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SRC-I Quarterly Technical Report

April-June 1980

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SRC-I QUARTERLY TECHNICAL REPORT

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**SRC-I
Solvent-Refined Coal**

QUARTERLY

TECHNICAL

REPORT

APRIL—JUNE 1980

by

INTERNATIONAL COAL REFINING COMPANY
P. O. Box 2752, Allentown, PA 18001

to

UNITED STATES DEPARTMENT OF ENERGY
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SEPARATING SOLIDS AND COAL LIQUIDS: AN EVALUATION OF CURRENT TECHNIQUES

G. C. Rappe*

As part of the conceptual design phase for a 6000 ton per day (TPD) Demonstration Plant for the Solvent Refined Coal (SRC-I) Process, an evaluation of various solid/liquid separations techniques was made.

The evaluation included Kerr-McGee's Critical Solvent Deashing (CSD) Process and Lummus's Anti-Solvent Deashing Process and was based on operating data from pilot plant operations—Kerr-McGee at Wilsonville and Lummus at Fort Lewis.

Mechanical methods for ash separation were also investigated. These included:

(1) Centrifugal techniques:

- Centrifuges
- Hydroclones

(2) Filtration techniques:

- Pressure leaf filters (United States Filter vertical leaf, Funda horizontal leaf)
- Pressure candle filters (Bergbau/Boll and Kirch, Industrial)
- Pressure rotary drum precoat filters (Goslin-Birmingham, Johns-Manville/Stearns-Roger)

The initial analysis indicated two promising approaches: Kerr-McGee's CSD Process and filtration on a vertical leaf filter (U.S. Filter). As a result, the conceptual design phase of the 6000 TPD SRC-I Demonstration Plant included both techniques in a parallel design effort.

Recent candle filter tests have also been extremely encouraging.

The choice of an ash separation technique has a significant effect on the design of the entire process and an economic comparison, including capital and operating cost factors, must take into account all differences rather than changes in the deashing area only.

THE SRC-I PROCESS

There are several coal liquefaction processes in various stages of development in the United States at this time: SRC-I (see Figure 1), SRC-II, H-Coal, and Exxon Donor Solvent (EDS). Aside from other differences in the processes, the one striking difference that sets SRC-I apart is this: only the SRC-I Process requires a solid/liquid separation by a technique other than distillation. Thus, solid/liquid separation is critical to a successful demonstration of the SRC-I Process.

*ICRC. This article originated as a paper presented at the International Conference on Solids Separations in Dublin, 17 April 1980.

The conceptual design (Phase 0) for a 6000 TPD SRC-I Demonstration Plant as submitted to the U.S. Department of Energy (DOE) in July 1979, included an expanded-bed hydrocracker in order to increase the flexibility of the process to make liquids as well as solid SRC.⁹ DOE authorized the Joint Venture of Air Products/Wheelabrator-Frye to proceed with Phase 1 (detailed design and engineering) in October 1979.

In the SRC-I Process, coal ground to 200 mesh is slurried with recycled process solvent, heated to 825°F in a fired heater in the presence of hydrogen, and sent to a dissolver. In the dissolver at 1700 psi, coal liquefaction takes place simultaneously with desulfurization and solvent rehydrogenation. After pressure letdown and light gas removal, the coal liquid, solvent, ash, and unconverted carbon are sent to solids separation. The clean liquids are fractionated and SRC is solidified. The residue from solid/liquid separation is gasified to produce an environmentally acceptable slag and to generate hydrogen for coal dissolution. If the residue does not supply sufficient hydrogen, coal is gasified with the residue.

The solid/liquid separation technique must recover as much of the SRC as possible because it is significantly cheaper to gasify coal than mineral ash residue (MAR). Therefore, separation techniques that yield slurries of diluted or low solids concentration are at an economic disadvantage compared to those methods that provide a "dry" cake discharge. One possible method eliminating this problem is to install a low temperature carbonizer (LTC) to recover additional liquids. However, the LTC adds technical uncertainties and it is by no means clear that it improves the overall economics. Many techniques for removing ash from coal liquefaction liquids have been tried, with varying degrees of success.

CENTRIFUGAL TECHNIQUES

Centrifugal techniques include hydroclones and centrifuges. Hydroclones were tested at Cresap, WV and at Wilsonville, AL (6 TPD SRC Pilot Plant).

Results from Hydroclone Systems

Several different hydroclone systems were tested at Wilsonville in 1975.¹ (See Figure 2 for a summary of data and Table 1 for a schematic of the hydroclone test system.) Solids removal efficiencies were 70-80% and centrifugal efficiencies were 60-75%. However, the ash content of the overflow was still substantially higher than the ash specification of 0.16 wt % in SRC.

One way to improve ash recovery is to add an anti-solvent (AS) to the feed to modify the agglomeration of the ash particles. Three hydroclone systems—a 10 mm dia Dorr-Oliver TML-4, a 1 inch dia Borg-Warner, and a 2 inch Heyl-Patterson—were tested to determine the efficiency of the removal of solids from the feed slurry. The effects of adding a wash solvent (350-450°F bp) and Soltrol 130 (Phillips Petroleum) were investigated.² (See Table 2 for a summary of the effect of a wash solvent addition with the Borg-Warner hydroclone.) Without the addition of a wash solvent, the best separation efficiency was 49%. With the addition of a wash solvent, the separation efficiency improved to 56-68% for a wash solvent/feed slurry ratio of 0.1 to 0.6.

The conclusion was that hydroclone separation is not suitable in the SRC-I Process for two basic reasons: the overflow clarity contains too much ash and the underflow ash concentration is too dilute; i.e., one cannot afford to gasify this stream directly.

Centrifuge Tests

Two different types of centrifuges were tested at Wilsonville—a Sharples P-850 solid bowl conveyor and a Dorr-Oliver Merco PC-9 nozzle-disc centrifuge.³ These centrifuges were operated in series (see Figure 3). The overflow from the Sharples centrifuge was the feed to the Merco centrifuge. (See Table 3 for summary of solids recovery efficiencies and overflow ash content in SRC from both centrifuges—and Figure 4 for the effect of the feed rate on solids recovery efficiency of both centrifuges.²) The Sharples centrifuge efficiencies in August-September 1977 were lower than those in June-July due to lower feed temperatures. (See Figure 5 for the effect of feed rate on overflow ash content of SRC.)

Centrifuge Tests with the Addition of an Anti-Solvent

In early 1978 tests with an AS addition to the reaction product did improve the separation performance of the Sharples centrifuge.⁴ (See Table 4 for the data for centrifuge tests.)

At a total feed rate of 1250 lb/hr and a wash solvent input of 0.3 lb/lb of reaction product (RP), the ash in SRC was 0.79% compared to 1.67% for reaction product only. An (AS) addition further improved overflow clarity. For an AS/RP ratio of 0.3 at a feed rate of 1100 lb/hr, 0.2% ash in the SRC was produced. For an AS/RP ratio of 0.4 and a feed rate of 2100 lb/hr, less than 0.1% ash in the SRC was produced. Mineral residue separation was considerably poorer at AS/RP ratios below 0.25.

Without an AS addition, the low feed rates necessary to achieve 0.16% ash in the SRC with either type of centrifuge makes centrifugation economically unattractive. With an AS addition, the process would be somewhat similar to Lummus' Anti-Solvent Deashing Process but using a centrifuge to effect the separation instead of a gravity settler. No detailed economic comparison between Lummus and anti-solvent centrifugal separation is available but it would be difficult to visualize a significant economic incentive for the centrifuge system. From a mechanical, maintenance, and operating standpoint, the gravity settler would appear to be a better choice.

THE FILTRATION TECHNIQUE

Filtration as a solid/liquid separation technique for coal liquids has been more extensively studied and tested than any other method. Here the emphasis is on the more recent filtration test results and plans at Wilsonville, although the earlier filtration work is reviewed. (See Table 5 for the various filters which have been tested in SRC-I.)

Rotary Drum Pressure Precoat Filtration

A comprehensive review of rotary drum pressure precoat filtration was presented recently by Smith.⁵ (See Figure 6 for the basic method of operation.) The drum is prepared with a 4-6 inch layer of precoat. During the filtration portion of the cycle, the drum is submerged in the filter feed slurry. As the drum rotates, a thin layer of solids is built up on the precoat. As the drum rotates out of the liquid, additional solvent and SRC are displaced by hot nitrogen passing through the cake. The filtrate and nitrogen pass through the precoat

layer into the drum and then to a filtrate receiver where the gas is separated, recompressed, and recycled. The cake solids can be washed with solvent by spray nozzles and then discharged by a doctor knife blade. However, to remove the solid residue from the filter housing, it is necessary to reslurry with solvent.

Operating Results with the Goslin-Birmingham Rotary Filters. Two rotary precoat filters (A & B) built by Goslin-Birmingham were installed at Fort Lewis in 1976. (See Figure 7 for a schematic flow diagram for the "B" filter installation.) A detailed experimental program was carried out on the "B" filter (41.6 ft²).⁶ (See Table 6 for some representative data from this test period.) The key parameters are knife advance rates, drum submergence, drum speed, and pressure drop.

(a) Knife Advance Rate. Rates of 1, 2 and 3 mil/min were tested at 1.75 rpm drum speed. The filtration rate increased linearly with the knife advance. This indicates that more of the solids penetrating the precoat are removed as the knife advance rate is increased.

(b) Drum Submergence. The total filtration rate increases with increased drum submergence but the filtrate rate per unit area submerged decreases as the submergence is increased.

(c) Drum Speed. For a given knife advance rate, the highest filtration rate will occur at the highest drum speed. At a given drum speed, filtration rates may be increased by increasing the knife advance rate.

(d) Pressure Drop. The filtration rate increased with increasing pressure drop (30 to 40 psi) but less than proportionally.

Johns-Manville/Stearns-Roger Rotary Precoat Filter. In 1977 the Electric Power Research Institute (EPRI) sponsored the construction of a 50 ft² rotary pressure filter by Johns-Manville and Stearns-Roger. Many innovative features were included in this filter.⁵ It was installed at Fort Lewis and operated from May-August 1978 (see Figure 8).

Many mechanical and process problems were encountered with the filter, as well as with upstream and downstream equipment, which severely limited the actual operating time of the filter. A few of the problems were due to operator inexperience but most indicated the need for design modifications (see Table 7).

Very limited data at 5 rpm tended to support high rates (200 lb/hr/ft²) obtained in bench scale tests by Johns-Manville.⁷ Most of the previous work had emphasized high filtration rates but solvent washing to remove SRC and the ability to discharge a dry cake would have required a significant developmental effort. Due to the limited operating time in the SRC-I mode at Fort Lewis and a commitment to test the Lummus Anti-Solvent Deashing Process there, DOE decided not to authorize filter modifications nor additional tests.

However, even if the mechanical problems could have been resolved and the high filtration rates achieved, the expense of the rotary drum pressure precoat filter (\$1280/ft²), its small size (300 ft²), and considerable operating costs would make it economically unattractive compared to other separation techniques, including other filters.

The Funda Filter

The Funda filter was installed at Wilsonville in 1974 and completed 8879 cycles before it was removed in August 1978. (See Figure 9 for a schematic flowsheet of its operation.⁸) The Funda filter is a horizontal leaf filter which contained 37 screens (100 ft²) on a 1.6 inch spacing. The filter operated at 500 to 580°F and 150 to 200 psig with a pressure drop of 15 to 65 psi.

Batch filtration was automatically controlled by a programmer with the following steps:

- (a) Precoating
- (b) Filtration
- (c) Washing
- (d) Cake blowing
- (e) Depressurization
- (f) Vacuum flash drying
- (g) Cake discharging
- (h) Repressurization

The Funda filter operation has recently been reviewed in depth. (See Table 8 for the large variety of precoats evaluated at Wilsonville.) Different coals were also tested to determine specific cake resistance. (See Figure 10 for the effect of coal type and conversion on the cycle filtration rate.)

Screen blinding was a problem that was never completely resolved. Filtration rates were higher at the start of a run but then decreased with the increasing number of cycles, even with a precoat (see Table 9).¹⁰ The Funda filter did not have a positive system for cake discharge but relied on centrifugal force (leaf rotation at 400 rpm) to remove the sticky SRC solids. Furthermore, it was not equipped to clean the screens in situ.

Basically, the Funda filter performed quite well as a pilot plant filter but it had three major limitations which made it unsuitable for scale-up to commercial size:

- (1) It did not have a means for positive cake discharge
- (2) Its method of cake discharge (high speed leaf rotation) was a severe mechanical risk in a large filter (3000 ft²)
- (3) Since it filtered on one side of a leaf only, it was more expensive than a vertical leaf filter

The U.S. Filter

The U.S. Filter (see Figure 11) was installed at Wilsonville in August 1978. Its selection as a successor to the Funda filter was based on these considerations:

- (a) U.S. Filter had successfully filtered coke breeze from tar/ pitch at similar temperatures but lower pressure.
- (b) It had a positive means of cake discharge—a doctor knife blade.
- (c) High rotational speeds for cake discharge were not required.
- (d) The filter could be scaled-up to 3000-5000 ft².
- (e) Since both sides of the leaves were available for filtration, a smaller filter volume/ area was necessary.
- (f) Substantial cost reductions compared to Funda were expected.

U.S. Filter Pressure Leaf Construction. (See Figure 12 for a schematic of the leaf construction used for the tests at Wilsonville in November-December 1978.) The seven-ply construction consisted of a center tubular slit screen, a 21% open area perforated plate (3/32 inch dia holes on 3/16 inch center-to-center), and a fine mesh outer screen. Three different outer screens were used. (See Table 10 for a summary of results of the tests.)

What no one realized at the time of the tests was that diffusion bonding of the entire leaf package was a new and untried technique. Diffusion bonding was successful in effectively sealing the leaf at the hub and periphery but undulations were present in the leaf surface. This necessitated moving the knife back to within 1/8 inch of the high spots. As a

result, more cake was retained on the leaf after cake discharge, perhaps as much as $\frac{3}{8}$ inch in some areas.

Successful tests on the 80 X 80 mesh and 60 X 60 mesh screen in the precoat mode formed the basis for the values of specific cake resistance, $\alpha = 5.6 \times 10^{11}$ ft/lb_m and media resistance, $R_m = 3.1 \times 10^{11}$ ft⁻¹ used in the Phase 0 design.

Tests without precoat on an extremely fine Dutch twill (94 X 720) showed rapid binding and plugging in 15 filter cycles. Modifications to the U.S. Filter (see Table 11) were proposed in January-February 1979 and were implemented over a three-month period. These included the design and installation of a modified sluice system (see Figure 13). This sluice system was successfully tested at low pressure during a one-week test in May 1979. At that time, DOE authorized the installation of a high pressure pump and associated piping to provide sluicing pressures of up to 2000 psi.

Modifications were also made to the filter leaves for the May test period but the leaves were not acceptable due primarily to problems associated with the diffusion bonding furnaces at Michigan Dynamics. As a result, two new sets of leaves were constructed. For these leaves, the outer two- or three-ply laminate was diffusion bonded and then spot welded to the leaf substructure.

Filter Screen Selection. A 24 X 110 Dutch twill wire cloth calendered on one side to an aperture of 83 μ m was selected, based on lab scale tests at Wilsonville which showed that a filter cake could be formed on this screen with desired clarity.¹¹ Thus, this outer screen was suitable with or without precoat. Two sets of four leaves were prepared by U.S. Filter for the October-December 1979 test period. The leaf construction was as follows:

Set #1	Set #2
Tubular slit screen	Tubular slit screen
8 X 8 mesh spacer screen	8 X 8 mesh spacer screen
Three-ply laminate	Two-ply laminate
24 X 110 Dutch twill calendered one side to 83 μ m	24 X 110 Dutch twill calendered one side to 83 μ m
16 X 16 mesh calendered screen	6 X 6 mesh calendered screen
40% open area perforated plate	

U.S. Filter Test Program. (See Table 12 for the objectives for the October-December 1979 U.S. Filter tests at Wilsonville and Figure 14 for a contingency plan for accomplishing these objectives.) All of the objectives in the current plan relate to increasing the cycle filtration rate by reducing filter downtime. However, a large body of recent data has shown that body feed addition (0.5 lb/lb solids) has increased filtration rates by a factor of 3-6.¹²⁻¹⁴ It is expected to include body feed capability at Wilsonville early in 1980.

From an operating cost standpoint, eliminating precoat from the filter cycle and optimizing the sluice variables can reduce costs by \$9.6 MM for the 6000 TPD Demonstration Plant (see Table 13).

The Candle Filter

Late in 1978 it was learned that a candle filter, developed specifically for SRC-like materials by Bergbau Forschung in Essen and Boll and Kirch in Koln, was available. En

couraging results based on preliminary screening tests were reported to APCI in February 1979.¹² A consensus of attendees at the 1979 Conference on Filtration of Coal Liquids, sponsored by Air Products, agreed that the candle filter was best suited for implementing the filtration of SRC.

A hot SRC test (200°C) at Bergbau in May 1979 on a 0.21 m² BBK candle filter indicated high cycle filtration rates (5.4-5.6 gal/hr/ft²) with the use of inert body feed. (See Figure 15 for the filter cycles.) Based on these results, an optimized cycle filtration rate of 10 gal/hr/ft² is expected. For the 6000 TPD Demonstration Plant it is anticipated that filtration area requirements can be reduced 60-70% with a similar reduction in capital cost.

In May 1979 Industrial Filter and Pump Company (IFPCO) agreed to supply a 6 ft² candle filter with vibration discharge for Wilsonville with EPRI and DOE concurrence. Also, a 0.75 m² BBK candle filter has been constructed for online testing at Wilsonville. The installation and testing of the IFPCO candle filter began in April 1980.

Cake Discharge from BBK Candle Filter. Cake discharge from the Bergbau/Boll and Kirch (BBK) filter is described briefly here (see Figure 16). The candle filter consists of cylindrical tubular wedge wire elements. Each element is subjected to a rapid downward impulse which moves the element 1-2 mm. The impulse is removed and the element moves rapidly back to its original position. The motion shears the cake at the element surface. After this cycle is repeated a number of times, the cake breaks off the element in 1-2 inch pieces and discharges through the bottom of the filter. This method of cake discharge is so effective with SRC filter cake from Kentucky #9 coal that one can see indentations of the wedge wire on the inside annular surface after cake discharge. The advantage of this method is that sluicing of the elements is not required.

U.S. FILTER/CANDLE FILTER COMPARISON

Here are the major differences between filtration on a vertical leaf filter (U.S. Filter) and a candle filter (BBK and/or IFPCO) (see Table 14).

Filter Media

The construction of a seven or nine-ply screen for U.S. Filter is discussed above. Due to the large internal surface area, there is a potential for long-term blinding even if the sluicing system successfully keeps the outer leaf clean.

The two sets of leaves constructed in August 1979 were flatter with less distortion than any other previous set of screens. However, the leaves were still not perfectly flat but were slightly concave/convex, about 1/16-1/8 inch/radial ft.

The candle filter medium is a single-ply construction consisting of a cylindrical, spirally-wound wedge wire with a trapezoidal slot. The slot width (100 μ m) is narrowest at the surface and increases in the direction of filtration (see Figure 17). Thus, a small particle will tend to pass through the flow channel and not get lodged in the trapezoidal opening. This construction reduces blinding.¹⁵

Cake Discharge

The effectiveness of cake discharge in SRC filtration is as important as the filter media in determining long-term successful filter operation. Filter cake is discharged from the U.S.

Filter by leaf rotation at 1 rpm past a doctor knife blade. To prevent metal-to-metal contact between the outer fine mesh wire cloth and the knife, the doctor blade is set back 1/8 inch from the highest spot. Therefore, a flat, rigid leaf is necessary to minimize the thickness of the filter cake remaining after cake discharge.

Cake discharge by inertial impact from the BBK filter results in a more effective, more complete cake discharge.

No Precoat Operation

For the U.S. Filter, successful operation without precoat had not yet been demonstrated as of November 1979. However, it was included as one of the objectives of the recent filter test program at Wilsonville.

A no-precoat operation is the "modus operandi" of the candle filter and this was successfully tested with the BBK filter at Bergbau in May 1979. Eliminating precoat significantly simplifies the filter cycle and cuts 15-20 minutes from the downtime portion of the cycle.

Mechanical Comparison

The candle filter is a much simpler filter from a mechanical standpoint. It has no knife blade discharge, high pressure sluice system, or screw conveyor. Furthermore, the candle filter does not require construction of large, rigid, and flat multilayered screens, as does the U.S. Filter.

Economic Comparison

Based on competitive bids, vertical leaf filters were 20-40% more expensive than candle filters on a \$/ft² basis.

Process Comparison

For a constant feed of a concentration of solids, cake thickness is proportional to the volume filtered on a leaf filter. For a cylindrical element, the surface area increases during filtration. As a result, cake thickness increases less rapidly (see Figure 18). Hence, for a given volume filtered, the cake is thinner on the candle filter, or for the same cake thickness the candle filter has a greater throughput. This is particularly significant for cake thicknesses greater than 1 inch.

Also, since the surface area for filtration increases on the tubular element, the instantaneous filtration rate drops off more slowly for the candle filter (see Figure 19).

Effect of Filtration Improvements on Filter Downtime

(See Table 15 for the sequence of operations for the U.S. Filter at Wilsonville.) By reducing the sluicing time and filling and draining more rapidly, the base case for the U.S. Filter in the Phase 0 design was reduced to 81 minutes.

(See Figure 20 for specific changes in the filter sequence which are necessary to reduce downtime from 81 minutes to 39.5 minutes for the leaf filter and 32 minutes for the candle filter.)

Effect of Downtime on Cycle Filtration Rates

A comparison of cycle filtration rates for both the leaf and candle filter has been made using the values of specific cake resistance ($\alpha = 5.6 \times 10^{11}$ ft/lb_m) and medium resistance ($R_m = 3.1 \times 10^{11}$ ft⁻¹) determined from U.S. Filter test data at Wilsonville in November-December 1978 (see Figure 21—and Figures 22 and 23 for results). For the leaf filter, the basis for the Phase 0 Design was for a downtime of 81 minutes. This corresponds to a cycle filtration rate of about 3 gal/hr/ft². If precoat can be eliminated from the filter cycle, downtime can be reduced to 40 minutes and the cycle filtration increased to 4.2 gal/hr/ft².

For the candle filter, downtime could be reduced to as low as 32 minutes. A cycle filtration rate of about 5.5 gal/hr/ft² would then be predicted. This is a substantial increase from the Phase 0 Design and higher than the best projections for the U.S. Filter.

Body Feed Increases Filtration Rate

Recent data by Bergbau, Industrial Filter, and the National Coal Board have shown increases in SRC filtration rates by a factor of 3-6 with inert and carbonaceous filter aids.¹²⁻¹⁴ To be economically attractive, an inexpensive filter aid based on coal must be available. Nerofil (Great Lakes Carbon) and a proprietary carbon filter aid have performed well in laboratory-scale tests, have a desirable pore structure, and are being evaluated as part of the candle filter test program at Wilsonville (see Figures 24 and 25).

LUMMUS ANTI-SOLVENT DEASHING

The application of the Lummus Anti-Solvent Deashing Process to Solvent Refined Coal has been reported recently.¹⁶⁻¹⁸ These studies were based on laboratory-scale testing in a 0.006 cu m gravity settler, and these test results were used as a design basis for the 50 TPD SRC-I Pilot Plant at Fort Lewis.

Work on the Fort Lewis Deashing Unit was started in March 1977. Construction began in November 1977 and mechanical completion was accomplished in August 1978. The first test at Fort Lewis was started in April 1979. A second run began on 15 June and ended 7 July. A 30-day continuous run has also been completed (21 July-20 August). The Lummus Unit is now shut down during operation at Fort Lewis in the SRC-II mode. The next run period has tentatively been set for April-May 1980.

Test Results from Fort Lewis

(See Figure 26 for a schematic flow diagram for the Lummus Anti-Solvent Process.) The dissolver effluent passes to the high and low pressure separator where light gases, including unreacted hydrogen and H₂S, are removed. The slurry is sent to a fractionator

to remove light liquids with a boiling point below 500°F. The resulting slurry is sent to a feed surge drum. The feed consists of process solvent (500°-850°F), SRC, mineral residue, and unconverted coal. (See Table 16 for a typical feed composition.)

The feed is mixed in-line with anti-solvent and distributed within the gravity settler to each of the settling chambers. Each chamber consists of a section of an inclined conical surface. The heavy phase flows to an underflow outlet pipe. The clear, light, liquid phase flows to an overflow collection pipe.

The overflow is sent to an anti-solvent fractionator to recover anti-solvent. The anti-solvent overflow (ASOF) is sent to a clean column to recover process solvent for recycle. SRC from the clean vacuum column is cooled and solidified.

The settler underflow is sent to an underflow stripper to recover anti-solvent (ASUF). The stripper bottoms are sent to a vacuum unit to recover process solvent. The vacuum bottoms containing up to 40 wt % solids is pumped to a gasifier.

The initial conditions selected for startup of the Lummus unit were the full design AS flow but only 50% of the SRC slurry feed. The larger AS/Feed ratio caused excessive precipitation of the marginally soluble SRC components. This caused plugging of the underflow lines and accumulation of solids in the settler. An ash balance around the settler also confirmed an accumulation of solids. The unit was shut down for cleaning.

A 30-day continuous test (Run #3) was carried out from 21 July-20 August 1979. (See Figure 27 for preliminary results for the ash content of SRC from the anti-solvent free settler overflow.) The results indicate that the ash specification (0.16 wt %) was met about one-third of the time.

(See Figure 28 for the recovery of SRC for Run #3.) The data indicate a sporadic operation. Part of this was due to day-to-day operating changes. First, an attempt was made to reduce ash in the anti-solvent free overflow to less than 0.05 wt % (this corresponds approximately to 0.16 wt % ash in SRC). When a low ash overflow was obtained, the overflow/underflow split was increased from 50/50 to 60/40 and then to 65/35. It was apparent that the OF/UF ratio is an important parameter in the stability of the process. A satisfactory, sustained operation has not yet been achieved for splits greater than 65/35. Upsets have repeatedly occurred at this value forcing a reduction in SRC recovery.

More data from Fort Lewis are needed with acceptably low ash in the overflow and with a consistent SRC recovery before claims of SRC recoveries above 70% can be substantiated. The data collected thus far support an average SRC recovery of 65% and that is uneconomic compared to filtration (U.S. Filter or candle filter) or the Kerr-McGee Critical Solvent Deashing Process.

KERR-McGEE CRITICAL SOLVENT DEASHING

CSD is a continuous deashing process for separating solids from coal liquids. Unlike the Lummus process which is an agglomerative gravity sedimentation process, Kerr-McGee CSD is a solvent extraction process operated near the critical point of the deashing solvent in which two liquid phases form rapidly.¹⁹

(See Figure 29 for a schematic flow diagram of the Kerr-McGee CSD Process.) Dissolver effluent is let down to remove light gases and then fractionated to remove almost all of the process solvent. The feed slurry to the CSD unit is a vacuum bottoms product with 3-10% process solvent.

The vacuum bottoms product is mixed in-line with deashing solvent at temperature-pressure conditions near the critical point of the deashing solvent and fed to the first stage

settler. The overflow light liquid phase contains deashing solvent, soluble components of the SRC, and less than 0.1 wt % ash. The underflow heavy liquid phase contains deashing solvent, unextracted portions of SRC, ash, and unconverted carbon.

The overflow is heated above the critical point of the deashing solvent to reject the SRC. This can be done in one or more stages to separate lighter SRC (LSRC) components from the heavier, less soluble components. LSRC typically contains 25-50% process solvent and has been found to be effective for dissolving coal.¹⁹⁻²⁰ The SRC product is cooled and solidified.

The underflow from the first stage settler is flashed to recover deashing solvent. The ash, unconverted carbon, and SRC form a finely divided powder. The ash concentrate is then fed to a gasifier to convert the ash to an environmentally acceptable slag and to generate hydrogen for coal liquefaction.

Wilsonville Test Results

A Kerr-McGee CSD unit was installed at Wilsonville in June 1978. The experimental program began in August 1978. The unit has effectively deashed Indiana #5, Kentucky #6 and #11, and Kentucky #9 coals with similar results.

SRC Recovery. SRC recovery depends on conditions in the dissolver. Dissolver parameters that increase the solubility of vacuum bottoms are: residence time, pressure, and solids concentration in the dissolver (see Table 17 and Figure 30 for summary of results). Increasing residence time (lower space rate) in the dissolver generally increases SRC recovery, as does an increase in dissolver pressure. The effect of solids withdrawal from the dissolver on SRC recovery is particularly significant at 1700 psig. SRC recovery seems to be less susceptible to low solids concentration at 2100 psig.

(See Figure 31 for typical operating results during a 12-day run with the CSD unit.) Usually within two days the unit is lined out and day-to-day variations in SRC recovery are quite small. The low SRC recovery initially is attributed to the presence of insoluble material present in the system at start-up. It also takes about two days for an equilibrium solids concentration to be achieved in the dissolver.

Although there is some variation in the ash content of SRC, all the SRC produced in Run 160 was within the 0.16 wt % ash specification.

Due to an improved SRC recovery at 2100 psig at a coal space rate of 38 lb/hr/ft³, these conditions were selected as a design basis for Phase 0. An SRC recovery of 81% is anticipated.

SRC can be subdivided into three classes of compounds by solubility (see Table 18): oils, asphaltenes, and preasphaltenes. The deashing solvent dissolves most of the oils and asphaltenes but only a portion of the preasphaltenes. Typically about 90% of the oils and asphaltenes are extracted but only 25-60% of the preasphaltene fraction. Therefore, conditions in the dissolver which reduce the concentration and molecular weight of preasphaltenes will yield a higher SRC recovery in the CSD unit. (See Table 19 for a typical material balance for feeds of high and low solubility.)

Deashing Solvent Recovery. Deashing solvent is unlikely to be coal-derived. Therefore, recovery of deashing solvent from the ash concentrate is important. From an economic standpoint, it is desirable to reduce residual deashing solvent levels to less than 1 wt %. Due to the finely divided nature of the particulate, a fluidized bed dryer is not appropriate. Dryers with longer residence time are being evaluated and plans are for this type of dryer to be installed and tested at Wilsonville in 1980, if necessary.

TWO VIABLE CANDIDATES

The Kerr-McGee Critical Solvent Deashing process and filtration with a candle or leaf filter are both viable candidates as deashing options for the SRC-I 6000 TPD Demonstration Plant. A recent economic comparison of the Kerr-McGee process at an 81% SRC recovery was a standoff against 12 5000 ft² U.S. Filters (95% SRC recovery).⁹ The higher capital costs for filtration were balanced by the higher losses of SRC in the Kerr-McGee Process.

The Lummus Anti-Solvent Deashing Process is at an earlier stage of process development but at a larger operating scale. It has not yet demonstrated sustained operation simultaneously producing (a) an acceptably low ash in the overflow; and (b) a high (70%) SRC recovery. This data would be required before the Lummus Process could be seriously considered as a design option for deashing in the 6000 TPD Demonstration Plant.

A preliminary cost estimate indicates capital costs for Lummus are similar to filtration (U. S. Filter) but the lower SRC recovery is a significant economic penalty.

The other mechanical methods, such as the rotary drum and horizontal leaf filter and the centrifugal techniques, have been eliminated from consideration due to one or more of the following problems: high capital and operating costs, low mechanical reliability, inability to meet process ash specifications, production of a dilute underflow, or inability to scale-up to commercial size.

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Table 1
Summary of Hydroclone Tests
May 1975

	Run #1	Run #2	Run #3
Operating Conditions			
Pressure drop, psi	73	76	95
Temperature, °F	410	450	475
Flow rates, lb/hr			
Feed	2100	2865	2545
Overflow	1430	2265	1755
Volume split			
Overflow/underflow	68/32	79/21	69/31
Analyses			
Feed			
Ash, wt %	1.4-1.5	1.6-1.3	1.7-2.0
Unconverted carbon, wt %	1.2-1.3	1.3-1.3	1.4-1.6
Overflow			
Ash, wt %	0.45-0.63	0.46-0.44	0.46-0.52
Unconverted carbon, wt %	0.45-0.67	0.44-0.46	0.44-0.38
Underflow			
Ash, wt %	4.3-3.4	5.8-	5.1-5.3
Unconverted carbon, wt %	3.2-2.8	4.7-	4.5-4.2
Ash removal from feed, %	77	77.5	81
Carbon removal from feed, %	69.5	72.5	81
Hydroclone efficiency, %			
Ash removal	63	69	74
Carbon removal	56	66	76

Table 2

Hydroclone Operating Data
January 1978
Indiana #5 Coal

Test #	Total feed, gpm	r ¹	Feed temp, °F	Pressure drop, psi		Overflow, % feed	Feed ⁴		Overflow			SRC		Overflow viscosity, ⁵ cP
				p ²	p ³		Cl, %	Ash, %	Cl, %	Ash, %	SRC, %	Ash, %	S, %	
1	3.5	-	500	50	40	70	7.0	4.8	2.44	1.68	28.6	5.4	-	-
2	3.4	0.33	550	49	40	64	6.1	3.76	2.98	1.97	19.7	8.68	-	2.1
2	3.5	0.3	545	51	42	86	3.9	2.7	1.84	1.20	13.6	7.77	1.53	2.0
3	3.4	0.32	547	49	41	83	4.22	2.88	2.36	1.60	12.7	10.62	1.60	1.8
4	3.6	0.32	545	50	40	77	4.44	3.08	2.28	1.57	15.9	8.63	1.38	1.9
5	3.8	0.32	548	50	40	75	4.8	3.28	2.66	1.74	16.8	8.94	1.79	1.8
6	2.7	0.66	548	50	43	74	4.96	3.47	1.12	0.77	13.1	5.41	-	0.8
7	3.2	0.50	550	50	40	67	4.15	2.87	0.89	0.61	11.9	4.77	-	-
8	2.7	0.66	532	50	40	79	4.81	3.35	0.51	0.34	8.2	4.53	0.87	0.9
9	2.8	0.63	550	50	40	71	3.62	2.49	0.16	0.11	14.3	0.76	-	1.1
10	4.3	1.11	530	48	40	55	2.87	1.78	0.14	0.09	16.6	0.58	0.68	-

1. Ratio of anti-solvent to filter feed by weight.
2. Pressure drop across feed and overflow.
3. Pressure drop across feed and underflow.
4. After mixing with anti-solvent.
5. At 400°F.
6. Wash solvent used in place of Soltrol 130.

Table 3

Centrifuge Performance Summary

Date (1977)	Sharples centrifuge			Dorr-Oliver centrifuge		
	Feed rate, gpm	Recovery, efficiency, %	Ash in SRC, %	Feed rate, gpm	Recovery, efficiency, %	Ash in SRC, %
27 July	2	95.1	0.5	-	-	-
28 July	2.2	96	0.83	-	-	-
26 Aug	3.6	83.6	2.76	4.2	23.5	3.9
27 Aug	4.3	79.6	-	4.2	37	-
27 Aug	3.6	78.7	-	-	-	-
28 Aug	3.1	82.6	3.42	4	24.8	1.54
31 Aug	3.2	90.5	0.7	4.6	25.1	0.76
1 Sept	2.5	83.9	2.38	3.4	44.5	0.32
1 Sept	3.1	86.6	-	2.2	61.2	0.38
1 Sept	3.3	84	-	2.3	73.4	0.81
2 Sept	1.9	91.3	-	2.3	41.9	-

Table 4

Centrifuge Operating Data
Indiana #5 Coal

Date 1978	Rates, gpm		Underflow, wt % of feed	VIII feed				Total feed		Overflow			
	VIII Deashing feed	solvent		Cl, %	Ash, %	SRC, %	<450°F, %	Cl, %	Ash, %	Cl, %	Ash, %	SRC, %	<450°F, %
Jan													
28	2	0.7	9	-	-	-	-	2.74	1.66	0.2	-	15.4	29.16
29	1.6	0.5	7	3.7	2.6	17.9	-	2.61	1.8	0.2	0.1	18.5	21.03
30	2.6	0.9	8	5.19	3.67	20.5	3.32	4.94	3.4	0.23	0.1	15.8	21.39
30	1.7	0.6	13	7.44	5.22	22.2	10.59	5.55	3.88	0.12	-	14.3	34.9
31	2	1.3	17	6.4	4.42	20.1	3.42	5.36	3.7	0.35	0.22	18.8	12.78
Feb													
1	2.9	1.7	18	7.54	5.36	21.4	3.68	5.64	3.9	0.38	0.24	16.6	22.3
2	1.7	0.6	24	6.8	4.8	20.1	5	6.1	4.3	0.19	0.11	19	40.4
2	1.2	0.9	19	6.2	4.4	21.2	7	5.1	3.5	0.04	0.01	13.8	38.7
3	2.3	0.5	18	6.3	4.4	20.2	4.7	5.6	3.9	0.1	0.03	17.2	33.8
3	1.7	1.3	25	6.6	4.6	22.2	4.7	4.4	3	>0.01	-	12.2	9.8
Mar													
8	2.5	1.1	21	7.6	5	25.8	15	6	3.7	0.1	-	26.8	18.3
8	1.3	0.7	17	8	5.1	27.6	4.1	5	3.2	0.02	-	21.6	19
9	2.3	1	17	6.9	4.5	21.9	2.1	5.6	3.5	0.04	-	25.5	28.4
9	4.7	2.4	21	7	4.5	25.8	2.2	4.6	2.8	0.05	-	19.4	31.8
9	6.3	3.2	14	6.9	4.5	23.8	3.2	5.6	3.5	0.08	0.04	17.8	28.9
10	4.5	2.9	15	7.2	4.8	22.3	2.7	5.7	3.6	0.04	-	13	46.9
11	4	3.2	25	8	5.2	19.3	2.5	2.8	1.8	0.02	-	-	48
11	4.2	2.9	18	7.5	4.9	25.5	1.2	5.3	3.5	0.04	0.013	18.3	45.6
11	5.1	2.1	24	7.5	4.9	22.4	2.6	6.4	4	0.63	0.41	23.5	29.6
12	2.8	1.8	18	7.5	4.8	21.6	5.2	5.5	3.5	0.05	-	20.4	36.1
12	3.4	1.4	21	8	5.1	23.5	3.4	6.3	4	0.26	0.16	20.4	35.7
12	2	1.3	21	7.7	4.9	23.3	3.3	6.1	3.9	0.17	-	21.3	21
13	2.4	1	22	-	-	-	3	6.4	4	0.19	0.12	23.3	22.7
13	5.1	2.1	24	6.5	4.2	-	-	4	2.5	0.39	0.25	23.7	29.4
13	4.6	2.5	16	7.1	3.3	23.1	3.8	5.9	3.7	0.34	0.21	18.5	20

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Table 5

Filters Tested With SRC-I

Filter type	Manufacturer	Data source	Dates tested
Rotary drum precoat	Goslin-Birmingham	Fort Lewis	1976-1978
Rotary drum precoat	Johns-Manville/ Stearns-Roger	Fort Lewis	May-Aug 1978
Horizontal leaf (Funda)	Chemap	Wilsonville	1975-June 1978
Vertical leaf	U.S. Filter	Wilsonville	Nov 1978-Feb 1980
Candle filter	Industrial Filter	Wilsonville	Mar 1980-present
Candle filter	Bergbau/Boll-Kirch	Wilsonville	TBD-present

Table 6

Goslin-Birmingham Rotary Drum Tests at Fort Lewis

Filter Test Run Operating Conditions

Run 6 12/16 and 17/76

Constant conditions				
Basecoat: 10 lb 11-C Precoat: 500 lb Speedex Filter feed temperature: 525°F Filter temperature: 490°F Slurry feed rate: 17,000 lb/hr Pyridine insolubles in filter feed: 7.44% 30% SRC Filter feed viscosity: 19.7 cS @ 210°F				
Variable conditions and results:				
	Run period			
	1	2	3	4
Duration, hours	8	8	7	4
Vat pressure, psig	111	111	111	111
Delta P, psi	30.5	30.5	30.5	30.5
Drum submergence				
% diameter	29.6	29.6	29.6	29.6
% area ¹	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	0.7	1	1.4
Cake wash, lb/hr	870	950	940	900
Gas rate, lb/hr	530	580	1115	<1100
Filtrate rate, ² lb/hr	5900	5500	6800	5000
% P.I.	trace	trace	trace	trace
% ash	0.03	0.03	0.03	0.03
Viscosity @ 100°F, cS	317	280	117	983

1. Total screen area = 41.6 ft².

2. Includes cake wash.

Table 7

Modifications to JM/SR
Rotary Pressure Precoat Filter

-
1. Add supports to prevent screens from sagging
 2. Ensure safety of sight glass
 3. Modify design of packing glands
 4. Modify knife edge advance drive; provide electrical interlock and reduce speed of high speed knife advance
 5. Modify drum drive to 4-speed (2.5, 5, 7.5, 10 rpm) in lieu of variable speed drive
 6. Eliminate leakage in main flange
 7. Modify sluicing system to prevent base coat precoat from separating from the drum in large chunks
 8. Add fogging nozzles to speed up cooldown of filter
-

Table 8

Precoats for SRC Filtration at Wilsonville

Coal type	Precoat	Precoat rate (lb/ft ²)
Illinois #6	Hyflo Supercel	0.09-0.18
Emery	Hyflo Supercel	0.26
Illinois #6, Indiana #5, Emery	Celite 560	0.23-0.45
Illinois #6, Kentucky #9 and 14, Pittsburgh #8	Fiberflo 7-C	0.25-0.27
Amax, Emery, Indiana #5 Kentucky #6 and 11		0.25-0.45
Emery	Fiberflo 7-C and Celite 560	0.30-0.67
Indiana #5	Fiberflo 7-C and filter cell (25%)	0.48
Indiana #5	Celite 560 and asbestos	0.30-0.36
Indiana #5	Perlite	0.23-0.36
Indiana #5	Perlite and asbestos	0.23
Indiana #5	Flyash	0.63-0.8
Indiana #5	Flyash and asbestos	0.63-0.79
Indiana #5	Perlite 4156	0.32-0.63
Indiana #5, Kentucky #9	Perlite 436	0.46-0.79
		0.22-0.33
Indiana #5	Solka-Floc and Hyflo Supercel	-

Table 9

Funda Horizontal Pressure Leaf Filter
Fibra Flo 7-C Precoat at 0.2-0.5 lb/ft²

Coal	Year	Area, ft ²	No. of cycles		Cycle rate, lb/hr/ft ²		
			Total	Longest ¹ continuous	Start	End	Optimum ²
Kentucky #9 and 14	1976	54	1589	401 ³	26	19	28
Monterey	1976	100	1017	454 ⁴	24	20	25
Illinois #6				351	22	17	
Amax Belle AYR	1976-	100	773	235	15	16	35
	1977	54 ⁵		124		30	
Emery	1977	54 ⁶	806	214	33	20	39
Indiana #5	1977-	100	1861	194	18	13	27
	1978			103	18	18 ⁷	
			6046				

1. Without cleaning.
2. Delta P = 60 psi, filtration time = downtime = 50 minutes, base conditions: clean screens.
3. Filter not drained after precoating; rate data does not include precoat solvent filtered.
4. Celite 560 used as precoat during several test periods.
5. 3-inch spacing instead of usual 1.5-inch spacing.
6. Precoat tank filtered regularly to clean.

Table 10

U.S. Filter Tests at Wilsonville
Nov-Dec 1978

Screens tested	Precoat	Grade
80 x 80 mesh	Yes	Fibre-Flo
60 x 60 mesh	Yes	Perlite 436
60 x 60 mesh	No	
94 x 720 (HF-34)	No	

Demonstrated

- ° High clarity--ash content 0.015%
- ° Filter can be drained after precoating
- ° Cake retention during washing and drying
- ° Cake composition <5% SRC--98-99% SRC recovery
- ° High clarity without precoat

Problem areas

- ° Screens blind/plus readily without precoat
- ° Screen washing (sluicing) not adequate
- ° High pressure drop across leaf

Table 11

U.S. Filter Modification

<u>Screen blinding</u>	<u>Screen plugging</u>
Solids accumulation on screen surface	Solids accumulation between screen and perforated plate
<u>Remedy</u>	
<ul style="list-style-type: none"> ◦ Improved nozzle/slucice design ◦ Use coarser media ◦ Wash with clean solvent ◦ Provide flatter screen surface 	<ul style="list-style-type: none"> ◦ Use coarser outer screen ◦ Insert coarse screen between outer screen and perforated plate
<u>Evaluation of coarser media</u>	
<ul style="list-style-type: none"> ◦ 60 x 60 mesh calendered to 106µm ◦ 24 x 110 Dutch twill calendered to 78µm ◦ 24 x 110 Dutch twill calendered on one side to 83µm ◦ 30 x 150 Dutch twill--80µm ◦ 60 x 60 mesh calendered on one side 	

Table 12

U.S. Filter Test Program

1. Preliminary checkout and precoat demonstration--2 to 3 weeks

The objective is to demonstrate the operability of the U.S. Filter while using precoat and the high pressure (1500 psi) sluice system. The objectives will include meeting product clarity and producing filtration and blinding rate data. The differential pressure during filtration will be maintained at 50 psi. The filter will be shut down and opened when necessary to observe the filter screens.

2. Demonstration of no-precoat operations

If the precoat tests are successful, the no-precoat operation will be tested next. Initial tests will be with high-pressure sluice at 2000 psi and filtration differential pressure of 50 psi. Objectives for this period include:

- (a) minimize sluice pressure
- (b) maximize leaf rotation rate during sluicing
- (c) minimize wash solvent usage

It is estimated that 7 to 8 weeks will be needed to complete these optimizations. If it is obvious that the filter cannot be operated in the no-precoat mode, the objectives for this period will be to optimize performance with precoat.

Table 13

Incentives for Eliminating Precoat

° Reduce capital costs	
Eliminate tanks, pumps, bulk handling and storage facilities 0.33 x \$40 MM	13.2 MM
Add high pressure sluice pumps 8 x \$0.22 M	(-1.76) MM
Shorten downtime on filter, increase throughput	
Eliminate precoat 18/150 x \$40 MM	4.8 MM
Increase sluice time 12/150 x \$40 MM	(-3.2) MM
Net reduction in capital costs	13.0 MM
Equivalent reduction in operating cost	
\$13.0 MM (0.16 + 0.06)	2.87 MM
° Reduce operating cost	
Precoat cost (220/ton)	5.73 MM
Fractionation costs for sluice solvent recovery	(-2.54) MM
(5.83-3.0) gal/ft ² x 5000 ft ² /filter x 10 filters	
x 9.6 $\frac{\text{cycles}}{\text{day}}$ x 0.74 x 8.337 $\frac{\text{lb}}{\text{gal}}$ x 200 $\frac{\text{Btu}}{\text{lb}}$ x	
\$4.60/MM Btu x 330 $\frac{\text{day}}{\text{yr}}$	
Pumping costs for high pressure sluice	(-0.25) MM
Reduce solids loading to gasifier	0.14 MM
	3.08 MM
Minimum operating cost reduction	5.95 MM
Maximum operating cost reduction	9.64 MM

Table 14

U.S. Filter/Candle Filter Comparison

U.S. filter	Candle filter
<ul style="list-style-type: none"> ◦ <u>Filter Media</u> <ul style="list-style-type: none"> Complicated 7/9 ply screen Blinding, plugging Distortion ◦ <u>Cake discharge</u> <ul style="list-style-type: none"> Doctor blade 1/8-3/8 inch cake residue High pressure sluice ◦ <u>No precoat operation</u> <ul style="list-style-type: none"> Yet to be demonstrated ◦ <u>Mechanical comparison</u> <ul style="list-style-type: none"> Knife blade discharge Large complicated screens High pressure sluice Screw conveyor ◦ <u>Economic comparison</u> <ul style="list-style-type: none"> 30-40% more expensive/ft² 	<ul style="list-style-type: none"> Simple wedge wire construction Trapezoidal slot reduces blinding Rugged, durable construction Inertial impact/vibration Essentially complete removal No sluice required Small pilot plant tests successful

Table 15

U.S. Filter
Sequence of Operations
Precoat Mode

Operation	Current cycle	Minimum cycle	Design cycle
Sluice with filter feed	3	14 ¹	6 ²
Drain filter feed sluice	5	-	-
Sluice with precoat	3	-	-
Precoat fill completion	2	5	3
Precoat	20	20	9
Precoat drain	10	7	6
Fill with filter feed	7	5	6
Drain filter feed	10	6	9
Fill with wash solvent	6	4	9
Cake wash	11	11	17
Drain wash solvent	11	8	9
Cake blow	1	-	2
Depressure and dry	8	8	20
Discharge	10	5	7
Repressure to 130 psig	<u>3</u>	<u>3</u>	<u>2</u>
Total	110	96	105

1. Sluice with wash solvent.

2. Sluice with filtrate.

Table 16

Lummus Anti-Solvent Deashing at Fort Lewis
August 1979

Feed composition	7899 lb/hr
	Run = 3 wt %
Process solvent (bp 500°-850°F)	61.3
SRC (bp + 850°F)	32.2
Solids (ash + unconverted coal)	<u>6.5</u>
	100.0
Process solvent/SRC = 1.9	
Anti-solvent free overflow	5320 lb/hr
	wt %
Process solvent	67.2
SRC	32.6
Ash and unconverted coal	<u>0.2</u>
	100.0
Process solvent/SRC = 2.06	
Anti-solvent free underflow	2579 lb/hr
	wt %
Process solvent	49.1
SRC	31.5
Ash and unconverted coal	<u>19.4</u>
	100.0
Process solvent/SRC = 1.56	
SRC recovery = 68.1%	
OF/UF split = 67.3/32.7	

Table 17

Kerr-McGee Critical Solvent Deashing

Run #	Coal	Dissolver conditions ¹			SRC recovery ²	
		Pressure, psig	% volume	Solids concentrate	Mean	Median
143	Indiana #5	1700	100	High	81.4	81.4
147	Indiana #5	1700	100	Low	71.6	72.2
150	Kentucky #6 and 11	1700	50	High	75.9	76.8
151	Kentucky #6 and 11	2100	50	High	77.6	78
157	Kentucky #6 and 11	1700	100	High	81.9	83.2
159	Kentucky #9	1700	50	High	74.5	74.6
160	Kentucky #9	1700	75	High	78.9	80.6
161	Kentucky #9	1700	75	Low	71.6	73.1
162	Kentucky #9	2100	75	Low	83	83.4
163	Kentucky #9	1700	75	Low	78	79.5
166	Kentucky #9	2100	75	Low	78.8	80.2
166A	Kentucky #9	1700	75	Low	70.5	69.1
167	Kentucky #9	1700	75	Low	71.3	72

1. Dissolver outlet temperature 825°F.

2. % Cresol soluble in feed by forced ash balance.

Table 18

SRC Fractions Defined by Solubility

SRC component	Soluble in		
	n-Pentane	Benzene	Cresol
Oil	Yes	Yes	Yes
Asphaltene	No	Yes	Yes
Preasphaltene	No	No	Yes
Unconverted carbon	No	No	No

Table 19

SRC Recovery by Component

Soluble feed			
Run 143	Feed, wt %	Ash concentrate, wt %	SRC recovery, %
Oil	19.89	6.34	88.7
Asphaltene	36.03	5.61	94.5
Preasphaltene	20.58	19.88	60.4
Undissolved coal	6.80	22.32	
Ash	16.70	45.85	
	100.00	100.00	83.8

Ash concentrate/feed = 35.6%

Less soluble feed			
Run 147	Feed, wt %	Ash concentrate	SRC recovery, %
Oil	19.03	4.93	87.7
Asphaltene	28.48	3.55	94.1
Preasphaltene	28.29	33.63	34.3
Undissolved coal	8.47	23.42	
Ash	15.73	34.47	
	100.00	100.00	70.2

Ash concentrate/feed = 47.7%

Figure 1

Solvent Refined Coal

Figure 2
Hydroclone Schematic

Figure 3
Flow Diagram for Combined Operation
Of Solid and Disc Nozzle Bowl Centrifuges

Figure 4
Effect of Feed Rate
On Solids Recovery Efficiency of Centrifuges

Figure 5
Effect of Feed Rate
On Ash in SRC From Overflow of Centrifuges

Figure 6
Rotary Precoat Filter

Figure 7
Rotary Drum Filter Installation at Fort Lewis

Figure 8
JM/SR Rotary Precoat Filter at Fort Lewis

Figure 9
Funda Filter Installation at Wilsonville

Figure 10

Effect of Conversion and Coal Type
On Filtration Rates

Figure 11
Auto Jet Vertical Leaf Filter

Figure 12
U.S. Filter
Pressure Leaf Construction

Figure 13
U.S. Filter
Sluice System Modification

Figure 14
U.S. Filter Program Objectives

Figure 15
SRC-I Filtration on BBK Candle Filter

Figure 16
Cake Discharge
BBK Candle Filter

Figure 17
Candle Filter Construction

Figure 18
Volume Filtered vs Cake Thickness

Figure 19
Filtration Rate vs Cake Thickness

Figure 20
Effect of Filtration Improvements
On Filter Downtime

Figure 21
Filtration Data at Wilsonville

Screen blinding

Solids accumulation on
screen surface

- Improved nozzle/slucice design
- Use coarser media
- Wash with clean solvent
- Provide flatter screen surface

Screen plugging

Solids accumulation between screen
and perforated plate

Remedy

- Use coarser outer screen
- Insert coarse screen between outer screen
and perforated plate

Evaluation of Coarser Media

- 60 X 60 mesh calendered to 106 μm
- 24 X 100 Dutch twill calendered to 78 μm
- 24 X 100 Dutch twill calendered on one side to 83 μm
- 30 X 150 Dutch twill - 80 μm
- 60 X 60 mesh calendered on one side

Figure 22
Effect of Downtime
On Cycle Filtration Rates
Leaf Filter

Figure 23
Effect of Downtime
On Cycle Filtration Rates
Candle Filter

Figure 24
Nerophil

Figure 25
Proprietary
Carbon Filter
Aid

The diagram illustrates a coal liquefaction process. It begins with a 'From dissolver' input to a 'Separator'. The 'Separator' produces 'Off-gas' and 'Light oil' (top product). The bottom stream from the separator goes to a 'Light oil fractionation' column. The top product of this column is 'Recycle anti-solvent', which is recycled back to the 'Separator'. The bottom product of the 'Light oil fractionation' column goes to a 'Settler'. The 'Settler' produces an 'Overflow' stream that goes to an 'Overflow fractionation' column and an 'Underflow' stream that goes to an 'Underflow fractionation' column. The 'Overflow fractionation' column produces 'Solidified' material, which goes to a 'Solidifier' and then to 'Solvent Refined Coal'. The 'Underflow fractionation' column produces 'Fuel oil' and 'Recycle process solvent'. The 'Recycle process solvent' is recycled back to the 'Settler'. The 'Underflow' stream from the 'Settler' goes to a 'Gasifier'. The 'Gasifier' produces a 'Slurry' stream that goes to the 'Underflow fractionation' column and a 'Gas' stream that goes to a 'Gas treating' unit. The 'Gas treating' unit produces 'H₂', 'Fuel gas', and 'Low Btu gas'. The 'Gas treating' unit also produces 'Acid gas', which goes to a 'Claus plant'. The 'Claus plant' produces 'Sulfur'. The 'Gasifier' also produces a 'Solid' stream that goes to a 'Coker or LTC'. The 'Coker or LTC' produces a 'Solid' stream that is recycled back to the 'Gasifier'.

Figure 28
Lummus Anti-Solvent Deashing
SRC Recovery
Run #3

Figure 29
Schematic Flow Diagram
Kerr-McGee Critical Solvent Process

Figure 30
Kerr-McGee Critical Solvent Deashing
Effect of Coal Space Velocity
On SRC Recovery

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SURVEY TAKEN OF DOMESTIC VENDORS OF THICK-WALL PRESSURE VESSELS

B. Lubliner*
and K. L. Baumert**

The design for the SRC-I Demonstration Plant includes two large thick-wall vessels which push the state of the art for construction and handling. The vessels, coal dissolvers, have these physical and design characteristics (Kerr-McGee Case):

Diameter:	11 ft 0 inches i.d. (inside diameter)
Height:	72 ft 0 inches straight side
Material:	SA-387 Grade 22, Class 2 (2¼Cr-1 Mo) with TP-347 weld overlay
Thickness:	9 inches approximately
Weight:	600 ton approximately
Design code:	ASME (American Society of Mechanical Engineers), Section VIII, Division 2
Design pressure:	2040 psig
Design temperature:	900°F

Because there are a limited number of vendors capable of fabricating and handling thick-wall dissolvers, ICRC initiated an early investigation (1) to determine those vendors capable of fabricating these two vessels; (2) to inspect their manufacturing facilities; and (3) to decide whether they had the technical documentation necessary to engage in procurement. Domestic vendors with large-vessel capabilities were visited by a joint ICRC/Catalytic team of vessel engineering, materials, and purchasing specialists.

In addition, the use of layered-construction vessels was looked at and vendors of layered vessels visited.

(For a summary of the construction preferences of each vendor, along with general comments, see Table 1.)

The recommendation was against the use of layered vessels, despite their appeal financially, because the lack of experience with them at elevated temperatures presents too high a risk.

MONOWALL CONSTRUCTION

There was general agreement among the vendors of solid, thick-wall vessels that the thickness limit for hot rolling of plate into shells was between 6 and 7 inches. Those vendors

*Catalytic, Inc.

**ICRC

who have the equipment for press forming of thick plate will evaluate the plate cost and availability against that for forged rings. The evaluation will also consider the available plate sizes and longitudinal seam welding required. Where the capability for press forming is lacking or marginal, shell construction will be from forged rings.

Hemispherical vessel heads will either be die-formed in sections and then welded, or formed in one piece, depending upon press capabilities.

Internal surfaces of shell courses and heads will be given a weld overlay of austenitic material such that diffusion will be minimized and approximately 3/16 inch of homogeneous overlay material will be guaranteed. Depending upon the vendor, shell overlay will be either multiple wire or strip, deposited by automatic equipment. Head overlay will be either single or double wire deposited. From actual overlay deposits inspected, it appears that the "as deposited" surface can be put into service without machining.

Courses of shell and heads will be assembled using full penetration girth welds. The shell course lengths may be from 10 to 20 ft long, depending upon vendor and material availability. The dissolvers will be supported by short cylindrical skirts, attached in the area of the bottom head to the shell juncture.

Temper Embrittlement

Temper embrittlement is a phenomenon defined as a decrease in the notch toughness (an increase in transition temperature) of alloy steels when they are heated in the approximate temperature range of 750°-1100°F. This deterioration occurs in the welds as well as the plate metal.

Temper embrittlement can be minimized by controlling metal chemistry and heat treatment. This can be achieved by specifying a Watanabee or Brusecato number (a measure of the susceptibility to temper embrittlement based on chemistry) for the plate metal. For weld metal, a maximum transition temperature shift based on a step aging method will be specified. A typical specification for weld metal would be:

$$T_f = T_t + (X)(\Delta T) = 100^\circ\text{F} \quad 1.5 \leq X \leq 2.5$$

where T_f is the final transition temperature, T_t is the transition temperature before step aging, and ΔT is the change in transition temperature after step aging.

All of the fabricators were comfortable with $X = 1.5$ and could achieve $T_f = 100^\circ\text{F}$ with $X = 2.5$, if we required it.

Weld Overlay

The requirements for the dissolvers will include an 18-8 stainless steel material on the i.d. to prevent excessive corrosion from the H_2 - H_2S atmosphere. This can be achieved by weld overlay, roll bonding, or explosive bonding. All of the surveyed fabricators were capable of meeting the necessary specifications of chemistry, thickness, and ferrite number.

All use a single or double torch for weld overlaying the heads or nozzles. A number of techniques have been demonstrated for shells. These have been single-wire, multiple-wire, and strip techniques using flux or inert gas for protection of the molten deposit. It appears that any of the techniques can be made to perform satisfactorily, if adequately

controlled. The preferred technique has been the 90 mm-wide strip deposited as a submerged arc technique with the chromium level enhanced through additions in the flux.

It is expected that a hard zone will result when the post weld heat treatment is undertaken due to the diffusion of carbon from the low alloy steel into the austenitic material. There has been sufficient service experience in the refineries with austenitic weld deposit on low alloy steel stress-relieved vessels to inspire confidence in the techniques available.

Vendors

Combustion Engineering (C.E.). The Chattanooga, TN, plant offers excellent facilities and capabilities for the fabrication of the coal dissolvers. The company is experienced with nuclear vessel fabrication and has established a technology for large vessel design and fabrication with appropriate check points and laboratory control. Many large, thick-wall vessels, up to 14 ft 6 inches o.d. (outside diameter) and 12 inches thick, were seen in fabrication. The average weight of these vessels is 500-800 tons. Water access for barge shipment is available.

Material procurement is expected to be from Lukens, Marrel (French), or Japan Steel Works for thick plate material; and Ladish, U.S. Steel, Bethlehem, Marrel, and Kobe Steel for ring forgings. A 14,000 ton press makes plate fabrication attractive and radiography requirements would be satisfied with a 15 MeV linear accelerator. C.E. estimated that four to five shell courses, each with either two or three welded long seams, would be used for each dissolver. Courses would be assembled by submerged-arc welding. The time for design, fabrication, and delivery of two coal dissolvers is estimated at less than two years, assuming material availability. Fabrication of the remaining dissolver units for a full size SRC-I plant can all be accomplished at this facility with reasonable delivery expectancy.

Chicago Bridge and Iron (CB&I). After a tour of the Memphis, TN, shop, we had no doubt that the coal dissolvers can be designed and built at this facility. Established under a partnership agreement between CB&I and General Electric (G.E. would not be involved in dissolver procurement) for fabrication and assembly of nuclear equipment, this facility offers 1000 ton handling capabilities, barge-loading accessibility, a 12,000 ton press for shell forming, a 12 MeV linear accelerator supplemented by gamma ray radiographic facility, and an experienced manufacturing and engineering team. A nearly completed 14 ft o.d. X 80 ft long hydrocracker built with an 11-inch wall thickness simulated the appearance of the subject dissolver. Dissolver shell courses would be assembled from thick-formed plate with long seams electro slag-welded.

Scheduling for the delivery of two dissolvers will be 18 months, with reasonable plate delivery. Delivery of subsequent units can also be expected to start 18 months after the order, but will be subject to material availability. A sample material procurement specification was obtained for reference in preparing the users specification. CB&I claims to be capable of erecting large, thick-wall vessels in the field with equipment and quality equal to that done in the shop.

Babcock & Wilcox (B&W). Being landlocked has not stopped this Barberton, OH, facility from shipping vessels in excess of 550 tons and up to 15½ ft o.d. by rail. Radiant boilers and steam drums with 8-inch thick walls are evident throughout the shop. Other larger diameter vessels with thinner walls are also in fabrication, some for the Navy Trident Program. Heads of up to 8-9-inch thickness are formed in a 6500 ton die press, starting with up to 16 ft blanks. A 7½ MeV linear accelerator is used for radiography. Large nozzles

are automatically sub-arc welded into heads. Although capable of using formed thick plate with electro slag long welds, B&W prefers to consider forgings for the shell courses. Assembly would be by circumferential submerged-arc welding. Tentative scheduling for the delivery of two dissolvers is 33 months. This includes an expected one year for receipt of materials (seven forgings) and 72 weeks for fabrication.

This facility is diversified and exhibits inventiveness in its machining, forming, and assembly areas for nuclear and fossil requirements. Engineering and quality appear to be of exceptionally high caliber. But, since the current maximum rated crane capacity is only 450 tons, the problems of handling capacity and shipping will have to be worked out.

Wyatt. This Houston, TX, shop has nearby access to barging facilities. It is expected that the coal dissolvers will be fabricated from forgings even though a thick plate-forming capability of up to 14-inch thickness exists. The final decision will depend upon cost and availability. An 8 MeV linear accelerator is available for radiography and 700 ton handling capacity is more than sufficient for the dissolvers. Several large vessels in the 20 ft o.d. range were seen in the shop, along with thick-wall vessels, but the overall shop load appeared extremely light. Scheduling of two dissolvers is 18 months, with reasonable material availability.

Belmas-Jovel. Also based in Houston, TX, this shop has nearby access to barging facilities. Unlike other vendors visited, Belmas does not have a capability to form thick plate for the dissolvers, and would, therefore, quote on the basis of forged courses for the shell. A handling capability of up to 600 tons is available and many large 18-20 ft o.d. vessels were seen in the shop. To date, Belmas has not produced a vessel with a wall thickness over 6½ inches. To fabricate and inspect a 9-inch vessel would require additional radiographic equipment.

General

A tabulation of vessels built to date in or about the size range of that of the dissolver was requested of each vendor and the information received is attached to this report (Appendix 1).

A certified users specification will be prepared in accordance with the requirements of ASME, Section VIII, Division 2. This specification and the code documentation required of the vessel manufacturer will cover the design analysis, material, fabrication, inspection, and testing of the coal dissolvers.

Discussions with all the vendors visited have led to questions regarding foreign competition. All appear to resent foreign competition, especially from Japan; one vendor was reluctant to bid at all against such competition. The reasons for this concern are:

- (a) Foreign governments subsidize their vendors, thus enabling them to bid lower than American vendors
- (b) American government restrictions increase prices for American vendors but do not affect foreign shops or prices
- (c) The domestic fabricators must consider a foreign supply of materials, possibly from the same firms quoting against them on fabrication work; they could expect inflated prices from those sources

If American shops close because of foreign competition, thick-wall and large vessel fabrication technology will be lost to this country.

LAYERED CONSTRUCTION

Favorable pricing experience for layered vessels led to the consideration of layered vessel vendors along with those fabricating monowall vessels.

Of the vendors visited, Hahn and Clay and Struthers Wells are experienced in fabricating large layered vessels and would pursue the layered approach. These vendors use different methods of fabrication.

Vendors

Hahn and Clay. This company in Houston, TX, starts shell segment fabrication with a pressure-tight innermost shell or layer of $\frac{3}{8}$ -1-inch thickness made from clad plate. A series of outer concentric layers of approximately $\frac{1}{4}$ inch nominal thickness are then added in one or more pieces and welded. The outer layers are vented to the atmosphere through a series of small vent holes. Lengths of shell segment and heads are assembled with circumferential weld seams through the built-up thickness. An alternate approach allows staggering of the circumferential seams for each of the thicknesses.

Struthers Wells. This company in Warren, PA, also starts with a pressure tight inner shell made from clad plate (or unclad plate to be weld overlayed), but the plate is about 2.3 inches thick. Additional cylindrical layers of approximately the same thickness are rolled and welded and the welds radiographed. These layers, fabricated with selected diametric tolerancing, are then successively heated and slid over the previous inner assembly for a shrink fit, until the entire shell segment is completed (in four thicknesses). The outer layers are vented to the atmosphere. To ensure proper venting, two thin perforated layers (with elongated slots) are positioned circumferentially between the inner and second layers. Lengths of shell segments and heads are assembled with circumferential weld seams through the built-up thickness. This fabrication method is called Multiwall.

Others. A variation of the preceding method of fabrication is Plywall construction—a thin plate type—done by Nooter Corp. of St. Louis, MO.

Still another method of layered construction called Coilayer has been brought to ICRC's attention by Mitsubishi Heavy Industries of Japan. This technique also starts with a pressure tight shell but has a clock spring like a spiral coil of plate wound around the inner shell. Tapered plates start and finish this wrap. No welding is used except as required to make the wrapping continuous with the tapered sections and along the sides of the inner tapered section to the edges of the inside shell cylinder. An outside shell made in two halves is then welded in place over the tightly wound spiral. Inner shell thickness ranges from $\frac{3}{4}$ to $1\frac{1}{2}$ inches while wrap thickness is from $\frac{1}{8}$ to $\frac{1}{4}$ inches. Outer layers are vented to the atmosphere and the lengths of shell segments and heads are assembled with circumferential weld seams through the built-up thickness of wall. The ends of the cylindrical shell segments are machined, weld buttered, and then remachined before the circumferential weld seams are made. Other foreign vendors fabricate layered vessels to the methods herein described or variations thereof.

Conclusions on Layered Construction

Although the initial reason for considering layered construction was financial, it was learned that many technical advantages exist. The primary one appears to be the use of

readily available thinner plate. In addition, material properties through the entire thickness are more uniform. And there is greater ease in detecting flaws that may exist in the plate. Depending upon the particular layered construction method employed, inspection of the cylindrical layer components may be considered closer than that for a standard thin-wall vessel. Full penetration thick-wall longitudinal weld seams are eliminated. Inner cylinders can be clad as well as weld overlayed. Venting of outer layers eliminates potential buildup of hydrogen which has penetrated through the inner shell. If a thick-wall vessel fails, it will probably fail in a catastrophic manner, whereas failure of a thin-wall or layered vessel will probably be in a ductile fashion.

Even with these advantages, plus acceptance of layered construction by the ASME Code and limited use for industrial and nuclear high-pressure application, there still remains the fact that there is no track record for this type of vessel construction at this level of temperature. Full penetration butt weld circumferential seams between shell segments and seams between forged solid nozzles and layered shell walls raise a question regarding creep and crack propagation at high temperature and pressure due to the inherent notches.

An additional worry regarding layered construction is that the proposed size of the coal dissolver is pushing the limit of the state of the art. (If layered construction can be shown to be acceptable for this application, it is expected that the technology could be quickly expanded to produce vessels with up to 30-inch thick walls.)

Therefore, the potential offered by layered construction should not be considered for direct use in the SRC-I Demonstration Plant. The lack of experience at elevated temperature presents too high a risk factor in this program.

Use of a layered vessel as an experimental unit in the SRC-I Demonstration Plant would be acceptable if all necessary precautions were taken and a standby backup vessel were available on line. Functioning in place of a vessel such as the High Pressure Flash Tank, the layered vessel could be instrumented to gather data while exposed to practical high temperature operating conditions.

Table 1

Thick Wall Vessel Fabricators Visited

Fabricator	Type of construction	Comments
Babcock & Wilcox Barberton, OH	Shell from forged rings or formed plate	Must rely on rail shipment to Ohio River (Mt. Vernon, IN). Developing strip overlay process. 7-ft dia reactors with 8-inch thick walls in shop. Inventive machine shop.
Belmas-Jovel, Inc. Houston, TX	Shell from forged rings	Many large vessels up to 6-inch thick in fabri- cation. Uses multiple wire open arc overlay. Requires additional radiographic equipment.
Chicago Bridge & Iron Company Memphis, TN	Shell from formed plate or forged rings	Duplicate to reactor seen on shop floor. 15-inch (hot) press forming capacity. Uses plasma arc weld overlay for shell and wire overlay for heads.
Combustion Engineering Chattanooga, TN	Shell from formed plate or forged rings	1-ft thick nuclear vessels in fabrication. 17-inch (hot) press forming capacity. Uses strip overlay. Makes own welding rods and flux. Excellent engineering backup facility.
Hahn & Clay Houston, TX	Layered construction (A.O. Smith method-- thin plate type)	No large vessels seen. Vessels not main manufacturing product line. X-ray capabilities to 9-inch thickness.
Nooter Corp. St. Louis, MO	Layered construction-- Trademark Plywall (thin plate type)	Potential for large-size layered construction. Capability still about one year away.
Struthers Wells Corp. Warren, PA	Layered construction-- Trademark Multiwall	Over 2-inch thick wall, shrink fit construction. Potential size and weight problem for shipment from Warren, PA.
Wyatt Houston, TX	Shell from forged rings or formed plate	14-inch (hot) press forming capacity. Large thick-wall vessels in fabrication. Uses multiple wire open arc overlay.

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GKT COAL GASIFICATION PROCESS IS RECOMMENDED OVER TEXACO

**A. D. Bixler
and A. R. Kuhns***

As the Phase 0 Report points out, the quantity of hydrogen required for the 6000 TPD SRC-I Demonstration Plant, 50 MM scfd, is sufficient to justify onsite production as an integral part of the SRC facility.**

After considerable study, ICRC has selected the commercially proven GKT Gasification Process.*** Because GKT is commercially viable now, the choice will allow ICRC to concentrate its Demonstration Plant effort on developing liquefaction technology to produce significant quantities of product.

ICRC expects that a pressurized gasification process (preferably with a dry feeding system) will ultimately be the economic choice for the Commercial Plant. Many pressurized gasifiers are currently being developed on a demonstration scale and some commercial scale projects have been announced. These efforts by others will reduce the risk associated with developing gasifier technology; and the SRC-I Demonstration Plant can reduce the related risk associated with uncertainties in the composition and properties of Kerr-McGee ash concentrate feedstock.

Also important, selection of the GKT Process can lead to an advanced position with respect to the Shell-Koppers gasification technology—which is being developed jointly by Shell Oil and Krupp-Koppers on the basis of a pressurized extension of the GKT technology.

Technical Considerations. ICRC recommends proceeding with the GKT gasifier for these reasons:

- (1) The probability of achieving a reliable hydrogen supply is greater because the designs used have been proven at full scale in similar service.
- (2) Long-term life of the gasifier refractory is expected, based on the Krupp-Koppers design concept of a layer of frozen slag protecting the refractory.
- (3) Krupp-Koppers has successfully resolved previous problems with its gasifiers even in very remote locations.
- (4) A relatively low penalty, approximately 4% in overall efficiency, for the production of hydrogen is expected. The nature of the Kerr-McGee feedstock adversely penalizes the Texaco Process relative to a low ash coal feed.

*ICRC. The authors acknowledge the excellent work done by The Ralph M. Parsons Company in preparing the detailed trade-off studies that formed the basis for ICRC's final evaluation. This paper was initially drafted in February 1980; the final version was completed in June 1980.

**SRC-I Coal Refining, Tasks 1 and 2, 31 July 1979, p. 273.

***A subsidiary company, GKT (Gesellschaft für Kohle Technologie), has been organized by Krupp-Koppers to market its technology in the U.S. under the name GKT Gasification Process. Originally, this process was called the Koppers-Totzek (K-T) Gasification Process and early correspondence used this name.

In any event, ICRC would be unwilling to recommend use of the Texaco Gasification Process until the successful conclusion of gasification tests with the Kerr-McGee feed. Further, these tests would have to demonstrate that the reactivity of this feed is sufficiently high to obtain adequate carbon conversion at a sufficiently low temperature so that the risk of refractory failure is judged acceptable. These tests would also have to confirm the successful mixing and pumping of slurry at or near proposed design concentration.

Economic Considerations. Using a 1979 dollar basis, the GKT Process is projected to add only about 11¢/MM Btu to the SRC product cost compared to the Texaco design slurry concentration case, but is about 27¢/MM Btu less expensive in SRC product cost than the 70% of design slurry concentration case.

Scheduling. Since a delay in reaching the gasifier selection decision would have an economic impact in the range of \$95,000/day to \$400,000/day, depending on the extent of the delay in construction, the design of the Demonstration Plant is proceeding, with Department of Energy concurrence, based on recommendations in this report. Therefore, only a reversal of this basis would have a negative effect on the schedule.

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The following sections of the report discuss advantages and disadvantages of two gasification processes—first, Texaco and, second, GKT—as each applies to gasification of Kerr-McGee feedstock for the SRC-I Process.**** Uncertainties are also listed, with an approximate timetable for their resolution.

The technical evaluations have been limited (1) to the major uncertainties associated with each process; and (2) to the use of that process with high ash Kerr-McGee feedstock. Other considerations do not have major impact and require more detailed design information than is available for this evaluation. For example, the equipment for removing particulates from the gas stream in the Texaco Process is simpler than that for the atmospheric pressure GKT Process—a potential operating advantage for Texaco—but the information available is insufficient to quantify the advantage. Furthermore, both Texaco Partial Oxidation plants and GKT Coal Gasification plants have been successful in obtaining adequate particulate removal.

TECHNICAL SUMMARY OF THE TEXACO COAL GASIFICATION PROCESS

Firm Advantages

Efficiency. A comparison of the Texaco and GKT gasification processes for gasifying the Kerr-McGee material with small amounts of supplemental coal gives the Texaco Process an advantage in overall efficiency of approximately 4% and in cold gas efficiency of approximately 1%. Overall efficiency is defined as the higher heating value (HHV) of product hydrogen plus the HHV of export steam divided by the sum of the HHV of all feed and fuel materials plus the HHV of imported power (i.e., 9500 Btu/kW).

****Proprietary information has been excluded.

Cold gas efficiency is defined as the HHV of the product hydrogen divided by the HHV of the gasifier feedstock.

The comparison was made using information from detailed studies performed by Parsons during and subsequent to the Phase 0 engineering work on this project.^{1, 2} Type I evaluations of Kerr-McGee ash concentrate (MAR) at Texaco's Montebello Laboratory indicated that the slurry concentration used during the Phase 0 study cannot be obtained without briquetting and regrinding the Kerr-McGee MAR to optimize the particle size.^{3, 4} The efficiency comparison quoted above includes briquetting and regrinding of the Kerr-McGee MAR to the Texaco Unit rather than reducing the slurry concentration. Without this extra processing step the slurry concentration will be limited to 70% of the design concentration, thus greatly decreasing the efficiency of the Texaco Process.

The higher overall efficiency of the Texaco Process arises from operation of the gasifier at pressure, as the decreased power for product compression is only partially offset by the higher air and oxygen compression cost.

A detailed comparison of Texaco and GKT gasification processes for gasifying a mixture of filter cake and coal was made during the Phase 0 engineering work on the project.⁵ In this study, the Texaco Process was shown to have a higher overall efficiency by 3-4% but a lower cold gas efficiency by 2-3%. The higher ash content of this feedstock causes the cold gas efficiency to decrease substantially for Texaco but the overall efficiency decrease is offset by increased production of export steam.

Feed Reactivity. Due to the higher operating pressure of the Texaco gasifier, the feed material is expected to have a higher degree of carbon conversion at a given temperature.^{6, 7, 8} Alternately, the exit gas temperature from the reaction zone of the Texaco gasifier will be lower than the GKT gasifier exit temperature at a given carbon conversion.

Reactor Size. Fewer gasifiers would be required in the Commercial Plant for the Texaco Coal Gasification Process: approximately five operating gasifiers with Texaco, compared with 10 operating four-headed GKT gasifiers.⁹ The number of gasifiers is based on the largest size Texaco Partial Oxidation reactor as well as the largest size GKT gasifier in operation today. But it is difficult to use this advantage effectively in the Demonstration Plant due to reliability considerations.

Firm Disadvantages

Commercial Experience. Texaco has limited commercial experience with its Coal Gasification Process. In January 1980, a 150 ton per day (TPD) coal feed demonstration plant operated by Ruhrchemie in Germany had been onstream for two years with 4000 hours of operation. The longest run to date was 500 hours. However, Ruhrchemie has stated that the primary emphasis of its program to date has not included making long runs.¹⁰

Dow Chemical is currently operating an air blown unit of unknown size at Plaquemine, LA, to produce low Btu gas; TVA will be starting up an 8 ton/hr (192 TPD) coal feed unit this summer; and Tennessee Eastman has announced a large commercial chemicals facility based on this technology.

Refractory Life. Although Texaco has a program of testing refractories in its Montebello pilot unit and providing information on the performance to the refractory manufacturer, it has not undertaken the task of demonstrating long-term refractory life testing in slagging service. Because the Texaco coal gasifier operates at inside vessel wall temperatures above the slagging temperature of the ash in the feedstocks, the refractory is exposed

to erosion, corrosion, solution, and chemical attack by the molten slag. This represents substantially more severe service than that experienced in a partial oxidation reactor in low ash residual oil service.

The plant at Ruhrchemie has experienced severe problems with refractory life during its relatively short onstream time. The refractory bricks currently in the reactor were installed recently. Representatives of Ruhrchemie have stated that the problems with refractory at its plant have not yet been fully solved.¹⁰

Scale-Up Risk. Although the Texaco Coal Gasification Process makes use of the Texaco Partial Oxidation Equipment design technology, a number of the essential equipment items have not been demonstrated, to ICRC's knowledge, on a commercial scale by Texaco. These include the slurry pumps and the ash removal system. The economics of the Texaco Coal Gasification Process requires that the slurry concentration be maintained near the limit of pumpability.

The slag removal system uses a lockhopper to accomplish the pressure letdown of the slag from a water quench system. Valves in this service are subject to severe erosion if they do not seal tightly on every closing. Both the slurry pumps and the lockhopper valves are judged to pose extra risk in initially achieving a satisfactory design on a large scale. This could lead to reduced onstream factors for the hydrogen generation system until any unforeseen problems are resolved.

Unresolved Issues

Gasification Tests on Kerr-McGee Feed. Gasification tests on the Kerr-McGee feedstock at Montebello have been delayed pending successful completion of tests on SRC-II materials there. But planning for SRC-I tests is currently under way. ICRC is investigating facilities for briquetting the Kerr-McGee material and Texaco has indicated that these tests may be conducted in September.

Slag Leaching Tests. Slag leaching tests will be made at Oak Ridge National Laboratory when the Montebello gasification tests are done. These tests will probably take two months including shipping samples and scheduling and completing analysis.

Commercial Acceptance of Texaco Gasifier by DOE. The commercial acceptance of the Texaco Coal Gasification Process by DOE is required.

Slurry Mixing and Pumpability. The design of a briquetting and regrinding system for the Kerr-McGee feedstock has not been demonstrated. This system will enhance the slurry concentration that can be obtained without optimizing the feed particle size. An early test of Kerr-McGee ash concentrate indicated a larger particle size and a higher slurry concentration (55 wt %) than the most recent test by Texaco.^{3, 11}

The design of a blending system for mixing the fine Kerr-McGee feed with water and achieving a pumpable slurry poses an additional risk area as this system has not yet been demonstrated. Laboratory testing has shown that dispersing agents can significantly increase the concentration of a pumpable slurry.³ The effective use of these agents on a large scale in this service remains to be determined.

Pumpability of the Kerr-McGee ash concentrate water slurry, as proposed for our process, is currently at the stage of bench testing. The Montebello tests will confirm pumpability on a small scale. Obtaining a commercially-sized pump with long life components remains a technical risk. Testing on a commercial scale is difficult as the slurry exhibits both pseudo-plastic and thixotropic behavior. Although pseudo-plasticity (shear thinning) is quite com-

mon for slurries, the thixotropic (time dependent) characteristic of the slurry is an indication that the slurry could "set up" in pipelines. The characteristics of the slurry will change with time if tested in a pump loop and only limited quantities of material are available for testing on a once through basis.

TECHNICAL SUMMARY OF THE GKT COAL GASIFICATION PROCESS

Firm Advantages

Commercial Experience. As of January 1979, the daily output of ammonia from gasification of coal in Koppers-Totzek gasifiers was nearly 4000 TPD, with operation at seven plants in six countries.¹² Since 1951 more than 50 gasifiers have been delivered to 14 clients, with a total capacity of 470 MM scfd of synthesis gas ($H_2 + CO$).¹²

GKT plants operating on coal are located in Greece, Thailand, Turkey, Zambia, South Africa, and two locations in India. A plant in Egypt operates on gaseous hydrocarbons and one in East Germany operates on fuel oil. Large-scale tests on petroleum coke and Char-Oil-Energy-Development Process (COED) char were conducted in Spain with good results. Prior to shutdown, the plant in Finland operated on coal, oil, and peat.¹³

Full-Scale Equipment. With the exception of one or two items of equipment, all major equipment items have been demonstrated at the scale of the GKT facility. One item which has not been demonstrated, an indirect water cooler to meet increased environmental concerns, has been tested successfully on a small scale at the commercial plant in Greece.¹⁴

It would be ICRC's intent that any additional items representing evolutionary improvements in the design would be carefully reviewed and, where practical, the concept of being able to operate without them would be retained.

Refractory Life. The Krupp-Koppers design for the GKT gasifier uses a low-pressure steam jacket external to the gasifier to maintain a frozen layer of slag on the gasifier wall. This slag layer minimizes the potential for chemical attack and solution at the refractory slag interface and is self-restoring with regard to erosion.

The refractory support pins also serve as heat transfer fins and, by tailoring the design of this system, refractory problems can and have been eliminated for most coal feeds.¹⁵ Based on experimental tests of samples of Kerr-McGee feed material, the feedstock is judged to be well-suited to this gasifier with respect to refractory design.¹⁶ At Modderfontein, South Africa, severe refractory problems occurred during the initial operation of GKT gasifiers. After the problem was recognized, the refractory support system was modified. The current refractory has been in service for nearly three years and the plant manager expects to perform minor repairs at four years and replacement at six years.¹⁷

Firm Disadvantages

Efficiency. The overall efficiency of the GKT gasifier in delivering hydrogen to the liquefaction loop at pressure is lower than the Texaco Process by approximately 4%. This was discussed in detail in Section I as an advantage for the Texaco Process. The cold gas efficiency of the Texaco Process is approximately 1% higher than the GKT Process with Kerr-McGee feed.

Feedstock Reactivity. Although the reactivity of a given feedstock is apparently lower at low gasification pressures, as discussed earlier, this only becomes a serious problem if the feedstock reactivity is sufficiently low so that the refractory design is unable to accommodate the higher gasification temperatures. Based on tests completed in September 1978 by Krupp-Koppers and recently confirmed by gasification tests, the Kerr-McGee feed is sufficiently reactive that this is expected to be a minor disadvantage.^{16, 18}

Reactor Capacity. For the Phase 0 design based on filters, three onstream GKT two-headed gasifiers were required, compared to one onstream Texaco gasifier.⁵ Each design included one spare gasifier. Further consideration of the base Texaco design, however, led to the subsequent incorporation of a liquid hydrogen backup system to provide additional reliability.¹⁹ To improve the reliability of the Texaco design without the need for a backup hydrogen supply, this study considered the use of two 50% Texaco gasifiers along with two 50% spare gasifiers for the Demonstration Plant. To meet the additional hydrogen requirement with the Kerr-McGee deashing process, two operating GKT four-headed gasifiers plus one spare will be required.

Coal gasification reactors are subject to frequent trips—this being in the nature of a plant handling solids, having a high degree of moving parts, and coupled with the need for an extensive safety interlock system required by the process. Operating multiple gasifiers minimizes the seriousness of these trips in downstream equipment operation; only part of the production is lost and flow through the downstream units can be maintained. This avoids lost time involved with sequentially restarting each unit, as well as reducing potential for equipment failure due to cycling.

For the commercial design, the gasifier decision should be reviewed based on the commercial experience available then. Other technologies may merit more serious consideration at that time; i.e., Shell-Koppers.

Unresolved Issues

Handling Characteristics of Kerr-McGee Feedstock. To perform the gasification tests in Essen, it was necessary to predry the feed material at 110°C—because of a high concentration of deashing solvent.¹⁶ Much of that problem is believed to have been caused by the present design of the ash concentrate flash drum and downstream solids-handling equipment at the Wilsonville Kerr-McGee unit. The maximum allowable deashing solvent concentration and storage temperature to ensure flowability in the Demonstration Plant is uncertain at this point. Qualified facilities have been identified for performing solids flowability tests and a program is being planned. These tests will determine a maximum storage temperature and deashing solvent content of Kerr-McGee ash concentrate to assure flowability of the solids.

Gasification Tests on Kerr-McGee Feed. Tests on a bench scale gasification reactor in Essen, Germany, have been completed and the full report on these tests, as well as a preliminary process design balance resulting from the test work, have been received. Krupp-Koppers states that the results of the pilot scale tests can be used for making a good prediction of the efficiency and operation of the Commercial Plant.¹⁴ Further work in evaluating the consistency of the Pilot Plant tests and the implications to the Demonstration Plant design is under way.

Slag Leaching Tests. Slag leaching tests were run in mid-March at Oak Ridge National Laboratory. A preliminary analysis of the leachate from these tests indicates that

none of the samples produced an extract which would cause the waste to be considered hazardous under the Resource Conservation and Recovery Act (RCRA) toxicity standard.²⁰ However, the analytical program and the interpretation of the results are not yet complete.

If these tests indicate that the reactor slag is acceptable but the flyash material is unacceptable, then the capability for recycle or other means of making the flyash acceptable would need to be investigated. While recycle is judged to be feasible, it is likely to be more costly for GKT due to the higher fraction of flyash produced in the GKT Process compared to the Texaco Process.

Commercial Acceptability of Kerr-McGee Feedstock in the GKT Process. GKT gasifiers have been run on a wide range of coals and other feedstocks, including coals with ash contents as high as 40%, but there has not been any commercial experience with this particular feed material. Furthermore, sufficient quantities of feed are not available for any meaningful commercial testing. However, based on the experience with laboratory tests, Krupp-Koppers is willing to warrant the process without commercial testing.

COMPARATIVE GASIFIER ECONOMICS

Demonstration Plant

The GKT case was compared with two cases based on Texaco water slurry gasification. In one case, the Kerr-McGee ash concentrate is first briquetted and crushed and then slurried to the design concentration between 60-70% solids. In the second case, the briquetting and crushing step is excluded, thus allowing a slurry concentration of only 70% of the design concentration (between 40 and 50% solids).

The GKT Process is projected to add only about 11¢/MM Btu to the SRC product cost compared to the Texaco design slurry concentration case—but is about 27¢/MM Btu less expensive in SRC product cost than the 70% of design slurry concentration Texaco case. Both differences in product cost are on a 1979 dollar basis.

In the absence of a successful demonstration of the briquetting and crushing operation in commercial size units, the potential economic penalty incurred by the choice of GKT technology is small compared to the enhanced reliability and decreased technical risk achieved.

The GKT case is based on three four-headed gasifiers installed, with two operating. Both Texaco base cases include four gasifiers installed, with two operating. The design slurry concentration case necessitates the use of nominal 8 ft gasifiers; the 70% of design slurry concentration case necessitates the use of nominal 9 ft gasifiers. The additional spare gasifier for each Texaco base case is necessary to achieve the required plant onstream factor while allowing time for changeouts of gasifier refractories. Both Texaco cases are based on two refractory changeouts per year for each operating gasifier, corresponding to a refractory life of about 4000 hours.

The Capital Investment Summary. Estimates for each base case are presented in Table 1 in 1979 dollars. The projected capital requirement for the GKT case is about 6% greater than the Texaco case with briquetting and crushing but about 11% less than the Texaco case for the 70% of design slurry concentration.

The cost estimates shown in Table 1 are not restricted to hydrogen generation alone, but rather encompass the entire gasification and gas treating sections of the plant, simply

because these costs are readily available to ICRC in that form. This reporting technique will not distort the gasifier comparisons; those areas which are unrelated to hydrogen generation will cancel each other (e.g., the DEA unit).

The Annual Operating Costs. These costs, also in 1979 dollars, are shown in Table 2. Based on SRC product cost, the GKT case is estimated to be 11¢/MM Btu more expensive than the Texaco design slurry concentration case but 27¢/MM Btu less costly than the 70% of design slurry concentration case.

In addition to the reliability and technical risk advantages which favor the selection of GKT gasification, another mitigating factor in the apparent economic advantage for the Texaco design slurry concentration case could be the costs for briquetting and crushing of the Kerr-McGee material. These costs have not been assessed in detail as yet. For example, a 100% increase in briquetting and crushing capital would reduce the projected advantage of the Texaco case by \$1.7 MM annually—1979 dollars—and the SRC cost differential to less than 7¢/MM Btu.

Two Sensitivity Cases Also Evaluated. Table 3 shows that a reduction in refractory life to 2000 hours decreases the advantage of the design slurry concentration Texaco case over the GKT case to 8¢/MM Btu—and increases the disadvantage of the 70% of design slurry concentration case to the GKT case to 31¢/MM Btu.

Table 4 illustrates that a 1000 hour refractory life shifts both Texaco cases to an inferior position compared to GKT. The Texaco disadvantages are 1¢/MM Btu for the design case and 45¢/MM Btu for the 70% of design case. The 1000 hour refractory life Texaco cases require five installed gasifiers due to the increase of refractory changeout frequency.

Commercial Plant

The relative economics of GKT and Texaco gasification was also assessed for SRC-I Commercial Plants (30,000 TPCD). It was assumed that the design slurry concentration Texaco case with briquetting and crushing would be available at the time of SRC-I commercialization. Seven nominal 9 ft gasifiers (five operating) are required for this case. The corresponding GKT case requires 12 four-headed gasifiers (10 operating).

Table 5 shows GKT requires about 10% more capital investment than Texaco. Table 6 presents the annual operating costs for the same cases. The Texaco case, based on a 4000 hour refractory life, is estimated to have an 11¢/MM Btu advantage in SRC product cost in 1979 dollars.

As stated at the beginning of this article, a dry-feed, pressurized gasification process would be preferred as the economic choice.

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Table 1

Base Kerr-McGee Demonstration Plant Cases
Capital Investment Summary (MM 1979 \$)

	GKT	Texaco design slurry concentration with briquetting	Texaco 70% of design slurry concentration
Gasification and shift	75.4	52.0	71.4
Selexol	17.7	18.4	19.8
H ₂ S compression	8.6	8.6	8.6
BSR unit	7.3	7.5	7.5
Claus units (2-100%)	9.8	9.8	9.8
DEA unit	6.8	6.8	6.8
Ammonia-sulfide stripping	1.4	1.4	1.4
O ₂ plant	26.1	33.0	47.4
Briquetting/crushing	-	7.0	-
Subtotal	153.1	144.5	172.7
Contingency (@ 20%)	30.6	28.9	34.5
Total	183.7	173.4	207.2

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Table 2

Base Kerr-McGee Demonstration Plant
Annual Operating Cost (M 1979 \$)¹

	GKT	330 operating days/yr 4000-hour refractory life for Texaco cases	
		Texaco design slurry concentration with briquetting	Texaco 70% of design slurry concentration
Coal @ \$1.40/MM Btu	\$ 171	\$ 108	\$ 4,630
Fuel gas @ \$4.60/MM Btu	1,093	386	386
Power @ \$0.036/kwh	16,255	13,640	19,115
Steam credits ²	(2,414)	(2,611)	(5,475)
Catalysts and chemicals	1,055	1,443	1,730
Refractory changeouts	-	1,000	1,600
Capital charges @ 20%	<u>36,740</u>	<u>34,680</u>	<u>41,440</u>
Total	\$52,900	\$48,646	\$63,426
Delta annual cost ³	-	-\$ 4,254	+\$10,526
Delta SRC product cost ³ (¢/MM Btu)	-	- 11	+ 27

1. Annual operating costs are not given for those items unaffected or only slightly affected by the choice of gasifiers.

2. 450 psig (sat) @ \$2.00/M lb
150 psig (sat) @ \$1.80/M lb
75 psig (sat) @ \$1.80/M lb
35 psig (sat) @ \$1.50/M lb

3. Relative to GKT case.

Table 3

Kerr-McGee Demonstration Plant Cases
Annual Operating Cost (M 1979 \$)¹

330 operating days/yr Sensitivity case: 2000-hour refractory life for Texaco cases			
	GKT	Texaco design slurry concentration with briquetting	Texaco 70% of design slurry concentration
Coal @ \$1.40/MM Btu	\$ 171	\$ 108	\$ 4,630
Fuel gas @ \$4.60/MM Btu	1,093	386	386
Power @ \$0.036/kwh	16,255	13,640	19,115
Steam credits ²	(2,414)	(2,611)	(5,475)
Catalysts and chemicals	1,055	1,443	1,730
Refractory changeouts	-	2,000	3,200
Capital charges @ 20%	<u>36,740</u>	<u>34,680</u>	<u>41,440</u>
Total	\$52,900	\$49,646	\$65,026
Delta annual cost ³	-	-\$ 3,254	+\$12,126
Delta SRC product cost ³ (¢/MM Btu)	-	- 8	+ 31

1. Annual operating costs are not given for those items unaffected or only slightly affected by the choice of gasifiers.

2. 450 psig (sat) @ \$2.00/M lb
150 psig (sat) @ \$1.80/M lb
75 psig (sat) @ \$1.80/M lb
35 psig (sat) @ \$1.50/M lb

3. Relative to GKT case.

Table 4

Kerr-McGee Demonstration Plant Cases
Annual Operating Cost (M 1979 \$)¹

		330 operating days/yr Sensitivity case: 1000-hour refractory life for Texaco cases	
		Texaco design slurry concentration with briquetting	Texaco 70% of design slurry concentration
GKT			
Coal @ \$1.40/MM Btu	\$ 171	\$ 108	\$ 4,630
Fuel gas @ \$4.60/MM Btu	1,093	386	386
Power @ \$0.036/kwh	16,255	13,640	19,115
Steam credits ²	(2,414)	(2,611)	(5,475)
Catalysts and chemicals	1,055	1,443	1,730
Refractory changeouts	-	4,000	6,400
Capital charges @ 20%	<u>36,740</u>	<u>36,360</u> ⁴	<u>43,800</u> ⁴
Total	\$52,900	\$53,326	\$70,586
Delta annual cost ³	-	+\$ 426	\$17,686
Delta SRC product cost ³ (¢/MM Btu)	-	+ 1	+ 45

1. Annual operating costs are not given for those items unaffected or only slightly affected by the choice of gasifiers.
2. 450 psig (sat) @ \$2.00/M lb
150 psig (sat) @ \$1.80/M lb
75 psig (sat) @ \$1.80/M lb
35 psig (sat) @ \$1.50/M lb
3. Relative to GKT case.
4. Includes five installed gasifiers due to refractory changeout frequency.

Table 5

30,000 TPCD Kerr-McGee Commercial Plant Cases
Capital Investment Summary (MM 1979 \$)

	GKT	Texaco design slurry concentration with briquetting
Gasification and shift	244	153
Selexol	53	56
H ₂ compression	22	22
BSR units	27	28
Claus units	20	20
DEA unit	20	20
Ammonia-sulfide stripping	3	3
O ₂ plant	70	89
Briquetting/crushing	-	23
Subtotal	459	414
Contingency (@ 20%)	92	83
Total	551	497

Table 6

30,000 TPCD Kerr-McGee Commercial Plant Cases
Annual Operating Cost (M 1979 \$)₁

	<u>330 operating days/yr</u>	
	GKT	Texaco design slurry concentration with briquetting
Coal @ \$1.40/MM Btu	\$ 950	\$ 600
Fuel gas @ \$4.60/MM Btu	6,080	2,150
Power @ \$0.036/kwh	90,380	75,840
Steam credits ²	(13,420)	(14,520)
Catalysts and chemicals	5,870	8,020
Refractory changeouts ³	-	4,000
Capital charges @ 20%	<u>110,200</u>	<u>99,400</u>
Total	\$200,060	\$175,490
Delta annual cost ⁴	-	-\$ 24,570
Delta SRC product cost (¢/MM Btu) ³	-	- 11

1. Annual operating costs are not given for those items unaffected or only slightly affected by the choice of gasifiers.
2. 450 psig (sat) @ \$2.00/M lb
150 psig (sat) @ \$1.80/M lb
75 psig (sat) @ \$1.80/M lb
35 psig (sat) @ \$1.50/M lb
3. Texaco based on 4000-hour refractory life.
4. Relative to GKT case.

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HOW BEST TO DISPOSE OF MAR: FOUR PIECES IN THE PUZZLE

The Ralph M. Parsons Company

MAR, the mineral ash residue left over from the deashing process in the production of SRC-I, is also known as "filter cake" when the work is done by filtration and "K-MAC" (for Kerr-McGee ash concentrate) when it is done by Kerr-McGee Critical Solvent Deashing. By any name, MAR poses a problem.

If it were an environmentally acceptable substance, it could be allowed to accumulate as a relatively remote mountain. But it is pyrophoric (at least, K-MAC at elevated temperatures is and filter cake may be). It generates dust. And it is probably carcinogenic. As it comes from the deashing area, it is not environmentally acceptable.

This creates the puzzle of how best to dispose of it. The following studies, undertaken by The Ralph M. Parsons Company at ICRC's request, have turned up some of the pieces of the puzzle.

In the first study, Parsons examines the original concept, that MAR could be efficiently gasified into makeup hydrogen. As the Phase 0 Report said: "Gasification serves two purposes. First, it converts mineral ash residue (MAR) . . . into inert slag for disposal or sale. Second, gasification of the MAR, plus supplemental coal, generates makeup hydrogen."¹ But in this study Parsons finds that as gasifier feed MAR has a negative value. Disregarding for the moment the expense of an alternative disposal method, it would cost less to make hydrogen from coal purchased at market than it would to make it from MAR, which comes free. The MAR here is filter cake. The study was initiated as a Bridging Task following Phase 0 and dealt with filter cake from filtration deashing and disposal by Texaco Gasification, which were the Base Case then.

In the second study Parsons reviews the literature on alternative schemes of disposing of K-MAC and offers a preliminary assessment of them.

The third study is a more detailed examination of one alternative and, again, derives a negative value for MAR—K-MAC this time.

However, the fourth study, in which GKT Gasification disposes of K-MAC, comes out positive. In this case K-MAC would have a value of +\$1.39/MM Btu—which brings us back to the original concept and makes it economically viable.

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¹Phase 0 Report, 31 July 1979, page 21.

TEXACO GASIFICATION: FILTRATION MAR/COAL FEED vs COAL FEED

The objective here was to investigate the actual cost involved in disposing of filter cake by Texaco Gasification. The approach was to derive a delta cost estimate from the study of the following two cases in producing the same amount of the makeup hydrogen requirement (50.90 MM scfd) for the 6000 TPD SRC-I Demonstration Plant:

- (1) Gasifying a mixture of coal and filter cake via Texaco Gasification
- (2) Gasifying coal alone via Texaco Gasification

For the purpose of comparison, a total plant concept for the production of hydrogen was considered—including facilities for oxygen production, coal and filter cake gasification, shift, acid gas removal (Selexol Unit), sulfur recovery, sour water stripping, and hydrogen compression—and also the DEA Unit in the Gas Treating Area, even though it is not part of makeup hydrogen production.

The evaluation was made in terms of feed requirements, process capacity requirements, product purity, overall utility, and construction cost.

The resulting delta cost estimate showed that to dispose of the filter cake MAR by Texaco Gasification would increase the cost of the project by approximately \$29.6 MM. Further, gasifying the filter cake would produce only about 9% of the total makeup hydrogen requirement, although it would consume an additional 48% oxygen. Gasifying the filter cake would also require a substantial increase in the consumption of utilities, raising annual utility costs by \$1.04 MM. With capital related charges of 20% of the total delta project costs, gasifying filter cake this way would increase the annual project costs by \$6.95 MM. This translates into a derived filter cake value of $-\$2.60/\text{MM Btu}$. The implicit cost of filtration MAR disposal by Texaco Gasification amounts to 1¢ per pound.

Four Plant Areas Studied

The study covered four areas of the Demonstration Plant: Gasification, Gas Treating, Compression, and Sulfur Recovery. The DEA (diethanolamine) Unit in the Gas Treating Area was also included, because it is pertinent to the data developed for gas treating and sulfur recovery.

The design was based on single-train operation, although the cost estimate allows for a complete spare gasification train and compression train together with adequate spares for the other areas.

Gasification (Area 15). The system selected for the study was the Texaco Coal Gasification Process with total direct gas quench. Five sections in this area—Slurry Preparation, Gasification, Gas Cleaning and Scrubbing, Wastewater Treatment, and Shift—are designed for the required makeup hydrogen production, allowing 1% total gas losses through the entire makeup hydrogen producing facility. The design capacity for both the Base Case (filtration MAR and coal) and the Coal Feed Case follows:

<u>Plant capacity</u>	<u>Base Case Required/design</u>	<u>Coal Feed Case Required/design</u>
(H ₂ +CO) moles/hr	5,534/5,592	5,534/5,592
MM scfd	50.40/50.90	50.40/50.90

Gas Treating (Area 16). Three units: Selexol, DEA, and Ammonia Sulfide Water Stripping.

The Selexol Unit removes acid gases from the shifted gasifier effluent. The design capacity for both the Base Case and the Coal Feed Case follows:

<u>Plant capacity</u>	<u>Base Case</u>	<u>Coal Feed Case</u>
Shifted syngas		
moles/hr (dry)	11,469	9,844
MM scfd (dry)	104.45	89.65
Product hydrogen (no loss basis)		
<u>Capacity:</u>		
(H ₂ +CO) moles/hr	5,592	5,592
MM scfd	50.90	50.90

The specifications were the same for both cases:

Hydrogen	95.0 mole % (min)
Carbon monoxide	3.0 mole % (max)
Nitrogen and argon	2.5 mole % (max)
Carbon dioxide	2,000 ppmv (max)
Methane	1,600 ppmv
Hydrogen sulfide	5 ppmv (max)

The DEA Unit treats the HP gas and LP fuel gas streams from the SRC Process Unit (Area 12) and the LP fuel gas stream from the Coker Calciner Unit (Area 21). The design capacity for both the Base Case and the Coal Feed Case follows:

<u>Plant capacity</u>	<u>Base Case</u>	<u>Coal Feed Case</u>
Total DEA circulation rate	600 gpm	600 gpm
HP gas purity:		
H ₂ S	1 ppmv	1 ppmv
CO ₂	10 ppmv	10 ppmv
LP gas purity:		
H ₂ S	160 ppmv	160 ppmv

The Ammonia Sulfide Water Stripping Unit processes all contaminated water originating in the entire SRC facility. Design capacity and product stream purity follow:

	<u>Base Case</u>	<u>Coal Feed Case</u>
Design capacity	160 gpm	160 gpm
Product stream purity:		
H ₂ S	3 ppmv	3 ppmv
NH ₃	30 ppmv	30 ppmv

Compression (Area 17): The Hydrogen Compression Unit compresses the makeup hydrogen from the Selexol Unit and the recycle hydrogen from the Hydrogen Purification. Design capacity follows:

<u>Makeup hydrogen</u>	<u>Base Case</u>	<u>Coal Feed Case</u>
Capacity, moles/hr	5,719	5,631
lb/hr	18,412	15,100
MM scfd	52.09	51.28
Suction, psig/°F	685.5/84.0	685.5/84.0
Discharge, psig		
normal/max	1,850.5/2,250.5	1,850.5/2,250.5
<u>Recycle hydrogen (the same for both cases)</u>		
Capacity, moles/hr	7,485	
lb/hr	33,341	
MM scfd	68.17	
Suction, psig/°F	1,445.50/63.0	
Discharge, psig		
normal/max	2,300.5/2,700.4	

Sulfur Recovery (Area 18): The Claus Unit and Beavon Sulfur Removal Unit in the Sulfur Recovery Area process all acid gases generated in the Selexol Unit, the DEA Unit and the Ammonia Sulfide Water Stripping Unit.

	<u>Base Case</u>	<u>Coal Feed Case</u>
Claus Unit		
Standard plant size, LTPD ²	299	242
Sulfur in feed, LTPD	191	133
Sulfur recovery, %	95.6	95.4
Beavon Sulfur Removal Unit		
Claus tail gas capacity, MM scfd	25.09	20.59
Export fuel gas, MM scfd	5.13	5.13
Design sulfur recovery rate, LTPD	11.50	8.54

Process in Four Areas Described

Gasification Area (Area 15): Here the filter cake from the filtration deashing unit in the Liquefaction Area is gasified with the addition of some coal into environmentally acceptable slag for disposal and synthesis gas by partial oxidation. The carbon monoxide

²The standard plant size is used for a curve-type cost estimate. It is calculated on the volume of tail gas and represents the LTPD of sulfur which would be in the feed if it contained 90% H₂S and no NH₃.

and water vapor in the synthesis gas are converted to hydrogen and carbon dioxide by passing the gas over a sulfur resistant shift catalyst.

The Feed Preparation Section produces a water slurry from coal, filter cake, and a clarifier underflow.

The Gasification Section produces hot, steam-saturated, particulate-free synthesis gas containing a total of 50.90 MM scfd hydrogen and carbon monoxide for the subsequent shift section.

The Shift Section shifts more than 97% of the carbon monoxide in the POX effluent gas to hydrogen in the presence of sulfur—resulting in a product gas, after acid gas removal, that contains 50.90 MM scfd hydrogen and carbon monoxide, with an overall hydrogen purity of about 96%.

Gas Treating (Area 16): Gas treating removes contaminants from the various gas streams generated in the Demonstration Plant—contaminants including H_2S , COS, CO_2 , and HCl.

Of the three major units of the area, the Selexol Unit removes acid gases (H_2S , CO_2 , and COS) from the makeup hydrogen stream generated in the Gasification Area. The two-stage DEA Unit purifies an HP hydrogen-rich stream as well as an LP fuel gas stream. The Ammonia Sulfide Water Stripping Unit treats the sour water produced in the Demonstration Plant.

Selexol is a proprietary process of the Allied Chemical Corporation, using an organic solvent that absorbs acid gases from HP gas streams. The Selexol Unit treats the HP makeup hydrogen stream generated in the Gasification Area, selectively removing H_2S from the gas stream, then removing CO_2 in bulk. The process generates an H_2S -rich acid gas that can be fed directly to a conventional Claus Sulfur Recovery Unit, as well as a CO_2 -rich gas stream that can be vented to the atmosphere without further processing. The sweetened hydrogen gas after desulfurization and CO_2 removal is sent to the Compression Area.

The DEA process has been used commercially for many years to remove acid gases from natural gas and petroleum refinery gas systems. It treats two different gas streams in the SRC facility: an HP hydrogen-rich stream from the SRC Process Area and an LP combined fuel gas stream.

The sour water streams generated in the SRC facility are combined before entering the Ammonia Sulfide Water Stripping Unit. The stripped water contains about 3 ppmw H_2S and 30 ppmw NH_3 . The stripped gases from the top of the stripper combine with the gases from the flash drum and then flow to the Claus Unit (Area 18).

Compression (Area 17): The makeup hydrogen from the Selexol Unit in the Gas Treating Area and recycle hydrogen from the Hydrogen Purification Area, which are compressed here, provide the hydrogen required in the SRC Process Area.

Sulfur Recovery (Area 18): The Sulfur Recovery Unit makes the plant tail gases environmentally acceptable, so they can be vented directly to the atmosphere. It also produces 200 LTPD of salable sulfur. A portion of the SRU treats an LP fuel gas stream for removal of residual sulfur compounds. The SRU contains two processing units: a Claus Unit and a Beavon Sulfur Removal Unit (BSRU). The Claus Unit recovers the bulk of the sulfur from the SRC plant acid gases. The BSRU provides the final step of sulfur removal.

Delta Construction Costs Summarized

On the basis of the design and process described above for the four areas, there would be substantial differences in the cost of constructing the makeup hydrogen facility, de-

pending on whether gasifier feed was a combination of filter cake and coal (Base Case) or just coal alone (Coal Feed Case). The delta values, in millions of October 1979 dollars and all favoring the Coal Feed Case, follow:

Makeup hydrogen facility	
Gasification Unit	\$ -7.15
Selexol Unit	-2.70
Claus Unit	-0.60
Beavon Sulfur Removal Unit	-0.82
Compression Unit	0.00
Total delta construction cost	\$-11.27
Other delta project costs	
Home office	\$ -2.14
Oxygen plant	-9.00
Turbogenerator Unit	-2.24
Contingency	-4.93
Total delta project cost	\$-29.58

* * *

ALTERNATIVE SCHEMES FOR MAR DISPOSAL

This study, which dealt with K-MAC from the Kerr-McGee Critical Solvent Deashing Process, was a literature review and preliminary assessment of the processing schemes available to render the residue environmentally acceptable as landfill or for similar disposition.

In the absence of a definition of what exactly was environmentally acceptable, Parsons assumed that when sulfur compounds have been thermally reduced to a level that was stable at ambient conditions, the residual carbonaceous material was also acceptably inert. Similarly, it was assumed that when most of the volatile matter was removed by combustion, the remaining carbonaceous material and sulfur would have been reduced to acceptable levels.

Four Alternatives Identified

As a result of the literature review, Parsons determined that there were several alternative processes to treat the mineral ash residue. The most promising of these are:

- (1) **Fluid Bed Combustion and Desulfurization.** This is a singlestep processing scheme that reduces the carbonaceous material and sulfur in the K-MAC by combustion in a fluidized bed of dolomite, which absorbs the sulfur oxides from the flue gas. Heat is recovered by generating steam. Being a commercial process and single-step, it should require a minimum operational staff. Impure gypsum is a byproduct.

- (2) **Powdered Coal Combustion in Utility Boiler.** Also a well-established commercial reality. A process for purifying mineral ash residue, it produces a product of nearly pure ash. It involves no feed preparation, but stack gas cleanup will be required to remove sulfur oxides from the flue gas. Several stack gas desulfurization processes are available.
- (3) **The IMP/CE-Lummus Thermal Desulfurization Process.** An interesting new process. It gasifies the carbonaceous material and sulfur. With conventional gas processing, it produces elemental sulfur and sweet fuel gas as byproducts. But it has been demonstrated only in a small pilot plant.
- (4) **Incineration, Steam Generation, and Flue Gas Scrubbing.** A three-step processing sequence. All three separate steps are currently offered commercially. This scheme probably requires more operating personnel than either the Fluid Bed or the Utility Boiler alternatives.

Any of the above processes, and probably others, should be capable of rendering the mineral ash residue environmentally acceptable. The preliminary review selected the Fluid Bed Combustion and Utility Boiler Cases in particular as worthy of further consideration.

Feed Composition

In evaluating these four schemes, it was assumed that the K-MAC would be received as a dry powder at 400°F under an inert gas blanket. The powder size would be 100% less than 50 microns and 50% less than 5 microns. It would probably be pyrophoric.

The composition on a critical solvent-free basis is:

Unconverted coal	23.36 wt %
Ash	39.15
SRC	37.18
Heavy oil	0.31
Total	100.00

Ultimate analyses:

	Unconverted coal	SRC	Heavy oil	Total
C	72.66 wt %	86.87 wt %	87.30 wt %	81.43 wt %
H	4.54	6.01	7.80	5.46
N	1.64	2.05	0.75	1.89
S	16.64	0.82	0.47	6.88
O	4.52	4.25	3.68	4.34
MAF ³	100.00	100.00	100.00	100.00

³MAF: moisture and ash free.

Heating values (Dulong formula): 9232 Btu/lb (gross) and 8924 Btu/lb (net).

Ash analysis:

P ₂ O ₅	0.2 wt %
SiO ₂	44.6
FeO	25.5
Al ₂ O ₃	22.2
TiO ₂	0.7
CaO	1.6
MgO	0.6
SO ₃	1.3
K ₂ O	2.2
Na ₂ O	0.8
Undetermined	0.3
Total	100.00

Fluid Bed Combustion and Desulfurization

This is a combined single-step process. It devolatilizes and desulfurizes the fuel feed by combustion and removes the sulfur oxides from the flue gas as they are produced. The heat of combustion is absorbed in a heat exchange coil. The heat is generally used to raise steam.

The process carries out combustion in a fluidized bed of pulverized dolomite or limestone. The stone picks up the sulfur oxides essentially as sulfate. At present, this is a once-through process discarding a gypsum product. Some licensors are developing processes to regenerate and recycle the dolomite or limestone.

Several proprietary processes are in development. At least one, using Foster-Wheeler Energy Corporation techniques, has reached the commercial stage. A 30 MW boiler has been in operation by the Monongahela Power Company in their Rivesville, WV, power station since 1977.

The process offers several advantages. Combustion and desulfurization take place in the same vessel. The presence of the steam generating coil allows positive temperature control without wasting water in quench sprays. Steam production, or other process heat use, is an energy-conserving byproduct. Combustion can be carried out at low temperature levels, avoiding ash fusion and minimizing NO_x production.

A disadvantage in the present state of the art is the continuous consumption of dolomite or limestone and the need to dispose of the gypsum produced in the flue gas desulfurization.

Because of the MAR's high (4.2%) sulfur and very high (39%) ash, specific licensor information will be needed to develop valid economic data.

Process Description. The fluidized bed combustion process seems to be a practical method of cleaning up mineral ash residue without using separate flue gas scrubbing.

The process block flow diagram is shown in Figure 1. The MAR is burned in a fluidized bed, a term applied to a method of burning a fuel, usually solid, in a bed of other fluidized solids. For flue gas desulfurization, the other fluidized solid would be limestone or dolomite, which react with sulfur dioxide, as follows:

When dolomite or limestone is used on a once-through basis to remove 90% sulfur dioxide, the result is a high waste disposal burden. To reduce the high limestone feed rate, a system is now under study in which the CaSO_4 would be regenerated back to CaO in a separate fluidized bed by reaction with a reducing gas, as follows:

The regenerated CaO would be returned to the boiler where it would again react with SO_2 and O_2 . SO_2 in the regenerator off-gas is at a high enough concentration for sulfur recovery. Since the regeneration section is still in the pilot stage, it is shown as a future item to be added after it has been demonstrated commercially.

The K-MAC, which consists of 50 micron and smaller particles, will be burned to a bed temperature of 1500°-1600°F, which is below its ash fusion point. Because of the low bed temperature, NO_x formation will be well within current EPA standards. Most of the ash particles will end up in the flue gas to be removed as flyash by a conventional electrostatic precipitator. Spent limestone and dense pyritic ash will be withdrawn from the bed by a gravity drain system.

High heat transfer coefficients between the fluidized bed and immersed steam generation surfaces reduce the boiler cost. Capital cost savings on boiler surface and elimination of the scrubber system may make the fluid bed system cheaper than a conventional fired boiler with a scrubber. Operating costs are estimated to be roughly half those of a conventional boiler with a scrubber system.

Pilot test work will be required to obtain process data based on the fluid bed combustion of K-MAC to produce an environmentally acceptable residue.

References

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- E. C. McKenzie, "Burning Coal in Fluidized Beds," Woodall-Duckham, Ltd., Sussex, England, *Chemical Engineering*, August 1978.
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The IMP/CE-Lummus Thermal Desulfurization

In this intriguing alternative, the only solid waste stream for disposal is the residual ash. Sulfur is removed from the K-MAC by pyrolysis and recovered from the resulting gas stream

by conventional means. Elemental sulfur and sweet low-or-medium-Btu fuel gas are recovered as byproducts.

Process heat needs are supplied by partial oxidation of the K-MAC. Either air or oxygen may be used for this purpose, yielding the corresponding low-or-medium-Btu gas product.

But the literature indicates that this process has not developed beyond a pilot plant scale. Further, the process was conceived as a means of upgrading high sulfur petroleum coke. K-MAC would have to be tested to determine the effectiveness of the process. Until it has been tested in this application, it is not possible to develop cost and utility estimates.

Process Description. As presently conceived, the process utilizes a three-zone moving bed reaction chamber. The coke feed is heated up to desulfurization temperature in the upper zone. The middle zone is the desulfurization reactor. The desulfurized coke passes down into the bottom zone for cooling by heat exchange with recycling gas.

The coke is heated and pretreated by partial oxidation using a stream of air and gas from the product cooler. The offgas, containing distilled volatile matter from the coke, passes through a dust separator and a scrubber to remove condensable hydrocarbons. The remaining uncondensed gas is compressed and recycled to the bottom zone.

Preheated coke flows into the reactor for desulfurization. The degree of sulfur removal is dependent on a time-temperature relationship. A coke of 4.7 wt % sulfur is shown to be reduced to 2.6% in three hours at 2400°F, or to 0.5% in nine hours at 2730°F. The offgas consists of pyrolyzed sulfur, as H_2S , COS and CS_2 , light hydrocarbons, and some purge gas from the bottom zone. This gas is dedusted, cooled, compressed, and combined with the preheater offgas. The total gas is then desulfurized and recycled to the bottom zone to cool the product coke. Excess sweetened gas is withdrawn for use as fuel.

This looks like a clean, simple concept. The most conspicuous drawback is the very large reactor needed to provide three-to-nine hours of residence time. The pilot plant tests revealed a few other drawbacks. The petroleum cokes tend to get "tacky" on heat-up, although K-MAC might not have this problem. Pilot plant tests would be required to determine whether agglomerated K-MAC would maintain particle integrity through the heating, desulfurizing, and cooling steps.

Tests would also be needed to determine if the K-MAC has enough hydrogen to satisfactorily desulfurize itself. If not, additional hydrogen would have to be added to the recycle gas stream.

More fundamental is the need to demonstrate that the process can handle a material with 39 wt % ash and about 50% carbon. Figure 2 shows the facilities needed to process K-MAC using the IMP/CE-Lummus process. The existing sulfur recovery unit should be able to handle the acid gases from the added units without an increase in size. However, if more coal were to be gasified to supply the hydrogen that had been obtained from K-MAC in the Base Case, the sulfur yield would be increased. The fractionation and acid gas removal facilities shown here could possibly be integrated into the Base Case units. These points can only be cleared up by tests with K-MAC.

References

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Utility Boiler With Flue Gas Scrubbing

The relatively high heating value of the K-MAC suggests that it could simply be burned as a powdered fuel in a utility boiler. If the large output of about a million pounds per hour of steam could be used, such a scheme could well be the most economical alternative.

The residue left for disposal would be little more than pure ash. However, the problem of disposal of the sulfur extracted from the flue gas sulfur would remain, as in the incineration case.

Combustibility of this high ash fuel (39%) could be easily tested and proven before proceeding with any design.

* * *

COSTS, ENVIRONMENTAL IMPACT OF THE THREE-STEP ALTERNATIVE

The fourth alternative mentioned above—Incineration, Steam Generation, and Flue Gas Scrubbing—was the subject of a separate study by Parsons. The purpose was to assess this alternative's costs and environmental impact.

Results of the study indicate that the proposed multihearth incinerator/incineration boiler/flue gas desulfurization (FGD) system would convert approximately 50% of the carbon and 30% of the sulfur contained in the feed K-MAC to combustion products. The resulting solid to be disposed of would be reduced by about 41,300 lb/hr.

Incineration produces approximately 3300 lb/hr of SO_2 , which is treated in a double alkali FGD system. Stack emissions from the process are approximately 330 lb/hr as SO_2 . Assuming that regulations limit the SO_2 emission to 1.2 lb/MM Btu heat release, the allowed stack emission can be as high as 640 lb/hr.

To translate this conceptual process into a plant would cost approximately \$61 MM. The economic data presented herein should be used as a "best estimate" only, adequate for a conceptual design feasibility study. A more reliable cost estimate would require extensive piloting and scale-up efforts.

Chemical requirements are about 90 lb/hr Na_2CO_3 and 3160 lb/hr CaO. Energy requirements are about 3700 kw and water requirements exceed 970 gpm. The system produces 380,000 lb/hr of superheated steam.

The net annual operating cost of the system—adding up chemicals, power, water, and operating labor, plus allowances for maintenance and capital charges, and taking a credit for steam—would be \$5.11 MM. This translates to a negative derived value for K-MAC, $-\$0.53/\text{MM Btu}$. Costs are calculated in 1979 \$ and based on 330 operating days per year.

The apparatus for the system is readily available, but the system as such has never been proved. Piloting would be needed prior to final scale-up design.

Combustion Sequence

The proposed scheme for producing an environmentally acceptable K-MAC consists of a minimum combustion process using a multihearth incinerator with limited oxygen sup-

ply, followed by combustion of the volatiles in a conventional incinerating boiler. It is possible to reduce the carbon content of the ash to very low values through additional combustion; however, it is suspected that a certain amount of residual carbon in the treated K-MAC may be desirable to retard leaching by water. The multihearth incinerator/boiler combination is an attractive sequence from an economical point of view since steam is produced and quenching water is minimal.

Roasting in a multihearth incinerator eliminates free reacting sulfur compounds and reduces undesirable products, which might be generated at higher combustion temperatures.

Minimal treatment would require heating the material to a temperature in the range of 1000° to 1200°F to devolatilize it and combust the volatiles evolved. Such a treatment might increase the ash content from approximately 39% by weight to over 50% by combusting about half of the carbonaceous (ash-free) K-MAC.

For this analysis, it was assumed that approximately 50% of the carbon would be converted in the above process while combusting with limited oxygen supply. This and other assumed conversions were based on comparable results from similar types of incineration devices.

After treatment the K-MAC would contain approximately 35 wt % carbon and 4.3 wt % sulfur. The overall ash content of the treated product will be about 57 wt % (dry basis).

The estimated composition of the treated K-MAC follows:

	Converted		Ash to disposal		
	%	lb/hr	lb/hr	wt %	wt %
C	51	33,664	31,732	81.39	35.0
H	79	3,475	907	2.33	1.0
N	52	789	725	1.86	0.8
S	30	1,637	3,898	10.00	4.3
O	51	1,772	1,723	4.42	1.9
MAF		41,337	38,985	100.00	43.0
Ash	0	0	51,678		57.0
Dry			90,663		100.00

Process Description

Refer to Figure 3, the conceptual process flow diagram.

The K-MAC is received as a relatively fine dry powder at 400°F under an inert gas blanket, pelletized by pressure, and fed to a multihearth incinerator. The pellets enter through the top of the unit where mechanical rakes attached to a centrally rotating shaft slowly rabble the material to either the periphery or center of consecutive hearths and there the material falls to the next hearth. This process continues in a zigzag fashion until the material exits the last hearth to a hopper area within the unit. The pellets are combusted during the rabbling and the falling from one hearth to the next. Typically the upper hearths serve to heat the material to combustion temperatures while the lower hearths serve to eliminate volatiles and sulfur.

The multihearth incinerator is "oxygen starved" because the air supply is limited. The air-to-K-MAC ratio is varied to control the temperature of the devolatilized product. The

conceptual design assumes the treated material is produced at a temperature of 1200°F. However, if piloting demonstrated the necessity, the process could be designed to produce solid product at temperatures up to about 2000°F.

Hot gas from the roaster is essentially oxygen-free and contains combustible gases, high molecular weight hydrocarbons, and tar vapors. The following balance was used in the analysis:

Ash-free K-MAC combusted	6%
Ash-free K-MAC evolved as vapor	45
Ash-free K-MAC remaining as solid	49
	<hr/> 100

The hot K-MAC from the roaster is cooled by water quenching, filtered, and disposed of as landfill. The filtered water is cooled and returned to the quench.

The hot gas is further combusted in an incinerating boiler to generate steam. The boiler operates with 10% excess air. Flue gas from the boiler may be cooled to about 500°F in the convection section and further cooled to about 325°F through heat exchange with air and scrubber flue gas. A temperature of at least 325°F is desired to avoid condensation of sulfuric acid from any sulfur trioxide formed during combustion. The amount of actual sulfur trioxide formation is indeterminate, but usually in the range of 10 to 15% of the sulfur in the fuel.

Particulates are removed in an electrostatic precipitator prior to treatment in a double alkali flue gas desulfurization system for SO₂ removal, which is commercially available and can remove in excess of 90% of the sulfur contained in the feed gas. For this analysis, a 90% efficiency was assumed.

The double alkali FGD system is applicable to a variety of industrial processes with SO₂ emissions as well as to boilers burning sulfur-laden fuels. In the process, sulfur dioxide is converted to a solid disposable material by first absorbing it into a highly reactive sodium scrubbing solution and then using calcium to form the solids and recover sodium for reuse.

Sulfur dioxide is absorbed in a solution containing sodium sulfite (Na₂SO₃), sodium bisulfite (NaHSO₃), and sodium sulfate (Na₂SO₄). As SO₂ is absorbed, the sodium sulfite is converted to sodium bisulfite and a very small percentage is oxidized to sodium sulfate.

The scrubbing solution is regenerated by adding lime to precipitate calcium sulfite and sulfate. It is possible that some of the lime may be added as calcium carbonate rather than calcium oxide. The slurry solution is settled in a thickener and the supernatant liquid is returned to the scrubber. The thickened sludge is filtered and the resulting filter cake (approximately 70% solids) is disposed of along with the treated MAR. Filtrate is returned to the scrubber. Water is required for makeup losses and sodium carbonate is added to maintain solution balance. Sodium may be added as sodium hydroxide or bicarbonate.

The scrubbing solution is normally controlled at a pH of 6-7. Above a pH of 7, carbon dioxide absorption becomes significant and can lead to formation of calcium carbonate scaling. Below a pH of 6, the increase in SO₂ vapor pressure reduces the system's ability to absorb SO₂.

The regeneration of sodium takes place in the lime reactor where the reaction of sodium bisulfite with lime is controlled at a pH of 8.5. This is effectively the titrimetric endpoint for sodium bisulfite and operating at this pH assures excellent responsiveness of the control system.

Equipment List

MAR filtrate receiver (1)	8 ft dia × 16 ft T-T
Scrubber (1)	18 ft dia × 24 ft T-T
Lime storage (1)	
Crystallizer (1)	10 ft dia × 16 ft T-T
Soda storage (1)	
Sulfite receiver (1)	3 ft dia × 3 ft T-T
Residue quench tank (2)	8 ft dia × 18 ft T-seam
Multihearth incinerator (2)	71 MM Btu/hr (liberated)
Incinerating boiler (1)	535 MM Btu/hr (liberated)
Slurry water cooler (1)	32.2 MM Btu/hr
Air heater (1)	16.5 MM Btu/hr
Flue gas reheater (1)	7.4 MM Btu/hr
MAR slurry pump (4)	1,500 gpm, 25 psi
MAR filtrate pump (2)	1,300 gpm
Scrubber bottoms pump (2)	650 gpm
Slurry pump (2)	650 gpm
Slurry pump (2)	50 gpm
Scrubber feed pump (2)	700 gpm
Filtrate pump (2)	35 gpm
Flue gas blower (2)	126,125 scfm
Pelletizer (1)	72 tons/hr
Precipitator (1)	116,500 scfm
Flyash conveyor (2)	5 tons/hr
MAR filter (1)	
Stack (1)	
Mixer (1)	
Mix tank (1)	7,000 gallon capacity
Thickener (1)	
Sulfate filter (1)	

Capital Costs (1979 \$)

Equipment	\$18,986,000
Bulk materials	19,324,000
Indirect costs	9,057,000
Total constructed cost	\$47,367,000
Home office/overhead	5,684,000
Total capital cost	53,051,000
Contingency	7,949,000
Total plant cost	\$61,000,000

Operating Costs (1979 \$)

Chemicals	
90 lb/hr Na ₂ CO ₃ @ \$60/T	\$ 21,000
3160 lb/hr CaO @ \$30/T	375,000
Power	
3720 kw @ \$0.036/kwh	1,060,000
Water	
Boiler feed, 840 gpm @ \$2/Mg	798,000
Process, 134 gpm @ \$0.45/Mg	29,000
Operating labor	
One person/shift	110,000
Maintenance (4% of investment)	2,440,000
Total operating costs	\$ 4,833,000
Capital charges @ 16%	9,760,000
Total annual cost	\$14,593,000
Steam credit	
380 M lb/hr @ \$3.15 M lb	(9,480,000)
Net annual cost	\$ 5,113,000

Derived K-MAC value

1585 TPD @ 1847 MM Btu/T	
or 9.66×10^{12} Btu/yr	\$0.53/MM Btu

Other Data

Solid waste	45 tons/hr (excluding water)
SO ₂ emissions	330 lb/hr
Allowable SO ₂ emissions	640 lb/hr
Steam generated	380,000 lb/hr, 650 psig

The multihearth incineration/steam generation/flue gas scrubbing process appears feasible for rendering the K-MAC "environmentally acceptable for disposal." The costs reported should be within a reasonable range of accuracy for a budget-type cost estimate. But additional testing and piloting are necessary to establish final design parameters and these could affect the actual costs.

The total constructed cost of \$61,000,000 includes redundancy for mechanical equipment which might fail under normal use such as pumps, blowers, etc. Accordingly, the process should have a fairly high reliability rating.

Chemical requirements for the flue gas scrubbing sub-system are available and not excessive considering the quantity of gas to be treated.

The main point of uncertainty here is the definition of "environmentally acceptable for disposal." The assumptions made for this analysis appear to be valid, but current or future environmental regulations may require different standards.

GKT COAL GASIFICATION: K-MAC FEED vs COAL FEED

As in the first study above, the objective in this later study was to investigate the actual cost of disposing of a mineral ash residue by gasification—except that here the material was K-MAC and the process GKT Coal Gasification.

The approach was also similar—to derive a delta cost estimate by comparing two cases producing the same makeup hydrogen requirement (56.92 MM scfd $H_2 + CO$) for the Demonstration Plant:

- (1) Gasifying K-MAC exclusively via GKT Coal Gasification.
- (2) Gasifying coal exclusively via GKT Coal Gasification.

Again, for the purpose of comparison, a total plant concept for the production of hydrogen was considered and, therefore, facilities for oxygen production, coal and K-MAC gasification, shift, acid gas removal (Selexol Unit), sulfur recovery, sour water stripping, and hydrogen compression were included in the evaluation. Although the DEA Unit in the Gas Treating Area is not part of the makeup hydrogen producing facility, it was also included in both cases.

The evaluation was made in the same terms as the first study: feed requirements, process capacity requirements, product purity, overall utility, and construction cost.

The delta cost estimate that came out of this work gave the advantage to K-MAC. Specifically, gasifying K-MAC by the GKT process would increase the cost of the project by approximately \$0.6 MM, compared to gasifying coal by that process—but K-MAC would require less service from utilities than would coal, substantially decreasing the annual cost of utilities by \$13.514 MM. With capital-related charges of 20% of the total delta project costs, GKT disposal of K-MAC would decrease annual operating costs by \$13.438 MM—which works out to a positive derived value for K-MAC, +\$1.33/MM Btu. In sum, gasification of K-MAC using the GKT technology is far more economical than disposal by the three-step alternative (Incineration, Steam Generation, and Flue Gas Scrubbing) discussed above.

A net deficit of HP steam (1050 and 835 psig) resulted from the high steam requirements of the shift section. Because of this, capital costs for a boiler and accessories were included in these estimates (and a study has been initiated of a saturator-cooler scheme for the shift section with the goal being to minimize HP steam consumption).

Plant Areas Studied

The same plant areas were examined as in the first study, but the design of the Gasification Area was based on four-train operation. The other areas—Gas Treating, Compression, Sulfur Recovery—were single train.

Gasification (Area 15). In the GKT Coal Gasification Process, the area consists of six sections: Solid Feed, Gasification and HP Steam Generation, Gas Cleaning and Scrubbing, Wastewater Treatment, Compression, and Shift. The design capacity for both cases follows:

<u>Plant capacity</u>	<u>K-MAC feed case</u>	<u>Coal feed case</u>
Design		
($H_2 + CO$), moles/hr	6,249	6,249
MM scfd	56.92	56.92

Gas Treating (Area 16). Three units: Selexol, DEA, and Ammonia Sulfide Water Stripping.

Design capacity for the Selexol Process:

<u>Plant capacity</u>	<u>K-MAC feed case</u>	<u>Coal feed case</u>
Shifted syngas		
moles/hr (dry)	11,402	11,311
MM scfd (dry)	103.84	103.02

Product hydrogen (no loss basis)

<u>Capacity:</u>		
(H ₂ +CO) moles/hr	6,249	6,249
MM scfd	56.92	56.92

Specifications (same for both cases):

Hydrogen	95.0 mole % (min)
Carbon monoxide	3.0 mole % (max)
Nitrogen and argon	2.5 mole % (max)
Carbon dioxide	2,000 ppmv (max)
Methane	1,600 ppmv
Hydrogen sulfide	5 ppmv (max)

DEA Gas Treating Process design capacity:

<u>Plant capacity</u>	<u>K-MAC feed case</u>	<u>Coal feed case</u>
Total DEA circulation rate	650 gpm	650 gpm
HP gas purity:		
H ₂ S	1 ppmv	1 ppmv
CO ₂	10 ppmv	10 ppmv
LP gas purity:		
H ₂ S	160 ppmv	160 ppmv

Ammonia Sulfide Water Stripping Process capacity and product stream capacity:

	<u>K-MAC feed case</u>	<u>Coal feed case</u>
Design capacity	170 gpm	170 gpm
Product stream purity:		
H ₂ S	3 ppmw	3 ppmw
NH ₃	30 ppmw	30 ppmw

Compression (Area 17). Design capacity:

<u>Makeup hydrogen</u>	<u>K-MAC feed case</u>	<u>Coal feed case</u>
Capacity, moles/hr	6,302	6,283
lb/hr	18,918	18,275
MM scfd	52.40	51.32
Suction, psig/°F	685.5/73.8	685.5/73.5
Discharge, psig normal/max	1,850/2,250.6	1,850.5/2,250.5

Recycle hydrogen (the same for both cases)

Capacity, moles/hr	11,241
lb/hr	49,774
MM scfd	102.38
Suction, psig/°F	1,445.50/63.0
Discharge, psig normal/max	2,300.5/2,700.4

Sulfur Recovery (Area 18). Design capacity:

	<u>K-MAC feed case</u>	<u>Coal feed case</u>
Claus Unit		
Standard plant size, LTPD	287	263
Sulfur in feed, LTPD	168	142
Sulfur recovery, %	95.7	95.4
Beavon Sulfur Removal Unit		
Claus tail gas capacity, MM scfd	24.6	22.39
Export fuel gas, MM scfd	6.17	6.17
Design sulfur recovery rate, LTPD	10.90	9.26

Process Variations Described

Gasification (Area 15): In this GKT scheme, a Solid Feed Section delivers dry solid feed to the gasifier. The section includes equipment for surge storage and feeding of ground coal and K-MAC.

The Gasification Section produces cooled, particulate-free synthesis gas containing a total of 56.92 MM scfd hydrogen and carbon monoxide for the subsequent shift section.

The oxygen, steam, and K-MAC or coal react at a slight positive pressure (about 0.6 psig) and high temperature. The hot syngas exiting the gasifier is water-quenched to solidify entrained slag droplets, then further cooled by generating HP saturated steam via a waste heat boiler (WHB). The WHB effluent gas is next directed into a cyclone-scrubbing system for final carbon and particulate removal. The particulate-free syngas is then ready for further processing in the compression section.

The Compression Section compresses the total particulate-free syngas (71.48 to 73.81 MM scfd) to design pressure prior to shift. This section consists of a single compression train together with adequate spares aiming at the achievement of the overall facility on-stream factor.

The Shift Section shifts better than 96% of the carbon monoxide in the POX effluent gas to hydrogen in the presence of sulfur. This results in a product gas, after acid gas removal, containing a total of 56.92 MM scfd hydrogen (no loss basis) and carbon monoxide with an overall hydrogen purity of about 96%.

The particulate-free syngas from the Compression Section is first mixed with the required shift steam. The mixed shift feed is next preheated by exchange with the first shift reactor effluent, then shifted in three stages with interstage cooling. The hot shifted gas from the third stage, after cooling to the design temperature, is ready for further processing in the Selexol Unit (Area 16).

Gas Treating, Compression, and Sulfur Recovery (Areas 16, 17, 18). With the changes in design capacity noted above, these areas function essentially as described in the first study.

Delta Construction Costs Summarized

The values shown below, in millions of first quarter 1979 dollars, are the differences realized for the GKT Coal Gasification Process when comparing the Coal Feed Case with the K-MAC Feed Case. All negative values represent savings for the Coal Feed Case:

Makeup hydrogen facility

Gasification Unit	\$+0.02
Selexol Unit	-0.04
Claus Unit	-0.25
Beavon Sulfur Removal Unit	-0.40
Compression Unit	0.00
Boiler and accessories	+0.06
Total delta construction cost	<u>\$-0.61</u>

Figure 1
MAR Disposal: Fluid Bed Combustion and Desulfurization

Figure 2
MAR Disposal: IMP/CE-Lummus Thermal Desulfurization

Figure 3 (Sheet 1 of 2)
MAR Disposal: Multihearth Treatment

Pelletizer 72 tons/hr.	Multi-hearth incinerator 71 MMBtu/hr. (liberated)	Residue quench tank 8'-0" Dia. X 18'-0" T-ssm	Slurry water cooler 32.3 MMBtu/hr.	Incinerating boiler 535 MMBtu/hr. (liberated) 515 MMBtu/hr. (absorbed and lost)	MAR Filtrate receiver 8'-0" Dia. X 16'-0" T-T	MAR filter	Precipitator 116,500 SCFM 231,000 ACFM	Flue gas blower 126,125 SCFM 193,600 ACFM 2875 Theo. HP 4100 Act. HP	Flue gas heater 7.4 MMBtu/hr.	Scrubber 18'-0" Dia. X 24'-0" T-T
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Figure 3 (Sheet 2 of 2)
MAR Disposal: Multihearth Treatment

NOTES:

1. Material balance assumes all sulfur becomes CaSO_3 - actually 10-15% is expected as CaSO_4 .
2. Material balance is for 100% sulfur recovery, approx. 90% is expected.

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TWO SLURRY SURGE TANKS RECOMMENDED OVER ONE

Lalitkumar H. Udani*

A comparison of two cases in the slurry preparation system of the 6000 tons per day (TPD) SRC-I Demonstration Plant was made to determine the advantages in operation and economics.

The two cases evaluated were the base case (Case I), provided for in the Phase 0 design, using two Slurry Surge Tanks (V-1201 A and B)—and the alternate case (Case II), using a single Slurry Surge Tank (V-1201).

The trade-off study (80-02-06), done by Catalytic, Inc., indicated that the two-tank system was considerably more reliable.

It was found that the use of a single Slurry Surge Tank would result in marginal capital savings (\$125,000—1979 \$) and minimal energy savings (10 hp). See Tables 2 and 3. Identical home office costs were applied in each case.

But more important, with the use of the single-tank system, repairs to the Slurry Tank or to the Agitator (A-1201) could result in coal feed interruptions and loss of SRC production. For minor repairs to the Agitator, the plant could be kept operating by feeding process solvent to the coal feed pumps. But if there were a major Agitator breakdown, the plant would shut down.

With the two-tank system, on the other hand, the plant operation could be maintained without interrupting the coal feed. If an Agitator were to fail, the plant would not shut down.

Moreover, in the Phase I design a much finer coal particle size (70% through 200 mesh) will be used, as compared to that used in the Phase 0 design (90% through 20 mesh). To maintain the same reliability of the slurry preparation system as in Phase 0 design, a greater equipment redundancy is required for the Phase I design, making a system with two Slurry Surge Tanks even more desirable.

CASE I—THE BASE CASE WITH TWO SLURRY SURGE TANKS

In the base case, the slurry preparation system is divided into two trains, each designed to operate at 50% of full load but capable of operating at full load (see Figure 1).

In each train, pulverized coal is charged to the agitated Slurry Surge Tank—by means of a Bin Discharge Screw Feeder (CV-1201) and a Weigh Belt Feeder (CV-1202) under ratio-control with process solvent feed, to produce a 38.5% slurry.

Water and hydrocarbon vapors and dust leaving the Tank are scrubbed in a Scrubbing Eductor (J-1201) with process solvent circulated by a Scrubber Circulating Pump (P-1203).

*Catalytic, Inc.

The vapors are then separated from the solvent in the Scrubbing Solvent Separator (V-1202).

A recovery system—consisting of a Mixed Phase Condenser (E-1201), a Mixed Phase Receiver (V-1203), and a Mixed Phase Condensate Transfer Pump (P-1204)—common to both trains is designed to condense vapors and separate the condensate from non-condensables.

From the Slurry Tank, slurry is pumped by a Slurry Recycle Pump (P-1201) to a set of three high-pressure reciprocating Slurry Feed Pumps (P-1202).

Slip streams—from the Slurry Recycle Pump discharge and high-pressure Slurry Feed Pump suction—are recycled to prevent dead-heading of the suction manifolds of the Slurry Feed Pumps.

Process Solvent is charged to the Scrubbing Solvent Separator first and then fed to the Slurry Surge Tank by gravity, along with coal dust recovered in the Scrubbing Eductor.

Equipment that is critical to the reliable operation of the slurry preparation system is:

- Coal Feed System
- Slurry Surge Tank
- Slurry Surge Tank Agitator
- Slurry Recycle Pumps
- Scrubbing System

Coal Feed System

The coal feed system is used to weigh and feed coal to the Slurry Surge Tank. It includes a Screw Feeder and Weigh Belt Feeder, Rotary Air Lock Valve (AL-1201), slide valves, and controls. The system is designed for a full load of 500,000 lb/hr with a 2:1 turndown ratio. Experience has indicated that these feeders handling coal dust are high maintenance items due to possible mechanical failures, drive failure, wear and tension loss on the belt, weight control, etc.

In the base case, each Slurry Surge Tank is equipped with a feed system that can handle a full load of 500,000 lb/hr but normally operates at 50% capacity. It is, therefore, not necessary to provide a spare feed system to each Tank as both Tanks and their auxiliary equipment are mutual spares. On failure of one feed system the second Tank is operated at full load without any downtime or loss of production.

Slurry Surge Tank

The Slurry Surge Tank is an atmospheric, vertical tank designed to provide a nominal 15-minute residence time, allowing for coal particles to be wetted and form the slurry. In case of an Agitator failure, the Slurry Tank can be pumped out in a relatively short time before the solids settle out in the tank. Normally, the liquid level in the Tank is maintained at the half-full mark. When operating at full load, the level can be raised to the three-quarter-full mark to allow a 10-minute residence time. A single blade turbine Agitator (15 hp) with four baffles in the Tank is deemed sufficient to maintain the slurry integrity. The Surge Tank used is 18 ft 6 inches in diameter and 18 ft 6 inches T to T, with a 46,000 gallon capacity.

The Tanks are installed within a 120 ft X 52 ft X 38 ft 6 inch structure available in Area 11 at grade. The auxiliary equipment is mounted on the adjoining structure.

Agitator

The Slurry Surge Tank Agitator is a single-blade turbine agitator. Each of the two agitators is equipped with a 15 hp motor. These Agitators are considered highly reliable. The SRC-I Pilot Plant at Wilsonville has not experienced any breakdown or maintenance problems with the Slurry Tank Agitator, which is run continuously. Modern agitator design, where the shaft is installed separately from the drive mechanism, makes for easy maintenance and extreme reliability. Agitator manufacturers promise a prompt supply of spare parts from Parts Exchange Programs maintained by equipment manufacturers. Good service will, of course, minimize downtime. Maintaining warehouse spares for Agitator parts will further increase the reliability and availability of the Agitator.

With the two Slurry Surge Tanks, any maintenance to an Agitator can be made without a loss of coal feed to the process by operating the Slurry Surge Tank of the functioning train at full load.

Slurry Recycle Pumps

The Slurry Recycle Pumps are centrifugal pumps. Each Slurry Recycle Pump feeds a 38.5% coal slurry to a set of three Slurry Feed Pumps at a minimum pressure of 50 psig. A 10% slip stream from each Slurry Feed Pump is returned to the Slurry Surge Tank to keep the coal slurry moving in the suction line and prevent "dead heading" the suction manifold. A bypass stream of 100 gpm is returned to the Surge Tank from the discharge of the Slurry Recycle Pump. This latter slip stream can be increased when it becomes necessary to reduce the forward flow. The Recycle Pump is rated at 1420 gpm-1200 gpm forward flow and a total of 220 gpm recycle.

Each Recycle Pump is provided with a 100% spare unit connected separately to the same Tank. This has been specified in order to avoid cross-connection of pump suction lines that may lead to line pluggage. The spare units are connected so that they can supply either set of Slurry Feed Pumps. This arrangement ensures full production even when one of the Tanks is out of commission.

Scrubbing System

The scrubbing system includes one Eductor, one Separator, and one Circulating Pump, sized for 100% of full load. Each Slurry Tank has a separate scrubbing system with a common spare Circulating Pump. The Eductor is not spared as the solids content of the scrubbing fluid is not expected to exceed 1% and the problem of erosion is minimal.

Slurry Preparation System

The slurry preparation unit in the base case, therefore, includes:

- (a) Two Slurry Surge Tanks sized for 15-minute residence time at half the full load capacity and half-full level
- (b) Two full-size coal feed systems, one for each Slurry Surge Tank, including a Bin Discharge Screw Feeder, a Weigh Belt Feeder, and auxiliaries

- (c) Two Slurry Recycle Pumps, each with a 100% spare (four pumps)
- (d) Two full-size scrubbing systems with a common spare Scrubber Circulating Pump

CASE II—ALTERNATE CASE WITH SINGLE SLURRY TANK

In Case II the slurry preparation system is designed as a single train operating at 100% of full load (see Figure 2).

Pulverized coal is charged to the agitated Slurry Surge Tank, by means of a Bin Discharge Screw Feeder and a Weigh Belt Feeder under ratio-control with process solvent feed, to produce a 38.5% slurry.

Vapors and dust leaving the tank are scrubbed in a Scrubbing Eductor with process solvent circulated by a Scrubber Circulating Pump. The vapors are then separated from the solvent in the Scrubbing Solvent Separator.

A recovery system—consisting of a Mixed Phase Condenser, a Mixed Phase Receiver, and a Mixed Phase Condensate Transfer Pump—is designed to condense vapors and separate the condensate from non-condensables.

The Slurry Surge Tank is equipped with two Slurry Recycle Pumps (P-1201 A and C). Each pump delivers slurry to a set of three high-pressure reciprocating Slurry Feed Pumps.

Slip streams from the Slurry Recycle Pump discharge and Slurry Feed Pump suction are recycled to prevent dead-heading of the suction manifolds of the Slurry Feed Pumps.

All equipment except the Slurry Recycle Pump is sized to handle a full load; this pump is sized for half the full load.

Process solvent is charged to the Scrubbing Solvent Separator first and then fed to the Slurry Surge Tank by gravity, along with dust recovered in the Scrubbing Eductor.

The critical equipment of the alternate slurry preparation system is examined below.

Coal Feed System

As in Case I, the coal feed system, consisting of a Screw Feeder, Weigh Belt Feeder, Rotary Air Lock Valve, etc., is a high maintenance item. If only one coal feed system were used for the Single Slurry Surge Tank Case, its failure would result in total loss of coal feed and would severely affect production, depending on the extent of maintenance required. Therefore, a 100% spare coal feed system is specified for this case.

The result is that there is no difference in the number of coal feed systems that would be installed for either case of the slurry preparation system.

Slurry Surge Tank

The Slurry Surge Tank volume for a 15-minute residence time at half-full level is 69,000 gallons. A 23 ft diameter X 23 ft T to T vertical Tank was chosen. A single turbine agitator (20 hp) with four baffles will be required. The single Tank can be installed within a 67 ft X 52 ft X 42 ft-high structure, as compared to a 120 ft X 52 ft X 38 ft 6 inch-high structure necessary for the two-tank case. The single Tank, just like the two Tanks, is installed at grade, with the coal feed and vapor recovery systems mounted on an adjoining structure

in Area 11. However, no structural cost benefits would accrue with the single Slurry Surge Tank, since the structural size of 120 ft X 52 ft X 38 ft 6 inches is fixed by other units in Area 11.

Agitator

The Slurry Surge Tank Agitator is a single blade turbine agitator equipped with a 20 hp motor. As noted in Case I, the agitator is highly reliable.

In the single Slurry Surge Tank system, the Agitator is the most critical piece of equipment—and there is no backup. Therefore, a major breakdown, due to damage to the shaft or blade, would cause a plant shutdown. A backup pump-around system to maintain the slurry integrity without an Agitator, as will be seen later, would be very large, expensive, and impractical.

Slurry Recycle Pumps

In the single Slurry Surge Tank case, two operating and two spare Slurry Recycle Pumps have been provided. They are separately connected to the Tank rather than through a common manifold. This is necessary in the slurry service as continuous flow cannot be maintained in all the branches of suction manifold. Using one Pump as a common spare, instead of two, would reduce the spare capacity by 50% and affect the reliability of the system. Therefore, in this case also, four Slurry Recycle Pumps have been provided.

Evaluation of Slurry Recycle Pumps as Mixers. The possibility of using the Slurry Recycle Pumps to maintain the slurry in the Slurry Surge Tank, in case of Agitator failure, was investigated. It was determined that for a Recycle Pump to be used as a backup for the Agitator it should be capable of turning over one-tank volume per minute. For the base case, a pumping capacity of 20,000 gpm is required. However, the pump size can be reduced by a factor of four by pumping the slurry through a set of submerged mixing Eductors at the bottom of the Tank.

For example, in the base case, to replace an Agitator, 33 2-inch mixing Eductors (SS316) are required per Tank along with a 5000 gpm Slurry Recycle Pump. The Pump motor is rated at 320 bhp. This is 21 times the power required for an Agitator. For Case II, the number of Eductors and motor horsepower is doubled. These facts indicate that a recycle Pump is not an economical or practical backup for an Agitator.

Scrubbing System

The scrubbing system includes one Scrubbing Eductor, one Separator, and one Circulating Pump. All equipment is sized for a 100% full load and the Circulating Pump has a 100% spare unit. As in Case I the Eductor is not spared; the solids content of the scrubbing fluid is not expected to exceed 1% and the problem of erosion is minimal. Thus, the equipment saved in the single Slurry Surge Tank system, as compared to the base case, includes one Eductor, one Separator, and one Circulating Pump.

Slurry Preparation System

To sum up, the slurry preparation unit using a single Slurry Surge Tank (see Figure 2) includes the following:

- (a) A single Slurry Surge Tank sized for 15-minute residence time at half-full level
- (b) Two full-size coal feed systems, including a Bin Discharge Screw Feeder, a Weigh Belt Feeder, and auxiliaries
- (c) Two Slurry Recycle Pumps, each with a 100% spare
- (d) One full-size scrubbing system and a spare Circulating Pump

ECONOMIC ANALYSIS—1979 \$

For the base case, the cost is \$1,525,000. For Case II, the cost is \$1,400,000. A saving of \$125,000, or about 8.2%, is realized in Case II for an order-of-magnitude estimate. These estimates exclude home office costs.

A summary of equipment required for both cases is given in Table 1. Order-of-magnitude cost estimates ($\pm 30\%$) for the slurry preparation system equipment affected by this trade-off study are given in Table 2. Structural costs for the two cases are not included as the equipment will be installed in Area 11 (by Rust Engineering). Home Office engineering costs, etc., are not included either, as they are assumed to be identical for both cases.

CASES I AND II COMPARED

The comparison between Case I and Case II (see Table 3) shows that use of a single Slurry Surge Tank brings about some reduction in equipment. However, the economic advantages in terms of the cost of the system are marginal (see Table 2). Energy economy amounts to reduction in the total agitator horsepower from 30 hp to 20 hp, or 7.5 kwh, other power requirements remaining unchanged.

In Case II, equipment reliability and availability are seriously compromised. With a single Tank, any breakdown in the agitator system will directly affect production. For short-term maintenance on the Agitator, the plant can continue to be run without coal feed by charging process solvent directly to the coal feed pumps. For extensive repairs on the Agitator or Slurry Surge Tank the plant must be shut down.

On the other hand, a two-tank system is unaffected when one Tank goes out of commission for any reason. Maintenance of the Tank and auxiliaries can be carried out without plant interruptions by operating one train at full load. If one Tank loses both Pumps, the loss is overcome by an interconnecting balance line between two Tanks. Coal slurry can continue to be fed from both Tanks without interruption.

PHASE I DESIGN CONSIDERATIONS

As noted above, this study is based on the coal feed characteristics as defined in Phase 0; i.e., coal particle size is 90% through 20 mesh. For the Phase I design the coal particle size will be 70% through 200 mesh, with nearly 50% of the feed being less than 30 microns in size. Such a fine coal feed is expected to increase the maintenance requirements of the feed system due to dust and compaction.

The Slurry Surge Tank design will have to be modified to reduce dust carryover and provide more agitation for particle wetting and mixing. Energy requirements will, therefore, increase. Raising the slurry temperature from 280°F in Phase 0 to 350°F in Phase I will cause more vapors to be given off and will increase dust entrainment to some extent.

The scrubbing Eductor will handle more solids and may require greater maintenance. The solids content of the scrubbing solvent may increase to such a level that, instead of feeding it to the Slurry Surge Tank by gravity, it may have to be pumped by the Scrubber Circulating Pump. The Slurry Recycle Pump rating and piping will be upgraded to handle fine slurry.

In short, in order to maintain the same reliability of the slurry preparation system as in Phase 0, a greater equipment redundancy is required for the Phase I design. Therefore, the desirability of a system with two Slurry Surge Tanks is even greater for the Phase I design than for the Phase 0 design.

EQUIPMENT FAILURE ABATEMENT PROCEDURES IN TWO-TANK SYSTEM

Some of the failures that may occur in each train of the slurry preparation system would be in the following equipment:

- (a) The coal feed system
- (b) An operating slurry Circulating Pump
- (c) Both operating and spare slurry Circulating Pumps
- (d) Slurry Surge Tank or Agitator

The following abatement procedures are recommended.

Coal Feed System Failure

Assume that the coal feed system of the A-train breaks down. Capacity of the B-train coal feed system is then jacked up to 100% full-load and the level in V-1201B is reset to 75% full. P-1201C is brought onstream and connected to the A-train Slurry Feed Pumps. V-1201 is run out and P-1201A turned off.

Operating Slurry Pump Failure

Assume that P-1201A fails. Immediately the low pressure switch on the discharge line will automatically shut off the line and simultaneously open the flush solvent valve to supply process solvent to the Slurry Feed Pumps. Pump P-1201B is activated manually and put onstream. The pump discharge line is opened and flush solvent turned off.

Failure of Both Operating and Spare Slurry Circulating Pumps

Assume that the spare Pump in the A-train fails before a malfunction in the operating Pump is corrected. The low-pressure switch will automatically shut the discharge line and open the flush solvent valve to supply process solvent to the Slurry Feed Pumps. The balance line between the two Slurry Surge Tanks is opened and slurry continues to be made in both

tanks. The spare slurry Circulating Pump of the B-train is started and connected to the Slurry Feed Pumps of the A-train.

Failure of a Slurry Surge Tank or Agitator

If the Slurry Surge Tank of the A-train is to be taken out of service or if its Agitator needs repair, the Slurry Surge Tank of the B-train is operated at 100% full load. The coal feed system of the B-train is operated at full capacity and the slurry level in the Surge Tank is raised to 75% full level. The spare Slurry Recirculating Pump is put onstream and connected to the A-train Slurry Feed Pumps. The changeover is done manually while the contents of the A-train Slurry Surge Tank are run out.

Table 1

Equipment List for Slurry Preparation Area
Cases I and II

Item #	Name	# req'd	Case I (Phase 0)	# req'd	Case II
			Size		Size
A-1201	Slurry Surge Tank Agitator	2	One turbine blade, 15 hp each	1	One turbine blade, 20 hp
AL-1201	Rotary Air Lock Valve	2	510,000 lb/hr max	2	Same as Case I
C-1201	Slurry Vent Fan	2	130 acfm, 3 hp	2	Same as Case I
CV-1201	Bin Discharge Screw Feeder	2	24 inches dia x 8 ft long, 10 hp	2	Same as Case I
CV-1202	Weigh Belt Feeder	2	38 inches wide x 7 ft long, 2 hp	2	Same as Case I
E-1201	Mixed Phase Condenser	1	Shell tube, CS 1875 ft ² , 1 shell	1	Same as Case I
J-1201	Scrubbing Eductor	2	11,728 lb/hr capacity, 1.5 inch WG Draft, 472 gpm Motive Liquid	1	Same as Case I
P-1201	Slurry Recycle Pump	4	38.5% slurry, centrif pump, 140 gpm, 123 ft TDH, 150 hp	4	Same as Case I
P-1203	Scrubber Recirculating Pump	3	Centrif pump, 236 gpm, 326 ft TDH, 40 hp	2	Same as Case I
P-1204	Mixed Phase Condensate Transfer Pump	2	Centrif pump, 37 gpm, 180 ft TDH, 7.5 hp	2	Same as Case I
V-1201	Slurry Surge Tank	2	18 ft 6 inches dia x 18 ft 6 inches T-T, FV/15 psig, 400°F, Agit 4 baffles, CS	1	23 ft 0 inches dia x 23 ft 0 inches T-T, FV/15 psig, 400°F, Agit 4 baffles, CS
V-1202	Scrubbing Solvent Separator	2	8 ft 0 inches dia x 15 ft 0 inches T-T, FV/15 psig, 400°F, CS	1	Same as Case I
V-1203	Mixed Phase Receiver	1	3 ft 0 inches dia x 7 ft 0 inches T-T, FV/15 psig, 400°F, CS	1	Same as Case I
X-1201	Pulverized Coal Slide Valve	2	Part of coal weigh system package	2	Same as Case I
X-1202	Slurry Surge Tank Slide Valve	2	Part of coal weigh system package	2	Same as Case I

Table 2

Cost Estimate Comparison Between Case I and Case II¹

Item	Cost, \$	
	Case I	Case II
Process equipment	551,000	513,800
Materials	268,000	239,200
Subcontracts and other costs	<u>227,000</u>	<u>201,000</u>
Subtotal	1,046,000	954,000
Field labor	360,000	330,000
Other field charges	<u>119,000</u>	<u>116,000</u>
Total	1,525,000	1,400,000

1. Above cost estimate is an order-of-magnitude study ($\pm 30\%$) based on First Quarter 1979 prices. In the estimate, materials include piping, instrumentation, electrical and field supplies. Other costs include risk insurance, taxes, etc. Field labor includes labor for equipment installation, piping, instrumentation, and electrical. Other field charges include field supervision and construction equipment and tools. Materials and labor for structures were not included as the system will be installed in Area 11 (by Rust Engineering). Engineering expenses, contingencies, escalation, overhead, and fees were not included as they would be incremental and are assumed to be identical for the two cases.

Table 3

Cases I and II Compared

Case I (Base Case)	Case II	Change from Base Case
1. Two full capacity coal feed systems required (one per train). Coal feed system failure in one train is compensated by second train.	1. Two full capacity coal feed systems required; one operating, one spare. Failure of coal feed system is compensated by the spare unit.	1. No change in number of units.
2. Two Slurry Surge Tanks (V-1201) 18 ft 6 inches dia x 18 ft 6 inches T-T. Each can be operated at half or full load. Tank failure is compensated by second train.	2. One Slurry Surge Tank, 23 ft dia x 23 ft T-T. Operated at full load. Tank failure would result in plant shutdown.	2. One less tank. Bigger size for Case II reduces cost advantage. No spare tank.
3. Two Slurry Surge Tank Agitators (A-1201); 15 hp each. Agitator failure compensated by second train. Effective backup. Reliability of Agitator high.	3. One Slurry Surge Tank Agitator (A-1201); 20 hp. Agitator failure would result in stoppage of slurry feed and, hence, plant interruption or shutdown. Reliability of Agitator high. But there is no effective backup.	3. One less Agitator. Power requirements reduced by 10 hp.
4. Four Slurry Recycle Pumps (P-1201)--two operating, two spares. Any two Pumps can feed both trains.	4. Four Slurry Recycle Pumps (P-1201)--two operating, two spares. Any two Pumps can feed both trains.	4. No change in number of units.
5. Two scrubbing systems--each sized for full capacity with a common spare Pump. Failure of an Eductor will close down one train, but not the plant. Eductor failure is not expected to be a problem.	5. One full-size scrubbing system with one spare pump. Eductor failure will interrupt or shut down coal feed. Eductor failure is not expected to be a problem.	5. Reduction in equipment as follows: One Scrubbing Eductor One Separator One Circulating Pump
6. If the system were installed independently, a 125 ft x 52 ft x 38 ft 6 inch-high structure would be required.	6. If the system were installed independently, a 67 ft x 52 ft x 42 ft-high structure would be required.	6. No cost advantage as the system is installed in Area 11.
7. Order-of-magnitude cost is \$1,525,000.	7. Order-of-magnitude cost is \$1,400,000.	7. Reduction in cost of \$125,000, or 8.2%.

Figure 1
Base Case: Two Slurry Surge Tanks

Figure 2
Alternative Case: One Slurry Surge Tank

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TWIN FULL-TRAIN OPTION SELECTED TO MEET CSD FRONT END PROBLEM

James H. Harkins*

The Kerr-McGee Critical Solvent Deashing (CSD) Unit has been operating at the Wilsonville Pilot Plant for two years. During that time, numerous shutdowns have been caused by plugging and operating difficulties in the first stage and downstream solids-handling systems. The second and third stage systems and the deashing solvent recovery system have exhibited a high level of operating reliability.

Three alternate unit designs of the CSD Unit were compared for cost, operating flexibility, and their effect on the SRC Liquefaction Area throughput and onstream time.

Of the three cases studied, Case 3—two 100% front end trains without reclaim—is recommended for the Demonstration Plant. This scheme (1) has the lowest capital cost; (2) has the lowest manufacturing cost; (3) avoids the complexity of the reclaim system included in Cases 1 and 2; and (4) eliminates turndown of the SRC Liquefaction Unit required in Case 1.

All three cases achieve an increase in the Demonstration Plant's capacity to produce spec product . . . offer protection against a total facility shutdown in the event of the CSD Unit going down . . . and have built-in design features for keeping the CSD Unit operating after problems occur. The study found:

Case 1. The 120% train with reclaim results in a reduced SRC production rate during each unscheduled CSD shutdown that exceeds the capacity of the Vacuum Tower Bottoms Hold Tank.

Case 2. The two 60% trains with reclaim permit SRC production at the 60% level after shutdown of one train for maintenance and result in no loss of SRC production during unscheduled shutdowns. However, there is no difference in manufacturing costs between Cases 1 and 2 if the number of shutdowns per month exceeds one.

Case 3. The two 100% front end trains without reclaim permits the SRC Liquefaction Area and the CSD Unit to operate at 100% throughput even if operating difficulties develop in the front end of one train of the CSD Unit. Manufacturing costs are essentially independent of the number of shutdowns, assuming that both front ends are not shut down at the same time and that the back end is not shut down.

The capital requirements for each case expressed as a percentage of the capital costs of the base case (Case 1) are:

Case 1	100%
Case 2	103%
Case 3	95%

*Catalytic, Inc.

Relative manufacturing costs for each case have been developed for either 0, 1, or 3 shutdowns/month. The costs are expressed as a decimal fraction of the Case 1-0 shutdowns.

	No. of shutdowns/month		
	0	1	3
Case 1	1.00	1.09	1.22
Case 2	1.02	1.09	1.22
Case 3	0.98	1.00	1.00

CASE 1—120% TRAIN WITH RECLAIM

The flow scheme is similar to the Phase 0 design but the capacity of the CSD Unit is increased to 120% of the Vacuum Tower Bottoms flow to allow for reclaiming off-spec material—and the capacity of the solidifiers is increased to 120%. The 20% rate of reclaim chosen is an attempt to keep the size of the reclaim equipment reasonable and yet takes into consideration the CSD Unit's frequency of operation. Although it is not possible to predict the frequency or time length of CSD Unit shutdowns, a 20% rate of reclaim will cover the CSD Unit's performance adequately.

If there is a system upset within the CSD Unit resulting in a flow stoppage and this upset is expected to be short, a Vacuum Tower Bottoms Hold Tank has been provided to receive hot liquid bottoms from the Vacuum Tower and maintain it in liquid state. The capacity of this tank is six hours at full normal SRC Liquefaction Unit throughput.

But if the CSD Unit shutdown extends beyond six hours, the Vacuum Tower Bottoms must then be diverted to the SRC solidifiers and stockpiled as off-spec solid SRC. If this happens, the SRC solidifiers are not designed for the full Vacuum Tower Bottoms rate; hence the SRC Liquefaction Area must be turned down to 72% of normal throughput to conform to the capacity of the SRC solidifiers. The SRC Liquefaction Unit can then continue to operate at this throughput, until the CSD Unit is again ready for operation.

In case of CSD Unit shutdown, the above procedure allows for six hours of additional operation of the SRC Liquefaction Area at 100% throughput, followed by operation at 72% throughput for as long a period as required for restarting the CSD Unit. It is possible for the operation of the SRC liquefaction area to be extended to 21 hours of 100% operation, if immediately on shutdown of the CSD Unit, 72% of the Vacuum Tower Bottoms is diverted to the product solidifiers, with the remaining 28% flowing as liquid product to the Vacuum Tower Bottoms Hold Tank. At the end of 21 hours, if the CSD Unit is still down, the SRC liquefaction area will then have to be turned down to 72% capacity.

On restart of the CSD Unit, the off-spec liquid and solid SRC must be worked off as part of the feed to the Kerr-McGee Unit. The liquid off-spec material can be directly pumped to the Kerr-McGee Unit and blended with the net Vacuum Tower Bottoms. The solid off-spec must first be remelted by passing it through the reclaim system before adding it to the CSD feed.

CASE 2—TWO 60% TRAINS WITH RECLAIM

Here, two 60% CSD trains are provided, each handling 50% of the normal flow rate. A reclaim system identical to that provided in Case 1 is also included here.

Analysis of this scheme is straightforward. If one 60% CSD train goes down, the other train continues to produce spec SRC at a 60% rate. The remaining 40% Vacuum Tower Bottoms will be collected in the Vacuum Tower Hold Tank for 15 hours and then, if the shutdown CSD train has not been restarted, this 40% of the Vacuum Tower Bottoms is solidified. The SRC Liquefaction Unit can continue to operate at 100% throughput until the CSD Unit is again ready for operation. In the unlikely event of both units shutting down simultaneously, the sequence of operations described in Case 1 would apply here.

On restart of the shutdown CSD train, the off-spec liquid and solid SRC must be worked off as part of the feed to both CSD trains. The liquid off-spec material can be directly pumped to the Kerr-McGee Unit and blended with the net Vacuum Tower Bottoms. The solid off-spec SRC must first be remelted by passing it through the reclaim system before adding it to the CSD feed.

CASE 3—TWO 100% FRONT END/ONE 100% BACK END CSD UNIT/NO RECLAIM EQUIPMENT

The scheme here maintains 100% spec SRC production without the need to produce a solid off-spec material, and without the expense of an off-spec solids reclaim system. Two 100% front end and one 100% back end CSD units are provided. This was done because Wilsonville showed that the major difficulties had occurred in the front end of the CSD Unit where solids are being handled. The back end of the CSD Unit, in which liquid hydrocarbon fractions and deashing solvent are processed, would be expected to have the long onstream periods experienced in conventional petroleum refining.

If difficulties do occur in the front end of the CSD Unit causing one train to come down, the other train can be started up to permit the Liquefaction Unit to run at 100% of capacity. The Vacuum Tower Hold Tank will provide three hours storage capacity to make the switch-over and put the standby front end train on-line. The Vacuum Tower Bottoms will be slowly blended into the CSD feed after the second train has been brought onstream.

ECONOMIC ANALYSIS

Order-of-magnitude $\pm 30\%$ estimates of capital, operating costs, and manufacturing costs were developed for all three cases.

The economics of each case is sensitive to the frequency and length of unscheduled shutdowns. Therefore, to fairly weigh economic advantages and disadvantages, three sub-cases within each case were defined and evaluated:

Subcase 1—0 unscheduled shutdown/month

Subcase 2—1 unscheduled shutdown/month

Subcase 3—3 unscheduled shutdowns/month

Each shutdown is assumed to last 48 hours.

The capital requirement is summarized in Table 1. Operating costs are presented in Tables 2, 3, and 4. Manufacturing costs are presented in Tables 5, 6, and 7.

Table 1

Total Capital Requirement¹

	Case 1	Case 2	Case 3
Installed cost of CSD equipment	73	76	73
Delta installed cost of large solidification equipment	4	4	0
Initial charge of deashing solvent	1	1	1
Home office cost	9	9	9
Contingency @ 15% of the above	<u>13</u>	<u>13</u>	<u>12</u>
Total capital	100	103	95

1. These costs are expressed as a % of total for Case 1.

Table 2
Case 1 Operating Costs¹

330 Days/Yr

	Subcase 1 No unscheduled shutdowns	Subcase 2 1 unscheduled shutdown/month	Subcase 3 3 unscheduled shutdowns/month
Fuel gas @ \$6.20/MM Btu	.74	.75	.75
Electric power @ \$0.04/kwh	.23	.24	.26
Boiler feed water @ \$0.50/1000 gal	.00	.00	.00
150 psig steam @ \$2.15/MM Btu	(.07) ²	(.07) ²	(.07) ²
Dowtherm @ \$9.40/MM Btu		.09	.28
Cooling water @ \$0.06/1000 gal		.00	.00
Operating labor @ \$110,000/yr (4-shift)	.05	.05	.05
Deashing solvent makeup	<u>.05</u>	<u>.05</u>	<u>.05</u>
	1.00	1.11	1.32

1. These costs are expressed as decimal fractions of Case 1, Subcase 1 (no unscheduled shutdowns).
2. Credit.

Table 3

Case 2 Operating Costs¹

330 Days/Yr

	Subcase 1	Subcase 2	Subcase 3
	No unscheduled shutdowns	1 unscheduled shutdown/month	3 unscheduled shutdowns/month
Fuel gas @ \$6.20/MM Btu	.74	.75	.77
Electric power @ \$0.04/kwh	.23	.24	.26
Boiler feed water @ \$0.50/1000 gal	.00	.00	.00
150 psig steam @ \$2.15/MM Btu	(.07) ²	(.07) ²	(.07) ²
Dowtherm @ \$9.40/MM Btu		.10	.28
Cooling water @ \$0.06/1000 gal		.00	.00
Operating labor @ \$110,000/yr (4-shift)	.06	.06	.06
Deashing solvent makeup	<u>.05</u>	<u>.05</u>	<u>.05</u>
	1.01	1.13	1.35

1. These costs are expressed as decimal fractions of Case 1, Subcase 1 (no unscheduled shutdowns)--see Table 2.
2. Credit.

Table 4

Case 3 Operating Costs¹

330 Days/Yr

	Subcase 1 No unscheduled shutdowns	Subcase 2 1 unscheduled shutdown/month	Subcase 3 3 unscheduled shutdowns/month
Fuel gas @ \$6.20/MM Btu	.74	.74	.75
Electric power @ \$0.04/kwh	.23	.23	.23
Boiler feed water @ \$0.50/1000 gal	.00	.00	.00
150 psig steam @ \$2.15/MM Btu	(.07) ²	(.07) ²	(.07) ²
Dowtherm @ \$9.40/MM Btu			
Cooling water @ \$0.06/1000 gal			
Operating labor @ \$110,000/yr (4-shift)	.05	.05	.05
Deashing solvent makeup	<u>.05</u>	<u>.05</u>	<u>.05</u>
	1.00	1.06	1.01

1. These costs are expressed as decimal fractions of Case 1, Subcase 1 (no unscheduled shutdowns)--see Table 2.

2. Credit.

Table 5

Case 1 Manufacturing Costs¹

330 Days/Yr

	Subcase 1 No unscheduled shutdowns	Subcase 2 1 unscheduled shutdown/month	Subcase 3 3 unscheduled shutdowns/month
Total products (10 ¹² Btu/yr)	28.38	28.08	27.49
	\$/MM Btu Product		
Operating costs	.53	.60	.73
Capital charges @ 20% of total capital	<u>.47</u>	<u>.49</u>	<u>.49</u>
Manufacturing costs	1.00	1.09	1.22

1. These costs are expressed as decimal fractions of Case 1, Subcase 1 (no unscheduled shutdowns).

Table 6

Case 2 Manufacturing Costs¹

330 Days/Yr

	Subcase 1 No unscheduled shutdowns	Subcase 2 1 unscheduled shutdown/month	Subcase 3 3 unscheduled shutdowns/month
Total products (10^{12} Btu/yr)	28.38	28.38	28.38
	<u>\$/MM Btu Product</u>		
Operating costs	.53	.60	.73
Capital charges @ 20% of total capital	<u>.49</u>	<u>.49</u>	<u>.49</u>
Manufacturing costs	1.02	1.09	1.22

1. These costs are expressed as decimal fractions of Case 1, Subcase 1 (no unscheduled shutdowns)--see Table 5.

Table 7

Case 3 Manufacturing Costs¹

330 Days/Yr

	Subcase 1 No unscheduled shutdowns	Subcase 2 1 unscheduled shutdown/month	Subcase 3 3 unscheduled shutdowns/month
Total products (10^{12} Btu/yr)	28.38	28.38	28.38
	<hr/> \$/MM Btu Product <hr/>		
Operating costs	.53	.53	.53
Capital charges @ 20% of total capital	<u>.44</u>	<u>.44</u>	<u>.44</u>
Manufacturing costs	.97	.97	.97

1. These costs are expressed as decimal fractions of Case 1, Subcase 1 (no unscheduled shutdowns)--see Table 5.

ANALYSIS OF PROCESS STREAMS FOR CAUSES OF CORROSION IN THE FRACTIONATING TOWER

**K. L. Baumert, B. Davis,
C. Jewitt, A. Sagues,
and G. Thomas***

Severe corrosion has beset the fractionating and vacuum towers in the Pilot Plants at Wilsonville and Fort Lewis.^{1, 2, 3} Probes at the Wilsonville fractionating tower have measured corrosion rates exceeding 1000 mils a year. The consequence has been the relining, re-traying, and even replacement of the towers—although currently Wilsonville is holding corrosion down to manageable levels by adding sodium carbonate to the coal feed.

The immediate cause of corrosion has not yet been determined, although earlier work has suggested the synergistically corrosive effects of chloride and phenols.¹⁰ Wilsonville found that a period of high corrosion coincided with the use of coal of high chloride content. Investigators suggested the possibility of an interaction between chloride compounds and organic acids that could have resulted in increased corrosion in the fractionating column. They hypothesized that chlorinated naphthenic acids might be responsible, since naphthenic acids are a common corroding species in petroleum distillation columns. But experiments by Ganesan and others showed no significant increase in the corrosivity of naphthenic acids combined with various chloride compounds in a heavy naphtha base at 190°C.⁴

The present investigation addresses the question of how inorganic chloride gets to the fractionating and distilling towers. Samples of process streams from Wilsonville were analyzed for chloride and acidic and basic compounds. The samples were taken both when the rate at which sodium carbonate had been added to the coal was low and when it was high.

The analyses lead to seven conclusions:

- (1) Inorganic chloride builds up in the bottom of the fractionating tower to concentrations significantly higher than those in the liquid feed to the tower.
- (2) The inorganic chloride is carried to the fractionating tower by the bottoms coming from the high pressure separator. The reason for so concluding is that the overhead from the high pressure separator, as sampled from the solvent decanter, contains little water-soluble inorganic chloride.
- (3) Differences in the amount of sodium carbonate added to the feed coal make little, if any, difference in the total phenolic content or in the distribution of phenolic compounds in fractionating tower liquids.

*Baumert is from ICRC, Jewitt from Ashland Petroleum Research, and Davis, Sagues, and Thomas from the Institute for Mining and Minerals Research, University of Kentucky. The authors acknowledge the help of the staffs of the Institute and of the Wilsonville Pilot Plant, particularly H. E. Lewis, P. Barnett, R. G. Boykin, J. Bresowar, and J. Sapp at Wilsonville.

- (4) It is unlikely that chloride reacts selectively with one compound to form a highly corrosive organic chloride species. The evidence here is that when liquids from the top, middle, and bottom of the fractionating column (Trays 1, 9, and 15) are separated into a phenolic, an amine, and an oil fraction, each fraction contains a similar amount of organic chloride. (The samples were collected when the addition of sodium carbonate was low.)
- (5) Basic nitrogen compounds make up 4-10 wt % of the fractionating tower liquids—and are present in only about a tenth the amount of the phenolic compounds. Amines are present in the fractionating tower liquids in much larger concentrations than the chlorides. The amines could form organic chloride salts similar to ammonium chloride, which would increase the corrosivity of such liquids.
- (6) The maximum amount of carboxylic acids present in the fractionating tower liquids is 0.6 wt %—and even this, because of the separation scheme used, may be an overestimate.
- (7) By tracking the chloride content of the process streams, we have deduced the path along which water-soluble inorganic chloride flows from the dissolver to the fractionating tower. Refer to Figure 1 (Wilsonville flow chart) and Figure 2. The path follows the bottoms from the high pressure separator (V-103) through the flash tank (V-110) and the reclaim tank (V-111), thence along Tray 8 of the vacuum tower (T-102) through the storage vessel (V-160) to the fractionating tower (T-105).

Sampling Includes Period of Severe Corrosion

Most of the analytical results in this report are for two sets of samples collected at the Wilsonville Pilot Plant (Tables 1 and 2). One set was collected in July 1979, when the Wilsonville unit was processing Kentucky #9 coal with the maximum addition of sodium carbonate (40 lb of sodium carbonate per two-ton batch of coal). The other set of samples was collected in December 1979, when Kentucky #9 coal was being processed with a relatively small addition of sodium carbonate.

The corrosion rate in the fractionating tower (T-105) was low when the maximum amount of sodium carbonate addition was added. But when the smaller amounts of sodium carbonate were used, the corrosion rate became unacceptably high. In the period after the samples were collected, larger dosages of sodium carbonate were resumed and the corrosion rates decreased to an acceptable level. Thus, the material or materials responsible for the severe corrosion should have been present in the samples collected in December 1979.⁵

The samples were analyzed for total chloride, for water-soluble inorganic chloride, and for chloride remaining in the water-washed samples. The water-soluble inorganic chloride was removed from the organic sample by washing it three times with distilled water—the volume of water used in each washing being equal to the volume of the organic sample. (During the course of the analysis it was found that one washing was enough to remove all but traces of the water-soluble inorganic chloride. Nevertheless, each sample was washed three times, so each would have uniform treatment.)

The organic samples, as-received and after washing, were burned in a Parr oxygen bomb. The bomb, after adding 10mL of a scrubber solution (0.25g NaHCO₃, 0.25g Na₂CO₃, and 200g H₂O₂/L) to the organic sample in the metal sample holder, was pressurized to 300 psig with oxygen and the sample ignited. The bomb was then cooled to room tem-

perature, gas was vented through a bubbler filled with the scrubber solution, and the liquids in the vent bubbler and the bomb were rinsed into a volumetric flask.

The bomb wash solution was injected into a Dionex Ion Chromatograph and analyzed under the following instrument conditions:

Eluent: 0.0015M NaHCO_3

Flow rate: 230mL/hr

Separator column: 3 × 250mm anion separator

Suppressor column: 6 × 250mm anion suppressor

Conductivity meter setting: 10 μmho full scale

Standard chloride samples were prepared from reagent grade NaCl in eluent solution.

Distribution of Chlorides in and around T-105 Tower

The results for the chloride analysis around the T-105 tower for samples collected in December 1979 are in Figure 3. All chloride concentrations are reported as parts per million of the process streams around the tower. As Table 2 shows, the sodium carbonate addition was being changed during the interval sampled and the analyses may reflect this. However, the three sampling periods are consistent and the results follow the same trend. The chloride data demonstrate a number of important points regarding the tower corrosion:

- (1) The chloride content in Tray 1 of the tower is lower than that in the tower feed coming from the storage vessel (V-160). The chloride is present in organic forms, not as water-soluble chlorides.
- (2) The chloride content of the liquid in the tower's Tray 15 is two to three times higher than that in the tower feed and more of it is present as inorganic than as organic chloride.
- (3) There is a chloride buildup in the tower, which operates so the flow from the bottom is at least twice the flow overhead—usually more than twice. The bottoms flow goes over Tray 15 and the overhead passes Tray 1. Using a total chloride content for the storage vessel of 350 ppm—and 94 ppm for the overhead—then, with a 2-to-1 split (bottoms to overhead), by chloride balance we can calculate the maximum chloride content of Tray 15: $(350)(3) = 2(x) + (1)(94)$ —or x (the maximum chloride in Tray 15) would be 478 ppm. As the bottom flow increases to more than that of the overhead, the maximum chloride in Tray 15 decreases accordingly.
- (4) Chloride concentration buildup in Tray 15 does not appear to carry through, since the chloride content of the fractionating tower bottoms is less than that of the chloride in the Tray 15 liquid.

Samples of liquids in Trays 1, 9, and 15 were collected during a period of maximum sodium carbonate addition—July 1979—and analyzed. As Table 3 shows, adding sufficient sodium carbonate to the coal feed prevents chloride from appearing in T-105 tower liquids.

In most samples the sum of chloride measured in the water-washed organic layer, plus the inorganic chloride, is less than the total chloride measured in the as-received sample. One reason for this is the extraction of organic chlorides into the water layer; our analytical method would not detect this water-soluble organic chloride.

To verify this, low chloride (< 10 ppm) liquid from Tray 9 was spiked with p-chlorophenol and p-chlorobenzoic acid—and then put through the same extraction procedure. The data in Table 4 indicate that 15 to 20 percent of the organic chloride compound was extracted by the three washings. Similarly, as Table 5 indicates, in the December 1979 high

chloride samples, the loss of chloride by extraction from the liquids of Trays 1, 9, and 15 is in that range—although the data here exhibit scatter.

Several Possible Causes of Corrosion Investigated

A concern raised by the severe corrosion problem at Wilsonville and Fort Lewis was that the chloride might be reacting with some component of the organic liquid to form a highly corrosive material. Table 6 suggests that this is not the case.

A bottoms sample had been collected from the T-104 distillation tower on 13 October 1978, when Wilsonville was processing a blend of Kentucky #6 and #11 coal from the Pyro Mine. The coal blend contained 0.25 wt % chloride. Sodium carbonate was not being added at the time. The phenolic and oil fractions from this T-104 bottoms sample contained, within experimental error, concentrations of chloride similar to that of the water-washed sample from the same bottoms.

Liquids from Trays 1, 9, and 15, collected 12 December 1979, also had similar chloride concentrations in the phenolic and oil fractions and in the water-washed organic sample. Thus, the phenolic and oil fractions, after separation, contained the same amount of chloride that had been present as organic chlorides in the sample as-received. Therefore, it is unlikely that the chloride in either fraction is concentrated in one or a few organic compounds.

Small portions of a few samples were obtained from Wilsonville for the period prior to the addition of sodium carbonate to the coal feed. Two samples (approximately 5mL) of the feed liquid for the fractionating tower were obtained from J. R. Keiser and V. B. Baylor of the Oak Ridge National Laboratory. One of the samples had been collected when Indiana #5 coal was the feed at Wilsonville and no severe corrosion problems existed. The other sample was collected 1 February 1979 while Wilsonville was processing Kentucky #9 coal and the corrosion rate was unacceptably high. The Indiana coal had contained 0.02% chloride, the Kentucky coal 0.25%.

These samples were analyzed for total chloride by coulometry. The water-soluble chloride was extracted from the organic layer by washing the samples with an equal volume of water three times. Then the chloride in the water was determined by a potentiometric titration technique (Table 7).

Total chloride reported in Table 7 agrees with values reported by Keiser and Baylor in their analysis of portions of the same samples.⁷ The surprising feature of the Table 7 data, apart from the difference in the total chloride, is that the major portion of the chloride in the liquid derived from the Kentucky #9 coal is present in a water-soluble inorganic form. Organic chloride is present in similar amounts in the liquids derived from both coals. This indicates that inorganic chloride may be an important factor in the enhanced corrosion rate observed during Kentucky #9 coal runs at Wilsonville.

Sulfur and nitrogen were determined for the two T-105 liquid feeds and the results are reported in Table 8. There does not appear to be a significant difference in the nitrogen and sulfur content of either the feed liquid or the wash water from the liquids derived from the two coals.

Two Methods of Analysis Used for Ammonia Content

Comparisons of ammonia content with inorganic chloride content have varied in these samples.

For example, samples were obtained for the period 24-27 June 1979, while Wilsonville was processing a Kentucky #9 coal (Lafayette coal). This was shortly after the rebuilt T-105 tower was put back into operation, but prior to the first sodium carbonate addition on 28 June 1979.

Table 9 shows that corrosion probes in the tower indicated a high corrosion rate during this period.⁵ In Table 10, the total chloride values of the tray samples collected on 24 and 26 June follow the same trend that marked the samples collected six months later in December 1979. The Tray 1 liquid on both June days contained less than 100 ppm chloride—essentially all the chloride was organic chloride. Tray 15 (or Tray 20) had a much higher total chloride content and a high water soluble inorganic chloride content (although not as high as in the December 1979 sampling).

The analyses in Table 10 were done by a commercial laboratory and not all the details are available. But we know that the T-105 tower samples of 24-28 June 1979 were extracted with water . . . that the analyses, except for total chloride and ammonia, were carried out on the wash water . . . that the ammonia content was determined by a distillation technique . . . and that a portion of the sample was made basic with a caustic solution and refluxed to distill out the ammonia that had been absorbed in water.

The ammoniacal aqueous solution was then titrated with a standard acid solution. But the ammonia so determined did not correlate with the inorganic chloride; in fact, just the opposite appears to be true. The Tray 1 and overhead samples had the highest ammonia concentration, but essentially no inorganic chloride. On the other hand, the Tray 15 and Tray 20 samples, with the highest inorganic chloride content, had the lowest ammonia concentration. Other anions reported in Table 10 do not show a trend that would account for this ammonia distribution.

We followed a different method in analyzing for ammonia some of the samples collected on 14 and 15 December 1979. The as-received sample was washed with water. The wash water was made basic with sodium hydroxide and the ammonia determined by an NH_3 -sensitive electrode. See Table 11 for the results: the ammonia measured by this method is low; for the samples with 100 to 530 ppm chlorine, the ammonia concentration is only about 10 percent of the amount of the inorganic chloride present.

We need further evaluation of this method before we draw the obvious conclusion from Table 11: that the inorganic chloride is not present as ammonium chloride. However, ammonia and chloride data obtained by three laboratories using different techniques show the chloride content to be higher than the ammonia content.

T-105 liquids contain basic nitrogen compounds in much larger quantities than the amount of ammonia reported by the commercial laboratory in Table 10. It is likely that some of these basic compounds, such as anilines, would distill with ammonia—which would result in the high ammonia values in Table 10. The fact that the highest ammonia values are obtained for Tray 1, which contains the most volatile basic nitrogen compounds, is consistent with the view that basic compounds, in addition to ammonia, distill over and are measured as ammonia to give the results reported in Table 10.

The data in Tables 10 and 11 suggest that much of the inorganic chloride is not present as ammonium chloride. However, basic compounds can function in the same manner as ammonia. For example, aniline can react with HCl the same way it reacts with ammonia:

Note that aniline hydrochloride is used only as an example of one possible nitrogen compound. More likely there is a range of nitrogen compounds, as indicated by the amounts identified in Table 18.

From a chemical point of view, it makes little difference whether the inorganic chloride, if HCl, is present as the ammonium salt. However, in terms of corrosion this may be important since there could be differences in solubility and volatility so that corrosion would appear at different points in the process stream, depending on the salt that the inorganic chloride is in.

Sample Liquids Collected from Four Process Sections

In addition to the samples already described for the T-105 fractionating tower, samples were collected 10-15 December 1979 from major process streams around the R-101 dissolver, the T-102 vacuum column, and the T-104 distillation tower (see Figure 1). Unfortunately, these samples cover a period when the amount of sodium carbonate added to the slurry was being changed, so comparisons of chloride data are uncertain for these samples from the four process sections.

Chloride determinations for samples from the process streams around T-102 are presented in Figure 4. The chloride content of the samples from V-120 (vacuum preheater surge drum) and from Trays 3 and 8 of T-102 suggest that chloride builds up in the lower portion of the tower. However, limited data and different sample collection times make the conclusion less definite here than in the case of the T-105 tower. Data for the 17 December 1979 samples from T-102 show a surprisingly low level of water-soluble inorganic chloride. A five-gallon sample collected that day from the V-160 solvent feed tank contained only 180 ppm total chloride. The sample collected 14-15 December 1979 from T-105 contained this much or more water-soluble inorganic chloride. But the time lag between the collection of the T-102 and T-105 tower samples precludes us from a detailed tracing of the water-soluble inorganic chloride through the distillation towers.

The chloride balance around the T-104 distillation tower is presented in Figure 5, from samples taken during the same period as the T-105 tower samples. Total chloride is lower than that in V-160 solvent feed tank and there is essentially no water-soluble inorganic chlorides in these liquids.

The chloride flow around the R-101 dissolver is shown in Figure 6. The overhead from the high pressure separators goes to solvent decanter V-105, where the water is separated from the oil. Water from V-105 is high in chloride, 4660 ppm, but the oil layer from V-105 contains only 10 ppm water-soluble inorganic chloride and 110 ppm total chloride. The fraction of the high pressure separator that ends up as bottoms of the flash tank V-110 contains 710 ppm total chloride. It is difficult to obtain a water-soluble inorganic chloride value for this sample since the neat organic liquid, when shaken with water, forms a very stable emulsion. Attempts to decrease the emulsion problem by dissolving the V-110 bottoms in a solvent prior to extraction caused precipitation of some of the sample.

Chloride Content of Kentucky Coals

The Kentucky coals processed at Wilsonville since June 1979 have contained 0.15-0.25 wt % of chloride. The operators have found the addition of sodium carbonate to be

an expedient means of reducing corrosion in the Pilot Plant. However, adding sodium carbonate, because of the tonnage involved and the processing problems, would not be a viable solution for the Demonstration Plant or a commercial operation.

Chloride in Western Kentucky coals covers a wide range, as seen in Table 12.⁹ Many Kentucky coals have as low a chloride content as some other coals that were processed at Wilsonville without severe corrosion problems.

Several Procedures Used for Phenols Extraction

Liquids from Trays 1, 9, and 15 of the T-105 fractionating tower were analyzed for acidic compounds. A common practice separates carboxylic acids from mixtures by extraction with 5-10 wt % aqueous sodium bicarbonate.⁸ After extraction of the carboxylic acids, phenols can then be extracted with a more basic solution, e.g., 5 to 10 wt % aqueous sodium hydroxide. These extraction schemes are outlined in Figures 7 and 8A. Two variations (Figures 8B and 8C) were used for our extractions: in one case the extraction was performed on neat T-105 tower liquid and in the other the T-105 tower liquid was dissolved in isopentane prior to extraction. The total content of phenols and the phenolic compound distribution were similar for both methods.

The procedure described here for the neat liquid is representative of the extractions performed during this investigation. Approximately 200g of the Tray 9 liquid was extracted twice with 200ml of a 5 wt % aqueous sodium bicarbonate solution. The bicarbonate fractions were combined and extracted with hexanes; the aqueous fraction was then neutralized with a 20% sulfuric acid solution; and the carboxylic acids were extracted into a commercial hexane solvent. The residue was collected and weighed after removal of the hexane by vacuum distillation with a Rotovap.

The data in Table 13 show that the carboxylic acids are not present as a major fraction of the T-105 liquids. It is likely that the bicarbonate solution removed some water-soluble phenols; the carboxylic acids in Table 13 should be viewed as the maximum amount that may be present. In separate experiments it has been found that water will extract the compound phenol from the hydrocarbon layer; the data in Table 13 are consistent with the carboxylic acids being contaminant with phenols.

The organic fraction remaining after the bicarbonate extraction was then extracted twice with 200 mL portions of a 10 wt % sodium hydroxide solution. Neutralization and phenol recovery followed the procedure described above for carboxylic acids.

Gas Chromatography for Phenols Analysis

Phenols were analyzed by gas chromatography, employing different columns and conditions, but producing similar results.

Figure 9 is a typical gas chromatogram for Tray 9 liquid before phenols extraction, using a glass column 3 ft X 2mm filled with helium gas, the temperature being 100°C for one minute and then increasing to 212°C at 6° per minute.

Peaks eluting prior to about eight minutes are due to the oils; peaks after this time are due to phenols. The inset table gives retention times and areas calculated when peaks eluting prior to eight minutes are neglected in the calculation.

Figure 10 is a gas chromatogram (same conditions) of the phenol fraction extracted by the procedure of dissolving the sample in a solvent prior to extraction. A comparison

of Figures 9 and 10 and of the inset tables shows that the extracted phenol fraction does not contain significant hydrocarbons—and the individual phenol peak areas are nearly identical, which indicates that the extraction does not alter the original phenol distribution. The chromatogram in Figure 11 traces small amounts of the hydrocarbon layer (less than 5%) that were carried through when the neat oils were extracted.

Percentages given in the gas chromatograms are calculated without regard to relative response factors, although Table 14 shows that there are variations in the response factors. When calculating ratios of phenol to hydrocarbons in the mixtures, response factors become important; for example, the response factor for benzene (1.12) is nearly twice that of phenol (0.61) o-cresol (0.66) and p-cresol (0.60).¹¹ Some of the peaks in Figure 10 represent the sum of two or more compounds not resolved under the conditions governing. Note the retention times given in Table 15, which shows that the peaks in Figures 9, 10, and 11 with approximately 9.91 minutes retention time result from both m- and p-cresols.

The gas chromatography trace presented in Figure 12 was obtained with an OV-17 capillary column and a slower temperature program. The superior resolution reveals a third peak due to the compound, 2,4,-dimethylphenol, in the 9.91 region, in addition to the peaks from the m- and p-cresols. Some of the peaks shown in Figures 10 and 11 result from other unresolved phenols, as well as those listed in Table 15.

Phenols comprise 35 to 55 wt % of the T-105 tray liquids; see Table 16. The phenol content is highest for Tray 1 and decreases as the tray number increases (the trays are numbered from the top so that Tray 1 is uppermost and the bottom tray is Tray 20). The phenol content of the sample collected 20 July 1979 when the maximum amount of sodium carbonate was being added, is very similar to the sample collected 15 December 1979 when sodium carbonate addition was low (Table 2). The ratios of individual phenolic compounds in samples with the same tray number are also nearly identical (Table 17). Differences in the composition of liquids collected on the two dates are within the experimental error associated with the analysis methods.

Therefore we do not believe the differences in corrosion rates measured by corrosion probes in the bypasses of Trays 1, 9, and 15 can be attributed to differences in the phenol content (cresolic acids) or the distribution of phenol compounds.

Chlorides and Phenols Blamed for Corrosion

It is clear there is a buildup of water-soluble inorganic chloride in the bottom part of the T-105 tower to values much greater than those in the T-105 feed stream. The buildup may exceed 500 ppm of water soluble chloride; this amount of chloride in T-105 tower liquids gives a laboratory corrosion rate approximating the rate observed at Wilsonville when the plant processes coal containing 0.25% chloride and does not add sodium carbonate.

The 35 to 55 wt % phenolic compounds in the T-105 tower trays and the synergistic effect of chloride and phenols probably account for a significant amount of the corrosion in the T-105 tower.¹⁰

The following scenario is one explanation for the accumulation of chloride in the lower portion of the T-105 fractionating tower, which is oversized, operates at a high reflux ratio, and has a heated reboiler. The liquid withdrawal from the bottom of the column is at least twice the withdrawal from the overhead of the column. The chloride is apparently in a volatile form, such as ammonium chloride, and it distills from the reboiler and the bottom of the tower. As the chloride distills up the column, it dissolves in the highly phenolic liquid; am-

monium chloride is readily soluble in the highly phenolic liquid at the 1000 ppm level.¹⁰ The combination of chloride solubility and the greater liquid withdrawal from the bottom of the column prevents the volatile chloride from distilling to the top of the column and being removed in the overhead.

This scenario suggests ways of dealing with the corrosion problem. Removal of the phenolic fraction of the liquid feed to the T-105 column would not seem to be feasible, but it may be possible to remove the water-soluble inorganic chloride by a washing technique such as steam stripping or by a chemical reaction.

Interpreting present data to determine the pathway of inorganic chloride through the SRC process streams has been complicated by the fact that sampling took place at various process points on different days and while the sodium carbonate addition rates were being changed. Also there was the possibility of flashing off volatile HCl during sample collection. However, it appears reasonable to set the certain limitations to the pathway of inorganic chloride in the process.

The feed to, and bottoms from, T-104 are low in water-soluble chlorides. Since these were sampled during the same time period as T-105, it appears that the only possible source of the water-soluble inorganic chloride in T-105 is the bottoms from V-103.

If the finding of low inorganic chloride content for the T-104 bottoms is valid, then the inorganic chloride in T-105 must be coming from Tray 8 of the T-102 tower. Unfortunately, the sample of Tray 8 liquid we analyzed was collected 17 December 1979, rather than on 14-15 December 1979, when V-160 and V-164 were sampled. On 17 December the water-soluble chloride in the liquid of Tray 8 of the T-102 tower was much lower than that found in the V-160 two days earlier. The water soluble inorganic chloride we measured in Tray 8 was too low to account for the inorganic chloride in V-160. However, a sample of V-160 collected on 17 December also contained much less chloride than the V-160 sample collected two days earlier. The chloride content of the vessels and the process streams apparently was changing during mid December 1979 and samples collected more than one day apart should not be compared.

We place greater reliance on the T-104 data than the T-102 data, because the samples were collected during the same time period as the T-105 column samples. Hence, we believe that the major source of the water-soluble inorganic chlorides in the T-105 fractionating tower is liquid from Tray 8 of the T-102 vacuum distillation tower.

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Table 1

SRC Wilsonville Pilot Plant SRC Process Samples Used in Investigation

Sample number	Sample origin	1979 collection date
51927	T-102 tray 3	12/17
51928	T-102 tray 8	12/17
51746	T-104 overhead	12/12-14
51747	T-104 bottom	12/12-14
47176	T-105 tray 1	7/20
51726	T-105 tray 1	12/14
51824-1	T-105 tray 1	12/15 (1)
51824-2	T-105 tray 1	12/15 (2)
51940	T-105 tray 1	12/18
47177	T-105 tray 9	7/20
51727	T-105 tray 9	12/14
51825-1	T-105 tray 9	12/15 (1)
51825-2	T-105 tray 9	12/15 (2)
52015	T-105 tray 9	12/17
47178	T-105 tray 15	7/20
51728	T-105 tray 15	12/14
51826-1	T-105 tray 15	12/15 (1)
51826-2	T-105 tray 15	12/15 (2)
52016	T-105 tray 15	12/17
51743	T-105 bottom	12/12-14
51739	V-105 water	12/12-14
51656	V-105 oil	12/13
51595	V-110	12/12
51924	V-120	12/17
51744	V-1318	12/12-14
51741	V-160	12/12-14
51725	V-160	12/14
51722	V-160	12/14
51823	V-160	12/15
51926	V-160	12/17
51722	V-164	12/14
51925	V-164	12/17
51698	V-178	12/13
51913	K-125	12/17
51724	P-171	12/14
51754	Tote bin feed coal	12/12-14

Table 2

Plant Feed Parameters and T-105 Corrosion Rates
During Periods of Sample Collection¹

Date	Na ₂ CO ₃ added (1b/2 tons coal)	Chlorine in coal (wt %)	Corrosion rate (mpy) measured at	
			Tray #9	Tray #15
7/24/1979	40	0.25	< 1	< 1
12/12/1979	10	0.18	9	27
12/13/1979	10	0.18	37	120
12/14/1979	10	0.18	37	186
12/15/1979	15	0.18	93	436
12/16/1979	15	0.18	205	321
12/17/1979	15	0.18	130	204
12/18/1979	15	0.18	56	77

1. Corrosion rates by 321 SS Corrosometer (c) probe wire. mpy = mils per year. In all instances feed coal was Kentucky #9 (Lafayette).

Table 3

Analysis of T-105 Liquids Collected 20 July 1979¹

Sample	IMMR		Ashland	
			Acid number	Saponification number
Tray 1	< 10 ppm Cl	7 ppm Cl	0.36	14.4
Tray 9	< 10 ppm Cl	3 ppm Cl	0.23	18.5
Tray 15	< 10 ppm Cl	23 ppm Cl	0.21	12.0

1. Acid number is in mg KOH/g sample determined by ASTM D-664. Saponification number is in mg KOH/g sample by ASTM D-939. Addition of Na_2CO_3 was the maximum of 40 lb/2 tons coal.

Table 4

Water Extraction of Organic Chloride
from Spiked Tray 9 Liquid, T-105¹

Added compound	Before extraction (ppm)	After extraction (ppm)	% loss
p-Chlorophenol	590	477	20
p-Chlorobenzoic acid	652	547	16

1. Each sample was extracted three times with an equal volume of water.

Table 5

Loss of Chloride by Water Extraction
in Tray 1, 9, and 15 Liquids, T-105

			Chloride	
			Total (ppm)	% loss
Tray 1	12/15/1979	Sample 1	89	50
Tray 9	12/15/1979	Sample 1	303	28
		Sample 2	239	+ 3
Tray 15	12/14/1979	Sample 1	454	- 3
		Sample 2	560	17
Tray 15	12/15/1979	Sample 1	871	38
		Sample 2	961	29

Table 6

Chloride Content of Phenol and Oil Fractions

Sample	Chloride, ppm			
	Organic layer			
	after water washing	Amine fraction	Phenolic fraction	Oil fraction
T-104 Bottoms 10/13/1978	143	--	149,121	111,105
Bottoms 12/12-14/1979	130	170	--	--
T-105 Tray 1 12/15/1979 Sample 1	67	220	75	100
Tray 9 12/15/1979 Sample 2	81	--	150	100
Tray 15 12/15/1979 Sample 2	151	120	75	130
V-160 12/15/1979 Sample 2	30	170	--	--

Table 7

Chloride in T-105 Feed

	Chloride (ppm)	
	Indiana #5	Kentucky #9
T-105 feed, total sample	133	820
T-105 feed, water-washed	141	254
Wash water	6	570

Table 8

Sulfur and Nitrogen in T-105 Feed

	Indiana #5		Kentucky #9	
	Sulfur	Nitrogen	Sulfur	Nitrogen
T-105 feed, total sample	0.26%	0.59%	0.30%	0.61%
T-105 feed, water-washed	--	0.53%	--	0.60%
Wash water	trace	29 ppm	trace	96 ppm

Table 9

Effect on T-105 Corrosion
of Adding Na_2CO_3 to Coal Feed¹

Date	Na_2CO_3 added (1b/2 tons coal)	Average corrosion rate (mpy) measured at Tray 15
6/24-27/1979	none	339
6/28 to 7/1/1979	40	N/A
7/1-6/1979	40	44
7/6 to 8/7/1979	40	< 10

1. Corrosion rates by 321 SS Corrosometer (c) probe wire. mpy = mils per year.

Table 10

Constituents in T-105 Liquids
Collected Prior to Carbonate Additions¹

Date and location	Total		Sulfate	Ammonia	Thiocyanate	Cyanide	Na	Ca	Mg
	Chloride	chloride							
6/24/1979 Tray 1	<2	41	<10	550	390	1.40	14	8.4	4.7
Tray 15	170	410	<10	480	360	1.30	10	3.7	<0.5
Bottoms Tray 20	280	510	no extract	310	670	0.32	14	7.0	10
6/26/1979 Overheads	<2	270	insufficient sample	1450	370	0.86	40	8.8	16
Tray 1	<2	74	<10	650	300	0.72	16	<1.0	<0.5
Tray 9	140	620	<10	580	400	1.40	37	1.6	13
Tray 15	220	1350	<10	420	360	0.80	8	1.7	<0.5
6/28/1979 B131 B	420	900	no extract	300	460	1.20	27	10.2	9.6

1. All values in parts per million.

Table 11

Ammonia Extracted with Water from Organic Layer, T-105

				Water extract	
				Cl (ppm)	NH ₃ (ppm)
Tray 1	12/15/1979	Sample 1		1.9	2.2
		Sample 2		1.8	2.3
	12/14/1979			2.5	2.1
Tray 9	12/15/1979	Sample 2		151	3.8
	12/14/1979			42	2.2
Tray 15	12/15/1979	Sample 1		294	26
		Sample 2		532	30
	12/14/1979			370	17
V-160	12/14/1979			212	17

Table 12

Chloride Content of Some Kentucky Coals

KCERL number	Coal seam	County	Chloride (wt %)
7237	11	Webster	0.18
7239	11	Webster	0.18
7242	4	Butler	0.02
7245	9	Muhlenberg	0.08
7246	9	Muhlenberg	0.11
7244	11	Muhlenberg	0.05
7248	9	Muhlenberg	0.04
7253	9	Henderson	0.05
7224	9	Hopkins	0.03
7227	14	Hopkins	0.02
7229	4	Hopkins	0.03
7232	11	Union	0.04
7235	9	Union	0.14
7222	11	Hopkins	0.03
7122	12	Hopkins	0.03
7124	14	Hopkins	0.02

Table 13

Carboxylic Acids in T-105 Liquids
Based on Sodium Bicarbonate Extraction

Sample	Collection date	Carboxylic acid (wt %)
Tray 1	7/20/1979	0.6
Tray 9	7/20/1979	0.2
Tray 15	7/20/1979	0.1

Table 14

Gas Chromatography: Relative Response Factors¹

Compound	Average response factor	Deviation
o-Cresol	0.928	±0.007
m-Cresol	0.924	0.008
p-Cresol	0.953	0.012
4-Ethylphenol	0.893	0.005
2,3-Dimethylphenol	0.942	0.016
2,4-Dimethylphenol	0.958	0.012
2,5-Dimethylphenol	0.986	0.021
2,5-Dimethylphenol	0.980	0.005
3,4-Dimethylphenol	0.960	0.002
3,5-Dimethylphenol	0.926	0.002
2-n-Propylphenol	0.963	0.006
4-n-Propylphenol	0.933	0.004
Phenol	0.882	0.012

1. Standard: 2,6-Di-tert-butyl-4-methylphenol. Response factors determined for Carbowax gas chromatography column at IMMR, March 1980.

Table 15

Gas Chromatography Retention Data for Phenols and Carbazole¹

Column: 3 ft x 2mm glass
 Packing: 4% and 9% Carbowax 20m on 100/120 mesh Supelcoport
 Carrier: helium
 Temperature: 100°C for 1 minute, increasing by 6° per minute to 212°C

	Retention time (min)	Relative time
2-Chlorophenol	6.51	0.85
2,6-Dimethylphenol	7.35	0.96
2,6-Di-tert-butyl-4-methylphenol	7.64	1.00
Phenol	8.75	1.15
2-Methylphenol (o-cresol)	8.81	1.15
4-Methylphenol (p-cresol)	9.91	1.30
3-Methylphenol (m-cresol)	10.03	1.31
3,5-Dimethylphenol	11.29	1.48
3,4-Dimethylphenol	11.94	1.56
4-(2-Propyl)phenol	12.01	1.57
4-t-Butylphenol	13.02	1.70
4-Methoxyphenol	14.37	1.88
4-Chlorophenol	14.83	1.94
4-Chloro-3-methylphenol	15.71	2.05
1-Naphthol	20.71	2.17
2-Naphthol	21.43	2.80
Carbazole	28.72	3.76

1. See Figures 9-12.

Table 16

Phenol Composition of T-105 Trays

	7/20/1979	12/15/1979 both samples (wt %)
Tray 1	51	45
Tray 9	47	43
Tray 15	37	34

Table 17

Phenol Compositions of T-105 Tower

Phenol ¹	7/20/1979 samples ²			12/15/1979, sample 1 ²		
	(wt %)			(wt %)		
	Tray 1	Tray 9	Tray 15	Tray 1	Tray 9	Tray 15
2,6-Dimethylphenol	1.5	0.5	--	1.6	0.3	0.4
Phenol and o-cresol	15.7	3.1	2.6	13.2	3.0	2.4
m- and p-cresol; 2,4- and 3,5-dimethylphenol	42.8	16.9	7.1	40.0	11.4	4.8
2,3-Dimethylphenol; 2-n-propylphenol	4.2	8.0	6.6	5.5	8.9	6.4
3,5-Dimethylphenol; 4-ethylphenol	29.1	43.3	29.8	28.8	36.0	24.2
3,4-Dimethylphenol	4.2	13.4	15.6	5.6	15.0	15.3
4-n-Propylphenol	1.5	13.0	31.3	2.6	17.6	31.8

1. The phenols listed are the ones possible; individual phenols may or may not be present.
2. Based on gas chromatographic analyses with a Carbowax column. The phenol fraction was extracted from the total sample prior to analysis. The percentages do not add to 100% because some compounds are not included.

Table 18

Basic Nitrogen Compounds from T-105 Column Liquids
Samples Collected 12/12-14/1979

	Basic nitrogen compounds (wt %)
T-105 Tray 1 Sample 1	6.5
T-105 Tray 1 Sample 2	2.5
T-105 Tray 15 Sample 1	8.0
T-105 Tray 15 Sample 1	14.5
V-164	8.5
T-104	5.0

Figure 1
Wilsonville Pilot Plant Flow Chart

Figure 2
Flow of Water-Soluble Inorganic Chloride From Dissolver to Fractionating Column

Figure 3
Chloride Distribution in Process Streams Around T-105 Fractionating Column

Figure 4
Chloride Distribution in Process Streams Around T-102 Vacuum Column

Figure 5
Chloride Distribution in Process Streams Around T-104 Distillation Tower

All quantities are parts per million

Figure 6
Chloride Distribution in Process Streams Around R-101 Dissolver

All quantities are parts per million

Figure 7
Carboxylic Acid Extraction

Figure 8A
Phenol Extraction

Figure 8B
Alternative Phenol Extraction

Figure 8C
Alternative Phenol Extraction

Figure 9
Gas Chromatograph Trace of Sample Liquid
From Tray 9, T-105 Fractionating Column

Figure 10
Gas Chromatograph Trace of Phenol Fraction
From Tray 9, T-105 Fractionating Column

Same gas chromatograph conditions as in Figure 9. Phenol fraction extracted using procedure of dissolving sample in solvent prior to extraction

Figure 11
Gas Chromatograph Trace of Phenol Fraction
From Tray 9, T-105 Fractionating Column
(Extracted From Neat As-Received Sample)

Figure 12
Gas Chromatograph Trace of Phenol Fraction
From T-105 Fractionating Tower
(Using OV-17 Capillary Column)

Conditions: Same sample as in Figure 10, but Gas Chromatography was done using capillary OV-17 0.3 mm column.
Temperature initially 50°C for two minutes, increased to 180° at 2° per minute

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QUANTIFYING AND CONTROLLING DEMONSTRATION PLANT POLLUTION: A PRELIMINARY STUDY

**A. F. Yen,
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The SRC-I Demonstration Plant will generate air pollutants, wastewater, and solid wastes—the environmental and socioeconomic effects of which are being assessed in the Environmental Impact Statement (EIS) now in preparation.

This article, presenting work done for one section of the EIS, estimates emission and effluent rates for various pollutants and discusses ways of controlling them. The basis of the estimates and discussions is the Phase 0 design, but variations and alternatives are also considered.

The air pollutants that will be subject to Environmental Protection Agency (EPA) criteria include sulfur dioxide, nitrogen oxides, particulates, carbon monoxide, and hydrocarbons.

In addition, about 1.5 million (MM) gallons a day of wastewater will be produced, the major streams being sour water, cooling tower blowdown, and coal pile rainwater runoff. Untreated wastewater is expected to contain biodegradable organics, hydrogen sulfide, phenol, ammonia, cyanides, metals, and trace organics not subject to biological degradation but amenable to activated carbon adsorption.

Solid waste will amount to approximately 800 tons (dry basis), mostly slag from the gasifier. The water and wastewater treatment plant will produce another 6-10 tons per day (TPD) (dry basis), including about 2 TPD of biological solids.

AIR

Air Emissions During Normal Operation

Basis for Emission Estimates. Two plant-derived fuels are combusted in the plant's fire heaters: fuel gas and desulfurized fuel oil. The anticipated fuel distribution is 100% fuel gas for continuous operation and fuel oil during startup. The sulfur content of fuel gas is less than 5 parts per million (ppm)—the sulfur content of the fuel oil is in the 300 to 1000 ppm range, depending on the amount of fuel oil actually used. The sulfur content of the

*ICRC

fuel oil will be decreased as oil consumption increases so that the total emissions rate of sulfur from oil combustion remains less than 10 lb/hr.

The estimates of NO_x , total suspended particulates (TSP), carbon monoxide (CO), and nonmethane hydrocarbon (NMHC) are based on factors taken from numerous EPA publications, such as AP-42, reports on combustion of synthetic fuels, and vendors' data.

Fugitive gas emissions—from leaks in equipment, valves, pump compressor seals, and open and drain valves—are based mainly on "Controlling Fugitive Emissions," *Chemical Engineering*, 27 August 1979.

The estimates for fugitive TSP are based on "EPA Technical Guidance for Control of Industrial Process Fugitive Particulate Emissions" (EPA-450/3-77-010), March 1970.

Emissions Summary. For a summary of the emission rates of sulfur dioxide (SO_2), nitrogen oxides calculated as nitrogen dioxide (NO_2), CO, TSP, and NMHC during normal operation, see Table 1.

Estimated emissions from combustion and baghouses in Table 1 are based on conservative firing rates and particulate evolution. The resulting emission rates are believed to be maximum expected rates of emission. The SO_2 emission rates from the heaters are based on 30% fuel oil with 300 ppm of sulfur.

In addition to the above criteria pollutants, emissions of these non-criteria pollutants are added: hydrogen sulfide (H_2S)—0.17 g/sec—and carbonyl sulfide (COS)—4.84 g/sec. Most of these pollutants are emitted from the gas treatment and sulfur recovery sections through the CO_2 vent stack. For the coal composition and sulfur content, see Table 2.

Fugitive Emissions. In contrast to point source emissions which can be monitored relatively easily—such as emissions from baghouses and combustor stacks—fugitive emissions coming from equipment leaks and wind loss from outdoor coal and product storage are difficult to estimate.

Although current and projected environmental quality standards and emission limitations require state-of-the-art pollution control techniques, these standards and limitations do not exist for most NMHCs which will be found in the streams in the SRC-I Demonstration Plant.

Fugitive NMHC gas emissions are estimated to be less than 1.9 g/sec (60 tons per year (TPY))—and fugitive emissions are estimated to be 3.0 g/sec (95 TPY). See Table 1. Information on mitigation of fugitive emissions is given later.

Air Emissions During Startup

The objective of the present design effort is to provide a Demonstration Plant that will start up in a controlled manner at rates below the design specifications for normal operation.

Re-evaluation will be made as detailed design proceeds to ensure that the discharges during startup will meet applicable environmental standards.

The two types of emissions during startup will be (1) emissions from process equipment sources, and (2) emissions from flaring of process streams. Emissions from process equipment sources will be slightly higher during startup than emissions during normal operation because the startup fuel will use desulfurized oil rather than the process-derived fuel gas. The fuel gas, which will be used to a great extent during normal operation, has a much lower sulfur content than the oil. However, the increase of emissions due to the higher sulfur fuel oil will be counterbalanced to some degree by the lower flow rates during startup. The

sulfur dioxide emission during startup from all sources other than the flare is shown in Figure 1. The other gaseous emissions of NO_x , CO, NMHC, and TSP will follow the same pattern of emission rate.

Normal, safe startup procedure requires flaring of process streams. The flare system will burn the H_2S to SO_2 and the hydrocarbons to CO_2 and H_2O . Flaring of process streams occurs while one section of the plant is being started up at low process flow rates and while its operation is being stabilized. The products of the section being started up are flared, rather than sent to further downstream processing for impurity removal, because flow rates are low and compositions have not reached a steady state. Startup of the gasifier, shift converter, and Selexol Unit require flaring in a controlled manner as each unit is brought onstream separately. Table 3 shows these startup conditions and the resulting SO_2 emission rate and estimated duration. The total flaring time is estimated at 10 hours. For the emission rates of pollutants from the flare, see Table 4.

Air Emissions During Shutdown

The emission rates during shutdown will be similar to those during startup, except that the duration will be much shorter.

Air Emissions During Upsets and Emergencies

The most frequent upsets which will cause a change from normal air emissions are leaking relief valves, compressor shutdowns, and upsets in the gas treating plant. These occurrences will send gases to the flare system. To ensure that these gases, including heavy hydrocarbons, are fully burned with a minimum of effect on the environment, a controlled combustor system is being evaluated as part of the flare system (see section on control alternatives). Atmospheric discharge from the controlled combustor would be smokeless and flameless and it would be designed to handle approximately 90% of all the incidents requiring the use of the flare system.

During upsets, the flare system is used to release gases in a safe manner to prevent equipment damage and personnel hazard. The rate of release of contaminants such as SO_2 may be large, but the length of time will be limited because correction will be made quickly or the plant will be shut down.

Three cases of emergency discharging are:

- (1) Blowdown of the dissolver: This would result from a major equipment failure. About 500,000 lb of hydrocarbon gas mixture would be burned at the flarestack within half an hour. The estimated SO_2 emissions (see Condition A in Table 5) represent a worst-case for this incident; it assumes that all the gas in the dissolver section of the plant is flared.
- (2) Emergency venting of the gasification unit: This may occur more frequently resulting from flow blockage downstream of the gasifier unit (see Condition B in Table 5). The flarestack would be essentially smokeless because the gasifier output consists of low molecular weight combustible gases. Total SO_2 emission is estimated at 18,800 lb per incident.
- (3) Emergency venting of the dissolver unit to keep it running while clearing a downstream upset (see Condition C in Table 5). This may occur as often as three or

four times in the first months of operation. Total SO_2 emission is estimated at 32,400 lb per incident.

Control Alternatives

Alternatives for Controlling Emissions of Sulfur Oxides and Reduced Sulfur Compounds. *Sulfur Recovery Plant.* Sulfur enters the plant as a component in the coal. Approximately 92% of the total sulfur entering in the coal feed will be recovered as elemental sulfur from the Sulfur Recovery Area. Most of the remaining 7.8% stays in the plant products. The total sulfur emitted to the atmosphere is estimated at 776 lb/day, or about 0.2% of the total sulfur entering the plant. This includes sulfur in the fuel gas to be used in-plant (169 lb/day), in the vent from the gas treatment (570 lb/day), and in the stack from the Sulfur Recovery Area (37 lb/day). See Figure 2 for the plant-wide sulfur balance.

Through a series of processing steps in the plant, the sulfur that was contained in the coal is hydrogenated to H_2S . Two parallel acid-gas removal processes are planned to remove H_2S , COS, and CO_2 from the process streams: the Selexol Process, which uses a proprietary physical solvent; and a diethanolamine (DEA) solvent process. Both these processes produce a concentrated $\text{H}_2\text{S}/\text{CO}_2$ stream which is sent to the Sulfur Recovery Area. No atmospheric emission is evolved from the DEA Unit; however, some carbonyl sulfide is emitted to the atmosphere from the Selexol Unit in a CO_2 and nitrogen waste stream (570 lb sulfur/day).

CLAUS AND BEAVON-STRETFORD COMBINATION. The sulfur recovery plant consists of two (100% spare) three-stage straight-through Claus Units and a Beavon-Stretford System. The concentrated H_2S stream from Selexol and DEA Units is converted to elemental sulfur in one of the Claus Units; and the tailgas from the Claus Unit is further treated in Beavon-Stretford. Based on data of several operating plants, typical concentrations of sulfur compounds in the Beavon-Stretford effluent are in the 50 to 100 ppmv range (as SO_2)—normally at the low end of this range. Although many alternative processes for sulfur removal are available, the combination of Claus and Beavon-Stretford was chosen because it has demonstrated high reliability in full-scale operation in similar services and because it provides high removal efficiency.

OTHER COMMERCIAL TECHNOLOGIES CONSIDERED. A combination of Claus and Shell Superscott was not selected because it has a lower efficiency; the concentration is typically about 250 ppmv (as SO_2). Another alternative, Giammarco-Vetrocoke, could replace the Stretford Unit in the Beavon-Stretford System, reducing the SO_2 concentration further, but a major drawback is the use of arsenic compounds which would appear in trace quantities in all effluents, including the sulfur product.

Two commercially available alternative technologies exist which could potentially replace the Claus Sulfur Recovery System: Stretford and Giammarco-Vetrocoke. Both of these systems have demonstrated lower sulfur emissions than Claus systems in some applications, although they would be considerably more costly for the sulfur throughput of this plant. Also, the performance of these systems is sensitive to feed stream composition and the feed stream in the proposed plant may contain process contaminants that would preclude the effective use of these alternative systems.

The Chiyoda Thoroughbred 101 Process is a potential alternative which would provide a gypsum product from the remaining sulfur in the tailgas instead of elemental sulfur. Sulfur dioxide in the offgas would be higher than with the proposed system. Other potential al-

ternatives include the Dual Alkali Process, the Cleanair Process, the Wellman-Lord System, the IFP-2 System, and a lime-limestone slurry system. All have been ruled out because they are either less efficient or more difficult to operate.

Fuel Burning Devices. Operation of the SRC-I Process and supporting processes requires significant quantities of heat which must be supplied by combustion of fuel in indirect heat exchangers (fired heaters). Four alternative fuel sources were considered: purchased gaseous fuels (natural gas or LPG), purchased petroleum distillate fuels, coal, and process-generated fuels.

Use of purchased gaseous and distillate fuels was rejected because it would consume resources which are already in short supply. Use of process-generated heavy distillate fuels or coal would require extensive facilities for control of SO_2 and particulate emissions to meet emission limitations and prevention of significant deterioration (PSD) requirements. Use of process-generated gaseous and light distillate fuels allows compliance with applicable environmental standards without requiring additional control equipment; therefore, this source was selected.

Alternatives for Controlling Emission of Total Suspended Particulates. Four particulate control alternatives are available: cyclones, scrubbers, electrostatic precipitators, and baghouses (or filters). Although cyclones could probably achieve compliance with applicable emission standards in some applications, they are generally only moderately efficient. Both electrostatic precipitators and baghouses are significantly more efficient. Baghouses were selected for particulate control in the proposed Demonstration Plant—except for the cleanup of the calciner flue gas where a scrubber would be used. That stream also contains a significant quantity of SO_2 and a scrubber can remove both the TSP and the SO_2 at the same time.

Coal Storage. If no controls are used, total fugitive particulate emissions from the active and reserve coal piles are estimated at 400 TPY. Dust suppressant systems will be employed to reduce the emissions.

Using water as the dust suppressant typically reduces emissions by 60% for the active pile and 75% for the reserve. Employing a water suppressant system for both the active and reserve piles would reduce the emissions to 130 TPY.

The emission rate can be reduced further with an application of a polymer dust suppressant system to the reserve pile. The polymer system would reduce emissions to 90% for the reserve pile. However, the polymer system is not effective for use on the active pile because the pile is continually being disturbed.

The proposed design is a water system for the active pile and a polymer system for the reserve. This alternative would reduce the fugitive emission from these coal piles to 95 TPY.

SRC Storage. The solid SRC product will be stored in open piles. A polymer dust suppressant system will be used to control fugitive particulate emission. This control method would reduce the fugitive emission rate to 17 TPY. The other, more efficient, alternative for fugitive emission control (up to 99%) would be enclosed storage. However, storage bins or silos for this material would be very costly because of the large volume involved. Therefore, this alternative is not selected.

Coke Storage. Because of its small volume and powdery nature, coke will be stored in silos and there will be virtually no fugitive emission.

Sulfur Storage. The elemental sulfur will be stored in a molten state in a closed tank.

Coal Drying. Two methods of coal drying are available:

(a) The currently proposed design uses conventional technology, with wet coal obtained from the stockpile simultaneously pulverized and dried in a system heated by combustion

flue gases. In the system of conventional technology, the feed coal is both ground and dried to 2-5% moisture in an enclosed system of mill, size classifier, hot air furnace, duct work, and baghouse. Drying temperatures in the mill are well below the oxidation temperature of the coal and sulfides. The exhaust air contains the combustion products of the furnace, the water removed from the coal, entrained particulates, volatiles from the coal and, potentially, entrained sulfurous water present in the feed coal. The baghouse is designed to remove 99+ % of the particulates.

(b) An alternative is flash drying, with wet coal crushed and dried separately. In the flash dryer system, crushed coal is injected into warm solvent. The coal-solvent mixture flows to the slurry mixing vessel, where a hot process recycle solvent and a recycle slurry are added and slurried. The water flashes off as steam and is later condensed, leaving a hydrocarbon gas that would then be injected into the vapor recovery system. The flash drying system would not produce a particulate emission, whereas the proposed conventional dryer would generate approximately 27.4 lb of particulates/hr (25% of the plant's overall emission rate).

In both systems, feed coal is obtained from an outdoor stockpile. The washed feed coal (1¼ inch × 0 inch) has a moisture content which varies from 10 to 15% during rainy or snowy seasons. This coal is fed to a ball mill and is reduced to nominal 200 mesh. The mill provides the grind size required and operates at a relatively low noise level compared to other grinders.

Grinding and drying tests will be performed on the design coal to determine the makeup of the exhaust air and to quantify the concentrations of acid gases, H₂S, HCN, hydrocarbons, particulates, and other components. The exhaust air system will be designed to capture pollutants as required by EPA and state air quality standards.

Because the flash drying system retains potential pollutants within the process, it is the most environmentally desirable option, given the present information. However, the systems currently being considered require further testing by the Pilot Plant to define the process design and operating parameters. A choice will involve trade-offs between capital cost, environmental impact, maintenance, process benefits, and operational liability. Until this is done, conventional drying-grinding has been incorporated into the current design.

Coal and Product Transferring. Fugitive emissions will be minimized at transfer points by enclosed chutes or hoods. An enclosed conveyor system will be used to contain the fugitive dust and particulates in the exhausts from the conveyors will be controlled by a baghouse.

Alternatives for Controlling Emissions of Nitrogen Oxides (NO_x). *Fuel Burning Devices.* The NO_x in flue gas is the result of oxidation of the nitrogen in combustion air and of the chemically-bonded nitrogen in the fuel. In most combustion processes, the primary oxide of nitrogen is NO, which oxidizes in air to NO₂.

The present Demonstration Plant design includes these measures to minimize NO_x emissions:

(a) **OXYGEN CONTROL.** Large process heaters and the auxiliary boiler are expected to have automatic air control. Automatic controls are not cost effective for the smaller process heaters; therefore, operators of these units will adjust air intake manually.

(b) **BURNER DESIGN AND FIRING.** On the large process heaters and the auxiliary boiler, burners yielding low NO_x emission will be used. Where possible, firing patterns such as tangential or corner firing, which are known to give lower NO_x emissions than patterns such as cyclone firing, will be used—and numerous small burners rather than a few large burners will be used.

(c) **FUEL TYPE.** Fuels containing low levels of chemically-bound nitrogen will be consumed in preference to high nitrogen fuels. The fuel gas contains less nitrogen than the fuel oil and is preferred, especially in those combustion processes that are constrained to use firing methods promoting NO_x formation (i.e., well-mixed air and fuel or high temperature combustion).

Other methods for NO_x control—in addition to the combustion control mentioned above (temperature and fuel/oxygen mixing)—are available but they have not been commercially tested in the U.S. NO_x reduction has been realized by ammonia injection into the flue gas and both noncatalytic and catalytic processes are available. Noncatalytic ammonia injection is effective only over a narrow range of high temperatures—a difficult control problem. Catalytic ammonia injection is effective at lower, more easily controlled temperatures; however, low temperatures induce corrosion problems when sulfur oxides are present in the flue gas.

Flares. No control techniques exist for reducing NO_x emissions from combustion of pilot and purge gas from flares beyond the normal design features of controlling the proportion of fuel and air to lower flame temperature. However, the contribution of the flare makes up only a very small fraction (less than 1% during normal operation) of the plant's overall NO_x emission rate.

Alternatives for Controlling Emissions of Carbon Monoxide (CO). The principal sources of CO are the vents from the Gas Treatment System and the Beavon Sulfur Removal Unit. They emit about 70% of the total CO emissions from the entire plant. The CO emission from these vents will not have adverse environmental consequences. The CO could be reduced by replacing the Selexol with other solvents—such as a Benfield solution with a lower solubility for CO—or by installing an after-burner to combust the CO. Neither method is desirable. Other solvents cannot concentrate H_2S as much as Selexol does. As a result, the feed stream to the Claus Unit would be more dilute in H_2S ; this would substantially increase the cost of the Sulfur Recovery System. Combusting the CO with an after-burner is not desired either because a great quantity of fuel would be required and would generate increased NO_x emissions.

Other sources of CO are the fire heaters, although quantities are small. There are no satisfactory control measures for reducing CO emissions beyond an efficient fire heater operation, which also minimizes fuel consumption. Most control techniques for reducing CO emission result in increased NO_x emission. EPA's policy has been to stress control of NO_x even when this might result in increased CO emissions.

Alternatives for Controlling Emissions of Nonmethane Hydrocarbons (NMHC). *Fuel Burning Devices.* Nonmethane hydrocarbon emissions from fuel burning devices will be controlled by proper applications, installation, operation, and maintenance of all related equipment and by minimizing fuel consumption.

Mitigation of NMHC Emissions during Off-Normal Operation. A controlled combustor is being evaluated for mitigating NMHC emissions during off-normal operation of the proposed Demonstration Plant. A controlled combustor is a highly efficient combustion system, similar to a gas-fired boiler burner and firebox. It would serve two purposes: (1) to efficiently destroy a wide range of materials, including polynuclear aromatic compounds (PNAs) in the flare gases, with essentially no smoke; and (2) to mitigate the illumination nuisance involved by frequent flaring.

A practicable procedure for determining the required size of the controlled combustor is to assume several emergency situations and estimate their frequency and flaring rates. Since this is a prototype Demonstration Plant, no historical data are available. The closest

types of installations for such an evaluation are refineries where some empirical operating data are available. On the basis of this data, the prediction is for a controlled combustor sized for about 5% of the maximum flaring rate to handle over 90% of the flaring incidents.

The controlled combustor, which has a substantial turndown capability, would accommodate (1) all upsets wherein recovery of normal operation without ceasing operation is attempted; (2) over 90% of all releases due to off-normal operation, including small emergency dumps; and (3) all leaks from components, such as safety valves, that can be piped to the system. Gas releases to the atmosphere from the controlled combustor system would present a low potential for contamination. Preliminary estimates show that the NMHC emissions would be a small portion of the estimated uncontrollable fugitive NMHC emissions for the plant.

The relief discharge system would include knockout vessels upstream of the elevated flarestack, where most entrained solids and liquid droplets would be removed, with water seal legs for diverting gases to the controlled combustor equipment located at ground level. At high vent gas rates, the increased backpressure would blow the water seal and allow the gas to flow to the elevated flare.

THE LARGEST PRACTICAL CONTROLLED COMBUSTOR. The controlled combustor under consideration is the largest practical size; emergency releases that would exceed that capacity will go to the elevated flare. Use of multiple controlled combustors would complicate the emergency relief system and is not technically feasible.

Emergency releases can occur almost instantaneously—and no controlled combustor design can accommodate the largest of these releases, which would produce a big flame momentarily. The flare, therefore, must be elevated to a level that is safe. Past industrial experience has shown that, if a release were too low, grass and brush at ground level may ignite and personnel below the flare would not survive.

But the controlled combustors available are all designed for installation at ground level for easier maintenance of burners and controls. There is no system that could safely handle a release such as that described above.

A 99% EFFICIENCY. A controlled combustor of the size considered is estimated to be 99% efficient in burning NMHC at its design rate. For the release rate described for the elevated flare, the combustor response would be too slow to provide good burning. More important, at these rates, air availability represents a physical constraint. Such a release into the controlled combustor would require more air than can physically be made available.

By including both a controlled combustor and an elevated flare, efficiency of combustion would be optimum for the full range of releases. The controlled combustor is efficient throughout its design rates, but an elevated flare sometimes is inefficient at low rates because material bypasses the small flame as it spills off the very large flare tip. At the larger flows, the elevated flare provides maximum efficiency because the tip is designed to ingest large air flows.

Mitigation of Fugitive Emissions. A recent study of Radian Corporation (EPA-600/2-79-044, February 1979), involving a number of petroleum refineries, found that over a third of the valves encountered leaked, contributing 60% of the "baggage" source emissions. Preliminary studies showed that good maintenance would significantly reduce these emissions.

To minimize fugitive emissions associated with leaks, a monitoring and maintenance program will be implemented and strictly enforced at the plant. In addition, these mechanical design provisions will reduce the possibility of leaks: pumps, valves, and compressors will have double mechanical seals . . . use of flanged connections will be minimized . . . sampling

connections will have double valving . . . drains and sumps will be totally enclosed and diluted with washdown water to control the emissions.

With the above measures, the total fugitive emissions for the entire plant are estimated to be less than 60 TPY. Less than 15 TPY of this will be of the NMHC material with a boiling point above 600°F. Alternatives for reducing the NMHC emission even further are now being evaluated.

TANKAGE. Vapor recovery systems are provided on all hydrocarbon storage tanks and at truck and railroad loading racks. The recovered vapor will be sent to the controlled combustor for combustion. No other technology exists for controlling these emissions.

COAL DRYING. If the conventional coal drying technology is employed, NMHC emission is expected to be negligible.

If flash drying is selected, the NMHC gas will be injected into the vapor recovery system, and the recovered hydrocarbons combusted in the controlled combustor. The amount of NMHC released to the environment would be negligible. No other alternatives for NMHC control are considered because the proposed alternative represents the best available technology.

Vehicular Emissions During Operation

Mobile Plant Equipment. Approximately 25 plant vehicles—including trucks, cranes, and maintenance vehicles—will be used by the SRC-I Demonstration Plant. For estimated air emissions from these, see Table 6. Each vehicle is assumed to travel about 8 miles per day and consume one-half gallon of diesel fuel per mile.

Highway Vehicles. During operation of the plant, the total average work force is conservatively estimated to be 500, including contract maintenance personnel. Assuming an average of two persons per vehicle and a round-trip commuting distance of 30 miles, plus an 8% additional allowance for delivery vehicles, would result in 2.2 MM vehicle miles per year, with estimated emission rates shown in Table 7.

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WATER

Assuming GKT gasifiers are used, water intake to the plant averages approximately 3445 gpm. Approximately 70% of this intake is consumed as cooling tower makeup; the balance is consumed by various process areas. Discharge of treated effluent from the wastewater treatment facility averages about 930 gpm, resulting approximately in a 2515 gpm net water consumption. The major water flows into the plant are makeup from the Green River and rainfall.

If the zero discharge alternative discussed below is implemented, the water intake would average approximately 2520 gpm. Cooling tower makeup would consume about 60% of this water. Discharge of water to the Green River would occur only when rainfall exceeds the 10-year, 24-hour storm. Up to this quantity, the rainwater is retained and sent to waste-

water treatment at a rate up to 2000 gpm. To achieve zero process water discharge, the cooling tower blowdown must be evaporated and all the treated wastewater effluent must be used as makeup water for the cooling tower. Zero discharge will affect the amount of non-evaporative cooling that can be used.

The plant includes design provisions to:

- (a) Preclude contamination of the cooling tower water by the process
- (b) Ensure that the plume of evaporated water from the cooling tower does not descend to the ground outside the plant
- (c) Prevent drift from the cooling tower from falling outside the plant site boundary

Water Treatment During Normal Operation

Major Wastewater Streams and Contaminants to Wastewater Treatment. The expected normal flow to be handled by wastewater treatment will be about 930 gpm on a yearly average (or a nominal rate of 1.5 MM gpd). Major wastewater sources are:

Process sour water	340 gpm
Cooling tower blowdown	307 gpm
Rainwater runoff	218 gpm
Steam boiler blowdown	31 gpm
Brine	16 gpm
Steam stripper top	18 gpm
	<hr/>
	930 gpm

As shown, about a third of the total wastewater flow will be sour water, the most heavily contaminated stream. Hydrogen sulfide, ammonia, and phenolics should be the major contaminants in this stream, along with some cyanide, thiocyanate, and metals. Trace complex organics, such as polynuclear aromatic compounds, will probably be present in the stream, also.

Another third of the flow wastewater will be the cooling tower blowdown. The most significant contaminant in this stream is hexavalent chromium, which is added to the cooling system for corrosion control. The common inorganic constituents originating in the makeup water to the cooling system will be concentrated and present in the blowdown. The contamination of the cooling tower blowdown with the hexavalent chromium could be avoided if nonchromate anticorrosion chemicals could be used in lieu of chromate. The possibility of using a phosphate inhibitor is being evaluated.

The remaining third of the wastewater will come from various sources: runoffs from raw coal and SRC piles, runoffs from the diked areas where the process equipment and storage and shipping areas are located, sanitary waste, landfill leachate, oil-bearing wastes, and such lightly contaminated waters as boiler blowdown and water-filter backwash.

The contaminants in the runoffs from the coal piles should be mostly dissolved inorganic common solids, although trace quantities of heavy metals may also be present. The process area runoff should be contaminated with process liquids, including certain high-boiling point hydrocarbons. The landfill leachate should contain the contaminants common to the coal pile runoff. The lightly contaminated wastewaters should contain chiefly inorganics and suspended solids.

The major contaminants of the combined wastewater stream are:

- phenolics
- ammonia
- cyanide
- thiocyanate
- toxic metals
- dissolved solids
- suspended solids
- oil and grease
- trace organics

Conceptual Design of the Wastewater System. All wastewaters generated in the plant, as well as the contaminated runoff, will be sent to the wastewater treatment facility. Figure 3 shows the conceptual diagram for the treatment facility. The treatment processes include separation of oily wastes, precipitation and flocculation of toxic metals, two-stage aerobic biological treatment followed by filtration, and a final polishing of the effluent by activated carbon and disinfection. The system provides equalization and retention basins to promote mixing and thereby prevent upset of the biological system due to shock by toxic components.

The sour water will initially be steam-stripped in the ammonia sulfide water stripping unit to remove hydrogen sulfide and ammonia. After stripping, the stream will be combined with the landfill leachate and then fed into a first-stage precipitator for nickel removal. The optimal pH for nickel removal is about 11, considerably higher than the optimal pH, 8.5, for removing other metals.

If chromate is used as the corrosion inhibitor in the cooling system, cooling tower blowdown will be treated in an electrolytic reduction unit in which the soluble Cr^{6+} is reduced to trivalent chromic (Cr^{3+}) oxide precipitates. The blowdown then will flow into a second-stage precipitator for solids removal.

Chemicals Added in Second-Stage Precipitator. The pH in the second-stage precipitator will be maintained at 8.5 for a maximum removal of other metals. Various chemicals will be added to facilitate the removal. For example, iron will aid in removing arsenic and vanadium, and polymer or alum will enhance sedimentation. After the precipitation stage, the wastewater will flow into an equalization pond where it will come in contact with other waste streams prior to biological treatment.

Also feeding into the second-stage precipitator is the coal pile runoff. Because of its high fluctuation in flow rate and concentration, it is equalized in a surge pond before feeding to the precipitator.

Process area runoff, which is likely to be contaminated with hydrocarbons, will be combined with other oily wastes for emulsion breaking and oil removal. The treated oily waste will be introduced to the equalization pond upstream of the biological system. There will be an oil skimming device in this pond to remove any floating oil not removed upstream.

Two Separate Biological Systems. The two-stage biological system consists of two separate systems, each with a reactor followed by a clarifier. The purpose of the first-stage biological system will be to remove mostly phenolics and other easily biodegradable materials. Removal of ammonia and thiocyanates, as well as refractory organics, should occur principally in the second-stage biological system. Since the microorganisms in the second stage are usually more sensitive to environmental changes and more susceptible to inhibitions, the first stage should provide stability for the second stage. To further protect the biological system from potential inhibition and toxicity, provisions will be made for adding powdered activated carbon.

An alternative to the proposed design is being considered—use of oxygen rather than air in the biological systems. Because oxygen is readily available onsite, an oxygen activated sludge system could be more cost effective than an air activated sludge system.

The effluent from the biological system will be further polished before discharge by filtration and activated carbon adsorption. Filtration removes residual suspended solids and adsorption greatly reduces trace organics, particularly polynuclear aromatics. Sludges and precipitates from water treatment are disposed as discussed in the section below on solid waste disposal.

Sanitary waste will be treated separately in a package biological unit. Because of fecal coliforms, disinfection of this stream is required before discharge. Chlorination or ozonation will be employed. The decision between the two will be made during detail engineering.

After final treatment, effluent from the wastewater treatment facility on an average of 1.3 MM gpd would enter the Green River at approximately Mile Point 19 via a diffuser discharge structure.

Anticipated Treated Effluent Quality. Table 8 shows the anticipated composition of the treated wastewater. The estimated trace organics, polynuclear aromatics (PNAs), concentrations are listed in Table 9. The values shown in these two tables have been extracted from the literature. Based on this information, the wastewater treatment system should virtually remove all the major and trace contaminants. PNAs, too, should be removed substantially in the biological treatment system and the activated carbon adsorption.

A wastewater characterization and treatability study is being conducted to obtain more definitive information on the effluent composition. A major effort will be made to identify the trace contaminants and to determine their removal efficiencies.

Wastewater Treatment Alternatives

Three alternatives to the base case of discharge of treated effluent to the Green River were considered:

- (1) Holding untreated process area runoff and wastewater for natural evaporation. But this was not feasible because the natural evaporation rate is lower than the precipitation rate for the proposed plant site.
- (2) Putting the coal pile inside a building, thus decreasing the quantity of runoff requiring treatment to about 80 gpm. But this alternative was rejected because of high costs.
- (3) Replacing the proposed discharge to the Green River with a zero wastewater discharge system. This is being evaluated.

Zero Wastewater Discharge. Figure 4 shows a simplified flow diagram of a possible zero wastewater discharge alternative. Here, wastewater receives primary, secondary, and tertiary treatment similar to that described in the preceding section.

The treated effluent is then used as partial makeup water to the cooling tower. The balance of makeup water comes from treated process water and condensate from an evaporation system. Blowdown from the cooling tower, along with a small brine stream, is passed into an evaporator where the solids are concentrated for disposal.

The advantages of this type of system are: no pollutant discharge to navigable waters, reduced river water intake, simplified wastewater treatment system design work, and the potential for integrating the zero discharge system with other portions of the Demonstration Plant. The disadvantages are: water soluble salts are difficult to dispose of; evaporator

downtime may be high due to scaling, fouling, and corrosion problems; and high capital and operating costs. Several cooling alternatives, discussed below, are considered because the leakage of heavy hydrocarbons into an evaporative cooling system could pose a potential health hazard.

Disposal of Water-Soluble Solid Wastes. Approximately 35 TPD of residue will leave the evaporator. Disposal of the highly water-soluble solid waste is a major problem for zero discharge.

Major waste streams originate from the gasification section, the dissolver section, the cooling tower blowdown, and the boiler feed water treatment effluent.

Waste from the gasification section results from ash leaching. Heavy metals contained in the ash will be leached into this waste stream along with other constituents from the ash. The amount of blowdown of wastewater from the gasification is set by a maximum chloride concentration that determines the amount of water-soluble solids in this stream.

Water from the dissolver section will also contain chlorides. The chlorides and other water-soluble solids will consist only of those that volatilize in the dissolver. Thus, heavy metals and nonvolatile solids will be in low concentrations compared to the gasification waste.

Cooling tower blowdown and boiler feedwater treatment residues will contain inorganic compounds of calcium, sodium, magnesium, sulfate, and chloride in essentially the same proportion as they enter with the river water.

Precipitation runoff from the coal pile and from the slag landfill will also contain dissolved inorganic solids.

The residue from the evaporator is water-soluble; it dissolved in the wastewater. Consequently, the solid cannot be disposed of by conventional landfill. Precipitation on such a landfill would redissolve the solids, causing an endless recycle through the water reclamation system.

Several methods of managing these solids are under evaluation. Currently, a method of encapsulation of the solid residue is being considered, with final disposal in a secure landfill. Other alternative disposal methods will be considered during Phase I engineering.

Disposing of these water-soluble salts is one of the key environmental decisions to be made during the detailed design phase and one of the major factors in the ultimate decision to use a zero discharge water treatment system.

Water Balance

Figure 5 shows a plant water balance for the wastewater treatment system which discharges treated wastewater to the Green River. This figure shows that an average water use from the Green River is approximately 3445 gpm. Average wastewater flowrate to wastewater treatment is 931 gpm. This consists of cooling tower blowdown at an average rate of 307 gpm and other wastewater streams at a rate of 624 gpm.

Figure 6 shows a plant water balance if a zero wastewater discharge system is selected. This figure shows that the average water withdrawn from the Green River is approximately 2500 gpm. Treated wastewater effluent at a flowrate of 608 gpm is used as makeup water to the cooling tower. Blowdown from the cooling tower enters an evaporator where solids are concentrated and sent to a secure landfill. Condensate from the evaporator at a flowrate of 290 gpm is sent back to the cooling tower as additional makeup water. Since this condensate will contain very low total dissolved solids levels, the blowdown is 27 gpm less than the blowdown shown in Figure 5.

As seen from Figures 5 and 6, the largest use of water is from the cooling tower (2407 gpm of makeup in the base case and 1482 gpm of makeup in the zero wastewater discharge case). Most of the water to the cooling tower is lost through evaporation. The second largest use is the boiler feed water system (350 gpm).

A comparison of Figures 5 and 6 shows that consistent water capacities were used in both cases for total makeup water to the cooling tower for wastewater treated. In Figure 5, the total water makeup to the cooling tower is 2407 gpm, with a blowdown of 307 gpm. In Figure 6, the total water makeup to the cooling tower (including process water, wastewater effluent, and condensate) is 2380, with a blowdown of 280 gpm. The difference in makeup rates, 27 gpm, is accounted for in the differences in blowdown rates. In Figure 5, the total wastewater treated is 931 gpm. Excluding the blowdown of 307 gpm and a brine stream of 16 gpm, the total water treated would be 608 gpm. As shown in Figure 6, this is the amount treated in the wastewater treatment process. The blowdown and brine are sent to the evaporation, as shown in Figure 6.

Basis for Estimates. Water balance can be affected by many factors: enthalpy balance, the heat rejection alternative employed and its operating conditions, the type of gasifiers used, and rainfall intensity and duration.

The balance, as shown in Figures 5 and 6, is based on the enthalpy balance presented in Table 10. The conventional wet evaporative cooling tower is chosen as the primary heat rejection device for the balance. Evaporation from the cooling tower varies with air humidity and temperature; the average evaporation of 2100 gpm, as shown in these figures, is for summer conditions found at the site. These assumptions are made: that the cooling water is operated at about seven cycles of concentrations—and that GKT is the gasifier. The rainfall used for the balance is the 10-year, 25-hour storm.

Balancing Water Use and Demand. Balancing of water supply and demand is critical for the zero discharge alternative. At this time, insufficient design work has been completed to permit an accurate water balance. If the zero discharge alternative is selected for the plant, final decisions on the design of the plant cooling system will require a thorough evaluation of the rate at which the water retention basin for rainfall can be emptied. Rainfall patterns and plant operation modes, and their effect on water consumption, must be thoroughly evaluated to ensure that, under various combinations of weather conditions and operating modes, all wastewater can be consumed adequately within the plant to accomplish zero discharge to the river.

Cooling Tower Drift Quantity and Composition. Drift is discharged from the cooling tower. The drift portion of the draft and evaporation in the water balance is about 60 gpm. Minerals in the drift normally consist of those in the treated river water concentrated seven times. The estimated composition of the drift is shown in Table 11.

Concerns have been raised about the potential of carcinogens contaminating the cooling water as a result of leaks in the proposed cooling system and ultimately escaping to the atmosphere through cooling tower drift and evaporation. The alternative design provisions discussed below in this article would preclude contamination of the cooling tower water by the process. A final choice cannot be made until the detailed design is completed.

Waste Heat Rejection Alternatives

Available Cooling Tower Alternatives. Several alternative waste heat rejection methods are available. Five types are considered here, each involving different amounts

of water usage and different risks of hydrocarbon release to the atmosphere through the cooling system. As discussed above, the choice of a cooling system has a large effect on the choice of a wastewater treatment system. Figure 7 shows these alternative configurations.

Aerial Coolers. These are preferred and feasible for cooling process streams to about 150° or 140°F, thus reducing the size of the cooling water system and the makeup water requirements. Process fluids flow inside finned tubes and atmospheric air is circulated around them by motor-driven fans. Aerial coolers require an extensive plot area. By using welded connections, the possible leak points at flanges are minimized. Leaks of process materials can be readily detected with monitoring equipment and visual inspection.

Wet Evaporative. These cooling towers are the most widely used type. Cooling water is circulated through exchangers to remove heat from the process fluid and is cooled by contacting air in a cooling tower that is open to the atmosphere. The circulating water may become contaminated with process materials introduced through cooling system leaks from higher-pressure process environments. These contaminants could escape to the atmosphere through the cooling tower evaporation and drift. Drift losses may be minimized by high-efficiency drift eliminators. This type of system is the lowest in overall cost and is suitable for cooling down to 85° or 95°F, depending on atmospheric wet-bulb temperatures. It also has the highest makeup water requirements.

Dry Indirect. This is a method in which the process heat exchangers would be cooled by a circulating heat transfer fluid isolated from the environment. The circulating water would be cooled inside tubes by air in an aerial cooler. The dry indirect system eliminates evaporation and drift losses of water to the air with the advantage of reducing the plant makeup water requirements and any subsequent impact on the river supply.

The heat transfer fluid is low-pressure, moderate-temperature, corrosion-inhibited water so that leakage into this water would not be a problem. If a leak occurs, the process materials would be separated from the heat transfer fluid in a disengaging drum. Vapors would be flared and liquids would be recovered until the plant could be shut down for repairs. Any contaminated heat transfer fluid (water) would be sent to the water treatment system. The dry indirect system thus prevents risk of contaminating the atmosphere by leaks of certain process hydrocarbons into the circulating cooling water.

A major disadvantage of the dry indirect system is the cost involved, which is considerably more than for the conventional wet (evaporation) water cooling tower system.

Wet/Dry. This is a type of cooling tower that has a dry upper section (cooling water in tubes, cooled by upward air flow) and a lower wet evaporating cooling section, with the water in open contact with air in order to cool by evaporation. The principal advantages of this type are:

- Diminish visible water-vapor plume and reduce fog
- Obtain a colder fluid temperature that approaches ambient wet bulb
- Conserve makeup water as compared with the 100% wet evaporative cooling tower

Direct Contact After Cooler (DCAC). This is a method of cooling the compressed air feeding into the oxygen plant. Here, air is passed up a packed or tray tower and cooled with a recirculating water stream that passes countercurrently down the tower. The circulating water in turn is cooled with the cooling water system through a heat exchanger.

Cooling process water requirements for the DCAC heat exchanger are only 3% of the total process water requirements of the Demonstration Plant. The use of DCAC would not significantly affect the water balance for the zero wastewater discharge alternative.

Base Case and Alternative Designs. The base case design of the Demonstration Plant uses wet evaporative cooling towers for process needs, a direct contact after-cooler for the oxygen plant, and some aerial coolers for streams containing heavy hydrocarbons.

Environmental concerns with evaporative cooling are: blowdown disposal, drift losses, visible plumes, noise, and the release of toxic and carcinogenic compounds to the environment through contamination of the cooling water by polynuclear aromatic (PNA) compounds and organics.

The possibility that leakage into cooling water would contaminate the atmosphere can be almost eliminated by providing a separate aerial cooling system with a dry direct cooling tower for part of the process. The sections served would be the coal dissolver, hydrogen recovery, expanded-bed hydrocracker, and coker-calciner.

Alternative A. By using the all-dry indirect type, the risk of atmospheric contamination by the cooling water system is minimal. However, since there is no wet evaporative cooling tower, consumption of treated and recycled wastewater as cooling tower makeup would be eliminated. Since the zero wastewater discharge system uses treated wastewater as partial cooling tower makeup, this system would have to be modified if dry indirect cooling were used. A possible modification would be recycling the treated wastewater to the process, coal preparation, and slag disposal areas—or as partial makeup as boiler feed water.

Alternative B. Dry indirect cooling systems would be provided for all the process cooling water users, with a wet/dry cooling tower system for the oxygen plant.

Alternative C. Dry indirect coolers would be provided for cooling process streams that have appreciable concentrations of objectionable hydrocarbon materials—PNA. A conventional wet cooling tower system would be provided for the rest of the process users, and a wet/dry cooling tower for the oxygen plant.

Table 12 is a preliminary comparison of several factors for the base case and the three alternatives. Based on these factors, the base case and Alternative C appear to be better than the others. Before a final decision is made, a more detailed engineering and environmental review will be conducted.

PNA Isolation. As discussed earlier, a major potential source of environmental pollution with hydrocarbons containing PNA is leakage from process into circulating cooling water. They could escape to the atmosphere through evaporation and drift from the cooling tower. In the final design, PNA hydrocarbons will be isolated to prevent such escape. Design and operating features can affect PNA isolation. Indirect heat exchange can also be used in two different types of cooling systems. Thus, three alternatives are available.

The first alternative involves specific design and operating features, including the following:

- (1) A mechanical design will minimize mechanical failures
- (2) Materials of construction will be chosen for appropriate resistance to corrosion and erosion
- (3) Thermal stresses in water coolers will be minimized by placing heat recovery exchangers and aerial coolers ahead of water coolers to limit the inlet temperatures
- (4) Heat recovery exchangers and aerial coolers will be used to the fullest extent economically and operationally feasible and this will greatly decrease the potential for leakage into the cooling water system
- (5) A monitoring system at the cooling tower will alert the operators so that the plant can be shut down with negligible loss to the environment if a major leak into the cooling water occurs

These features have the advantage of being conventionally used in many plants. The disadvantage of relying entirely on them is the high risk of not achieving PNA isolation, which would cause frequent plant shutdowns for repair.

The second alternative is the use of dry indirect type of cooling systems. A potential advantage is that water usage is decreased; however, the cost is very high. A minimum

water evaporation requirement is necessary in the plant in order to achieve zero process water discharge. The use of this type of cooling exclusively would not preclude the zero discharge alternative.

The third alternative available is the use of wet indirect type of cooling systems. This type is similar to the dry indirect type, except that the heat is ultimately rejected through an evaporative cooling tower rather than an aerial cooler. The incremental cost is moderate as compared to conventional evaporative systems. A potential disadvantage of this method of cooling is that water usage is not reduced as compared to conventional wet evaporative cooling tower systems.

During detailed design work, repeated evaluations will be made to obtain an optimum, feasible, and operable overall combination of types to prevent environmental impact from escaping PNA hydrocarbons.

The Effect of Startup on Wastewater Treatment

Startup of the physical-chemical portion of the treatment system would take about one week; normal startup of the biological portion would take a few weeks. To shorten the time period, seeding micro-organisms will be obtained from a wastewater plant treating a similar waste.

Prior to the full establishment of the acclimated biomass, the treated wastewater would contain relatively high concentrations of biodegradable impurities. The partially-treated effluent will be recycled to a maximum extent during this period of time.

Holding the partially-treated wastewater in open basins could pose some odor problems and increase chances of releasing volatile organic compounds to the atmosphere. These problems, if they occur at all, would be temporary.

If a large rainfall (10-year, 24-hour storm) occurs during the startup period, it could be stored in the surge basins and then withdrawn at a steady rate from the basins to the treatment system. The basins can store the process water and the rainfall up to three weeks. The impact of the storm on startup is expected to be small.

If the zero discharge alternative is selected, normal startup of the evaporator would take about one week. However, until the process heat load reaches a certain level and the wet cooling system has the evaporation capacity needed, treated wastewater will have to be stored or reused in the process, coal preparation, and slag disposal areas to eliminate discharge to the river.

The Effect of Shutdown on Wastewater Treatment

During shutdown, process stream flows entering the treatment facility will be low but flows of coal and SRC pile runoff, secure landfill runoff, and process area runoff could continue. The major contaminants in these streams are inorganics and they can be removed by the physical-chemical section of the treatment system as in normal operation. The biological portion of the treatment systems needs food to sustain itself. External food would be added to support the growth during the shutdown period.

The zero discharge operating mode cannot be practiced during shutdown, because the evaporative capacity of the cooling tower will not exist. Either storage or reuse of the wastewater would be required.

The Effect of Emergencies on Wastewater Treatment

During emergencies, several sources of contaminated water could exceed the capabilities of the storage system and, later, treatment and reclaiming systems. Analyses are only partially complete on environmental criteria.

There are three principal sources of contaminated water during emergencies, in addition to those discussed under startup and shutdown:

- (1) Massive leaks to the cooling water system may occur. This would require emergency shutdown, followed by reclaiming or treatment of all the water in the cooling system.
- (2) Oil-water mixtures could be produced as a result of equipment failure—during a fire, for example.
- (3) Tankage or loading leaks could occur after shutdown of the plant. Though reclaiming the waste produced during normal operation would be practical, the quantities may exceed storage capacity for contaminated water during shutdown—and this would constitute an emergency.

Part of the problem in accommodating the storage of water produced during emergencies is the type of storage required. Over 17 acre-feet of water retention capacity is available as open storage, many times the capacity available as closed storage. Much of the wastes produced during the period described in the preceding sections cannot be kept in open storage without processing because volatile organic compounds are present that would vaporize. One alternative available is to include processing adequate to convert some or all wastes to forms that can be stored in the open.

Activated carbon treatment and filtration of many streams would make them acceptable for open storage and possibly for cooling makeup. Treatment data are needed to define the systems required.

Preliminary information and designs are available on carbon treatment and on neutralization and precipitation of coalpile runoff. These designs are now only suitable for reclamation and cleanup to provide low-pressure boiler feedwater. Ongoing work is directed at defining criteria for treatment with these systems for cooling water makeup.

Slag leachate can be processed for discharge using existing technology, as can effluents generated by coal receiving, handling, and storage. Providing a small treating system would permit extended shutdown of the plant and avoid concern over pollution due to leachate.

The biological treatment system with completely mixed reactors can tolerate moderate shock loads, but severe spillage of toxic materials can destroy the biomass. This problem can be alleviated by a spill prevention and detection program, coupled with provisions to prevent the toxic material from reaching the biological system. The capability of adding powder-activated carbon to the biological system is also being considered. Adding powder-activated carbon could, in time of emergency, prevent some irreversible damage to the biomass.

* * *

SOLIDS

The three principal types of solid wastes generated at the SRC-I Demonstration Plant will be flydust and slag from the gasifier (700 TPD), metal bearing sludge from the wastewater

treatment area (8 TPD), and biological sludge also from the wastewater treatment system (2 TPD). If the zero discharge concept is implemented at the plant, 35 TPD of water-soluble solid wastes will be generated, in addition to the three types discussed above.

Disposal methods, discussed below, were chosen after testing the wastes to determine if they will be classified as hazardous or nonhazardous under the Resource Conservation and Recovery Act (RCRA).

Slag and Flyash Leaching Tests

Preliminary findings based on the portion of a leaching test program on gasifier slag and flydust, completed 25 May 1978, indicate that the materials are well within the bounds of the nonhazardous criteria defined by RCRA.

Kerr-McGee ash concentrate from the Wilsonville Pilot Plant and Kentucky #9 coal were sent to the laboratory scale, GKT gasifier in Essen, Germany. Three pounds of gasifier flydust and slag were received by Oak Ridge National Laboratory (ORNL) on 18 February 1980. The material breakdown was:

Flydust—Run 175	466.73 g
Flydust—Run 179	479.92 g
Slag—Run 179	483.73 g

Run 175 in the GKT gasifier was a lower temperature, lower oxygen/coal, moisture and ash free test than run 179. The operating conditions for run 179 are more representative of the projected actual gasification operation at the Demonstration Plant. Table 13 is a general chemical composition of the gasifier slag and flydust. Table 14 is a breakdown of selected elements in both the Kentucky #9 coal, feedstock, and the resulting slag and flydust.

The EPA-EP batch leaching tests were conducted on the slag and the flydust samples. This test meets the toxicity testing criteria outlined in the 19 May 1980 RCRA regulations.

The air-dried samples of slag and flydust were combined with 16 times their weight of deionized water and the mixture was agitated for 24 hours. During this agitation, if the initial pH of the mixture was above 5.0, it was adjusted and controlled at 5.0 plus or minus 0.1 unit by the addition of 0.5N acetic acid.

The results of the chemical analysis of leachates for the eight trace elements included in the Primary Drinking Water Standards and in the RCRA standards appear in Table 15. In addition to the eight priority trace elements, copper, nickel, vanadium, and zinc are also analyzed because of their environmental importance and the consequential possibility that they may be added to the list of eight trace hazardous elements in future legislation. Parameters measured and controlled during the extraction appear in Table 16. The RCRA standards listed as a comparison basis in Table 15 are simply the Primary Drinking Water Standards multiplied by a factor of 100.

Batch leach testing using the EPA extraction procedures and the interim RCRA hazardous waste criteria indicates that the gasifier slag and flydust will not be classified as hazardous wastes. Priority pollutant trace elements are leachable in quantities much less than those specified by the RCRA regulations. Flydust, the finest fraction of waste generated, releases more soluble trace elements than the coarse slag. Cadmium and lead concentrations in the flydust exceeded Primary Drinking Water Standards, but all other trace element concentrations were well within these standards.

Additional gasifier material has been obtained for column leaching tests which are next on the leaching study agenda. The columns in which the tests will be conducted will have a 30-cm (12-inch) depth of slag or flydust and will be dosed daily with one liter of demineralized water (about one column volume). Generally, column leachate concentrations are less than batch leachates, which may be due to shorter contact time between the water and the slag.

Slag and Flydust Disposal

Even though the waste does not exceed RCRA criteria for toxicity and consequently may not be classified as a hazardous waste, any nonhazardous waste must be disposed to minimize or eliminate ground water contamination. Figure 8 shows the design of the slag/flydust storage pond. The pond will have a 15 cm (6-inch) clay liner resting on compacted natural soils. Slag settling to the bottom of the ponds will be under several feet of water. The pond will be diked and will be installed onsite to provide better quality control.

Another alternative being considered is to construct the first ash pond with a design similar to one for hazardous wastes. Because the laboratory batch testing procedures may not be indicative of slag leachate resulting from prolonged storage under chemically reducing conditions, long-term leachate data would then be collected on the first pond to determine conclusively if it is necessary to construct later ponds in a manner closely parallel to the secure landfill for the metal bearing and biological sludges. The disadvantage of constructing the first pond like a hazardous landfill is the high cost.

Metal-Bearing and Biological Sludges

No leaching tests of metal-bearing sludges and biological sludges have been conducted or are planned because it is certain that traces of toxic metals and organics will be present in these sludges and that they will accordingly receive a hazardous classification under RCRA.

The quantities of these sludges are relatively small and they will be stored in a secure landfill as illustrated in Figure 9. The landfill liner will consist of a 20-mil synthetic material which will be covered with 0.9 meters (3 ft) of compacted clay.

Leachate collection systems above the clay will collect leachate generated in the landfill and return it to the wastewater treatment area. A leachate detection system will also be installed below the synthetic liner and the leachate analyzed to determine if any leaks are present in the liner. Monitoring wells will be installed upstream and downstream of the landfill, in accordance with the RCRA regulations, for early detection of contamination of the ground water.

Stabilization of Solid Waste

In addition to the above alternatives, the feasibility of stabilizing the gasifier solid wastes, the metal-bearing sludge, and the water-soluble zero discharge residuals will be evaluated. Several commercial processes are available for stabilizing solid wastes. Some do it by greatly decreasing the permeability of the materials, thereby limiting the leaching of the materials

to the surface. Others "fix" toxic metals by chemical reactions and render the metals insoluble.

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Table 1
Air Emissions Estimate (Normal Operation)

Equipment description	Estimated maximum emissions (g/sec)				
	SO ₂	NO ₂	CO	TSP ¹	NMHC ²
Flue gas from combustion					
12-1 Preheater Stack A	1.742	8.625	1.895	0.215	0.109
12-2 Preheater Stack B	1.742	8.625	1.895	0.215	0.109
12-3 Heater Stack	0.105	0.979	0.129	0.03	0.020
12-4 Heater Stack	0.109	0.759	0.146	0.164	0.026
13-1 Heater Stack	0.906	1.88	0.157	0.043	0.053
15-3 General Startup Aspirator	--	0.101	0.013	0.015	0.003
21-1 Coking Heater	0.357	1.76	0.578	0.088	0.045
21-2 Scrubber Stack	2.19	1.89	0.201	0.645	0.023
23-1 Dowtherm Heater	0.162	4.8	0.41	0.30	0.064
23-5 Auxiliary Boiler Stack	0.293	0.31	0.15	0.08	0.019
28-1 Common Heater Stack	0.292	1.44	0.133	0.063	0.013
Subtotal	7.898	32.17	5.707	1.858	0.484
Emissions from baghouses					
11-1 Tunnel Vent Exhauster	--	--	--	0.001	--
11-2 Exhauster	--	--	--	0.002	--
11-3 Exhauster	--	--	--	0.002	--
11-4 Tunnel Exhauster	--	--	--	0.018	--
11-5 Tunnel Exhauster	--	--	--	0.018	--
11-6 Tunnel Exhauster	--	--	--	0.002	--
11-7 Exhauster	--	--	--	0.009	--
11-8 Pulverized Bag Collector	--	--	--	0.777	--
11-9 Pulverized Bag Collector	--	--	--	0.777 ₃	--
11-10 Coal Bunker Dust Filter	--	--	--	0.005 ₃	--
11-11 Coal Silo Vent Filter	--	--	--	0.075 ₃	--
11-12 Coal Silo Vent Filter	--	--	--	0.075 ₃	--
11-13 Gasification Coal Silo Vent	--	--	--	0.013 ₃	--
11-14 Rod Mill Bin Vent Filter	--	--	--	0.002	--
11-15 Pulverized Prefeed Dust Filters	--	--	--	0.0044	--
11-16 Coal Bin Vent Filters	--	--	--	0.002 ₃	--
13-2 Blower	--	--	--	0.001	--
15-1 Coal Bin Baghouse	--	--	--	0.145	--
21-3 Dust Collector	--	--	--	0.252	--
21-5 Dust Collector	--	--	--	0.121	--
21-6 Dust Collector	--	--	--	0.121	--
24-1 Tunnel Exhaust Fan	--	--	--	0.043	--
24-2 Dust Collector Exhaust Fan	--	--	--	0.017 ₃	--
24-3 SRC Silo Vent Filters	--	--	--	0.033 ₃	--
Subtotal	0	0	0	2.515	0
Emissions from other sources					
16-1 CO ₂ Vent Stack ⁵	--	--	11.6	--	--
25-1 Oil Storage Tank Vent	--	--	--	--	0.466
26-1 Flare	1.5993 ⁴	0.066	--	--	--
Fugitive losses	--	--	--	3.0	1.9
TOTAL	9.50	32.24	17.31	7.373	2.85

1. Total suspended particulates.
2. Nonmethane hydrocarbons.
3. Intermittent source, averaged over 24 hours.
4. Intermittent source, included with continuous source, averaged over 3 hours.
5. This stack includes emission from the sulfur recovery area; see text for the reduced sulfur emission from this stack. SO₂ is also present but amount is negligible.

Table 2

Feedstock Composition (Wt %, Moisture-Free Basis)

Carbon	71.0
Hydrogen	5.0
Nitrogen	1.5
Oxygen	8.4
Sulfur	3.4
Pyrite	1.50
Organic	1.80
Sulfate	0.10
Chlorine	0.2
Ash	<u>10.5</u>
Total	100.0

Table 3

SO₂ Emissions from Flarestack during Startup

Condition*	Description	Rate (g/sec)	Time (hr)
A	Gasifier startup	1188	4
B	Shift startup	1188	2
C	Shift stabilize	1188	2
D	Selexol startup	0.008	2

*Combustion is based on no supplemental fuel.

Table 4

Flarestack Emissions during Startup

Pollutant	g/sec	lb/hr	Duration (hr)	Total lb
SO ₂	1,180	9,400	8	75,200
NO _x	39	310	10	3,100
CO	38	300	10	3,000
NMHC	0	0	10	0
TSP	1	8	10	80

Table 5

SO₂ Emissions from Flarestock during Emergency Upsets

Condition	Description	Emission Rate		Time (hr)	Total (lb)
		(lb/hr)	(g/sec)		
A	Dissolver unit blowdown	27,000	3,402	1/2	13,500
B	Gasification unit without purification	9,400	1,184	2	18,800
C	Dissolver unit without purification	16,200	2,041	2	32,400

Table 6

Emissions from Mobile Plant Equipment during Operations

Pollutant	Emissions		
	lb/10 ³ gal*	lb/yr	t/yr
CO	92.2	3,400	1.7
HC	300	11,000	5.5
NO _x	524	19,000	9.6
SO _x	31.2	1,100	0.6
TSP	17.7	650	0.3

*From EPA publication AP-42, Table 3.2.7-1, p. 3.2.7-3.

Table 7

Emissions from Highway Vehicles during Operation

Pollutant	Emissions* **		
	g/mi	g x 10 ⁶ /yr	t/yr
CO	23.0	49	55
HC	4.2	9	10
NO _x	3.1	7	7
SO _x	0.20	0.4	0.5
TSP	0.44	0.9	1.0

*Emission factors for all highway vehicles are given in EPA publication AP-42, Table D.7.1, p. D.7.1. The mix includes about 8% heavy-duty vehicles, which commuters would not use. However, because many of the deliveries to the plant will be heavy-duty vehicles, the referenced basis was used for these emission determinations.

**Only a small portion, perhaps 10% of the above emissions, would occur at the Demonstration Plant site.

Table 8*

Liquid Effluents--Anticipated Effluent Contaminants after All Treatments

Parameter	Concentration (mg/l)	
	Average	Maximum
Temperature	61°F	100°F
Flow	1.3 mgd	2.88 mgd
pH (units)	6-9 units	6-9 units
BOD	20	40
COD	50	100
TOC (2.2 x BOD)	44	88
Oil and grease	10	20
TSS	20	35
TDS	TBD	TBD
Chlorides	TBD	TBD
Phenolics	0.1	0.6
Ammonia as N	5	20
Phosphate as P	5	10
Nitrate	30	60
Sulfide	0.04	0.09
Arsenic	0.1	0.25
Barium	1	2
Cadmium	0.15	0.30
Chromium (total)	1	2
Cyanide	0.45	0.9
Fluoride	7	14
Lead	0.25	0.4
Selenium	0.35	0.7
Silver	0.1	0.2
Zinc	1	2
Nickel	0.5	1.6
Iron	2	4
Vanadium	1	5
Copper	1	2
Manganese	2	4
Mercury	0.001	0.002
Cl ₂ residual (total)	1	2
Beryllium	0.005	0.01
Fecal coliform	TBD	(below 400/100 ml)
Boron	TBD	TBD
Alkalinity	TBD	TBD
Thiocyanate	TBD	TBD
Sulfate	TBD	TBD

*BOD, biochemical oxygen demand; COD, chemical oxygen demand; TOC, total organic content; TSS, total suspended solids; TDS, total dissolved solids; mgd, million gallons per day; TBD, to be determined.

Table 9

Organic Compounds in the Projected SRC-I Effluent¹

Chemical compound	Untreated effluent concentration, mg/l	Bio-Unit effluent, ² mg/l	After activated carbon filtration-- 75% reduction, ³ mg/l	After dilution by 7-day, 10-year low flow, mg/l
Methyl-indene	15	2.4-8.4	2.1	13
Tetralin	0.1	0.5 (15%) ⁴	0.01	0.1
Dimethyltetralin	0.5	0.08-0.4	0.10	0.6
Naphthalene	5	0.81 (80%) ⁵	0.20	1.2
Dimethylnaphthalene	0.3-2	0.05-1.12	0.28	1.7
2-Isopropylnaphthalene	0.7	0.11-0.39	0.10	0.6
1-Isopropylnaphthalene	2	0.32-1.12	0.28	1.7
Biphenyl	0.2	0.03-0.11	0.03	0.2
Acenaphthalene	<0.1	0.01-0.06	0.02	0.1
Dimethylbiphenyl	0.2-0.5	0.79-1.1	0.28	1.7
Dibenzofuran	0.6	0.09-0.32	0.08	0.5
Xanthene	0.1	0.01-0.06	0.02	0.1
Dibenzothiophene	1.5	0.24 (60%)	0.01	0.4
Methyldibenzothiophene	<0.1	0.01-0.06	0.02	0.1
Thioxanthene	0.1	0.01-0.06	0.02	0.1
Fluorene	0.3	0.08 (64%) ⁵	0.02	0.1
9-Methylfluorene	0.3	0.05-0.17	0.04	0.2
1-Methylfluorene	0.2	0.04 (76%) ⁵	0.01	0.1
Anthracene/Phenanthrene	1.1	0.26 (70%) ⁴	0.07	0.4
Methylphenanthrene	0.2-0.3	0.05-0.11	0.03	0.2
C ₂ -Anthracene	<0.05	0.01 (62%) ⁵	0.003	0.02
Fluoranthene	0.4	0.06-0.63	0.16	1.0
Bihydropyrene dihydropyrene	<0.05	0.007-0.03 ⁵	0.008	0.1
Pyrene	0.6	0.24 (50%) ⁵	0.06	0.4
Dimethyldibenzothiophene	<0.05	0.007-0.03	0.008	0.1

1. Table taken from EAR, Appendix Q, Table Q.1.
2. A 30 to 80% degradation is assumed unless otherwise noted (from Table Q.1).
3. Taken from W. G. Schlenger, "Coal Gasification Development and Commercialization of the Texaco Coal Gasification Process." Presented at the Sixth International Conference on Coal Gasification, Liquefaction, and Conversion (no date or place).
4. Isolated organisms known to degrade naphthalene and/or phenanthrene (from Table Q.1).
5. Removal efficiencies for a sludge which degrades petrochemicals in Colgate Creek, LA (from Table Q.1).

Table 10

Approximate Enthalpy Balance

Input	MM Btu/hr
Electricity	192
Fuel	914
Heat of reaction	<u>976</u>
Total	2,082
<u>Output</u>	
Products	12
Air coolers	667
Cooling tower	1,054
Heat and surface losses	133
Stack losses	<u>216</u>
Total	2,082

Table 11

Estimated Drift Composition

Components (mg/l)	Average	Maximum
Calcium	190	380
Magnesium	50	100
Sodium	60	120
Manganese	1	2
Phosphorus	1	2
Potassium	0	0
Zinc	13	26
Aluminum	0.5	1
Bicarbonate	144	288
Sulfate	600	1,200
Nitrate	7	14
Chloride	100	200
Ammonia	0	0
Organic phosphate inhibitor	10	10
Silica	0	0
Total dissolved solids	1,100	2,200
Total suspended solids	5	10
pH	7.2	6.5-7.5

Table 12

Summary of Base Case and Alternate Cooling Systems*

Case description	Base	Alternate A	Alternate B	Alternate C
Main cooling system	Wet (evaporative)	Dry indirect	Dry indirect	Wet (evaporative)
Oxygen plant cooling system	DCAC**	Dry indirect	Wet/dry	Wet/dry
Additional cooling	Aerial	No	No	Dry indirect
Water rates (gpm)				
Circulation	78,000	170,000	160,000	131,000
Evaporated	2,100	0	250	2,000
Drift	60	0	10	60
Fits with zero discharge	Yes	Yes	Yes	Yes

*Design load is 1.72 billion Btu/hr in all cases.

**Direct contact after cooler.

Table 13

Chemical Composition of Slag and Flydust
From GKT Gasifier (SRC-I Residue)

Element	Concentration (%)		
	Slag Run #179	Flyash Run #179	Flyash Run #175
Aluminum	14.78	7.16	5.99
Calcium	0.50	0.43	0.40
Iron	11.80	8.89	7.66
Potassium	0.85	0.73	0.63
Magnesium	0.38	0.36	0.30
Sodium	0.34	0.29	0.25
Titanium	0.36	0.32	0.26
Silicon	21.74	18.13	14.91
Sulfur*	0.50	3.74	4.04
Carbon	0.53	22.44	35.71

*The sulfur content in the flydust will be significantly lower from the prototype plant because of different operating conditions.

Table 14

Trace Element Concentrations in Gasifier Coal Feedstock,
Gasifier Slag, and Flydust
(all concentrations in ppm)

Element	Kentucky #9 Coal	Slag Run #179	Flyash Run #179	Flyash Run #175
Arsenic	8.9			
Barium	83.0	383.00	316.00	250.00
Beryllium	1.3	8.7	7.4	6.0
Chromium	21.0	3500.00	890.00	749.00
Copper	8.5	57.00	106.00	84.00
Manganese	42.0	298.00	159.00	138.00
Lead	2.9	4.00	287.00	192.00
Nickel	10.7	1860.00	260.00	213.00
Silver	0.02	2.00	0.7	0.3
Zinc	55.0	33.00	365.00	253.00

Table 15

EPA Extraction Procedure Results for Ash and Flydust
Produced by GKT Gasifier on SRC-I Residue

Element	Slag Run #179	Flyash Run# 179	Flyash Run #179	RCRA* standard
Arsenic, ppb	4	4	5	5,000
Barium, ppb	3.3	73	125	100,000
Cadmium, ppb	0.098	45	27	1,000
Chromium, ppb	0.46	8.5	5.3	5,000
Lead, ppb	1.1	78.5	13.5	5,000
Mercury, ppb	0.022	0.047	0.053	200
Selenium, ppb	5	5	5	1,000
Silver, ppb	0.01	0.01	0.01	5,000
Copper, ppb	0.0013	1.53	0.58	**
Nickel, ppm	0.10	3.71	1.82	**
Zinc, ppm	0.012	7.9	2.1	**
Vanadium, ppm	0.27	1.6	2.0	**

*RCRA standard is based on 100 times the limits set in the Primary Drinking Water Standards.

**Elements not listed in the Interim Primary Drinking Water Standard.

Table 16

Parameters Measured during EP Extractions on SRC-I
Gasifier Slag and Flydust

	Slag Run #179	Flydust Run #179	Flydust Run #175
Initial pH (distilled H ₂ O)	5.2	3.8	4.0
Final pH (24-hour shaking)	5.1	4.2	4.2
Total 0.5 N Acetic acid added (mequiv/g sample)	0.004	0	0
Electrical conductivity (μ mhos/cm)	13.4	369	410

Figure 1
Startup Sequence

1. Pre-startup—auxiliary boiler at 10% capacity
2. Begin startup—operate auxiliary boiler at 100% capacity
3. Startup dissolver, deashing, and gasifying sections—feed at 60% normal rate
4. Gradually raise feed rates to 100% normal rate
5. Start coker and expanded bed hydrocracker
6. Feed coke to coker at 100% normal rate—feed gas to reformer at 60% normal rate, then gradually increase feed to 100% normal rate
7. Start heating calciner by firing desulfurized oil
8. Convert heater fuel from desulfurized oil to gas
9. Normal operating level

Figure 2
Sulfur Balance

Product slate:
 1/3 SRC for coke production
 1/3 SRC for clean fuels production
 1/3 SRC solid

Figure 3
Conceptual Wastewater Treatment System

Figure 4
Zero Wastewater Discharge

Figure 5
Preliminary Water Balance--River Discharge

Figure 6
Preliminary Water Balance--Zero Wastewater Discharge

Figure 7
Alternative Cooling Systems

Figure 8
Typical Ash Pond Dike

Figure 9
Typical Section of Waste Sludge Landfill

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CHARACTERIZING WASTEWATER AND MONITORING ITS TREATMENT

**O. J. Noichl
and D. M. Nicholas***

A project has been set up by ICRC to identify likely contaminants in the wastewater from the SRC-I Demonstration Plant—and to monitor the effectiveness of methods proposed for treating these contaminants.

The work, which began in April, is being done by Air Products and Chemicals (APCI) with the cooperation of several universities. So far there has been progress in two tasks—characterization and physical-chemical pretreatment. Other tasks will include simulation of the wastewater and biological treatment.

Samples are being collected from the Wilsonville and Fort Lewis Pilot Plants and from suppliers of critical processes: GKT, Texaco, Lummus, and Mitsui.

Preliminary analyses have measured the samples received for pH, oil and grease, metals, total organic carbon, chemical oxygen demand, etc.

A tentative finding, based on Pilot Plant samples, is that oil and grease can be readily removed with an American Petroleum Institute mechanical separator. Nonetheless, a more elaborate separation technique for removing dispersed oil, such as DAF (dissolved air flotation), may be recommended to serve units like the expanded-bed hydrocracker in the Demonstration Plant.

Where the Samples Come From

The Sampling points are those identified as containing representative organic compounds, metals, and product oils. Fort Lewis and Wilsonville are providing samples of the overhead flash wastewater streams that comprise most of the sour water expected from the SRC-I Demonstration Plant. Wastewater streams originating from the GKT and Texaco gasification tests, the Mitsui solidification tests, the Kerr-McGee deashing tests, and coal and SRC pile runoff should contain the majority of the metal contaminants. Oil-containing wastewater sources are the Fort Lewis surge reservoir and an overhead flash stream from the Wilsonville pilot plant. Samples received to date include sour water from the K-111 vacuum jet and the V-105 solvent decanter at Wilsonville; surge reservoir wastewater, Sandvik belt cooling water, SRC and coal pile leachate, and SRC-II recycle process water from Fort Lewis; gasification wastewater from GKT; and vapor condensate from APCI's Coal Process Development Unit.** Still to come are SRC-I recycle process water from Fort Lewis;

*Noichl is from Air Products and Chemicals, Inc., and Nicholas from ICRC.

**SRC-I Monthly Technical Progress Report, October 1979, page 35.

gasification wastewater from Texaco; solidification wastewater from Mitsui; and expanded-bed hydrocracker and coker wastewater from Lummus.

Analytical Techniques and Problems

Table 1 presents the data generated to date on sample analysis. The types of analysis used to generate the data are shown in Table 2.

The nature of the samples has created technical challenges. For example, obtaining homogeneous samples from the container was difficult due to the heterogeneity of the material, which generally contained solids, an aqueous layer, and an oil layer. This led to significant variations in such measurements as pH, oil and grease, and total organic carbon (TOC).

The organic fraction of a sample appears to create significant interferences with the ion analysis. Problems have occurred using a Technicon autoanalyzer (coating the optical flow cells with the sample) and specific ion electrodes (sample reaction with the sensing element or by membrane damage).

In addition, TOC measurements decrease 25 to 30% in 15 minutes when a fresh sample is exposed to air. New preparation techniques will be tried to circumvent the suspected problem of volatile loss or lack of a homogeneous sample.

Ammonia analyses of a Wilsonville sample (from the V-105 solvent decanter) have resulted in concentrations that are vastly different when the Technicon autoanalyzer was used (15,000 mg/l $\text{NH}_3\text{-N}$), compared to those measured with the specific ion electrode (150 mg/l $\text{NH}_3\text{-N}$). The autoanalyzer value is similar to measurements obtained by Catalytic during its sampling program at Wilsonville. Discrepancies between the autoanalyzer and ion electrode analysis may be caused by interference from other compounds in the sample and these problems are currently being investigated.

Chemical oxygen demand (COD) analysis has proven difficult on sour water samples; due to interference from high levels of hydrogen sulfide. Of primary importance is the reaction converting silver sulfate to silver sulfide, thereby removing the silver from the COD dichromate oxidation solution. Silver sulfate is added as a catalyst to oxidize straight chain alcohols and acids, which are expected in quantity in these wastewater samples.

Screening Tests for Physical-Chemical Pretreatment

The purpose here is to develop the physical-chemical pretreatment methods required prior to biological treatment to remove toxic metals and oil and grease—although preliminary results indicate low metal concentrations.

Again, preliminary results indicate that any oil present is mostly product oil that can be readily separated from the wastewater by conventional separation equipment. Oil was present in all samples received. Grease, as a floating solid, was not observed. Most of the oil was contained in a film or floating layer; very little was emulsified. The oil content of the total sample was measured by the *Standard Methods* freon extraction test for oil and grease. The total amount of freon-extractable material is reported in Table 3. It was found that oil removal could be readily accomplished. Standard additives were tested for maximum oil removal ability. The amount of oil removed was determined by a weight measurement of the precipitate, as shown in Table 3. As can be seen, all additives operated with the same

efficiency, removing all of the oil and leaving the remaining water with a TOC of 400-500 ppm carbon.

All toxic metals (those classified as priority pollutants) are present in concentrations of less than 10 ppm in the overhead sour water streams and less than about 100 ppm in the GKT gasification wastewater as shown in Table 1. At these concentrations pretreatment for the removal of toxic metals may not be required before the biological treatment basins because they may be removed without adverse effects by the sludge within the biological treatment basins. Any residual trace concentrations of these metals may then be removed by activated carbon treatment which would follow the biotreatment.

Table 1
Wastewater Analytical Results*
(sheet 1 of 3)

A Sample	B pH	C Alkalinity (mg/l as CaCO ₃)	D Oil and grease (mg/l freon extractable)
1. CRDD Pilot Reactor XCL23	9.5	26,260	-
2. V-105 4/17/80 #55804	-	60,450	-
3. V-105 4/18/80 #55834	8.8	59,460	412
4. V-105 4/19/80 #55870	-	52,520	-
5. V-105 4/20/80 #55913	8.7	59,460	1,300
6. V-105 4/21/80 #55933	-	58,970	-
7. V-105 5/6/80	8.5	-	480,000
8. V-105 5/8/80	8.55	-	1,640
9. K-111 4/17/80 #55803	8.6	54.6	1,110
10. K-111 4/18/80 #55835	-	-	-
11. K-111 4/19/80 #55869	8.6	63.0	920
12. K-111 4/20/80 #55914	-	50.4	-
13. K-111 4/21/80 #55933	7.35	50.4	848
14. K-111 5/5/80	8.4	-	360
15. K-111 5/7/80	7.6	-	757
16. Fort Lewis recycle process water, SRC-II	9.45	145,700	705
17. Fort Lewis Surge Reservoir, SRC-II	8.25	147	1,530
18. GKT Gasifier water	3.0	-	351
19. Sandvik cooling water	7.15	39.9	378

*Alkalinity (Column C) determined by titration to pH 4. Freon extractables (Column D) as defined by Method A, Standard Methods (see reference in Table 3).

Table 1
Wastewater Analytical Results
(sheet 2 of 3)

	E			F	G	H	I	J
	Major (>1%)	Minor (0.01-1%)	Trace (<0.01%)	TSS (mg/l)	VSS (mg/l)	NH ₃ -N (mg/l)	Total COD (mg C/l)	Filtered COD (mg C/l)
1. Sn*	Fe,B,Na	Al,Mg,Cu, Ni,Ca	113	172	-	-	-	
2. -	-	-	-	-				
3. -	Cu,Mg,B, Na	Fe,Si,Ag, Al,Ni,Ca	27.6	25.4				
4. -	-	-	-	-				
5. -	Cu,Mg,B, Na	Fe,Si,Ag, Al,Ni,Ca	48.6	27.6				
6. -	-	-						
7. -	-	-						
8. -	-	-						
9. Fe,Ca	Si,Mg,Cu, Na,Al,Mn	Ni,Cr,Ti	82.6	17.6	12.2	2,940	2,610	
10. -	-	-	-	-	8.1	-	-	
11. -	Fe,Si,Mg, Na	Cu,Ca,Al	28.0	16.8	11.3	3,200	2,940	
12. -	-	-	-	-	11.3	-	-	
13. -	-	-	36.0	12.6	9.7	2,750	2,450	
14. -	-	-						
15. -	-	-						
16. -	Mg,B,Fe, Si,Na	Cu,Ag,Cr, Ni,Ca,Al	33.0	35.6				
17. Fe,Si,Na, Ca,K	Mg,V,B, Ca,Al	Ag,Ti,Ni, Mn,Pb,Ca	47.4	36.8	342			
18. Fe,Si,K, Na,Al,Ca	Mg,Mo,Pb, Cr,V,Ti,Mn	Ga,Sn,Cu, Sr,Ni						
19. -	-	-						

*Artifact from storage container.

Table 1
Wastewater Analytical Results
(sheet 3 of 3)

	K*	L	M	
			-----Observed HPLC fractions----- Hexane extractables	Aqueous phase EtOH extractables
1.	350	-	1-, 2-, 3-, 4- member rings	
2.	350	17,100		
3.	-	15,500		
4.	350	15,150	1- and 2- member rings	-10 polar compounds
5.		16,100		
6.		14,900		
7.				
8.				
9.	-			
10.	200		1-, 2-, 3- member rings	polar compounds acids and phenols
11.	-			
12.	200			
13.	200			
14.				
15.				
16.		30,200		
17.				
18.				
19.				

*GC (Column K): 30m SE54 capillary column gas chromatograph.

Table 2
Techniques of Wastewater Sample Analysis

Instrument	Location	Data
<u>Metals</u>		
Emission spectrometry	APCI	Identification of metals present; semiquantitative
Atomic absorption	APCI	Quantitative for specific metals
Neutron activation	TBD	Quantitative for almost all elements
<u>Organics</u>		
Capillary GC	APCI	Preliminary profiles. Identification of 25% of the components by retention indices.
Capillary GC-MS	APCI	Further identification of specific components.
HPLC	Seton Hall U.	Preliminary profiles; fractions separated and identified by ring size and class. Approximate number of components in each fraction determined.
GC-MS	Brigham Young U.	Identification of components from HPLC fractions. Some quantification of components.
HRMS	U. of Nebraska	Further identification of components from fractions. Further quantification.
GPC-GC-MS	Texas A&M	GPC separation of fractions followed by GC-MS identification.
<u>Traditional parameters</u>		
Specific ion electrodes	APCI	Ammonia, cyanide, hardness, nitrate and nitrite, sulfide.
Specific probes	APCI	pH
Wet chemistry	APCI	Alkalinity, biological oxygen demand (BOD), chemical oxygen demand (COD), oil and grease, phenolics, thiocyanate, total inorganic phosphate, total Kjeldahl nitrogen
TOC analyzer	APCI	Total organic carbon (TOC)
Turbidimeter	APCI	Turbidity
Weight measurements	APCI	Total dissolved solids, total suspended solids, volatile suspended solids

Neutron activation analysis insures that no elements are overlooked in the screening procedure.

Gas chromatography and high pressure liquid chromatography are to be done concurrently.

Wet chemistry and weight measurements are to be done as prescribed in Standard Methods for the Examination of Water and Wastewater, 14th edition (1976), American Public Health Association, Washington, DC.

Table 3
Wastewater Pretreatment Results
(Oil and Grease Removal)

Sample	Date	Method	Total sample oil and grease (mg/L)	Total sample TOC (mg/L)	Extracted water TOC (mg/L)
Wilsonville					
K-111	4-19	Freon extraction	920	>1000	425
	4-21		850	>1000	334
	5-5		360	>1000	266
	5-7		760	>1000	427
Fort Lewis					
Surge	(prefiltered)	Acidification pH 2.	500	>1000	500
		Alum (sat)	500	>1000	-
		FeCl ₃ (sat)	500	>1000	400
		CaO (sat)	500	>1000	500
		Ultracentrifuge	500	>1000	400

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