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SOLVENT REFINED COAL (SRC) PROCESS

QUARTERLY TECHNICAL PROGRESS REPORT
FOR THE PERIOD
JULY 1, 1978 THROUGH SEPTEMBER 30, 1978

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

Work Performed Under Contract EX-76-C-01-0496

PREPARED FOR THE
U. S. DEPARTMENT OF ENERGY
DIVISION OF COAL CONVERSION AND UTILIZATION
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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 July 1, 1978 through September 30, 1978. Major activities at the Fort Lewis Pilot Plant during the quarter included SRC I operation and the test operation of a new type of rotary drum filter, Filter C. A period of SRC II operation using a Pittsburgh seam coal from the Powhatan No. 5 mine was initiated at the end of the quarter. Merriam Laboratory activities included testing of novel reactors and very short residence times and a study of solvent behavior at SRC II reaction conditions. The Process Development Unit P-99 at Harmarville tested a series of Pittsburgh seam coals in the SRC II process to study both feedstock effects and process variable effects.

I. SUMMARY OF FORT LEWIS PILOT PLANT OPERATIONS

From June 25 through August 23, 1978, the Fort Lewis Pilot Plant continued to process Kentucky Nos. 9 and 14 coal in the SRC I mode to provide coal solution slurry for the testing of a newly installed filter designated as Filter C. During this period approximately 1,640 tons of coal were processed in 974 hours of operation for an adjusted hourly on-stream factor of 66.5%. The pilot plant was shut down on August 23 after the vacuum flash preheater became plugged as a result of severe coking. It became apparent that decoking of the vacuum flash preheater would extend beyond September 1, the scheduled date for termination of filter testing operation, and the shutdown was modified to a plant turnaround and conversion to SRC II processing. On September 21 the plant began SRC II processing of Pittsburgh seam coal from North American Coal Corporation's Powhatan No. 5 mine. The plant was subsequently shut down to clear a plugged dissolver sample probe. The adjusted hourly on-stream factor for the period of SRC II operation between September 21 through September 24 was 49.3%. At the end of the reporting period the plug had been removed and preparations were being made to resume feeding Powhatan No. 5 coal.

In spite of a number of operational difficulties with Filter C, eight filtration experiments were completed. These experiments were designed to evaluate the effects of knife advance rate on filtration rate at a drum speed of 5 rpm. The greatest difficulty experienced during these runs was repeated failure of the filter screen, the result of a design defect in the screen support system. Other operating problems were frequent plugging of the cake wash and sluice nozzles, unreliable operation of the cake auger and the filter tub level indicator, and leaks at all shaft and sight port glands. None of the short runs produced filtrate of sufficient clarity to assure continuous production of specification SRC I.

In meetings held at Fort Lewis on August 31 and September 1 with representatives from Johns-Manville Corporation, EPRI and Stearns-Roger, it was generally agreed that major design changes would be required for Filter C to become a reliable process unit. Improved design is needed for the screen support system, the filter drum sluice and wash systems and the filter tub level control. Further consideration must also be given to the design of the drum drive mechanism, the filter sight glasses and the shaft packing assemblies. Until these design problems have been resolved, further testing of Filter C is not anticipated.

Operating problems, related to corrosion and coking, were experienced in the solvent recovery area. Between August 13 and August 19 coal feed was stopped to repair leaks which had developed in the wash solvent column overhead cooler. Following the repair of the cooler, attempts to restart the solvent recovery area failed when the old vacuum flash preheater plugged. X-rays indicated that the coil was severely coked from the seventh to the twentieth coil. Work to unplug the vacuum flash preheater coil was started August 25. Eventually, the coil was cut in eight places to facilitate hydroblasting. By

September 11 all preheater coil rewelding and stress relieving was complete. All welds were x-rayed and found to be within code specifications. After repair and dryout of the heater refractory September 21, the old vacuum flash system was put on circulation and held in reserve.

A system was installed and successfully operated to withdraw samples from the dissolver during operation. One sample was taken during well lined-out operation on July 11 and another at a lower pressure and gas rate on July 19, both from a point about 15 inches above the dissolver inlet. It was not possible to obtain satisfactory analyses of the gas phase of these samples, but various analyses of the liquid phase were carried out. In addition, samples of stripper bottoms were taken at the same time to represent dissolver outlet composition. The data show that the dissolver samples contain much more ash and pyridine insolubles than the comparable stripper bottoms, which confirms other studies indicating that there is significant settling of mineral within the dissolver in SRC I operation.

Studies to determine the effects of raw solvent additions to the process stream on solvent yields and on solvent quality were conducted in the months of June, July and August. These studies indicated that in the SRC I mode very little of the raw solvent is converted to wash solvent. During the study the wash solvent inventory increased by 487 barrels. This increase represents a calculated yield of 4.9 wt % based on moisture-free coal. Process solvent inventory yields, exclusive of raw solvent additions, were negative. Process solvent quality, as measured by IR ratio, showed steady improvement during the overall test period except that pronounced declines in IR ratio were observed when raw solvent was introduced into the process stream.

A study on the swelling properties of coal suggests that coal swelling at elevated temperatures (above 350°F) can be minimized by increasing the diameter of the coal particles fed to the mix tank or by decreasing the residence time of the slurry in the blend tank. Study of the solvation of large coal particles confirmed the feasibility of feeding larger sized coal to the plant reaction system.

At the end of August overall construction of the C-E Lummus antisolvent deashing system was essentially complete, and two Lummus representatives were at the plant supervising precommissioning activities. Work through September consisted of clearing punch-list items submitted to Lummus by P&M operations. At the end of the quarter, water-flushing and piping dry-out operations were in progress. Systems testing of this unit will begin once the current SRC II test program has been completed and the plant has been converted to the SRC I mode.

II. PILOT PLANT OPERATIONS, ENGINEERING AND MAINTENANCE

A. Coal Receiving and Preparation (Area 01)

1. Operation June 25 through August 23, 1978

One thousand six hundred forty tons of Kentucky coal were processed in 49 days of SRC I operation for an on-stream factor

based on days of operation of 80.3%. The adjusted hourly on-stream factor for the period of SRC I operation from June 25 through August 23, 1978 was 67.6%. A coal processing summary covering the third quarter and overall process operating histories is shown on Table 1. The primary objectives for the first two months of the third quarter were to provide coal reaction slurry for testing of a newly installed filter and to process raw solvent to increase the pilot plant process solvent inventory. Target coal feed rates ranged from 2,500 lb/hr to 4,250 lb/hr. Coal slurry concentration ranged from 33% to 40%.

Area 01 problems caused several major coal outages. After start-up June 26 the pulverizer inert gas heater tripped off when a relay coil in the control circuitry burned out. Since no replacement coil was immediately available, coal feed was out 11 hours. Coal feed was resumed the afternoon of June 27 and continued until gas circulation through the dehumidification loop was lost June 28 when the impeller on the inert gas blower worked loose. After maintenance had repaired the blower, it was discovered that on low fire the inert gas heater would not shut down when gas flow through the coil was lost. Modifications were then made to the heater circuit to correct this problem. These combined problems resulted in 21 hours of lost production.

Electrical power failures caused two curtailments and upset the entire plant operation. Several power interruptions (transients) on July 26 delayed the starting of coal feed 12 hours. On August 8 a complete power failure required the use of the emergency generating system. On demand, the emergency generators synchronized and came on line immediately. Once the generators were adjusted so that total output was equally balanced between the three generators, the system stabilized and performed as required.

2. Turnaround Maintenance

Converted pulp dryers previously used to pre-dry Blacksville Pittsburg seam coal were reinstalled next to the track hopper since Powhatan No. 5 coal also required drying. The dryers were fed using a front-end loader instead of conveyors which had been a source of operating problems in previous experience.

During the turnaround to convert to SRC II operation, additional piping was installed to complete the coarse coal (-1/8") circulation test loop. Two capillaries were installed in parallel downstream of the high pressure charge pumps and transfer line going to the slurry preheater. Capillary A, made from 75' of 9/16" O.D. (0.312" I.D. stainless steel autoclave tubing, was installed to provide back pressure to the high pressure charge pumps. Capillary B, made from 160' of 3/4" schedule 160 pipe, was installed to provide an alternate route if capillary A plugged. New 1" piping was installed from the capillary discharge to the inlet of the existing quadrant edged orifice meter (FE-1152). Existing piping from the meter back to the

TABLE 1

COAL PROCESSING SUMMARY

	1974	1975	1976	1977	June 24- July 25 1978	July 25- Aug. 24 1978	Aug. 25- Sep. 24 1978	1978	Total
Coal Processed, Tons	678	8,021	6,559	4,776	999	636	13	4,005	24,039
On-stream Days	30	241	234	216	29	20	1	147	868
On-stream Hours [a]	-	-	-	3,990	596	378	13	2,854	6,844
Average Feed Rate Per On-stream Day, Tons/Day	22.3	33.3	28.0	22.1	34.4	31.8	13.0	27.2	27.7
Average Feed Rate Per On-stream Hour, Lb/Hr [a]	-	-	-	2,115	3,352	3,365	2,000	2,807	2,566
On-stream Factor, Days %	32.6	66.0	63.9	59.2	96.7	64.5	3.2	55.1	59.7
On-stream Factor, Hours, % [a]	-	-	-	67.9	82.8	50.8	1.7	44.5	57.1
On-stream Factor, Adjusted Hours, % [b]	-	-	-	-	82.8	50.8	12.9	70.6	70.6

[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.

slurry blend tank was not modified. (The Area 02 slurry preheater is isolated from this circulation loop.) To prevent large coal particles from settling in the pump, a 2" bypass from the charge pump to the slurry blend tank was installed. For test purposes only, high pressure (bottled) nitrogen was piped into the system to supply cushion gas to the high pressure charge pump pressure snubbers.

On September 2 and 3 a successful test run was completed pumping minus 1/8 inch coal in process solvent around the existing feed slurry preparation systems. The test system included the new mix tank, the slurry blend tank, the centrifugal circulating pumps and the high pressure charge pumps. The slurry was pumped at ambient temperature up to the inlet of the slurry preheater where it was diverted through a 0.312" ID capillary line back to the slurry blend tank. A maximum of 49 percent coal in the slurry was achieved and best operation was at concentrations above 30 percent. The system was able to resume normal operation after stopping flow for up to 30 minutes, and an inspection of the slurry blend tank on September 7 revealed no significant buildup of deposits. Engineering is being accelerated to provide minus 1/8 inch crushed coal for an on-stream test with Powhatan coal.

3. Operation September 21 through September 24, 1978

Startup in the SRC II mode using Pittsburgh seam coal supplied by North American Coal Corporation from their Powhatan Mine No. 5 began September 21 but was interrupted after 13 hours of operation to replace the bottom sample tap on A dissolver which had plugged. Area 02 was repaired by September 24, but repacking of recycle compressor A, as well as the repair of the valves and rings in the fresh hydrogen compressor, delayed startup 16 additional hours. The adjusted hourly on-stream factor for the period of SRC II operation between September 21 through September 24 was 49.3%. At the end of the period preparations were being made to resume coal feed.

Routine maintenance activity included replacement of the pulverizer rotary air lock with the rebuilt spare and replacement of high pressure charge pump plungers, plunger packing and ball checks.

4. Pump Performance

a. Preheater Charge Pumps A and B (01D56101, 01D56102)

High pressure charge pump plunger packing failed six times between June 25 and July 24. Three of these failures occurred on plungers having an experimental Triballoy 400 metal spray coating rather than the nickel-chrome-boron coating usually used.

Triballoy 400 exhibited poor bonding and high wear rate characteristics. The other failures occurred because the chevron ring packing previously used was not available and standard packing had to be substituted.

In July two discharge valve balls were replaced on Charge Pump A. They were scratched and indented.

b. Slurry Blend Circulation Pumps (01D56047, 01D56080)

These Durco centrifugal pumps required minimal maintenance during the reporting period.

B. Slurry Preheating and Dissolving (Area 02)

1. Operation June 25 through August 23, 1978

Area 02 processed coal slurry 49 days of this period during the Filter C testing phase. All operation was in the SRC I mode with half dissolver. The dissolver operated at pressures between 1500 psig and 1800 psig while the dissolver outlet temperature was maintained at 850°F. Eight attempts to complete portions of the Filter C test program were made under these conditions.

When Area 04 was shutdown July 24 to repair a leaking cooler, Area 02 was shutdown to repair both the high pressure (LCV-166A) and the intermediate pressure (LCV-175) level control valves. Trim made from tungsten carbide was installed at LCV-166A, but experimental trim made from boron carbide, which was being considered a possible substitute for tungsten carbide, was installed at LCV-175. The boron carbide tip failed after twenty minutes of operation on solvent flow. Since it failed so quickly, no additional tests were made using boron carbide trim. Repair of LCV-175 was completed using trim made of tungsten carbide.

Area 02 operation was seriously upset July 26 because of a succession of power dips and again on August 8 because of a power failure. Area 02 was also shutdown several times during the period from July 26 to August 13 to complete repairs in Areas 03 and 04. Area 02 was shutdown August 23 along with the rest of the plant to prepare for SRC II operation.

2. Turnaround Maintenance

B dissolver head was removed from A dissolver and reinstalled on B dissolver. The A dissolver was hydroblasted and external piping was switched for full dissolver operation (SRC II mode). The original head from A dissolver, which had been removed to repair a collapsed middle quench line, was reinstalled on A dissolver. Replacement of the middle quench line was completed during the SRC I run. The middle quench line was modified with a T-shaped gas sparger to test a proposed design for demonstration plant spargers.

Installation of a second solids sampling probe in the side/center outlet of A dissolver as well as a high pressure nitrogen purge system was also completed. The high pressure nitrogen from a tube trailer will serve as an emergency purge for the dissolver quenches and sample probes if the hydrogen purge is lost.

Other maintenance work completed during the shutdown included:

- Removal and replacement of broken plug tips on the trim of the high pressure let-down valve (LCV-166A), the intermediate pressure let-down valve (LCV-175A) and the water quench temperature control valve (TCV-167).
- Reinstallation of the Willis variable orifice control valve (LCV-166B) and block valves in parallel with the Fisher DBAQ valve at LCV-166A.
- Refinishing of the sealing surfaces on the head flange of the intermediate pressure flash drum.
- Replacement of the corrosion racks in all Area 02 high pressure vessels.
- Relocation of the dissolver D.P. cell.
- Installation of a new head on the high pressure flash drum.
- Installation of a 2" diameter erosion/corrosion test loop in the dissolver effluent line.

3. Operation September 21 through September 24, 1978

Startup September 21 was interrupted after 13 hours of operation to unplug the dissolver sample tap and to clear solvent from the hydrogen purge system. Fifty-one hours of lost production were attributed to the plugged sample tap. Slurry processing was scheduled to begin again September 24, but compressor problems in Area 05 delayed the startup.

C. Mineral Separation and Drying (Area 03)

After hydraulic test No. 3 was completed June 16 (see second quarterly report), coal slurry was fed to Filter C for 3.5 hours to gain operating experience before beginning the second phase of the Johns-Manville filter test program. At the end of the run the precoat could not be sluiced from the screen indicating possibly plugged sluice nozzles. Between June 16 and June 26 the filter was shut down to clean the screen and address the problem of tub flushing during the sluicing cycle. Problems in Area 01 with the inert gas heater delayed the start of filtration test No. 1A until June 27.

1. Operation June 25 through August 23, 1978

Eight filtration test runs were conducted with Filter C at various conditions in order to obtain data for determining the optimum

knife advance rate, differential pressure and precoat type. Operating parameters for these test runs are given in Table 14.

On June 27 Filter C was precoated using 150 pounds of Celite 550 basecoat and 750 pounds of Standard Supercel for precoat. During the precoating operation large dark spots were observed on the precoat surface indicating possible blinding. At the request of the Johns-Manville representative, the precoat was cut off with a rapid knife advance and the heel was successfully sluiced with the high pressure sluicing system.

On June 28 the filter was again precoated using the same procedure and mix as in the previous attempt. During this operation there were no indications of blinding, and the first filtration test (1A) with coal slurry was begun. The test was conducted with knife advance as the variable and the following constants:

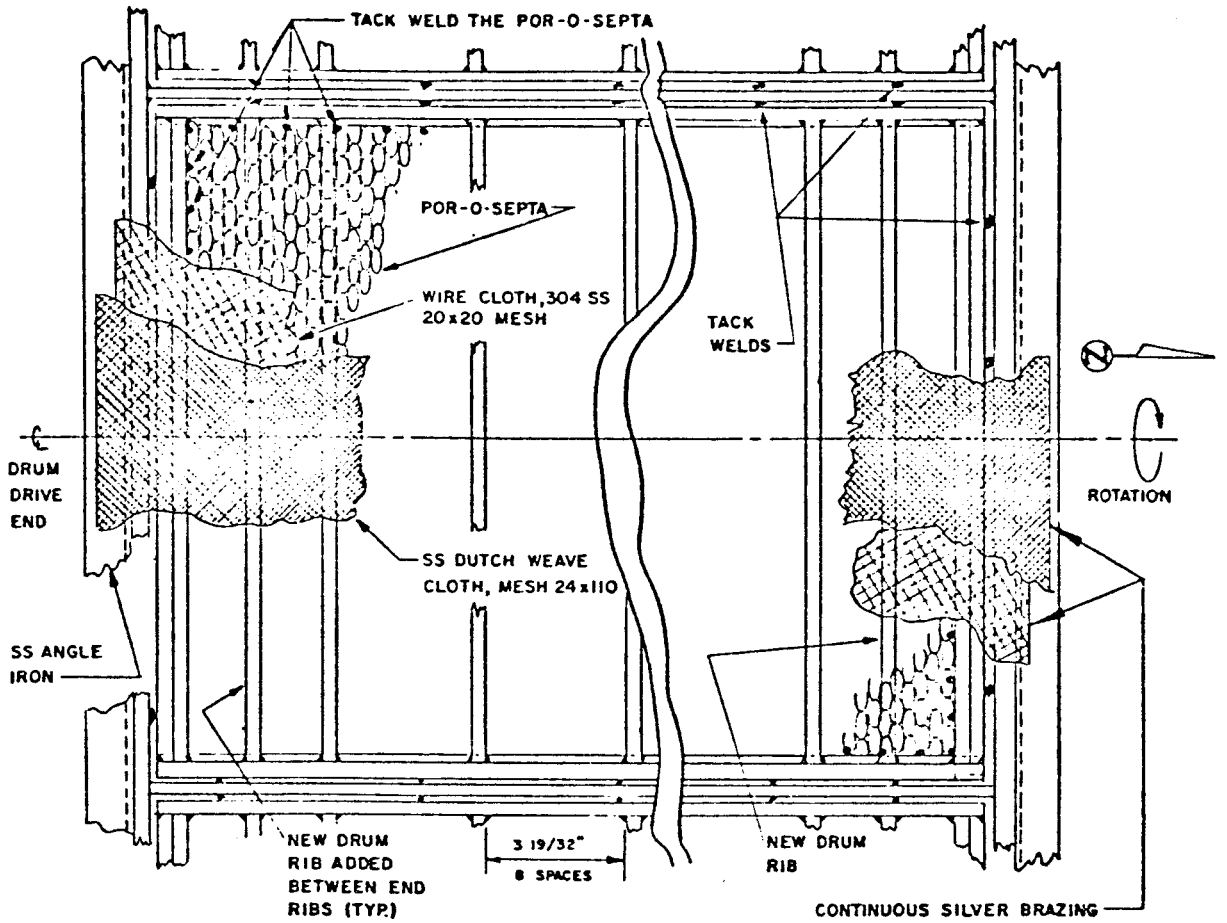
Filter temperature	600°F
Delta P	95 psi
Drum submergence	40%
Cake wash rate	1,000 lb/hr
Drum speed	5 rpm
Gas humidity	Dry

The test was completed on June 30, but because the high pressure sluice nozzles were plugged, the heel could not be sluiced from the drum. After the filter had been hydroblasted it was opened for inspection to determine the cause of poor filtrate clarity during the run. As suspected, several holes had developed in the screen along the outer circumference of the drum. In addition, the filter cloth (septum) and backing screen (Figure 1) were distorted in a scalloped pattern against the support ribs and the segmented knife blades were bowed upward in a similar scalloped pattern. The holes in the dutch weave filtering screen apparently developed when the Por-O-Septa support screen pulled away from the edge support member, allowing the septum to tear on the exposed edges. Support failure was caused by permanent deflection of the Por-O-Septa between the positive support bars.

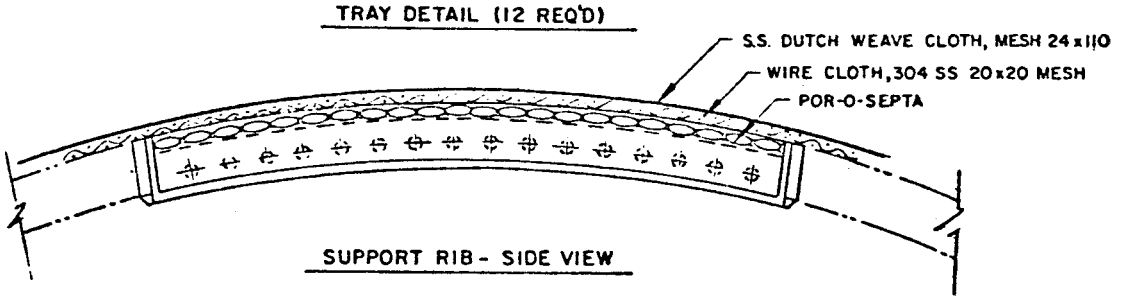
The Johns-Manville filter consultant believed that the events leading to the collapse of the support screen began when the rate of knife advance was reduced from 2 mils/rev to 1/2 mil/rev in a single step. (The knife advance rate was changed to determine its effect on filtration rates.) The abrupt reduction in the knife advance allowed time for coal solids (filter cake) to build up and penetrate the precoat causing the precoat to contract. Contraction of the precoat increased the distance between the knife and the precoat allowing additional time for solids to build up and further compact the precoat. Eventually compaction of the precoat stopped, and the knife which was continually advancing began to contact the filter cake. Unfortunately, in this test the knife was unable to penetrate the hard filter cake and began skimming over the surface of the filter

Figure 1

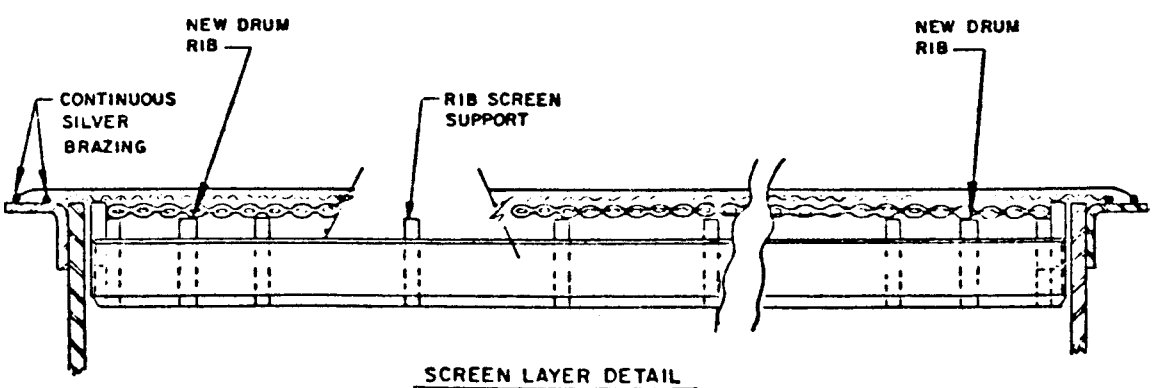
FILTER "C" SCREEN ASSEMBLY



TRAY DETAIL (12 REQ'D)



SUPPORT RIB - SIDE VIEW



SCREEN LAYER DETAIL

cake. The filter cake was not cut smoothly but, instead, spalled off in advance of the knife and coal solids severely abraded the knife segments while uneven pressures distorted the knife segments. Ideally, the resultant force on the knife should be transmitted to the advance mechanism along the plane of the knife support blade; however, during this test, the knife angle was too shallow and the resultant force exerted on the knife deflected it away from its support blade.

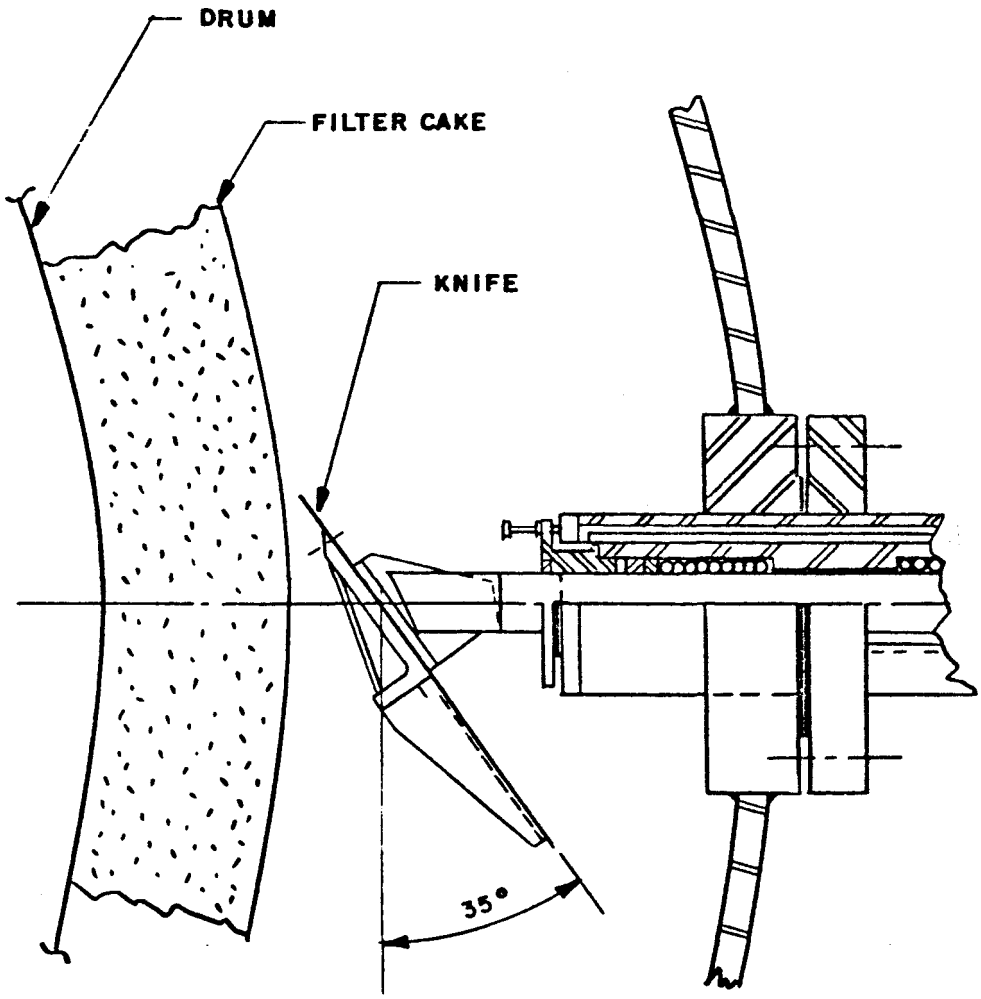
Between July 2 and July 14 several modifications were made to resolve the problems described above. First, in order to align the resultant force in the appropriate direction, the angle of the knife attack was increased from 30° to 35°, as shown in Figure 2. Second, to prevent distortion of the knife segment under stress, a section of spare knife blade, in stock as a replacement for Filter A and fabricated from 1/8" Stellite, was installed. The original knife segments for Filter C were made of 1/16" Stellite. Third, based upon the manufacturer's recommendation, a new and stronger Por-O-Septa screen having larger openings (0.125") was purchased and installed. According to the vendor, the new screen was capable of supporting twice the load of the original screen. Fourth, instead of re-installing the 20 mesh back-up screen in segments, this screen was installed in one piece around the whole circumference of the drum. It was anticipated that this change would help to support the septum by bridging across the uneven areas at the edge of the drum and between sections.

At the end of run 1A the cake wash nozzles and the high and low pressure sluice nozzles were plugged. All filter nozzles and headers were cleaned and the spray patterns checked with water. Plugging in these nozzles had been a constant problem in previous precoating attempts and hydraulic tests. To alleviate this problem, parallel Y-strainers were installed on all supply headers and piping was installed to provide for blow down of these strainers. Piping was also installed to provide for reverse flow of inert gas and wash solvent vapor through the spray nozzles using internal filter pressure.

All of the above modifications were completed by July 15. In reassembling the filter, maintenance inadvertently bent one of the packing gland retainer bolts on the filter end where the shaft goes from the filter to the filtrate receiver. The entire filter had to be disassembled to repair the damage. The filter was turned over to operations July 17, but problems with packing leaks and shaft vibration prevented any precoating activity for several days. During July 18 and 19 the filter was down to replace drum shaft packing on the north shell gland and in the receiver gland. Packing in the north shell gland was 7/8" Chesterton Style 1 pure graphite. All of the external braid had been worn off the two outer rings; the inner rings, however, were not worn. Based on this observation, it appeared that all of the effective sealing occurred in the outer rings of the gland and that the packing would not compress evenly in the stuffing box. The filtrate

Figure 2

MODIFIED KNIFE BLADE ANGLE



receiver gland had been packed with 1" Garloc lubricated graphite packing. This packing was not severely worn but, as a precaution, was replaced. Upon reassembly, both glands were repacked with 1" Garloc.

The filter was precoated July 19 using 100 pounds of Celite 550 basecoat and 750 pounds of Standard Supercel for precoat. Although the precoat was uneven and discolored with black spots, the Johns-Manville representative recommended beginning filter test 1B. The drum speed was 5 rpm and the knife advance was varied from 0.3 to 2.4 mils per revolution. The other constants were the same at test 1A. Mechanical problems interrupted the test on two occasions, but the test was completed on July 21. Once again the heel could not be sluiced because the high pressure sluice nozzles were plugged.

After filtration run 1B the filter was shutdown to hydroblast the screen and determine the cause of poor filtrate clarity. Inspection of the screen revealed that the Por-O-Septa had again deflected between the positive support bars and that the septum had pulled away from the drum edge in at least three places. Three pinhole leaks were also observed in the screen. These holes were resoldered, but the repair was considered to be marginal because the areas could not be cleaned sufficiently to prepare a proper bonding surface for the silver solder.

Since back blowing the spray nozzle proved to be only marginally effective, nitrogen was piped into all spray nozzle headers to allow the nozzles to be continuously purged while the filter is in operation and the nozzles are not in use. This was done to prevent accumulation of condensed material which might then leave a deposit of coke due to the high operating temperature in the filter.

Final repair of the filter consisted of replacing the Chesterton Style 1 packing at the drive end with Garloc packing to stop the leaks which had developed in run 1B.

Repairs and modifications to the filter were completed July 25 and the filter was again turned over to operations. Electrical power problems caused by bad weather on July 26 delayed coal feed by 12 hours which in turn delayed steady state operation until July 27. During basecoating operations on July 27, three 75 pound batches of Johns-Manville Celite 550 were added before the screen picked up any basecoat. Finally, the filter was precoated with 850 pounds of Johns-Manville Standard Supercel. After filtration test 1C began July 27, packing leaks developed on both ends of the filter and on the filtrate receiver. Although the packing could have been compressed enough to allow the addition of another ring of packing by further tightening of the packing gland, this action was not taken because the added drag on the shaft would have exceeded drum driver horsepower availability. Instead, the filter was removed from service to completely repack all of the leaking glands (at the filtrate separator and at the north and south ends of the filter vessel).

When the filter was opened July 29 to determine the cause of poor filtrate clarity in test 1C, the shell was full of basecoat and precoat. In addition, maintenance found that the drum interior inspection plate had not been reinstalled in the tub. These observations explain why precoating was only marginally successful on July 27 and July 28. Although cake sluice was not successful during shutdown of the filter, the high pressure sluice nozzles were clear. After the screen was hydroblasted and all nozzles were cleaned and reinstalled, the filter was ready for testing July 30.

The fourth attempt (test 1D) to run filtration test 1 began late July 31 but was terminated August 1 when neither the filtration rate nor the filtrate clarity were satisfactory. This time the heel was successfully sluiced before the filter was turned over to maintenance for repair. A large hole had developed in the screen. After the screen was patched, the spray nozzles were steam cleaned and checked with water. All five of the top cake wash nozzles and two of the five east cake wash nozzles were plugged. In addition, two of the five auger wash nozzles and one of the four knife wash nozzles were plugged. While removing the cake wash nozzles for cleaning, two were dropped and lost. Because duplicate replacements were not immediately available, the middle three top cake wash nozzles were replaced with 1/4" PSS-5010 flat jet nozzles. Three nozzles were replaced because, in addition to the two that had been lost, one of the remaining original nozzles had a poor spray pattern.

During startup late August 4, a loud noise developed in the packing gland at the north end of the filter. After adjustments were made to the packing gland to quiet the shaft noise, a gasket began leaking at the north manway. Once the leak at the north manway was repaired, the filter was ready for testing. For precoating operation, 100 pounds of Celite 503 and 850 pounds of Celite 512 were used instead of Celite 550 and Standard Supercel. Test 2A began late August 4 and lasted approximately ten hours. Although no operating difficulties were encountered, filtrate clarity remained unsatisfactory ranging from 0.20% to 0.81% pyridine insolubles.

After test 2A the heel was successfully sluiced and the filter was precoated for test 2B (a repeat of 2A) without shutting down for maintenance. During test 2B the filtrate solids increased to 4.0%. On August 6, after test 2B had been completed, the filter drum was inspected to detect the source of solids leakage. Once again, as in previous runs, the septum had pulled away from the south edge of the drum and the backing screen was partially collapsed. The severity of the deflection was less than that of run 1A but again indicated that Por-O-Septa made from carbon steel lacks sufficient strength to withstand the forces exerted on it. After the filter was hydroblasted, the tear along the edge and several holes in the screen were patched. Even though extensive efforts were made to clean the areas to be patched, the

necessary cleanliness required for good brazing was not achieved. Consequently, the repairs were considered to be adequate only for short-term operation. At this time it was observed that the filter cloth and backing screen were starting to distort again into a scalloped pattern against the support ribs. While the filter was down, all of the cake wash solvent nozzles were replaced with new SS 5010 nozzles and the auger wash manifold was removed and relocated above the knife support. In addition, 100 mesh (Tyler) in-line strainers were added to the wash solvent, the flush solvent and the high pressure flush supply lines downstream of the Y strainers previously installed. These screens were installed to determine if the solids which plug the nozzles are migrating past the Y strainers or, alternately, if they are forming by coking or corrosive action in the internal filter piping.

During preparations for test run 3A, August 10, maintenance adjusted the packing at the filtrate separator to eliminate shaft vibration. At the same time, leaking packing in the newly installed sight ports was tightened. The filter was basecoated with 200 pounds of Celite 503 and precoated with 800 pounds of Celite 512. The precoat developed dark spots and some pieces of precoat fell off the screen. Because of these difficulties, operations began removing the precoat. At this time the sluice nozzles plugged so the heel was removed by submerging the drum in solvent and washing the screen with cake wash. The second basecoating attempt proved successful and filter test 3A started August 11. As this run progressed, leaks developed at both ends of the filter and filter separator. These leaks were controlled by further tightening of the packing glands. When the filtrate solids (pyridine insolubles) went over 0.5%, 100 pounds of Celite 550 were injected with the feed. After the Celite 550 was injected, the filtrate solids dropped to about 0.15%. The filter knife advance was stopped twice during test 3A; once because of low levels in the feed vessels, and once to remove a plug in the cake leg. The plug developed when a stray bolt lodged in the valve seat of the filter cake leg control valve. Test 3A was concluded August 12. No attempt was made to sluice the cake, instead the filter was recoated with Standard Supercel on top of the basecoat heel.

Test run 3B (a repeat of 3A) began August 13 and once again leaks developed on the drum drive end of the filter. These leaks, as well as leaks from the sight glasses, were controlled by tightening the packing glands. Exceedingly high pyridine insoluble values (4.0%) in the filtrate during this run indicated the possibility of another screen failure.

When the filter was cleaned and inspected, several holes were found. Most of these holes were located at points where the screen had previously been patched. To maximize the chances for completing the additional tests, the Johns-Manville representative requested that the septum, the backup screen and the Por-O-Septa support screen be replaced. Before the support panels were rescreened, they were modified to provide additional

support for the Por-O-Septa near the drum edge. Two new support ribs were installed in each panel. These new ribs were installed at the ends of each panel and positioned half-way between the end of the panel and first support rib. The modified support panel is shown in Figure 1.

The support screen which failed August 13, as well as the original support screen which failed in July, were made of carbon steel. Because Por-O-Septa made from carbon steel lacks sufficient strength to support the filtering screens at operating temperature, it was not used a third time. Instead, Por-O-Septa made from stronger 316 stainless steel was ordered August 10. This new stainless steel screen was installed on the support panels August 16. Filter C was completely rescreened August 20. During the next two days the high pressure sluice nozzles which had plugged were replaced and the drum drive sheaves were modified to increase the maximum drum speed from 5 rpm to 7 rpm.

Leaking sight glasses were a persistent problem. From the beginning of the test program efforts to stop leaks between the sight glasses and the retainer were unsuccessful. Unless these leaks can be stopped and the safety of these glasses proven, continued use of these units is not recommended. If the leaks cannot be stopped, the possibility exists that not enough compression can be maintained on the glass to prevent it from disintegrating if it fractures (quartz glass fractures easily). Unfortunately, the safety of these sight ports is predicated upon maintaining the glass under compression. Safety, although a major problem, was not the only problem. Assembly of these units without chipping the edges of the glasses was quite difficult. On August 15 a representative of Pres-Sure Products visited the plant and demonstrated the assembly of one sight glass to selected P&M technicians. He then directly supervised the assembly of two other units by the P&M technicians. The vendor's representative was confident that in the future these technicians would be able to properly assemble the units without supervision. The vendor recommended that after the units are installed the packing glands be tightened at intervals of 100°F as filter temperature increased. He thought this technique would assure that the packing would hold leak tight. The three sight ports which were assembled during the representative's visit were installed August 21. The filter was operationally ready for testing August 23; however, problems in Area 04 prevented startup. Consequently, the new screen, the new support ribs, the new sight ports, and the new nozzles remain untested.

2. Turnaround Maintenance

Because the trim in the high pressure quench water control valve (PCV-1309A) often requires replacement, a second parallel pressure control loop (PCV-1309B) was installed. Installation of a second loop eliminates the need for manual operation or a plant shutdown when trim replacement is necessary. The control valve outlet piping was routed directly back to the pump suction manifold rather than into the recycle process water tank (RPWT). There

was concern that water return to the tank was disturbing the oil-water interface and making separation more difficult.

All activity on Filter C has been suspended indefinitely.

3. Pump Performance

a. Filter Feed Surge Vessel Pump (03D56067)

This pump required one new seal during the reporting period. In September the pump case was replaced as a hole had developed in the pump suction nozzle.

A new Lawrence heavy duty slurry pump, Model 1-1/2 AL, was installed for future evaluation in this service.

b. Filter Feed Flash Vessel Recirculation Pump (03D56006)

This pump required two new seals and a pump realignment during the reporting period.

c. Recycle Water Booster Pumps A and B (03D56109, 03D56120)

These pumps are both triplex plunger pumps. They supply high pressure recycle process water to the dissolver quench in Area 02. Each pump was repacked once during the reporting period. During the September shutdown the drive end of A pump was rebuilt. The pressure control of the discharge of these pumps continued to be erratic and caused numerous head gasket failures.

D. Solvent Recovery (Area 04)

1. Operation June 25 through August 23, 1978

Startup of the new vacuum flash drum in the wash solvent preflash mode of operation had to be aborted on June 27 because an air leak into the bottoms line at the point where a tie-in for the Lummus unit was made caused the bottoms pumps to cavitate. By June 29 the new vacuum flash system was repaired and was back in service. The main operating problem encountered with this system through July was control of the vacuum in the range of 5-10 inches of mercury.

The light ends column reboiler developed a Dowtherm leak on August 1 causing 41 hours of lost production. The bundle was replaced with a spare and another bundle was placed on order.

The old vacuum flash system, which had been out of service since June 21 for maintenance, was returned to service June 25. This system had been shutdown to replace two collapsed sections of Dowtherm jacketed piping below the vacuum flash drum. Repair of this line also included installation of several inspection plugs for closer monitoring. An additional inspection plug was also installed above level control valve LCV-219A.

Based on inspection during the last shutdown, it was predicted that the wash solvent overhead air-fin cooler would require replacement in six months. Unfortunately, the prediction proved to be optimistic. The first indication of problems occurred during July. On July 4, and again on July 24, the wash solvent column had to be taken out of service to repair leaks in the overhead with weld metal on July 25. On August 15 a leak developed in one of the tube rolls. While rolling the leaking tube, several leaks developed in adjacent tubes. Eventually twenty tubes were rolled to keep the unit from leaking. On August 16 several new leaks developed in the tubes not previously rolled; therefore, the remaining 54 tubes were rolled and the unit was tested for leaks and returned to service. Two tubes began leaking August 17 after only several hours of operation. These tubes failed because they lacked sufficient strength to resist cracking when rerolled. (All tubes were stainless steel which is susceptible to cracking when work hardened.) Initially, the tubes that failed were replaced with plugs made from carbon steel tube material. One of these plugs, however, could not be rolled tight and required removal. Repairs were completed by threading the tube sheet and installing a 1" hollow bull plug. Repairs to the cooler accounted for 168 hours of lost production during the quarter.

The solvent fractionation section was back onstream August 19 but attempts to start Area 04 during the weekend of August 19 and 20 failed when the old vacuum flash preheater plugged with coke, apparently the result of high solids filter cake leg material settling in the vacuum feed flash accumulator and subsequently being charged to the preheater. After efforts to steam clean the coil were unsuccessful, x-rays were taken of the coils to determine the severity of the plugging. The x-rays confirmed other indications that the coil was plugged from the seventh coil through the twentieth coil. The seventh coil through the fourteenth coil were full of coked material. Although the remaining coils were not full, each was severely restricted. Difficulties experienced in the initial attempts to hydroblast the coil indicated the coil would have to be cut in several places. Shutdown activities to prepare the plant for SRC II operation began August 23 after it became obvious that decoking of the heater coil would extend beyond September 1.

2. Turnaround Maintenance

Cutting and hydroblasting of the old vacuum flash preheater coil began August 25. Cuts were made in the seventh, ninth, eleventh, fifteenth, seventeenth, eighteenth, and nineteenth coils. By September 1 the coil which had been cleaned and x-rayed was ready for welding. By September 11 all preheater coil welding and stress relieving was complete. After repair of the heater refractory was completed, steam flow was established through the coil to remove any residual debris and for refractory dryout.

Two new Dowtherm tempering coolers were installed on the downlegs of the new and old vacuum flash drums in order to match the downleg temperature to the drum flash zone temperature. This change should result in smoother operation by minimizing vaporization in the downleg. The level control valve for the new vacuum flash drum was changed from a 1" Fisher Vee Ball to a 2" Fisher Vee Ball to make it full line size to prevent solids bridging. Eventually the 1" Fisher Globe Valve in the old vacuum flash drum will be changed to a 2" Fisher Vee Ball valve. For greater safety, the control valve piping for both systems was redesigned to allow rodding of the valve from the upstream side.

Other maintenance work during the shutdown included:

- Installation of a coke trap in the new vacuum flash drum.
- Replacement of the badly corroded wash solvent column overhead cooler with a new one.
- Installation of upgraded Dowtherm valve jackets on the new vacuum flash drum system. The new jackets are designed to withstand pressures of 350 psig at 800°F.
- Installation of new demister mats in both vacuum flash drums.

3. Operation September 21 through September 24, 1978

The new vacuum flash system was brought on-line September 21 in the SRC II mode, but was shut down September 22 because of problems in Area 02. At the end of the reporting period, both vacuum flash systems and the columns were on circulation awaiting the resumption of slurry feed.

4. Pump Performance

a. Old Vacuum Flash Drum Bottoms Pumps (04D56083, 04D56084)

In July a hole developed in the suction side of A pump casing. Four new seals were required on these pumps during the reporting period.

b. New Vacuum Flash Drum Bottoms Pumps (04D56325, 04D56326)

These pumps required six new seals during the reporting period. This is due to the fact that the new vacuum flash system was operating in a pre-flash mode and the pumps were operating continuously. Under normal operation they operate only on startup and shutdown of the system.

c. Other Pumps

The wash solvent column bottoms pump required two new seals during the reporting period, and the vacuum flash accumulator pump required three.

E. Gas Recovery and Recompression (Area 05)

1. Operation June 25 through September 24, 1978

Valve failure continued to be a problem with the Chicago pneumatic compressors. In June the fresh hydrogen and A recycle compressors were each repaired three times because of valve failure. One of these failures resulted in a 1-1/2 hour production curtailment. The valves on A recycle compressor required repair two times in July. These repairs were made with no loss in production. Repacking of A recycle compressor as well as the repair of the valves and rings of the fresh hydrogen compressor on September 24 delayed startup 16 hours.

In September a cooling water tempering device was installed on the inlet of the fresh hydrogen compressor. Its purpose is to ensure that condensation does not form in the gas inlet chamber of the compressor by maintaining the inlet chamber at a higher temperature than the incoming gas. Elimination of condensate formation is expected to decrease the frequency of intake valve failures.

B recycle compressor made by Ingersoll Rand was shutdown twice in June; once for replacement of rings and once for repacking. Maintenance repacked it again in July and at the same time replaced the valves.

Area operation was upset on July 26 and again on August 8 because of electrical power failures.

The Naphtha unit, which is not required for SRC I mode operation, was recommissioned for the SRC II run on September 21. No significant problems were encountered, although #2 and #3 circulation pumps did require repair.

F. Product Solidification and Storage (Area 08)

1. Operation June 24 through August 23

The Sandvik belt operated routinely throughout the reporting period with only minor tracking problems.

A request from DOE for 400 tons of SRC I high ash solid product was filled during the period of July 9 to 24.

2. Turnaround Maintenance

The interlock system on both the old and new vacuum flash level control valves was modified to allow the valves to operate independently of the Sandvik belt. This modification will permit continued operation of either flash drum if it becomes necessary to remove the Sandvik belt from operation for cleaning or maintenance.

G. Waste Treatment (Area 09.1)

1. Operation June 25 through September 24, 1978

Operation of the waste treatment area was stable with the exception of periods of higher than normal oil and solids loading during vessel clean-out. A program was initiated to anticipate such periods and increase chemical injections to the reactivator accordingly. Minor equipment problems were experienced with the alum injection system, the reactivator drive unit and the reactivator feed pump.

Maintenance modified the piping on the charcoal filters to permit series or parallel operation of these filters. The charcoal filters were used as necessary to meet plant effluent guidelines.

While the plant was shut down, ammonia and wash solvent were added to the Oxycontact Unit as supplement to the feed in order to maintain biomass.

H. Cooling Water System (Area 09.3)

1. Operation June 25 through September 24, 1978

On August 22 the plant lost its raw water supply for a short time while the Fort Lewis Military Reservation made repairs to its water system. During this outage the level in the cooling tower dropped about five inches, indicating that the cooling tower would be one of the first places an extended water outage would become a major problem.

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

1. Inert Gas

The following items were completed during the pre-SRC II shutdown:

- A leak in the #1 inert gas compressor interstage cooler was repaired.
- The inert gas filter element was replaced.
- The DeOxo unit catalyst was screened to remove fines.
- A gas leak around the outside flange of the burner plate on the stripper was repaired.

2. Hydrogen Unit

On July 5 another leak developed on the outlet flange of reformer tube 1E. Steam spargers were installed and are in service to minimize the possibility of fire. To date this leak and a previous leak have not increased in severity. Repairs will be made at the next unit shutdown.

3. Stretford Unit

The unit operated sporadically until July 6 when it was shut down and drained for modifications which are designed to improve operability. The major change was replacement of the air spargers in the oxidizer to improve sulfur frothing. Other work included increasing the size of the sulfur slurry pump discharge line from 1" to 2" and repairing the circulation loop controllers. The unit was returned to service July 19 and remained in operation until August 23 when the plant was shut down. The new air spargers and careful process monitoring have resulted in substantially improved sulfur recovery. At present approximately 95% of the hydrogen sulfide in the feed gas is recovered as sulfur.

J. Dowtherm System (Area 09.8)

1. Operation June 25 through September 24, 1978

A six hour coal curtailment occurred August 17 when a pinhole leak developed on the line into the bottom of the Dowtherm heater. The leak was caused by a steam tracing leak impinging on the line. When the plant was shut down August 23, the Dowtherm unit was shut down to replace the heater burner refractory block and gas spider, which were cracked and damaged. After refractory dry out was complete, the unit was returned to service September 9. The main problems with the Dowtherm system continue to include Dowtherm leaks to the process and intermittent flame-outs of the Dowtherm heater.

III. PROCESS ANALYTICAL DATA

The plant operated in the SRC I mode during July and August using Kentucky Nos. 9 and 14 coal from the P&M Colonial Mine. During the month of September, when the plant was down for turnaround maintenance, the laboratory continued to monitor the operation of the waste treatment area.

The average analyses of the raw coal used during July and August are shown in Table 2.

TABLE 2

Average Raw Coal Analyses, Wt %

	July	August
Ash	8.56	8.48
Moisture	6.01	6.63

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

TABLE 3

Average Dried, Pulverized Coal Analyses, Wt %

	July	August
Carbon	71.47	70.37
Hydrogen	5.37	5.16
Nitrogen	1.50	1.47
Sulfur	3.28	3.61
Oxygen (by difference)	8.81	9.00
Ash	9.16	9.78
Moisture	0.41	0.61

TABLE 4

Average Analyses of Forms of Sulfur, Wt %

	July	August
Pyritic Sulfur	1.61	1.99
Sulfate Sulfur	0.19	0.30
Organic Sulfur	1.44	1.36
Total Sulfur	3.24	3.65

TABLE 5

Average Sieve Analyses of Dried, Pulverized Coal, Wt %

	July	August
- 40 mesh	99.51	99.31
- 100 mesh	97.84	97.48
- 140 mesh	92.97	91.95
- 200 mesh	81.02	78.63

The average analyses of the stripper bottoms in this quarter are shown in Table 6.

TABLE 6

Average Analyses of Stripper Bottoms, Wt %

	July	August
Water	0.32	0.28
Light Oil	0.13	0.05
Wash Solvent	0.89	0.03
Process Solvent	64.49	63.34
Vacuum Bottoms (PI included)	27.16	28.44
Pyridine Insolubles (as received)	7.01	7.86
Ash in Pyridine Insolubles (PI)	62.31	63.71

Laboratory determined coal conversion on a moisture-ash-free (MAF) coal basis are reported by month in Table 7.

TABLE 7

Average MAF Coal Conversion, Wt %

July	94.1
August	94.3

Typical analyses of the laboratory vacuum bottoms obtained from the work-up of the recycle stripper bottoms are shown in Table 8.

TABLE 8

Typical Analyses of Laboratory SRC I Vacuum Bottoms, Wt %

	July	August
Carbon	87.77	86.92
Hydrogen	5.82	5.75
Nitrogen	2.05	2.10
Sulfur	0.84	0.75
Oxygen (by difference)	3.34	4.32
Ash	0.18	0.16
Fusion Point	338°F	341°F

The average analyses of the recycle process water (RPWT) oil and water phases for this quarter are reported in Tables 9 and 10.

TABLE 9

Average Analyses of RPWT Oil Phase, Wt %

	July	August
Light Oil	20	15
Wash Solvent	25	33
Process Solvent	55	52
Specific Gravity @ 60/60°F	0.952	0.980

TABLE 10

Average Analyses of RPWT Water Phase, Wt %

	July	August
Phenols	0.49	0.73
Nitrogen	0.97	0.64
Sulfur	0.76	0.52

Typical fractional analyses of liquid products, based on ASTM D-86 distillation data, are reported in Table 11.

TABLE 11

Typical Fractional Analyses of Pilot Plant Liquid Products, Vol %

Laboratory Distillation Fractions	Light Oil	Wash Solvent	Process Solvent
July			
IBP - 380°F	95	7	0
380 - 480°F	5	93	0
480 - 850°F	0	0	100
August			
IBP - 380°F	88	2	0
380 - 480°F	12	96	2
480 - 850°F	0	2	98

The average analyses of the pilot plant vacuum bottoms produced during this quarter are reported in Table 12.

TABLE 12

Average Analyses of Pilot Plant Vacuum Bottoms, Wt %

	July	August
Carbon	76.75	78.85
Hydrogen	5.15	5.09
Nitrogen	1.72	1.78
Sulfur	2.07	1.94
Oxygen (by difference)	2.22	1.06
Ash	12.09*	11.28*
Fusion Point (Gradient Bar Method)	332°F	353°F

* Even though the plant was in the SRC I mode of operation, the feed to the vacuum flash drum had not been totally filtered. This is the reason for the high ash values obtained on the plant vacuum bottoms. The previously reported laboratory vacuum bottoms were produced from filtered material and predict what a plant filtered product would have yielded.

The analyses from the waste treatment units reflects that excellent control was achieved throughout the quarter. Typical analyses of waste water streams are shown in Table 13.

TABLE 13

Process Waste Treatment Analyses

	Bio Unit Feed	Bio Unit Effluent	Plant Effluent (Composite)
July			
pH	6.4	6.2	6.6
Total Suspended Solids, ppm	489	49	4
Phenol, ppm	99	3.2	0.3
Chemical Oxygen Demand, ppm	5111	487	39
Biological Oxygen Demand, ppm	219	49	8
August			
pH	7.1	6.9	7.0
Total Suspended Solids, ppm	152	67	17
Phenol, ppm	74	3.9	0.4
Chemical Oxygen Demand, ppm	1929	983	38
Biological Oxygen Demand, ppm	95	128	3

September	Bio-Unit Feed	Bio Unit Effluent	Plant Effluent (Composite)
pH	7.0	7.1	7.0
Total Suspended Solids, ppm	55	25	2.8
Phenols, ppm	40	2.5	0.1
Chemical Oxygen Demand, ppm	309	91	49
Biological Oxygen Demand, ppm	20	9	11

During the quarter, plant effluent had an average flow of 307,560 GPD with a minimum of 207,360 GPD and a maximum of 547,200 GPD. Phenol content in effluent water averaged 0.4 ppm, with a minimum of 0.04 ppm and a maximum of 1.0 ppm. The oil and grease concentration did not exceed 9.7 ppm and no sheen was visible.

IV. PILOT PLANT SPECIAL STUDIES

A. Filter C Test Program

Hydraulic capacity tests on the experimental Filter C were completed in the second quarter 1978 and in the third quarter filtration experiments were started during processing operations on Kentucky Nos. 9 and 14 coal in the SRC I mode. Eight tests were attempted but none were satisfactory because of various filter design and operating problems. A brief summary of the filtration conditions used in the experimental runs and comments on each run are shown in Table 14. A complete discussion of the filtering operations is given in Section II-C of this report.

Filtration rates and operating conditions during Test 1A are shown in Figure 3. The filter appeared to operate satisfactorily and produced filtrate of acceptable clarity until 0100 on June 30 when an abrupt reduction in knife advance rate from 12 mil/min to 2 mil/min was followed by increased solids in the filtrate and finally total loss of filtration. The loss of filtrate clarity in the latter stages of Test 1A precludes the use of these data for filter evaluation. Various mechanical operational problems also limit the value of the early data from this run, although Figure 3 does show qualitatively that increased knife advance rates resulted in increased filtration rates which is in agreement with filtration theory and previous coal slurry filtration results.

The filter was repaired after Test 1A, and Test 1B was started at the same conditions to establish reliable baseline filtration rates. The filtration data for Test 1B are shown in Figure 4. Pyridine insolubles show that filtrate clarity was poor at the beginning of this run and, although clarity improved as the run progressed, it was still unsatisfactory at the end. Operational problems and poor filtrate clarity make the results of Test 1B of limited value also.

Six additional filtration tests were completed during July and August, but operational problems caused by poor performance of the

TABLE 14

FILTER C TESTS

DATE	TEST NO.	FILTRATION CONDITIONS						COMMENTS
		BASECOAT	PRECOAT	TEMP. °F	PSID	DRUM SPEED RPM	KNIFE ADVANCE RATE	
6/29-6/30	1A	Celite 550	Standard Supercel	600	95	5	Vary	Precoat applied 24 hours before filtration. Tear in screen resulted in poor filtrate clarity.
7/20-7/21	1B	Celite 550	Standard Supercel	600	95	5	Vary	Filtration was stopped twice during the run due to mechanical problems. Filtrate clarity was unacceptable to produce on-spec SRC.
7/27-7/28	1C	Celite 550	Standard Supercel	600	95	5	Vary	Had basecoating/precoating difficulties. Clarity was poor. Found filter tub inspection door open after completion of test.
7/31-8/1	1D	Celite 550	Standard Supercel	600	95	5	Vary	Very high solids concentration in filtrate. Found tear in screen when filter was opened after test.
8/4-8/5	2A	Celite 503	Celite 512	600	95	5	Vary	Filtrate clarity poor.
8/5-8/6	2B	Celite 503	Celite 512	600	95	5	Vary	Very high solids concentration in filtrate. Found tear in screen when filter was opened. Tear due to deflection of screen support.
8/11-8/12	3A	Celite 503	Celite 512	600	70	5	Vary	Poor filtrate clarity. Tried to improve clarity by adding precoat to feed. Clarity remained poor.
8/13	3B	Recoat over Run 3A	Standard Supercel	600	70	5	Vary	Attempted to recoat over old basecoat. Precoat mix was poor resulting in a lumpy precoat cake. Had very high solids concentration in filtrate. Found major screen tear when filter was opened.

FIGURE 3

FILTRATION EXPERIMENT 1A.

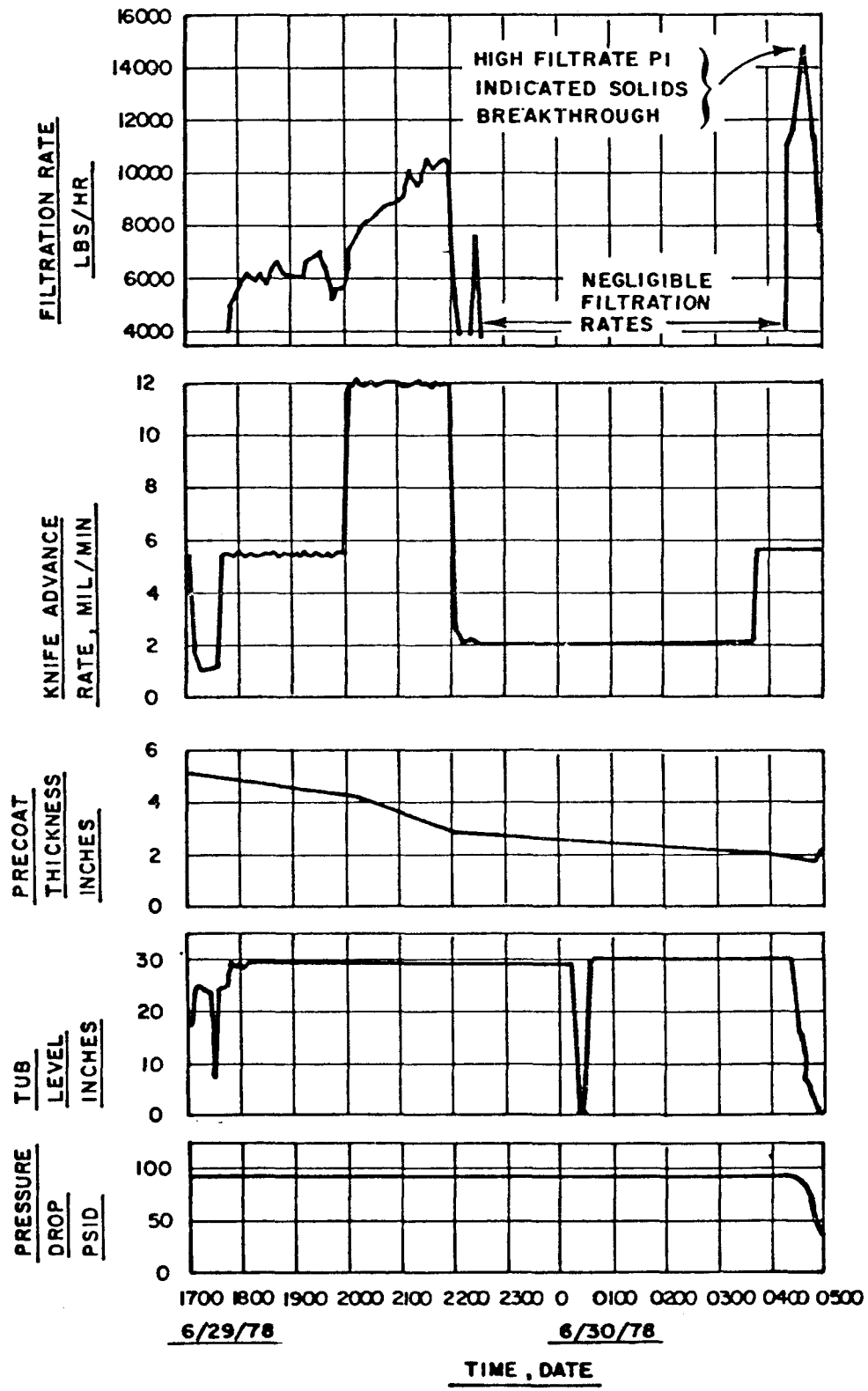
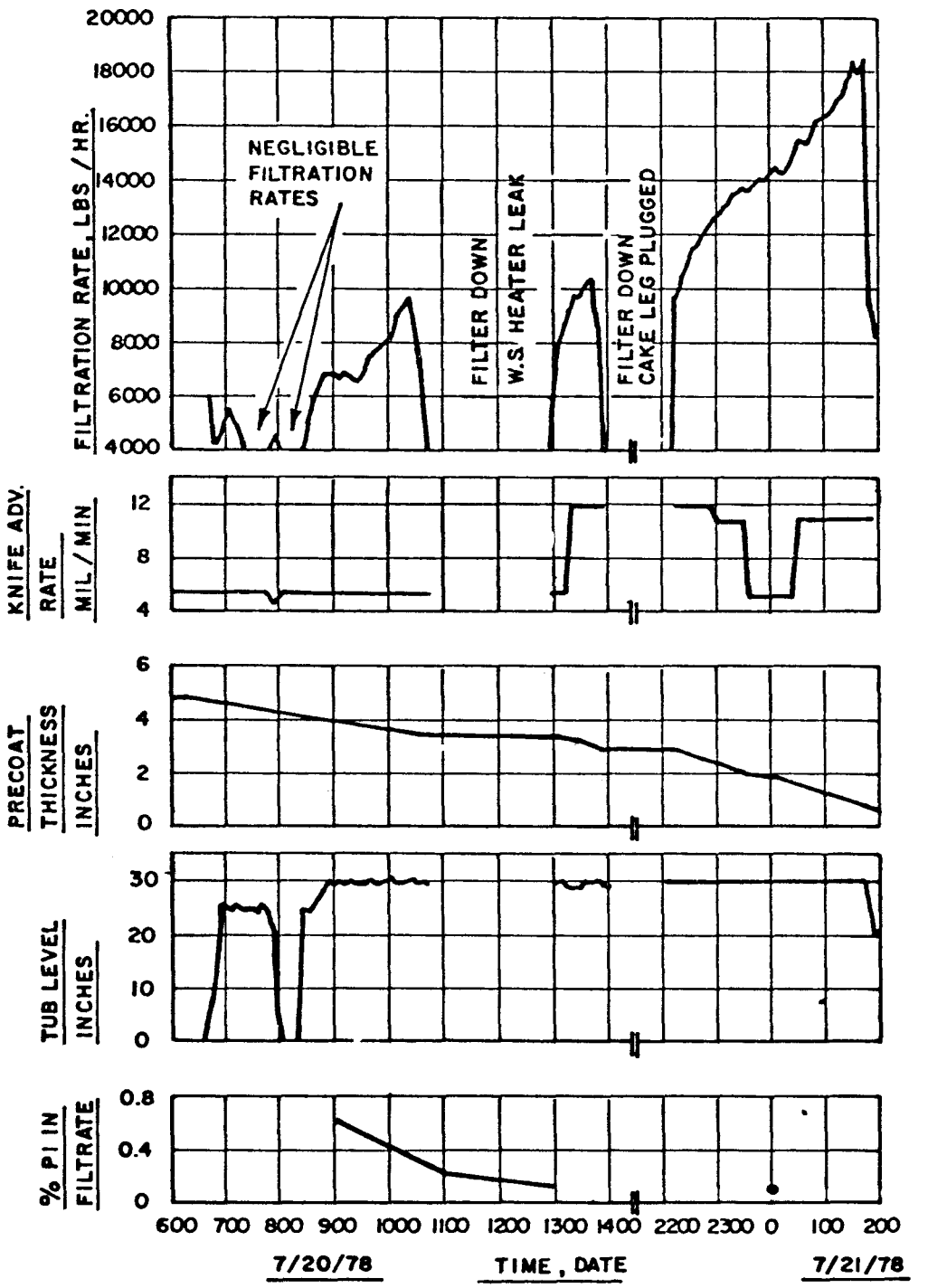


FIGURE 4

FILTRATION EXPERIMENT 1B.



screen support resulted in unsatisfactory filtrate clarity in all cases. Measurements of filtrate clarity, expressed as weight percent pyridine insolubles (solids) in the filtrate, are shown in Figure 5 for these six tests. In the past it has been found that 0.05% pyridine insolubles represents the maximum concentration at which the SRC ash specification (0.16%) can be achieved. The results in Figure 5 indicate that this limit was exceeded in all six tests.

The presence of pyridine insoluble material in the filtrate suggests that some filter feed material bypassed the filter, therefore the measured filtrate rates during these tests are biased high. In general, filtrate rates ranged from 150-250 pounds of filtrate per hour per square foot of filter surface area.

B. SRC I Solvent Inventory and Quality

Studies to determine the effects of raw solvent additions to the process stream on solvent yields and on solvent quality were conducted in the months of June, July and August. To derive the desired data, weekly inventory balances of wash solvent, process solvent and raw solvent were made by measuring the volume and analyzing the contents of each plant vessel. Pertinent data and calculations based on these data are given graphically in Figure 6. The IR ratio (a measure of the transferrable hydrogen content) of the process solvent fraction was measured daily. These data are graphically presented in Figure 7.

The results shown in Figure 6 indicate a relatively steady rate of increase in wash solvent inventory during the entire period. The net increase from June 12 to August 21 is 487 barrels, which represents a yield on moisture-free coal processed during the period of 4.9% by weight. Since the increases in wash solvent inventory occur independently of raw solvent additions to process, it appears that little wash solvent was directly produced from raw solvent. In support of this, the calculated yields of wash solvent (4.9% M.F. coal) is in accord with previous yield measurements made by material balance techniques[1].

In contrast to the observations made concerning the inventory of wash solvent, the process solvent inventory showed increases only when raw solvent was being added to the reaction system. When the addition of raw solvent was discontinued, the inventory of process solvent steadily decreased. This indicates that the coal-derived process solvent balance was negative during the period.

The process solvent quality data presented in Figure 7 show a gradual increase in transferrable hydrogen level (increasing IR ratio) during the period. This increasing trend occurred in spite of sharp reductions in IR ratio which accompany each period of raw solvent addition. It appears that the depression in transferrable hydrogen level which results from raw solvent addition is more substantial at lower levels of initial IR ratio. In addition, Figure 7 indicates that the recovery in IR ratio which follows raw solvent addition occurs more rapidly at high initial levels of transferrable hydrogen.

FIGURE 5

FILTRATE CLARITY (WT. % PI) FOR FILTER C TEST RUNS

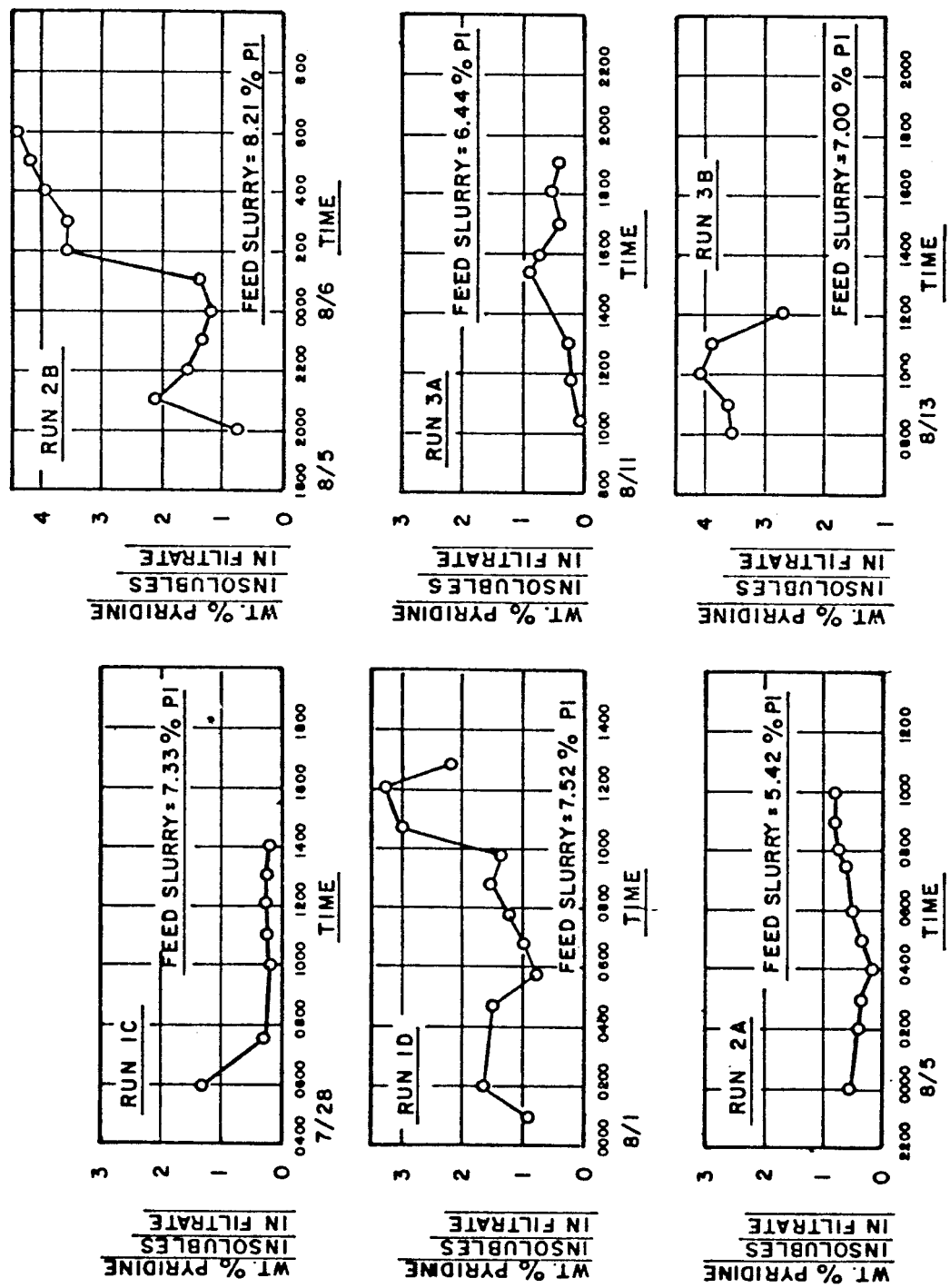


FIGURE 6

CUMULATIVE INVENTORY CHANGE - SRC I LIQUIDS

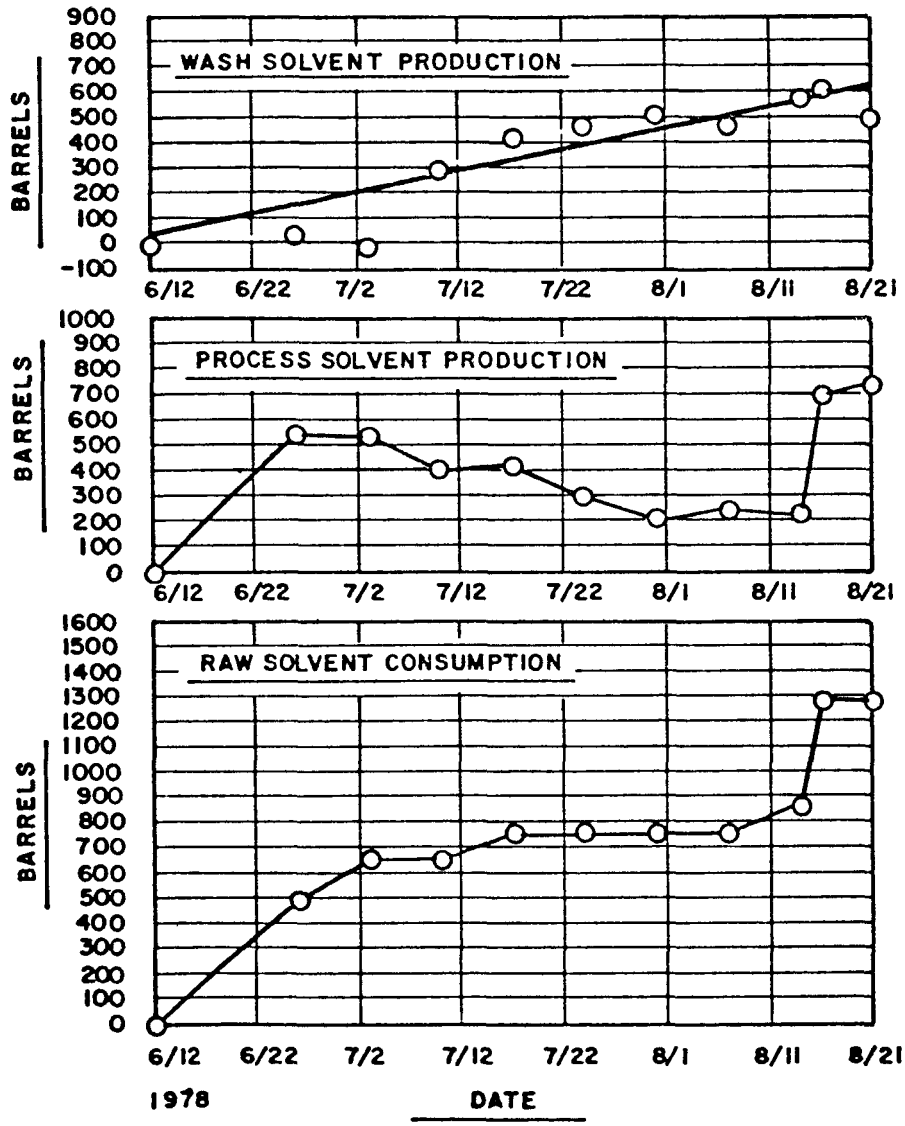
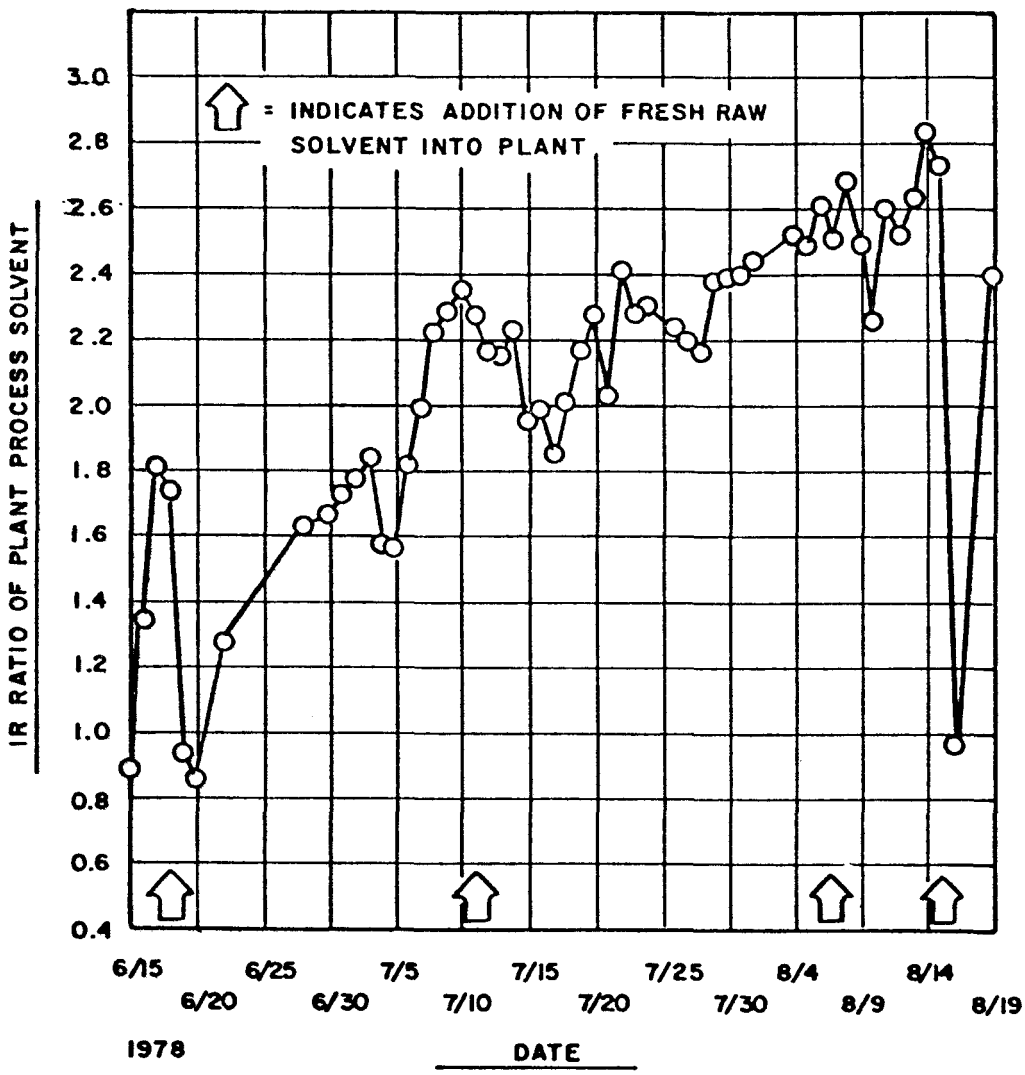


FIGURE 7

PROCESS SOLVENT IR RATIO DURING SRC I OPERATIONS



C. Dissolver Solids Sample Analysis

A system was designed and constructed to take samples from the bottom of A dissolver during operation. A simplified schematic diagram of the system is shown in Figure 8. The sample point is about 15 inches above the inlet to the reactor. Hydrogen and flush solvent purges are used to reduce plugging problems, and multiple manually operated valves, as well as a remotely controlled valve, are used to ensure safety. In operation the sample was taken through the lower cylinder and into the upper cylinder until pressure was equalized. The cylinders were then isolated and taken to the laboratory for analysis. The contents of the lower cylinder were considered representative of the dissolver contents.

Samples were taken on July 11 and July 19, 1978 during well lined-out SRC I operation. The total gas feed rate and dissolver pressure were significantly higher during the first sampling period as shown by the reaction condition data in Table 15. For analysis, the gas phase was vented and collected and the slurry phase was displaced from the cylinder using inert gas. The gas volume and slurry weights were determined and are shown in Table 16.

1. Analytical Results

It was not possible to analyze the gas phase because of contamination with other gases during sampling and pressure letdown and because of problems with the laboratory chromatograph. It was also not possible to filter the slurry phase at laboratory conditions, and the analyses for pyridine insolubles content, ash and distillation were carried out on the as-received samples.

Ash, pyridine insolubles, and iron analyses of the dissolver samples are presented in Table 17 along with similar analyses from the stripper bottoms sample of the same day. It should be noted that some difficulty was experienced in reproducing ash determinations on the as-received samples. This would indicate that the samples were somewhat heterogeneous despite careful mixing. In addition, wide discrepancies were found in pyridine solubility determinations for the July 19 samples which were apparently due to solids in this sample bypassing the filter membrane on some analyses. The data in Table 17 show that there is appreciably more insoluble and mineral matter in the material taken from the dissolver than in the stripper bottoms. This may be taken as evidence that there is significant settling of minerals within the dissolver in SRC I operation.

Table 18 contains data from vacuum distillation of the dissolver samples together with data for stripper bottoms samples from the same day for comparison. Analyses of the process solvent fractions obtained from distillation are shown in Table 19.

Since extraction with specific solvents is frequently used to characterize coal liquefaction products, the dissolver samples were analyzed by extraction with n-pentane, benzene, and pyridine. The extract data are shown in Table 20.

FIGURE 8

BOTTOM DISSOLVER SAMPLING INSTALLATION SCHEMATIC

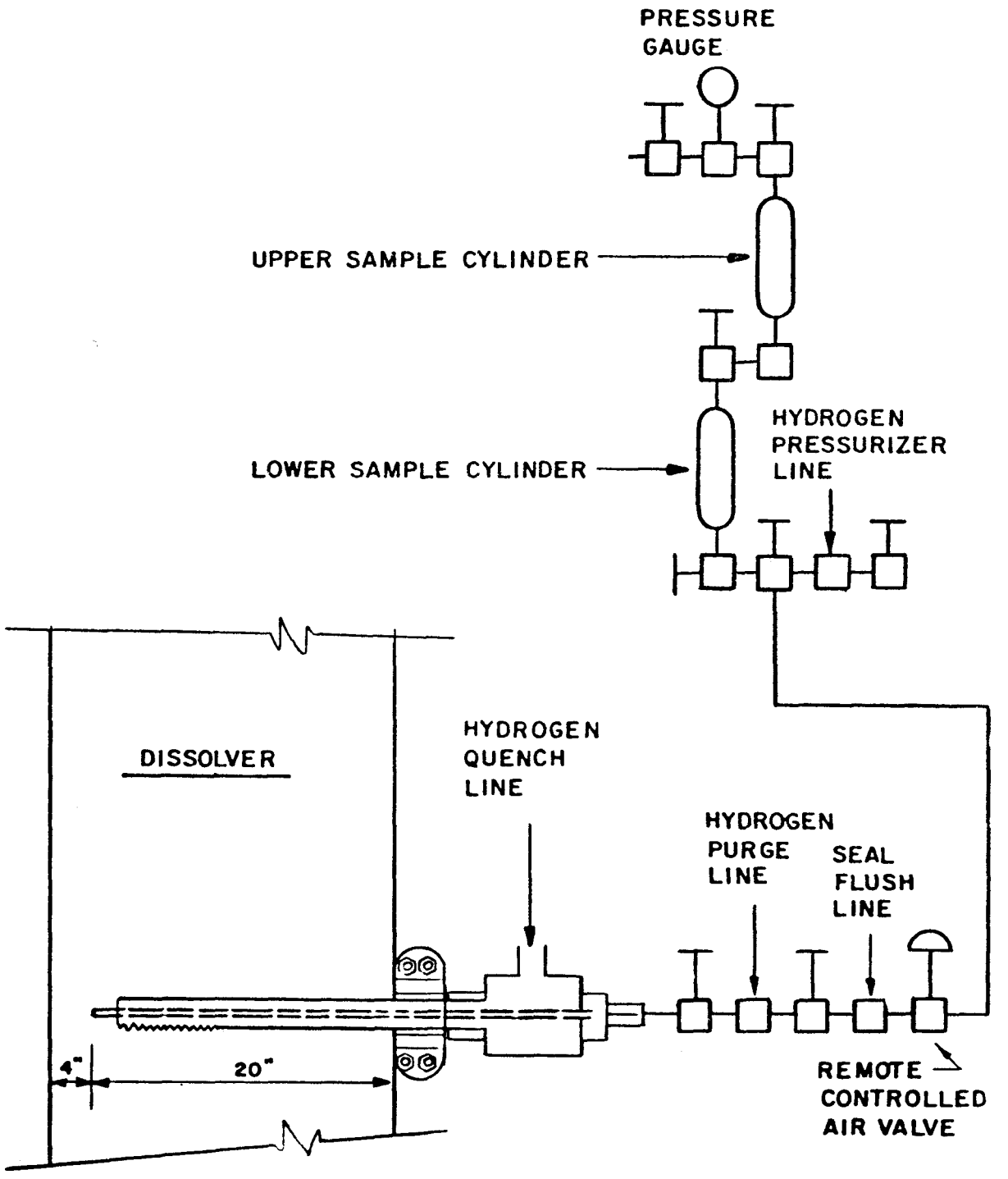


TABLE 15

REACTION CONDITIONS AT THE TIME OF DISSOLVER SAMPLING

SAMPLE DATE	7/11/78	7/19/78
Coal feed rate, lb/hr		
Raw	3774	3816
Dehumidified	3559	3580
Ash in Dehumidified feed coal, %	8.71	8.83
Solvent feed rate, lb/hr	5310	5296
Total gas feed rate *		
lb/hr	407	351
SCFH	44,200	40,000
Dissolver Pressure, psig	1812	1536
Temperature **, °F	816	827

*Includes instrument purges

**Measured approximately 3 feet above sample point near the wall of the dissolver.

TABLE 16

CONTENTS OF DISSOLVER SAMPLE CYLINDERS

Date of Sample	7/11/78	7/19/78
Gas Volume (liters at STP)		
Upper cylinder	34.2	29 (8.91 M.W.*)
Lower cylinder	5.7	4 (17.27 M.W.*)
Slurry Weight (gms)		
Upper cylinder	47.3	678.3
Lower cylinder	920.1	1308.8

*Obtained by the method described in ERDA R&D Report No. 53 Interim Report No. 7, FE-496-3, pages 74 and 75.

TABLE 17

PROXIMATE ANALYSIS OF INSOLUBLE MATERIAL

FROM DISSOLVER SAMPLES

Date	7/11/78	7/19/78
Dissolver Samples		
Pyridine Insolubles (Wt %)	32.9	51.1
Ash (Wt %)	23.3	38.5
Iron* (Wt %)	3.1	12.7
Stripper Bottoms, Same Day		
Pyridine Insolubles (Wt %)	6.0	6.4
Ash (Wt %)	3.8	4.2

* Values presented for iron are based on total sample, but were calculated from the average of iron determinations made on the ash.

TABLE 18

VACUUM DISTILLATION OF DISSOLVER SAMPLE

(Lower Cylinder)

Fraction (wt %)	Boiling Range (°F)	7/11/78	7/19/78
Water		1.51	0.29
Light Oil	< 380	1.24	0.06
Wash Solvent	380 - 480	0.10	0.06
Process Solvent	480 - 850	39.92	27.21
SRC	> 850	24.26	21.27
Pyridine Insolubles		32.90	51.10
Comparable Stripper Bottoms (wt %)			
Water		0.17	0.13
Light Oil		0.0	0.0
Wash Solvent		0.27	0.28
Process Solvent		64.07	64.86
SRC		22.86	21.97
Pyridine Insolubles		6.00	6.40

TABLE 19

ANALYSIS OF PROCESS SOLVENT FRACTION

DATE	7/11/78	7/19/78
Dissolver A Sample		
% Carbon	89.41	87.80
% Hydrogen	7.35	6.57
% Nitrogen	0.83	0.81
% Sulfur	0.55	0.56
IR Ratio	2.77	2.23
Comparable Stripper Bottoms (Process Solvent Fraction)		
% Carbon	*	87.22
% Hydrogen	*	6.18
% Nitrogen	0.81	0.93
% Sulfur	0.54	0.52
IR Ratio	2.28	2.68

* Not obtained because of instrument malfunction.

TABLE 20

SOLVENT EXTRACTION OF DISSOLVER SAMPLES

Fraction Name	Solvent	7/11/78 Sample	7/19/78 Sample
		Wt %	Wt %
Asphaltol	Benzene Insoluble Pyridine Soluble	14.4	8.7
Asphaltene	Benzene Soluble Pentane Insoluble	9.1	6.5
Maltene	Pentane Soluble	44.3	33.7

Density measurements were made on the dissolver samples at several temperatures using a calibrated pycnometer submersed in a constant temperature bath. These data are shown in Table 21. Also shown in Table 21 are viscosity data taken with a Brookfield Model HAT-200 viscometer using spindle No. SC4-21.

2. Particle Size Analysis

One of the more important reasons for obtaining these dissolver samples is to determine the size distribution of mineral matter particles found in the reactor under different conditions. In an effort to characterize this distribution, aliquots of each dissolver sample (7/11/78 and 7/19/78) were prepared and sent to several outside laboratories for particle size distribution (PSD) analysis. Three different types of particle size analysis were compared using the first dissolver sample.

a. PSD Analysis of the 7/11/78 Sample

As soon as the as-received sample was released from the pressure cylinder, it was stirred throughly, but not violently, by hand for several minutes. Then several aliquots of approximately 5 grams were removed to a centrifuge tube of about 60 cc capacity. The tubes were each filled with pyridine and then placed individually in an ultrasonic bath. After approximately five minutes of ultrasonic agitation, the tubes were centrifuged for one hour at 1800 rpm. The supernatant liquid was carefully removed from each tube by aspiration. This treatment was repeated for each tube until the supernatant was clear and nearly colorless. The first time the tubes were aspirated the supernatant from eight tubes was combined, evaporated and ignited. The ignition residue from this was 46 mg indicating satisfactory recovery of ash by centrifugation. The last two dispersion/centrifugation cycles were done with methanol rather than pyridine. The resulting methanol slurries were divided into four groups which were sent out for analysis by three different techniques, as described below.

i. X-ray Sedimentation

This work was provided by Micron Data Laboratories of Mission Viejo, California. X-ray sedimentation provides a direct measurement of mass fraction of particles as a function of equivalent Stokes diameter. Equivalent Stokes diameter is defined as that diameter which satisfies Stoke's-law for the system in question.

The diameter of a particle as determined by sedimentation in the Stoke's-law region is given by:

$$D = \sqrt{\frac{18\eta v}{(\rho - \rho_o)g}} \qquad (1)$$

TABLE 21

DENSITY AND VISCOSITY OF DISSOLVER SAMPLES

DENSITY	Temperature °F	Density, gm/cm^3
7/11/78 Sample	237	1.344
	247	1.305
	254	1.312
	263	1.299
7/19/78 Sample	275	1.520
	305	1.508
	336	1.469

VISCOSITY	Temperature °F	Shear Rate sec^{-1}	Viscosity cp
7/11/78 Sample	210	93	716
	275	93	144
	275	186	93.5
7/19/78 Sample	250	93	489
	275	93	632
	350	93	445

Where: D is the diameter of a spherical particle
v is the equilibrium sedimentation velocity (terminal settling velocity)
η is the fluid medium viscosity
ρ is the particle density
ρ_o is the fluid density
g is the gravitational acceleration

In practice the particles are suspended in a sedimentation fluid whose viscosity and density are precisely known. The particles are suspended by circulation through the instrument in turbulent motion. Under those conditions the instrument is set to 0% transmittance. One hundred percent transmittance has been previously set with the sedimentation fluid alone. The circulation is then abruptly stopped and the unit continuously measures the x-ray extinction as a function of time and vertical position in the measuring cell. These data are automatically converted to a cumulative mass distribution with the aid of a microprocessor using appropriate Stoke's-law parameters.

Figure 9 shows the particle size distributions obtained from two separate aliquots (A and B) of the 7/11/78 sample. These distributions are in roughly the same range but indicate substantially different median particle diameters (8μm for A and 21.5μm for B). They both, however, suggest a polymodal distribution as the curves are not smooth with a single inflection. For comparison, the pyridine insoluble material, which was obtained by boiling in pyridine and filtering, was also analyzed. This distribution is shown in Figure 9 as curve C. It can be seen that this curve is somewhat intermediate between A and B. The implications of Figure 9 are that filtration is as effective as centrifugation for particle recovery for this sample and that analyses are not perfectly reproducible either due to sampling error or sample work up and analysis. Micron Data Laboratories asserts that their method is reproducible for a given sample to less than 1%. If this is true, then Figure 9 may indicate a measure of non-homogeneity in the original sample.

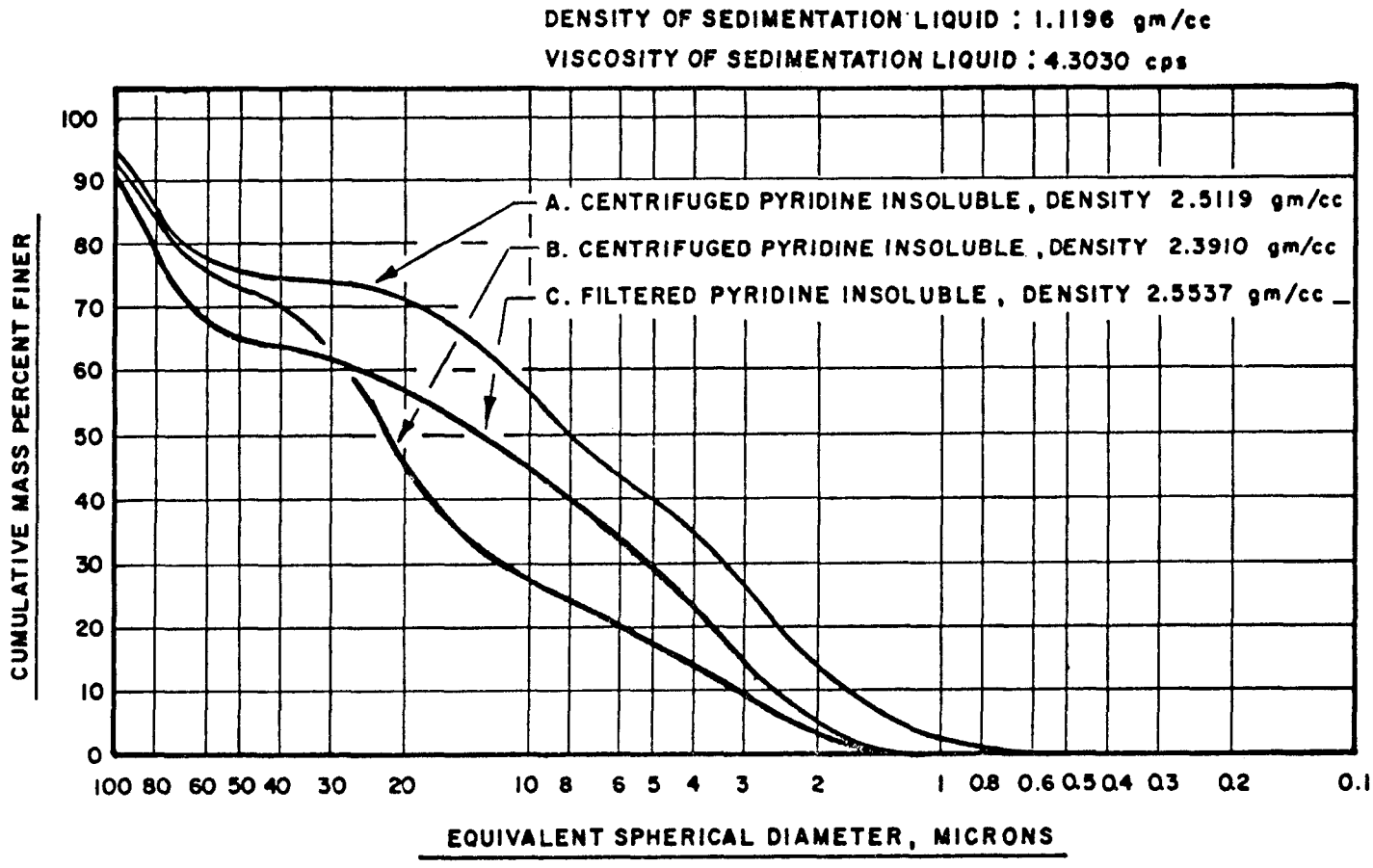
ii. Scanning Electron Microscopy

Another portion of solids from the 7/11/78 sample was sent to Gulf Science and Technology Company for PSD analysis by scanning electron microscopy. In this computer assisted technique the particles are dispersed and mounted on a two dimensional surface. The surface is then viewed in a scanning electron microscope. The scan data from certain frames selected by the operator are fed to a computer which calculates and stores diameters measured along four axes which pass roughly

FIGURE 9

PARTICLE SIZE DISTRIBUTION BY X-RAY SEDIMENTATION

DISSOLVER SAMPLE 7/11/78



through the center of the projected image of each particle. In this manner a distribution can be computed showing the relative frequency of particles as a function of average diameter.

The distribution is prepared by dividing the range of measured particle diameters into some number, say n, of equal intervals and counting the number of particles to be found within each interval. Thus, the relative frequency distribution is approximated by the equation:

$$f(\bar{D}_i) \simeq \frac{X_i}{\sum_{i=1}^{n} X_i} \qquad (2)$$

where: $f(\bar{D}_i)$ is the relative frequency (number % ÷ 100) of particles within an interval i, whose mean diameter is $\bar{D}i$.

$\bar{D}_i$ is the mean of the diameters representing the upper and lower boundaries of the ith interval.

X_i is the number of particles whose average diameter falls within the ith interval.

n is the number of equal intervals in the range.

The distribution of interest, however, is that which relates the volume fraction or mass fraction to the average particle diameter. For this it is usually assumed that for a collection of similar particles the volume of any particle may be given by:

$$V = k_a D^3 \qquad (3)$$

where: V is the volume of the particle

D is the average diameter of the particle

k_a is a constant for all particles in the collection

Since equation (3) indicates the volume contribution of a class of particles varies as the cube of the diameter and since the largest particles are usually fewest in number, it can be seen that a significant potential for error exists with this method. This error is typically manifested in the volume distribution as a skewness toward the larger particles.

It should also be noted that in the process of mounting the particles on a two dimensional surface, these particles will tend to come to rest in a "lowest potential energy" configuration. This may result in a bias in the measured average diameter.

A volume frequency distribution may be calculated by the following equation:

$$V(\bar{D}_i) = \frac{k_a X_i \bar{D}_i^3}{\sum_{i=1}^{n} \left(k_a X_i \bar{D}_i^3\right)} = \frac{k_a X_i \bar{D}_i^3}{k_a \cdot \sum_{i=1}^{n} \left(X_i \bar{D}_i^3\right)} = \frac{X_i \bar{D}_i^3}{\sum_{i=1}^{n} \left(X_i \bar{D}_i^3\right)} \quad (4)$$

where: $V(\bar{D}_i)$ is the volume fraction assigned to the ith interval whose mean diameter is $\bar{D}_i$.

$\bar{D}_i, X_i, k_a$ are defined as above.

In Figure 10 the cumulative number distribution and cumulative volume distribution are presented as obtained for the 7/11/78 dissolver sample by applying equations (2) and (4) respectively to the scanning electron microscope data. In this case the particle densities are assumed to be constant and thus volume and mass distributions are directly comparable. It is seen that the volume (or mass) distribution of Figure 10 is similar to the x-ray sedimentation distributions given in Figure 9.

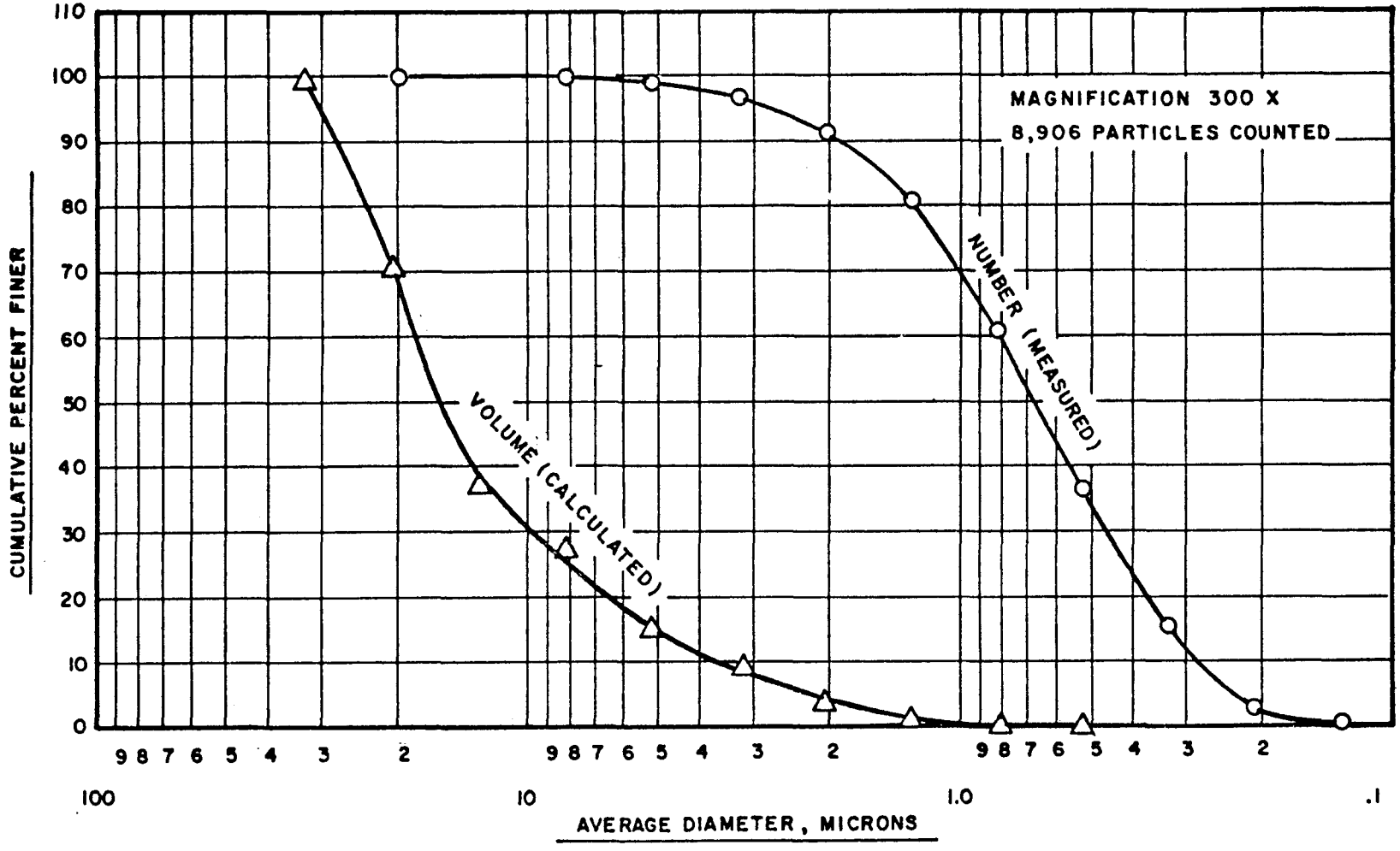
iii. Coulter Counter Analysis

Another portion of the solids from the 7/11/78 sample was sent to Coulter Electronics, Inc., Fine Particle Lab in Hialeah, Florida. The Coulter Counter operates with a dilute suspension of sample solids in an electrolyte fluid (in this case, 60% of a 5% lithium chloride solution in methanol with 40% of glycerol). This suspension is pumped through an aperture. A constant electrical current is maintained through the aperture at right angles to the flow of slurry. When a particle passes through the aperture the voltage required to maintain the constant current is changed by the resistivity of the particle. For particles whose resistivity is nearly constant the resistance change across the cell is approximately proportional to the particle volume. Each voltage pulse thus generated by the passage of a particle through the cell is recorded and scaled by the instrument which then gives a frequency distribution as a function of particle volume. The major difficulty of

FIGURE 10

PARTICLE SIZE DISTRIBUTION BY SCANNING ELECTRON MICROSCOPY

DISSOLVER SAMPLE 7/11/78



the Coulter method arises from the necessity to suspend the hydrophobic coal solids in a hydrophilic electrolyte system. Thus, agglomeration is probably present in the actual slurry analyzed. Figure 11 gives the volume distribution obtained on the 7/11/78 sample. The mean and range of the distribution are at much larger particle diameters than those given by x-ray sedimentation and electron miscroscopy.

b. PSD Analysis of the 7/19/78 Sample

The 7/19/78 sample was worked up for PSD Analysis in exactly the same way as the 7/11/78 sample except that centrifugation was continued for two hours rather than one hour. This was necessitated by the observation that considerable turbidity remained in the supernatant liquid after one hour of centrifugation. The PSD analysis by x-ray sedimentation, given in Figure 12, indicates a smaller average particle diameter than any of the distributions for the 7/11/78 sample. Median particle diameter is 2.6 microns. No other PSD analyses were performed on this sample.

D. Chlorine Concentration in Powhatan Coal

Preliminary analyses of Powhatan No. 5 coal processed in the Gulf Science & Technology SRC Pilot Plant showed an unusually high concentration of chlorine in the coal (0.4-0.5% wt). Since this might tend to increase the potential of chloride stress corrosion cracking, additional measurements of the chlorine level were made to further study the situation.

Composite samples of Powhatan No. 5 coal received at Fort Lewis were sent to an independent testing laboratory where chlorine composition was determined by ASTM procedure D-2361. The results showed an average chlorine concentration of 0.07 wt % in the moisture free coal. This concentration is approximately equal to that of the other Pittsburgh seam coal processed at Fort Lewis (Blacksville No. 2).

To verify the results, the same composite sample was sent to Washington State University for analysis by the neutron activation technique. The measured concentration by this technique was 0.04 wt %, in approximate agreement with the results obtained by ASTM D-2361 procedure.

In conclusion, it appears that the chlorine concentration in the Fort Lewis Powhatan No. 5 coal is not greatly different from that of previously processed Pittsburgh seam coal. Although the concentration is slightly higher than that of previously processed Kentucky and Illinois coals (approximately 0.04 wt % chlorine), no special processing difficulties related to chlorine content of Powhatan No. 5 coal are foreseen.

FIGURE 11

PARTICLE SIZE DISTRIBUTION BY COULTER COUNTER

DISSOLVER SAMPLE 7/11/78

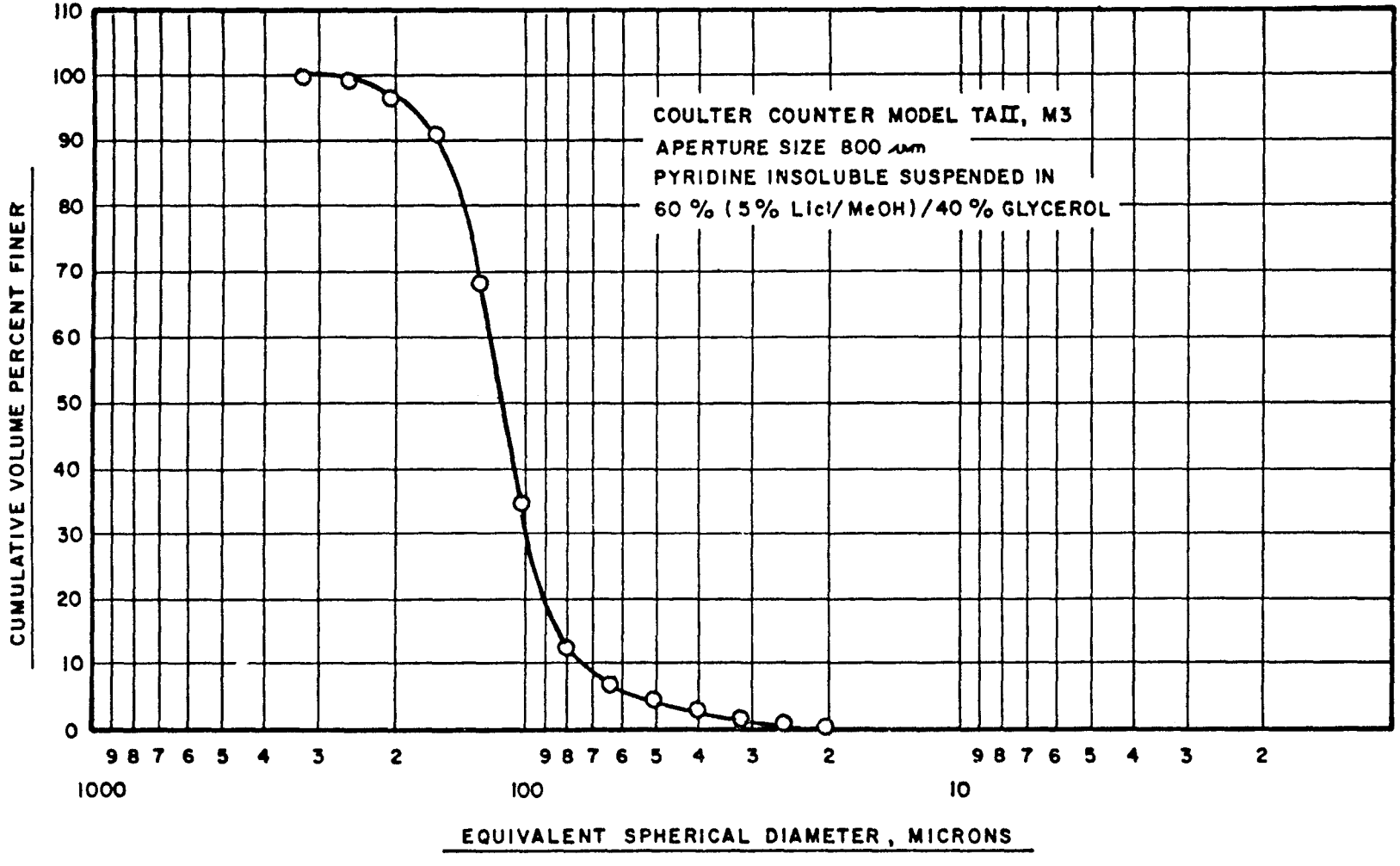
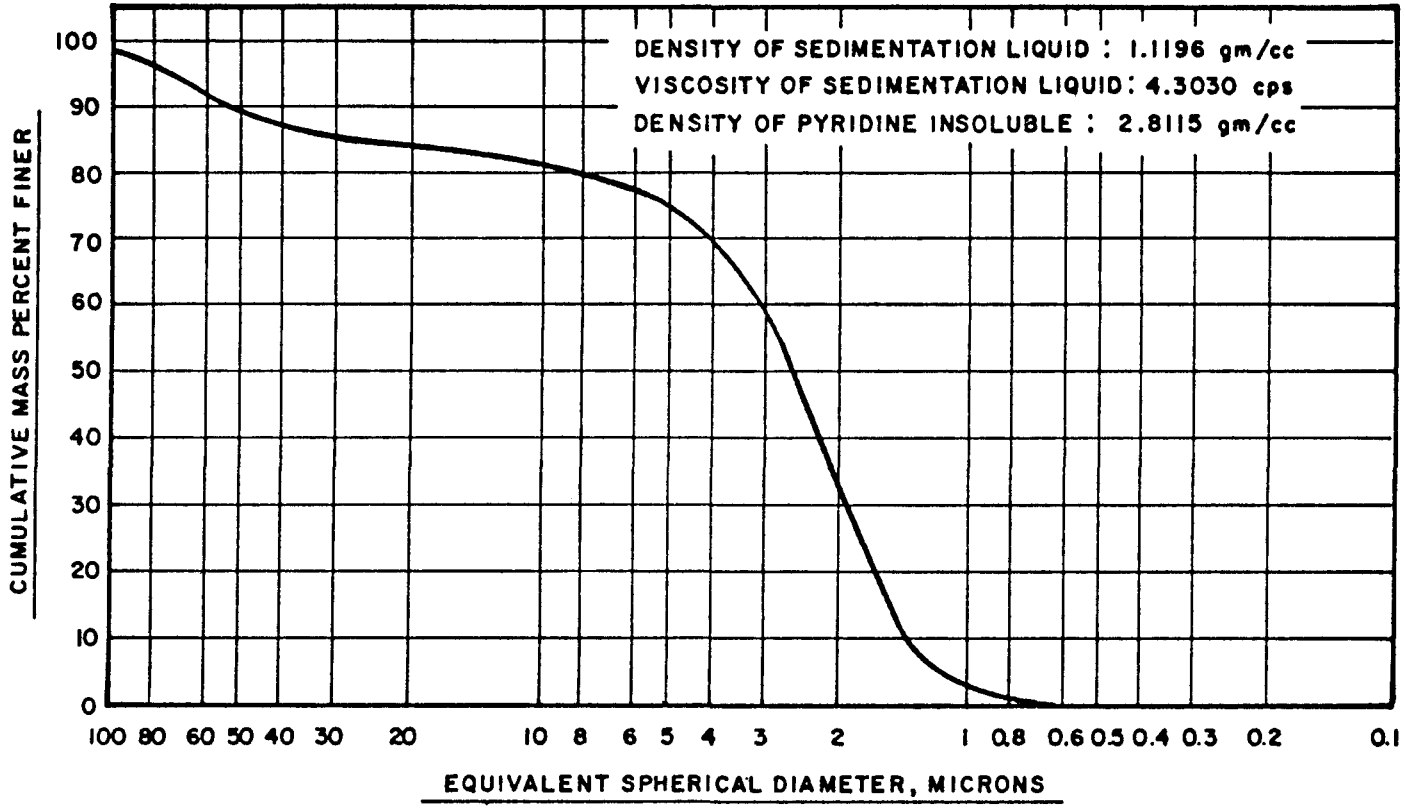


FIGURE 12

PARTICLE SIZE DISTRIBUTION BY X-RAY SEDIMENTATION

DISSOLVER SAMPLE 7/19/78



E. Swelling Properties of Coal

A recently concluded study determined the effects of time and temperature, coal particle size, and coal solution composition on the swelling behavior of three coals.

Swelling behavior is an indication of the interaction between the coal and the agent used as a slurrying medium. At an elevated slurry blending temperature, the coal structure disintegrates rapidly as the coal dissolves in the solvent. At lower temperatures, however, the disintegration is a slow process characterized by absorption of solvent into the coal. This solvent absorption, or "coal swelling", can become very important in the SRC II process as swelling depletes the free liquid components of the feed slurry. This, in turn, increases the viscosity of the blend, making processing more difficult.

In the study, pre-sized coal particles were submerged in heated coal derived liquids. At discrete times the coal particles were removed and the absorbance of solvent was measured by a standardized procedure. Coal swelling was then calculated as:

$$W_{gt} = \frac{W_t - W_D}{W_D} \times 100$$

Where: W_{gt} = Weight gain percentage at elapsed time t (min) after coal particles were submerged into the coal solution.

W_t = Weight of coal solution soaked coal particles at elapsed time t (min).

W_D = Weight of dry coal particles before submersion into coal solution.

A series of tests was made with Western Kentucky, Blacksville No. 2 and Powhatan No. 5 coal in which coal swelling was measured at: slurry residence times from 5 to 200 minutes; slurry temperatures of 325°F to 450°F, coal particle sizes of 12 to 16 mesh, 7 to 8 mesh, and 3 1/2 to 4 mesh; and "solvent" solution containing SRC II heavy distillate (550° to 850°F boiling range) blended with up to 35 wt % of ash-free SRC I solvent refined coal. The results of these tests are shown in Figure 13 through 15.

The following conclusions are based on the results of these tests:

(1) Coal does not swell significantly below 325°F at residence times of up to 3 hours (see Figure 13).

(2) Coal swelling increases with temperature; at temperatures above 400°F, the coal swelling occurs very rapidly (see Figure 13).

(3) Coal swelling increases with decreasing coal particle size; however, at high temperature (450°F) and long residence time (3 hours), this effect becomes insignificant (see Figure 14).

FIGURE 13

INCREASE IN COAL PARTICLE WEIGHT WITH SUBMERGENCE TIME AT VARYING SOLUTION TEMPERATURES

SOLVENT MIXTURE: 10% SRC, 90% HEAVY DISTILLATE

PARTICLE SIZE: 12-16 MESH

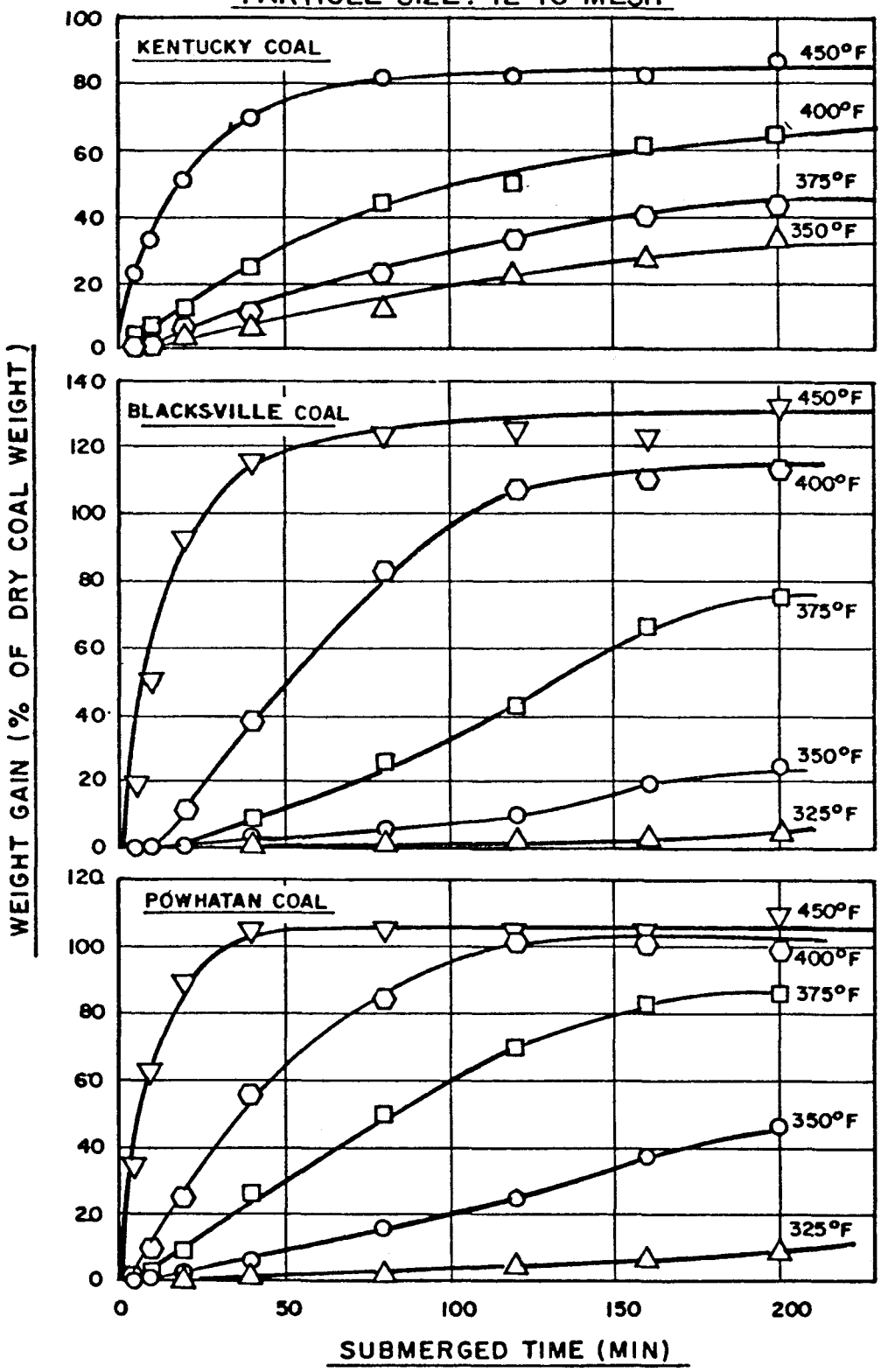


FIGURE 14

INCREASE IN COAL PARTICLE WEIGHT WITH SUBMERGENCE TIME FOR VARYING INITIAL PARTICLE SIZES SOLVENT MIXTURE : 10 % SRC, 90 % HEAVY DISTILLATE

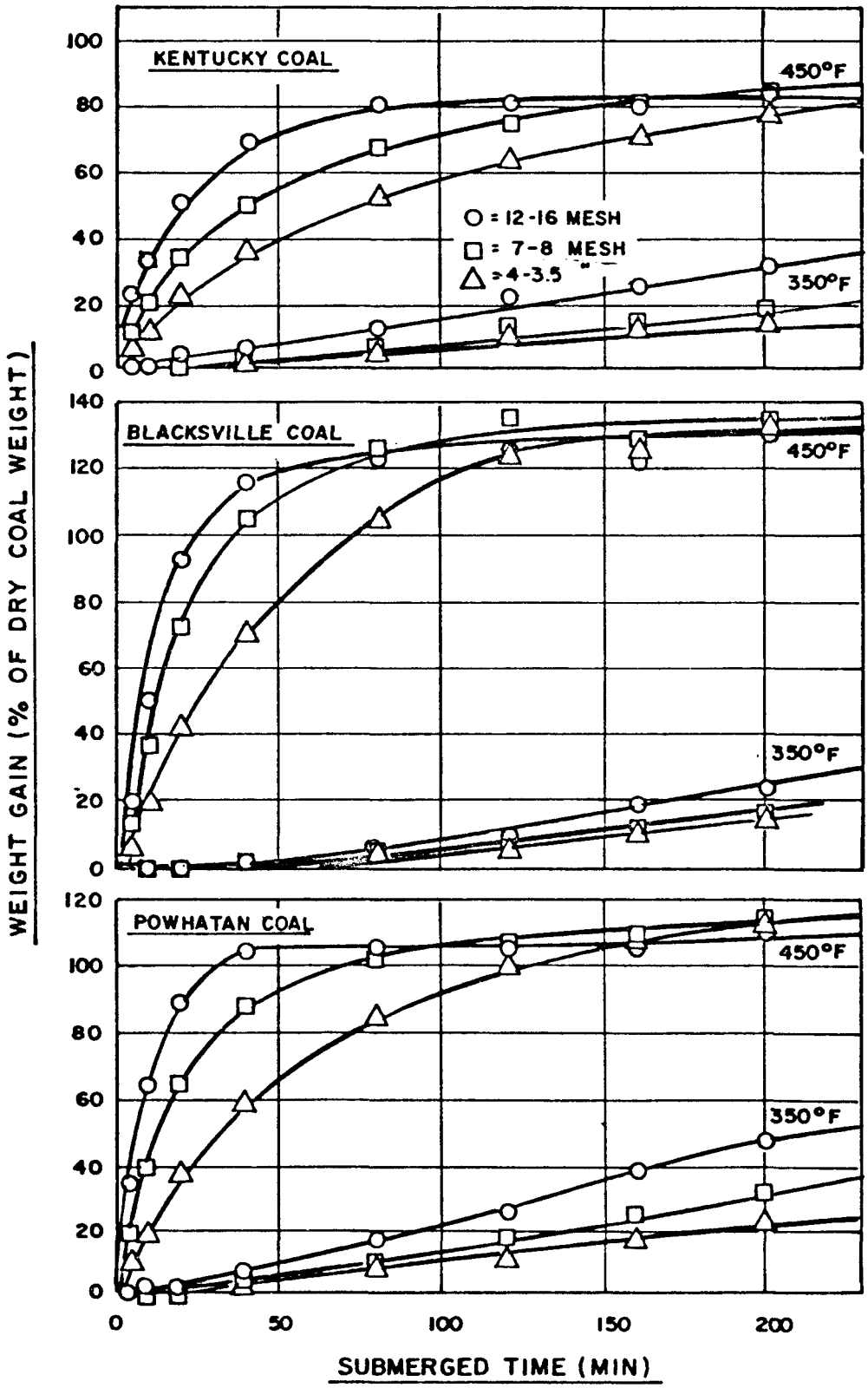
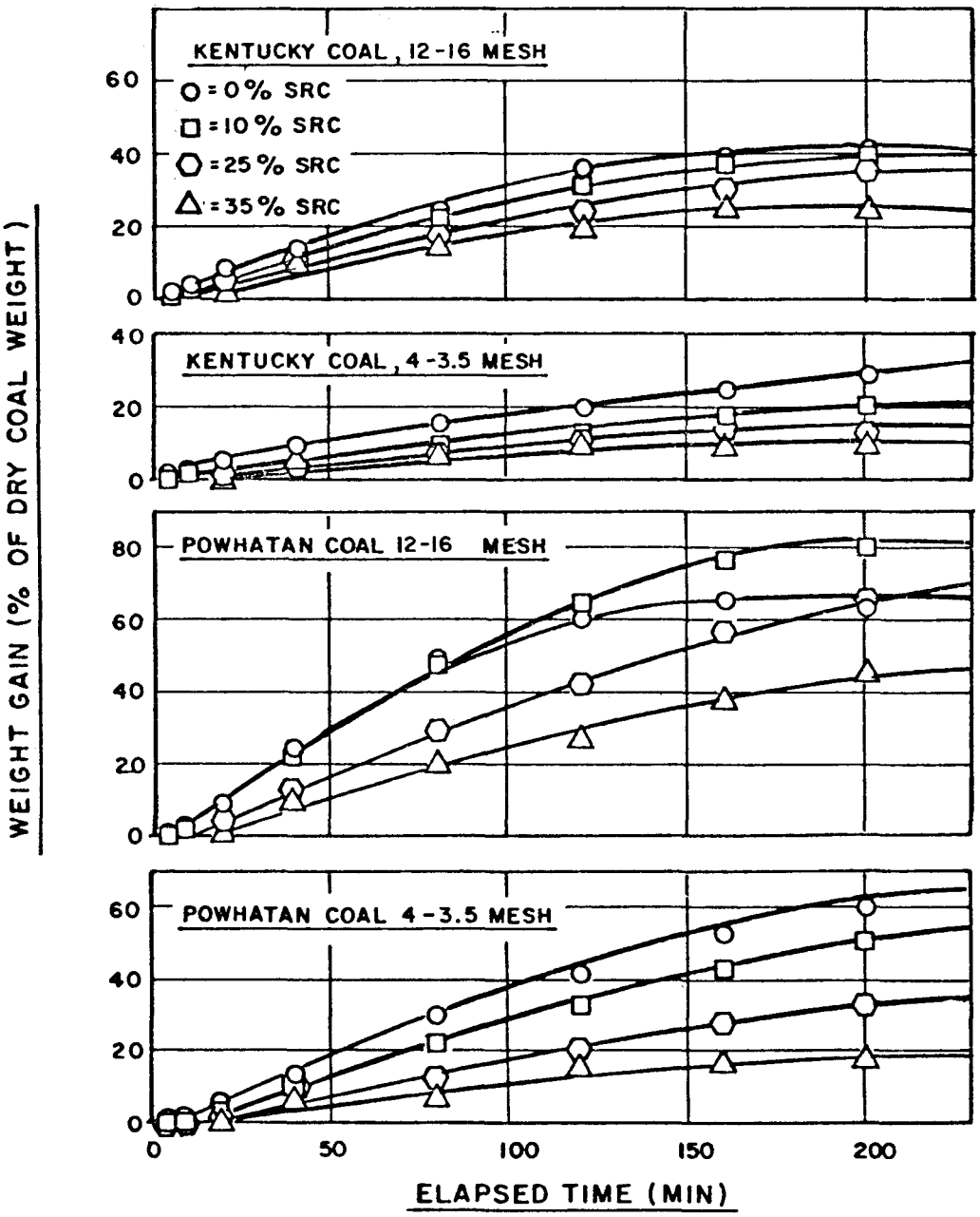


FIGURE 15

INCREASE IN COAL PARTICLE WEIGHT WITH SUBMERGENCE

TIME-VARYING SOLVENT MIXTURE COMPOSITIONS

TEMPERATURE: 375°F



(4) Coal appears to swell more slowly at higher SRC concentrations in the "solvent" solution (see Figure 15).

These results suggest that at elevated slurry temperatures (>350°F) the slurry viscosity can be minimized by short slurry residence times in the slurry vessel and by the use of large diameter coal particles.

F. Solvation of Large Coal Particles

An experiment was performed to investigate the relative dissolution characteristics of coarse and finely ground particles of Powhatan No. 5 coal. The object of the study was to verify that large coal particles are subject to ready solvation in process derived solvent, leaving a minimal residue of large particles.

In the experiment, two samples of Powhatan coal were prepared, one of which was classified to minus 1/4" plus 1/8", and the other was pulverized in the plant to 80% minus 200 mesh. The samples were blended separately with heavy distillate in about 10 wt % concentration then heated under reflux (pot temperature approximately 600°F) for 48 hours. Aliquots were then collected from each slurry and submitted for particle size distribution analysis by x-ray sedimentation.

The results, shown in Figure 16, indicate that in both samples the coal particles were comminuted to approximately the same degree. In Figure 16, curve A represents the 200 mesh (75 μm) sample and curve B represents the 1/4-1/8" sample. In the case of the 1/4-1/8" coal sample (curve B), the distribution indicates about 24 wt % of the particles is found between 15 μm and 25 μm diameter, whereas, only about 6 wt % is found in the same region for the 200 mesh coal sample (curve A). This is the only significant difference seen in these data.

It is apparent that in both samples essentially all of the particles are less than 25 μm in diameter. This observation tends to confirm the viability of feeding larger size coal to the plant reaction system.

G. Dissolver Volume Measurement

On August 30 a measurement was made to the active dissolver volume. Using a previously developed water filling technique, a bottom section volume of 46.2 ft^3 was obtained.

The following table compares the August 30 measurement with previous determinations. It appears that an accumulation of 3-4 cubic feet of static solids can be expected in normal operations.

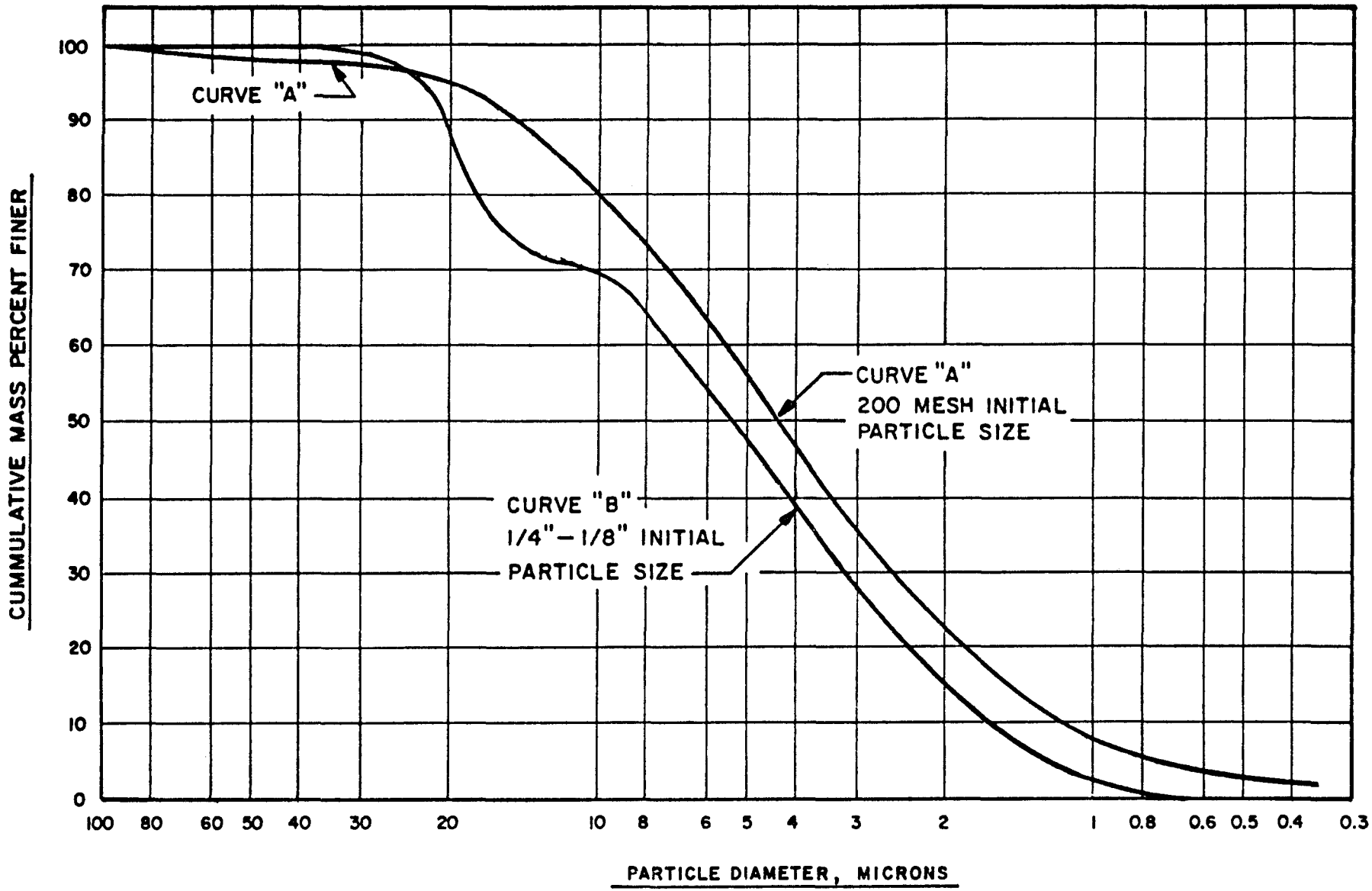
DISSOLVER VOLUME MEASUREMENTS

Volume of Clean Bottom Section of Dissolver: 50 ft^3

Date of Measurement	Measured Volume of Bottom Section of Dissolver
09/20/77	47.3 ft^3
12/09/77	47.7 ft^3
04/07/78	45.7 ft^3
08/30/78	46.2 ft^3

FIGURE 16

COAL SLURRY PARTICLE SIZE DISTRIBUTIONS



H. Data Acquisition System

1. System Reliability

Several computer shutdowns occurred in the first half of the third quarter 1978 but, in most cases, the system restarted automatically. On two occasions the operating system made erroneous entries into the system cross reference table and the system had to be reloaded. This resulted in the loss of some data.

On August 18 the system failed and could not be restarted. A Foxboro Company service representative replaced a defective power supply and redistributed the power supply loadings. The system was restarted on August 20 and had only one problem since which was caused by a fuse failure on the process interface rack resulting in a short interruption in data collection. A summary of system reliability is shown in Table 22.

2. System Modifications

A major activity during the third quarter 1978 involved modification of the computer system in preparation for Lummus deashing unit data acquisition. The required changes included expansion of both hardware and software to accommodate 96 additional process inputs. By the end of the reporting period the Lummus unit was interfaced with the computer system and programs were completed which retrieve and store the unit operating data. Programming modifications required to provide operator assistance and data reports remain to be completed.

3. Programming Activities

In conjunction with the modifications required for the Lummus deashing unit, several changes were made in the data collection routines to improve their efficiency and to enlarge the capacity of drum data storage. First, the process data file structure was changed from floating to fixed point in order to compress data storage space requirements. This change was accomplished with no loss of precision since the resolution of fixed point notation is within that of the analog-to-digital conversion of transduced measurements. Although the fixed point format results in a loss of absolute magnitude, a scale factor file was created to provide this information in stored and printed data. An additional change in the data processing scheme was made to delete the calculation and storage of statistical data regarding process measurement accuracy. The changes permitted an expansion of on-line process data storage from four to ten days.

Modifications were also made to two operator assistance programs: the scan program, which monitors selected process measurements, and the current loop value program, which returns the current value of any process measurement requested by the user. Both programs were changed to use the current process temperature and pressure to calculate flows rather than averages from the

TABLE 22

COMPUTER SYSTEM RELIABILITY FOR

JULY 1 THROUGH SEPTEMBER 24, 1978

EQUIPMENT TYPE	% AVAILABILITY
INPUT DEVICES:	
A.) Operators Console CRT	100
B.) Tape Reader	100
C.) Decwriters (Avg. 3)	100
D.) Magnetic Tape	100
E.) Process/Computer Interface	99.9
OUTPUT DEVICES:	
A.) Tape Punch	100
B.) Decwriter (Avg. 3)	100
C.) Line Printer	100
D.) Magnetic Tape	100
E.) Operators Console CRT	100
CENTRAL PROCESSOR	95.34
OVERALL SYSTEM	95.29

previous hour. The scan program was also modified to enable the operator to change as many of the values being scanned as desired without reinitiating the entire scan. The current loop value program was modified to provide additional information when flows are reported. This information includes the current temperature and pressure of the stream as well as calculated values of specific gravity, viscosity and Reynolds number.

Several revisions were made to the flowrate calculation programs during the period. The changes were necessitated by the installation of corner tap orifice plate flowmeters and segmental wedge flow meters in the plant.

Plant solvent inventory calculations were resumed during this reporting period. The computer programs which are used to calculate and report the solvent inventory were modified to report the inventories of SRC I and SRC II liquids separately.

V. PILOT PLANT SPECIAL PROJECTS

A. Dissolver Quench Modifications

A prototype sparger was designed and installed as a replacement for the middle hydrogen quench line which had collapsed during previous SRC II runs. The new sparger is intended to simulate a proposed design for the demonstration plant dissolvers. The new sparger is expected to provide improved distribution of the hydrogen as the principal advantage over the previous serrated open pipe sparger. Installation of a nitrogen tube trailer to supply emergency purge to the dissolver quenches and sample probes in the event of a hydrogen purge failure was also completed.

B. Relocation of the Dissolver Differential Pressure Cells

The original dissolver differential pressure (DP) cells were unreliable because of recurrent plugging of the taps leading to the dissolver. The primary causes of the plugging have been that the DP cells were located below the dissolver connections and that hydrogen purges were not available for several tap lines. In order to alleviate these problems the dissolver DP cell was relocated above the dissolver (on the top floor of the 02 structure) and hydrogen purge supply (with nitrogen backup) was installed in the sensing lines.

C. Middle Dissolver Sample Point

A sample point installation from the side/center outlet on A dissolver was designed and an operating procedure for this sample point was prepared.

Installation of the sample point began August 18 and was completed during the September shutdown.

D. Ground Coal Studies

Implementation of a new plan to feed minus 1/8" coal to the process began with the leasing of an impact type coal crusher from Jodal Manufacturing, Seattle, Washington. Major modifications to Area 01 for the installation of the Jodal crusher included construction of a protective structure over the track hopper to keep the coal dry during unloading, erection of a support for the feed hopper of the Jodal crusher and installation of a dynamometer for weighing the contents of the feed hopper.

E. Area 02 Preheater

Approval of the new slurry preheater project was received from DOE and, after resolution of a possible patent infringement, an order for its fabrication was placed with Heat Research Corporation, Houston, Texas. Delivery is expected no later than May 1979.

F. Cornucopia Vacuum Flash Preheater

A bid for the fabrication of a newly designed vacuum flash preheater coil was received from Piping Engineering Company, Sand Springs, Oklahoma, but placement of the order has been suspended until the performance of the present 4-inch coil can be evaluated.

G. Lummus Antisolvent Deashing Unit

At the end of August, overall construction of the C. E. Lummus antisolvent deashing system was essentially complete and two Lummus representatives were at the plant supervising precommissioning activities. Work through September consisted of clearing punch-list items submitted to Lummus by the P&M Operations section. At the end of the quarter, water flushing and piping dry-out operations were in progress. Systems testing of this unit will begin once the current SRC II test program has been completed and the plant has been reconverted to the SRC I mode.

VI. MERRIAM LABORATORY OPERATIONS

A. Introduction

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

- Operation of a new one liter upflow dissolver in both the SRC I and SRC II modes.
- Operation of a concentric tube dissolver including a standard upflow center zone with cocurrent hydrogen flow and a downflow outer zone with countercurrent hydrogen flow. This dissolver was also operated in both the SRC I and SRC II modes.
- A brief investigation of solvent hydrogenation and cracking under SRC II processing conditions.

Preliminary investigations of SRC I operation with short residence times.

Conditions and results for all runs reported this quarter are summarized in Table A-1.

B. Evaluation of a One Liter Standard Upflow Dissolver

A new one liter standard upflow reactor was first used in July 1978. The volume of this dissolver is similar to that of the previously used GU 5 reactor, but the new dissolver has a larger diameter and lower L/D and is a single vessel while the GU 5 reactor consisted of two dissolvers operated in series. In addition, the new dissolver has a port at half-length so that it can also be operated as a half-liter vessel. The new dissolver, designated DOE 1, is shown in Figure 17. The dissolvers of the old GU 5 reactor were heated by strap resistance heaters while the new dissolver is heated in the air circulation furnace which was described in the previous quarterly report[1]. Two SRC I and three SRC II runs were made to compare operation of the new dissolver with that of the GU 5 reactor.

Results for the SRC I comparisons are shown in Table 23. Conditions for DOE 234 and 235 (DOE 1 dissolver) were selected to match those of GU 213 and 214 (GU 5 dissolver), respectively. Yields with the two reactors are similar but with minor differences in the SRC and oil yields. With the DOE 1 dissolver, the oil yield is slightly higher and the SRC yield slightly lower. The slightly higher conversion of SRC to oil in the new dissolver could be due to variations in hydrodynamics of the two reactors or could be due to variation in temperature profiles. The cooling which occurs between the first and second dissolvers and results in a low temperature at the bottom of the second dissolver in the old GU 5 reactor is absent with the new reactor. The higher conversion in the new reactor could also be due in part to the slightly longer residence time.

Both the SRC and recycle solvent obtained with the new dissolver are somewhat more hydrogenated than those obtained with the GU 5 reactor. However, the sulfur content of the SRC obtained with the new reactor in DOE 235 is somewhat higher than that obtained in GU 214. Although the differences are sufficiently small that they may not be significant, the higher sulfur content of the SRC and the slightly higher insoluble organic matter yield with the new reactor could be attributed to a changed residence time distribution with more material getting through the reactor with a very short residence time in the single tube dissolver.

Runs DOE 237R and 238R (DOE 1 dissolver) were SRC II runs with conditions matching those of GU 216R (GU 5 dissolver) and DOE 239R (DOE 1 dissolver) was an SRC II run with conditions matching those of GU 212R (GU 5 dissolver). The latter run was a simulation of Fort Lewis Material Balance Run 78SR-17. Results for the SRC II runs are compared in Table 24. Run DOE 237R was terminated due to a partial plug at the preheater inlet before satisfactory steady state operation had been demonstrated so run DOE 238R was made under the same nominal conditions.

Figure 17

Upflow Dissolver (DOE 1)

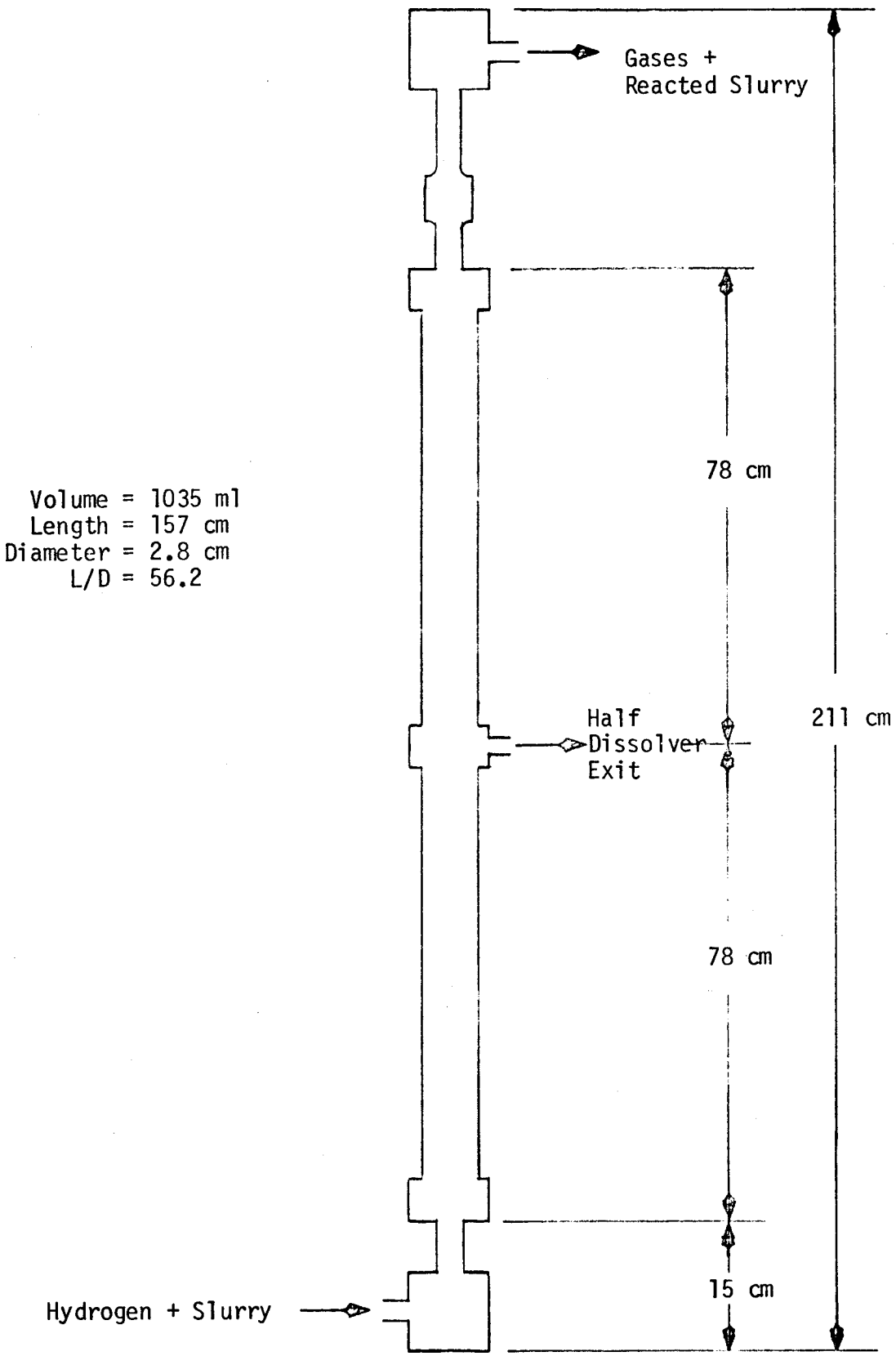


TABLE 23

SRC I Comparison of the GU 5 and DOE 1 Dissolvers

Conditions	GU 213	DOE 234	GU 214	DOE 235
Coal	← Kentucky Nos. 9 & 14 Colonial Mine, Lot 7 →			
Reactor	GU 5	DOE 1	GU 5	DOE 1
Nominal Residence Time, hr	0.98	1.03	0.99	1.04
Coal Feed Rate, lb/hr/ft^3	21.9	20.9	32.2	31.0
Slurry Formulation				
% Coal	30.0	30.0	45.0	45.0
% Recycle Solvent	70.0	70.0	55.0	55.0
Hydrogen Feed Rate				
Wt % based on slurry	4.55	4.20	4.62	4.22
Dissolver Temperature, °C	455	455	455	455
, °F	851	851	851	851
Pressure, psig	2000	2000	2000	2000
Results				
Yields, wt % MF Coal Basis				
C_1-C_4	10.5	9.9	10.5	10.1
Recycle Solvent	4.0	4.1	8.1	12.3
Total Oil	25.9	28.9	27.4	28.9
SRC	42.7	39.4	41.4	38.2
Insoluble Organic Matter	4.1	4.3	4.4	4.6
Product Properties				
% S in SRC	0.49	0.50	0.48	0.55
% H in SRC	5.70	5.92	5.52	5.83
% S in Recycle Solvent	0.28	0.30	0.33	0.37
% H in Recycle Solvent	7.96	8.01	7.84	7.94

TABLE 24

SRC II Comparison of the GU 5 and DOE 1 Dissolvers

Conditions	GU 212R	DOE 239R	GU 216R	DOE 237R	DOE 238R
Coal	←Pittsburgh Seam→ Blacksville Mine No. 2		←Kentucky Nos. 9 & 14→ Colonial Mine		
Reactor	GU 5	DOE 1	GU 5	DOE 1	DOE 1
Nominal Residence Time, hr	0.99	1.00	1.00	1.07	1.05
Coal Feed Rate, lb/hr/ft^3	21.7	21.4	21.6	20.1	20.5
Slurry Formulation					
% Coal	30.0	30.0	30.0	30.0	30.0
% Unfiltered Coal Solution	64.0	64.0	70.0	70.0	70.0
% Recycle Solvent	6.0	6.0	--	--	--
Dissolver Temperature, °C	461	461	455	455	455
, °F	862	862	851	851	851
Pressure, psig	1860	1860	2000	2000	2000
Results					
Yields, wt % MF Coal Basis					
C_1-C_4	11.0	13.0	16.1	14.6	12.6
Heavy Distillate	12.9	15.4	21.8	24.6	24.8
Total Oil	28.2	32.6	38.9	39.5	39.2
SRC	33.9	30.4	21.0	22.5	28.0
Insoluble Organic Matter	9.3	7.5	5.1	4.6	5.0
Heavy Distillate Analyses					
% H	7.56	7.33	7.90	7.72	7.69
% S	0.48	0.37	0.29	0.36	0.37

As was observed in the SRC I comparison runs, in the Pittsburgh seam SRC II comparison, yields are similar but with somewhat higher conversion of SRC to oil with the DOE 1 reactor. In this particular comparison, the insoluble organic matter yield is lower with the DOE reactor; this observation is of limited significance as the insoluble organic matter yield with Blacksville Mine No. 2 coal has been found to vary widely with little apparent change in conditions. In contrast to the SRC I work, hydrogen content of the heavy distillate product is lower with the DOE reactor. With the Pittsburg seam coal, the heavy distillate product from the DOE 1 reactor is better desulfurized. This is in direct contrast to all other comparisons.

Results of the SRC II work with Kentucky coal are uncertain. Run DOE 237R was terminated prematurely due to experimental difficulties. The calculated and actual feed slurry ashes are in good agreement which is an indication of steady state operation; however, the product distribution was calculated based on the results of a single distillation of a small sample so there is no assurance that reliable yields were obtained. Yields obtained in DOE 237R are similar to those obtained in GU 216R.

Due to the uncertainty in the DOE 237R results, the same nominal conditions were investigated in run DOE 238R. A comparison of DOE 238R yields with those of GU 216R shows a significantly higher SRC yield (28.0 vs 21.0%) with the DOE reactor. (The 7.0% increase in SRC yield is counterbalanced by 3.5% and 3.3% reductions in gas and water yields, respectively.) The higher SRC yield obtained with the DOE reactor in this comparison is in direct contrast to the other three comparisons which sheds some doubt on either GU 216R or DOE 238R. Some problem with the DOE 238R data is apparent in the ash balance. Actual feed slurry ash was 15.15 while the calculated feed slurry ash was only 13.55% (lineout index = 1.12). Actual ash output was 112.5% of theory. Similar discrepancies have been observed where ash balances were calculated on single samples (as was done for GU 237R) but are not usually observed where several samples are available for analysis. (The ash balance for DOE 238R is based on three distillations and three vacuum bottoms ash analyses; good precision was observed for both the distillations and ash analyses.) The reason for the reported high ash output has not been found but it casts some doubt on the reliability of the DOE 238R results.

The GU 216R results have been compared with those of other runs to see if they appear reasonable. Runs under similar conditions with the same lot (lot 7) of coal are not available but runs made under similar conditions with other lots of Colonial Mine coal indicate that the GU 216R yields are not unreasonable but the SRC yield predicted from other runs would be about 2% higher and the C_1-C_4 yield about 2% lower. An SRC yield as high as 28% under these conditions with the GU reactor does not appear likely.

As was observed with the Pittsburgh seam coal, the heavy distillate product obtained in SRC II operation with the DOE reactor is less well hydrogenated and, as was observed in the SRC I runs, the heavy distillate product is more poorly desulfurized.

In summary, yields obtained with the new DOE 1 reactor are similar to those obtained with the GU reactor, and, in three of the four available comparisons, a slightly better conversion of SRC to oil was observed with the new reactor. Results of the fourth comparison are inconclusive.

C. Upflow-Downflow Reactor Evaluation

A new reactor design, including both upflow and downflow reaction zones, was evaluated. A diagram of this reactor, designated DOE 2, is shown in Figure 18. The reactor consists of two concentric tubes. Slurry and hydrogen from the preheater (the standard preheater was used) enter the center tube of the dissolver at the bottom and travel up the center tube as in a standard dissolver. When the liquid reaches the top of the inner tube, it spills into the downflow section of the dissolver. Another hydrogen stream enters the downflow section near the bottom so hydrogen flow is countercurrent to the slurry in the outer zone. All vapor exits the dissolver at the top. The reacted slurry is removed from the dissolver at the bottom of the downflow section.

The expected advantage of a downflow dissolver is that product gases formed in the initial reaction stages are removed at the top of the dissolver and as hydrogen is added at the bottom of the dissolver, the slurry is subjected to the maximum hydrogen partial pressure in the final stages of the reaction. In earlier work[2] with a downflow reactor, it appeared that this potential advantage was more than offset by the lack of accumulation of mineral matter (catalyst) which is experienced in a standard upflow reactor. The lack of accumulation of mineral matter appeared to be of less significance in SRC II than in SRC I.

With the DOE 2 reactor design, the slurry is subjected to the maximum hydrogen partial pressure in the final stage of the reaction and the upflow section of the dissolver does allow the accumulation of mineral matter in that section of the reactor. However, mineral matter accumulation might be more beneficial later in the reaction where it is assumed that most solvent rehydrogenation reactions are taking place.

Results of SRC I operation with the upflow-downflow reactor (run DOE 240C) are compared with those of standard upflow reactors in Table 25. All runs were made with Kentucky Nos. 9 and 14 coal and slurry composition was 30% coal, 70% recycle solvent. A somewhat higher hydrogen feed rate was used with the upflow-downflow reactor as the hydrogen feed was split into two streams with about 1/3 entering the preheater and 2/3 entering the bottom of the downflow section of the dissolver. In all cases, target dissolver temperature was 455°C (851°C) and pressure was 2000 psig. These results show a definite degradation in performance of the upflow-downflow reactor in comparison to the standard upflow reactors. While recycle solvent was obtained in 4% excess with the standard reactors, a 10% deficiency of solvent was noted with the DOE 2 reactor. An increase in

Figure 18

Upflow-Downflow Dissolver (DOE 2)

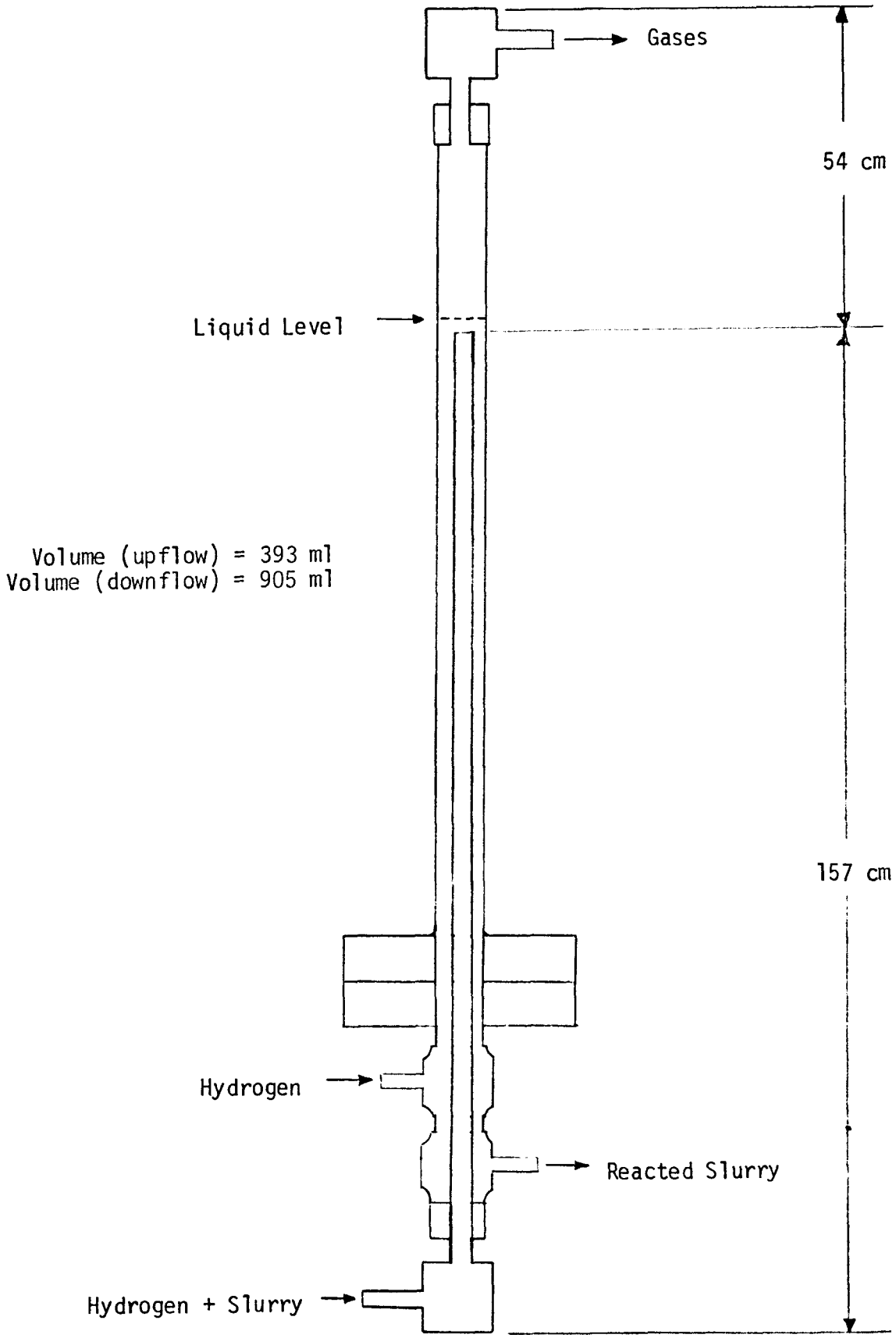


TABLE 25

SRC I Comparison of Standard Upflow Reactors (GU 5 and DOE 1) with an Upflow-Downflow Reactor (DOE 2)

Conditions	GU 213	DOE 234	DOE 240C
Reactor	GU 5	DOE 1	DOE 2
Nominal Residence Time, hr	0.98	1.03	1.00
Coal Feed Rate, lb/hr/ft^3	21.9	20.9	21.4
Hydrogen Feed Rate, Wt % Based on Slurry	4.55	4.20	5.46
Results			
Yields, wt % MF Coal Basis			
C_1-C_4	10.5	9.9	[a]
Recycle Solvent	4.0	4.1	(10.4)[b]
Total Oil	25.9	28.9	13.0
SRC	42.7	39.4	52.7
Insoluble Organic Matter	4.1	4.3	5.6
Product Properties			
% S in SRC	0.49	0.50	0.74
% H in SRC	5.70	5.92	5.72
% S in Recycle Solvent	0.28	0.30	0.41
% H in Recycle Solvent	7.96	8.01	7.31

a) C_1-C_4 yield not determined due to gas chromatograph failure.

b) Loss

SRC yield from approximately 40% to over 50% was noted. There was also a higher insoluble organic matter yield with the DOE 2 reactor. Sulfur content of both the SRC and recycle solvent obtained with the DOE 2 reactor were higher, and hydrogen content of the recycle solvent obtained with the DOE 2 reactor was much lower (7.3 vs 8.0%). In SRC I operation, performance of the upflow-downflow reactor is clearly inferior to that of a standard upflow reactor.

Results of SRC II operation with the upflow-downflow reactor are compared with those of standard upflow reactors in Table 26. Run DOE 240R (DOE 2 dissolver), made with Kentucky coal, was initiated under conditions corresponding to those of GU 216R and DOE 238R (runs made with the GU 5 and DOE 1 dissolvers, respectively). However, under these conditions the viscosity of the feed slurry became excessively high and it was necessary to formulate feed slurry with 5% distillate recycle solvent in order to reach steady state operation. Consequently, run DOE 240R was completed under conditions varying from those of the control runs GU 216R and DOE 238R. The results for DOE 240R indicate poorer performance for the DOE 2 reactor than for the standard upflow reactors. With the upflow-downflow reactor, total oil yield was 33% in comparison to 39% for the standard upflow reactors. SRC yield was 35% in comparison to an average SRC yield of 25% for the upflow reactors. This degradation in yields is significantly greater than would be anticipated on the basis of the changed feed slurry composition alone. In the run with the Kentucky coal, heavy distillate from the DOE 2 reactor is more poorly hydrogenated and more poorly desulfurized than that from the upflow reactors.

Run DOE 241R (DOE 2 dissolver), made with the Pittsburgh seam coal from the Blacksville Mine No. 2, was made under conditions similar to those of GU 212R and DOE 239R (GU 5 and DOE dissolvers, respectively). Run DOE 241R was made with a pressure of 2000 psig in place of the 1860 psig used in the control runs; after data were obtained at 2000 psig the pressure was dropped to 1860 psig and the run continued as DOE 242R. No degradation in yields is apparent with the drop in pressure, although, at the lower pressure, hydrogenation of the heavy distillate product was poorer.

Runs GU 212R and DOE 239R were made with a temperature profile (with a maximum temperature of 460-461°C) to match that of the Fort Lewis pilot plant. In run DOE 242R, the temperature was somewhat lower with the temperature of the top zone being 456°C. In this comparison, there is less difference apparent between the upflow-downflow and the standard upflow reactors. In the DOE 2 reactor the total oil yield averaged 28% in comparison to an average of 30% for the standard upflow reactors. SRC yield averaged 37% for the upflow-downflow reactor in comparison to an average SRC yield of 32% for the upflow reactors. These differences in yields may be attributable largely to differences in temperature.

While in SRC I operation there is a large degradation in performance of the upflow-downflow reactor in comparison to the performance of

TABLE 26

SRC II Comparison of Standard Upflow Reactors (GU 5 and DOE 1) with an Upflow-Downflow Reactor (DOE 2)

Conditions	GU 216R	DOE 238R	DOE 240R	GU 212R	DOE 239R	DOE 241R	DOE 242R
Coal	← Kentucky Nos. 9 & 14, Lot 7 →			← Pittsburgh Seam Blacksville Mine No. 2 →			
Reactor	GU 5	DOE 1	DOE 2	GU 5	DOE 1	DOE 2	DOE 2
Nominal Residence Time, hr	1.00	1.05	0.99	0.99	1.00	0.98	1.00
Coal Feed Rate, lb/hr/ft^3	21.6	20.5	21.5	21.7	21.4	21.9	21.6
Slurry Formulation							
% Coal	30.0	30.0	30.0	30.0	30.0	30.0	30.0
% Unfiltered Coal Solution	70.0	70.0	65.0	64.0	64.0	63.0	64.0
% Recycle Solvent	--	--	5.0	6.0	6.0	7.0	6.0
Hydrogen Feed Rate							
Wt% Based on Slurry	4.63	4.27	5.42	5.83	5.84	5.37	5.02
Dissolver Temperature, ^oC	455	455	455	461	461	455	455
, ^oF	851	851	851	862	862	851	851
Pressure, psig	2000	2000	2000	1860	1860	2000	1860
Results							
Yields, wt % MF Coal Basis							
C_1-C_4	16.1	12.6	11.6	11.0	13.0	9.6	9.5
Heavy Distillate (>249^oC, 480^oF)	21.8	24.8	17.7	12.9	15.4	12.3	14.0
Total Oil	38.9	39.2	32.5	28.2	32.6	27.4	28.8
SRC	21.0	28.0	34.8	33.9	30.4	38.3	36.0
Insoluble Organic Matter	5.1	5.0	5.4	9.3	7.5	7.4	7.8
Heavy Distillate Analyses							
% H	7.90	7.69	7.54	7.56	7.33	7.70	7.43
% S	0.29	0.37	0.49	0.48	0.37	0.40	0.44

a standard upflow reactor, the degradation of performance of the DOE 2 reactor in SRC II operation is less apparent. With Kentucky coal in SRC II operation there is a definite decrease in conversion of SRC to oil but the decrease is less significant than was observed in SRC I operation. However, with the Pittsburgh coal there is a smaller difference in yields and, in fact, much of the difference in results with the two types of reactor may be attributed to a 5°C difference in the dissolver temperatures.

D. Effect of Pyrite and Mineral Residue on Hydrogenation and Cracking of Recycle Solvent

Runs DOE 243-246 were made to determine the level of hydrogenation and cracking of recycle solvent taking place under normal SRC II reaction conditions and to determine the effect of pyrite and mineral residue on the hydrogenation and cracking. These runs were made following the investigation of the DOE 2 reactor with the DOE 2 reactor still in service. Run DOE 243 was made as a continuation of DOE 242R with no change in conditions except in the change of feed to recycle solvent only. This run was terminated after about 20 hours for replacement of pressure letdown valves and repair of slurry recirculation pumps. Results of this run could be influenced by coal minerals not flushed from the reactor after stopping coal feed. After reactor repairs, the same conditions were investigated in run DOE 243A. This run was continued for about 16 hours after which pyrite (2.5g pyrite per 97.5g solvent) was added to the solvent feed for run DOE 244. Run DOE 245 investigated the effect of added mineral residue at a 5.7% level (based on slurry weight). Pyrite was used at a 5.7% level in run DOE 246; in the latter run, temperature was dropped to 400°C (752°F) from the 455°C (851°F) used in the earlier runs. Conditions and results are summarized in Table 27.

In all cases, except for the 400°C run, a mild hydrogenation of the recycle solvent was observed. Very moderate desulfurization also took place. Results are summarized below:

	Feed Solvent	DOE 243A	DOE 244	DOE 245	DOE 246
% H	7.48	7.72	7.61	7.62	7.47
% S	0.47	0.44	0.40	0.38	0.44

The light hydrogenation with the pyrite or mineral residue was expected; the hydrogenation without additives was not. This run may have been influenced by mineral residue left in the reactor from the previous run.

In addition to hydrogenation, low levels of cracking were also observed. C_1-C_4 yields of about a half a percent based on input solvent were observed; these yields do not appear to be correlated with the presence or absence of additives. Negligible hydrocarbon gas yields were observed at 400°C. Low level formation of light

TABLE 27

Effect of Pyrite and Mineral Residue on Hydrogenation and Cracking of Recycle Solvent

Conditions	DOE 243	DOE 243A	DOE 244	DOE 245	DOE 246
Additive	None	None	(Pyrite)	(Mineral Residue)	(Pyrite)
Slurry Formulation					
% Recycle Solvent	100.0	100.0	97.5	94.3	94.3
% Additive	--	--	2.5	5.7	5.7
Nominal Residence Time, hr	1.00	0.98	0.98	0.95	0.96
Hydrogen Feed Rate					
Wt %, based on slurry	5.03	5.36	5.37	5.22	5.28
Pressure, psig	1860	2000	2000	2000	2000
Target Temperature, °C	455	455	455	455	400
, °F	851	851	851	851	752
Results					
Recycle Solvent Analyses					
% H	7.72		7.61	7.62	7.47
% S	0.44		0.40	0.38	0.44
Yields, wt % solvent basis					
H_2O		0.50	0.86	0.66	0.25
CO		0.01	0.01	0.04	--
CO_2		--	0.02	0.02	--
H_2S		0.24	0.71	0.29	1.70
NH_3		--	--	--	--
C_1		0.36	0.34	0.32	--
C_2		0.21	0.21	ND	--
C_3		0.06	0.04	0.04	--
C_4		0.03	0.03	0.02	--
Total C_1-C_4		0.66	0.62	0.38	--
C_{5+} (gas)		0.53	0.31	0.80	--
Naphtha, <193°C		0.69	0.73	0.66	0.31
Middle Distillate, 193-249°C		3.11	3.06	3.30	2.28
Heavy Distillate, >249°C		94.63	94.94	94.24	97.15
Total Oil (C_{5+}-heavy distillate)		98.96	99.04	99.00	99.74
SRC		--	--	--	--
Insoluble Organic Matter		--	--	--	--
Ash		--	--	--	--
Total		100.37	101.26	100.39	101.69
H_2 Reacted (gas balance)		0.37	0.58	0.39	0.10

oils was also observed. Naphtha yields of about 0.7% and middle distillate yields of about 3% were observed (lower yields were observed at 400°C). Again, these yields appear independent of the absence or presence of additives. It was not unequivocally established that the reported yields of light oils are due entirely to cracking in the reactor; there is a possibility that the reported yield of light oils may have resulted at least in part due to refractionation of the recycle solvent which takes place in the letdown system of the reactor.

E. Short Residence Time Runs

Eleven short residence time SRC I runs were made during the quarter. Discussion of these runs will be delayed until a later report.

F. Merriam Maintenance and Modifications

1. Maintenance

Principle areas of maintenance were:

a. Providing reliable slurry letdown service from the Research Control Valves required frequent replacement of trim. Trim in these valves were constructed of tungsten carbide, but average service was only three to five days.

b. Improper placement of thermocouples caused overheating of a preheater tube and the high temperature separator. The preheater tube was discarded while the separator was inspected, pressure tested and placed back in service.

c. Level control in the high temperature separator was degraded due to slurry buildup in the hydrogen purged pressure sensing lines. The separator was removed on two occasions to clean these lines.

d. Two motor failures on the electrically heated air circulation furnace were caused by frozen and improperly installed bearings. Fan shafts became excessively worn and were turned end for end before new bearings could be installed.

e. When ambient temperature rose above 80°F and 80% relative humidity was reached, the Fluke datalogger malfunctioned. A printed circuit board was replaced and the datalogger was again operational.

f. Frequent rupture disc failures in July were attributed to excessive vibration on the rupture disc vent lines, irregularities in the rupture disc holders, and improper torque in assembly procedures. Corrective actions were taken and the problem was largely eliminated.

g. In run DOE 239R the refrigerated separator became plugged with material that was water soluble and easily removed when

the vessel was taken off line. Provisions were made in the piping to bypass the vessel if plugging occurs in the future.

h. After two runs in which pyrite and mineral matter were added to process solvent to observe hydrogenation effects, the unit was shutdown to remove the additives accumulated throughout the system. The process solvent was not viscous enough to suspend the additives and they settled in many parts of the unit.

2. Modifications

Numerous modifications were completed this quarter. The major items were:

a. The vacuum flash system was brought on line during DOE 236. Failure of mechanical timers and undersized vacuum condensor receiver vessels caused the run to be terminated. The receiver vessels were enlarged and a search for different timers was started.

b. A slurry return line from the bottom of the atmospheric flash vessel to the slurry mix vessel was installed. This eliminated the need to enter the high pressure dissolver bay to remove products. A back pressure regulator was installed on the atmospheric flash overhead vapor line to maintain 3 to 20 psig on the flash vessel. This facilitates the removal of coal solution from the vessel.

c. The hydrogen compression and metering system was modified to obtain two independent hydrogen feed streams. The two streams may be supplied by the same compressor or supplied individually from the Pressure Products Industries and Aminco compressors.

d. A ceramic sheathed capacitance probe was installed in the atmospheric flash vessel to indicate the level of coal solution in the vessel. The signal from the probe is used to control the valve which drains product from the atmospheric flash vessel.

e. Poor performance of Research Control Valves in slurry letdown service prompted the installation of a Fisher Gismo Valve in place of one of the Research Control Valves. The Gismo valve provides two to three times the length of service before trim replacement is required.

f. An absolute pressure transmitter and controller were installed in the distillation laboratory in addition to a new vacuum pump. This will maintain a constant pressure on the still heads during vacuum distillations.

VII. P-99 Unit

During the second and third quarters of 1978, three different coals and nine different run conditions were tested in the SRC II mode on the continuous 1 T/D pilot plant P-99. The three coals were all from the Pittsburgh seam, but they were from different mines: the Powhatan No. 5 Mine; the Valley Camp Mine; and the Robinson Run Mine. Inspections for these three coals are given in Table 28.

Three runs were made feeding the Powhatan coal, two feeding Valley Camp coal, and four feeding Robinson Run coal. Operating conditions for these nine runs are given in Table 29. The main objective of this work was to assess the suitability of Pittsburgh seam coal as a feedstock for SRC II process and to learn something about variability from one part of the Pittsburgh seam to another.

Normalized and elementally balanced yields from the nine runs are summarized in Table 30. Examination of the data in Table 30 shows that yields from the three Pittsburgh seam coals are similar if they are compared on an ash-free basis. Thus, although the Robinson Run coal shows lower distillate yields on a moisture-free basis, the yields are comparable to the other coals on an ash-free basis. This work shows the suitability of several Pittsburgh seam coals as feed for the SRC II process.

Runs 35 and 36 were made to determine the effect of recycle gas rate on products yields. Run 35 was made at a relatively high dissolver gas rate typical of previous SRC II runs on P99; whereas, run 36 was made at a lower dissolver gas rate in the range proposed for the demonstration plant design. To eliminate partial pressure effects, the hydrogen purity of the dissolver gas was increased in run 36 to give the same reactor outlet hydrogen partial pressure for the two runs. The data in Table 30 shows that, within experimental error, yields were identical for runs 35 and 36.

Runs 36 and 37 and runs 38 and 39 show the effect of decreasing hydrogen partial pressure. A comparison of runs 36 and 37 shows about a 1.8% decrease in distillate and 1.3% increase in 900°F+ product for a decrease in hydrogen partial pressure of about 180 psia. Comparing runs 38 and 39 shows a decrease in distillate of 1.9% and an increase in 900°F+ product of 2% for a 70 psia decrease in hydrogen partial pressure. However, run 39 had to be terminated prematurely, due to a unit problem, and may not have been fully lined out. If the run 35-36 comparison is considered to be the more accurate, then it appears that distillate yield increases about 1% for each 100 psia increase in hydrogen partial pressure.

The four runs on Robinson Run coal (runs 41 to 44) represent a 2^2 factorial experiment varying coal concentration in the feed slurry and dissolver outlet partial pressure of hydrogen. An analysis of the data shows about a 2.4% increase in distillate, a 1% increase in C_1-C_4 gas, and 3.9% decrease in 900°F+ product for a 5% decrease in coal concentration. This trend of increased distillate yield with decreased feed coal concentration is consistent with previous results.

With respect to partial pressure effects, however, the Robinson Run data conflict with previous results. These data show a slight decrease in distillate yield and a slight increase in 900°F product yield with increasing partial pressure of hydrogen, which is the opposite of the effect of hydrogen partial pressure observed in other work. There are two possible explanations for these results. First, considerable difficulties were encountered during runs 41 and 42 with the vacuum column, which did not function properly and finally plugged completely. These problems may have contributed to the low distillate yields in runs 41 and 42. Second, the Robinson Run coal may be unusual and increased partial pressure of hydrogen does not have a beneficial effect with this coal. The first explanation appears more likely.

TABLE 28

PROPERTIES OF PITTSBURGH SEAM COALS

Mine	Powhatan	Valley Camp	Robinson Run
Elemental Analysis, Wt% of Dry Coal			
Carbon	73.4	74.0	68.7
Hydrogen	5.1	5.2	4.8
Nitrogen	1.3	1.4	1.3
Oxygen (by diff.)	7.3	7.6	4.9
Sulfur	3.4	2.9	3.6
Ash	9.5	8.9	16.7
Moisture	0.8	0.7	1.0
Spectral Ash Analysis			
Metals, Wt% of Ash			
Al	12	15	15
Ba	Trace	Trace	Trace
B	0.1	0.1	0.1
Ca	2	1	3
Cr	Trace	Trace	Trace
Cu	Trace	Trace	Trace
Fe	18	15	10
Mg	0.5	0.8	2
Mn	Trace	0.1	0.1
K	1.5	1	1.3
Si	20	25	20
Na	0.5	0.3	0.4
Sr	Trace	Trace	0.2
Ti	Trace	1	1
Proximate Analysis			
Moisture	0.8	0.7	1.0
Ash	9.5	8.9	16.7
Volatile	38	38	36
Sulfur Types			
Pyritic Sulfur	1.66	1.47	2.23
Sulfate Sulfur	0.04	0.03	0.07
Inorganic Sulfur	1.70	1.50	2.3
Organic Sulfur	1.70	1.40	1.3
Total Sulfur	3.40	2.90	3.6
Particle Size Distribution			
>80 Mesh	0	0	0
75 Microns	6	6	4
45 Microns	24	23	21
20 Microns	36	35	35
<20 Microns	34	36	40

TABLE 29

SUMMARY OF OPERATING CONDITIONS

Run No. P99-	35	36	37	38	39	41	42	43	44
Coal	<——— Powhatan ———>			<—Valley Camp —>		<——— Robinson Run ———>			
Temperature, °F									
Dissolver	851	851	851	851	851	851	851	851	851
Preheater Outlet	730	716	721	738	727	730	734	738	730
Pressure, psig	2000	2000	2000	2000	2000	2000	2000	2000	2000
WHSV, lb/ht/ft^3									
Slurry	76.1	76.1	76.1	76.1	76.1	76.1	76.1	76.1	76.1
As Received Coal	22.6	22.6	22.6	22.6	22.6	22.6	18.8	18.8	22.6
Nominal Slurry Residence Time, hr	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
Slurry Composition, Wt%									
Coal (Moisture Free)	29.5	29.5	29.5	29.3	29.4	29.4	24.5	24.4	29.5
Process Solvent	36.0	35.9	36.8	36.3	35.6	30.4	29.3	27.7	28.8
900°F+ Pyridine Solubles	21.7	22.1	21.2	22.7	23.4	24.1	25.2	26.7	24.3
Pyridine Insolubles	12.8	12.5	12.5	11.7	11.6	16.0	20.9	21.1	17.3
Dissolver Inlet Gas									
10^3 scf/ton feed coal	70.4	48.6	49.5	49.7	49.0	50.3	50.8	48.2	45.8
H_2 Content, Vol %	85	94	85	94	91	94	94	90	90
Hydrogen Partial Pressure* at Dissolver Outlet, psia	1325	1334	1152	1378	1308	1428	1401	1296	1298

*Calculated values, based on vapor-liquid equilibrium

TABLE 30

SUMMARY OF SRC II YIELDS WHEN FEEDING PITTSBURGH SEAM COALS

Run No. P99-	35	36	37	38	39	41	42	43	44
Coal	<—— Powhatan ——>			<- Valley Camp ->		<———— Robinson Run ——>			
Yield, Wt% of Moisture-Free Coal									
Hydrogen Comsumption	-4.3	-4.3	-4.3	-4.2	-4.0	-3.8	-4.1	-4.0	-3.6
Methane	5.9	5.6	5.8	5.8	5.6	5.1	5.3	5.3	4.9
Ethane	4.4	4.4	4.4	4.4	4.2	3.7	3.9	3.9	3.5
Propane	4.2	4.2	4.1	4.1	4.0	3.7	3.9	3.9	3.4
I-Butane	0.2	0.3	0.3	0.3	0.2	0.3	0.2	0.2	0.2
N-Butane	2.0	2.0	1.9	2.1	2.0	2.1	2.1	2.1	1.8
Total C_1 to C_4	16.7	16.5	16.5	16.7	16.0	14.9	15.4	15.4	13.8
Ammonia	0.4	0.4	0.4	0.5	0.5	0.5	0.5	0.5	0.5
Hydrogen Sulfide	2.3	2.3	2.3	1.9	1.8	2.1	2.3	2.3	2.2
Carbon Dioxide	1.1	1.1	1.2	1.0	1.0	0.7	0.6	0.8	0.8
Carbon Monoxide	0.2	0.2	0.2	0.1	0.1	0.2	0.2	0.2	0.2
Total Other Gases	4.0	4.0	4.1	3.5	3.4	3.5	3.6	3.8	3.7
Water	5.9	5.9	6.3	5.3	5.7	3.8	4.4	4.4	3.6
C_5-380°F	8.9	9.4	9.5	8.0	8.3	8.7	9.5	8.8	7.6
380-550°F	13.6	13.9	13.0	16.6	13.6	12.1	15.8	14.3	14.4
550-900°F	15.0	13.8	12.8	12.8	13.5	12.7	10.7	14.1	12.9
Total C_5+ Distillate	37.5	37.1	35.3	37.4	35.4	33.5	36.0	37.2	34.9
900°F+ Pyridine Solubles	25.3	26.0	26.4	24.9	27.5	23.9	21.8	20.6	24.0
Insoluble Organic Matter	5.4	5.3	6.2	7.5	7.1	7.5	6.2	5.9	6.9
Ash	9.5	9.5	9.5	8.9	8.9	16.7	16.7	16.7	16.7
Total 900°F+ Product	40.2	40.8	42.1	41.3	43.5	48.1	44.7	43.2	47.6
Fuel Oil (380-990°F)	28.6	27.7	25.8	29.3	27.1	24.8	26.5	28.4	27.3

REFERENCES

1. 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., May 1979, FE/496-157.

2. SOLVENT REFINED COAL (SRC) PROCESS, Quarterly Technical Progress Report for the Period January - March 1977. The Pittsburg & Midway Coal Mining Co., July 1977, FE/496-137.

APPENDIX A

TABLE A-1

Summary of Merriam Process Conditions, Yields, and Product Analyses

Run No.	DOE 234	DOE 235	DOE 237R	DOE 238R
Conditions				
Coal	Kentucky[a]	Kentucky[a]	Kentucky[a]	Kentucky[a]
Reactor	DOE 1	DOE 1	DOE 1	DOE 1
Nominal Liquid Residence Time, hr	1.03	1.06	1.07	1.05
Coal Feed Rate, lb/hr/ft^3	20.9	30.4	20.1	20.5
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	4.20	4.30	4.35	4.27
MSCF/ton of coal	52.7	36.0	54.6	53.7
Additive	--	--	--	--
Slurry Formulation, wt %				
Coal	30.0	45.0	30.0	30.0
Recycle Coal Solution	--	--	70.0	70.0
Recycle Solvent	70.0	55.0	--	--
Additive	--	--	--	--
Slurry Blend Composition, wt % (For SRC II runs)				
Coal			30.0	30.0
Solvent			25.4	24.3
SRC			26.9	29.8
Ash (from recycle coal solution)			12.2	10.6
Insoluble Organic Matter (from recycle coal solution)			5.5	5.3
Total Solids			47.7	45.9
Yields, wt % Based on MF Coal				
H_2O	9.4	8.7	8.9	7.1
CO	0.4	0.3	0.1	0.1
CO_2	0.6	1.1	1.0	1.0
H_2S	1.3	1.7	2.1	2.0
NH_3	--	0.4	0.6	0.5
C_1	4.0	4.1	5.8	5.1
C_2	2.9	2.9	4.1	3.5
C_3	2.1	2.1	3.2	2.8
C_4	0.9	1.0	1.5	1.2
Total C_1-C_4	9.9	10.1	14.6	12.6
Naphtha, C_5-193°C	10.7	8.0	7.4	6.9
Middle Distillate, 193-249°C	14.1	8.5	7.5	7.5
Recycle Solvent (heavy distillate), >249°C	4.1	12.4	24.6	24.8
Total Oil	28.9	28.9	39.5	39.2
SRC	39.4	38.2	22.5	28.0
Insoluble Organic Matter	4.3	4.6	4.6	5.0
Ash	9.9	10.2	10.2	9.9
Total	104.1	104.2	104.1	105.4
H_2 Reacted, gas balance	4.1	4.2	4.1	5.4
Lineout Index	--	--	1.01	1.12
Product Analyses				
Recycle Solvent (or Heavy Distillate) Analyses				
% C	88.37	87.72	88.48	88.27
% H	8.01	7.94	7.72	7.69
% S	0.30	0.37	0.36	0.37
% N	0.96	1.07	1.32	1.41
% O (By difference)	2.36	2.90	2.12	2.26
Specific Gravity	1.0414	1.0472	1.0674	1.0639
SRC (or Vacuums Bottoms) Analyses				
% C	88.47	88.82	60.67	65.11
% H	5.92	5.83	3.61	3.56
% S	0.50	0.55	3.08	2.77
% N	2.20	2.25	1.50	1.52
% Ash	0.16	0.10	30.28	26.01
% O (By difference)	2.75	2.45	--	--

a) Kentucky Nos. 9 & 14, Colonial Mine.

TABLE A-1 (Continued)

Run No.	DOE 239R	DOE 240C	DOE 240R	DOE 241R	DOE 242R
Conditions					
Coal	Pittsburgh[b]	Kentucky[a]	Kentucky[a]	Pittsburgh[b]	Pittsburgh[b]
Reactor	DOE 1	DOE 2	DOE 2	DOE 2	DOE 2
Nominal Liquid Residence Time, hr	1.00	1.00	0.99	0.98	1.00
Coal Feed Rate, lb/hr/ft^3	21.4	21.4	21.5	21.9	21.6
Nominal Dissolver Temperature, °C/°F	461/862	455/851	455/851	455/851	455/851
Dissolver Pressure, psig	1860	2000	2000	2000	1860
H_2 Feed, wt % based on slurry	5.84	5.46	5.42	5.37	5.02
MSCF/ton of coal	73.3	68.6	68.1	67.4	63.0
Additive	--	--	--	--	--
Slurry Formulation, wt %					
Coal	30.0	30.0	30.0	30.0	30.0
Recycle Coal Solution	64.0	--	65.0	63.0	64.0
Recycle Solvent	6.0	70.0	5.0	7.0	6.0
Additive	--	--	--	--	--
Slurry Blend Composition, wt % (For SRC II runs)					
Coal	30.0		30.0	30.0	30.0
Solvent	32.0		28.9	27.8	26.2
SRC	22.8		28.4	27.6	27.8
Ash (from recycle coal solution)	9.6		8.3	9.3	10.0
Insoluble Organic Matter (from recycle coal solution)	5.6		4.4	5.3	6.0
Total Solids	45.2		42.7	44.6	46.0
Yields, wt % Based on MF Coal					
H_2O	4.8	7.1	6.0	4.2	4.5
CO	0.4		0.3	0.4	0.3
CO_2	0.9		1.0	0.7	0.8
H_2S	1.9		1.9	1.9	1.6
NH_3	0.3		0.3	0.3	0.3
C_1	5.5	14.5	4.4	3.4	3.2
C_2	3.5		3.2	2.5	2.6
C_3	2.7		2.7	2.4	2.4
C_4	1.3		1.3	1.3	1.3
Total C_1-C_4	13.0		11.6	9.6	9.5
Naphtha, C_5-193°C	7.0	8.6	7.5	8.0	8.1
Middle Distillate, 193-249°C	10.2	14.8	7.3	7.1	6.7
Recycle Solvent (heavy distillate), >249°C	15.4	(10.4)[c]	17.7	12.3	14.0
Total Oil	32.6	13.0	32.5	27.4	28.8
SRC	30.4	52.7	34.8	38.3	36.0
Insoluble Organic Matter	7.5	5.6	5.4	7.4	7.8
Ash	12.9	10.1	10.1	12.9	12.9
Total	104.7	103.0	103.9	103.1	102.5
H_2 Reacted, gas balance	4.7	--	3.9	3.1	2.5
Lineout Index	0.98		1.15	1.03	0.98
Product Analyses					
Recycle Solvent (or Heavy Distillate) Analyses					
% C	88.84	88.83	88.59	89.06	88.69
% H	7.33	7.31	7.54	7.70	7.43
% S	0.37	0.41	0.49	0.40	0.44
% N	1.15	0.93	1.27	1.31	1.24
% O (By difference)	2.31	2.52	2.11	1.53	2.20
Specific Gravity	1.0709	1.0703	1.0782	1.0814	1.0840
SRC (or Vacuums Bottoms) Analyses					
% C	66.83	87.43	67.87	69.22	69.41
% H	3.77	5.72	3.87	4.03	4.00
% S	2.31	0.74	2.57	2.15	1.91
% N	1.41	2.13	1.57	1.44	1.41
% Ash	24.00	0.18	23.44	22.65	22.40
% O (By difference)	--	3.80	--	--	--

a) Kentucky Nos. 9 & 14, Colonial Mine. b) Pittsburgh seam, Blacksville Mine No. 2 c) Loss

TABLE A-1 (Continued)

Run No.	DOE 243	DOE 243A	DOE 244	DOE 245	DOE 246
Conditions					
Coal	--	--	--	--	--
Reactor	DOE 2	DOE 2	DOE 2	DOE 2	DOE 2
Nominal Liquid Residence Time, hr	1.00	0.98	0.98	0.95	0.96
Coal Feed Rate, lb/hr/ft^3	--	--	--	--	--
Nominal Dissolver Temperature, °C/°F	455/851	455/851	455/851	455/851	400/752
Dissolver Pressure, psig	1860	2000	2000	2000	2000
H_2Feed, wt % based on slurry	5.03	5.36	5.37	5.22	5.28
MSCF/ton of coal	--	--	--	--	--
Additive	--	--	Pyrite	Mineral Residue	Pyrite
Slurry Formulation, wt %					
Coal	--	--	--	--	--
Recycle Coal Solution	--	--	--	--	--
Recycle Solvent	100.0	100.0	97.5	94.3	94.3
Additive	--	--	2.5	5.7	5.7
Yields, wt % Based on Solvent					
H_2O		0.50	0.66	0.66	0.25
CO		0.01	0.01	0.04	--
CO_2		--	0.02	0.02	--
H_2S		0.24	0.71	0.29	1.70
NH_3		--	--	--	--
C_1		0.36	0.34	0.32	--
C_2		0.21	0.21	ND	--
C_3		0.06	0.04	0.04	--
C_4		0.03	0.03	0.02	--
Total C_1-C_4		0.66	0.62	0.38	--
Naphtha, C_5-193°C		1.22	1.04	1.46	0.31
Middle Distillate, 193-249°C		3.11	3.06	3.30	2.28
Recycle Solvent, >249°C		94.63	94.94	94.24	97.15
Total Oil		98.96	99.04	99.00	99.74
SRC		--	--	--	--
Insoluble Organic Matter		--	--	--	--
Ash		--	--	--	--
Total		100.37	101.26	100.39	101.69
H_2 Reacted, gas balance		0.37	0.58	0.39	0.10
Product Analyses					
Recycle Solvent					
% C	88.52		88.59	88.76	88.87
% H	7.72		7.61	7.62	7.47
% S	0.44		0.40	0.38	0.44
% N	0.78		0.79	0.81	0.85
% O (By difference)	2.54		2.61	2.43	2.37
Specific Gravity	1.0600		1.0560	1.0562	1.0612

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