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SOLVENT REFINED COAL (SRC) PROCESS

Quarterly Technical Progress Report

for the Period

January - March 1977

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PREPARED FOR THE UNITED STATES
ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION

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CONTENTS

LIST OF TABLES

LIST OF TABLES (Cont.)

LIST OF FIGURES

ABSTRACT

This report summarizes the progress of the Solvent Refined Coal (SRC) Project by The Pittsburg & Midway Coal Mining Co. for the Energy Research and Development Administration for the period January 1, 1977 to March 31, 1977. Activities include the operation and modification of the Solvent Refined Coal Pilot Plant at Fort Lewis, Washington and process development work at the laboratory in Merriam, Kansas.

I. Summary of Operations

The Fort Lewis SRC Pilot Plant was operated only 14 days during January prior to being shut down for SRC II modification. During the January operating period, the filtration test program begun in late 1976 was completed. Results of the program are presented in this report.

For the remainder of the quarter the pilot plant underwent extensive inspection, repairs, and modification in preparation for SRC II operation. In the SRC II process, formerly referred to as the slurry recycle mode of operation, a portion of unfiltered coal solution will be recycled to the slurry preparation area. The longer residence time of the slurry, the somewhat higher reaction pressure, and the recycle of unfiltered coal solution yield a liquid fuel as the principal product.

Process development work at the Merriam Laboratory has defined the operating parameters for SRC II operation using Kentucky No. 9 coal. A brief series of trials using the Amax coal in an SRC I mode of operation was also begun. A summary of the Merriam SRC II work is presented in this report.

II. Pilot Plant Operations

The plant operated for 14 days during January to generate filtration data using "B" filter and the Johnson Screen. Reactor area conditions were held at a constant 3500 lb/hr coal feed and a 1.6 solvent-to-coal ratio to provide a consistent filter feed. Ninety-four tons of specification SRC were produced during this period.

Analytical data from the January operating period is presented in the following tables. Only waste treatment units were in operation the entire quarter.

Table 1

Average Coal Analysis - January 1977 (wt %)

Carbon	71.35
Hydrogen	5.07
Nitrogen	1.44
Sulfur	3.50
Oxygen (by difference)	7.55
Ash	10.12
Moisture	0.97

Table 2

Sulfur Forms (wt % on coal)

Pyritic Sulfur	1.63
Sulfate Sulfur	0.09
Organic Sulfur	1.76
Total Sulfur	3.48

Table 3

Average Mineral Residue Analyses (wt %)

Carbon	27.61
Hydrogen	1.39
Nitrogen	0.54
Sulfur	7.29
Ash	63.17
Pyridine Insoluble	96.98

Table 4

Solvent Distillation*

Distillation Product	Distillation Fraction		
	Up to 380°F	380 to 480°F	480 to 850°F
Light Oil	45%	47%	8%
Wash Solvent	24%	75%	1%
Process Solvent	0%	11%	89%

* During this period the Light Ends Column was operated unpacked and without reflux as a topping still.

Table 5

Elemental Analyses of Plant Solvents

Description	Light Oil	Wash Solvent	Process Solvent
% Carbon	84.17	83.17	87.81
% Hydrogen	9.05	8.82	7.66
% Nitrogen	0.37	0.67	0.66
% Sulfur	0.26	0.14	0.45
% Oxygen (by difference)	6.15	7.20	3.42
% Dowtherm	--	--	1.52

Table 6

Average Analysis of SRC

% Carbon	86.46
% Hydrogen	5.61
% Nitrogen	1.97
% Sulfur	1.00
% Oxygen (by difference)	4.72
% Ash	0.24
Heating Value (Btu/lb) - (Dulong)	15,750
Fusion Point (^{0}F) (Gradient Bar)	350

Analyses of the waste unit effluents is given in Table 7. Operation of the units has been erratic during the quarter due to the cleaning of the surge reservoir and the plant cooling system. When necessary, effluent from the bio-unit was recycled.

Table 7

Process Waste Treatment Analyses

	Bio-Unit Feed	Bio-Unit Effluent	Plant Effluent (Composite)
pH	7.0	6.4	6.7
Total Suspended Solids, ppm	184	51	5.5
Phenol, ppm	6.3	0.17	0.06
Chemical Oxygen Demand, ppm	529	141	31
Biological Oxygen Demand, ppm	24	2.5	0.9

III. Pilot Plant Engineering, Maintenance and SRC II Modifications

A. Introduction

After completion of the filtration test runs in January all process areas were shut down for SRC II modification and maintenance. Utility units also were shut down for varying lengths of time during the quarter for inspection and maintenance.

B. Coal Receiving and Preparation - (Area 01)

The following work was performed during the first quarter of 1977:

1. A new belt was installed and the gravimetric feeder calibrated in January.
2. New rotary air locks were installed at the inlet to the coal pulverizer and the discharge of the pulverized coal bag house.
3. One new pulverizer ball was installed.
4. The pulverized coal scrubber shell and eductor were replaced with new parts.
5. Cooling water jacketed packing glands were installed on both high pressure charge pumps.
6. The dehumidifier cooling water exchanger was pressure tested and numerous tube leaks were discovered. The severe tube side corrosion is believed to have been caused by high concentration of carbon dioxide and water vapor in the circulating inert gas stream. A used replacement exchanger of stainless steel construction was located and was prepared for installation.

C. Preheating and Dissolving - (Area 02)

In the reaction area, the following occurred:

1. The 1/4" micro form trim set installed in LCV-166B in December performed successfully during start-up operation in January. This was a test to determine the feasibility of using smaller trim in LCV-166A in an effort to lengthen the valve service life. One-fourth (1/4) inch tungsten carbide and 1/4" ceramic trim sets have been ordered for LCV-166A, for installation at a later date.

2. Coke deposits were found in the bottom six to seven feet around the wall of "A" dissolver after completion of the January filtration test. Coke was also found in the last 25 feet of the 4" SL-13 line upstream of "A" dissolver, the 4" SL-15 and the 4" SL-16 lines between "A" dissolver, and the dissolver product quench cell. The coke was removed by hydroblasting.

3. "A" dissolver, the high pressure flash vessel, the intermediate pressure flash vessel, and the recycle condensate separator were hydroblasted and inspected for metal loss. The only item requiring further attention will be the intermediate flash separator head which will require replacement in the near future. Bids are currently being requested. Corrosion racks were installed in each vessel and the vessels were readied for service. A new head, with hydrogen quench connections, will be installed on dissolver "A" prior to SRC II startup. The unmodified original head will be used during startup in the SRC I mode.

4. The emergency isolation valve between the dissolver and the high pressure flash vessel (FCV-2119) was removed and replaced by a spool piece.

5. The wide radius elbow on the inlet to "A" dissolver was removed and sent to the Oak Ridge National Laboratory for destructive testing.

6. New EBV isolation valves were installed on the high pressure flash letdown valves (LCV-166 A&B) and the intermediate pressure flash letdown valve (LCV-175).

D. Mineral Separation and Drying - (Area 03)

Filter "A" was brought down twice during the January operating period for suspected knife malfunction. When opened the second time, a loose knife wash solvent header mounting bracket was discovered which had allowed the header to rotate. The bracket was welded and the header and nozzles were cleared. New flexible grease lines were installed, the screen hydroblasted and the drive chain was shortened prior to returning the filter to operation.

Other maintenance and modification changes made during the quarter were as follows:

1. "A" and "B" filter screens were hydroblasted and the vats washed out and made ready for service.

2. "A" and "B" wash solvent heater tube bundles were removed, hydroblasted, and made ready for service.

3. "A" and "B" vapor scrubbers and vapor surge drums were cleaned, inspected, and made ready for service.

4. The filter feed surge vessel agitator motor and drive were inspected and found to be satisfactory. The agitator bottom steady bearing was repaired. The vessel was inspected and returned to service.

5. The mineral residue dryer was manually cleaned. The vapor line was hydroblasted from the dryer outlet breech to the scrubber.

6. SRC II related modifications were begun on the recycle process water tank, the Nos. 1 and 2 flash condensate separators and the new tie-ins to the Dowtherm headers.

7. All pressure relief valves were tested and reinstalled.

8. Double block valves and bleeders were installed on the Dowtherm inlet lines to "A" and "B" wash solvent heaters.

9. Both shell-to-head gaskets were replaced on "A" filter gas heater.

10. The filter feed flash vessel was inspected and returned to service.

11. A new demister pad was installed in the dryer condensate drum. The vessel was inspected and returned to service.

12. The drive chain on the mineral residue cooler was repaired and all cooling water spray nozzles replaced.

13. The emergency flare vent line from the mineral residue dryer was cut for the installation of a block valve to facilitate blinding procedures during dryer shutdown.

E. Solvent Recovery - (Area 04)

A substantial amount of cleanup, repair, and modification was needed in the solvent recovery area. After completion of the modification work, the solvent recovery area will be returned to its original lineup, and the light ends column will be repacked with Pall rings. When operating in the SRC I mode, the new small vacuum flash drum, which was installed as part of the SRC II construction, will be used as a preflash vessel to remove low boiling fractions ahead of the vacuum flash preheater. The overhead pressure control systems of the light ends column and wash solvent column were separated to permit better pressure control.

Work performed in the 04 Area is as follows:

1. An agitator was installed in the wash solvent accumulator. Also, a new pressure control loop was installed.
2. Tie-ins from the new vacuum flash system into the old were made. The new vacuum flash heater and drum were set on the sixth floor of the Area 04 structure.
3. Double block valves and bleeders were installed on the five new tie-ins off the main Dowtherm headers. This will allow startup of the Dowtherm system before construction is completed.
4. Two new pressure control loops were installed on the wash solvent column reflux drum (PCV-3014 "A" and "B").
5. New tracks were welded in the wash solvent column reboiler shell. The old reboiler tube bundle was hydroblasted. A new stainless steel tube bundle was installed and new corrosion racks welded to the shell. (The old bundle will be installed as a temporary light ends column preheater bundle until a new bundle arrives in April.)
6. The old light ends column reboiler shell was removed and replaced with a stainless steel shell. Corrosion racks and probes were installed in the column and also on the reboiler tube bundle and shell. The column was reassembled and Pall rings installed.
7. The light ends column flare line was reinstalled with new heat tracing.
8. Revisions were begun for the temporary light ends column feed exchanger.
9. The suction line for the vacuum bottoms recirculation pumps was moved from the mezzanine deck to a "Y" immediately above the vacuum flash drum level control valve which is just above the Sandvik belt.
10. A corrosion rack was installed in the vacuum flash condensate drum.
11. The line from the old vacuum flash preheater to the vacuum flash drum was replaced because of excessive thinning.
12. New filter elements were installed in the seal flush filter, and the seal flush cooler was pressure tested. One tube was found to be leaking and was plugged.
13. The flush solvent heat exchanger was removed due to severe fouling on the process side. A spool piece was installed and will be used until a new exchanger is delivered. The

flush solvent piping was modified so that the electric heaters could heat the flush solvent. A leaking thermowell was also replaced above the exchanger.

14. All steam leaks on the main steam header were repaired.

15. Orifice flanges were installed in the Dowtherm return lines on the light ends column feed exchanger and reboiler and the wash solvent column reboiler.

16. Manways were reinstalled on all vessels except the vacuum flash drum and the wash solvent accumulator.

17. Installation was begun on new lines to bypass the wash and process solvent accumulators.

18. The sewer line headers between Areas 03 and 04 were hydroblasted as were several small plugs in lines around the old vacuum preheater and vacuum flash drum down leg to the Sandvik belt.

F. Gas Recovery and Recompression - (Area 05)

Items in the 05 Area completed or started during the quarter were:

1. The suction and discharge valves were replaced in both the fresh and recycle hydrogen compressors. Mechanical and electrical work was begun on the new recycle hydrogen compressor.

2. Work continued on the new naphtha absorber and the spare recycle hydrogen compressor.

3. All pressure relief valves were tested and reinstalled.

4. The fresh hydrogen scrubber and the hydrogen cooling water exchangers were inspected and prepared for service.

G. Product Solidification and Storage - (Area 08)

The Aercology Centri-Clean centrifugal separator, installed during the last quarter to remove particulate-mist from the Sandvik belt exhaust, was run on the fumes from the Sandvik belt hood at various times during January after the original configuration was modified for better performance. The drain lines and filter element housing were steam-traced and insulated. The outlet secondary filter was removed to prevent plugging with entrained droplets from the oil drainage chamber. New duct work was installed to route the exhaust through a catch pot. After a day and a half of operation with the exhaust flowing through the catch pot, the catch pot had not been dirtied by oil indicating that mist from the Sandvik belt is being captured.

The amount of oil recovered varied with the operation of the vacuum tower. As much as three quarts per hour have been collected. The oil recovered was tested in the laboratory and found to be predominantly process solvent.

A gravimetric weigher was installed to weigh the SRC product to the dump truck loading area for mass balance runs. To keep the weigher inlet from plugging due to large pieces of SRC, the SRC breaker was reinstalled on the Sandvik belt discharge.

H. Waste Treatment - (Area 09.1)

The surge reservoir and the reactivator were bypassed and cleaned during the quarter. The reactivator internals were inspected and found to be in good condition. Four sample taps were installed which will allow better control of the circulating solids concentration and volume.

The activated sludge (Oxycontact) unit was drained and the defective air sparger system was repaired. Inspection revealed the unit was in good structural condition. Some pitting corrosion was found on the tank walls. The pitted areas were wire brushed and painted. A block valve was installed on the inlet line.

The activated charcoal filter media was replaced. Sand filter media replacement will be made upon delivery of specification sand.

I. Cooling Water and Flare Systems - (Area 09.3)

The cooling water system was acid cleaned during February using a solution consisting of 5% hydrochloric acid, stannous chloride and ammonium bifluoride. The system was returned to normal operation after the chemical cleaning was completed.

The entire flare system was removed from service and prepared for SRC II modifications. The system was blinded from all process equipment and steam purged for hot work. All pressure relief valves will be removed, checked, and repaired prior to reinstallation.

J. Gas System - (Area 09.5)

1. Inert Gas Unit

The inert gas unit was shut down for inspection and repairs early in March. On March 2 the Deoxo catalyst vessel was relocated to grade level which should eliminate the compacting and attrition of the catalyst that occurred in the past due to excessive vibration.

Inspection of the firebox revealed loss of and damage to the burner refractory and the burner end of the heat shield. The burner refractory was repaired by replacing about 75% of the old refractory and extending it approximately two feet farther into the firebox. The heat shield was repaired by installing a new shroud over the old one.

The inert gas surge vessel was modified so that now all the gas flows through the vessel rather than the vessel acting as an out-of-the-line pressure reservoir. This should help eliminate the carryover of moisture into the system. Finally, a new tube bundle was installed in the west MEA cooler.

2. Hydrogen Unit

The charcoal desulfurizers were opened for inspection in early February. Severe attrition of the charcoal was evident as the particles were smaller than original with a large amount of fines present. Both the upper and lower support screens in the west vessel were torn loose, and the bottom screen on the east vessel was ruptured. New screens were installed and the support rings modified to withstand greater loading. Both vessels were recharged with charcoal (Girdler 32-J and 32-W) and returned to service.

Thermocouples were installed in both the high and low temperature shift catalyst beds. The new thermocouples will record the inlet and outlet temperature of each bed. Prior to the installation there was no temperature indication on either bed.

The high temperature shift converter catalyst was dumped. A four inch deep layer of high temperature alumina support balls was placed in the vessel and the converter reloaded with new Girdler 3A catalyst.

The low temperature shift converter was loaded with fresh Girdler G-66RS catalyst in early March. On March 14 it was discovered that the catalyst temperature had suddenly increased. The cause of the temperature excursion and its potential effect on the catalyst are under review.

The transfer line from the reformer to the high temperature shift reactor was replaced with 304 stainless steel. Two pinhole leaks were found in the original carbon steel line during the past year.

The primary reformer was started in order to decoke, desulfurize and to oxidize the catalyst prior to opening for inspection. During this time, the shift converters were bypassed. Burners were lit on March 20 to start this procedure and it was completed on March 24.

Inspection of the top section of the reformer showed that several expansion joints between the tube outlets and the reformer cooler had to be replaced. Of the ten expansion joints, eight will be replaced. On March 25, the top tube flanges were removed. There were signs of coke formation on the catalyst and piping. Several of the tube centers (refractory heat shields) on the outlet piping had reached excessive temperature and had crystalized or become embrittled. (These tube centers will be replaced during the next shutdown). The catalyst in the tubes appeared to be in satisfactory condition although the outage distance was very erratic, ranging from 15" to 35". A pressure drop survey was carried out on each tube, and the maximum pressure drop was 8% greater than the mean pressure drop. The tube with the highest pressure drop also had the greatest outage measurement. Although the pressure drop variances were above 5%, it was decided not to reload the catalyst. Catalyst hold down grids will be placed inside the tubes to eliminate catalyst carryover from the tubes.

Other items worked on in the hydrogen plant area included:

1. The shift converter aftercooler was tested and found to have several tube to tubesheet leaks. This bundle was sent out for repair.
2. The quench chamber was opened for inspection and repairs. The inlet line elbow was replaced, a missing quench nozzle was replaced and an access manway was installed.
3. A flow transmitter and indicator was installed on the quench steam to the high temperature shift.
4. Preventive maintenance work was completed on the hydrogen compressors.
5. A modified pressure control station was installed to vent excess hydrogen when process demands are less than production. This loop will be completed in early April.

3. DEA Unit

The DEA unit was shut down as scheduled for SRC II modifications. Routine repairs and some preventive maintenance on pumps were also accomplished.

Based on wall thickness measurements and engineering requests, some high pressure piping connected to the absorber and flash drum was replaced.

K. Dowtherm System - (Area 09.8)

Items worked during the quarter on the Dowtherm system included:

1. Eight tie-ins to the main headers were made for SRC II modifications.
2. The surge drum was inspected and approved.
3. New check valves were installed on the discharge of the circulating pumps.
4. All leaking valves on the system were repacked, repaired, or replaced.
5. All flow indicators were cleaned and checked.
6. All pressure relief valves were removed, tested, and reinstalled.
7. Raw water lines were run to the circulating pump seals to eliminate cooling tower water loss via those seals.
8. A new temperature element was installed on the reclaimer unit.

IV. SRC II Modifications

A. Summary

The SRC pilot plant was shut down during most of the quarter and extensive modifications were made to the plant to provide the operating flexibility necessary for the SRC II mode of operation. The SRC II operation differs from the SRC I mode in that unfiltered coal solution, in whole or in part, is used to slurry feed coal rather than a recycled distillate solvent. This, together with higher hydrogen partial pressures and longer slurry residence times than typically used in the SRC I process, provide a distillate oil product as the principal fuel produced and reduces distillation residue yields sufficiently that under some process conditions they are in balance with feed stock requirements for hydrogen generation.

A brief SRC II experimental program at the pilot plant in 1975 indicated that the plant, as then configured, could operate in the SRC II mode only at relatively low coal concentrations and relatively short slurry residence times. It also showed that the coal eductor used in SRC I operation to slurry coal in the recycle solvent was not really suitable for operation with the more viscous recycle unfiltered coal solution, that hydrogen availability might limit plant operation in the SRC II mode, that better temperature control in the dissolver by provision for quench hydrogen addition would be quite helpful, or perhaps necessary, for SRC II operation, and that a scaled down vacuum preheater and vacuum flash drum would be needed to obtain data useful in scale-up to commercial design in the SRC II mode.

B. Description of SRC II Modifications

In order to provide capability of operation in the SRC II mode over a fairly wide range of conditions, the following conditions and modifications were made.

1. Slurry Preparation - Area 01

A mix-tank system, in which a high speed agitator creates a vortex in the slurry and coal is added into the vortex, was installed for mixing coal and recycle slurry in the SRC II process. A vent condenser for refluxing solvent vapors to the slurry and an additional recirculation pump were also installed.

2. Dissolver - Area 02

A new head, having connections for three separate hydrogen quench locations at differing elevations in the dissolver, was installed on Dissolver "A" to allow dissolver temperature control by quench hydrogen addition. The unfiltered coal solution stripper internals were modified and a new oil-water separator which will allow adequate separation of oil and water in the dissolver effluent was installed. The original separator system has been inadequate since water injection was adopted for dissolver effluent cooling shortly after initial plant start up.

3. Solvent Recovery - Area 04

A new smaller vacuum flash drum and a smaller Dowtherm heated vacuum flash preheater and associated piping was installed. The smaller drum will provide data at vapor velocities anticipated for commercial design. The new preheater and flash drum will be used as an atmospheric flash when operating in the SRC I mode. The light ends column, wash solvent column, and the original vacuum flash system were returned to their original flow scheme. This will allow better fractionation of the liquid products while retaining the atmospheric flash found necessary to prevent recurring coking of the vacuum flash preheater.

4. Gas Treating and Compression - Area 05

A naphtha scrubbing system was installed to remove hydrocarbon impurities from the recycle hydrogen stream so less hydrogen-rich gas would need to be purged and more would be available for recycle. A larger recycle hydrogen compressor was installed and the fresh hydrogen compressors were modified to increase their capacity by about 20%.

V. Pilot Plant Special Studies

A. Filtration Test Program

1. Objectives

The objectives of the Ft. Lewis study were to observe overall effects of filter operating variables on filtration rates and to investigate alternatives for filtration optimization.

2. Accomplishments

Twelve runs were completed during the test period. Each run was designed to investigate the effects of a variable on filtration rates. A factorial experimental design was not chosen due to the time constraints on the program which precluded performing enough experiments in a factorial design to adequately explore the recognized process variables over their ranges. Attempts were made to begin filtration optimization toward the end of the program, but plant operating problems prevented this.

It was not possible to evaluate individual filtration resistance terms in this study.

3. Discussion

Toward the end of the SRC I production run, a test program was carried out to obtain filtration data on the plant rotary pressure precoat filters. Two filters were in use in the plant, one having a nominal screen area of 80 sq. ft. and the other a nominal screen area of 40 sq. ft. All of the tests described were conducted on the smaller filter, Filter B, which had an actual screen area of 41.6 sq. ft. Prior to the start of these tests, a Johnson screen had been installed on Filter B. The Johnson screen is a slotted screen, with trapezoidal shaped bars, which should make screen blinding less likely to occur.

No comparative tests have been made with 110 x 24 mesh Dutch weave screens, which have had widespread use at Ft. Lewis. The Johnson screen is an experimental screen in this service that has been installed for evaluation of its operability, potential to minimize screen blinding, and to increase screen life.

The filtration rate of the rotary precoat pressure filter was modeled in the following manner. The filtration rate (F) was assumed to be affected by three resistance variables, expressed as:

F = f (precoat resistance, cake resistance, interface resistance)

Precoat resistance can be described as the flow resistance due to the filter aid itself. This term would be strongly related to the viscosity of the filtrate and the pore size of the precoat. Cake resistance results from formation of a layer of solids on the surface of the precoat. In a typical filter feed slurry, the distribution of particle size shows a substantial particle number fraction in the submicron range. Such a fine material could form a cake with very small pore size, much smaller than that of the filter aid. The last resistance term, interface resistance, results from penetration of solid material into the precoat.

The principle behind operation of the rotary precoat filter is to deposit solids and then shave off a thin layer of material each time the drum rotates. It is possible to shave off all the cake formed during the revolution and a small increment of precoat. If there is no penetration of solids beyond that precoat which is removed by the knife, the only resistance to flow immediately after cutting the cake is the precoat resistance. In some instances this is not the case since some solids have penetrated beyond the depth of the knife cut and, as a result, the pore size of the precoat layer is reduced.

In all, seven operating variables were tested in this program. A description of these variables follows.

a. Precoat Application: Two precoat application procedures were followed. The standard P&M procedure was used as a base condition and the second procedure was used for comparison. It was expected that the two procedures would yield precoats which were packed to different degrees.

b. Precoat Grade: Two grades of precoat, Speedex and Speedplus, both flux calcined diatomite filter aids manufactured by Dicalite, were used in these tests. Speedex is a coarser filter aid (mean particle size of 10-12 microns) than Speedplus (mean particle size of 8.8-9.4 microns). Manufacturer's data also state that Speedex has a higher relative flow rate than Speedplus (1030 versus 700). From this information, one would expect a cake with smaller pores from Speedplus if precoating procedures were held constant. The small pore size was expected to limit solids penetration into the precoat. This benefit could be offset to some degree by the greater precoat resistance of the finer Speedplus.

c. Knife Advance Rates: Previous experience at Ft. Lewis and elsewhere had shown that increases in knife advance rates in the range of 1-5 mil/min resulted in increased filtration rates. Most runs were at constant drum speed, which would result in comparable knife penetrations per drum revolution. When drum speed was changed, knife advance rates were expressed in units of mils per revolution which provides information on the unit rate of knife penetration per revolution.

The deeper the knife cut the more likely that all of the penetrated solids will be removed. At some point, all of the solids will be removed and additional increases in knife advance would serve only to decrease precoat resistance.

d. Drum Speed: Drum speeds in the range of 1-6 RPM were available for Filter B. The higher drum speeds result in shorter drum submergence times on each cycle at a constant vat level. By minimizing submergence time, two effects are realized:

   1. The time available for solids penetration into the precoat is minimized.

   2. The thickness of cake built up over the precoat is decreased.

e. Drum Submergence: Another variable which was studied is drum submergence, which can be varied between 9% diameter submerged to about 40% submerged. The upper limit on this range is due to the location of the top of the cake leg. At a drum submergence of more than about 40% diameter, the slurry in the filter overflows into the cake leg. At submergences much in excess of 30% diameter, the drum bearings are submerged in filter feed. Due to continual lubrication problems in the filter, a decision was made to conduct these tests at drum submergences of 30% diameter or less.

f. Pressure Drop: The driving force which causes the filtrate to flow through the cake and precoat is pressure differential. Increased pressure differential in the filter can promote several effects, some of which occur simultaneously. First, higher pressure differentials provide more driving force to cause the filtrate to flow through the cake. Second, higher pressure differential may cause additional compacting which would result in a precoat and cake with smaller pore sizes and, hence, lower relative flow rates. Third, due to the increased driving force causing higher filtrate rates, solids penetration into the precoat may increase, resulting in a higher level of interface resistance. Alternatively, if the precoat is compacted, then smaller pore sizes might limit solids penetration.

g. Cake Wash Rate: The rate at which wash solvent is sprayed on the cake is another variable which was taken into consideration in this study. The cake wash is used ostensibly to wash out the process solvent and SRC imbibed in the filter cake so that, when the cake is removed, only wash solvent remains to be removed in an atmospheric dryer. The cake wash also serves to dilute or wash out the liquids in the precoat. It has been felt that by washing out process solvent and SRC, the viscosity of the liquid imbibed in the precoat will be very low and the initial filtration rate, as it dips into the slurry, will be very high. The extent to which this is true was studied in these tests.

At the beginning of these tests, it was apparent that parameters would have to be chosen for comparing and analyzing the data obtained during the runs. Three parameters for comparison were chosen: filtration rate, solids removal rate per unit of precoat, and gas flow rate through the filter. The filtration rate was taken as the flow rate from the receiver and was assumed to consist of the filtered coal solution plus the amount of wash solvent sprayed on the cake. Throughout this discussion, filtration rate always includes the cake wash.

The solids removal rate was calculated by computing the rate of filtered coal solution and using the average pyridine insolubles content of the filter feed for the run to estimate the quantity of feed solids removed. There were some minor variations in feed composition between runs. The volumetric rate of precoat removal was calculated by measuring the total distance of knife travel during an hour and computing the corresponding volume which would have been shaved under these conditions. The rate of solids removal per unit volume of precoat removed was then calculated. The last parameter used for comparison of filtration operating conditions was gas rate through the filter in units of pounds per hour. The gas rate was found to be a good measure of cake permeability and probably can be used as an estimate of relative flow rate.

4. Experimental

An experimental program, consisting of twelve runs, was carried out to obtain process information on the variables previously described. Each run was designed to provide data on the primary effects of at least one variable, and, in several experiments secondary, interactive effects were also investigated. At the beginning of the program, it was recognized that some severe problems existed in obtaining data adequate for analysis. First, precoat application had the potential to result in precoats with different relative flowrates. Second, the effect of run duration or precoat aging on filtration rate was unknown. This second effect would potentially result in drifting filtration rates during a run even though all operating conditions were held constant. The possible combined effects of these two problems greatly influenced the experimental program.

A decision to investigate the effects of different levels of a single variable in one run was made, based on an analysis of the problems listed above. In addition, each run was begun with a fresh precoat. To provide a means of comparing data from different runs, a set of base conditions was established. Each run in this program started at those base conditions, and the base conditions were maintained until filtration rates stabilized. The selection of a set of base conditions permits comparison of the filtration rate at the same point in each run, the only difference being due to precoat.

The base conditions and a brief description of the variables investigated in each run are listed in Table 8. The base conditions were chosen to represent what was felt to be midrange operating conditions when the program started.

Each run began with a fresh application of precoat. A simplified flow sheet representing the precoat loop is shown in Figure 1. Precoat application was preceded by introduction of a basecoat slurry (containing 10 lbs. of a diatomite-asbestos fiber mixture) into the filter. The precoat filter aid was then charged into the filter through a piston-type positive displacement pump. The filter aid slurry was diluted to a concentration of approximately 5 wt % solids in the filter by process solvent recirculation from the precoat slurry pressure vessel. Typically, this process would result in precoat application over a span of 3-4 hours.

Upon completion of the precoat cycle, filter feed, containing some solid material, was introduced into the filter. The resultant filtrate solids concentration (clarity) was monitored until acceptable clarity (trace amounts of pyridine insolubles) was obtained. When the clarity was acceptable, the filter was lined up to start the filtration run.

Figure 2 schematically shows the flow scheme which was followed during the tests. The filter was fed directly from the filter feed flash vessel (FFFV). The feed to the flash vessel consisted of undiluted unfiltered coal solution from the reaction area and overflow from the filter. (Overflow from the filter was provided for filter temperature control and to minimize settling of pyridine insolubles in the filter). It was felt that the best way of controlling the feed composition during these tests was to prevent contamination of the unfiltered coal solution and to feed only fresh material. Attempts were also made to limit feed composition variations by holding reaction conditions constant.

Figure 2 shows that wash solvent is used in the filter as cake wash, knife wash and auger wash. Of these three streams, the cake wash is the only one which should influence filtration rate. In all of these tests, the assumption was made that all of the cake wash passes through the precoat and is included in the filtration rate. Filtration rate was measured as the liquid flow out of the filtrate receiver. Any liquid carried out of the filtrate receiver in the gas stream and condensed upstream of the compressors was reinjected into the gas stream recirculated into the filter.

Figure 1

PRECOAT CYCLE FLOW SCHEME

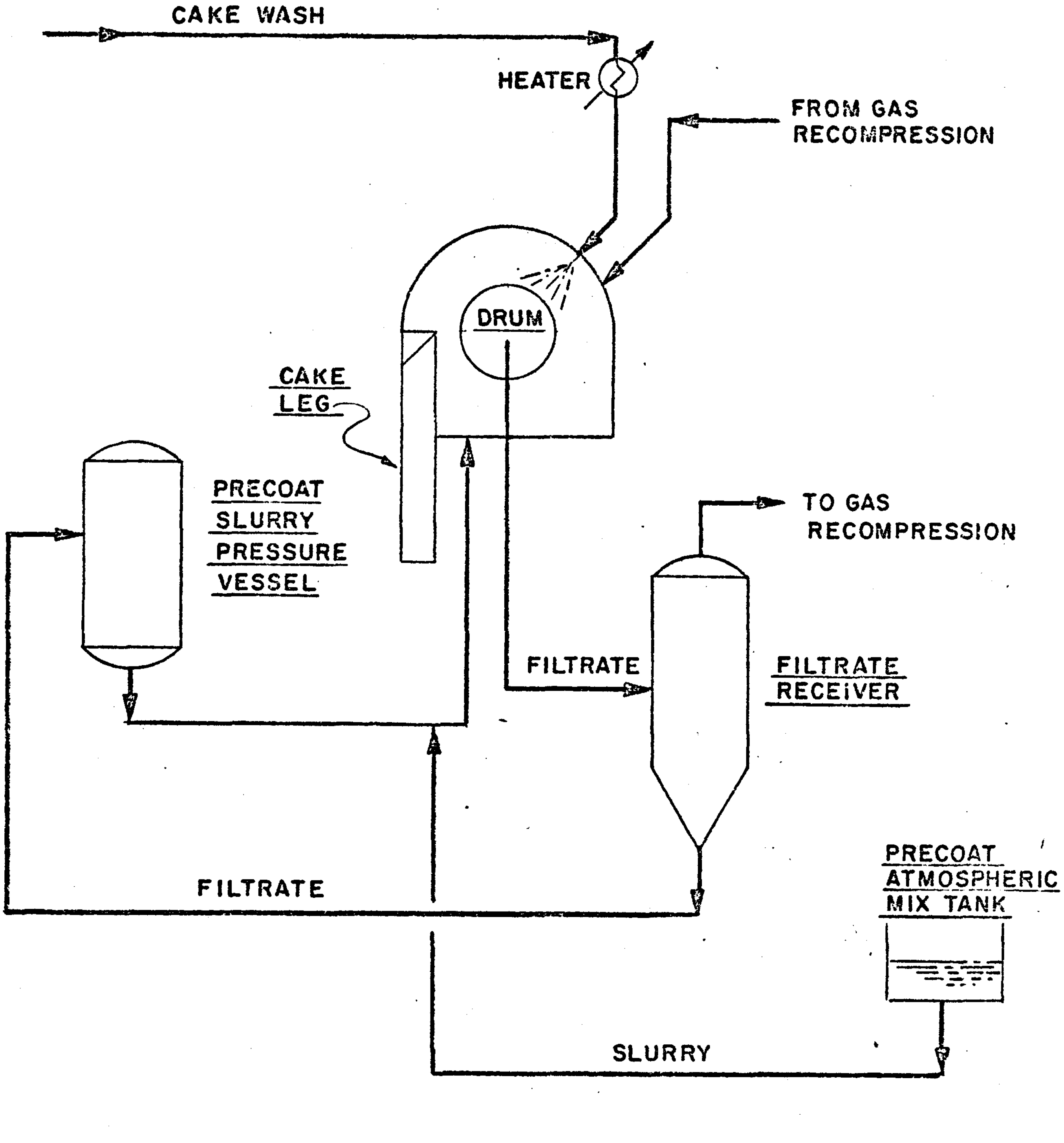


Figure 2

FILTRATION CYCLE FLOW SCHEME

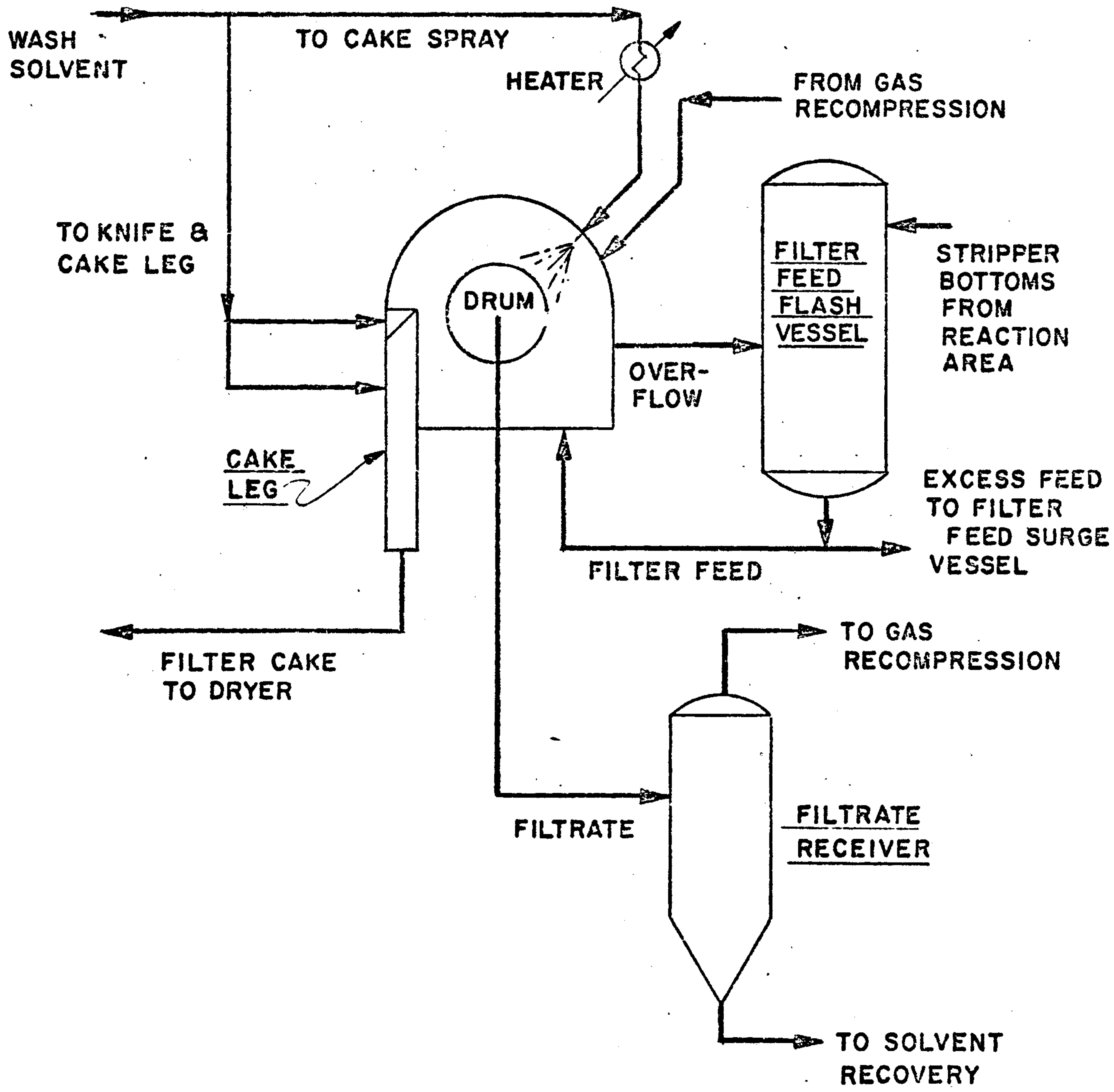


TABLE 8

FILTRATION TEST RUNS
FOR
KENTUCKY COAL UNFILTERED COAL SOLUTION

BASE CONDITIONS

Knife Advance:	2 mil/min (.002 inches/min)
Drum Speed	1.75 RPM
Drum Submergence	30% diameter
Vat Pressure	110 psig
Pressure Differential	30 psi
Filter Temperature	490°F
Slurry Feed to Filter	25000 lbs/hr
Cake Wash Rate	850 lbs/hr Wash Solvent
Precoat	500 lbs Speedex
Base Coat	10 lbs 11-C

RUN NUMBER	DESCRIPTION
1A	Alternate Precoating Procedure
1B	Base Conditions
2A	Vary Knife Advance Rate
2B	Vary Knife Advance Rate
3	Vary Drum Submergence at lcw Knife Advance Rate
4	Vary Drum Speed and Knife Advance
5	Vary Pressure Differential and Knife Advance
6	Vary Knife Advance
7	Alternate Precoat Procedure Vary Knife Advance and Cake Wash Rate
8	Base Conditions
9	Speedplus Precoat - Vary Knife Advance
10	Speedplus Precoat - Base Conditions

REACTION CONDITIONS

Kentucky Coal Feed Rate	2500 to 3500 lbs/hr
Solvent to Coal Ratio	1.5/1
Dissolver Outlet Temperature	850-860°F
Reactor Pressure	1500 psig

Samples of the filtrate and filter feed were routinely analyzed to determine viscosity and the concentration of vacuum bottoms and solids in the sample. By carefully planning the operating procedures for these runs, it was possible to obtain data at many different conditions. Each of the variables discussed above was investigated and some interesting relationships, most of which verified previous hypotheses, were observed. The scope of this test can only be considered to be preliminary in that attempts were not made to optimize overall operating parameters. Instead, the primary emphasis was placed on developing qualitative relationships between various levels of the variables and filtration results.

5. Results

Each run was split into several run periods during which operating conditions were held relatively constant. Tables A-1 to A-12 in Appendix A list the operating conditions and filtration data obtained during each run period.

Unusual procedures, run objectives, and comments on each run are listed in the appropriate tables. Evaluation of the data has revealed some interesting relationships.

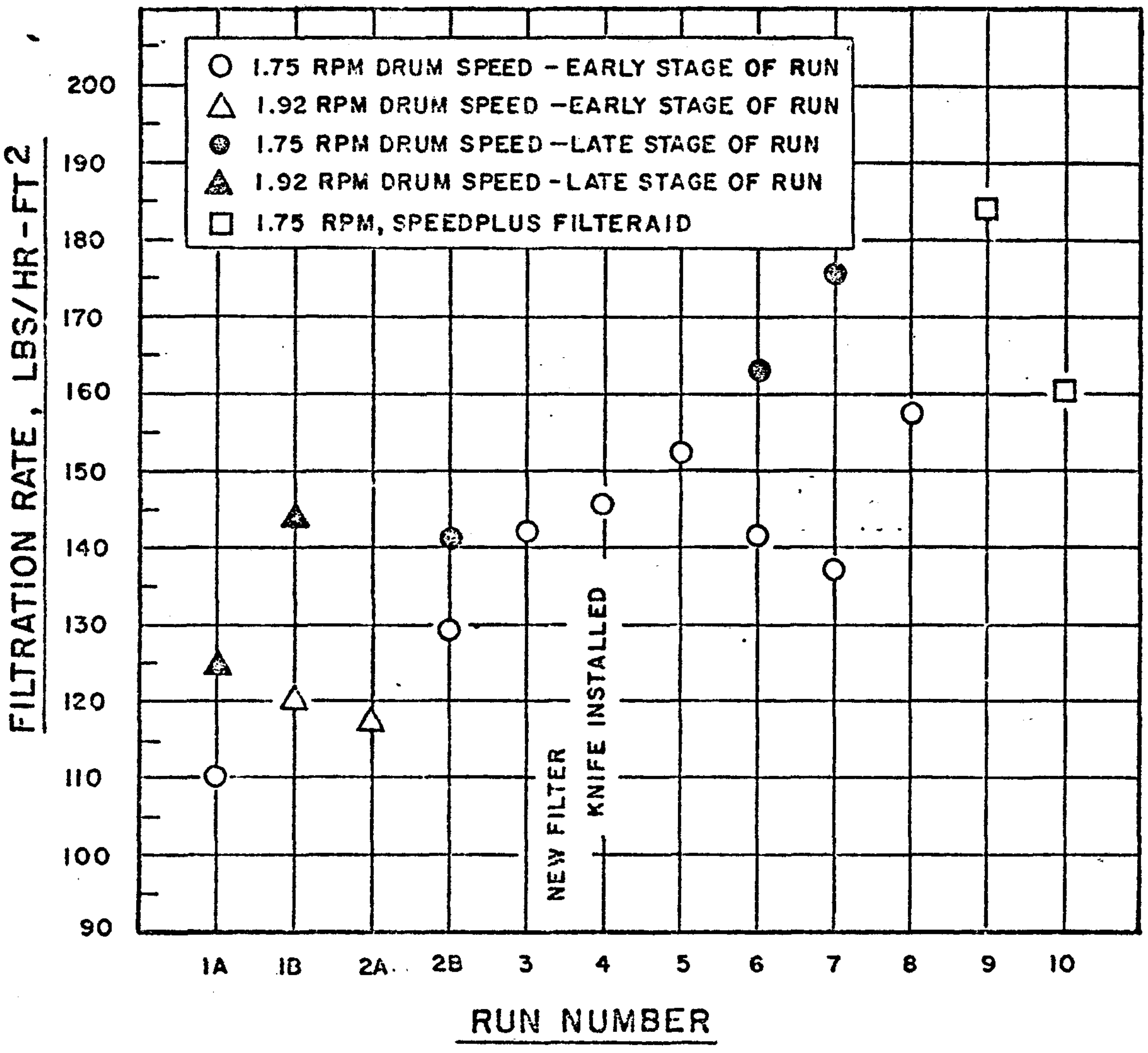
a. Precoat: The effects of precoat on filtration rate were never quantitatively determined during these runs. A comparison of the filtration rates, which were obtained while operating at base conditions during each run has been made. The data, which are shown in Figure 3, indicate that for the last five runs in which a Speedex precoat was used the filtration rate at the base conditions in the early stages of the run were comparable and averaged 146 lb/hr ft^2 (including cake wash). The first five runs were not included in the analysis because prior to Run 4, a new knife was installed in the filter and filtration rates in subsequent runs seemed higher than in previous runs.

Figure 3 also shows data which were obtained at base conditions but in the latter stages of the runs. These data show that filtration rates toward the end of the run tend to be higher than at the early stages of a run. The average filtration rates for base conditions in the runs in which Speedplus was used as a precoat material are also shown in Figure 3, and the rates appear to be higher than for Speedex precoat runs. However, the Speedplus data was obtained at later stages in the run than was normal for the Speedex runs. This fact makes comparison of the data for the different precoat materials difficult and no definitive conclusion is possible.

Precoat application can also be evaluated based on the degree to which the cake is packed. This "packing" effect can be compared between various runs by comparing the gas flow

Figure 3

COMPARISON OF BASE CONDITION FILTRATION RATES FOR FILTER RUNS 1A THROUGH 10



rate through the filter at the base conditions. Gas rates for all the runs at base conditions varied from 300 to 500 lbs/hr, indicating that there was some variation in precoat permeability due to the precoat application. This difference between runs probably can be attributed to subtle differences in precoat application procedures.

Observation of the gas rate during the runs indicates that, as the run progresses and the precoat thickness decreases, the gas rate increases, especially during the later stages of the filtration cycle. The increases in gas rate are probably due to decreased precoat resistance and may be a function of the residence time of the precoat in the vapor region of the filter. When the precoat thickness decreases sufficiently, there may be enough time to "blow the precoat dry" before the precoat reenters the slurry. By removing the liquid imbibed in the precoat, the resistance of the precoat to gas flow is decreased. Figure 4 shows gas flow rates during Run 1B versus precoat thickness. (All operating conditions were held constant during Run 1B.)

The data shows an increase in gas flow rates in the latter stages of the run. This corresponds with a slight increase in filtration rate during Run 1B. From this information it seems probable that precoat thickness has an effect on filter performance. To simplify the analysis of later runs, this effect was assumed to be negligible.

b. Knife Advance Rate: The effect of the knife advance rate on filtration was investigated in several runs. Along with the runs in which the knife advance rate was the only operating variable investigated, knife advance was also varied in the runs in which drum speed and pressure drop were changed. The results of all of these tests indicate that knife advance rates have a strong influence on the filtration process.

Figure 5 is a plot of filtration rate as a function of knife advance rate for Runs 3 through 8 in which Speedex filter aid was used as precoat material. The data shown in Figure 5 were all obtained with all of the other operating variables in the precoat loop at the base condition levels. The curve drawn through the data indicates increasing filtration rates with increasing knife advance rates. This increase would be expected to continue until the knife advance rate is high enough to permit deep enough knife penetration to remove all of the filter cake (pyridine insolubles in the coal) and cake-precoat interface during each drum revolution. This principle of "diminishing returns" in filtration rate with increasing knife advance is represented in Figure 5 by the approach to an asymptotic value of filtration rate at high knife advance rates. Attempts to operate the filter at knife advance rates in

Figure 4

GAS FLOW RATE vs KNIFE POSITION FOR RUN IB

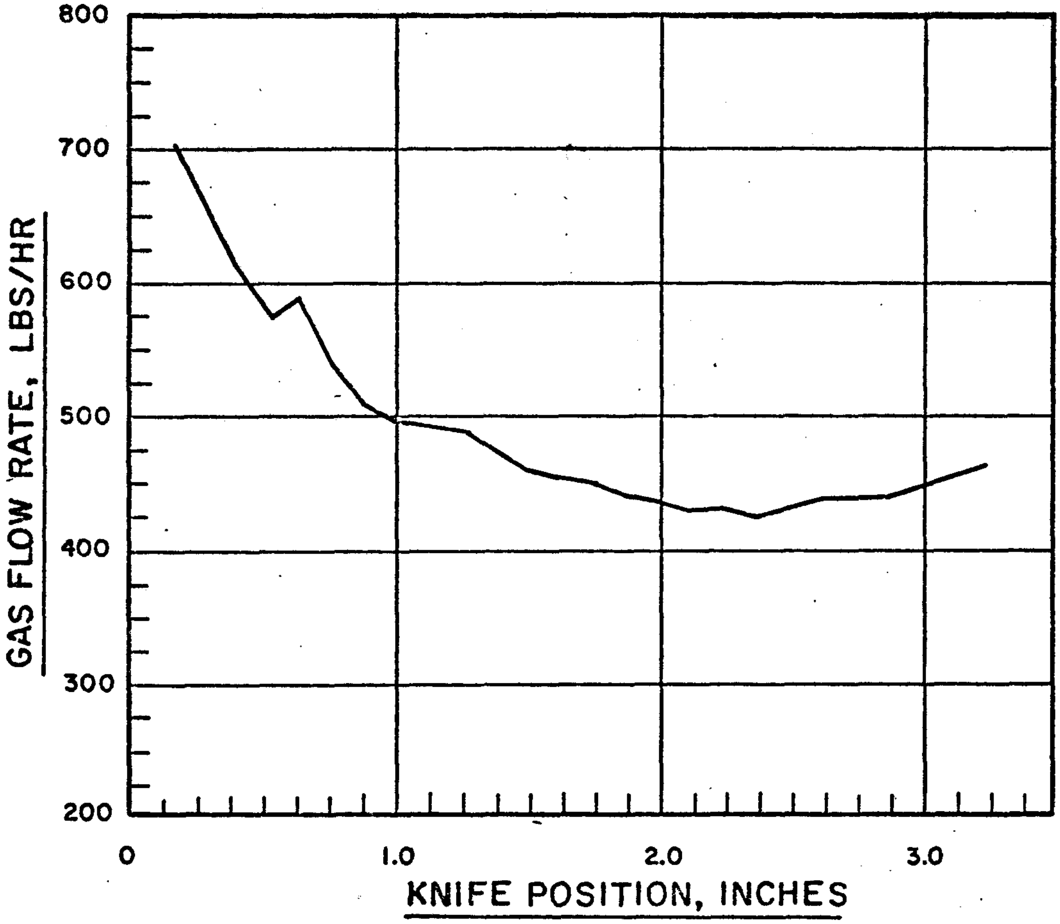
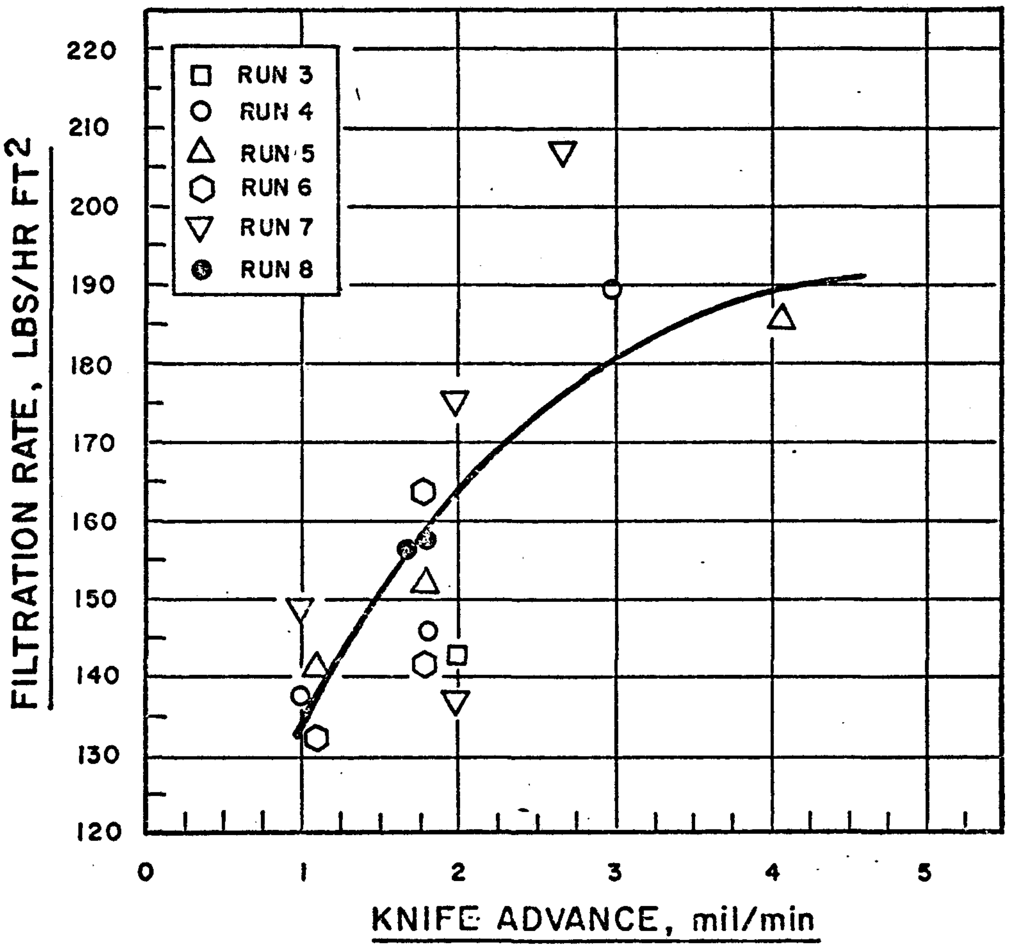


Figure 5

FILTRATION RATE vs KNIFE ADVANCE AT BASE CONDITIONS IN RUNS 3 TO 8



excess of 4 mil/min were not made, and, as a result, the point at which knife advance rate increases do not result in increased filtration rates has not been defined.

To determine the efficiency of precoat usage in these tests, the rate of removal of the solids (pyridine insoluble matter) in the filter feed was estimated from the filtration rate and average solids concentration during each run. An hourly precoat usage, in units of cubic feet, was calculated from knife position data recorded each hour. The ratio of solids removal to precoat usage was then computed for each run. This ratio, with units of lbs. solids/cubic ft. precoat was defined as a measure of filter efficiency and was used in comparison of the data obtained for the various operating periods. A comparison was made of these filter efficiency data for the runs in which knife advance was varied. The results showed that the ratio of solids removed from the filter feed per unit of precoat volume decreased as knife advance increased. This indicates that more precoat is consumed in producing the same quantity of filtrate at high knife advance rates than at low knife advance rates. However, at the low knife advance rates the filtration rate is lower requiring more filtration time to produce the comparable quantity of filtrate.

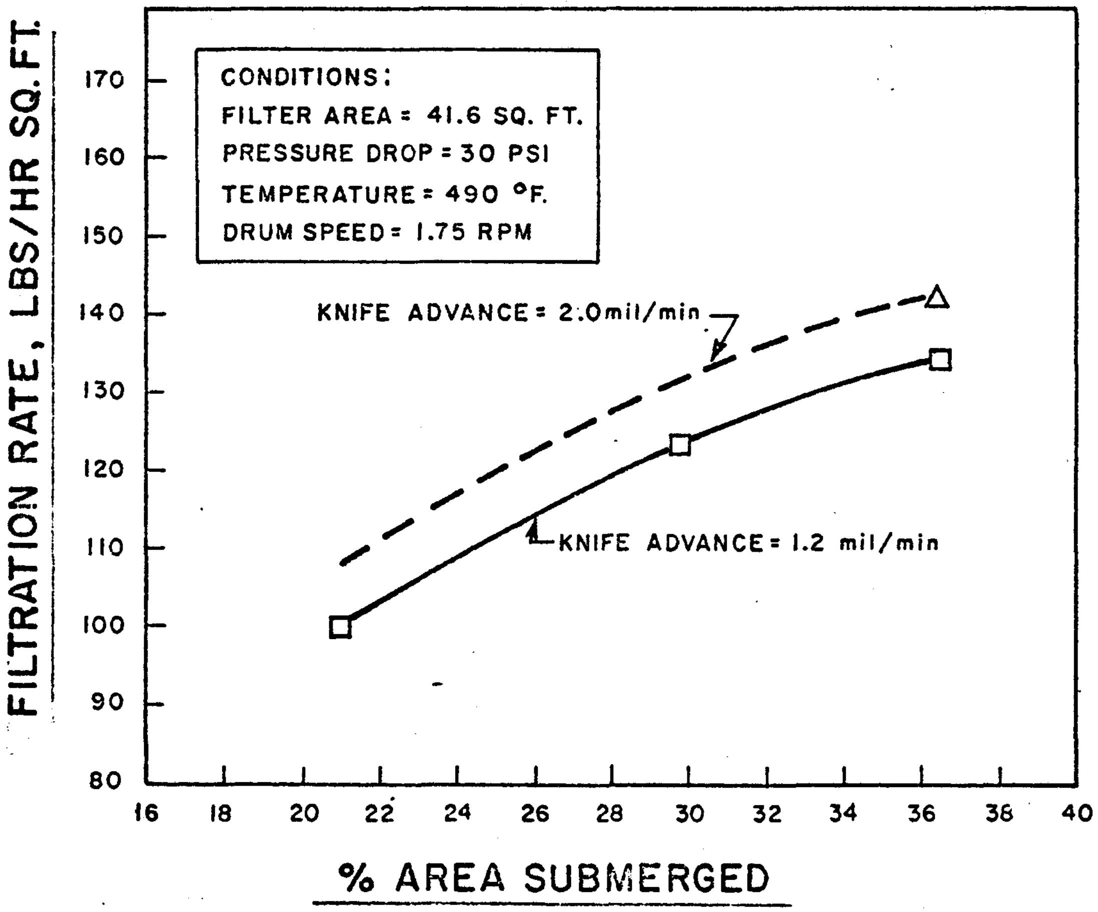
The potential interaction of knife advance with other operating variables was believed to be strong prior to the start of these tests, so several variables were examined at different levels with various knife advance rates at each level. Those results are presented in subsequent sections.

c. Drum Submergence: Figures 6 and 7 show the effect of drum submergence of filtration rate. Figure 6 shows the filtration rate as a function of submergence. The data indicate that increased submergence resulted in higher rates. The fact that higher rates were achieved at higher submergence demonstrates that filter cake is not completely blinding the precoat and preventing additional filtration. By increasing the drum residence time in the slurry, additional filtrate will be produced. However, a trend toward some maximum filtration rate seems apparent in Figure 6. Each additional unit of area results in a smaller gain in filtration rate at high submergence. As drum submergence is increased, the cake thickness will also increase due to longer drum residence time in the slurry. As the cake thickness increases, the cake resistance should also increase, resulting in a gradual decrease in instantaneous rate through the precoat. This phenomenon apparently caused the leveling off of filtration rate as seen in Figure 6.

The filtration rate per unit of area submerged is another variable which can be used to describe the filter performance at various levels of submergence. Figure 7 shows that the

Figure 6

FILTRATION RATE vs. % AREA SUBMERGED



rate per unit area submerged decreased with increased submergence (the same data set plotted in Figure 6 was used to generate Figure 7). This decrease in utility of submerged area with increased submergence can be explained by the same phenomenon described above in the discussion of Figure 6; namely, that the marginal increase in filtration rate decreases as submergence increases.

Both Figure 6 and 7 show that increased knife advance rates result in higher filtration rates at higher submergence than low knife advance rates. Insufficient data at high knife advance were generated during the submergence study to provide parametric curves of the effect of knife advance on filtration at various levels of submergence. Therefore, the filtration rates which might be expected at various levels of submergence and at 2 mil/min knife advance rate have been extrapolated and plotted as broken lines in Figure 6 and 7.

d. Drum Speed: During Run 4 the combined effects of drum speed and knife advance rate on filtration rates were investigated. The run was divided into seven periods in which various combinations of drum speed and knife advance rates were studied. Three levels of drum speed (1.0, 1.75, and 2.85 RPM) were chosen and at each level knife advance rates per drum revolution were chosen to facilitate comparisons between the runs.

The results of Run 4 are illustrated in Figure 8 in which filtration rate has been shown as a function of drum speed with knife advance rate as a parameter. Figure 8 suggests two conclusions. First, at the knife advance rates studied the highest drum speed resulted in the highest filtration rate. Second, at the drum speeds studied the highest knife advance rate always resulted in the highest filtration rate. The obvious conclusion from this study is that to maximize filtration rates, maximum knife advance rates and drum speeds should be utilized.

This same conclusion on the effects of knife advance rate and drum speed has been reached elsewhere using coal solutions in bench scale tests on a simulated rotary pressure precoat filter.[1]

Obviously, high knife advance rates will consume substantial amounts of precoat and, therefore, the efficiency of solids removal by the precoat must be considered. Solids removal per cubic foot of precoat usage were calculated for the data discussed above and are shown in Figure 9. The data suggest

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[1] Smith, G.R.S., and Martin, P.C., "Filtration Process and Equipment Studies for Coal Liquefaction Processes," Report No. FE-2007-31, Johns-Manville Sales Corp., under ERDA Contract EX-76-C-01-2007, March, 1977.

Figure 7

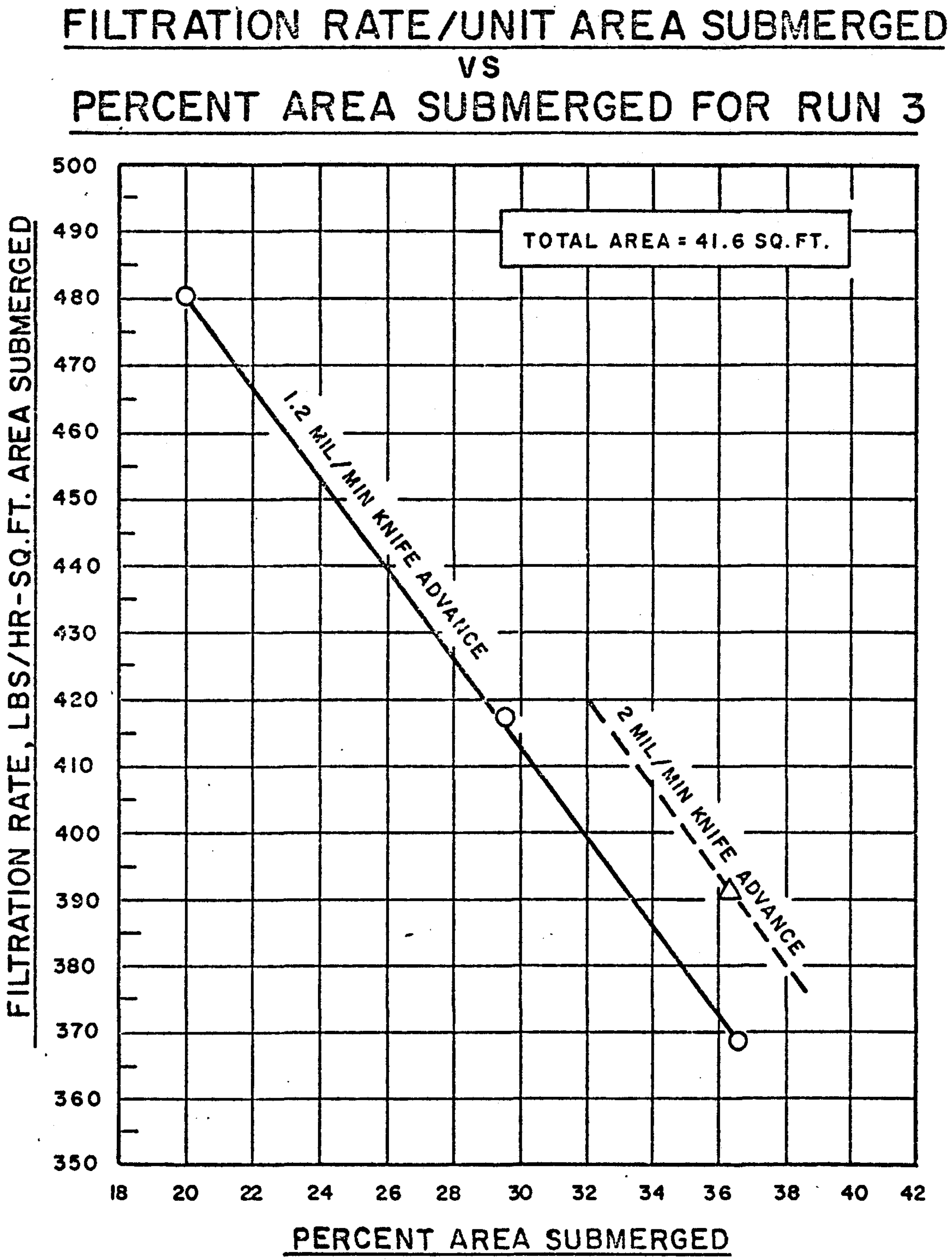
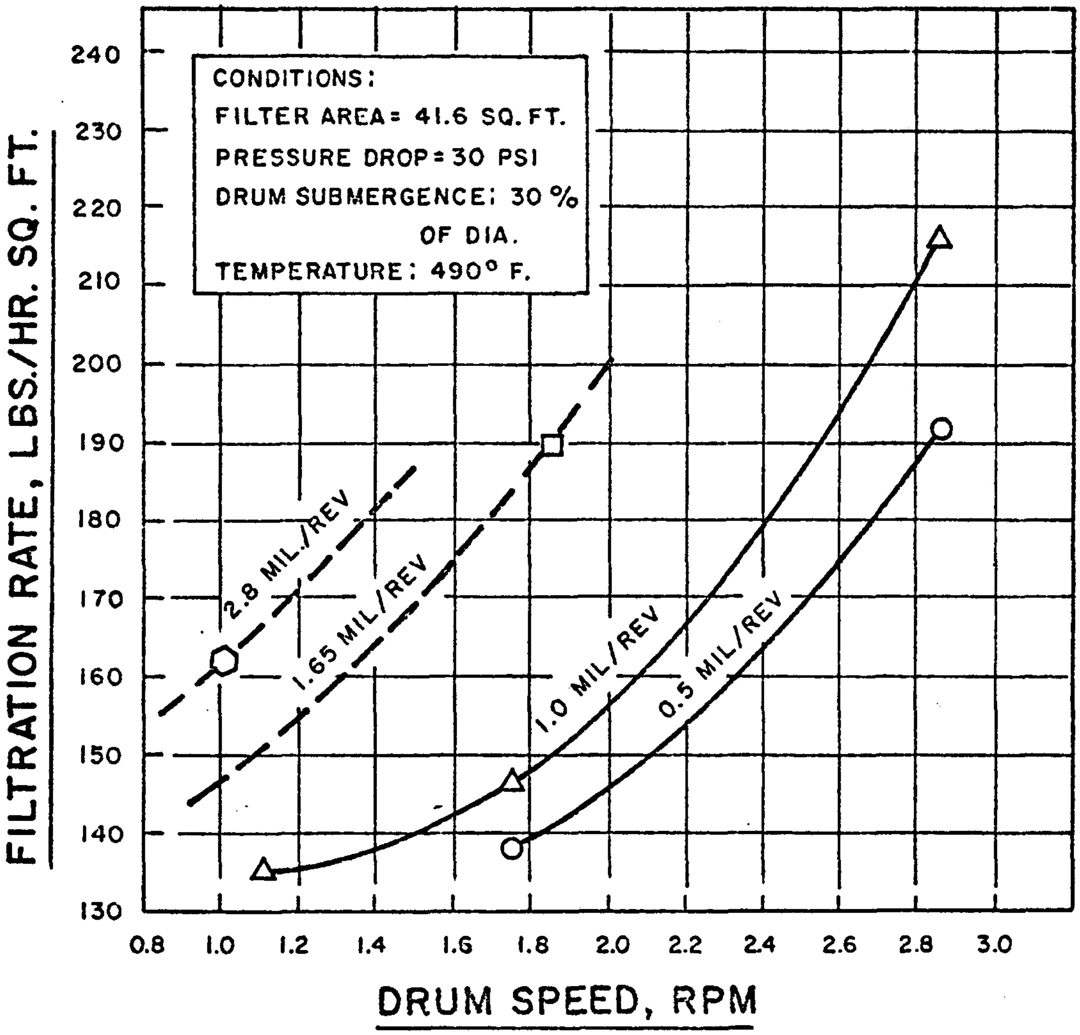

FILTRATION RATE/UNIT AREA SUBMERGED
VS
PERCENT AREA SUBMERGED FOR RUN 3
TOTAL AREA = 41.6 SQ.FT.
1.2 MIL/MIN KNIFE ADVANCE
2 MIL/MIN KNIFE ADVANCE
FILTRATION RATE, LBS/HR-SQ.FT. AREA SUBMERGED
500
490
480
470
460
450
440
430
420
410
400
390
380
370
360
350
18
20
22
24
26
28
30
32
34
36
38
40
42
PERCENT AREA SUBMERGED

Figure 8

FILTRATION RATE vs. DRUM SPEED



that the solids removal efficiency was not a function of drum speed but was a strong function of knife advance rate. As the knife advance rate increased, the solids removal efficiency decreased.

The fact that solids removal efficiency was independent of drum speed would suggest that it would be advantageous to obtain the high filtration rates associated with high drum speeds. At the same time, the data in Figure 9 indicate that high knife advance rates result in a rapid drop in efficiency when rates increase above 1 mil/rev. Therefore, it might not be advantageous to maximize knife advance rate. It would be most useful to optimize the knife advance rate to produce a high filtration rate at a high solids removal efficiency. The data in Figure 9 imply that the optimum knife advance rate should be in the 0.5-1.0 mil/rev range, the range where the solids removal efficiency drops rapidly as knife advance is increased. Further work is necessary to explore possible optimum conditions.

e. <u>Cake Wash Rate</u>: A brief test was made to determine the effects of cake wash rate on filtration rates. The results are shown in Figure 10. In the figure, line A represents a least squares linear regression fit of the data. Line B has been drawn to illustrate the position and slope of a line which would indicate a gain in filtration rate equal to the gain in cake wash rate (recall that the filtrate has been assumed to include the cake wash to the filter). The slope of line A is less than the slop of line B, suggesting that increasing the amount of cake wash in the range studied does not increase the true filtration rate. In fact, if the cake wash is subtracted from the filtrate, the actual filtration rate decreases as the cake wash increases. The results of this cake wash study indicates that more work needs to be done to determine the minimum amount of cake wash needed to adequately remove imbibed process solvent and SRC from the cake. If the quantity of cake wash can be reduced significantly without deleterious effects, as these data seem to suggest, the amount of solvent that has to be distilled in the fractionating towers can be reduced.

f. <u>Pressure Differential (ΔP)</u>: In Run 5, the effects of pressure differential (ΔP) and knife advance rate on the filtration rate were investigated. The results are illustrated on Figure 11. Indications are that increasing the differential pressure from 30 to 40 psi acts to increase the filtration rate at any knife advance rate. In addition, the data in Run 5 indicated that knife advance rates resulted in a greater increase in filtration rate at 40 psi than at 30 psi.

The fact that high knife advance rates seem to result in higher filtration rates at 40 psi than at 30 psi has been

Figure 9

SOLIDS REMOVAL EFFICIENCY
VS
KNIFE ADVANCE FOR RUN 4

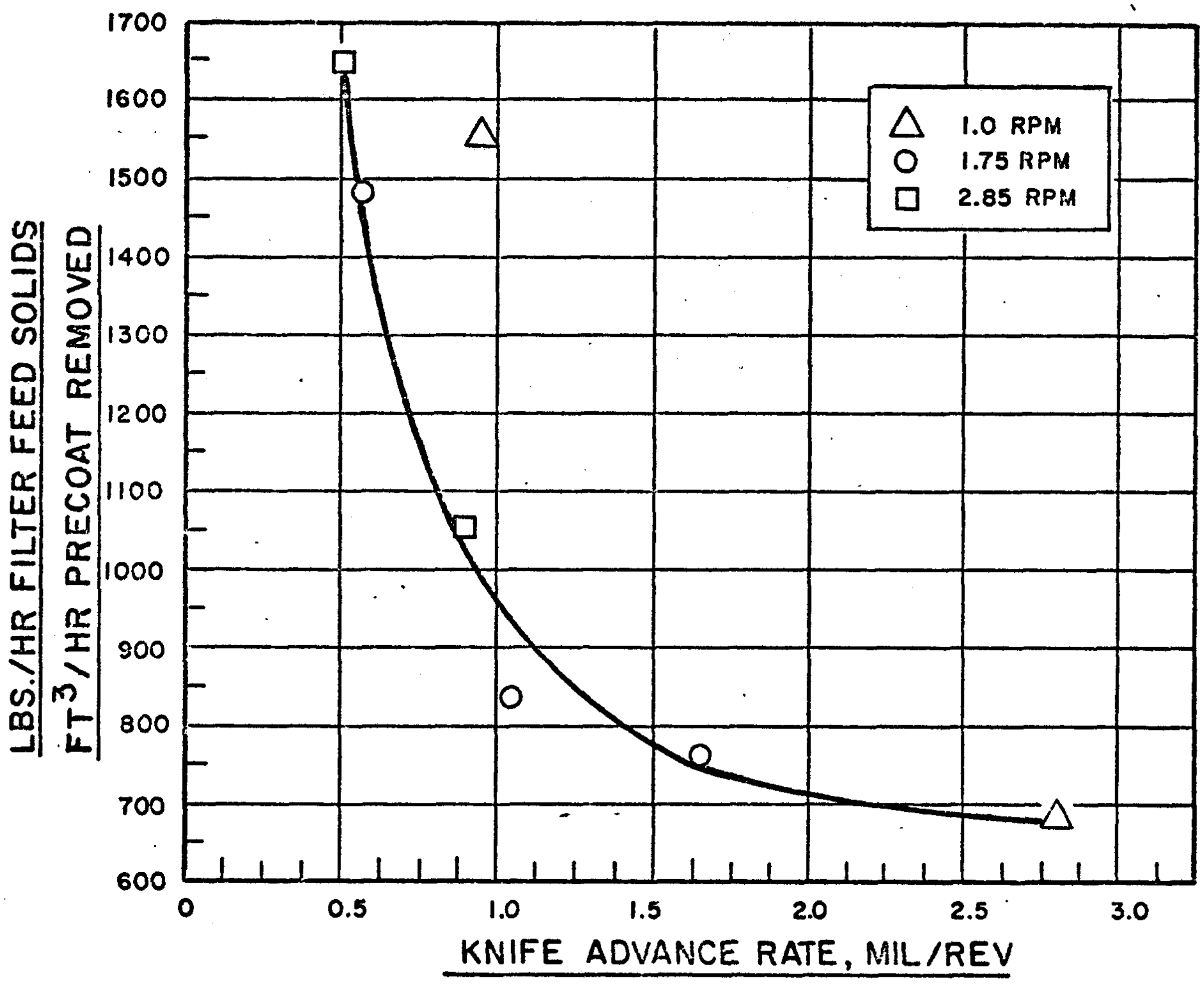


Figure 10

EFFECT OF WASH SOLVENT RATE ON FILTRATION RATE DURING RUN 7

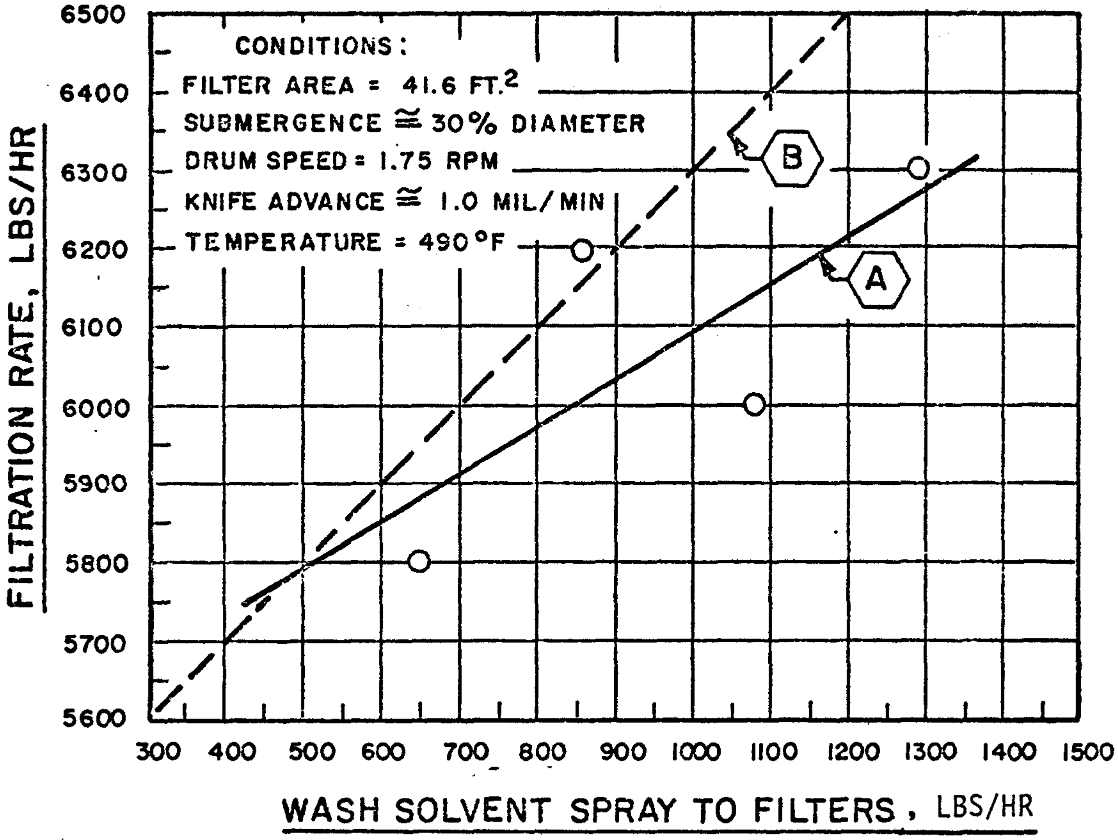
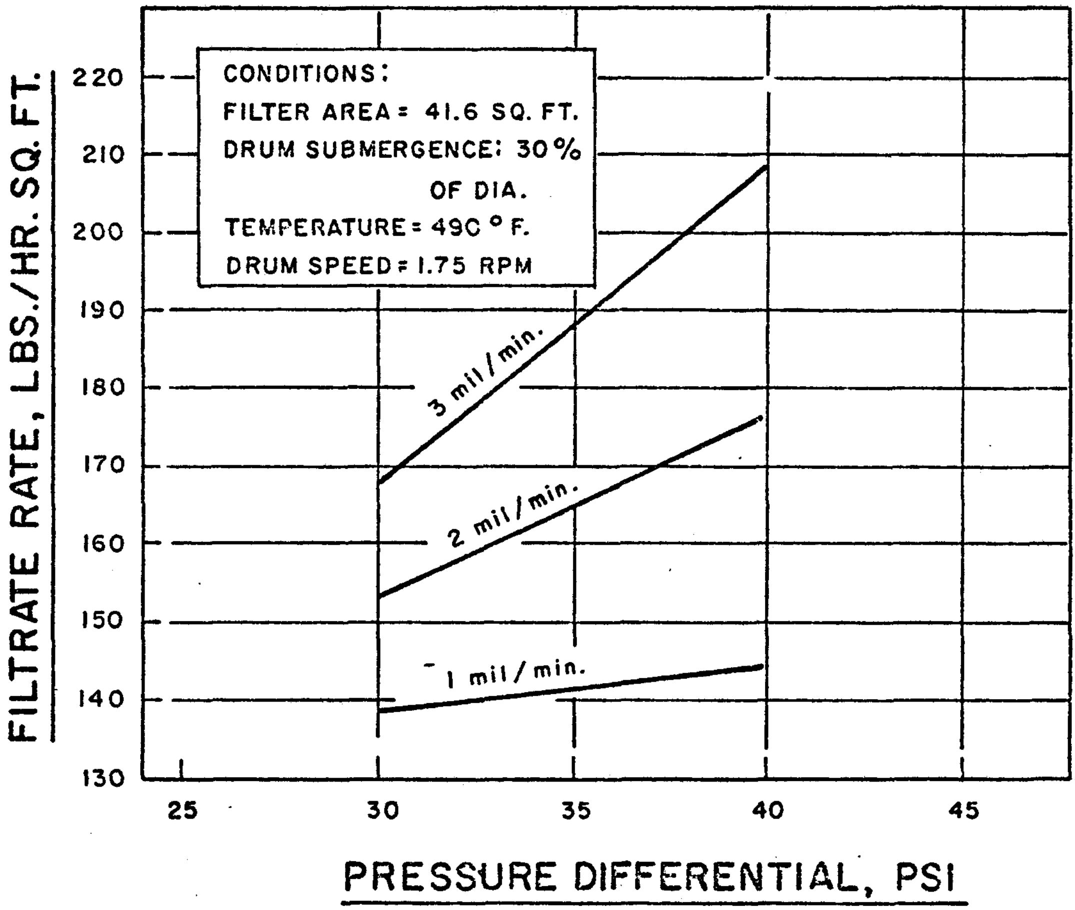


Figure 11

FILTRATION RATE vs. PRESSURE DIFFERENTIAL



attributed to more efficient solids removal at the higher pressure, possibly due to the formation of a cleaner cake-precoat interface than at the lower pressure.

The dramatic effect of knife advance rate at 30 and 40 psi pressure differential is shown in Figure 12. The increase in slope of the constant knife advance curves as the knife advance increases is evidence of the strong supplemental effect of knife advance on the filtration rate.

The solids removal efficiency parameter was calculated for the data shown in Figures 11 and 12, and the results have been plotted in Figure 13. As with all other solids removal efficiency data at different knife advance rates, the implication from Figure 13 is that solids removal efficiency decreases as knife advance increases, and the decrease is rapid between 0.5 and 1.0 mil/rev. (The drum speed in Run 5 was 1.74 RPM.) Figure 13 shows that there is a positive effect of pressure differential on solids removal efficiency. At the 40 psi differential, the efficiency of solids removal was much higher than at the 30 psi differential.

Based on this data, it can be concluded that the most efficient filter operation will be obtained at high pressure differential. The precoat efficiency is highest at high pressure differential and low knife advance rates. More data are now needed at pressure differentials in excess of 40 psi to determine if filtration rates continue to increase and if cake compression becomes a serious problem.

6. Optimization

The data from all of the periods of all twelve runs were analyzed and estimated filter feed rates and solids removal efficiencies calculated for each period. These data were then classified into two groups: periods in which Speedex precoat was used and periods in which Speedplus precoat was used. These two groups were each broken into four subgroups according to knife advance rates of 0.5-1.5, 1.5-2.5, 2.5-3.5, and 3.5-5.0 mil/min. The data were then plotted to determine if any unusual correlations existed. Some interesting trends in the data were noted and are shown by least squares regression curves in Figure 14.

The data show a interrelationship between solids removal efficiency and filtration rate at each knife advance rate. These data should be useful in future filter designs.

Based on the results of this program, conditions for an optimum run have been formulated. These conditions, which are listed in Table 9, assume that maximum filtration rate is an important, but not singular, objective and that increasing the solids removal efficiency is an important objective.

Figure 12

FILTRATION RATE vs KNIFE ADVANCE RATE

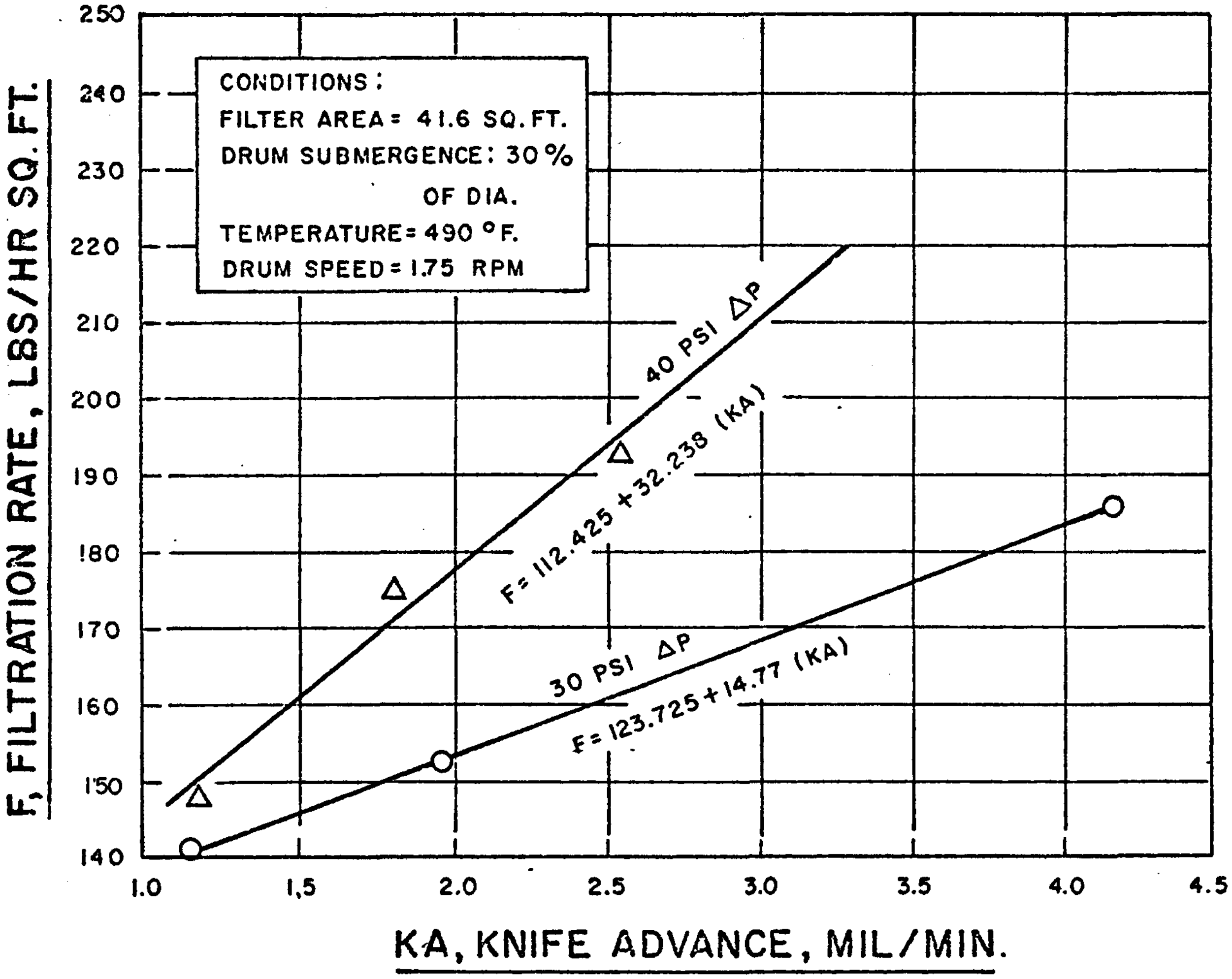


Figure 13

SOLIDS REMOVAL EFFICIENCY vs KNIFE ADVANCE RATE AT VARIOUS LEVELS OF PRESSURE DIFFERENTIAL DATA FROM RUN 5

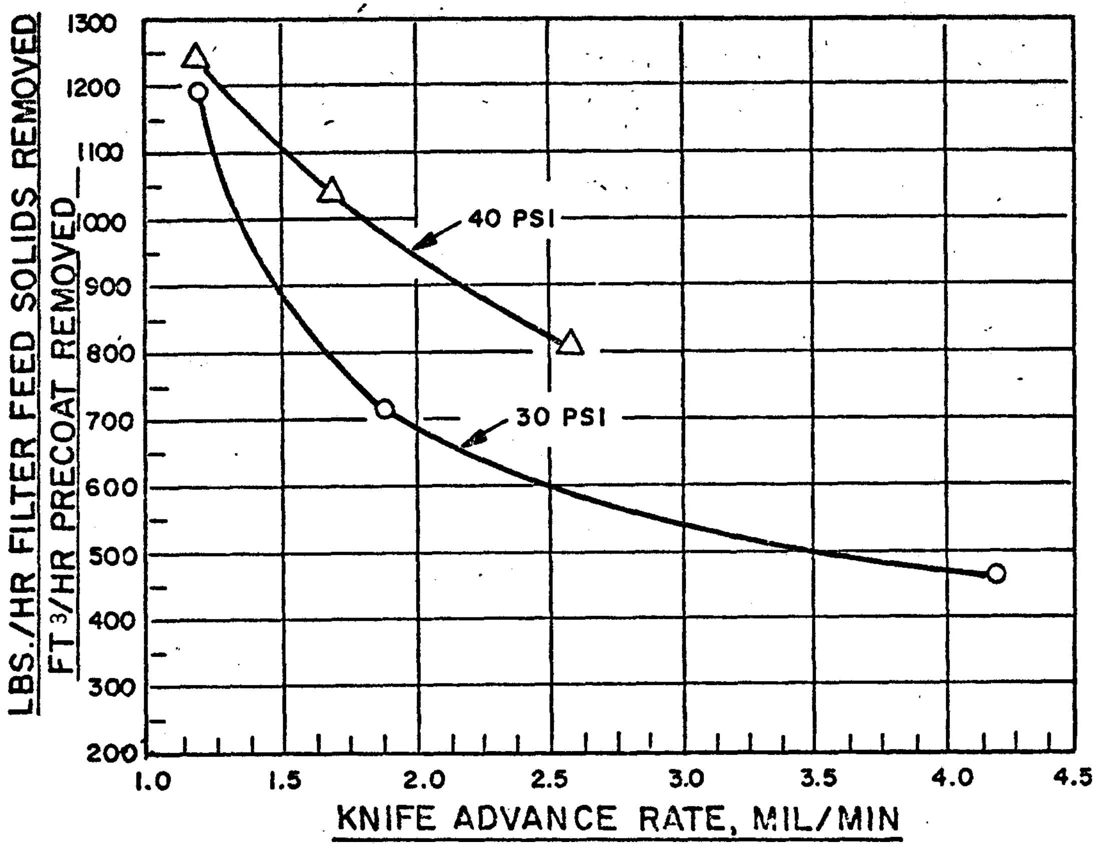


Figure 14

FILTRATION vs. SOLIDS REMOVED EFFICIENCY

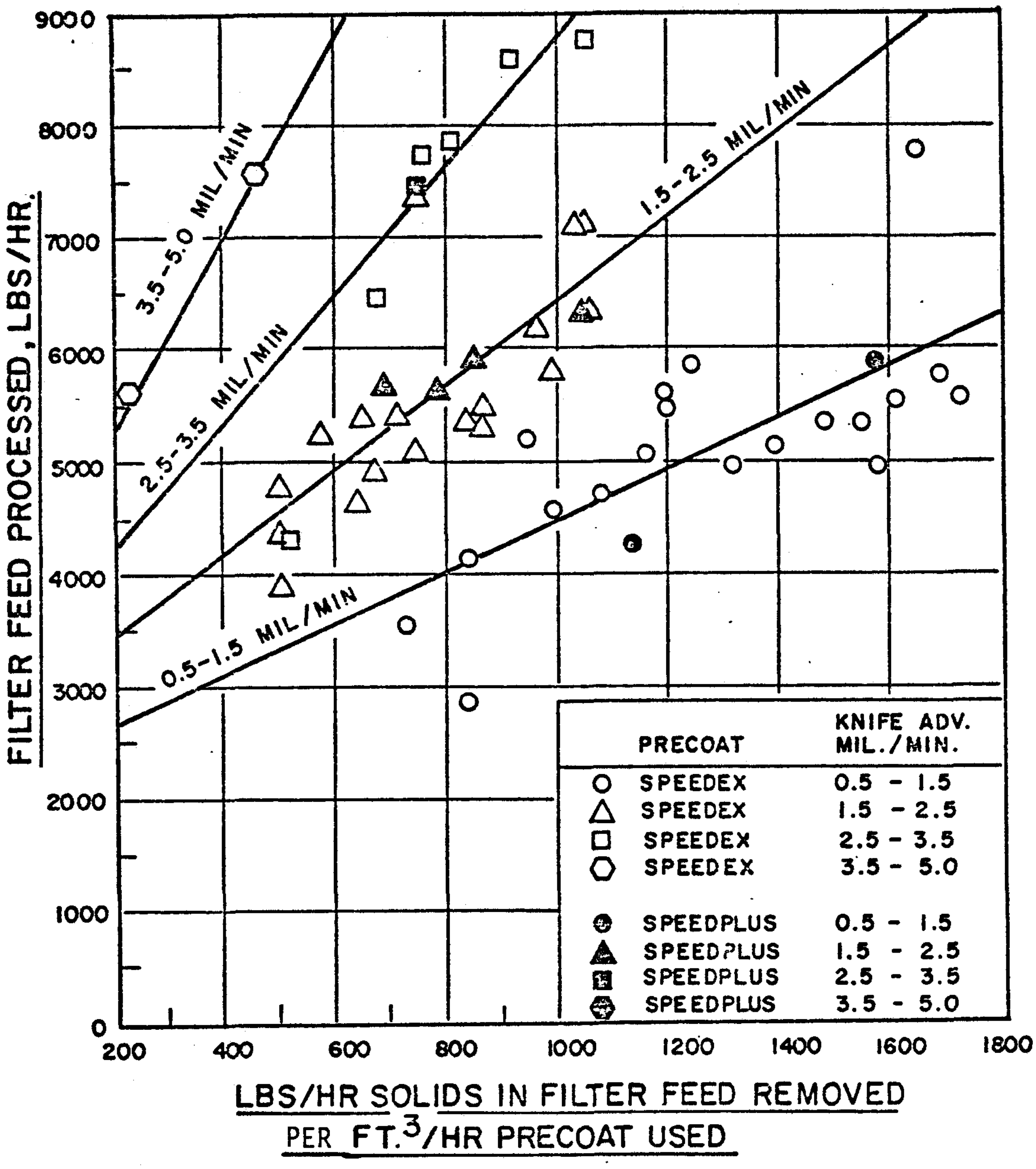


TABLE 9

PROPOSED OPTIMUM FILTRATION RUN

Filter Temperature	500°F or higher
Drum Submergence	35% diameter (up to bearings)
Pressure Differential	40 psi or higher
Drum Speed	3-4 RPM
Knife Advance	1 mil/rev
Wash Solvent to Cake	600 lbs/hr
Precoat	Speedplus
Basecoat	Dicalite 6000 (No Asbestos)
Slurry Feed[1]	25,000 lbs/hr

[1] Includes filter feed overflow.

B. Heat Capacity of SRC Products

Samples of wash solvent, process solvent, and SRC were sent to an outside laboratory for determination of heat capacity. Problems resulting from high solvent vapor pressures prevented measuring heat capacities at temperatures as high as desired. However, some low and moderate temperature data were obtained.

Figure 15 and Table 10 show heat capacity versus temperature relationships for wash solvent, process solvent, and SRC which were obtained using a differential scanning calorimeter. As previously noted, measurements were not obtained above the ranges shown due to problems of increasing vapor pressure and thermal expansion. It is believed that the results shown may be extrapolated to higher temperatures with reasonable confidence. SRC shows an inflection in heat capacity curve in the region of its fusion point.

C. SRC Agglomeration Study

An experimental study was carried out to investigate the tendency of SRC flakes to agglomerate or consolidate into a solid mass during storage. This agglomeration of SRC has been observed in the Ft. Lewis storage silos.

A laboratory test apparatus was constructed to simulate SRC storage at various combinations of temperature and pressure. Specification grade SRC product with a fusion point of 320°F was selected for the study and the material was classified to seven or eight mesh prior to use. Figure 16 illustrates the effect of temperature and pressure on the tendency of SRC flakes to agglomerate. At the higher pressures tested, agglomeration was noted as low as 230°F, some 90° below the nominal fusion point of the SRC, while at low pressures agglomeration did not begin until about 250°F, still some 70° below the nominal fusion point.

Figure 17 shows the observed effect of time on SRC agglomeration at various temperatures and a constant pressure (118.5 psig). The results show that agglomeration might be dependent on elapsed time at the elevated temperature and pressure. The apparent effect of time on agglomeration may not be significant and may be attributable to delays in temperature equilibration of the specimen resulting from poor thermal conductivity of the SRC and the poor heat transfer between the SRC and the sample container. An additional test, not shown in Figure 17, confirmed that at room temperatures the effect of time on agglomeration is negligible. In that test, a sample of SRC was stored at room temperature under a pressure of 118.5 psig for a period of approximately four months. The SRC showed no signs of agglomeration at the end of the period.

The following conclusions can be drawn regarding agglomeration tendency of SRC:

1. The tendency of stored SRC to agglomerate is related to its storage temperature and its storage pressure.

Figure 15

HEAT CAPACITY vs TEMPERATURE

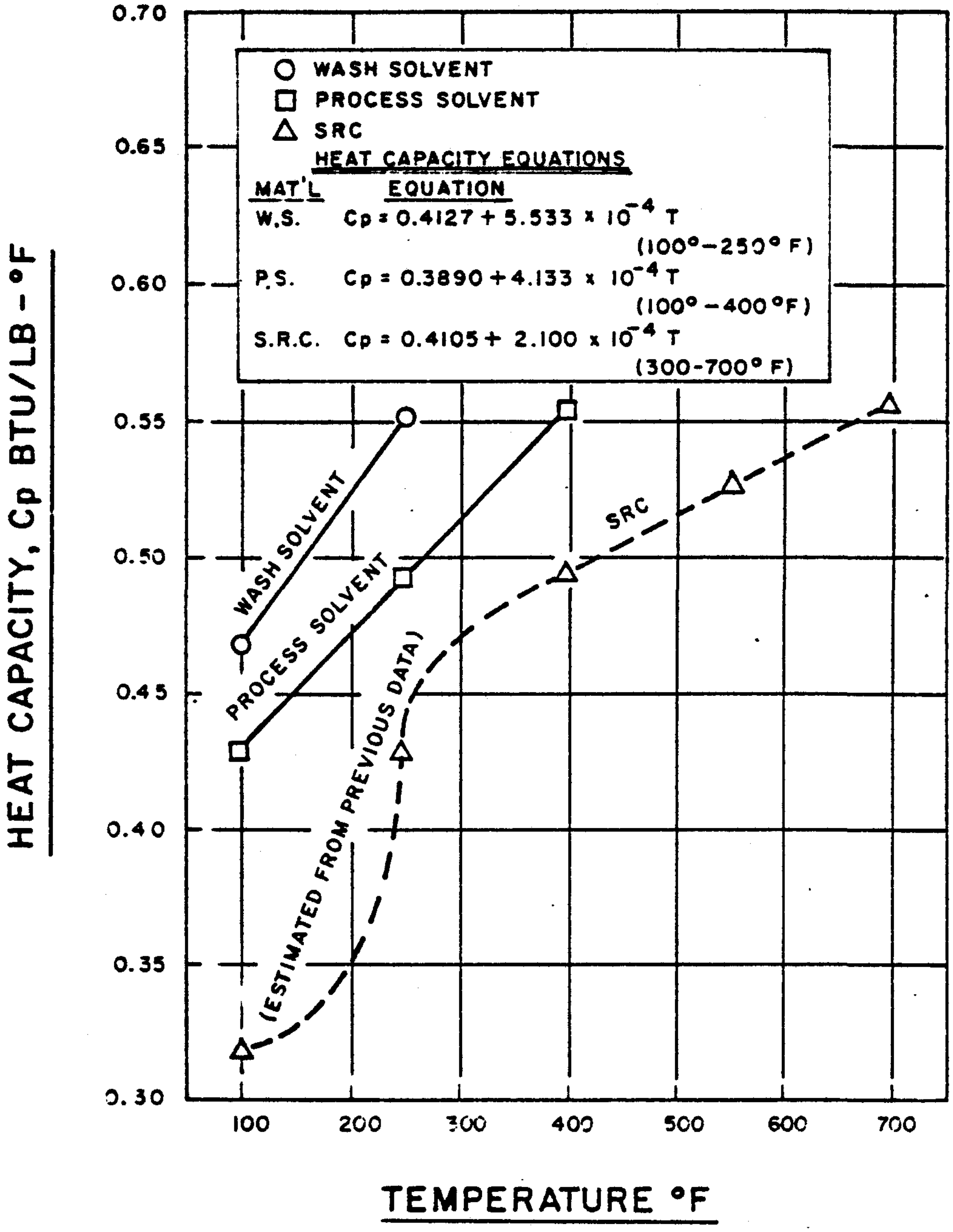


TABLE 10

HEAT CAPACITIES OF SRC AND SRC SOLVENTS, BTU/LB-°F

Temp. °F	SRC	Process Solvent	Wash Solvent
100	0.319	0.430	0.468
250	0.429	0.493	0.551
400	0.494	0.554	--
450	--	--	--
550	0.527	--	--
600	--	--	--
700	0.557	--	--

Figure 16

SRC AGGLOMERATION vs PRESSURE AND TEMPERATURE

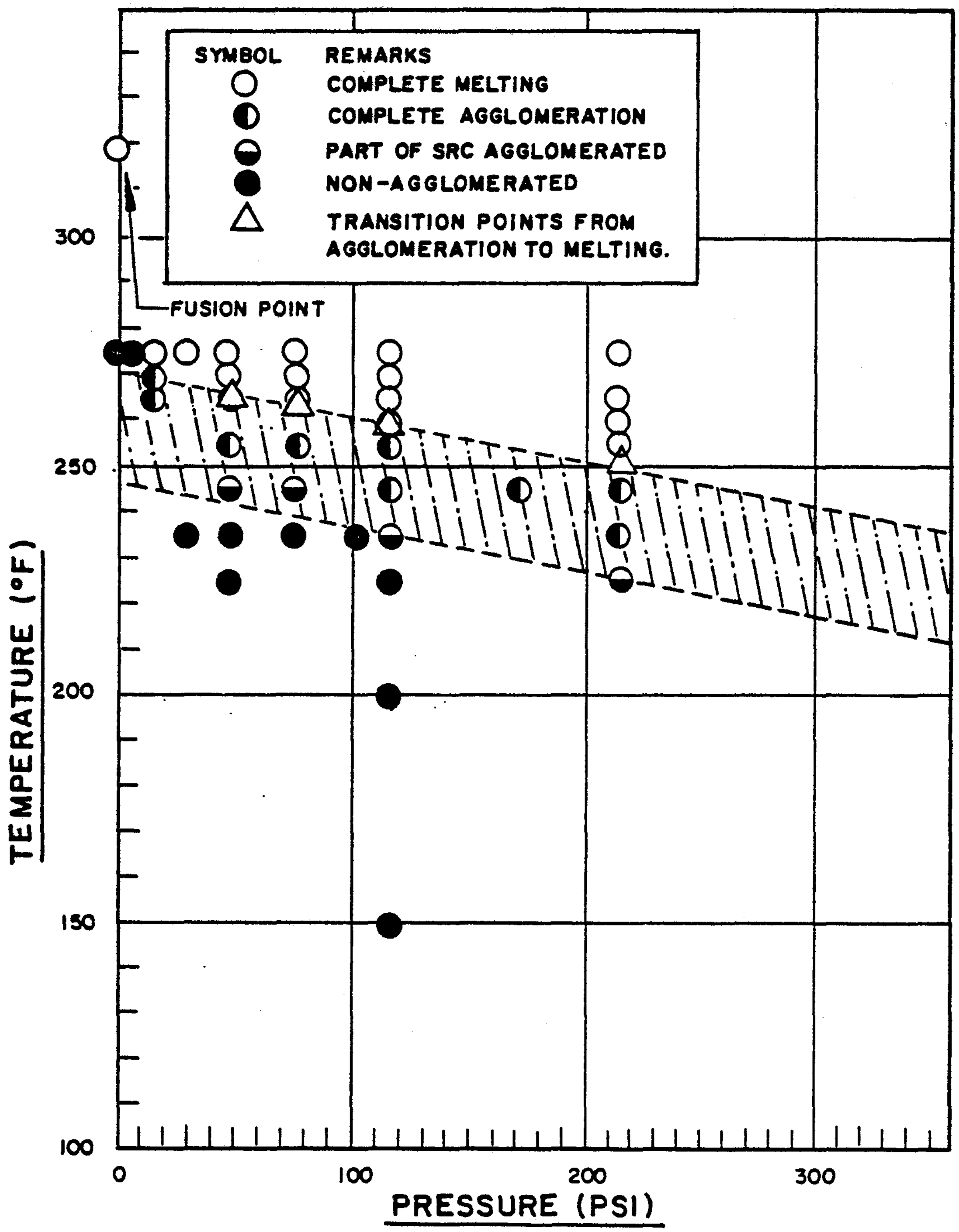
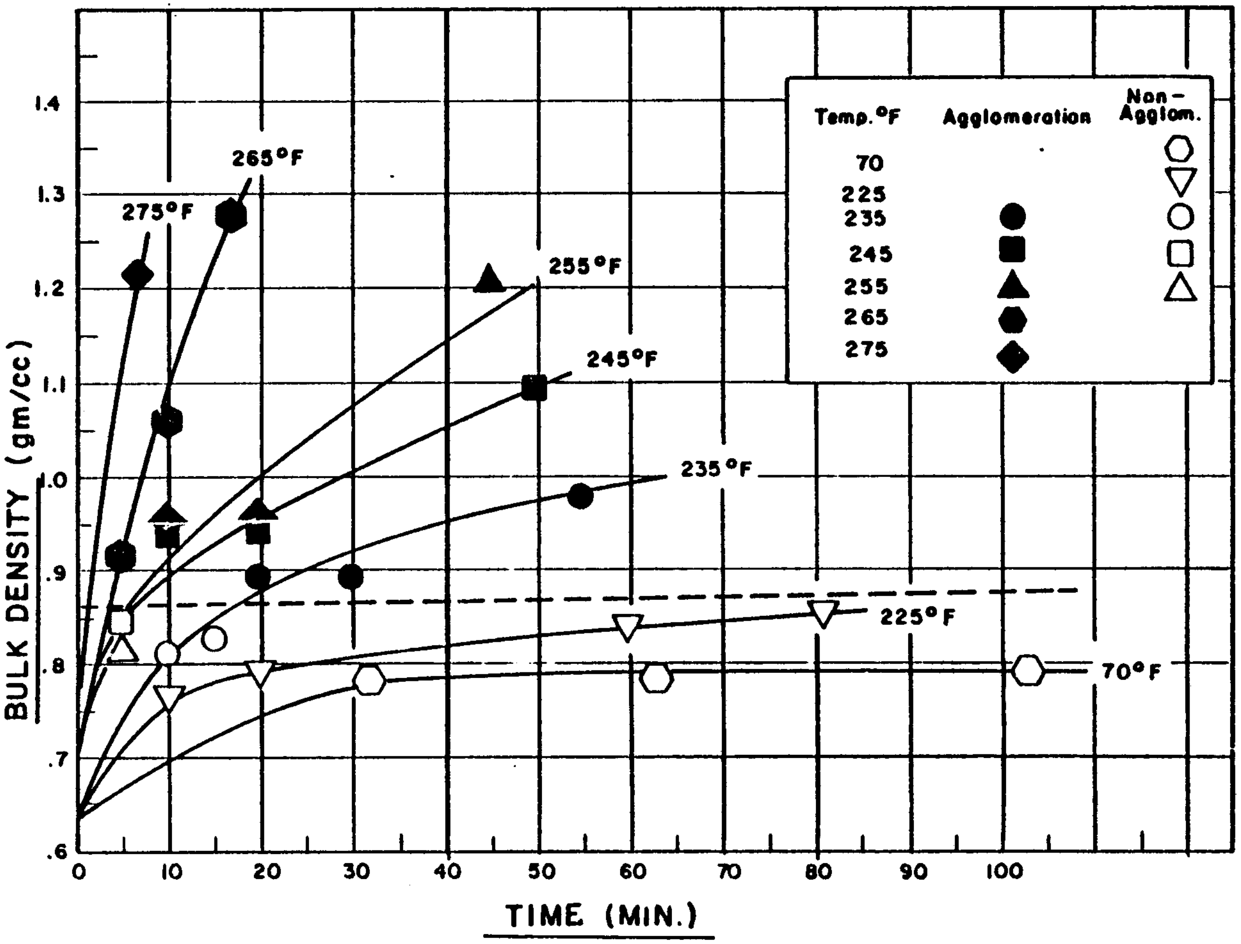


Figure 17

TIME EFFECT ON SRC AGGLOMERATION
AT 118.5 PSI



2. Normally, SRC agglomeration should not be a problem, since the conditions that favor agglomeration are far beyond those found in the SRC pilot plant storage silos.

3. To prevent problems of SRC agglomeration, the SRC product should be well stripped to ensure an adequately high fusion point. The SRC should also be stored at conditions of lower temperatures and pressures than those in the shaded region of Figure 16.

4. The SRC agglomeration which was previously seen in storage silos at Ft. Lewis was likely caused by unusual material such as poorly cooled or poorly stripped SRC being placed in storage.

5. The effect of time on agglomeration is negligible.

VI. Merriam Laboratory Operations

A. Introduction

During the first quarter of 1977, work in three areas was carried out. An extended set of runs in support of the Ft. Lewis SRC II development program was completed. Preliminary results of this study, which was designed to determine the effect of certain variables in the SRC II process, were reported in the 1976 Annual Report. The completed study is discussed in detail in Section VII. Two exploratory runs were made to evaluate a downflow, countercurrent hydrogen flow reactor, and an initial run was made with a new coal sample, a subbituminous coal from the Amax Coal Co., Belle Ayr Mine.

B. Completion of SRC II Studies with Kentucky Coal.

Runs GU 156R through GU 160RB complete the series beginning with GU 137R which has investigated the effect of the following variables on SRC II operation: coal concentration in the feed slurry, residence time, dissolver temperature, and hydrogen feed rate. One lot of coal has been used throughout the series (fifth lot,[2] Kentucky Nos. 9 and 14 from P&M's Colonial Mine) and solids level ($\leq$ 48%) in the feed slurry was maintained. Pressure has been constant at 1900 psig except for minor variations in GU 160R. The results of the complete study are discussed in Section VII.

In runs reported earlier, hydrogen feed rates of 4.9 and 6.4 wt % based on feed slurry were investigated. At the levels studied, hydrogen feed rate appeared to have no significant effect on product yields in the laboratory reactor. The objective of run GU 156R was to determine the effect of a still higher hydrogen feed rate (7.8 wt % based on slurry) and the objective of GU 157R was to determine the effect of a decreased hydrogen feed rate (4.2 wt % based on feed slurry). Severe temperature control problems prevented meeting the stated objectives, but results of GU 156R are still of interest. (Run GU 157R was terminated before satisfactory lined out operation was achieved due to slurry pumping problems.) Temperature control problems experienced during these two runs were due to improved insulation installed on the reactor before run GU 156R. The problem was corrected by reinstallation of the old style insulation following GU 157R.

Nominal conditions for GU 156R were similar to those of GU 138R and GU 154R except for hydrogen feed rate. Results for these runs are summarized below. (Conditions and results for all runs are summarized in Table B-1 in Appendix B.) The significant observation to be made is the increased oil yield and decreased SRC yield for GU 156R in comparison to GU 138R and GU 154R. Gas yield for GU 156R is also substantially increased. It is believed that the increased conversion to oil in GU 156R should be attributed to

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[2] See Table 14, 1976 Annual Report to ERDA under Contract E(49-18)-496 (Report No. FE-496-131) for a complete analysis.

dissolver temperature rather than the increased hydrogen feed. Although temperatures in the dissolvers were very erratic, the slurry was subjected to a relatively high temperature in the top half of the first dissolver (≈463°C) and then subjected to a somewhat cooler temperature in the second dissolver. Such a temperature profile has previously been observed to improve oil yield.

	GU 138R	GU 154R	GU 156R
C_1-C_4 Yield	10.0	9.6	14.3
Total Oil Yield	29.5	30.5	33.8
SRC Yield	34.1	35.0	25.2

Run GU 158R was made under the same nominal conditions as GU 140R. Results of the earlier run were suspect due to a temperature recorder malfunction and the possibility of overheating. Results of GU 158R confirm the suspicion of overheating in GU 140R. A comparison of GU 158R with GU 139R indicates that increasing the residence time from 1.0 to 1.5 hr results in increases in gas and oil yields and a decrease in SRC yield.

Run GU 159R was made under conditions similar to those of GU 138R but with the dissolver temperature decreased from 455 to 445°C. In this run, satisfactory steady state operation was not achieved due to slurry pumping problems. The primary pumping problems were due not to excessively high slurry viscosity but rather to the formation of gel-like particles in the slurry which plugged pumps and lines.

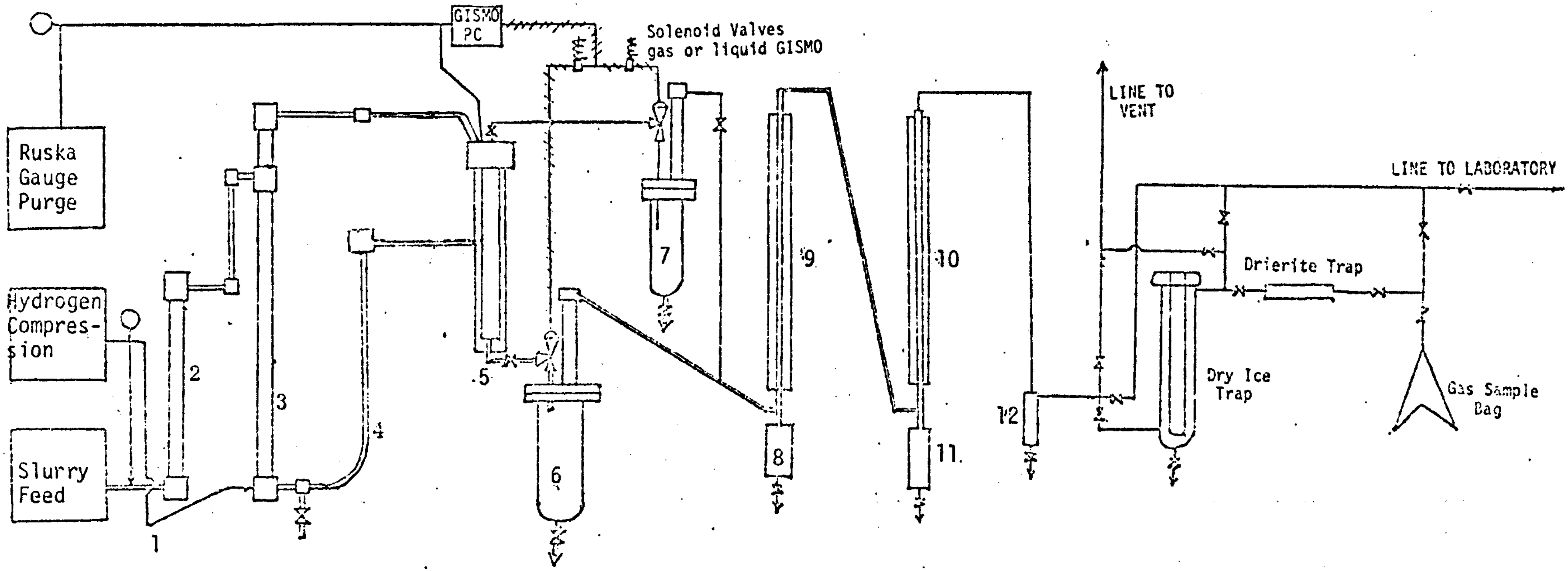
Run GU 160R was made with 2/3 hr residence time, a coal concentration of 30% and a temperature of 465°C. These conditions are similar to those of GU 150R where the run was terminated due to an excessively high feed slurry viscosity as steady state operation was being approached. Run GU 160R was initiated with a pressure of 2000 psig rather than the normally used 1900 psig. Satisfactory steady state operation was maintained for about 12 hours during which data for a product distribution were obtained (GU 160RA). Pressure was then reduced to 1800 psig (GU 160RB) and the run was continued for an additional 12 hours before the run was terminated due to slurry pump failure associated with excessively high feed slurry viscosity. Again, the 2/3 hour residence time with a 30% coal concentration and ≈1900 psig pressure was found to be on the borderline of operability in small equipment.

C. Exploratory Runs with a Downflow Reactor

Two exploratory runs were made with a downflow, countercurrent hydrogen flow reactor. A simplified schematic of this reactor, GU 6, is shown in Figure 18. The standard preheater (with both hydrogen and slurry entering at the bottom) was used. From the preheater exit the slurry flows to a tee near the top of the downflow dissolver. Hydrogen flow in the dissolver is countercurrent

Figure 18

GU 6 REACTOR DIAGRAM (COUNTERCURRENT FLOW REACTOR)



1. Capillary flow split for hydrogen to preheater and dissolver.
2. Preheater (335 gm to fill)
3. Dissolver (433 gm to fill)
4. Product transfer line and pressure equalization loop
5. High pressure separator vessel
6. Liquid product atmospheric pressure receiver
7. Light liquid atmospheric pressure receiver
8. Flash condensate receiver
9. Flash condenser
10. Knock out condenser
11. Knock out condensate receiver
12. Drip Leg

Note: Downstream vessels are a dry ice trap, gas drying reagent, a gas sample bag, and tubing and valves to allow delivery of the gas sample to the laboratory for volume or compositional analysis.

(about 2/3 of the hydrogen is added at the bottom of the dissolver while the remaining 1/3 is added with the slurry at the bottom of the preheater). The anticipated advantage of this dissolver modification is associated with a higher hydrogen partial pressure. Product gases formed in the initial reaction stages (preheater) are removed from the reaction zone at the preheater exit (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 where it is believed to be most effective. Aside from possible operational problems, the anticipated disadvantage of the downflow reactor was the lack of accumulation of mineral matter (catalyst) which is experienced in the standard upflow reactor.

Conditions and results for these runs and appropriate control runs are summarized in Table 11. Run GU 161 was an SRC I run and GU 161A was a continuation of the same run with an increased hydrogen flow while GU 162R was an SRC II run.

Probably the most significant findings of these exploratory runs was that the downflow countercurrent reactor can be run without significant operating problems and that the yield structure is modified. Other conclusions must be regarded as tentative. The lack of accumulation of mineral matter in the downflow reactor appears to be of less significance in the SRC II run which is not unreasonable in view of the increased concentration of mineral matter in the feed slurry in SRC II operation.

In SRC I operation, replacement of the standard reactor with the countercurrent flow reactor resulted in:

1. Run control data that indicated less favorable conditions (increased blackness and decreased $\overline{\text{IR}}$).
2. Decreased hydrogenation level of recycle solvent and SRC.
3. A small increase in sulfur level of SRC (a small decrease in sulfur level of recycle solvent was noted, however).
4. Decreased MAF (moisture-ash-free) conversion (increased yield of insoluble organic matter).
5. Increased SRC yield and decreased oil yield.

In SRC II operation with the countercurrent reactor, the following were observed:

1. Decreased hydrogen content and increased sulfur content in the SRC II distillate product.
2. Little change in oil yield.
3. Significantly increased gas yield and decreased solid SRC yield.

TABLE 11

COMPARISON OF STANDARD AND DOWNFLOW REACTORS

	GU 127	GU 131	GU 161	GU 131A	GU 161A	GU 145RB[b]	GU 162R[c]
Conditions							
Reactor	Standard	Standard[a]	Downflow	Standard[a]	Downflow	Standard	Downflow
Coal (Kentucky Nos. 9&14)	Lot 5	Lot 5	Lot 5	Lot 5	Lot 5	Lot 6	Lot 6
Coal Feed Rate, lb/hr/ft^3	78.4	77.3	79.5	76.8	76.2	25.5	22.5
Nominal Residence Time, hr	0.35	0.36	0.35	0.36	0.36	0.98	1.11
Pressure, psig	1410	1410	1410	1410	1410	1900	1900
Nominal Dissolver Temp, $^\circ$C	452	454	455	454	455	455	455
Slurry Composition							
% Coal	39	39	39	39	39	35	35
% UFCS	--	--	--	--	--	60	60
% Recycle Solvent	61	61	61	61	61	5	5
Hydrogen Feed Rate							
Wt % based on slurry	1.79	1.85	2.04	3.84	4.12	4.62	6.30
MSCF/ton of coal	17.4	18.0	19.8	37.5	40.1	49.7	67.7
Yields, wt % based on coal							
C_1-C_4	3.3	4.7	4.8	3.6	4.8	10.5	16.7
Total gas[d]	7.5	9.9	8.3	10.2	7.4	17.6	27.0
Water	5.6	4.7	5.2	4.1	6.6	9.9	13.1
Light Distillate, <249°C	6.4	8.8	3.6	9.7	4.1	8.3	8.7
Heavy Distillate, >249°C	5.2	0.7	0.9	2.5	5.6	19.9	20.7
SRC	61.4	61.0	65.8	58.7	60.6	31.8	22.1
Insoluble Organic Matter	5.5	5.4	8.5	5.1	7.6	6.5	5.2
Ash	9.6	9.6	9.5	9.7	9.6	9.1	9.1
Workup Loss[e]	0.6	1.4	--	1.4	--	0.5	--
H_2 Reacted, gas balance	1.8	1.5	1.8	1.4	--	3.6	--
MAF Conversion, %	93.9	94.1	90.6	94.2	91.6	92.8	94.0
Recycle Solvent Analyses							
% C	87.59	87.86	88.20	87.87	88.03	87.16	88.30
% H	7.78	7.61	7.60	7.85	7.66	8.06	7.86
% S	0.46	0.48	0.38	0.46	0.38	0.33	0.52
% N			0.98		0.91	1.33	1.06
% O			2.84		3.02	3.12	2.26
Specific Gravity	1.0485	1.0598	1.0488	1.0526	1.0489	1.0412	1.0454
Vacuum Bottoms Analyses							
% C	86.35	86.96	86.90	86.48	87.44	89.10	89.66
% H	5.79	5.76	5.56	6.01	5.53	5.30	5.31
% S	0.84	0.77	0.98	0.81	0.88		0.52
% N			2.11		2.13	2.18	2.26
% Ash (Filtered product)	0.23	0.16	0.24	0.29	0.32		0.54

a) Dissolver with baffles

b) Yields reported for GU 145RB in this table differ somewhat from those reported in Section VII. For internal consistency, yields of C_5+ material in the output gas are included with the gas yield in this Table; in Section 7, these materials are included with the naphtha yield.

c) Yields for GU 162R are approximate only; steady state operation was not achieved.

d) Includes C_5+ material present in the vent gas.

e) Yields for GU 161, GU 161A, and GU 162R were calculated on a loss free basis.

The high gas yield and low reported SRC yield for GU 162R are of interest, but the SRC yield is approximate as steady state operation was not reached.

D. Subbituminous Coal (Amax Coal Co., Belle Ayr Mine)

Run GU 163 was the initial run (SRC I) with a subbituminous coal from the Amax Coal Co. Belle Ayr Mine. Due to the anticipated low reactivity of the Amax coal, this run was made at a longer residence time (1.3 hr) and higher pressure (2050 psig) than is typically used in SRC I operation with Kentucky coal. The most significant observation of this run was the gradual improvement in operability which occurred throughout the 72 hours of continuous operation before the run was terminated by a plugged reactor. Improvement during the run was shown by the run control data (increasing $\overline{IR}$ and decreasing blackness), increased MAF conversion (from 86.2% to 89.2%), and substantial improvements in recycle solvent recovery which in the early stages of the run was as low as 83% while for the final several products of the run a large excess of recycle solvent was obtained (13 wt % based on coal). Conditions and yields for the latter part of the run are summarized in Table 12.

Analysis of solids removed from the reactor after the run was terminated by the plug indicated a high calcium content. Iron content of the ash of the deposit was also increased over the iron content of the coal ash. The iron classification may be related to the gradual improvement in operability observed throughout the run.

TABLE 12

SRC I RUN WITH AMAX BELLE AYR MINE COAL

Run No.	GU 163
Conditions	
Coal	Amax
Coal Feed Rate, lb/hr/ft^3	18.1
Nominal Residence Time, hr	1.27
Pressure, psig	2050
Nominal Dissolver Temperature, °C	455
Slurry Composition	
% Coal	32.1
% Recycle Solvent	67.9
Hydrogen Feed Rate	
Wt % based on slurry	3.68
MSCF/ton of coal	43.2
Yields, wt % based on coal	
C_1-C_4	9.1
Total gas[a]	15.6
Water	11.9
Light Distillate, <249°C	7.6
Excess Recycle Solvent,[b] >249°C	12.9
SRC[c]	39.4
Insoluble Organic Matter	10.1
Ash	7.1
H_2 Reacted	
gas balance	4.6
product analysis	3.6
MAF conversion	89.2

a) Includes C_5+ material present in the vent gas.
b) Average recycle solvent analyses, %: C, 88.23; H, 7.59; S, 0.28; N, 0.77; O, 3.13.
c) Average SRC analyses, %: C, 89.39; H, 5.30; S, 0.20; N, 1.76; Ash, 0.34; O, 3.01.

VII. Summary and Analysis of SRC II Studies on Kentucky No. 9 Coal

A. Introduction

In a modified version of the Solvent Refined Coal Process, known as the SRC II process, a portion of unfiltered coal solution is recycled as the feed coal slurrying media in place of the distillate solvent used in the original SRC I process. This improved process increases the conversion of dissolved coal to lower molecular weight fuels; the primary product of this process is a liquid fuel in place of the solid product of the SRC I process.

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

1. Pressure and residence time used in the SRC II process are increased over those typically used in the SRC I process.
2. Recycle of the unfiltered coal solution allows additional reaction time for the conversion of high molecular weight dissolved coal to lower molecular weight products.
3. The concentration of mineral matter, which is known to function as a catalyst for SRC reactions, is increased.

The primary product of the SRC II process is a low sulfur distillate fuel oil. The residue from the vacuum distillation consists of three components: ash, insoluble organic matter, and material which has dissolved but is not distillable; i.e., similar to the solid SRC of the SRC I process. By appropriate selection of reaction conditions, the distillation residue yield can be reduced to that required for hydrogen generation for the process by partial oxidation.

The recently completed extended series of SRC II runs on Kentucky No. 9 coal carried out at the Merriam Laboratory was designed to support the planned SRC II work on Kentucky No. 9 coal at the SRC pilot plant. The results of this series of experiments have been analyzed and will be discussed in detail later in this report.

B. Objectives and Accomplishments

The principal objective of this series of SRC II trials on Kentucky No. 9 coal was to provide information for operation and experimental design for the Ft. Lewis Solvent Refined Coal Pilot Plant for their planned operation in the SRC II mode on Kentucky No. 9 coal. In May of 1976, Ft. Lewis personnel requested a series of six experimental runs at the Merriam Laboratory varying the coal concentration in the feed slurry, and the residence time of the slurry in the dissolver. The results of those runs were intended to satisfy three objectives:

1. An improved product yield data base.
2. An understanding of dissolver residence time effects.
3. An understanding of coal concentration effects.

After the conclusion of those six experiments in August, 1976, Ft. Lewis personnel requested an additional eight experimental runs whose principal objectives were:

1. To examine the effect of hydrogen feed rate.
2. To examine combined low coal concentration and short residence time.
3. To evaluate the effect of changing dissolver temperature.

The set of suggested experiments has been completed, including some replication and some repeats of experiments that had some mechnical problem. Steady state operation, providing reliable yield data, was achieved on most of the longer residence time trials and on one low concentration short residence time trial. In general, it was not possible to attain steady state operation at combined high coal concentrations and short residence times as the feed slurry viscosity became unmanageable, and the experiments were terminated by the inability to pump the excessively viscous slurries.

Substantial insight has been gained into the effect of coal concentration, residence time, dissolver temperature, and hydrogen circulation rate. Hydrogen partial pressure was not deliberately explored in this series of experiments. The limited data available are consistent with the general belief that increased hydrogen partial pressure increases reaction rates and improves operability. Some information was obtained on the effect of dissolver configuration, though this was not an explicitly studied variable. Accuracy of yield data for the principal yields was generally good with relative standard deviations of yields as measured in a set of replicate runs of 3-9%.

Accuracy of hydrogen consumption measurements is poorer for reasons discussed in detail in Section VII-E-5.

C. Conclusions

This series of SRC II runs on the Kentucky No. 9 coal have demonstrated that it is possible to sustain satisfactory SRC II operation at an inlet hydrogen partial pressure of 1900 psig at slurry residence times ranging from about one hour to 1-1/2 hours and at dissolver temperatures of 445 (833) to 465°C (869°F). Experimental results are reproducible and the precision is quite reasonable for the difficult materials handlings involved. Over the range of process conditions studied, simple linear correlations and first order reaction kinetics adequately describe the yields of major components. Hydrogen consumption estimates are not satisfactory for adequate estimates of process economics and substantial equipment modifications would appear to be necessary to achieve that level of accuracy.

Among specific conclusions drawn are:

1. Distillate oil yields decrease linearly with increasing coal concentration in the feed slurry while SRC and total vacuum

residual yields increase linearly over the range studied. Little can be said about the response of light gas yields or hydrogen consumption to varying coal concentrations.

2. First order kinetics adequately describe the dependence of distillates and residual yields on dissolver residence time.

3. Process yields are sensitive to dissolver temperature. SRC and total distillation residue yields decrease significantly with increasing temperature while total distillate yield and C_1-C_4 hydrocarbon yields increase moderately with increasing temperature.

4. No significant effect of hydrogen feed rate on any yields was seen in the ranges studied. The applicability of this conclusion to larger reactors should be viewed with special caution because the reactor hydrodynamics may change dramatically with reactor scale.

5. Increased hydrogen partial pressure generally improves operability of the process though this phenomena was not explored in detail in this study.

6. Substantial equipment and analytical modifications would be necessary to provide hydrogen consumption measurements adequate for process economic evaluation or design work.

7. At a fixed slurry residence time, dissolver temperature, and hydrogen partial pressure, oil production rates expressed as units of production per unit of volume of dissolver are insensitive to coal feed rate when expressed as units of feed per unit of volume of dissolver.

D. Experimental Technique and Equipment

1. Description of Unit

A simplified schematic drawing of the laboratory scale continuous reactor used in the present work is shown in Figure 19. The slurry feed subsystem, hydrogen compression and metering subsystem, and gauge purge subsystem exist as previously described[3] with only minor modifications. Some modifications have been made in the slurry feed system in attempts to improve operability with viscous feed slurries. The most significant modification is the replacement of the feed slurry recirculation pump with either a Moyno progressing cavity pump, type CDO, with a stainless steel rotor and Viton stator, or Tuthill lobe pump, Model 4A. Both of these pumps have proved to be more satisfactory in this service than the previously used Viking F-32 gear pumps.

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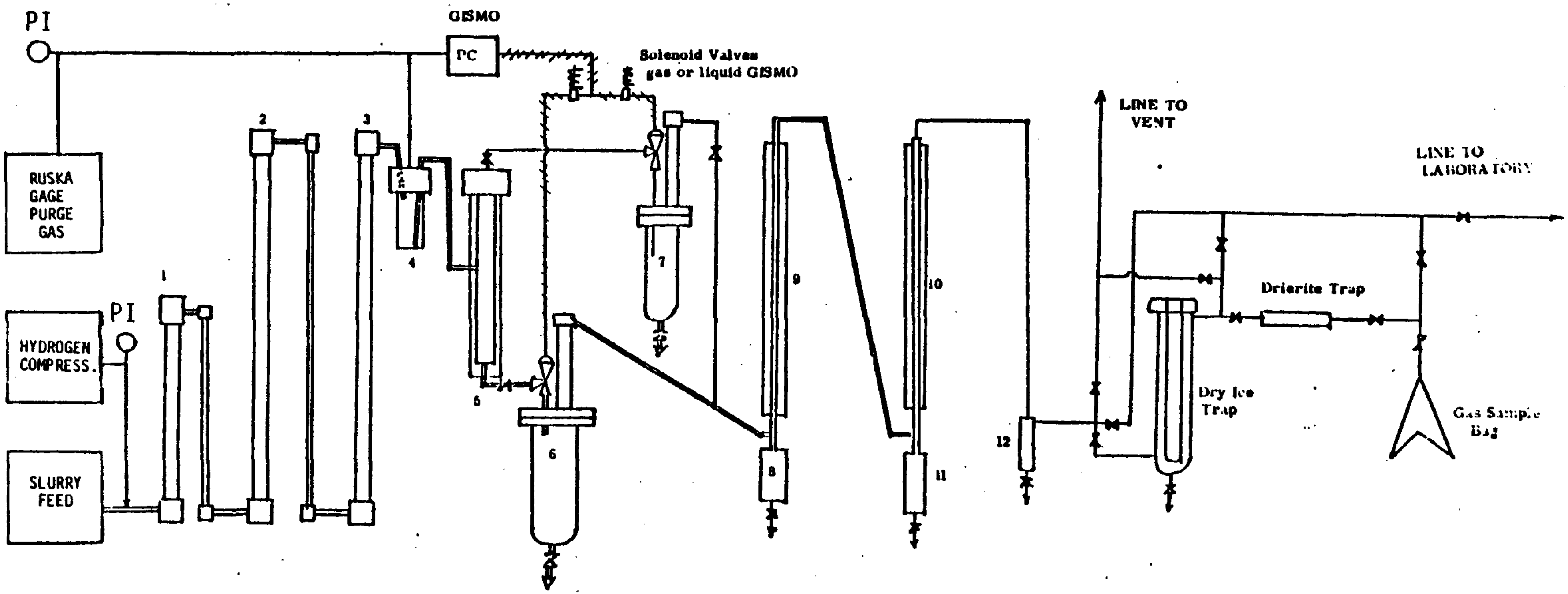
[3] Anderson, R. P., and Wright, C. H., ERDA R & D Report No. 53, Interim Report No. 8, Volume II, Part 3, FE 496-T1. Continuous Reactor Studies Using Petroleum Derived Solvents.

Figure 19

GU 5 REACTOR DIAGRAM



1. Preheater (335 gm to fill)
2. First Dissolver (520 gm to fill)
3. Second Dissolver (520 gm to fill)
4. 1 liter Pressure Buffer Vessel
5. High Pressure Separator
6. Product Slurry Atmos. Flash Vessel
7. Overhead Separator
8. Vapor Condensate Receiver #1
9. Vapor Condensor #1
10. Vapor Consensor #2
11. Vapor Consensate Receiver #2
12. Drip Leg Receiver

Note: Downstream vessels are a dry ice trap, gas drying reagent, a gas sample bag, and tubing and valves to allow delivery of raw or processed gas samples to the laboratory or to the gas bag.

The standard reaction subsystem consists of a preheater and either one or two dissolvers. The preheater is a 4.5 ft section of 11/16" ID pressure tubing and each of the dissolvers is a 7 ft section of the same diameter tubing. The preheater is typically operated with a temperature profile to allow a temperature of 400°C in the top zone (438°C for the five runs with lot 6 coal). Due to the low temperature in this vessel, its volume is not included in calculation of reactor volume, residence times, or feed rates expressed in lb/hr/ft^3. Reaction volume for each dissolver (corrected for thermowell volume) is 452 ml (0.01596 ft^3) which corresponds to 520 g of slurry to fill. Thus, a slurry feed rate of 1040 g/hr corresponds to a nominal liquid residence time of 1 hr with both dissolvers in use. The two dissolver configuration was used in most cases with the one dissolver reactor being used with some of the short residence time (2/3 hr) runs (GU 142R, GU 149R-GU 151R) and also with two of the 1 hr residence time runs (GU 143R, GU 144R).

In two runs, GU 147R and GU 148R, a modified preheater was used in place of the standard preheater. This modified preheater was a coil 20 ft in length by 0.30" ID. This preheater was designed to more closely match the flow parameters of the Ft. Lewis Pilot Plant preheater.

The pressure letdown and gas and liquid separation systems have been extensively modified. Prior to this series of runs, all reactor effluent (gas, liquid, and solid) flowed through a single pressure control valve which resulted in substantial wear to the tungsten carbide trim. This resulted in an unsatisfactory life of this valve which often necessitated shutdown due to valve failure. Installation of the high pressure separator (vessel 5, Figure 19) and separate gas and liquid control valves has extended the life of the pressure control valves considerably.

Reactor products include the stripped unfiltered coal solution (vessel 6), light oils and an aqueous phase containing dissolved ammonia, sulfides, and carbonates (from vessels 7, 8, 11, and 12, and the dry ice and Drierite traps) and the vent gas containing unreacted hydrogen and product gases.

During this series, the gas cleanup system (removal of light hydrocarbon oils from the gas stream) was improved and the gas bag sampling technique was replaced with on-line gas sampling techniques.

2. Operating Techniques

A typical run in which a single condition is investigated typically requires on the order of 60-100 hours of continuous operation to reach steady state conditions followed by a period of steady state operation during which data for yield determinations and product properties are collected.

The most satisfactory method to reach steady state operation in the SRC II mode has been found to begin operation with a slurry of distillate solvent and coal. As coal solution is collected, the required amount is blended with coal and added to the reactor slurry feed vessel. This process is continued until a satisfactory period of steady state operation is achieved. The approach to lineout is followed by empirical run control measurements which have been discussed previously and will not be reiterated here. A typical run control chart is shown in Figure 20. For this run it can be seen that feed slurry ash increased as coal solution was recycled until a steady state concentration of about 9% was observed, blackness[4] increased to a steady state value slightly below 20 and IR[4] (except for the low value for the initial sample which is a reflection of the lower hydrogenation state for the startup solvent) shows some oscillation but no long term trends. These control measurements together with gas yields, distillation results, etc., give rapid indications of the progress of the run and indicate when steady state operation is achieved; steady state operation is later confirmed by constancy of product yields and compositions.

A portion of the coal solution is used in slurry formulation; the remainder is available for product isolation. Yields presented in this report are for the process involving distillation of unfiltered coal solution to produce a distillate fuel product and a distillation residue. Yields of the three components of the distillation residue--ash, insoluble organic matter, and solid SRC (dissolved but not distillable)--are reported separately. In the actual laboratory workup, a filtration step may precede the distillation. In either case, the distillation is a batch operation carried out in laboratory equipment with an oil vacuum pump. Typically, pressure early in the distillation is below 1 mm Hg and increases as the distillation temperature exceeds 250°C. Distillations are terminated before serious decomposition takes place; the normal distillation endpoint is about 270°C head temperature at which time the distillation pressure has often increased to about 3-5 mm Hg.

In the GU 137R-GU 160R series, solids level in the feed slurry was not allowed to exceed 48%. (Solids include ash and insoluble organic matter in the unfiltered coal solution used in a slurry formulation as well as the added coal). This constraint was imposed by consideration of possible operational constraints for the Ft. Lewis Pilot Plant. At the higher coal concentrations used, this limitation necessitates the use of some recycle solvent in slurry formulation. This is illustrated in the simplified flow diagram below where solids level in the feed slurry can be controlled at a constant coal concentration by varying the relative amounts of unfiltered coal solution and recycle solvent used in slurry preparation. At a coal concentration of 30%, no recycle solvent is

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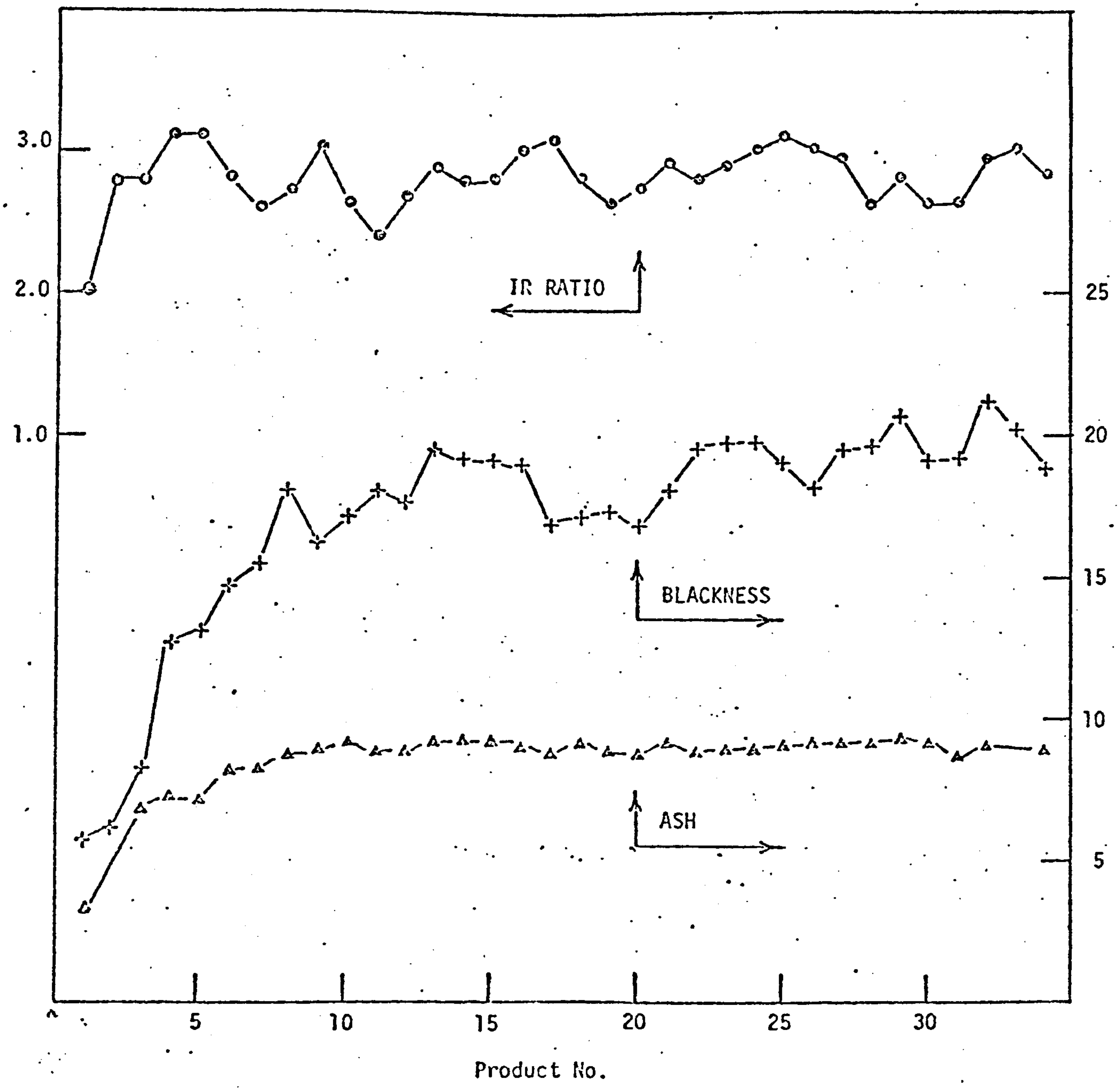
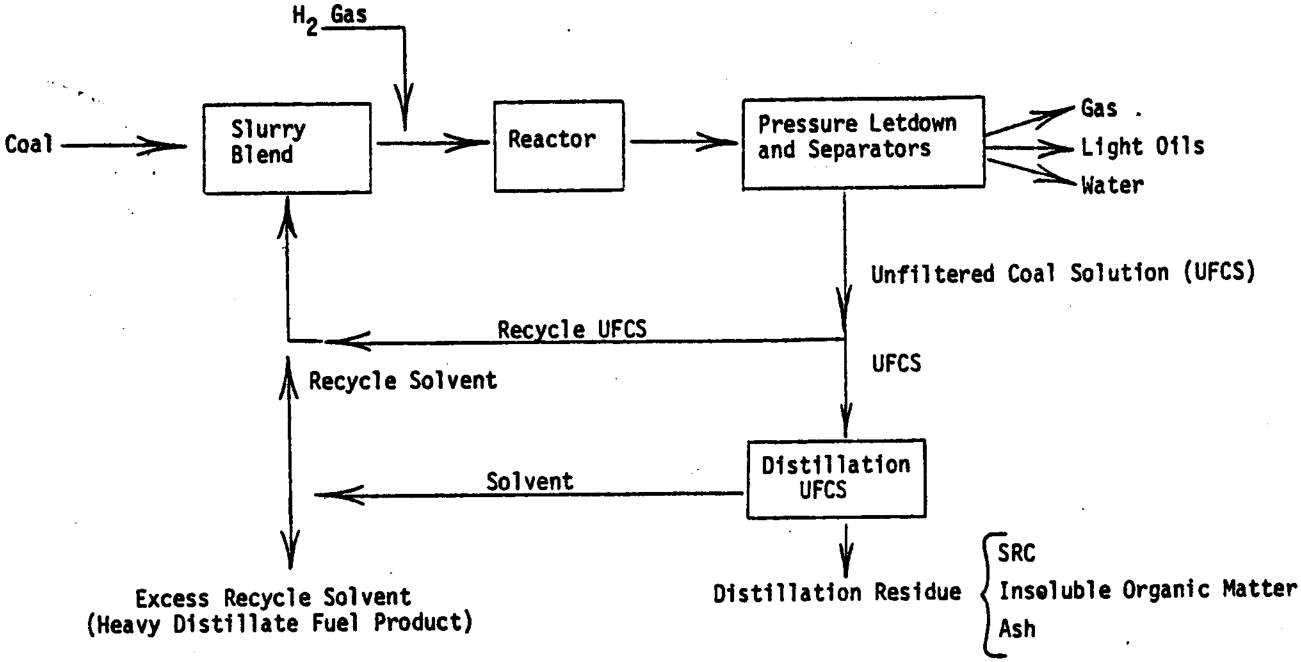
[4] Blackness is a measure of absorbance of the coal solution in the visible region. IR, determined from the infrared spectrum of the coal solution, is a measure of the hydrogenation state of the coal solution and increases as the degree of hydrogenation of the coal solution increases. See the following for a more complete description of these measurements: Wright, C. H., Pastor, G.R., Perrussel, R. E., ERDA R&D Report No. 53, Interim Report No. 7, Volume II, Part 2 - Continuous Reactor Experiments Using Anthracene Oil Solvent.

Figure 20

GU 146R RUN CONTROL DATA



required. At a coal concentration of 40%, other components of the feed slurry were 51% unfiltered coal solution and 9% recycle solvent, and at a coal concentration of 45%, other components of the feed slurry were 27.5% unfiltered coal solution and 27.5% recycle solvent. As currently practiced, the recycle solvent used in a slurry formulation is the same material as the distillate fuel product of the SRC II process. Unfiltered coal solution is distilled; the required amount of distillate is used in slurry formulation and the remainder is available as product.



3. Analytical Techniques

This section will be limited to a brief discussion of the analytical techniques used in determination of product distributions.

a. Gas Analyses

Gas output weight is determined from gas output volume (moles/hr) and the gas molecular weight as determined by the gas density method. As gas output rates (determined via wet test meter) are determined while the liquid level control valve is closed, gas output rates must be corrected for gas displaced from the high pressure separator by accumulating liquid. Concentration of the following gases in the output gas are determined by gas chromatography: methane, ethane, propane, iso- and n-butane, carbon monoxide, carbon dioxide, nitrogen, and oxygen. Nitrogen and oxygen are generally present in very small amounts; results are normalized to give compositions on an air free basis. Hydrogen sulfide is determined by an iodimetric technique and ammonia is determined by infrared. Hydrogen is determined by difference; i.e., by subtracting all identified gases from 100. The gas output of each gas in g/hr is determined from its concentration, molecular weight and the total gas output. The total weight of identified gases is compared with the weight of gas determined by the gas density method. Weight by the gas density method exceeds the total weight of identified gases; this quantity has been tabulated in past reports as "unidentified gas." This material has been shown by gas chromatography-mass spectrometry to consist primarily of C_5-C_8 hydrocarbons. In this report this material has been included with the naphtha fraction (C_5-193°C).

b. Water and Light Oil Fractions

Water and light oil yields are determined from analysis of composite samples from the various light oil and water receivers. Yields are reported for oil cuts as follows: naphtha, <193°C; wash solvent, 193-249°C; and recycle solvent (or heavy distillate fuel product), >249°C. The ranges were selected to correspond to conventional cuts in the SRC I process. These yields are reported in the tabular data summary in Appendix B. In the discussion of process variable effects, only total distillate yields are generally considered rather than yields of discrete distillation fractions; this eliminates problems which might be introduced by variability in the fractionation procedure.

All oil fractions are distilled except for material collected in the Drierite trap. As it is not possible to analyze this material conveniently and as the amount is small (typically 1-3 g/hr), it was assumed to consist of equal amounts of water and naphtha (<193°C). This assumption is based in part on the results of early runs in the series where a charcoal trap was used in place of the Drierite trap.

Water yields reported in the results summary are for yields of the crude aqueous phase. This phase is typically about 92% water containing dissolved ammonia, sulfides, carbonates, and phenolic material.

Distillation procedures, as described in Section VII-D-2, are used in the determination of yields of recycle solvent (or heavy distillate fuel product) and distillation residue. Breakdown of the distillation residue yield into the components (ash, insoluble organic matter, and SRC) is described in Section VII-E-1.

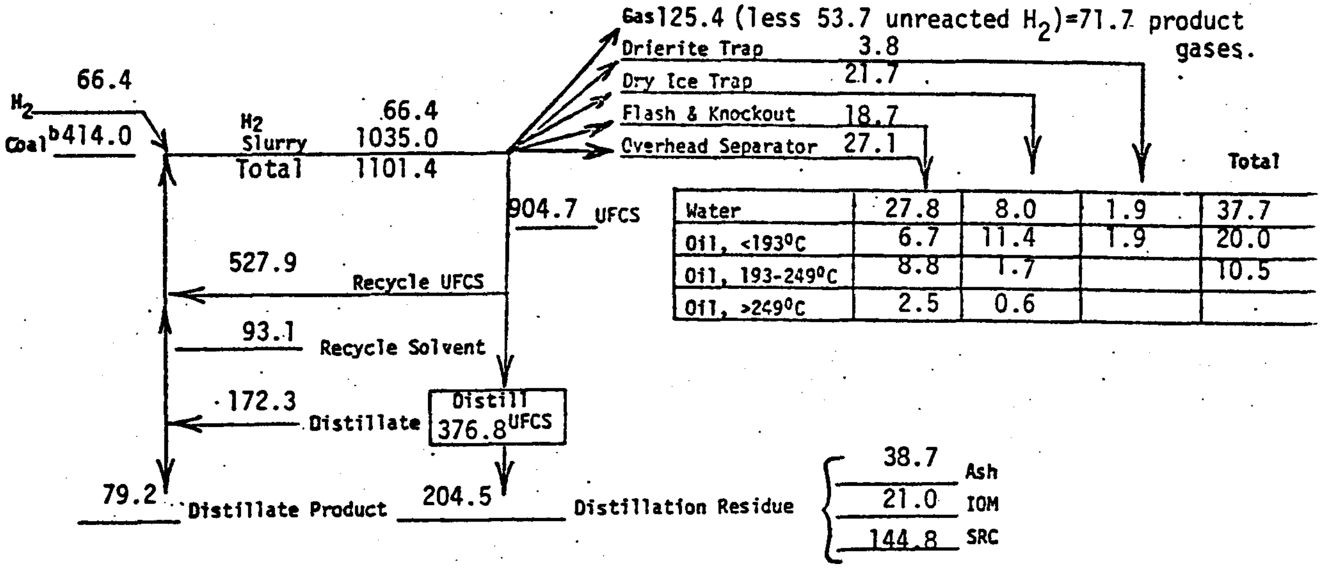
E. Material Balances

1. Methodology

The technique used in determination of product yields is best illustrated by example. A flow diagram for run GU 154R is shown in Figure 21. The flow diagram was constructed for a 40-hour period of stable operation. Stable operation was indicated by run control measurements (Section VII-D-2), gas compositions, distillation

Figure 21 GU 154R Hourly Flow Diagram[a]

Reactor Balance, Products 18-33 (40.0 hr); Results Based on Distillation of UFCS



				Total
Water	27.8	8.0	1.9	37.7
Oil, <193°C	6.7	11.4	1.9	20.0
Oil, 193-249°C	8.8	1.7		10.5
Oil, >249°C	2.5	0.6		

Yield Summary

Material	g/hr	Wt % based on dry feed coal
H_2O	37.7	9.11
C_1	14.39	3.48
C_2	9.16	2.21
C_3	9.99	2.41
C_4	6.01	1.45
CO	2.84	0.69
CO_2	4.85	1.17
H_2S	9.30	2.25
NH_3	1.77	0.43
C_{5+}[c] (gas)	13.4	3.24
Naphtha, <193°C	20.0	4.83
Lt. oil, 193-249°C	10.5	2.54
Heavy distillate, >249°C	82.3	19.88
SRC	144.8	34.98
IOM	21.0	5.08
Ash	38.7	9.35
Total	426.7	103.10

	g/hr	wt%
Total C_1-C_4	39.55	9.55
Total Oil (C_{5+}- heavy distillate)	126.2	30.49

a) All flows are given in g/hr. b) Dry coal basis, includes 38.7 ash.
c) Difference between total gas weight and weight of quantified gases.

results, and MAF conversion. Material balance data for this period show a total reactor input of 44051 g and a total output of 44056. Reactor input is forced to equal reactor output by the appropriate adjustment in slurry input weight; in this case, a correction of only 5 g is required. All quantitites given in the flow diagram are in g/hr.

Hydrogen input (controlled by pressure differential across a metering valve or capillary) is determined by calibration before and after a run; in some cases, only a single calibration is available. The hydrogen input shown in the flow diagram includes one mole/hr added as a gauge purge; as it does not have an opportunity to react, this quantity is excluded from the hydrogen feed rate expressed as wt % based on slurry or as MSCF/ton of coal.

Slurry input is determined by weight loss from the slurry feed vessel with appropriate correction to force a mass balance as noted above. The components of the feed slurry (coal, unfiltered coal solution, recycle solvent) are precisely known as slurries formulated batchwise using manual gravimetric techniques. Slurries are formulated to contain the required amount of coal on a dry basis. Water added with the coal is assumed to be lost during the slurry formulation procedure which involves heating the slurry to ≈110°C and then cooling (before the slurry is added to the reactor slurry feed vessel).

Gas output weights were determined as discussed in Section VII-D-3. Gas output weights shown in the flow diagram are averages of five complete gas analyses. The total gas output of 125.4 g/hr includes 53.7 g of unreacted hydrogen and 71.7 g of product gases. Hydrogen input was 66.4 g/hr while measured hydrogen output was 53.7 g/hr. This was a hydrogen consumption of 12.7 g/hr or 3.1 wt % based on feed coal.

Yields of individual gases were determined as discussed in Section VII-D-3. The total yields of identified gases, excluding hydrogen, is 58.3 g/hr while the total yield of product gases by gas density is 71.7 (125.4 g/hr total - 53.7 g hydrogen). The 13.4 g of "unidentified gas" is assumed to consist of C_5-C_8 hydrocarbons and is included in the naphtha yield.

The Drierite trap condensate of 3.8 g/hr is assumed to consist of equal amounts of water and naphtha.

Compositions of materials collected in the light oil and water receivers were determined by distillations of three individual composite samples: a dry ice trap sample, a flash and knockout composite sample (vessels 8, 11, and 12, Figure 19) and an overhead separator composite sample (vessel 7, Figure 19). Water was separated before distillation. A preliminary distillation is carried out under vacuum. Products of this distillation are a cold trap sample containing water and oil, and a distillate oil sample (e.g., <90°C at 3mm Hg). Cold trap oil and water are separated; all other materials are recombined and distilled at atmospheric pressure. The purpose of the preliminary vacuum distillation is to circumvent foaming problems which are experienced if it is omitted. Atmospheric distillation cuts collected are: naphtha, <193°C; wash solvent, 193-249°C; and a residue, >249°C. The small losses experienced in the distillations are presumed to be due to the loss of light hydrocarbons. In construction of the flow diagram, these distillation results are calculated on a loss-free basis with the loss included with the cold trap oil category. Actual losses for the three samples were 0.9%, 1.4%, and 7.2% for the overhead separator, flash knockout, and dry ice trap composites, respectively. Unfiltered coal solution was produced at a rate of 904.7 g/hr. Of this, 529.7 g/hr is required for slurry formulation with the remaining 376.8 g available for product recovery. Yields considered are for those for distillation of the unfiltered coal solution (distillation procedures are discussed in Section VII-D-2). Distillation results are based on the distillation of seven individual samples with a total weight of 15258 g. A total of 8280.4 g or 54.27% of vacuum distillation residue was obtained.

As noted previously, the distillation residue may be considered to consist of three components: ash, insoluble organic matter, and SRC. Yields of the three components are reported individually. A forced ash balance calculation is used. As input ash was 38.7 g, (414.0 g of coal, 9.35% ash), ash in the products is also assumed to be 38.7 g. This theoretical ash output is compared with the actual experimental ash output. Distillation of 376.8 g of unfiltered coal solution produces a distillation residue of 204.5 g. Actual ash content of the vacuum bottoms (average of four samples) is 18.83% which provides an actual ash output of 38.5 g or 99.5% of the theoretical ash input.

Yield of insoluble organic matter (IOM) is determined by analysis of pyridine insolubles samples. A sample of pyridine insolubles is obtained from a sample of unfiltered coal solution or preferably from a wet filter cake sample if some of the coal solution was filtered. The pyridine insoluble fraction consists of two materials: ash and insoluble organic matter. In GU 154R, the pyridine insoluble fraction contained 64.79% ash. The ash yield of 38.7 g divided by 0.6479 indicated a pyridine insolubles yield of 59.7 g including 38.7 g of ash and 21.0 g of insoluble organic matter. Subtraction of these quantities from the total distillation residue yield leaves an SRC yield of 144.8 g.

Subtraction of the distillation residue yield of 204.5 g from the weight of 376.8 g leaves a loss free distillate yield of 172.3 g. Actual distillation losses are small; in this case the actual distillation loss was 0.26% and an additional 0.36% of the material distilled was collected in a cold trap. Of the loss free distillate yield of 172.3 g, 93.1 g was required for slurry formulation, and the remainder of 79.2 g was available as the heavy distillate product. The weight of >249°C oil from distillation of the light oil fractions was added to the heavy distillate yield.

As all operations are corrected to a loss-free basis, the total weight of products is equal to the weight of input coal plus the weight of hydrogen reacted. Weights of all products are divided by the weight of input coal to give percentage yields based on dry feed coal.

2. General Closure

Techniques in use allow excellent mass accountability. In over half of the runs reported, reactor output is within 0.5% of the reactor input. In about one-fourth of the runs, the variation between input and output exceeded 1.0%, and in only two cases did the variation exceed 2% (a 2.4% loss in GU 158R and a 3.8% gain in GU 137R). There is no tendency for either a negative or positive bias to predominate with about equal numbers of runs showing gains or losses.

3. Estimated Precision and Reproducibility

A brief set of near replicate runs is available from which precision and reproducibility of yields can be estimated. Estimated relative standard deviations for the principal yields range from 1% for the total distillation residue to 6% for the total distillate yield. Individual component yields within the distillate and residual categories range from 2 to 21% with the 21% occurring with the wash solvent, a material present in relatively low yield and subject to distillation errors on both the low and high boiling side of its range. The reproducibility of water yields within this set of runs is consistent with the levels of precision indicated for other component yields, having a relative standard deviation of 10%. Hydrogen consumption reproducibility varied substantially depending upon which technique was used to estimate the consumption. In this set of data, the gas balance technique exhibited a relative standard deviation of 17% while the product analysis approach yielded a relative standard deviation of only 5%, a substantially greater reproducibility. The hydrogen consumption measurement is discussed in substantially greater detail in Section VII-E-5.

4. Estimated Accuracy - General

All yield estimates involve measurement of both a flow rate and a composition in some sense. The feed rates and production rates of liquid streams are determined gravimetrically and are generally known within 1% relative. The principal source of error in the determination of the yields of the various distillate cuts is the precision of the distillation process itself; hence, the intermediate stream, wash solvent, has an indicated relative standard deviation of 21% in the series of replicates while the overall distillate yield had an indicated relative standard deviation of only 6%.

Total distillation residue yield has, in the set of replicates, a relative standard deviation of 1%, essentially the level of gravimetric precision available. The reproducibility of the partition of the total distillation residue amongst SRC, insoluble organic matter, and ash has somewhat less reproducibility, and the results are analytically interdependent. The indicated variability of the determination of the insoluble organic matter between the runs is substantially larger than that measured on repeat analyses on the product of a single run indicating that within the set of the replicate runs there are some real differences in insoluble organic matter yields. The sum of SRC plus insoluble organic matter yield, which is the material available for hydrogen generation, is not sensitive to this partition and the relative standard deviation of the sum of those yields in the replicate set was 1.7%.

The accuracy with which gaseous product yields can be determined is dependent on a more complex sequence of measurements. The gaseous product flow rate is sampled, measured periodically via a wet test meter, and not accumulated continuously. The gas stream is also sampled periodically for chromatographic analysis. With the present equipment typically five to eight chromatograph samples are analyzed in a run. The chromatographic determination of component concentration depends on calibration with standard gas blends. Combined analytical and flow related uncertainty is estimated to range from about 5% for major components such as methane to about 12% for minor components such as butane.

The accuracy with which the yields of major products can be estimated is adequate for the scale at which the coal processing is carried out in the Merriam bench scale unit. The accuracy with which hydrogen consumption can be estimated, and to which process economics are very sensitive, is discussed in detail in the following section.

5. Accuracy of Hydrogen Consumption Measurements

The accuracy with which hydrogen consumption can be measured is of substantial interest for several reasons. Included among them are:

1. The sensitivity of process economics to hydrogen requirements.
2. The apparent scatter in hydrogen consumption data.
3. The experimental difficulty associated with precise measurements of gas volume flows in a small high temperature, high pressure unit.

Two techniques were used to measure hydrogen consumption. The first, the gas balance technique, involves measuring the flow rates and compositions of the gases entering and leaving the process unit. The hydrogen consumption rate is the difference of the two measured component flow rates. The second technique, referred to as the product analysis technique, does not require the measurement of the inlet hydrogen flow but does require the measurement of the rates and hydrogen contents of all products and the rate and analysis of the feed coal. Estimates of the anticipated error of hydrogen consumption measurement by the gas balance technique and by product analysis technique are presented in detail in Appendix B. These analyses indicate that the 95% confidence interval on hydrogen consumption is about 2% absolute by the gas balance technique and about 1.5% absolute for the product analysis technique with the current flow measurement and analytical capabilities of the Merriam facility. These estimates of anticipated error, while not large in an absolute sense, are substantial relative to the expected consumption of hydrogen. Both approaches are sensitive to error in flow measurement and in composition of the product gases from the unit while the gas balance technique is particularly sensitive to error in the measurement of the input hydrogen flow rate and the product analysis technique is sensitive to flow rate and analytical accuracy of the off gas and of the coal feed. Estimated relative standard deviations for the two techniques are 0.15 and 0.10 for the gas balance and product analysis techniques, respectively. Actual reproducibility in a short series of replicate runs (see Table 26) produced relative standard deviations of 0.17 and 0.05, respectively, for the gas balance and product analysis techniques. This may indicate that, for the product analysis technique, the reproducibility is significantly better than the ultimate accuracy, while the reproducibility of hydrogen consumption by the gas balance technique in this small series is about equal to the estimated accuracy.

The hydrogen consumption measurements for the set of replicate runs indicate that there may be some bias between the gas balance and product analysis technique. A statistical test for this bias by the t-Test indicates that, at a 5% significance level, the difference in gas balance and product analysis techniques is real. This is detailed in Table 13. Further analysis of all runs of this set, for which both gas balance and product analysis estimates of hydrogen consumption were available, is shown in Table 14. For the entire set of data, the average difference of the two techniques is quite small and is not significant at the 5% level. The apparent bias present in the replicate runs is believed to originate with a gas leak problem discovered during run 145R but which may have been present earlier. Such a leak would have had the effect of creating a bias in the observed direction.

Hydrogen consumption can be correlated with several other variables. Figures 22 and 23 present plots of hydrogen consumption vs SRC yield by the gas balance and product analysis techniques, respectively. In both cases, the data was fitted by least squares to a linear curve and a 95% confidence band shown. Similar correlations with similar levels of scatter can be made against methane or total C_1-C_4 hydrocarbon yields and, perhaps, other yields. The principal source of scatter in these correlations is due to the scatter in the measurement of hydrogen consumption, though some real chemical effects may be present beyond the simple relationship implied by the correlation.

TABLE 13

ANALYSIS OF H_2 CONSUMPTION
COMPARISON OF GAS BALANCE & PRODUCT ANALYSIS TECHNIQUE

t - TEST ON PAIRED DATA

Run No.	Gas Balance Y_1	Product Analysis Y_2	Δ $Y_1 - Y_2$
143 R	4.33	3.12	1.21
144 R	4.93	2.81	2.12
145 RB	3.57	3.03	0.54
148 R	3.42	2.86	0.56
			$\bar{d} = 1.11$
			$S_d = 0.74$

Confidence interval $\bar{d} \pm t\ \nu,\ \alpha/2 \sqrt{\frac{1}{n} S_d^{\,2}} = 1.11 \pm 1.03$

Where $t\ \nu,\ \alpha/2 = t_4,\ .025 = 2.776$

$P\ (0.08 \leq \delta \leq 2.14) = 0.95$

There is a difference in the two techniques of measuring H_2 consumption in this subject of data, and the difference is significant at the 5% level; i.e., can reject the hypothesis that $\delta \simeq 0$ with $P \geq 0.95$.

TABLE 14

ANALYSIS OF H_2 CONSUMPTION MEASUREMENT BY GAS BALANCE AND PRODUCT ANALYSES

t - TEST ON ALL AVAILABLE PAIRED DATA

Run No.	Gas Balance Y_1	Product Analysis Y_2	Δ $Y_1 - Y_2$
135 R	4.0	4.3	-.3
137 R	3.8	4.0	-.2
138 R	3.6	2.9	.7
139 R	3.6	3.3	.3
141 R	4.1	4.5	.4
142 R	4.8	4.0	.8
146 R	3.4	3.3	.1
150 R	3.9	4.6	-.7
152 R	3.3	3.7	-.4
153 R	2.7	2.7	0
154 R	3.1	3.2	-.1
155 R	3.1	3.6	-.5
158 R	3.1	4.1	-1.0
160 RA	4.5	4.6	-0.1
160 RB	4.5	4.5	0
134 R	4.5	4.8	-.3
136 R	4.1	4.3	-.2
140 R	5.4	5.1	0.3
143 R	4.3	3.1	1.2
145 RB	3.6	3.0	.6
149 R	4.7	4.8	-.1
151 R	4.0	3.7	0.3
157 R	4.1	3.6	0.5
			$\bar{d}$ = .0217
			S_d = 0.51

$t_{22,.025} = 2.074$

Interval estimate $\bar{d} \pm t_{22,.025} \sqrt{\frac{1}{23}(.51)^2} = 0.02 \pm 0.22$

$$P\ (-0.20 \le \delta \le 0.24) = 0.95$$

Data do not warrant rejecting the hypothesis that $\bar{d} = 0$; i.e., there is no significant difference between the techniques.

Figure 22

CORRELATION OF HYDROGEN CONSUMPTION WITH SRC YIELD
HYDROGEN CONSUMPTION BY GAS BALANCE

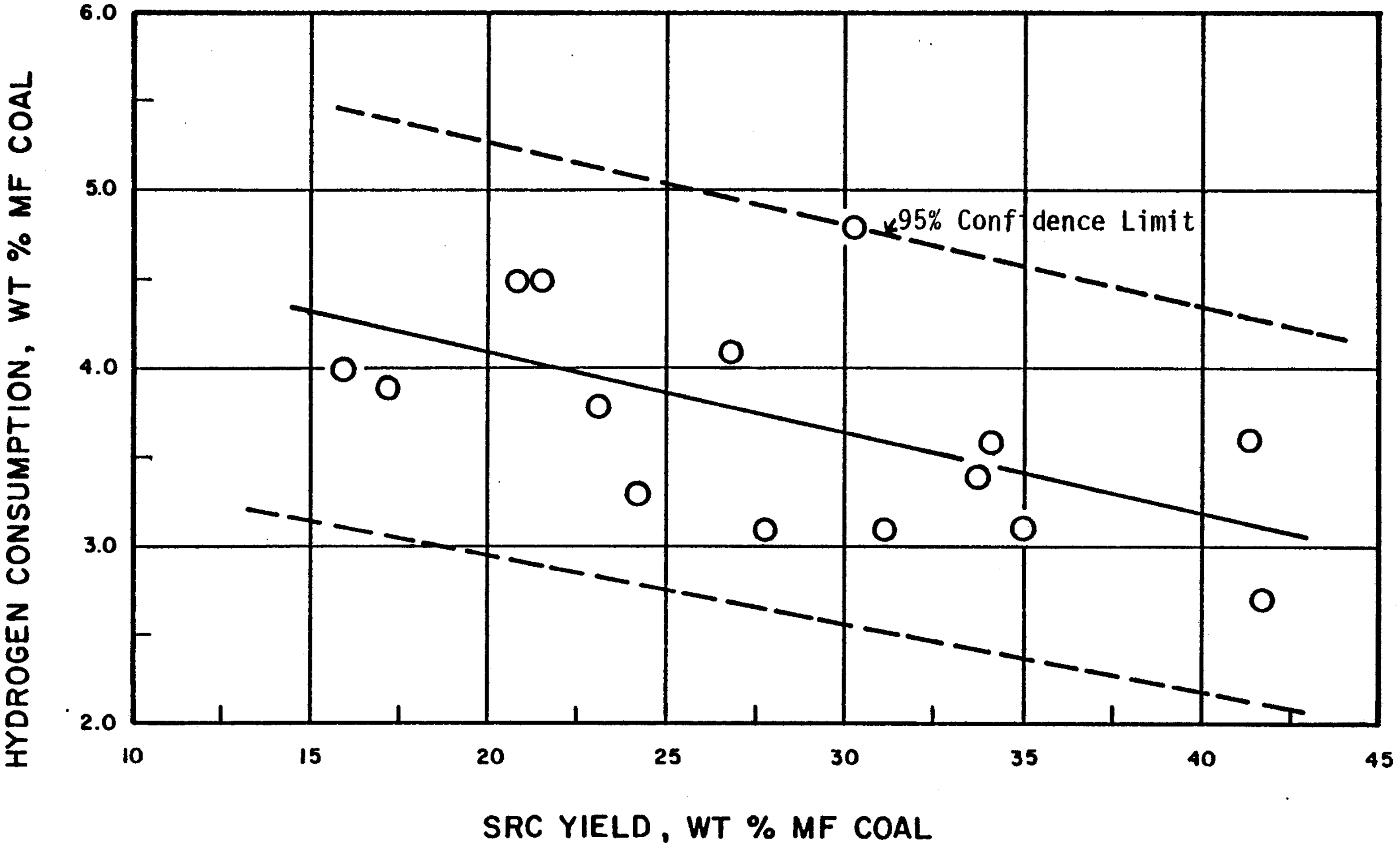
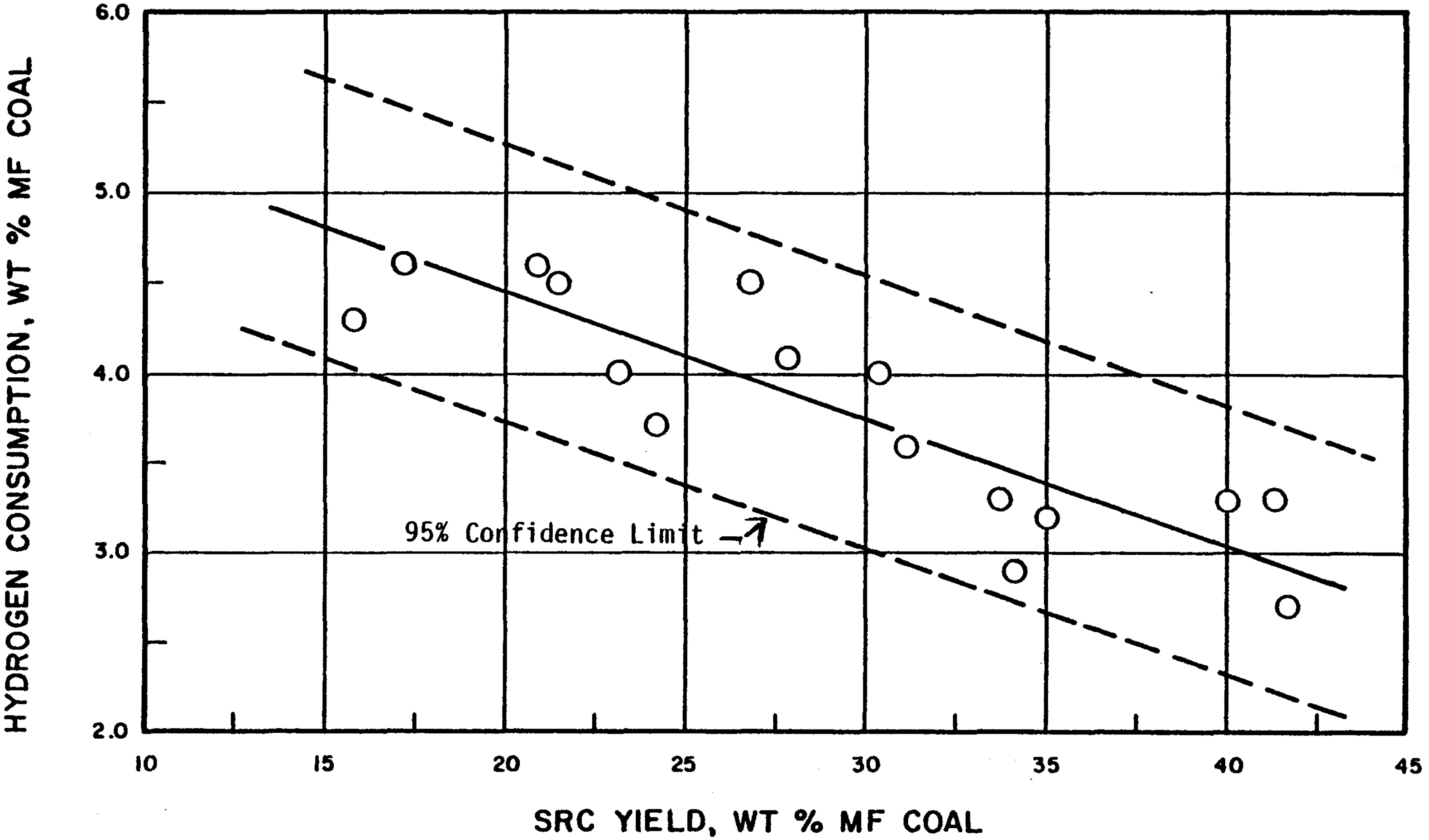


Figure 23

CORRELATION OF HYDROGEN CONSUMPTION WITH SRC YIELD
HYDROGEN BY PRODUCT ANALYSIS



F. Process Variable Effects

1. Effect of Coal Concentration

Coal concentration in the feed slurry has a substantial effect on distillate and residual yields when processing Kentucky No. 9 coal at otherwise uniform processing conditions. Figure 24 illustrates the effect of coal concentration over a range of coal concentration from 30-45 wt % of the slurry at process conditions that were standard at 455°C (851°F) dissolver temperature, 1 hr nominal slurry residence time, 1900 psig inlet hydrogen partial pressure and the use of a single lot (No. 5) of coal from the Colonial Mine. Process solvent and total distillate yields decrease linearly with decreasing coal concentration while yields of SRC and total distillation residue increase linearly with increasing coal concentration. There is no apparent direction to the C_1-C_4 yields over this range of coal concentration.

Table 15 presents the results of linear least-squares curve fit of the yield data presented in Figure 24. Two things are apparent from these fits. First, that for distillate and residual yields a linear curve gives excellent fit to the data over the range studied, and second, that little correlation appears between the light gas yield and the coal concentration. The differing slopes for process solvent yield and total distillate yield indicate a shift toward higher molecular weight and higher boiling solvent production with increasing coal concentration. It can be physically argued that total distillate residue yield, hence also SRC yield, should be zero when the feed coal concentration is zero and that the process solvent and total distillate yields must be zero or less as when coal concentration is zero, hence, the linear behavior exhibited in the region studied must break down as coal concentration becomes progressively lower.

Table 16 presents runs at high and low levels of coal concentration together with the average process conditions and yields at these high and low levels. These results are consistent with those in Figure 24 and reinforce the conclusion that total distillate yield and process solvent yield decrease with increasing coal concentration in the feed slurry, and that SRC and total vacuum residual yields increase with increasing coal concentration in the feed slurry. Little can be said about the response of light gas yield or hydrogen consumption to varying coal concentration.

Figure 25 illustrates the effect of coal feed rate on the production rate of various products at a fixed nominal slurry residence time of 1 hr, a dissolver temperature of 455°C, and 1900 psig inlet hydrogen partial pressure. Higher coal feed rates (as lb/hr/ft^3 dissolver volume) are proportional to coal concentration in the feed slurry as the total slurry charge rate is fixed.

Under the conditions outlined, the production rate of distillate liquids is insensitive to the coal feed rate while the production rate of distillation residue is proportional to the coal feed rate.

TABLE 15

EFFECT OF COAL CONCENTRATION ON SRC II YIELDS - KENTUCKY NO. 9 COAL

Linear Least-Square Fit of Yield vs. Coal Concentration

Dissolver Temperature - 455°C (851°F)   Inlet P_{H_2} - 1900 psig

Nominal Slurry Residence Time - 1 hr.   Lot No. 5 Coal

Yield Wt % MF Coal	Slope	Intercept	Coefficient of Determination
Process Solvent	-0.624	43.33	0.92
Total Distillate	-0.897	65.64	0.98
SRC	1.148	-10.89	0.99
Total Distillation Residue	1.176	2.24	0.99
C_1-C_4 Gases	-0.02	11.41	0.02

Equation form $Y = mx + b$

where Y = yield wt % MF coal

m = slope

x = coal concentration

b = intercept

TABLE 16

EFFECT OF COAL CONCENTRATION ON SRC II YIELDS - KENTUCKY NO. 9 COAL

Inlet P_{H2} - 1900 psig   Lot No. 5 Coal

					Yields, Wt % MF Coal					
Run No.	Nominal Residence Time, Hr.	Temperature °C	Coal Concentration in Feed, Wt %	Hydrogen Feed Wt % Slurry	C_1-C_4	Process Solvent	Total Distillate	SRC Yield	Distillate Residue	Hydrogen Consumption
134R	1.0	465	35	4.7	16.6	23.7	39.6	18.5	33.0	4.5
137R	0.97	455	30	4.6	10.9	23.8	37.4	23.2	36.7	3.8
152R	1.0	455	30	6.3	10.7	25.5	39.9	24.2	38.5	3.3
155R	1.0	445	30	6.6	10.0	22.7	35.1	31.1	44.7	3.1
Avg's at low concentration	0.99	455	31.2	5.6	12.05	23.93	38.0	24.25	38.2	3.68
138R	1.01	455	40	4.6	10.0	16.5	29.5	34.1	48.7	3.6
139R	1.04	↓	45	4.8	11.6	14.4	24.5	41.3	55.5	3.6
147R	0.99		45	4.6	11.6	15.5	25.6	40.0	54.7	--
153R	1.0		45	6.1	8.2	16.0	25.4	41.6	55.6	2.7
154R	1.0		40	6.2	9.6	19.9	30.5	35.0	49.4	3.1
156R	1.0		40	7.8	14.3	20.3	33.8	25.2	40.4	--
157R	1.1		40	4.2	12.7	18.5	34.3	28.9	44.9	4.1
Avg's at high concentration	1.01	455	43.0	5.3	10.2	16.5	27.1	38.4	52.8	3.2

Figure 24

EFFECT OF COAL CONCENTRATION ON SRC II YIELDS
KENTUCKY NO. 9 COAL

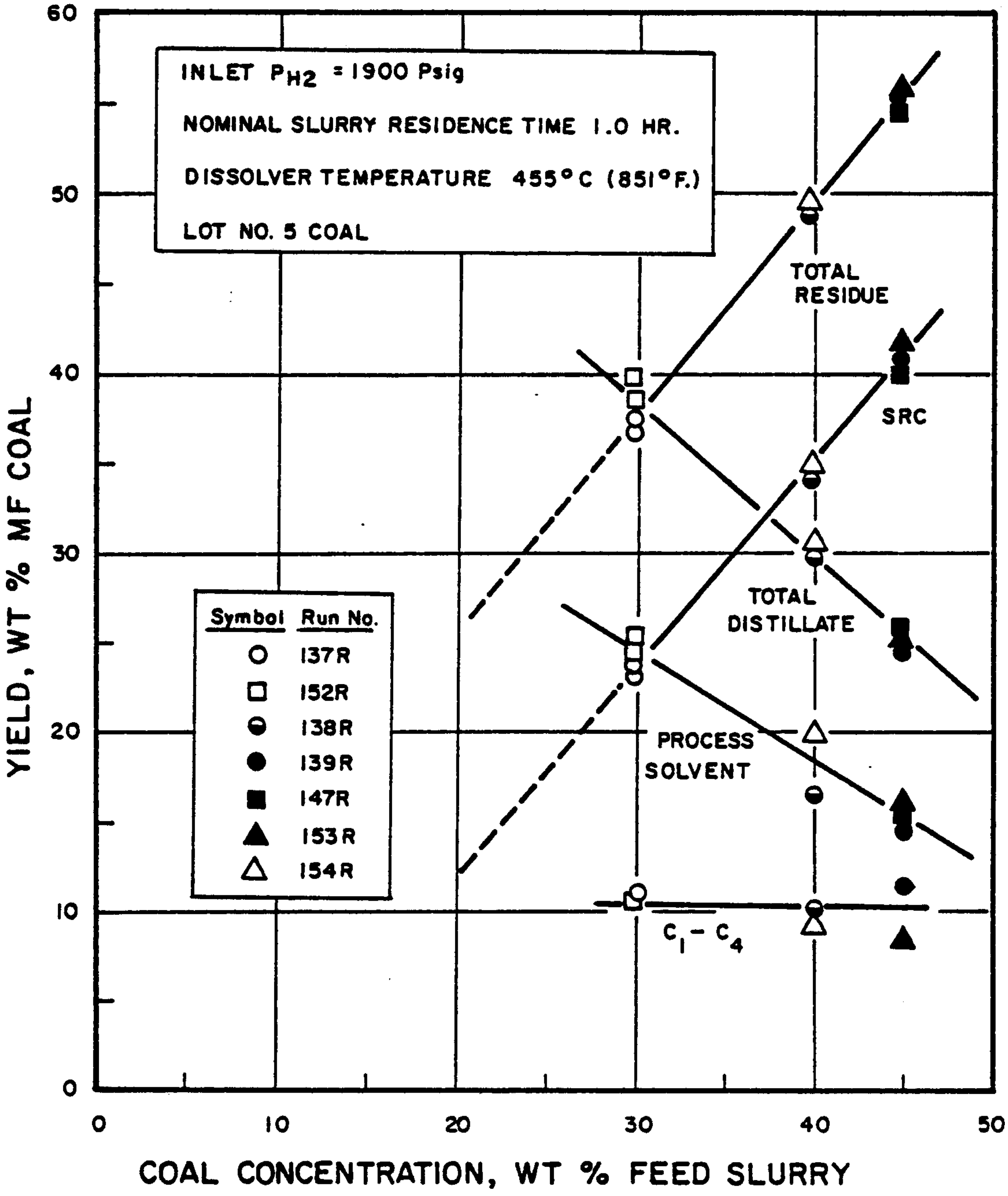
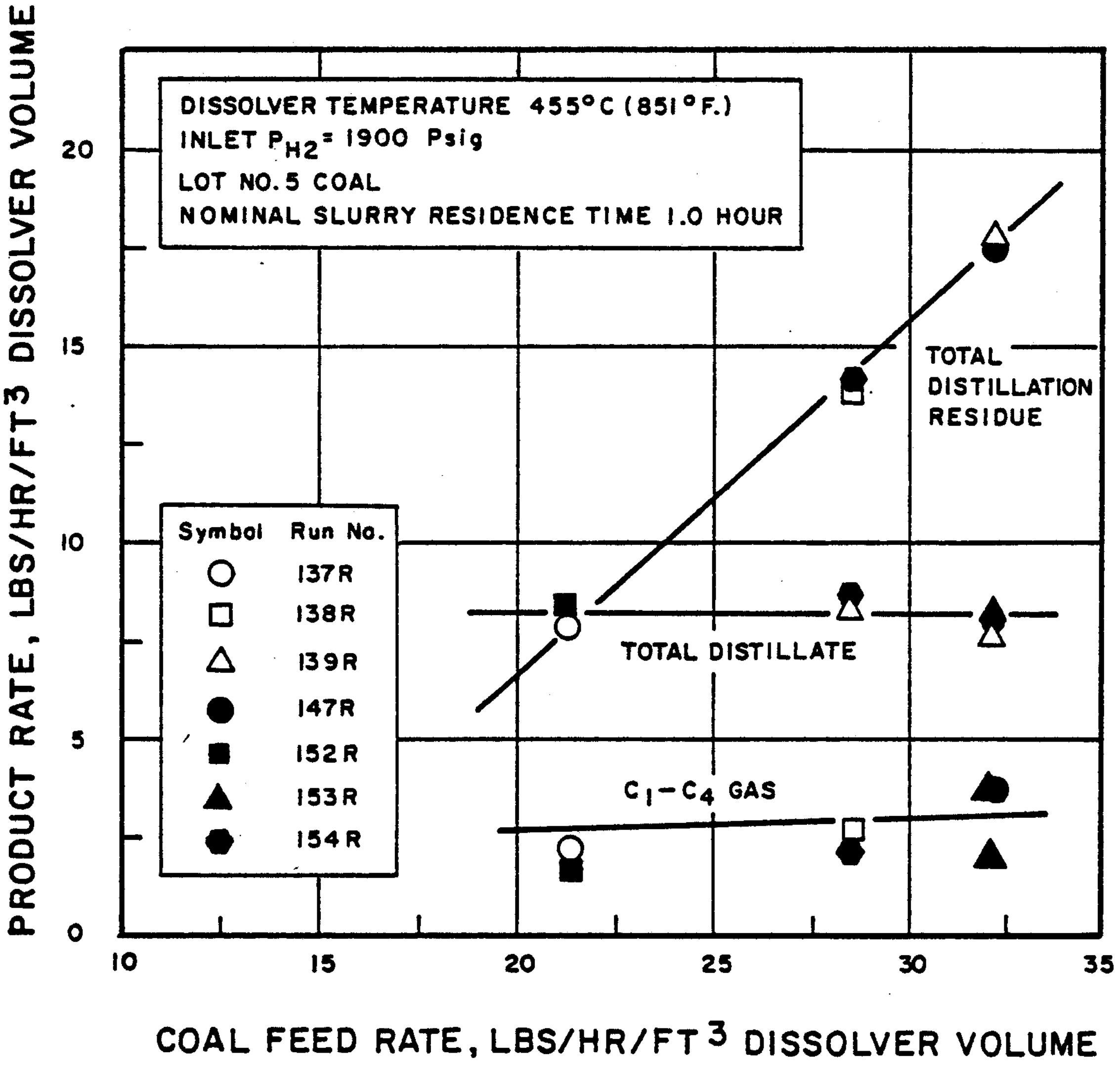


Figure 25

EFFECT OF COAL FEED RATE ON SRC II PRODUCTION RATES
KENTUCKY NO. 9 COAL



Within this range relative production rates of solid and liquid materials can be varied substantially by altering feed composition, but little change in the distillate production rate is possible. This relationship must break down at sufficiently high and low coal feed rates and may break down at differing temperatures, residence times, or pressures.

2. Effect of Dissolver Residence Time

Residence time of the coal slurry in the dissolver has a major effect on yields, and it can be varied over a fairly wide range. Figure 26 illustrates the effect of slurry residence time at a dissolver temperature of 455°C (851°F), 1900 psig inlet partial pressure of hydrogen with lot No. 5 coal and data adjusted for a 40% coal concentration in the feed slurry. The data are plotted as log yield vs residence time and are well fit by straight lines indicating that within the range and precision of the data available here that first order kinetics adequately describe both the disappearance of vacuum residual material and the production of total distillate range material.

Data from certain runs was adjusted to a 40% coal in the feed slurry basis using yield vs coal feed concentration correlations previously determined (see Table 15). Table 17 details the adjustments made. The close grouping of data at equivalent residence times shown in Figure 26 indicates that the linear adjustments made are valid. The substantial values of the correlation coefficients obtained on linear least-squares curve fitting of the logarithms of total distillate product yield and total residual product yield vs residence time (Table 18) reinforce this conclusion.

Table 18 details the fits obtained when linear least-squares fitting was applied directly to the yields and to the logarithms of the yields, both vs residence time as the independent variable. Direct fitting of the yield data produced linear correlations that poorly represented the data as evidenced by both the low values of the correlation coefficient, r^2, and by the fit when plotted against the data. The linear least-squares fit of the logarithm of the total residual yield vs residence time gave an excellent fit, both as measured by the correlation coefficient and as illustrated in Figure 26. A similar fit of the distillate yield data gives a much better fit than the simple linear correlation, although it is not as good as the fit of the residual yield data. This is consistent with the hypothesis that residual materials are produced by a rapid reaction and disappear by a relatively slow reaction while the rates of production and disappearance of distillate materials are of similar magnitude to one another.

Table 19 compares in more detail the average behavior of several runs at 1 hr residence time with a run at 1.6 hr residence time and the same coal concentration in the feed. Light gas, process solvent, and total distillate yields are seen to increase with increasing time while SRC and total distillation residue yields decrease. Hydrogen consumption increases with increasing residence time.

TABLE 17

EFFECT OF RESIDENCE TIME ON SRC II YIELDS - KENTUCKY NO. 9 COAL
ADJUSTMENT OF DATA TO 40% COAL IN FEED

Run No.	Nominal Slurry Residence Time, Hr	Actual Coal Concentration Wt %	Total Residue Yield Wt % Actual	Total Residue Yield Wt % Adj.	Total Distillate Yield Wt % Actual	Total Distillate Yield Wt % Adj.
137R	1.0	30	36.7	48.5	37.4	28.4
152R	1.0	30	38.5	50.3	39.9	30.9
142R	0.7	30	44.6	56.4	34.7	25.7
149R	0.7	20	34.7	58.3	40.1	22.2
158R	1.5	45	33.0	38.9	42.1	37.6

Adjusted using slopes of yield vs coal concentration determined in Table 15.

TABLE 18

EFFECT OF RESIDENCE TIME ON SRC II YIELDS - KENTUCKY NO. 9 COAL

Linear and Exponential Fits of Data
All Yields Adjusted to 40% Coal in Feed

Run No.	Nominal Slurry Residence Time, Hr.		Yield, wt % MF Coal Total Distillate	Yield, wt % MF Coal ln (Total Dist.)	Yield, wt % MF Coal Total Residue	Yield, wt % MF Coal ln (Total Res.)
142R	.7		25.7	3.24	56.4	4.03
147R	.7		22.2	3.10	58.3	4.07
137R	1.0		28.4	3.35	48.5	3.88
152R	1.0		30.9	3.43	50.3	3.92
138R	1.0		29.5	3.38	48.7	3.89
154R	1.0		30.5	3.42	49.4	3.90
141R	1.6		35.4	3.57	41.1	3.72
158R	1.5		37.6	3.63	38.9	3.66
Linear Fit						
$y = mx + b$		m	5.16	0.432	-1.63	-0.409
		b	23.70	2.92	53.69	4.32
		r^2	0.20	0.59	0.23	0.95

TABLE 19

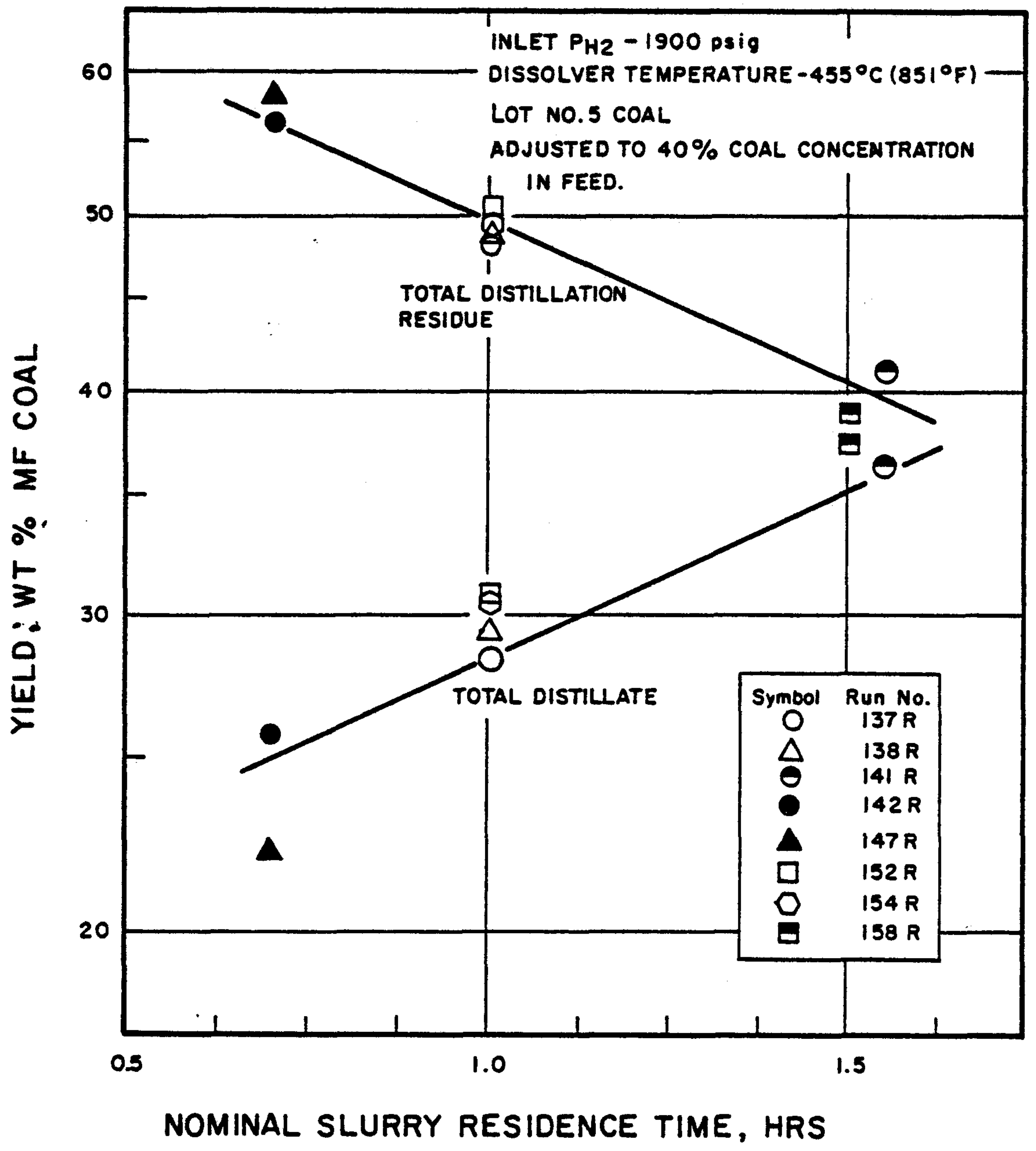
EFFECT OF RESIDENCE TIME ON SRC II YIELDS - KENTUCKY NO. 9 COAL

Inlet P_{H_2} - 1900 psig   Lot No. 5 Coal

Run No.	Nominal Slurry Residence Time, hr.	Dissolver Temp., °C	Coal Concentration in Feed, wt %	Hydrogen Feed wt % slurry	Yield, Wt % MF Coal					
					C_1-C_4 Gas	Process Solvent	Total Distillate	SRC	Distillation Residue	Hydrogen Consumption
141R	1.59	455	40	4.8	13.9	20.9	35.4	26.8	41.1	4.1
138R	1.01	455	40	4.6	10.0	16.5	29.5	34.1	48.7	3.6
139R	1.04	↓	45	4.8	11.6	14.4	24.5	41.3	55.5	3.6
146R	1.03	↓	40	5.1	11.7	18.7	28.7	33.7	49.8	3.4
147R	.99	↓	45	4.6	11.5	15.5	25.6	40.0	54.7	--
154R	1.00	↓	40	6.2	9.6	19.9	30.5	35.0	49.4	3.1
155R	1.00	↓	30	6.6	10.0	22.7	35.1	31.1	44.7	3.1
Avg's for short residence time	1.01	455	40	5.3	10.7	16.3	29.0	35.9	51.5	3.4

Figure 26

EFFECT OF DISSOLVER RESIDENCE TIME ON SRC II YIELDS–KENTUCKY NO.9 COAL



3. Effect of Dissolver Temperature

Dissolver temperature is generally believed to have a major effect on both reaction rates and yields in the SRC II process. However, the temperature range under which the process is operable at the low hydrogen partial pressure utilized is quite narrow and limits any extended exploration of temperature as a process variable.

Figure 27 illustrates the effect of temperature on various yields. All yields shown were adjusted to a constant 40% coal concentration in the feed slurry. Total distillation residue and SRC yields can be seen to be quite sensitive to temperature, decreasing significantly with increasing temperature. Total distillate yield and C_1-C_4 hydrocarbon gas yield increase with increasing temperature, although the effect is less pronounced.

The effect of temperature is also shown in Table 20 where discrete runs at similar coal concentrations and residence times are compared. It is seen that, over the range investigated, increasing temperature generally results in increases in gas and oil yields and decreases in SRC yield. The temperature effect noted in the 2/3 hr residence time runs is of particular interest. Oil and SRC yields for the higher temperature (465°C) run appear more favorable, but the increase in insoluble organic matter (IOM) yield is regarded as being of particular significance. This substantial increase in IOM yield is believed to be an indication of generally unsatisfactory operating conditions; conditions which may be near those which result in coke formation, destruction of catalyst activity, and reactor plugging.

Table 21 further illustrates the effect of temperature on yields by comparing averaged yields from a series of high temperature (465°C) runs with those from a series of lower temperature (455°C) runs. Average residence times, feed coal concentrations, and hydrogen feed rates are similar. These results show that C_1-C_4 gas yield and total distillate yield increases substantially with the increasing temperature and that SRC yield and total distillation residue yield decrease with increasing temperature. Process solvent yield and hydrogen consumption show only minor increases. Table 22 details the adjustments made to the yields shown on Figure 27 to put all the data on the same feed concentration basis. The linear adjustments made are based on the slopes determined from yield vs concentration data described fully in Section VII-F-1. The close grouping of the yield data at the 455°C dissolver temperature where several runs are available indicates this is a valid adjustment. The table also shows an adjustment of the total distillation residue yields to a 30% feed coal concentration. Here also the data group well. Run GU 146R, which appears anomalous on Figure 27 and in this table, also appears anomalous in Table 20 where it is directly compared to a run at 455°C and the same coal concentration and residence time. The source of this behavior is presently unknown, but may be related to a degraded *in situ* catalyst.

Figure 27

EFFECT OF DISSOLVER TEMPERATURE ON SRC II YIELDS-KENTUCKY NO. 9 COAL

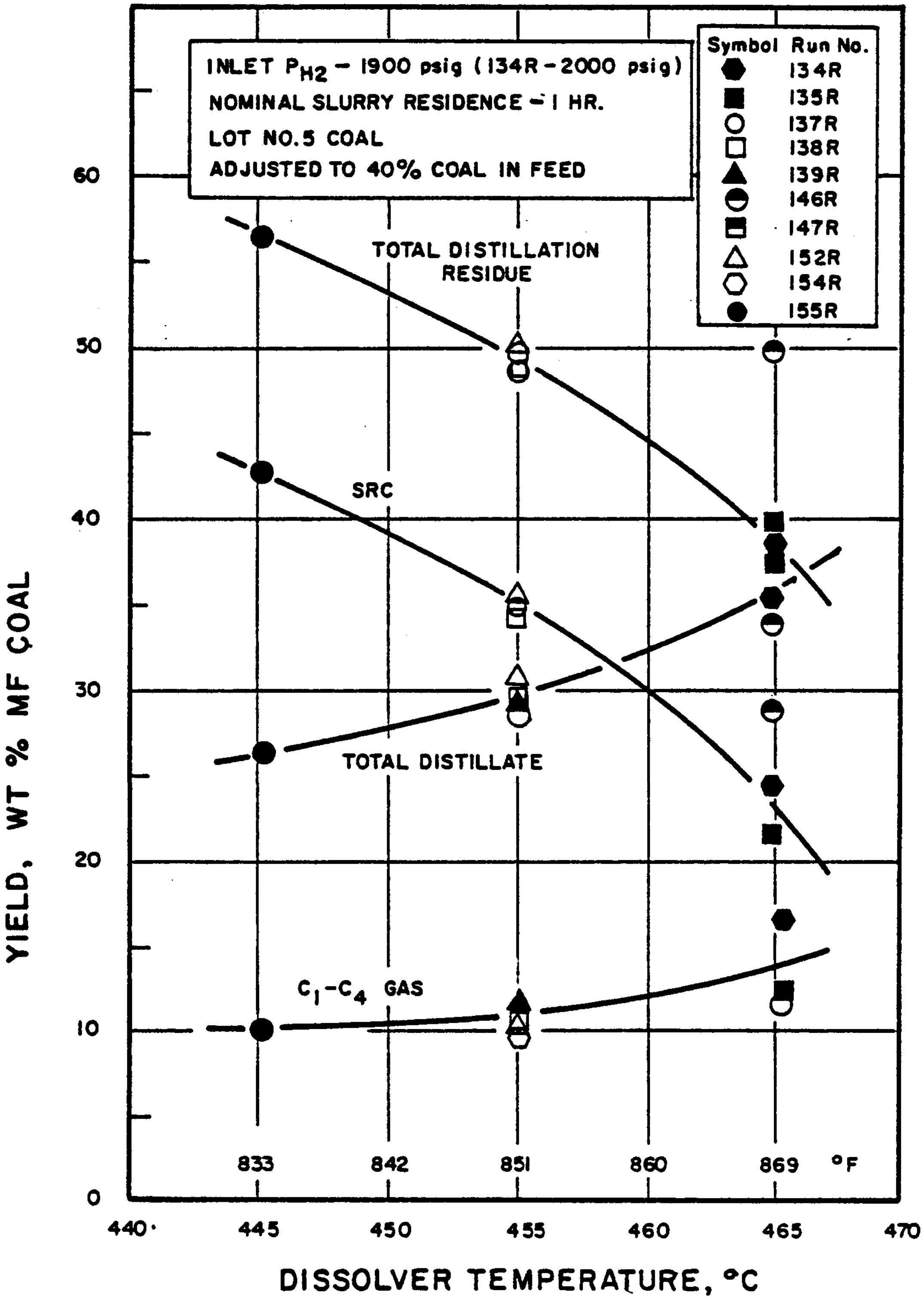


TABLE 20

EFFECT OF TEMPERATURE

A. 30% Coal Concentration, 2/3 hr Residence Time

Run No.	Temp, °C	C_1-C_4 Yield	Total Oil Yield	SRC Yield	IOM[1] Yield
GU 142R	455	12.9	34.7	30.4	4.8
GU 150R	465	15.3	39.0	17.2	7.8
GU 160RA[2]	465	16.9	38.0	20.9	6.3
GU 160RB[2]	465	(16.9)[3]	37.3	21.5	7.2

B. 30% Coal Concentration, 1 hr Residence Time

Run No.	Temp, °C	C_1-C_4 Yield	Total Oil Yield	SRC Yield	IOM[1] Yield
GU 155R	455	10.0	35.1	31.1	4.3
GU 137R	455	10.9	37.4	23.3	4.3
GU 152R	455	10.7	39.9	24.2	4.9

C. 40% Coal Concentration, 1 hr Residence Time

Run No.	Temp, °C	C_1-C_4 Yield	Total Oil Yield	SRC Yield	IOM[1] Yield
GU 138R	455	10.0	29.5	34.1	5.4
GU 146R	465	11.7	28.7	33.7	6.7

[1] Insoluble organic matter

[2] GU 160RA was made at a pressure of 2000 psig and GU 160RB was made at a pressure of 1800 psig. All other runs were made at the normal 1900 psig.

[3] Estimated from GU 160RA

TABLE 21

EFFECT OF DISSOLVER TEMPERATURE ON SRC II YIELDS - KENTUCKY NO. 9 COAL

Inlet P_{H_2} - 1900 psig   Lot No. 5 Coal

Run No.	Nominal Residence Time, hr.	Dissolver Temp, °C	Coal Concentration in Feed wt %	Hydrogen Feed wt % Slurry	Yields, wt % MF Coal					
					C_1-C_4	Process Solvent	Total Distillate	SRC	Distillate Residue	Hydrogen Consumption
134R[1]	1.0	465	35	4.7	16.6	23.7	39.6	18.5	33.0	4.5
146R	1.03	465	40	5.1	11.7	18.7	28.7	33.7	49.8	3.4
150R	.72	465	30	5.2	15.3	22.3	39.0	17.2	34.0	3.9
Avg's at high temperature										
	.92	465	35	5.0	14.5	21.6	35.8	23.1	38.9	3.9
137R	.97	455	30	4.6	10.9	23.8	37.4	23.2	36.7	3.8
138R	1.01	↓	40	4.6	10.0	16.5	29.5	34.1	48.7	3.6
139R	1.04		45	4.8	11.6	14.4	24.5	41.3	55.5	3.6
142R	.69		30	5.0	12.9	23.9	34.7	30.4	49.6	4.8
147R	.99		45	4.6	11.6	15.5	25.6	40.0	54.7	--
151R	.68		30	6.5	10.8	20.3	33.8	25.2	40.4	3.8
152R	.97		30	6.3	10.7	25.5	39.9	24.2	38.5	3.3
Avg's at low temperature										
	.91	455	35.7	5.2	11.2	20.0	32.2	31.2	45.6	3.8

[1] Inlet P_{H_2} - 2000 psig

TABLE 22

EFFECT OF DISSOLVER TEMPERATURE ON SRC II YIELDS - KENTUCKY NO. 9 COAL

Adjustment of Data to Standard Feed Concentration

			Yields, Wt % MF Coal							
	Dissolver	Coal Concen-	C_1-C_4	Total Distillate		SRC		Total Residue		
Run No.	Temp, °C	tration wt %	Actual	Actual	Adj. to 40%	Actual	Adj. to 40%	Actual	Adj. to 40%	Adj. to 30%
137R	455	30	10.9	37.4	28.4	23.2	34.7	36.7	48.5	36.7
138R	455	40	10.0	29.5	29.5	34.1	34.1	48.7	48.7	36.9
146R	465	40	11.7	28.7	28.7	33.7	33.7	49.8	49.8	38.0
152R	455	30	10.7	39.9	30.9	24.2	35.7	38.5	50.3	38.5
155R	445	30	10.0	35.1	26.1	31.1	42.6	44.7	56.5	44.7
154R	455	40	9.6	30.5	30.5	35.0	35.0	49.4	49.4	37.6
147R	455	45	11.6	25.6	30.1	40.0	34.3	54.7	48.8	37.1
139R	455	45	11.6	24.5	29.0	41.3	35.6	55.5	49.6	37.9
135R	465	35	12.2	44.3	39.8	15.9	21.6	31.9	37.8	26.0
134R	465 (2000 psig)	35	16.6	39.6	35.1	18.5	24.2	33.0	38.9	27.1

Adjustments based on the slopes of the linear yield vs coal concentration in the feed correlations. (See Table 15).

4. Effect of Hydrogen Feed Rate

The effect of hydrogen feed rate on yields is shown in Figure 28 and the effect both on yields and product properties is shown in Table 23. Figure 28 shows total distillation residue yield, SRC yield, total distillate yield, and C_1-C_4 gas yield vs hydrogen feed rate over a range of hydrogen feed from 40-80 mscf/ton coal. All runs were at 455°C dissolver temperature, 1 hr nominal slurry residence time, and 1900 psig inlet hydrogen partial pressure with lot No. 5 coal. All yields were adjusted to a 40% coal concentration in the feed basis as previously described and detailed in Table 24. No significant effect of hydrogen feed rate is seen on any of the yields.

Table 23 compares three pairs of runs, each pair of which differs only in the hydrogen feed rate. All runs were at 455°C dissolver temperature, 1900 psig inlet hydrogen partial pressure, 1 hr slurry residence time, with lot No. 5 coal and ranged from 30-45% coal in the feed slurry. The data are consistent with a hypothesis that process solvent, total distillate, SRC, and distillation residue yields all increase very slightly with increasing hydrogen feed rate and that C_1-C_4 gas yield and hydrogen consumption both decrease slightly. The changes are so small, however, that they might equally reasonably be attributed to random error or to a blocking effect between an early series and a later series of experiments.

The elemental analyses of the recycle solvent material and its specific gravity, which are included in Table 23, also show no clear effect of hydrogen feed rate. Only nitrogen content shows a consistent pattern; it decreased slightly with increased hydrogen feed in all cases.

Adjustment of the yield data shown in Figure 28 to a uniform 40% coal concentration is as described previously and is detailed in Table 24.

5. Effect of Hydrogen Partial Pressure

Pressure was not among the process variables included in this SRC II development study. It is generally recognized that, as pressure is increased, desired hydrogenation rates are increased and operability is improved. The objectives of this study included surveying the effects of several other process variables at a processing pressure consistent with equipment constraints of the SRC pilot plant at Ft. Lewis. Essentially all work was carried out at 1900 psig, the upper limit of the dissolver working pressure at the pilot plant.

Some information on pressure effects is available from scoping experiments that preceded this set of experiments and a single short residence time experiment carried out at two pressures at the end of the series. During the exploratory experiments, it was

Figure 28

EFFECT OF HYDROGEN FEED RATE ON SRC II YIELDS KENTUCKY NO. 9 COAL

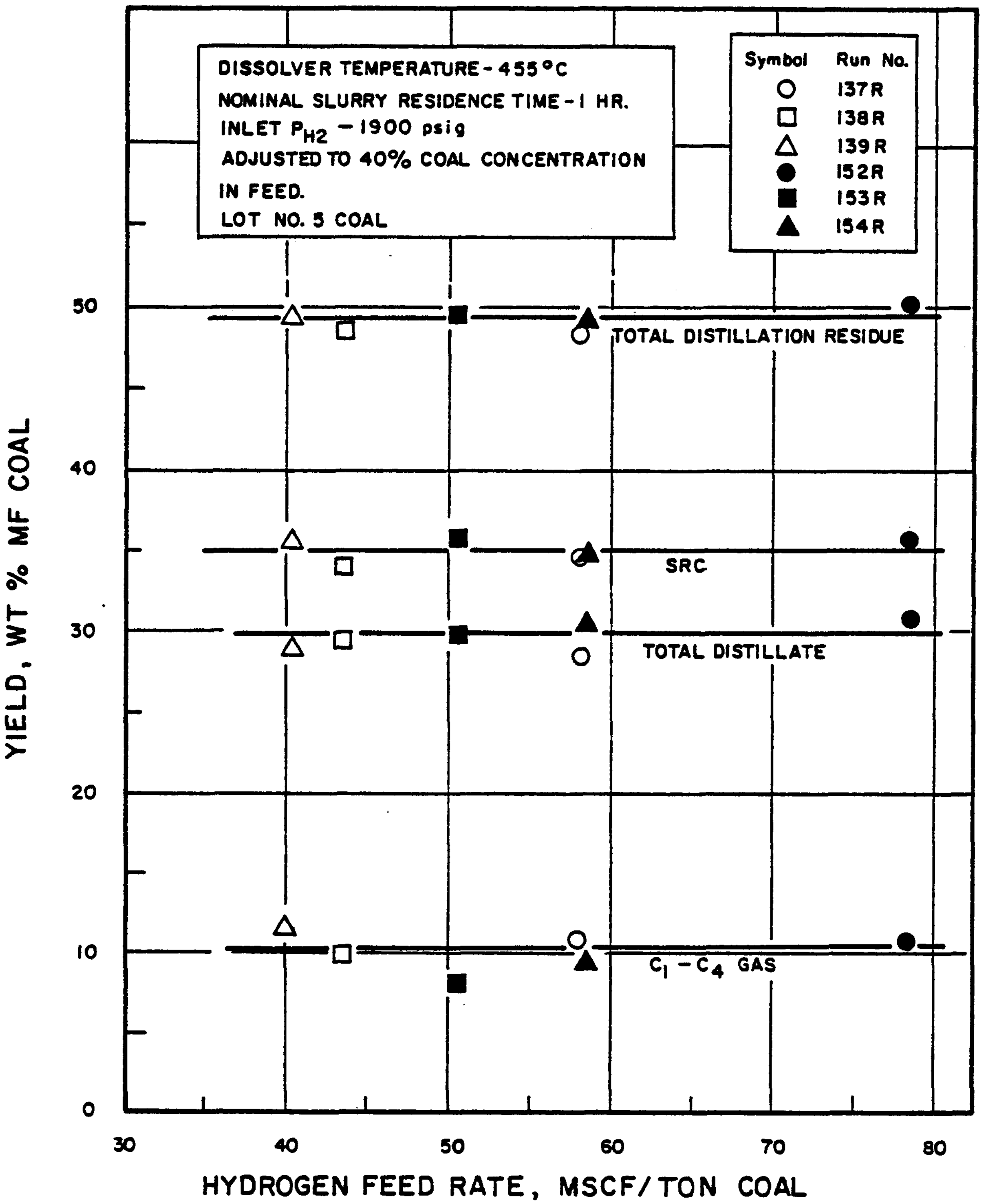


TABLE 23

EFFECT OF HYDROGEN FEED RATE ON SRC II YIELDS - KENTUCKY NO. 9 COAL

Dissolver Temperature - 455°C
Inlet P_{H_2} - 1900 psig

Nominal Slurry Res. Time - 1 hr.
Lot No. 5 Coal

	Hydrogen Feed Rate		Coal Concentration	Yields, Wt % MF Coal						Distillate Fuel Analysis, Wt %					
Run No.	Wt % Slurry	MSCF/ Ton Coal	Wt % Slurry	C_1-C_4 Gas	Solvent	Total Distillate	SRC	Distillation Residue	Hydrogen Consumption	Carbon	Hydrogen	Sulfur	Nitrogen	Oxygen	Specific Gravity
GU 137R	4.63	57.9	30	11.0	23.8	37.4	23.2	36.7	3.8	87.34	8.15	0.27	1.35	2.89	1.043
GU 152R	6.28	78.5	30	10.7	25.5	39.9	24.2	38.5	3.3	87.57	7.88	0.23	1.25	3.12	1.046
GU 138R	4.63	43.4	40	10.0	16.5	29.5	34.1	48.7	3.6	87.40	8.02	0.30	1.26	3.02	1.040
GU 154R	6.23	58.4	40	9.6	19.9	30.5	35.0	49.4	3.1	86.77	8.17	0.29	1.21	3.56	1.034
GU 139R	4.83	40.2	45	11.6	14.4	24.5	41.3	55.5	3.6	87.17	8.14	0.23	1.16	3.30	1.027
GU 153R	6.06	50.5	45	8.2	16.0	25.4	41.6	55.6	2.7	87.21	8.24	0.29	1.11	3.15	1.035

TABLE 24

EFFECT OF HYDROGEN FEED RATE ON SRC II YIELDS - KENTUCKY NO. 9 COAL

Adjustment of Data to 40% Coal Feed Basis

Run No.	Feed Concentration Wt % Coal	Total Distillate		SRC		Total Residual		H_2 Feed	
		Actual	Adj. to 40%	Actual	Adj. to 40%	Actual	Adj. to 40%	Wt % Slurry	MSCF/T Coal
137R	30	37.4	28.4	23.2	34.7	36.7	48.5	4.63	57.9
138R	40	29.5	29.5	34.1	34.1	48.7	48.7	4.63	43.4
139R	45	24.5	29.0	41.3	35.6	55.5	49.6	4.83	40.2
152R	30	39.9	30.9	24.2	35.7	38.5	50.3	6.28	78.5
153R	45	25.4	29.9	41.6	35.9	55.6	49.7	6.06	50.5
154R	40	30.5	30.5	35.0	35.0	49.4	49.4	6.23	58.4

Adjusted to 40% coal in feed slurry on slopes of yield vs coal concentration previously obtained. (See Table 15).

found to be impossible to maintain operation at 1500 psig at 1 hr residence time and 465°C. In order to keep viscosity of the feed slurry manageable, residence time was gradually increased. It was eventually found to be possible to maintain satisfactory operability, but only after the residence time was increased to 1.7 hrs. These conditions resulted in a high conversion to lighter products, but only with excessively high light gas yield. Runs GU 133R and GU 134R were made at inlet hydrogen partial pressures of 1500 and 2000 psig, respectively. They also differed in slurry residence time, that of 133R being 1.7 hrs, while that of 134R was 1 hr. C_1-C_4 gas yields were 24.4% in run 133R but 16.6% in 134R, while recycle solvent yields were 16.5% and 23.7%, respectively.

Run GU 160A and B explored the value of hydrogen pressure in maintaining operability at short slurry residence times. Satisfactory operability was found at 2000 psig hydrogen partial pressure, but slurry viscosity grew at the 1800 lbs psig pressure ultimately forcing a shutdown of the run because of inability to pump the viscous slurry.

Increased hydrogen partial pressure enhances operability in the bench scale unit over the narrow range studied. At hydrogen pressures of 1500 psig substantially greater residence times are required to maintain slurry viscosity adequately low to pump in the small scale equipment. The long residence times give rise to high yields of undesired light gas materials. Greater operating flexibility at low pressures may exist in larger scale equipment that is capable of routinely pumping highly viscous slurries.

6. Effect of Reactor Configuration

As described in section VII-D-1, two dissolver configurations were used in this series of runs. The most commonly used configuration consisted of two 7' x 11/16" ID dissolvers in upflow connected by a small diameter transfer line. The other configuration consisted of a single dissolver of the same dimensions. The use of the differing dissolver configurations allowed the maintenance of more consistent pumping rates and product receiver and feed slurry pot retention times over the span of slurry residence times employed but raised the question of the comparability of data from runs employing the two differing dissolver configurations. Four runs that differed principally only in the dissolver configuration employed were compared to determine if any bias in yields existed between the one dissolver and two dissolver configurations.

Table 25 indicates the differences of the averages of the principal yields in the single dissolver and two dissolver configurations, the standard deviation of the pooled measurements, and the interval estimate on the difference of the means by the Student's t-test. None of the differences in yields or hydrogen consumptions are significant at the 5% level. Only the difference in hydrogen consumption by the gas balance technique becomes significant at a 40% probability level and hydrogen consumptions are suspect because of a gas leak detected during run 145R. Table 26 presents the data,

TABLE 25

Effect of Dissolver Configuration on SRC II Yields
Kentucky No. 9 Coal
Comparison of Averages of Replicates of
Single and Double Reactor Configurations

Yields, wt % MF Coal	Difference of Averages $\overline{Y_1}-\overline{Y_2}$	Standard Deviation of Pooled Measurement S_{pooled}	Interval Estimate at 5% Significance Level
Process Solvent	0.9	1.41	6.06
Total Distillate	2.72	2.04	8.78
SRC	-0.74	.88	3.78
Total Distillate Residue	-0.53	.56	2.41
Hydrogen Reacted (Gas)	1.13	.70	3.01
(Elemental)	.02	.15	0.64

No differences between Single and Double Tube dissolvers are significant at the 5% level

Interval estimate for the difference of the two means

$$(\overline{Y}_1-\overline{Y}_2) \pm t\, \nu\, \alpha/2 \sqrt{\left(\frac{1}{n_1}+\frac{1}{n_2}\right) S^2_{pooled}}$$

TABLE 26

SUMMARY OF REPLICATE SRC II RUNS

All runs at 1900 psig inlet hydrogen pressure, 455°C dissolver temperature
using Lot No. 6, Kentucky No. 9 and 14 coal (P&M Colonial Mine)

	143R	144R	145RB	148R	Single Tube 143R 144R		Two Tube 145RB 148R		Pooled Results		
Reaction Conditions					$\bar{X}$	Sx	$\bar{X}$	Sx	$\bar{X}$	Sx	Sx/$\bar{X}$
Nominal Slurry Residence Time, hr	.97	1.00	.98	1.04							
H_2 Feed, wt % of slurry	4.66	4.86	4.68	4.49							
Reactor Configuration	----1 Tube----		----2 Tube----								
Yields, wt % MF coal											
CH_4	4.11	4.04	3.91								
C_1-C_4	11.96	10.24	10.46								
CO	.40	.31	.28								
CO_2	.72	.88	1.09								
H_2S	1.53	1.25	2.22								
Total Gas	14.61	12.68	14.05	14.05							
Naphtha C_5-193°C	9.75	9.96	8.74	7.91	9.86	.15	8.32	.58	9.09	0.95	.10
Wash Solvent 193-249°C	3.20	3.67	3.59	2.21	3.43	.33	2.90	.98	3.17	0.67	.21
Process Solvent 249-454°C	21.10	23.09	19.94	22.46	22.10	1.41	21.20	1.78	21.65	1.41	.06
Total Distillate C_5-454°C	34.05	36.72	32.27	32.58	35.88	1.89	32.66	1.21	34.02	2.04	.06
Solvent Refined Coal	32.34	31.51	31.80	33.51	31.92	.59	32.66	1.21	32.29	.88	.03
Insoluble Organic Matter	5.59	6.84	6.52	5.97	6.21	.88	6.24	.39	6.23	.56	.09
Ash	9.11	9.30	9.07	8.89	9.20	.13	8.98	.13	9.09	.17	.02
Total Distillation Residue	47.04	47.65	47.39	48.37	47.35	.43	47.88	.69	47.61	.56	.01
Water	8.63	7.88	9.86	8.42	8.25	.53	9.14	1.01	8.70	.84	.10
Total Yield	104.33	104.93	103.57	103.42							
Hydrogen Reacted (gas balance)	4.33	4.93	3.57	3.42	4.63	.42	3.50	.11	4.06	.70	.17
(product analysis)	3.12	2.81	3.03	2.86	2.97	.22	2.95	.12	2.96	.15	.05

their means, standard deviation, pooled means, pooled standard deviations, and relative standard deviations of the pooled results in more detail. As previously mentioned, a leak was discovered in the gas system during Run 145R. On the basis of the known leak and of statistical tests for outliers, Run 145R was excluded from the set of replicate runs considered.

G. Recommendations

The following are recommended based on the work reported here:

1. That the effect of hydrogen feed rate be explored to lower hydrogen feed rates to determine the minimum hydrogen feed rate that can be employed without serious degradation of yields or operability. This information may not be directly transferrable to larger reactors because of differing hydrodynamics; it would, however, provide guidance for the SRC Pilot Plant in seeking a desirable level of hydrogen gas circulation.

2. Similar but not as extensive process variable surveys should be made on other coals intended for SRC II operation at the Ft. Lewis SRC Pilot Plant.

3. At the present time, no major changes should be instituted in the Merriam bench scale unit to attempt to substantially improve the accuracy of hydrogen consumption measurements, but accurate hydrogen consumption measurements should be sought on the larger scale pilot units.

APPENDIX A

SRC PILOT PLANT DATA

TABLE A-1

FILTER TEST RUN OPERATING CONDITIONS

RUN 1A 0000 HRS 11/18/76 to 0000 HRS 11/19/76

CONSTANT CONDITIONS:

BASECOAT: 10 lbs 11-C
PRECOAT: 500 lbs Speedex
FILTER FEED TEMPERATURE: 520°F
FILTER TEMPERATURE: 480-485°F
SLURRY FEED RATE: 23000 lbs/hr
AVERAGE PERCENT PYRIDINE INSOLUBLES IN FILTER FEED: 7.6; 28% SRC
FILTER FEED VISCOSITY: ---

VARIABLE CONDITIONS & RESULTS:

	RUN PERIOD	
	1	2
DURATION, HOURS	8	16
VAT PRESSURE, PSIG	110	110
Δ P, psi	29.5	29.5
DRUM SUBMERGENCE,		
Percent Diameter	29.4	29.4
Percent Area*	36.5	36.5
DRUM SPEED, RPM	1.72	1.89
KNIFE ADVANCE RATE,		
mil/min	2.3	2.2
mil/rev	1.3	1.2
CAKE WASH, LBS/HR	850	840
GAS RATE, LBS/HR	380	380-560
FILTRATE RATE, **LBS/HR	4600	5200
Percent P.I.	Trace	Trace
Percent Ash	---	0.04
Viscosity @ 100°, cSt	---	---

* Total Screen Area = 41.6 ft^2
**Includes Cake Wash

RUN OBJECTIVES & PROCEDURE: Maintain operating conditions throughout filtration run at the base conditions. Attempt to precoat the filter at high drum speed, low submergence, 5% filter aid in the precoat slurry, and rapid rise to the desired operating Δ P. Analyze effects of duration of run on the filtration rate.

COMMENTS: Inadequate precoat instrumentation resulted in the lack of appropriate control during precoating. As a result, the precoat application did not follow the requested procedure. Run conditions were held fairly constant near the pre-established "base conditions". Filtration rates of approximately 120 lbs filtrate per hour per square foot of total screen area (41.6 ft^2) were observed using a feedstock consisting of undiluted unfiltered coal solution.

TABLE A-2

FILTER TEST RUN OPERATING CONDITIONS

RUN 1B 1100 HRS 11/20/76 - 1300 HRS 11/21/76

CONSTANT CONDITIONS:

BASECOAT: 10 lbs 11-C
PRECOAT: 500 lbs Speedex
FILTER FEED TEMPERATURE: 520°F
FILTER TEMPERATURE: 485-490°F
SLURRY FEED RATE: 25000-27000 lbs/hr
AVERAGE PERCENT PYRIDINE INSOLUBLES IN FILTER FEED: 6.9; 21% SRC
FILTER FEED VISCOSITY: ---

VARIABLE CONDITIONS & RESULTS:

	RUN PERIOD 1
DURATION, HOURS	26
VAT PRESSURE, PSIG	116
Δ P, psi	29.8
DRUM SUBMERGENCE,	
Percent Diameter	29.6
Percent Area*	36.6
DRUM SPEED, RPM	1.92
KNIFE ADVANCE RATE,	
mil/min	2.0
mil/rev	1.0
CAKE WASH, LBS/HR	865
GAS RATE, LBS/HR	423-706
FILTRATE RATE, **LBS/HR	5000-6000
Percent P.I.	0.002
Percent Ash	---
Viscosity @ 100°, cSt	---

* Total Screen Area = 41.6 ft^2
**Includes Cake Wash

RUN OBJECTIVES & PROCEDURE: Precoat using standard P&M basecoating and precoating procedures. Hold operating conditions constant throughout the run, and at the base conditions. Observe effects of run time on the filtration rate. Compare average filtration rate with Run 1A.

COMMENTS: A low concentration (0.002%) of pyridine insoluble material was detected in the filtrate, suggesting a possible solids breakthrough in the filter. During this run, it was noted that both the gas flowrate through the filter and the filtrate rate increased as the run progressed. This suggested a gradual decrease in the precoat or interface resistance as the precoat thickness decreased. Further study of this phenomenon is planned for future runs. Filtration rates during this run were higher than in Run 1A, evidently due to the different precoat application technique.

TABLE A-3

FILTER TEST RUN OPERATING CONDITIONS

RUN 2A 2100 HRS 11/21/76 - 0800 HRS 11/23/76

CONSTANT CONDITIONS:

BASECOAT: 10 lbs 11-C
PRECOAT: 500 lbs Speedex
FILTER FEED TEMPERATURE: 520°F
FILTER TEMPERATURE: 490°F
SLURRY FEED RATE: 26000 lbs/hr
AVERAGE PERCENT PYRIDINE INSOLUBLES IN FILTER FEED: 5.8; 18% SRC
FILTER FEED VISCOSITY: ---

VARIABLE CONDITIONS & RESULTS:

	RUN PERIOD 1	2	3	4
DURATION, HOURS	3	7	7	18
VAT PRESSURE, PSIG	116	116	116	116
Δ P, psi	29.7	29.7	29.7	29.7
DRUM SUBMERGENCE,				
Percent Diameter	29.6	29.6	29.6	29.6
Percent Area*	36.6	36.6	36.6	36.6
DRUM SPEED, RPM	1.92	1.92	1.92	1.92
KNIFE ADVANCE RATE,				
mil/min	0.8	2.0	1.2	1.4
mil/rev	0.4	1.0	0.6	0.7
CAKE WASH, LBS/HR	880	830	850	920
GAS RATE, LBS/HR	420	540	580	600-930
FILTRATE RATE, **LBS/HR	3500	4900	4200-5000	5000-6400
Percent P.I.	Trace	Trace	.03	.05
Percent Ash	---	---	---	---
Viscosity @ 100°, cSt	---	---	137	---

* Total Screen Area = 41.6 ft^2
**Includes Cake Wash

RUN OBJECTIVES & PROCEDURE: Analyze the effects of knife advance rate on filtration.

COMMENTS: Noted detectable concentration of solids in the filtrate during much of the run. Observed a very strong effect of knife advance rate on filtrate rate. Initial filtration rates were comparable to those observed at the initial stages (first six hours) of Run 1B. This run will be repeated to minimize fluctuation in operating conditions.

TABLE A-4

FILTER TEST RUN OPERATING CONDITIONS

RUN 2B 1000 hrs 11/25/76 to 0100 11/27/76

CONSTANT CONDITIONS:

BASECOAT: 10 lbs 11-C
PRECOAT: 500 lbs Speedex
FILTER FEED TEMPERATURE: 520-535°F
FILTER TEMPERATURE: 420-495°F
SLURRY FEED RATE: 26000 lbs/hr
AVERAGE PERCENT PYRIDINE INSOLUBLES IN FILTER FEED: 5.3; 21% SRC
FILTER FEED VISCOSITY: ---

VARIABLE CONDITIONS & RESULTS:

	RUN PERIOD		
	1	2	3
DURATION, HOURS	8	8	13
VAT PRESSURE, PSIG	116	116	116
Δ P, psi	29.8	29.8	29.8
DRUM SUBMERGENCE,			
Percent Diameter	29.4	29.4	29.4
Percent Area*	36.5	36.5	36.5
DRUM SPEED, RPM	1.74	1.74	1.74
KNIFE ADVANCE RATE,			
mil/min	1.9	2.0	1.0
mil/rev	1.1	1.1	0.6
CAKE WASH, LBS/HR	860	860	860
GAS RATE, LBS/HR	650	710	720-800
FILTRATE RATE, **LBS/HR	5390	5840	5310
Percent P.I.	Trace	Trace	Trace
Percent Ash	---	0.03	0.03
Viscosity @ 100°, cSt	---	---	---

* Total Screen Area = 41.6 ft^2
**Includes Cake Wash

RUN OBJECTIVES & PROCEDURE: Duplicate Run 2A. Observe effects of various knife advance rates on filtration.

COMMENTS: Filter temperature declined toward the end of this run. A ten hour plant shutdown occurred in the early stages of this run, but the precoat did not appear to be affected much by the shutdown. A strong knife advance effect on filtration rate is apparent from the data obtained in this run.

TABLE A-5

FILTER TEST RUN OPERATING CONDITIONS

RUN 3 1300 HRS 11/23/76 to 300 HRS 11/25/76

CONSTANT CONDITIONS:

BASECOAT: 10 lbs 11-C
PRECOAT: 500 lbs Speedex
FILTER FEED TEMPERATURE: 520°F
FILTER TEMPERATURE: 480-490°F
SLURRY FEED RATE: 26000 lbs/hr
AVERAGE PERCENT PYRIDINE INSOLUBLES IN FILTER FEED: 6.17; 22% SRC
FILTER FEED VISCOSITY: ---

VARIABLE CONDITIONS & RESULTS:

	RUN PERIOD			
	1	2	3	4
DURATION, HOURS	11	10	10	7
VAT PRESSURE, PSIG	116	116	116	116
Δ P, psi	29.9	29.9	29.9	29.9
DRUM SUBMERGENCE,				
Percent Diameter	29.2	10.5	20.2	29.5
Percent Area*	36.4	21.0	29.6	36.6
DRUM SPEED, RPM	1.75	1.75	1.75	1.75
KNIFE ADVANCE RATE,				
mil/min	2.0	1.2	1.2	1.2
mil/rev	1.1	0.7	0.7	0.7
CAKE WASH, LBS/HR	860	850	860	850
GAS RATE, LBS/HR	630	760	720-790	740-760
FILTRATE RATE, **LBS/HR	5930	4195	5140	5615
Percent P.I.	Trace	Trace	Trace	Trace
Percent Ash	0.02	---	---	---
Viscosity @ 100°, cSt	120	---	---	---

* Total Screen Area = 41.6 ft^2
**Includes Cake Wash

RUN OBJECTIVES & PROCEDURE: Observe the effects of drum submergence on filtration rate.

COMMENTS: The filtrate rate responded markedly to changes in drum submergence, varying directly with changes in submergence. The gas rate seemed to increase throughout the run, but was observed to decrease when submergence increased.

TABLE A-6

FILTER TEST RUN OPERATING CONDITIONS

RUN 4 1300 HRS 12/9/76 to 0000 HRS 12/11/76

CONSTANT CONDITIONS:

BASECOAT: 10 lbs 11-C
PRECOAT: 500 lbs Speedex
FILTER FEED TEMPERATURE: 520°F
FILTER TEMPERATURE: 495°F
SLURRY FEED RATE: 25500 lbs/hr
AVERAGE PERCENT PYRIDINE INSOLUBLES IN FILTER FEED: 7.23; 20% SRC
FILTER FEED VISCOSITY: ---

VARIABLE CONDITIONS & RESULTS:

	RUN PERIOD							
	1	2	3	4	5	6	7	8
DURATION, HOURS	11	10	2	3	2	2	2	2
VAT PRESSURE, PSIG	110	110	110	110	110	110	110	110
Δ P, psi	29.9	29.9	29.9	29.9	29.9	29.9	29.9	29.9
DRUM SUBMERGENCE,								
Percent Diameter	29.6	29.6	29.6	29.6	29.6	29.6	29.6	29.6
Percent Area*	36.6	36.6	36.6	36.6	36.6	37.6	36.6	36.6
DRUM SPEED, RPM	1.74	1.74	1.10	1.02	1.02	1.82	2.85	2.85
KNIFE ADVANCE RATE,								
mil/min	1.83	1.00	1.05	1.4	2.8	3.0	2.6	1.43
mil/rev	1.05	0.57	0.95	1.4	2.8	1.65	0.9	0.5
CAKE WASH, LBS/HR	880	860	860	860	850	850	850	850
GAS RATE, LBS/HR	500	500	620	700	1000	900	950	820
FILTRATE RATE, **LBS/HR	6070	5720	5590	5700[a]	6740	7870	8970	7950
Percent P.I.	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace
Percent Ash	---	---	---	---	---	---	---	---
Viscosity @ 100°, cSt	---	125	---	---	---	---	---	---

* Total Screen Area = 41.6 ft^2
**Includes Cake Wash
a. Flow did not level off this run. Value shown is a rough estimate.

RUN OBJECTIVES & PROCEDURE: Installed new knife before this run. This run was designed to distinguish the effects of drum speed on filtrate rate. In addition the interactive effect of knife advance rate will also be measured, in an attempt to define solids penetration into the precoat at different drum speeds. To accomplish this the time required to line out the filter after a change in operating conditions will be studied.

COMMENTS: During this run the flow from the filtrate receiver was set to hold a constant level in the receiver. This resulted in the capability of observing sudden changes in filtrate rate. Using this technique it was possible to establish eight combinations of knife advance rate and drum speed. Comparable knife advance rates (in mil/rev) at each drum speed were chosen for data analysis. Observation of the instantaneous filtration rates at each condition indicated that filtration rates lined out rapidly (within 15 minutes). The data obtained in this run indicate that both high knife advance rates and high drum speeds aid filtration. The effect of increasing knife advance rate did not seem to change at different levels of drum speed.

TABLE A-7

FILTER TEST RUN OPERATING CONDITIONS

RUN 5 0900 HRS 12/11/76 to 1800 HRS 12/12/76

CONSTANT CONDITIONS:

BASECOAT: 10 lbs 11-C
PRECOAT: 500 lbs Speedex
FILTER FEED TEMPERATURE: 525°F
FILTER TEMPERATURE: 495°F
SLURRY FEED RATE: 14000-20000 lbs/hr
AVERAGE PERCENT PYRIDINE INSOLUBLES IN FILTER FEED: 6.27; 28% SRC
FILTER FEED VISCOSITY: ---

VARIABLE CONDITIONS & RESULTS:

	RUN PERIOD						
	1	2	3	4	5	6	7
DURATION, HOURS	10	2	3	3	3	2	3
VAT PRESSURE, PSIG	110	110	110	120	120	120	129
Δ P, psi	29.6	29.6	29.6	39.8	39.8	39.8	49
DRUM SUBMERGENCE,							
Percent Diameter	29.6	29.6	29.6	29.6	29.6	29.6	29.6
Percent Area*	36.6	36.6	36.6	36.6	36.6	36.6	36.6
DRUM SPEED, RPM	1.74	1.74	1.74	1.74	1.74	1.74	1.74
KNIFE ADVANCE RATE,							
mil/min	1.9	4.2	1.2	1.2	1.7	2.6	2.4
mil/rev	1.1	2.4	0.7	0.7	1.0	1.5	1.4
CAKE WASH, LBS/HR	870	860	850	860	850	860	850
GAS RATE, LBS/HR	575	800	700	920	1060	>1100	>1100
FILTRATE RATE, **LBS/HR	6340	7720	5870	6170	7280	8060	7640
Percent P.I.	Trace	Trace	Trace	Trace	Trace	Trace	Trace
Percent Ash	---	---	---	---	---	---	---
Viscosity @ 100°, cSt	---	---	---	---	---	---	---

* Total Screen Area = 41.6 ft^2
**Includes Cake Wash

RUN OBJECTIVES & PROCEDURE: Observe effects of pressure drop and the interaction of knife advance rates on filtration rates.

COMMENTS: Using the same flow control scheme from the filtrate receiver as in Run 4, it was possible to try several combinations of knife advance rate and pressure drop. The data indicate that at higher levels of pressure drop in the filter, increasing the knife advance rates results in a greater increase in filtrate rate than at lower levels of pressure drop. A seven hour shutdown during the early stages of this run did not seem to affect precoat properties. This was probably due to the fact that the pressure differential, drum submergence, and gas rate were all maintained during the shutdown.

TABLE A-8

FILTER TEST RUN OPERATING CONDITIONS

RUN 6 0500 HRS 12/16/76 to 0900 HRS 12/17/76

CONSTANT CONDITIONS:

BASECOAT: 10 lbs 11-C
PRECOAT: 500 lbs Speedex
FILTER FEED TEMPERATURE: 525°F
FILTER TEMPERATURE: 490°F
SLURRY FEED RATE: 17000 lbs/hr
AVERAGE PERCENT PYRIDINE INSOLUBLES IN FILTER FEED: 7.44; 30% SRC
FILTER FEED VISCOSITY: 19.7 cSt @ 210°F

VARIABLE CONDITIONS & RESULTS:

	RUN PERIOD			
	1	2	3	4
DURATION, HOURS	8	8	7	4
VAT PRESSURE, PSIG	111	111	111	111
Δ P, psi	30.5	30.5	30.5	30.5
DRUM SUBMERGENCE,				
Percent Diameter	29.6	29.6	29.6	29.6
Percent Area*	36.6	36.6	36.6	36.6
DRUM SPEED, RPM	1.73	1.73	1.73	1.73
KNIFE ADVANCE RATE,				
mil/min	1.8	1.2	1.8	2.5
mil/rev	1.0	0.7	1.0	1.4
CAKE WASH, LBS/HR	870	950	940	900
GAS RATE, LBS/HR	530	580	1115	>1100
FILTRATE RATE, **LBS/HR	5900	5500	6800	5000
Percent P.I.	Trace	Trace	Trace	Trace
Percent Ash	0.03	0.03	0.03	0.03
Viscosity @ 100°, cSt	317	280	117	983

* Total Screen Area = 41.6 ft^2
**Includes Cake Wash

RUN OBJECTIVES & PROCEDURE: Duplicate Runs 2A and 2B. Varied knife advance rate from 1.2-2.5 mil/min.

COMMENTS: Changes in knife advance rate did not result in any clear trend in changes in filtrate rate. There was a very large increase in gas rate toward the end of the run. Level control in the filtrate receiver was poor duing this run, probably causing the apparently high filtrate rates at the low knife advance rates. The wash solvent rate fluctuated considerably during this run. Overall, with the exception of the initial filtration rate, the data from this run is considered to be questionable.

TABLE A-9

FILTER TEST RUN OPERATING CONDITIONS

RUN 7 1400 HRS 12/17/76 to 2200 HRS 12/18/76

CONSTANT CONDITIONS:

BASECOAT: 10 lbs 11-C
PRECOAT: 500 lbs Speedex
FILTER FEED TEMPERATURE: 525°F
FILTER TEMPERATURE: 495°F
SLURRY FEED RATE: 17000-25000 lbs/hr
AVERAGE PERCENT PYRIDINE INSOLUBLES IN FILTER FEED: 7.43; 20% SRC
FILTER FEED VISCOSITY: 16.47 cSt @ 210°F

VARIABLE CONDITIONS & RESULTS:

	RUN PERIOD							
	1	2	3	4	5	6	7	8
DURATION, HOURS	4	4	3	3	3	4	4	7
VAT PRESSURE, PSIG	110	110	110	110	110	110	110	110
Δ P, psi	29.6	29.6	29.6	29.6	29.6	29.5	29.5	29.3
DRUM SUBMERGENCE,								
Percent Diameter	29.8	31.7	29.4	29.4	29.4	29.6	29.6	29.6
Percent Area*	36.8	38.1	36.5	36.5	36.5	36.6	36.6	36.6
DRUM SPEED, RPM	1.75	1.76	1.76	1.76	1.76	1.76	1.76	1.76
KNIFE ADVANCE RATE,								
mil/min	2.0	0.9	0.9	0.9	1.0	1.0	2.0	2.7
mil/rev	1.1	0.5	0.5	0.5	0.6	0.6	1.1	1.5
CAKE WASH, LBS/HR	870	870	650	1080	1290	860	860	860
GAS RATE, LBS/HR	510	490	485	475	465	470	490	1030
FILTRATE RATE, **LBS/HR	5700	5400	5800	6000	6300	6200	7300	8600
Percent P.I.	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace
Percent Ash	---	---	---	---	---	0.05	---	0.02
Viscosity @ 100°, cSt	---	---	---	---	---	251	---	245

* Total Screen Area = 41.6 ft^2
**Includes Cake Wash

RUN OBJECTIVES & PROCEDURE: Alternate precoat procedure from Run 1A to be used this run. Effect of knife advance rate will also be studied. Cake wash rate will be varied between 850 and 1100 lbs/hr to determine its effect.

COMMENTS: The effect of changing the wash solvent spray rate was studied in this run. The test showed that the effect of wash solvent spray in the range studied was insignificant. The effect of high knife advance rate was also investigated, and the results show that the filtrate rate continued to increase at knife advance rates up to 3 mil/min.

TABLE A-10

FILTER TEST RUN OPERATING CONDITIONS

RUN 8 0700 HRS 1/13/77 to 0800 HRS 1/14/77

CONSTANT CONDITIONS:

BASECOAT: 10 lbs 11-C
PRECOAT: 500 lbs Speedex
FILTER FEED TEMPERATURE: 500°F
FILTER TEMPERATURE: 470-475°F
SLURRY FEED RATE: 23000 lbs/hr
AVERAGE PERCENT PYRIDINE INSOLUBLES IN FILTER FEED: 7.34; 23% SRC
FILTER FEED VISCOSITY: 31.3 @ 210°F

VARIABLE CONDITIONS & RESULTS:

	RUN PERIOD		
	1	2	3
DURATION, HOURS	13	4	8
VAT PRESSURE, PSIG	111	111	111
Δ P, psi	30.2	30.2	29.4
DRUM SUBMERGENCE,			
Percent Diameter	29.6	25.0	29.6
Percent Area*	36.6	33.3	36.6
DRUM SPEED, RPM	1.73	1.73	1.73
KNIFE ADVANCE RATE,			
mil/min	1.8	1.9	1.7
mil/rev	1.0	1.1	1.0
CAKE WASH, LBS/HR	830	835	830
GAS RATE, LBS/HR	310	370	550
FILTRATE RATE, **LBS/HR	6550	5730	6510
Percent P.I.	Trace	0.01	Trace
Percent Ash	0.04	---	0.08
Viscosity @ 100°, cSt	829	---	---

* Total Screen Area = 41.6 ft^2
**Includes Cake Wash

RUN OBJECTIVES & PROCEDURE: Observe effects of run duration on filtrate rate by holding all conditions constant.

COMMENTS: This run was conducted to determine the effect of run duration on the filtrate rate. Conditions were to be held constant throughout the run. The data show that the initial and final filtrate rates were not significantly different. (The final rate shown is for the period up to 3 hours before run termination.) The conclusion from this run is that run duration may not have a strong effect on filtrate rates. However, the gas flow data indicate that the gas rate during this run was lower than for comparable runs with Speedex precoat. This might have been due to a more tightly packed precoat during this run than for previous runs. It was noted that even though the initial gas rate was lower than in previous runs, the gas rate increased during the later half of the run, a phenomenon also observed in all other runs.

TABLE A-11

FILTER TEST RUN OPERATING CONDITIONS

RUN 9 1200 HRS 1/24/77 to 1800 HRS 1/25/77

CONSTANT CONDITIONS:

BASECOAT: 10 lbs 11-C
PRECOAT: 500 lbs Speedplus
FILTER FEED TEMPERATURE: 513°F
FILTER TEMPERATURE: 475-485°F
SLURRY FEED RATE: 26000 lbs/hr
AVERAGE PERCENT PYRIDINE INSOLUBLES IN FILTER FEED: 6.99; 24% SRC
FILTER FEED VISCOSITY: 19.8 @ 210°F

VARIABLE CONDITIONS & RESULTS:

	RUN PERIOD					
	1	2	3	4	5	6
DURATION, HOURS	4	8	2	11	2	3
VAT PRESSURE, PSIG	111	111	111	111	111	111
Δ P, psi	30.4	30.4	30.4	30.4	30.4	30.2
DRUM SUBMERGENCE,						
Percent Diameter	21.8	29.4	29.4	28.1	28.1	18.8
Percent Area*	30.9	36.5	36.5	35.6	35.6	28.5
DRUM SPEED, RPM	1.72	1.73	1.73	1.73	1.73	1.73
KNIFE ADVANCE RATE,						
mil/min	2.7	1.1	1.1	1.1	2.0	2.0
mil/rev	1.6	0.6	0.6	0.6	1.2	1.2
CAKE WASH, LBS/HR	720	900	1160	870	870	870
GAS RATE, LBS/HR	---	340	340	---	---	---
FILTRATE RATE, **LBS/HR	7500	5800	6600	5100	7700	6400
Percent P.I.	Trace	Trace	Trace	Trace	Trace	Trace
Percent Ash	---	0.04	---	0.03	---	---
Viscosity @ 100°, cSt	---	105	---	175	---	---

* Total Screen Area = 41.6 ft^2
**Includes Cake Wash

RUN OBJECTIVES & PROCEDURE: Test effectiveness of Speedplus filter aid as a precoat material. Test effect of knife advance rate on filtration with Speedplus filter aid.

COMMENTS: This initial Speedplus precoat run was hampered by problems in other areas of the plant. As a result, it was necessary to cut the filtrate rate. This was done by decreasing the knife advance rate to 1 mil/min. Comparable data with Speedex precoats for long operating periods at low knife advance rates are not currently available. However, it should be noted that the filtration rates observed with a Speedplus precoat at a knife advance rate of 1 mil/min were comparable to those observed with Speedex precoats at 2 mil/min knife advance rates. Further analysis of precoat grades is warranted, based on the results of this run.

TABLE A-12

FILTER TEST RUN OPERATING CONDITIONS

RUN 10 1300 HRS 1/26/77 to 0800 HRS 1/27/77

CONSTANT CONDITIONS:

BASECOAT: 10 lbs 11-C
PRECOAT: 500 lbs Speedplus
FILTER FEED TEMPERATURE: 513°F
FILTER TEMPERATURE: 485°F
SLURRY FEED RATE: 27000 lbs/hr
AVERAGE PERCENT PYRIDINE INSOLUBLES IN FILTER FEED: 5.8
FILTER FEED VISCOSITY: 26.7 @ 210°F

VARIABLE CONDITIONS & RESULTS:

	RUN PERIOD 1	2	3
DURATION, HOURS	2	3	15
VAT PRESSURE, PSIG	113	113	111
Δ P, psi	32.4	32.3	31.1
DRUM SUBMERGENCE,			
Percent Diameter	10.7	3.2	29.2
Percent Area*	21.1	11.4	36.4
DRUM SPEED, RPM	1.74	1.74	1.74
KNIFE ADVANCE RATE,			
mil/min	5.7	4.3	2.0
mil/rev	3.3	2.5	1.2
CAKE WASH, LBS/HR	920	930	870
GAS RATE, LBS/HR	420	580	350-580
FILTRATE RATE, **LBS/HR	6220	4211	6700
Percent P.I.	Trace	Trace	Trace
Percent Ash	---	---	0.04
Viscosity @ 100°, cSt	---	---	238

* Total Screen Area = 41.6 ft^2
**Includes Cake Wash

RUN OBJECTIVES & PROCEDURE: Duplicate of Run 9. Test effectiveness of Speedplus Filter aid.

COMMENTS: The precoat application in this run resulted in a grooved and uneven precoat. As a result, 1½ inches of precoat were removed prior to beginning the run. This resulted in the run being of rather short duration. The data obtained during lined out operation indicate that the filtration rates with Speedplus filter aid are comparable to those obtained with the most favorable Speedex precoats. The gas flow through the filter was noted to increase at the end of the run, as had been the case in the Speedex precoat runs.

APPENDIX B

MERRIAM LABORATORY DATA

TABLE B-1

SRC II PROCESSING OF KENTUCKY NOS. 9 & 14 COAL
SUMMARY OF PROCESS CONDITIONS, YIELDS AND PRODUCT ANALYSES

	GU 133R	GU 134R	GU 135R	GU 136R	GU 137R	GU 138R
Reaction Conditions						
Coal (Kentucky Nos. 9 & 14)	Lot 5	Lot 5	Lot 4	Lot 4	Lot 5	Lot 5
Nominal Liquid Residence Time, hr	1.7	1.0	1.0	1.0	1.0	1.0
Nominal Dissolver Temperature, °C	465	465	465	455	455	455
Reactor Pressure, psig	1500	2000	2000	1900	1900	1900
H_2 Feed, wt % based on slurry	6.6	4.7	4.9	5.1	4.6	4.6
Slurry Composition, wt %						
Coal	35.0	35.0	35.0	30.0	30.0	40.0
Recycle Coal Solution	62.0	65.0	65.0	70.0	70.0	51.0
Distillate Recycle Solvent	3.0	--	--	--	--	9.0
Yields, wt % of feed coal						
C_1	9.1	5.8	4.1	3.9	3.7	4.0
C_2	6.1	4.1	2.9	2.5	2.5	2.4
C_3	5.6	4.0	3.0	3.1	2.8	2.2
C_4	3.6	2.7	2.2	2.1	1.9	1.4
Total C_1-C_4	24.4	16.6	12.2	11.6	10.9	10.0
CO	0.5	0.6	0.3	0.5	1.0	0.3
CO_2	1.1	1.1	1.1	1.1	1.1	1.1
H_2S	2.3	1.9	2.1	2.1	2.0	1.8
NH_3	(0.5)	0.9	0.7	0.7	0.7	0.5
Total Gas	28.8	21.1	16.4	16.0	15.7	13.7
Water	10.9	10.8	11.4	11.1	14.0	11.7
Naphtha, C_5-193°C	17.5	10.4	10.6	10.9	10.2	9.4
Wash Solvent, 193-249°C	1.6	5.5	8.9	2.4	3.4	3.6
Recycle Solvent, >249°C	16.5	23.7	24.8	25.3	23.8	16.5
Total Oil	35.6	39.6	44.3	38.6	37.4	29.5
SRC	12.7	18.5	15.9	22.6	23.2	34.1
Insolubles Organic Matter	7.2	5.2	4.5	4.5	4.3	5.4
Ash	9.5	9.3	11.5	11.3	9.2	9.2
Total Distillation Residue	29.4	33.0	31.9	38.4	36.7	48.7
Total	104.7	104.5	104.0	104.1	103.8	103.6
H_2 Reacted, wt %						
gas balance	4.7	4.5	4.0	4.1	3.8	3.6
product analysis	--	4.8	4.3	4.3	4.0	2.9
Recycle Solvent Analyses						
% C	88.63	88.04	87.92	87.57	87.34	87.40
% H	7.66	7.57	7.84	8.25	8.15	8.02
% S	0.27	0.27	0.22	0.22	0.27	0.30
% N				1.40	1.35	1.26
% O				2.56	2.89	3.02
Specific Gravity	1.0503	1.0534	1.0396	1.0400	1.0426	1.0400

TABLE B-1 (Cont.)

	GU 139R	GU 140R[a]	GU 141R	GU 142R[g]	GU 143R[b]	GU 144R[c]
Reaction Conditions						
Coal (Kentucky Nos. 9 & 14)	Lot 5	Lot 5	Lot 5	Lot 5	Lot 6	Lot 6
Nominal Liquid Residence Time, hr	1.0	1.5	1.6	0.7	1.0	1.0
Nominal Dissolver, Temperature, °C	455	455[a]	455	455	455	455
Reactor Pressure, psig	1900	1900	1900	1900	1900	1900
H_2 Feed, wt % based on slurry	4.8	4.7	4.8	5.0	4.7	4.9
Slurry Composition, wt %						
Coal	45.0	45.1	40.1	30.0	35.0	35.0
Recycle Coal Solution	27.6	26.7	50.3	70.0	60.0	60.0
Distillate Recycle Solvent	27.4	28.2	9.6	--	5.0	5.0
Yields, wt % of feed coal						
C_1	4.6	8.8	5.7	5.8	4.1	4.0
C_2	2.6	5.1	3.2	2.5	2.9	2.5
C_3	2.9	4.4	3.2	2.8	2.9	2.6
C_4	1.5	2.3	1.8	1.8	2.1	1.1
Total C_1-C_4	11.6	20.6	13.9	12.9	12.0	10.2
CO	(0.5)[e]	0.4	(0.5)[e]	(0.5)[e]	0.4	0.3
CO_2	1.1	1.0	1.1	1.1	0.7	0.9
H_2S	1.6	1.7	1.4	1.8	1.5	1.3
NH_3	0.6	0.5	0.5	0.6	0.5	0.4
Total Gas	15.4	24.2	17.4	16.9	15.1	13.1
Water	8.2	12.1	10.2	8.6	8.6	7.9
Naphtha, C_5-193°C	8.1	8.9	10.9	8.3	9.3	9.5
Wash Solvent, 193-249°C	2.0	2.9	3.6	2.5	3.2	3.7
Recycle Solvent, >249°C	14.4	13.4	20.9	23.9	21.1	23.1
Total Oil	24.5	25.2	35.4	34.7	33.6	36.3
SRC	41.3	28.6	26.8	30.4	32.3	31.5
Insolubles Organic Matter	4.7	5.9	4.9	4.8	5.6	6.8
Ash	9.5	9.4	9.4	9.4	9.1	9.3
Total Distillation Residue	55.5	43.9	41.1	44.6	47.0	47.6
Total	103.6	105.4	104.1	104.8	104.3	104.9
H_2 Reacted, wt %						
gas balance	3.6	5.4	4.1	4.8	4.3	4.9[c]
product analysis	3.3	5.1	4.5	4.0	3.1	2.8
Recycle Solvent Analyses						
% C	87.17	87.81	87.70	86.77	87.62	87.56
% H	8.14	7.89	8.31	8.24	7.61	7.89
% S	0.23	0.20	0.23	0.31	0.35	0.33
% N	1.16	1.18	1.25	1.24	1.43	1.29
% O	3.30	2.92	2.51	3.44	2.99	2.93
Specific Gravity	1.0325	1.0322	1.0364	1.0377	1.0524	1.0454

TABLE B-1 (Cont.)

	GU 145R[c]	GU145RB	GU 146R	GU 147R	GU 148R[d]	GU 149R[g]
Reaction Conditions						
Coal (Kentucky Nos. 9 & 14)	Lot 6	Lot 6	Lot 5	Lot 5	Lot 6	Lot 5
Nominal Liquid Residence Time, hr	1.0	1.0	1.0	1.0	1.0	0.7
Nominal Dissolver, Temperature °C	455	455	465	455	455	455
Reactor Pressure, psig	1900	1900	1900	1900	1900	1900
H_2 Feed, wt % based on slurry	4.8	4.7	5.1	4.6	4.5	4.8
Slurry Composition, wt %						
Coal	35.0	35.0	40.0	45.0	35.0	20.0
Recycle Coal Solution	60.0	60.0	51.0	27.5	60.0	80.0
Distillate Recycle Solvent	5.0	5.0	9.0	27.5	5.0	--
Yields, wt % of feed coal						
C_1	3.1	3.9	4.7	(4.6)[e]	3.9[e]	5.0
C_2	2.0	2.6	3.0	(2.6)[e]	2.6[e]	3.2
C_3	2.1	2.5	2.6	(2.9)[e]	2.5[e]	3.8
C_4	1.4	1.5	1.4	(1.5)[e]	1.5[e]	2.3
Total C_1-C_4	8.6	10.5	11.7	(11.6)[e]	10.5[e]	2.3
CO	0.4	0.3	0.4	(0.5)[e]	0.3[e]	0.6
CO_2	0.9	1.1	1.2	(1.1)[e]	1.1[e]	1.2
H_2S	1.4	2.2	1.7	(1.6)[e]	2.2[e]	2.9
NH_3	0.4	0.4	0.7	(0.6)[e]	0.4[e]	0.7
Total Gas	11.7	14.5	15.7	(15.4)[e]	14.5[e]	19.7
Water	9.0	9.9	9.2	7.7	8.4	10.2
Naphtha, C_5-193°C	7.6	8.3	7.1	8.2	7.5	13.5
Wash Solvent, 193-249°C	3.0	3.6	2.9	1.9	2.2	5.4
Recycle Solvent, >249°C	22.1	19.9	18.7	15.5	22.5	21.2
Total Oil	32.7	31.8	28.7	25.6	32.2	40.1
SRC	34.4	31.8	33.7	40.0	33.5	19.6
Insolubles Organic Matter	6.6	6.5	6.7	5.3	6.0	5.7
Ash	9.4	9.1	9.4	9.4	8.9	9.4
Total Distillation Residue	50.4	47.4	49.8	54.7	48.4	34.7
Total	103.8	103.6	103.4	103.4	103.5	104.7
H_2 Reacted, wt %						
gas balance	3.8[c]	3.6	3.4	--	--	4.7
product analysis	2.7	3.0	3.3	3.3	2.9	4.8
Recycle Solvent Analyses						
% C	86.91	87.16	87.19	87.15	86.86	87.38
% H	8.09	8.06	8.09	8.15	8.17	7.89
% S	0.32	0.33	0.29	0.29	0.29	0.28
% N	1.25	1.33	1.26	1.20	1.24	1.27
% O	3.43	3.12	3.17	3.21	3.44	3.18
Specific Gravity	1.0326	1.0412	1.0377	1.0323	1.0321	1.0446

TABLE B-1 (Cont.)

	GU 150R[g]	GU 151R[g]	GU 152R	GU 153R	GU 154R	GU 155R
Reaction Conditions						
Coal (Kentucky Nos. 9 & 14)	Lot 5	Lot 5	Lot 5	Lot 5	Lot 5	Lot 5
Nominal Liquid Residence Time, hr	0.7	0.7	1.0	1.0	1.0	1.0
Nominal Dissolver, Temperature °C	465	455	455	455	455	445
Reactor Pressure, psig	1900	1900	1900	1900	1900	1900
H_2 Feed, wt % based on slurry	5.2	6.5	6.3	6.1	6.2	6.6
Slurry Composition, wt %						
Coal	30.0	30.0	30.0	45.0	40.0	30.0
Recycle Coal Solution	70.0	70.0	70.0	27.5	51.0	70.0
Distillate Recycle Solvent	--	--	--	27.5	9.0	--
Yields, wt % of feed coal						
C_1	5.6	3.9	3.9	3.0	3.5	3.4
C_2	3.6	2.4	2.3	1.9	2.2	2.3
C_3	3.8	2.7	2.7	2.1	2.4	2.3
C_4	2.3	1.8	1.8	1.2	1.5	2.0
Total C_1-C_4	15.3	10.8	10.7	8.2	9.6	10.0
CO	0.7	0.5	0.9	0.6	0.7	0.6
CO_2	1.3	1.3	1.2	1.3	1.2	1.3
H_2S	2.6	2.2	2.4	2.2	2.2	2.6
NH_3	0.6	0.5	0.6	0.4	0.4	0.4
Total Gas	20.5	15.3	15.8	12.7	14.1	14.9
Water	10.4	7.6	9.1	9.0	9.1	8.4
Naphtha, C_5-193°C	10.7	11.0	9.1	6.5	8.1	8.7
Wash Solvent, 193-249°C	6.0	3.6	5.3	2.9	2.5	3.7
Recycle Solvent, >249°C	22.3	27.3	25.5	16.0	19.9	22.7
Total Oil	39.0	41.9	39.9	25.4	30.5	35.1
SRC	17.2	25.1	24.2	41.6	35.0	31.1
Insolubles Organic Matter	7.8	4.7	4.9	4.7	5.1	4.3
Ash	9.0	9.4	9.4	9.3	9.3	9.3
Total Distillation Residue	34.0	39.2	38.5	55.6	49.4	44.7
Total	103.9	104.0	103.3	102.7	103.1	103.1
H_2 Reacted, wt %						
gas balance	3.9	4.0	3.3	2.7	3.1	3.1
product analysis	4.6	3.7	3.7	2.7	3.2	3.6
Recycle Solvent Analyses						
% C	87.57	87.31	87.52	87.21	86.77	86.58
% H	7.71	8.08	7.88	8.24	8.17	8.49
% S	0.29	0.30	0.23	0.29	0.29	0.35
% N	1.17	1.25	1.25	1.11	1.21	1.30
% O	3.26	3.06	3.12	3.15	3.56	3.28
Specific Gravity	1.0491	1.0405	1.0458	1.0351	1.0345	1.0329

TABLE B-1 (Cont.)

	GU 156R[f]	GU 157R[fg]	GU 158R	GU 159R	GU160RA	GU160RB
Reaction Conditions						
Coal (Kentucky Nos. 9 & 14)	Lot 5	Lot 5	Lot 5	Lot 5	Lot 5	Lot 5
Nominal Liquid Residence Time, hr	1.0	1.1	1.5	1.0	0.7	0.6
Nominal Dissolver Temperature, °C	455[f]	455[f]	455	445	465	465
Reactor Pressure, psig	1900	1900	1900	1900	2000	1800
H_2 Feed, wt % based on slurry	7.8	4.2	5.0	4.6	5.7	5.1
Slurry Composition, wt %						
Coal	40.0	40.0	45.0	40.0	30.0	30.0
Recycle Coal Solution	51.0	51.0	27.5	51.0	70.0	70.0
Distillate Recycle Solvent	9.0	9.0	27.5	9.0	--	--
Yields, wt % of feed coal						
C_1	5.5	5.0	5.5	Not Lined Out	6.6	6.6[e]
C_2	3.9	3.5	3.9		4.6	4.6[e]
C_3	3.1	2.7	3.4		3.7	3.7[e]
C_4	1.8	1.5	1.5		2.0	2.0[e]
Total C_1-C_4	14.3	12.7	14.3		16.9	16.9[e]
CO	0.4	0.4	0.4		0.4	0.4[e]
CO_2	0.8	0.8	0.8		1.1	1.1[e]
H_2S	3.3	1.9	2.2		2.2	2.2[e]
NH_3	0.6	0.4	0.4		0.7	0.7[e]
Total Gas	19.4	16.2	18.1		21.3	21.3[e]
Water	9.4	8.7	9.9		9.4	8.4
Naphtha, C_5-193°C	8.2	10.3	10.5		9.5	9.2
Wash Solvent, 193-249°C	5.3	5.5	4.3		4.9	5.2
Recycle Solvent, >249°C	20.3	18.5	18.2		23.6	22.9
Total Oil	33.8	34.3	33.0		38.0	37.3
SRC	25.2	28.9	27.9		20.9	21.5
Insolubles Organic Matter	6.0	6.6	4.8		6.3	7.1
Ash	9.2	9.4	9.4		8.6	8.9
Total Distillation Residue	40.4	44.9	42.1		35.8	37.5
Total	103.0	104.1	103.1		104.5	104.5
H_2 Reacted, wt %						
gas balance	--	4.1	3.1		4.5	4.5[e]
product analysis	3.8	3.6	4.1		4.6	4.5
Recycle Solvent Analyses						
% C	87.58	87.77	88.07	87.54	88.07	88.15
% H	7.94	7.83	7.85	7.88	7.83	7.62
% S	0.25	0.28	0.22	0.28	0.25	
% N	1.24	1.23	1.19	1.12	1.39	1.43
% O	2.99	2.89	2.67	3.18	2.46	
Specific Gravity	1.0365	1.0409	1.0349	1.0385	1.0500	

a) Recorder malfunction, dissolver overheated - nominal conditions repeated in GU 158R.
b) Single dissolver
c) Reported gas yields and H_2 consumed (by product analysis) low due to leak in gas sampling system. H_2 consumed (by gas analysis) may be high for the same reason.
d) Coiled preheater
e) Estimated from results for a similar run.
f) Results influenced by very poor temperature control.
g) Approximate yields, steady state operation not reached.

TABLE B-2

ESTIMATION OF ANTICIPATED ERROR OF HYDROGEN CONSUMPTION BY GAS BALANCE TECHNIQUE

Method Hydrogen In - Hydrogen Out = Consumption
% Consumption = Consumption x 100 ÷ Coal Fed

Hydrogen fed by capillary tube. Accuracy estimated to be $\pm$ 1 g mole/hr

Rate 22 g mole/hr $\bar{X} = 22.0$ $Sx = 0.5$
$\longrightarrow$ ≈95% of time $21.0 \leq x \leq 23$

Similarly for gauge purge
Rate 1.00 g mole/hr $\pm$ 0.05

Typical reacted H_2 3.5 wt % coal on 350 g/hr coal fed
or 12.25 g/hr

Outlet H_2 $23.0 - 12.25/2.02 = 16.94$ g mole/hr

Outlet gas typically 90 mole % H_2

Outlet gas volume $16.94/.9 = 18.82$ g mole/hr

Volume measurement by wet test meter $\pm$ 0.25 g mole/hr
Chromatographic analysis of outlet gas for hydrogen
accurate to $\pm$ 3 % relative
Actual H_2 concentration 87.3 - 92.7 mole %
Range of H_2 rate out
Low $(18.82-0.25) \div .873 = 16.21$ g mole/hr
High $(18.82+0.25) \div .927 = 17.68$
Actual and error 16.95 $\pm$ 0.73

Uncertainty in H_2

In 23.00 $\pm$ 1.05 g mole/hr
Out 16.94 $\pm$ 0.73

Consumed 6.06 $\pm$ 1.78

Accuracy of coal feed rate 1 % relative
Actual 350 $\pm$ 3.5 g/hr

% Consumption
Low $100 \ (6.06-1.78) \ .2.02 \ / \ (350+3.5) = 2.45$
High $100 \ (6.06+1.78) \ 2.02 \ / \ (350-3.5) = 4.57$
% Consumed 3.5 $\pm$ 1.06
$\longrightarrow P\ (2.45 \leq x \leq 4.57) = 0.95 \rightarrow Sx = 0.54 \ \ Sx/\bar{X} = 0.15$

Analysis is conservative in that worst case variants are used; e.g., high flow with high concentration. Solution loss of H_2 ignored which may bias result to higher than actual (chemical) hydrogen consumption.

TABLE B-3

ESTIMATION OF ANTICIPATED ERROR OF HYDROGEN CONSUMPTION BY PRODUCT ANALYSIS TECHNIQUE

Method Hydrogen in Products - Hydrogen in Coal = Hydrogen Reacted

GAS PRODUCT

Component	Component Concentration Mole %	Analytical Error % Relative	Analytical + Volume %	Component Accuracy g/hr	Contained Hydrogen Accuracy g/hr
C_1	4.38	3.7	5.03	0.663	0.167
C_2	1.44	4.9	6.23	0.137	0.028
C_3	0.66	4.5	5.83	0.318	0.058
C_{4+}	0.23	10.8	12.13	0.304	0.053
C_{5+} (as C_6)	0.47		5.43[1]	0.413	0.068
H_2S	0.61	16.0	17.33	0.675	0.040
NH_3	0.28	10.7	12.03	0.108	0.019
Rate Mole/hr	18.8	1.33		---	
					Σ 0.433

[1] Based on gas density measurements

LIQUID & SOLID FEEDS & PRODUCTS

Component	Rate g/hr	Rate Accuracy % Relative	Hydrogen Content Wt %	Analytical Accuracy % Relative	Analytical & Rate %	Contained Hydrogen Accuracy g/hr
Coal	350	1.0	5.1	1.96	2.96	.497
Water	20.6	17.0	11.2	0	17.0	.392
Naptha	20.6	6.0	11.8	0.85	6.85	.163
Solvent	49.1	6.0	7.6	1.32	7.32	.273
SRC	156.1	3.0	5.0	2.02	5.02	.392
IOM	24.5	9.0	2.0	5.00	14.0	.069
						Σ 1.289

ΔH products + ΔH coal = ΔH reacted
$0.433 + 1.289 + 0.497 = 2.219$ g/hr
1.10 g mole/hr

% Consumption
Low $100 \cdot (12.25-2.22) \div (350+3.5) = 2.84$
High $100 \cdot (12.25+2.22) \div (350-3.5) = 4.18$

% Consumed 3.5 ± 0.67

Assuming
$P(2.84 \leq x \leq 4.18) = 0.95$
$\longrightarrow Sx = 0.34 \quad Sx/\overline{X} = 0.098$