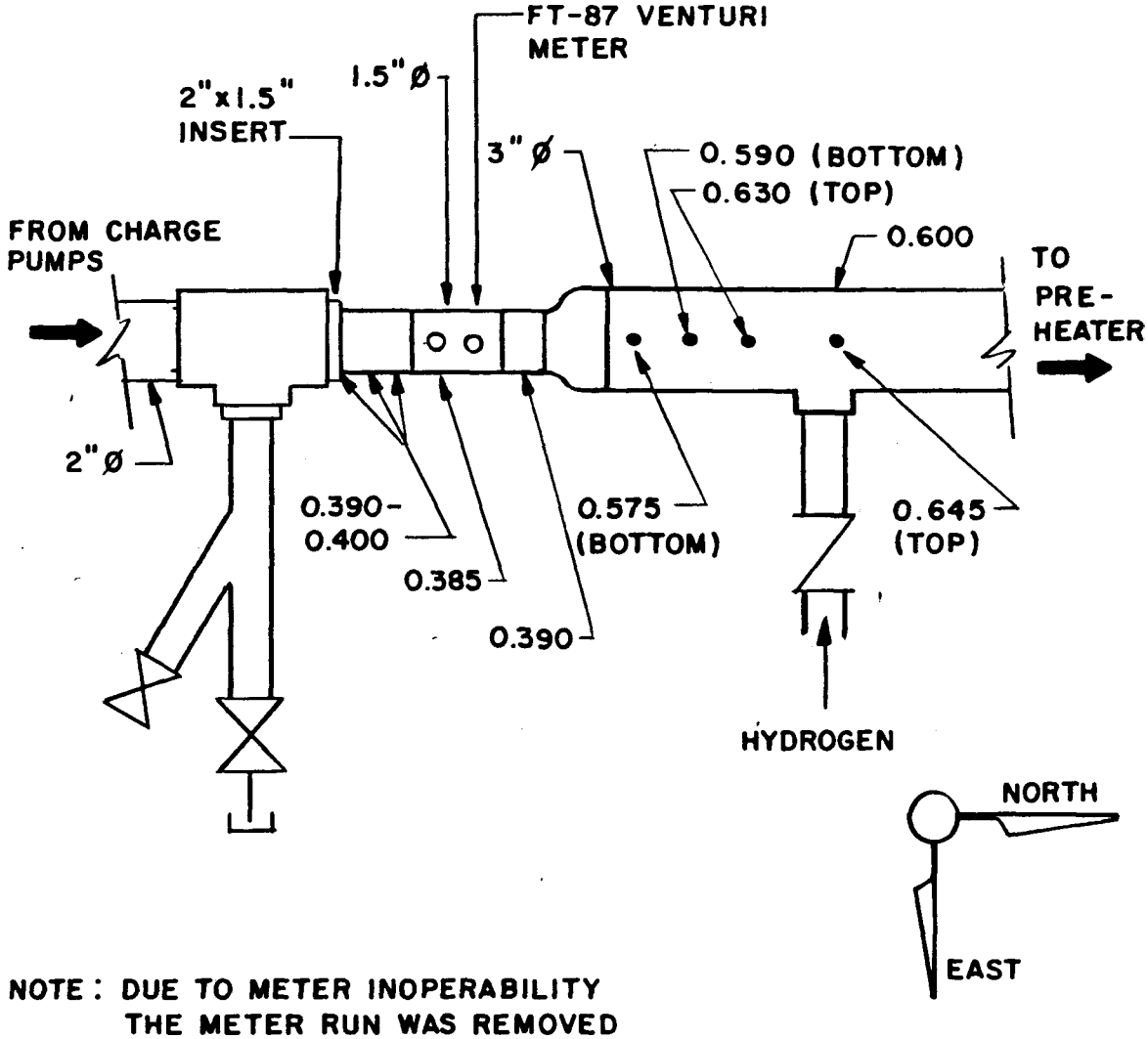


FIGURE 6

2" SL-II-AI FT-87 (VENTURI METER)



NOTE: DUE TO METER INOPERABILITY THE METER RUN WAS REMOVED AND REPLACED WITH 2" SCH. XX CS PIPE.

NOMINAL 1 1/2" SCH. XX = 0.400"
3" SCH. XX = 0.600"

B. Slurry Preheating and Dissolving - (Area 02)

1. Operation March 25 through April 1, 1978

All operation during the first eight days of the reporting period was with full dissolver at 2000 psig and 860°F outlet temperature. Operation from March 25 to March 31 was in the SRC II mode with Blacksville No. 2 coal. On March 31 and April 1, operation was in the SRC I mode with Kentucky coal.

During shutdown on April 1, new flushing procedures were used in an attempt to more thoroughly clean the 02 area vessels for inspection. After an initial flush of 8,000-10,000 lbs/hr with flush solvent for three hours, the system was flushed with wash solvent for one hour at 10,000 lbs/hr. This was followed by a four-hour period on total recycle with wash solvent at 350-400°F. When opened for inspection, the vessels were considerably cleaner than in prior shutdowns.

2. Turnaround Maintenance

During the turnaround, the following maintenance work was completed in Area 02:

a. Most of the Grayloc flanges were refaced by Gray Tool Company.

b. The middle hydrogen quench sparger assembly which had collapsed was removed from dissolver A when the vessel was opened for inspection. Because of procurement problems, incorporation of a dissolver sampling tube as part of the middle quench line (which is part of "A" head) was delayed. To meet the scheduled startup date for SRC I mode operation, dissolver B head was temporarily installed on dissolver A. Dissolver A head will not be reinstalled until it is required for SRC II operation.

c. A new head was installed on the intermediate pressure flash vessel.

d. Modifications were completed to seven process gas flowmeters to improve their serviceability and to improve the overall gas balance accuracy. Orifice dimensions were carefully checked and orifice flanges were relocated to downflow configurations to minimize buildup of entrained or condensed material at the orifice plate. Machined orifice runs were installed to improve meter accuracy and double block and bypass piping was installed to permit servicing the orifice plate without shutting down.

e. Double block and bleed valves were installed in slurry blowdown lines to the flare.

f. The high pressure flash drum sample point (204) was relocated and the stripper side draw accumulator and associated piping were removed.

g. Parallel letdown valves with double block and bleed valves upstream and downstream were installed for intermediate pressure flash drum level control in place of the single letdown valve LCV-175.

h. A new type of control valve, known as a variable orifice valve manufactured by the Willis Oil Tool Company, was installed in parallel to the existing level control valve on the high pressure flash drum (LCV-166). Two Willis block valves were installed on each side of the control valve. This assembly will test the Willis valve design which is expected to demonstrate significant improvement of control valve life in this service. Prior to the June 10 startup, however, a hydrotest revealed that the Willis valves did not hold pressure and they were taken out of service and returned to the vendor. The vendor modified the valves and returned them, but they again failed the hydrotest. At this time it was discovered that the valves leaked between the tungsten carbide seat and its stainless steel retainer which were bonded together with silver solder. With this information, the vendor was able to repair the valves and they were tested to hold leak tight at a pressure of 6000 psi in the forward direction and 3000 psi in the reverse direction. The Willis valve assembly (LCV-166B) will be reinstalled and placed in service as soon as possible.

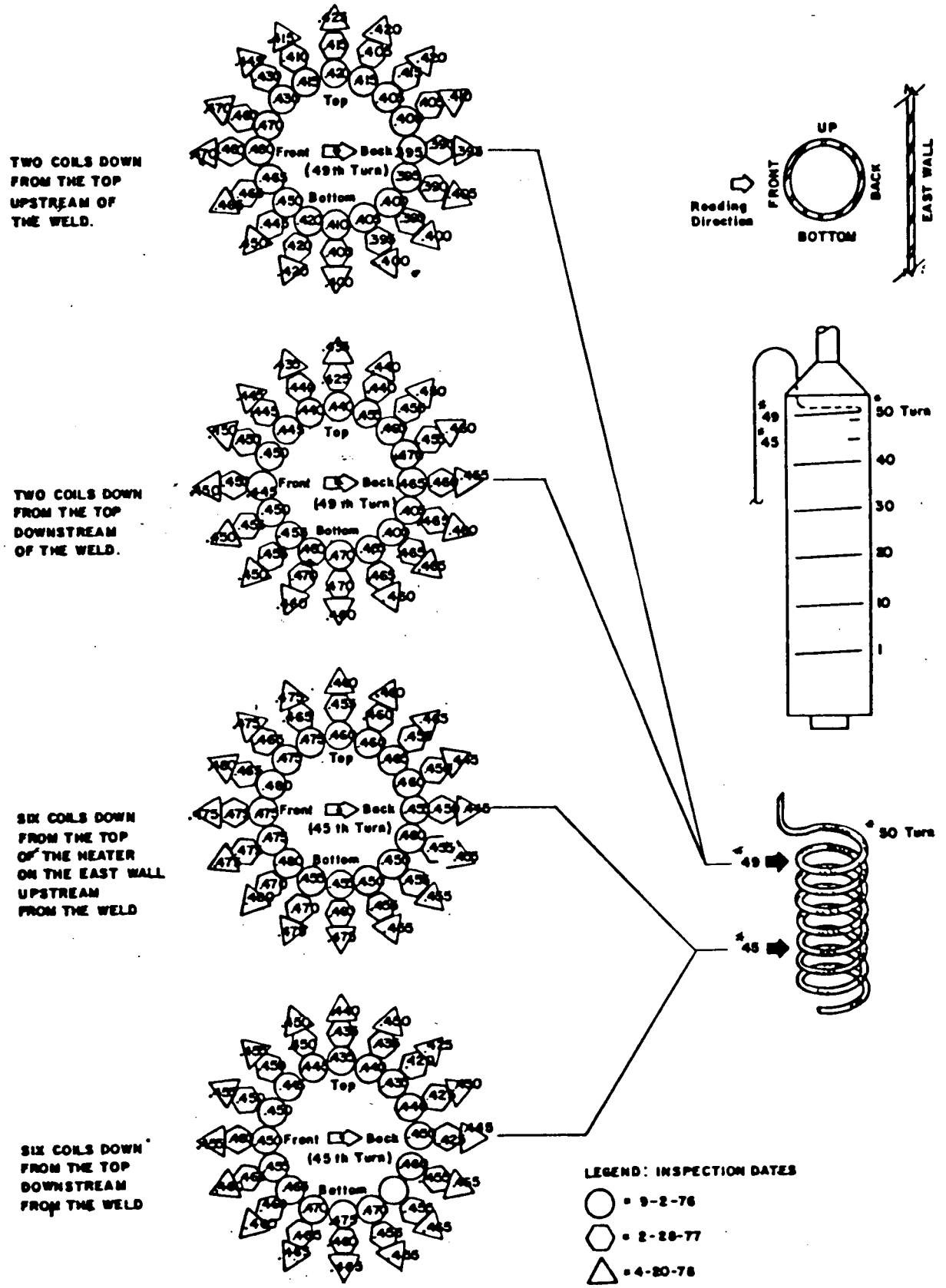
3. Inspection of Piping and Vessels

During the plant turnaround, considerable inspection effort was devoted to the reaction area. Vessels examined were the slurry preheater, the A and B dissolvers, the high pressure flash drum, the intermediate pressure flash drum, the recycle condensate separator, and the slurry recycle stripper. In addition, extensive inspection of piping and all Grayloc flanges was completed. All corrosion racks were removed and new ones installed except in the dissolver. These corrosion racks could not be installed because the quench tube on which they are mounted was damaged and was not reinstalled.

The slurry preheater coil was ultrsonically inspected for metal thinning and x-rayed to determine if substantial coking had occurred. As shown in Figure 7, no significant loss was detectable. There was an indication from radiography that some coke was deposited in the coil, but decoking was not advised.

FIGURE 7

SLURRY PREHEATER COIL INSPECTION DATA



Both the weld overlay and the cracked breather rings of dissolver A were inspected. Although the on-site metallurgical examination of the 347 SS overlay in dissolver A did not reveal any transgranular or intergranular cracks, a general attack of the 347 SS overlay was noted. The overlay displayed a feathery carbide microstructure, believed to be the result of carbon penetration. The carbon penetration appears to be greater at the bottom of the dissolver than in the upper areas. Since cracks were not detected in the overlay, the carbon penetration does not seem to have had any adverse effects on the overlay properties. The base metal underneath the cracked breather rings on the bottom and side nozzles was checked for metal loss using ultrasonics and was found to be in good condition. Since there was severe corrosion underneath the cracked breather ring on the intermediate pressure flash drum, the side outlet nozzle of dissolver A was further examined with radiography. The radiographs did not reveal any corrosion, but they did reveal a slag inclusion that has been present since fabrication. Radiographs will be made of this area during the next annual shutdown to determine if this condition is worsening.

The results (see Table A-1) from the corrosion racks which were removed from dissolver A indicate a higher corrosion rate for the base metal (2 1/4 Cr- 1/2 Mo) than what has been observed on the base metal beneath the cracked and separated breather ring on the side nozzle. The difference in rates can possibly be explained by the fact that the breather ring was protecting the base metal and, as a result, the corrosion rate was less. New corrosion racks were not installed in dissolver A.

An on-site metallurgical inspection was also made of dissolver B which has a total service history of only a few hours. Cracks have been found at the arc stops in both dissolvers A and B. However, the cracks appear to be hot tear cracks that have been present since the original fabrication.

The high pressure flash drum was examined and found to have transgranular (probably chloride) and intergranular (probably polythionic) cracking in the 304 cladding on both the head and the shell. Extensive ultrasonic inspection of the 304 SS cladding verified that none of the cracks had propagated to the base metal (2 1/4 Cr- 1/2 Mo). However, a cracked breather ring on the head was bulged and ultrasonic examination verified that approximately 0.140" of base metal had been lost. Since the head thickness was still greater than the minimum thickness specified in the data sheet, the vessel was put back into service without derating.

Inspection of the intermediate pressure flash drum revealed polythionic and chloride stress corrosion cracking of the 304 SS cladding on both the head and the shell, corrosion

(about 0.123") behind a cracked and separated head breather ring, and some cracking (probably chloride) of the 347 SS overlay on the head flange. As in the case of the high pressure flash drum, none of the cracks had penetrated the cladding. There was, however, considerable corrosion (0.123") underneath the cracked breather ring on the head. Apparently this corrosion has taken place since the last inspection (February 1977) as no corrosion was detected at that time. The on-site metallography of the 347 SS overlay of the flange did reveal some newly developed chloride cracks. Filings of this wall overlay were analyzed and confirmed to be 347 SS. The corroded 304 SS clad head was replaced with a new 347 SS weld overlayed head.

In order to minimize further corrosion of the base metal, breather rings in the vapor space of both the high pressure and intermediate pressure flash drums were modified by welding a 1/16" thick Incoloy 800 plate over the existing breather ring. A total of six breather rings on the high pressure flash drum and three on the intermediate pressure flash drum were repaired in this manner.

All the rings were dye penetrant tested and any defects were repaired. However, the ring on the HP head leaked on startup. The leak was stopped by placing a valve on the weep hole and shutting the valve. A new head without breather rings (overlayed nozzles) is expected to arrive in time for installation during the next shutdown.

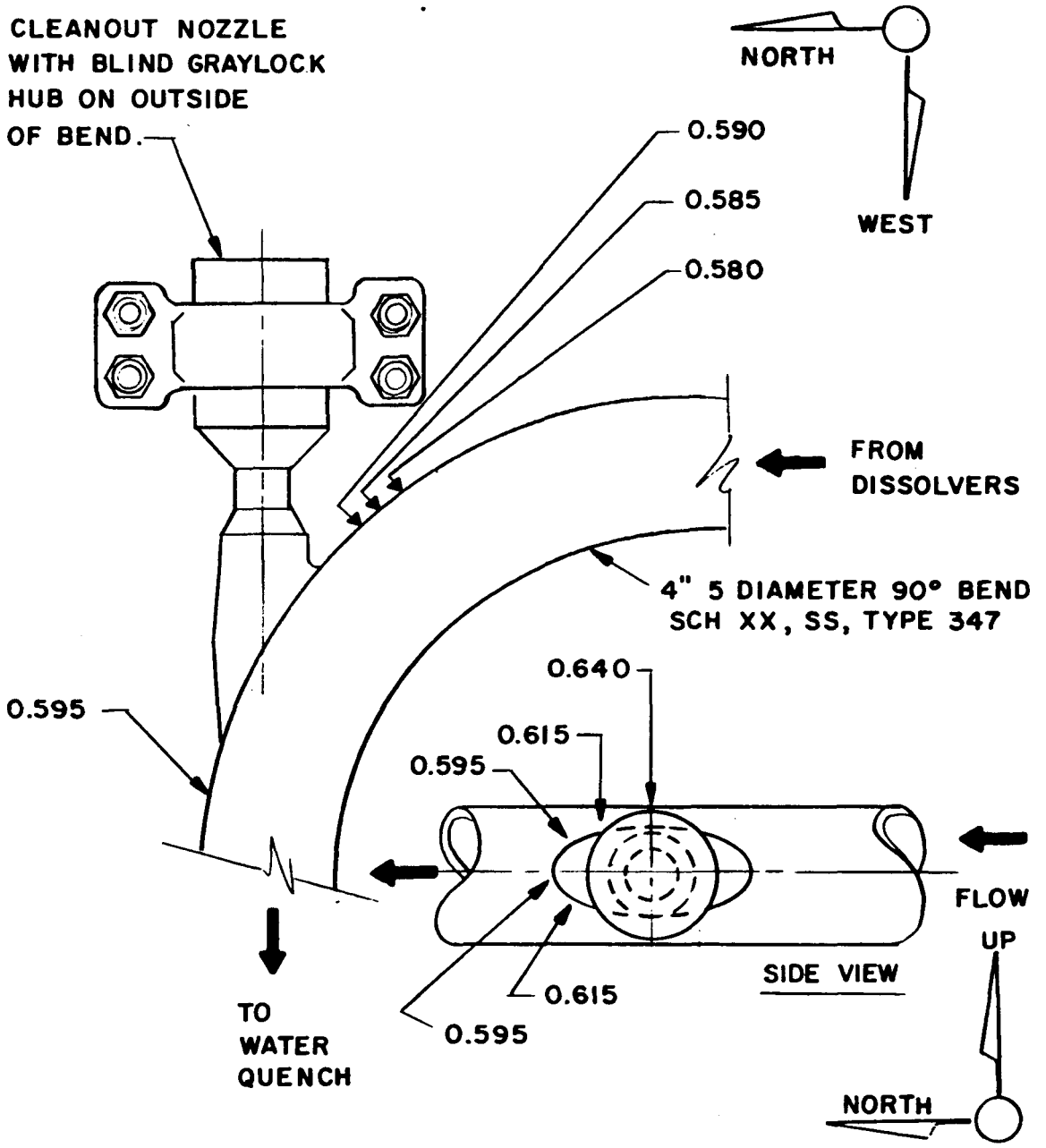
The recycle condensate separator was inspected and found to be in good condition. The only cracks found were in the 304 SS clad heads. Table A-1 shows the results from the corrosion racks which were removed.

An inspection of the slurry recycle stripper revealed no noticeable corrosion of the 304 L SS vessel. Corrosion rack data from this vessel are also shown in Table A-1. The indicated corrosion rate for this vessel may be on the low side since the corrosion rack is located at the top of the column at a relatively low temperature.

All stainless steel piping (type 347 and 304L) in Area 02 shows minimal wear. Critical areas of this piping are shown in Figures 8 through 12. Most of the metal thinning at five diameter bends is probably the result of the bending of the pipe which stretches the outside radius. All of the five diameter bends were fabricated from nominal pipe. The long radius inlet nozzle to the IP flash drum, as shown in Figure 12, shows no metal loss. All piping shown is original plant construction, except the FT-157 meter run, Figure 11A, which was installed during the SRC II modification in early 1977. The 1" pipe in this meter run was removed and inspected

FIGURE 8

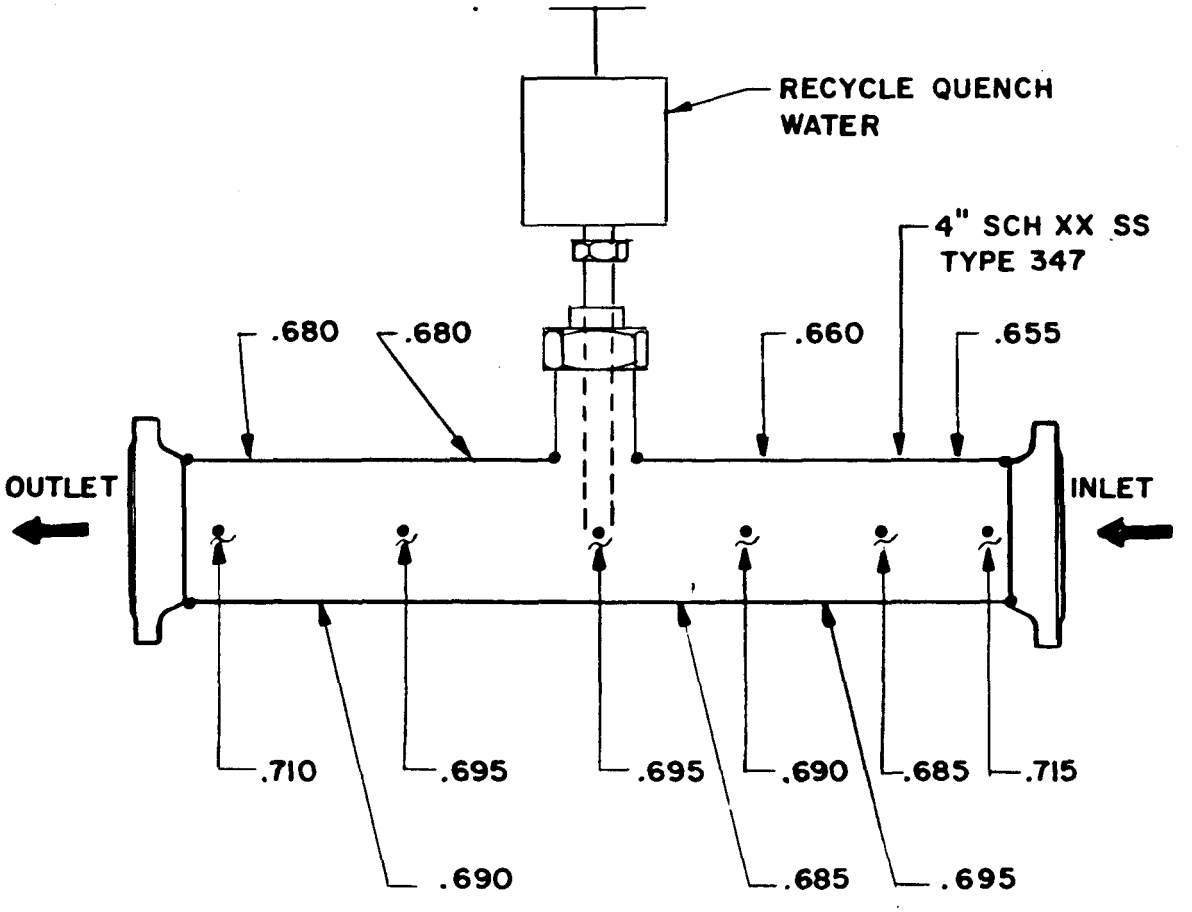
4" SL-16-A6 AT CLEANOUT NOZZLE



4" SCH. XX NOMINAL = 0.674"

FIGURE 9

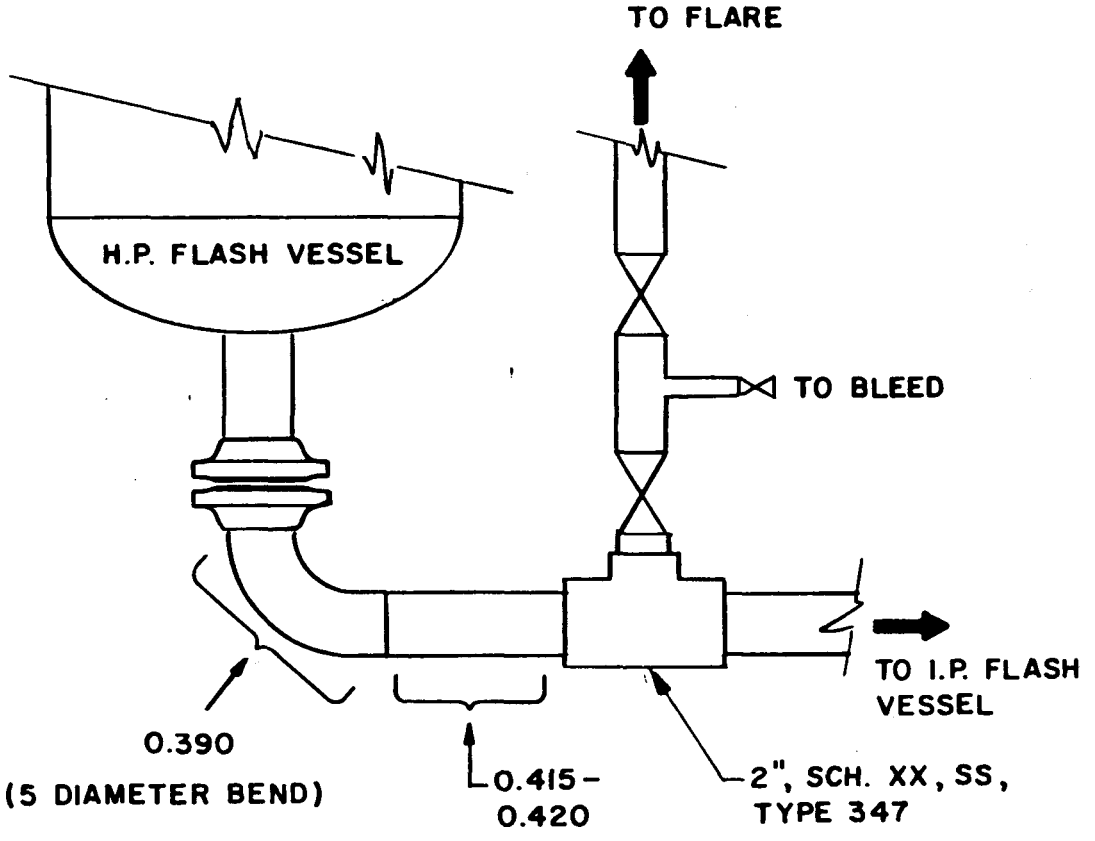
4" SL-16-A6 RECYCLE WATER QUENCH SPOOL PIECE



NOMINAL 4" SCH. XX, SS = 0.674"

FIGURE 10

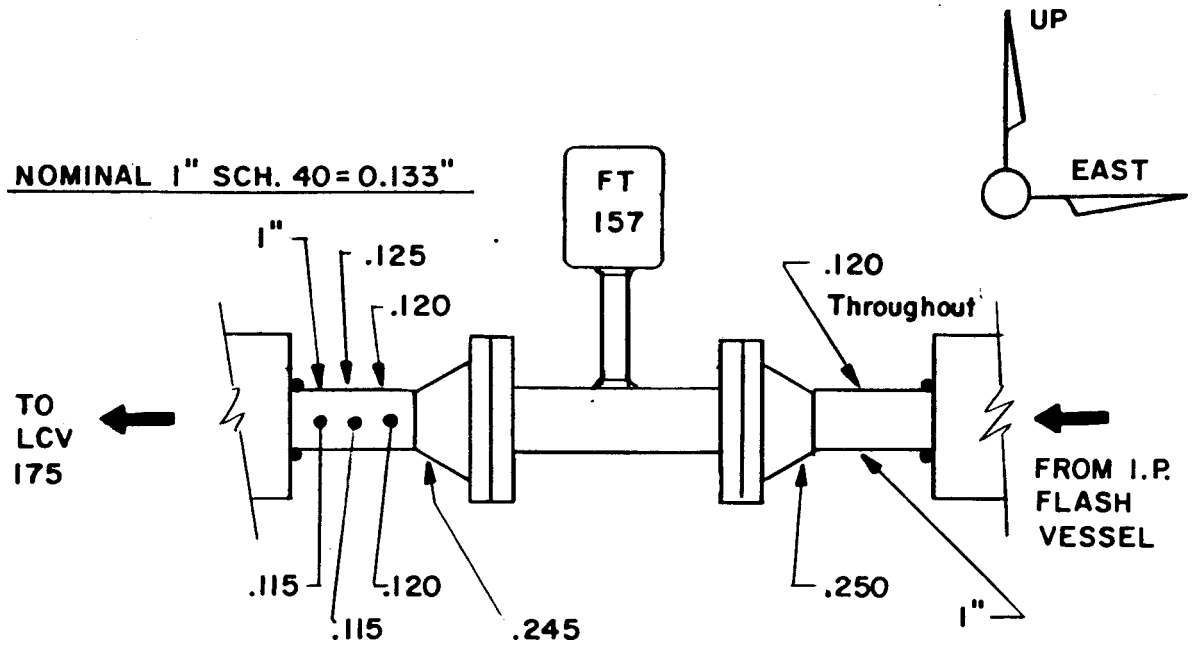
2" SL-20-A6 AT H.P. FLASH VESSEL



NOMINAL 2" SCH. XX SS = 0.436"

FIGURE II

A.- AT FT 157 (TURBINE METER)



NOTE:
FT. 1127 & 1" PIPING WAS REMOVED AND REPLACED WITH 2" SCH. 160 S.S. PIPE (TYPE 304 L)

B.- 2"-SL-21-B3, IP FLASH DRUM BOTTOMS

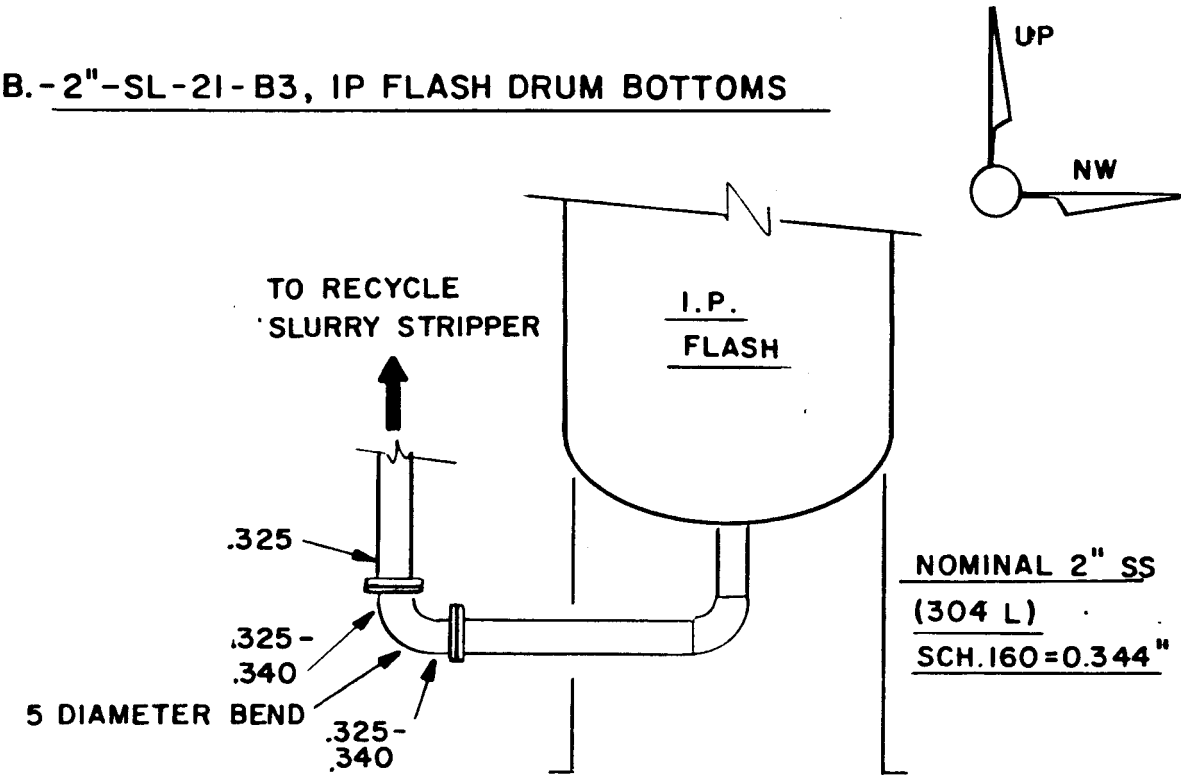
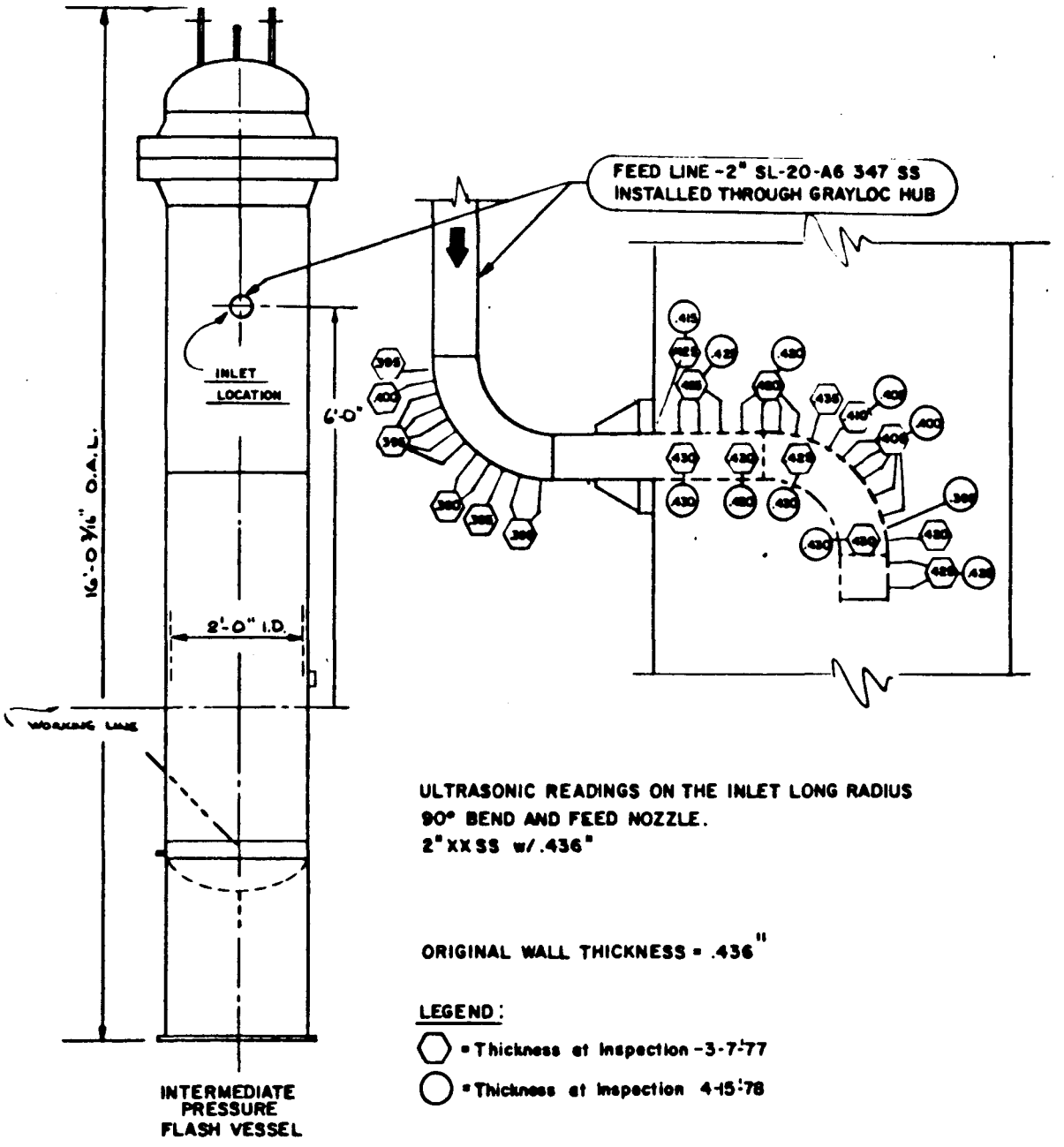


FIGURE 12

2" SL-20-AG AT I.P. FLASH VESSEL



ULTRASONIC READINGS ON THE INLET LONG RADIUS
90° BEND AND FEED NOZZLE.
2"XXSS w/.436"

ORIGINAL WALL THICKNESS = .436"

LEGEND:

⬡ = Thickness at Inspection -3-7-77

◯ = Thickness at Inspection 4-15-78

internally. It showed no observable erosive type wear. A normally observed protective scale was seen. The unusually thin wall thickness of this pipe was attributed to the installation of schedule 40 pipe instead of specification schedule 80 pipe.

The carbon steel piping between the intermediate pressure flash drum level control valve (LCV-175) and the slurry recycle stripper and the carbon steel piping between the slurry recycle stripper and Areas 01, 03, and 04 has been severely thinned. Figure 13 shows a schematic of the survey of the affected piping. Figures A-1 through A-11 show the data obtained in greater detail. Most of this piping is approximately three years old. Exceptions are the first 90° bend downstream of LCV-175 in Figure A-1 and 1" HL-134-E1 in Figure 13 which are only one year old, and 2" SL-21-E1 in Figure 13, A-10 and A-11 which is original plant piping. Experience has shown that carbon steel gives good performance where fluid velocities are low.

Corrosion in unjacketed lines was most severe where process temperatures were highest (i.e., just downstream of LCV-175 and the slurry recycle stripper). Corrosion rates decreased along the lines as the process temperature decreased. The maximum corrosion rate at the five diameter bend downstream of LCV-175 was 0.063"/year. The maximum corrosion rate at the first 90° bend downstream of the slurry recycle stripper was 0.040"/year.

Thinning in the Dowtherm jacketed line from the slurry recycle stripper to the new vacuum flash drum in Area 04 (1" SL-134-E1, Figure 13) was most severe at the discharge or hot end of the line where it exhibited a maximum corrosion rate of 0.168"/year. Considerable vaporization and resultant higher slurry velocities may have contributed to the temperature effect in this high wear area.

4. Operation June 10 through June 24

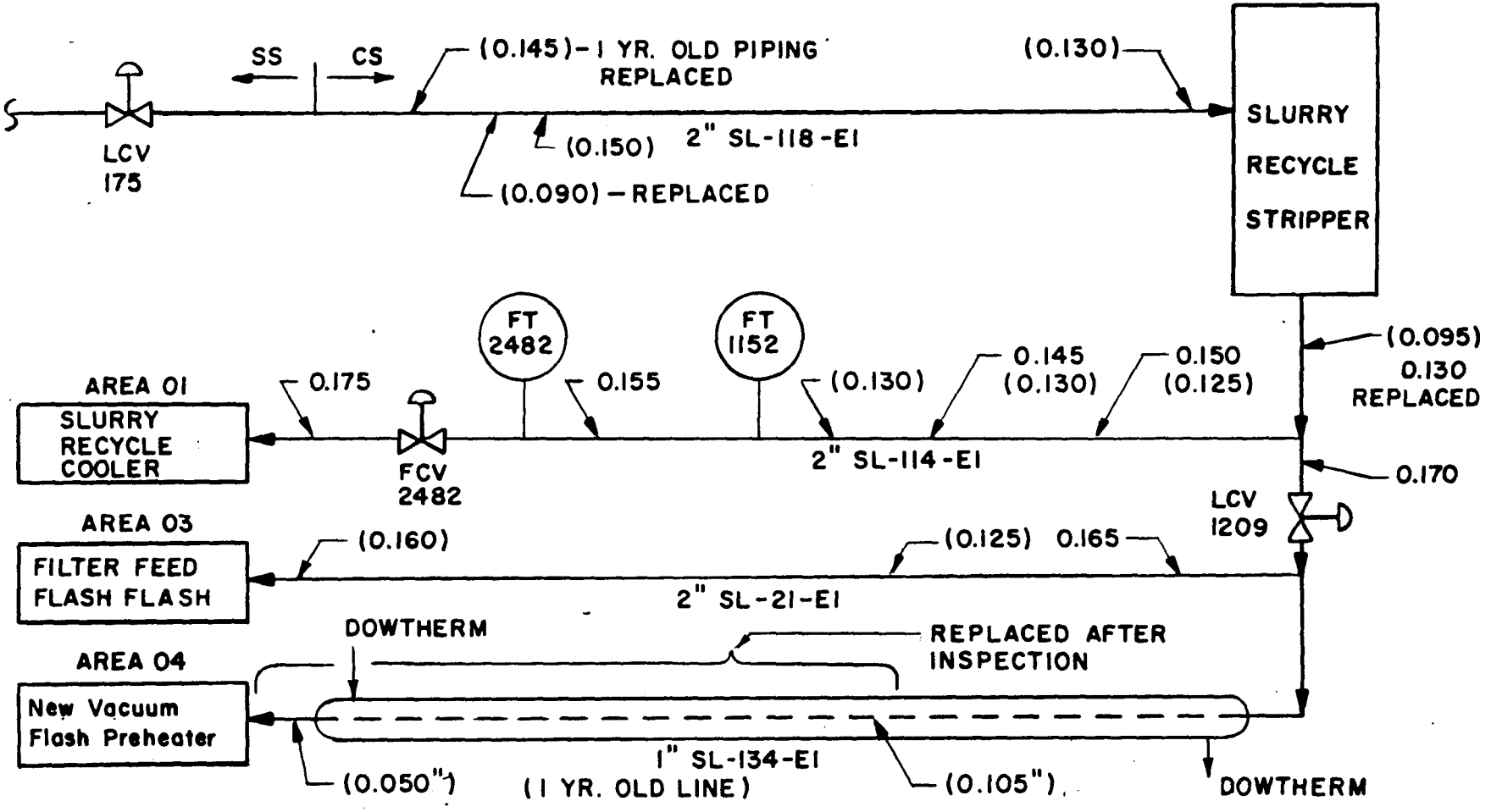
Coal slurry processing was resumed on June 10 after a number of processing problems with slurry level control valves were resolved. Problems with plugging and trim breakage in these level control valves are still frequently encountered during startup after a prolonged shutdown. All operation was in the SRC I mode using one half of dissolver A (outflow through the side nozzle). Dissolver target conditions varied between 840° and 860°F outlet temperature and 1500 and 1800 psig dissolver pressure. The hourly on-stream factor adjusted for downtime not related to process requirements was 26.3%.

FIGURE 13

SURVEY OF CARBON STEEL SLURRY PIPING

NOMINAL WALL THICKNESS 2" SCH. 80 = 0.218"

THICKNESS NOTED IN ( ) ARE TAKEN AT LONG RADIUS BENDS



C. Mineral Separation and Drying - (Area 03)

1. Operation March 25 through April 1, 1978

The only vessels in Area 03 necessary for SRC II operation are the filter feed surge vessel, the filter feed flash vessel, and the recycle process water tank. The filter feed surge vessel and the filter feed flash vessel were used to accumulate off-spec oil. The recycle process water tank was used in its normal function as a quench water reservoir and as a final oil-water separator.

The major engineering and maintenance efforts in Area 03 were directed toward the startup of Filter C, a new design rotary precoat filter. Because the objective of the scheduled Filter C test program was to demonstrate the operability of the unit and not to produce specification product (SRC I, low ash vacuum bottoms), a plan to mix the cake leg with the filtrate downstream of the filtrate receiver was adopted. These modifications eliminated the necessity of using the mineral residue dryer which in the past has been a major source of production curtailment. In addition, the mineral residue dryer would have required extensive precommissioning maintenance which would have delayed the filter test program.

An alternate method for preflashing the filtrate stream was also designed. In previous SRC I operation this preflashing was done in the light ends column in order to prevent substantial build-up of coke in the preheater. To avoid this problem during the Filter C test program, modifications were made to preflash the cake leg/filtrate stream in the small vacuum flash drum before it entered the fired preheater, thereby reducing the sensible heat required for operation of the larger vacuum flash drum.

2. Turnaround Maintenance

Inspection, modification and maintenance work in Area 03 from April 1 through May 3, 1978 included the following:

a. Most of the vessels in Area 03 were opened and inspected.

b. New tubes were installed in wash solvent exchanger A and it was relocated to facilitate the installation of double block and bleed valves on the Dowtherm supply and return lines.

c. Exchanger B was cleaned by hydroblasting.

d. The filter feed surge vessel recirculation exchanger was hydroblasted, inspected and four 90^{0} bends with flanges were replaced.

e. The filter feed flash recirculation exchanger was hydroblasted and one tube was replaced.

f. The bypass block valve around the high pressure water pump pressure controller was replaced.

g. Safety valves were removed, inspected, and reinstalled.

h. Utility tie-ins for the Lummus antisolvent deashing system were completed.

Before initial testing of Filter C began, the drum was modified to relocate filtrate drain points from the leading to the trailing edge of the filter drum panels. New piping for Filter C was flushed with water and the filter was closed and ready for checkout May 3. A representative from Reliance Electric checked and adjusted the drive system on May 4. Operator training sessions were held May 4 and May 5.

3. Inspection of Piping and Vessels

The major items that were inspected in the mineral separation area were the filter feed flash vessel, filter feed flash recirculation exchanger, filter feed surge vessel recirculation exchanger, and the precoat slurry recirculation exchanger. As shown in Figure 14, the filter feed flash vessel has lost some metal. Data from the corrosion racks (see Table A-1) indicate a high rate of corrosion. All of the above heat exchangers have a history of piping wear due to high operating temperatures, high liquid velocities, and high solids content. Wear is of both the erosive and corrosive types. There is a general thinning of the entire pipe wall as well as severe goughing downstream of surface irregularities. Figures 15 through 18 show the data obtained upon inspection of these exchangers. They were repaired as noted and returned to service.

4. Operation May 5 through June 24, 1978

The initial pressure test of Filter C revealed many leaks. The most serious leaks were in the packing glands of the shaft and sight glasses. Examination of the packing from the shaft packing glands indicated that very little of the original Grafoil ribbon packing had been compressed. The initial shape of the ribbons was still intact in most cases. After all the shaft packing had been replaced with braided graphite packing, the drum shaft bound up in the packing gland on the filtrate separator. The shaft was realigned, but continued to bind when the packing follower was tightened to more than half of its design loading.

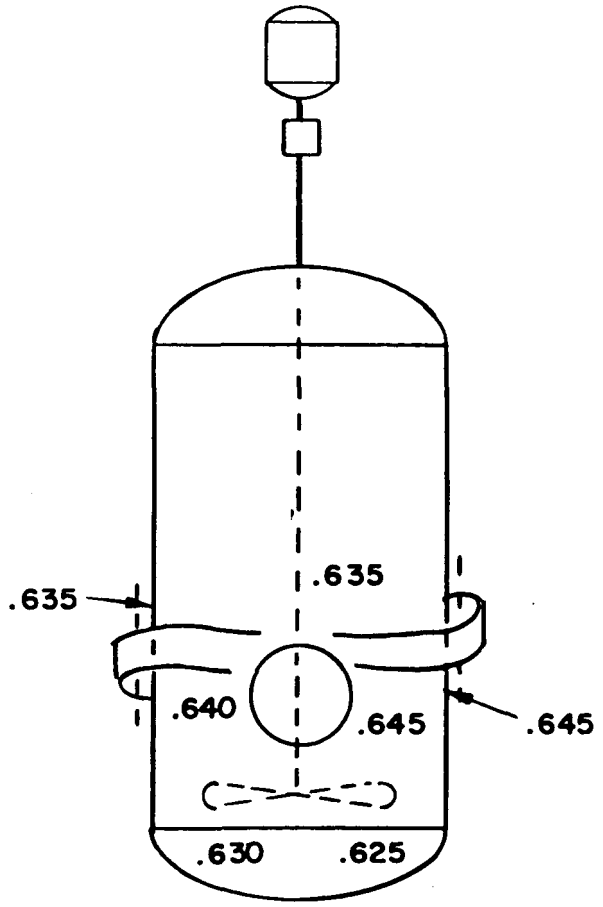
On May 10, the carbon thrust bearing on the drive end of the shaft failed at 50 psig filter pressure. (The shaft thrust was calculated to be over 28,000 lbs at 250 psig design

FIGURE 14

FILTER FEED FLASH VESSEL , EQUIP. NO. 03D75506

NOMINAL WALL
THICKNESS = 0,687 "

TOP & BOTTOM =0.687"



6'-0" I.D. x 9'-0"
DESIGN 175 PSIG @ 175°F

FIGURE 15

FILTER FEED FLASH VESSEL SIDE-ARM HEAT EXCHANGER, (EAST BANK)

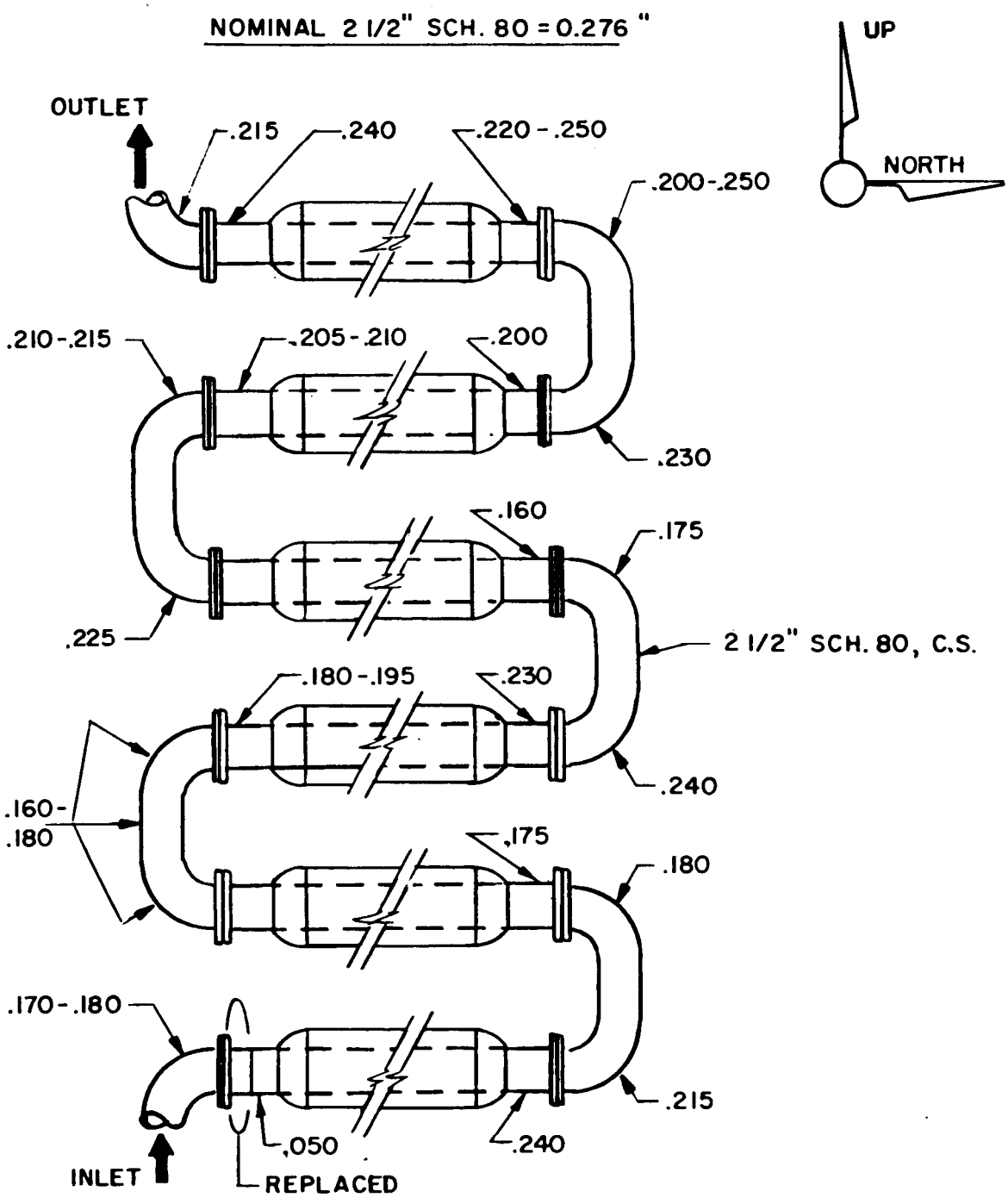
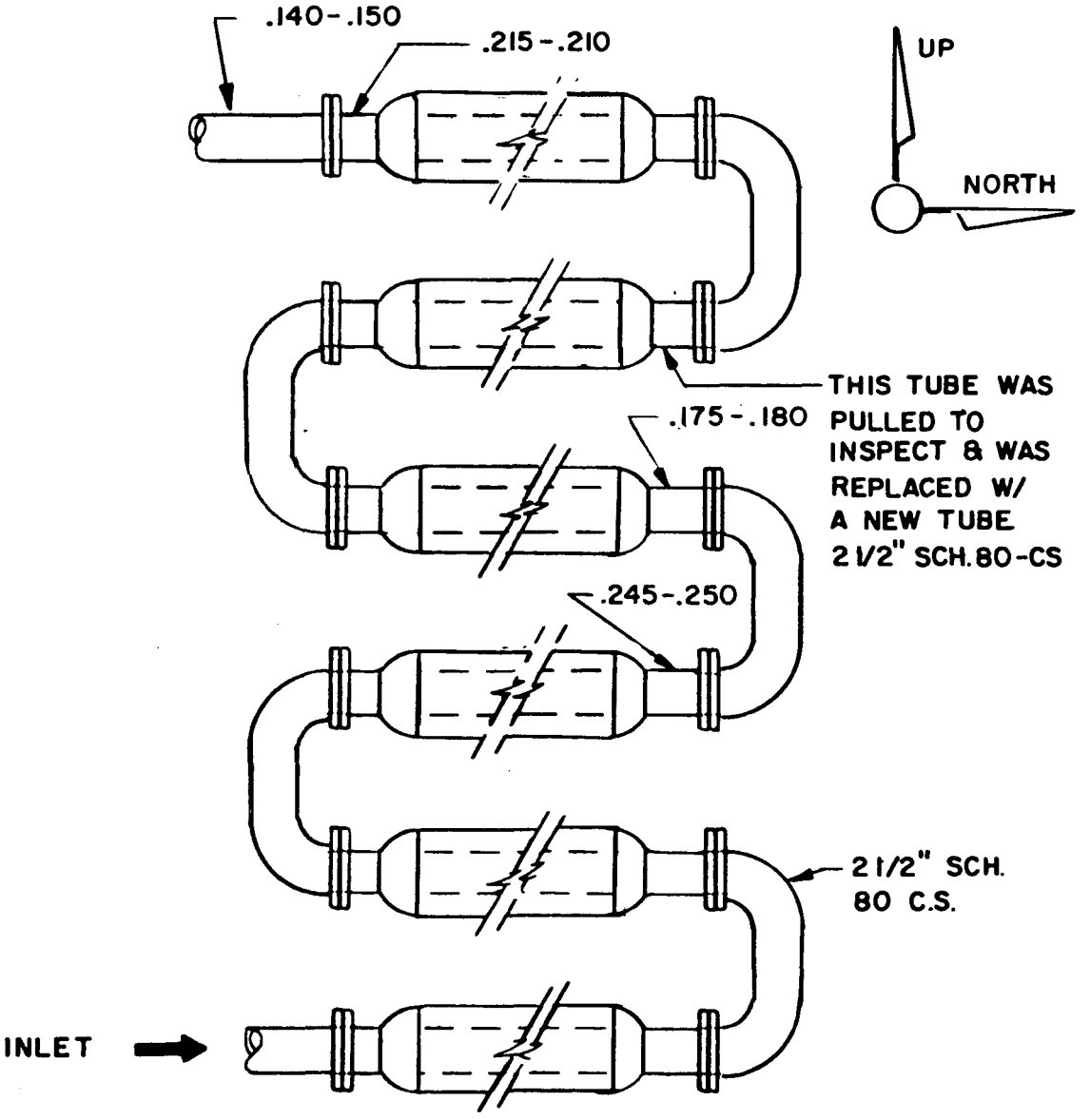


FIGURE 16

FILTER FEED FLASH VESSEL SIDE-ARM HEAT EXCHANGER, WEST BANK



NOMINAL 2 1/2" SCH. 80 = 0.276"

FIGURE 17

PRECOAT SLURRY RECIRCULATION EXCHANGER (03D30603)

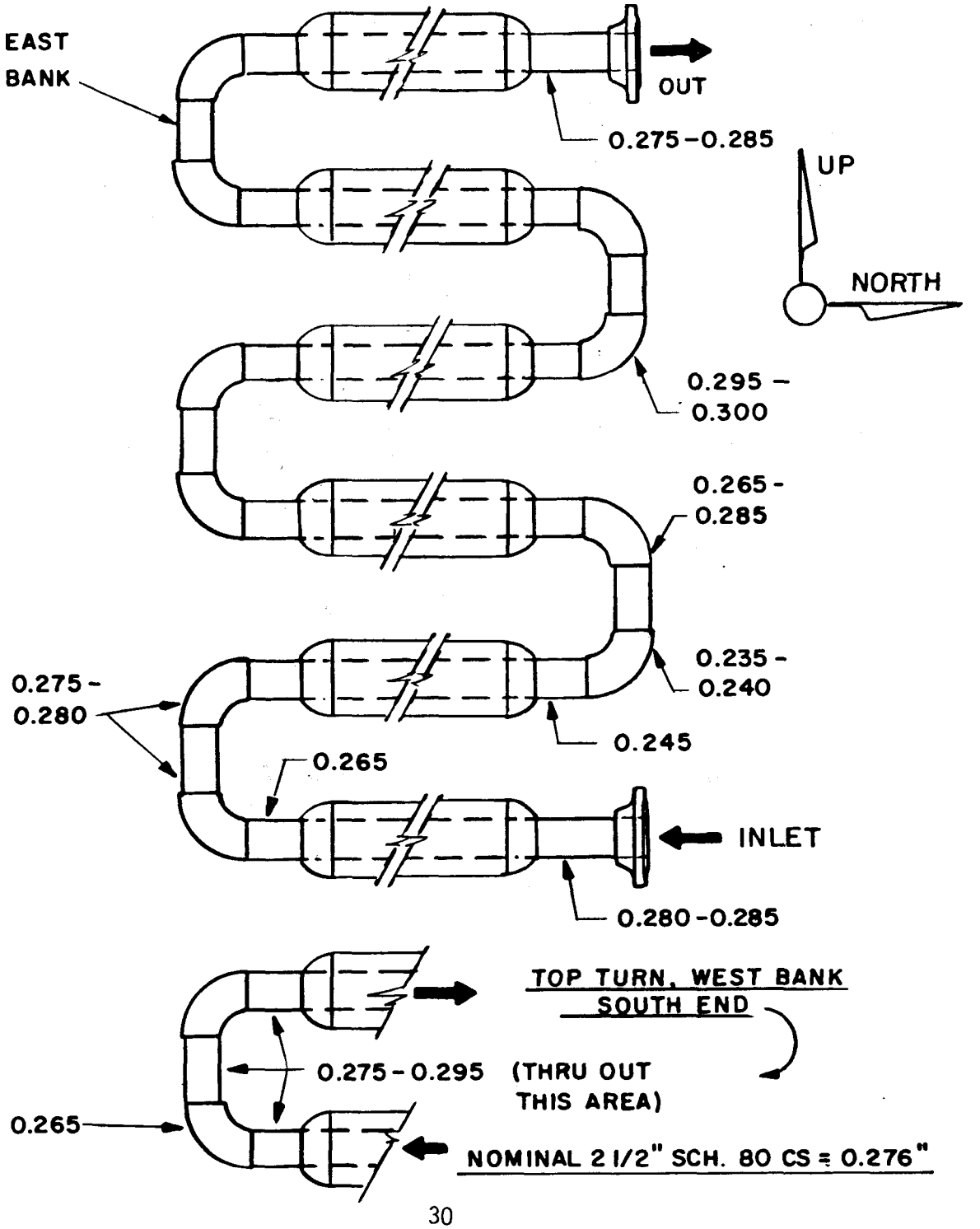
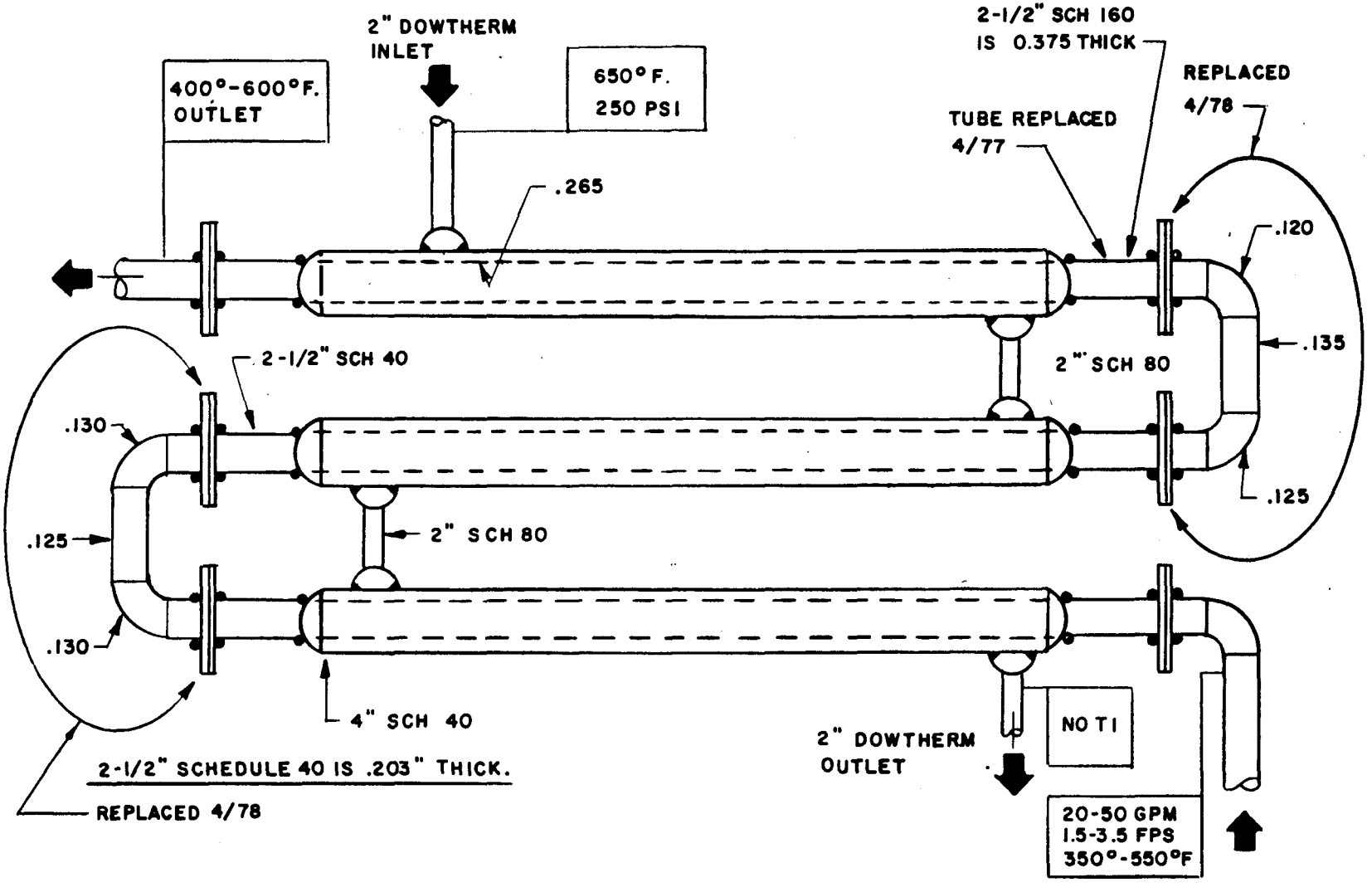


FIGURE 18

FILTER FEED SURGE VESSEL RECIRCULATION EXCHANGER



pressure.) The carbon thrust bearing had not been designed for the high thrust developed on the shaft from differential pressure between the filter tub and the filtrate receiver. A new roller type thrust bearing and channel iron support member were designed by Stearns-Roger and installed by P&M maintenance. The support member was subsequently reinforced and additional holddown bolts were installed by P&M maintenance after it was discovered that the support deflected over 1/8" when filter pressure was increased to 100 psig.

While the problems with the thrust bearing were being resolved, all seven of the leaking sight glasses were removed, repacked, bench tested to 300 psig and reinstalled. As a result of excessive leakage during operation, the sight glasses were again removed June 20. Inspection of the sight glasses showed extensive chipping. Two of the seven glass assemblies were salvageable. These were installed above the knife and at the knife sight port. New sight glasses were expected to be available during July.

As the filter drum packings were tightened to prevent excessive leakage at operating pressure (50 psig), problems with the drum drive system developed. The dual drive belts were undersized for the drum horsepower requirements. The belts slipped and had to be replaced several times. At drum speeds under 5 rpm, the drive motor overheated and tripped out. Subsequent tests showed that at 10 rpm the drive output was 21 horsepower; approximately 7 horsepower over the maximum rating for the gear reducer. On May 27, the filter drum drive unit was modified with new four belt sheaves to transmit power from the motor to the gear reducer. To eliminate overheating, the drive motor sheaves were resized. Resizing the drum sheaves changed the drum speed range from 2 through 10 rpm to 1.5 through 7 rpm.

Modifications to the filter were completed May 30. After several hours of solvent circulation, however, the screen appeared to be blinded by solids in the solvent loop. At the request of Johns-Manville, the filter was shut down, the screen hydroblasted, and all spray nozzles removed, cleaned, and checked for proper orientation. On June 2, again at the request of the Johns-Manville representative, the filter was precoated without a basecoat in an attempt to remove solids from the process solvent. Failure of the tub level indicator allowed the tub to overflow during precoating, resulting in a precoat of approximately one inch in thickness. After an attempt at filtration and subsequently poor results, the filter was shutdown for inspection. Again the screen was hydroblasted, the tub and shell were cleaned and the sluice nozzles were removed and cleaned. On June 7, a successful precoat was applied and first hydraulic test was completed. The filter was again precoated on June 8 and second hydraulic

test was completed. While raw solvent was conditioned to replenish depleted process solvent inventory, the filter was shutdown for inspection and necessary maintenance work. Because of recurring high pressure sluice nozzle pluggage, two fiber-glass cartridge filters were installed in parallel on the supply line near the filter. Unfortunately, these cartridges disintegrated at operating pressures and had to be removed. The filter was precoated again on June 15 and the third hydraulic test was completed on June 16. Prior to sluicing the heel, coal slurry was fed to the filter for several hours. After that, the remaining heel of precoat could not be sluiced from the screen, indicating possible plugged sluice nozzles. The filter was shut down to clean the screen and solve the problem of tub flushing during the sluicing cycle.

To minimize the possibility of plugging in the filter tub drain, the external piping from the tub drain to the mineral residue feed surge vessel was re-routed. In addition, feed to the tub trough sluice nozzle was changed from the low pressure to the high pressure header and the tub sluice nozzle size was increased from 0.087" to 0.125".

At the end of the reporting period the filter was awaiting feed which had been interrupted because of problems in the vacuum flash system.

5. Pump Performance

Pump maintenance in this area was primarily required for the replacement of seals on pumps used in the operation of Filter C. Each of the pumps listed below required at least one new seal during the reporting period:

- Precoat Slurry Circulation Pump
- Filter Feed Surge Vessel Circulation Pump
- Filter Feed Flash Vessel Circulation Pump
- "A" Filtrate Pump
- "A" Filter Feed Pump

D. Solvent Recovery - (Area 04)

1. Operation March 25 through April 4, 1978

Both vacuum flash systems were in service early in the reporting period with reactor product being processed in the new system and reclaimed solvent in the old. After all SRC II products had been fractionated and transferred to the tank farm, SRC I products were processed through the vacuum flash and fractionation systems. Fractionation of all products was completed April 4 and the columns were shut down.

2. Turnaround Maintenance

Work completed between April 4, 1978 and June 6, 1978 included the following:

a. Most of the plugged tubes in the vacuum flash overhead air-cooled exchangers were cleared by steaming and hydroblasting. Several plugged lines in area 04 were also cleared by hydroblasting.

b. The light ends column reboiler bundle and the wash solvent column reboiler bundle were removed and hydroblasted to remove normal coke deposits.

c. The light ends column pressure control loop was relocated, heat traced and insulated to prevent plugging by condensed material.

d. The demister pad in the new vacuum flash drum was removed because of differential pressure problems experienced during previous operation.

e. New nozzles were installed in the primary and secondary jets of the vacuum flash steam eductor system.

f. A new 1 1/2" Dowtherm jacketed line was installed to replace the one-inch jacketed feed line from area 03 to the new vacuum flash preheater. The one-inch line (SL-134) had shown significant metal loss, and the larger line was designed to keep velocities below 10 ft/sec with the increased flow due to blending filter cake slurry with filtrate during SRC I operation.

g. Replacement of the old vacuum flash drum bottoms recirculation line (CP-27) with 316L SS was completed during the reporting period, but the clamp-on Dowtherm jackets purchased for heating the line were not installed because of serious defects in their construction.

h. The new four-inch alonized coil was installed in the new vacuum flash drum preheater. This four-inch coil was originally ordered for SRC II operation but was not available during the first SRC I to SRC II conversion. A back-up six-inch carbon steel coil was used throughout SRC II operation but was replaced with the four-inch coil to improve heat transfer. Heat transfer was a problem with the six-inch coil, not only because of its size, but also because neither the Thermon heat transfer cement nor the tracing would remain bonded to its surface.

i. All block valves with welded Dowtherm jackets were replaced with new valves and clamp-on type jackets.

j. The main vacuum flash feed accumulator pump and vacuum flash bottoms pump B were rebuilt.

k. All Lummus project tie-ins were completed.

l. Tie-ins were completed to the new vacuum flash drum down leg and recirculation pump suction piping for the future connections to the Rexnord cooler. The new vacuum flash drum level control valve to the Sandvik belt (LCV-1465) was removed from the down leg for use on the new line to the Rexnord cooler.

3. Inspection of Piping and Vessels

The vacuum flash system, the wash solvent column, and the light ends column were thoroughly inspected. The only items that required any maintenance were the wash solvent overhead cooler and the trays in the wash solvent column (WSC). The cooler was replaced with a 316L SS cooler because of severe corrosion of the carbon steel tube sheet.

Both vacuum flash drums, the vacuum flash preheater, the preheater transfer line (CS-4-E2) and the vacuum flash condensate drum were inspected and only the preheater transfer line was found to have any metal loss. This line, as shown in Figures 19 and 20, was installed one year ago to replace a previously corroded/eroded line. It was used intermittently during the year on SRC II operation. Some wear is evident at 90° bends. The 316L SS test spool piece installed in this line one year ago shows no signs of wear. The results obtained from the corrosion racks that were removed from the old and new vacuum flash drums and from the vacuum flash condensate drum are shown in Table A-1. The corrosion rates are similar to previous experience and appear to be representative of vessel performance.

Inspection data for the wash solvent column show some corrosion (approximately 0.005"-0.015") of the upper half of the column but no corrosion of the lower half. However, the carbon steel reboiler has lost most of the 0.125" corrosion allowance and will require replacement within a year. As shown in Figure 21, only the 316 SS tubes in the WSC reboiler tube bundle are showing any signs of corrosion and that is only light pitting. All the remaining tubes (317 SS, Incoloy 800, Hastelloy G) are not corroded. In order to test the various tray materials, the column was retrayed with five different materials (316 SS, Hastelloy G, Hastelloy C, Incoloy 800, and Incoloy 825). All of the downcomers, the hardware, and the majority of the trays were made from Hastelloy G. One tray of each of the above

FIGURE 19

3" C.S.-4-E2 AT OLD VACUUM FLASH DRUM PREHEATER

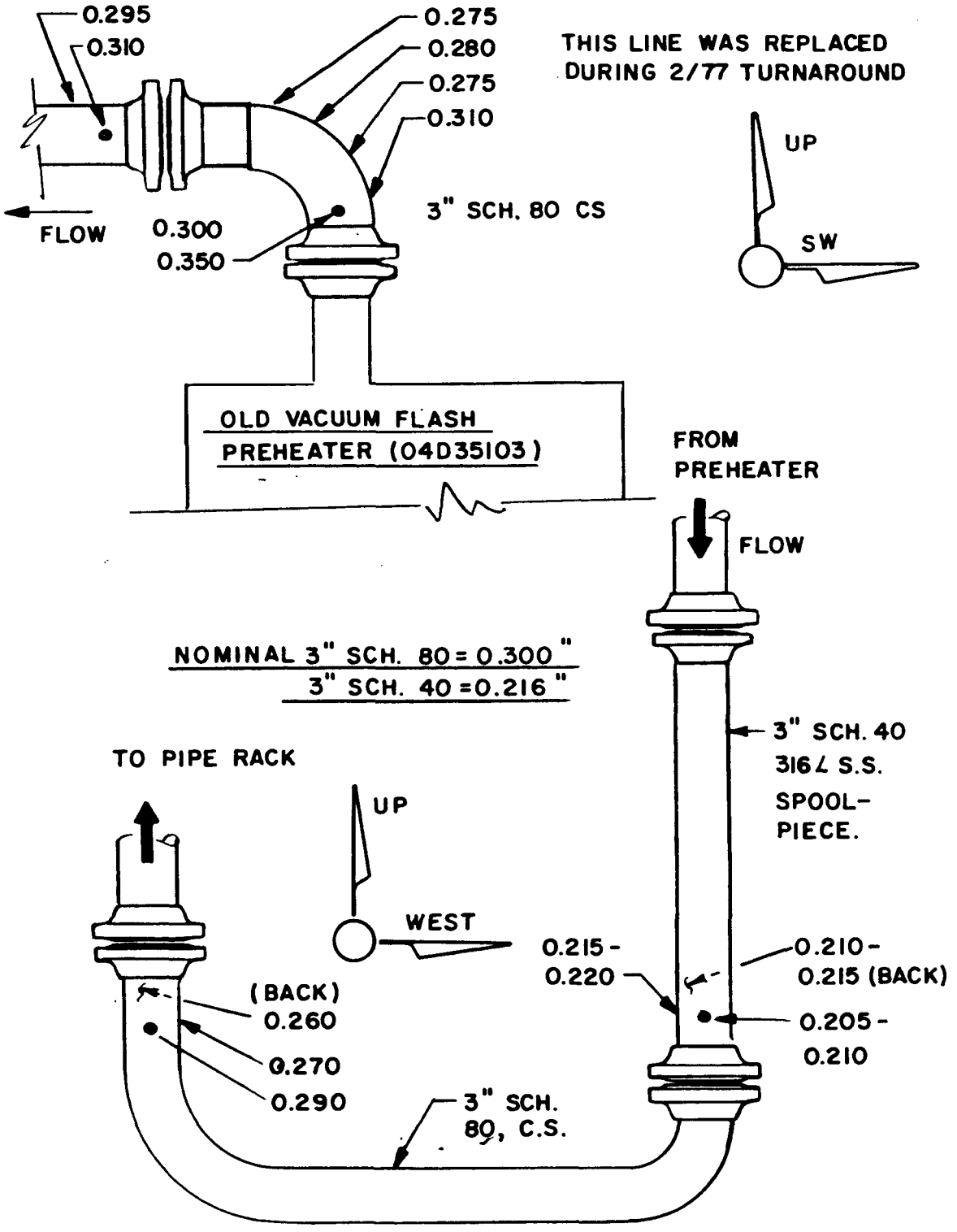
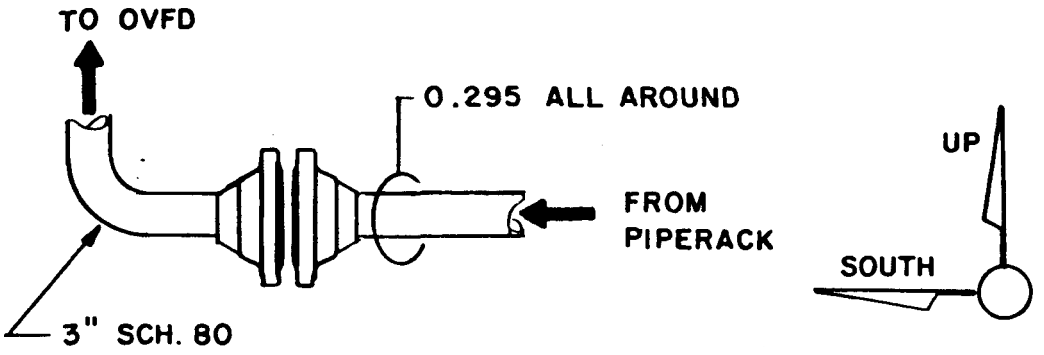


FIGURE 20

A.- 3" C.S.-4-E2



B.- 3" C.S.-4-E2 (DOWNSTREAM)

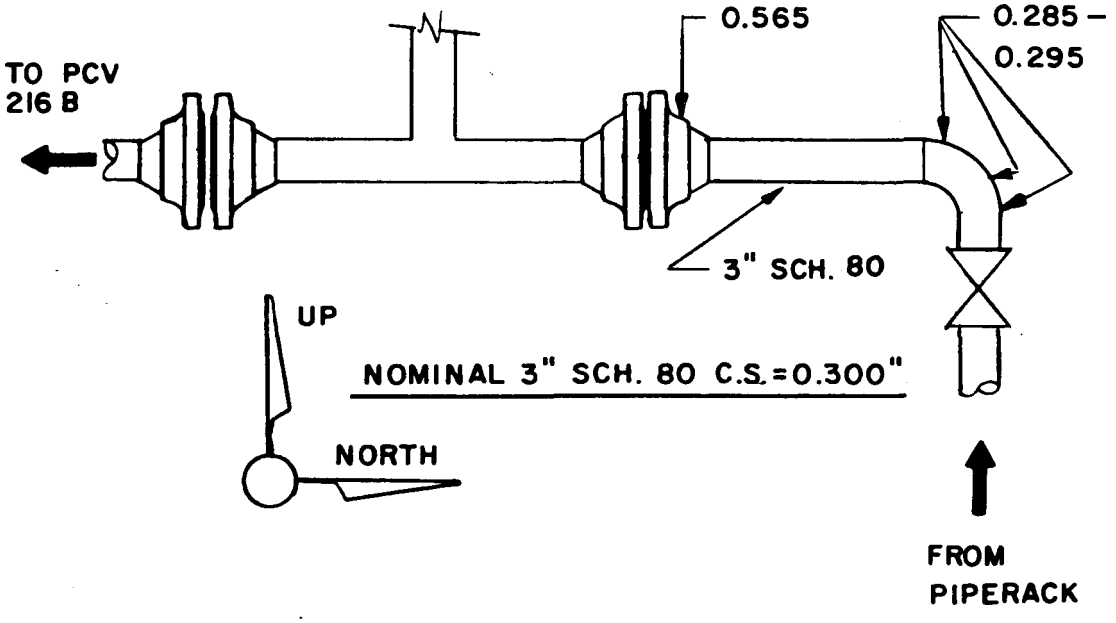
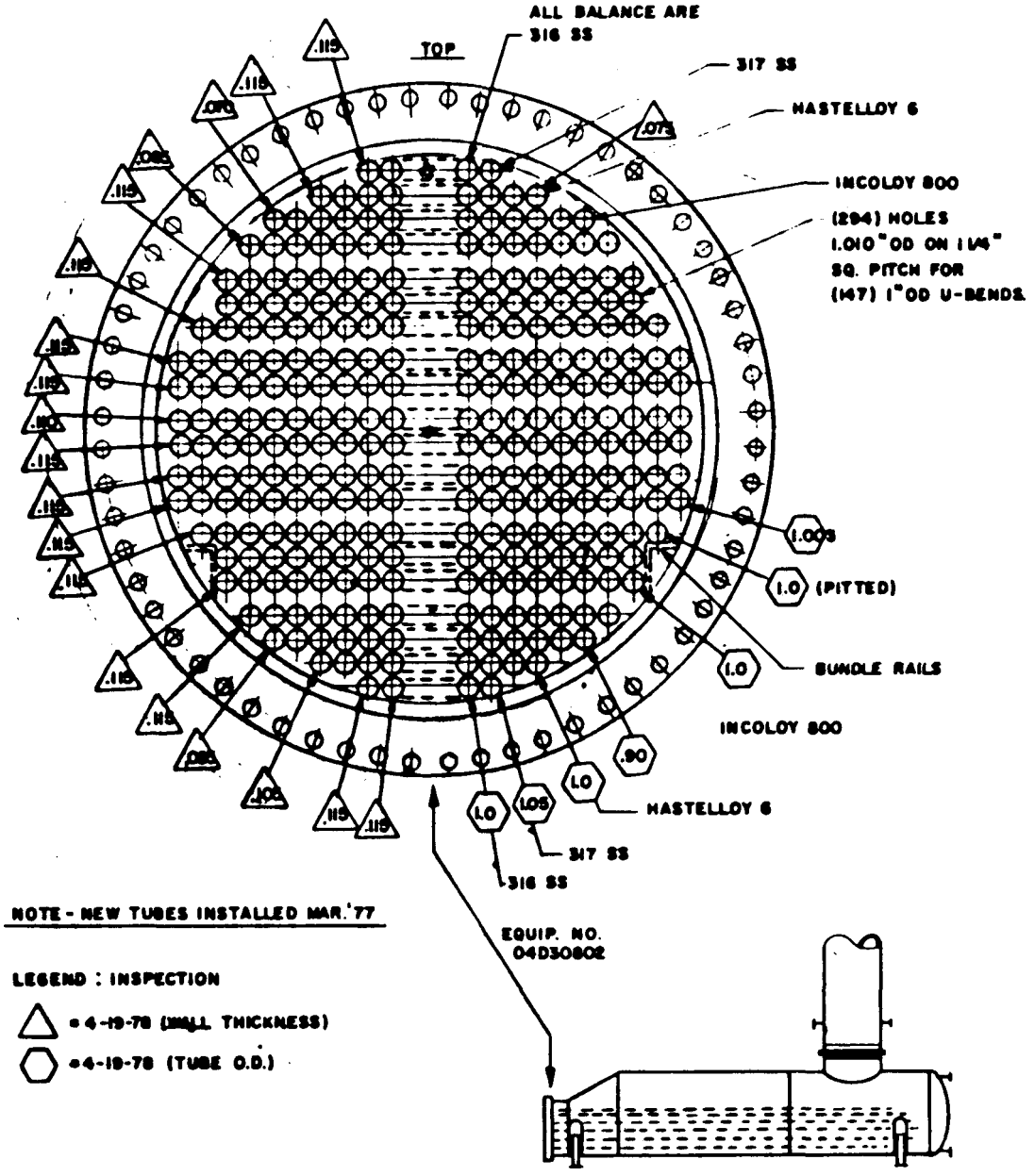


FIGURE 21

WASH SOLVENT COLUMN REBOILER TUBE BUNDLE



metals was installed for testing with the exception of 316 SS. Two trays of 316 SS were installed. One of the 316 SS trays had valves like the majority of the trays and the other was a sieve tray. Since the trays wear at a higher rate in the area where there is metal to metal contact (valve area), it was thought that the sieve tray could be a superior design. The corrosion rates from the corrosion racks are shown in Table A-1. The rates of corrosion for 316 SS (1.6 - 18.0 mpy) seem to be in good agreement with actual experience.

A diagram of the WSC overhead cooler as it was when it was removed is shown in Figure 22. The 316 SS tubes were in good condition; however, the carbon steel tube sheet was corroded extensively and could no longer be used. A new WSC overhead cooler, fabricated from 316L SS, was installed as a replacement.

The only significant corrosion of the light ends column shell was found at the very bottom of the column. As shown in Figure 23, the bottom of the column has lost from 0.005" to 0.010" in about seven months. The remaining portion of the column appeared to be in good condition.

The 304 SS tubes in the LEC reboiler bundle were severely corroded along with some pitting. Two of the 304 SS tubes were sealed off to prevent Dowtherm leakage to the process. The 321 SS tubes were corroded to some extent and the remaining experimental tubes (316 SS, Incoloy 825, and Hastelloy G) were in essentially new condition. The corrosion rack results, as shown in Table A-1, appear to substantiate the corrosion rates observed for carbon steel.

4. Operation June 6 through June 24, 1978

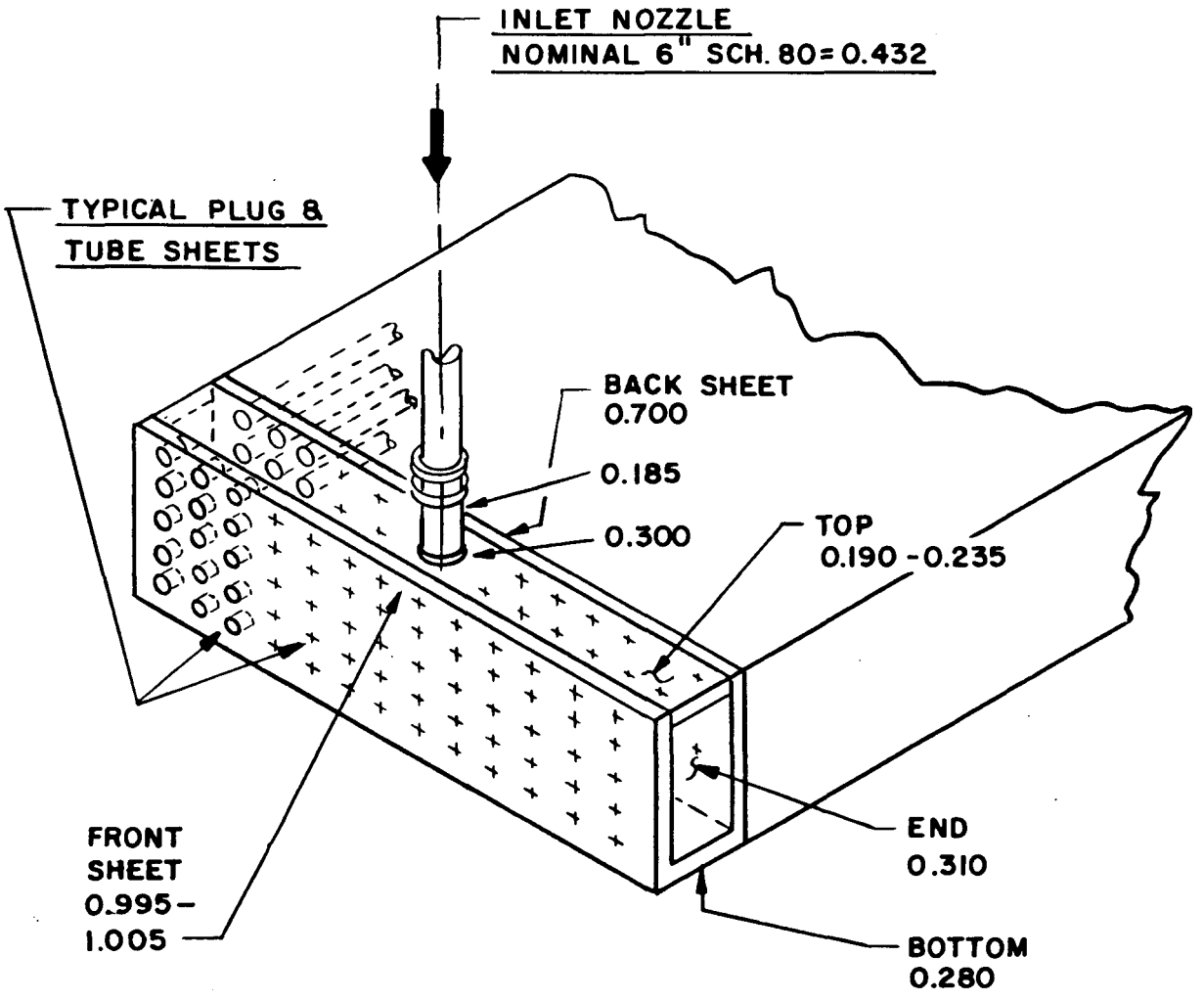
During the intital startup of the old vacuum flash system June 6, it was difficult to pull over four inches of vacuum on the system. After various procedures were tried to determine what the difficulty might be, it was found, by disassembling the eductor system, that the secondary jet nozzle had been left out.

By June 10, startup of the system was reattempted. When the Dowtherm jacketed piping on the bottoms system was put into service, cracks were discovered in the clamp-on jackets of the drum level control bypass valve (LCV-219) and the control valve on the discharge of one of the bottoms pumps. The jackets were replaced in kind and the entire jacketed system was pressure tested to 450 psig and returned to service.

Each half of a clamp-on jacket assembly consists of a small carbon steel plate exchanger surrounded by an aluminum casting which conforms to the shaft of the particular valve being

FIGURE 22

WASH SOLVENT COLUMN OVERHEAD FIN COOLER

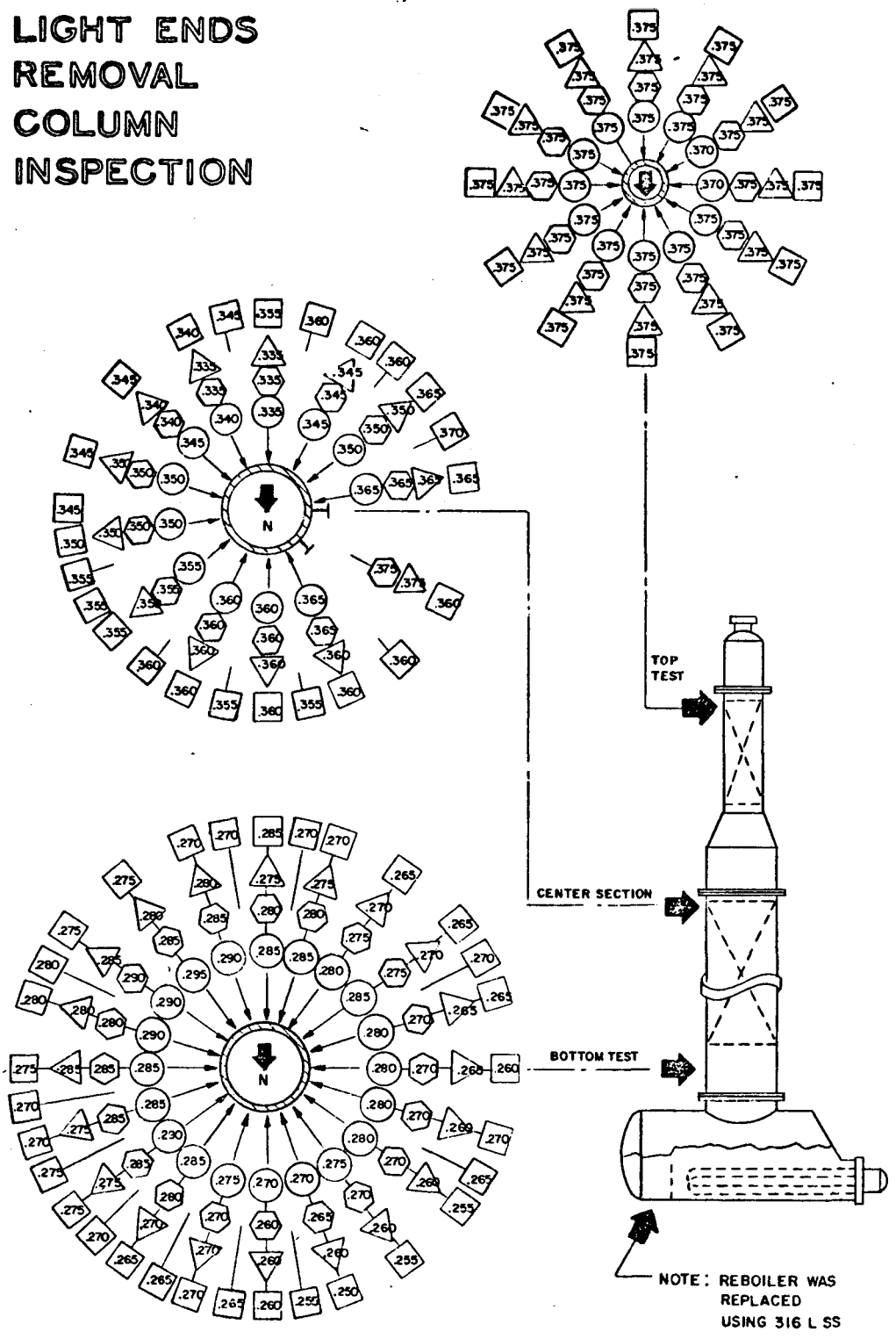


NOMINAL WALL THICKNESS

TOP = 0.375"
ENDS = 0.375"
BOTTOM = 0.375"
FRONT SHEET = 1.125"
TUBE SHEET = 1.125"

Figure 23

LIGHT ENDS REMOVAL COLUMN INSPECTION



NOTE: ALL READINGS ARE TAKEN BY ULTRASONIC.
ORIGINAL READINGS = 0.375"

LEGEND:

- ○ = THICKNESS AT INSPECTION 2-25-77
- ⬡ = " " " 4-14-77
- △ = " " " 10-15-77
- □ = " " " 5-3-78

jacketed. The plate exchanger bulged on overpressure causing the casting to crack. The most probable cause of failure was that during startup the Dowtherm was blocked in and hot process fluid was circulated through the inner process piping on the jacketed line causing thermal expansion of the Dowtherm heating medium.

To determine the reliability and safety of the clamp-on jackets one of the plate exchangers was taken out of its aluminum casing and hydrotested. The plate exchanger started to deflect at 450 psig and permanently yielded at 750 psig. When the pressure was raised to 1500 psig, the plate exchanger deformed badly but did not rupture. In order to assure a greater margin of safety, all of the jackets will eventually be sent back to the manufacturer to be rebuilt to a rating of 350 psig at 800°F.

The old vacuum system was returned to service on June 12, but operation was extremely unstable. Starting about mid-June and continuing through June 22, it became increasingly difficult to maintain suction to the bottoms pumps or flow to the cooling belt. The vacuum flash system was brought down and the piping inspected for pluggage. The jacketed bottoms line inner pipe had collapsed about a foot below the bottom of the drum and in a separate section of piping about 20 feet below the drum. By June 25, the entire inner pipe had been replaced and the old vacuum flash drum was returned to service.

5. Pump Performance

All of the pumps listed below required minimal maintenance during the reporting period. Each of these pumps required one new seal during the reporting period.

- Light Ends Column Bottoms Pump (04D56024)
- Light Ends Column Reflux Pump (04D56025)
- Wash Solvent Column Reflux and Product Pump (04D56027)
- Light Ends Product Pump (04D56032)
- Process Solvent to Storage Pump (04D56031)
- Wash Solvent Accumulator Pump (04D56033)
- Hotwell Water Pump (04D56039)
- Hotwell Oil Pump (04D56082)
- Light Ends Column Feed Pump (04D56043)
- Old Vacuum Flash Drum Bottoms Pumps (04D56083, 04D56084)

E. Gas Recovery and Recompression - (Area 05)

1. Operation March 25 through April 1, 1978

No significant operating problems were encountered during the early part of the reporting period. On April 1, the unit was removed from service, steamed, and prepared for inspection.

2. Turnaround Maintenance

During the shutdown the following work was completed in Area 05:

a. As part of an improved gas balance project, five gas meter runs were revised to improve meter accuracy and to allow maintenance of the orifice plate during plant operation.

b. The recycle hydrogen bypass loop exchanger carbon steel tube bundle was replaced with one made from 316 SS.

c. Recycle compressor A as well as the fresh hydrogen compressor were completely rebuilt.

3. Operation June 4 through June 24, 1978

After startup, valve spring failures continued to be a problem with the Chicago Pneumatic compressors. Maintenance repaired the springs on the third stage suction valves of the fresh hydrogen compressor five times during the reporting period. Two of the three valve failures delayed coal processing and caused approximately six hours of lost production. The continuing problem with valves on this compressor and on recycle compressor A is now believed to be the result of faulty valve design and/or the presence of condensate in the compressor valves, either of which may lead to spring breakage and valve failure. We continue to search for a means of preventing this high incidence of valve failure. Recycle compressor A was shut down once to repair loose packing and once to repair valves. No production was lost as a result of those problems.

The naphtha scrubber is not required for SRC I mode operation and, consequently, the naphtha circulation pumps were not in operation after startup.

F. Product Solidification and Storage - (Area 08)

1. Operation March 25 through April 1, 1978

Equipment in this area operated with only minor problems until plant shutdown. Repeated failure of the drive chain on the Sandvik belt was the most persistent problem.

2. Turnaround Maintenance

Items completed during the shutdown to improve performance included:

a. Modification of the fume hood from side to top vapor draw to reduce vapor duct restrictions.

b. Installation of water spray headers under the perimeter of the fume hood to aid in solidification and containment of liquid SRC at the feed point.

c. Installation of a steam spray header at the head of the belt to aid in cleaning.

d. Installation of a new knife and a new belt.

e. Installation of a Rexnord direct water quench vibrating cooler as an alternate solidification system. This unit was acquired to solidify the residue obtained from vacuum flashing the stripped underflow from the Lummus antisolvent deashing unit scheduled for startup in the latter part of this year.

3. Operation June 10 through June 24, 1978

The Sandvik belt was returned to service June 10. After initial belt training and adjustment, the unit performed satisfactorily.

G. Waste Treatment - (Area 09.1)

1. Operation March 25 through June 24, 1978

The plant effluent was within guidelines most of the reporting period with the exception of a few days in April. The biological oxidation unit functioned well until April 4. At that time the phenol content of the effluent began increasing as various tanks were emptied, cleaned, and flushed to the waste disposal system. At the same time, the reactivator was not operating because of problems with metering pumps for acid, alum and polyelectrolyte. As a result, a high pH solution with an increased amount of suspended solids entered the bio-unit overloading the bio-mass and allowing the phenol, COD, BOD and TSS values to increase. After new bio-mass was added to the system, the metering pumps repaired and the load reduced, the system again performed satisfactorily.

2. Turnaround Maintenance

A new metering pump was installed to inject phenols into the biological oxidation unit feed line to maintain bio-mass during periods of low phenol feed.

3. Inspection of Piping and Vessels

The charcoal and sand filters were emptied for vessel inspection. Corrosion in the sand filters was minimal and they were returned to service after refilling with filter media. Both charcoal filters, however, were severely corroded in a shotgun

pattern where the protective liner has been damaged. Electrolyte action between the steel tank and the charcoal and stainless steel charcoal support was responsible for the pit corrosion. After the Union Tank Company repaired these vessels, maintenance installed magnesium anodes in each vessel for electrolysis protection. Charcoal filter B was returned to service, but filter A could not be used because of a leaking flange on the inlet nozzle. At the end of the reporting period, preparations were being made to modify piping for operation of the filters, either in series or in parallel.

H. Tank Farm - (Area 09.2)

Final inventory of SRC II liquids consisted of 355 bbl of naphtha, 5059 bbl of middle distillate, and 905 bbl of heavy distillate.

Blends of middle distillate and heavy distillate were prepared and shipped to Babcock and Wilcox and to Consolidated-Edison for burn test studies. In both cases the blends were 5.75:1 middle distillate to heavy distillate. Blending of approximately 4,500 barrels of distillate for the Consolidated-Edison burn test was completed May 19.

Rail cars were loaded by first filling with water, then displacing the water with nitrogen and then bottom loading the fuel oil blend. After loading was completed, the cars were pressurized to 50 psig with nitrogen and sealed.

I. Inert Gas, Hydrogen Production and Desulfurization - (Area 09.5)

1. Operation March 25 through April 1, 1978

On March 29, the loss of instrument air and steam caused an upset of the hydrogen reformer which resulted in a coal outage of 3.25 hours. On the same day, the syn-gas compressor valves failed resulting in an additional two-hour coal outage.

2. Turnaround Maintenance

The inert gas unit was removed from service April 23 for replacement of the combustion barrel in the MEA stripper and for retubing of the flue gas cooler. The original Admiralty brass tubes in the inert gas flue cooler were retubed using 304 SS. The original tubes were not suitable in wet CO_2 service.

The hydrogen reformer was removed from service on April 1 after decoking and desulfurizing operations. The reformer was opened for inspection, but the shift convertors remained bottled up under positive nitrogen pressure. The outlet pipes on all reformer tubes were removed and the depth of each catalyst bed was measured. Only minor changes (less than one inch) were noted from the 1977 annual shutdown. Based on

the small change in catalyst depth, a differential pressure survey was not made.

A flange leak on a hydrogen reformer tube, which had been leaking for some time, was repaired in place. All of the remaining eight expansion joints on the hydrogen reformer tube piping were replaced with units identical to the two test Zallea Brothers expansion joints which had been in service during all of the SRC II runs. The eight-inch 304L SS piping from the reformer to the shift converter, which had cracked in several welds, was replaced with carbon steel piping.

The Worthington syn-gas compressor was completely overhauled during the shutdown.

The Stretford unit was removed from service April 1 and remained out of service until June 19, 1978. While the unit was down, a Pritchard representative inspected the unit and proposed solutions for several operating problems. His recommendations for improving the performance of the unit were as follows:

a. Maintain chemical balances and unit conditions within tolerances.

b. Modify the oxidizer and air spargers to improve solution regeneration.

c. Procure proper materials and follow standard precautions to minimize corrosion.

3. Operation May 5 through June 24, 1978

The inert gas unit was returned to service May 5. The No. 1 inert gas compressor was the only problem area during this period. On May 21, and again on May 30, this compressor was shut down for valve repair.

Hydrogen unit repairs were completed and the unit was returned to service May 15. On May 27 the unit was shut down to repair leaks that had developed on the outlet flanges of two reformer heater tubes. One of the leaks occurred in a flange repaired during shutdown. Engineering is investigating the possiblities for making acceptable field repair of these flanges.

The Stretford unit was started June 19 and in service nine hours when a leak at the separator head, a safety valve failure on the melter charge pump, and corrosion of the melter feed line interrupted operation.

The DEA unit was returned to service June 5. The unit was out of service for eight hours June 15 to repair a leak on the circulation pump discharge piping and to unplug the suction strainers. On June 19, the unit was out of service four hours to repack the absorber level control valve.

J. Dowtherm System - (Area 09.8)

1. Operation March 25 through April 1, 1978

Operation of the Dowtherm system continued normally until it was shut down with the rest of the pilot plant for the scheduled turnaround.

2. Turnaround Maintenance

The following maintenance work was completed on Area 09.8 during the turnaround:

a. Valves were replaced on the suction and discharge of the Dowtherm circulation pumps and around the minimum flow control valve.

b. The Dowtherm surge tank was steam cleaned and a vortex breaker was installed in the bottom of the tank. Also, new level taps and a D/P cell were installed for more reliable level indication.

c. The heater was opened and inspected for coke formation. No significant amount was found.

d. Two six-inch tie-ins were made to the Dowtherm supply and return headers for the Lummus unit.

3. Operation May 6 through June 24, 1978

Shutdown work on the Dowtherm system was completed and the unit was recommissioned May 6. On May 11, the system was again shutdown to repack several of the valves which had been installed during the shutdown. On June 8, it was discovered that the burner sparger, burner throat, and the adjacent refractory were seriously damaged. Replacement parts were ordered for repair at the earliest opportunity and the heater is being closely monitored for further deterioration or flame impingement.

III. PROCESS ANALYTICAL DATA

The plant was down during the months of April and May; therefore, most of the data contained in this report was obtained during the month of June. When the plant started up in June, Kentucky Nos. 9 and 14 coal from P&M's Colonial Mine and raw solvent were used.

The typical analyses of the raw coal during the month of June are shown in Table 2.

TABLE 2

Average Raw Coal Analyses, Wt %

Ash	9.02%
Moisture	6.99%

The typical analyses of the dried, pulverized coal are reported in Tables 3, 4 and 5.

TABLE 3

Typical Dried, Pulverized Coal Analyses, Wt %

Carbon	71.51
Hydrogen	5.30%
Nitrogen	1.51%
Sulfur	3.42%
Oxygen (by diff.)	8.25%
Ash	9.43%
Moisture	0.58%

TABLE 4

Typical Analyses for Forms of Sulfur, Wt %

Pyritic Sulfur	1.50%
Sulfate Sulfur	0.44%
Organic Sulfur	1.40%
Total Sulfur	3.34%

TABLE 5

Typical Sieve Analyses of Dried, Pulverized Coal, Wt %

- 40 mesh	99.39%
-100 mesh	97.24%
-140 mesh	91.18%
-200 mesh	90.10%

The typical analyses of the stripper bottoms in the month of June are shown in Table 6.

TABLE 6

Average Analyses of Stripper Bottoms, Wt %

Water	2.06%
Light Oil	0.97%
Wash Solvent	1.38%
Process Solvent	60.64%
Vacuum Bottoms (P.I. Included)	34.94%
Pyridine Insolubles (as rec'd)	5.79%
Ash in Pyridine Insoluble	54.49%

The typical moisture-ash-free coal conversion in June was 92.8%.

The typical analyses of the laboratory vacuum bottoms obtained from the work-up of the recycle stripper bottoms are shown in Table 7.

TABLE 7

Typical Analyses of Laboratory SRC I Vacuum Bottoms, Wt. %

Carbon	88.53%
Hydrogen	5.74%
Nitrogen	1.98%
Sulfur	0.78%
Oxygen (by diff.)	2.82%
Ash	0.15%
Fusion Point	302°F

The typical analyses of No. 3 separator oil phase for the month of June are reported in Table 8.

TABLE 8

Typical Analyses of No. 3 Separator Oil Phase, Wt %

Specific Gravity @ 60/60°F	1.004
Light Oil	19%
Wash Solvent	19%
Process Solvent	62%

The typical analyses of oil and water phase from the recycle process water tank (RPWT) for the month of June are reported in Tables 9 and 10.

TABLE 9

Typical Analyses of RPWT Oil Phase, Wt %

Specific Gravity @ 60/60°F	0.982
Light Oil	18%
Wash Solvent	31%
Process Solvent	51%

TABLE 10

Typical Analyses of RPWT Water Phase, Wt %

Phenols	0.31%
Nitrogen	0.48%
Sulfur	0.53%

Typical fractional analyses of liquid products, based on ASTM-D86 distillation data, are reported in Table 11.

TABLE 11

Typical Analyses of Pilot Plant Liquid Products, Vol. %

	Light Oil	Wash Solvent	Process Solvent
IBP - 380°F	100	4	0
380 - 480°F	0	95	3
480 - 850°F	0	1	97

During this current SRC I production, the filtrate was recombined with the cake-leg stream before entering the vacuum flash drum. Therefore, the ash content of the vacuum bottoms is higher than normal for SRC I operation. Average analyses for the pilot plant vacuum bottoms solid product produced in June are reported in Table 12.

TABLE 12

Average Analyses of Pilot Plant Vacuum Bottoms, Wt %

Ash	10.15%
Fusion Point	300°F

The daily analyses of samples from the waste treatment units have shown that it was within the guidelines set for this area throughout the quarter. Average analyses of waste water streams for the months of April, May and June are shown in Tables 13, 14 and 15.

TABLE 13

Waste Water Analyses

April	Bio-Unit Feed	Bio-Unit Effluent	Plant Effluent (Composite)
pH	7.1	7.1	6.7
Phenols, ppm	64	2.9	0.48
Chemical Oxygen Demand, ppm	1086	434	52
Biological Oxygen Demand, ppm	201	64	5.9
Total Suspended Solids, ppm	193	195	12

TABLE 14

Waste Water Analyses

May	Bio-Unit Feed	Bio-Unit Effluent	Plant Effluent (Composite)
pH	6.6	7.7	7.0
Phenols, ppm	19	0.9	0.11
Chemical Oxygen Demand, ppm	510	267	34
Biological Oxygen Demand, ppm	77	16	12
Total Suspended Solids, ppm	47	76	7.3

TABLE 15

Waste Water Analyses

June	Bio-Unit Feed	Bio-Unit Effluent	Plant Effluent (Composite)
pH	7.0	6.4	6.5
Phenols, ppm	86	0.83	0.22
Chemical Oxygen Demand, ppm	1713	211	42
Biological Oxygen Demand, ppm	149	23	11
Total Suspended Solids, ppm	171	37	4

At the request of the Department of Energy, middle distillate and heavy distillate were blended in a ratio of 5.75/1 in anticipation of future burn tests. A blend of 408 barrels was prepared in April and shipped to Babcock & Wilcox in Alliance, Ohio. An additional blend

of 4500 barrels was subsequently prepared for shipment to Consolidated-Edison in New York. As this blend was being transferred from storage tank 045 to railroad cars, samples were taken for analysis. Even though blending operation for these two shipments were completed at different times, the final analyses of the separate blends were essentially equivalent. For this reason, only the composite analyses for the shipments sent to Consolidated-Edison will be presented in this report. These results are given in Table 16.

TABLE 16

COMPOSITE ANALYSES OF SRC II LIQUID PRODUCT

Sample Kind: 5.75/1 v/v Blend of Middle Distillate to Heavy Distillate

Spec. Gravity 60/60°F	0.992	% Pyridine Insolubles	0.06
°API @ 60°F	11.2	% Conradson Carbon	0.45
Viscosity @ 100°F cSt	4.07	% Ash	0.06
@ 210°F cSt	1.20	% Water	Trace
Pour Point °F	-54	% Carbon	85.54
Flash Point °F	173	% Hydrogen	8.77
Btu/lb	17,100	% Nitrogen	0.97
Coal Tar Acid % v/w	27.0	% Sulfur	0.20
		% Oxygen	4.46

Distillation	ASTM D-1160 °F	ASTM D-86 °F
I.B.P.	-	292
5%	135	389
10%	144	403
20%	158	416
30%	169	428
40%	180	440
50%	193	454
60%	208	471
70%	225	491
80%	250	522
90%	320	596
95%	390	698
End Point	445	722
Recovery	97%	97%
Residue	2%	2%
Loss	1%	1%
Distilled under	2.0 mm Hg	758 mm Hg

IV. PILOT PLANT SPECIAL STUDIES

A. Filter C Hydraulic Capacity Tests

Three tests were conducted to evaluate the baseline hydraulic performance of filter C with feed material nominally free of solids. These tests, using process solvent feed and minimal knife advance rate, served as shakedown runs for the filter. During the tests, precoating techniques were also developed. The results of these tests provide data concerning the effect of precoat thickness on hydraulic flow rate. A nominal operating temperature of 465°F was chosen for these tests because the viscosity of process solvent at that temperature is estimated to be the same as that of coal solution at a temperature of 600°F, the target temperature for process filtration experiments. Table A-2 lists pertinent operating conditions for these tests and Figures A-12 through 14 present operating data obtained during hydraulic tests 1, 2 and 3, respectively. Target drum submergence during these runs was 30% of drum area. However, erratic flowrates resulted from an inability to maintain a steady tub level at the higher differential pressures. This difficulty seriously limited the value of the data obtained because the quantitative relationship between tub level and hydraulic flow rate is not known.

1. Hydraulic Test No. 1

The data in Figure A-12 indicate that drum speed has no significant effect on hydraulic flow rate at 40 psi differential pressure. The effect of knife advance appears to be masked by the effect of knife position. This tentative conclusion may be drawn by observing in Figure A-12 that rate of filtrate flow is nearly always increasing at a given knife advance rate. Minimal effect of knife advance is, of course, expected when filtering a virtually clean fluid. The basic effect demonstrated in the first hydraulic test is that for a "clean" filter feed, the precoat thickness significantly affects the overall rate of filtrate flow.

2. Hydraulic Test No. 2

The maximum hydraulic flow rate achieved during this run (400 lbs/hr/sq ft) occurred at 30% drum area submergence and a knife advance rate of 1.6 mil/min with 2.9 inches of precoat remaining. The trend of increasing flow rates with decreasing precoat thickness that was observed in the first hydraulic test also was apparent in the second test. Comparison of the results of these tests to determine the effect of differential pressure on precoat hydraulic capacity was not attempted because of level control problems which could be expected to cause decreased flowrates at the low drum submergence levels.

3. Hydraulic Test No. 3

In the early stages of this run, the differential pressure was increased from 40 to 95 psig and the rate of filtrate flow dropped dramatically. Flow rates were not re-established until the knife advanced one inch, suggesting that the change in differential pressure caused a 20% compression of the five-inch precoat.

When the precoat thickness had decreased to approximately two inches in test No. 3, coal solution was introduced into the filter at 550°F and a 30% drum area submergence. As the data in Figure A-12 indicate, the filtration rate at 6.9 mil/min knife advance rate and 2 rpm drum speed decreased to approximately 150 lb/hr/sq ft. Filtrate solids increased to 0.2% during this period.

4. Conclusions

The hydraulic capacity tests lead to the following conclusions:

a. The precoat thickness is very significant to hydraulic flow rate. Figure 24 shows rate data selected from the hydraulic tests plotted versus the inverse of measured precoat thickness. The non-linearity shown in Figure 24 is indicative of some compressibility of the precoat bed. In actual coal solution filtration, however, this resistance characteristic is expected to be of minimal importance when compared to the resistances of filter cake and cake-precoat interface (penetration).

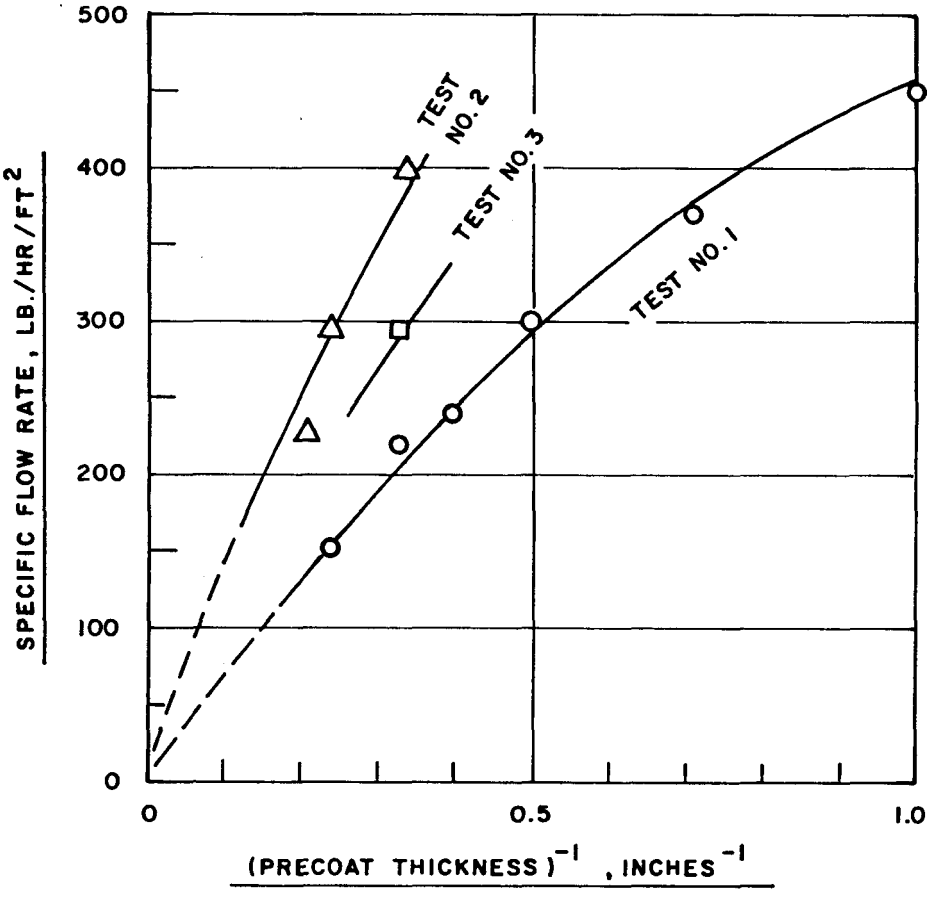
b. The maximum hydraulic flow rate achieved with "clean" filter feed in the range of conditions studied (1-5 rpm drum speed, 1.5 mil/min knife advance rate, 40-95 psig differential pressure, and 30% drum area submergence) was 450 lb/hr/sq ft at a 1.125 inch precoat thickness and 40 psi differential pressure.

B. High Temperature Coal Slurry Blending Tests

Prior to the plant shutdown on April 1, a brief test was made to determine the operability of the existing coal slurry blending system at higher temperature (375-400°F) with Blacksville No. 2 mine coal. Starting at a base temperature of about 350°F, three approaches to high temperature operation were attempted. Each attempt was terminated as temperature reached the 375-390°F range due to loss of slurry flow from the centrifugal circulation pumps.

FIGURE 24

EFFECT OF PRECOAT THICKNESS ON HYDRAULIC TEST FLOW RATE



Estimated slurry composition at the time of the tests was:

Component	Wt %
Dried Pulverized Coal (Blacksville No. 2)	30.6
Solvent (from recycle slurry and seal flush)	27.4
SRC (from recycle slurry)	25.5
Insoluble Organic Matter (from recycle slurry)	7.4
Ash (from recycle slurry)	9.1

The loss of slurry circulation apparently was caused by pump cavitation due to either vaporization of volatile slurry components or extremely high slurry viscosity. Past feed slurry viscosity data tend to corroborate the latter theory, since analysis of this data indicates a minimum slurry viscosity at approximately 340°F with powdered Blacksville No. 2 coal. In addition, after the first two approaches to high temperature operation had proven unsuccessful, heavy distillate was substituted for seal flush to determine if vaporization of volatiles in the circulation pump had caused cavitation and a subsequent loss of circulation. The final approach to high temperature operation did not prove to be any more successful than previous attempts, with the loss of slurry circulation occurring at 385°F slurry blend tank temperature.

The slurry viscosity measured on the plant on-line capillary viso-meter prior to loss of circulation during the first approach to high temperature operation is compared in Figure 25 with the viscosity measured at a similar slurry composition, but lower blending temperature, during a material balance run. The viscosity data indicate that, at 370°F, the slurry viscosity was nearly an order of magnitude greater than at 345°F.

C. Material Balance Run Accuracy Analysis

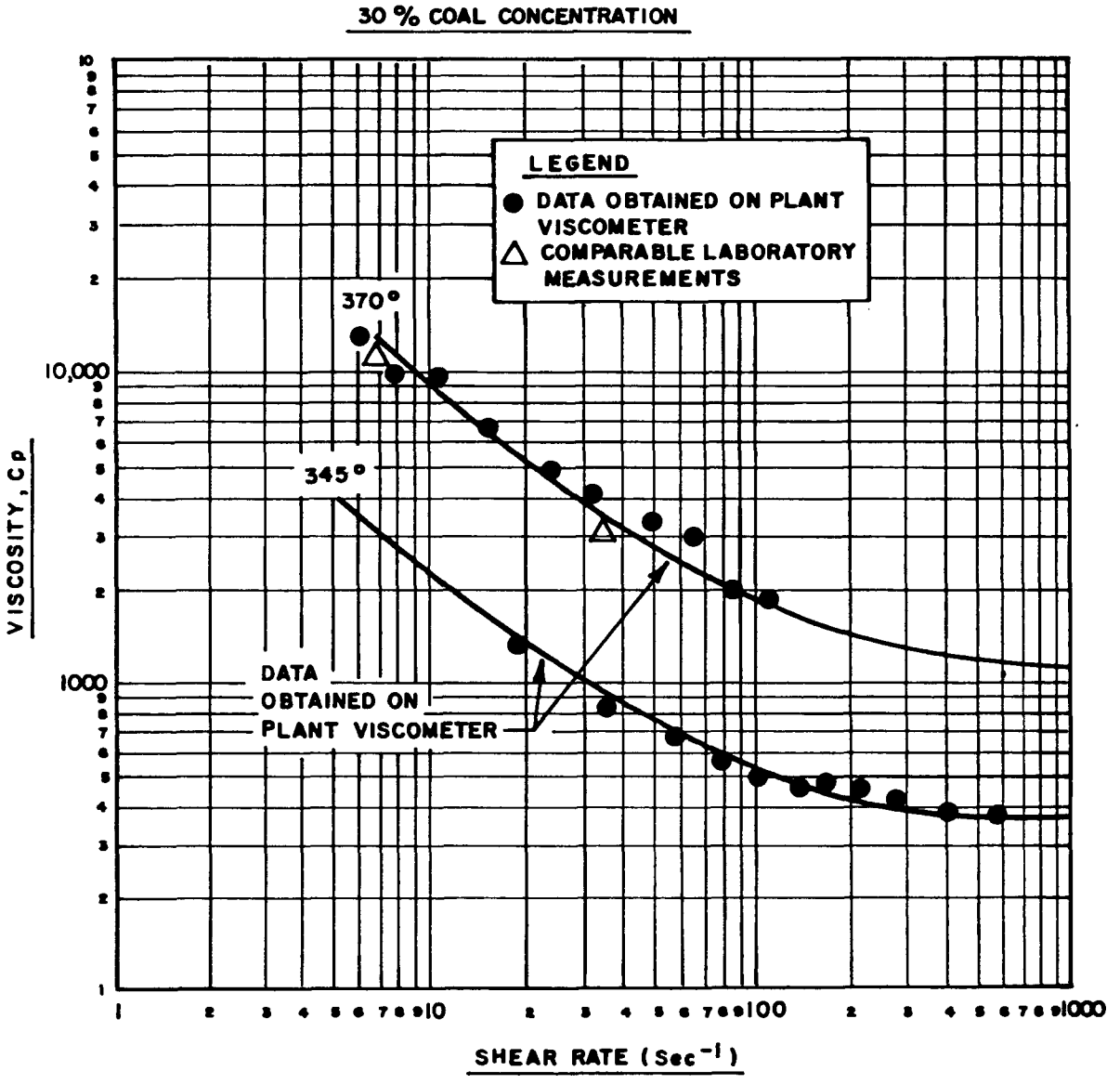
In order to obtain a statistical measure of the significance of process yield variations observed in different material balance runs and to assign confidence limits to the observed data, it is necessary to have a measure of material balance run accuracy. Potential inaccuracies in the measured data may be categorized in two classes: systematic error or bias, and nonsystematic or random error. Systematic error cannot be estimated with a stochastic model, but it is possible to estimate nonsystematic error with such a model.

To estimate the random error in liquid yields, it is necessary to understand the method by which the actual yields are calculated. The liquid balance procedure used in Ft. Lewis can be described by the following relationship:

Mass Accumulation of Product in Vessel j = Mass of Product in Vessel j at End of Run − Mass of Product in Vessel j at Start of Run, Or,

FIGURE 25

EFFECT OF TEMPERATURE & SHEAR RATE ON VISCOSITY OF SRC II FEED SLURRY (BLACKSVILLE NO. 2 COAL)



$$L_j = \begin{pmatrix}\text{fraction of}\\ \text{product in}\\ \text{vessel at EOR}\end{pmatrix}\begin{pmatrix}\text{liquid}\\ \text{level at}\\ \text{EOR}\end{pmatrix}\left(\frac{\text{lb}}{\text{inch}}\right) - \begin{pmatrix}\text{fraction of}\\ \text{product in}\\ \text{vessel at SOR}\end{pmatrix}\begin{pmatrix}\text{liquid}\\ \text{level at}\\ \text{SOR}\end{pmatrix}\left(\frac{\text{lb}}{\text{inch}}\right)$$

$$= S_f L_f W - S_i L_i W \qquad (1)$$

Where: L_j = The mass of a given component accumulated in vessel j during a run period.

S_f = The weight fraction of a given component in vessel j at end of run.

S_i = The weight fraction of a given component in vessel j at start of run.

L_f = The level in vessel j at the end of the run.

L_i = The level in vessel j at the start of the run.

W = Vessel j strapping, mass per unit of level.

EOR = End of Mass Balance Run.

SOR = Start of Mass Balance Run.

The total mass accumulation, L, of a given product would therefore be the sum of the L_j for all vessels in the material balance loop:

$$L = \Sigma L_j$$

Each L_j has associated with it an S_f, L_f, S_j, L_i and W (5 variables). Therefore, the total number of variables in a total yield calculation is then:

$$n = \text{No. of variables} = 5j$$

Renaming each variable X_i, the variance in the total yield can be estimated[1] as:

$$\sigma^2_L = \sum_{i=1}^{n} \left(\frac{\partial L}{\partial X_i}\right)^2 \sigma^2_{X_i}$$

Where: σ^2_L = variance in the total component yield

x_i represents X_{Sf}, X_{Si}, X_{Lf}, X_{Li}, X_W for each vessel j.

$\sigma^2_{X_i}$ = variance of each x measurement.

Simplifying this to one vessel, let $X_1 = S_f$

$X_2 = L_f$

$X_3 = S_i$

$X_4 = L_i$

$X_5 = W$

then: $$\sigma^2_{L_j} = \sum_{1}^{5} \left(\frac{\partial L}{\partial X_i}\right)^2 \sigma^2_{X_i}$$

or, substituting variables of equation (1)

$$\sigma^2_{L_j} = (S_f L_f - S_i L_i)^2 \sigma^2_W + (L_f W)^2 \sigma^2 S_f + (S_f W)^2 \sigma^2 L_f + (L_i W)^2 \sigma^2 S_i + (S_i W)^2 \sigma^2 L_i \quad (2)$$

(Note that all signs of terms are positive since variances are accumulated.)

Since the sampling and level variances do not change from start to end of run, the following equations may be defined:

$$\sigma^2 S_f = \sigma^2 S_i = \sigma^2 S \quad (3)$$

$$\sigma^2 L_f = \sigma^2 L_i = \sigma^2 L \quad (4)$$

The general equation for vessel j variance can be simplified by substituting (3) and (4) into (2):

$$\sigma^2_{L_j} = (S_f L_f - S_i L_i)^2 \sigma^2_W + \{(L_f W)^2 + (L_i W)^2\} \sigma^2 S + \{(S_f W)^2 + (S_i W)^2\} \sigma^2 L$$

which can be simplified to

$$\sigma^2_{L_j} = (S_f L_f - S_i L_i)^2 \sigma^2_W + (L_f^2 + L_i^2) W^2 \sigma^2 S + (S_f^2 + S_i^2) W^2 \sigma^2 L \quad (5)$$

This equation represents the variance in product yield in vessel j.

The total plant variance is simply

$$\sigma^2_L = \sum_j \sigma^2_{L_j}$$

By estimating the variance of each X_i measurement (variance in level, variance in sampling and analysis, and variance in strapping) for each vessel, it is possible to estimate the total variance for each liquid component. At at given level of significance it is then possible to estimate the random error in the component yield. This procedure was used to estimate the inaccuracy in the total distillate, naphtha, middle distillate and heavy distillate yields for SRC II Material Balance Runs. It was also possible to determine the contribution of each vessel in the material balance loop to the total component variance and the contributions to variance due to vessel strapping error, level measurement error, and sampling and analysis error.

Selected results of this error analysis for Material Balance Run 78SR-20 are shown in Table A-3 where the contributions to the total variance for each vessel are listed by component. The inaccuracy in determining the amount of a given component in the wash solvent accumulator represented the major source of random error. For every vessel, sampling and analysis represented the main source of error. The results in Table A-3 also show the

relative accuracy of each component expressed as the ratio of the error term to the total recovery. The error terms shown in Table A-3 were calculated to give a 95% confidence level in the liquid yield. The percent error for each component is an indication of a 95% confidence limit for the component. For example, the total distillate yield for run 78SR-20, which was found to be 33.7% of moisture free coal feed, is accurate at a 95% confidence level to 10.8%, or has a range of 33.7$\pm$ 3.6% of moisture free coal feed according to this analysis. The results for other components can be interpreted in the same manner.

Several conclusions appear warranted based on this study of material balance accuracy. First, the fact that the wash solvent accumulator represents the most significant error contribution justifies the design of the SRC II material balance envelope, including provision of both recirculation capabilities and mixing in the wash solvent accumulator to help ensure that a representative sample is obtainable. In addition, since the error analysis points out the desirability of accumulating as much of the liquid as possible in a single vessel, additional emphasis can be placed on draining most of the liquid produced during a material balance run into the wash solvent accumulator. A further refinement in liquid yield accuracy would result by beginning each material balance run with an empty wash solvent accumulator to minimize errors associated with beginning level measurement and sampling and analysis of the vessel contents.

The error analysis discussed above deals strictly with random measurement error. Other errors, such as variability in coal composition and any bias in the material balance run calculation procedure, are sources of systematic error and cannot be estimated by a model similar to the one presented above. Those types of errors are unique to each run and must be individually measured.

This analysis has shown that the main source of random error in the Fort Lewis material balance procedure for liquids is the sampling and analysis error in the wash solvent accumulator. In addition, the results of the study indicated that the total liquid yields for the material balance runs typically are accurate at a 95% confidence level to within $\pm$ 10% of the observed yields.

D. Comparison of SRC II Liquid Fuel with Petroleum-Derived Fuel

Selected properties of a blend of SRC II middle and heavy distillates are compared with specifications and typical values for petroleum-derived No. 6 fuel oil in Table A-4. These data show that both filtered and unfiltered SRC II fuel meet or exceed the ASTM standards for sediment, water and ash. The sulfur level is below that of the typical low sulfur No. 6 fuel oil. The pour point and viscosity are lower than those of the No. 6 fuel oil, which should be a benefit since heated storage and fuel preheat may not be required for the SRC II fuel.

Table A-4 also indicates that the gravity (°API) of the SRC II fuel oil is significantly lower than that of the petroleum-derived oil. This is not significant in itself, but does reflect the high aromaticity of the SRC II oil. Despite the low gravity of the SRC II oil, the heating value per barrel of the fuel is slightly lower than that typical for petroleum oil. This is due to the high aromaticity and oxygen content of the SRC II oil, which are also reflected in the high carbon to hydrogen ratio.

A comparison of typical ash constituents for SRC II oil and a petroleum-derived fuel is presented in Table A-5. The ash from SRC II oil, like that from coal, is high in iron, silicon, aluminum, and titanium. More importantly, the ash is low in vanadium, nickel, and sodium which often contribute heavily to fire side tube fouling. In addition, the presence of substantial quantities of silicon, aluminum and titanium might be expected to further inhibit the fire side deposition of alkaline ash deposits. Finally, it is expected that SRC II fuel ash would exhibit a higher fusion temperature than that from petroleum fuel which would further limit tubeside scale formation. In summary, the ash inspection data indicate little potential for the formation of fire side deposits which would impede heat transfer and cause firebox corrosion problems.

E. SRC II Reactor Effluent Particle Size Analysis

A sample of SRC II recycle slurry was sent to Micron Data Laboratories for a particle size distribution analysis by x-ray sedimentation. This technique was chosen because particle size analyses of previous samples of product slurries by scanning electron microscopy and Coulter Counter had not produced acceptable particle distributions.

Measurements based on the scanning electron microscopy procedure are influenced by particle size and particle shape. Because the particles for analysis are mounted on a flat surface, the determination of particle diameter may be biased by particle shape, resulting in an inaccurate estimate of particle size. Also, because the volume of a particle varies as the cube of its diameter, a relatively small number of large particles results in an unacceptably skewed distribution when particle number distributions are converted to mass or volume distributions.

The Coulter Counter analysis of particle size is limited in sensitivity to particles with diameter greater than approximately one micron. The presence of a significant number of particles with diameters less than one micron in coal solution slurries limit the usefulness of this technique in determining particle size distribution.

The x-ray sedimentation method has the potential to analyze a reasonably large sample of particles with good sensitivity to both small and large particle diameters and to yield a mass

distribution directly. Figure 26 represents the cumulative mass distribution of particles in the recycle slurry sample analyzed. The results presented in Figure 26 are biased by the incomplete dissolution of the original sample by the proprietary solvent used to dissolve the oils and resins of the sample (the solids which produced the results of Figure 26 were found to contain 49.8% pyridine insolubles). However, the continuous distribution produced by this analysis suggests that this technique for determining particle distribution may be applicable to coal solids once a suitable technique is developed for dissolving the oils and resins of the coal solution slurry. In the future, this dissolution step will be performed in the plant laboratory using pyridine as a solvent and the resulting solids will be sent out for x-ray sedimentation analysis.

F. Heat Capacity of SRC II Materials

Samples of SRC II vacuum bottoms, stripper bottoms, and pyridine insolubles were sent to Bartlesville Energy Research Center for measurements of heat capacity versus temperature by differential scanning calorimeter. All of the samples tested were obtained while processing Illinois No. 6 coal. Figure 27 shows the calorimetric data plotted versus temperature. A fair amount of scatter is indicated, especially for stripper bottoms. As previously observed, the vacuum bottoms heat capacity curve shows an inflection in the vicinity of the fusion point. The stripper bottoms sample also seems to show this characteristic. This suggests that it may be possible to accurately measure the point at which the change of state usually referred to as "fusion" takes place through differential thermal analysis. The characterization data for the samples tested are given in Table A-6.

G. Viscosity Studies

A series of viscosity studies was undertaken to define the effects of certain variables on the viscosity of solutions commonly processed in the SRC II operating mode. These viscosity studies are especially important in the coal blending system in the SRC II operating mode, where control of the coal-slurry viscosity is a crucial factor in maintaining system operability.

The relationship between viscosity and the concentration of vacuum bottoms or SRC was studied in the laboratory for unfiltered coal solution (recycle slurry in the SRC II mode) and solvent-low ash SRC mixtures. The viscosity of feed coal slurry was studied in the plant using an on-line viscometer and freshly prepared coal slurry that was circulating in the coal blending system. Each of these studies is discussed separately below.

1. Process Solvent - SRC Mixtures

The relationship between absolute viscosity and the concentration of SRC in process solvent was investigated in the

PARTICLE SIZE DISTRIBUTION BY X-RAY SEDIMENTATION

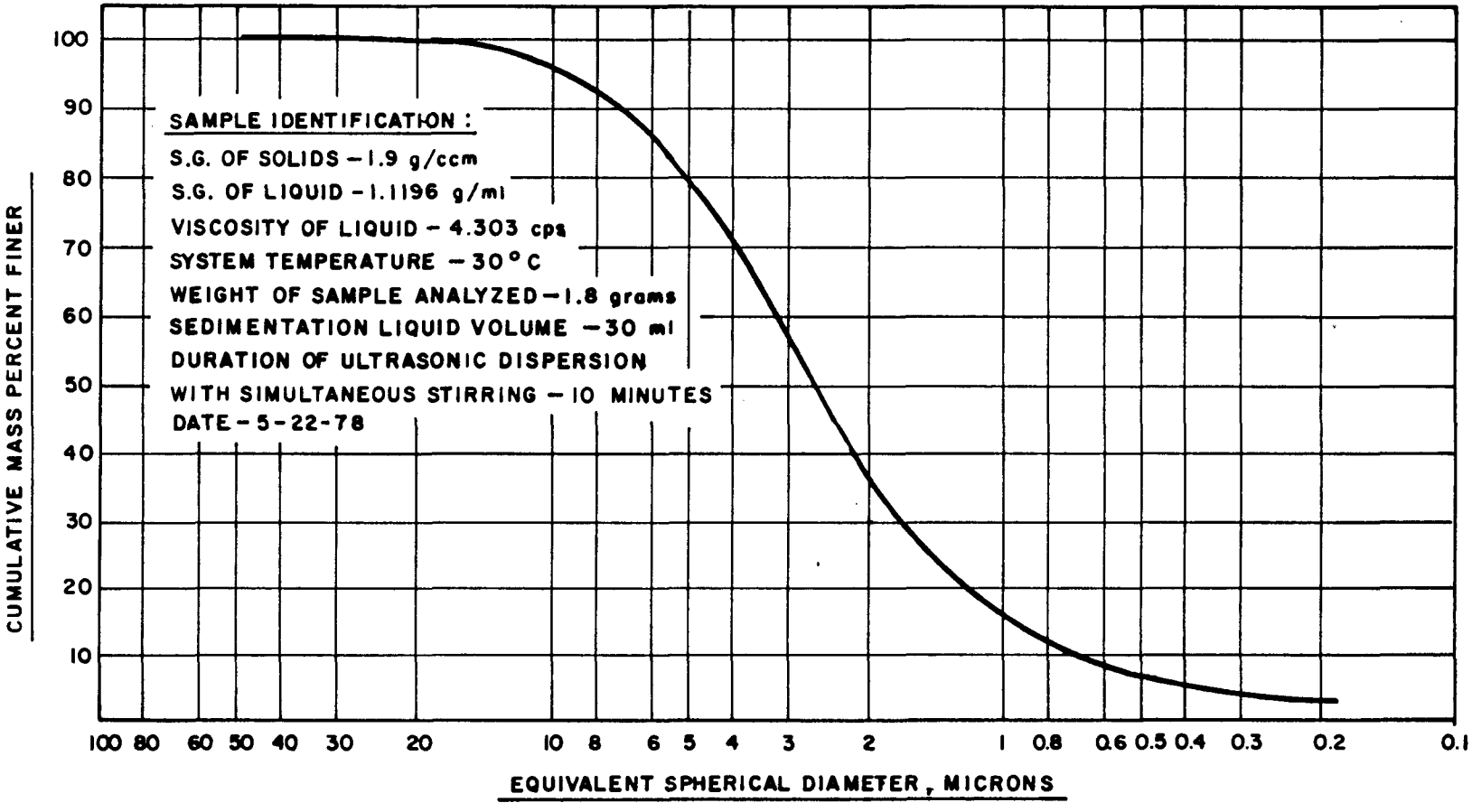


FIGURE 26

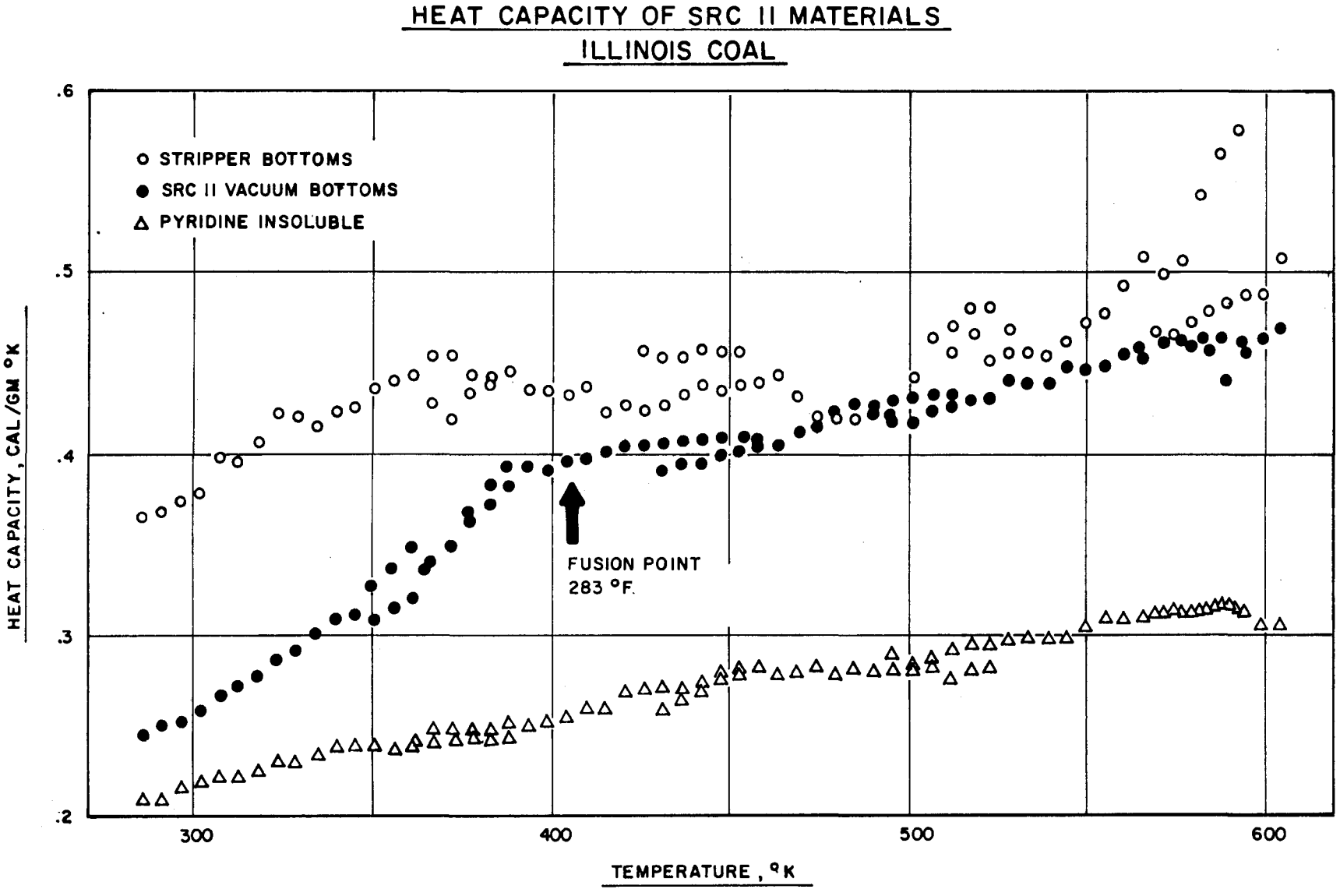

HEAT CAPACITY OF SRC II MATERIALS
ILLINOIS COAL
STRIPPER BOTTOMS
SRC II VACUUM BOTTOMS
PYRIDINE INSOLUBLE
FUSION POINT
283 °F.
HEAT CAPACITY, CAL/GM °K
.6
.5
.4
.3
.2
300
400
500
600
TEMPERATURE, °K

FIGURE 27

laboratory on a Brookfield model HA viscometer equipped with a No. 21 spindle. The viscometer was operated at a speed of 200 rpm resulting in a 186 sec^{-1} shear rate. The samples which were analyzed consisted of low ash SRC dissolved in process solvent. The results from this study are presented in Figure 28.

The "Huggins equation"[2] for predicting the viscosity of dilute polymer solutions was found to provide a suitable fit of the data shown in Figure 28. This equation has the following form:

$$\frac{1}{c} \ln \frac{\eta}{\eta^o} = \hat{\eta} + k\hat{\eta}^2 c,$$

where η is the absolute viscosity of the solution
η^o is the absolute viscosity of the pure solvent
c is the solute concentration
k is a constant
$\hat{\eta}$ is the "intrinsic viscosity"

Rearrangement of this equation results in the following quadratic equation:

$$\ln \eta = \ln \eta^o + \hat{\eta}c + k\hat{\eta}^2 c^2.$$

By applying polynomial regression to a data set comprised of viscosity measurements and SRC concentrations, a "best fit" equation of the form

$$y = a + b\chi + c\chi^2$$

was obtained where y is the $\log_e$ of absolute viscosity at a constant temperature and χ is the SRC concentration. Interpreting this by the Huggins equation then, "a" is the $\log_e$ of the viscosity of the pure solvent and "b" is the intrinsic viscosity, $\hat{\eta}$. k can be determined as

$$\frac{c}{b^2} = \frac{k\hat{\eta}^2}{\hat{\eta}^2} = k.$$

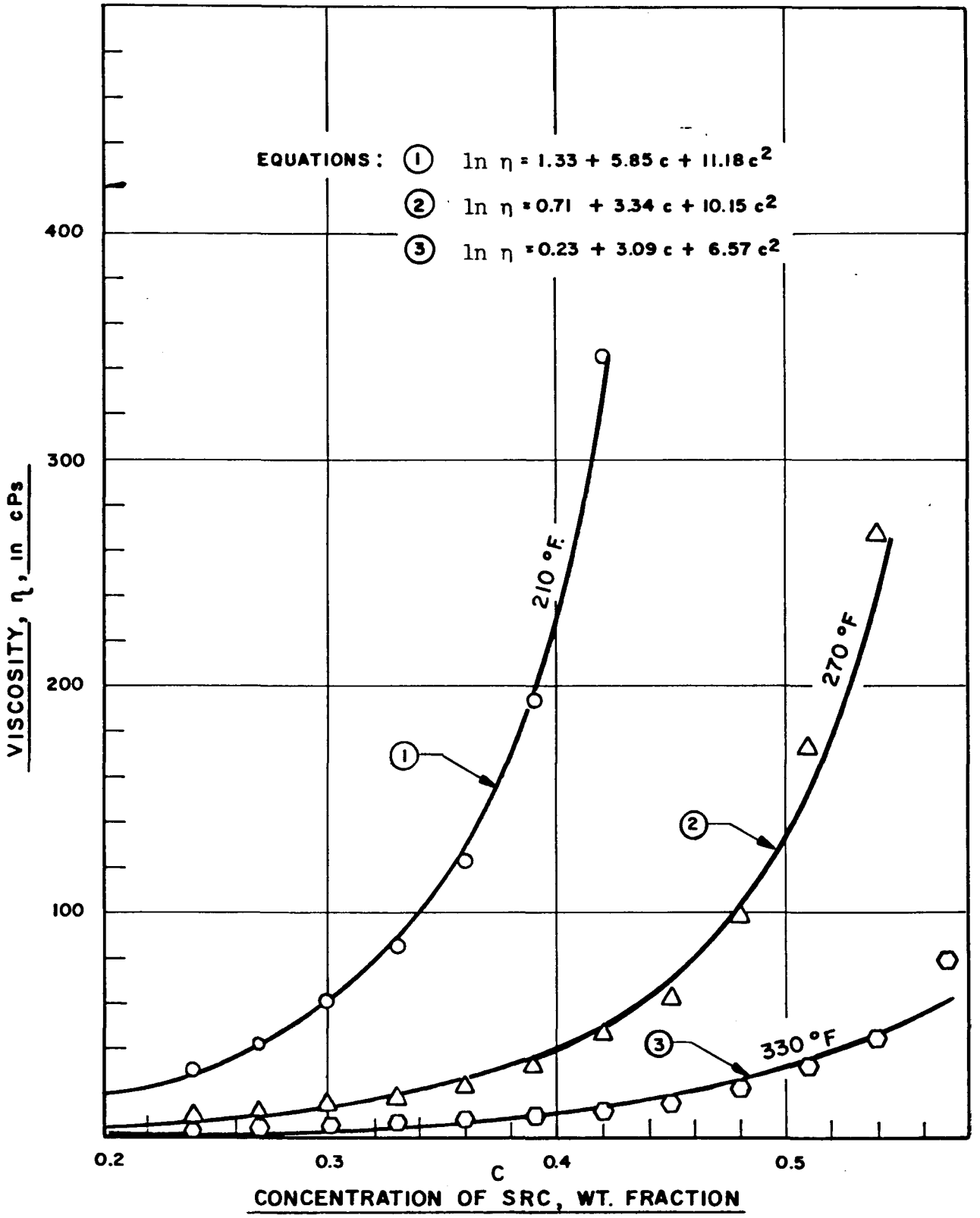
Use of the Huggins model in this case gives a very good fit to the data shown in Figure 28. Also, the values of "a" obtained from polynomial regression yield reasonable values for pure solvent viscosity. The observations lead to the conclusion that the solvent-low ash SRC system viscosity behaves in a manner quite similar to that of dilute polymer systems.

2. Unfiltered Coal Solution (Recycle Slurry)

Using the same apparatus as in the solvent-low ash SRC studies, the relationship between the apparent viscosity (the viscosity

FIGURE 28

VISCOSITY OF SRC – PROCESS SOLVENT BLENDS
VISCOSITIES AT 186 SEC^{-1} SHEAR RATE



which would satisfy Poiseuille's equation at the specified shear rate) and vacuum bottoms concentration (SRC plus pyridine insolubles) in an unfiltered coal solution was investigated. Samples of unfiltered coal solution from the pilot plant were obtained during SRC II operation with both Western Kentucky and Illinois coals. The results from this study are presented in Figure 29. This data was fit using the Huggins equation as discussed above. While the Huggins equation fits the trends in the data, there is a significant amount of dispersion among the data points. This effect is believed to be due to the non-Newtonian nature of the unfiltered coal solution and the assumptions inherent in the Huggins model of a dilute homogeneous mixture.

One of the most interesting aspects of this study is the very strong effect of vacuum bottoms concentration on apparent viscosity in the range of 55-60% vacuum bottoms in the unfiltered coal solution. This same effect can also be observed for the process solvent-low ash SRC solution data in Figure 28. Recycle slurry compositions in this range are typical during SRC II operation. Additional studies of the effects of vacuum bottoms concentration might prove useful in identifying conditions that minimize viscosity in the coal blending system.

3. Coal Slurry

The relationship between shear rate and coal slurry apparent viscosity was studied using an on-line capillary tube viscometer in the coal blending area. Seven sets of data were collected during operation with Blacksville No. 2 coal. During each test period, the shear rate was varied by changing the flowrate through the capillary tube. Operating parameters for each test period are shown in Table A-7, and the relationship between measured apparent viscosity and shear rate for each test period is shown in Figure 30.

The results are presented on a log-log plot of viscosity versus shear rate and indicate that straight lines with large negative slopes fit most of the data. In two runs the slope decreases substantially around a shear rate of 120 sec^{-1} and may approach zero. This type relationship between viscosity and shear rate is typical of non-Newtonian fluids classified as pseudoplastics.

The power law model is commonly used to describe the relationship between apparent viscosity and shear rate for pseudoplastics. The following equation desribes this relationship:

$$\mu_A = k\,(\text{shear rate})^{n-1}$$

where k is the "consistency index"
n is the "flow behavior index"
μ_A is the apparent viscosity

FIGURE 29

VISCOSITY OF UNFILTERED COAL SOLUTION
AT 210 °F AND 186 SEC^{-1} SHEAR RATE

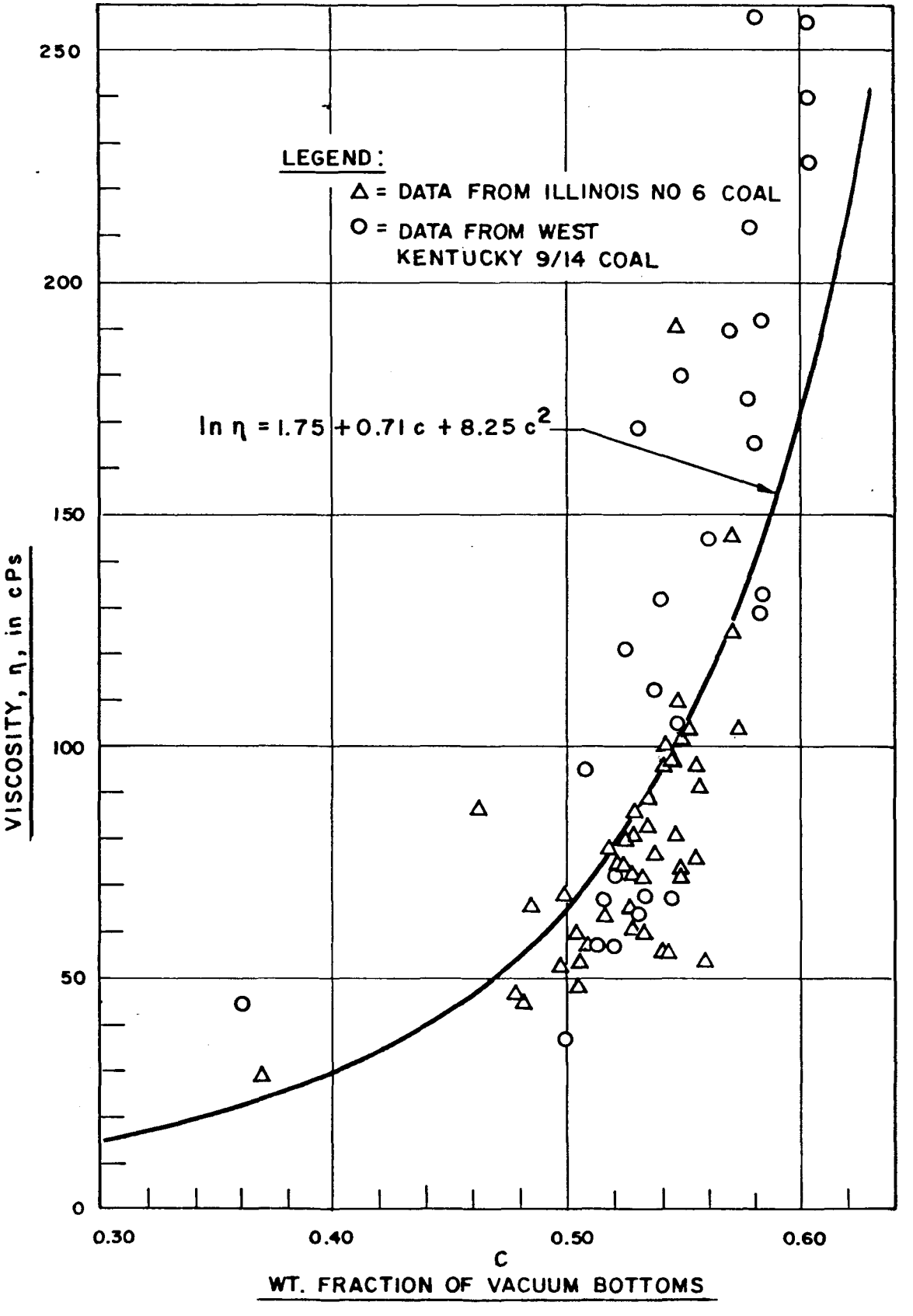
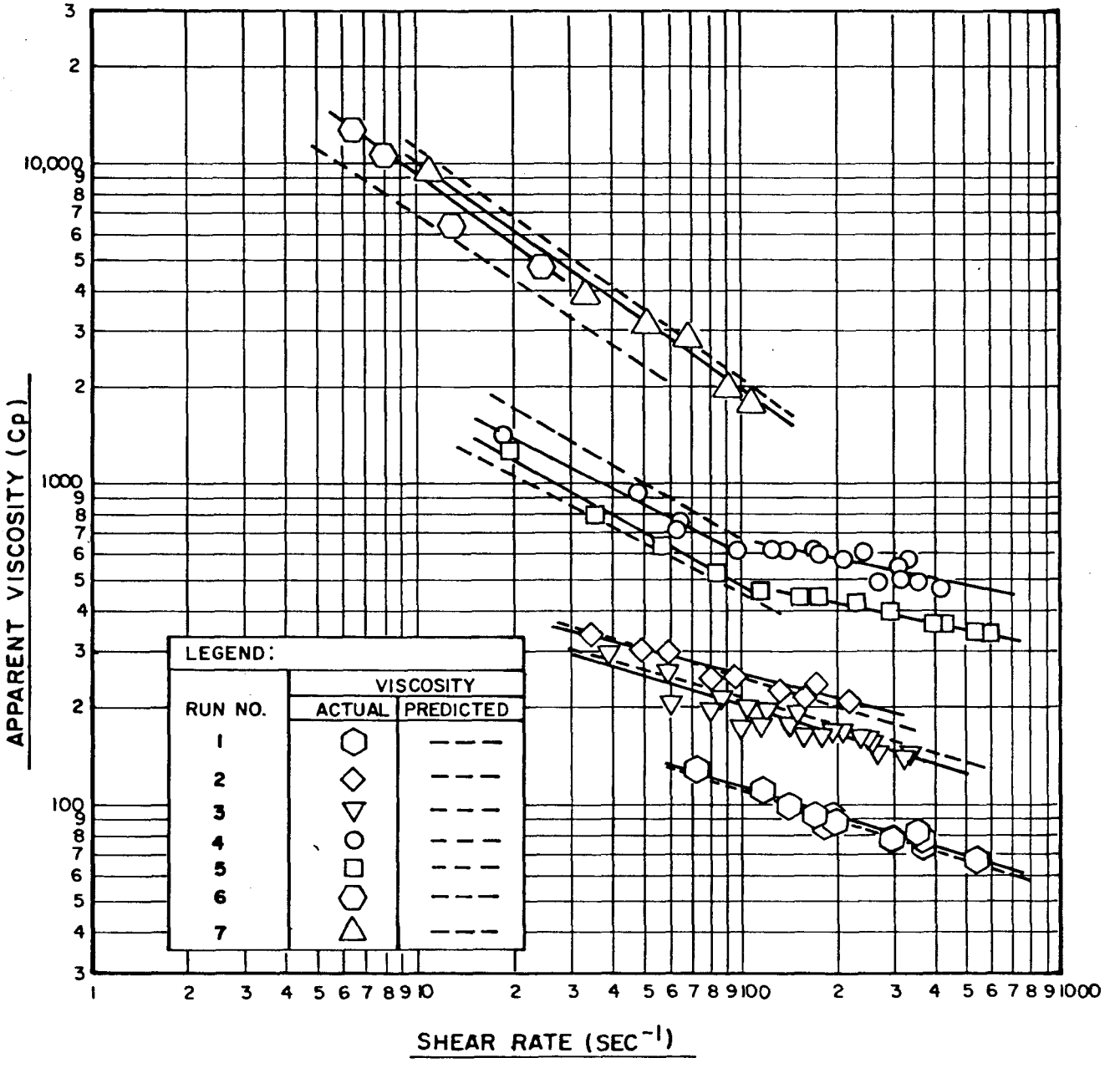


FIGURE 30

VISCOSITY vs SHEAR RATE OF BLACKSVILLE NO. 2 SRC II COAL SLURRY



Generally, the values of n and k are estimated from the slope and intercept, respectively, of a logarithmic plot of shear stress versus shear rate.

From the data shown in Table A-7, empirical models were developed which relate n and k to temperature, the concentration of coal in the feed slurry, and the concentration of SRC and pyridine insolubles in the recycle slurry. These relationships are shown below:

$$n = 1.43393 \times 10^{8} C_c^{-1.0479} C_{SR}^{-7.7201} C_{PI}^{-0.3974} \exp^{-0.022938(T+460)}$$

$$k = 3.6376 \times 10^{-17} C_c^{3.4014} C_{SR}^{-7.201} C_{PI}^{4.7827} \exp^{0.05667(T+460)_n - 2.7076}$$

Where C_c is the feed slurry coal concentration (wt. fraction)
C_{SR} is the recycle slurry SRC concentration (wt. fraction)
C_{PI} is the recycle slurry PI concentration (wt. fraction)
T is the feed slurry temperature (^{o}F)

A comparison of the apparent viscosity predicted by this model with the measured apparent viscosity is shown in Table A-8.

Additional laboratory studies are now in progress to define the effects of temperature, residence time and particle size on coal swelling which is believed to have a strong effect on the overall apparent viscosity in the coal blending system.

H. Heating Value of Solvent Refined Coal Products

As a part of the continuing characterization of SRC products, a collection of samples was sent to a local testing laboratory for heat of combustion by ASTM D-240 and D-2015 methods. Description of samples and results are listed below.

Sample Type	Heat of Combustion (Btu/lb) Values Analyzed
Total Solvent* from Wash Solvent Accumulator, MBR 78SR20	17,200
Naphtha (lab distilled) 78SR20	18,926
Middle Distillate (lab distilled) 78SR20	17,140

Heavy Distillate (lab distilled) 78SR20	17,190
5.75 to 1 blend of Middle Distillate to Heavy Distillate (Shipment Sample #1198)	17,325
SRC II Vacuum Bottoms (Ky. Nos. 9 & 14 feed coal) - Ash content: 28.15%	11,300
SRC I Solid Product, composite of shipments for Southern Services burning tests	15,854

*Total solvent from MBR 78SR20 contained 1.4% water, 26.0% naphtha, 52.8% middle distillate and 19.8% heavy distillate.

I. Dissolver Volume Measurement

The volume of the bottom section of dissolver A, as measured on April 7, was 45.7 ft^3, a loss of 2 ft^3 since December 1977. Total loss of dissolver volume due to the buildup of solids during the entire period of SRC II operation was 4.3 ft^3, or 4.7% of the full dissolver volume.

J. Data Acquisition System

1. System Reliability

During the April plant shutdown, the Fox 2/30 computer was taken off-line several days so that Foxboro service representatives could thoroughly clean and check both the central processor and the analog input modules (process interface). Although no apparent reason was found for the numerous previous system failures, only one failure occurred after cleaning. A problem with the No. 3 Decwriter terminal was also corrected at this time.

On June 5 and June 19, the system shut down with no diagnostic messages printed. The system was successfully restarted on both occasions. On June 20, the system failed to output data properly to the Decwriter terminal causing an output buffer overflow. The situation was corrected by restarting the system. On June 21 and 23, the operating system made erroneous entries in the drum storage cross reference table, making some programs inoperable. On both occasions the drum was reloaded from magnetic tape, resulting in the loss of some data. These problems have been reported to the Foxboro Company for study. A list of system reliability is reported in Table A-9.

2. Programming Activities

The major programming activity during the period concerned modification of the data acquisition system in preparation for

monitoring the performance of filter C. Programs completed in this regard included:

a. A program to tabulate daily temperatures, flows and pressures associated with the operation of filter C.

b. Program expansion to permit flowrate calculation for an increased number of flowmeters associated with the filter, including expansion of the associated data files which contain pertinent information on each flowmeter. The program used to average data over a user-specified period was also expanded to handle the increased number of flowmeters.

c. A program which calculates the rate of filter knife advance using a count of the revolutions of the knife advance shaft over a specific period of time.

d. A program to store the knife position at the beginning of each hour in the process data files and eventually in permanent storage on magnetic tape.

e. A program to calculate the average value of seventeen filter-operating parameters over a specified period of one to sixty minutes. Process parameters available include temperatures, pressures, levels, knife position and knife advance rate, and calculated mass flowrates.

f. Program modification to add the calculated filter knife advance rate to the "current loop value" operator assistance routine.

g. A program to report volumetric flow rates of the filter precoat loop was created to provide operator assistance during filter precoating.

In addition to the modifications detailed above, several revisions of existing programs were necessary to handle operation in the SRC I mode. The program to calculate solvent inventory was expanded to include six additional vessels in the filtration area. Calculation of slurry blend tank composition also required revision since recycle slurry is not used for coal slurry blending in the SRC I mode.

The corrosion-erosion monitoring program was modified to handle measurements of trim life and operating limits for the new I.P. flash letdown valve (LCV-175B). Another test was also added to this program to record the time-temperature history of the slurry experience of the valve.

A program was written and installed in the computer system to produce a report of 142 current operating parameters for use by plant operators, and a series of programs was written to assist the purchasing department in preparing their quarterly report.

V. PILOT PLANT SPECIAL PROJECTS

A. Lummus Antisolvent Deashing Studies

Work on the Lummus antisolvent deashing system continued during the second quarter. At the end of the quarter, the overall project was about 72% complete. The mechanical and electrical contractors had completed 88% and 25% of their work, respectively. After the unit is mechanically complete, precommissioning activities will begin. Flushing of the system and mechanical run-in should be completed by late September.

B. New Slurry Preheater Design

Bids were received in April for a smaller slurry preheater for dissolver feed. The present preheater has proven to be oversized and a new smaller coil is necessary to obtain suitable scale-up data for a commercial plant heater.

Major studies to be conducted with the new preheater include the effects of slurry velocity on erosion. New and improved instrumentation will also help to resolve questions related to the formation and location of gels (an intermediate step in the coal dissolving process) as well as heat transfer effects.

In June, a proposal for its purchase was submitted to DOE for approval.

C. Dissolver Solids Sampling

A system was designed and installed to sample the contents of dissolver A while on-stream. Results of the work-ups on these samples will be contained in subsequent reports.

D. Vacuum Flash Drum Preheater

An improved preheater design for use in the small vacuum flash drum was completed and at the end of the period a proposal for its construction was in the final stages of drafting. The new coil was designed with increasing incremental volume along its length in order to maintain more constant vapor and slurry velocity. The new coil consists of pipe of different diameters, increasing from two inches at the inlet of the coil to four inches at the outlet. Because of previous problems with the Thermon heat tracing used on the original six-inch coil, the new coil is to be Dowtherm jacketed.

VI. MERRIAM LABORATORY OPERATIONS

A. Introduction

During the second quarter of 1978, activities at the Merriam Laboratory included the following:

- A study of the various factors which contribute to an increased oil production in SRC II vs SRC I operation.
- Startup of the new bench scale continuous unit.
- Exploratory short residence time runs.

A series of runs was made to explore the importance of various factors which increase the yield of volatile products in the SRC II process over that obtained in the SRC I process. It was again demonstrated that under similar conditions of pressure, temperature, and residence time the oil yield in SRC II operation is substantially higher than that obtained in SRC I operation. Additional runs were made to separate the effects of recycle of SRC (pyridine soluble vacuum residue) and recycle of mineral residue (pyridine insolubles). The results of this series support the concept that both the recycle of coal solids (mineral and insoluble organic matter) and the recycle of soluble but non-distillable organic matter (SRC) significantly affect process yields. It should be particularly noted that recycle of either component alone failed to produce the yield pattern observed when the combination was recycled (SRC II).

The continuous bench scale coal processing unit was started up in May. Much of the month was spent in shakedown trials and operator training. Initial operation of the unit involved preliminary runs investigating SRC operation with short residence times. Discussion of results of the short residence time runs will be deferred until a later report.

B. Recycle Studies: A Study of Factors Contributing to Increased Oil Production in SRC II vs SRC I Operation

1. Introduction

In the original P&M Solvent Refined Coal Process, now designated SRC I, coal is dissolved under moderate hydrogen pressure in a heavy aromatic solvent derived from the process. This results in solution of most of the organic matter in coal and produces a filterable liquid. Mineral matter is removed by filtration and the filtrate is subjected to a vacuum flash operation for recovery of solvent for recycle. The vacuum flash residue is a low-ash, low-sulfur solid material known as Solvent Refined Coal.

In an improved version of the process, designated SRC II, a portion of the coal solution is recycled as the coal slurrying

media in place of the distillate solvent used in the original SRC I process. This improved process increases the conversion of dissolved coal to lower molecular weight materials. The primary product of this process is a liquid fuel in place of the solid product of the SRC I process. In the modified process, a fraction of the coal solution is used in feed slurry formulation. The remainder is available for product recovery. The fuel value could be recovered by filtration in which case the primary fuel product would be the filtrate, a heavy viscous liquid product.

Laboratory and pilot plant work have demonstrated, however, that under appropriate reaction conditions the conversion of high molecular weight dissolved coal to lower molecular weight products is adequate to allow recovery of the fuel product by distillation, thereby eliminating the difficult and expensive filtration step. The primary product is a low-sulfur distillate fuel oil.

The residue from the vacuum distillation consists of three components: ash, insoluble organic matter, and material which has dissolved but is nonvolatile; i.e., similar to the solid SRC of the SRC I process. By appropriate selection of reaction conditions the distillation residue can be reduced to that required for hydrogen generation for the process.

The increased conversion to light products in the SRC II process is caused by a combination of three factors:

a. Pressure and residence time used in the SRC II process are increased over those typically used in SRC I operation.

b. Recycle of the coal solution allows additional reaction time for conversion of high molecular weight dissolved coal (SRC) to lower molecular weight products.

c. The concentration of mineral matter, which is known to function as a catalyst for SRC reactions, is increased.

The objective of the work reported in this section was to develop a better understanding of the SRC II process by separating these factors and demonstrating the relative importance of each.

Coal used was a blend of Kentucky Nos. 9 and 14 from P&M's Colonial Mine. This is the coal which has been most extensively investigated in the SRC process, both at the Merriam Laboratory and the Fort Lewis Pilot Plant. Analyses of the coal are given in Table B-1.

Conditions and results for the runs made in this study are summarized in Table B-2. In all runs, pressure was 2000 psig, dissolver temperature was 455°C (851°F), coal concentration in the feed slurry was 30%, and coal feed rate was 21.3 lb/hr/ft^3. This feed rate corresponds to a nominal slurry residence time (with no allowance made for volume occupied by gas) of 1.0 hr.

2. SRC I - SRC II Comparison

The first factor which contributes to the increased conversion to light products in the SRC II process is that SRC II operation is conducted with longer residence times and at higher pressures than are normally used in SRC I. In normal operation, conditions for SRC I processing are selected to obtain a breakeven yield of recycle solvent but with no attempt to significantly increase the solvent yield beyond that required for recycle. The effect of this factor (increased residence time and pressure) may be separated from the remaining two factors by conducting SRC I and SRC II runs under identical process conditions. Runs GU 213 and GU 216R were made under identical conditions except for the composition of the slurry feed. GU 213 was a normal SRC I run with a slurry feed of 30% coal and 70% distillate recycle solvent and GU 216R was a normal SRC II run made with a slurry feed of 30% coal and 70% recycle coal solution.

Yields for the two processes are compared in Table 17. Increasing pressure and residence time to those normally used in SRC II operation contributes to substantially increased conversion of SRC to light products in SRC I operation. Hydrocarbon gas yield is increased by several percent, recycle solvent yield is increased from approximately breakeven to a 4% excess, and total oil yield is approximately doubled. There is an accompanying decrease in SRC yield from near 60% to 43%.

A comparison of the yields in Table 17 shows that under similar conditions of temperature, pressure and residence time, there is a substantially lower yield of SRC in SRC II operation in comparison to that obtained in SRC I operation. This decrease in SRC yield from 42.1 to 21.0% is due to a combination of the second and third factors listed above; i.e., recycle of SRC which allows additional reaction time for the nonvolatile SRC to be converted into lighter products and/or to the increased concentration of mineral matter which functions as a catalyst for SRC reactions.

The relative importance of these two factors has aroused considerable speculation. Although the effect of process variables on SRC II product distributions has been thoroughly investigated, none of this work has allowed an unequivocal separation of these two factors. In normal SRC II operation (with a constant feed coal composition), ash concentration and SRC concentration in the slurry feed are not independent

TABLE 17

YIELD COMPARISON: SRC I VS SRC II[a]

	Yields, wt % MF Coal Basis	
	SRC I (GU 213)	SRC II (GU 216R)
C_1-C_4	10.5	16.1
Heavy Distillate (Recycle Solvent)	4.0	21.8
Total Oil	25.9	38.9
SRC	42.7	21.0
Insoluble Organic Matter	4.1	5.1
H_2	-2.4	-5.6

a) Conditions were identical except for the composition of the slurry feed.

variables. Earlier studies have led to opposing opinions; two extreme opinions have been expressed. At one extreme there is the opinion that mineral matter has no effectiveness as a catalyst and that the increase in conversion in SRC II is only a consequence of the second factor: increased reaction time for the conversion of SRC to lighter products. At the other extreme there is the opinion that only the mineral matter recycle is of consequence. In this view, once SRC is formed it is stable and undergoes no conversion to lighter products.

In this work these two effects have been separated by two additional runs. In one run the high molecular weight dissolved coal (SRC) was recycled but not mineral matter. And, in the other run, mineral matter was recycled but not SRC.

3. Recycle of SRC Without Mineral Residue Recycle

Recycle of SRC without recycle of mineral matter was accomplished as shown in Figure 31. Effluent from the reactor was filtered. The filtrate contained the recycle solvent range material and dissolved SRC but not coal minerals or undissolved coal. Slurry was formulated as 70% filtrate and 30% coal and fed back into the reactor.

Recycle of SRC without mineral matter recycle was shown to be impractical from an operational standpoint in the bench scale unit. The oil and SRC yields could not be quantitatively determined. However, the available results do provide an indication that SRC recycle does promote conversion of SRC to lighter products. Yields in Table 18 show a substantial increase in hydrocarbon gas yield and an increase in hydrogen consumption.

Quantitative oil and SRC yields were not obtained because, as steady state operation was approached, the bench scale system became unmanageable. The unit was operated for over 100 hours but, as steady state composition was approached, the time required for filtration became excessive and the entire inventory of process material was eventually tied up in the filtration process and no material for slurry formulation was available. Other measures indicated that these operating conditions were unsatisfactory even if the filtration could be improved so that adequate material was available for feed slurry blending.

An indication of this unsatisfactory operation is provided in the run control data which are shown in Figure 32. The IR ratio is a measurement derived from the infrared spectrum of a sample of unfiltered coal solution. The ratio is a rough indication of the ratio of non-aromatic to aromatic hydrogen.

Figure 31

FLOW DIAGRAM FOR SRC RECYCLE WITHOUT MINERAL RECYCLE
(GU 215)

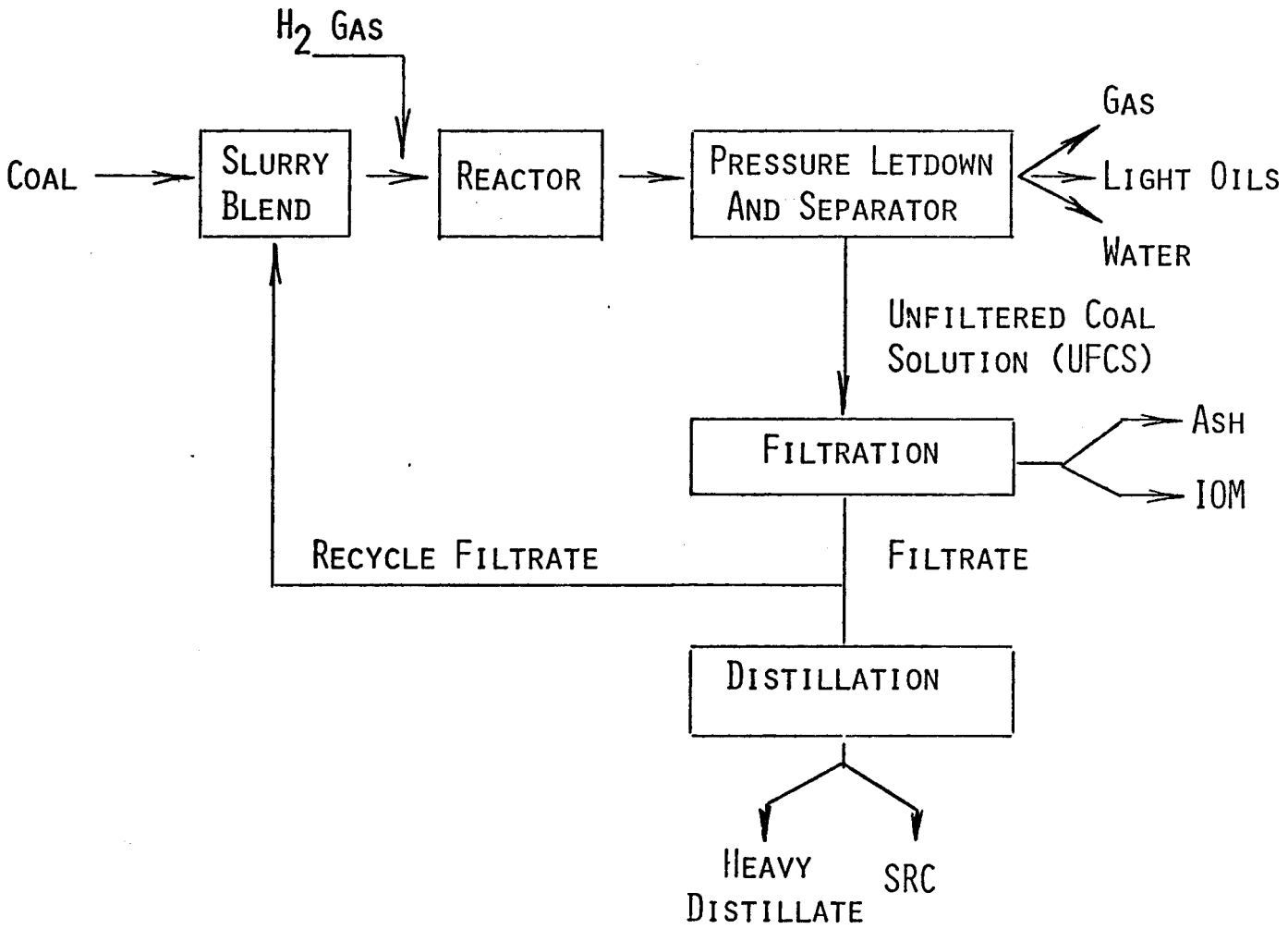


TABLE 18

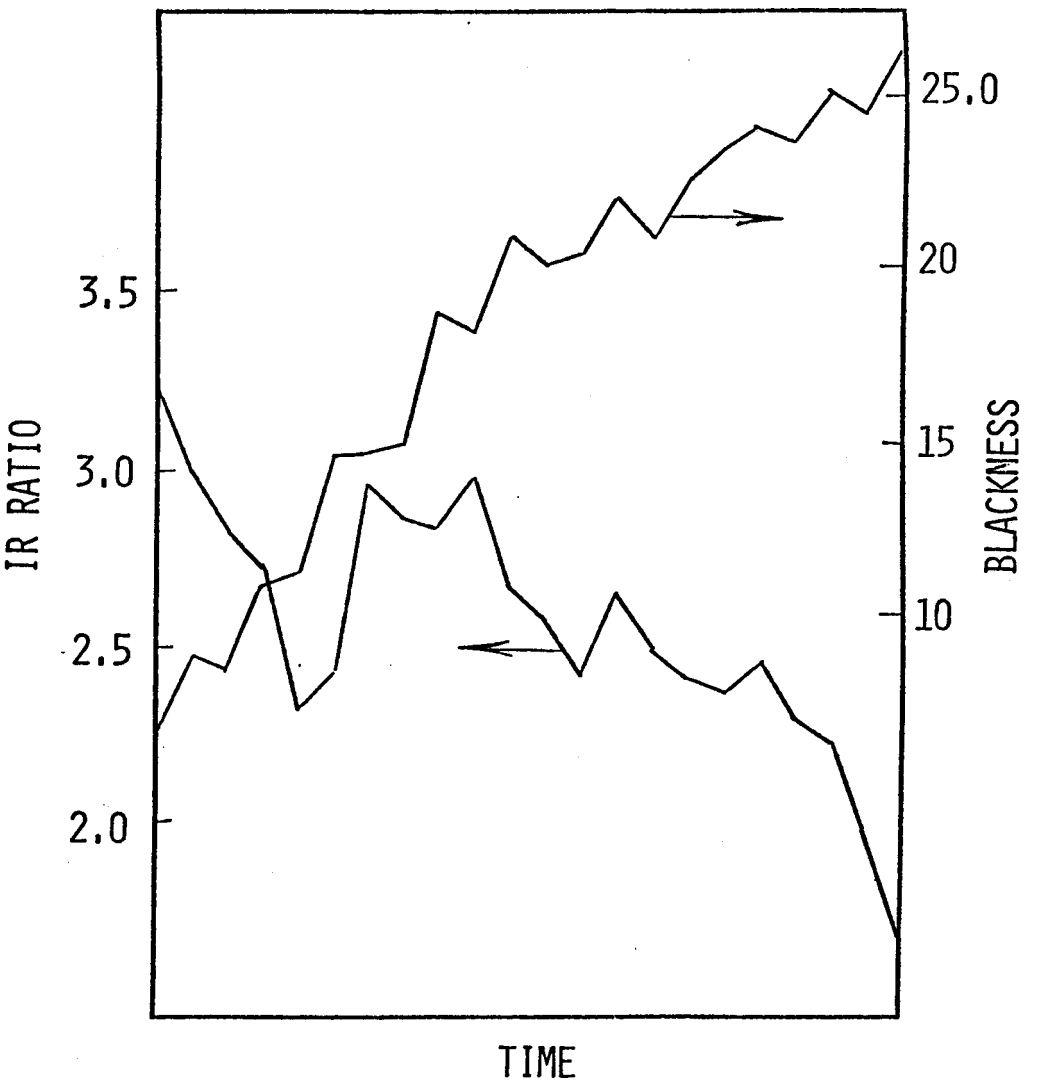
YIELD COMPARISONS: SRC I, SRC RECYCLE
WITHOUT MINERAL RESIDUE RECYCLE, SRC II

	Yield, Wt % MF Coal Basis		
	SRC I (GU 213)	SRC Recycle (GU 215)	SRC II (GU 216R)
C_1-C_4	10.5	15.4	16.1
Total Oil	25.9	--*	38.9
SRC	42.7	--*	21.0
Insoluble Organic Matter	4.1	7.0	5.1
H_2	-2.4	-5.0	-5.6

* Meaningful SRC and heavy distillate yields could not be determined.

Figure 32

GU 215 RUN CONTROL DATA
(SRC RECYCLE WITHOUT MINERAL RECYCLE)



Thus, a declining IR ratio indicates a declining degree of hydrogenation. The run control chart indicates that the degree of hydrogenation was decreasing throughout the run and at the end of the run was decreasing quite sharply.

Blackness is a measurement of absorbance in the visible region of a sample of coal solution in pyridine. Increasing blackness is an indication of increasing concentration of high molecular weight material. In addition to the declining IR and the high blackness measurement made at the end of the run, a further indication of the nonsuitability of these conditions was provided by a declining MAF conversion at the end of the run. For example, ash concentration in the pyridine insoluble fraction decreased from 62 to 53% in the final ten hours of the run. This corresponds to a decrease of MAF conversion from approximately 93.2 to 89.8% in only ten hours of operation.

4. Recycle of Mineral Residue Without SRC Recycle

Yields for the high mineral residue addition run (GU 219D) are compared with those for SRC I and SRC II operations in Table 19. It is readily apparent that a substantial increase in conversion of SRC to volatile products was brought about by the addition of a high level of mineral matter. Total oil yield increased from 25.9 to 37.0% and SRC yield decreased from 42.7 to 32.1% with mineral addition. The increased conversion is accompanied by the expected increase in hydrogen consumption. However, the yield of SRC with mineral residue addition is still appreciably higher than that obtained in SRC II operation. Thus, recycle of both SRC and mineral matter is required in order to reduce SRC yield to the desired level.

The relationship between yields in SRC I and SRC II operation with varying levels of mineral residue addition are best seen in graphical form; this is shown in Figure 33. Three levels of mineral residue addition were investigated. Yields for the SRC I process are shown on the left side of the figure. It is readily apparent that with 5% mineral residue addition (5% based on slurry) there is a significant reduction in SRC yield, accompanied by a significant increase in oil yield and a moderate increase in hydrocarbon gas yield.

With a further increase in mineral residue level to 10%, there is a further decrease in SRC yield and an accompanying increase in oil yield. However, with a further increase in mineral residue level to 15%, there is no further improvement in conversion of SRC to lighter products. In comparison, in SRC II operation, at a mineral matter level similar to that of the highest mineral residue addition, there is a further

TABLE 19

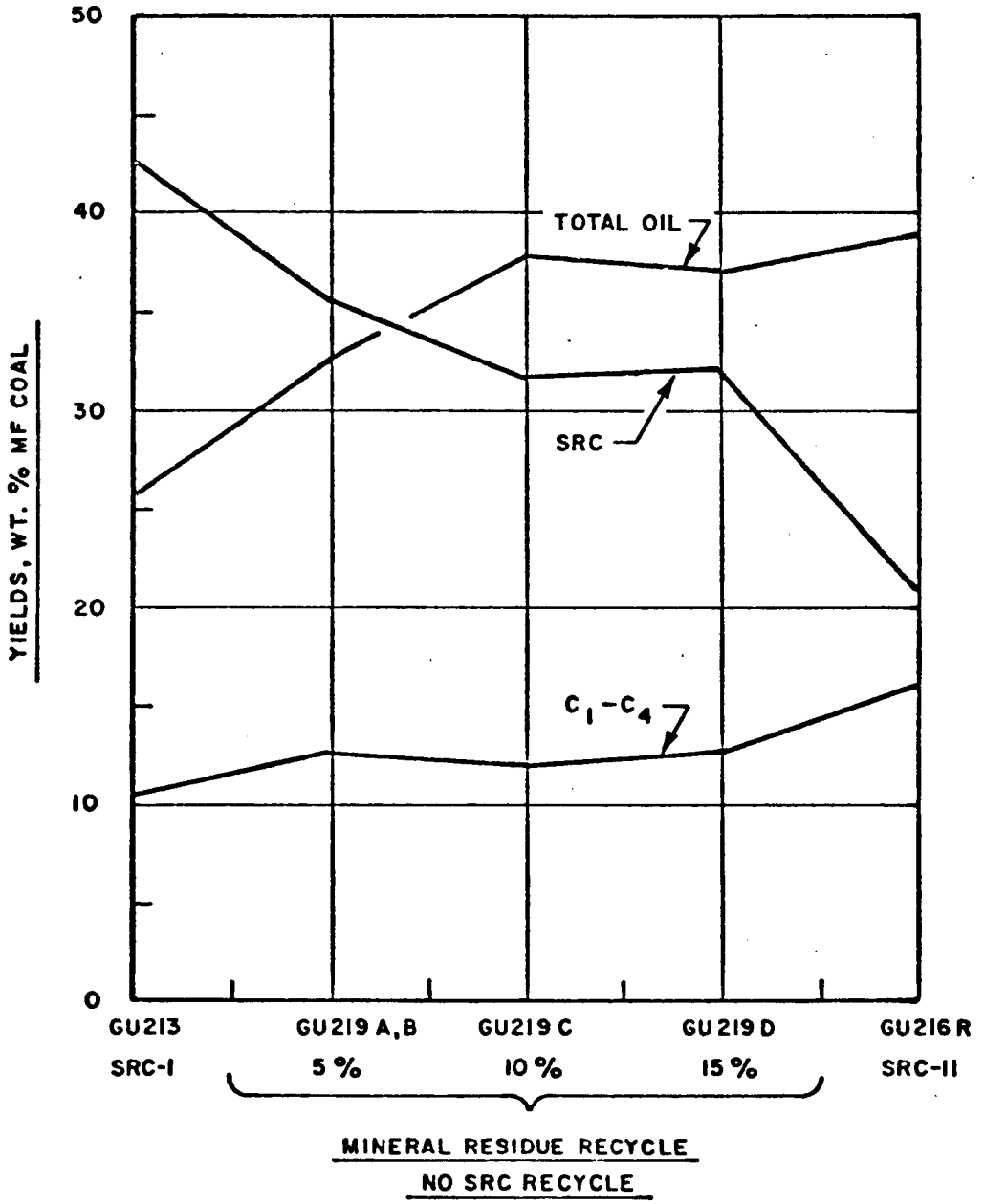
YIELD COMPARISON: SRC I, MINERAL RESIDUE RECYCLE WITHOUT SRC RECYCLE, AND SRC II

	Yield, Wt % MF Coal Basis		
	SRC I (GU 213)	Mineral Residue Recycle (GU 219D)	SRC II (GU 216R)
C_1-C_4	10.5	12.7	16.1
Total Oil	25.9	37.0	38.9
SRC	42.7	32.1	21.0
Insoluble Organic Matter	4.1	1.3	5.1
H_2	-2.4	-4.8	-5.6

FIGURE 33

YIELD COMPARISON:

SRC I, MINERAL RESIDUE ADDITION, SRC II



decrease in SRC yield and increase in hydrocarbon gas yield and a small increase in oil yield.

This work demonstrates that recycle of both SRC and mineral matter is important in the SRC II process. Recycle of SRC alone, without mineral matter, was shown to be unsatisfactory from an operational standpoint at the bench scale and, likely, also at larger scale. Addition of mineral matter alone causes a substantial increase in conversion of SRC to lighter products, but the SRC yield is still well above that required for hydrogen generation for the process. In order to obtain the conversion necessary for a plant in hydrogen balance, recycle of both SRC and mineral matter are required.

The flow scheme for mineral matter recycle is shown in Figure 34. In this case, slurry feed consists of coal, recycle distillate solvent, and recycle mineral residue. Due to the high level of mineral residue recycle required in order to match the mineral residue concentration obtained in SRC II operation, it was necessary that mineral residue be prepared from earlier runs. Two samples of mineral residue were used in this study. In both cases, the mineral residue was from Kentucky Nos. 9 and 14 coal. Mineral residue from Fort Lewis was used in two runs, and mineral residue from the Merriam operation was used in the remaining two runs of this series. Preparation and analyses of these mineral residue samples are discussed in the Experimental Section (Section VI-B-7).

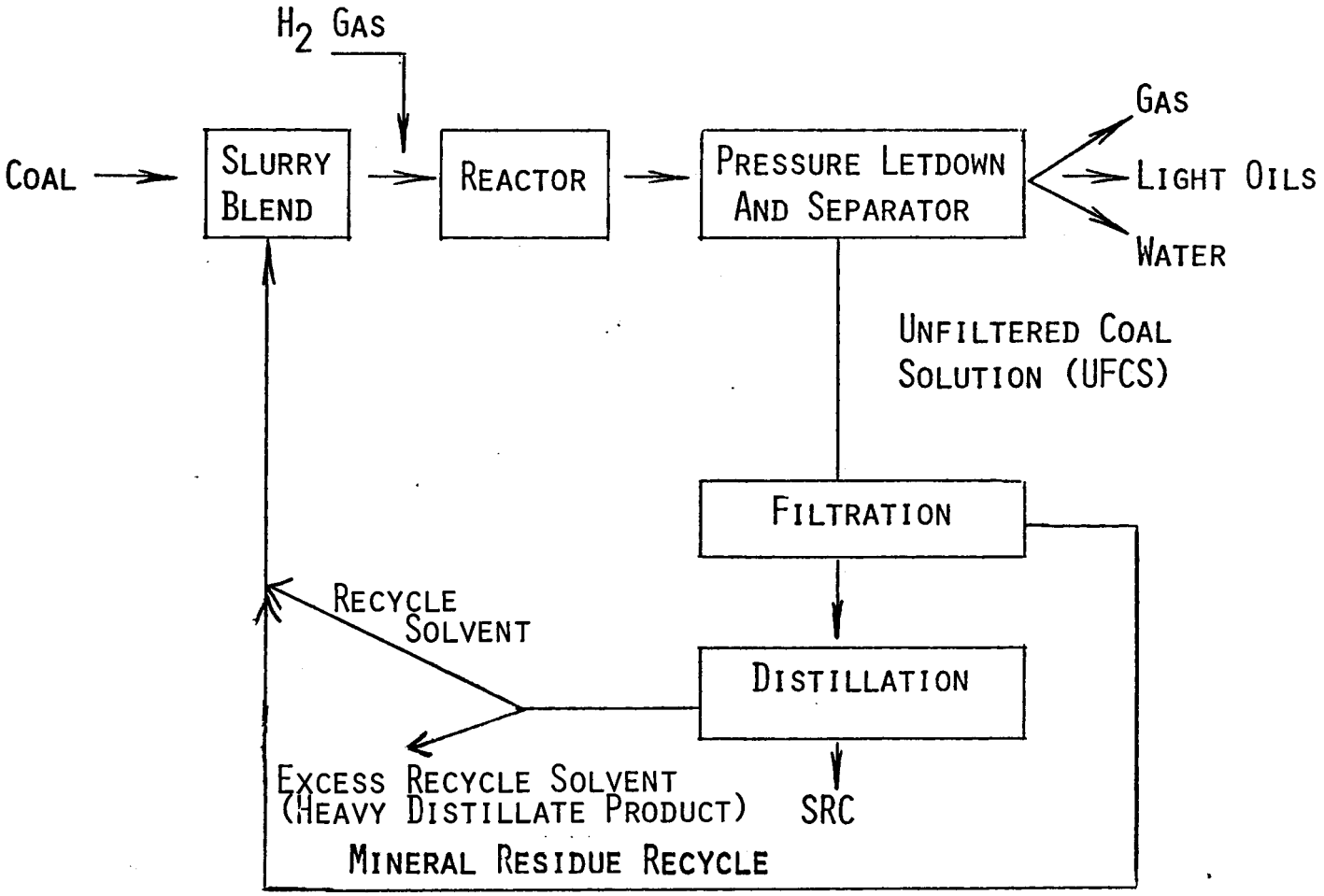
There was some concern that the Fort Lewis material might have been deactivated due to the severe temperature used in the mineral residue dryer. In addition, the Fort Lewis mineral residue contains filter aid. However, it was necessary to use mineral matter from Fort Lewis as it was not practical to prepare an adequate amount of material in the Merriam operation. The Fort Lewis mineral residue had a higher iron content (22.7 vs 14.5%) which could compensate for some lowering of catalytic activity, possibly caused by the drying operation.

Two runs were made (GU 219A, Fort Lewis mineral residue; GU 219B, Merriam mineral residue) to compare activity of the two mineral residue samples. The only variation between the runs was the source of the mineral residue. Similar results were obtained in each case. Total oil yield was 32.7% for each run. SRC yield was 1.9% lower and gas yield 1.2% higher with the Merriam mineral residue.

Results summarized in Table B-2 indicate that insoluble organic matter yield decreases with increasing mineral residue addition. This is an indication that the insoluble organic matter added with the mineral residue is not completely unreactive, but that some of it reacts thereby decreasing apparent insoluble organic matter yields.

Figure 34

MINERAL RESIDUE RECYCLE WITHOUT SRC RECYCLE



An additional aspect of mineral residue catalysis should be noted. This is, that in addition to catalyzing hydrogenation and liquefaction, mineral residue may also catalyze decomposition reactions during distillation. This point is discussed further in Section VI-D.

5. Effect of Recycle on Product Compositions

The effect of recycle on product composition is summarized in Table 20. As expected, mineral matter recycle was shown to increase the degree of hydrogenation although the increase is not large. In the heavy distillate product, hydrogen content increased from 7.96 to 8.18%. SRC hydrogen content increased from 5.70 to 5.84% with mineral recycle. With mineral recycle, there was a small improvement in desulfurization with oil sulfur content decreasing from 0.28 to 0.22%. Similarly, SRC sulfur content decreased from 0.49 to 0.38%. Little change in nitrogen content is apparent with mineral recycle.

Compositions of the product obtained with SRC recycle without mineral recycle are of significant interest. The hydrogen level of the products obtained with SRC recycle were the lowest obtained in this study. The presence of mineral matter apparently is required to maintain a satisfactory hydrogenation level. The high sulfur and nitrogen contents of the heavy distillate obtained with SRC recycle are consistent with an increase in conversion of the more refractory SRC to distillate material in comparison to SRC I. However, due to the absence of a high level of mineral matter, the heavy distillate is more poorly denitrogenated, hydrogenated and desulfurized than that obtained in SRC II operation.

6. SRC I with High Coal Concentration

One additional run was made in this series. This was a conventional SRC I run made with an increased coal concentration. In this run (GU 214), coal concentration in the feed slurry was increased to 45% so that the solids level was similar to that reached in the SRC II or high level mineral residue addition runs. Yields for this run (Table B-2) were essentially equivalent to those obtained in the SRC I run with a 30% coal concentration.

7. Experimental

a. Reactor

The recycle studies were carried out in the standard GU 5 reactor system. This reactor was described in the first quarter 1977 technical progress report[3]. The reaction subsystem consists of a preheater and two dissolvers operated in series. The preheater is a 4½-foot section of 11/16 inch pressure tubing and each of the dissolvers is a 7-foot section of the same diameter tubing. The

TABLE 20

EFFECT OF RECYCLE ON PRODUCT COMPOSITIONS

	SRC I (GU 213)	SRC Recycle (GU 215)	Mineral Recycle (GU 219D)	SRC II (GU 216R)
Heavy Distillate				
% Hydrogen	7.96	7.56	8.18	7.90
% Sulfur	0.28	0.36	0.22	0.29
% Nitrogen	0.98	1.45	0.94	1.21
SRC				*
% Hydrogen	5.70	5.08	5.84	
% Sulfur	0.49	0.40	0.38	
% Nitrogen	2.11	2.28	2.23	

* No solids-free SRC was available for analysis

preheater was operated with a temperature profile to produce a temperature of 400°C in the top zone. Due to the low temperature of this vessel, preheater volume is not included in calculation of reactor volumes, residence times, or feed rate expressed in lb/hr/ft^3. Reaction volume for each dissolver is 452 ml which corresponds to 520 grams of slurry. Thus, a slurry feed rate of 1040 grams per hour corresponds to a nominal liquid residence time of one hour. Reactor effluent passes from the dissolver to a pressure letdown and sample collection system. This system accomplishes the separation of gases, water, and light oil from the coal solution. Further workup of the coal solution (filtration, distillation, slurry formulation) is performed manually in batch procedures in the laboratory.

b. Mineral Residue Preparation

Mineral residue from the Merriam operation was prepared from wet filter cake collected in earlier runs in this series. Filtrations were carried out in the normal manner using glass fiber filter papers, electrically heated Buchner funnels, and house vacuum. No filter cake washing was attempted during filtration. Wet filter cakes were dispersed in wash solvent (bp 193-249°C), filtered through a screen to remove glass fibers, and then refiltered. Insolubles were washed with light solvent (bp <193°C), then with acetone, and air dried. The dried mineral residue was ground to pass through a 150 mesh sieve. Analysis of the mineral residue showed that it contained 5.3% pyridine soluble material, indicating that pyridine is a better solvent than the wash solvent used in the mineral matter preparation.

The only preparation required for the Fort Lewis mineral residue was sieving to pass 150 mesh. Compositions of the Merriam and Fort Lewis mineral residues are shown in Table 21.

TABLE 21

MINERAL RESIDUE COMPOSITION

	% Ash	Insoluble Organic Matter %	% SRC	% Wash Solvent	% Iron In Ash
Merriam	65.1	29.6	5.3	---	14.5
Fort Lewis	73.6	25.0	---	1.4	22.7

The Fort Lewis mineral residue contains 98.6% pyridine insolubles; in this case the other material is primarily wash solvent which is not totally removed in the drying step. The higher ash content of the Fort Lewis mineral residue is due partly to the presence of filter aid. The use of a higher ash coal or a slightly higher conversion could also contribute to the higher ash. Iron content of the Fort Lewis mineral residue was 22.7% in comparison to 14.5% in the Merriam residue. Fort Lewis mineral residue was used in runs DOE 219A and 219D, and Merriam mineral residue was used in runs DOE 219B and 219C.

C. Short Residence Time Runs

A series of short residence time SRC I runs was initiated during the second quarter. Discussion of these runs will be delayed until a later report.

D. Comparison of Results: Distillation of Filtrate vs Distillation of Unfiltered Coal Solution

As was reported in the previous quarterly report[4], product distributions can be based on distillation of either filtered or unfiltered coal solution. Yields reported in the summary tables for all runs, other than SRC II runs, are based on distillation of filtrate.

A comparison of yields based on the two methods showed that in SRC I operation yields of SRC based on distillation of unfiltered coal solution averaged 3.2% (absolute) higher than those based on distillation of filtrate. This increase in SRC yield is assumed to be due in part to the higher pressure in vacuum distillation of unfiltered coal solutions. Distillations are carried out under vacuum and are terminated at a head temperature of 270°C (518°F). It is possible to maintain a better vacuum when filtrates are distilled rather than unfiltered coal solution. When filtrates are distilled, the pressure at the end of a distillation is normally about 2-3mm Hg. The presence of mineral matter apparently accelerates decomposition reactions during distillation. Distillations of unfiltered coal solutions were normally terminated at the same endpoint (270°C, 518°F) but pressure at the endpoint normally reached 3-5mm Hg and in some cases reached 7-8mm Hg. The increase in SRC yield based on distillation of unfiltered coal solution is not believed to be due exclusively to difference in distillation pressure, however. Part of the increase in SRC yield is believed to be due to repolymerization reactions occurring during the distillation which may be catalyzed by the mineral matter present in the unfiltered coal solution.

The effect of mineral residue on distillation results was found to be even more pronounced in the runs with mineral residue addition. These results are summarized in Table 22.

TABLE 22

COMPARISON OF RESULTS: DISTILLATION OF FILTRATE VS DISTILLATION OF UNFILTERED COAL SOLUTION (Mineral Residue Addition Studies)

	Mineral Residue Level*	SRC Yield (Based on Filtrate)	SRC Yield (Based on UFCS)	SRC (UFCS) -SRC (Filtrate)
GU 219A, Filtrate	5.0	36.4	42.2	5.8
GU 219B, Filtrate	5.0	34.5	41.8	7.3
GU 219C, Filtrate	10.0	31.6	41.9	10.3
GU 219D, Filtrate	15.0	32.1	44.1	12.0

* Wt % of feed slurry

With the 5% mineral matter addition, the increased SRC yield based on distillation of unfiltered coal solution is only slightly greater than differences reported earlier for SRC I work. However, at the 10 and 15% mineral residue levels, the increase in SRC yield is larger than any observed previously. This comparison is of interest in consideration of forthcoming studies on the effect of additives in SRC II work.

In recent work, yields of SRC II operation have been based exclusively on the distillation on unfiltered coal solution. In studying the effect of additives, there is a possibility that, if the same procedures were followed, the beneficial effect of some additives could be overlooked as they might increase the conversion to distillate in the reactor, but then might also catalyze polyermization during distillation so that the increase in distillate yield would not be apparent. Alternate methods of unfiltered coal solution workup will have to be explored to avoid this possible problem.

E. Description of the DOE Continuous Reactor

1. Introduction

An upgraded continuous bench scale process development unit was put into operation in May 1978. It is designated the DOE unit to differentiate it from the earlier CU and GU units at Merriam. Four basic systems constitute the continuous unit:

a. Gas compression and metering
b. Slurry mixing, pumping, and metering
c. Reaction vessels
d. Product separation and recovery

Control and monitoring instruments for these four systems are centrally located at the control panel. Electronic controllers and instruments are employed on all major control loops and data acquisition equipment. From the control panel the operator monitors all vessel temperatures, pressures, levels, and pumping rates. In addition to basic control equipment, the panel contains an explosive gas monitor and equipment failure alarms.

Each major system is described in detail with drawings to provide an understanding of the unit and its capabilities. Volumes of major vessels and their typical operating temperatures and pressures are listed in Table 23.

2. Feed Gas Compression and Metering System

Gas supply to the compression and metering system is available from a bank of six tanks with a total capacity of 30 MSCF or from a manifold of standard 2000 psig gas cylinders. Since hydrogen is the primary gas used in the unit, the large storage tanks are reserved for hydrogen. This hydrogen source is maintained at a minimum purity of 99.95%. Hydrogen or other gases may also be supplied to the unit by the use of conventional 2000 psig cylinders.

Gas is removed from storage at a low pressure and recompressed to desired pressure. When the feed gas is pure hydrogen, the Pressure Products Industries (PPI) Model 1047 diaphragm-type compressor is the sole compressor operating. When a dual component feed gas is desired, the Aminco Model 46-14025 diaphragm-type compressor must be operated in addition to the PPI compressor. The Aminco compressor also serves as a backup for the PPI compressor when only hydrogen is being compressed.

The two gas compression subsystems differ slightly, as indicated in Figure 35. The PPI compressor receives 250 psi hydrogen and delivers 3000 psi hydrogen to two high pressure reservoirs. One high pressure reservoir supplies hydrogen for the purge of a reactor pressure gauge and a differential

Table 23

DOE REACTOR SYSTEM

Volumes and Typical Operating Conditions

	Volume	Temperature	Pressure
Preheater	300 cc	400°C	R
Dissolver	1000 cc	455°C	R
High Temperature, High Pressure Separator	675 cc	375°C	R
Intermediate Temperature, High Pressure Separator	330 cc	330°C	R
Ambient Temperature, High Pressure Separator	25 cc	25°C	R
Refrigerated Separator	500 cc	0°C	R
Atmospheric Flash	1600 cc	220°C	A + 5
Vacuum Flash	325 cc	220°C	2-10 mm Hg
Distillation Feed Accumulator	200 cc	25°C	A
Distillation Preheater	15 cc	250°C	A
Distillation Column	375 cc	200-350°C	A

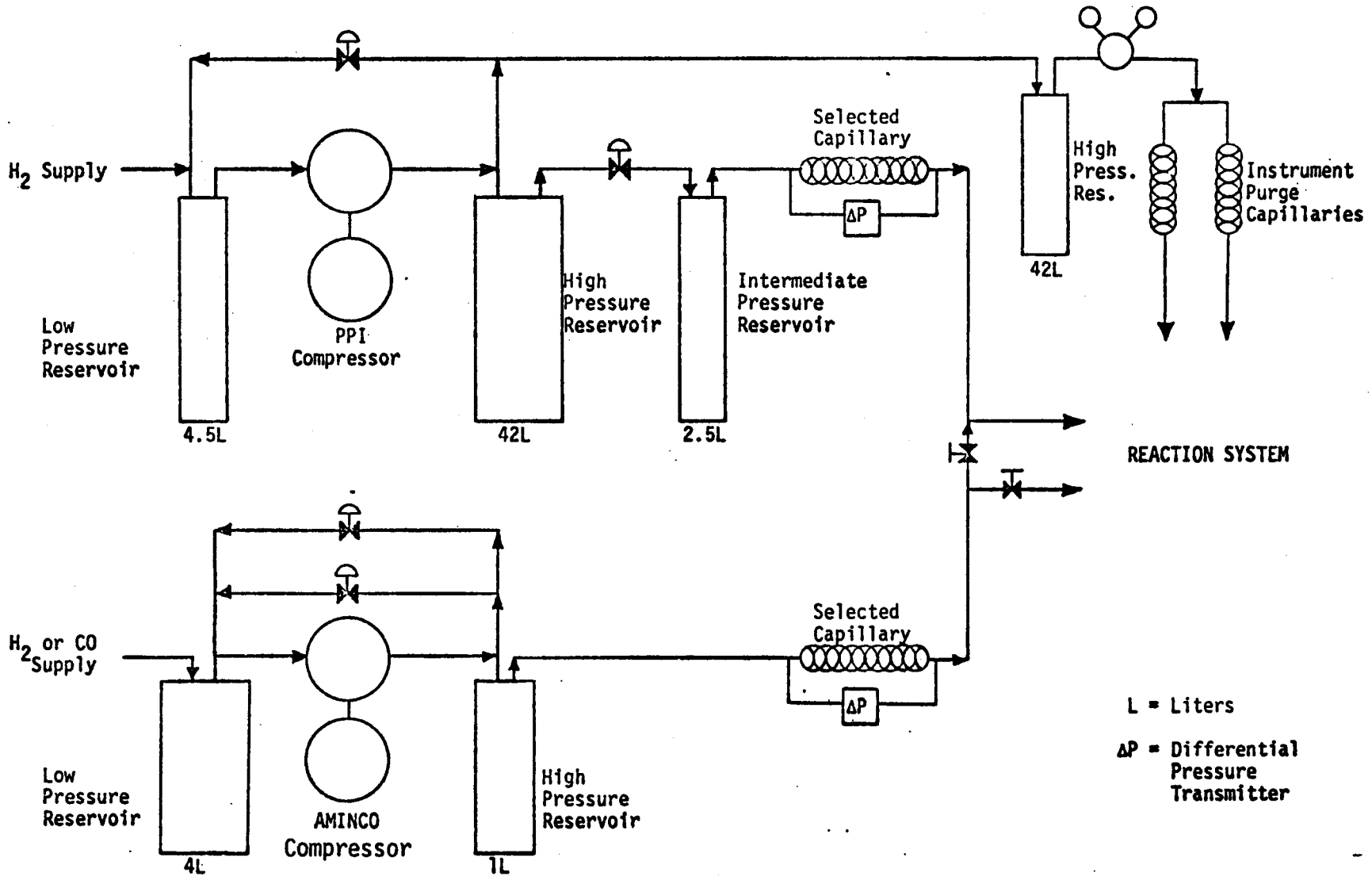
R = Reactor Pressure

A = Atmospheric Pressure

A + 5 = Atmospheric Pressure + 5 psi

Figure 35

GAS COMPRESSION AND METERING SYSTEM



pressure transmitter on the high pressure separator. Downstream pressure of these capillaries is reactor pressure while the upstream pressure is normally set 250 psi above reactor pressure. Three to five gram moles per hour of hydrogen are used as purge gas, depending on reactor pressure.

Hydrogen feed to the reaction system from the second PPI high pressure reservoir is controlled by regulating the pressure drop across a selected capillary. This differential pressure is monitored by a Rosemount Model 1151 DP transmitter. A Leeds and Northrup 440 Centry controller uses this signal to actuate a valve between the high pressure reservoir and intermediate pressure reservoir. The pressure drop across the capillary is controlled by regulating the pressure of the intermediate pressure reservoir. This reservoir is usually maintained 100 to 500 psi above reactor pressure, depending on the desired feed rate. With the high volume, high pressure reservoirs, the system can continue to deliver hydrogen for several hours after the compressor is shut down. In the event of a compressor failure, this allows time to cool the reactor before hydrogen flow is lost.

The Aminco compression system operates in much the same manner as the PPI system with the major difference being a small high pressure reservoir. This prevents any large amounts of gas from entering the reactor or building from this system in emergency situations. Both compression systems employ pneumatic Fisher pressure controllers on the high pressure reservoirs. In the PPI system, the Fisher pressure controller regulates the pressure of the two high pressure reservoirs. Compressed hydrogen from the high pressure reservoirs is returned to the low pressure reservoir when pressure in the high pressure reservoir exceeds a predetermined value.

The pressure of the high pressure reservoir in the Aminco system is maintained by a Leeds and Northrup 440 Centry controller which controls a second control valve between the high pressure reservoir and the low pressure reservoir. The Centry controller obtains its signal from a Rosemount Model 1151 DP transmitter connected across the feed capillary. The Fisher pressure controller operates only if the high pressure reservoir exceeds normal pressure.

Feed from the Aminco compression system may be combined with the feed from the PPI compression system or may be introduced to the reaction system separately.

3. Slurry Mixing, Pumping and Metering

Slurry mixing, pumping and metering equipment is enclosed in a plexiglas hood equipped with an exhaust fan. Plexiglas panels on the sides of the hood may be removed for easy access

to the equipment. Three BLH Electronics 40 pound capacity strain gauge scales are located inside the hood to provide weights of material in the coal solution product flask, slurry mix vessel and slurry feed vessel. Figure 36 shows the layout of equipment in this system.

Slurry is formulated by adding minus 150 mesh coal to the slurry mix vessel which already contains recycle solvent or unfiltered coal solution from the unit. An air motor driven agitator continuously stirs the mixture while a Tuthill Model 4A lobe pump circulates the slurry. The stainless steel mix vessel has an eight liter capacity and is heated by band heaters. Temperatures are controlled by Love Model 49 or 51 proportional temperature controllers.

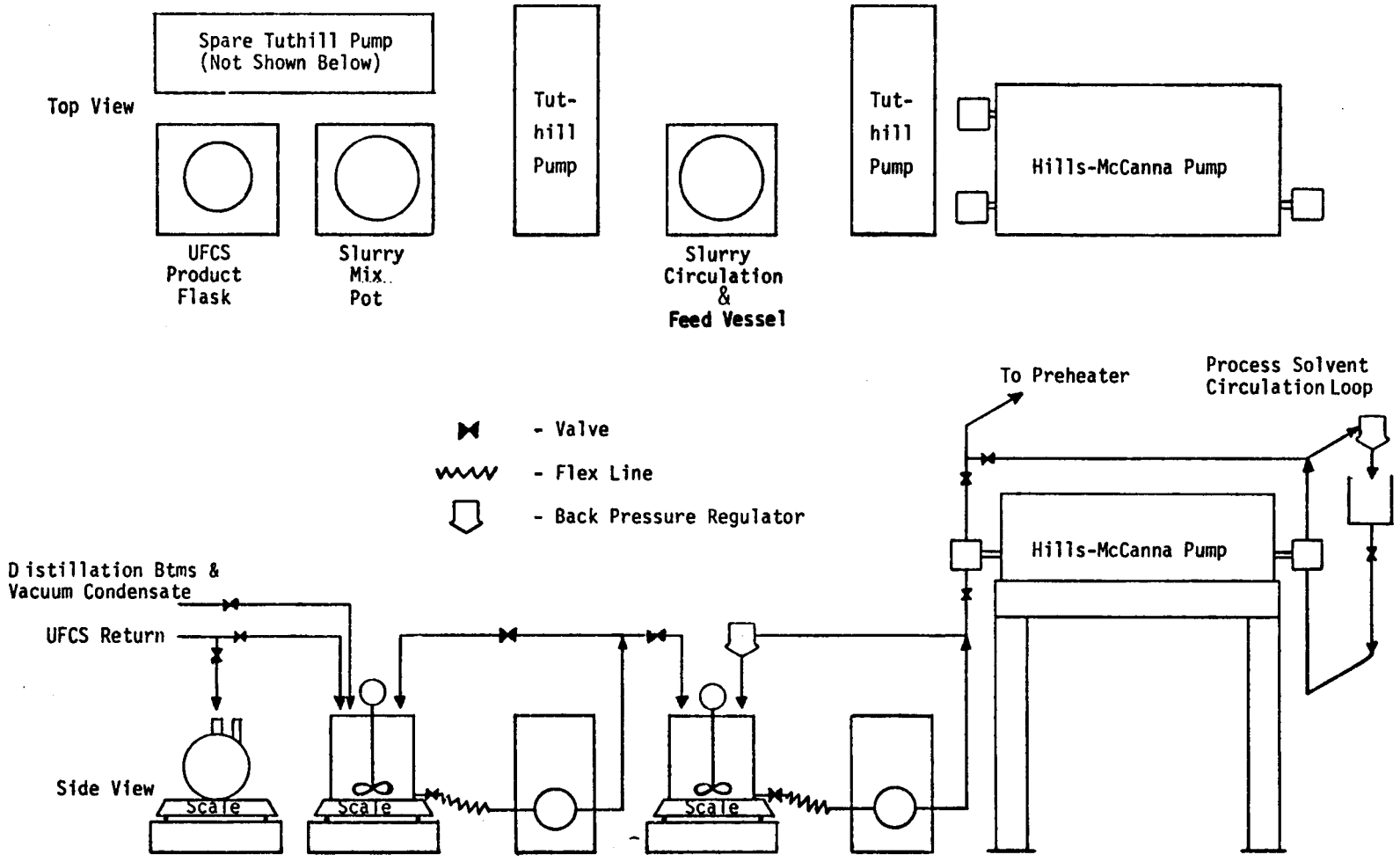
When slurry formulation is complete, it is transferred to the slurry feed vessel. Another Tuthill lobe pump continually circulates the slurry and provides pressure in the loop to feed a dual head Hills-McCanna Model U positive displacement pump. Pressure on this circulation loop is maintained between 10 and 100 psi, depending on the viscosity of the slurry. A tungsten carbide trimmed back pressure valve regulates the pressure in the loop. Slurry from this loop is pumped to the preheater in the reaction system by the Hills-McCanna positive displacement pump. This pump elevates the slurry pressure to reactor pressure while simultaneously metering it. The pump rate is controlled by adjusting the pump stroke or altering the motor speed. The General Electric DC motor that drives the Hills-McCanna pump is controlled by a Louis Allis Saber 3200 DC static drive. The motor speed may be set manually or may be controlled automatically by the Louis Allis drive.

The BLH Electronic's weigh system calculates the flow rate from the decreasing weight in the slurry feed vessel and sends a proportional current to a Leeds and Northrup Centry controller. The Leeds and Northrup controller conditions the signal and transfers it to the Louis Allis drive. The motor speed is adjusted by the Louis Allis to maintain a constant flow rate. Accuracy of the BLH Electronics calculated flow rate has been at best $\pm 5\%$, and usually no better than $\pm 10\%$. Modification of this system is under review. Presently, manual pump rate control is used, giving adequate control.

A third pump head on the Hills-McCanna pump circulates process solvent at reactor pressure to provide an alternate feed to the preheater. During heat-up and shutdown procedures, process solvent is pumped into the reaction system. If slurry feed is stopped for any reason, process solvent will be pumped into the reaction system to prevent coking or material buildup. All piping from the Hills-McCanna pump to the reaction system is 3/8 inch Autoclave high pressure tubing. The unit contains appropriately rated rupture discs at possible overpressure areas.

Figure 36

SLURRY MIXING, PUMPING AND METERING



4. Reaction System

The reaction system contains two vessels: a preheater and a dissolver. The dissolver used in this system may be changed to provide a wide range of reactor volumes or to study reactor designs. A 300cc preheater is located upstream from the dissolver. Slurry and feed gas enter the reaction system continuously at the base of the preheater at a temperature below 200°C. Strip heaters supply heat to the one-inch Autoclave high pressure tubing preheater. Flow through the preheater is upward and a transfer line delivers the slurry and gas to the base of the dissolver. Slurry temperature inside the preheater is monitored by three thermocouples in a thermowell. Temperatures are controlled based on the internal thermocouples while external wall temperatures are also monitored.

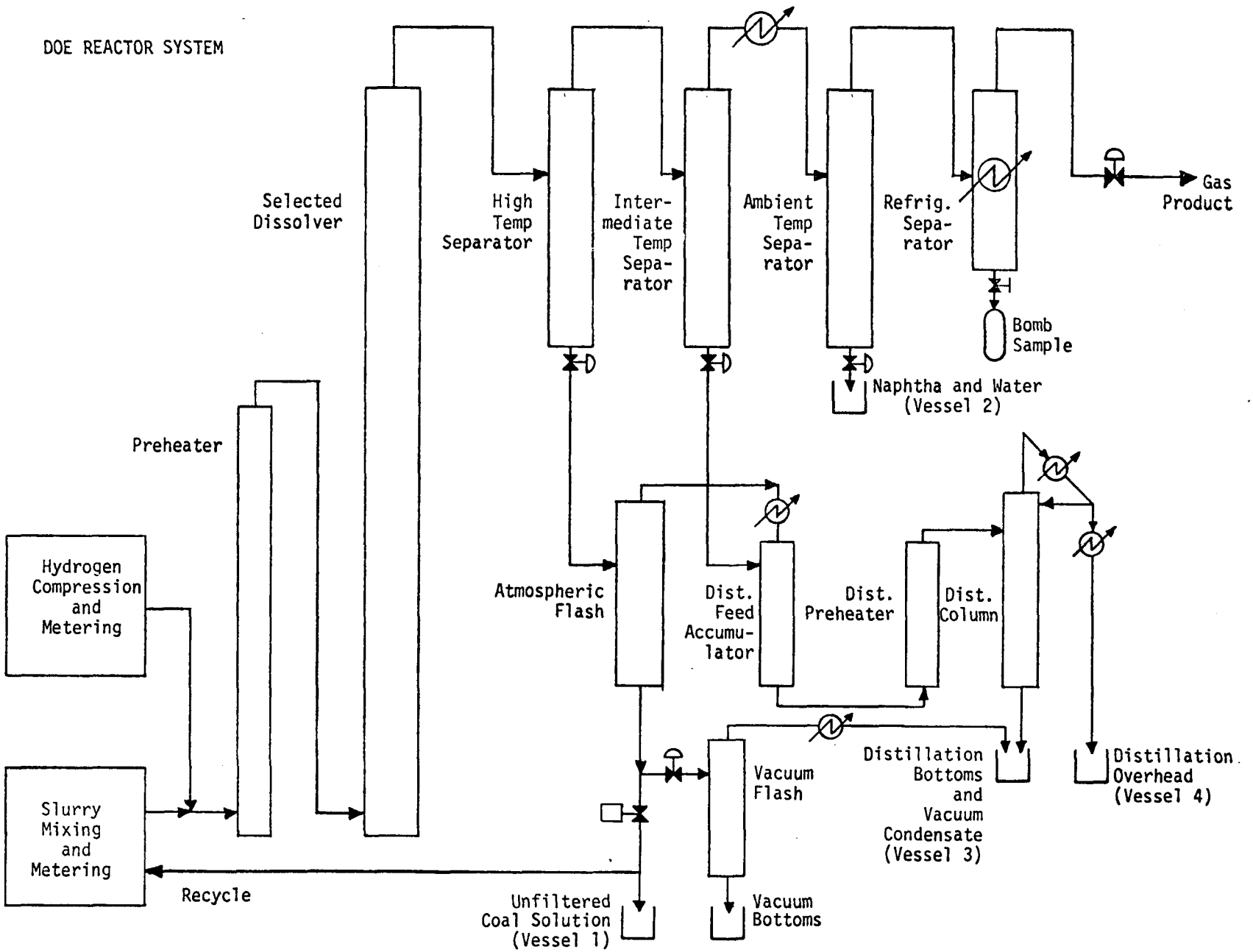
The dissolver is located inside an electrically heated air circulation furnace. The furnace, custom built by Heat Enterprises, circulates hot air in six independent zones. Temperatures of each zone are controlled by Love temperature controllers and are relatively independent of adjacent zone temperatures. The space inside the furnace is one foot square and seven feet tall. Smaller dissolvers are usually selected lengths of one-inch Autoclave high pressure tubing while larger dissolvers are custom built. Like the preheater, thermowells with thermocouples are installed in the dissolvers to monitor the slurry temperature in each of the six heating zones. Dissolver surface temperatures are also monitored to prevent excessive external temperatures. Gas and slurry effluent from the dissolver flow to the high pressure, high temperature separator.

5. Product Separation and Recovery

This system, shown in Figure 37, may be divided into two categories: reactor-pressure vessels and low-pressure vessels.

Product from the dissolver first enters the high temperature separator (HT separator) where the initial vapor-liquid separation occurs. The temperature of the product typically is reduced to approximately 375°C with vapor exiting at the top and slurry being removed at the bottom. At this point the slurry will be referred to as unfiltered coal solution (UFCS) to differentiate it from the feed slurry. The level of UFCS in the HT separator is maintained by a level control valve. Two valves are mounted in parallel with the second serving as a backup. The primary level control valve is a Fisher Model 530 Gismo valve. The spare valve, a one-fourth inch Research Control valve, may be put into operation when the Gismo valve fails to operate properly. Since the UFCS is high in solids and the pressure drop across these valves is high, all trim is constructed from tungsten carbide.

Figure 37


DOE REACTOR SYSTEM
Selected Dissolver
High Temp Separator
Inter-mediate Temp Sepa-rator
Ambient Temp Sepa-rator
Refrig. Sepa-rator
Gas Product
Bomb Sample
Naphtha and Water (Vessel 2)
Preheater
Hydrogen Compression and Metering
Slurry Mixing and Metering
Recycle
Atmospheric Flash
Dist. Feed Accumu-lator
Dist. Preheater
Dist. Column
Vacuum Flash
Distillation Bottoms and Vacuum Condensate (Vessel 3)
Distillation Overhead (Vessel 4)
Unfiltered Coal Solution (Vessel 1)
Vacuum Bottoms

Operation of the UFCS letdown valve depends on the level sensing device on the HT separator. A hydrogen purged Beckman Model 8610 differential pressure transmitter is used. The liquid pressure tap is a one-fourth inch stainless steel dip tube extending from the top of the vessel. The HT separator is fabricated from 1½ inch schedule XX 321 stainless steel pipe with a ring joint flange for easy cleaning and inspection. A Leeds and Northrup Centry controller monitors the signal from the Beckman differential pressure transmitter and actuates the UFCS letdown valve to maintain constant level.

Overhead vapors from the HT separator enter the intermediate temperature separator (IT separator) while the UFCS flows to the atmospheric flash vessel. The overhead vapors are condensed in three stages and the remaining gases are piped to a gas chromatograph for analysis and to a wet test meter for flow rate determination. Temperature of the gas stream is typically reduced to 330°C in the IT separator. Condensate accumulates in this vessel while the gas stream flows to a high pressure condensor. The IT separator is constructed from an Autoclave one-inch high pressure tube. Liquid is removed from this vessel through a one-fourth inch Research Control Valve (RCV) and transferred to the distillation feed accumulator. Similar to the control loop of the HT separator, a Beckman differential pressure transmitter and a Leeds and Northrup Centry controller actuate the liquid letdown RCV. The liquid level is maintained at the elevation set on the controller. The pressure taps on the IT separator do not require a purge.

In the high pressure condenser vapor temperature is reduced to approximately 25°C. Liquids formed in the high pressure condenser are separated from the gas stream in the ambient temperature separator (AT separator). The AT separator is 9/16 inch high pressure tube. Liquid level control and liquid removal is identical to that of the IT separator except the product is removed from the system. The liquid, which is primarily water and naphtha, accumulates in a collection vessel until an operator drains the material for analysis.

The gas stream from the AT separator flows to a refrigerated separator. The refrigerated separator is a 500cc autoclave immersed in a cooling bath of water and ethylene glycol. The cooling bath is maintained at a temperature of 0-5°C by a Neslab Model PBC-2 refrigeration unit. The vessel is drained by opening a valve and allowing the collected liquid to flow into a 90cc sample bomb. The primary function of the refrigerated separator is to serve as a gas cleanup; this extends the life of packing in the gas chromatograph columns. Reactor pressure is monitored by a Beckman Model 8645 pressure transmitter located between the AT separator and the refrigerated separator. This transmitter sends a signal to a Leeds and Northrup Centry controller which actuates one of

two RCV's on the gas effluent line of the refrigerated separator. The Research Control Valves are mounted in parallel similar to the UFCS letdown valves to allow for the failure of one valve. Pressure of the entire high pressure reaction system is controlled by this valve.

There are five vessels in the low pressure portion of the product separation and recovery system. The atmospheric flash and the vacuum flash separate oil from the UFCS while the distillation column and associated vessels separate the oil into process solvent and lighter oils. The atmospheric flash is constructed from two-inch schedule 40 stainless steel pipe. A Robertshaw Model 160 ceramic coated capacitance probe is installed in the vessel to provide an indication of UFCS level. When the UFCS exits the bottom of the atmospheric flash, it may be directed in three directions. It can be transferred to the vacuum flash vessel, to the slurry mix vessel, or to the UFCS product flask. When the vacuum flash vessel is used, the capacitance probe signal is used by a Leeds and Northrup Centry controller to regulate the RCV between the two vessels. This controls the level of UFCS in the atmospheric flash and the feed to the vacuum flash. If the vacuum flash is not operated, then UFCS is removed from the atmospheric flash by opening a one-half inch Whitey ball valve. This valve is actuated by an adjustable timer and the UFCS can be transferred to a product flask or to the slurry mix vessel. Pressure in the atmospheric flash is maintained at about five psig by a back pressure regulator on the gas exit line. The vacuum flash vessel is constructed from one-inch schedule 40 stainless steel pipe and is connected to a Kinney Model KC-15 vacuum pump. The vacuum flash system is not currently operated due to the unreliability of the original cycle timer and delays in the delivery of a programmable electronic timer. When the system is operated, UFCS enters the vacuum flash vessel where process solvent range liquid is flashed. The vapors are condensed before they reach the vacuum pump. This process is interrupted several times an hour to remove vacuum bottoms from the vacuum flash vessel and condensate from the vacuum flash condensate receiver. The timer coordinates the opening and closing of four valves. Five psig nitrogen forces the vacuum bottoms from the system and transfers the condensate to the atmospheric distillation bottoms receiver. During this product removal process, the vacuum pump and feed are isolated from the system.

IT separator bottoms and condensed vapors from the atmospheric flash are combined in the distillation feed accumulator and pumped into the distillation preheater. A Beckman Model 8610 differential pressure transmitter monitors the level in the distillation feed accumulator. A Leeds and Northrup Centry controller adjusts the speed of the Lewa Model FLK-1 feed pump, depending on the level in the accumulator.

The distillation column is constructed from one-inch schedule 40 stainless steel pipe and packed with miniature stainless steel helices. The overall volume of the column is 375cc with the reboiler constituting one-third of the volume. Temperatures in the column typically range from 350°C in the reboiler to 200°C in the overhead line. Feed to the column is normally maintained at 250°C. Overhead vapors are condensed and recycled to the top of the packing. A Whitey three-way ball valve is actuated by an adjustable Lab Line timer to remove a small amount of reflux as distillation overhead product. The purpose of this on-line distillation column is the removal of lighter oils from the process solvent. Distillation bottoms may be combined with the vacuum condensate and returned to the slurry mix pot for formulation or removed from the system as product. Distillation overhead oil is usually considered as light oil product but may also be returned to the slurry mix pot for formulation.

F. Merriam Maintenance, Modifications and Construction

1. GU 5 Unit

Operation of the GU 5 unit continued until the first of May when startup of the DOE unit commenced. Maintenance on the GU 5 unit was limited to replacement of packings on charge and recycle pumps and replacement of slurry transfer lines which became plugged in an attempt to add 15 percent mineral residue to the feed slurry. No modifications were performed on this equipment during the quarter.

2. DOE Unit

a. Construction

Construction of all major systems on the upgraded bench scale unit were completed by May 1 with the following items completed during the quarter:

1. High pressure hydrogen lines in the gas compression and metering system were completed to allow pressure testing of the system.

2. A hydrogen purged differential pressure transmitter was installed in the high-pressure separator to measure liquid level.

3. A refrigerated bath was constructed for the one-half liter high pressure Autoclave vessel used as a final high pressure, vapor liquid separator.

4. The air circulation furnace for temperature control of the dissolver was installed and dissolver supports inside the furnace were completed.

5. Framework for the fume hood over the slurry mixing and pumping station was erected.

6. Excess flow check valves and bypass lines were installed on all lines leading to pressure gauges on the control panel.

7. The Aminco compressor was connected to the hydrogen metering capillaries to provide a back-up compressor for the DOE unit.

b. Maintenance

Principal areas of maintenance during the quarter were:

1. Differential pressure transmitters on the high temperature separator, room temperature separator and distillation feed vessel were replaced during the period. Faulty diaphragms in the new Beckman D/P cells were the cause of the instrument failure in each case. The BLH flow rate calculator and the Fluke datalogger malfunctioned during the period. The BLH instrument required shipment to the manufacturer for repairs.

2. Trim in the slurry letdown Research Control Valves was replaced weekly due to their failure to hold pressure caused by erosion.

3. An overflow of slurry from the atmospheric flash vessel and the high-temperature separator contaminated downstream vessels. Since these vessels were not designed to handle slurry, the system was disassembled and cleaned.

4. Stainless valve trim on the reactor pressure control valve was replaced by a stellite trim of the same C_V.

5. The liquid product removal line from the atmospheric flash vessel plugged on three occasions and additional heat tracing was installed.

6. Non-uniform wrapping of a heat tape caused the line between the atmospheric flash vessel and the distillation feed vessel to fill with coke. The line was replaced and the heat tracing was modified to prevent future hot spots.

c. Modifications

Several modifications to the DOE unit were made to improve performance of the upgraded unit. These were:

1. Slurry was found to be present in the hydrogen purged lines of the Beckman differential pressure transmitter on the high temperature separator. Reorientation of the transmitter, alteration of the purge lines, and installation of an independent hydrogen feed for the purge improved the performance of the system.

2. Ruby ball checks in the Lewa distillation feed pump were replaced with tungsten carbide balls due to the inability of the ruby balls to handle low viscosity fluid.

3. To prevent solids from entering the pump head of the Lewa distillation feed pump, a filter was installed on the inlet line.

4. The Louis Allis slurry feed controller and the Lewa pump controller required the installation of isolation transformers on their power supply lines to prevent their interference with the Leeds and Northrup process controllers.

VII. PROCESS DEVELOPMENT UNIT - P99

A. Introduction

The P99 unit is a nominal one ton per day process development unit configured for SRC II operation. The unit is located at Gulf Science & Technology Co., Harmarville, Pennsylvania. Operation of the P99 unit has become part of the Solvent Refined Coal (SRC) Process contract effective April 1, 1978 by a modification of the contract between DOE and P&M.

The following sections provide a description of the P99 unit and of activity on the unit during the second quarter 1978.

B. Description of P99 Unit and Process Flow

P99 is an integrated pilot plant with recycle of gas, process solvent, and reactor effluent slurry being carried out on a continuous basis. The flow scheme in P99 essentially duplicates that of the SRC II module of a commercial plant; however, holdup relative to feed rate is higher in P99 than it would be in a commercial unit so that lineout time is somewhat longer. Also, the distillation scheme in a commercial unit would probably be somewhat different from that in P99.

Coal is delivered to P99 in aluminum tote bins already dried and ground. Hydrogen is provided from the central system which services the entire Gulf Science & Technology Company Research Center. Waste gas is sent to the Research Center boiler house where it is

burned along with the regular fuel. Waste water is collected in 55-gallon drums and hauled away for disposal. Other products are also collected in 55-gallon drums and stored until used or disposed of. The feed rate to P99 is generally in the range of a half a ton of coal a day.

Figure 38 presents a simplified flow diagram of P99. A discussion of the process flow is presented in the following sections.

1. Coal Handling

The dried, powdered coal is received in aluminum tote bins which are approximately 4 x 4 feet square by 6-1/2 feet high. Each bin contains approximately 1.5 tons of coal. These bins are moved by a fork lift truck to the dumping station. From the dumping station, the coal is conveyed to a 30-ton hopper by means of a series of screw feeders and a gas lift. A star feeder in the bottom of the hopper dumps the coal onto a weigh belt which controls the coal feed rate to the unit. The weigh belt dumps the coal into a screw feeder which conveys it to the unit. The coal enters the feed mix tank through a funnel-shaped "coal chute."

2. Feed Mix Section

The mix tank has a capacity of 100 gallons, but it is operated at as low a level as possible to keep slurry inventory and residence time to a minimum. This tank is equipped with a stirrer and a pumparound system to mix the coal with the slurry and to prevent settling. The tank is Teflon lined to help prevent buildup of material on the walls. Also, the discharge from the pumparound system is returned to the mix tank tangentially near the top of the tank to keep the walls washed down.

The mix tank and all the associated pumps and lines are steam-traced to prevent plugging caused by solidification of the feed slurry. Normal mix tank operating temperature is approximately 100-110°C. The mix tank is connected to an eductor system which maintains a slight vacuum in the tank. This prevents vapors from getting into the coal chute, condensing, and causing plugging problems. The eductor gas from the mix tank is passed through a freon-cooled scrubber for recovery of hydrocarbon vapors. Any recovered material is returned to the fractionation system.

Recycle slurry is prepared by mixing bottoms from the atmospheric flash column and recycle process solvent in Tanks 4 and 5. Two tanks are used to get accurate feed rates (one tank is being filled while the other is emptied). These tanks are mounted on weigh cells, and tank weights are recorded each hour. After the weights are recorded the tanks are switched. Tanks 4 and 5 are fitted with stirrers and pump-around systems to keep the contents well mixed. The slurry from these tanks is sent to Tank 1 by means of a metering pump.

MAKE-UP H_2

COAL

TO VAPOR SCRUBBER

LIQUIDS TO L.P. SEPARATIONS SYSTEM

COAL THROAT

CONTINUOUS BLEND AT MINIMUM INVENTORY #1

50 GAL FEED TANK

150# STM

#1FS

#2FS

TUTHILL 30A

TUTHILL 30A

TARE

#12 FS

250 GAL HOT FLUSH TANK #2

150# STM

TUTHILL 30A

TO FLUSH SYSTEM OF H.P. FLUSH PUMP

#3FS

#4FS

H.P SLURRY PUMP

RECYCLE GAS

QUENCH

REACTOR & FURNACE

AGITATION GAS

H_2O PUMP & WEIGHT TANK

HEATER

C. OIL

INTER. SEP.

C.W.

H.P HOT SEPARATOR

HEATER

TO WATER STR.

H_2O REC.

COLD H.P SEPARATOR

LOW PRESS SURGE TANK

C. OIL

THROTTLING VALVE

OSCILLATING VALVES

500-650°F

DP

FLASH COL.

HIGH TEMP. VALVE

TO DECUT.

SPLITTER

#13FS

#14FS

TO RECYCLE SLURRY

TO VAC. COLUMN

EMERGENCY DUMP VALVE

BLOW DOWN TANK

FLUSH SOLVENT

FRACTIONATOR

C.W.

WATER SEP.

H_2O

DEBUTANIZER

WEIGH TUBES

BLEED GAS

AMINE SCRUBBER

AMINE REACTIVATOR COL

AMINE FEED TANK

KEROSENE SCRUBBER

KEROSENE REACTIVATOR COL.

SUCTION SURGE TANK

COMPRESSORS

DISCHARGE SURGE TANK

TO VAC STM JETS

VAC OVHD RECYCLE

NORTHERN PUMP/WITH AIR MOTOR

VAC. FLASH COL.

REHEATER

50 GAL HOLDUP TANK

VAC. BTMS. RECVS.

#11FS

SLURRY RECYCLE

RECYCLE SLURRY

STEAM TRACE

PROCESS SOLVENT WT. TANK 276 GAL #9

PROCESS SOLVENT WT. TANK 276 GAL #8

PROCESS SOLVENT PRODUCT WEIGH SYSTEM

TANK #6

TANK #7

FRACTIONATOR WEIGH SYSTEM

ATM BOTTOMS PRODUCT

METERING PUMP

50 GAL PROCESS SOLV. PRODUCT STORAGE #11

TO DRUMS

DILUTION STACK

250 GAL. UPSET SURGE TANK OFF-LINE DUTY #3

TO VAPOR SCRUBBER VENT

#4

#6FS

TUTHILL 15A

TO VAPOR SCRUBBER VENT

#5

#7FS

TUTHILL 15A

#8FS

#9FS

HILLS McCANNA DUPLEX (PLUS SPARE)

RECYCLE SLURRY WEIGH AND BLEND SYSTEM

#5FS

TUTHILL 15A

#10FS

HILLS McCANNA DUPLEX

OFF-LINE MATERIAL DRAW OFF

REVISIONS

A

REV 6 - GENERAL REVISION S.L.M. 6-25-78 APR'VD - W.G. 6/26/78

REV 5 - GENERAL REVISION S.L.M. 11-1-77 APR'VD - "

REV 4 - GENERAL REVISION S.L.M. 5-2-77 APR'VD - W.G./F.T.R.

REV 3 - GENERAL REVISION S.L.M. 2-26-76 APR'VD - T.M.

REV 2 - GENERAL REVISION S.L.M. 2-19-76 APR'VD - W.G.M.

REV 1 - GENERAL REVISION S.L.M. 1-29-76 APR'VD - T.D.A.

REVISIONS

REF. DWGS.

SRC II - MODIFICATION
P-99 PILOT PLANT
ONE T/D - BASE CASE
CONTINUOUS SYSTEM

GULF RESEARCH & DEVELOPMENT CO.
PITTSBURGH, PA.

DATE 1-21-76 SCALE —

DRAWN S.A.P. - S.L.M. APR'VD. W.G. 4/26/78

TRACED APR'VD.

CHK'D. APR'VD.

DWG. No. ES-D-756

Figure 38

3. High-Pressure System

The pump-around loop on Tank 1 flows past the suction of the high-pressure slurry pump. This is a two-sided hydraulic pump. Both sides can be operated simultaneously, but normally only one side is operated with the other side being kept as a spare. The speed of this pump is controlled by a radioactive level detector (Kay-Ray) on Tank 1. Thus, the reactor feed rate is controlled by the metering pump on Tanks 4 and 5 and the weigh belt feeder on the coal hopper.

After being pumped to system pressure, the feed slurry is mixed with recycle gas and passed downflow through the reactor preheater. This preheater is an electrically heated coil. It has five heating zones so that the temperature profile in the preheater can be controlled as desired. From the preheater the reactor feed enters the bottom of the reactor. The reactor is an empty vessel and agitation is achieved by the upward flow of gas. There is one quench port, at about the middle of the reactor, for temperature control. There is an emergency dump valve at the bottom of the reactor, and a small stream of flush oil is pumped through the line containing this valve to make sure it will be free to operate, if needed.

Reactor effluent leaves the top of the reactor and flows to the hot high-pressure separator. In this vessel, vapor-slurry separation occurs. The hot separator is provided with a hydrogen sparge to keep the slurry well mixed. The slurry from the separator flows through a hot oil cooled exchanger and then to the letdown valve system. This system consists of two valves in series with a small surge tank in between. These valves are operated alternately so that the surge tank is first filled from the hot separator and then emptied to the atmospheric flash column. Operation of these valves is controlled by a level controller on the hot separator. Two sets of letdown valves are piped in parallel with each other so that a spare is available at all times.

The vapor from the hot separator is cooled and sent to the intermediate separator where condensed hydrocarbons are removed. The vapor from the intermediate separator is further cooled and sent to the cold separator where both water and liquid hydrocarbons are removed. The hydrocarbon stream is combined with that from the intermediate separator and sent to the fractionation system. The water is combined with the water from the low-pressure system and is collected in a product drum.

The gas from the cold separator is sent to the amine and naphtha scrubbers. These scrubbers are manifolded so that they can be operated in either order. Typically, the amine scrubber precedes the naphtha scrubber. Each scrubber has its own regenerator. The volumes of the off-gas streams from

the regenerators are measured by McAfee gauges, and their compositions are measured by on-line chromatographs.

Make-up hydrogen is added to the scrubbed gas, and the combined stream is sent to the suction of the recycle compressor. Two compressors are available, one operated on line while the other is kept as a spare. Prior to addition of the make-up gas, a bleed-off stream is removed to maintain the desired hydrogen concentration in the recycle gas. The quantity of bleed-off gas is measured by a gas meter, and its composition is determined by an on-line chromatograph.

4. Fractionation System

From the let-down system, the slurry flows to a small surge vessel. Then, it goes through a preheater to the atmospheric flash column. Overhead from the flash column goes through a preheater to the fractionator. The fractionator is packed with Goodloe packing to achieve good separation efficiency. The overhead from the fractionator goes to the debutanizer. In the debutanizer, C_4 and lighter gases are taken overhead, and C_5 and heavier come off the bottom. Valving is available so that the liquid hydrocarbon stream from the intermediate and cold separators can be sent to either the atmospheric flash column or the fractionator. Normally this stream goes to the fractionator. The volume of debutanizer overhead gas is measured by a gas meter, and its composition is determined by on-line chromatograph.

The bottoms from the flash column goes to the "recycle splitter." The splitter is a vessel fitted with a vertical plate which divides the bottom portion into two chambers. There is a flapper just below the inlet to the vessel which directs the flow to one or the other of the bottom chambers. The fraction of time that the flapper directs flow to recycle is controlled by a timer which can be set anywhere between 0 and 100%. The liquid from one of the bottom chambers is sent to Tanks 4 and 5 for recycle to the reactor, and the liquid from the other chamber is sent to the vacuum flash column. Thus, the percentage of the reactor effluent slurry that is recycled can be set to any desired value by adjusting the timer.

In the vacuum flash column, solvent boiling range material is taken overhead, and SRC, undissolved coal, and ash are removed from the bottom. The bottoms from the vacuum column goes to a receiver which is manually drained very hour. The fractionator bottoms and vacuum column overhead are combined into a stream referred to as "process solvent" and sent to Tanks 8 and 9. Tanks 8 and 9 operate in a manner similar to Tanks 4 and 5; that is, one tank collects product while recycle solvent is being obtained from the other tank. At the end of each hour, the tank from which recycle process solvent was being obtained is pumped down to a fixed level. The material removed from

the tank, which, in effect, represents net process solvent production for that hour, is sent to Tank 11. Tank 11 is drained at the beginning and end of each material balance period to obtain the weight of process solvent produced during the period.

5. Data Acquisition

P99 is tied into an IBM 1800 computer which logs the data and prints it out hourly on a typewriter located in the control room. All pertinent temperatures, pressures, flow rates, and gas compositions are logged. In calculating gas yields, gas stream volumes are reduced by their nitrogen content before being printed. Gas compositions from the chromatograph are normalized to 100% on a nitrogen-free basis before being printed out.

In addition to printing out an hourly log, the 1800 computer also punches a series of IBM cards each hour. The data punched on these cards consist of reactor temperatures, reactor pressure, rates of the various feed components, rates of all product streams, and gas compositions.

During the course of a run, selected periods of stable operation are chosen for further data work-up. All of the debutanizer bottoms, water, process solvent, and vacuum bottoms produced during one of these material balance periods are collected, and samples of these streams are submitted for elemental analyses. These data, plus the data cards from the 1800 computer, are used to calculate normalized and elementally balanced yields for the period. This is done by means of an off-line data reduction computer program. This program also prints out tables of gas stream compositions and feed and product properties.

The data reduction program first calculates a yield structure based only on the flow rate data from the unit and the waste water analysis. Then an elementally balanced yield structure is calculated which uses the elemental analyses of the coal and the product streams in addition to the flow rate data. Finally, a yield structure is calculated which provides estimated distillate yields for particular boiling ranges. For data analysis purposes, the elementally balanced, distillation-adjusted yields are generally used since these are believed to be the most representative of what would be achieved in a commercial unit.

C. P99 Activity During the Second Quarter 1978

P99 was down the full month of April for unit maintenance and modification. It came back up early in May with the feed being Pittsburgh seam coal from Powhatan No. 5 mine. Three runs were completed on this coal (Runs 35, 36 and 37). At the beginning of

June, feed coal was changed to Pittsburgh seam coal from the Valley Camp Mine. Two runs (Runs 38 and 39) were made on this coal. A leaking valve in the high pressure system forced a premature end to Run 39 on June 15, and P99 then remained down for the rest of June.

Table C-1 gives the analyses of the Powhatan and Valley Camp coals; Table 24 presents a summary of the operating conditions for Run 35 through 39; and Table 25 presents a summary of the elementally-balanced, distillation-adjusted yields for these runs.

TABLE 24

NOMINAL OPERATING CONDITIONS

Run No. P99-	35	36	37	38	39
Coal	<------Powhatan------>			Valley Camp	
Average Dissolver Temp., °F	851	851	851	851	851
Dissolver Pressure, PSIG	2000	2000	2000	2000	2000
WHSV, lb as received coal per ft^3 dissolver vol per hr	22.5	22.5	22.5	22.5	22.5
Nominal slurry residence time, hr	1.0	1.0	1.0	1.0	1.0
Coal in Feed Slurry, wt.%	30	30	30	30	30
Dissolver Inlet Gas					
10^3 SCF/T feed coal	70.5	48.6	49.5	49.8	49.0
H_2 content, Vol.%	85	95	85	93	90

TABLE 25

SUMMARY OF SRC II YIELDS

Run No. P99-	35	36	37	38	39
Yield, Wt.% of Moisture-Free Coal					
Hydrogen	-4.3	-4.3	-4.3	-4.2	-4.0
Methane	5.9	5.6	5.8	5.8	5.6
Ethane	4.4	4.4	4.4	4.4	4.2
Propane	4.2	4.2	4.1	4.1	4.0
I-Butane	0.2	0.3	0.3	0.3	0.2
N-Butane	2.0	2.0	1.9	2.1	2.0
Total C_1 to C_4	16.7	16.5	16.5	16.7	16.0
Ammonia	0.4	0.4	0.4	0.5	0.5
Hydrogen Sulfide	2.3	2.3	2.3	1.9	1.8
Carbon Dioxide	1.1	1.1	1.2	1.0	1.0
Carbon Monoxide	0.2	0.2	0.2	0.1	0.2
Total Other Gases	4.0	4.0	4.1	3.5	3.5
Water	5.9	5.9	6.3	5.3	5.7
C_5-380°F	9.4	10.2	10.3	9.4	9.1
380-550°F	14.1	14.4	13.0	16.0	14.2
550-900°F	14.0	13.5	11.5	11.0	10.6
Total C_5 -900°F Distillate	37.5	38.1	34.8	36.4	33.9
900°F+ Pyridine Solubles	25.3	25.0	26.9	25.8	28.9
Insoluble Organic Matter	5.4	5.3	6.2	7.6	7.1
Ash	9.5	9.5	9.5	8.9	8.9
Total 900°F+ Product	40.2	39.8	42.6	42.3	44.9

REFERENCES

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2. Rosen, Stephen L., Fundamental Principles of Polymeric Materials for Practicing Engineers, Barnes & Noble, Inc., New York, NY, 1971. Chapter 6, pp. 52-54.

3. Solvent Refined Coal (SRC) Process, Quarterly Technical Progress Report for the Period January 1977-March 1977, The Pittsburg & Midway Coal Mining Co., July 1977, FE/496-134.

4. Solvent Refined Coal (SRC) Process, Quarterly Technical Progress Report for the Period January 1978-March 1978, The Pittsburg & Midway Coal Mining Co., February 1979, FE/496-155.

APPENDIX A

TABLE A-1

CORROSION RACK SUMMARY

RACK NO.	LOCATION	TEMPERATURE °F	MATERIAL	INSTALLED TIME, DAYS	OPERATING TIME, DAYS	CORROSION RATE MILS/YR INSTALLED	CORROSION RATE MILS/YR OPERATING
212	Dissolver A Top	800-860	2 1/4 Cr-1 Mo	370	285	24.2	31.4
			9 Cr-1 Mo			12.3	16.0
			Type 410			2.6	3.4
			Type 321			3.2	4.2
			Incoloy 800			3.4	4.4
211	Dissolver A Bottom	800-860	2-1/4 Cr-1 Mo	370	285	35.4	46.0
			9 Cr-1 Mo			13.0	16.9
			Type 410			4.2	5.4
			Type 321			4.9	6.3
			Incoloy 800			5.9	7.7
233	High Pressure Flash Drum, Top	630-700	2-1/4 Cr-1 Mo	202	163	25.3	31.3
			9 Cr-1 Mo			2.7	3.4
			Type 410			2.4	3.0
			Type 321			0.3	0.4
			Incoloy 800			0.2	0.2
234	High Pressure Flash Drum, Bottom	630-700	2-1/4 Cr-1 Mo	202	163	12.3	15.2
			9 Cr-1 Mo			1.6	2.0
			Type 410			0.2	0.2
			Type 321			0.2	0.2
			Incoloy 800			0.3	0.4
235	Intermediate Pressure Flash Drum, Top	630-700	1-1/4 Cr-1/2 Mo	202	160	14.6	18.5
			Type 405			2.1	2.6
			Type 321			0.2	0.2
			Incoloy 800			0.2	0.3
			1 Cr-1/2 Mo			18.1	22.9

TABLE A-1 (Cont'd)

CORROSION RACK SUMMARY

RACK NO.	LOCATION	TEMPERATURE °F	MATERIAL	INSTALLED TIME, DAYS	OPERATING TIME, DAYS	CORROSION RATE MILS/YR INSTALLED	CORROSION RATE MILS/YR OPERATING
236	Intermediate Pressure Flash Drum, Bottom	630-700	1-1/4 Cr-1/2 Mo	202	160	9.4	11.9
			1 Cr-1/2 Mo			8.6	10.9
			Monel			111.1	140.3
			Type 321			0.1	0.1
237	Recycle Condensate Separator, Top	60-90	Carbon Steel	199	199	0.4	0.4
			Type 405			0.1	0.1
			Type 304			0.0	0.0
			Monel			0.2	0.2
238	Recycle Condensate Separator, Bottom	60-90	Carbon Steel	199	199	0.4	0.4
			Type 405			0.1	0.1
			Type 304			0.0	0.0
			Monel			0.2	0.2
206	Stripper Column, Top	360-480	Carbon Steel	370	271	5.0	6.8
			Type 405			0.5	0.7
			Type 316			0.1	0.1
			Type 321			0.1	0.1
			Incoloy 800			0.0	0.0
			26 Cr-1 Mo			0.0	0.0
209	Filter Feed Flash Vessel	540-600	Carbon Steel	370	224	11.3	18.6
			Type 405			0.1	0.1
			Type 316			0.1	0.1
			Type 321			0.1	0.1
			Incoloy 800			0.0	0.0
			26 Cr-1 Mo			0.0	0.0

TABLE A-1 (Cont'd)

CORROSION RACK SUMMARY

RACK NO.	LOCATION	TEMPERATURE °F	MATERIAL	INSTALLED TIME, DAYS	OPERATING TIME, DAYS	CORROSION RATE MILS/YR INSTALLED	CORROSION RATE MILS/YR OPERATING
227	Vacuum Flash Drum Column	80-160	Carbon Steel	365	365	0.2	0.2
			Type 405			0.0	0.0
			Type 304			0.0	0.0
			Type 316L			0.0	0.0
			Type 317			0.0	0.0
			Incoloy 800			0.0	0.0
229	Vacuum Flash Drum, Old	560-630	Carbon Steel	370	145	6.3	16.0
			Type 405			0.0	0.1
			Type 304			0.0	0.0
			Type 316L			0.0	0.0
			Type 317			0.0	0.0
			Incoloy 800			0.0	0.0
249	Vacuum Flash Drum, New	560-630	Carbon Steel	203	118	2.9	5.0
			Type 410			0.9	1.6
			Type 304			0.1	0.1
			Type 316L			0.0	0.0
			Type 317			0.0	0.0
			Incoloy 800			0.1	0.1
243	Wash Solvent Column, Top	420-480	Carbon Steel	180	98	42.9	78.8
			Type 405			28.8	52.8
			Type 304			5.9	10.8
			Type 316L			9.8	18.0
			Type 317			11.2	20.6
			Incoloy 800			7.6	13.9
			Incoloy 825			0.9	1.7
			Hastelloy C			0.0	0.0
			Hastelloy G			0.0	0.0

TABLE A-1 (Cont'd)

CORROSION RACK SUMMARY

RACK NO.	LOCATION	TEMPERATURE °F	MATERIAL	INSTALLED TIME, DAYS	OPERATING TIME, DAYS	CORROSION RATE MILS/YR INSTALLED	CORROSION RATE MILS/YR OPERATING
244	Wash Solvent Column, Middle	420-480	Carbon Steel	180	98	29.5	54.2
			Type 405			16.1	29.6
			Type 304			7.8	14.4
			Type 316L			4.6	8.4
			Type 317			3.5	6.5
			Incoloy 800			26.0	47.8
			Incoloy 825			1.0	1.8
			Hastelloy C			0.0	0.0
			Hastelloy G			0.1	0.2
225	Wash Solvent Column, Reboiler Wall	600-700	Carbon Steel	370	193	11.5	22.0
			Type 304			4.3	8.2
			Type 316L			0.8	1.6
			Type 317			0.1	0.2
			Incoloy 800			0.2	0.3
			Hastelloy C			0.0	0.0
239	Light Ends Column Top	200-400	Carbon Steel	171	113	1.1	1.7
			Type 405			0.2	0.3
			Type 304			0.0	0.0
			Type 316L			0.0	0.0
			Type 317			0.0	0.0
			Incoloy 800			0.0	0.0
			Incoloy 825			0.0	0.0
			Hastelloy G			0.0	0.0

TABLE A-1 (Cont'd)

CORROSION RACK SUMMARY

RACK NO.	LOCATION	TEMPERATURE °F	MATERIAL	INSTALLED TIME, DAYS	OPERATING TIME, DAYS	CORROSION RATE MILS/YR INSTALLED	CORROSION RATE MILS/YR OPERATING
220	Light End Column, Middle	200-400	Carbon Steel	370	235	4.3	6.7
			Type 405			0.0	0.1
			Type 304			0.0	0.0
			Type 316L			0.0	0.0
			Type 317			0.0	0.0
			Incoloy 800			0.0	0.0
221	Light Ends Column, Reboiler	400-600	Carbon Steel	370	199	23.3	43.3
			Type 405			15.3	28.4
			Type 304			1.1	2.0
			Type 316L			0.3	0.5
			Type 317			0.1	0.1
			Incoloy 800			0.2	0.4
222	Light End Column, Reboiler Bundle	400-600	Carbon Steel	370	199	16.2	30.2
			Type 405			7.8	14.5
			Type 304			0.3	0.6
			Type 316L			0.1	0.2
			Type 317			0.0	0.0
			Incoloy 800			0.0	0.1

TABLE A-2

OPERATING DATA FOR "C" FILTER HYDRAULIC TESTS

	Test No. 1	Test No. 2	Test No. 3
Date:	0600,6/7/78- 0500,6/8/78	1700,6/8/78- 1800,6/9/78	1900,6/15/78- 0500,6/17/78
Test Temperature (°F, TE 3033)	488	465	490
Differential Pressure (psi, PDT 3022)	40	95	40 & 95
Precoat Type	Std. Supercel	Std. Supercel	512
Kinematic Viscosity (cSt @ 100°F)	15.6	19.0	--
Specific Gravity (60°F)	1.077	1.082	1.106
Estimated Viscosity at Test Temperature (cp)	0.37	0.46	--
Feed Insolubles (wt%)	0.02	0.06	0.09
Filtrate Insolubles (wt%)	Trace	Trace	Trace

TABLE A-3

SUMMARY OF LIQUID YIELD ERROR ANALYSIS
Ft. Lewis Material Balance Run 78SR-20

Error Contribution by Vessel, % of Total Accumulation

Vessel	Naphtha	Middle Distillate	Heavy Distillate	Total Distillate
SBT	0.52	0.53	0.52	0.40
HPF	0.05	0.05	0.05	0.00
IPF	0.03	0.03	0.03	0.01
RCS	0.01	0.01	0.01	0.00
STRPR	0.00	0.00	0.00	0.01
St.Ref.	0.01	0.01	0.01	0.00
W.S.A.	92.71	92.66	91.92	95.80
P.S.A.	0.00	0.00	0.00	0.00
F.S.A.	0.02	0.02	0.01	0.02
S.F.A.	0.00	0.02	0.01	0.02
V.F.C.D.	4.27	4.31	4.29	0.26
R.P.W.T.	0.19	0.11	0.08	0.19
Hotwell	0.00	0.00	0.00	0.00
S.B.C.T.	0.00	0.01	0.00	0.01
Widing	2.19	2.24	2.19	1.87
TOTAL	100.00	100.00	100.00	100.00
Error (lbs. for run)	2113	7155	8077	6340
Total Recovery lbs.	10494	32534	15715	58743
% Error(Based on Total Recovery)	20.1%	22.0%	51.4%	10.8%

TABLE A-4

COMPARISON OF FT. LEWIS SRC II LIQUID FUEL WITH PETROLEUM-DERIVED FUEL

Test	SRC II Liquid Fuel (5:1 MD:HD Volumetric Blend) Not Filtered	Filtered Thru 100μ	Filtered Thru 25μ	Filtered Thru 5μ	Petroleum Derived Fuel[1]
Sediment (by Extraction-Toluene), Wt. Pct.	0.14	0.05	0.06	0.05	No Spec
Sediment (by Extraction-Pyridine), Wt. Pct.	0.08	0.04	0.08	0.08	No Spec
Water & Sediment (BS&W), Vol. Pct.	0.18	0.14	0.10	0.10	Max 2.0
Ash (ASTM D-482), Wt. Pct.	0.015	0.016	0.003	0.005	No Spec.
Sulfur (GRM 1156), Wt. Pct.	0.28	0.17	0.21	0.21	0.84[2]
Viscosity, SFS @ 122°F	6.5[3]	6.5[3]	6.5[3]	6.5[3]	45-300
Heat of Combustion, FOE/Barrel[4]	0.95	0.96	0.95	0.95	1.0[2]
Flash Point, °F	165	168	160	168	150 Min.
Gravity, °API	10.6	10.5	10.5	10.5	12.6[2]
Compatability with Bunker C (Spot Test)	1	1	1	1	No Spec.
Pour Point, °F	-60.	-60.	-60.	-52.	Approx 75[2]
Conradson Carbon Residue, Wt. Pct.	0.34	0.36	0.34	0.34	No Spec.
Carbon/Hydrogen Weight Ratio	10.0	10.0	10.0	10.0	8.3[2]
Nitrogen, Wt. Pct.	0.87	0.90	1.04	0.94	0.28[2]

[1] A.S.T.M. No. 6 Fuel Specification D-396.

[2] No Specification - "Typical Value" (reference: Chemical Engineers Handbook, Perry, 5th Ed. Section 9).

[3] Calculated from Measured Viscosity of 4.3 cs @ 100°F.

[4] 1 FOE (Fuel Oil Equivalent Barrel) = 6.25 x 10^6 BTU.

TABLE A-5

COMPARISON OF TYPICAL ASH COMPOSITION

Component	SRC II Oil, PPM in Oil	No. 6 Fuel, PPM in Oil
Platinum	0.04	ND
Zirconium	0.15	ND
Vanadium	0.55	15.
Iron	77.	12.
Nickel	0.28	4.6
Calcium	13	12
Magnesium	2.3	7.8
Sodium	3.7	12.
Silicon	99	15
Manganese	0.29	0.18
Aluminum	83	3.2
Barium	ND	1.0
Lead	0.14	<1.2
Tin	0.10	0.11
Molybdenum	ND	0.027
Copper	0.08	0.059
Silver	0.005	0.0034
Zinc	ND	0.54
Titanium	9.9	0.086
Cobalt	0.03	0.66
Potassium	9.4	Trace
Chromium	1.0	0.042
Strontium	0.10	0.082
Boron	0.88	ND
Phosphorous	8.3	ND

Source: KVB Co. Report 19900-733 (December, 1977)

TABLE A-6

ANALYSIS OF SAMPLES USED FOR HEAT CAPACITY STUDY

Sample No.	900	882	883
Sample Type	Pyridine Insolubles	SRC II Vac. Bottoms	SRC II Stripper Bottoms
% Ash	73.77	25.81	14.96
% Pyridine Insoluble	100	35*	29.92
% Carbon	18.90	--	--
% Hydrogen	1.04	--	--
% Nitrogen	0.30	--	--
% Sulfur	5.11	--	--
% Water	--	--	0.07
% Naphtha	--	--	0
% Middle Distillate	--	--	9.38
% Heavy Distillate	--	--	32.18
% Vacuum Bottoms	--	--	58.36
Fusion Point (°F)	--	283	--

* Estimated

TABLE A-7

CONDITIONS OF VISCOSITY MEASUREMENT RUNS

Date	Run No.	Coal Concentration, wt %	SRC, Wt %	PI Wt %	(°F)	Residence Time in blend tank (min.)
11/8/77	1	17.13	34.32	20.29	356	23
11/16/77	2	21.44	35.90	25.61	343	25
11/25/77	3	19.38	37.29	26.63	348	22
1/18/78	4	30.76	41.03	25.72	355	36
1/25/78	5	30.80	38.01	23.37	347	35
3/29/78	6	31.17	41.32	24.35	368	47
3/30/78	7	31.75	40.28	22.77	373	36

TABLE A-8

Actual Apparent Viscosity and Predicted Apparent Viscosity

RUN 1			RUN 2			RUN 3			RUN 4			RUN 5		
Shear Rate (sec^{-1})	Apparent Viscosity (cp) Actual	Expected	Shear Rate (sec^{-1})	Apparent Viscosity (cp) Actual	Expected	Shear Rate (sec^{-1})	Apparent Viscosity (cp) Actual	Expected	Shear Rate (sec^{-1})	Apparent Viscosity (cp) Actual	Expected	Shear Rate (sec^{-1})	Apparent Viscosity (cp) Actual	Expected
191	92	92	219	216	199	260	159	163	433	480	---	616	348	---
117	112	108	176	240	213	266	157	162	367	496	---	547	352	---
73	131	125	160	218	219	203	170	175	320	512	---	444	369	---
142	102	101	133	228	231	149	196	192	274	503	---	400	377	---
186	88	93	96	251	255	121	202	204	183	601	---	297	396	---
370	80	75	60	303	294	84	225	226	141	617	---	235	428	---
360	83	76	35	345	346	39	302	283	98	629	691	182	448	---
547	68	66	50	308	311	60	263	250	63	711	894	153	452	---
370	79	75	82	249	268	106	199	212	19	1410	1797	117	475	420
297	79	80	---	---	---	141	180	195	49	944	1035	84	534	497
172	95	95	---	---	---	196	168	177	66	765	870	37	795	768
195	89	92	---	---	---	238	158	167	128	630	---	20	1295	1041
						273	152	161	175	626	---	58	647	604
						336	146	151	216	593	---			
						327	140	152	250	617	---			
						263	151	162	320	562	---			
						183	163	180	342	586	---			
						101	178	215						
						79	197	230						
						59	207	251						
						112	177	208						
						157	169	189						
						194	163	177						

RUN 6		
Shear Rate (sec^{-1})	Apparent Viscosity (cp) Actual	Expected
25	4846	3768
16	6493	5116
6.5	12930	9486
8.0	10850	8200

RUN 7		
Shear Rate (sec^{-1})	Apparent Viscosity (cp) Actual	Expected
92	1996	2317
119	1798	1923
34	3988	4768
11	9664	10804
68	2891	2885
52	3237	3504

TABLE A-9

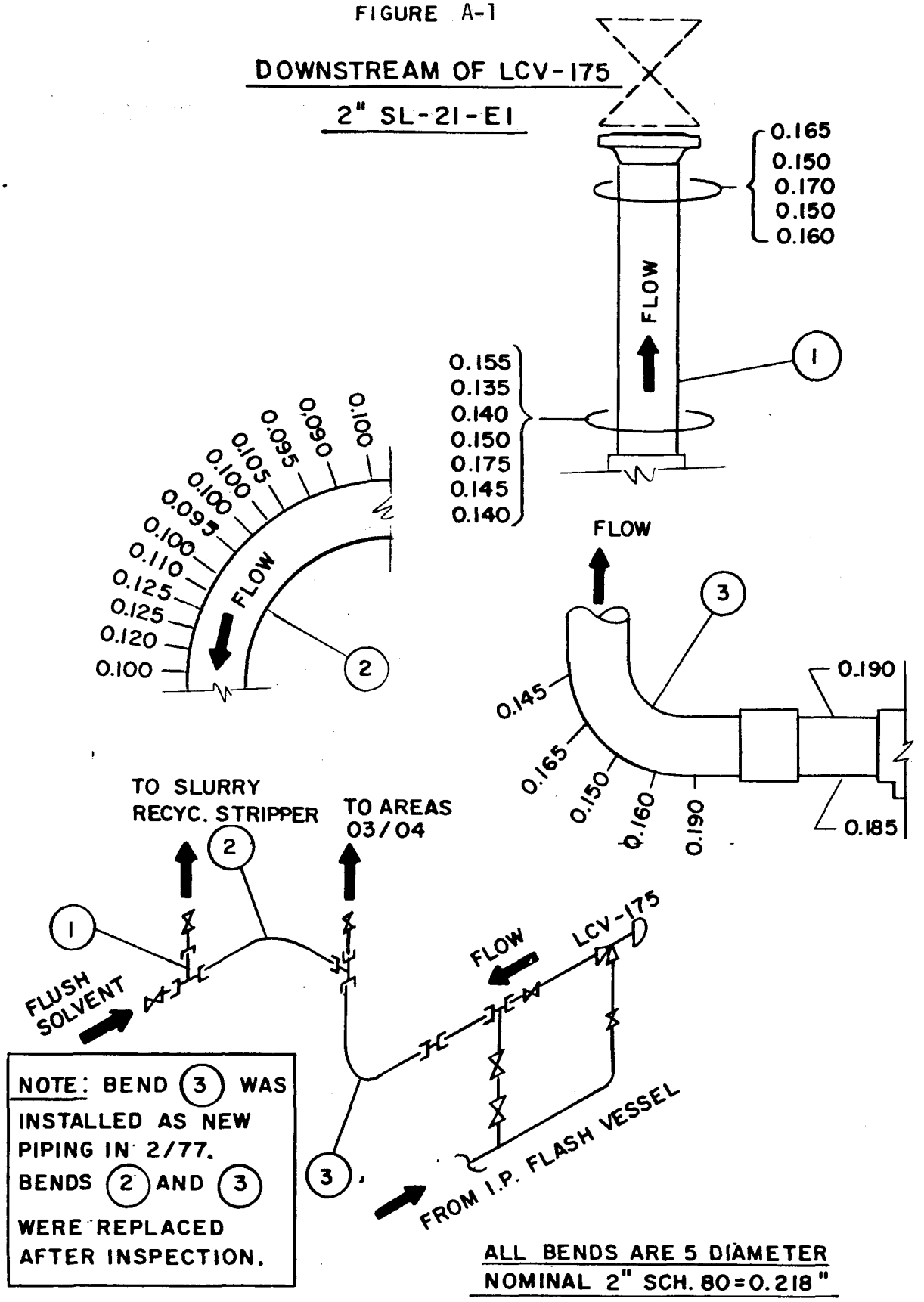
COMPUTER SYSTEM RELIABILITY FOR
April 1 to June 30, 1978

EQUIPMENT TYPE	% AVAILABILITY
INPUT DEVICES:	
A.) Operators Console CRT	100
B.) Tape Reader	100
C.) Decwriters (Avg. 3)	98.4
D.) Magnetic Tape	100
E.) Process/computer Interface	99.9
OUTPUT DEVICES	
A.) Tape Punch	100
B.) Decwriters (Avg. 3)	98.4
C.) Line Printer	100
D.) Magnetic Tape	100
E.) Operators Console CRT	100
CENTRAL PROCESSOR	99.1
OVERALL SYSTEM	99.0

FIGURE A-1

DOWNSTREAM OF LCV-175

2" SL-21-E1

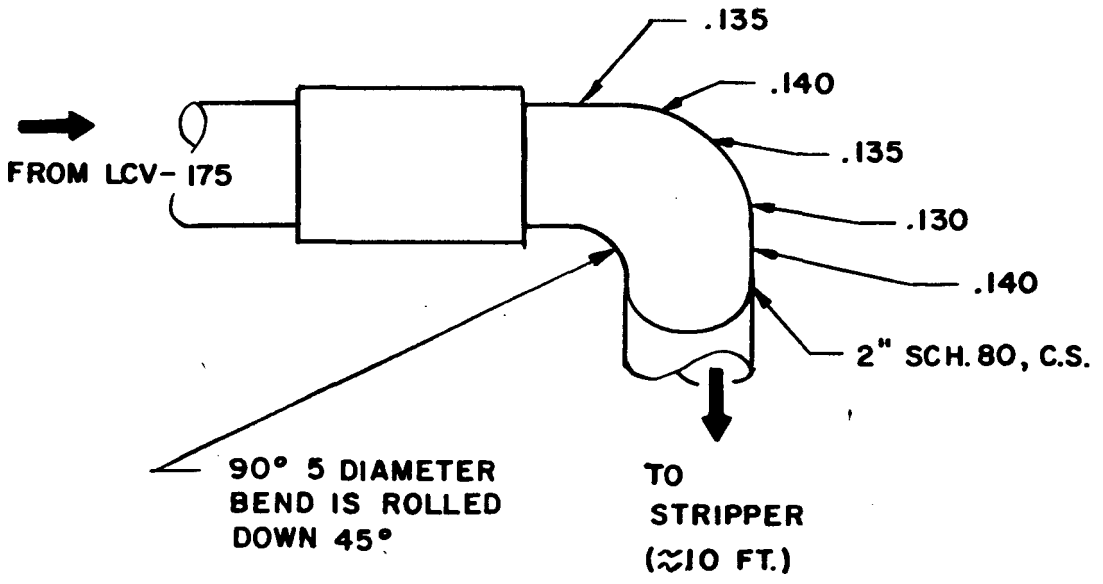


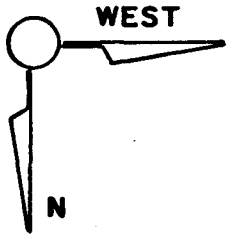
NOTE: BEND (3) WAS INSTALLED AS NEW PIPING IN 2/77. BENDS (2) AND (3) WERE REPLACED AFTER INSPECTION.

ALL BENDS ARE 5 DIAMETER
NOMINAL 2" SCH. 80=0.218"

FIGURE A-2

2" SL-118-E1 AT SLURRY RECYCLE STRIPPER

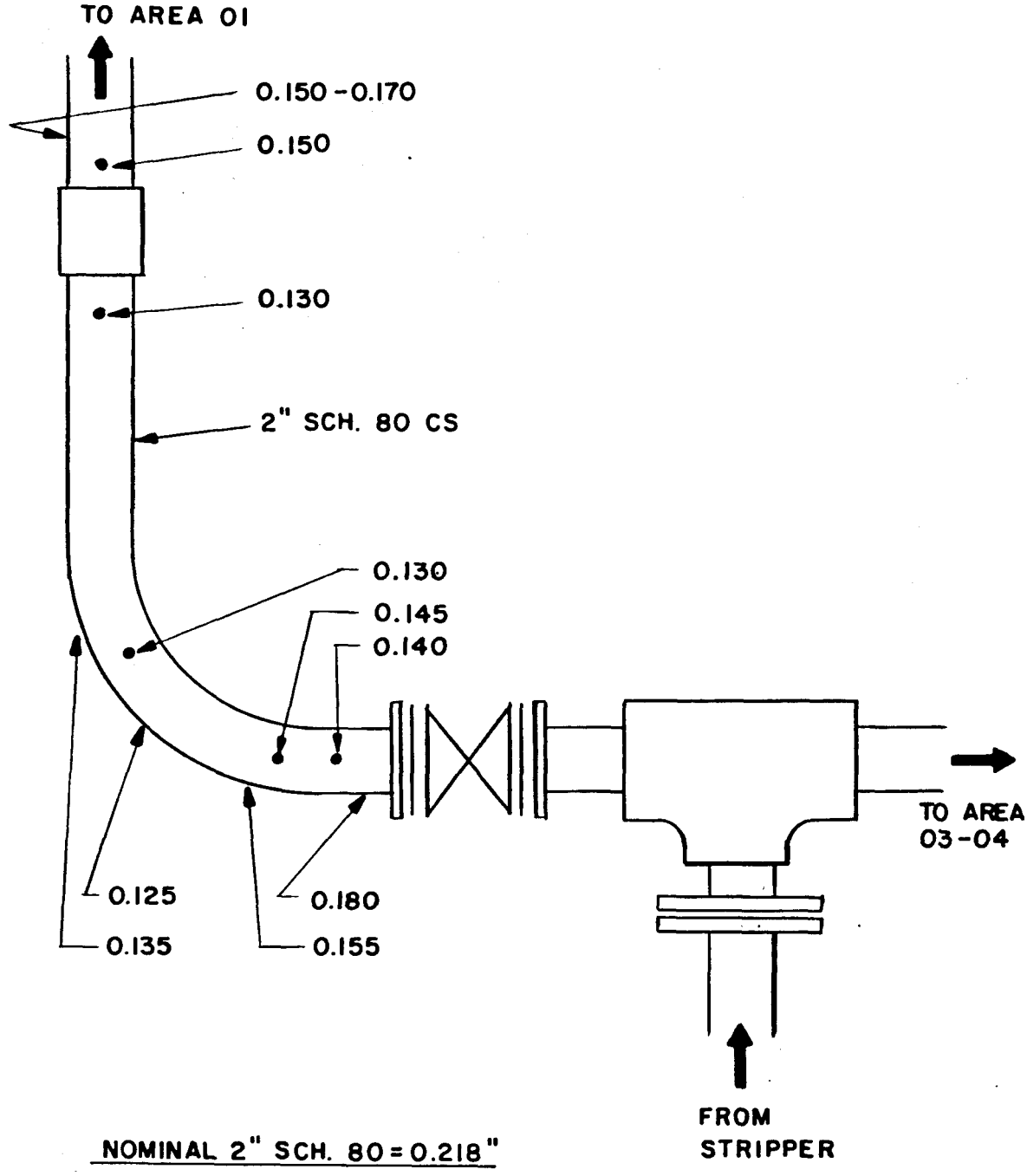




NOMINAL 2" SCH. 80 = 0.218"

FIGURE A-3

2" SL-114-E1

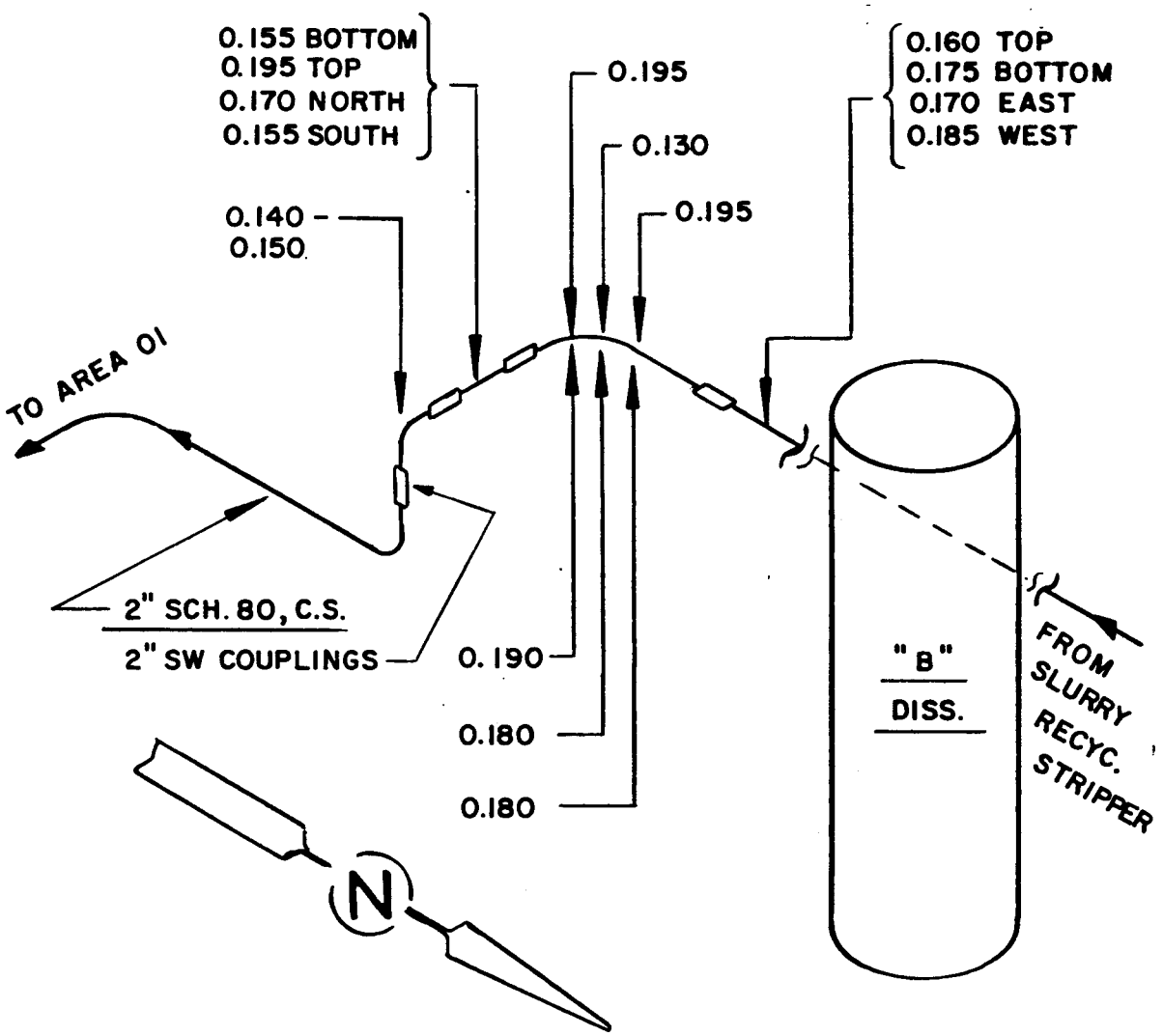


NOMINAL 2" SCH. 80 = 0.218"

NOTE: THIS 5 DIAMETER BEND WAS REPLACED.

FIGURE A-4

2" SL-114-E1 ON PIPERACK



NOMINAL 2", SCH. 80 = 0.218"

ALL BENDS ARE 5 DIAMETER

FIGURE A-5

2" SL-114 E1 AT FT 1152

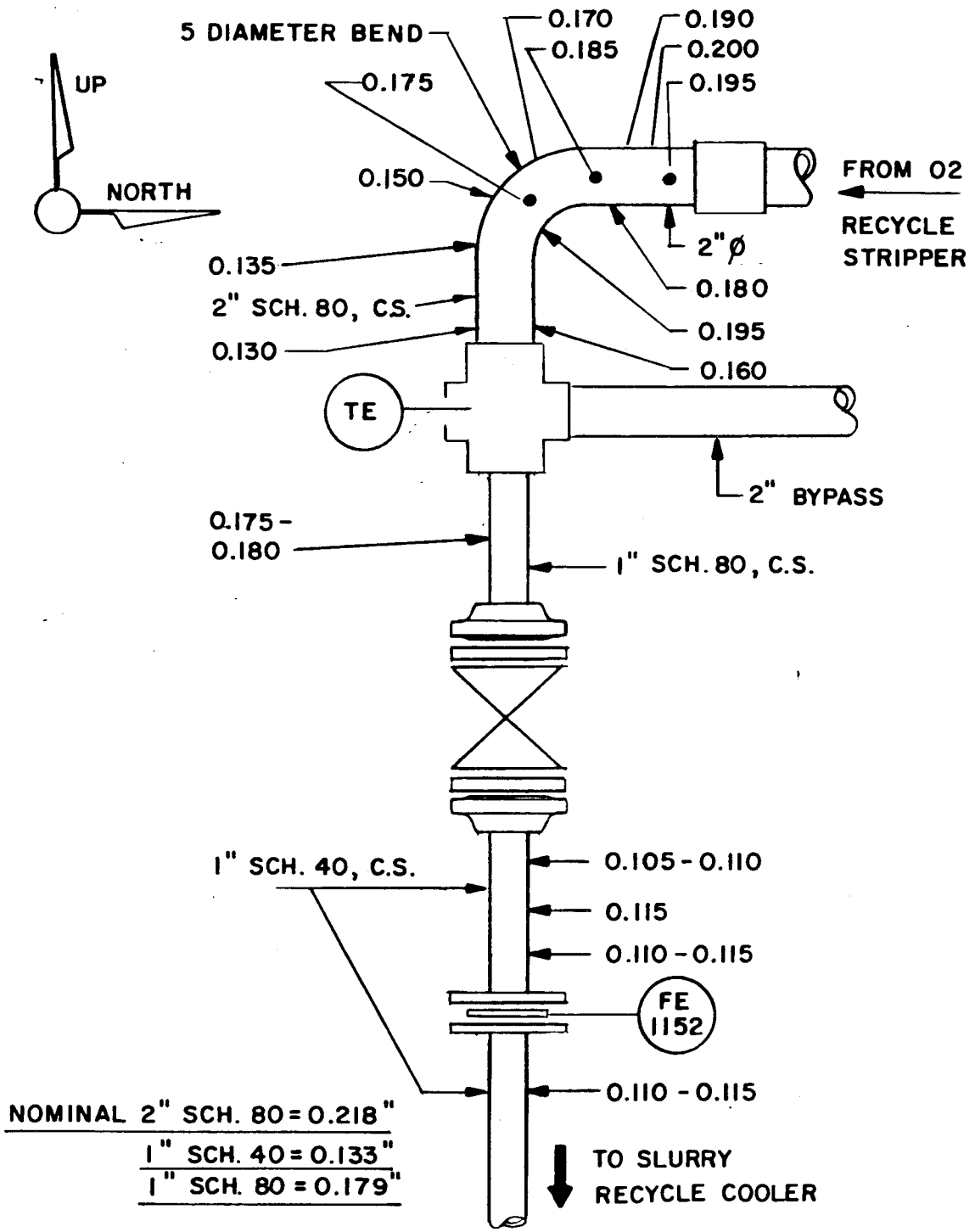
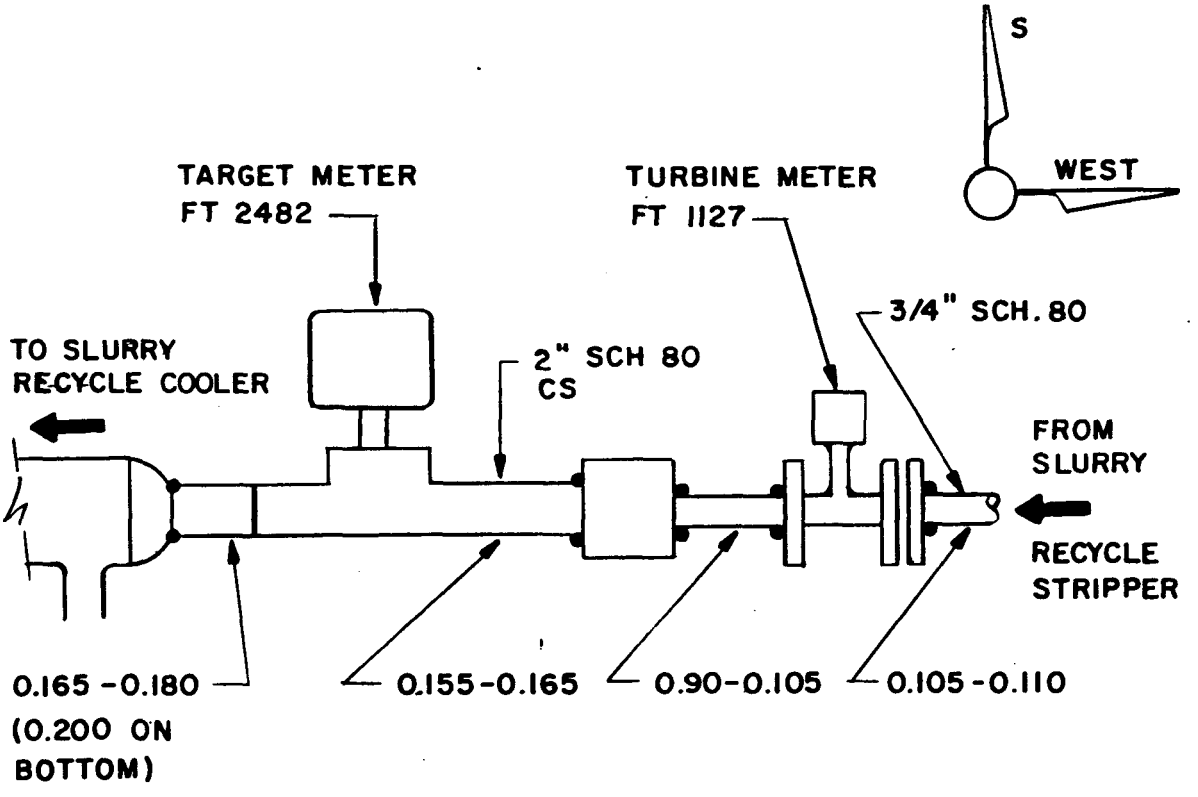


FIGURE A-6

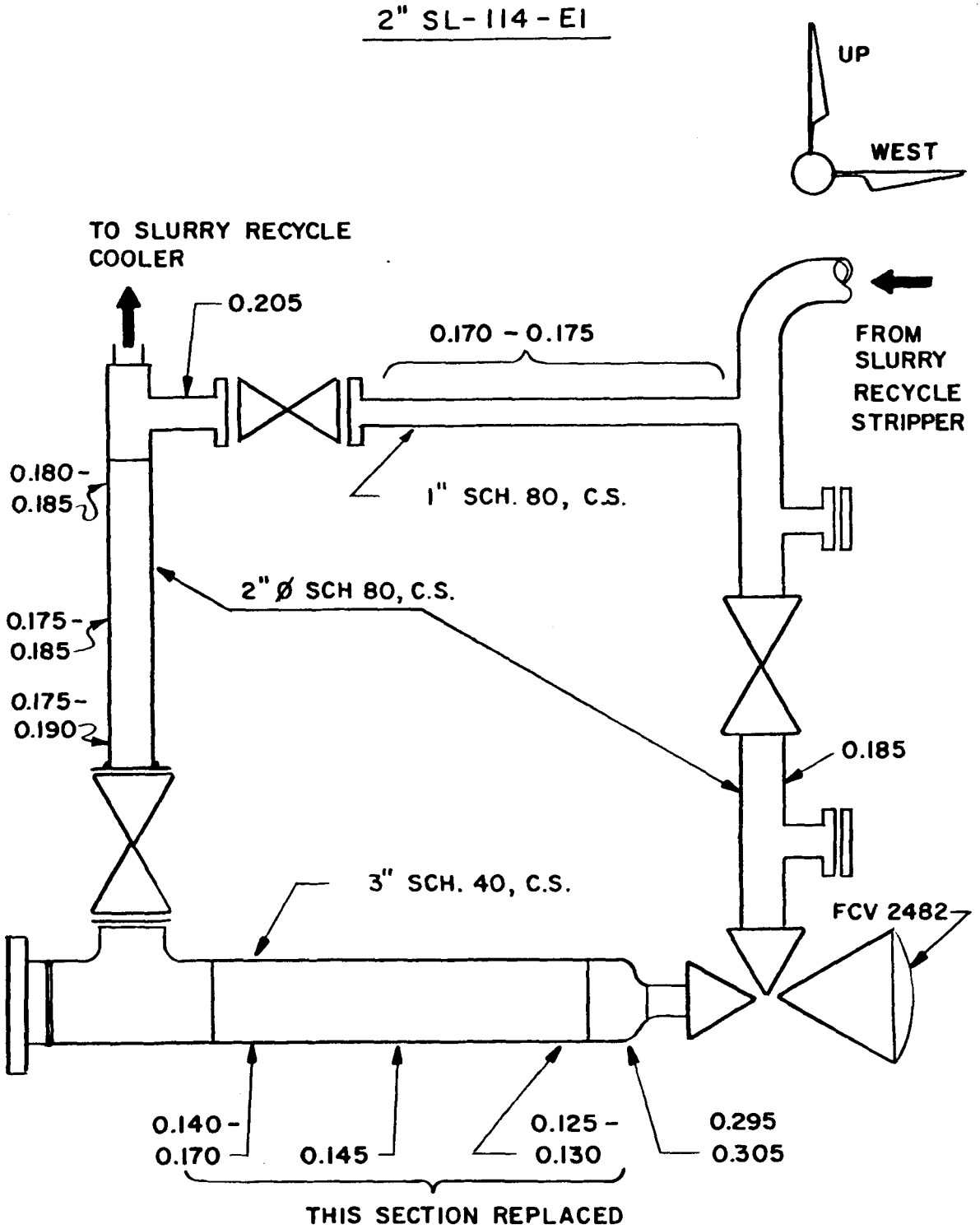
2" SL-114-E1 AT FT-2482



NOTE: TURBINE 1127 AND 3/4" PIPING WAS REMOVED AND REPLACED WITH A 1" SCH. 80 CS EROSION TEST SPOOLPIECE.

NOMINAL 2" SCH 80 = 0.218"
3/4" SCH 80 = 0.154"

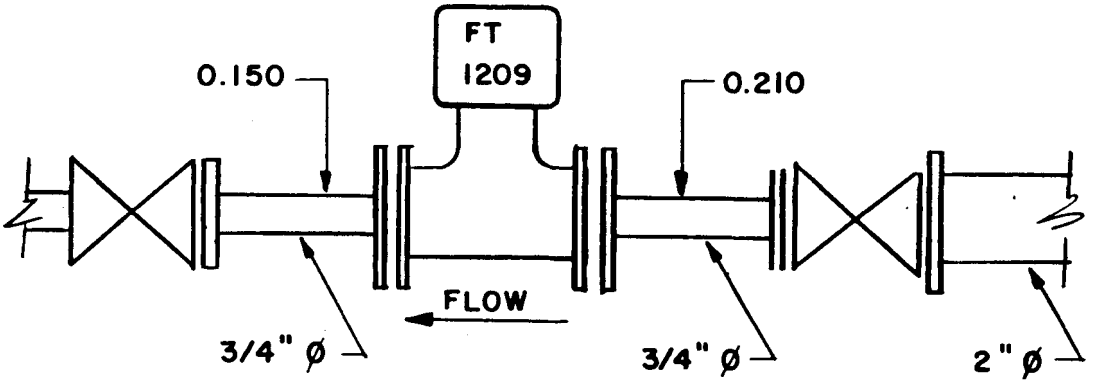
FIGURE A-7



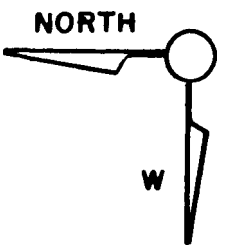
3" SCH 40 - NOMINAL 0.216"
2" SCH 80 - NOMINAL 0.218"
1" SCH 80 - NOMINAL 0.179"

FIGURE A-8

2" SL-133-E1 AT FT 1209



NOMINAL 3/4" SCH. 80 = 0.154"
3/4" SCH. 160 = 0.219"

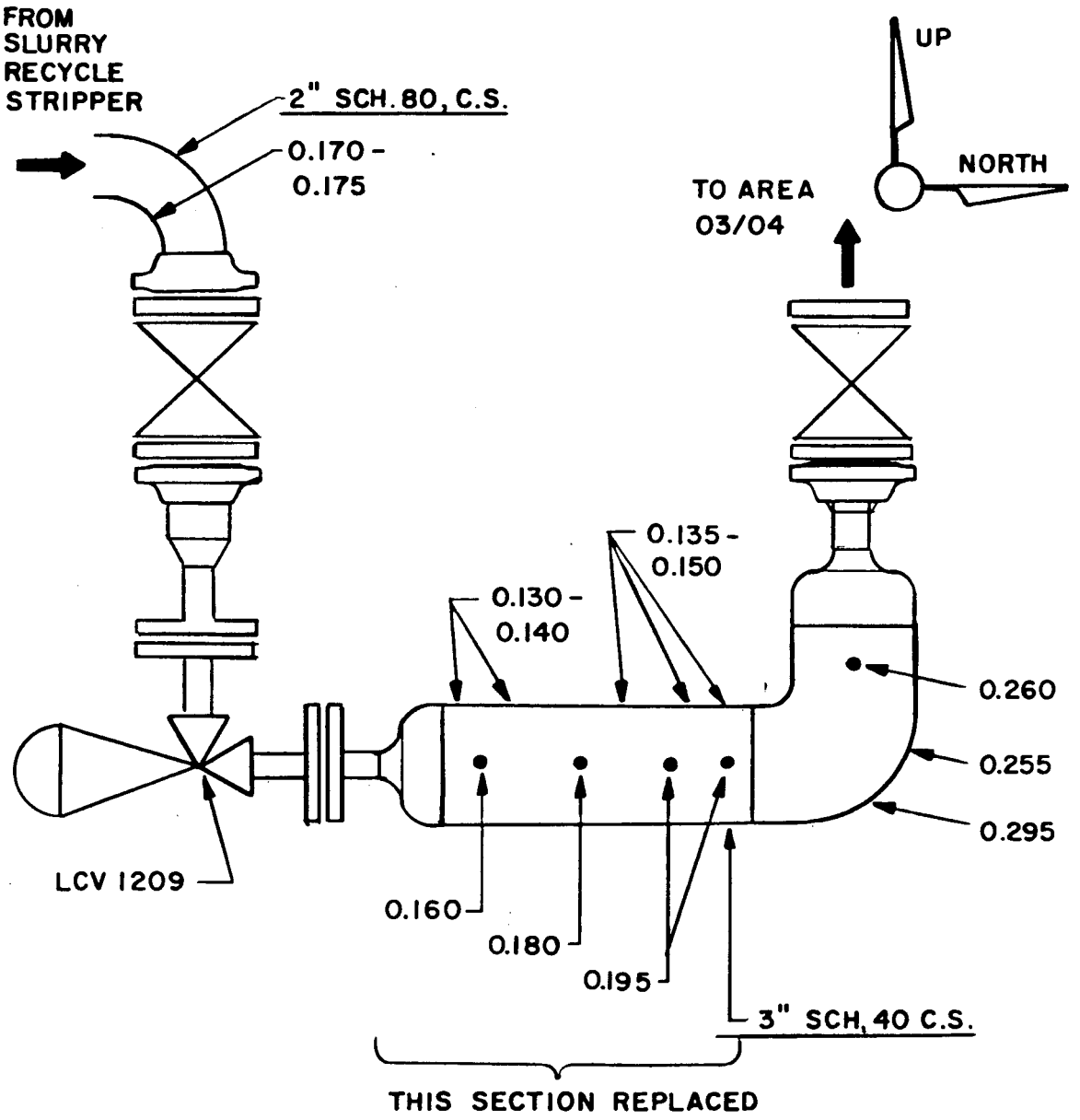


NOTE:

THIS METER WAS INOPERABLE IN THIS SERVICE. THE METER RUN WAS REMOVED AND REPLACED WITH STRAIGHT 2" SCH. 80 CS PIPE.

FIGURE A-9

2" SL-133-E1 AT LCV-1209

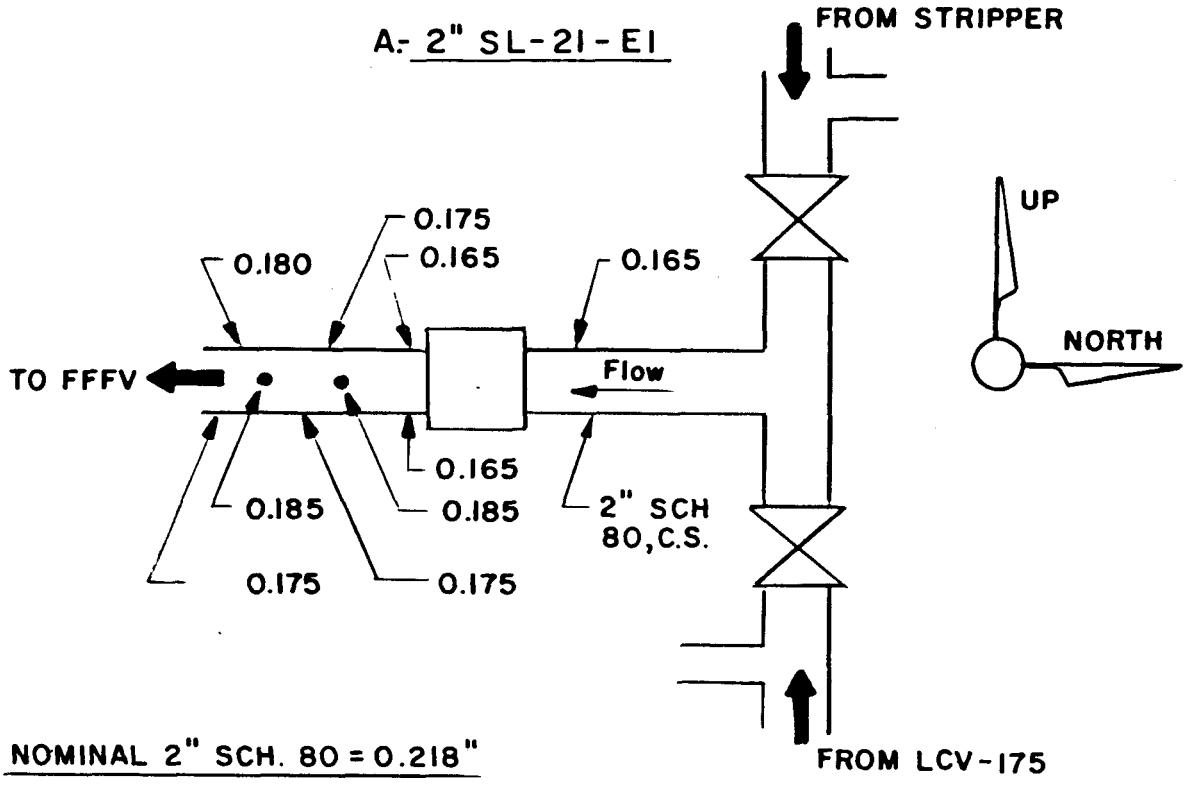


NOMINAL 2" SCH. 80 = 0.218"

3" SCH. 40 = 0.216"

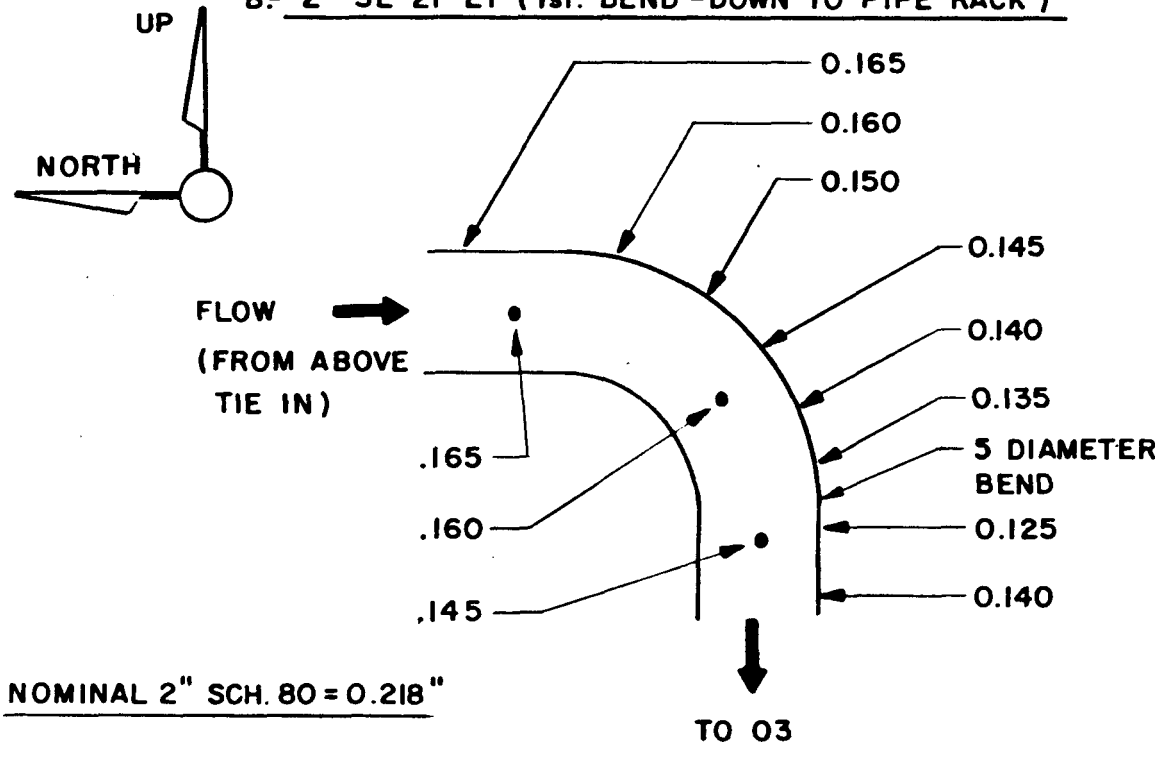
FIGURE A-10

A:- 2" SL-21-E1



NOMINAL 2" SCH. 80 = 0.218"

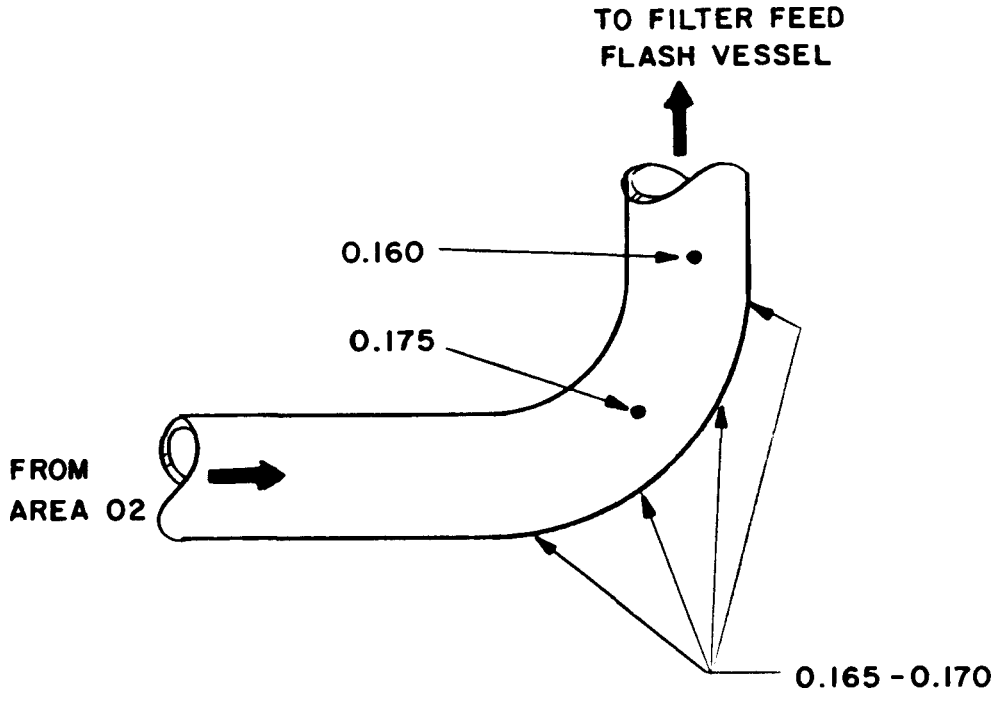
B:- 2" SL-21-E1 (1st. BEND -DOWN TO PIPE RACK)



NOMINAL 2" SCH. 80 = 0.218"

FIGURE A-11

2" SL-21-E1 AT LAST LONG RADIUS 5 DIAMETER BEND BEFORE FILTER FEED FLASH VESSEL



NOMINAL
2" SCH. 80 C.S.
= 0.218"

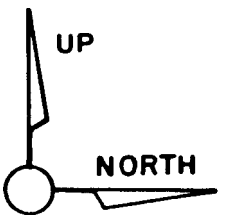


FIGURE A-12

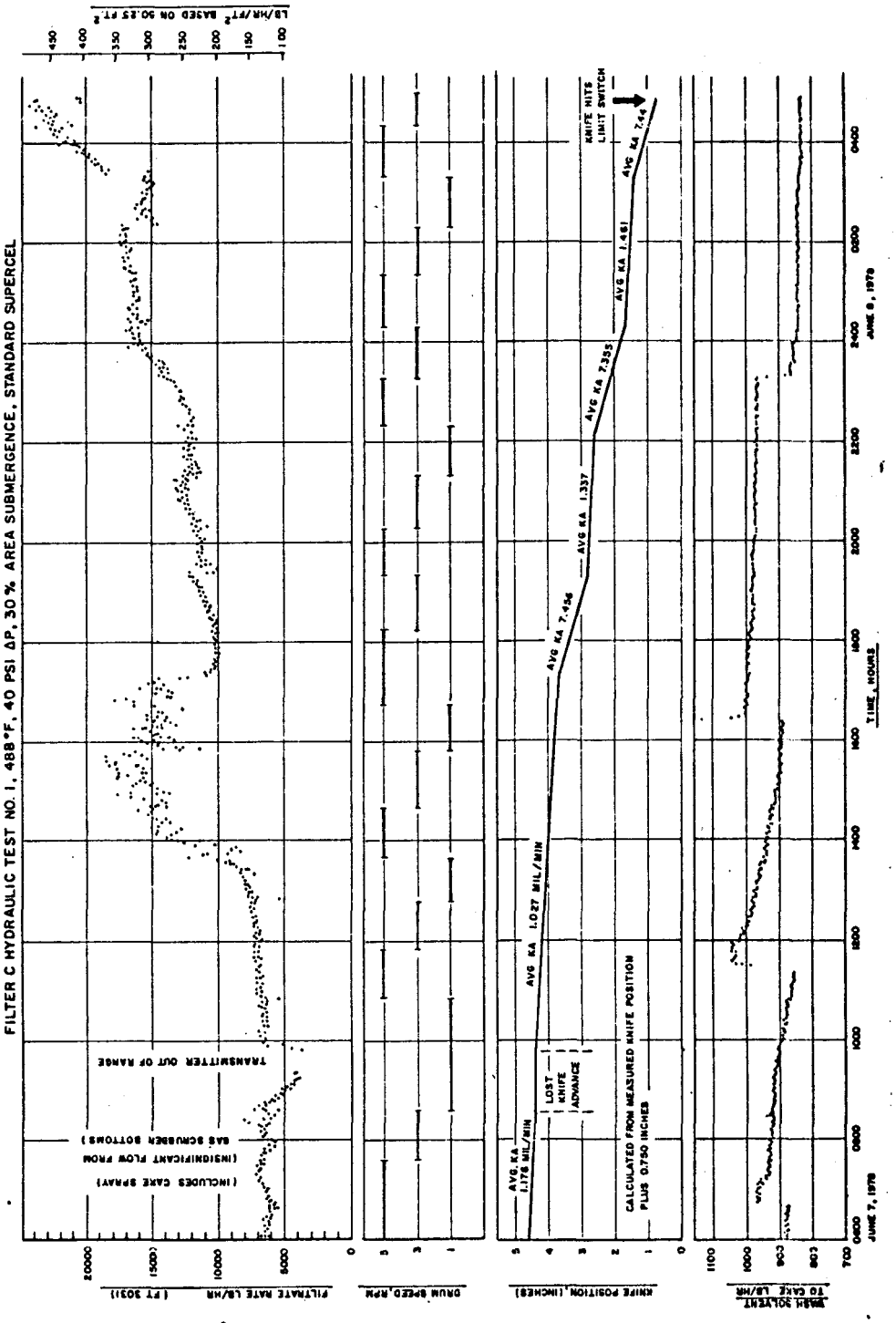

FILTER C HYDRAULIC TEST NO. 1, 488 °F, 40 PSI ΔP, 30 % AREA SUBMERGENCE, STANDARD SUPERCEL
LB/HR/FT² BASED ON 50.83 FT²
FILTRATE RATE LB/HR (FT 3031)
(INCLUDES CAKE SPRAY)
(INSIGNIFICANT FLOW FROM GAS SCRUBBER BOTTOMS)
TRANSMITTER OUT OF RANGE
DRUM SPEED, RPM
KNIFE POSITION, (INCHES)
AVG. KA 1.178 MIL/MIN
AVG KA 1.027 MIL/MIN
LOST KNIFE ADVANCE
AVG KA 1.434
AVG KA 1.337
AVG KA 1.355
AVG KA 1.481
AVG KA 7.44
KNIFE HITS LIMIT SWITCH
CALCULATED FROM MEASURED KNIFE POSITION PLUS 0.750 INCHES
WASH SOLVENT TO CAKE LB/HR
TIME, HOURS
JUNE 7, 1978
JUNE 8, 1978

FIGURE A-13

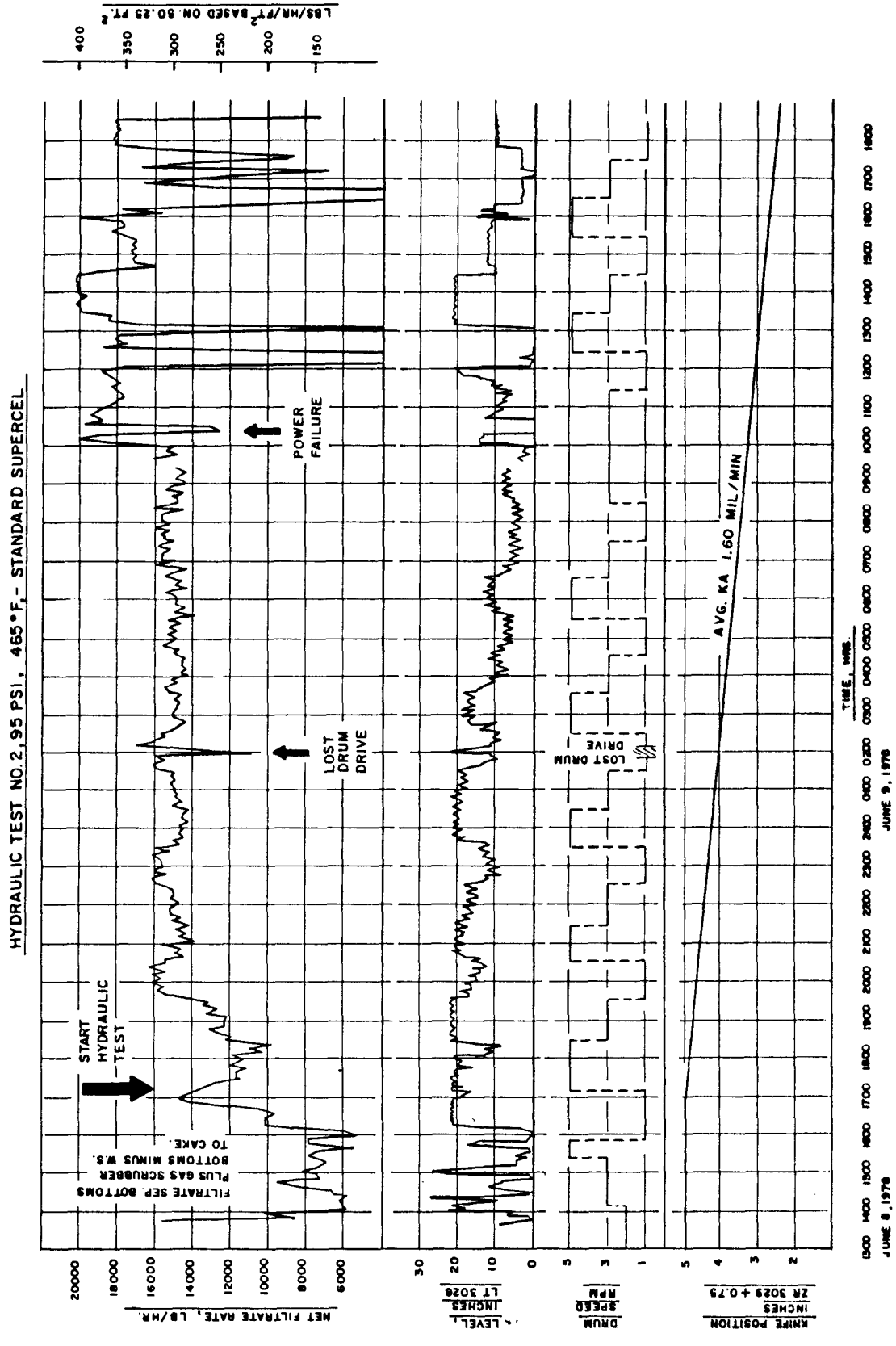
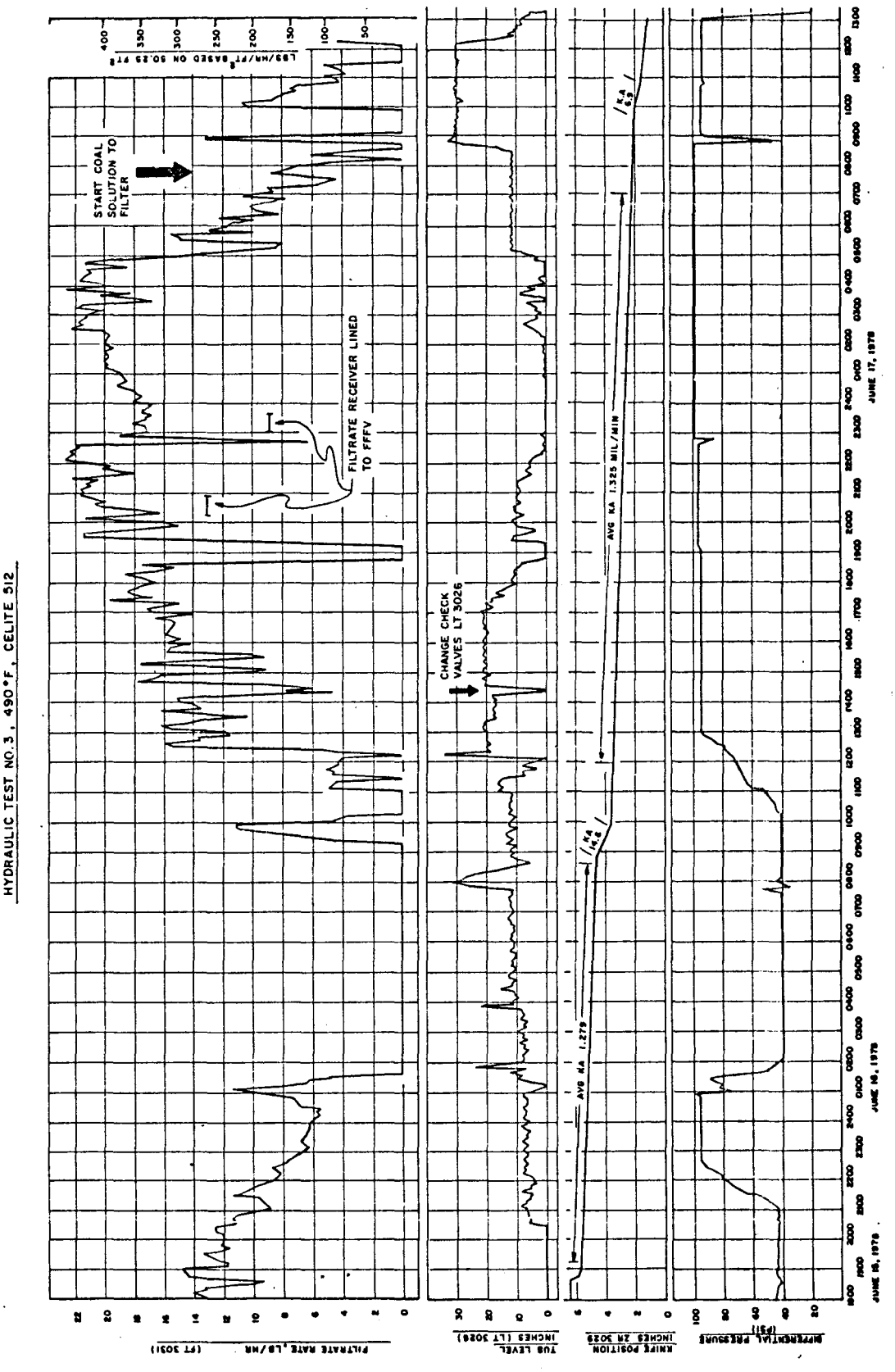

HYDRAULIC TEST NO.2,95 PSI, 465°F - STANDARD SUPERCEL
LBS/HR/FT² BASED ON 80.25 FT.²
400
350
300
250
200
150
NET FILTRATE RATE, LB/HR.
20000
18000
16000
14000
12000
10000
8000
6000
FILTRATE SEP. BOTTOMS PLUS GAS SCRUBBER BOTTOMS MINUS W.S. TO CAKE.
START HYDRAULIC TEST
POWER FAILURE
LOST DRUM DRIVE
LEVEL, INCHES LT 3026
30
20
10
0
DRUM SPEED RPM
5
3
1
LOST DRUM DRIVE
KNIFE POSITION INCHES ZR 3029 + 0.75
5
4
3
2
AVG. KA 1.60 MIL/MIN
TIME, HRS.
1500 1600 1700 1800 1900 2000 2100 2200 2300 2400 0100 0200 0300 0400 0500 0600 0700 0800 0900 1000 1100 1200 1300 1400 1500 1600 1700 1800
JUNE 8, 1978
JUNE 9, 1978

FIGURE A-14


HYDRAULIC TEST NO.3, 490°F, CELITE 512
START COAL SOLUTION TO FILTER
FILTRATE RECEIVER LINED TO FFFV
CHANGE CHECK VALVES LT 3026
AVG KA 1.325 MIL/MIN
AVG KA 1.279
JUNE 16, 1978
JUNE 17, 1978

APPENDIX B

TABLE B-1

Analysis[a] of Kentucky Nos 9 & 14 Coal
P&M Colonial Mine, Lot 7[b]

Proximate Analysis	
% Ash	11.41
% Volatile	36.73
% Fixed Carbon	51.86
	100.00
BTU	12793
Sulfur Forms	
% Pyritic Sulfur	1.13
% Sulfate Sulfur	0.25
% Organic Sulfur	1.99
% Total Sulfur	3.37
Free Swelling Index	3
Ultimate Analysis, wt %	
Carbon	71.17
Hydrogen	5.08
Nitrogen	1.50
Chlorine	0.04
Sulfur	3.37
Ash	11.41
Oxygen (diff)	7.43
	100.00
Mineral Analysis of Ash, wt % Ignited Basis	
Silica, SiO_2	48.93
Alumina, Al_2O_3	18.93
Titania, TiO_2	0.98
Ferric Oxide, Fe_2O_3	21.68
Lime, CaO	3.35
Magnesia, MgO	0.90
Potassium Oxide, K_2O	1.92
Sodium Oxide, Na_2O	0.09
Sulfur Trioxide, SO_3	2.60
Phos. Pentoxide, P_2O_5	0.11
Undetermined	0.51
	100.00
% Iron in Coal	1.73

a) Performed by Commercial Testing & Engineering Co. All analyses are presented on a dry coal basis.

b) The sample analyzed was a preliminary composite of samples obtained from each of 30 drums. After blending and sieving, average ash content was 10.07% and iron content (dry coal basis) was 1.51%.

TABLE B-2

Summary of Merriam Process Conditions, Yields, and Product Analysis

Run No.	GU 213	GU 214	GU 215	GU 216R
Conditions				
Coal Seam	Kentucky Nos. 9 & 14, Colonial Mine, Lot 7			
Nominal Liquid Residence Time, hr	0.98	0.99	0.99	1.00
Coal Feed Rate, lb/hr/ft^3	21.9	32.2	21.7	21.6
Nominal Dissolver Temperature, °C/°F	456/851	456/851	455/851	455/851
Dissolver Pressure, psig	2000	2000	2000	2000
H_2 Feed, wt % based on slurry	4.55	4.62	4.63	4.63
MSCF/ton of coal	57.1	38.7	58.1	58.2
Slurry Formulation, wt %				
Coal	30.0	45.0	30.0	30.0
Recycle Coal Solution	--	--	--	70.00
Recycle Solvent	70.0	55.0	--	--
Recycle Filtrate	--	--	70.0	--
Slurry Blend Composition, wt %				
Coal	30.0	45.0	30.0	30.0
Solvent	70.0	55.0	31.4	23.9
SRC	--	--	38.6	27.1
Ash (from recycle coal solution)	--	--	--	12.5
Insoluble Organic Matter (from recycle coal solution)	--	--	--	6.5
Total Solids	30.0	45.0	30.0	49.0
Yields, wt % based on MF coal				
H_2O	6.1	6.8	9.7	10.4
CO	0.4	0.5	0.5	0.4
CO_2	0.8	1.0	1.0	1.0
H_2S	1.9	1.8	2.1	2.3
NH_3	0.5	0.3	0.6	0.7
C_1	3.7	3.9	5.6	5.7
C_2	2.9	2.8	4.0	4.1
C_3	2.3	2.3	3.5	3.7
C_4	1.6	1.5	2.3	2.6
Total C_1-C_4	10.5	10.5	15.4	16.1
Naphtha, C_5 - 193°C	10.6	10.3	14.0	10.0
Middle Distillate, 193-249°C	11.3	9.0	12.3	7.1
Heavy Distillate (Recycle Solvent), >249°C	4.0	8.1	16.6*	21.8
Total Oil, C_5+	25.9	27.4	42.9*	38.9
SRC	42.7	41.4	16.0*	21.0
Insoluble Organic Matter	4.1	4.4	7.0	5.1
Ash	9.5	9.7	9.8	9.7
Total	102.4	103.8	105.0	105.6
H_2 Reacted (gas balance)	2.4	3.8	5.0	5.6
MAF Conversion, %	95.5	95.1	92.2	94.4
Lineout Index	--	--	--	1.00
Recycle Solvent (or Heavy Distillate) Analyses				
%C	88.04	87.56	88.15	87.68
%H	7.96	7.84	7.56	7.90
%S	0.28	0.33	0.36	0.29
%N	0.98	1.14	1.45	1.21
%O (By difference)	2.74	3.13	2.48	2.92
Specific Gravity	1.0327	1.0389	1.0611	1.0383
SRC (or Vacuum Bottoms) Analyses	← Filtered →			Unfiltered
%C	88.45	89.09	90.58	64.50
%H	5.70	5.52	5.08	3.55
%S	0.49	0.48	0.40	2.95
%N	2.11	2.27	2.28	1.49
% Ash	0.16	0.07	0.23	27.60
%O (By difference)	3.09	2.57	1.43	--

* These values do not represent lined out operation. At lineout the yield of SRC will be higher and the yield of heavy distillate will be lower.

TABLE B-2 (Continued)

Run No.	GU 219 A	GU 219B	GU 219C	GU 219D
Conditions				
Coal Seam	←——Kentucky Nos. 9 & 14, Colonial Mine, Lot 7——→			
Nominal Liquid Residence Time, hr	1.00	1.03	0.99	0.99
Coal Feed Rate, lb/hr/ft^3	21.4	20.8	21.2	21.6
Nominal Dissolver Temperature, °C/°F	455/851	455/851	455/851	455/851
Dissolver Pressure, psig	2000	2000	2000	2000
H_2 Feed, wt % based on slurry	4.64	4.77	4.70	4.61
MSCF/ton of coal	58.2	59.9	59.0	57.9
Additive	Mineral Residue	Mineral Residue	Mineral Residue	Mineral Residue
Slurry Formulation, wt %				
Coal	30.0	30.0	30.0	30.0
Recycle Solvent	65.0	65.0	60.0	55.0
Additive (Mineral Residue)	5.0	5.0	10.0	15.0
Slurry Blend Composition, wt %				
Coal	30.0	30.0	30.0	30.0
Solvent	65.1	65.0	60.0	55.2
SRC	--	0.3	0.5	--
Ash (from recycle coal solution)	3.7	3.2	6.5	11.0
Insoluble Organic Matter (from recycle coal solution)	1.2	1.5	3.0	3.8
Total Solids	34.9	34.7	39.5	44.8
Yields, wt % based on MF coal				
H_2O	6.2	6.5	7.3	7.6
CO	0.4	0.5	0.4	0.5
CO_2	0.8	0.9	0.8	0.9
H_2S	2.3	2.7	2.1	2.0
NH_3	0.4	0.5	0.6	0.5
C_1	4.3	4.7	4.2	4.5
C_2	3.2	3.5	3.2	3.3
C_3	2.7	3.0	2.7	2.9
C_4	1.8	2.0	1.9	2.0
Total C_1-C_4	12.0	13.2	12.0	12.7
Naphtha, C_5 - 193°C	11.5	13.7	17.8	13.9
Middle Distillate, 193-249°C	9.1	10.7	16.7	12.9
Heavy Distillate (Recycle Solvent) >249°C	12.1	8.3	3.3	10.2
Total Oil, C_5+	32.7	32.7	37.8	37.0
SRC	36.4	34.5	31.6	32.1
Insoluble Organic Matter	2.9	3.1	2.2	1.3
Ash	10.2	10.2	10.2	10.2
Total	104.3	104.8	105.0	104.8
H_2 Reacted (gas balance)	4.3	4.8	5.0	4.8
Recycle Solvent (or Heavy Distillate) Analyses				
% C	87.70	87.73	87.79	87.61
% H	8.22	8.27	8.09	8.18
% S	0.28	0.24	0.24	0.22
% N	0.93	0.91	0.94	0.94
% O (By difference)	2.87	2.85	2.94	3.05
Specific Gravity	1.0241	1.0156	1.0216	1.0197
SRC (or Vacuum Bottoms) Analyses	←———	Filtered	———→	
% C	88.93	89.03	89.11	89.00
% H	5.65	5.56	5.71	5.84
% S	0.47	0.39	0.41	0.38
% N	2.24	2.30	2.24	2.23
% Ash	0.23	0.53	0.47	0.15
% O (By Difference)	2.48	2.19	2.06	2.40

APPENDIX C

TABLE C-1

COAL ANALYSES

Coal	Powhatan LR-24924	Valley Camp LR-24925
Elemental Analysis, moisture free basis		
Carbon	73.41	74.03
Hydrogen	5.10	5.19
Nitrogen	1.29	1.43
Sulfur	3.37	2.88
Oxygen (by diff)	7.36	7.59
Ash	9.47	8.88
Moisture	0.80	0.73

DISTRIBUTION LIST

Standard Distribution Category UC-90d - 309

Additional External Distribution:

Clifford N. Click
Pullman Kellogg Research & Development Center
16200 Park Row, Industrial Park Row
Industrial Park Ten
Houston, TX 77084

Everett Huffman
Southern Services, Inc.
P. O. Box 2625
Birmingham, AL 35202

Sidney Marks
Environmental & Safety Research
Battelle Pacific NW Laboratories
Richland, WA 99352