# THE SRC II PROCESS

R. P. Anderson

The Pittsburg & Midway Coal Mining Co.
9009 West 67th Street
Merriam, Kansas 66202

## I. INTRODUCTION

In the original P&M Solvent Refined Coal Process,[1] now designated SRC I, coal is dissolved under moderate hydrogen pressure in an internally generated heavy aromatic solvent to produce a liquid from which mineral matter is removed by filtration. Solvent is recovered for reuse by vacuum distillation; the distillation residue which is a solid under ambient conditions is a very low ash, low sulfur fuel known as Solvent Refined Coal. The current state of development of the SRC I process is indicated by the highly successful operation of two pilot plants; a 6 ton/day pilot plant at Wilsonville, Alabama,[2] which is currently sponsored by ERDA and EPRI and is operated by Catalytic, Inc., and a larger 50 ton/day pilot plant at Fort Lewis, Washington,[3] which is sponsored by ERDA and is operated by The Pittsburg & Midway Coal Mining Co., a subsidiary of Gulf Oil Corp.

In an improved version of the process, designated SRC II,[4] a portion of coal solution is recycled as solvent in the process in place of the distillate solvent of 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.

In the modified process, a fraction of the coal solution is used in feed slurry formulation; the remainder is available for product isolation (Figure 1). The fuel value could be recovered by filtration in which case the primary fuel product would be the filtrate, a heavy, sometimes viscous liquid product. Laboratory work has demonstrated, however, that under appropriate reaction conditions the conversion of high molecular weight dissolved coal to lower molecular weight products is adequate to allow recovery of the fuel product by distillation, thereby eliminating the difficult and expensive filtration step. The primary product is a low sulfur distillate fuel oil with a distillation range corresponding roughly to that of recycle solvent of the SRC I process. 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.

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 coal solution allows additional reaction time for 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.

In this paper, development of the SRC II process at the P&M Merriam Laboratory is described. In the first stage of development, this work defined in general terms conditions under which the SRC II process could be conducted and product yields and properties were determined. In later work, which was designed to provide information for SRC II operation at the Fort Lewis Pilot Plant, a systematic study was made to determine the effect of the following variables: coal concentration in the feed slurry, dissolver residence time, dissolver temperature, and hydrogen feed rate. Pressure effects were not a part of the latter study (the objective was to make the study at the minimum practical pressure) but some comparisons between runs at varying pressures are available.

## II. EXPERIMENTAL

Materials: Coal used was a blend of Kentucky Nos. 9 and 14 from P&M's Colonial Mine in Hopkins County. Most of the work described in this paper was carried out with one lot, designated lot 5, but several runs (GU 120, GU 4A, GU 135R) carried out with another lot, lot 4, which was of higher iron content are also presented. Coal analyses are given in Table I. Recycle solvent was generally internally generated. When insufficient internally generated solvent was available for start up of a run, recycle solvent from the Fort Lewis Pilot Plant was used.

Equipment: A simplified schematic of the laboratory scale continuous reactor used in the present work is shown in Figure 2. The reaction subsystem consists of a preheater and one or two dissolvers operated in series. The preheater is a 4.5 ft section of 11/16 inch ID pressure tubing (316 stainless steel) 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. Due to the low temperature of this vessel, preheater 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 cu ft) 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.

TABLE I

Feed Coal Analysis
(Dry Basis)

| PROXIMATE ANALYSIS | Lot 4 | Lot 5 |
|---|---|---|
| % Ash | 11.39 | 8.87 |
| % Volatile | 38.12 | 38.12 |
| % Fixed Carbon | 50.49 | 53.01 |
| | 100.00 | 100.00 |
| Btu/lb | 12700 | 13238 |

| ULTIMATE ANALYSIS | % Weight Lot 4 | % Weight Lot 5 |
|---|---|---|
| Carbon | 69.54 | 71.89 |
| Hydrogen | 4.94 | 5.10 |
| Nitrogen | 1.22 | 1.24 |
| Chlorine | 0.03 | 0.06 |
| Sulfur | 4.11 | 3.21 |
| Ash | 11.39 | 8.87 |
| Oxygen (diff) | 8.77 | 9.63 |
| | 100.00 | 100.00 |

| SULFUR FORMS | Lot 4 | Lot 5 |
|---|---|---|
| % Pyritic Sulfur | 2.65 | 1.87 |
| % Sulfate Sulfur | 0.54 | 0.27 |
| % Organic Sulfur | 0.92 | 1.07 |
| % Total Sulfur | 4.11 | 3.21 |
| Free Swelling Index | 3.5 | 5 |

| MINERAL ANALYSIS | % Weight Ignited Basis Lot 4 | % Weight Ignited Basis Lot 5 |
|---|---|---|
| Phos. pentoxide, $P_2O_5$ | 0.37 | 0.14 |
| Silica; $SiO_2$ | 31.56 | 39.10 |
| Ferric oxide, $Fe_2O_3$ | 38.76 | 31.78 |
| Alumina, $Al_2O_3$ | 22.93 | 20.91 |
| Titania, $TiO_2$ | 0.98 | 0.98 |
| Lime, CaO | 1.79 | 2.00 |
| Magnesia, MgO | 0.45 | 0.65 |
| Sulfur trioxide, $SO_3$ | 1.27 | 1.71 |
| Potassium oxide, $K_2O$ | 1.34 | 2.20 |
| Sodium oxide, $Na_2O$ | 0.31 | 0.38 |
| Undetermined | 0.24 | 0.15 |
| | 100.00 | 100.00 |

Reactor effluent passes from the dissolver to a pressure letdown and sample collection system. This system accomplishes the separation of gases, water, and light oil from the coal solution. The resultant stripped coal solution is collected for intervals of from 2 to 4 hrs; further workup of the coal solution (slurry preparation and product isolation) is performed manually in batch procedures.

Operating Procedures: 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 is 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[5] and will not be reiterated here. A typical run control chart is shown in Figure 3. 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[a] increased to a steady state value slightly below 20 and $\overline{IR}$[a] (except for the low value for the initial sample which is a reflection of the lower hydrogenation state for the startup solvent) showed 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.

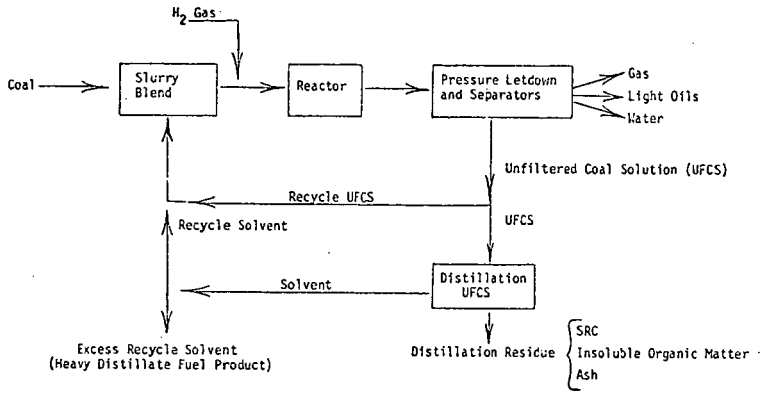
A portion of the coal solution is used in slurry formulation; the remainder is available for product isolation. Yields presented in this paper are for the process involving distillation of unfiltered coal solution to produce a distillate fuel product and a distillation residue. Yields of the three components of the distillation residue--ash, insoluble organic matter, and solid SRC (dissolved but not distillable)--will be 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 slurry formulation as well as the added coal). This constraint was imposed by consideration of possible operational constraints for the Fort 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 shown on the following page 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

---

[a]Blackness is a measure of absorbance of the coal solution in the visible region. $\overline{IR}$, 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 reference 5 for a complete description of these measurements.

concentration of 30%, no recycle solvent is 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.




Conditions Explored: Factors and levels which were investigated in the GU 137R-GU 160R series are as follows:

| Factor | Levels |
|---|---|
| Coal concentration in feed slurry, % | 30, 40, 45 |
| Dissolver Temperature, $^0C$ | 445, 455, 465 |
| Residence Time, hr | 2/3, 1, 1 1/2 |
| Hydrogen Feed Rate, wt % based on feed slurry | 4.9, 6.3 |

A complete factorial would require an excessive number of runs, so only selected conditions were investigated. Pressure which is known to be an important variable was not investigated as the objective was to make the current study at the minimum practical pressure. Several runs outside of this series (made at other conditions including other pressures) are included in this paper for comparison purposes.

Variables examined were not varied over a wide range; previous work had defined in general terms conditions over which the SRC II process was operable. For example, at temperatures below about 445$^0$C, the rate for

conversion of asphaltene to oil is too low to sustain satisfactory operation without an excessively long residence time and at temperatures much in excess of 465°C, coking becomes a problem.

It was generally possible to attain satisfactory steady state operation and obtain reliable yield data for the 1 hour and 1.5 hour residence time runs but experimental difficulties were often experienced with shorter residence times. The primary problem experienced in the shorter residence time runs was with feed slurry viscosity. At the shorter residence time, conversion of SRC to oil is lower and feed slurry viscosities are higher. Under such conditions, feed slurry viscosity would lead to slurry pump failure which would force run termination before a satisfactory period of steady state operation was achieved.

Yield Reporting: Yields are reported on a loss free, forced ash balance basis. Actual material accountability was excellent with reactor output normally varying from input by only a few tenths of a percent. Yields considered in this paper are hydrocarbon gas yields ($C_1$-$C_4$), oil yields, SRC yields, and, in one comparison, insoluble organic matter (IOM) yields. Methane through butane yields are considered rather than total gas yields or total hydrocarbon gas yields as $C_1$-$C_4$ yields are not subject to variability in operation of the reactor sampling system as are total gas yields or total hydrocarbon gas yields. (The latter yields are subject to variable efficiency of removal of heavier hydrocarbon gases from the product gas stream.)

Total oil yields are considered rather than yields of discrete distillation fractions; this eliminates any problems which might be introduced by any variability in the fractionation procedure.

Total oil yields include the heavy distillate fuel cut (>249°C, equivalent to recycle solvent) and the cuts which correspond to the wash solvent (193-249°C) and light oil (<193°C) cuts of the SRC I process. The fraction noted as "unidentified gas"[a] has been shown to consist primarily of $C_5$-$C_8$ hydrocarbons so this category is included with the total oil yield.

Distillation residue produced from distillation of unfiltered coal solutions contains ash, insoluble organic matter, and SRC. As ash is independent of processing conditions and the insoluble organic matter yield varies over only a small range, distillation residue and SRC yields respond to changes in operating conditions in a parallel manner. In this treatment, SRC yields rather than total distillation residue yields are considered. Insoluble organic matter yields are also considered where they are of interest.

Water yields appear anomalously high in some cases. The high water yields may be related to hygroscopic properties of the slurry or, more likely, may be due to oxidation of hot feed slurry. Forced carbon

[a]Difference between total weight of reactor output gas and weight of identified gases.

balance yields have been calculated[7] to correct for the high water yields but are not used in this paper. Reported water yields are for the crude aqueous phase; the aqueous phase typically is about 92% water containing dissolved ammonia, hydrogen sulfide, carbonate and phenol.

Hydrogen consumptions are calculated by two methods. In the hydrogen gas balance method, consumption is determined by subtracting the amount of hydrogen in the output gas from the calibrated hydrogen gas input. This method involves the determination of a small difference between two large numbers, both of which are subject to a high experimental uncertainty. As this method is generally considered unsatisfactory due to the large experimental uncertainty, hydrogen consumptions are also calculated by an alternate method.

A somewhat improved estimation of hydrogen consumption is obtained by product analysis. The amount of hydrogen in the products is determined from the product distribution (forced carbon balance basis) and the composition (% hydrogen) of each product. Hydrogen consumption is then equal to the amount of hydrogen in the products less the hydrogen content of the feed coal.

## III. RESULTS

SRC I - SRC II Comparison: SRC I runs are generally made at shorter residence times and lower pressures than are SRC II runs. In normal operation, conditions for SRC I runs are selected to obtain a breakeven yield of recycle solvent but with no attempt to significantly increase the solvent yield beyond that required for recycle. Three laboratory runs are compared in Table II. These runs represent an SRC I run made under typical SRC conditions; an SRC I run made under conditions typical of SRC II operations, and a typical SRC II run. These runs were made with lot 4 coal. (All other runs discussed in this paper were made with lot 5 coal.) Under the typical SRC I conditions, a breakeven yield of recycle solvent is obtained but there is no excess heavy oil while the primary product of the process is the solid SRC which is obtained in 56% yield. In the SRC I run made under the conditions typical of the SRC II process, a 16% yield of excess recycle solvent is obtained (total distillate oil yield of 29%) and the SRC yield is decreased to 35%. Under the same conditions but in the SRC II mode of operation the SRC yield is decreased to only 16% and the total distillate oil yield is increased to 44%. The improved conversion of solid SRC to lighter products in the SRC II runs is due to a combination of the increased mineral matter (catalyst) concentration and the recycling of the coal solution which allows additional reaction time for conversion of solid SRC to distillate products.

The results of this SRC II run indicate that the concept of the SRC II operation with recovery of the fuel product by distillation is practical and that the yield of distillation residue can be reduced to that required for hydrogen generation for the process. It also indicates that the activity of the naturally occurring catalyst in the coal is adequate to accomplish the required conversion. That the use of a

separate catalyst is not required is a major advantage of the SRC II process. As the conversion to low molecular weight products is adequate to allow isolation of the fuel product by distillation, the difficult and expensive solids removal step as well as mineral residue drying and product solidification steps may be eliminated which greatly simplifies the process. Due to the elimination of filters, dryers, and solidification equipment, it has been estimated that the cost of the SRC II product should be no more than that of the solid SRC I product.[4]

Pressure: As noted previously, pressure was not among the variables included in the current study. It is generally recognized that as pressure is increased, desired hydrogenation rates are increased and operability is improved. The objective of this study was to study other variables at the minimum practical pressure. Work at pressures lower than the 1900 psig of this study had shown that it was possible to sustain SRC II operation at a lower pressure but only with significant increases in residence time. It was found to be impossible (in laboratory equipment) to maintain operation at 1500 psig with other conditions similar to those of GU 135R (Table II). In order to keep the viscosity of the feed slurry manageable, residence time was gradually increased. It was eventually found to be possible to maintain satisfactory operation but only after the residence time was increased to 1.7 hr. These conditions resulted in a high conversion of SRC to lighter products (SRC yield of only 13%) but only with an excessively high gas yield, ($C_1$-$C_4$ yield of 24.4%, twice that reported for GU 135R).

In view of unsatisfactory results at pressures significantly below 1900 psig and the desire to operate at the minimum practical pressure, all further work was carried out at 1900 psig (with the exception of GU 160A and GU 160B which carried out pressures of 2000 and 1800 psig, respectively).

Coal Concentration: The effect of coal concentration is readily apparent from Table III. These runs were all made with a dissolver temperature of 455°C, a pressure of 1900 psig, and a residence time of one hour. No correlation between hydrocarbon gas yields and coal concentration is apparent, but there are near linear relationships between coal concentration and yields of SRC and oil. Oil yield decreases with increasing coal concentration while SRC yield increases with increasing coal concentration.

Residence Time: Three levels of residence time were investigated. Most runs were made with a nominal one hour liquid residence time with a limited number of runs made with a 1.5 or 2/3 hour residence time. The 2/3 hour residence time runs were made only with low coal concentrations and were generally subject to operating difficulties. Results in Table IV show that the reduction in residence time from one hour to 2/3 hour results in a significant increase in SRC yield and a smaller

TABLE II

Comparison SRC I and SRC II Runs

| | Typical SRC I Run (GU 120) | SRC I Under Typical SRC II Conditions (GU 4A) | SRC II (GU 135R) |
|---|---|---|---|
| Reaction Conditions | | | |
| Pressure, psig | 1425 | 2000 | 2000 |
| Dissolver Temperature, $^0C$ | 450-455 | 455-465 | 465 |
| Nominal Residence Time, hr | 0.4 | 1.0 | 1.0 |
| Hydrogen Feed, wt % of slurry | 2.1 | 5.3 | 4.9 |
| Feed Slurry Composition | | | |
| % Coal | 39.0 | 35.0 | 35.0 |
| % Coal Solution | -- | -- | 65.0 |
| % Distillate Solvent | 61.0 | 65.0 | |
| Yields, Wt % Based on Coal | | | |
| $C_1$-$C_4$ | 2.9 | 12.5 | 12.2 |
| CO, $CO_2$, $H_2S$ | 3.0 | 3.6 | 3.5 |
| Water | 6.4 | 7.6 | 11.4 |
| Light Oil, $C_5$-249$^0$C | 16.4 | 13.6 | 19.5 |
| Heavy Distillate, <249$^0$C | -- | 15.7 | 24.8 |
| Total Oil | 16.4 | 29.3 | 44.3 |
| SRC | 56.3 | 34.8 | 15.9 |
| IOM | 5.3 | 4.4 | 4.5 |
| Ash | 11.3 | 11.3 | 11.5 |
| $H_2$ Reacted (gas balance) | 1.6 | 3.5 | 4.0 |

TABLE III

Effect of Coal Concentration in Feed Slurry

| Run No. | % Coal | $C_1$-$C_4$ Yield | Oil Yield | SRC Yield |
|---|---|---|---|---|
| GU 137R | 30 | 10.9 | 37.4 | 23.2 |
| GU 152R | 30 | 10.7 | 39.9 | 24.2 |
| GU 138R | 40 | 10.0 | 29.5 | 34.1 |
| GU 154R | 40 | 9.6 | 30.5 | 35.0 |
| GU 139R | 45 | 11.6 | 24.5 | 41.3 |
| GU 147R | 45 | (11.6)[a] | 25.6 | 40.0 |
| GU 153R | 45 | 8.2[b] | 25.4 | 41.6 |

a) Estimated b) Reported value is suspect

but still significant decrease in oil yield. It is suspected that the increased gas yield reported for the 2/3 hour run (GU 142R) is erroneous. This suspicion is confirmed by another run made under the same conditions except for an increased hydrogen feed rate (GU 151R). The latter run is excluded from the following comparison as the run was terminated before satisfactory steady state operation was achieved. (A run which approaches and does not reach steady state operation may produce satisfactory gas yield data although reliable oil and SRC yields are not available.)

An increase in residence time from 1 to 1 1/2 hour resulted in increases in gas and oil yields and the expected decrease in SRC yield.

TABLE IV

Effect of Residence Time

A. 30% Coal Concentration

| Run No. | Residence Time, Hr | $C_1$-$C_4$ Yield | Oil Yield | SRC Yield |
|---|---|---|---|---|
| GU 142R | 0.69 | 12.9[a] | 34.7 | 30.4 |
| GU 137R | 0.97 | 10.9 | 37.4 | 23.2 |
| GU 152R | 1.00 | 10.7 | 39.9 | 24.2 |

B. 40% Coal Concentration

| Run No. | Residence Time, Hr | $C_1$-$C_4$ Yield | Oil Yield | SRC Yield |
|---|---|---|---|---|
| GU 138R | 1.01 | 10.0 | 29.5 | 34.1 |
| GU 154R | 1.00 | 9.6 | 30.5 | 35.0 |
| GU 141R | 1.59 | 13.9 | 35.4 | 26.8 |

C. 45% Coal Concentration

| Run No. | Residence Time, Hr | $C_1$-$C_4$ Yield | Oil Yield | SRC Yield |
|---|---|---|---|---|
| GU 139R | 1.02 | 11.6 | 24.5 | 41.3 |
| GU 158R | 1.48 | 14.3 | 33.0 | 27.9 |

All runs at 455°C

[a]Reported value is suspect

Temperature: The effect of temperature is shown in Table V. Most runs were made at a dissolver temperature of 455°C with a limited number of runs at temperatures of 445 or 465°C. Although the magnitude of the effect of temperature change is quite variable, it is seen that, over the range investigated, increasing temperature generally results in increases in gas and oil yields and decreases in SRC yield. At a 30% coal concentration and one hour residence time, increasing the temperature from 445 to 455°C results in a significant decrease in SRC yield and increase in oil yield while an increase from 455 to 465°C (1 hour residence time, 40% coal) results in only a small (probably insignificant) decrease in SRC yield and increase in oil yield.

The temperature effect noted in the 2/3 hour runs is of particular interest. Oil and SRC yields for the higher temperature (465°C) runs appear more favorable (although the magnitude of the improvement is questionable due to the usual mechanical problems experienced with 2/3 hour runs), 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 may be near those which will result in coke formation, catalyst destruction, and reactor plugging.

TABLE V

Effect of Temperature

A. 30% Coal 2/3 Hr

| Run No. | Temp, °C | $C_1$-$C_4$ Yield | Oil Yield | SRC Yield | IOM 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[a] | 465 | 16.9 | 38.0 | 20.9 | 6.3 |
| GU 160RB[a] | 465 | (16.9)[b] | 37.3 | 21.5 | 7.1 |

B. 30% Coal 1 Hr

| Run No. | Temp, °C | $C_1$-$C_4$ Yield | Oil Yield | SRC Yield | IOM Yield |
|---|---|---|---|---|---|
| GU 155R | 445 | 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 |

[a]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.

[b]Estimated.

C. 40% Coal 1 Hr

| Run No. | Temp, $^0C$ | $C_1$-$C_4$ Yield | Oil Yield | SRC Yield | IOM Yield |
|---|---|---|---|---|---|
| GU 138R | 455 | 10.0 | 29.5 | 34.1 | 5.4 |
| GU 146R | 465 | 11.7 | 28.7 | 33.7 | 6.7 |

The normal hydrogen feed rate was about 4.9 wt % based on feed slurry. In several runs a hydrogen feed rate increased by about 30% (6.4 wt % based on feed slurry) was used. Results for three of the increased hydrogen feed rate runs are compared with the appropriate control runs in Table VI. Any improved reaction due to increased hydrogen feed rate is apparently nullified by the reduction in liquid residence time caused by the increased gas volume. At the levels studied, hydrogen feed rate appears to have no significant effect on product yields. This conclusion should not be considered necessarily valid for larger reactors because of anomalous bubble behavior in the small diameter reactor used in this work.

TABLE VI

Effect of Hydrogen Feed Rate

| Run No. | % Coal | Residence Time | $H_2$ Feed: Wt % Based on Slurry | $H_2$ Feed: MSCF Ton of Coal | $C_1$-$C_4$ Yield | Oil Yield | SRC Yield |
|---|---|---|---|---|---|---|---|
| GU 137R | 30 | 0.97 | 4.63 | 58.1 | 10.9 | 37.4 | 23.2 |
| GU 152R | 30 | 1.00 | 6.28 | 78.9 | 10.7 | 39.9 | 24.2 |
| GU 138R | 40 | 1.01 | 4.63 | 43.6 | 10.0 | 29.5 | 34.1 |
| GU 154R | 40 | 1.00 | 6.23 | 58.6 | 9.6 | 30.5 | 35.0 |
| GU 139R | 45 | 1.04 | 4.83 | 40.4 | 11.6 | 24.5 | 41.3 |
| GU 153R | 45 | 0.99 | 6.06 | 50.7 | 8.2[a] | 25.4 | 41.6 |

All runs at 455$^0$C

[a]Value suspect.

Hydrogen Consumption: Hydrogen consumptions generally vary in a manner consistent with the varying conditions. Precise correlations are difficult, however, in view of the relatively high experimental error in the hydrogen consumption determination. A discussion of this topic is beyond the scope of this paper. Hydrogen consumptions of the run in this report range from under 3% to 4.6%. In a typical run in which a satisfactory conversion of SRC to lighter products is achieved, hydrogen conversion is generally 4.0 to 4.5 wt % based on the feed coal.

Product Properties: Product properties have generally been observed to vary in a rational manner with operating conditions, but a detailed discussion of this topic is beyond the scope of this paper. However, properties have varied over only a relatively narrow range. The following ranges of elemental composition and specific gravity for the heavy distillate fuel product (>249°C) have been observed in the GU 136R-GU 160R series.

| | | | |
|---|---|---|---|
| Carbon | 86.8 | - | 88.1 |
| Hydrogen | 7.6 | - | 8.5 |
| Sulfur | 0.22 | - | 0.35 |
| Nitrogen | 1.1 | - | 1.4 |
| Oxygen | 2.5 | - | 3.6 |
| Specific Gravity | 1.027 | - | 1.052 |

## IV. CONCLUSIONS

It has been possible to sustain satisfactory SRC II operation at a pressure of 1900 psig and residence times of about 2/3 to 1 1/2 hours and temperatures of 445 to 465°C. Under the better conditions, distillation residue yield does not exceed that required for hydrogen generation for the process. Increasing coal concentration in the feed slurry results in increasing the yield of vacuum residue. Increasing temperature results in decreased vacuum residue yield and increased yield of light products but 465°C appears to be approaching the maximum operable temperature. Over the range investigated, hydrogen addition rate does not appear to be a significant variable in the laboratory apparatus. Increased residence time results in decreased yields of vacuum residue and increased yields of light products.

An interesting observation on this series of runs is the apparent near invariance of distillate oil yield with coal feed rate when both are expressed in terms of productivity per unit of reactor volume per hour.[6] This is illustrated in Figure 4.

Properties of the distillate fuel products vary in a predictable manner but compositions vary over a limited range. In all cases, the distillate fuel readily surpasses EPA standards for $SO_2$ emission and also meets many of the more stingent requirements for major metropolitan areas.

Hydrogen consumption measurements are subject to a relatively high experimental error but generally vary in a manner consistent with condition variations and fall in the range of 4.0-4.5% for runs with satisfactory conversion of SRC to lighter products.

## REFERENCES

[1]Kloepper, D. L., Rogers, T. F., Wright, C. H., and Bull, W. C., "Solvent Processing of Coal to Produce a De-Ashed Product." Report to Office of Coal Research, U. S. Department of Interior, by Spencer Chemical Division of Gulf Oil Corporation. Contract 14-01-0001-275, OCR R&D Report No. 9, (1965) Clearinghouse No. PB 167809.

[2]Wolk, R., Stewart, N., and Alpert, S., "Solvent Refining for Clean Coal Combustion," ERRI Journal, May 1976.

[3]Chenoweth, G. E., "Progress Report of Pilot Plant Work on the Solvent Refined Coal Process," presented at the Second Annual Conference on Coal Gasification and Liquefaction at the University of Pittsburgh, Pittsburgh, Pennsylvania on August 5-7, 1975.

[4]Schmid, B. K., Jackson, D. M., "The SRC II Process," presented at the Third Annual International Conference on Coal Gasification and Liquefaction; University of Pittsburgh, Pittsburgh, Pennsylvania, August 3-5, 1976.

[5]Anderson, R. P., and Wright, C. H., "Coal Desulfurization in the P&M Solvent Refining Process," American Chemical Society, Division of Fuel Chemistry Preprints, Vol. 20, No. 1, p 2 (1975).

[6]Wright, C. H., unpublished.

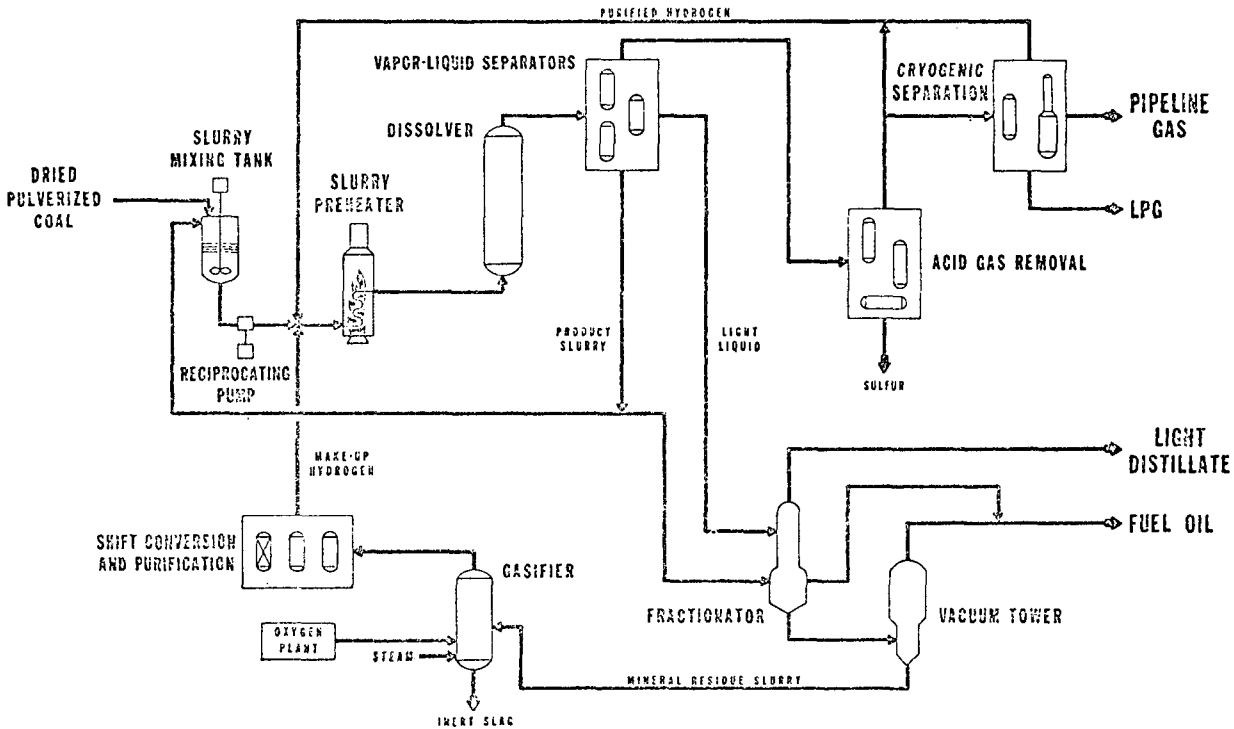
[7]Solvent Refined Coal (SRC) Process Annual Report for 1976, Pittsburg & Midway Coal Mining Co., ERDA Contract No. E(49-18)-496, FE-496-131.

## ACKNOWLEDGEMENT

This paper is based on work carried out by The Pittsburg & Midway Coal Mining Co. with support of the United States Energy Research & Development Administration under Contract No. E(49-18)-496.

FIGURE 1

# THE SRC II PROCESS



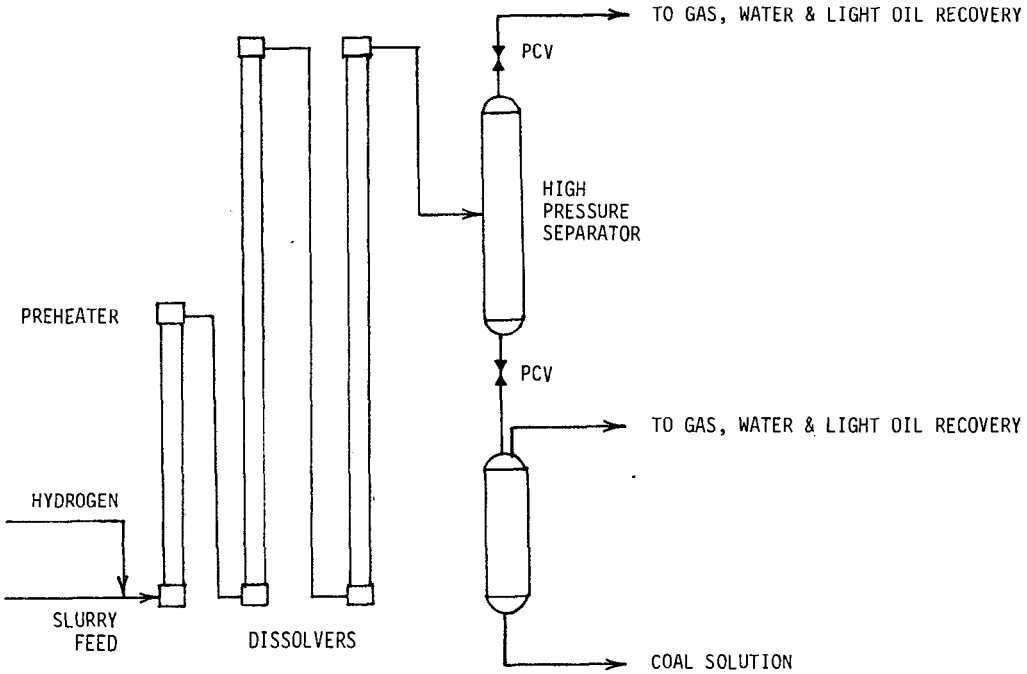



FIGURE 2 GU 5 REACTOR

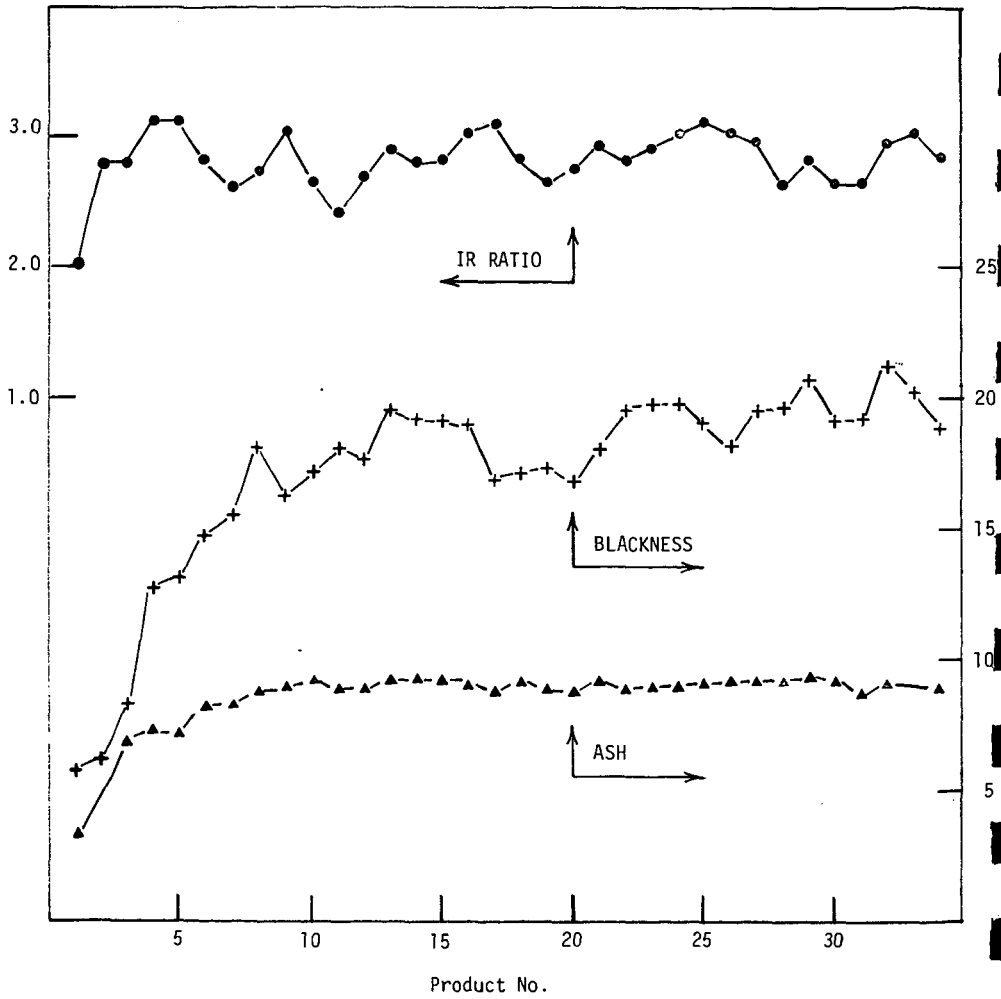
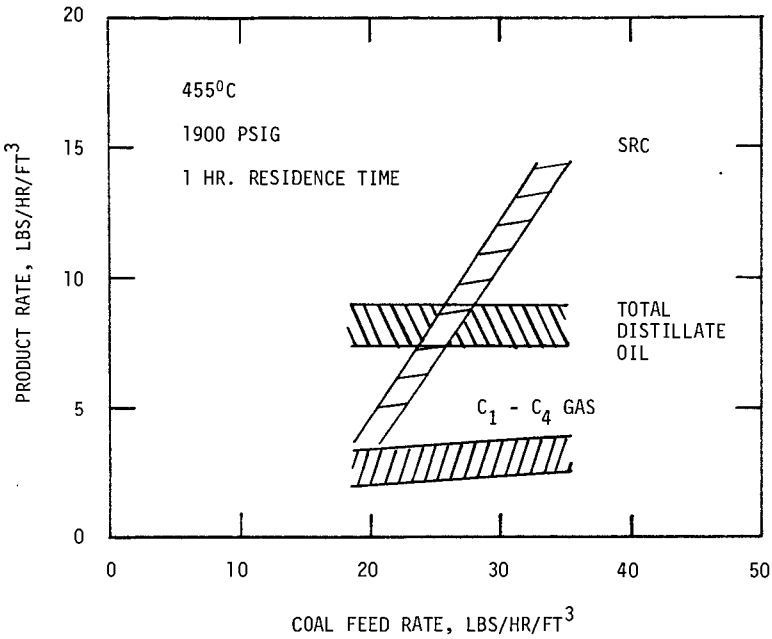



FIGURE 3 GU 146R RUN CONTROL DATA

Figure 4

PRODUCTION RATES VS COAL FEED RATE