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SRC-I TECHNICAL REPORT, JULY–DECEMBER 1983

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Work Performed Under Contract No. AC05-78OR03054

International Coal Refining Company
Allentown, Pennsylvania

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SRC-I TECHNICAL REPORT

JULY–DECEMBER 1983

Prepared By
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Allentown, Pennsylvania 18001

for the
UNITED STATES DEPARTMENT OF ENERGY
Office of Solvent-Refined Coal Products
under Contract DE-AC05-78OR03054

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THE SRC-I PROJECT

To help reduce America's dependence upon imported petroleum, the U.S. Department of Energy (DOE) initiated several synthetic fuel projects to demonstrate the technical, environmental, and economic feasibility of converting high-sulfur, bituminous coals into environmentally acceptable solid and liquid fuels.

One of these projects involves the design (Phase I), construction (Phase II), and operation (Phase III) of a demonstration-sized solvent-refined coal (SRC-I) facility near Newman, in Daviess County, Kentucky. The demonstration plant, to be located on a 1,500-acre site on the Green River, will convert 6,000 tons per day (tpd) of coal into the equivalent of 20,000 barrels (bbl) per day of petroleum in the form of clean solid and liquid fuels.

HISTORICAL BACKGROUND

The proposed demonstration plant is the culmination of nearly a decade of work in synthetic fuels development by Wheelabrator-Frye Inc. and Air Products and Chemicals, Inc. (APCI). In 1973, Catalytic, Inc., an APCI subsidiary, engineered and built a pilot plant in Wilsonville, Alabama to convert approximately 6 tons of high-sulfur coal per day into clean-burning SRC solid, one of the products of direct coal liquefaction. During the same year, Wheelabrator-Frye, through its Rust Engineering Company subsidiary, began building a larger pilot plant in Ft. Lewis, Washington to convert 50 tons of high-sulfur coal per day into solvent-refined coal products. These two plants have logged over 10 operating years, and have provided the data base for the demonstration plant design.

In 1977, Wheelabrator-Frye and the Kentucky Center for Energy Research began working in partnership to design a solvent-refined coal facility that would convert 2,000 tons per day of high-sulfur coal into energy products. In 1980, Wheelabrator-Frye and Air Products and

Chemicals pooled their knowledge and formed a Joint Venture which later became the International Coal Refining Company (ICRC) to demonstrate this promising, clean-coal technology on a larger scale.

During Phase 0, ICRC prepared the conceptual design, preliminary cost estimates, marketing assessments, economic evaluation, and environmental appraisal. Process options were evaluated, critical technology areas requiring additional data were identified, and the economics were assessed for both a 30,000-tpd commercial plant and a commercial plant of the same size expanded from a 6,000-tpd demonstration plant. The latter option was chosen.

Phase 0 work was completed in July 1979, and in October 1979, the DOE authorized Phase I, the detailed engineering of the project. On August 7, 1980, a cost-sharing agreement was signed between ICRC and DOE, covering the remainder of the demonstration plant program through start-up and operation of the facility. Under the terms of the agreement, ICRC will invest $90 million in the project, the Commonwealth of Kentucky will invest $30 million, and DOE will fund the balance. The contract states that ICRC will eventually own the coal refinery after buying out the Federal and state governments' interest.

Under the cost-sharing agreement, ICRC became the prime contractor on the project. As an important subcontractor to ICRC, Southern Company Services, Inc., the principal contractor to DOE for Phase 0, will continue its pioneering role in the development of the SRC technology by providing broad technical reviews and product use studies.

Aluminum Company of America (ALCOA) is also negotiating with representatives of ICRC and the government to share the cost of building and operating the facility. Because ALCOA has experience in the technology and products of direct coal liquefaction, its participation will strengthen the pool of knowledge and expertise that already exists within ICRC.

PROJECT MANAGEMENT

The engineering/construction approach to the project is unique. Overall project management is the responsibility of ICRC. Each of six contractors provide the engineering and major equipment procurement for a defined area of the plant as follows:

1)	Air Products and Chemicals, Inc. Allentown, Pa.	oxygen plant and and hydrogen purification equipment
2)	Catalytic, Inc. Philadelphia, Pa.	SRC area
3)	C-E Lummus Co. Bloomfield, N.J.	coking, calcining, and LC-Finer
4)	The Ralph M. Parsons Co. Pasadena, Calif.	gasification, acid gas treatment, compression, and sulfur recovery
5)	The Rust Engineering Co. Birmingham, Ala.	coal preparation, utilities, and off-sites
6)	Johnson Controls, Inc. Philadelphia, Pa.	instruments and controls

As the construction manager/constructor, Stone & Webster Engineering Corporation will be responsible for procuring standard equipment and bulk materials, for establishing and managing all construction subcontractors, and for direct-line management of all force account labor.

A 30-month period for start-up and operation in a demonstration mode will follow mechanical completion of the plant. Provisions are available to extend the demonstration period by as much as 3 years. Following this period, ICRC could expand the plant as much fivefold to produce a nominal 100,000 bbl/day of products from 30,000 tpd of coal.

DESIGN IMPACTS OF OPERATING SRC-I GAS SYSTEMS AREA EQUIPMENT UNDER MAXIMUM AND MINIMUM FLOW CONDITIONS

T. C. Li*

The Baseline Design (ICRC, 1982) for the gas systems area of the SRC-I Demonstration Plant was based only on normal flow conditions, specified in ICRC's Design Basis Memorandum (ICRC, 1981). However, following submission of the Baseline Design to the Department of Energy in 1982, all equipment in the gas systems area was re-evaluated to assess the design impact of operation at maximum and minimum flow conditions. The impact on the operating philosopy of each unit was also investigated. As the result of these evaluations, several additional tie lines were added for recycle, and some equipment was re-sized to accommodate the maximum and minimum flows.

This article documents those cases that have been studied for Gas Systems minimum/maximum operations, and the design or operational changes that result. It is recognized that there may be other cases which should be considered prior to completion of the design. However, it is not believed that any significant cost increases will result from consideration of these other cases.

The basis for evaluating the maximum and minimum flow rates and descriptions of controlling factors for both cases are presented in this report for each unit in the gas systems area.

BACKGROUND DESCRIPTION

The SRC-I Demonstration Plant is divided into five major areas, each of which was designed by a different subcontractor:

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*International Coal Refining Co. (ICRC).

° Area 12:	SRC process
° Area 13:	Expanded-bed hydrocracker (EBH) and coker/calciner
° Area 14:	Cryogenic systems
° Area 15:	Gas systems
° Areas 11, 16, and 17	Utilities and off sites

Figure 1 shows the major process flow scheme for the overall demonstration plant and identifies each subcontractor area.

The only area discussed in this report is the gas systems area (15), which was subcontracted to the Ralph M. Parsons Company, of Pasadena, California. This area is divided into four subareas, each consisting of several process units:

1. Gasification area
   - Dust preparation unit (including nitrogen recycle/solvent recovery)
   - Gesellschaft für Kohle Technologie (GKT) coal gasification unit
   - Wash water treatment unit
   - Raw syngas compression unit
   - Shift unit
   - Methanation unit

2. Gas treating area
   - Selexol unit
   - DEA unit
   - Ammonia/sulfide water stripping unit
   - Liquefied petroleum gas (LPG) recovery unit

3. Compression area
   - Hydrogen compression unit

4. Sulfur recovery area
    - Claus unit
    - Beavon sulfur removal unit
    - Caustic storage and distribution

The dust preparation unit blends supplemental coal from the coal preparation area and KMAC from the SRC deashing area to use as feed for the coal gasification unit. The coal gasification unit generates makeup hydrogen from the KMAC/coal mixture for the SRC process area and the expanded-bed hydrocracker and naphtha hydrotreater. The wash water treatment unit removes solid material (fly ash and slag) from the raw water used in the coal gasification unit for various quenching, cooling, and washing steps.

The raw syngas compression unit boosts the pressure of the raw syngas from the gasification unit, and a shift unit converts most of the carbon monoxide in the raw syngas to hydrogen. The Selexol process removes acid gases from the makeup hydrogen gas. Part of the Selexol-treated makeup hydrogen is further processed in the methanation unit to remove CO and CO_2 before it is charged to the expanded-bed hydrocracker and the naphtha hydrotreater.

The DEA process removes acid gases from the high-pressure raw recycle hydrogen-rich gas (generated in the SRC process area and in the expanded-bed hydrocracker area) and treats various low-pressure raw fuel-gas streams generated in the plant. The hydrogen compression unit compresses part of the treated makeup hydrogen from the Selexol unit, part of the treated hydrogen-rich gas from the DEA unit, and the recycle hydrogen stream from the hydrogen purification unit for use in the SRC process area.

Ammonia and acid gases contained in sour water generated throughout the plant are removed in the ammonia/sulfide water stripping unit. Bulk sulfur is further removed from acid gases in a three-stage, straight-through ammonia-burning Claus unit. Claus plant tail gas is then treated using the Beavon sulfur removal process, so that it can be safely vented to the atmosphere.

Finally, LPG is recovered from the low-pressure fuel reject stream of the hydrogen purification unit, supplementing start-up and/or emergency fuel requirements as well as ensuring a reliable pilot gas system.

EVALUATION OF MAXIMUM AND MINIMUM FLOW CASES

Gasification Area

The gasification area unit design is based on KMAC/coal mixed feed to produce 77.5 MM scfd of hydrogen plus carbon monoxide. The dust preparation unit and the gasification unit are designed to be capable of operating at 100% KMAC feed, or 100% coal feed, or at any blend of KMAC and coal dust. The coal-feed rate decreases as KMAC feed increases. The maximum flow case would occur when the system is handling a mixture of high-ash KMAC and high-ash coal (see Table 2) at the rated capacity. The high-ash coal composition was defined after surveying various coal supplies near the SRC-I Demonstration Plant site. Compositions are listed in Table 2; H, O, and N were normalized. High-ash KMAC is produced when the first-stage recovery unit (in the SRC process area) is processing high-ash coal. High-ash KMAC compositions are also shown in Table 2. The minimum flow case would occur when one gasifier is operated at 70% design load during plant start-up. (Three gasifiers are running under normal conditions.)

Dust Preparation Unit (DPU)

Process Description. Figure 2 diagrams process flow for the DPU and associated solvent recovery and nitrogen recycle. KMAC enters the DPU battery limits and is routed to the KMAC receiving bunker. The KMAC is humidified to obtain the proper moisture content for use as feed to the gasification reactors.

Supplemental coal is conveyed from the coal preparation area to the coal dust bunker using nitrogen. The coal is then properly blended with the humidified KMAC and the combined dust is routed to the finished dust bunker. During plant start-up or when KMAC is unavailable, the coal is fed directly from the coal dust bunker to the finished dust bunker. The coal/KMAC dust mixture or coal dust is conveyed via pneumatic pumps in N_2 to the service bunkers in the gasification unit.

The nitrogen used for conveying and bunker fluidization is combined after use, filtered to remove dust, and routed to the solvent recovery/nitrogen recycle section of the dust preparation unit. The nitrogen is compressed, cooled to recover water and solvent condensate, and recycled for re-use in the dust preparation unit.

Controlling Factors for the Maximum and Minimum Flow Cases. The DPU consists of two parallel trains, each operating at 50%. Feed streams per train under normal operating conditions using both KMAC and coal dust are listed in Table 1.

The KMAC- and coal-handling facilities are sized to handle 155,380 lb/hr (77,690 lb/hr per train) of dry KMAC and 142,684 lb/hr (71,342 lb/hr per train) of coal, respectively, for 100% KMAC feed and 100% coal feed based on the design criteria established early in the project design phase. Coal feed rates higher than that required for the normal mixed feed case (Table 1) are needed to operate the SRC-I Demonstration Plant during start-up, when KMAC is not available. However, the coal feed rate at 142,684 lb/hr to produce the rated hydrogen (i.e., 77.5 MM scfd of H_2 + CO) would probably not occur. For the KMAC-handling facilities, the available maximum KMAC at 144,300 lb/hr (shown in Table 2) was defined later in the design phase. It appears that both the KMAC- and coal-handling facilities are oversized for the maximum case. However, the equipment has not been resized due to time limitations of the project schedule and the uncertainties of the dissolver's operation in the SRC unit.

The nitrogen recycle/solvent recovery (NRSR) unit has been designed for the normal KMAC/coal mixed feed case with no margin. Solvent in the KMAC feed is assumed to be 1 wt % maximum. Solvent is assumed to be stripped out completely in the DPU by nitrogen and is recovered in the NRSR unit. These assumptions lead us to believe that the NRSR unit design is very conservative for the normal mixed feed case, and it should be able to handle the maximum KMAC case, which represents only a 6% increase in the KMAC feed rate. The nitrogen flow will remain relatively constant with a small change in KMAC feed. Under the minimum flow case, one of the two DPU trains should be operated at 46% of its design load. This unit can also be operated in a semicontinuous mode.

Equipment Design Factors. The design factor for each piece of equipment is shown in Table 3.

Coal Gasification Unit

Process Description. See Figure 3 for the process flow diagram. The coal dust (KMAC, coal dust, or KMAC/coal mixture) is conveyed by nitrogen from the DPU to the service bunker for the gasifier burner. The dust is then passed to the feed bunkers and fed via a variable-speed screw feeder to one of the four burners of each four-headed GKT gasifier.

In the gasifier, solids are partially oxidized in the presence of oxygen and steam, and the slag exits from the bottom of the gasifier. The raw syngas generated in the gasifier is quenched and then cooled in a waste heat boiler (WHB). The syngas leaving the WHB passes through a series of gas-washing, -cleaning, and -cooling steps. Particulate-laden water from these steps is sent to the wash water treatment unit. Finally, the raw syngas is routed to the common raw gas holder (in the raw syngas compression unit) through the raw gas blower.

Controlling Factors for Maximum and Minimum Flow Cases. The coal gasification unit is designed as three 33-1/3% gasification trains, providing 77.5 MM scfd of hydrogen plus carbon monoxide. Feed streams in normal operation using both KMAC and coal dust are shown in Table 4. The unit is designed to operate at its rated capacity with 100% KMAC feed, 100% coal feed, or with any blend of KMAC and coal dust.

The maximum flow case would occur when the gasification unit is operated under one of the following feed conditions: (a) high-ash coal feed; (b) high-ash KMAC feed; (c) mixture of high-ash KMAC and high-ash coal feeds. Each piece of equipment in the gasification trains was designed to accommodate these three conditions; each train can be operated under any one of the conditions during start-up and normal operation.

Operating the unit with a mixture of high-ash KMAC and high-ash coal provides a maximum flow for the downstream raw syngas compression, shift, and Selexol units.

Minimum flow would occur when one gasifier is operated at 70% design load during start-up. The gasification unit consists of three 33-1/3% trains and each train can be operated at 70% of its normal design load, no design modification is required to accommodate the minimum flow case.

Equipment Design Factors. Table 5 lists detailed design factors for all pieces of equipment, which were assessed individually to accommodate the three maximum flow conditions described.

Wash Water Treatment Unit (WWT)

Process Description (See Figure 4). Raw wash water from the coal gasification unit and recycle water from the settling pond (in the utility and off-sites area) enter a common enclosed clarifier (settling basin). Vapors released from the wash water are drawn off by fans and sent to the plant boiler system (for use as supplemental combustion air). Solids are separated from the wash water in the clarifier by gravity, and a mechanical scraper continuously rakes the solids settled on the bottom to the slurry pump receivers (funnel tops), from which the slurry is continuously pumped to the settling pond by ash slurry pumps.

The clarified wash water is passed over a weir at the end of the clarifier into a discharge system, from which it flows by gravity to the wash water sump. Wash water is pumped through an indirect plate-type cooler and returned to the coal gasification unit. The coolers are periodically back-flushed with clarified wash water and nitrogen to minimize fouling due to solids deposition.

Controlling Factors for Maximum and Minimum Cases. The WWT unit design consists of two parallel 50% settling basins and multiple heat exchangers and pumps. The system is designed to remove ash and unconverted carbon solid constituents in the wash water feed to a level of 100 ppmw (normal) and 150 ppmw (maximum), and to maintain the overall concentration of chloride in the wash water at a level of 5,070 mg/L (normal) and 6,491 mg/L (maximum). Maximum flow will occur when the coal gasification unit is gasifying a mixture of high-ash KMAC and high-ash coal derived from feed coal with a high chloride content.

Minimum flow conditions will exist when one gasifier is operating during plant start-up. (Three gasifiers are operating under normal conditions.) No problems are anticipated since the wastewater unit design is based on a multiple-unit philosophy.

Equipment Design Factors. The design factor for each piece of equipment was assessed individually to accommodate the maximum flow case. Details are listed in Table 6.

Raw Syngas Compression Unit

Process Description. Raw syngas from the raw gas holder is sent to a single electrostatic precipitator for final particulate removal. The raw syngas is then compressed from 14.5 to 825 psia using a single five-stage centrifugal machine. A catalytic reactor is installed between the first and second stages of the compressor to remove traces of NO_x, O_2, and SO_2 contained in the raw syngas. An aftercooler and condensate knockout pot are provided after each stage of compression except the last. Condensate is routed to the ammonia/sulfide stripping unit. The hot raw syngas leaving the final stage of compression is sent to the shift unit. The process flow diagram is shown in Figure 5.

Controlling Factors for Maximum and Minimum Flow Cases. This unit is based on a single train. The centrifugal compressor is driven by a steam turbine. Maximum flow will occur when the gasifier is fed with a mixture of high-ash KMAC and high-ash coal to provide 77.5 MM scfd of H_2-plus-CO gas. During such conditions, the raw syngas flow rate will be 1.5% higher and the gas will be 1.8% heavier than normal.

Minimum flow will occur during plant start-up, when one gasifier is operated at 70% load. The raw syngas compression unit will be operated on a recycle mode, because of the flow limitation (about 75% of normal flow) of the centrifugal compressor.

Impacts for the Maximum and Minimum Flow Cases. The equipment design based on normal flow rate was assessed for compatibility with maximum and minimum flow conditions. No modification of any equipment is required to accommodate the maximum flow case since the flow increase is small. Also, no modifications are required for the minimum flow case, since a recycle mode for handling the start-up case is implemented for this unit.

Shift Unit

Process Description (See Figure 6). Raw synthesis gas from the raw syngas compression unit is combined with superheated steam and heated before entering the first of three shift converter stages. The superheated steam is generated by mixing superheated steam imported from the boiler section of the utilities area and saturated steam produced in the shift and methanation units. In a series of steps, the gas is passed over a sulfur-resistant catalyst, whereupon carbon monoxide and steam react to generate hydrogen and carbon dioxide. Heat generated by the exothermic reaction is used to preheat the feed gas and also to generate steam.

Effluent from the third shift converter, after steam generation, boiler feedwater and CO_2 waste gas stream (from the Selexol unit) preheating, and final cooling, is sent to the Selexol unit. Condensate separated from the gas splits into two streams, one going to the GKT gasifier for hot raw gas quenching and the other to the ammonia/sulfide water stripping unit.

Controlling Factors for Maximum and Minimum Flow Cases. The shift unit is designed as one 100% train. To minimize the need for oversizing equipment, the total quantity of contained hydrogen plus carbon monoxide has been established at 77.5 MM scfd for both normal and maximum flows. Therefore, the feed rate to the shift unit increases only because of changes in feed gas composition.

The maximum flow case will occur when the gasification unit is gasifying the mixture of high-ash KMAC and high-ash coal. Minimum flow will occur during start up of a single gasifier. However, to meet the 50% turndown limitation in the Selexol unit, the system including the raw syngas compression unit (RSGC), the shift unit, and the Selexol unit will be recycling part of the treated hydrogen and flash gases from the Selexol unit to the suction of the RSGC unit. Therefore, the minimum flow case for the shift unit will also be the 50% turndown case.

Equipment Design Factors. The design of each piece of equipment was assessed individually to accommodate the maximum and normal flow cases. For the maximum case, the CO_2 vent-gas stream flow rate for the shell side of E-15263 would increase by about 5%. However, the CO_2 vent gas can be bypassed around E-15263, and the CO_2 gas vent would not exceed the emission limit. Therefore, the size of E-15263 has not been changed. No equipment modification is required to accommodate the minimum flow case. The design factors are listed in Table 7.

Methanation Unit

Process Description (Figure 7). The sweetened hydrogen gas from the Selexol unit, containing approximately 3% CO, 0.2% CO_2, and 6 ppmv sulfur compounds, is preheated to 485°F and fed to a CoMo reactor bed for COS conversion. The gas is then passed through a ZnO bed for trace sulfur compound removal. The desulfurized hydrogen gas mixture is then methanated in the methanator. The effluent from the methanator is used to generate high-pressure steam and preheat the feed gas. The methanated gas is then cooled to 100°F and sent to the EBH area and the naphtha hydrotreater.

Description of Controlling Factors for Maximum and Minimum Flow Cases. The methanation unit is designed for one 100% train and is capable of being operated at turndown to 20% of its design capacity. No maximum flow case is anticipated for this unit, since the upstream Selexol unit is designed to provide a fixed amount of hydrogen plus carbon monoxide. The minimum flow case will occur when the SRC unit is operated at 50% turndown condition and the naphtha hydrotreater is operated at 33% rate. The demand of H_2 from the methanator is about 6% of normal operating flow. Under this condition, the methanation unit can be operated at about 20% of its design capacity with no equipment modification, and the excess H_2 will be sent for fuel. Therefore, no modification of equipment design is required to accommodate this minimum flow case.

Gas Treating Area

Selexol Unit

Process Description. The Selexol unit treats the high-pressure shifted syngas from the shift unit. As shown in Figure 8, the shifted syngas first passes through an H_2S absorber, in which CO_2-saturated Selexol solvent selectively removes H_2S. The rich solvent is flashed twice before it enters the H_2S stripper. The vapors generated in the flashing steps are compressed and recycled to the H_2S absorber.

Acid gases are stripped by steam in the H_2S stripper and sent to the sulfur recovery area for sulfur recovery. Cold, lean solvent removes CO_2 from the desulfurized gas in the CO_2 absorber. The rich solvent is flashed twice before entering the CO_2 stripper; the high-pressure flash gas is recycled to the H_2S absorber.

CO_2 is stripped from the rich solvent with N_2 in the CO_2 stripper. The gas mixture leaving the CO_2 stripper is combined with the CO_2-rich low-pressure flash vapors and heated to 280°F (in the shift unit) prior to being vented. The sweetened hydrogen gas leaving the top of the CO_2 absorber is then split. Part is routed to the methanation unit and the rest goes to the hydrogen compression unit.

Controlling Factors for Maximum and Minimum Flow Cases. The Selexol unit consists of one 100% operating train and is capable of being operated at turndown to 50% of its design capacity.

Maximum flow will occur when the upstream gasification unit is gasifying a mixture of high-ash KMAC and high-ash coal to produce syngas containing 77.5 MM scfd of hydrogen plus carbon monoxide. The shifted syngas fed to the Selexol unit increases by about 5% over the normal case, and the H_2S and CO_2 contents in the feed increase by 25 and 5%, respectively.

Minimum flow will occur during start-up of a single gasifier. The raw syngas generated from the GKT area under this condition represents about 33% of its design capacity. To accommodate this minimum flow case, the flash gas from the H_2S recycle compressor and part of the treated hydrogen gas must be recycled through the raw syngas compression and shift units in order to attain enough gas flow for good vapor/liquid contact in the H_2S and CO_2 absorber towers. To maintain the raw syngas compressor suction molecular weight close to that of the normal case, CO_2 flash gas from the CO_2 absorption loop is also recycled to the RSGC. A block flow diagram showing recycle loops from the Selexol unit to the RSGC and shift units for the minimum flow case is shown in Figure 9. Table 8 lists the material balance for the start-up case.

Equipment Design Factors. In order to evaluate the impact of operating the Selexol unit with maximum feed gas, a computer simulation was carried out to obtain the overall heat-and-material balance for the assessment of individual equipment. Results indicate that the compressor and heat exchangers handling the flashed gas recycle loop must be increased by about 10%. To eliminate these increases in equipment sizes and their costs, the operating pressures of the flash vessels V-15401, V-15402, and V-15407 can be increased from 250, 50, and 250 psia to 262, 56, and 262 psia, respectively, to reduce the flash gases to the same quantities as those for the design case. The current vessels and compressor designs allow these changes in process operating conditions.

A new heat-and-material balance based on the above process conditions for the maximum flow case was used to assess individual equipment. The assessment can be summarized as follows:

- Compressors: All compressor selections for the design case are suitable for both maximum and minimum cases.
- Heat Exchangers: Table 9 compares heat duties and flow rates for the design and maximum cases. A detailed analysis indicated that all heat exchangers rated for the design case are adequate for the maximum and minimum cases.
- Pumps: The capacities for all pumps in the H_2S and CO_2 absorption loops must be increased by 7.6 and 2.4%, respectively, for the maximum case. According to Mechanical Design Criteria specifications, all pumps when installed with a new impeller size should be able to deliver a 5~10% capacity increase in head at rated conditions. All pumps rated for the design case are adequate for the maximum case if new impellers

are used. All pumps will also be operated satisfactorily at 50% turndown under a recycle mode.

- Hydraulic Turbines: All hydraulic turbines rated for the design case are adequate for both maximum and minimum cases.
- Towers: All towers sized for the design case are adequate for the maximum case, and the minimum case under a recycle mode.
- Vessels, Tanks, and Filters: All vessels, tanks, and filters sized for the design case are adequate for both the maximum and minimum cases.
- Packaged Refrigeration Systems: The refrigeration requirement for the maximum case is about 2% higher than that for the design case, a small increase. The recommended packaged refrigeration system has sufficient capacity to meet the design requirements for the maximum case, and will also operate satisfactorily for the minimum case.
- CO_2 Vent Stack: The CO_2 vent stack sized for the design case is adequate for both the maximum and minimum cases.

The equipment design factors for the design case are listed in Table 10.

DEA Unit

Process Description. The DEA process is designed to treat high-pressure (HP) raw hydrogen-rich gas streams and low-pressure (LP) raw fuel gas streams in HP and LP absorbers, respectively. As shown in Figure 10, the HP raw H_2-rich gas stream is a combination of two independent streams, one originating from the SRC process area and the other from the EBH area. The LP raw fuel gas streams originate from the SRC, EBH, and coker/calciner areas. Sweetened HP H_2-rich gas is split: part is bypassed directly to the hydrogen compression unit and the rest is routed to the hydrogen purification unit (HPU).

Both the HP raw H_2-rich gas and the LP raw fuel gas are scrubbed with water for NH_3 removal. The HP raw H_2-rich gas and the LP raw fuel gas then flow to the HP and LP absorbers, respectively, where acid gases are chemically removed by DEA solution. The HP gas leaving the top of the HP absorber is washed with caustic solution for final H_2S and CO_2 removal. The HP rich solution leaving the bottom of the HP absorber is flashed and combined with the LP rich solution. The combined solution, containing acid gas removed from both the HP and LP gas streams, is sent to the DEA regenerator, where acid gases are stripped from the solution by steam. The overhead acid gases are rerouted to the downstream Claus unit for sulfur recovery.

Controlling Factors for Maximum and Minimum Flow Cases. The DEA unit is designed for one 100% operating train and is capable of being operated at turndown to 50% of its design capacity.

Maximum flow will occur when the upstream SRC unit is processing high-oxygen and high-sulfur coal at high severity conditions. The HP and LP feed gas flow rates will increase by 4.2 and 17.8%, respectively. The acid gases (CO_2 and H_2S) increase by 21.5 and 47.8% for HP and LP feeds, respectively, but the actual quantity is small. For processing coal which contains sulfur and oxygen higher than the maximum allowable values, blending this coal will be necessary to reduce the maximum flows.

The minimum flow case will occur when the SRC process area is operated at 50% turndown and both the EBH and coker/calciner are not operating. The feeds decrease to 47 and 12% of the normal design flow case for HP and LP feeds, respectively.

Equipment Design Factors. The process equipment requirements for both maximum and minimum cases were compared with those for normal design:

- Heat Exchangers: All heat exchangers rated for the design case will meet the requirements for the minimum case, but increased surface areas are required for all heat exchangers for the maximum case (see Table 11).
- Pumps: All pumps will operate satisfactorily under minimum flow conditions, but an increase in capacity of about 10% will be required for all the pumps, except P-15306, P-15311, P-15314, and P-15319, for the maximum case. To keep pump equipment cost increases to a minimum, installing new impel-

lers can be implemented. Running the spare pumps for P-15308, P-15309, and P-15313 can also be implemented to cope with flow.

- Hydraulic Turbines: The flow turndown from the design case to the minimum case is about 35%. The circulation rate of the DEA solution will be increased to 40% of its rated design capacity to meet the requirement of the maximum turndown for a hydraulic turbine. For the maximum case, the hydraulic turbine would be operated at its design capacity and the remainder of the flow would bypass the turbines.
- Tanks: All tanks rated for the design case are suitable for both maximum and minimum cases.
- Towers: For the maximum case, the diameter of the DEA regenerator (T-15306) must be increased from 5 ft 6 in. for the design case to 6 ft 0 in. For the minimum case, all towers except the low-pressure DEA absorber (T-15305) and the low-pressure NH_3 wash column (T-15302) have turndown capability to operate satisfactorily. About 120,000 scfh of high-pressure treated gas will be let down from the sweet gas knockout drum (V-15305) to the feed line of the T-15302 to achieve approximately 30% of flood for column operation.
- Vessels: All vessels except V-15308 rated for the design case are adequate for both the maximum and minimum cases. The size of V-15308 is increased from 4 ft i.d. x 15 ft TTL to 4 ft 6 in. i.d. x 17 ft 6 in. TTL to cope with the maximum case.
- Filters, Mixers, and Packaged Units: The filters, mixers, and packaged units will require no changes in either the maximum or minimum cases.

The equipment design factors for the DEA unit are listed in Table 12.

Ammonia/Sulfide Water Stripping (ASWS) Unit

Process Description. Sour water streams originate from the SRC process area, the EBH area, the coker/calciner area, the raw syngas compression unit, the DEA unit, and the sulfur recovery unit. As shown in Figure 11, the combined sour water streams are sent to a flash drum, where most of the dissolver light hydrocarbons and some CO_2, H_2S, and NH_3 are flashed off and combined with the overhead gases from the stripper. Ammonia, H_2S, and CO_2 are stripped from water in the ASWS by steam coming from the ammonia stripper. Water leaving the bottom of the ASWS is mixed with lime slurry in the settling tank to free the fixed NH_3. The free NH_3 is then stripped from the water stream produced from the reboiler. The water leaving the bottom of the stripper is cooled and sent to the wastewater treating area and the stripped gases from the stripper overhead are sent to the Claus unit.

Controlling Factors for Maximum and Minimum Cases. The ASWS unit is designed for one 100% operating train and is capable of being operated at turndown to 20% of design capacity.

The maximum flow case will occur when the SRC area is processed with coal containing maximum nitrogen and chloride, and when other units are operated under maximum flow conditions. For this case, the total sour water feed increases by 24.8%. In the contractual Design Baseline, the ASWS was sized to handle all the shift condensate. It was decided to reuse the shift condensate in the gasification unit, thereby reducing the requirements of the ASWS. The unit should be sized for 550 gpm (new maximum), instead of the old value of 595 gpm. However, the ASWS unit design for handling 595-gpm flow rate has not been changed due to the late arrival of ECP 6-1112 for flow reduction.

The minimum flow case will occur when the EBH and coker/calciner areas are not operating, while other units are operating at 50% turndown. The total sour water feed decreases to 30% of its design flow rate. Under this condition, steam to the reboiler, E-15504, can be adjusted to increase vapor rate inside columns T-15501 and T-15502 to satisfy the minimum flow constraints. Also, the facility exists to recycle stripped water to the front end of column T-15501 to maintain flows.

Equipment Design Factors. Process equipment requirements were compared with the design case. No modification is required for the minimum case, but most equipment has to be increased for the maximum case. Equipment design factors for the ASWS are listed in Table 13. It should be noted that the equipment sizes have not been revised to reflect the flow reduction due to the late arrival of ECP 6-1112, although their costs were.

LPG Recovery Unit

Process Description. As shown in Figure 12, the fuel gas reject stream from the hydrogen purification unit is first compressed in the feed compressor and then in the compressor side of the compander. The gas is then cooled and chilled by heat exchange with the residue gas from the deethanizer. Heavier components in the feed gas are, therefore, condensed. The liquid fraction is separated and fed to the deethanizer, while the vapor fraction is sent to the expander side of the compander. As the vapor expands in the expander section, its temperature is lowered because of the extraction of energy as mechanical work. Thus, more ethane and heavier components are condensed. This two-phase stream flows to the top of the deethanizer, where it combines with the top tray vapor. The resulting equilibrium produces reflux liquid for the deethanizer and cold residue gas. The residue gas exiting from the top of the deethanizer is warmed by the compressed feed gas and is sent to the hydrogen purification unit. Specified quality LPG is taken from the bottom of the deethanizer and pumped to LPG storage.

Controlling Factors for Maximum and Minimum Cases. The LPG unit is designed for one 100% operating train and is capable of being operated at turndown to 50% of its design capacity under a compressor recycle mode.

A review of all the variations in production of and demand for fuel gas for the overall SRC-I plant led to the following design philosophy for the LPG unit:

a. The front-end section (upstream of V-15552, HP feed gas compressor suction drum) of the LPG unit will be designed to accommodate both maximum and minimum flow. The LPG feed gas will be compressed to 127 psi, high enough to provide dry gas to the HPU unit for reactivation and to discharge gas into the plant fuel gas system.

b. Equipment downstream of V-15552 in the LPG unit will be designed based on the normal flow case. This unit can be operated under a recycle mode around C-15552, the HP compressor, to accommodate the minimum case. For the maximum case, the excess feed gas is bypassed around this downstream unit and is discharged into the plant fuel gas header.

In addition to the flow rate, the changes in molecular weight of the feeds play an important role in the selection of the compressor driver. The speed of the compressor can be varied to accommodate the changes in molecular weight to deliver the desired discharge pressure.

Maximum volume flow will occur when the upstream EBH unit is operated under a low-conversion mode. The maximum molecular weight case (maximum mass flow) will occur when the EBH unit is operated under a low-conversion mode and the HPU is operated under a liquid nitrogen addition mode. The volume flow rates increase by 12.3 and 3.77% for the maximum volume flow and maximum molecular weight cases, respectively. The molecular weights increase by 7.3 and 19.4% for the maximum volume flow and maximum molecular weight cases, respectively.

The minimum flow case will occur when the SRC unit is operated at a 50% turndown condition, and the EBH and coker/calciner units are not operating. The flow and molecular weight of the feed will be 30% of normal capacity and 2% heavier than that for the normal case, respectively. The demonstration plant is in a "fuel-short" condition under this minimum flow case. The front-end section of the LPG unit will be operated to compress the feed gas, and discharge it into the plant fuel-gas system. The downstream LPG unit (downstream of V-15552) will be shut down.

Equipment Design Factors. The process equipment requirements were evaluated for both cases according to the design philosophy outlined in the previous section. No modification is required to accommodate the minimum case. For the maximum case, C-15551 has to be changed from a centrifugal to a screw-type machine; V-15556 and V-15557 have to be added; and E-15551, E-15558, V-15551, and V-15552 have to be resized. To accommodate the feed gas molecular weight changes, a steam turbine driver using steam letdown from 850 to 417 psig is used to replace a motor to drive C-15552, the HP compressor. The design factors are listed in Table 14.

Compression Area

Hydrogen Compression Unit

Process Description. As shown in Figure 13, the hydrogen compression unit compresses a portion of the makeup hydrogen gas stream from the Selexol unit (SRC makeup hydrogen), the bypass hydrogen-rich gas stream from the DEA unit, and the recycle hydrogen gas stream from the HPU. The multiservice reciprocating compressor compresses the SRC makeup hydrogen gas stream in the first section with two compressor stages. The bypass hydrogen-rich gas and the recycle hydrogen gas streams are compressed in the second section with two separate single stages. All three hydrogen streams, after being discharged from the compressors, are combined and delivered to the SRC process area.

Controlling Factors for Maximum and Minimum Cases. The hydrogen compression unit is designed for two 50% operating trains plus one 50% spare train. The unit can be operated at turndown to 25% of its design capacity because of the multiple-train philosophy and the characteristics (pocket adjustment) of the reciprocating compressor.

The unit delivers about the same amount of total combined hydrogen to the SRC area under normal and maximum cases. The flow rate and composition of the combined hydrogen stream are expected to remain about the same. However, three individual feed streams (makeup hydrogen, bypass hydrogen, and recycle hydrogen) are expected to vary. The maximum makeup hydrogen flow rate to the SRC unit will occur when the SRC unit is operated at 100% load while the EBH is not operating. To avoid oversizing the compressor to meet this requirement, the extra hydrogen flow is routed through the methanator and a compressor in the EBH unit and then to the SRC unit. For this instance, a jumper line with associated control equipment is needed; the EBH interlocks must be set up such that the EBH compressor can be operated when the EBH is down; and double-block isolation of the EBH compressor from the rest of the

EBH unit is also required to allow maintenance of the EBH unit during this mode of operation. For the bypass and recycle hydrogen streams, the maximum flow for one stream will occur simultaneously with the minimum flow for the second stream. These conditions will occur when the operating mode of the EBH is switched between high and low conversions. A bypass line and control system will be installed to permit part of the bypass gas from the DEA unit to be compressed with the purified hydrogen stream from the HPU. In this fashion, the surplus flow of bypass H_2 can be compressed in the recycle hydrogen cylinder. This will provide more flexible operation, accommodating bypass hydrogen flow even higher than the maximum flow.

For the minimum case during plant start-up, the unit will handle about 38% of its design capacity. At this turndown operation, one compressor would be operated at 60% load.

Equipment Design Factors. The equipment is sized based on the design philosophy outlined above. Design factors are listed in Table 15.

Sulfur Recovery Area

Claus Unit

Process Description. An ammonia-burning three-stage Claus unit recovers most of the sulfur contained in the acid gases. As shown in Figure 14, NH_3-laden gas originating from the ASWS unit combines with the SRC sour water flash gas and is then combusted at high temperature in the front end of the reaction furnace. Part of the acid gas from the DEA unit is mixed with the NH_3 and also combusted in the front end of the reaction furnace. A smaller portion of DEA acid gas is sent to the in-line auxiliary burners. The remaining DEA acid gas and the Selexol acid gas are mixed before entering the second section of the reaction furnace. The heat generated by the exothermic reaction is used to generate steam. The gas leaving the reaction flows to the first sulfur condenser. From there, the process gas stream passes through three stages of catalytic conversion. Each stage consists of an in-line auxiliary burner, a catalyst bed, and a sulfur condenser. Condensed sulfur is drained to the sulfur pit. The tail gas from the last sulfur condenser is sent to the BSR unit for removal of residual sulfur.

Controlling Factors for Maximum and Minimum Cases. The Claus unit is designed for two 67% operating trains. When both Claus plants are operated, they will be able to process with up to 34% above the normal flow rate.

Maximum flow will occur when the SRC-I plant is processed with coal containing high sulfur and high oxygen. This results in higher quantities of H_2S and CO_2 in the DEA and Selexol acid-gas feed streams. Coal containing high levels of nitrogen will result in high NH_3 gas from the ASWS unit. Under these conditions, the feeds to the Claus facility can increase by 37% over that of the normal case. However, the total gas including air would increase by only 33.8%.

Minimum flow will occur when a single gasifier is being operated during start-up, resulting in a total gas throughput in volume equal to about 20% of a single Claus train design rate.

Equipment Design Factors. For the maximum flow case, both 67% Claus plants will be operated, providing 134% of their total design capacity. The total feed including air under this maximum condition would be about 133.8% of its design rate. Therefore, no modification is required to accommodate the maximum case.

For the minimum case, fuel gas will be fired in the reaction furnace to keep the temperature high enough to stabilize furnace operation. The gas processed through the Claus plant will be above 30% of its single-train design flow, which is slightly higher than the maximum turndown. Therefore, no modification is required to accommodate the minimum case.

The equipment design factors for the Claus unit are listed in Table 16.

Beavon Sulfur Removal (BSR) Unit

Process Description. Residual sulfur is removed from the Claus tail gas in the Beavon sulfur removal unit. As shown in Figure 15, the tail gas is first heated in a reducing gas generator. Steam is added to the generator to ensure adequate production of hydrogen. All sulfur compounds in the Claus tail gas are hydrogenated to H_2S in a catalytic reactor; the gas is then cooled before entering the Stretford portion of the unit, in which H_2S is removed from the gas by the circulating Stretford solution. The gas leaving the tail gas absorber is vented to the atmosphere.

Sulfur pit vapor is blown into an absorber, where it contacts oxidized Stretford solution for partial sulfur removal. The vapor effluent passes up through the upper bed of the tail gas absorber, where it again contacts oxidized Stretford solution. Reduced Stretford solution from the tail gas and pit vapor absorbers flows by gravity to the reaction tanks and then to the oxidizer tanks. Reduced Stretford solution is regenerated by air oxidation.

Sulfur is recovered as a slurry and pumped to the vacuum filter. The oxidized Stretford solution is separated and recovered from the vacuum filter. The sulfur filter cake of about 40 wt % sulfur is reslurried to about 22% and then pumped to a decanter, where steam injection is used to melt the sulfur. Pure sulfur is removed from the bottom of the decanter and sent to the sulfur pit. The oxidized Stretford solution from the decanter is returned to the balance tank and then pumped back to the absorbers.

The caustic storage and distribution facility is designed to provide a centralized caustic receiving, storage, dilution, and distribution system to serve the caustic requirements of several units in the SRC-I plant. Fifty percent caustic shipments will be pumped to the 50% caustic storage tank. Two similar caustic dilution systems are provided. One stream, using process water to dilute the strong caustic, serves the caustic requirements of the GKT gasification unit, the BSR unit, and the off-site usage for reactivating water demineralizers. The other dilution system uses steam condensate to dilute the strong caustic for usage in the DEA gas treating unit.

Controlling Factors for Maximum and Minimum Cases. The BSR unit is designed for one 100% operating train, capable of handling a 50% turndown condition.

Since the primary feed to the BSR unit comes from the Claus unit, it will vary as the Claus unit feed varies. The BSR unit should be able to handle the maximum flow condition when the upstream Claus unit is operating at the start of a run condition until it reaches about halfway between the start of a run and the end of a run condition (2/1.338 = 1.5), since the BSR unit design is based on the end-of-run condition in the Claus unit. This means that the H_2S and SO_2 leakages in the Claus tail gas stream are double those at the start of a run. When maximum flow occurs at the end of a Claus unit run, excess Claus tail gas will be flared through the thermal oxidizer. The minimum flow case will occur during SRC-I plant start-up. The Claus tail gas will be flared through the thermal oxidizer until the tail gas feed reaches 50% of its design capacity.

The Environmental Impact Statement for the SRC-I Demonstration Plant (DOE/EIS-0073) indicates that the vent from thermal oxidation of the Claus acid gas feed (H_2S content is about 30 lb-mol/hr) will not violate the emission regulations during the plant start-up with one gasifier operated at 70% load. The sulfur concentrations (as SO_2) in the thermal oxidizer vent under the conditions outlined above are anticipated to be 17 lb-mol/hr and 12.5 lb-mol/hr for the maximum and minimum cases, respectively. These numbers are much lower than that of the start-up case.

Equipment Design Factors. The equipment design factors for the BSR unit are listed in Table 17.

START-UP, NORMAL, AND MAXIMUM FEED CONDITIONS FOR GAS SYSTEMS UNITS

The feed conditions for the start-up (minimum), normal, and maximum cases for the process units in the gas system area are listed in Table 18.

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3. ICRC. 1980. Mechanical Design Criteria.
4. ICRC's ECP (Engineering Change Proposal) 6-1017, Rev. 1, Minimum/Maximum Modification.
5. ICRC's ECP 6-1108, Caustic Storage and Distribution.
6. ICRC's ECP 6-1112, ASWS Capacity Reduction.
7. Gas Systems Design Baseline Document, Vol. 1-10, December 1981.
8. Gas Systems Revised Design Baseline Document, Vol. 1-10.

Table 1

DPU Feed Streams, Normal Operating Condition, per Train

Component	lb/hr	wt %
	KMAC feed	
C	31,103	49.50
H	2,155	3.43
O	1,759	2.80
N	616	0.98
S	2,520	4.01
Cl	327	0.52
Ash	24,354	38.76
Total dry	62,834	100.00
Solvent	[a]	
Total wet	62,834	
Pressure, water gauge	1.2 in. (0.05 psig)	
Temperature (°F)	380	
	Coal dust feed	
C	13,202	71.0
H	930	5.0
O	2,562	8.4
N	279	1.5
S	632	3.4
Cl	37	0.2
Ash	1,952	10.5
Total dry	18,594	100.0
Water	283[b]	
Total wet	18,877	
Pressure, water gauge	1.2 in. (0.05 psig)	
Temperature (°F)	120	

[a] Will contain deashing solvent up to a maximum of 1 wt %.

[b] Moisture content may vary from a minimum of 1.5 to a maximum of 2.0 wt %.

Table 2

KMAC and Coal Feed Conditions per DPU Train

Component	Maximum KMAC[b] (wt %)	Max. ash mixed feed		100% coal (wt %)	
		High ash	High-ash coal	Normal ash	High ash
C	47.90	44.73	67.50	71.0	67.50
H	3.34	3.11	5.39	5.0	5.39
O	2.67	2.52	9.05	8.4	9.05
N	0.94	0.88	1.61	1.5	1.61
S	4.38	4.65	3.70	3.4	3.70
Cl	0.57	0.61	0.25	0.2	0.25
Ash	40.21	43.50[a]	12.50	10.5	12.50
H_2O	-	-	-	-	-
Subtotal (lb/hr)		66,713	22,686		
Total (lb/hr)	72,150	89,399		67,184	71,342

[a] Assume it is blended with coal to lower the ash content to 40.0%, or lower than 40% to produce the rated hydrogen.

[b] Maximum available KMAC (per train); the condition would occur when high-ash coal is processed in the SRC unit and the recovery in the deashing area is reduced by 5% from design.

Table 3

DPU Equipment Design Factors

Equipment no.	Equipment name	Design factors[a]	Remarks
A-15001	Coal/KMAC mixer	1.09	b
A-15002	KMAC humidifier	1.24	c
AL-15011	Mixed dust feeder	1.09	b
AL-15012	KMAC dust conveyor feeder	1.25	c
AL-15014	Filter dust feeder	1.67	b
AL-15016	Impure N_2 filter dust feeder	1.60	b
BH-15002	Specified KMAC receiving bunker	1.24	b
BH-15003	Coal dust receiving bunker	4.15	b
BH-15006	Finished dust bunker	1.06	b
C-15003	KMAC exhaust fan	1.00	b
C-15004	KMAC/coal dust exhaust fan	1.71	b
C-15005	Impure N_2 recycle fan	1.07	b
CV-15007	KMAC dust screw conveyor	1.25	b
CV-15008	Coal metering rooler	1.15	b
CV-15009	KMAC/coal dust screw conveyor	1.67	b
CV-15011	KMAC/coal dust screw conveyor	1.60	b
CV-15012	KMAC/coal dust screw conveyor	1.60	b
DC-15007	KMAC humidifier/cyclone dust separator	1.04	c
E- 15011	Nitrogen heater	1.42	b
FL-15003	KMAC exhaust filter	1.77	b
FL-15005	Impure N_2 filter	1.00	b

Table 3 (Continued)

Equipment no.	Equipment name	Design factors[a]	Remarks
X-15001	KMAC dosing package	1.24	c
X-15002	Coal dust dosing package	3.84	c
X-15003	Humidifier vent pipe	1.24	c
X-15004	Coal/KMAC mixer chute	1.09	b
X-15005	Coal/KMAC mixer vent pipe	1.09	b
C-15016	Recycle nitrogen LP compressor	1.0	b
C-15017	Recycle nitrogen HP compressor	1.0	b
E-15016	LP nitrogen air cooler	1.0	b
E-15017	LP nitrogen water cooler	1.0	b
E-15018	HP compressor effluent recycle N_2 heat exchanger	1.0	b
E-15019	HP nitrogen air cooler	1.0	b
E-15020	HP nitrogen water cooler	1.0	b
P-15017	Condensate pump	1.1	b
V-15016	Recycle H_2 1st K.O. pot	1.0	b
V-15017	Recycle nitrogen separator	1.0	b
X-15016	Recycle N_2 refrigeration unit	1.0	b

[a] Based on mixed-feed case flow conditions.
[b] Two 50% units.
[c] Four 25% units.

Table 4

Total Feed to the Gasification Unit; Mixed-Feed Case

Component (lb/hr)	Per train	Total (3 trains)	(wt %)
	A. Dust Feed		
C	29,536.29	88,608.88	54.41
H	2,056.61	6,169.82	3.79
O	2,214.13	6,642.40	4.08
N	596.41	1,789.24	1.10
S	2,101.25	6,303.74	3.87
Cl	242.65	727.94	0.44
Ash	17,538.51	52,616.24	32.31
Total solids	54,285.75	162,857.26	100.00
Nitrogen[a]	101.79	305.37	-
Water	1,251.99	3,755.97	-
Total solids & gas	55,639.50	166,918.60	-
Pressure (psia)[b]		14.55 (0.05 psig)	
Temperature (°F)[b]		206.0	
	B. Oxygen Feed		
O_2 (lb-mol/hr)	1,174.33	3,522.99	99.45
N_2 (lb-mol/hr)	0.59	1.77	0.05
Ar (lb-mol/hr)	5.90	17.70	0.50
Total (mol/hr)	1,180.82	3,542.46	100.0
Pressure (psia)		23.20	
Temperature (°F)		140	
	C. Process Steam Feed		
Total steam (lb/hr)	3,651	10,953	
Pressure (psia)		42.0	
Temperature (°F)		270.2	

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[a] N_2 assumed to be entrained in the pores of the dust feed.

[b] Estimated values.

Table 5

Gasification Equipment Design Factors

Equipment no.	Equipment name	Design factors[a]	Remarks
AL-15051	Coal/KMAC metering roller	1.36	
B-15051	Waste heat recovery boiler	1.18	c
B-15052	Fly dust separator	2.00	c
B-15053	Dust separator inlet	1.39	
BH-15051	Coal/KMAC feed service bunker	1.20	c
BH-15052	Coal/KMAC feed bunker	1.20	c
C-15051	Raw gas blower	1.04	b
CV-15051	Coal/KMAC feed pneumatic pump	1.36	d
CV-15052	Coal/KMAC feeder	1.38	d
CV-15053	Coal/KMAC double screw feeder	1.38	d
CV-15054	Slag belt conveyor	2.51	c
E-15051	O_2 heater	1.14	b
E-15052	Slag cooling water cooler	1.00	
E-15053	HP BFW preheater	1.18	c
FL-15051	Fly dust screen	2.00	
H-15051	Gasifier burner	1.14	
H-15052	Gasifier ignition burner	1.00	
H-15053	Gasifier sintering burner	1.00	
J-15051	Burner O_2/steam mixer	1.15	b
P-15051	Fuel oil pump	1.00	
P-15053	Drop separator wash water pump	1.34	c
P-15054	Slag cooling water pump	1.00	b
P-15055	Circulating pump	1.18	b

Table 5 (Continued)

Equipment no.	Equipment name	Design factors[a]	Remarks
R-15051	Gasifier	1.10	
S-15051	Raw gas cooling washer	1.39	b
S-15052	Ras gas disintegrators	1.04	b
S-15053	Raw gas drop separator	1.39	
SC-15051	HP steam sample cooler	1.00	
SC-15052	HP BW sample cooler	1.00	
SC-15053	LP steam sample cooler	1.00	
SC-15054	LP BW sample cooler	1.00	
ST-15051	Gasifier flare	1.02	
TK-15051	Fuel oil tank	1.00	
TK-15052	Overhead water tank	1.23	
TK-15053	Slag cooling water sump	1.26	
V-15051	Pneumatic pump N_2 vessel	1.00	
V-15052	Purge N_2 vessel	1.00	
V-15054	LP steam drum	1.44	b
V-15056	Fly dust discharge vessel	2.00	
V-15057	HP steam drum	1.18	c
V-15058	Vent N_2 seal pot	1.00	
V-15060	Flare seal pot	1.00	
V-15062	Steam impact separator	1.21	
X-15051	Slag extractor	2.34	b
X-15052	Raw gas quick seal	1.04	
X-15053	Coal/KMAC feed blow pipe	1.14	
X-15055	Gasifier ignition lance	1.00	

[a] Based on mixed-feed case.
[b] High-ash coal feed.
[c] High-ash KMAC feed.
[d] High-ash KMAC plus high-ash coal feeds.

Table 6

Equipment Design Factors for Wash Water Treatment Unit

Equipment	Equipment name	Design factor[a]
A-15151 A,B	Flocculating agent dosing drum agitator	1.11
C-15151 A,B	Channel exhaust vapor fan	1.0
C-15152 A,B	Settling basin vapor fan	1.0
E-15151 A through D	Wash water cooler	1.16
E-15152 A through D	Wash water cooler	1.16
P-15151 A,B	Flocculating agent dosing pump	3.1
P-15152 A through D	Sludge pump	1.31
P-15156 A through D	Sludge pump	1.31
P-15153 A through D	Wash water pump	1.34
P-15157 A through D	Wash water pump	1.34
P-15155 A,B	Drain water sump pump	1.0
TK-15151	Wash water sump	1.14
TK-15152	Drain water sump	1.0
V-15151 A,B	Flocculation agent dosing drum	1.11
X-15151 A,B	Settling basin scraper	1.13
X-15152 A,B	Settling basin	1.14

[a] Based on mixed-feed case flow conditions.

Table 7

Shift Unit Equipment Desgin Factors

Equipment	Equipment name	Design factor[a]
E-15252	1st shift feed/effluent heat exchanger	2.99
E-15253	1st shift effluent HP steam boiler	1.01
E-15254	2nd shift effluent HP steam boiler	1.39
E-15255	2nd shift effluent HP BFW heater	1.02
E-15256	3rd shift effluent HP BFW heater	1.02
E-15257	3rd shift effluent MP steam boiler	1.04
E-15258	3rd shift effluent LP steam boiler	1.04
E-15259	3rd shift effluent MP BFW preheater	1.04
E-15260	3rd shift effluent HP BFW preheater	1.01
E-15261	3rd shift effluent air cooler	1.26
E-15262	3rd shift effluent trim water cooler	1.13
E-15263	3rd shift effluent CO_2 exchanger	1.0
E-15264	Shift condensate/recycle condensate exchanger	1.35
E-15265	Recycle condensate water cooler	1.03
E-15266	Recycle condensate air cooler	1.04
H-15251	1st shift feed start-up heater	1.0
P-15255	Recycle condensate pump & spare	1.1
R-15251	1st shift reactor	1.0
R-15252	2nd shift reactor	1.0
R-15253	3rd shift reactor	1.0
V-15254	Shift effluent 1st K.O. pot	1.0
V-15255	Shift effluent 2nd K.O. pot	1.0
V-15256	Shift effluent/CO_2 exchanger K.O. pot	1.0
V-15257	Shift condensate flash drum	

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[a] Duty in controlling case/duty in normal SOR case.

Table 8

Material Balance for Start-up Case

	Stream no., stream description, temp (°F), and pressure (psia)									
	1	3	5	9	10	11	12	13	14	15
	Syngas from GKT unit	Syngas to RSGC	Syngas to shift	Shifted gas to Selexol	Treated H_2 gas	Treated H_2 gas to H_2 comp	Recycle H_2 gas	Recycle flash gas	Recycle gas from Selexol	Recycle gas from 5th st. of RSGC
	105	85	284	100	60	60	60	16.5	38.8	100
	14.5	14.5	852	720	675	675	675	50	50	845
lb-mol/hr										
H_2	887.67	3,083.34	2,374.10	4,283.33	4,228.69	2,800.58	1,428.11	58.32	1,486.43	709.24
N_2	30.04	62.69	48.27	48.27	53.06	35.14	17.92	0.31	18.23	14.42
Ar	6.25	12.35	9.51	9.51	9.47	6.27	3.20	0.06	3.26	2.84
CO	1,948.61	2,559.64	1,970.86	61.63	61.00	40.40	20.60	1.65	22.25	588.78
CO_2	322.25	1,630.88	1,255.74	3,169.78	4.60	3.05	1.55	931.94	933.49	375.14
H_2S	42.67	81.44	62.71	67.52	-	-	-	20.04	20.04	18.73
COS	4.78	6.31	4.86	0.05	Tr.	Tr.	Tr.	0.08	0.08	1.45
CH_4	3.31	9.42	7.25	7.25	6.92	4.58	2.34	1.60	3.94	2.17
H_2O	267.01	272.51	15.44	10.12	1.69	1.12	0.57	0.32	0.89	4.61
Total	3,512.59	7,718.58	5,748.74	7,657.46	4,365.43	2,891.14	1,474.29	1,014.32	2,488.61	1,717.38
MW (lb/lb-mol)	22.28	20.75	20.84	20.14	2.85	2.85	2.85	41.32	18.53	20.84

Table 9

Selexol Unit

Heat Exchanger Comparison

	Normal case		Maximum case		
Equip no.	Flow (lb/hr) (tube S/shell S)	Duty (MM Btu/hr)	Flow (lb/hr) (tube S/shell S)	Duty (MM Btu/hr)	Remarks
E-15401	169,874/27,352	2.610	180,880/25,844	2.704	The required additional duty load can be shifted to E-15410 and E-15412
E-15402	87,973/146,475	3.940	88,388/143,700	3.851	The current design can handle the maximum flow case
E-15403	59,979/air	1.860	59,981/air	1.614	"
E-15404	171,233/574,800	15.411	174,568/618,400	15.665	"
E-15405	29,020/528,156	25.143	29,931/568,110	25.931	"
E-15406	506,057/532,932	73.078	544,338/583,320	78.106	The current heat exchanger surface is enough to handle additional duty, since the flow rate increased by 8% would increase overall heat transfer coefficient
E-15407	2,820,140/1,848,860	16.372	2,897,410/1,902,340	17.161	The heat-transfer coefficient will increase by 2.5%, and the additional duty can be picked up by X-15401, the refrigeration unit
E-15409	98,557/30,274	0.431	107,777/31,935	0.360	The current design can handle the maximum flow case
E-15410	72,062/98,557	1.075	76,965/107,777	1.150	"
E-15412	153,132/184,694	2,313	151,426/180,233	2.262	"
E-15415	364,593/156,590	0.467	371,183/157,987	0.467	"
X-15401	2,820,140/241,275	16.052	2,897,410/246,080	16.372	"

Table 10

Selexol Equipment Design Factors

Equipment name	Design factor[a]	Remark
1. Compressors	1.00	
2. Exchangers	1.00	
3. Filters	1.00	
4. Hydraulic turbines	1.00	
5. Pumps		
5.1 P-15409	1.00	Impeller size for delivering additional 10% flow should be installed.
5.2 P-15401, P-15404, P-15405, P-15406, P-15407, P-15408	1.05	" "
5.3 P-15402, P-15403	1.10	
6. Towers	1.00	
7. Storage tanks and sumps	1.00	
8. Stacks	1.00	
9. Vessels	1.00	
10. Package items	1.00	

[a] Factor referred to normal design case requirement.

Table 11

Heat Exchanger Comparison

	Normal case		Maximum case	
Equip. no.	Flow (lb/hr) (tube side/shell side)	Duty (MM Btu/hr)	Flow (lb/hr) (tube side/shell side)	Duty (MM Btu/hr)
E-15301	Intermittent	8.170	Intermittent	8.170
E-15302	115,025/97,240	1.650	125,218/105,850	1.990
E-15304	106,490/98,423	5.980	138,693/128,030	7.420
E-15305	117,370/114,512	6.590	142,641/179,088	7.630
E-15306	128,196/508,037	13.720	161,672/640,600	17.300
E-15307	28,838/126,094	26.350	36,356/162,910	33.220
E-15308	98,243/air	5.760	128,029/air	6.330
E-15309	114,512/air	5.220	179,088/air	6.750

Table 12

DEA Equipment Design Factors

Equipment	Design factor[a]	Remarks
Mixers		
A-15302	1.0	
A-15303	1.0	
Exchangers		
E-15301	1.39	Rated to handle intermittent dilution of DEA shipments
E-15302	1.21	
E-15304	1.24	
E-15305	1.16	
E-15306	1.26	
E-15307	1.26	
E-15308	1.10	
E-15309	1.29	
Pumps		
P-15301 A,B	1.1	
P-15302 A,B	1.1	
P-15303 A,B	1.1	
P-15304 A,B,C	1.1	
P-15305 A,B	1.16	Sized for varying reflux
P-15306	1.0	
P-15307 A,B	1.2	Sized for uncertainty of feed gas contaminants
P-15308 A,B	1.14	
P-15309 A,B	1.14	
P-15310 A,B	1.08	
P-15311 A,B	1.0	

Table 12 (Continued)

Equipment	Design factor[a]	Remarks
Pumps (Continued)		
P-15313 A,B	1.16	
P-15314 A,B	1.0	
P-15318 A,B	1.37	Sized for uncertainty of feed gas contaminants
P-15319 A,B	1.0	
Hydraulic turbine		
HT-15301	1.0	
Towers		
T-15301	1.0	
T-15302	1.0	
T-15303	1.0	
T-15304	1.0	
T-15305	1.0	
T-15306	1.19	
Storage tanks	1.0	
Pressure vessels		
V-15308	1.48	
Other vessels	1.0	
Filter/separators	1.0	

[a] Factor referred to normal design case requirement.

Table 13

ASWS Unit Equipment Design Factors

Equipment	Design factor[a]	Remarks
Pumps		
P-15502 A,B	1.56	
P-15503 A,B	1.53	
P-15504 A,B	1.52	
P-15505 A,B	1.56	
P-15508 A,B	1.50	
P-15509 A,B	1.52	
Other pumps	1.0	
Exchangers		
E-15501	2.31	For winter condition
E-15503	2.12	For winter condition
E-15502	1.56	
E-15504	1.54	
Towers		
T-15501	1.56	
T-15502	1.56	
Tanks		
TK-15501	1.56	
TK-15502	1.56	
Vessels		
V-15501	1.56	
V-15502	1.56	
Slaker	1.0	

[a] Factor refers to new normal design case requirement. Equipment sizes have not been revised due to late arrival of ECP 6-1112 for flow reduction. However, their cost has been changed and incorporated in the Cost Baseline.

Table 14

LPG Equipment

Equipment	Design factor[a]	Remarks
	LPG Equipment Design Factors	
Compressors		
C-15551	1.12	Sized for maximum feed case
C-15552	1.0	
Exchangers		
E-15551	1.18	Sized for maximum feed case
E-15552	1.0	
E-15553	1.0	
E-15554	1.0	
E-15555	1.0	
E-15556	2.55	Sized for start-up duty
E-15557	1.0	
E-15558	1.17	Sized for maximum feed case
Pumps		
P-14441 A,B	1.0	
P-15552 A,B	1.0	
Columns		
T-15551	1.0	
Vessels		
V-15551	1.12	Sized for maximum feed case
V-15552	1.12	Sized for maximum feed case
V-15553	1.0	
V-15554	1.0	
V-15556	1.12	Sized for maximum feed case
V-15557	1.0	
Compander		
X-15551	1.0	
	LPG Storage, Vaporization, and Transfer Equipment Design Factors	
E-15559	1.0	
J-15551	1.0	
P-15553 A,B	1.25	Dictated by pump availability
P-15554 A,B	1.0	
V-15558 A,B,C,D	1.0	
V-15559	1.0	

[a] Factor refers to normal design case requirements.

Table 15

Area 15 Gas System H_2 Compression Unit Equipment Design Factors

Equipment no.	Equipment name	Design factors[a]	Remarks
C-15601	Hydrogen compressor		Three 50% units
	Makeup H_2 cylinder	1.0	
	Bypass H_2 cylinder	1.0	
	Recycle H_2 cylinder	1.05	
E-15601	Makeup H_2 compressor recycle cooler	1.0	Sized for 50% recycle
E-15602	Makeup H_2 interstage cooler	1.0	
E-15603	Recycle H_2 compressor cooler	1.05	Sized for 50% recycle
E-15604	Bypass H_2 compressor recycle cooler	1.0	Sized for 50% recycle
V-15601	1st stage makeup H_2 K.O. pot	1.0	
V-15602	2nd stage makeup H_2 K.O. pot	1.0	
V-15603	Recycle H_2 feed K.O. pot	1.0	
V-15604	Bypass H_2 feed K.O. pot	1.0	

[a] Factor refers to normal design case requirement.

Table 16

Claus Equipment Design Factors

Equipment no.	Design factor[a]	Remarks
C-15701 A,B,C	1.1	For variations in feed rate & composition
C-15702 A,B, 15722 A,B	1.0	
C-15703 A,B,C	1.0	
C-15704	1.0	
E-15701, 15721	1.0	
E-15702, 15722	1.0	
E-15703, 15723	1.0	
E-15704, 15724	1.0	
E-15705, 15725	1.0	
E-15706, 15726	1.0	
H-15701, 15721	1.0	
H-15702, 15722	2.0	For start-up & catalyst treatment operations
H-15703, 15723	2.0	For start-up & catalyst treatment operations
H-15704, 15724	2.0	For start-up & catalyst treatment operations
H-15705	1.0	
H-15706	1.0	
P-15701 A,B,C,D	-	Intermittent
P-15703 A,B, 15723 A,B	-	Intermittent
P-15704 A,B	1.1	
P-15705 A,B	1.1	
P-15706 A,B	1.1	
P-15707 A,B	-	Intermittent
TK-15701, 15721	-	Storage for 3-days sulfur production (each)
TK-15702 A,B	-	Storage for 10-days sulfur production (each)

Table 16 (Continued)

Equipment no.	Design factor[a]	Remarks
V-15701	1.0	
V-15702	1.0	
V-15703, 15723	1.0	
V-15704, 15724	1.0	
V-15705, 15725	1.0	
V-15706	1.0	
V-15707	1.0	
V-15708	1.0	
X-15701	1.0	

[a] Factor refers to the normal design case requirements.

Table 17
BSR Equipment Design Factors

Equipment	Design factor[a]	Remarks
Blowers		
C-15800 A,B	1.1	
Exchangers		
E-15801	1.53	Sized for uncertainty of E-15802 duty due to system reslurrying and water balance
E-15809	2.35	Sized for uncertainty in quantity of water required for reslurrying
All others	1.0	
Burners (H-15800)	1.1	Sized for start of run
Pumps	1.1	Rated for better process control
Reactor (R-15800)	1.0	
Venturi scrubbers	1.0	
Tanks	1.0	
Absorbers & vessels	1.0	
Filter	1.5	Sized for 16 hr/day operation
Caustic storage & distribution		
Mixer		
A-15809	1.0[b]	
A-15810	1.0	
Exchanger		
E-15810	1.18	
E-15811	1.00[b]	
E-15812	1.00[b]	
Pumps		
P-15816 A/B	1.25	
Other	1.00[b]	
Tanks	1.00[c]	

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[a] Factor refers to the normal design case requirement.
[b] Factor refers to the intermittent maximum flow requirement.
[c] Size is based on 15-day supply of caustic.

Table 18

Gas Systems Unit Feeds for Start-up, Normal, and Maximum Flow Cases

	Dust preparation unit			Gasification			Raw syngas compression		
	Start-up[a]	Normal[b]	Maximum[c]	Start-up	Normal	Maximum	Start-up[d]	Normal	Maximum
Temp (°F)				140	140	140		105	105
Press (psia)				23.2	23.2	23.2	14.5	14.5	14.5
lb-mol/hr									
H_2				-	-	-	3,083.34	2,652.57	2,606.45
N_2				0.45	1.77	1.85	62.69	89.83	96.64
Ar				4.45	17.70	18.54	12.35	17.70	18.36
CO				-	-	-	2,559.64	5,842.95	5,898.10
CO_2							1,630.88	699.39	793.44
H_2S							81.44	176.46	217.45
COS							6.31	20.01	24.16
CH_4							9.42	9.51	9.66
SO_2							0.01	0.03	0.03
HCN							0.25	0.72	0.72
NO							0.06	0.18	0.18
NH_3							0.06	0.18	0.18
H_2O							272.51	803.61	816.78
O_2				886.76	3,522.99	3,657.61	0.02	0.06	0.06
Steam				155.46	607.92	657.19	-	-	-
Total				1,047.12	4,150.38	4,335.20	7,713.98	10,313.20	10,482.21
lb/hr									
C	22,259.0	88,609.0	90,308.0	22,259.0	88,609.0	90,308.0			
H	1,569.0	6,170.0	6.595.0	1,569.0	6,170.0	6,595.0			
N	471.0	1,789.0	1,904.0	471.0	1,789.0	1,904.0			
S	1,066.0	6,304.0	7,882.0	1,066.0	6,304.0	7,882.0			
O	2,632.0	6,643.0	7,468.0	2,632.0	6,643.0	7,468.0			
Cl	64.0	728.0	927.0	64.0	728.0	927.0			
Ash	3,292.0	52,615.0	63,713.0	3,292.0	52,615.0	63,713.0			
Dry (total)	31,353.0	162,858.0	178,797.0	31,353.0	162,858.0	178,797.0			

Table 18 (Continued)

	Shift unit			Selexol unit			Methanation unit		
	Start-up	Normal	Maximum	Start-up[e]	Normal	Maximum	Start-up	Normal	Maximum
Temp (°F)	265	265	265	100	100	100	60	60	60
Press (psia)	810	810	810	720	720	720	670	670	670
lb-mol/hr									
H_2	2,374.10	2,651.91	2,604.16	4,283.33	8,264.33	8,319.59	642.88	3,214.38	3,214.38
N_2	48.27	89.83	96.63	48.27	89.80	96.62	7.53	37.64	40.46
Ar	9.51	17.70	18.36	9.51	17.69	18.36	1.38	6.88	7.13
CO	1,970.86	5,842.95	5,897.97	61.63	227.84	181.97	17.58	87.89	87.53
CO_2	1,255.74	699.39	793.65	3,169.78	6,304.82	6,518.49	0.67	3.35	3.35
H_2S	62.71	176.49	217.33	67.52	193.30	239.72	(0.24 ppmv)	(0.24 ppmv)	(0.24 ppmv)
COS	4.86	20.01	24.16	0.05	0.39	0.39	(1.1 ppmv)	(0.1 ppmv)	(0.1 ppmv)
CH_4	7.25	9.51	9.66	7.25	9.51	9.66	0.71	3.53	3.58
SO_2	-	-	-	-	-	-	-	-	-
HCN	0.25	0.72	0.72	-	-	-	-	-	-
NO	-	-	-	-	-	-	-	-	-
NH_3	0.12	0.36	0.36	-	-	-	-	-	-
H_2O	15.44	25.60	26.02	10.12	19.98	20.35	0.02	0.12	0.12
O_2	-	-	-	-	-	-	-	-	-
Steam	-	-	-	-	-	-	-	-	-
Total	5,749.11	9,534.47	9,689.00	7,657.46	15,127.64	15,405.13	670.77	3,353.79	3,356.55

Table 18 (Continued)

	DEA unit						LPG unit			
	Minimum[f]		Normal		Maximum		Minimum[f]	Normal	Maximum case	
	HP feed	LP feed	HP feed	LP feed	HP feed	LP feed			Max mol wt	Max flow
Temp (°F)	110	110	110	110	120	120	59.2	62	55	59.4
Press (psia)	1,810	100	1,810	100	1,910	115	18	18	18	18
lb-mol/hr										
H_2	4,807.0	14.25	9,948.00	107.0	10,034.21	122.89	145.5	439.0	180.40	365.0
N_2	282.5	7.50	599.0	17.2	672.99	13.11	67.4	88.0	161.26	95.0
Ar	43.0	0.50	96.0	1.7	91.09	1.38	9.5	30.0	27.77	30.0
CO	345.5	6.05	691.0	17.7	665.10	16.27	72.2	155.0	141.13	145.0
CO_2	45.5	0.25	91.0	4.9	108.50	6.37	-	-	-	-
H_2S	103.5	0.65	208.0	11.43	254.91	17.76	-	-	-	-
COS	-	-	-	-	-	-	-	-	-	-
CH_4	557.5	5.90	1,383.0	231.6	1,609.73	277.02	141.2	669.0	821.46	852.1
C_2H_6	164.5	8.40	410.0	82.8	486.53	94.09	60.8	239.0	305.65	306.0
C_3H_8	70.0	7.60	191.0	59.5	237.03	73.84	23.5	100.0	133.30	132.0
C_4H_{10}	30.0	7.65	86.0	54.9	121.36	72.95	7.1	30.0	44.89	39.9
C_5H_{12}	12.5	7.15	27.0	19.4	62.17	29.76	0.28	1.0	1.23	1.0
C_6^+~250°F	22.5	7.00	46.0	16.8	18.86	9.92	-	-	-	-
250-400°F	0.5	-	1.0	0.5	2.53	1.01	-	-	-	-
NH_3	2.5	0.05	6.0	0.22	0.71	0.81	-	-	-	-
H_2O	4.6	1.44	9.72	6.08	10.31	7.28	-	-	-	-
Total	6,491.6	74.39	13,792.72	631.73	14,376.03	744.46	527.48	1,751.0	1,817.09	1,966.0

Table 18 (Continued)

	H_2 compression unit								
	Startup			Normal			Maximum		
	Makeup H_2	Bypass H_2[f]	Recy. H_2[f]	Makeup H_2	Bypass H_2	Recy. H_2	Makeup H_2	Bypass H_2	Recy. H_2
Temp (°F)	60			60	110	62.4	64	120	55
Press (psia)	675			675	1,785	1,700	675	1,885	1,790
lb-mol/hr									
H_2	1,936.98			4,996.01	3,636.89	5,837.69	5,046.26	3,063.82	6,752.81
N_2	15.17			50.77	219.08	290.69	61.64	206.00	378.02
Ar	4.05			10.35	35.10	31.49	11.03	27.81	34.71
CO	43.09			153.84	252.64	281.82	109.69	202.90	316.15
CO_2	2.00			5.21	0.05	-	4.38	0.04	-
CH_4	2.14			5.49	505.51	191.38	5.63	491.04	274.14
C_2H_3	-			-	149.85	0.64	-	148.03	0.91
C_3H_8	-			-	69.80	0.03	-	71.72	-
C_4H_{10}	-			-	31.37	-	-	36.56	-
C_5H_{12}	-			-	9.87	-	-	16.69	-
C_6^+	-			-	17.19	-	-	4.75	-
H_2O	0.07			0.19	3.48	-	0.38	3.82	-
Total	2,003.50			5,221.86	4,930.83	6,633.74	5,239.01	4,273.18	7,756.78

Table 18 (Continued)

	ASWS unit[f]			Claus unit[g]			BSR unit		
	Minimum	Normal	Maximum	Startup	Normal	Maximum	Startup	Normal	Maximum
Temp (°F)	155	165	168	105	129.5	129.0		280	280
Press (psia)	25	25	25	24.5	24.5	24.5		17.5	17.5
lb-mol/hr									
H_2	2.20	4.40	4.53	-	2.74	4.92		42.96	58.0
N_2	0.05	0.10	0.12	-	0.09	0.18		1,372.89	1,806.95
Ar	0.01	0.03	0.02	-	-	0.03		-	0.03
CO	0.04	0.16	0.15	-	0.17	0.22		48.64	65.66
CO_2	29.96	59.91	68.60	107.83	602.09	871.84		559.93	818.31
H_2S	31.86	83.77	119.93	30.18	494.86	626.09		15.17	19.91
COS	0.01	0.02	0.02	0.02	0.26	0.19		0.14	0.15
CH_4	-	-	0.28		0.13	0.38			
C_2H_6	-	-	0.09		0.96	0.12			
HCN	0.01	0.06	0.18		0.11	0.23			
NH_3	46.65	171.31	222.02		172.19	238.91			
NH_4Cl	13.16	26.33	17.77		-	-			
CH_3SH	0.05	0.14	0.15		0.40	0.77			
Phenol	0.84	1.83	6.88		0.56	1.48			
SO_2	-	-	-		-	0.04		7.58	9.95
S_6	-	-	-		-	-		0.10	0.13
S_8	-	-	-		-	-		0.23	0.29
H_2O	3,543.75	12,727.5	14,551.78	6.51	178.55	246.55		934.40	1,179.41
Total	3,669.59	13,075.56	14.992.52	144.54	1,453.11	1,991.95		2,982.04	3,958.79

[a] 100% coal feed with one gasifier operating at 70%.
[b] KMAC/coal mixed feed case (125,668 lb/hr KMAC + 37,138 lb/hr coal, dry).
[c] Maximum ash KMAC/maximum ash coal mixed feed case (133,426 lb/hr KMAC + 45,372 lb/hr coal, dry).
[d] Including recycle gases from the Selexol unit and the discharge of the 5th stage of the RSGC.
[e] Including recycle gas from the Selexol unit to accommodate 50% turndown in Selexol.
[f] No flow during initial start-up.
[g] Combined flow rate.

Figure 1

Major Process Flowscheme--Overall SRC-I Plant

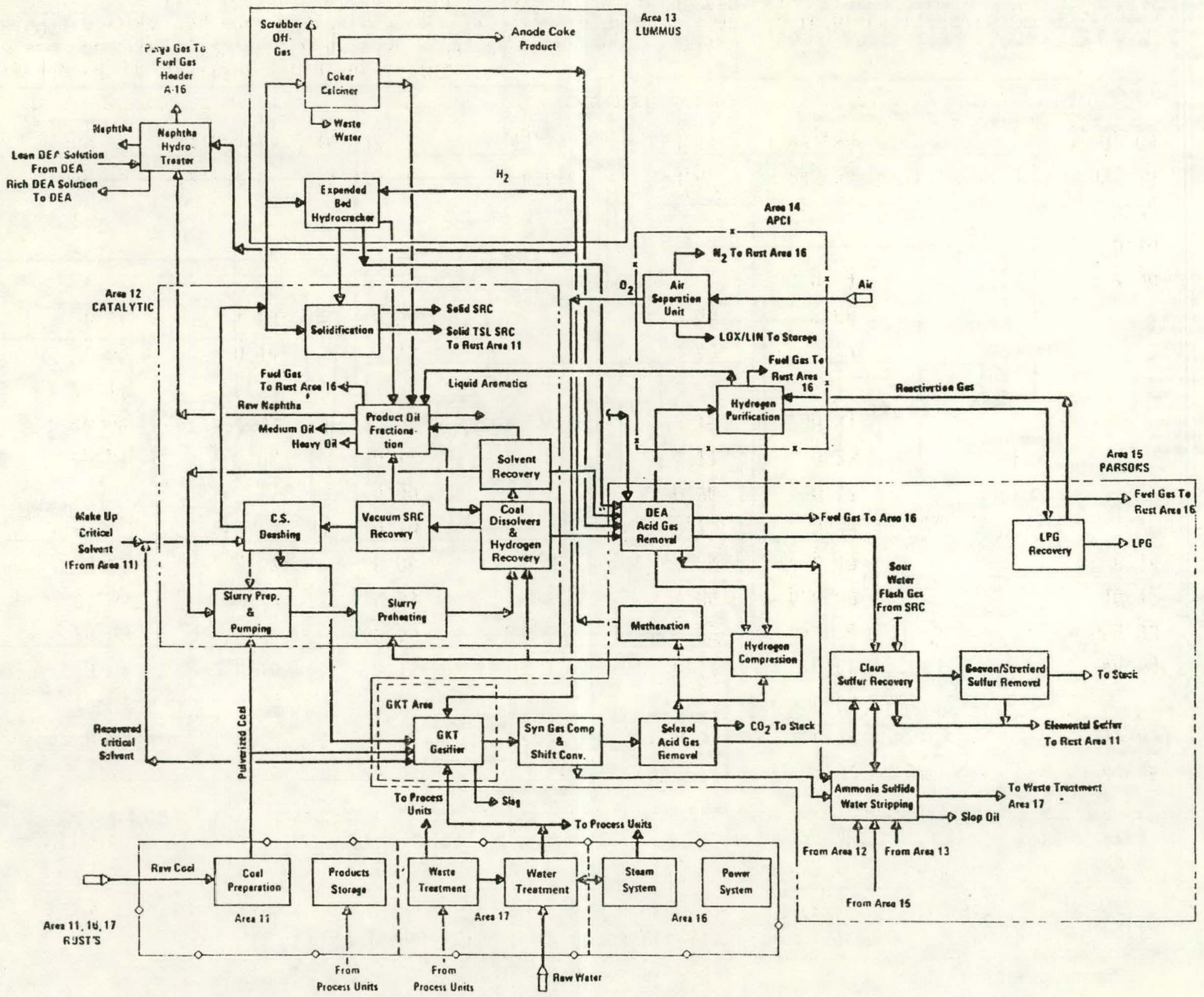


Figure 2A

Dust Preparation Unit (DPU) Process Flow Diagram

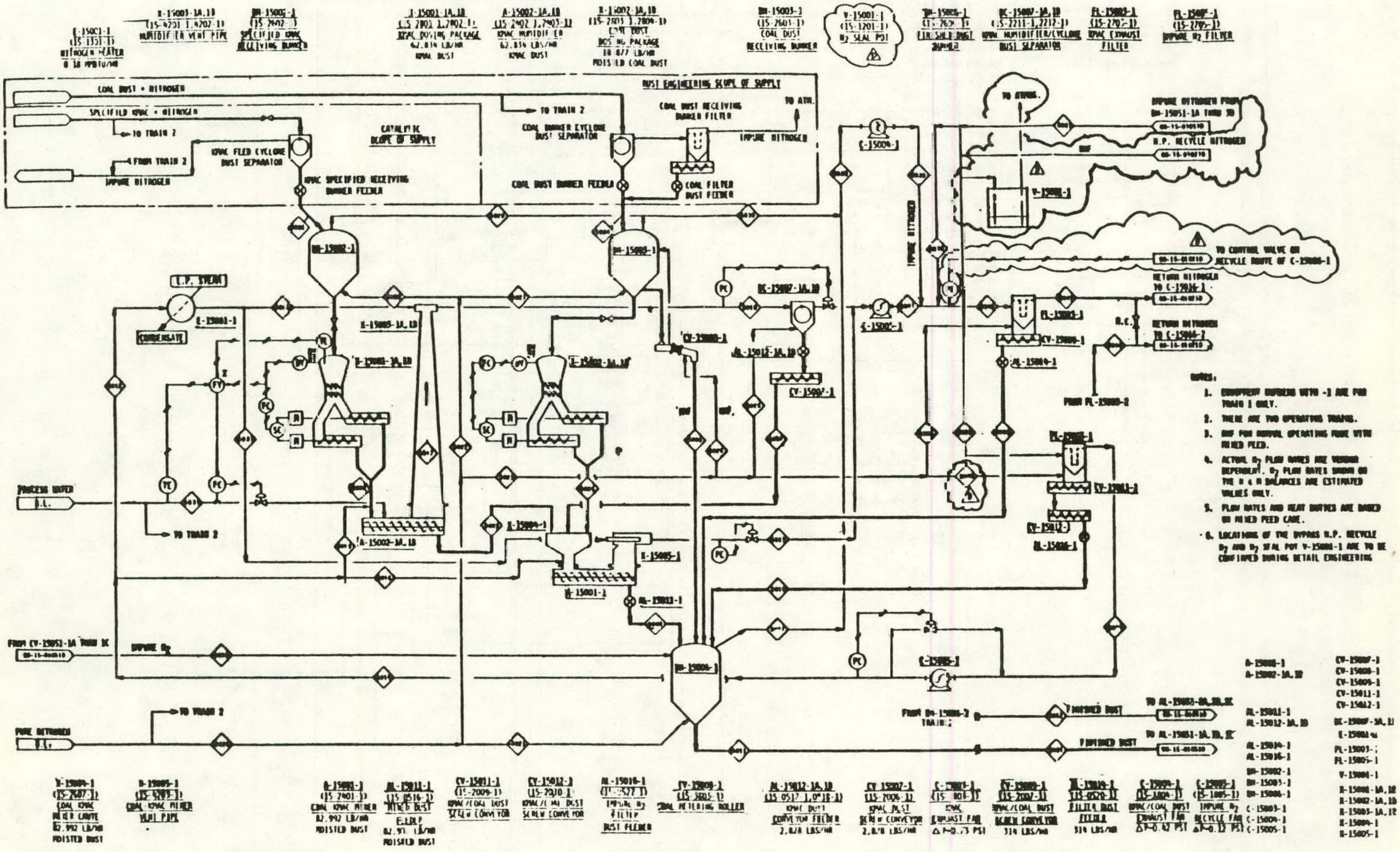


Figure 2B

DPU Nitrogen Recovery and Solvent Recovery Process Flow Diagram

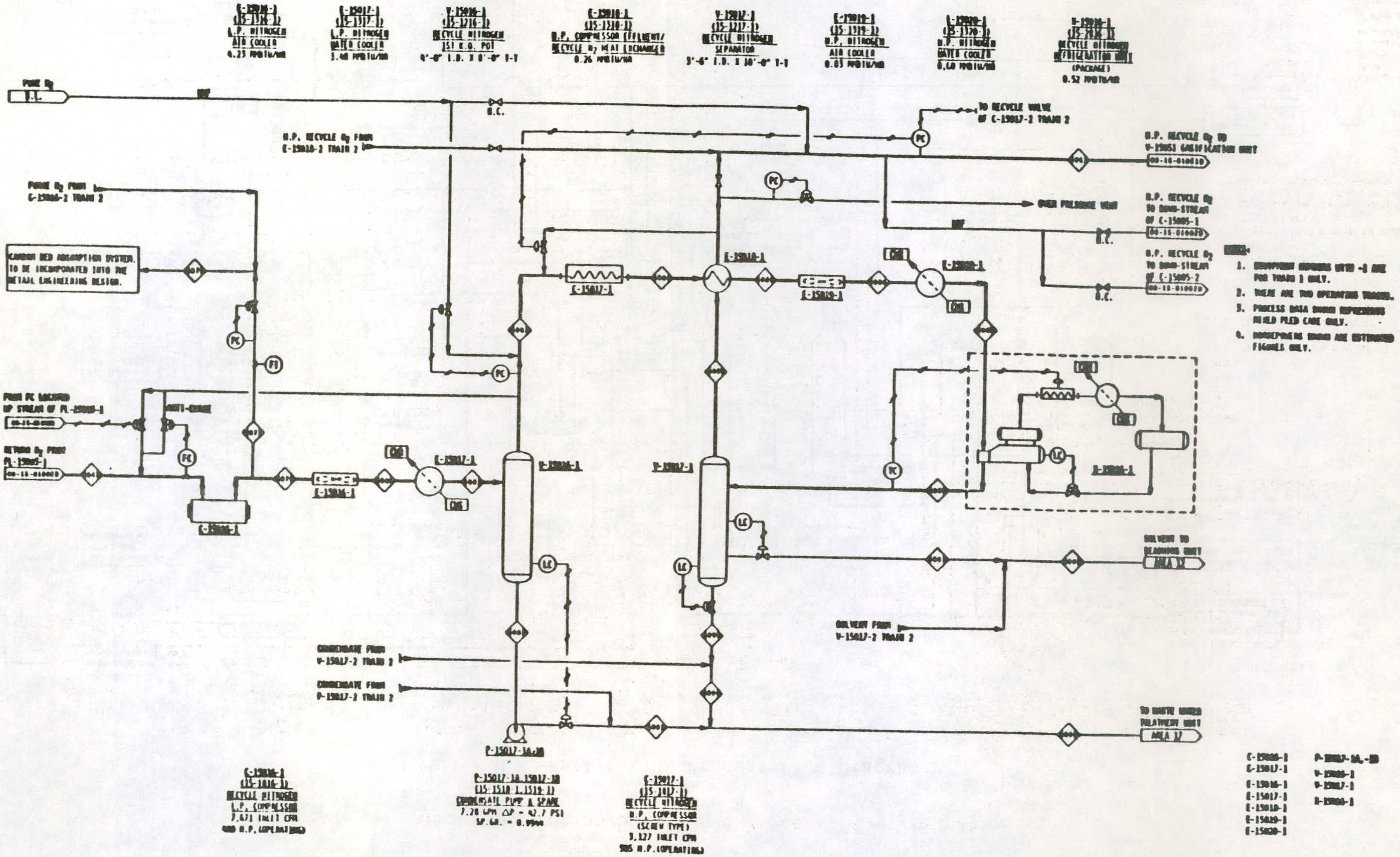


Figure 3

GKT Gasification Process Flow Diagram

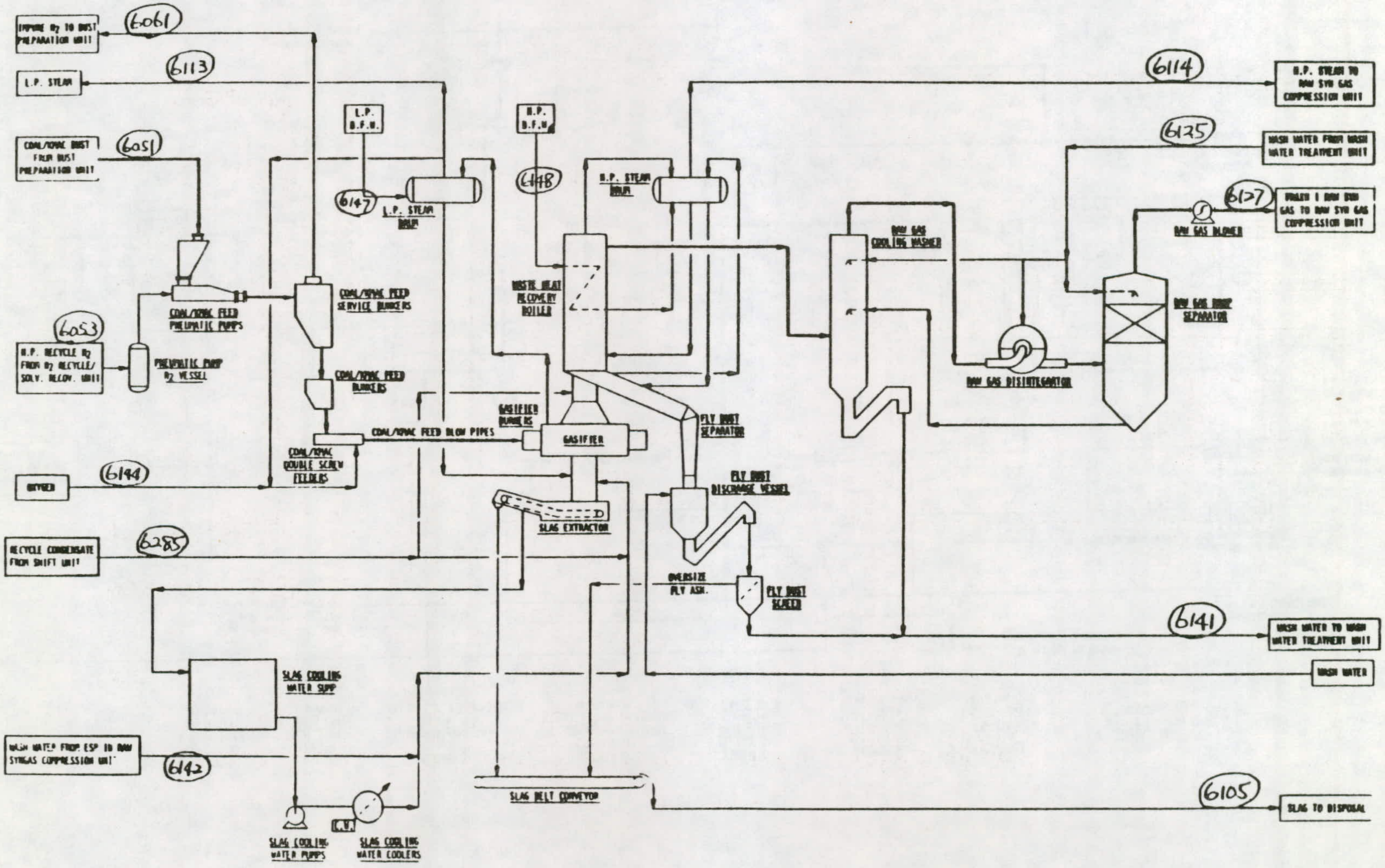


Figure 4

Wash Water Treatment Unit Process Flow Diagram

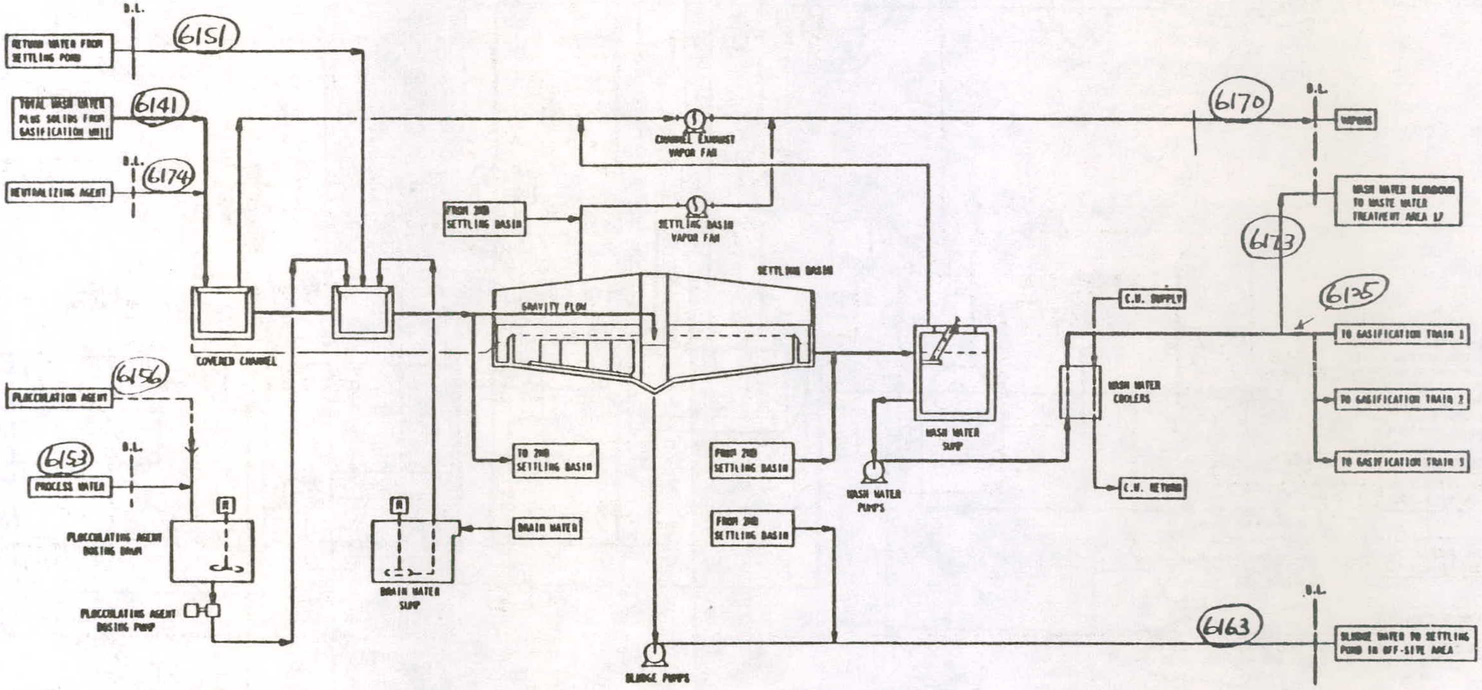


Figure 5

Raw Syngas Compression Unit Process Flow Diagram

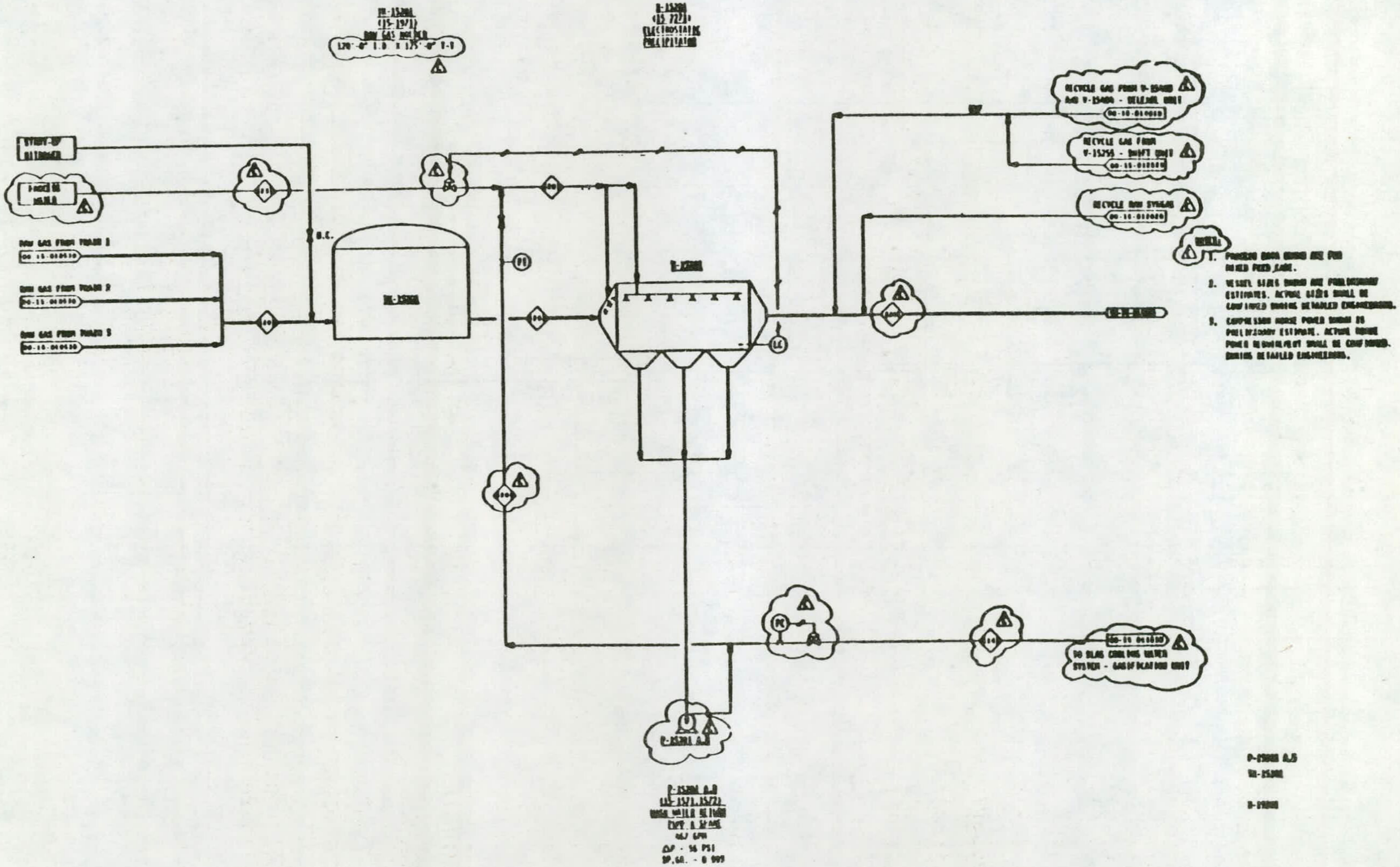


Figure 6A

Shift Process Flow Diagram

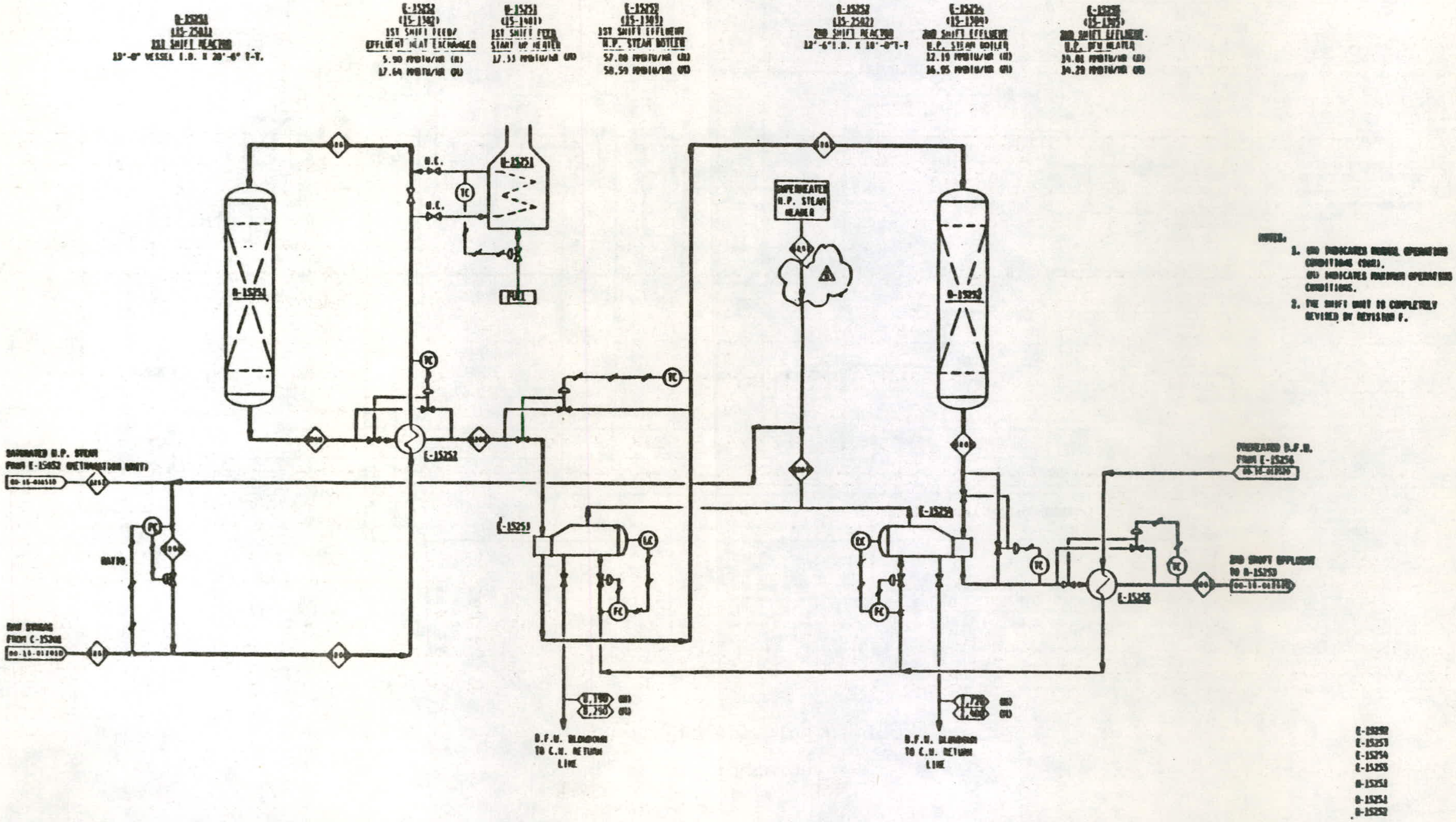


Figure 6B

Shift Process Flow Diagram

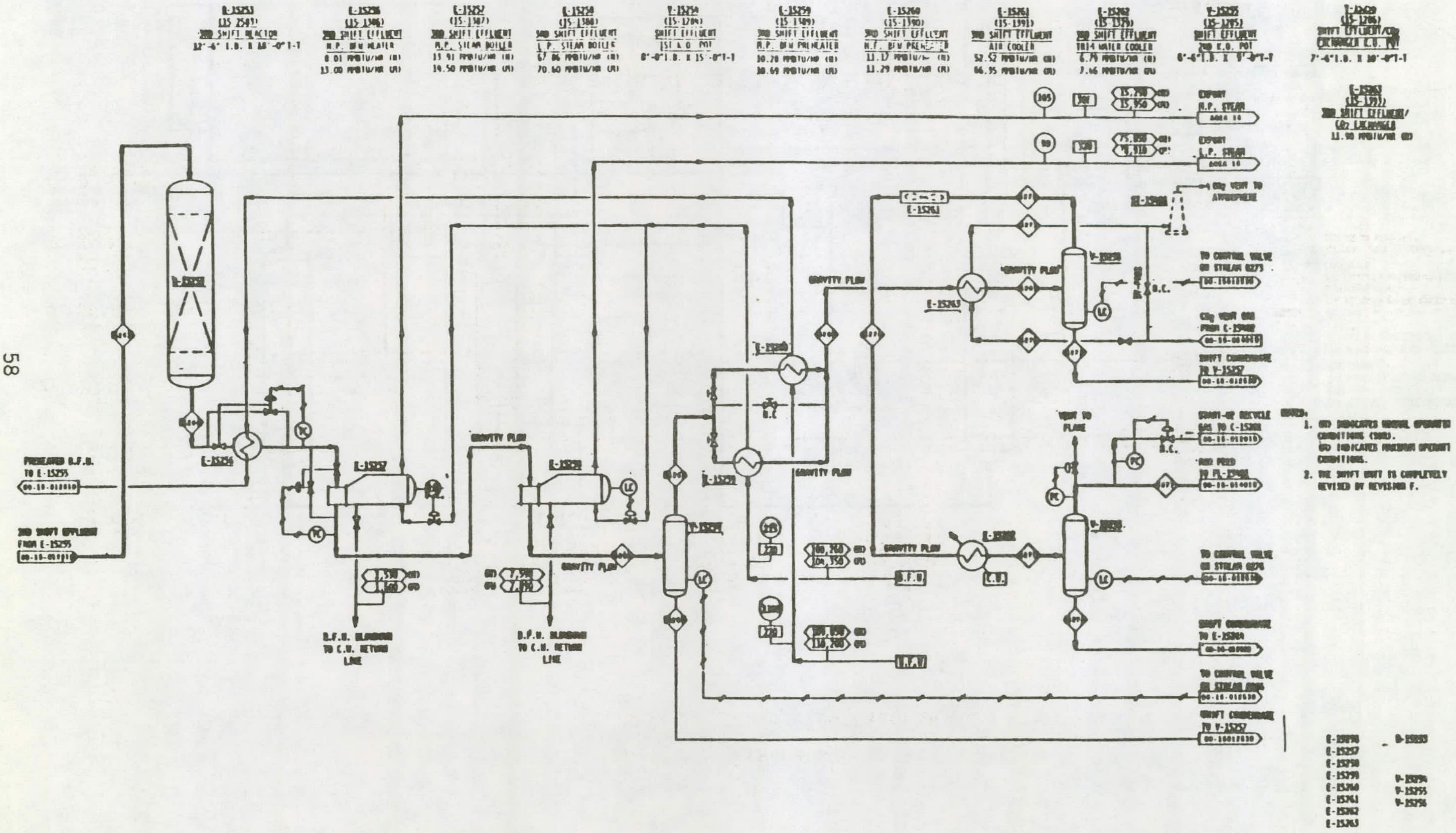
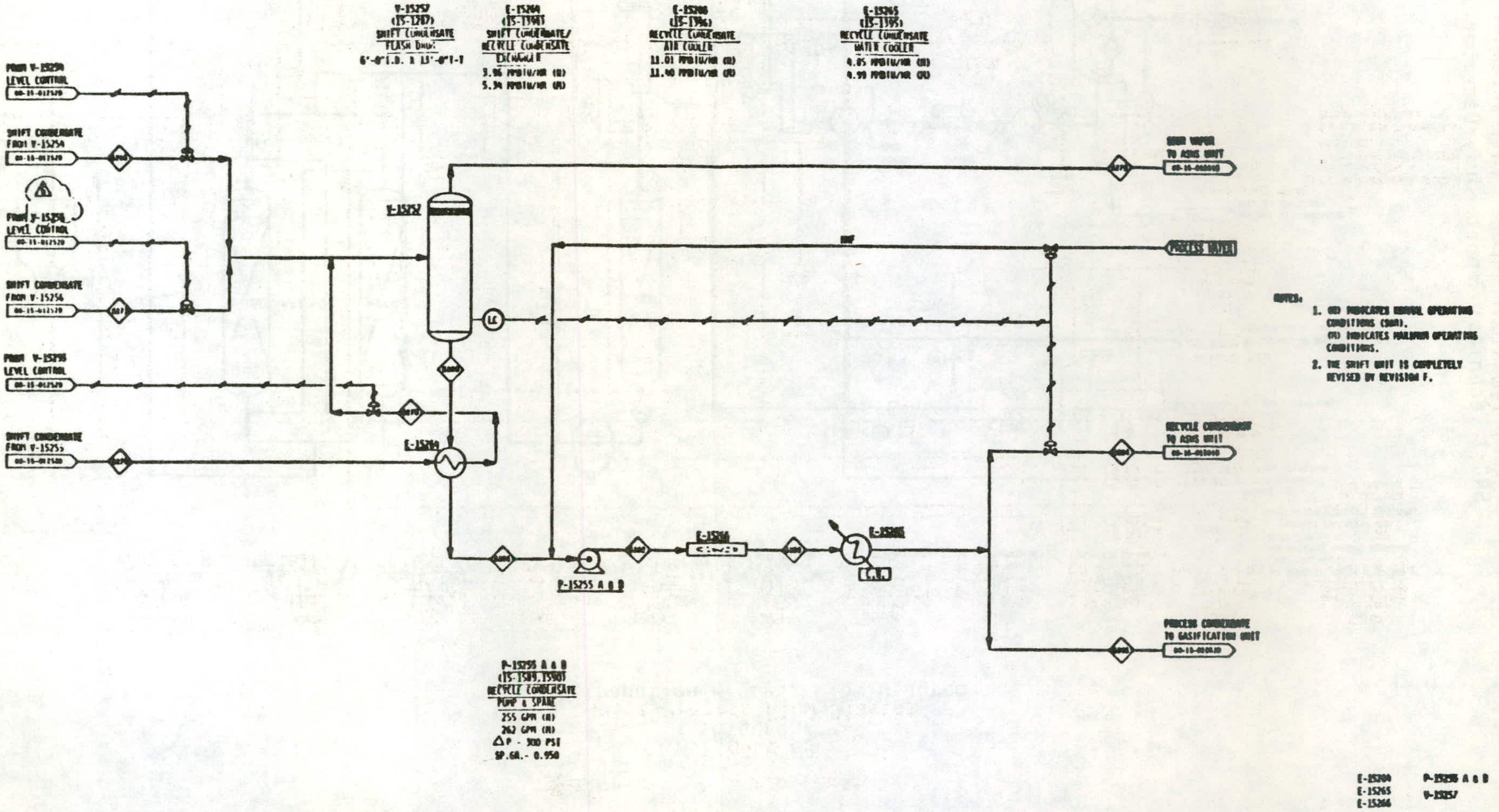


Figure 6C

Shift Process Flow Diagram



NOTES:

1. (N) INDICATES NORMAL OPERATING CONDITIONS (SOR).
   (M) INDICATES MAXIMUM OPERATING CONDITIONS.
2. THE SHIFT UNIT IS COMPLETELY REVISED BY REVISION F.

Figure 7

Methanation Process Flow Diagram

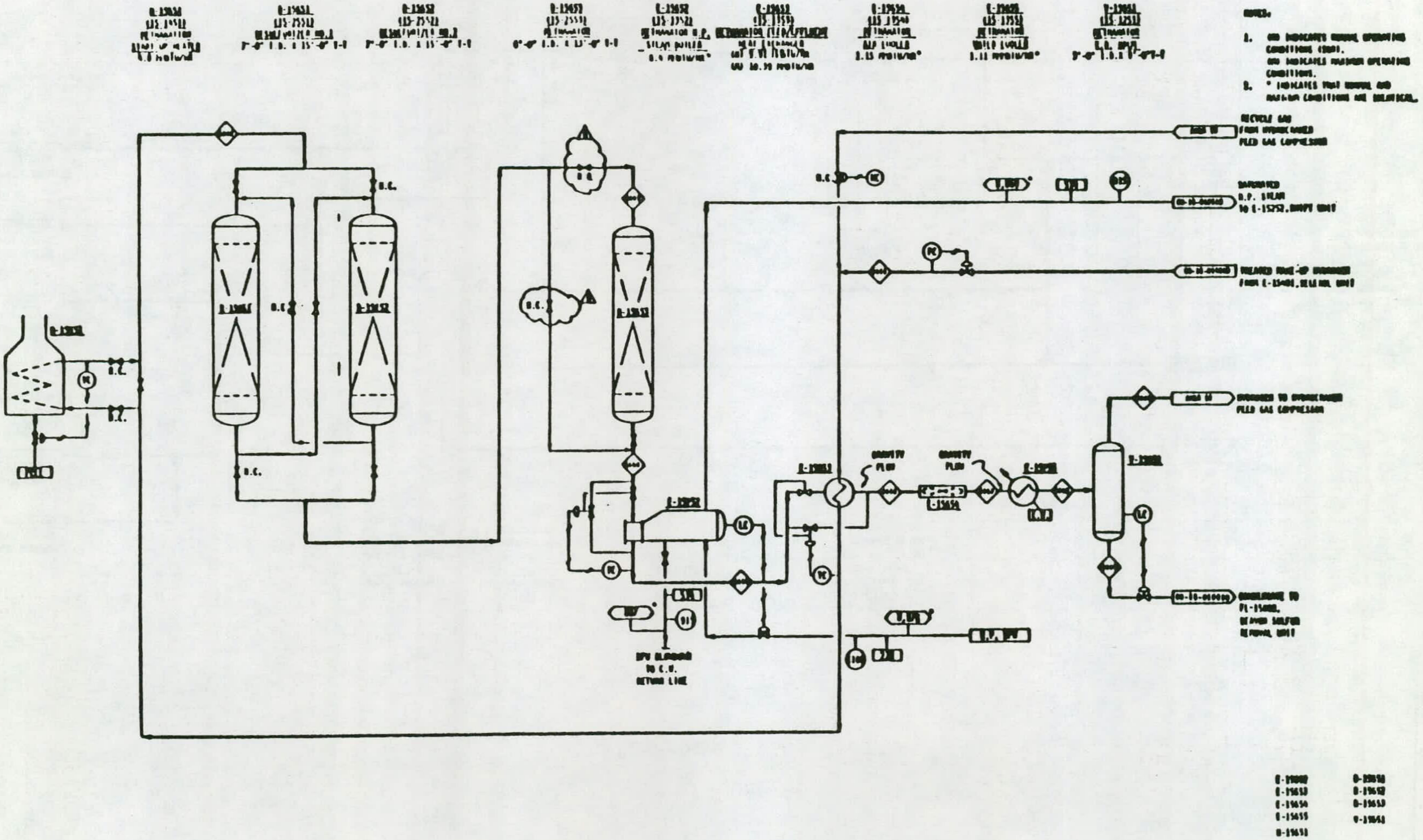


Figure 8

Selexol Process Flow Diagram

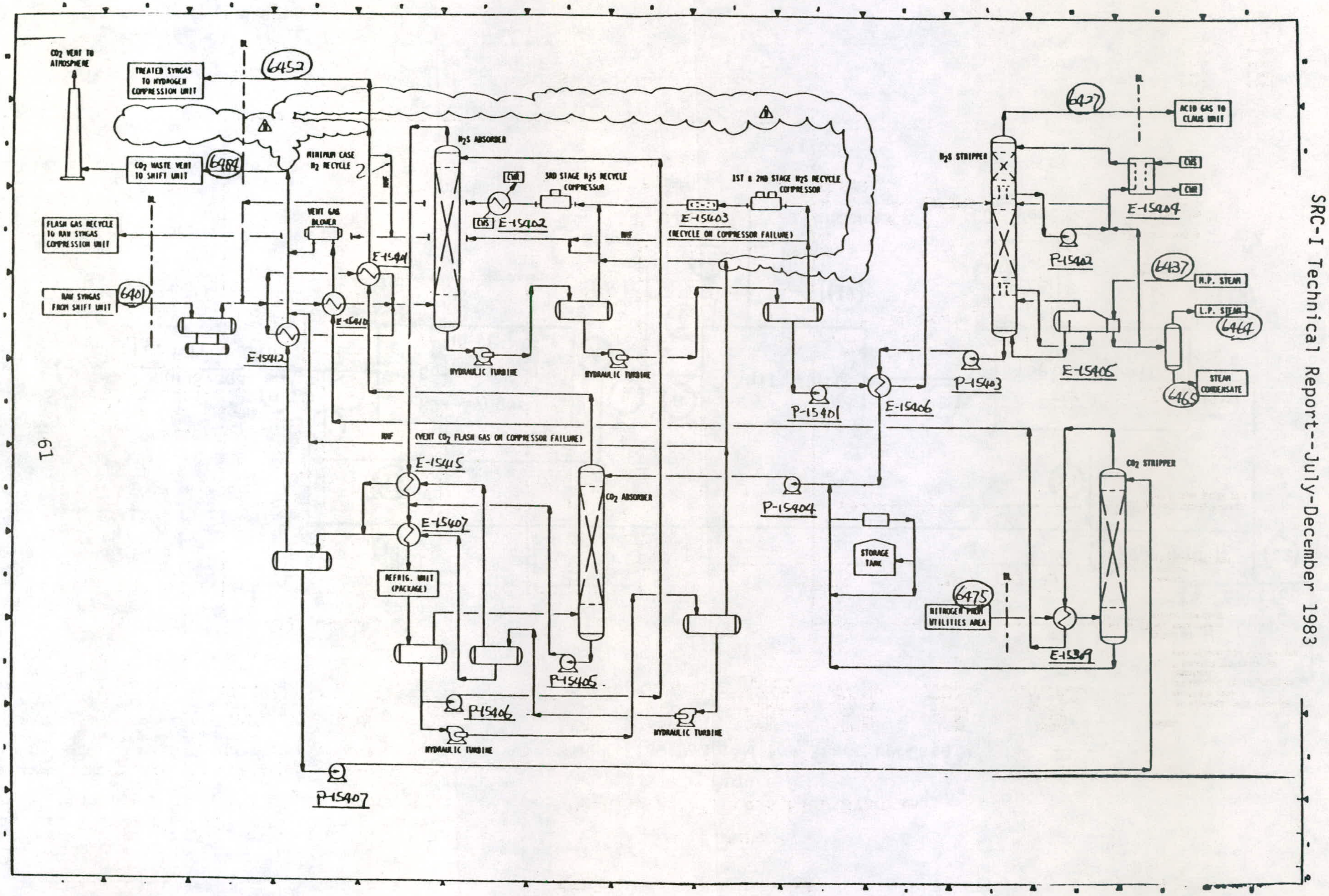


Figure 9

Block Flow Diagram for Start-up Case

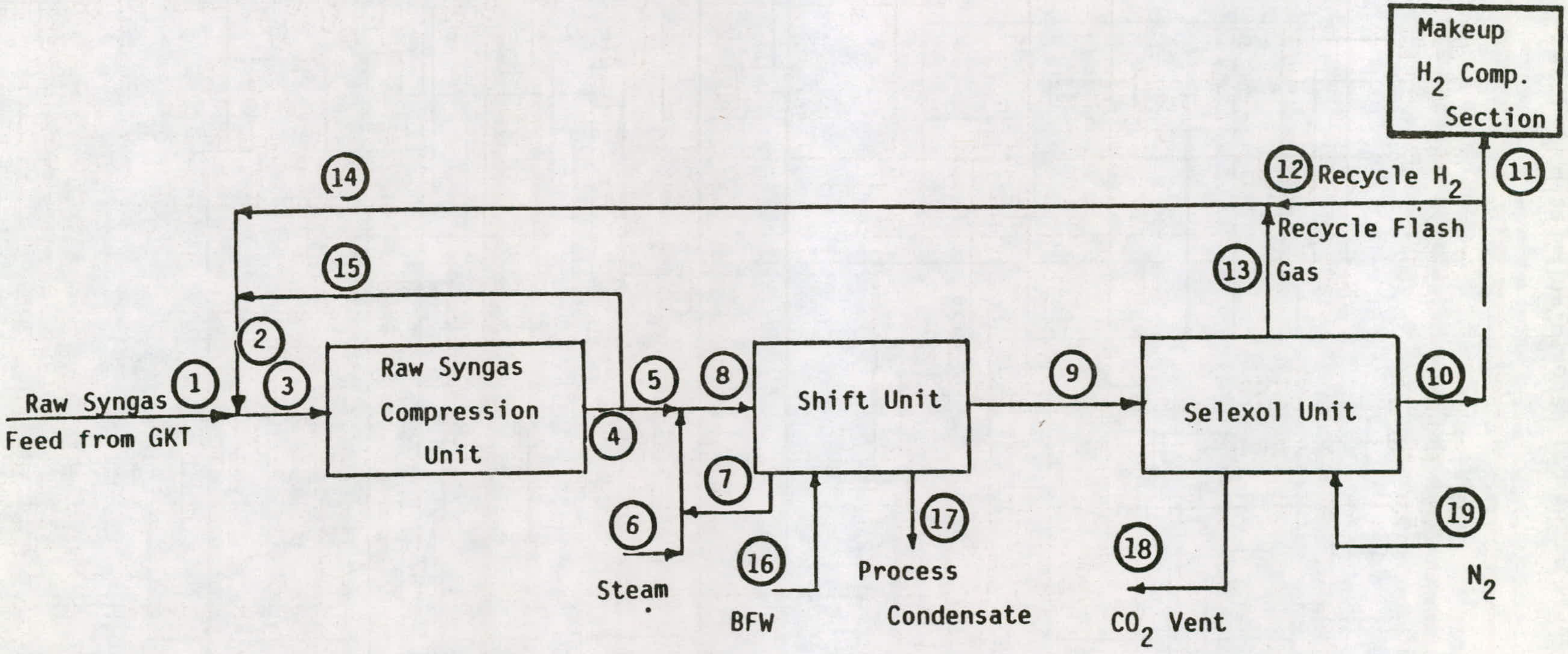
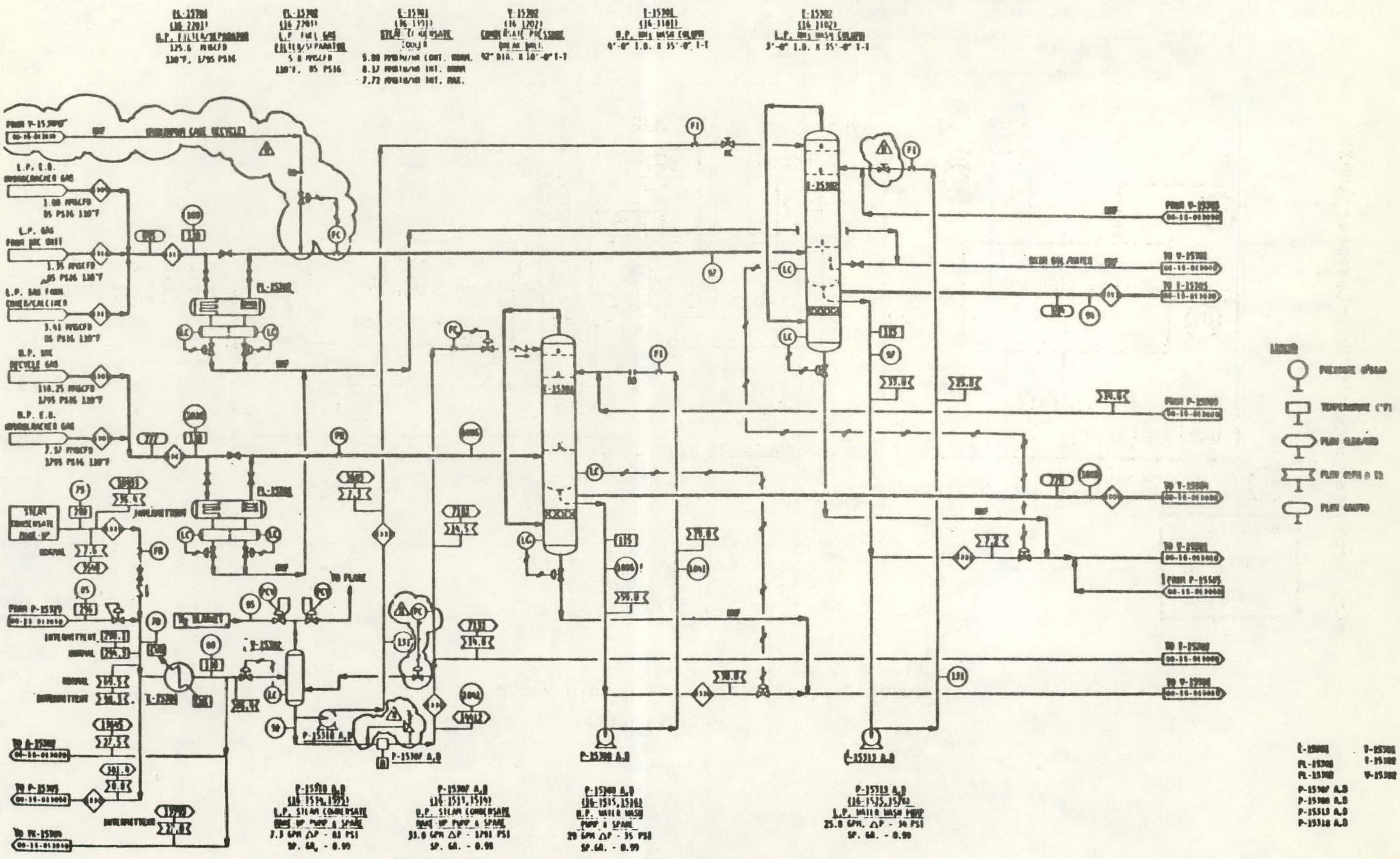


Figure 10A

Diethanolamine Unit Process Flow Diagram



`Figure 10B

Diethanolamine Unit Process Flow Diagram

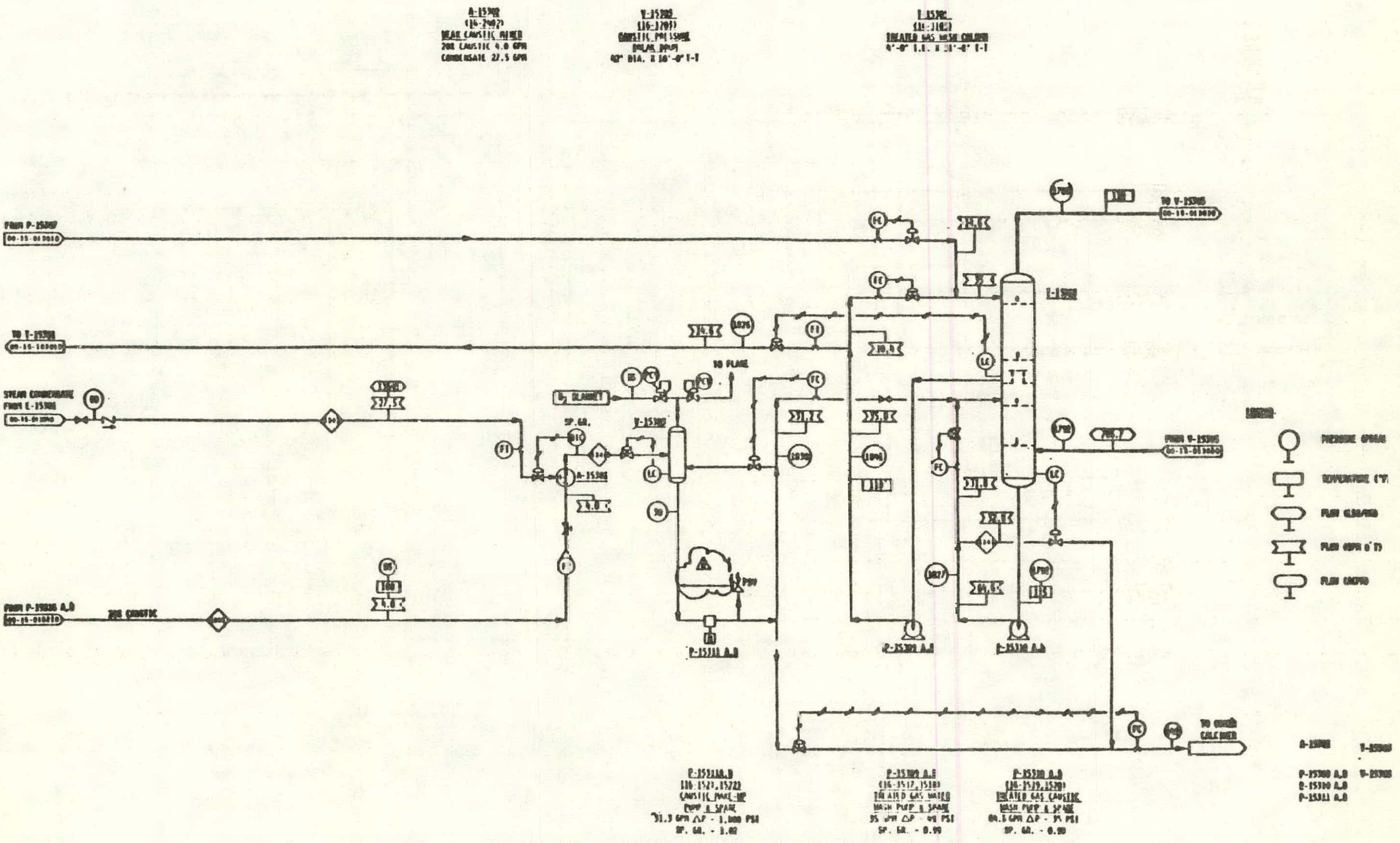


Figure 10C

Diethanolamine Unit Process Flow Diagram

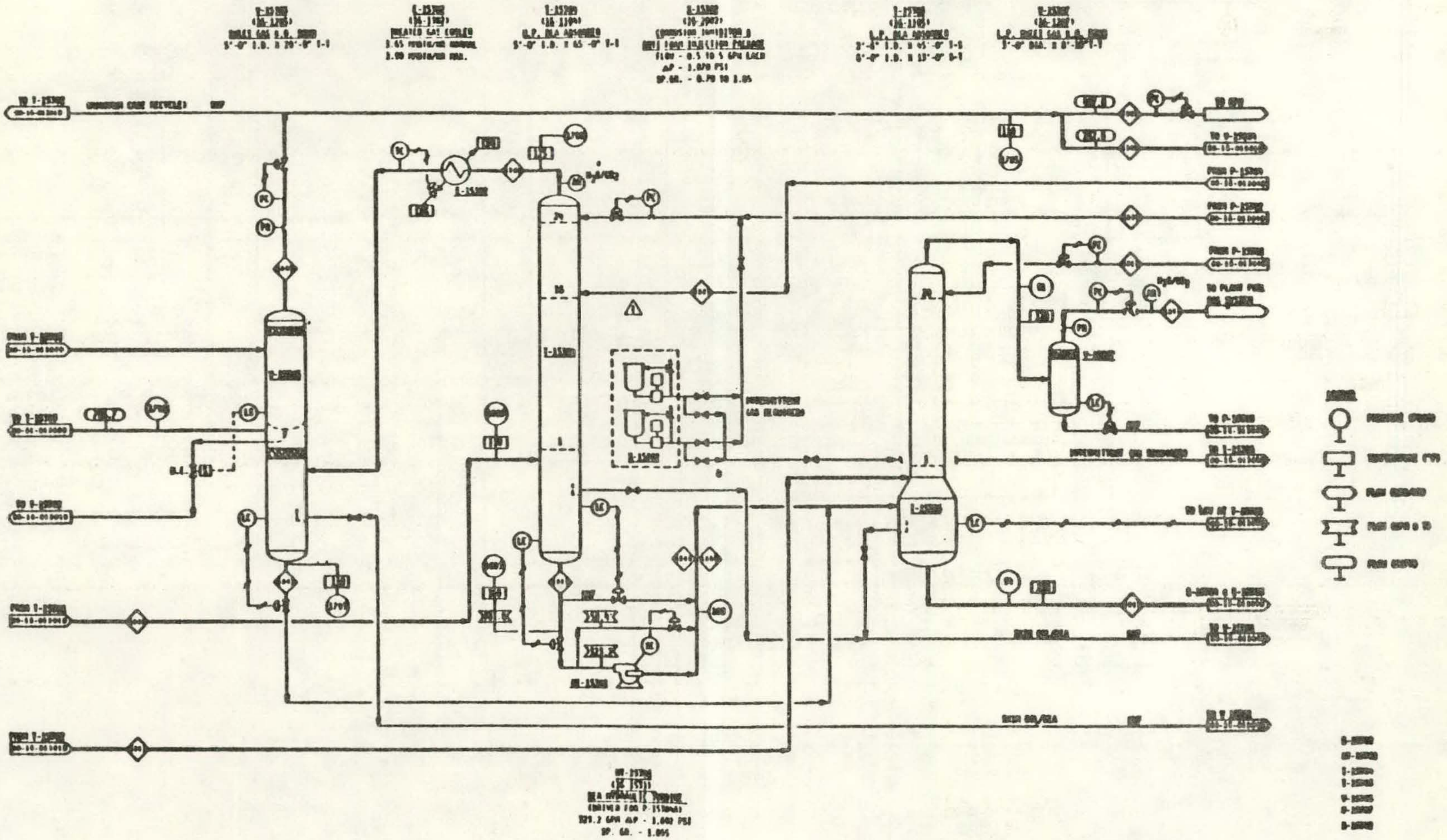


Figure 10D

Diethanolamine Unit Process Flow Diagram

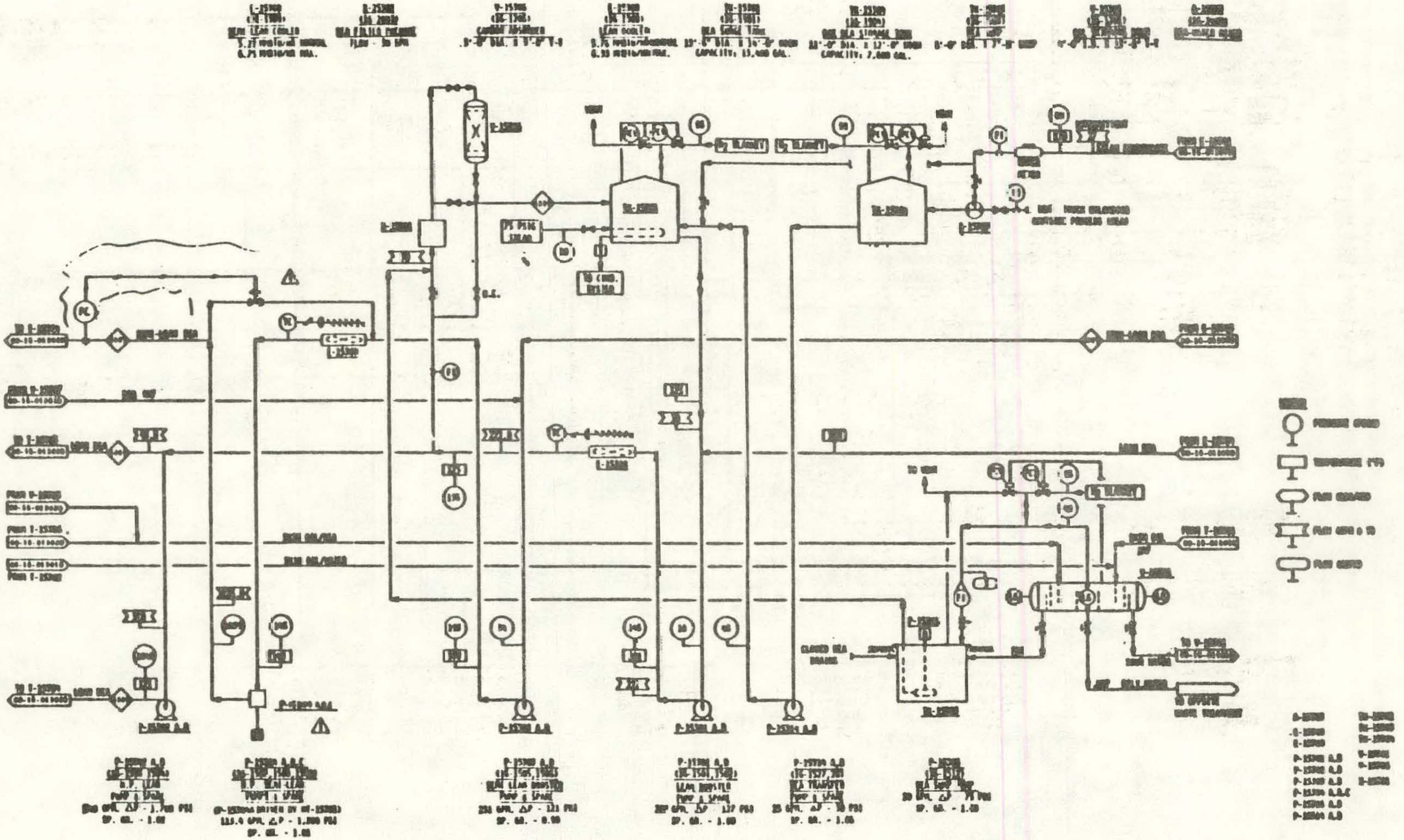


Figure 10E

Diethanolamine Unit Process Flow Diagram

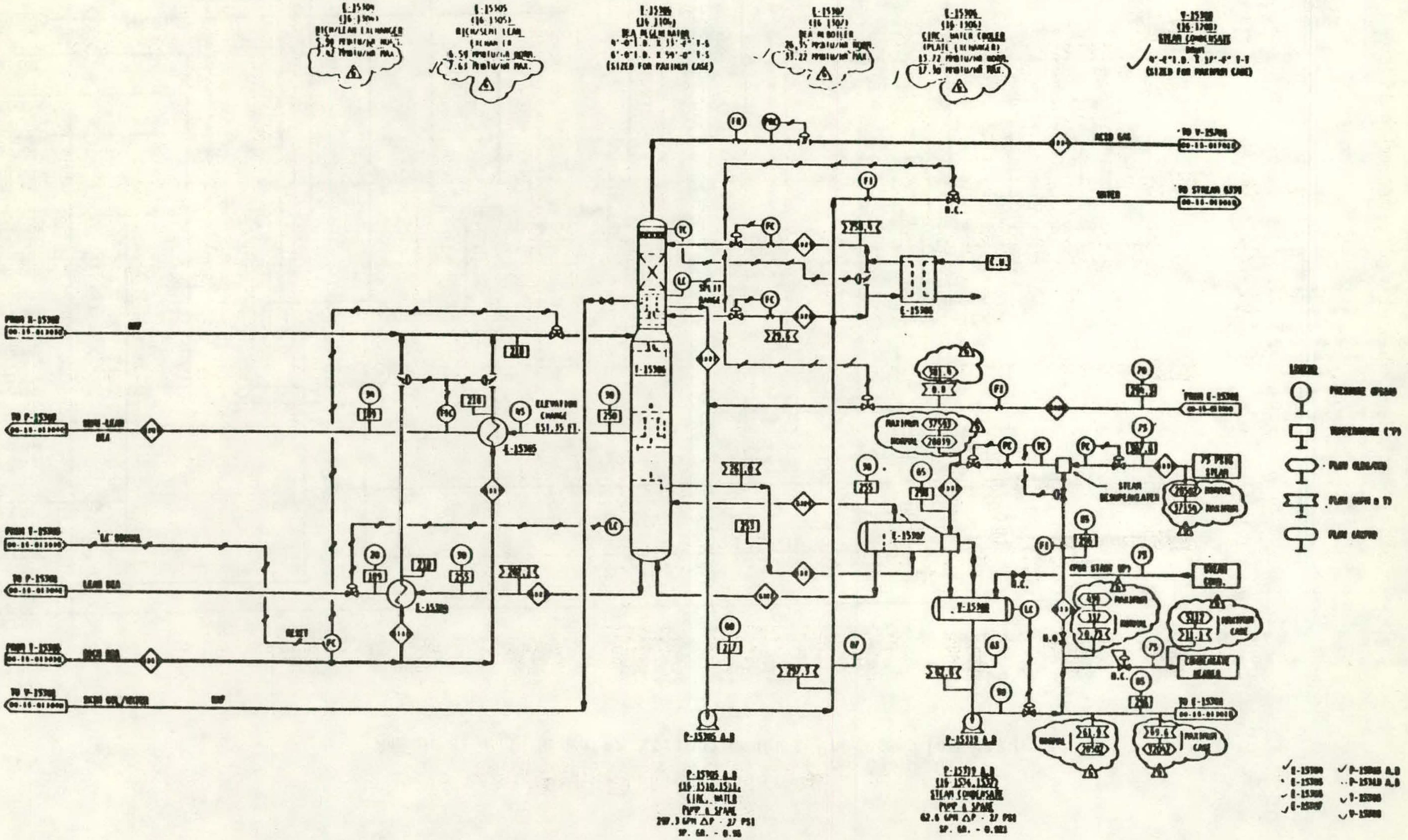


Figure 11A

Ammonia/Sulfide Water Stripping Unit Process Flow Diagram

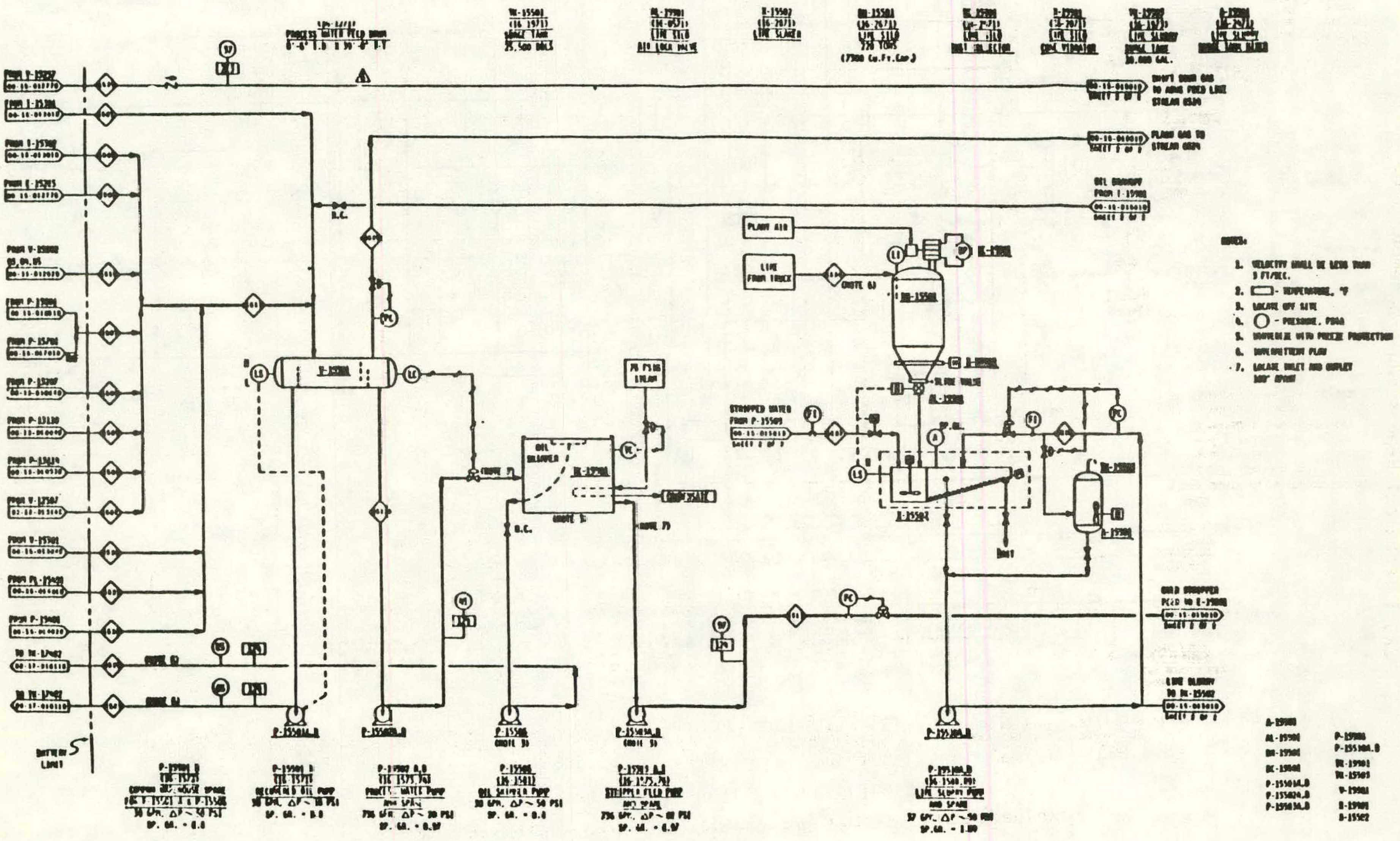


Figure 11B

Ammonia/Sulfide Water Stripping Unit Process Flow Diagram

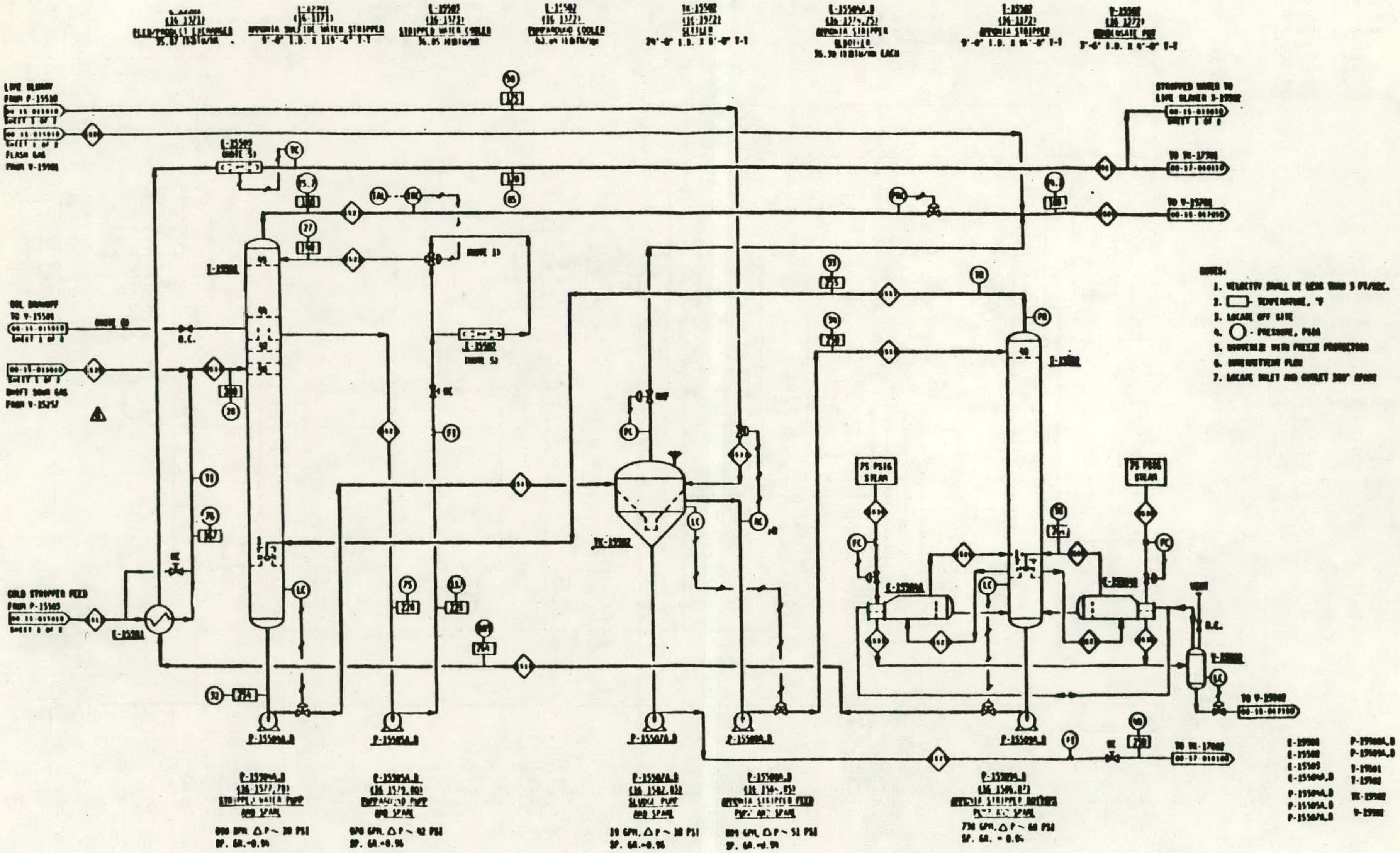


Figure 12A

Liquefied Petroleum Gas (LPG) Unit Process Flow Diagram

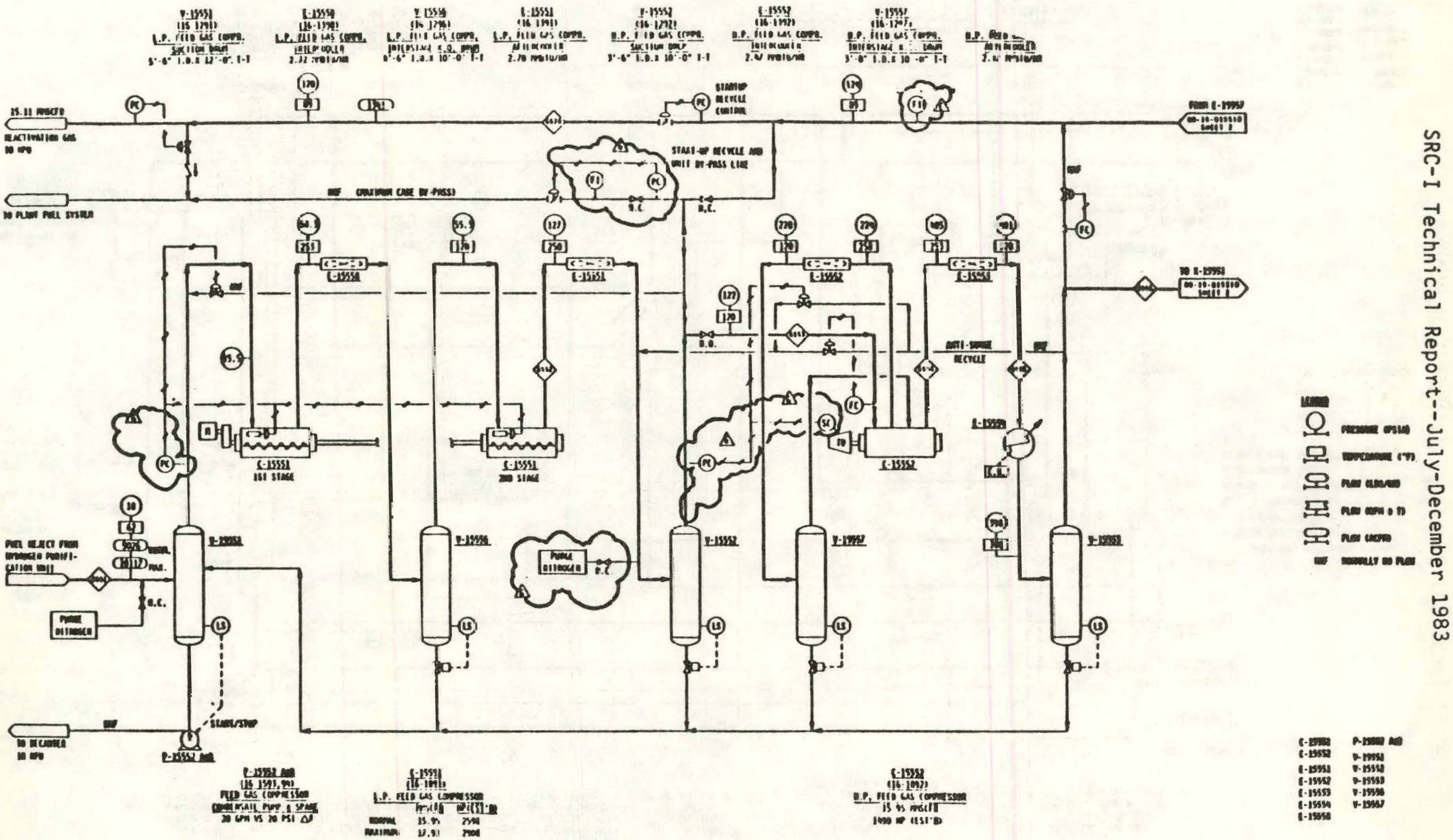


Figure 12B

Liquefied Petroleum Gas (LPG) Unit Process Flow Diagram

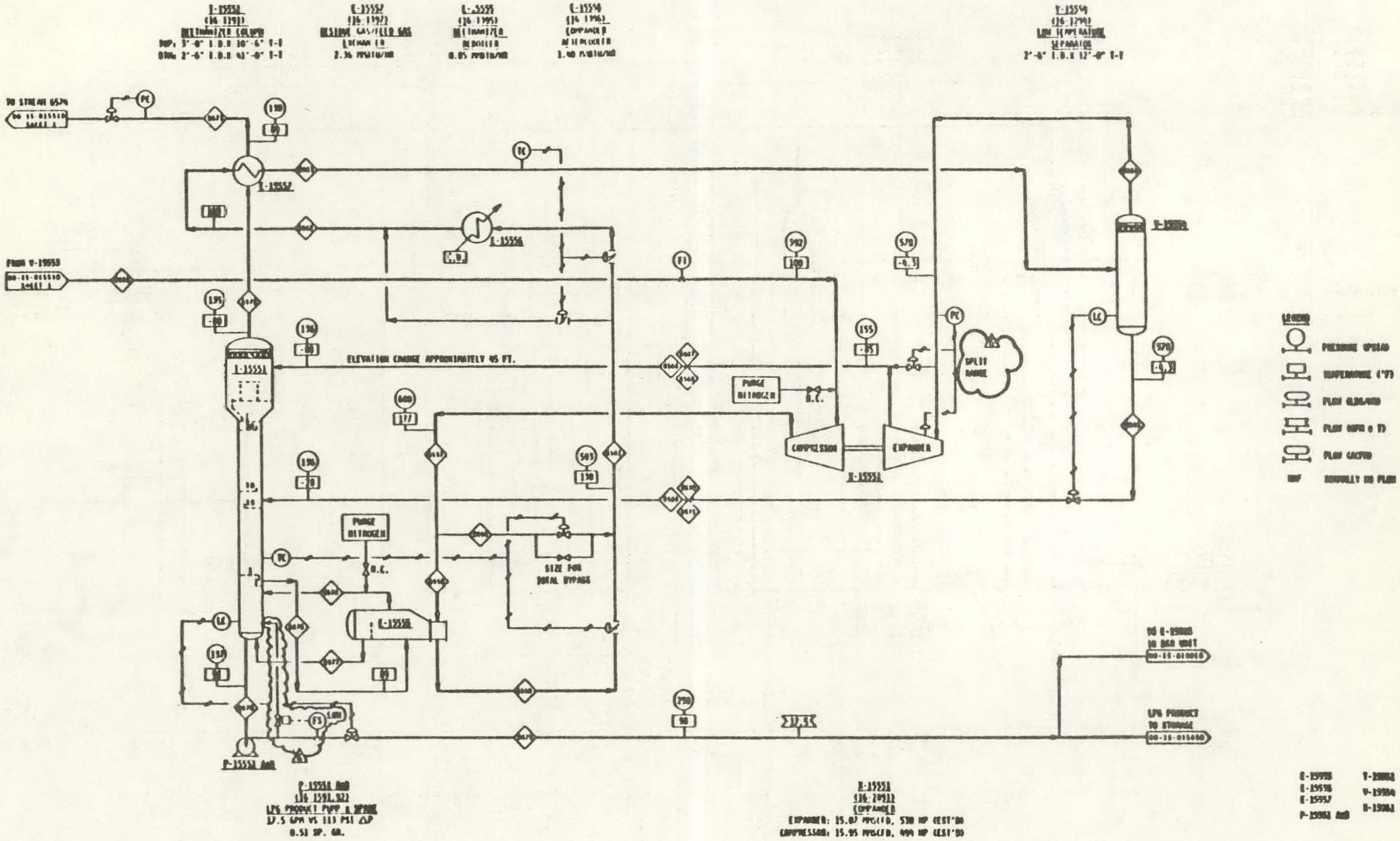


Figure 13

H_2 Compression Unit Process Flow Diagram

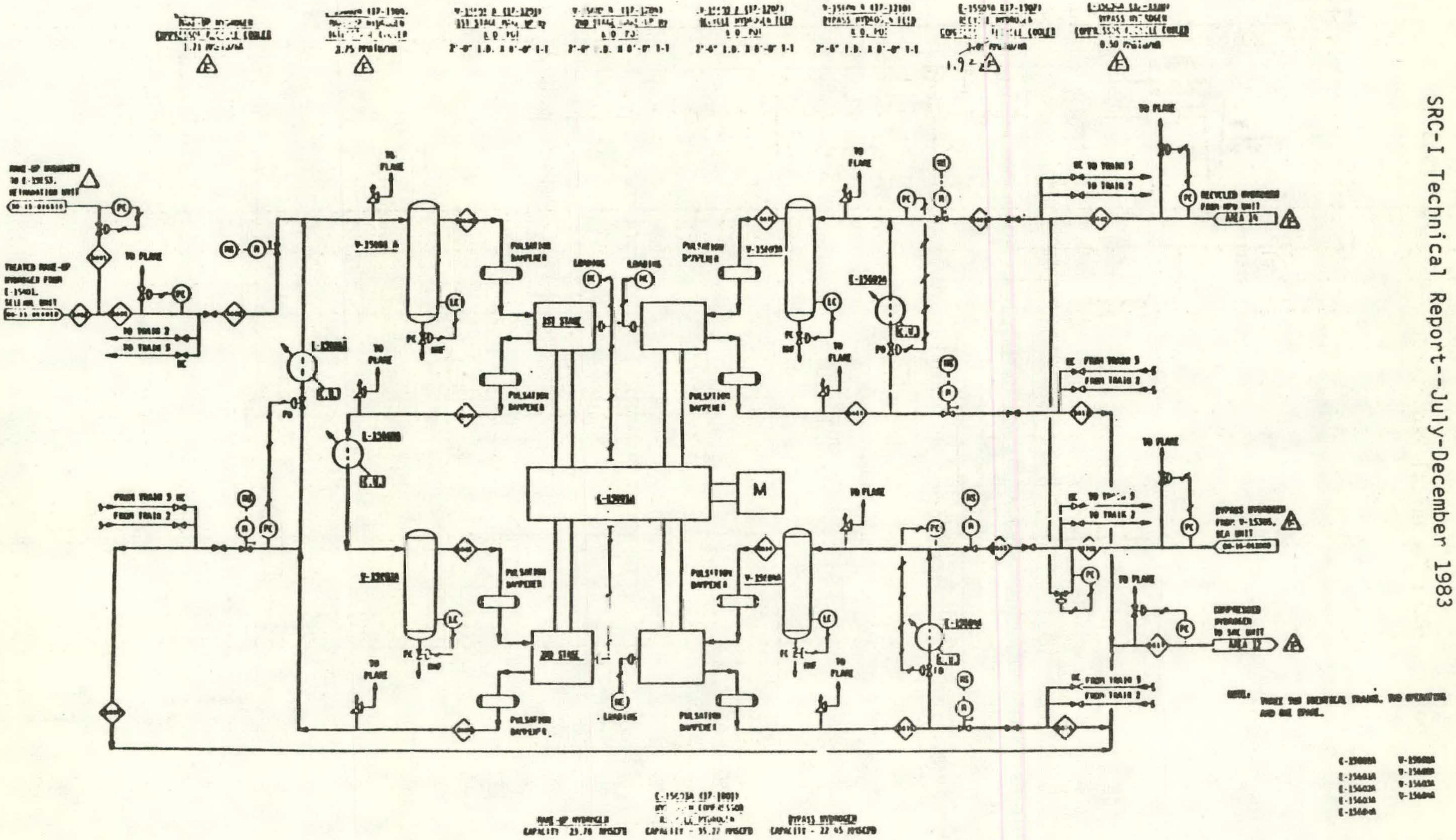


Figure 14

Claus Unit Process Flow Diagram

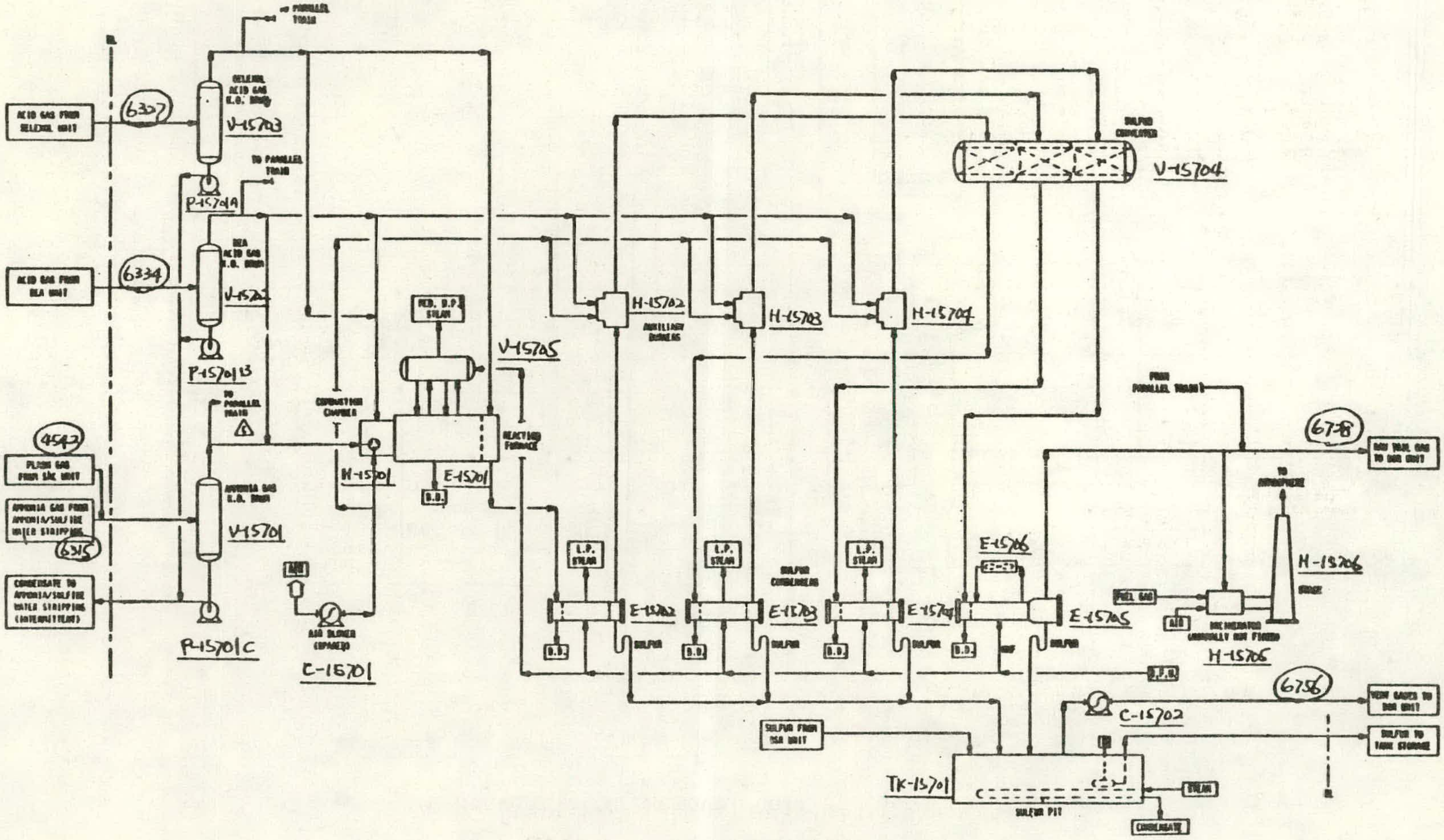


Figure 15A

Beavon Sulfur Removal Unit Process Flow Diagram

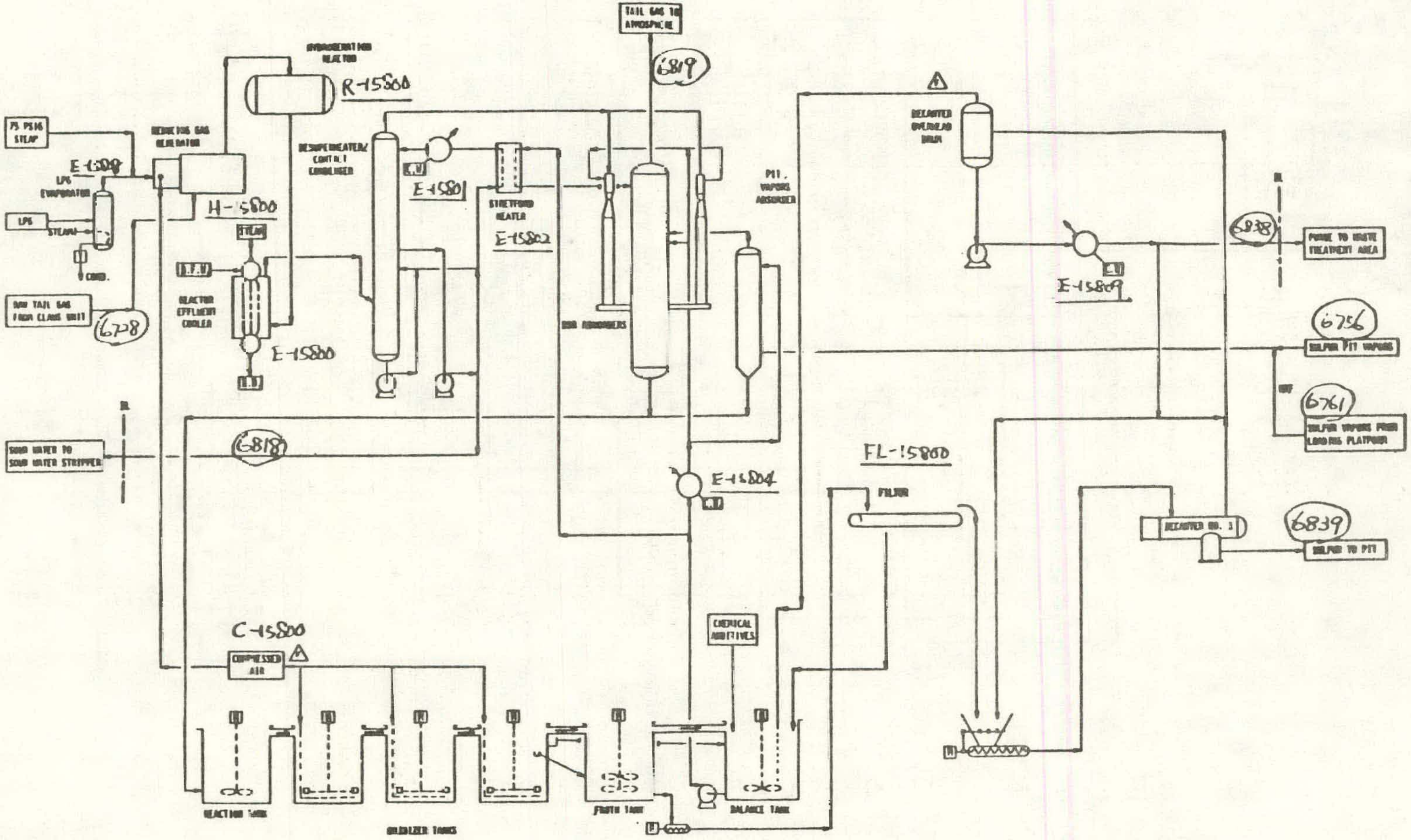
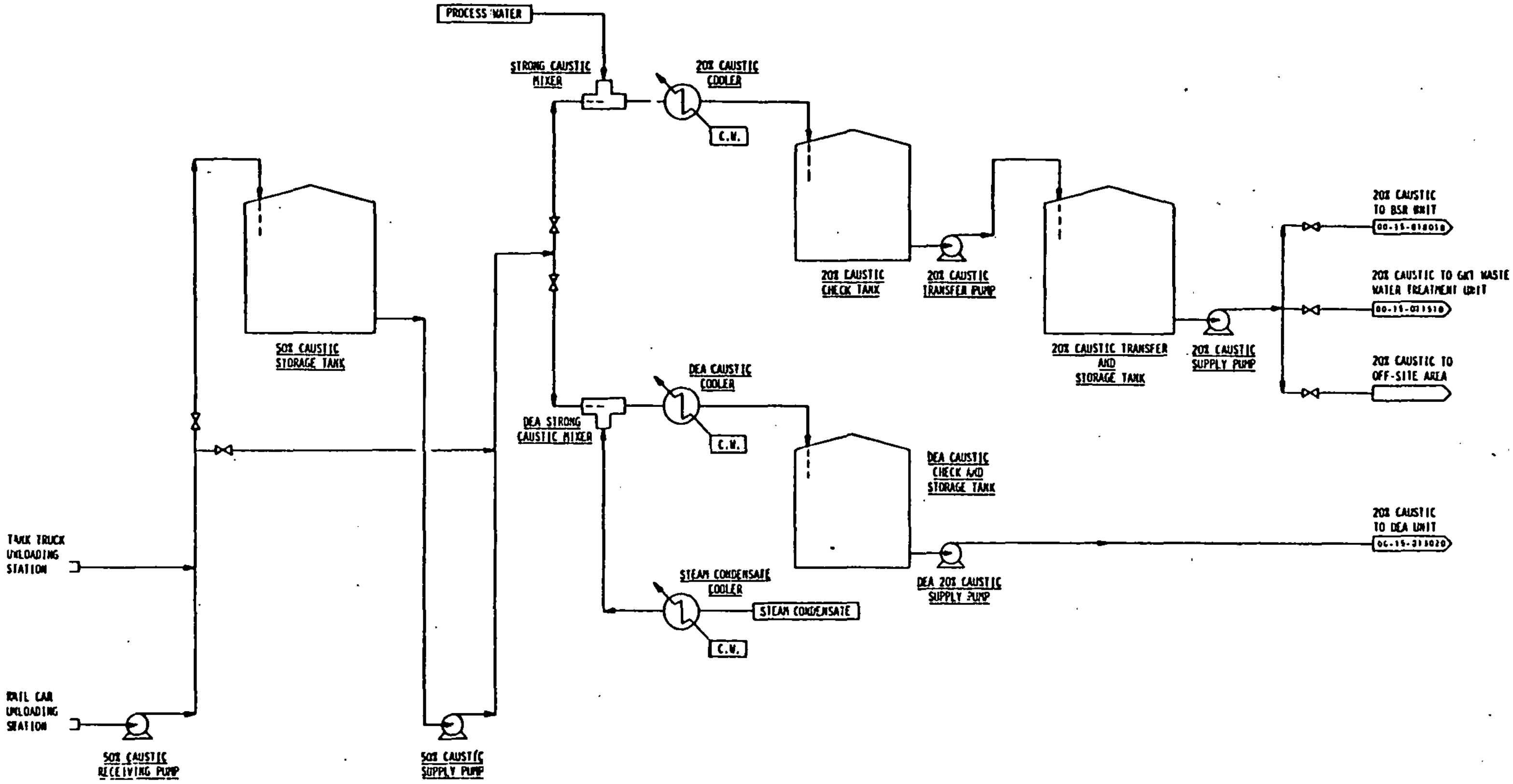


Figure 15B

Caustic Storage and Distribution



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LIQUID PRODUCT CHARACTERIZATION

R. W. Skinner,* W. W. Stawasz,** and
W. F. Tiedge**

Samples representing the various liquid streams from run 235 at the Wilsonville Advanced Coal Liquefaction Facility and of ALCOA coker condensate were fractionated into light-, middle-, and heavy-oil cuts, which were then characterized. Composite samples of all of the distillate products expected to be generated at the SRC-I Demonstration Plant were prepared from these cuts and characterized by a series of analytical tests. An alternate-boiling-range middle oil was also prepared and analyzed. In addition, middle- and heavy-oil samples were blended at various ratios with comparable petroleum liquids and aged for up to 5 months at 110°F with exposure to air. The storage stability of the original and blended samples was then determined by subjecting the aged samples to a series of analytical tests. Finally, a composite sample of naphtha was fractionated into 25°F boiling-range cuts and analyzed to determine the distribution of dicyclics and heteroatomic species.

INTRODUCTION

Effective marketing of liquid products from the SRC-I Demonstration Plant will depend on the ability of these liquids to meet the marketing specifications of potential customers, and on the compatibility of these liquids with equivalent petroleum liquids in the customers' storage tanks.

In support of International Coal Refining Company's (ICRC) Business Management Area, Corporate Research Services Department (CRSD) of Air Products and Chemicals, Inc. performed a two-year analytical research program entitled "Liquid Product Characterization Study." In FY 1981,

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*International Coal Refining Co. (ICRC).

**Corporate Research Services Department, Air Products and Chemicals, Inc. (APCI).

this program determined the physical and chemical properties of the various liquid streams expected to be generated in the demonstration plant (Tiedge and Slager, 1982). The FY 1982 study concentrated on characterizing composite samples of the total liquid product that would be generated in the demonstration plant, and included a limited program on individual liquid streams.

Most of the current (1982) program was devoted to testing the compatibility and stability of these liquids when blended with equivalent petroleum liquids. An accelerated aging procedure, by which liquids were stored at 110°F and exposed to air for up to 5 months, was used to assess the liquids' stability. Specific objectives of the 1982 program were to:

- Determine the effect upon naphtha and middle oil properties of decreasing the naphtha end point from 95% at 375°F to 95% at 350°F
- Determine the storage stability of the composite middle oil and heavy oil products
- Determine if the composite middle oil and heavy oil products are compatible with their petroleum counterparts.

To generate composite samples of naphtha and middle and heavy oils, first- and second-stage total liquid streams from the Wilsonville Advanced Coal Liquefaction Facility (run 235) and a sample of coker liquid obtained from ALCOA were fractionated, and the fractions were then blended together in a ratio representative of SRC-I Demonstration Plant yields. Because samples of some of the liquid streams were insufficient, the original FY 1982 program had to be modified somewhat. Tests that did not yield significant or relevant data were eliminated, based on evaluation of FY 1981 results.

The naphtha fraction's highly aromatic character qualifies it as a premium catalytic reformer feedstock that can be converted in high yield to an unleaded gasoline blendstock. The raw naphtha must be hydrotreated prior to reforming in order to remove nitrogen and sulfur, which are poisonous to bimetallic reformer catalysts. In addition, only a small concentration of dicyclic compounds, which form coke on the reformer catalyst, can be tolerated in the reformer feed.

The hydrotreater naphtha feed end point must be chosen so as to maximize the quantity of this valuable cut, while maintaining the dicyclics level below reformer tolerances and minimizing the quantity of heteroatoms to be removed during hydrotreating. ICRC's Baseline Design for the SRC-I plant uses a naphtha end point of 95% at 375°F. Analyses identifying the boiling-point fractions to which dicyclic and heteroatomic species report were conducted to confirm this choice. Clearly, changing the naphtha end point will also affect the middle-oil initial boiling point. In a related program, an alternate boiling range middle oil was prepared (based on a naphtha end point of 95% at 350°F) and characterized, and its storage stability was determined.

This report summarizes analyses of liquids generated during Wilsonville run 235, of naphtha and middle-oil cuts from an ALCOA coker liquid, and of composite samples prepared from these liquids. A detailed compatibility and storage stability study was performed on these liquids, and the composition and stability of an alternate middle oil are described. The naphtha end-point specification was investigated by fractionating a composite naphtha sample into 25°F cuts and analyzing them for dicyclic and heteroatomic species.

COLLECTION, FRACTIONATION, AND ANALYSIS OF COAL LIQUIDS

Collection of Coal Liquids

In November 1981, CRSD personnel travelled to the Wilsonville, Alabama pilot plant to collect samples of representative solid and liquid streams from run 235. Samples of first-stage light oil and process solvent and light oil from the low-conversion two-stage liquefaction (TSL) SRC run in progress at the time were collected in nitrogen-purged 1-gal glass bottles, wrapped with aluminum foil to exclude light. The filled containers were blanketed with nitrogen and cooled with dry ice while being transported to APCI's Linwood, Pennsylvania laboratories, where they were stored in a cold room maintained at 4°C until use. The process solvent from the low-conversion

TSL run was shipped by common carrier at a later date and also stored in the cold room at Linwood.

In December 1981, samples of light oil and process solvent from a high-conversion TSL run were gathered by Wilsonville personnel and shipped to Linwood. However, only a partially filled 5-gal pail of the high-conversion solvent was available from run 235, which was insufficient to complete all of the technical objectives outlined in the Technical Proposal for the FY 1982 study.

A sample of coker condensate oil from ALCOA run 50 was received by Air Products in September 1981.

Fractionation of Coal Liquids

Wilsonville Run 235 First-Stage Liquid Distillation. The Wilsonville run 235 front-end total distillate product was reconstituted by blending 57.3 wt % light oil with 42.7 wt % process solvent, as per the Wilsonville material-balance data. This blend was fractionated into naphtha, middle-oil, and heavy-oil cuts in the 50-L still using the procedure described in the FY 1981 final report (Tiedge and Slager, 1982). The naphtha cut was blended to an end point of 375°F at 95% distilled, and the middle oil was blended to an end point of 640°F at 90% distilled by Engler distillation. The heavy oil was taken as the still bottoms remaining after blending to the middle-oil end point. In order to achieve the middle oil end point, cuts up to an equivalent head temperature of 710°F had to be blended, leaving an insufficient amount of heavy oil to complete the FY 1982 program. This distillation was then repeated in order to obtain additional heavy oil. The heavy oils from these two distillations were blended together to produce a heavy-oil sample labeled 2076CB. Table 1 lists the cuts obtained from these distillations, and Table 2 summarizes the distillation results.

Wilsonville Hydrotreater Unit (HTU) Run 235 Distillation. All of the limited amount of process solvent available from high-conversion (~50%) HTU Run 235 was used to produce a blend containing 34 parts of 1994CB (HTU high-conversion light oil) and 66 parts of the high-conversion process solvent (1990CB), as per the Wilsonville material balance. This blend was fractionated into naphtha, middle-oil, and heavy-oil cuts in the 5-L still using the previously described procedure. The naphtha cut was blended to an end point of 375°F at 95% distilled and the middle oil was blended to an end point of 640°F at 90% distilled by Engler D-86 distillation. The heavy oil was taken as the still bottoms remaining after blending to the middle-oil end point. Since insufficient naphtha was obtained from this distillation to complete the FY 1982 program, additional light oil (1994CB) was distilled in a 5-L, packed-column glass distillation apparatus, using the procedure described in Tiedge and Slager (1982). The naphtha cut was blended to an end point of 375°F at 95% distilled by Engler distillation, and the still bottoms were discarded. The naphtha cuts from the two distillations were combined to form a single naphtha cut labeled 2053CB. Table 1 lists the fractions, and Table 3 summarizes the results of these distillations.

Wilsonville HTU Run 235 Low-Conversion Distillation. The program originally included analytical characterization only of the naphtha fraction from the low-conversion (~30%) HTU run 235. However, when it became evident that insufficient middle oil from the high-conversion run was available to complete the program, we decided to substitute the low-conversion middle oil.

The light oil (2020CB) was distilled in the 5-L glass still by the previously described procedure to obtain the naphtha cut, which was blended to an end point of 375°F at 95% distilled by Engler distillation. The still bottoms were later blended with process solvent (2041CB) to achieve a ratio of 34% light oil to 66% process solvent. This blend was then distilled twice in the 5-L glass still to obtain the middle-oil cut. The cuts from both distillations were combined, and we attempted to blend the middle oil to an end point of 640°F at 90% distilled by Engler distillation. However, severe foaming that could not be overcome resulted from use of Engler D-86 distillation, so this cut was blended to the specified end point by using the gas chromatographic simulated distillation D-86 correlation as a guide. Since the correlation was not exact, the D-86 correlation results from analysis of the high-conversion HTU middle oil were used as a guide. The still bottoms remaining after blending were taken as the heavy-oil cut. Table 1 lists the cuts obtained, and Table 4 summarizes results.

ALCOA Coker Liquid Distillation. The entire sample of coker condensate oil obtained from ALCOA (run 50, 1970CB) was fractionated into a naphtha and middle-oil cut in a 4-L, packed-column glass distillation apparatus using the previously described procedure. The heavy-oil fraction was not prepared, since the demonstration plant will have a zero net yield of coker heavy oil. The naphtha cut was blended to an end point of 375°F at 95% distilled by Engler distillation. All of the overhead cuts, up to an atmospheric equivalent head temperature of 775°F, were blended into the middle-oil cut; we were only able to achieve an end point of 622°F at 90% distilled by Engler distillation. The remaining still bottoms (31.1% of the charge), resembling SRC or partially coked SRC, were discarded. Table 1 lists the fractions generated, and Table 5 summarizes results.

Alternate Middle-Oil Distillation. Characterization of an alternate middle oil generated by lowering the naphtha end point to 350°F at 95% distilled was added to the FY 1982 program. A shortage of coker naphtha necessitated substituting first-stage SRC naphtha for coker naphtha using ICRC's Baseline Design yield structure. The coker naphtha is only 4.5% of the demonstration plant naphtha product.

A blend of 77.8% first-stage run 235 naphtha (2007CB) and 22.2% high-conversion HTU run 235 naphtha (2053CB) was distilled in two charges to a 5-L glass still (using the previously described procedure), and cuts from both distillations were combined. The naphtha cut was then blended to an end point of 350°F at 95% distilled by Engler distillation, and the remaining still bottoms were saved to be blended with a composite middle-oil sample, yielding the alternate middle oil. The composite middle-oil sample did not contain coker liquid middle oil; it was prepared by blending 61.9% first-stage middle distillate (2008CB) with 38.1% low-conversion HTU middle distillate (2024CB). The still bottoms were blended with the composite middle oil to yield the "alternate" middle oil. Unfortunately, cuts 4, 5, and 6, representing a head temperature from 380 to 410°F, were not mixed in with the still bottoms before the weight calculation and blending were performed. Therefore, the alternate sample tested under this program (2084CB) does not represent the true composition of the intended alternate middle oil. Table 1 lists the samples generated and Table 6 summarizes the distillation data.

Naphtha End-Point Distillation. To investigate the boiling-point distribution of heteroatomic and dicyclic species in the naphtha cut, a composite naphtha sample was fractionated into 25°F boiling-range cuts for analysis. Again, first-stage liquid naphtha was substituted for the coker liquid naphtha in the composite. The composite was prepared by blending 77.8% first-stage naphtha (2003CB) with 22.2% HTU high-conversion run naphtha (2053CB). The distillation was carried out in a glass, packed-column still of about 15 theoretical plates. The first cut was taken from the initial boiling point to a head temperature of 325°F, and consisted of 58.5% of the charge. The initial portion of the cut was taken at a 1:1 reflux ratio, which was increased to 5:1 and finally 10:1 as the 325°F head temperature was approached. The reflux ratio was maintained at 10:1 for the remaining cuts. Table 1 lists the samples, and Table 7 summarizes the distillation data.

Sample Analyses

Naphtha Analyses. (1) Wilsonville Run 235 First-Stage Naphtha (2003CB). Only limited analytical work was performed on this sample because two complete sets of data were generated in the FY 1981 program. Comparison of 1981 and 1982 data indicates that the samples are similar in composition and properties. Run 235 naphtha has more sulfur and oxygen and less nitrogen than last year's naphtha from runs 220 and 225. Although the oxygen content of run 235 naphtha is higher, the hydroxyl content, as determined by a near-infrared procedure, is lower. Since the complete IR spectrum was not examined, we do not know what other functional groups account for the higher oxygen content.

Properties such as the API gravity, smoke point, and existent gum strongly resembled those of last year's samples. The existent gum, at 83 mg/100 mL, is still quite high, and compares to values of 47 and 107 mg/100 mL measured in 1981. Analytical results are listed in Table 8.

(2) Wilsonville Run 235 HTU Naphtha, High and Low Conversion (2053CB, 2021CB). A complete set of analytical and physical property data for these two samples is listed in Table 8. There were no HTU samples in the FY 1981 program to compare these data with. The data support the hypothesis that the hydrocracker conversion level does not

affect the product properties of a given boiling-point cut, except for relatively minor changes in aromaticity. We found that the low- and high-conversion samples were quite similar, except for slightly more aromatics in the low-conversion sample.

The NMR results, along with physical properties such as a higher specific gravity and lower smoke point and higher carbon-to-hydrogen ratio, substantiate the conclusion that the low-conversion sample is more aromatic. Gas chromatographic/mass spectroscopic (GC/MS) identification showed that the 20 largest chromatographic peaks are essentially the same for both samples, and the components are those expected for a hydrotreated product. Toluene is the largest component at 15% in the low-conversion naphtha, compared to only 7.4% in the high-conversion sample, further confirming the more aromatic nature of this sample. The high-conversion naphtha has slightly less N, O, and OH (as measured by NIR); however, the phenol content measured by GC is higher than that of the low-conversion naphtha. The gravimetrically measured phenol content is significantly higher in high-conversion naphtha, and does not compare well with the GC results. This could be because an acidic species was extracted and thus was not chromatographed, or because a volatile part of the sample was lost during extraction. In any case, the phenol concentrations for both samples were low enough that none showed up in the GC/MS list of the 20 major components.

Both HTU samples have about the same nitrogen content as the first-stage naphtha; however, the oxygen and sulfur contents are an order of magnitude lower for the HTU samples, which also contain 1 wt % more hydrogen. The amount of existent gum in the HTU samples is 6-29 mg/100 mL, which is significantly less than we have observed for any first-stage naphtha sample, but still quite high.

(3) ALCOA Coker Liquid Naphtha (1996CB). Because of the limited amount of coker liquid available, this program was reduced to 11 tests, the results of which are listed in Table 8. GC/MS analysis of this sample shows it to be somewhat less complex and more aromatic than first-stage SRC naphtha. About 73% of the sample is represented in the 20 largest chromatographic peaks. Of this amount, phenols constitute about 18%, which compares well with the total phenols measured by GC and

gravimetric analysis (20.1 and 18.8%, respectively). The specific gravity, carbon-to-hydrogen ratio, and NMR and IR spectra all confirm the highly aromatic nature of this naphtha.

These data strongly resemble the GC/MS, elemental, NIR, IR, and phenol results obtained on an ALCOA coker naphtha sample in the FY 1981 program. The FY 1981 sample had an abnormally high level of chlorine at 220 ppm, thought to be due to contamination. However, the current sample of coker naphtha had only 18 ppm Cl, which is below the level of concern.

The coker naphtha is very similar in composition to the first-stage SRC naphthas studied in this and the FY 1981 program. A major difference is that the coker naphtha contains an order-of-magnitude lower sulfur concentration.

(4) Composite Naphtha Analysis (2077CB). The FY 1982 program called for characterization of a composite naphtha sample, representing the total liquid product of the SRC-I Demonstration Plant. Such a sample was prepared for analysis by blending the three liquid products, as per the demonstration plant Baseline yield structure, in the ratio 4.5 parts of coker naphtha (1996CB), 22.0 parts of high-conversion HTU naphtha (2053CB), and 73.5 parts of SRC first-stage naphtha (2003CB) by weight. Complete analytical and physical property characterization data are listed in Table 8.

In general, the data reflect what would be expected from a linear averaging of component properties for this composition. Significantly, the existent gum follows the averaging rule. The API gravity appears to be out of line; however, the specific gravity determined by the more accurate pycnometric procedure is in the correct range. Two values are reported for the heating value; one was determined by APCI and the other by Huffman Laboratories in Wheat Ridge, Colo. The values are within 60 Btu/lb, which is within accepted interlaboratory repeatability range. The GC/MS chromatogram is quite complex, as might be expected from a blend of this nature. The complexity of the chromatogram, including some apparently unresolved peaks, leads to difficulties in mass spectral interpretation and in assigning a ranking to the 20 largest peaks. The 20 largest peaks listed reflect the composition of this composite blend.

The existent gum level of the composite is very high, reflecting the values obtained for the components. The copper strip corrosion rating is surprisingly good, indicating that this sample is not corrosive to copper. The corrosion rating of the run 235 naphtha was not determined; however, the two first-stage naphtha samples in last year's program had a rating of 4B, which is extremely corrosive. Although the HTU naphtha used in this blend had an excellent rating of 1A, the amount of this material used in the composite (22%) could not be expected to overcome the corrosive nature of first-stage naphtha. The good copper corrosion rating is also surprising, considering the high sulfur content of the composite, which is usually associated with copper strip corrosion. The composite sample failed the thermal oxidation stability test for turbine fuels, as measured by ASTM D3241.

Middle-Oil Analyses. The middle oils tested in this program were blended to an end point of 640°F at 90% distilled by D-86, whereas the FY 1981 samples were distilled to an equivalent atmospheric head temperature of 640°F. The FY 1981 samples were lower boiling than the 1982 samples and did not achieve the specification of 640°F at 90% distilled. The D-86 and D-2887 GC-simulated distillation results in Table 9 show that higher boiling fractions (>640°F) reported to the middle-oil cut in this year's samples, but that the 50% point for the FY 1981 samples was actually higher.

In the FY 1981 program, the specific gravities of the middle-oil samples were so close to 1.0 that concern was expressed over the fate of excess water in a storage tank containing these oils, namely, whether the water would float, sink, or be emulsified. Consequently, a test was developed for this year's program that was designed to measure the tendency for water to emulsify in a middle-oil sample. The test consists of adding known amounts of water, ranging from 0.25 to 5%, to middle-oil samples, agitating them vigorously, allowing the samples to settle, and then determining the water concentration at various oil levels. The water concentration in the oil is expected to increase, up to the maximum solubility, and then remain relatively constant if the water/oil mixture does not shown any tendency to emulsify.

Three samples [2004CB SRC first-stage middle oil (M.O.), 2078CB composite M.O., and 2054CB HTU M.O.] were tested by this procedure; none showed any emulsification characteristics. All the samples had a specific gravity of less than one; therefore, the water sank to the bottom of the vessel. The maximum water solubility of each sample reflected its chemical composition; first-stage M.O. achieved a maximum water concentration of 0.9-1.2%, the composite M.O. 0.4-0.5%, and the HTU M.O. 0.1-0.2%. An obvious water layer was observed on the bottom of the container at excess water additions for the composite and HTU samples, but not for the first-stage M.O. Although the analytical data showed that excess water was present, no water layer was observed on the bottom of the container. Probably the mutual solubility of the water in oil, and water-soluble components of the oil in water, obscured the interface between the oil and water layers. The water solubilities in these three oil samples correlate well with their phenol contents, as measured by NIR.

A hot filtration sediment test developed primarily for heavy-oil samples in this program was also applied to these same three middle-oil samples. No sediment was detected in any of the samples, at a detection limit of 1.0 mg/100 g of sample.

Thermal conductivity and heat capacity data were requested by the ICRC engineering group to aid in designing the product storage facilities, and were measured for the first time this year. The thermal conductivity of the middle oils, at two temperatures, was determined by Dynatech and heat capacities were determined by thermal analysis (see Table 9 for results). Thermograms showed a broad/endothermic peak corresponding to vaporization of the sample. The specific heats were calculated at 100°C, which is just at the beginning of this endotherm and, therefore, valid; however, values at higher temperatures were not calculated. This analysis was performed using an open sample pan, which allowed vaporization. If heat capacity values are needed at higher temperatures, work should be conducted using a hermetically sealed pan or a pressure DSC cell.

Table 9 also lists the elemental analysis of these oils. Note that this analysis is on an "as-is" basis; that is, the results have not been corrected for water content. Water is a positive interference in both the hydrogen and oxygen analysis procedures; however, in the case of the hydrogen results, this effect is offset by the effect of correcting to a dry-weight basis. For oxygen, however, because of the high oxygen content of water, a significant negative correction can take place. Table 10 illustrates the effect of correcting for water content, where known, on the elemental analysis. Water solubility is a function of heteroatom content, ranging from 0.04% for hydrocracker middle oil to 0.5% for first-stage middle oil. Correcting to a dry-weight basis reduced all of the oxygen contents by about 10% (relative). Since this change is modest and predictable and the other elemental analyses are not significantly affected, the analysis of all product liquids for water content was not necessary. Results are reported on an "as-is" basis in Table 9 because the water content of all of the samples was not determined.

All chlorine elemental analyses, except that for sample 2084CB (the alternate middle oil), were conducted by APCI using the Dohrmann coulometric titration method. The initial results for Cl in 2084CB, reported by an outside laboratory, were an order of magnitude higher than APCI's chlorine results on the other middle oils. Therefore, this sample and 2004CB were sent to both Huffman and Galbraith Laboratories for N, S, and Cl analysis. Within the precision of the analytical methods, results from these laboratories confirmed APCI's results. Table 11 lists the results of the round-robin analysis for N, S, and Cl.

(1) Wilsonville Run 235 First-Stage Middle Oil (2004CB). Although higher boiling fractions were found in this cut when compared to the middle oil from last year's program (run 225), this sample was actually lower boiling overall. Thus, the lower specific gravity, freezing points, and pour point observed for this sample were not surprising. These property changes can also be partially attributed to the very high olefin content of this sample, which resulted in a lower paraffin and aromatic content, as measured by NMR. The viscosity, Conradson carbon residue, compatibility, and DuPont stability were the same as the run 225 middle oil. The phenol content, measured by NIR, was higher for 2004CB, reflecting its higher total oxygen content. The sulfur and chlorine contents are also slightly higher, but not enough to be of concern.

(2) Wilsonville Run 235 HTU High-Conversion Middle Oil (2054CB). In comparing the chemical and physical properties of this sample to last year's LC-Finer samples, we found that 2054CB is slightly more paraffinic and has a higher heteroatom content. This is reflected by a higher hydrogen-to-carbon ratio, heating value, pour point, neutralization number, and viscosity. The higher nitrogen and oxygen contents may be because a cobalt/molybdenum catalyst was used at Wilsonville, whereas nickel/molybdenum was used at Lummus. The sulfur content, at less than 0.1%, is the same as last year's samples, confirming that sulfur is easiest to remove.

All other physical properties measured were essentially identical to last year's samples. Identification of the 20 largest chromatographic peaks of sample 2054CB was complicated by the sample's complexity; however, the compounds identified were typical of those expected in a hydrotreated coal liquid. The 20 largest peaks represented only 20% of the total sample, with the highest peak concentration at 2.1%, compared to 3.8-4.2% for last year's LC-Finer samples.

The HTU middle distillate contains half as much nitrogen and about one-tenth as much oxygen and sulfur as the first-stage middle distillate. Surprisingly, although it contains over 1 wt % more hydrogen, the HTU middle distillate contains twice as many coke precursors as measured by the Conradson carbon test. The D-86 distillation shows these samples contain about the same concentration of high-boiling compounds, while the D2887 analysis shows more high-boiling samples in the HTU sample.

(3) ALCOA Coker Condensate Middle Oil (1997CB). No coker middle oil data are available from the FY 1981 program with which to compare the results on this sample. The sample is highly aromatic, as confirmed by NIR and GC/MS. Mass spectrometry shows that the 20 largest peaks are dominated by polynuclear aromatic hydrocarbons, although aromatic ethers, alkanes, and phenolic species are also represented. The sample also contains olefinic unsaturation, as measured by NMR, at about half the level of the first-stage M.O. The hydrogen/carbon ratio is low, as expected, and the oxygen content is less than the first-stage liquid, as confirmed by a lower phenol content (measured by NIR). The DuPont stability of this sample was rated as fair, although the color was 8+.

Note that this sample, and all other coal middle oils, have a very poor initial color, ranging from 6 for the hydrotreated liquid, to >8 for all of the other middle oils.

(4) Wilsonville Run 235 Composite Middle Oil Blend (2078CB). The FY 1982 program included the analysis of a composite middle oil sample representing the total liquid output of the proposed demonstration plant. The sample was prepared according to the demonstration plant Baseline yield structure, by blending coker middle oil (1997CB), hydrotreater middle oil (2054CB), and first-stage middle oil (2004CB) in the following proportions: 11:38.1:50.9. The blend's chemical and physical properties reflect its composition, although the API specific gravity is slightly high. The more accurate pycnometric specific gravity is in line with expectations.

The only property of the composite that appears to have degraded is the DuPont stability, which went from a good/fair rating for the constituents to marginal for the blend. We measured a 17,708-Btu/lb heating value for the composite. As part of our quality assurance procedures, this sample was also sent to Huffman Laboratories; they obtained a value of 17,685 Btu/lb.

The GC/MS ranking of the 20 largest peaks reflects the composition of this blend. The list is dominated by polynuclear aromatic hydrocarbons (PAHs), alkyl-PAHs, and partially hydrogenated PAHs; however, the concentration of tetralin and alkyltetralins clearly indicates that the sample contains hydrotreated material. The complexity of this sample dictates that the ranking of these 20 largest peaks is an approximation at best.

(5) Wilsonville Run 235 Alternate Middle Oil (2084CB, 2201CB). As reported earlier, because of blending errors, sample 2084CB does not represent the desired boiling range and chemical composition of the alternate middle oil. However, the sample was analyzed before this error was known; the results are reported in Table 9. The sample has a lower IBP, viscosity, and specific gravity. The oxygen and nitrogen content of 2084CB is slightly higher than that of the composite blend, as is the phenol content measured by NIR. Note that the alternate middle oil does not contain coker liquids, and that SRC first-stage

liquid was substituted for the coker middle oil. Also, the DuPont stability rating of the alternate sample maintained a "good" rating, whereas the composite blend (2078CB) was marginal.

When the error in blending the alternate middle oil was discovered, we decided to blend a sample to the desired composition of the alternate middle oil, since all of the needed fractions were available. We blended the fractions in the correct ratio of 61.6 parts of M.O. to 38.4 parts of naphtha. This true alternate boiling-range middle oil (2201CB) was analyzed by a limited number of tests; the results are reported in Table 9.

(6) Accelerated Stability Test Results. The FY 1982 program also included extensive testing of the middle oils under accelerated aging conditions by exposing them to air at 110°F. The protocol for the study was derived from one developed at BETC. The sample was filtered through the same porosity filter used to determine suspended sediment. Then 400 mL was placed in a 500-mL flask that was vented to the atmosphere through an inverted U tube in the cap, so that particulate matter could not fall into the sample bottle. This accelerated aging procedure is believed to be approximately equivalent to aging at ambient conditions with exposure to air for a period three times as long.

A sufficient number of bottles of each sample were placed in a thermostated oven so that they could be withdrawn and tested at 1-, 3-, and 5-month intervals. In some cases, such as the HTU middle oil (2054CB), there was insufficient sample for complete testing, so the analytical program was curtailed and only one storage sample was analyzed. The suspended sediment and adherent gum test was run on the entire 400-mL contents of one bottle.

The sample's total gum content was measured according to a procedure developed by BETC. A total gum level beyond about 3 mg/100 mL is considered undesirable for #2 diesel fuel. This test was initially run in duplicate, but reduced to a single determination for the 3- and 5-month testing, after the excellent reproducibility of the test had been established. Some tests were deleted from the 3- and 5-month schedule, because they did not appear to offer any useful information concerning storage stability. The deleted tests included the viscosity at two elevated temperatures, specific gravity, pour point, and the NIR procedure for hydroxyl and amine functionality. Those tests that appear to be a good measure of storage stability are the Conradson carbon residue, ASTM D-2781 compatibility, DuPont stability, and the suspended sediment and adherent gum test procedure.

Results on the accelerated aging testing are presented in Table 12 for four middle-oil samples. All four samples aged, but the HTU middle oil showed the greatest stability. The DuPont stability fuel rating, which in itself is an accelerated test, went from good or marginal to very poor in three months of aging for the other three samples, but only from good to fair for the hydrotreated sample (2054CB). In like manner, this sample showed no increase in viscosity or Conradson carbon residue and gave the lowest total gum value after 3 months.

The suspended sediment and adherent gum results offer the best measure of a fuel oil's stability, and the test results show that these coal liquids are surprisingly stable. The total gum content of the composite sample of the total liquid product from the proposed demonstration plant (2078CB) leveled off at 2 mg/100 mL of sample after 3 and 5 months of testing. Even the first-stage middle oil (2004CB), which was expected to be the least-stable sample tested, had only 5.4 mg/100 mL of total gum after 5 months of testing. Figure 1 illustrates the levels of total gum generated at each sampling period for the 5-month test period. Comparison of the aging properties of the alternate middle oil (2084CB) and the composite middle oil (2078CB) reveals no differences. Although our alternate middle oil was not of the desired chemical composition and boiling range, the additional phenols present in the sample did not adversely affect the aging process.

A 1- and 3-month storage test was performed on the "true" alternate middle oil (2201CB) to determine gum formation. The 1- and 3-month test results for sediment and gum are reported in a footnote to Table 12. Due to insufficient sample, the initial suspended sediment and adherent gum of sample 2201CB were not determined; however, this value is always close to zero because of the test protocol, which includes filtering the initial test sample before measurement. After 1 month of aging, the "true" alternate middle oil (2201CB) had 1.3 mg of suspended sediment/100 mL and less than 0.2 mg of adherent gum/100 mL. After 3 months of accelerated aging, the true alternate middle oil contained 0.8 mg/100 mL of suspended sediment and less than 0.2 mg/100 mL of gum. The 1-month and 3-month results are the same, within analytical precision, suggesting that sediment formation has leveled off. Thus, the storage stability of the alternate middle oil is the same or better than that of the standard middle oil (Blend B). We conclude that adding the 350-375°F boiling cut does not reduce middle oil quality.

Heavy-Oil Analyses. Three heavy-oil samples were generated in this program: the first-stage SRC heavy oil (2076CB), the high-conversion run hydrotreater unit heavy oil (2055CB), and a composite of the first two. The samples consisted of the still bottoms remaining after blending to the middle-oil end point, and they had a higher IBP than last year's samples because of the revised middle-oil end point.

The sample's thermal conductivity was measured at three temperatures by Dynatech (see Table 13). The heat capacities were measured by the same thermal analysis technique used for the middle oils; however, the results were reported at two temperatures (100 and 200°C). Although a vaporization endotherm was also noted for these samples, it occurred at higher temperatures, allowing the calculations to be performed at 200°C. The hot filtration sediment procedure was also run on all three samples, but the first-stage (2076CB) and composite-sample (2079CB) liquids could not be filtered using the procedure that had been developed. This was probably due to the heavy nature of the first-stage heavy oil, brought about by the change in the IBP. A value of 4.3 mg/100 mL of the sample was obtained for the hydrotreated heavy oil (2055CB); however, we have no values to compare this to.

(1) Wilsonville Run 235 First-Stage Heavy Oil (2076CB). A limited analytical program was run on this sample, since data existed from the FY 1981 program. However, the data cannot be directly compared because the cut point changed for this sample. The differences in the samples are manifested in the higher IBP, specific gravity, viscosity, and freezing point of 2076CB. The compatibility and Conradson carbon residue of 2076CB are the same as last year's run 225 heavy oil, as is the hydrogen/carbon ratio. The nitrogen and oxygen contents of this sample are higher than last year's samples, but the sulfur content is lower, as is the sediment obtained by ASTM D-1796.

(2) Wilsonville Run 235 Hydrotreater Unit Heavy Oil (2055CB) The differences in cut points are not apparent when comparing the GC simulated distillation and specific gravity results for this sample with last year's LC-Finer samples. However, the viscosity, freezing point, and pour point all reflect the higher initial cut point of the current sample. At 265, the molecular weight is also higher than that of the LC-Finer samples (215, 150). The hydrogen/carbon ratio of 2055CB is also much higher, indicating that this sample is more paraffinic, as confirmed by NMR. Properties such as the carbon residue, bottoms sediment and water analysis (BS&W), and ash content were very favorable (low), and the copper strip corrosion test result was excellent, as were last year's results.

The current sample's heating value of 18,195 Btu/lb is slightly higher than last year's, reflecting the more paraffinic nature of 2055CB; however, its compatibility was poor (a rating of 3) compared to the excellent rating of last year's sample. The neutralization number of 2055CB was also much higher, perhaps reflecting its higher oxygen and nitrogen content. GC/MS identification of the sample's 20 largest components did not yield much useful information; the sample's complexity resulted in poor chromatographic resolution and a lack of data in the mass spectral library on the many possible compounds present.

(3) Wilsonville Run 235 Composite Heavy Oil (2079CB). The composite heavy oil was generated by blending 21% first-stage heavy oil (2076CB) with 79% hydrotreater unit heavy oil (2055CB). The chemical and physical properties of this blend reflect its composition. For example, the composite's average molecular weight was 315. Although we did not determine the molecular weight of the first-stage heavy oil experimentally, the calculated value is 474, which is significantly higher than the value for last year's run 225 heavy oil (235). Since this composite sample was even more complex than the hydrotreater unit heavy oil, the mass spectral identification of the major peaks is even more tenuous.

(4) Wilsonville Run 235 Heavy-Oil Accelerated Aging Studies. All three heavy-oil samples were subjected to an accelerated aging study run

at 110°F with exposure to air. The samples were not filtered before being placed in 500-mL glass bottles and stored in a thermostated oven. Because the amount of sample was limited, only enough to run the limited analytical program was placed in each bottle.

Results (in Table 14) do not present a clear picture of the extent of aging. The viscosity data are ambiguous; the first-stage SRC heavy oil viscosity increased upon aging, the HTU heavy oil viscosity decreased, and that of the composite sample decreased but then sharply increased after five months of aging. Because the samples were too small, viscosities were not determined at 5 months for the first- and second-stage samples.

The Conradson carbon residue also appeared to increase with aging, with the composite sample again showing a sharp increase at 5 months. The bottoms sediment and water analysis (BS&W) showed no increase for the HTU sample and no definite trend for the composite sample; however, a dramatic increase in sediment was noted for the first-stage heavy oil after 5 months. The increase was so great that the test was repeated, with identical results. The cause of this large increase is unknown; however, no correspondingly large increase in sediment was noted in the composite sample. Note, however, that the composite sample exceeded the ASTM #6 final oil specification of no more than 2 wt % moisture and sediment even as a fresh sample. The composite sample has a significantly higher sediment concentration than either of its components, indicating that the first- and second-stage heavy oils are not fully compatible.

The nomenclature for the BS&W test as applied to heavy oils is misleading, since we are actually measuring a toluene-insoluble residue and no appreciable water. Because we wanted to determine what species were responsible for sediment formation, a limited analytical program designed to identify the sediment was added to the program. The sediment obtained from the 1-month aged samples was separated, washed with toluene, and then dried and analyzed. The program called for NMR analysis, molecular weight determination, elemental analysis, and simulated distillation by gas chromatography.

The residues from all three heavy-oil samples were so insoluble in all common solvents that the NMR and molecular weight determinations could not be run. The GC simulated distillation results indicated very low residue weights, and no unusual boiling-point distributions. Evidently, the intra- or intermolecular bonding causing the sediments to be so insoluble did not withstand the heat of the gas chromatograph's injection port. These results are reported in Table 15, along with the elemental analysis. Examination of the results shows that while the oxygen and nitrogen contents of the sediments are incrementally higher than those of the orginal heavy oils, the sulfur content is significantly higher, leading to the conclusion that sulfur species must be involved in sediment formation.

NAPHTHA END-POINT SPECIFICATION

Although naphtha from the SRC-I process is to be used as a reformer feedstock, preliminary contacts with hydrotreating/reforming licensors have elicited conflicting responses as to whether the dicyclic content of naphtha distilled to a 375°F head temperature at 95% distilled is too high for reformer feedstock. Preliminary Kellogg results have also indicated a very high concentration of phenolics (52%) in the 350-398°F fraction of first-stage SRC naphtha. This result must be confirmed, since eliminating a high phenolic fraction from the hydrotreater feed could significantly reduce hydrogen consumption.

In this task, a composite naphtha sample was fractionated into four 25°F boiling-range cuts, and a limited analytical program was performed to determine the dicyclic, phenolic, and heteroatom distribution within the cuts. When this distillation was carried out, as reported earlier, a significant amount of bottoms were recovered; therefore, this fifth cut was also analyzed.

Results of the analyses are reported in Table 16. In the following discussion on individual cuts, keep in mind that these cuts were generated by redistilling a naphtha cut at a high reflux ratio, thereby effectively more than doubling the efficiency of the 15-theoretical-plate distillation normally employed to obtain the naphtha cut. A simple lowering of the naphtha end point by 25°F will not, in all probability, produce the identical distribution of phenolics and dicyclics indicated by the analysis of these cuts.

A limited number of tests were run on these five cuts to characterize their physical properties. The results, as well as those from tests run to define the product distribution, are also listed in Table 16. The weight percent yield of each cut is also given. Properties such as boiling range, API gravity, and specific gravity followed trends to be expected for these successive 25°F boiling-range cuts. The phenolic and dicyclic content of each cut was obtained by GC/MS; the quantitative data may be in error because some GC peaks were unresolved and the response factors were not taken into account. These data and the heteroatom contents of the cuts are summarized in Table 17. Also presented in this table is the percent of the total component in each cut, and the ratio of this percent to the percent yield. This ratio is useful in evaluating the benefits of removing a cut from the product. The higher the ratio, the greater the benefit derived from removing the cut.

Lowering the naphtha end point by 25°F would probably cause most of the still bottoms (cut 5) and most of cut 4 to report to the middle-oil fraction. These two cuts account for 16.4% of the naphtha fractions; by removing them we can remove 64.6% of the naphtha dicyclic content and 54.6% of the nitrogen content. Examination of the results in Table 17 shows that the phenolic, oxygen, sulfur, and chlorine contents of the naphtha are distributed more evenly across the various cuts on a percentage basis. However, even on this basis there is some incremental value in excluding cuts 4 and 5 from the naphtha fraction. Although the phenolic content of the various fractions listed in Table 17 was obtained by GC/MS, note that Air Products' methods for measuring hydroxyl content by near-infrared and phenols by caustic extraction confirm the phenolic distribution obtained by GC/MS. Results of these analyses are presented in Table 16.

PETROLEUM/COAL LIQUID COMPATIBILITY

This part of the program was a detailed study to determine the compatibility of SRC-I middle- and heavy-distillate products with their petroleum counterparts. Ideally, it should be possible to substitute coal liquids for any petroleum liquid and mix them with their petroleum homolog in any proportion. We know that this ideal situation does not exist; however, results of the FY 1981 program indicate that the compatibility problems are less severe than expected.

In order to carry out this program, four petroleum fuels were obtained from the Sun Oil Corporation and analyzed to provide base-line data. These fuel oils consisted of a vacuum gas oil (2051CB), a No. 2 fuel oil (2050CB), a low-sulfur no. 6 fuel oil (2048CB), and a high-sulfur no. 6 fuel oil (2049CB). Results of the analyses of these samples are reported in Table 18. The results are as expected for these liquids; one surprising result was the incompatibility of the low-sulfur no. 6 fuel oil with another petroleum liquid.

Preparation of Petroleum/Coal Liquid Blends

The vacuum gas oil and no. 2 fuel oil were blended with 10, 25, 50, 75, and 90% by weight of the composite middle distillate (2078CB). The low- and high-sulfur no. 6 fuel oils were blended with the composite heavy-oil sample (2079CB) in the same weight ratios. The original program called for a blend of diesel fuel with the coal middle distillate; however, these blends were eliminated from the program, as were the blends at a concentration of 5% coal liquids. Added to the program were the blends containing 75 and 90% coal liquid.

Analysis of Petroleum/Coal Liquid Blends

The petroleum/coal liquid blends were analyzed as soon as possible after preparation, in order to determine compatibility and to provide a base point for the long-term stability studies. The blends were then stored at 110°F in glass, with exposure to air, in order to determine long-term storage stability. Since bulk properties are not good indicators of storage stability, these tests were not requested on the storage samples. In addition, if a test such as the DuPont stability test showed maximum incompatibility, it was also deleted from further testing. One test, the GC-simulated distillation test, is not reported in any of the tables on the analysis of these blends. Because of a number of problems with the analytical procedure, both instrumental and operator related, we determined that these results were so inconsistent that they could not be used as a measure of compatibility or stability. The results of all of the GC-simulated distillation tests are reported separately in Tables 19-24.

Vacuum Gas Oil/Coal Middle-Oil Blends. Results of compatibility and stability testing on these blends are listed in Table 25. The stability test was stopped after 1 month of storage, because of cost constraints. From the limited amount of testing performed, a compatibility problem is apparent, especially at low levels of coal oil (10 and 25%). At the 10% coal liquid level, the viscosity was too high to measure at 68°F. In an attempt to determine if the blend had gelled irreversibly, the viscosity was run at 104°F, with a value obtained. Although this sample was too gelled to flow through the capillary tube in the kinematic viscosity test, its measured pour point of 70°F is lower than the vacuum gas oil's pour point. Warming the sample to perform the pour point test probably broke the gel. The pour point was measured as the sample was being cooled, and it apparently had insufficient time to reestablish the gel state. This apparently indicates that the molecular attractions causing the viscosity increase are not permanent, as they are in polymerization.

Other indicators of incompatibility are the DuPont stability rating and the hot filtration sediment levels; both test results indicate maximum incompatibility to low coal-oil levels. The very limited testing program run on these blends after 1 month of storage at 110°F shows that all of the samples, including the unblended vacuum gas oil, are aging. The viscosity of the vacuum gas oil was too high to measure at 68°F after 1 month of storage, while the viscosities of most of the other blends also increased. The DuPont stability rating of all of the blends deteriorated to very poor, and the pour points increased, showing that all of the blends were becoming more incompatible upon aging.

No. 2 Fuel Oil/Coal Middle-Oil Blends. A comprehensive analytical program was performed on these blends, and the storage stability test was carried out to 3 months. An overall evaluation of the initial analytical results indicates incompatibility of the coal middle distillate with no. 2 fuel oil, especially at low coal oil concentrations. Results are listed in Table 26. Once again, bulk property tests are no measure of compatibility. Tests indicative of polymerization or gum formation, such as suspended sediment and adherent gum, and hot filtration sediment show a significant degree of incompatibility of the coal oil with no. 2 fuel oil, especially at low concentrations of coal oil. The total suspended sediment plus gum values seen in the BETC tests on the aged blends are unsatisfactory.

A plot of total suspended sediment and adherent gum vs. coal oil concentration, shown in Figure 2, demonstrates the incompatibility at low coal oil concentrations, which is further accentuated after storage at 110°F in glass for periods of 1 and 3 months. The DuPont stability rating of the blends has also decreased, although the maximum incompatibility, as measured by this test, appears to be at the 25-50% coal oil concentration. The storage stability of the unblended no. 2 fuel oil is excellent, as measured by all tests, and the storage stability of the composite coal middle distillate (Table 12) was good. However, that of the various blends is poor, with the aging accelerated in the more incompatible blends. This suggests that the poor storage stability of the blends results from incompatibility between the coal middle distillate and the no. 2 fuel oil.

No. 6 Fuel Oil/Coal Heavy-Oil Blends. A compatibility and storage stability study was also conducted on blends of heavy oil with both low- and high-sulfur no. 6 fuel oil (see Tables 27 and 28). The compatibility and stability of these heavy-oil blends are more difficult to measure, since we are dependent upon bulk property tests, rather than those that measure polymerization or gum formation. One test, the hot filtration sediment, was designed to measure this property; however, the test was found to be inapplicable because the liquids could not be filtered. We have to rely on a limited number of tests to measure the compatibility and stability of these blends. The results of the Saybolt viscosity, Conradson carbon, and bottoms sediment and water tests do not indicate any gross incompatibility of the coal heavy oil with either the low- or high-sulfur no. 6 fuel oil in any of the blend ratios. The storage stability test on the high-sulfur no. 6 fuel oil blends was terminated after 1 month of storage in glass at 110°F, with the limited analytical testing program indicating that no significant stability problems were encountered.

The Conradson carbon residue and the pour points of the various blend ratios did not change, while the viscosity and BS&W values showed minor fluctuations, both up and down, probably reflecting experimental and sampling error. The storage stability test on the blends prepared with the low-sulfur no. 6 fuel oil was carried out to 5 months on the unblended no. 6 fuel oil, and the 10, 50, and 90% coal oil blends. The stability test was terminated at 3 months for the 25 and 75% coal-oil blends. An examination of the stability results on those blends aged up to 3 and 5 months shows that while the blends are aging somewhat, as measured by a viscosity increase and a slight increase in the sediment as measured by the BS&W test at low coal oil concentrations, there does not appear to be any gross incompatibility of the coal heavy oil with the low-sulfur no. 6 fuel oil after 5 months of storage at 110°F. The BS&W values seem to be an algebraic average of the values for the parent liquids, based upon the fraction of each in the blend. Although many of the blends exceed the 2 wt % specification for BS&W, this is a consequence of the coal heavy distillate being high in BS&W and does not suggest that the no. 6 fuel oil and coal distillate are incompatible.

The viscosity measurements again are fluctuating somewhat, but it appears that only the high coal oil concentration blends exhibit significant viscosity increases at the 3-5-month time period. The 75% coal oil blend, however, does not show a viscosity increase after 3 months of storage. The Conradson carbon residue values remain constant for all samples, indicating no increase in coking properties of these blends, while the pour point results are ambiguous on the initial and 1-month samples. In an attempt to understand the nature of heteroatomic species involved in sediment formation, the sediment from the BS&W test was separated, washed with toluene, dried, and then analyzed by GC-simulated distillation for boiling point distribution and elemental analysis.

The boiling-point distribution of the BS&W sediment was essentially the same as the components of the blends, and no unusual residue weights were observed, indicating that the sediment is not a high molecular weight, or polymerized portion of the blend. Elemental analysis of the sediment shows that the nitrogen, oxygen, and sulfur contents of the sediment are higher than those of the blends, with the oxygen content significantly higher. Apparently, polymerization is not the primary mechanism for sediment formation; also, heteroatomic species, particularly oxygen, are involved. This is not too surprising, since the blends were exposed to oxygen; however, it should be emphasized that sediment formation was only minimal and does not appear to present major problems.

SUMMARY OF MAJOR FINDINGS

The following is a summary of the major findings from these studies:

- <u>Naphtha</u>: Second-stage naphtha contains less heteroatoms (except nitrogen) than first stage naphtha, while coker naphtha is similar to first-stage naphtha except for a lower sulfur content. Composite naphtha properties can be linearly blended. Composite naphtha has high existent gum and fails ASTM D3241 (thermal oxidation stability for turbine fuels). Its HHV is about 125,000 Btu/gal. Lowering the end point of composite naphtha reduces yield, phenolics, oxygen, sulfur, and chlorine by 15-20%, and removes two-thirds of the dicyclic content and one-half of the nitrogen, without significantly affecting middle oil properties.
- <u>Middle Oil</u>: Second-stage middle oil is lower in heteroatoms than first-stage but higher in coke precursors. Coker middle oil is highly aromatic but lower in oxygen than first-stage middle oil. Composite middle oil properties reflect the blend composition. The accelerated aging test (BETC) indicates that the SRC-I middle oils are relatively stable during storage under air. Thermal conductivity and specific heat data were collected and the composite HHV was measured at about 145,000

Btu/gal. Blends with VGO and No. 2 fuel oil show compatibility problems, especially at low coal oil contents (10%). No emulsification tendency was shown with water for the middle oils.

- Heavy Oil: Thermal conductivity and specific heat data were collected, and the composite HHV was measured at about 158,000 Btu/gal. Composite properties reflect the blends. First- and second-stage heavy oils do not appear to be fully compatible and the composite exceeds ASTM BS&W specs for No. 6 fuel oil. (Sulfur species appear to be involved in the formation of sediment.) However, blends with No. 6 fuel oil appear generally compatible. The sediment content increased significantly as SRC-I heavy oils aged.

LITERATURE CITED

Tiedge, W. F., and T. L. Slager. 1982. Liquid product characterization study. Pages 57-124 in SRC-I quarterly technical report, January-March 1982. DOE/OR/03054-7. International Coal Refining Co., Allentown, Pa.

TABLE 1

Samples for the Liquid Product Characterization Study

APCI No.	ICRC No.	Description
1970CB	--	ALCOA Coker condensate oil, Run 50, received 9/17/81.
1996CB	--	Light oil (naphtha) generated by distillation of 1970CB into a fraction having a boiling range of IBP to 95% distilled at 375°F.
1997CB	--	Middle oil generated by distillation of 1970 into a fraction having a boiling range of 375°F @ 95% to 90% distilled at 622°F.
1990CB	957-103A	Hydrotreater unit process solvent collected from sample point V-1074 during Wilsonville Pilot Plant Run No. 235 - High Conversion.
1994CB	--	Hydrotreater unit light oil (C_5-450°F) collected from sample point V-161 in December 1981 during Wilsonville Pilot Plant Run No. 235 - High Conversion.
2000CB	947-093A	Light oil (C_5-450°F) collected from sample point V-170 on November 18, 1981 during Wilsonville Pilot Plant Run No. 235.
2010CB	947-093B	Process solvent (450-850°F) collected from sample point V131A on November 18, 1981 during Wilsonville Pilot Plant Run No. 235.
2002CB	--	A blend of Wilsonville front end liquids prepared by blending 57.3 weight % of 2000CB and 42.7 weight of 2010CB.
2003CB	--	Light oil (naphtha) generated by distillation of 2002CB into a fraction having a boiling range of IBP to 95% distilled at 375°F.
2004CB	--	Middle oil generated by distillation of 2002CB into a fraction having a boiling range of 375°F @ 95% to 90% distilled at 640°F.
2005CB	--	Heavy oil generated by the distillation of 2002CB into a fraction having a boiling range of 640°F at 90% distilled to a nominal 850°F (still bottoms).
2006CB	--	A repeat of blend 2002CB, distilled to obtain additional liquid products.

TABLE 1 (Cont.)

Samples for the Liquid Product Characterization Study

APCI No.	ICRC No.	Description
2007CB	--	Light oil (naphtha) generated by distillation of 2006CB into a fraction having a boiling range of IBP to 95% distilled at 375°F.
2008CB	--	Middle oil generated by distillation of 2006CB into a fraction having a boiling range of 375°F @ 95% to 90% distilled at 640°F.
2009CB	--	Heavy oil generated by the distillation of 2006CB into a fraction having a boiling range of 640°F at 90% distilled to a nominal 850°F (still bottoms).
2020CB	947-093C	Hydrotreater unit light oil (C_5-450°F) collected from sample point V-161 on November 18, 1981 during Wilsonville Pilot Plant Run No. 235 - Low Conversion.
2021CB	--	Light oil (naphtha) generated by the distillation of 2020CB into a fraction having a boiling range of IBP to 95% distilled at 375°F.
2022CB	--	Still bottoms remaining after 2021CB (naphtha) was distilled.
2023CB	--	A blend of low conversion hydrotreater liquids prepared by blending 2022CB and 2041CB so that a ratio of 34% light oil and 66% process solvent was obtained.
2024CB	--	Middle oil generated by the distillation of 2023CB into a fraction having a boiling range of 375°F @ 95% to 640°F at 90% distilled.
2025CB	--	Heavy oil generated by the distillation of 2023CB into a fraction having a boiling range of 640°F at 90% distilled to a nominal 850°F (still bottoms).
2026CB	--	A repeat of distillation 2023CB using remainder of 2022CB bottoms.
2027CB	--	Middle oil generated by distillation of 2026CB into a fraction having a boiling range of 375°F @ 95% to 640°F at 90% distilled.
2028CB	--	Heavy oil generated by the distillation of 2026CB into a fraction having a boiling range of 640°F at 90% distilled to a nominal 850°F (still bottoms).

TABLE I (Cont.)

Samples for the Liquid Product Characterization Study

APCI No.	ICRC No.	Description
2041CB	940-086F	Hydrotreater unit process solvent collected from sample point V-1074 on November 18, 1981 during Wilsonville Pilot Plant Run No. 235 - Low Conversion.
2048CB-1,2,	--	0.5 weight %, sulfur, #6 fuel oil.
2049CB-1,2	--	2.0 weight % sulfur, #6 fuel oil.
2050CB-1,2	--	Straight run #2 fuel oil.
2051CB-1,2	--	Vacuum gas oil
2052CB	--	A blend of high conversion hydrotreater liquids prepared by blending 34 parts of 1994CB with 66 parts of 1990CB.
2053CB	--	Light oil (naphtha) generated by distillation of 2052CB into a fraction having a boiling range of IBP to 95% distilled at 375°F.
2054CB	--	Middle oil generated by distillation of 2052CB into a fraction having a boiling range of 375°F @ 95% to 90% distilled at 640°F.
2055CB	--	Heavy oil generated by distillation of 2052CB into a fraction having a boiling range of 640°F at 90% distilled to a nominal 850°F (still bottoms).
2076CB	--	A blend of heavy oils [640°F at 90% to a nominal 850°F (bottoms)] 2005CB and 2009CB.
2077CB	--	A blend of light oils (naphtha) designated "Composite Naphtha Blend A" prepared as follows: 90 grams of 1996CB, 440 grams of 2053CB and 1466 grams of 2003CB.
2078CB	--	A blend of middle oils designated "Composite Middle Oil Blend A" prepared as follows: 1540 grams-1997CB, 5334 grams-2054CB, 7126 grams-2004CB.
2079CB	--	A blend of heavy oils designated "Composite B, Heavy Oil, SRC/TSL" prepared as follows: 29% 2076CB, 71% 2055CB.

TABLE 1 (Cont.)

Samples for the Liquid Product Characterization Study

APCI No.	ICRC No.	Description
2080CB	--	A blend of light oils (naphtha) designated "Composite Naphtha Blend B-1, SRC/TSL" prepared as follows: 77.8 wt % (1867 grams) of 2002CB and 22.2 wt % 2053CB (533 grams). Will be distilled into 5 fractions 2080CB-1 IBP-325°F, 2080CB-2 325-350°F, 2080CB-3 350-375°F, 2080CB-4 375-400°F, 2080CB-5 400°F-BTMS.
2081CB	--	A blend of light oils (naphtha) designated Composite Naphtha Blend B-2" prepared as follows: 77.8% (5,446 grams) of 2007CB and 22.2% (1554 grams) of 2053CB.
2082CB	--	Light oil (naphtha) generated by distillation of 2081CB into a fraction having a boiling range of IBP to 95% distilled.
2084CB	--	A blend of middle oils designated "alternate middle oil" prepared by blending 435.2 grams 2083CB with 3818 grams of 2008CB and 2350 grams of 2024CB.

TABLE 2

WILSONVILLE RUN 235
FRONT-END LIQUID DISTILLATION

Charge Blend: 57.3% Light oil (2000CB, 947-093A)
42.7% Process solvent (2010CB, 947-093B)

Fraction	Grams	% of Charge	Boiling Range, °F	APCI No.
Blend Charge No. 1	29,964.0	100.0	--	2002CB
Naphtha	7,381.0	24.6	IBP - 400°F	2003CB
Middle Oil	21,792.0	72.7	400°F - 710°F	2004CB
Heavy Oil	774.5	2.6	710°F - Btms	2005CB
Loss	16.5	0.1	--	--
Blend Charge No. 2	39,120.0	100.0	--	2006CB
Naphtha	5,896.0	15.1	IBP - 400°F	2007CB
Middle Oil	30,982.0	79.2	400° - 740°F	2008CB
Heavy Oil	1,629.0	4.2	740° - Btms	2009CB
Loss	613.0	1.5	--	--

TABLE 3

WILSONVILLE HTU RUN 235
HIGH CONVERSION DISTILLATION

Charge Blend: 34 Wt. % HTU Light Oil (1994CB)
66 Wt. % HTU Process Solvent (1990CB)

Fraction	Grams	% of Charge	Boiling Range, °F	APCI No.
Blend Charge No. 1	20,600	100.0	--	2052CB
Naphtha	5,230	25.4	IBP - 375@95%	2053CB
Middle Oil	8,196	39.8	375@95% - 640@90%	2054CB
Heavy Oil	7,136	34.6	640@90% - BTMS	2055CB
Loss	38	0.2	--	--
Blend Charge No. 2*	3,576	100.0	--	1994CB
Naphtha	2,678	74.9	IBP - 375@95%	2053CB

*This charge was distilled from 1994CB only to make additional naphtha cut for testing purposes.

TABLE 4

WILSONVILLE RUN 235
TSL-LOW CONVERSION DISTILLATION

Fraction	Grams	% of Charge	Boiling Range, °F	APCI No.
Charge	3,258.0	100.0	C_5 - 450°F	2020CB
Naphtha	2,213.0	67.9	IBP - 375°F @ 95%	2021CB
Still bottoms	985.7	30.3	450°F	2022CB
Loss	59.3	1.8	--	--
	Charge Blend:	425 gms 2022CB Still Bottoms HTU Light Oil 3113.6 gms 2041CB HTU Process Solvent Low Conversion		
Charge A	3,531.6	100.0	400 - 850°F	2023CB
Naphtha	22.3	0.6	IBP - 400°F	--
Middle Oil*	2,393.1	67.8	400°F - 710°F	2024CB
Heavy Oil	1,108.2	31.4	690°F - 850°F	2025CB
Loss	8.0	0.2	--	--
Charge B	3,530.9	100.0	400 - 850°F	2026CB
Naphtha	66.5	1.9	IBP - 400°F	--
Middle Oil*	2,357.5	66.8	400 - 710°F	2027CB
Heavy Oil	1,092.2	30.9	710 - 850°F	2028CB
Loss	14.7	0.4	--	--

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*Due to foaming, M.O. cuts could not be blended by D-86. Blend was performed by GC-Simulated Distillation D-86 correlation.

TABLE 5

COKER CONDENSATE DISTILLATION

Fraction	Grams	% of Charge	Boiling Range*	APCI No.
Charge	3,703	100	--	1970CB
Naphtha	200.2	5.4	IBP - 400°F	1996CB
Middle Oil	2,257.5	61.0	400°F - 775°F	1997CB
Still Bottoms	1,152.7	31.1	775°F - Btms	--
Loss	92.6	2.5	--	

*Temperatures are equivalent atmospheric temperatures from the Maxwell-Bonnell Charts using a K factor of 12.

TABLE 6

COMPOSITE NAPHTHA BLEND B-2 DISTILLATION FOR ALTERNATE MIDDLE OIL

Charge Blend: 77.8 Wt. % Naphtha Cut from SRC Lite Distillation 2007CB
22.2 Wt. % Naphtha Cut from HTU High Conv. Distillation 2053CB

Fraction	Grams	% of Charge	Boiling Range, °F	APCI No.
Blend Charge	7000	--	--	2081CB
Naphtha	5697	81.4	IBP - 350 @ 95%	2082CB
Middle Oil	1162	16.6	350@95% - BTMS	2083CB
Loss	141	2.0	--	--

TABLE 7

NAPHTHA END POINT DISTILLATION

COMPOSITE NAPHTHA BLEND "B-1" - 2080CB

Charge Blend: 77.8 Wt. % Naphtha Cut,First Stage Liquid 2003CB
22.2 Wt. % Naphtha Cut HTU High Conv., 2053CB

Fraction	Grams	% of Charge	Boiling Range, °F	APCI No.
Blend Charge	2389	--	--	2080CB
--	1397	58.5	IBP - 325	2080CB1
--	283	11.9	325 - 350	2080CB2
--	280	11.8	350 - 375	2080CB3
--	249	10.5	375 - 400	2080CB4
--	141	5.9	400 - BTMS	2080CB5
Loss	39	1.7	--	--

TABLE 8
LIQUID PRODUCT CHARACTERIZATION - LIGHT OILS

Test #	ASTM #	Description	Wilsonville SRC run 235 (APCI 2003CB)	Wilsonville HTL run 235 High conversion (APCI 2053CB)	Wilsonville HTU run 235 Low conversion (APCI 2021CB)	Alcoa Coker Condensate oil Run 50 (APCI 1996CB)	Composite Naphtha Blend "A" (APCI 2077CB)
			Vapor temperature (°F)				
		Vol.% Dist.					
1	D86	IBP	118	149	172	230	151
		5	154	191	205	--	197
		10	181	206	216	275	223
		20	225	223	229	288	237
		30	252	239	242	294	267
		40	278	254	256	302	284
		50	300	272	272	311	304
		60	319	292	292	322	319
		70	335	313	314	334	335
		80	350	336	335	348	348
		90	368	357	355	365	370
		95	381	372	374	374	393
		FBP	398	390	384	387	410
	D2887	IBP	122	119	141	204	123
		5	157	187	187	255	158
		10	192	197	195	290	183
		15	217	223	215	305	204
		20	246	228	221	310	239
		30	274	255	249	319	267
		40	309	272	260	332	297
		50	339	296	295	352	324
		60	360	316	316	363	343
		70	381	339	342	380	364
		80	409	362	364	392	389
		90	497	387	391	405	426
		95	531	404	413	418	505
		FBP	874	446	547	688	757
		Residue (%)		3			
2	D445	Liquid viscosity (cSt) @68°F(20°C)	NR	1.0	1.0	NR	1.2
3	D1298	Liquid specific gravity @60°F	0.8393	0.8058	0.8165	0.8692	0.8448
		API gravity @60°F	37.1	44.1	41.3	31.3	36.0
3a	APCI Method	Specific gravity, pycnometer, @60°F (15.6°C)	--	0.8115	--	--	--
		@77°F (25°C)	0.8410	--	0.8158	0.8654	0.8311
6	D3828	Flash point (°F,°C)	NR	6,-14	11,-12	NR	2,-17

TABLE 8 (Cont.)
LIQUID PRODUCT CHARACTERIZATION - LIGHT OILS

Test #	ASTM #	Description	Wilsonville SRC run 235 (APCI 2003CB)	Wilsonville HTU run 235 High conversion (APCI 2053CB)	Wilsonville HTU run 235 Low conversion (APCI 2021CB)	Alcoa Coker Condensate oil Run 50 (APCI 1996CB)	Composite Naphtha Blend "A" (APCI 2077CB)
7	D1322	Smoke point(mm)	16.5	17.8	14.5	NR	16.3
9	D240	Heating value (Btu/lb)	NR	19,234	18,904	NR	17,798 17,740[a]
10	D130	Copper strip corrosion,rating	NR	1A	1A	NR	1B
11	D381	Existent gum (mg/100mL)	83	29	6	NR	65
14a	APCI method	Water by KF, wt %	NR	0.03	0.07	0.32	0.56
16	D2781	Compatibility Modified compatibility (blend 1/1-v/v with petroleum)	NR	1	1	NR	1
17	D664	Neutralization # (mg/KOH/gm)	NR	7.9	9.7	NR	7.2
19	APCI method	GC/MS analyses (Compound wt %) (20 major compounds)	NR				
		1		M-Cy-hexane(10.9)	Toluene(15)	Di-M-benzene (17.6)	Phenol(8.5)
		2		Cy-hexane(8.4)	M-Cy-hexane(9.3)	Phenol(8.1)	3-M-phenol(5.9)
		3		Toluene(7.4)	Cy-hexane(7.8)	Di-M-benzene(6.7)	M-benzene(5.9)
		4		E-Cy-hexane(4.8)	E-Cy-hexane(4.5)	3 & 4-M-phenol (5.9)	M-Cy-hexane(3.9)
		5		Di-M-E-benzene(3.0)	Propyl-Cy-hexane(3.0)	E-benzene(5.2)	2-M-phenol(3.4)
		6		P-Cy-hexane(3.0)	Cis-Octa-Hy-1H-indene(3.0)	2-M-phenol(4.3)	Di-M-benzene(3.1)
		7		Cis-Octa-Hy-1H-indene(1.9)	Di-M-benzene(2.6)	M-benzene(4.1)	Cy-hexane(2.4)
		8		Di-M-Cy-hexane(1.9)	E-M-benzene(2.1)	Undecane(2.9)	E-Cy-hexane(2.3)
		9		Di-M-Cy-hexane(1.8)	Bu-Cy-hexane(1.8)	Tri-M-benzene (2.8)	Octane(1.8)
		10		E-M-naphthalene (1.6)	Trans-deca-Hy-naphthalene(1.6)	Decane(2.7)	2,3-Di-Hy-1H-Indene(1.5)
		11		1,2,3,4,Te-Hy-naphthalene(1.6)	Di-M-Cy-hexane (1.6)	2,3,Di-Hy-1H-indene(1.8)	Heptane(1.4)
		12		E-benzene(1.6)	E-benzene(1.5)	E-M-benzene(1.7)	Di-M-Benzene(1.3)
		13		M-2,3,E-Di-Hy-1H-indene(1.4)	Di-M-Cy-hexane (1.5)	Nonane(1.7)	Nonane(1.3)
		14		Bu-Cy-Hexane(1.4)	Propylbenzene (1.4)	Dodecane(1.4)	M-2,3-Di-Hy-1H-indene & a C_4-alkylbenzene(1.3)
		15		2,3-Di-Hy-1H indene(1.4)	1,2,3,4,-tetra-Hy-naphthalene (1.4)	M-2,3-Di-Hy-1H indene & a C_4-alkylbenzene(1.4)	M-2,3,Di-Hy-1H-indene(1.3)
				(1.0)			

TABLE 8 (Cont.)
LIQUID PRODUCT CHARACTERIZATION - LIGHT OILS

Test #	ASTM #	Description	Wilsonville SRC run 235 (APCI 2003CB)	Wilsonville HTU run 235 High conv (APCI 2053CB)	Wilsonville HTU run 235 Low conv (APCI 2021CB)	Alcoa Coker Condensate oil Run 50 (APCI 1996CB)	Composite Naphtha Blend "A" (APCI 2077CB)
19(Cont)							
		16		M-Cy-pentane(1.4)	1- or 2-M-indan (1.2)	Tri-M-benzene (1.1)	E-M-benzene(1.3)
		17		Hexane(1.3)	E-M-Cy-hexane (1.1)	M-2,3,Di-Hy-1H-indene(1.0)	E-benzene(1.3)
		18		Trans-deca-Hy-naphthalene(1.2)	Di-M-benzene (1.0)	Octane(1.0)	P-Cy-hexane(1.3)
		19		E-M-Cy-hexane(1.2)	2,3-Di-Hy-1H-indene(indan)(1.0)	M-1,2,3-Di-Hy-1H-indene(0.9)	1,2,3,4-Te-Hy-naphthalene(1.2)
		20		Heptane(1.1)	1- or 2-M-indan & C_4-alkylbenzene	C4 alkyl-benzene(0.8)	Undecane(1.2)
20	APCI method	Elemental analysis					
		C (wt %)	82.75	87.06	87.05	85.57	83.85
		H (wt %)	11.56	12.82	12.38	10.60	11.73
		N (wt %)	0.23	0.18*	0.21*	0.23	0.24*
		O (wt %)	4.73	0.40	0.58	3.67	3.74
		S (wt %)	1.24	<0.1	<0.1	0.09	0.95
		Cl (ppm)	17	6	<2	18	12
21	APCI method	Proton NMR spectrum(vol %)	NR				
		Paraffins		79.1	71.4	32.1	57.4
		Olefins		0	0	16.4	13.6
		Aromatics		20.9	28.6	51.5	29.0
23	APCI method	Near infrared spectrum (wt %)					
		O as OH	2.33	0.15	0.26	2.06	1.86
		N as NH	<0.01	<0.01	<0.01	<0.01	<0.01
		N as NH_2	<0.01	0.04	0.08	<0.01	<0.01
24	APCI method	Complete infrared spectrum functional group	NR	CH(sat) CH(aro) C=C(aro)	CH(sat) CH(aro) C=C(aro)	CH(sat) CH(aro) C=C(aro) OH C-O	CH(sat) CH(aro) C=C(aro) OH C-O
25	APCI method	Caustic extraction with 20% NaOH (1/1-v/v)-GC analysis of extract (Compound wt %)	NR				
		Phenol		0.67	0.62	7.97	5.85
		x_1		0.05	0.10	0.04	0.10
		o-cresol		1.02	0.85	3.73	2.50
		x_2		0.04	0.04	-	-
		m&p cresol		1.05	0.86	6.66	4.25
		x_3		0.08	0.10	0.04	0.06

*Dohrman nitrogen

TABLE 8 (Cont.)
LIQUID PRODUCT CHARACTERIZATION - LIGHT OILS

Test #	ASTM #	Description	Wilsonville SRC run 235 (APCI 2003CB)	Wilsonville HTU run 235 High conversion (APCI 2053CB)	Wilsonville HTU run 235 Low conversion (APCI 2021CB)	Alcoa Coker Condensate oil Run 50 (APCI 1996CB)	Composite Naphtha Blend "A" (APCI 2077CB)
25(Cont)							
		2,6 xylenol		0.17	0.13	0.20	0.17
		x_4		-	0.01	-	-
		o-E-phenol		0.19	0.13	0.20	0.21
		2,4&2,5 xylenol		0.46	0.33	0.61	0.55
		x_5		0.01	0.01	-	-
		m&p-E-phenol		0.46	0.30	0.41	0.57
		x_6		-	0.01	-	-
		2,3 xylenol		0.05	0.04	0.05	0.05
		x_7		0.02	0.03	-	-
		3,4 xylenol		0.06	0.04	0.05	0.06
		x_8		0.20	0.13	0.08	0.14
		Total		4.53	3.73	20.08	14.51
		Gravimetric loss (wt %)		7.23	3.83	18.78	13.99
		x_n = unidentified components					
26	D3241	Oxidation stability					
		Heater tube deposit rating[b]	NR	4+	4+	NR	4+
		Pressure drop (mm Hg)[c]		85	0		254@9.9 min
28	D1323	Mercaptans (wt % S)	NR	<0.001	0.005	NR	0.27
29	D1500	Color (NPA)	NR	3.5	1.0[d]	8.0	3.5

[a] Repeat analysis obtained from Huffman Laboratories - Wheat Ridge, Colorado.
[b] Rating range 0 to 4 (Good to Poor)
[c] Pressure drop across filter at end of test or time to reach pressure differential of 250 mm Hg.
[d] Sample was pink and did not fit into range of yellow to orange colors.
NR Not requested

TABLE 9
LIQUID PRODUCT CHARACTERIZATION - MIDDLE OILS

Test #	ASTM #	Description	Wilsonville SRC run 235 (APCI 2004CB)	Wilscnville HTU run 235 High conversion (APCI 2054CE)	Alcoa coker Condensate oil Run 50 (APCI 1997CB)	Composite Blend "3" (APCI 2078CB)	Alternate Blend (APCI 2084CB)	True Alternate Blend (APCI 2201CB)
				Vapor temperature (°F)				
		Vol.% Dist.						
1	D86	IBP	423	447	416	442	396	356
		5	434	470	--	460	402	387
		10	440	484	448	462	404	399
		20	450	507	471	464	408	420
		30	459	527	490	480	436	440
		40	468	538	508	500	452	458
		50	481	568	526	520	480	489
		60	493	582	547	543	508	532
		70	521	602	569	572	540	-
		80	568	620	598	600	584	-
		90	639	640	622	638	635	-
		95	671	652	634	657	655	-
		FBP	672	659	636	663	659	-
	D2887	IBP	376	386	379	381	367	NR
		5	411	488	414	415	416	
		10	423	502	432	437	432	
		15	432	510	459	454	450	
		20	444	521	470	464	464	
		30	461	552	495	504	490	
		40	473	581	520	544	523	
		50	493	605	543	584	557	
		60	516	631	570	617	597	
		70	547	656	606	642	629	
		80	601	690	631	669	662	
		90	661	726	661	698	696	
		95	708	786	683	725	721	
		FBP	808	887	768	849	900	
		Residue (%)	--	--	--	<3	<3	
2	D445	Liquid viscosity (cSt)						
		68°F(20°C)	9.25	9.06	NR	9.24	8.20	8.22
		104°F(40°C)	4.45	4.63		4.50	4.08	4.15
		140°F(60°C)	2.76	3.02		2.84	2.64	2.81
3	D1298	Liquid specific gravity @60°F	0.9792	0.9580	0.9951	0.9806	0.9732	0.9679
		API gravity @60°F	13.0	16.2	10.7	12.8	13.9	14.7
3a	APCI Method	Specific gravity, pycnometer, @60°F(15.6°C)	--	--	0.9993	--	0.9717	NR
		@77°F(25°C)	0.9818	0.9540	--	0.9719	--	
5	D2386	Freeze point(°F,°C)	<-76,<-60	<-76,<-60	-74,-59	<-75,<-60	-72,-58	NR
6	D3828	Flash point (°F,°C)	NR	218,103	219,104	212,100	NR	NR

TABLE 9 (Cont.)
LIQUID PRODUCT CHARACTERIZATION - MIDDLE OILS

Test #	ASTM #	Description	Wilsonville SRC run 235 (APCI 2004CB)	Wilsonville HTU run 235 High conversion (APCI 2054CB)	Alcoa Coker Condensate oil Run 50 (APCI 1997CB)	Composite Blend "B" (APCI 2078CB)	Alternate Blend (APCI 2084CB)	True Alternate Blend (APCI 2201CB)
7	D1322	Smoke point(mm)	NR	12.0	NR	7.3	9.0	NR
8	D189	Carbon residue (%) (on 10% bottoms)	0.49	0.95	0.15	0.71	0.32	NR
9	D240	Heating value (Btu/lb)	NR	18,507	17,352	17,708 17,685[a]	17,879	NR
10	D130	Copper strip corrosion, rating	NR	D	NR	1A	D	NR
14	D1796	Bottoms sediment and water (vol %)	NR	<0.10	NR	<0.10	NR	NR
14a	APCI Method	Water by KF, (wt %)	0.50	0.04	NR	0.26	NR	0.21
15	D97	Pour point (°F, °C)	-10,-23	-35,-37	0,-18	-25,-32	-25,-32	NR
16	D2781	Compatibility Modified compatibility (blend 1/1-v/v with petroleum)	1	1	NR	1	1	1
17	D664	Neutralization # (mg/KOH/gm)	NR	14.76	NR	18.30	NR	NR
19	APCI Method	GC/MS analyses (Compound wt%) (20 major compounds)	NR				NR	NR
		1		Te-Hy-M-naphthalene (2.1)	Phenanthrene(7.2)	2-M-naphthalene (5.3)		
		2		Di-M-biphenyl(1.4)	2-M-naphthalene (6.2)	E-M-phenol(2.1)		
		3		2-M-naphthalene(1.3)	Di-benzofuran(2.7)	Naphthalene(1.8)		
		4		C4-alkyl-tetralin (1.2)	9-H-fluorene(2.4)	1,2,3,4,Te-Hy-M-naphthalene(1.8)		
		5		Octa-Hy-phenanthrene(1.1)	Naphthalene(2.4)	Phenanthrene(1.8)		
		6		1,2,3,4-Te-Hy-naphthalene(1.1)	1,2-Di-Hy-Acenaphthalene(2.1)	E-phenol(1.5)		
		7		P-Octa-Hy-phenanthrene(1.1)	Tetradecane(2.0)	Tetradecane(1.4)		
		8		Te-Hy-Di-M-napthalene(1.1)	1-M-naphthalene (1.9)	3 & 4-M-phenols (1.2)		
		9		Te-Hy-phenanthrene(1.0)	1,1'-Biphenyl (1.6)	Di-M-naphthalene(1.2)		
		10		3-(1,1-Di-M-E)1,2, Di-Hy-naphthalene (0.9)	Hexadecane(1.5)	M-9H-fluorene (1.0)		
		11		M-Octa-Hy-phenanthrene(0.9)	2-E-naphthalene(1.4)	Di-M-naphthalene(1.0)		

TABLE 9 (Cont.)
LIQUID PRODUCT CHARACTERIZATION - MIDDLE OILS

Test #	ASTM #	Description	Wilsonville SRC run 235 (APCI 2004CB)	Wilsonville HTU run 235 High conversion (APCI 2054CB)	Alcoa Coker Condensate oil Run 50 (APCI 1997CB)	Composite Run 50 (APCI 2078CB)	Alternate Blend "B" (APCI 2084CB)	True Alternate Blend (APCI 2201CB)
19 (Cont)								
		12		Unknown mixture (0.9)	3 & 4-M-phenols (1.4)	E-M-naphthalene(0.9)		NR
		13		Benzindan(0.9)	M-phenanthrene (1.3)	E-naphthalene(0.9)		
		14		Phenanthrene(0.8)	Heptadecane(1.2)	Te-Hy-Di-M-naphthalene(0.9)		
		15		1-Cy-He-3-M-benzene(0.8)	E-M-phenol(1.2)	1-M-naphthalene (0.8)		
		16		1,2,3,4,Te-Hy-2-M-naphthalene(0.8)	M-phenanthrene (1.1)	E-Te-Hy-naphthalene(0.8)		
		17		Unknown(0.8)	Di-M-naphthalene (1.1)	1,2,3,4-Te-Hy-naphthalene(0.8)		
		18		Unknown mixture (0.7)	E-phenol(1.1)	Di-M-naphthalene(0.8)		
		19		Unknown(0.7)	M-Debenzofuran (0.8)	Benzindan(0.8)		
		20		Unknown(0.7)	Di-M-naphthalene (0.8)	Benzindan(0.8)		
20	APCI method		Elemental analysis					
		C (wt %)	86.40	89.11	88.09	87.44	86.77	86.77
		H (wt %)	9.09	10.34	8.61	9.46	9.62	9.70
		N (wt %)	0.77	0.40	0.73	0.60	0.67	0.59
		O (wt %)	4.26	0.40	2.76	2.63	3.10	3.05
		S (wt %)	0.53*	<0.10	0.41	0.26	0.33*	0.38
		Cl (ppm)	29	9	23	22	20^b	-
21	APCI Method	Proton NMR spectrum(vol %)					NR	NR
		Paraffins	28.4	62.2	27.6	43.6		
		Olefins	15.8	0	6.7	7.5		
		Aromatics	55.8	37.8	65.7	48.9		
22	APCI Method	Molecular wt	NR	200	165	170	NR	NR
23	APCI Method	Near infrared spectrum(wt %)						
		O as OH	2.43	0.09	1.31	1.29	1.33	2.16
		N as NH	0.01	0.05	0.09	0.04	0.05	0.05
		N as NH_2	<0.01	0.09	<0.01	<0.01	0.01	0.15
24	APCI Method	Complete infrared spectrum functional group	NR	OH CH(aro) CH(sat) C=C(aro)	OH CH(aro) CH(sat) C=C(aro)	OH CH(aro) CH(sat) C=C(aro)	NR	NR
26	D3241	Oxidation stability						
		Heater tube deposit rating	NR	Too dark	NR	Too dark	Too dark	NR
		Pressure drop (mm Hg)	--	--	--	--	--	
29	D1500	Color (NPA)	NR	6.0	>8.0	>8.0	NR	NR

*Dohrmann Method

TABLE 9 (Cont.)
LIQUID PRODUCT CHARACTERIZATION - MIDDLE OILS

Test #	ASTM #	Description	Wilsonville SRC run 235 (APCI 2004CB)	Wilsonville HTU run 235 High conversion (APCI 2054CB)	Alcoa Coker Condensate oil Run 50 (APCI 1997CB)	Composite Blend "B" (APCI 2078CB)	Alternate Blend (APCI 2084CB)	True Alternate Blend (APCI 2201CB)
30	DuPont F21-61	DuPont stability (fuel rating/D1500 color ex)	Good/8+	Good/8+	Fair/8+	Marginal/8+	Good/8+	Marginal/7
33	APCI Method	Storage stability @ 110°F	See table 10	See table 10	NR	See table 10	See table 10	NR
34	D2117	Thermal conductivity (BTU, in/h, ft², °F)						
		at 60°F	0.846	0.790	0.853	0.825	NR	NR
		at 150°F	0.797	0.763	0.804	0.783		
35	APCI Method	Heat capacity (mcal/mg,°C) (Specific heat) at 100°C	0.58	0.52	0.55	0.59	NR	NR
36	APCI Method	Water/emulsion formation/ stability	Not emulsifiable	Not emulsifiable	NR	Not emulsifiable	NR	NR
37	BETC	Suspended sediment (mg/100ml)	0.02/0.03	NR	NR	0/0	0.05/0.05	-
		Adherent gum, (mg/100ml)	0.00/0.00			0/0	0/0.02	-
		Total (mg/100ml)	0.03			0/0	0.06	-
38	APCI	Hot filtration sediment (mg/100 ml)	01.0	01.0	NR	01.0	NR	NR

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[a] Repeat analysis obtained from Huffman Laboratories - Wheat Ridge, Colorado.
[b] Analysis obtained from Huffman Laboratories - Wheat Ridge, Colorado.
NR-Not requested
D-Deleted

Table 10

Effect of Correcting Elemental Analyses for Water Content

	2004CB (0.50% H_2O)		2054CB (0.04% H_2O)		2078CB (0.26% H_2O)	
	As is	Dry basis	As is	Dry basis	As is	Dry basis
C	86.40	86.83	89.11	89.15	87.44	87.67
H	9.09	9.08	10.34	10.34	9.46	9.46
N	0.77	0.77	0.40	0.40	0.60	0.60
O	4.26	3.84	0.40	0.36	2.63	2.41
S	0.46	0.46	<0.1	<0.1	0.26	0.26

Table 11

Round-Robin Analysis for N, S, and Cl

	2004CB	2084CB
% N		
APCI	0.77	0.67
Galbraith	0.75	0.77
Huffman	0.70	0.69
% S		
APCI (Leco)	0.46	0.41
APCI (Dohrmann)	0.53	0.33
Galbraith	0.53	0.36
Huffman	0.49	0.34
Cl (ppm)		
APCI	20	--
Galbraith	22	13
Huffman	20	20

TABLE 12
LIQUID PRODUCT AGING STUDY - MIDDLE OILS

Test			Wilsonville - SRC run 235 (APCI 2004CB)	Aged at 110°F			Wilsonville - HTU run 235 (APCI 2054CB)	Aged at 110°F	
no	ASTM	Description	Initial	1 Month	3 Months	5 Months	Initial	1 Month	3 Months
2.	D445	Liquid Viscosity (cSt)							
		68°F(20°C)	9.3	9.9	9.9	11.9	9.1	NR	9.4
		104°F(40°C)	4.5	4.7	D	NR	4.6	NR	D
		140°F(60°C)	2.8	2.9	D	NR	3.0	NR	D
3a	APCI method	Specific gravity							
		pycnometer @60°F(15.6°C)	--	0.9878	D	--	--	NR	D
		@77°F(25°C)	0.9818	0.9833	D	--	0.9540		D
8	D189	Carbon residue (%) (on 10% bottoms)	0.49	1.33	6.10	5.79	0.95	NR	0.64
15	D97	Pour point (°F,°C)	-10,-23	-10,-23	D	--	-35,-37	NR	D
16	D2781	Compatibility Modified compatibility Blend 1/1-v/v petroleum	1	2	2	--	1	NR	2
23	APCI method	Near infrared spectrum (wt %)							
		OH	2.43	2.17	D	--	0.09	NR	D
		NH	0.01	0.02	D	--	0.05	NR	D
		NH_2	<0.01	<0.01	D	--	0.09	NR	
30	DuPont F21-61	DuPont stability (fuel rating/D1500 color ex)	Good/8+	Poor/8+	V.Poor/8+		Good/8+	NR	Fair/8+
37	BETC	Suspended sediment (mg/100 ml)	0.02/0.03	1.4/0.9	2.5	4.8	NR	NR	1.1
		Adherent gum (mg/100 ml)	0/0	0.4/0.2	0.5	0.6	NR	NR	0.2
		Total (mg/100 ml)	0.03	1.5	3.0	5.4	NR	NR	1.3

TABLE 12 (Cont)
LIQUID PRODUCT AGING STUDY - MIDDLE OILS

Test No	ASTM	Description	Composite - Blend B (APCI 2078CB) Aged at 110°F Initial	Composite - Blend B (APCI 2078CB) Aged at 110°F 1 Month	Composite - Blend B (APCI 2078CB) Aged at 110°F 3 Months	Composite - Blend B (APCI 2078CB) Aged at 110°F 5 Months	Alternate - Blend (APCI 2084CB) Aged at 110°F Initial	Alternate - Blend (APCI 2084CB) Aged at 110°F 1 Month*	Alternate - Blend (APCI 2084CB) Aged at 110°F 3 Months
2	D445	Liquid Viscosity (cSt)							
		68°F (20°C)	9.2	9.5	10.0	10.1	8.2	8.6	9.1
		104°F (40°C)	4.5	4.7	D	--	4.1	4.7	D
		140°F (60°C)	2.8	2.9	D	--	2.6	2.7	D
3a	APCI method	Specific gravity							
		pycnometer @60°F (15.6°C)	--	0.9769	D	--	0.9717	0.9726	D
		@77°F (25°C)	0.9719	0.9724	D	--	--	0.9680	D
8	D189	Carbon residue (%) (on 10% bottoms)	0.71	1.42	1.72	2.53	0.32	1.05	2.09
15	D97	Pour point (°F, °C)	-25,-32	-15,-26	D	--	-25,-32	-45,-43	D
16	D2781	Compatibility Modified compatibility (blend 1/1-v/v petroleum)	1	2	2	--	1	3	2
23	APCI method	Near infrared spectrum (wt %)							
		OH	1.29	1.24	D	--	1.33	1.49	D
		NH	0.04	0.05	D	--	0.05	0.04	D
		NH_2	<0.01	<0.01	D	--	0.01	<0.01	D
30	DuPont F21-61	DuPont stability (fuel rating/D1500 color ex)	Marginal/8+	Poor/8+	V.poor/8+	--	Good/8+	Fair/8+	V.Poor/8+
37	BETC	Suspended sediment (mg/100 ml)	0/0	0.7/0.8	1.8	1.8	0.05/0.05	0.2/0.4	1.8
		Adherent gum (mg/100 ml)	0/0	0.1/0.1	0.2	0.1	0/0.02	0.2/0.4	0.3
		Total (mg/100 ml)	0/0	0.9	2.0	1.9	0.06	0.6	2.1

*New true alternate middle oil-aged has following BETC analysis:
Suspended sediment (mg/100 ml) 1.3 (1 month), 0.8 (3 month)
Adherent gum (mg/100 ml) <0.2 (1 month), <0.2 (3 month)
Total (mg/100 ml) 1.3 (1 month), 0.8 (3 month)

TABLE 13
LIQUID PRODUCT CHARACTERIZATION - HEAVY OILS

Test #	ASTM	Description	Wilsonville - SRC 235 APCI 2076CB (Blend of 2005CB & 2009CB)	Wilsonville - HTU run 235 (APCI 2055CB)	Composite - Heavy oil Blend "B" (APCI 2079CB)
		Vol % Dist		Vapor temperature, °F	
1	D2887	IBP	698	608	648
		5	744	681	702
		10	759	695	719
		15	772	705	731
		20	782	712	743
		30	800	723	767
		40	816	733	790
		50	833	743	811
		60	852	752	832
		70	874	761	855
		80	902	772	882
		90	945	791	920
		95	986	837	956
		FBP	1043	968	1038
		Residue (%)	4	<3	<3
2	D445	Liquid viscosity (cSt)			
		140°F(60°C)	--	286	564
		175°F(79°C)	687	--	--
		210°F(99°C)	114	--	--
3	D1298	Liquid specific gravity @60°F	Could not be measured	1.0261	1.0736
		API gravity @60°F		6.4	0.3
3a	APCI method	Specific gravity, pycnometer, @60°F (15.6°C)	--	--	1.068
		@77°F (25.0°C)	1.112	1.030	--
5	D2386	Freeze point(°F,°C)	127,53	18,-8	54,12
6	D3828	Flash point (°F,°C)	NR	>230,>110	>230,>110
8	D189	Carbon residue (%) (total sample)	3.44	0.06	1.32
9	D240	Heating value (Btu/lb)	NR	18,195	17,625
10	D130	Copper strip corrosion,rating	NR	1A	1B
14	D1796	Bottoms sediment and water (vol %)	0.8	<0.10	3.0
15	D97	Pour point (°F, °C)	>120,>49	50,10	100,38

TABLE 13 (Cont)

LIQUID PRODUCT CHARACTERIZATION - HEAVY OILS

Test #	ASTM	Description	Wilsonville - SRC 235 APCI 2076CB (Blend of 2005CB & 2009CB)	Wilsonville - HTU run 235 (APCI 2055CB)	Composite - Heavy oil Blend "B" (APCI 2079CB)
16	D2781	Compatibility Modified compatibility (blend 1/1-v/v with petroleum)	5	3	5
17	D664	Neutralization # (mg/KOH/gm)	NR	18.6	22.3
19	APCI method	GC/MS analyses (Compound wt %) (20 major compounds)	NR		
		1		M-pyrene(4.3)	M-pyrene(2.4)
		2		Pyrene(4.1)	Pyrene(2.4)
		3		Unknown mixture-MW218(3.5)	M-Tetra-Hy-pyrene(?)(2.0)
		4		Unknown mixture-MW220(3.3)	M-Phenyl naphthalene or M-Di-Hy-pyrene(?)(1.8)
		5		Unknown mixture(2.9)	M-Tetra-Hy-pyrene(?)(1.8)
		6		Unknown mixture(2.8)	Unknown M.W.232 species(1.4)
		7		Alkane (2.8)	Unknown mixture(1.4)
		8		Unknown mixture (2.7)	Unknown mixture(1.4)
		9		Unknown mixture-MW218(2.5)	No data available(1.4)
		10		Unknown mixture(2.4)	Unknown mixture(1.3)
		11		No data available(2.3)	E-pyrene(1.2)
		12		Complex mixture(2.3)	Unknown mixture(1.2)
		13		Complex mixture(2.3)	No data available(1.2)
		14		Complex mixture(2.0)	Unknown mixture(1.2)
		15		Unknown mixture(2.0)	Unknown mixture(1.2)
		16		No data available(1.9)	No data available(1.2)
		17		No data available(1.9)	M-pyrene(1.2)
		18		Complex mixture(1.9)	No data available(1.2)
		19		No data available(1.9)	No data available(1.1)
		20		No data available(1.9)	Unknown mixture(1.1)
20	APCI method	Elemental analysis			
		C (wt %)	87.27	90.14	89.28
		H (wt %)	7.57	9.10	8.58
		N (wt %)	1.57	0.64	0.98
		O (wt %)	3.75	0.77	1.41
		S (wt %)	0.31	0.01	0.15
		Cl (ppm)	16	<2	5

TABLE 13 (Cont)
LIQUID PRODUCT CHARACTERIZATION - HEAVY OILS

Test #	ASTM #	Description	Wilsonville - SRC 235 APCI 2076CB (Blend of 2005CB & 2009CB)	Wilsonville - HTU run 235 (APCI 2055CB)	Composite - Heavy oil Blend "B" (APCI 2079CB)
21	APCI method	Proton NMR spectrum(vol %)	NR		
		Paraffins		56.0	50.6
		Olefins		0	0
		Aromatics		44.0	49.4
22	APCI method	Molecular wt	NR	265	315
23	APCI method	Near infrared spectrum (wt %)			
		O as OH	NR	0.15	0.38
		N as NH		0.14	0.16
		N as NH$_2$		0.07	0.10
24	APCI method	Complete infrared spectrum functional group	NR	CH(aro) CH(sat) C=C(aro)	OH CH(aro) CH(sat) C=C(aro)
27	D482	Ash (wt %)	NR	0.002	0.023
33	APCI method	Storage stability @ 110°F	See table 14	See table 14	See table 14
34	D2117	Thermal conductivity (BTU, in/hr, ft^2, °F)			
		at 60°F (16°C)	0.998	0.804	0.825
		150°F (66°C)	1.012	0.783	0.783
		400°F (204°C)	1.040	0.770	0.721
35	APCI method	Heat capacity,(mcal/mg,°C)			
		(Specific heat) at 212°F (100°C)	0.42	0.49	0.46
		392°F (200°C)	0.49	0.65	0.56
38	APCI method	Hot filtration sediment (mg/100 ml)	NF	4.3	NF

NR-Not requested
NF-Not filterable

TABLE 14
LIQUID PRODUCT AGING STUDY - HEAVY OILS

Test no	ASTM	Description	Wilsonville - SRC 235 (APCI 2076CB) (Blend of 2005CB & 2009CB) Aged at 110°F Initial	1 Month	3 Months	5 Months	Wilsonville - HTU run 235 (APCI 2055CB) Aged at 110°F Initial	1 Month	3 Months	Composite - Heavy Oil "Blend B" (APCI 2079CB) Aged at 110°F Initial	1 Month	3 Months	5 Months
2	D445	Liquid viscosity (cSt)											
		140°F (60°C)	--	--	--	--	286	221	230	564	545	537	630
		175°F (79°C)	687	1219	D		--	47.6	--	--	91.3	--	--
		210°F (99°C)	114	182	180	Ins	--	40.3	--	--	28.0	--	30.7
8	D189	Carbon residue (%) (total sample)	3.44	3.47	8.06	7.50	0.06	0.47	0.67	1.32	2.31	2.76	6.20
14	D1796	Bottoms sediment and water (vol %)	0.80	4.0	4.0	20.4	<0.10	0.26	<0.10	3.0	6.0	4.0	7.0
15	D97	Pour point (°F,°C)	>120,>49	D	D	--	50,10	70,21	D	100,38	75,24	D	--
--	APCI Method	Pentane insolubles,wt%	--	--	--	14.6	--	--	--	--	--	--	4.1

D-Deleted
INS-Insufficient sample
*See Table 15 for sediment analysis

TABLE 15

ANALYSIS OF HEAVY OIL SRC SEDIMENT FROM TEST 14 (TABLE 14)

Test no	ASTM	Description	Wilsonville-SRC 235 (APCI 2076CB)	Wilsonville-HTU run 235 (APCI 2055CB)	Composite-Heavy Oil (APCI 2079CB)
		Vol % Dist			
1	D2887	IBP	258	652	580
		5	744	702	673
		10	770	720	689
		15	787	737	702
		20	801	753	714
		30	826	779	737
		40	849	800	761
		50	872	819	784
		60	894	838	809
		70	920	860	838
		80	954	885	871
		90	1005	921	914
		95	1039	952	949
		FBP	1073	1015	1015
		Residue (%)	5.9	<3	4
20	APCI method	Elemental Analysis			
		C	83.3	87.9	87.6
		H	6.9	8.4	8.0
		N	1.5	0.9	1.2
		O	4.3	1.7	2.2
		S	1.4	0.3	0.4

TABLE 16
LIQUID PRODUCT CHARACTERIZATION - NAPHTHA END POINT SPECIFICATION

Test #	ASTM #	Description	Cut 1 IBP-325°F (APCI2080 CB1)	Cut 2 325°F-350°F (APCI 2080 CB2)	Cut 3 350-375°F (APCI 2080 CB3)	Cut 4 375°F-400°F (APCI 2080 CB4)	Cut 5 400°F-BTMS (APCI 2080 CB5)
A	APCI method	Weight % distilled	58.5	11.9	11.8	10.5	5.9
				Vapor temperature (°F)			
		Vol.% Dist.					
1	D2887	IBP	110	306	338	329	300
		5	163	334	353	374	404
		10	183	341	363	383	412
		15	201	344	368	389	417
		20	212	348	373	393	422
		30	241	353	380	400	431
		40	250	358	386	405	438
		50	271	363	390	409	448
		60	295	367	394	413	462
		70	311	372	398	418	520
		80	334	378	405	423	659
		90	348	389	419	436	794
		95	370	426	492	586	912
		FBP	713	787	723	882	1027
		Residue (%)	<3	<3	<3	<3	<3
3	D1298	Liquid specific gravity @60°F	0.7905	0.8956	0.9129	0.9377	0.9465
		API gravity @60°F	47.5	26.5	23.5	19.4	18.0
3a	APCI method	Specific gravity, pycnometer, @60°F (15.6°C)	0.7891	0.8932	0.9113	0.9378	0.9489
11	D381	Existent gum (mg/100mL)	14	92	53	INS	46
19	APCI method	GC/MS analyses (Compound wt %) (20 major compounds)					
		1	Toluene and a C_8-alkane-10-20% (10.7)	Phenol(20)	3 & 4-M-phenol (17.2)	3 & 4-M-phenol (26)	Naphthlene(9.5)
		2	M-Cy-Hexane(7.6)	Cis-Octa-Hy-1H-indene and Bu-Cy-hexane 40%(5.6)	2-M-phenol(12.8)	2-M-phenol(7.5)	Di-M-phenol(6.9)
		3	Phenol(6.9)	Decane(5.6)	Undecane(6.9)	Di-M-indan & C_5-alkylbenzene (6.5)	1,2,3,4 Tetra-Hy-naphthalene(5.9)
		4	Cy-hexane and benzene-10%(6.5)	Tri-M-benzene (5.1)	M-indan & C_4-alkylbenzene(6.1)	Tetralin(6.4)	M-2,3-Di-Hy-1H-indene(4.0)
		5	E-Cy-hexane(4.0)	2-M-phenol(4.5)	Indan(4.8)	M-indan(6.3)	2-M-tetralin(3.4)
		6	Di-M-benzene(3.6)	Indan(4.2)	Phenol(4.3)	Dodecane(4.9)	4-E-phenol(3.3)
		7	M-Cy-pentane(3.3)	E-M-benzene(3.8)	M-propylbenzene (3.5)	M-indan & C_4-alkylbenzene(3.3)	Tridecane(3.0)
		8	Octane(3.1)	n-Bu-Cy-hexane(2.7)	E-Di-M-benzene(3.1)	Di-M-indan(1.9)	Bu-toluene(2.9)
		9	Hexane(2.8)	Octa-Hy-M-indene (2.4)	M-indan & Di-M-indan(2.5)	2,6-Di-M-undecane or C_{13}-alkane(1.8)	Di-M-indan(2.5)
		10	Heptane(2.3)	2-6-Di-M-nonane (2.3)	Di-E-benzene(1.4)	Naphthalene(1.7)	M-phenol(2.4)

TABLE 16 (Cont)
LIQUID PRODUCT CHARACTERIZATION - NAPHTHA END POINT SPECIFICATION

Test #	ASTM #	Description	Cut 1 IBP-325°F (APCI 2080 CB1)	Cut 2 325°F-350°F (APCI 2080 CB2)	Cut 3 350-375°F (APCI 2080 CB3)	Cut 4 375°F-400°F (APCI 2080 CB4)	Cut 5 400°F-BTMS (APCI 2080 CB5)
19(Cont)							
		11	Nonane(2.1)	Tri-M-benzene(2.0)	M-P-benzene(1.4)	Alkane & Bu-M-benzene(1.7)	5 or 6-M-tetralin(2.4)
		12	P-Cy-Hexane(1.9)	Undecane(2.0)	Bu-benzene(1.4)	Di-M-phenol & M-decalin or Di-M-Octa-Hy-indan (1.7)	Di-M-indan(2.1)
		13	Pentane(1.8)	Pentyl-Cy-pentane and a C_3-alkyl phenol(2.0)	Tri-M-benzene(1.4)	Di-M-phenol & C_4-alkyl-benzene or M-indanol(1.4)	1-M-tetralin(2.0)
		14	E-benzene(1.8)	E-M-benzene(1.9)	E-1,2,4-Tri-M-benzene(1.3)	p-Iso-Bu-toluene (1.2)	2-M-naphthalene(1.3)
		15	Di-M-Cy-hexane(1.6)	3 & 4-M-phenol(1.7)	Bu-M-Cy-hexane & M-decalin(1.3)	Undecane(1.2)	Di-M-indan(1.3)
		16	Di-M-benzene(1.6)	Tri-M-benzene(1.6)	C_4-alkylbenzene (1.2)	E-phenol(1.2)	Di-M-phenol(1.2)
		17	E-Cy-Pentane(1.4)	Octa-Hy-M-indene (1.5)	Alkane & 1-2 other components(1.2)	E-phenol(1.2)	M-indan(1.1)
		18	C_6-alkane(1.3)	E-M-benzene(1.4)	Tri-M-benzene(1.2)	Di-M-indan(1.1)	3-E-phenol(1.1)
		19	3-M-Cy-hexene(1.1)	M-isopropyl-Cy-hexane(1.4)	Dodecane(1.1)	Tetra-M-benzene (1.1)	Di-M-indan(1.1)
		20	E-M-or isopropyl benzene(1.1)	M-isopropyl benzene & a Bu-Cy-hexane-25% (1.4)	M-indan(1.1)	Di-M-indan(0.9)	Alkane(1.0)
20	APCI method	Elemental analysis					
		C (wt %)	85.15	83.17	83.43	82.94	83.82
		H (wt %)	13.01	10.80	10.47	9.81	9.85
		N (wt %)	0.09*	0.13*	0.26*	0.41	1.31
		O (wt %)	1.69	5.25	5.48	6.28	4.02
		S (wt %)	0.54	1.00	0.76	0.82	1.31
		Cl (ppm)	3	22	16	28	35
21	APCI method	Proton NMR spectrum(vol %)					
		Paraffins	68.4	48.8	36.9	28.6	37.9
		Olefins	7.8	15.9	20.8	24.4	20.0
		Aromatics	23.8	35.3	42.3	47.0	42.1
23	APCI method	Near infrared spectrum(wt %)					
		O as OH	0.57	4.04	3.00	3.43	1.65
		N as NH	<0.01	<0.01	<0.01	<0.01	0.07
		N as NH_2	<0.01	<0.01	<0.01	<0.01	0.13
25	APCI method	Caustic extraction with 20% NaOH (% wt loss)	6.7	25.9	34.8	43.6	35.0

INS Insufficient sample
*Dohrman N

Table 17

Analysis of Naphtha Cuts

Cut no.	% yield	Wt %	% of total	Total %/ % yield	Wt %	% of total	Total %/ % yield
		Phenolics			Dicyclics		
1	58.5	6.9	24.6	0.4	-	-	-
2	11.9	28.2	20.5	1.7	9.5	13.4	1.1
3	11.8	34.3	24.7	2.1	15.8	22.0	1.9
4	10.5	39.0	25.0	2.4	31.2	38.9	3.7
5	5.9	14.9	5.4	0.9	36.7	25.7	4.4
		Nitrogen			Oxygen		
1	58.5	0.09	24.1	0.4	1.69	31.2	0.5
2	11.9	0.13	7.3	0.6	5.25	19.9	1.7
3	11.8	0.26	14.1	1.2	5.48	20.5	1.7
4	10.5	0.41	19.6	1.9	6.28	20.8	2.0
5	5.9	1.31	35.0	5.9	4.02	7.6	1.3
		Sulfur			Chlorine		
1	58.5	0.54	45.9	0.8	3	15.6	0.3
2	11.9	1.00	17.3	1.5	22	23.2	2.0
3	11.8	0.76	13.1	1.4	16	16.8	1.4
4	10.5	0.82	12.5	1.2	28	26.1	2.5
5	5.9	1.31	11.2	1.9	35	18.3	3.1

TABLE 18
LIQUID PRODUCT CHARACTERIZATION - PETROLEUM OILS

Test	ASTM	Description	Vacuum Gas Oil (APCI 2051CB)	#2 Fuel Oil (APCI 2050CB)	#6 Fuel Oil Low Sulfur (APCI 2048CB)	#6 Fuel Oil High Sulfur (APCI 2049CB)
1	D2887	Vol % Dist		Vapor temperature (°F)		
		IBP	460	333	402	391
		5	553	493	577	555
		10	599	507	710	590
		15	623	515	806	674
		20	648	548	838	748
		30	687	568	875	843
		40	719	584	892	889
		50	749	601	908	923
		60	780	689	926	967
		70	813	743	948	1011
		80	851	810	978	1052
		90	904	844	1014	1094
		95	952	872	1035	1121
		FBP	1036	929	1055	1148
		Residue (%)	<3	<3	40	25
2	D445	Liquid viscosity (cSt)				
		68.5°F(20°C)	55.6	4.5	--	--
		175°F(79°C)	--	--	79.6	43.3
		210°F(99°C)	--	--	34.5	22.6
3	D1298	Liquid specific gravity @60F	0.8844	0.8408	0.9659	0.9745
		API gravity @60°F	28.5	36.8	15.0	13.7
3a	APCI Method	Specific pycnometer gravity @60°F(15.6°C)	0.8865	0.8385	0.9831	0.9781
5	D2386	Freeze point (°F,°C)	-37,-38	<-76,<-60	23,-5	0,-18
8	D189	Carbon residue, (%)				
		(on 10% bottoms)	0.36	0.15	--	--
		(total sample)	--	--	9.74	11.93
10	D130	Copper strip corrosion, rating	1A	1A	1B	1B
14	D1796	Bottoms sediment and water (vol %)	<0.10	<0.10	0.45	0.10
15	D97	Pour point (°F, °C)	85,29	0,-18	65,19	30,-1
16	D2781	Compatibility Modified compatibility (blend 1/1-v/v with petroleum)	2	NR	5	1
17	D664	Neutralization # (mg/KOH/gm)	0.11	0.04	3.06	1.26

TABLE 18 (Cont)
LIQUID PRODUCT CHARACTERIZATION - PETROLEUM OILS

Test	ASTM	Description	Vacuum Gas Oil (APCI 2051CB)	#2 Fuel Oil (APCI 2050CB)	#6 Fuel Oil Low Sulfur (APCI 2048CB)	#6 Fuel Oil High Sulfur (APCI 2049CB)
20	APCI method	Elemental analysis				
		C (wt %)	87.11	86.78	87.73	86.13
		H (wt %)	12.85	13.53	11.05	10.88
		N (wt %)	0	0	0.39	0.22
		O (wt %)	0.33	0.14	0.61	0.38
		S (wt %)	0.42,0.26[a]	0.14,0.12[a]	0.63,0.50[a]	2.98,2.60[a]
		Cl (ppm)	12	76	53	25
21	APCI method	Proton NMR spectrum (vol %)				
		Paraffins	85.9	86.4	77.5	78.2
		Olefins	0	0	0	0
		Aromatics	13.1	13.6	22.5	21.8
22	APCI method	Molecular wt	300	215	440	390
26	D3241	Oxidation stability	NR	4	NR	NR
		Heater tube deposit rating		94		
		Pressure drop, (mm Hg)				
30	DuPont method F21-61	DuPont stability (fuel rating/ D1500 color ex)	Poor/7.5	Excellent/1	NR	NR
33	APCI method	Storage stability @110°F	See table 25	See table 26	See table 27	See table 28
37	BETC	Suspended sediment (mg/100 ml)	NR	0.0/0.1	NR	NR
		Adherent gum (mg/100 ml)	--	0.0/00	--	--
		Total (mg/100 ml)	--	0.1	--	--
38	APCI method	Hot filtration sediment (mg/100 ml)	5.2	2.0	NF	24.9

[a] Repeat analysis by Huffman Laboratories - Wheat Ridge, Colorado.
NR-Not requested
NF-Not filterable

TABLE 19
LIQUID PRODUCT AGING STUDY - MIDDLE OILS
G.C. SIMULATED DISTILLATIONS

Test no	ASTM	Description	Wilsonville - SRC run 235 (APCI 2004CB) Initial	Aged at 110°F 1 Month	3 Months	5 Months	Wilsonville - HTU run 235 (APCI 2054CB) Initial	Aged at 110°F 1 Month	3 Months
1	D2887	IBP	376	377	387	464	386	NR	398
		5	411	405	428	547	488		451
		10	423	418	445	573	501		482
		15	432	429	460	595	510		506
		20	444	444	476	611	521		528
		30	461	470	498	635	552		563
		40	473	495	514	660	581		592
		50	493	522	534	683	606		615
		60	516	547	558	706	631		634
		70	547	583	594	730	656		653
		80	601	627	640	756	690		672
		90	661	692	696	789	726		694
		95	708	746	741	816	786		708
		FBP	808	909	836	888	887		741
		Residue (%)		<3	<3	6			<3

TABLE 19 (Cont)
LIQUID PRODUCT AGING STUDY - MIDDLE OILS
G.C. SIMULATED DISTILLATIONS

Test No	ASTM	Description	Composite - Blend B (APCI 2078CB) Aged at 110°F Initial	1 Month	3 Months	5 Months	Alternate - Blend (APCI 2084CB) Aged at 110°F Initial	1 Month	3 Months
1	D2887	IBP	381	370	371	532	367	339	378
		5	415	407	407	582	416	403	443
		10	437	421	422	603	432	419	461
		15	454	442	443	618	450	492	476
		20	464	458	459	629	464	500	492
		30	504	486	490	650	490	513	518
		40	544	516	516	668	523	561	538
		50	584	545	545	685	557	591	569
		60	617	579	579	701	597	714	609
		70	642	613	613	719	629	820	648
		80	669	646	646	748	662	870	687
		90	698	681	681	800	696	897	728
		95	725	706	705	898	721	915	754
		FBP	849	842	819	1017	900	1054	829
		Residue (%)	<3	9	<3	5	<3	<3	<3

TABLE 20
LIQUID PRODUCT AGING STUDY - HEAVY OILS
G.C. SIMULATED DISTILLATIONS

			Wilsonville - SRC 235 (APCI 2076CB) (Blend of 2005CB & 2009CB) Aged at 110°F				Wilsonville - HTU run 235 (APCI 2055CB) Aged at 110°F			Composite - Heavy Oil "Blend B" (APCI 2079CB) Aged at 110°F			
Test no	ASTM	Description	Initial	1 Month	3 Months	5 Months	Initial	1 Month	3 Months	Initial	1 Month	3 Months	5 Months
1	D2887	IBP	698	652	702	642	608	547	748	648	659	647	620
		5	744	704	759	753	681	695	813	702	714	706	696
		10	759	722	774	773	695	716	828	719	732	725	715
		15	772	738	785	787	705	733	840	731	748	740	731
		20	782	754	795	799	712	748	851	743	763	753	746
		30	800	783	813	821	723	774	875	767	791	779	774
		40	816	809	829	842	733	797	897	790	814	801	800
		50	833	832	846	863	743	817	918	811	832	822	825
		60	852	856	865	885	752	839	940	832	850	843	850
		70	874	882	887	909	761	863	964	855	871	866	878
		80	902	908	915	940	772	893	990	882	895	894	909
		90	945	945	958	984	791	937	1019	920	932	932	956
		95	986	978	1000	1016	837	1026	1035	956	967	972	994
		FBP	1043	1043	1068	1055	968	1142	1057	1038	1027	1057	1046
		Residue (%)	5	4	<3	8	<3	<3	27	<3	<3	<3	3

D-Deleted
INS-Insufficient sample
*See Table 14 for sediment analysis

TABLE 21
LIQUID PRODUCT AGING STUDY
PETROLEUM (VACUUM GAS OIL*)/COAL (MIDDLE OIL**) BLENDS
G.C. SIMULATED DISTILLATIONS

Test no	ASTM	Description	Vacuum gas oil (2051CB) Initial	Vacuum gas oil Aged at 110°F 1 month	10% Coal Oil (2119CB1) Initial	10% Coal Oil Aged at 110°F 1 month	25% Coal oil (2119CB2) Initial	25% Coal oil Aged at 110°F 1 month
1	D2887	**Vol % Dist**						
		IBP	460	413	518	557	408	430
		5	553	509	576	752	518	534
		10	599	547	720	769	591	580
		15	623	577	826	782	702	611
		20	648	603	860	794	788	635
		30	687	641	887	817	851	675
		40	719	675	906	840	884	707
		50	749	706	924	864	903	739
		60	780	736	943	888	924	771
		70	813	771	965	912	948	806
		80	851	814	991	939	978	847
		90	904	872	1031	977	1019	905
		95	952	910	1065	1007	1055	956
		FBP	1036	1007	1137	1049	1138	1030
		Residue (%)	<3	<3	<3	3	<3	<3

TABLE 21 (Cont)
LIQUID PRODUCT AGING STUDY
PETROLEUM (VACUUM GAS OIL*)/COAL (MIDDLE OIL**) BLENDS
G.C. SIMULATED DISTILLATIONS

Test no	ASTM	Description	50% Coal Oil (2119CB3) Initial	50% Coal Oil Aged at 110°F 1 month	75% Coal Oil (2119CB4) Initial	75% Coal Oil Aged at 110°F 1 month	90% Coal Oil (2119CB5) Initial	90% Coal Oil Aged at 110°F 1 month
1	D2887	Vol % Dist			Vapor Temperature, °F			
		IBP	408	410	505	393	363	397
		5	471	485	535	442	424	437
		10	520	534	574	469	458	465
		15	562	570	600	492	476	488
		20	593	597	703	515	502	512
		30	633	640	822	561	542	557
		40	665	675	868	601	584	598
		50	695	710	891	633	623	629
		60	729	744	912	665	655	660
		70	769	781	937	697	690	693
		80	815	824	968	752	735	746
		90	877	880	1010	834	819	824
		95	930	926	1047	898	882	885
		FBP	1031	994	1135	1030	1003	999
		Residue (%)	4	<3	<3	<3	<3	<3

*Vacuum Gas Oil - 2051CB
**Composite Middle Oil - 2078CB
NR-Not requested
TV-Too viscous

TABLE 22
LIQUID PRODUCT AGING STUDY
PETROLEUM (#2 FUEL OIL*)/COAL (MIDDLE OIL**) BLENDS
G.C. SIMULATED DISTILLATIONS

Test no	ASTM	Description	#2 Fuel Oil - (APCI 2050CB) Aged at 110°F Initial	1 month	3 months	10% Coal Oil (APCI 2120CB1) Aged at 110°F Initial	1 month	3 months	25% Coal Oil (APCI 2120CB2) Aged at 110°F Initial	1 month	3 months
1	D2887	Vol % Dist				Vapor Temperature, °F					
		IBP	333	412	309	345	386	314	360	357	311
		5	493	507	422	446	427	416	444	433	413
		10	507	546	462	474	457	449	471	461	445
		15	515	577	483	491	473	467	489	478	467
		20	548	602	496	504	493	481	502	492	484
		30	568	640	518	522	530	504	522	514	510
		40	584	667	535	539	567	523	539	534	529
		50	601	695	553	556	602	541	557	552	550
		60	589	731	572	576	630	560	578	573	571
		70	743	771	591	596	659	581	600	593	592
		80	810	817	613	616	691	608	621	615	615
		90	844	875	641	644	763	634	651	646	645
		95	872	921	656	668	834	659	675	671	668
		FBP	929	1011	805	843	971	811	811	792	759
		Residue (%)	<3	<3	5	<3	<3	4	<3	<3	15

TABLE 22 (Cont)
LIQUID PRODUCT AGING STUDY
PETROLEUM (#2 FUEL OIL*)/COAL (MIDDLE OIL**) BLENDS
G.C. SIMULATED DISTILLATIONS

Test no	ASTM	Description	50% Coal Oil (APCI 2120CB3) Aged at 110°F Initial	1 month	3 months	75% Coal Oil (APCI 2120CB4) Aged at 110°F Initial	1 month	3 months	90% Coal Oil (APCI 2120CB5) Aged at 110°F Initial	1 month	3 months
1	D2887	Vol % Dist				Vapor Temperature, °F					
		IBP	366	352	345	381	388	370	386	386	363
		5	440	434	414	431	428	412	428	425	412
		10	467	464	439	460	458	431	455	452	432
		15	486	484	457	476	474	450	471	468	451
		20	500	497	470	492	492	468	485	484	465
		30	522	520	497	519	526	497	516	513	492
		40	540	538	519	540	558	525	541	540	518
		50	559	558	542	564	587	553	569	567	542
		60	581	579	565	590	614	582	601	595	569
		70	604	599	592	616	637	612	627	619	598
		80	626	620	619	642	663	642	655	646	626
		90	656	651	653	676	691	674	685	675	662
		95	683	677	680	700	721	698	708	702	686
		FBP	844	811	782	813	862	887	880	835	807
		Residue, %	4	<3	<3	<3	6	<3	<3	3	<3

NR-Not requested
D-Deleted
* #2 Fuel Oil - APCI 2050CB
** Composite Middle Oil - APCI 2078CB

TABLE 23
LIQUID PRODUCT AGING STUDY
PETROLEUM (#6 FUEL OIL-LOW SULFUR*)/COAL (HEAVY OIL**) BLENDS
G.C. SIMULATED DISTILLATIONS

Test no	ASTM	Description	#6 Fuel Oil (Low Sulfur) (2048CB) Aged at 110°F Initial	1 Month	3 Months	5 Months	10% Coal Oil (2122CB1) Aged at 110°F Initial	1 Month	3 Months	5 Months	25% Coal Oil (2122CB2) Aged at 110°F Initial	1 Month	3 Months
1	D2887	Vol % Dist					Vapor Temperature, °F						
		IBP	402	378	419	367	515	411	451	401	520	468	501
		5	577	508	534	497	618	530	573	515	625	598	639
		10	710	559	582	541	647	578	620	567	658	644	701
		15	806	596	613	576	666	609	649	602	681	671	738
		20	838	616	634	602	679	633	670	627	699	692	762
		30	875	644	667	640	705	670	709	665	729	725	802
		40	892	669	695	669	730	701	737	697	757	756	833
		50	908	694	727	695	757	730	761	729	785	785	865
		60	926	726	764	723	784	766	805	763	812	812	897
		70	948	765	813	756	813	804	845	800	839	841	928
		80	978	814	875	794	845	848	894	842	870	874	964
		90	1014	874	954	841	882	908	955	890	907	912	1011
		95	1035	908	1003	866	902	949	998	919	929	935	1040
		FBP	1055	942	1054	891	920	989	1027	948	950	956	1073
		Residue (%)	40	29	41	20	34	25	13	31	25	24	24

TABLE 23 (Cont)
LIQUID PRODUCT AGING STUDY
PETROLEUM (#6 FUEL OIL-LOW SULFUR*)/(COAL (HEAVY OIL**) BLENDS
G.C. SIMULATED DISTILLATIONS

			50% Coal Oil (2122CB3) Aged at 110°F				75% Coal Oil (2122CB4) Aged at 110°F			90% Coal Oil (2122CB5) Aged at 110°F			
Test no	ASTM	Description	Initial	1 Month	3 Months	5 Months	Initial	1 Month	3 Months	Initial	1 Month	3 Months	5 Months
1	D2887	Vol % Dist				Vapor Temperature, °F							
		IBP	498	513	519	419	550	539	737	604	598	590	510
		5	636	643	668	576	684	683	815	699	698	714	677
		10	680	685	727	634	710	709	837	718	718	738	703
		15	704	707	759	670	727	727	848	734	735	754	720
		20	720	724	780	693	742	743	856	750	751	768	736
		30	751	755	810	727	769	773	873	770	777	796	765
		40	777	782	839	759	793	798	890	786	800	820	793
		50	802	806	866	789	814	820	906	801	820	843	818
		60	826	830	893	818	836	843	922	818	842	866	844
		70	853	857	919	849	861	869	941	837	866	892	873
		80	888	888	951	884	893	899	965	863	896	924	905
		90	937	928	997	929	941	946	1004	905	940	973	951
		95	973	953	1030	960	984	983	1037	935	974	1013	988
		FBP	1012	977	1065	997	1040	1026	1073	1007	1012	1057	1038
		Residue (%)	14	11	<3	14	4	12	5	<3	3	5	6

* #6 Fuel Oil (Low Sulfur) - APCI2048CB
** Composite Heavy Oil Blend "B" 2079CB
*** See Table 14 for sediment analysis
D-Deleted
NR-Not requested
NF-Not filterable
INS-Insufficient sample

TABLE 24
LIQUID PRODUCT AGING STUDY
PETROLEUM (#6 FUEL OIL-HIGH SULFUR*)/COAL (HEAVY OIL BLENDS**)
G.C. SIMULATED DISTILLATIONS

Test no	ASTM	Description	#6 Fuel Oil (High Sulfur) (2049CB) Aged at 110°F Initial	 1 month	10% Coal Oil (2124CB1) Aged at 110°F Initial	 1 month	25% Coal Oil(2124CB2) Aged at 110°F Initial	 1 month
1	D2887	Vol % Dist	Vapor Temperature, °F					
		IBP	391	300	427	419	417	437
		5	555	414	540	535	534	543
		10	590	454	581	575	575	590
		15	674	475	618	611	607	633
		20	748	489	654	647	642	671
		30	843	510	715	708	702	716
		40	889	525	767	758	751	754
		50	923	542	814	805	797	787
		60	967	559	856	846	839	817
		70	1011	578	897	887	881	850
		80	1052	600	939	927	923	885
		90	1094	626	986	969	969	920
		95	1121	645	1012	993	996	939
		FBP	1148	693	1037	1014	1022	957
		Residue (%)	25	<3	18	20	28	22

TABLE 24 (Cont)
LIQUID PRODUCT AGING STUDY
PETROLEUM (#6 FUEL OIL-HIGH SULFUR*)/COAL (HEAVY OIL BLENDS**)
G.C. SIMULATED DISTILLATIONS

Test no	ASTM	Description	50% Coal Oil (2124CB3) Aged at 110°F Initial	50% Coal Oil (2124CB3) Aged at 110°F 1 month	75% Coal Oil (2124CB4) Aged at 110°F Initial	75% Coal Oil (2124CB4) Aged at 110°F 1 month	90% Coal Oil (2124CB5) Aged at 110°F Initial	90% Coal Oil (2124CB5) Aged at 110°F 1 month
1	D2887	Vol % Dist			Vapor Temperature, °F			
		IBP	436	470	459	453	494	510
		5	549	595	586	601	666	687
		10	599	673	657	682	704	713
		15	644	709	690	707	723	729
		20	682	730	711	724	738	743
		30	731	767	750	756	768	773
		40	771	796	777	784	792	799
		50	806	823	801	810	813	821
		60	838	850	830	831	835	841
		70	892	880	863	852	860	862
		80	911	916	901	880	889	887
		90	958	968	951	928	931	923
		95	986	1006	985	966	962	958
		FBP	1015	1047	1021	1020	999	1022
		Residue (%)	11	11	12	13	10	<3

* #6 Fuel Oil (High Sulfur) - 2049CB
** Composite Heavy Oil Blend "B" - 2079CB
D-Deleted
INS-Insufficient Sample
NF-Not Filterable

TABLE 25
LIQUID PRODUCT AGING STUDY
PETROLEUM (VACUUM GAS OIL*)/COAL (MIDDLE OIL**) BLENDS

Test no	ASTM	Description	Vacuum gas oil (2051CB) Aged at 110°F Initial	Vacuum gas oil (2051CB) Aged at 110°F 1 month	10% Coal Oil (2119CB1) Aged at 110°F Initial	10% Coal Oil (2119CB1) Aged at 110°F 1 month	25% Coal oil (2119CB2) Aged at 110°F Initial	25% Coal oil (2119CB2) Aged at 110°F 1 month
2	D445	Liquid viscosity (cSt)						
		68°F(20°C)	55.6	TV	TV	TV	36.0	34.1
		104°F(40°C)	--	12.6	11.2	--	--	--
3a	APCI method	Specific gravity pychometer @60°F(15.6°C)	0.8865	NR	0.8982	NR	0.9111	NR
5	D2386	Freeze point, (°F,°C)	-37,-38	NR	-35,-37	NR	-33,-36	NR
10	D130	Copper strip corrosion,rating	1A	NR	1A	NR	1A	NR
14	D1796	Bottoms sediment and water (vol %)	<0.10	<0.10	<0.10	<0.10	<0.10	0.10
15	D97	Pour point, (°F,°C)	85,29	80,27	70,21	80,27	75,24	80,27
30	DuPont F21-61	DuPont stability (Fuel rating D1500 color ex)	Poor/7.5	Poor/8+	V. poor/8+	D	V. poor/8+	D
38	APCI method	Hot filtration sediment (mg/100 ml)	5.2	NR	13.0	NR	12.3	NR

TABLE 25(Cont)
LIQUID PRODUCT AGING STUDY
PETROLEUM (VACUUM GAS OIL*)/COAL (MIDDLE OIL**) BLENDS

Test no	ASTM	Description	50% Coal Oil (2119CB3) Initial	50% Coal Oil Aged at 110°F 1 month	75% Coal Oil (2119CB4) Initial	75% Coal Oil Aged at 110°F 1 month	90% Coal Oil (2119CB5) Initial	90% Coal Oil Aged at 110°F 1 month
2	D445	Liquid viscosity (cSt) 68°F(20°C)	5.6	20.4	10.9	12.4	9.2	11.2
3a	APCI method	Specific gravity pychometer 60°F(15.6°C)	0.9306	NR	0.9599	NR	0.9710	NR
5	D2386	Freeze point, (°F,°C)	-49,-45	NR	-51,-47	NR	-47,-44	NR
10	D130	Copper strip corrosion,rating	1A	NR	1A	NR	1A	NR
14	D1796	Bottoms sediment and water (vol %)	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10
15	D97	Pour point, (°F,°C)	50,10	70,21	40,4	60,16	15,-9	40,4
30	DuPont F21-61	DuPont stability (fuel rating D1500 color ex)	Marginal/8+	V.Poor/8+	Poor/8+	V.Poor 8+	Marginal/8+	V.Poor/8+
38	APCI method	Hot filtration sediment (mg/100 ml)	8.8	NR	4.9	NR	2.5	NR

*Vacuum Gas Oil - 2051CB
**Composite Middle Oil - 2078CB
NR-Not requested
TV-Too viscous

TABLE 26
LIQUID PRODUCT AGING STUDY
PETROLEUM (#2 FUEL OIL*)/COAL (MIDDLE OIL**) BLENDS

Test no	ASTM	Description	#2 Fuel Oil - (APCI 2050CB) Aged at 110°F			10% Coal Oil (APCI 2120CB1) Aged at 110°F			25% Coal Oil (APCI 2120CB2) Aged at 110°F		
			Initial	1 month	3 months	Initial	1 month	3 months	Initial	1 month	3 months
2	D445	Liquid viscosity, (cSt) 68°F(20°C)	4.5	4.6	4.4	4.6	5.0	5.2	5.0	6.9	5.7
3a	APCI method	Specific gravity pycnometer @60°F(15.6°C)	0.8385	NR	NR	0.8503	NR	NR	0.8666	NR	NR
5	D2386	Freeze point (°F,°C)	<-76,<60	NR	NR	<-76,<-60	NR	NR	<-76,-70	NR	NR
7	D1322	Smoke point (mm)	16.0	NR	NR	12.3	NR	NR	12.0	NR	NR
8	D189	Carbon residue (%) (on 10% bottoms)	0.15	0.03	0.01	0.22	NR	0.27	0.51	NR	0.75
10	D130	Copper strip corrosion, rating	1A	NR	NR	1A	NR	NR	1A	NR	NR
14	D1796	Bottoms sediment and water (vol %)	<0.10	<0.10	<0.10	0.10	0.30	0.4	0.16	1.0	0.2
15	D97	Pour point,(°F,°C)	0,-18	0,-18	D	5,-15	0,-18	D	0,-18	0,-18	D
30	DuPont F21-61	DuPont stability (fuel rating-(D1500 color ex)	Excellent/1	Excellent/0.5	Good/0.5	Poor/8	Poor/8	Poor/8+	V.Poor/8+	NR	NR
37	BETC	Suspended sediment (mg/100 ml)	0.0/0.1	0.5/0.3	0.5	4.6/3.8	9.5/10.2	11.1	2.7/2.9	9.6/8.9	8.0
		Adherent gum (mg/100 ml)	0.0/0.0	0.0/0.0	0.0	0.2/0.1	2.7/2.4	3.4	0.5/0.4	4.5/5.0	4.1
		Total (mg/100 ml)	0.1	0.4	0.5	4.4	12.4	14.5	3.3	14.0	12.1
38	APCI method	Hot filtration sediment (ml/100 ml)	2.0	NR	NR	5.0	NR	NR	8.2	NR	NR

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* #2 Fuel Oil - APCI 2050CB
** Composite Middle Oil - APCI 2078CB

TABLE 26 (Cont)
LIQUID PRODUCT AGING STUDY
PETROLEUM (#2 FUEL OIL*)/COAL (MIDDLE OIL**) BLENDS

Test no	ASTM	Description	50% Coal Oil (APCI 2120CB3) Aged at 110°F			75% Coal Oil (APCI 2120CB4) Aged at 110°F			90% Coal Oil (APCI 2120CB5) Aged at 110°F		
			Initial	1 month	3 months	Initial	1 month	3 months	Initial	1 month	3 months
2	D445	Liquid viscosity (cSt) @20°C(68°F)	5.2	4.8	6.4	6.6	8.9	9.5	8.5	7.5	7.6
3	APCI method	Specific gravity pycnometer @60°F(15.6°C)	0.8896	NR	NR	0.9440	NR	NR	0.9664	NR	NR
5	D2386	Freeze point (°F,°C)	<-76,<-60	NR	NR	-69,-56	NR	NR	-58,-50	NR	NR
7	D1322	Smoke point (mm)	11.3	NR	NR	11.0	NR	NR	11.8	NR	NR
8	D189	Carbon residue (%) (on 10% bottoms)	0.77	NR	1.95	NR	NR	3.91	NR	NR	INS
10	D130	Copper strip corrosion, rating	1A	NR	NR	1A	NR	NR	1A	NR	NR
14	D1796	Bottoms sediment and water (vol %)	0.10	<0.10	0.10	<0.10	<0.10	D	<0.10	<0.10	<0.10
15	D97	Pour point, (°F,°C)	0,-18	-5,-21	D	0,-18	-5,-21	D	-5,-21	-5,-21	D
30	DuPont F21-61	DuPont stability (fuel rating-D1500 color ex)	V.Poor/8+	NR	NR	Poor/8+	Poor/8+	Poor/8+	Poor/8+	V.Poor/8+	NR
37	BETC	Suspended sediment (mg/100 ml)	0.8/0.5	5.3/5.0	6.2	NR	NR	NR	NR	NR	NR
		Adherent gum (mg/100 ml)	0.1/0.1	1.0/1.2	1.3						
		Total (mg/100 ml)	0.7	6.3	7.5						
38	APCI method	Hot filtration sediment (ml/100 ml)	5.5	NR	NR	<1.0	NR	NR	1.1	NR	NR

NR-Not requested
D-Deleted
* #2 Fuel Oil - APCI 2050CB
** Composite Middle Oil - APCI 2078CB

TABLE 27
LIQUID PRODUCT AGING STUDY
PETROLEUM (#6 Fuel Oil-High Sulfur*)/Coal (Heavy Oil Blends**)

Test no	ASTM	Description	#6 Fuel Oil (High Sulfur) (2049CB) Aged at 110°F Initial	1 month	10% Coal Oil (2124CB1) Aged at 110°F Initial	1 month	25% Coal Oil(2124CB2) Aged at 110°F Initial	1 month
2	D445	Liquid viscosity,cSt,						
		@175°F(79°C)	43.3	51.2	54.9	49.9	46.1	53.1
		@210°F(99°C)	22.6	24.7	25.0	24.7	21.0	24.4
3	D1298	Liquid specific gravity @60°F	0.9745	NR	0.9902	NR	1.004	NR
		API gravity @60°C	13.7	NR	11.4	NR	9.5	NR
3a	APCI method	Specific gravity pycnometer @60°F(15.6°C)	0.9781	NR	0.9900	NR	1.003	NR
5	D2386	Freeze point (°F,°C)	0,-18	NR	19,-7	NR	32,0	NR
8	D189	Carbon residue (%)	11.9	11.9	11.4	12.2	10.7	10.8
10	D130	Copper strip corrosion,rating	1A	NR	1B	NR	1A	NR
14	D1796	Bottoms sediment and water (vol %)	0.10	<0.10	1.0	0.20	0.30	0.60
15	D97	Pour point (°F,°C)	30,-1	30,-1	40,4	35,2	40,4	40,4
38	APCI method	Hot filtration sediment (mg/100 ml)	24.9	NR	NF	NR	NF	NR

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* #6 Fuel Oil (High Sulfur)-2049CB
** Composite Heavy Oil Blend "B" - 2079CB
NR-Not Requested

TABLE 27 (Cont)
LIQUID PRODUCT AGING STUDY
PETROLEUM (#6 FUEL OIL-HIGH SULFUR*)/COAL (HEAVY OIL BLENDS**)

Test no	ASTM	Description	50% Coal Oil (2124CB3) Aged at 110°F Initial	50% Coal Oil (2124CB3) Aged at 110°F 1 month	75% Coal Oil (2124CB4) Aged at 110°F Initial	75% Coal Oil (2124CB4) Aged at 110°F 1 month	90% Coal Oil (2124CB5) Aged at 110°F Initial	90% Coal Oil (2124CB5) Aged at 110°F 1 month
2	D445	Liquid viscosity (cSt)						
		175°F(79°C)	55.9	56.6	76.4	71.1	87.4	79.4
		210°F(99°C)	23.6	24.2	24.4	26.2	27.1	26.6
3	D1298	Liquid specific gravity @60°F	1.020	NR	--	--	1.062	NR
		API gravity @60°F	7.1	NR	--	--	1.8	NR
3a	APCI method	Specific gravity pyconmeter @60°F(15.6°C)	1.029	NR	1.052	NR	1.070	NR
5	D2386	Freeze point (°F,°C)	50,10	NR	54,12	NR	61,16	NR
8	D189	Carbon residue (%)	8.4	8.4	5.6	5.7	4.4	4.3
10	D130	Copper strip corrosion, rating	1B	NR	INS	NR	1B	NR
14	D1796	Bottoms sediment and water (vol %)	1.0	2.0	1.0	2.8	3.8	3.4
15	D97	Pour point, (°F,°C)	65,18	65,18	INS	70,21	INS	70,21
38	APCI method	Hot filtration sediment (mg/100 ml)	INS	NR	INS	NR	INS	NR

* #6 Fuel Oil (High Sulfur) - 2049CB
** Composite Heavy Oil Blend "B" - 2079CB
D-Deleted
INS-Insufficient Sample
NF-Not Filterable

TABLE 28
LIQUID PRODUCT AGING STUDY
PETROLEUM (#6 Fuel Oil-Low Sulfur*)/Coal (Heavy Oil**) Blends

			#6 Fuel Oil (Low Sulfur) (2048CB) Aged at 110°F				10% Coal Oil (2122CB1) Aged at 110°F				25% Coal Oil (2122CB2) Aged at 110°F		
Test no	ASTM	Description	Initial	1 Month	3 Months	5 Months	Initial	1 Month	3 Months	5 Months	Initial	1 Month	3 Months
2	D445	Liquid viscosity, cSt											
		@175°F(79°C)	79.6	71.7	85.0	79.0	65.4	76.9	84.3	83.1	71.7	71.2	83.3
		@210°F(99°C)	34.5	32.3	D	36.0	36.3	34.3	D	36.6	31.6	29.9	D
3	D1298	Liquid specific gravity @60°F	0.9659	NR	--	NR	0.9606	NR	--	NR	0.9874	NR	NR
		API gravity @60°F	15.0	NR	NR	--	15.8	NR	NR	--	11.8	NR	NR
3a	APCI method	Specific gravity pycnometer @60°F(15.6°C)	0.9831	NR	NR	--	0.9979	NR	NR	--	1.009	NR	NR
5	D2386	Freeze point (°F,°C)	23,-5	NR	NR	--	25,-4	NR	NR	--	30,-1	NR	NR
8	D189	Carbon residue (%)	9.7	8.9	9.3	8.8	9.1	10.4	7.6	8.6	8.2	8.6	6.5
10	D130	Copper strip corrosion, rating	1B	NR	--	NR	1A	NR	--	NR	1B	NR	NR
14	D1796	Bottoms sediment and water (vol %)	0.45	0.30	2.50	0.90	0.10	0.10	0.2	0.50	0.50	0.5	1.0
15	D97	Pour point,(°F,°C)	65,19	85,29	D	--	85,29	70,21	D	--	70,21	75,24	D
38	APCI method	Hot filtration sediment (ml/100 ml)	NF	NR	NR	--	NF	NR	NR	--	NF	NR	NR
--	APCI method	Pentane insolubles,wt%	--	--	--	4.9	--	--	--	4.9	--	--	--

TABLE 28 (Cont.)
LIQUID PRODUCT AGING STUDY
PETROLEUM (#6 Fuel Oil-Low Sulfur*)/Coal (Heavy Oil**) Blends

Test no	ASTM	Description	50% Coal Oil (2122CB3)				75% Coal Oil (2122CB4)			90% Coal Oil (2122CB5)			
				Aged at 110°F				Aged at 110°F			Aged at 110°F		
			Initial	1 Month	3 Months	5 Months	Initial	1 Month	3 Months	Initial	1 Month	3 Months	5 Months
2	D445	Liquid viscosity, cSt											
		@175°F(79°C)	70.5	75.5	82.5	91.8	81.2	77.4	83.8	73.3	88.3	95.8	105.7
		@210°F(99°C)	28.4	30.2	D	33.2	28.8	28.8	D	27.9	29.8	D	32.6
3	D1298	Liquid specific gravity @60°F	1.022	NR	NR	--	1.036	NR	NR	--	NR	NR	--
		API gravity @60°F	7.0	NR	NR	--	5.1	NR	NR	--	NR	NR	--
3a	APCI method	Specific gravity pycnometer @60°F(15.6°C)	1.032	NR	NR	--	1.053	NR	NR	1.077	NR	NR	--
5	D2386	Freeze point (°F,°C)	34,1	NR	NR	--	48,9	NR	NR	54,12	NR	NR	--
8	D189	Carbon residue (%)	6.6	6.8	5.3	6.0	5.1	8.2	4.3	4.0	5.8	2.5	3.2
10	D130	Copper strip corrosion, rating	1B	NR	NR	--	1B	NR	NR	INS	NR	NR	--
14	D1796	Bottoms sediment and water (vol %)	2.2	2.6	3.6	2.8	2.4	3.8	3.4	3.4	4.4	4.0	2.2
15	D97	Pour point, (°F,°C)	75,24	80,27	D	--	85,29	75,24	D	95,35	85,29	D	--
38	APCI method	Hot filtration sediment, (mg/100 ml)	NF	NR	NR	--	NR	NR	NR	NR	NR	NR	--
--	APCI method	Pentane insolubles, wt%	--	--	--	6.1	--	--	--	--	--	--	4.6

* #6 Fuel Oil (Low Sulfur) - APCI2048CB
** Composite Heavy Oil Blend "B" 2079CB
*** See Table 14 for sediment analysis
D-Deleted
NR-Not requested
NF-Not filterable
INS-Insufficient sample

TABLE 29
ANALYSIS OF #6 FUEL OIL-LOW SULFUR/COAL (HEAVY OIL) BLENDS SEDIMENT FROM TEST 14 (TABLE 19)

Test no	ASTM	Description	10% Coal Oil (2122CB1)	25% Coal Oil (2122CB2)	50% Coal Oil (2122CB3)	75% Coal Oil (2122CB4)	90% Coal Oil (2122CB5)
1	D2887	Vol % Dist					
		IBP	519	594	583	610	601
		5	591	647	654	684	689
		10	624	672	689	707	710
		15	648	690	709	722	724
		20	669	706	724	737	738
		30	706	733	753	763	763
		40	743	759	780	787	786
		50	785	786	805	809	807
		60	829	813	830	831	828
		70	879	843	857	857	853
		80	940	880	891	889	882
		90	1008	931	941	937	924
		95	1042	963	980	975	964
		FBP	1073	994	1022	1021	1022
		Residue,%	<3	25	23	28	14
20	APCI method	Elemental Analysis					
		C	86.2	85.2	86.5	87.1	86.1
		H	8.4	9.8	9.3	8.6	8.0
		N	0.7	0.7	0.9	1.0	1.0
		O	2.8	2.3	2.0	2.5	3.0
		S	0.7	0.9	0.7	0.7	0.6

Figure 1

Middle Distillate Storage Stability

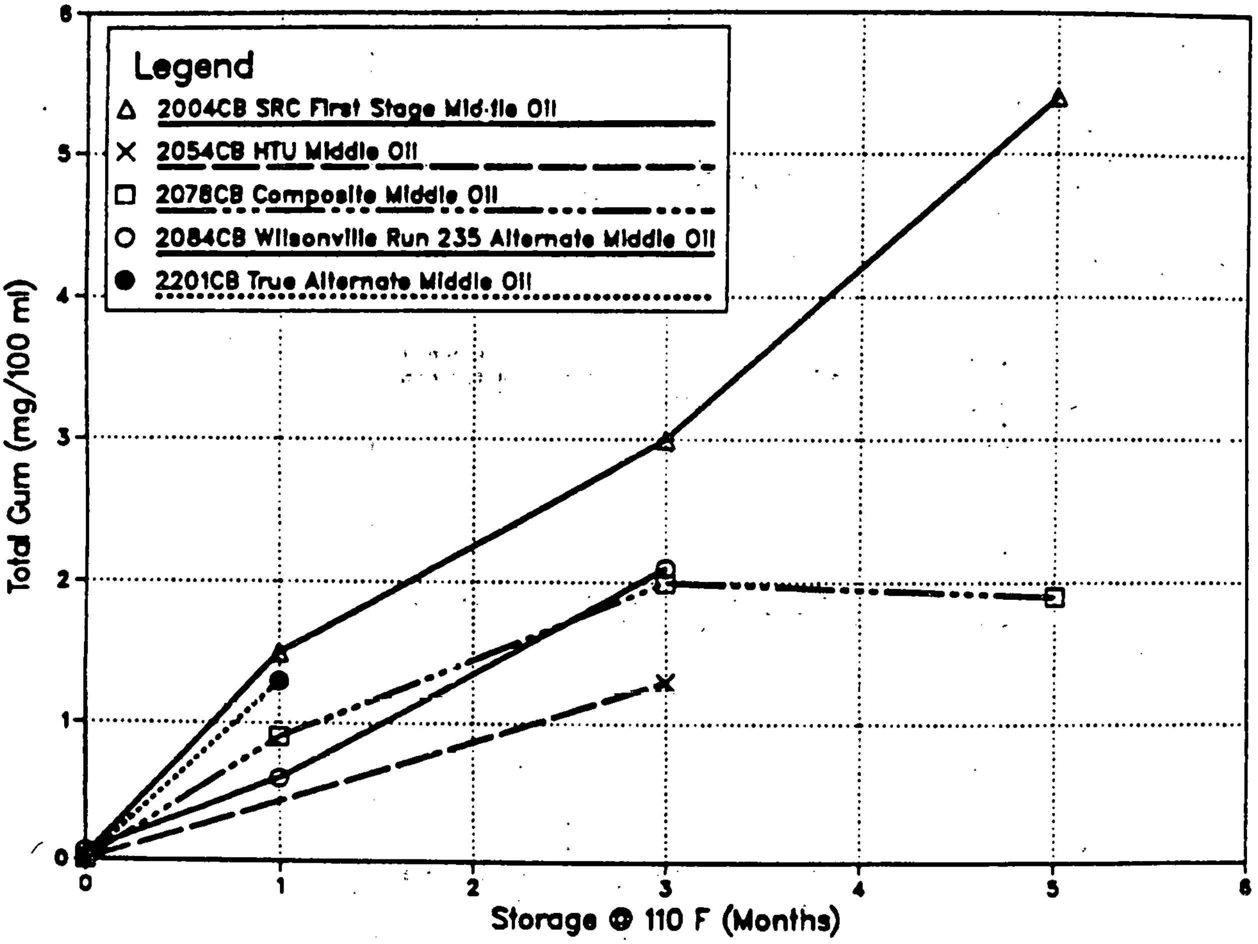
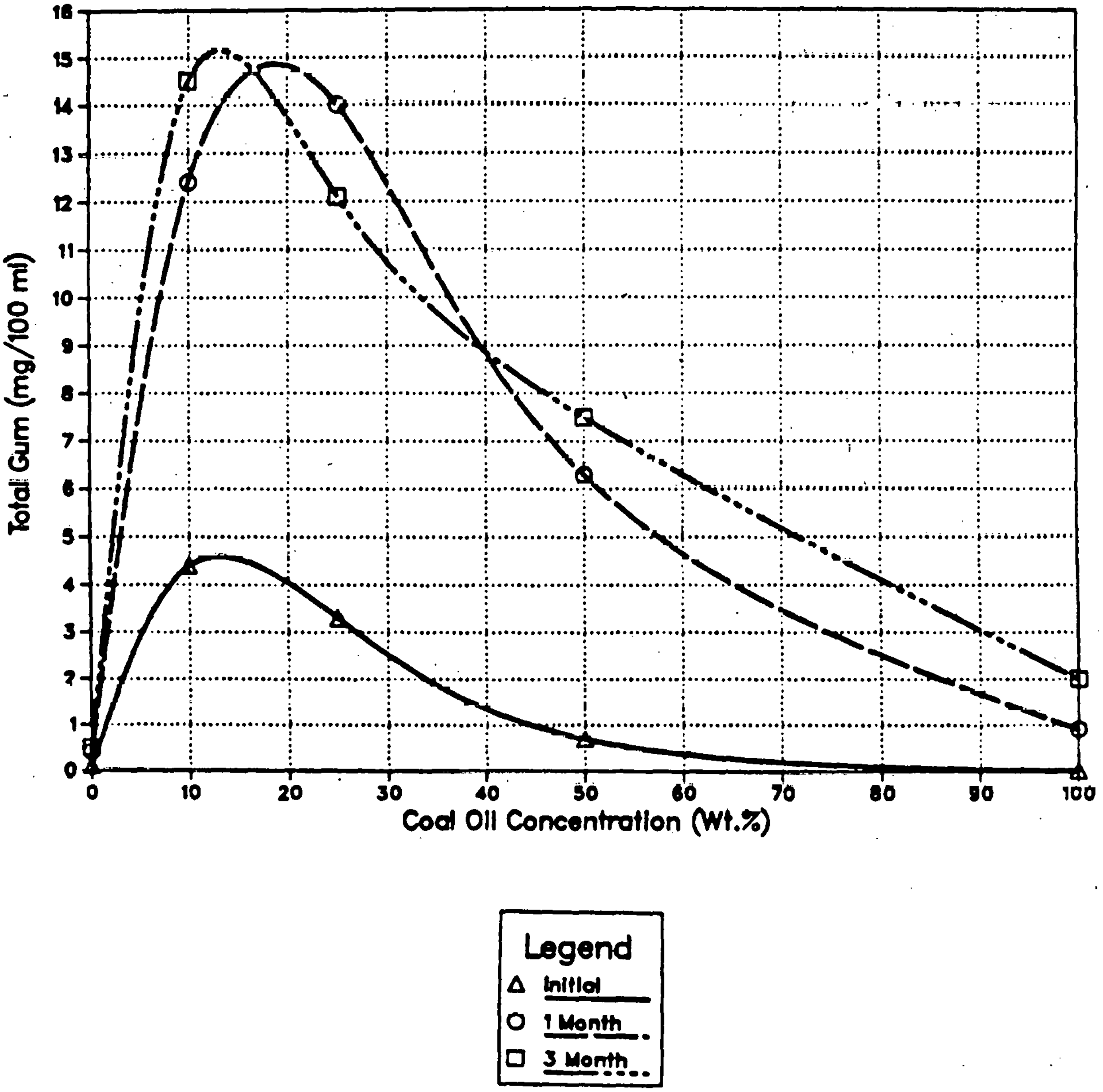


Figure 2

Coal Middle Distillate/Petroleum #2 Fuel Oil Compatibility/Stability Study. Gum Formation vs. Coal Oil Concentration



COAL DISSOLVER DESIGN BASIS

C. F. Harris* and A. D. Fazekas*

This report summarizes the coal dissolver design basis for the SRC-I project.

The severity of the dissolver operating conditions was selected to maximize SRC recovery using the Kerr-McGee critical solvent deashing unit. Those conditions chosen were 2,000 psia, 840°F, and a coal space rate of 38 lb coal/ft^3-hr. Based on these operating conditions, a conservative yield structure was selected using a single dissolver. However, auxiliary equipment included in the process flow sheet retained the flexibility to operate with two dissolvers in series to effect higher product yields. If the gas holdup times are higher than expected, the two-in-series dissolver arrangement can be switched to the two-in-parallel system.

A 2-ft-diam target plate at the dissolver entrance was selected as the optimum configuration for internal gas distribution, since the reaction is not mass transfer controlled. A batch solids withdrawal system was incorporated into the process design, and will be used on an as-needed basis.

In case an increase in dissolver severity would be required to improve product yield or satisfy increased fuel gas requirements, the maximum operating pressure was increased from 2,000 to 2,200 psig.

A monolithic wall was selected over the multiwall construction because of the lack of operating experience with a multiwall design at the SRC-I plant operating conditions.

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DISSOLVER OPERATING CONDITIONS

In the SRC-I process, high-sulfur coal is mixed with a process-derived solvent and heated under a hydrogen atmosphere. As heating occurs, coal, solvent, and hydrogen react to produce a deashed fuel known as solvent-refined coal (SRC). In this process, the coal dissolvers provide the slurry residence time needed to:

- produce an SRC product of less than 1.0% sulfur
- produce sufficient process solvent to maintain "solvent balance"
- maximize the recovery of SRC in a downstream processing unit.

Combustion of SRC with a sulfur content of less than 1% will meet environmental standards without the expensive flue gas desulfurization equipment required for direct combustion of the feed coal. In addition, this low-sulfur content will allow the SRC product to compete with low-sulfur fuel oils in applications in which feed coal could not be utilized at all. Thus, attaining this sulfur content specification is critical to the success of the process.

The process must also remain in "solvent balance." Processing of the feed coal requires slurrying with a process-derived solvent. If the reaction of coal does not produce enough solvent to make up for processing losses, it is not clear that sufficient quantities of solvent could be supplied from other sources. Even if an independent source of process solvent would be available, the costs would be prohibitive.

After the coal has reacted in the dissolvers, the SRC must be separated from the ash and unreacted coal. ICRC has selected the Kerr-McGee critical solvent deashing (CSD) system to perform this separation. O'Leary (1980, 1981) has documented the performance of the CSD unit at the Wilsonville Advanced Coal Liquefaction Facility and correlated the recovery of SRC to its asphaltene-to-preasphaltene ratio. While the CSD unit recovers almost all of the asphaltene found in the SRC, recovery of the preasphaltene fraction of SRC is limited to 65 to 70%. In order to ensure adequate recovery of the SRC product, the dissolver operating conditions must be selected to minimize the yield of preasphaltenes from the process.

Martin and Weber evaluated the performance of the Fort Lewis and Wilsonville pilot plants and developed a reaction severity model of the form:

$$\text{severity} = \left[P^{0.488} \times \frac{1{,}715}{1{,}715^{0.488}}\right] \times \text{constant} \times \frac{1}{\text{space rate}} \times \exp\left[\frac{-30{,}000}{T}\right]$$

where P = dissolver pressure in psia

T = dissolver temperature in °R

space rate = pound of coal per hour per cubic foot of reactor

With regression analysis, the sulfur content of the SRC, process solvent yield, and the ratio of asphaltenes to preasphaltenes, as well as the remainder of the yield structure, were correlated to reaction severity by an equation of the form:

$$\text{variable} = A + B\,(\text{severity})^{N}$$

where the value of N was 0.5, 1.0, or 2.0, and was selected to provide the best fit to the actual plant performance. Martin and Weber established that a minimum dissolver reaction severity of 0.5 was required to accomplish the production objectives of the SRC-I Demonstration Plant. The following normal operating conditions were then selected for the demonstration plant dissolver in order to obtain a reaction severity of 0.5:

Dissolver operating temperature	840°F
Dissolver normal operating pressure	2,000 psia
Dissolver coal space rate	38 lb coal/$\text{ft}^3\cdot$hr

A series of design confirmation runs was then conducted at the Wilsonville Pilot Plant (SCS, 1978 a,b,c,d) which demonstrated the reproducibility of sulfur removal, process solvent balance, and SRC recoverability at the above conditions. The data from these confirmation runs are presented in Table 1.

SELECTION OF TWO DISSOLVERS OPERATED IN SERIES

In addition to evaluating pilot plant data, ICRC studied coal liquefaction kinetics in a 1-liter continuous stirred tank reactor referred to as the Coal Process Development Unit (CPDU). The most important aspect of this work was the demonstration by Skinner (1979) that the coal liquefaction reaction yields could be predicted by assuming first-order kinetics. Skinner developed a sequential model (Figure 1) that suggested that series operation of multiple reactors would reduce the sulfur content of the SRC, as well as enhance SRC recovery by reducing preasphaltene yield compared with a single reactor. Table 2 presents a comparison of model predictions for single and multiple dissolvers. Harris (1982) demonstrated that the model predictions were consistent with Fort Lewis and Wilsonville pilot plant performance for single-reactor yields. Sivasubramanian (1983) demonstrated that the model accurately predicted the yield structure for series operation on smaller lab-scale equipment. Based on these data, operation of two dissolvers in series would result in a 3% MAF coal greater product recovery than a single dissolver at design reactor conditions. Operation of three dissolvers in series offered little benefit over two dissolvers operated in series. ICRC determined the installed capital cost of a single dissolver to be $5,396,000 (Table 3) and of two dissolvers to be $6,130,000 (Table 4). Based on the improved product recovery at minimal increased capital cost, ICRC elected to operate two dissolvers in series in the SRC-I Demonstration Plant.

However, it was not possible to test this configuration at Fort Lewis and Wilsonville. Because confirmation of the series yield structure could not be obtained from the pilot plants, ICRC elected to retain the more conservative single-dissolver yield structure produced by the pilot plants when designing the demonstration plant. However, sufficient flexibility was included in the design to accomodate the two-in-series yield structure predicted by the lab-scale data.

HEAT OF REACTION

Because the pilot plants could not be operated in the series mode, it was necessary to develop a model for the heat release in each demonstration plant dissolver in order to ensure safe control of dissolver temperatures. Harris (1981) and Phillips (1983) established the heat of reaction for the coal liquefaction reactions as 11,000 Btu/lb of hydrogen consumed based on the Fort Lewis and Wilsonville pilot plant performance. In conjunction with the Skinner model predictions of hydrogen consumption, this heat of reaction could be used to predict dissolver heat release under series operation. In order to confirm model predictions, a short residence time run was performed at the Wilsonville Pilot Plant which simulated the first dissolver in the demonstration plant. As indicated in Table 5, the Wilsonville run confirmed the model predictions of both hydrogen consumption and heat release within the accuracy of the Wilsonville data (Ying et al., 1980). (The Wilsonville short residence time run was performed at a dissolver temperature of 825°F rather than the demonstration plant design of 840°F due to mechanical difficulties at the Wilsonville Pilot Plant.)

The heat release predicted by the model for the demonstration plant dissolver is presented in Table 6. When operated in series, the second dissolver will have a heat release of 36.7 MM Btu/hr. ICRC has elected to quench this heat release with the recycle hydrogen gas that is required by the process to maintain hydrogen partial pressure. Up to 60 MM Btu/hr of quench is available from this stream, which provides an acceptable design safety margin.

DISSOLVER DIMENSIONS

Because the normal dissolver operating conditions were 840°F and 2,000 psig, the minimum acceptable mechanical design specifications for the dissolver vessel were established as 900°F and 2,200 psig. Based on a review of available pressure vessel operating data and construction capabilities, an 11-ft inside diameter vessel was selected for the demonstration plant dissolvers because it represented the limit of existing operating experience at dissolver design conditions. In addition, it corresponded to the maximum vessel diameter that could be shop-fabricated and shipped, thus avoiding the cost and potential schedule penalties associated with field fabrication of the dissolvers.

The determination of required vessel volume represents perhaps the greatest uncertainty in the design of the dissolvers. The selected coal space rate of 38 lb of coal/ft^3-hr was based on data available from the Wilsonville and Fort Lewis pilot plants. Both plants were operated at relatively low superficial gas velocities in the coal dissolvers. On scale-up to demonstration plant conditions, the superficial gas velocity increases by a factor of two to three and increased gas holdup reduces slurry residence time for the same reactor space rate. In order to account for the impact of gas superficial velocity on the gas holdup and available reactor volume, ICRC utilized gas holdup correlations based on air/water data. These correlations preducted a gas holdup of 17-20% at demonstration plant conditions, versus 5-10% gas holdup at pilot plant conditions. After the coal space rate was corrected for the differences in gas holdup, ICRC determined that a 67-ft dissolver height was required to provide adequate slurry residence time in the dissolvers.

However, tracer studies at the Exxon Coal Liquefaction Pilot Plant (ECLP) (Exxon, 1980, 1981b; ICRC, 1983) have indicated substantially higher gas holdup than predicted by air/water data. Figure 2 presents a comparison of the gas holdup correlation used in the design of the demonstration plant with those measured at ECLP under actual coal liquefaction conditions. As shown by Figure 2, the demonstration plant design gas holdups of 17 and 20% for the first and second dissolvers in series may be in error by a factor of 2 to 3. Table 7 compares parallel and series operation of the dissolvers based on the ECLP gas holdup data. If the ECLP data are accurate, parallel operation of the demonstration plant dissolvers would result in a higher product recovery than series operation due to the reduced residence time of series operation.

However, because of the uncertainty in gas holdup data, ICRC has elected to operate the dissolvers in series with the flexibility to switch to parallel operation should the ECLP gas holdup data be proven correct at the demonstration plant scale.

DISSOLVER INTERNALS

Normally, in two-phase (gas/liquid) reactor systems, a distributor is placed at the reactor inlet to ensure even flow distribution and enhance mass transfer between the two phases. The Wilsonville Pilot Plant employed a distributor plate in its 1-ft-diam coal dissolver for this purpose. The performance of the Wilsonville reactor demonstrated that its coal liquefaction reactions were kinetically controlled and that mass transfer rates were not affecting coal liquefaction rates. However, a distributor was not employed in the 2-ft-diam coal dissolver of the Fort Lewis Pilot Plant, and the coal liquefaction reactions for this reactor were also shown to be kinetically controlled without mass transfer effects. In order to establish the internals in the demonstration plant dissolver that would ensure adequate mass transfer, ICRC conducted several cold-flow experiments to measure the impact of various distributor systems on mass transfer in an air/water system for 1- and 6-ft-diam vessels (ICRC, 1983).

Figure 3 presents the results of this work for the 1-ft-diam column (with and without solids present to simulate the coal particles found in the coal dissolver). As demonstrated by the data, the absence of the distributor plate did not significantly reduce the mass transfer rate for the system. These results are consistent with the coal liquefaction pilot plant data, which show that neither the Wilsonville nor the Fort Lewis coal dissolver reactions are controlled by mass transfer, in spite of the fact that the Wilsonville dissolver has a distributor plate, while the Fort Lewis dissolver does not. In order to establish that the impact of internals is consistent with scale-up, ICRC also conducted cold-flow experiments with a 6-ft-diam column (Figure 4). As demonstrated by the data, the addition of target plates and bubble cap distributors showed a moderate improvement in the mass transfer coefficient for the system, while the use of gas spargers demonstrated the highest possible mass transfer coefficients for the system, roughly 150% that of no internals.

ICRC concluded that the use of a 2-ft target plate as the only internal in the dissolver represented the optimum configuration for the 11-ft-diam demonstration plant dissolvers. In the cold-flow studies, the use of a distributor did not affect mass transfer coefficients for the air/water system in the 1-ft vessel. This conclusion confirmed observed coal liquefaction pilot plant performance. The mass transfer coefficients for the 6-ft vessel were equivalent to or slightly greater than those for the 1-ft-diam vessel in the cold-flow study, demonstrating that scale-up would not effect mass transfer coefficients. Finally, the only internal that would provide greater mass transfer coefficients than the target plate would be gas spargers, which would greatly increase reactor complexity and potential for downtime.

SOLIDS WITHDRAWAL SYSTEM

The feed to the coal dissolver is a three-phase mixture of hydrogen gas, recycle process solvent, and solid coal particles. In the Wilsonville Pilot Plant, the solid coal and ash particles accumulated in the dissolver to the point of occupying as much as 50% of the reactor volume. This solids accumulation resulted from the relatively low superficial slurry and gas velocities, 0.008 and 0.048 ft/sec, respectively. In the Fort Lewis Pilot Plant, superficial slurry and gas velocities were substantially higher, 0.015 and 0.090, respectively, and solids accumulation was greatly reduced. In scale-up to demonstration plant conditions, the superficial slurry and gas velocities are further increased to as much as 0.055 and 0.28 ft/sec, respectively, and solids accumulation is expected to be minimal. The design of the demonstration plant dissolvers addresses this variation in solids accumulation in two ways.

The accumulated solids are believed to have a catalytic effect on coal liquefaction reactions. In order to ensure that the predicted demonstration plant dissolver performance was accurate, ICRC relied on the low solids accumulation data from the Fort Lewis Pilot Plant and selected Wilsonville data that were obtained with low solids accumulation in developing demonstration plant performance correlations. The low solids accumulation data from Wilsonville were acquired while a continuous solids withdrawal system was in operation, maintaining a 6% solids concentration in the dissolver slurry. This solids level is consistent with the solids content found in the Fort Lewis dissolver at end-of-run conditions, and is also consistent with the solids accumulation predicted for the demonstration plant dissolver based on cold-flow studies (ICRC, 1983).

As noted above, the Wilsonville Pilot Plant required a continuous solids withdrawal system to avoid filling the reactor with solids. The Fort Lewis Pilot Plant, with its higher superficial velocities, required solids removal only at the end of runs. However, both of these reactors operated at lower superficial velocities than the demonstration plant design conditions. In order to determine the need for a solids withdrawal system for the demonstration plant dissolver, ICRC evaluated the performance of the Exxon Coal Liquefaction Pilot Plant (ECLP), which operated on similar coal feedstocks. From August through October 1980, the ECLP reactors were operated at superficial velocities almost equivalent to those of the demonstration plant design. After operating for 52 days on -10 to -20 mesh coal and processing 7,000 tons, the unit was shut down and the accumulated solids removed. Table 8 provides the particle size distribution for the solids that had accumulated in the reactor. The sample taken 8 ft above the distributor plate was believed to be most representative. This sample indicated that less than 1.5 wt % of the accumulated solids was smaller than 100 mesh, while only 0.5 wt % was smaller than 200 mesh. The coal feed particle size for the demonstration plant is 70% less than 200 mesh. After considering the Fort Lewis and ECLP experience, ICRC elected not to include a specific solids withdrawal system for the demonstration plant dissolvers. Instead, solids will be removed through existing dissolver blowdown lines on a batch, as-needed basis. However, the dissolver design does include nozzles for the addition of a solids withdrawal system, since the use of 20-mesh feed coal may require a specific system.

DISSOLVER MAXIMUM OPERATING PRESSURE

The dissolver operating conditions required to attain the necessary reaction severity resulted in a minimum acceptable dissolver mechanical design of 900°F and 2,200 psig (Case A). ICRC also considered an alternate design (Case B) that increased the maximum mechanical design pressure to 2,400 psig, which would allow improved product yields by increasing reaction severity. In addition, the higher design pressure allowed the option to install rupture discs under the dissolver safety valves if required by the Process Hazards Review. The rupture discs may prevent solids buildup in the safety valve parts, due to leakage.

The incremental cost for Case B is $1.3 MM (1st-quarter FY 79) over Case A. These costs are limited to the Dissolver and Hydrogen Recovery Sections and the Hydrogen Recycle Compressor; although there will be a cost impact on the Gas Treatment Area and the Hydrogen Purification Unit, these costs are not included. A summary of the costs is provided in Table 9.

ICRC decided to design the coal dissolvers for 2,200 psig maximum operating pressure and 2,420 psig design pressure based on the ability to increase the yield by increasing this operating pressure. This higher design pressure will allow for increasing the severity of coal dissolver operation. However, the higher pressure will also produce more gas. If the fuel requirements exceed production, increasing the dissolver severity by raising the pressure will re-establish the fuel supply and demand balance, whereas increasing the temperature will require fuel gas. A higher temperature could drive the supply and demand further out of balance.

MONOLITHIC WALL

The physical and design basis of the coal dissolvers is as follows:

Internal diameter:	11 ft*
Shell wall thickness:	10.26-in. base wall*
Material:	SA - 387 GR22, CL2 with TP 347 weld overlay
Design code:	Section VIII, Division 2
Design pressure:	2,420 psig
Design temperature:	900°F

The 10-in.-thick wall of the dissolver required the consideration of multiwall construction because of the two distinct advantages of this type of construction: (1) approximately 20% savings in purchase price and (2) the use of readily available thinner wall plates. However, after considering these advantages, ICRC selected monolithic wall construction as the design basis for the coal dissolvers because of the following reasons:

(1) ICRC found no operating information on multilayer vessels under similar operating conditions of 900°F and 2,400 psig.

(2) The analyses for stress, temperature, and strain are very complicated in multilayered construction because additional assumptions have to be made concerning the transfer of these three parameters across each wall interface. In monowall construction, the stress and temperature patterns through the wall are continuous, since there is a continuous wall.

(3) Because of the current state-of-the-art of acoustic emissions monitoring, ICRC believes that interpretation of results is not possible. Therefore, in-service inspections or testing of the vessel would not be possible.

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*Neither of these values includes corrosion allowance, which is 3/16 in.

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Table 1

Design Configuration Runs with

Wilsonville Pilot Plant Data - Kentucky #9 Coal

Material-balance period	Temp (°F)	Residence time (min)	Total pressure (psig)	Hydrogen consumption (% MAF coal)	Yield structure (% MAF coal)							
					SRC	Distillate	Hydrocarbon gas	Heteroatom gas	Unconverted coal	Water	Preasphaltene[a]	% S in SRC
219 AB	840	37	2,100	2.5	59	28	4.1	3.7	6.4	2.5	21	0.83
220 AB	840	35	2,100	2.5	54	33	4.5	2.8	5.6	4.3	22	0.86
220 DE	840	35	2,100	2.3	54	30	4.6	3.5	6.4	5.0	21	0.90
225 BC	840	35	2,100	2.1	55	30	4.2	3.0	6.3	3.9	25	0.97
225 F	840	35	2,100	2.4	56	29	4.5	3.6	7.3	3.3	26	0.86
225 G	840	35	2,100	2.6	57	29	4.7	3.6	6.9	3.0	28	0.88
225 I	840	35	2,100	2.5	59	25	5.5	3.4	7.3	3.5	30	0.85
Demonstration plant design	840	36	2,100	2.5	56	25	5.0	3.9	6.7	5.4	26	1.0

[a] Based on Kerr-McGee analytical technique.

Table 2

Skinner Coal Liquefaction Model Predictions for 840°F Dissolvers

	One dissolver	Two dissolvers in series	Three dissolvers in series
Residence time (min)	36	18/19	12/12/12
Yield structure (% MAF coal)			
Unconverted coal	7	6	6
Preasphaltene	26	20	16
Asphaltene	25	29	30
Distillate	24	27	29
C_1 - C_5 gas	8	8	8
Heteratom products	12	13	14
H_2 consumption	2.2	2.4	2.6
% sulfur in SRC	0.84	0.76	0.74
Product recovery (% MAF coal)			
65% preasphaltene	17	13	10
95% asphaltene	24	28	29
98% distillate	24	27	29
Total	65	68	68

Table 3

Case I - One Coal Dissolver Installed Estimate[a]

Description	Material	Labor	Subcontract
R-1201 Coal Dissolver			
11 ft 0 in i.d. x 110 ft T/T (900°F & 2,200 psig design; 2 1/4% CR - 1% M with 347 SS overlay)	3,500,000		
Shipment	50,000		
Field erection (allow for poles, cranes, truck, etc.)	100,000		
Erect/dismantle poles (3,000 mhs[b])			
Install conc. deadman (600 mhs)			
Handle and install tower (1,000 mhs)		70,000	
Concrete			
Dissolver (160 yd^3)	10,000	23,000	
Deadman (80 yd^3)	3,000	4,000	
Stairstructure (10 yd^3)	600	3,000	
Structural steel (30 tons)			50,000
Insulation/painting			100,000
Piping			
Estimated material cost (inc. shop fab.)	1,000,000	50,000	
Instruments	3,000	1,000	
Electrical (lighting)	2,000	3,000	
	4,668,600	154,000	150,000
Markup to installed cost	50,000	154,000	10,000
Contingency (3% mat/15% labor/15% subcontract)	139,400	46,000	24,000
	$4,858,000	$354,000	$ 184,000
			354,000
			4,858,000
Total installed cost			$5,396,000

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[a] In first-quarter FY 1979 dollars.

[b] MHS - Manhours

Table 4

Case II - Two Coal Dissolvers Installed Estimate[a]

Description	Material	Labor	Subcontract
R-1201 A/B coal dissolver			
11 ft i.d. x 57 ft 6 in. T/T (900°F & 2,200 psig design; 2 1/4% CR - 1% M with 347 SS overlay)	4,216,000		
Shipment single barge	50,000		
Field erection (allow for poles, cranes, truck etc; assume same as Case I except 50% additional time)	150,000		
Erect/dismantle/relocate poles (4,500 mhs[b])			
Install conc. deadman (600 mhs)		106,500	
Handle and install tower (2,000 mhs)			
Concrete (230 yd^3)	12,000	28,000	
Structural steel (24 tons)			40,000
Insulation/paint			120,000
Piping	800,000	75,000	
Instruments	6,000	2,000	
Electrical (lighting)	1,200	2,000	
	5,235,200	213,500	160,000
Markup to installed cost	51,800	213,500	10,000
Contingency 3% mat/15% labor/15% subcontract	156,000	64,000	26,000
	5,443,000	491,000	196,000
			491,000
			5,443,000
Total installed cost			6,130,000

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[a] In first-quarter FY 1979 dollars.

[b] MHS-manhours.

Table 5

Wilsonville Short Residence Run Results

	Short residence time	Long residence time
Temperature (°F)	825	825
Residence time (min)	21	39
Hydrogen comsumption (% MAF coal)	1.4 ± 0.2	2.1 ± 0.2
Reactor heat release (M Btu/hr)	113	76
<u>Skinner model predictions</u>		
Hydrogen consumption (% MAF coal)	1.6	2.2
Reactor heat release (M Btu/hr)	110	77

Table 6

Demonstration Plant Dissolver Heat Release Predictions at 840°F

	One dissolver	Two dissolvers		
		No. 1	No. 2	Total
Residence time (min)	36	18	18	36
Hydrogen consumption (% MAF coal)	2.2	1.6	0.8	2.4
Heat release (MM Btu/hr)	100.1	73.4	36.7	110.1
Adiabatic temperature change (°F)	110	80	40	120

Table 7

Impact of Exxon Gas Holdup Data on Series/Parallel Dissolvers Yields at 840°F

	One dissolver	Two dissolvers in series
Superficial gas velocity (ft/sec)	0.14	0.21/0.28
Gas holdup (%)	34	49/68
Residence time (min)	30	11/7
Yield structure (% MAF coal)		
Unconverted Coal	8	7
Preasphaltene	28	36
Asphaltene	27	30
Distillate	21	12
C_1 - C_5 gas	7	5
Heteratom products	11	11
H_2 consumption (% MAF coal)	2.0	1.9
% Sulfur in SRC	0.94	1.12
Product recovery (% MAF coal)		
65% preasphaltenes	10	23
95% asphaltenes	26	29
98% distillates	21	12
Total	65	64

Table 8

ECLP Liquefaction Reactor Solids
October 1980 Monterey Mine Coal Operation[a]

	Solids from 8 ft above R101A distributor			Solids from R101A dump pile			Solids from R101B dump pile[b]	
Sieve fraction	Fraction (wt %)	Ash (wt %; SO_3-free)	Atomic hydrogen/ carbon ratio	Fraction (wt %)	Ash (wt %; (SO_3-free)	Atomic hydrogen/ carbon ratio	Ash (wt %; SO_3-free)	Atomic hydrogen/ carbon ratio
+8	3	26	0.38	2	76	0.46	--	--
-8+16	39	35	0.35	25	58	0.43	40	0.63
-16+30	49	66	0.39	64	77	0.64	79	0.75
-30+50	5	67	0.58	6	79	0.72	74	0.85
-50+100	3	60	0.60	2	58	0.74	53	0.83
-100+200	1	57	0.62	1	58	0.69	--	--
-200+325	0.4	62	0.69	--	--	--	--	--
-325	0.1	65	0.76	--	--	--	--	--

[a] From October - December 1982 quarterly technical report (DOE/ET/10069-T7).

[b] No sieve analysis.

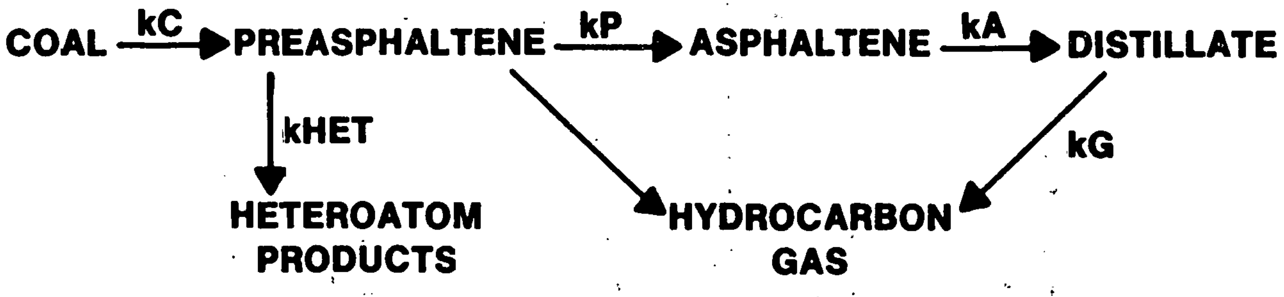
Table 9

Capital Costs of Increasing the Coal Dissolver Maximum Operating Pressure[a]

	Case A ($ MM)	Case B ($ MM)
Dissolver and hydrogen recovery area		
Installed cost	11.6	12.6
Home office allowance	1.8	1.8
Subtotal	13.4	14.4
Hydrogen compression	8.6	8.9
Total[b]	22.0	23.3

[a] First-quarter FY 1979 dollars.
[b] Excludes contingency, escalation, and additional ICRC costs.

Figure 1
Series Model Based on CPDU Data



RATE CONSTANTS	FIRST ORDER WITH RESPECT TO:	CORRELATION COEFFICIENT
$kC = 7.01 \times 10^{7} e^{(-22510/T^\circ R)}$	UNREACTED COAL	0.70
$kP = 1.05 \times 10^{6} e^{(-21450/T^\circ R)}$	PREASPHALTENE	0.89
$kHET = 2.08 \times 10^{5} e^{(-21550/T^\circ R)}$	PREASPHALTENE	0.73
$kH_2 = 7.90 \times 10^{3} e^{(-19550/T^\circ R)}$	PREASPHALTENE	0.94
$kA = 1.44 \times 10^{6} e^{(-23630/T^\circ R)}$	ASPHALTENE	0.54
$kG = 7.63 \times 10^{8} e^{(-28460/T^\circ R)}$	ZERO ORDER	0.81

Figure 2
Comparison of ECLP Gas Holdup Data With Demonstration Plant Design Basis

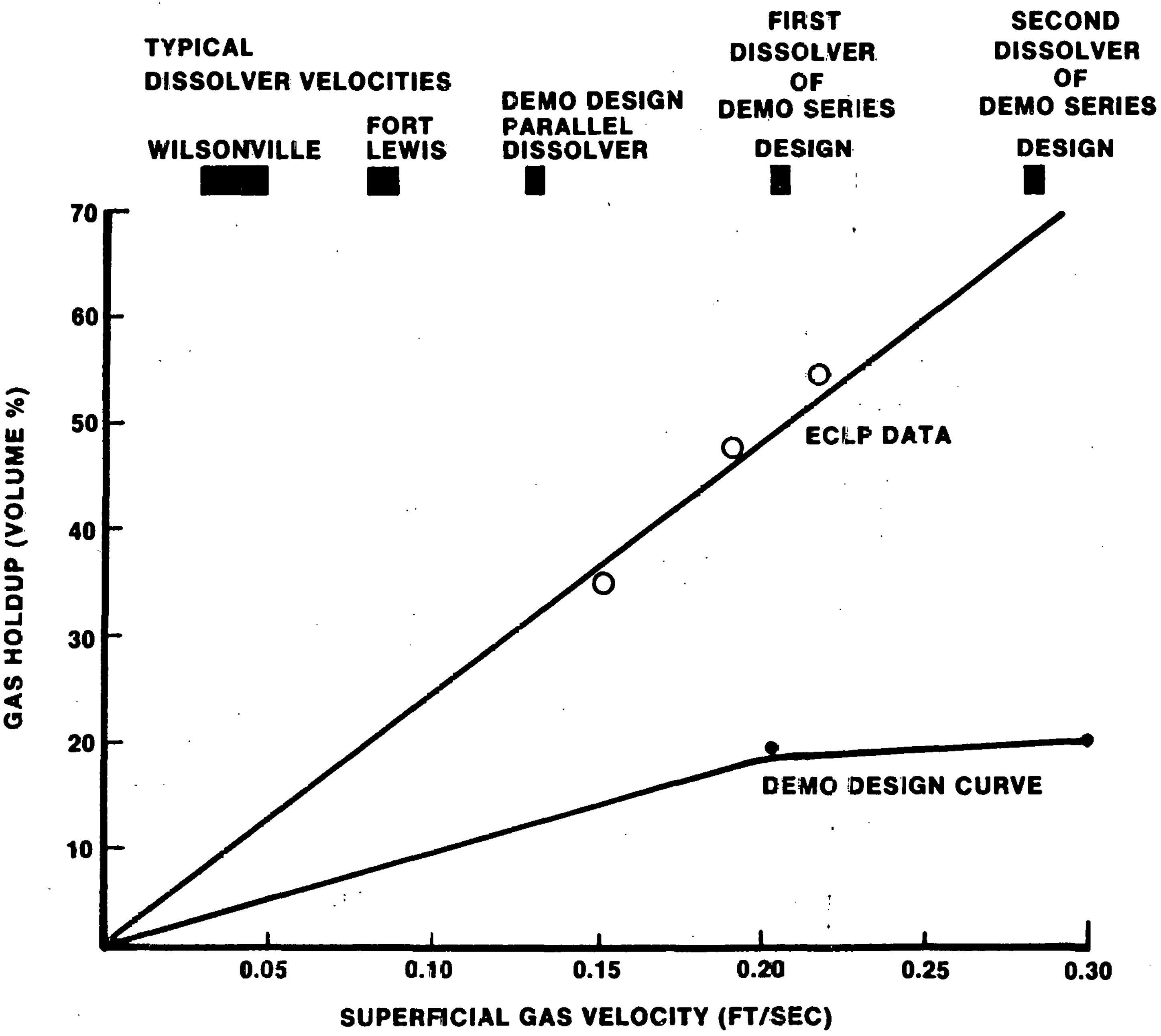


Figure 3
Effect of Distributors on Gas/Liquid Mass Transfer
in a 12-in. Column

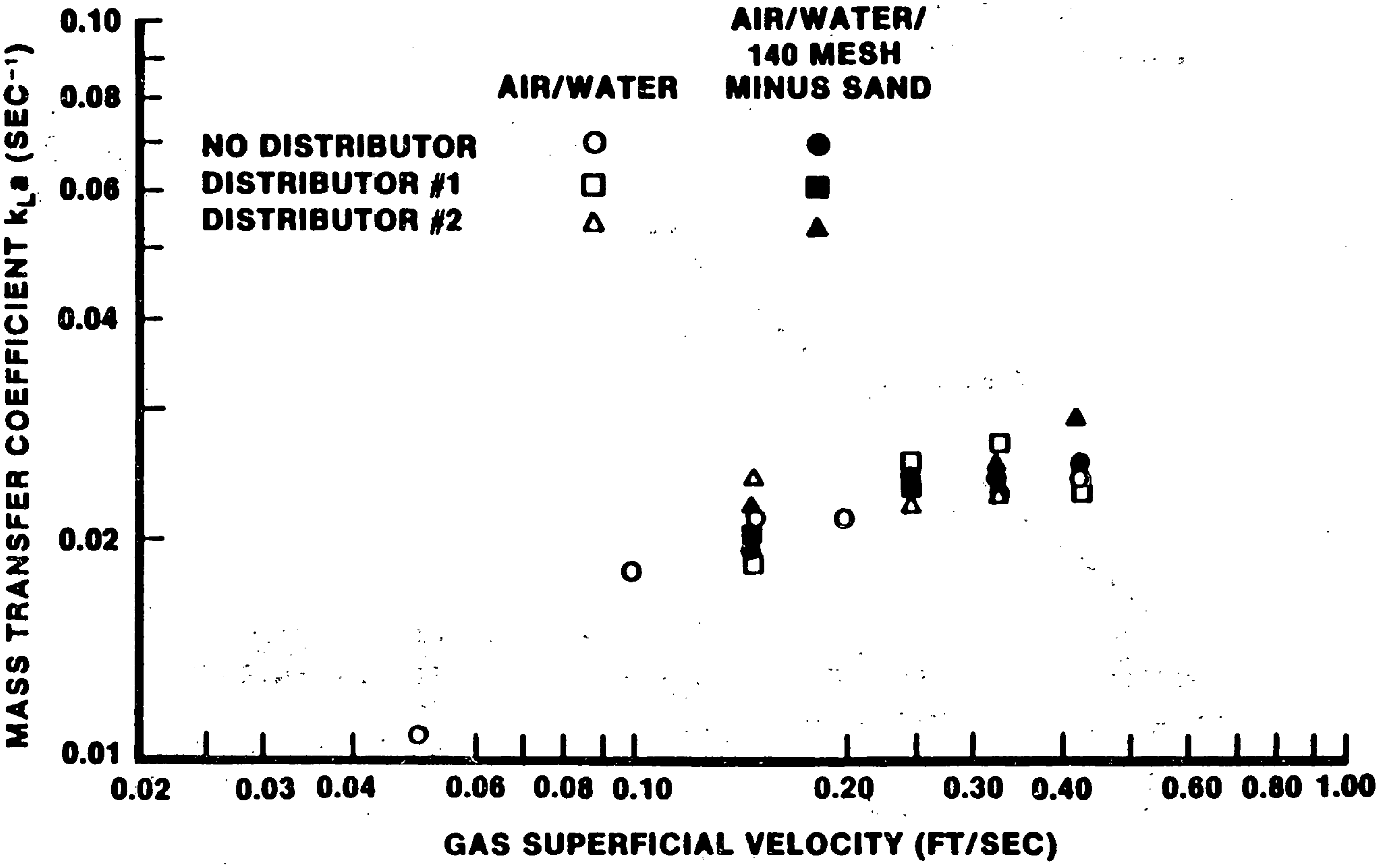
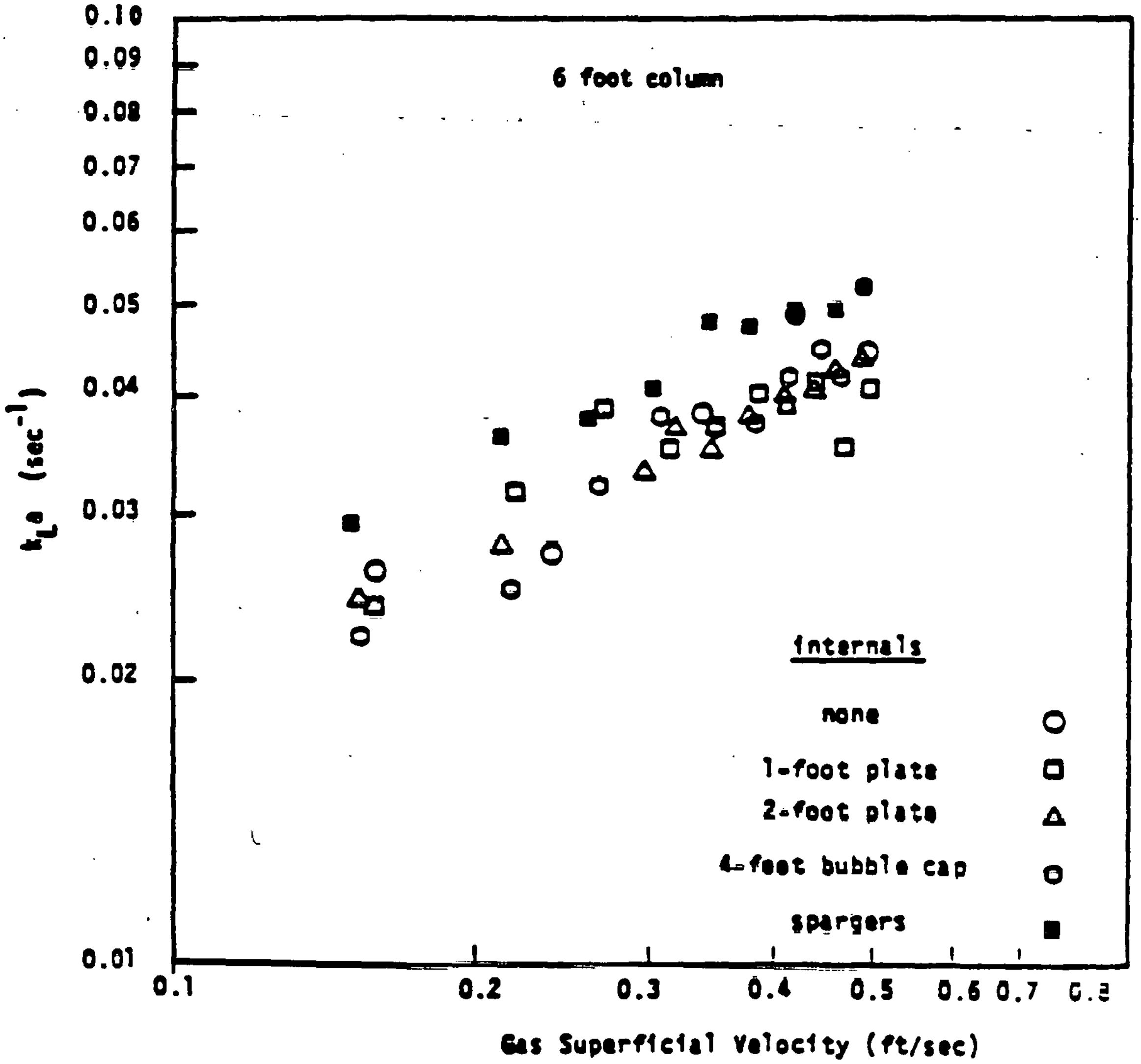


Figure 4
Effect of Different Internals on Gas/Liquid Volumetric Mass-Transfer Coefficient in a 6-ft Column



COAL SLURRY PREPARATION DESIGN USING 20- OR 200-MESH COAL

Catalytic, Inc.

Catalytic, Inc. conducted a study to determine the most flexible and efficient slurry piping system for the SRC-I Demonstration Plant that is capable of processing both 200- and 20-mesh coal, as well as allowing train capacity turndown to 50%. Critical velocities were calculated for d_{50}, d_{70}, and d_{max} particle sizes, and then compared with actual velocities from operating pipelines. It was determined that velocities based on d_{50} particle size were more in line with industrial experience. However, because operating pipelines are run on water slurry and the demonstration plant will be operated on oil slurry, it was deemed prudent to design the plant's slurry system at a critical velocity based on d_{70} particle size. On this basis, Catalytic designed a system for 20-mesh coal, which was determined to be adaptable to 200-mesh coal, and which allowed a 50% turndown in capacity.

INTRODUCTION

In order to design the slurry piping system for the SRC-I Demonstration Plant, Catalytic, Inc. studied the dimensions of several existing coal slurry pipelines, e.g., those of Black Mesa Pipe, Inc. and Consolidated Coal Co. This study discusses the available data and uses these data to predict critical velocity in the demonstration plant.

Critical Velocity

Critical velocity is the velocity in the slurry pipe below which solid particles begin to settle to the bottom of the pipe. The method previously used to calculate the critical velocity was based on Zandi's Correlation presented by Turian and Yuan (1977):

$$V_c^2 = 40 \frac{CDg\ (S-1)}{(C_d)^{1/2}}$$

where V_c = critical velocity (ft/sec)
C = solid concentration by volume
D = inside diameter of pipe (ft)
g = gravitational acceleration (ft/sec^2)
S = relative density ($\rho s/\rho l$)
ρ_s = density of solid (lb/ft^3)
ρ_l = density of liquid (lb/ft^3)
C_d = drag coefficient = $\frac{4}{3} \frac{gd\ (s-1)}{2}$
d = diameter of solid particles (ft)
v = terminal velocity of sphere, settling in an unbounded fluid (ft/sec)

The above critical velocity correlation was derived from a narrow size distribution of particles. In such a case, it is generally felt that only the coarser particles have an effect on critical velocity since those particles will settle out first. However, it is recognized that if the suspension contains enough fine particles, the mixture shows significant non-Newtonian properties, which reduces the settling rate of the coarser particles in the suspension.

In the mining industry, the "tailings" are disposed of by pumping them as water slurries. In the design of such systems (McElvain, 1974), Durand's Correlation is used:

$$V_c = F \quad [2gD \quad (S-1)]^{1/2}$$

Where F_L is a function of particle diameter and solids concentration by volume. In this case, the d_{50} particle size is used and denotes a size such that 50 wt % of the solids are coarser and 50 wt % are finer.

When the d_{50} particle size is used in Zandi's Correlation, the critical velocity calculated is very similar to that obtained by Durand's Correlation. Turian (personal communication) suggested that for a wide range of particle sizes, the d_{50} particle size should be used for calculating the critical velocity of suspended particles, whereas

d_{max} should be used only for a very narrow range of particle sizes. The distribution of 20- and 200-mesh coal particles used for the design of the SRC-I Demonstration Plant is shown in Figure 1.

Comparison of Operating Slurry Pipelines

Three operating slurry pipelines for which data on maximum particle sizes (d_{max}) and operating velocities were available were chosen for the study (Table 1). From the maximum particle size (d_{max}) and the normal distribution of the ground material, the d_{50} and d_{70} particle sizes were estimated. (The d_{70} particle size denotes a size such that 70 wt % are coarser and 30 wt % are finer.) The critical velocities calculated using Zandi's correlation for d_{50}, d_{70}, and d_{max} were then compared with the actual velocities in the three operating pipelines; the results are shown in Table 2. From this table, the following observations can be made:

- Critical velocities based on d_{max} are considerably higher than the actual velocities, indicating that the previous design of the demonstration plant slurry system, based on d_{max}, was extremely conservative.
- Excepting the Black Mesa pipeline, which has a critical velocity 3.3 ft/sec greater than the actual velocity, critical velocities based on d_{70} are only slightly higher than the operating velocities.
- Critical velocities based on d_{50} are lower than the actual velocities, which indicates that the prediction of critical velocity based on d_{50} particle size is more in line with industrial experience. The general practice for slurry pipelines is to design the system at a velocity 1 ft/sec higher than the d_{50} critical velocity. However, it appears that the margin of safety for the Black Mesa pipeline was reduced, probably for economic reasons.

One important difference between the operating pipelines and the proposed SRC-I Demonstration Plant slurry system is that the former operate on water slurry, which has different density and viscosity from oil slurry. These differences may affect the prediction of critical velocity for an oil slurry system, and because there are no published experimental oil slurry data, it is prudent to design the demonstration plant slurry system at a critical velocity based on d_{70} particle size. Because of this conservative approach, it was decided not to add any safety factor over the d_{70} critical velocity.

Basic Concepts

In this study, the basic concepts developed during previous work are summarized below and reproduced in Figure 2:

- Normally, two slurry drums, V-12201 A&B, are in operation, but each is capable of providing the total design throughput.
- Six separate slurry trains run from the slurry drums.
- From each slurry drum, there are actually three separate trains, each consisting of a low-pressure (LP) circulation pump (P-12201) and a high-pressure (HP) charge pump (P-12202). Only two trains are normally operating and the third one is a spare.
- Each HP charge pump is directly connected with the respective LP circulation pump on a one-to-one basis, with no interconnections between them Only the spare train joins at the discharge of the two normally operating HP charge pumps.
- LP circulation pumps P-12201 A-F are located in the space between the two slurry drums, and are arranged in two rows facing away from the drums for symmetry of suction lines. Each pump is provided with separate suction connections from both slurry drums.
- The four slurry heaters (H-12301) are fed by four operating HP charge pumps, with no interconnections between them.

The purpose of this study is to arrive at a system design capable of processing 200- as well as 20-mesh coal, and allowing a 50% turndown in train capacity.

NEW DESIGN

A single system must be designed to handle both 200- and 20-mesh coal so that no major modifications will be necessary when 20-mesh coal is processed in the future. For this purpose, the system is first designed to process 20-mesh coal, and then checked to determine if it can be adapted to the 200-mesh size.

Because there are four separate but identical operating trains from the slurry drums to the slurry heaters, the design of only one train will be discussed.

Design for 20-Mesh Coal

Table 3 lists the critical velocities calculated for both d_{50} and d_{70} particle sizes based on 8- and 6-in. pipe. The 8-in. schedule 20 pipe is used for the suction and discharge of pump P-12201, the 6-in. double extra strong (xxs) pipe is used for P-12202 discharge, and the 6-in. schedule 40 pipe is used in the recycle line from the P-12202 suction manifold to the slurry mix tanks, V-12201 A&B.

Capacity of LP Circulation Pump (P-12201). The design critical velocity on the basis of d_{70} particle size for 8-in. schedule 20 pipe is 8.3 ft/sec. The corresponding flow rate for pump P-12201 is 1,345 gallons per minute (gpm), which is explained under recycle line requirements below. At this high rate, the actual velocity is equal to the critical velocity.

HP Charge Pump (P-12202) Discharge Piping. The design flow rate at P-12202 discharge is 562 gpm. Under this condition, the actual velocity through 6-in xxs pipe is 9.6 ft/sec, which is well below the maximum allowable velocity of 18 ft/sec for slurry pipe. At 50% turndown, also a design consideration, the actual velocity through this discharge pipe is 4.8 ft/sec, which is lower than the design critical velocity of 6.4 ft/sec based on d_{70} particle size, and which indicates only 67% turndown. However, based on d_{50} particle size, the critical velocity is only 4.8 ft/sec. Therefore, in actual practice it is possible to turn down each pump to 50%.

Recycle Line. To prevent solids settling in the suction manifold of the HP slurry charge pump (P-12202), recirculation of a quantity of slurry is required through the suction manifold to the slurry mix tank. In addition to this normal circulating quantity, the system should be able to handle the full design flow of the LP slurry circulation pump (P-12201) in case the HP slurry charge pump is shut down.

The objective is to design the system with one recycle line to cover the whole range of flow. As shown in Table 4, the range of operability increases with the size of this recycle line; with 6-in. line, the desired range can be covered. However, in such a case, the normal circulation quantity is fairly high.

The normal recycle quantity is 822 gpm, and gives an actual velocity of 9.1 ft/sec through the 6-in schedule 40 pipe, which is above the d_{70} critical velocity of 7.1 ft/sec. However, when the slurry charge pump is down, the recirculation quantity will increase to 1,348 gpm, and the velocity through the 6-in recycle line will thus increase to an acceptable 15.0 ft/sec. The whole range of flow is therefore covered by a single 6-in recycle line.

The HP slurry charge pump requires about 50 psig pressure at the suction for proper operation. A pressure control valve (PCV) is located on the recycle line. When total recirculation is required through this recycle line during the shutdown of P-12202, the PCV is fully opened manually to keep the recycle system pressure drop low.

Pressure Drop. Table 2 also shows the $\Delta P/100$ ft based on the design critical velocity. Pump Calculation Work Sheet No. 1 gives the calculation for P-12201 pump head, brake horsepower (bhp), and the available net positive suction head (NPSH).

The total system design is shown in Figure 3.

Design for 200-Mesh Coal

Table 5 lists the critical velocities for d_{50} and d_{70} particle sizes based on the same pipe sizes used for 20-mesh coal design. In all cases, the actual velocities through these lines are higher than the critical velocities based on d_{70} particle size, and therefore create no problem.

Capacity of LP Circulation Pump (P-12201). The capacity of this pump is obtained indirectly by setting the velocity through the recycle line equal to the design critical velocity, which is 2.8 ft/sec. The normal recycle flow rate through the 6-in recycle line will thus be 253 gpm. To determine the flow rate for pump P-12201, the design flow rate of 562 gpm for pump P-12202 is added to the recycle line flow rate, and from this total the normal flush solvent injection of 36 gpm used in pump P-12202 is subtracted. The P-12201 pump capacity is thus 779 gpm at the discharge, which includes 3 gpm of flush solvent.

HP Charge Pump (P-12202) Discharge Piping. The actual velocities through the 6-in pipe for normal design flow and at 50% turndown are 9.6 and 4.8 ft/sec, respectively. The design critical velocity for this pipe size is only 2.6 ft/sec.

Recycle Line. The normal recycle quantity is 253 gpm, which gives the actual velocity equal to the design critical velocity of 2.8 ft/sec. The velocity for the maximum recycle flow of 779 gpm is 8.7 ft/sec.

Pressure Drop. The design pump head, brake horsepower, and the available NPSH for pump P-12201 are shown in Pump Calculation Work Sheet No. 2 based on the Δ P/100 ft shown in Table 3.

CONCLUSIONS

- The system designed for 20-mesh coal is also adaptable to 200-mesh coal.
- The system design allows 50% turndown.
- The previous recommendation of special 5-in. pipes for P-12202 pump discharge is no longer valid.
- No multiple recycle lines are required.

LITERATURE CITED

McElvain, R. E. 1974. High pressure piping. Skillings Mining Review, 26 January:63(4).

Turian, R. M., and T. Yuan. 1977. Flow of slurries in pipelines. AIChEJ 23(3).

Table 1

Coal Pipelines[a]

Transported solids	Pipe length and bore	Company and location	Size of solids	Slurry solids (wt %)	Specific gravity of the solids	Velocity (ft/sec)	Pumps capacity	Pipeline	Remarks
Coal	108 mi 10 in.	Consol. Coal Company Ohio coalfields to Cleveland, Lake Erie, U.S.A.	Up to 14 mesh (1.20 mm)	50-60	1.15 (1.4)	4.5-5.25	1.3×10^6 ton/yr (150 ton/hr)	Duplex double-acting. Three pumps/station at approx. 30-mi intervals	Operated 1956-1963. Closed due to poor economics.
Coal	273 mi 18 in. and 12½ in.	Black Mesa Pipe Inc. (Subsidiary of Pacific Pipelines Inc.) and Peabody Coal Co., Arizona, to Nevada/Calif. border, U.S.A.	-	-	-	6	4.8×10^6 ton/yr rising to 6×10^6 ton/yr	Total of thirteen 4,500 gal/min duplex pumps. 4 pumping stations	Operating
Coal	350 mi 20 in.	Consol. Coal Company and Texas Eastern Transmission Co. West Virginia to New York City and Baltimore area, U.S.A.	-	-	-	-	10×10^6 ton/yr	-	Proposed
Coal	38 mi 12 in.	Novovolynskaya Mine, U.S.S.R.	- 1/32 in.	50	-	4.75	220 ton/hr	-	Operating. Built 1957.
Coal	5½ mi 15 in.	Houillères du Bassin de Lorraine. Carling, France	16 mesh (1 mm)	25-30	-	7-10	250 ton/hr	-	Operating since 1952
Coal	490 mi	Cascade Pipeline Ltd. E. Kootenay, B.C., to Vancouver	-	-	-	-	-	-	Proposed by C.P.R.
Coal	126 mi 10 in.	Polish Central Mining Industry, Poland	Up to 2 in.	-	-	-	-	-	Operating

[a] Reprinted from Hazleton Pumps in "Hydraulic Transport of Minerals Magazine," April 1972, Vol. 126(4).

Table 2

Comparison of Operating Pipelines

Company	Maximum particle size	Particle mesh size		Operating velocity (ft/sec)	Critical velocity (ft/sec)		
		d_{50}	d_{70}		d_{50}	d_{70}	d_{max}
Consol Coal Co.	14 mesh 1,410 microns	180	80	4.5 to 5.25	3.6	5.7	12.8
Black Mesa Pipe, Inc.	7 mesh 2,830 microns	115	40	6	5.4	9.3	16.4
Novovolynskaya Mine, U.S.S.R.	22 mesh 800 microns	280	120	4.75	2.8	5.1	11.9

Table 3

20-Mesh Case

Line	Flow (gpm)	Velocity (ft/sec)	Critical velocity (ft/sec)		Pressure drop (psi/100 ft)
			d_{50}	d_{70}	
P-12201 suction (8-in. schedule 20)	1,345	8.3	6.2	8.3	2.6
P-12201 discharge (8-in. schedule 20)	1,348	8.3	6.2	8.3	2.6
P-12202 discharge (6-in. schedule xxs)	281 to 562	4.8 to 9.6	4.8	6.4	2.7 to 4.1
Recycle (6-in. schedule 40)	822 to 1,348	9.1 to 15.0	5.3	7.1	3.2 to 6.6

Table 4

P-12201 Recycle Line Data[a,b,c,]

Line sizes (in.)		Flow rate (gpm)		Actual velocity (ft/sec)		Critical velocity for D_{70} particle (ft/sec)		Pressure drop (psi/100 ft)		
Actual	Nominal	min/max 20 mesh	min/max 200 mesh	min/max 20 mesh	min/max 200 mesh	20 mesh	200 mesh	min/max 20 mesh	min/max 200 mesh	Remarks
2	1.939	84/610	84/610	9.1/66.3	9.1/66.3	4.0	1.6	10.1/409.4	10.5/422	Velocity exceeds 18 ft/sec and pressure drop is very high for 20-mesh coal
4	4.026	232/758	95/621	5.8/19.1	2.4/15.7	5.8	2.3	3.1/15.8	0.8/11.7	Velocity exceeds 18 ft/sec and pressure drop is on high side for 20-mesh coal
6	6.065	822/1,348	253/779	9.1/15.0	2.8/8.7	7.1	2.8	3.2/6.6	0.7/2.6	The most desirable operating conditions
8	8.125	1,340/1,866	540/1,066	8.3/11.5	3.3/6.6	8.3	3.3	2.6/3.3	0.7/1.2	Higher pump capacity and hp requirement for P-12201 compared to 6-in. line
10	10.25	2,390/2,916	975/1,501	9.3/11.3	3.8/5.8	9.3	3.7	2.4/2.8	0.6/0.8	Higher pump capacity and hp requirement for P-12201 compared to 6- and 8-in. line

[a] This table is valid when a single line for P-12201 pump is used, and P-12201 pump capacity is a variable.

[b] All minimum flow rates, except 2-in. recycle line for 20- and 200-mesh coal, were calculated such that no settling occurs in recycle, suction, or discharge lines of P-12201. Minimum flow of 84 gpm for 2-in. recycle line corresponds to 15% of P-12202 forward flow rate.

[c] Maximum flow rate is P-12201 capacity when P-12202 is down.

Table 5

200-Mesh Case

Line	Flow (gpm)	Velocity (ft/sec)	Critical velocity (ft/sec)		Pressure drop (psi/100 ft)
			d_{50}	d_{70}	
P-12201 suction (8-in schedule 20)	776	4.8	2.1	3.3	0.8
P-12201 discharge (8-in schedule 20)	779	4.9	2.1	3.3	0.8
P-12202 discharge (6-in schedule xxs)	281 to 562	4.8 to 9.6	1.6	2.6	1.2 to 3.9
Recycle (6-in schedule 40)	253 to 779	2.8 to 8.7	1.8	2.8	0.7 to 2.6

Figure 1
Coal Distribution

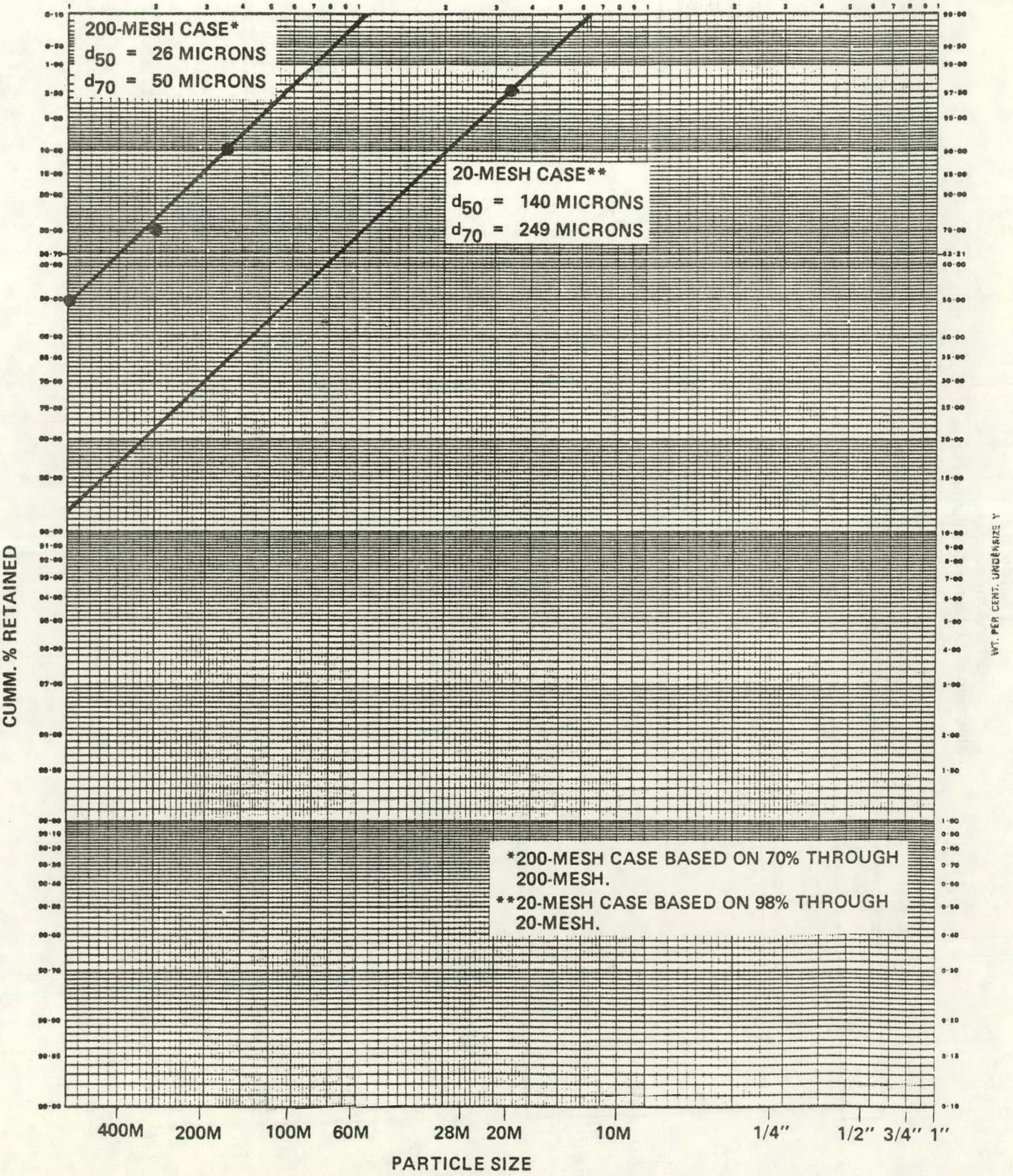


Figure 2
General Arrangement
Relocation of LP Slurry Circulation Pumps

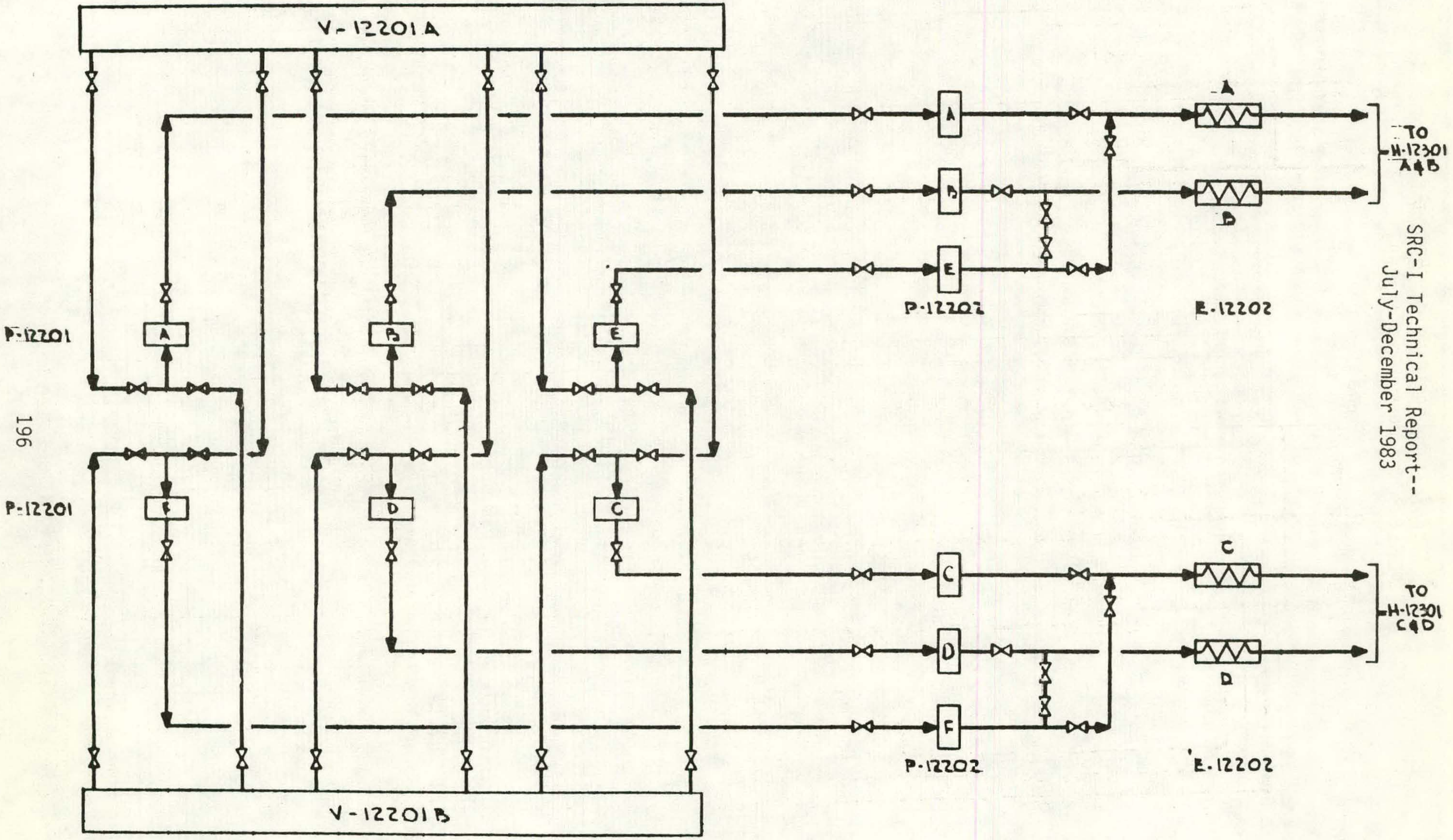
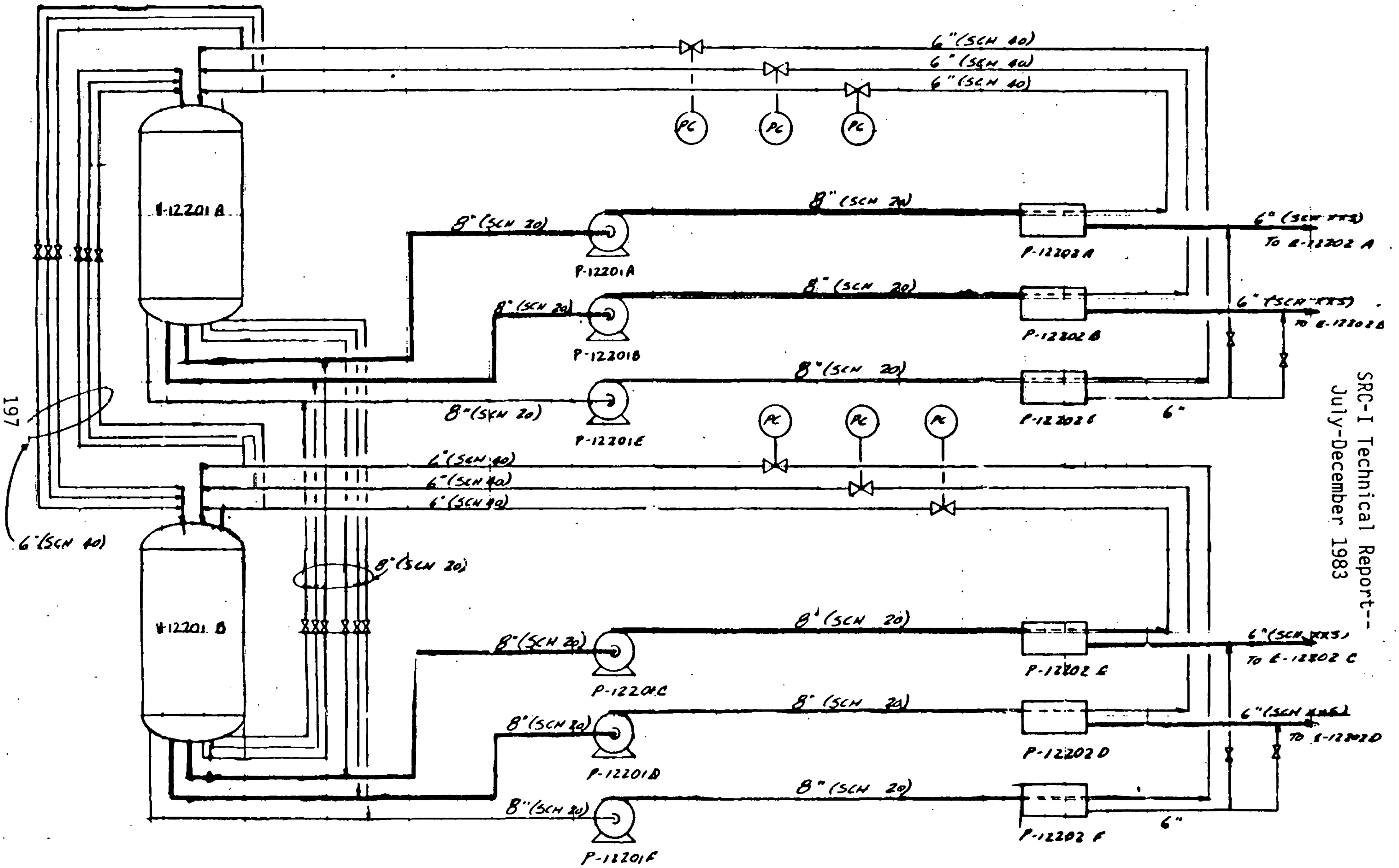


Figure 3
Single Recirculation Line Design



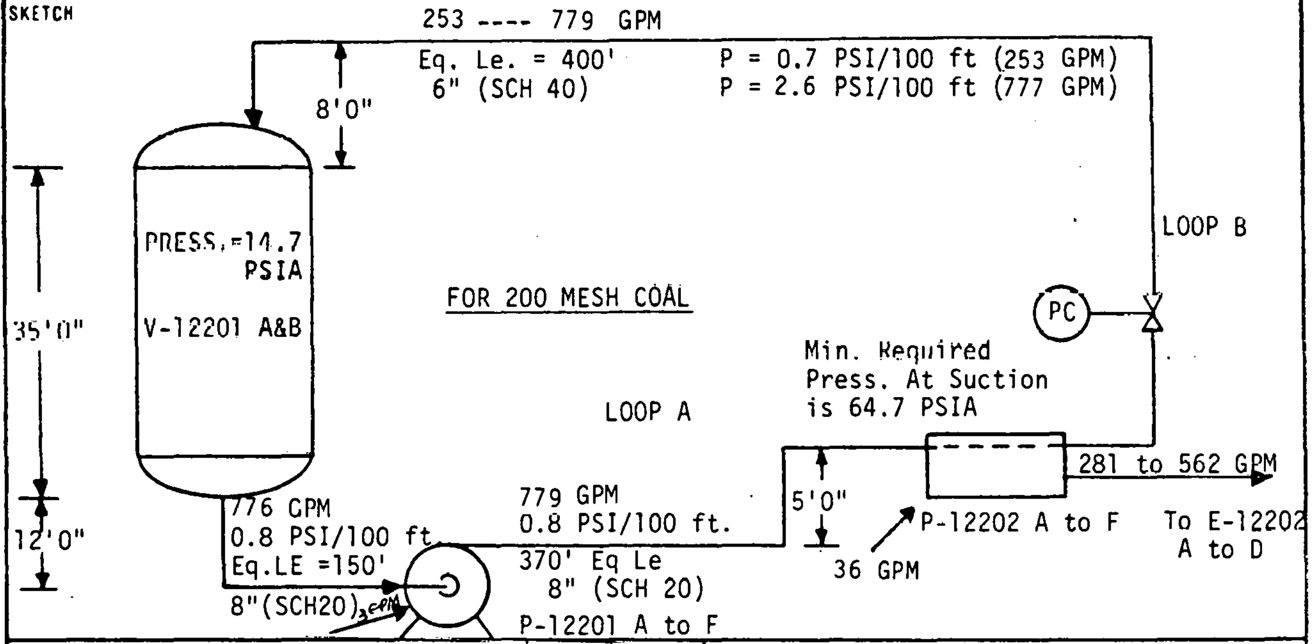
PUMP CALCULATION WORKSHEET
No. 1

CONTRACT NO ____
EQUIPMENT NO ____
NO. REQUIRED ____
SHEET ____ OF ____

CLIENT INTERNATIONAL COAL REFINING COMPANY

PROJECT 6000 TPSD SRC-I DEMONSTRATION PLANT PLANT LOCATION NEWMAN KENTUCKY

SKETCH



SUCTION PRESSURE	LOOP	A	B
ORIGIN PRESSURE	PSIA	14.7	
STATIC HD (FT X SP GR X .433)	PSI	5.7	
—LOSS (LINE + OTHER ΔP)	PSI	1.2	
PUMP SUCTION PRESSURE	PSIA	19.2	
NET POSITIVE SUCTION HEAD			
STATIC HD	FEET	12.0	
—LINE LOSS (PSI X 2.31/SP GR)	FEET	2.5	
+ (ORIG PR —VAP PR) (2.31/SP GR)	FEET	25.6	
AVAILABLE NPSH	FEET	35.1	
DISCHARGE PRESSURE			
DELIVERY PRESSURE	PSIA	64.7	14.7
STATIC HEAD	PSI	2.4	23.8
LINE LOSS	PSI	3.0	2.8
ΔP CONTROL VALVES	PSI		23.4
ΔP EXCHANGERS	PSI		
ΔP FURNACES	PSI		
ΔP ORIFICES	PSI		
ΔP OTHER*	PSI	10.0	
PUMP DISCHARGE PRESSURE	PSIA	80.1	64.7
DIFFERENTIAL PRESSURE			
DISCHARGE PRESSURE	PSIA	80.1	
SUCTION PRESSURE	PSIA	19.2	
TOTAL PUMP ΔP	PSI	60.9	
DIFFERENTIAL HEAD	FEET	127.9	
ESTIMATED BRAKE HORSEPOWER $\frac{\text{(GPM) X (PSI)}}{\text{1715 X 0.41 (PUMP EFF)}}$ =	BHP	67.5	

*MINIMUM OF 2 PSI FOR DYNAMIC HEAD

SERVICE COAL SLURRY CIRCULATION PUMPS

LIQUID PUMPED	SLURRY	
PUMPING TEMPERATURE (P.T.)	400	°F
VISCOSITY @ P.T.	56.0	LB/(FT-HR)
VAPOR PRESS. @ P.T.	2.5	PSIA
SPECIFIC GRAVITY @ P.T.	1.10	
FLOW NORMAL @ 60 F		GPM
FLOW NORMAL @ P.T.	779	GPM
FLOW DESIGN @ P.T.	779	GPM

REMARKS

1) Normal recycle rate is 253 GPM but if P-12202 is inoperative, the full 746 GPM can be recycled.

2) Flush solvent rates:
   P-12201 - 3 GPM
   P-12202 - 36 GPM

3) Line "B" is the recycle line from P-12202 manifold to V-12201.

4) During 100% recycle the pressure control valve will be opened fully to minimize pressure drop.

PREPARED BY	REVISION △	REVISION △	REVISION △
DATE			
APPROVED BY	BY DATE	BY DATE	BY DATE
DATE	APP'D DATE	DATE	APP'D DATE

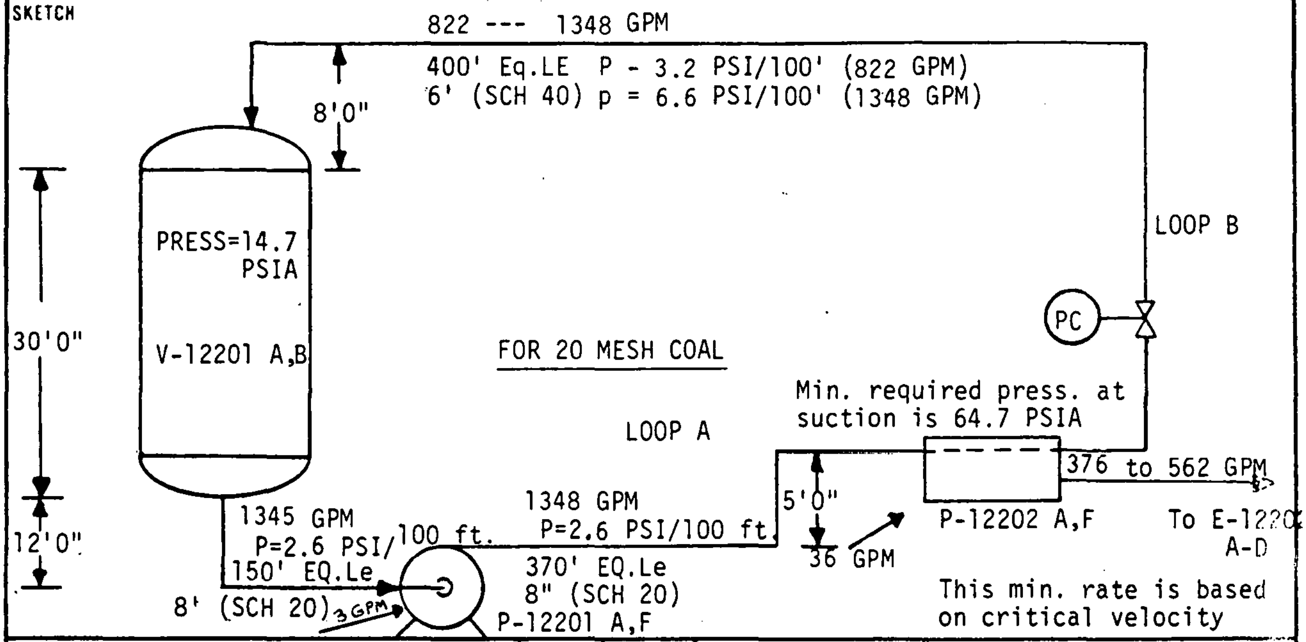
FORM 9315 (8/80)

	PUMP CALCULATION WORKSHEET No. 2	**CONTRACT NO.** **EQUIPMENT NO.** **NO. REQUIRED** **SHEET** ____ **OF** ____

CLIENT **INTERNATIONAL COAL REFINING COMPANY**

PROJECT 6000 TPSD SRC-I DEMONSTRATION PLANT **PLANT LOCATION NEWMAN, KENTUCKY**

SKETCH



	LOOP	A	B
SUCTION PRESSURE			
ORIGIN PRESSURE	PSIA	14.7	
STATIC HD (FT. X SP GR. X .433)	PSI	5.7	
—LOSS (LINE + OTHER) ΔP	PSI	3.9	
PUMP SUCTION PRESSURE	PSIA	16.5	
NET POSITIVE SUCTION HEAD			
STATIC HD.	FEET	12.0	
—LINE LOSS (PSI X 2.31/SP. GR.)	FEET	8.2	
+ (ORIG. PR —VAP. PR.) (2.31/SP. GR.)	FEET	25.6	
AVAILABLE NPSH	FEET	29.4	
DISCHARGE PRESSURE			
DELIVERY PRESSURE	PSIA	64.7	14.7
STATIC HEAD	PSI	2.4	23.8
LINE LOSS	PSI	9.6	10.4
ΔP CONTROL VALVES	PSI		15.8
ΔP EXCHANGERS	PSI		
ΔP FURNACES	PSI		
ΔP ORIFICES	PSI		
ΔP OTHER*	PSI	10.0	
PUMP DISCHARGE PRESSURE	PSIA	85.7	64.7
DIFFERENTIAL PRESSURE			
DISCHARGE PRESSURE	PSIA	86.7	
SUCTION PRESSURE	PSIA	16.5	
TOTAL PUMP ΔP	PSI	70.2	
DIFFERENTIAL HEAD	FEET	147.4	
ESTIMATED BRAKE HORSEPOWER $\frac{(GPM) \times (PSI)}{1715 \times 0.53 \text{ (PUMP EFF)}} =$	BHP	104.1	

***MINIMUM OF 2 PSI FOR DYNAMIC HEAD**

SERVICE COAL SLURRY CIRCULATION PUMPS

LIQUID PUMPED	SLURRY	
PUMPING TEMPERATURE (P.T.)	400	°F
VISCOSITY @ P.T.	56.0	LB(M)/FT-HR
VAPOR PRESS. @ P.T.	2.5	PSIA
SPECIFIC GRAVITY @ P.T.	1.1	
FLOW, NORMAL @ 60°F		GPM
FLOW, NORMAL @ P.T.	1348	GPM
FLOW, DESIGN @ P.T.	1348	GPM

REMARKS:

1) Normal recycle rate is 822 GPM but if P-12202 is down the full 1348 GPM can be recycled.

2) Flush solvent rates:
   P-12201 3 GPM
   P-12202 36 GPM

3) Loop "B" is the recycle line from P-12202 manifold to V-12201.

4) During 100% recycle the pressure control valve will be fully opened to minimize pressure drop.

PREPARED BY **DATE**	**REVISION** △	**REVISION** △	**REVISION** △
APPROVED BY	**BY** **DATE**	**BY** **DATE**	**BY** **DATE**
DATE	**APP'D** **DATE**	**DATE**	**APP'D** **DATE**

FORM 9315 (8/80)

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LC-FINER CATALYST TESTING

D. Garg*

During this final period of study, the activity and aging of Shell 324 Ni-Mo-Al catalyst in the hydroprocessing of heavy SRC was examined at 825°F. An SRC conversion of 54% occurred during the first 24 hr of operation, while overall desulfurization, denitrogenation, and deoxygenation were 93, 60, and 76%, respectively. SRC conversion, oils and hydrocarbon gas production, overall denitrogenation and deoxygenation, hydrogen consumption, and first-order rate constant for SRC conversion decreased slightly during the initial 96 hr of operation, (catalyst age = 59 weight units of SRC per weight unit catalyst), but decreased significantly thereafter. However, SRC sulfur content and overall desulfurization decreased gradually with time on stream. The decline in catalyst activity after 96 hr, as reflected in these results, could have been partly due to upsets in PDU operation.

Analysis of spent catalyst revealed a significant reduction in surface area and an almost total disappearance of pore structure. Furthermore, the spent catalyst showed heavy sodium deposition (2%), which was due to the presence of a high level of sodium in the feed. A mass balance around the system revealed that only minor portions of iron and titanium present in the feed SRC were deposited on the catalyst, whereas almost all of the sodium was retained by the catalyst. The reduction in surface area due to coke and metal deposition coupled with total loss of pore structure were the main reasons for the significant decline in catalyst activity.

INTRODUCTION

Production of distillate liquids by catalytic hydrocracking of solvent-refined coal (SRC) is a major processing step in the SRC-I

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Demonstration Plant. One of the few commercially available hydrocracking processes that can handle SRC is the Lummus-Cities Expanded-Bed Process. Design plans are to install an LC-Finer in the demonstration plant that is capable of handling 14,000 barrels/day (bpd) total feed, of which 30% is recycle distillate diluent oil.

The LC-Finer process was chosen because of its proven application for processing residual petroleum oils. The process employs conventional hydroprocessing catalysts in an ebullated-bed reactor, in which gas and slurry are fed upwardly through an expanded bed of catalyst. This process is exceptionally well-suited to the processing needs of a refractory stock because catalyst can be continuously added or withdrawn from the reactor without having to depressurize or cool it. Because SRC essentially comprises asphaltenes and preasphaltenes, its composition may be the major contributor to rapid catalyst aging.

To prove SRC hydrocracking technology in the demonstration plant, a catalyst having a suitable life for hydrocracking SRC must be specified before demonstration plant start-up. Prior LC-Finer process studies estimated that 0.7 lb of catalyst would be consumed per barrel of SRC feed, which is equivalent to a batch catalyst age of about 620 lb SRC/lb catalyst. This consumption level is equivalent to approximately $2.50 per barrel of feed, thereby making the catalyst cost a major factor in the hydrocracking process.

The major purpose of this program has been to develop a data base that will increase confidence in the process design by verifying Shell 324 Ni-Mo catalyst performance. The experimental program is designed to determine product distribution with catalyst age.

EXPERIMENTAL PROCEDURES

Run Conditions

Experimental run CCL-63, which hydroprocessed SRC using a novel fixed-bed catalyst basket reactor, was completed during this reporting period. Modified Shell 324 Ni-Mo, an alumina-supported catalyst, was used in the run. Typical reaction conditions were as follows: solvent/SRC ratio - 30 wt %/70 wt %, hydrogen pressure - 2,000 psig, H_2 feed rate - 8,000 scf/bbl total feed, temperature - 825°F, weight hourly space velocity (WHSV) - 1.0 g feed/g catalyst/hr. The run was carried out for 356 hr on stream, including 165 hr at reaction temperature. During this run, several samples were collected for detailed analysis.

Reactor Design

The reactor design was identical to that used in the previous run CCL-54 (Garg, 1982). The amount and volume of catalyst used in run CCL-63 are provided in Table 1.

Feed Materials

Heavy SRC (HSRC) and process solvent from the Wilsonville hydrotreater used in this program were collected during run 235 at the Wilsonville Advanced Coal Liquefaction Facility. Detailed analyses of the feed materials are reported in Table 2. The modified Shell 324 catalyst described previously (Garg, 1982) was used in run CCL-63.

Process Development Unit (PDU) Operation

Preparation. The catalyst basket was filled with 300 g (349 mL) of catalyst and placed in the 2-L autoclave. The autoclave was sealed, and the entire PDU was checked for proper operation of all controllers and also pressure-tested for possible leaks as discussed previously (Garg, 1982).

Sulfiding the Catalyst. The catalyst was sulfided at 600°F using creosote oil mixed with ethyl disulfide. The sulfiding procedure was identical to that followed for run CCL-54 (Garg, 1982).

Run Procedure. When catalyst sulfiding was completed, the pump was switched to start pumping SRC feed material, which consisted of 70 wt % SRC/30 wt % solvent from the Wilsonville hydrotreater. The reactor temperature was increased from 600 to 825°F and maintained at that level for the entire run. PDU operation was smooth for the first 138 hr on stream (including 102 hr at 825°F), after which the feed pump failed and the unit was shut down (Table 3). The temperature of the reactor was reduced to 550°F to prevent any catalyst deactivation during shutdown, and operation was resumed after 216 hr. However, operation was again halted at 241 hr (127 hr on stream at 825°F) due to a hydrogen leak, and the reactor temperature was again reduced to 550°F to prevent catalyst deactivation during shutdown. Operation was resumed at 318 hr and continued until 356 hr. Overall, the unit was on stream at the 825°F reaction temperature for 165 hr; during which several samples were taken for detailed analysis. A summary of sample numbers and detailed process conditions is given in Table 4.

Work-Up Procedure

The feed and product liquid samples were solvent-separated and analyzed by encapsulated gas chromatographic simulated distillation, as discussed previously (Garg, 1982), to determine 850°F- and 850°F+ fractions (see Table 5). Overall product distribution was calculated on the basis of conversion of 850°F+ material to gases and liquid, and SRC conversion was calculated using the following formula:

$$\%\ \text{SRC conversion} = \frac{(850^\circ\text{F+})_{\text{total feed}} - (850^\circ\text{F+})_{\text{total product}}}{(850^\circ\text{F+})_{\text{total feed}}} \times 100$$

RESULTS AND DISCUSSION

Catalyst Activity and Aging

Initially, the Shell 324 Ni-Mo-Al catalyst yielded an SRC conversion of 54% at 825°F (sample no. 63-24; Table 6). The production of oils (850°F- material) and hydrocarbon gases was 31 and 20%, respectively. Overall desulfurization, denitrogenation, and deoxygenation were 93, 60, and 76%, respectively. Hydrogen consumption, determined by elemental hydrogen balance, was 5%. The pentane-soluble and -insoluble fractions (Table 7) showed a significant decrease in hydrogen, oxygen, and sulfur compared to the total feed liquid. The decrease in hydrogen was probably due to catalytic dehydrogenation or cracking at the 825°F reaction temperature. Although the SRC sulfur content decreased from 1.0 to 0.1 wt %, the nitrogen content of the two fractions changed marginally compared to the feed material. The first-order rate constant for SRC conversion was calculated to be 1.12 hr^{-1}.

Overall SRC conversion changed slightly during the first 96 hr of operation at 825°F (catalyst age = 59 g SRC/g catalyst), but was lower thereafter, i.e., from 52% at 96 hr to 31% at 165 hr (Table 6 and Figure 1). Likewise, oil and hydrocarbon gas production remained constant during the first 96 hr, but was lower thereafter (Table 6 and Figure 2). Hydrogen consumption decreased slightly during the initial 96 hr, but had declined significantly by 151 hr (Figure 3). However, no significant variations in the hydrogen contents of the pentane-soluble and -insoluble fractions were noted with time (Table 7). The decrease in SRC conversion, oil and hydrocarbon gas production, and hydrogen consumption after 96 hr could partly be due to problems with the PDU after 138 and 216 operating hours.

Overall, desulfurization decreased gradually (Figure 4), while SRC sulfur content gradually increased (Table 6). Denitrogenation and deoxygenation generally decreased slightly up to 96 hr of operation at 825°F, but decreased sharply after 96 hr, as shown in Figure 4. As expected, the first-order rate constant for SRC conversion showed the same trend noted for SRC conversion (Figure 5). Finally, the sharp decline in catalyst activity after 96 hr could be partly due to problems with PDU operation.

Catalyst Analysis

A sample of the spent catalyst was recovered for detailed analysis after run CCL-63. The spent catalyst sample was first washed with pyridine and then with pentane to remove any residual SRC from the catalyst surface. The washed sample was then dried at room temperature under a continuous flow of nitrogen.

The detailed analysis of the fresh and spent catalysts is summarized in Table 8. The data indicated a significant amount of carbon deposition on the catalyst, as well as moderate metal deposition other than sodium. The metal and carbon deposition caused a significant reduction in the surface area and pore volume of the catalyst. In fact, the pore volume analysis of the spent catalyst revealed almost a total disappearance of pore structure. The reduction in surface area due to metal and coke deposition coupled with total loss of pore structure were the main reasons for the significant reduction in catalyst activity.

The significant deposition of sodium on the catalyst was due to the presence of a high level of sodium in the feed SRC (Table 2), which in turn resulted from Na_2CO_3 addition in the initial coal liquefaction step (Garg, 1982). A mass balance around the reactor revealed that minor portions of iron and titanium in the feed SRC were deposited on the catalyst, whereas a major portion of sodium in the feed was retained by the catalyst (Table 9). The amount of sodium deposited on the catalyst was higher than that present in the feed SRC because the amount of SRC hydroprocessed or passed through the reactor below 825°F was not included in the total amount of feed SRC. The significant sodium deposition on the catalyst may also be one of the reasons for severe catalyst deactivation.

LITERATURE CITED

Garg, D. LC-Finer catalyst testing. Pages 395-419 in SRC-I quarterly technical report, July-September 1982. DOE/OR/03054-9. International Coal Refining Co., Allentown, PA.

Catalytic, Inc. 1981. Wilsonville Pilot Plant monthly technical progress report. Prepared for U.S. Department of Energy and Electric Power Research Institute, December.

Table 1

Design Specifications of the Reactor

Total volume of reactor (mL)	1,524
Weight of catalyst (g)	300
Bulk volume of catalyst (mL)	349
Stirrer speed (rpm)	1,000

Table 2

Detailed Analysis of Feed

	Wt %		
	HSRC	Process solvent	Total feed
Carbon	86.9	89.3	86.8
Hydrogen	6.0	9.7	7.3
Oxygen	4.1	0.5	3.7
Nitrogen	2.0	0.5	1.6
Sulfur	1.0	<0.1	0.5
Pentane solubles	6.0	100.0	47.3
Pentane insolubles	94.0	0.0	52.7
Metals[a] (ppm)			
Arsenic	<1		
Vanadium	12		
Iron	150		
Sodium	200		
Titanium	120		
Chlorine	44		

[a] Data from Catalytic (1981).

Table 3

Run CCL-63 Operating Summary

Hr on stream	Hr on stream at 825°F	Mode
0-30	0	Catalyst sulfiding
30-35	0	Startup on SRC feed
35-138	0-102	Line-out
138-216	-	Pump failure; system down
216-241	102-127	Line-out
241-318	-	Hydrogen leak; system down
318-356	127-165	Line-out

Table 4

Summary of Sample Numbers and Process Conditions

Sample no.	Hr on stream	Temp (°F)	Pressure (psig)	g of 850°F+ material processed/ g of catalyst
CCL-63-24	24	825	2,000	16.0
CCL-63-48	48	825	2,000	29.5
CCL-63-72	72	825	2,000	44.0
CCL-63-96	96	825	2,000	58.5
CCL-63-151	151	825	2,000	99.1
CCL-63-165	165	825	2,000	108.4

Table 5

Analysis of Feed and Product Liquid Samples

	850°F- (oil)	850°F+ (SRC)
Total feed	36.3	63.7
CCL-63-24	65.0	35.0
CCL-63-48	63.0	37.0
CCL-63-72	64.6	35.4
CCL-63-96	62.7	37.3
CCL-63-151	52.9	47.1
CCL-63-165	49.9	50.1

Table 6

SRC Hydroprocessing Product Distribution

	Sample no.					
	63-24	63-48	63-72	63-96	63-151	63-165
Time on stream (hr)	24	48	72	96	151	165
Reactor temperature (°F)	825	825	825	825	825	825
Pressure (psig)	2,000	2,000	2,000	2,000	2,000	2,000
H_2 flow rate (scf/lb of feed)	16.0	18.7	18.5	17.7	12.4	14.6
WHSV (g of feed/g of catalyst per hr)	0.93	0.87	0.97	0.97	1.24	1.06
Catalyst age (g of SRC feed/g of catalyst)	16.0	29.5	44.0	58.5	99.1	108.4
Product distribution (wt % SRC feed)						
HC	20.37	21.28	17.64	22.31	14.77	15.40
CO, CO_2	0.00	0.00	0.00	0.00	0.00	0.00
H_2S, NH_3	2.57	2.32	2.18	2.28	1.13	1.28
H_2O	5.04	5.29	4.92	5.04	4.18	4.21
Oils	31.44	27.64	32.17	27.35	18.05	13.40
SRC	45.04	47.24	47.00	47.65	64.37	68.16
Conversion	54.67	52.05	53.02	52.35	35.63	31.84
H_2 consumption (wt % 850°F+ material)						
From gas	4.75	4.48	3.90	4.64	2.50	2.48
From solvent	0.22	0.26	0.30	0.27	0.42	0.42
Total	4.97	4.74	4.20	4.91	2.92	2.90
Desulfurization (%)	93.39	88.24	84.23	81.08	63.02	62.20
Denitrogenation (%)	59.89	52.81	49.29	53.67	20.06	25.35
Deoxygenation (%)	76.60	80.36	74.79	76.60	63.43	63.95
SRC sulfur (%)	0.10	0.18	0.23	0.23	0.38	0.38
First-order rate constant for SRC conversion (hr^{-1})	1.12	0.94	1.09	1.07	0.69	0.50

Table 7

Distribution of Elements in the Feed and Product Liquid Samples

	Total feed	Sample no.					
		63-24	53-48	63-72	63-96	63-151	63-165
Pentane solubles							
C	88.8	89.9	90.4	90.3	90.0	90.1	90.2
H	9.2	8.8	8.7	8.7	8.7	8.5	8.5
O	1.4	0.6	0.6	0.7	0.7	0.9	0.9
N	0.7	0.6	0.6	0.6	0.6	0.9	0.7
S	0.[illegible]	0.01	0.01	0.02	0.05	0.07	0.07
H/C	1.24	1.18	1.15	1.16	1.16	1.13	1.13
Pentane insolubles							
C	85.1	89.9	90.2	90.0	90.4	89.6	89.6
H	5.6	5.1	5.0	5.0	4.9	5.0	4.9
O	5.8	3.7	2.5	2.8	2.6	2.9	2.8
N	2.3	2.0	2.2	2.3	2.2	2.6	2.5
S	0.9	0.2	0.4	0.4	0.4	0.5	0.5
H/C	0.79	0.68	0.66	0.67	0.65	0.67	0.66

Table 8

Detailed Analysis of the Fresh and Spent Catalysts

	Wt %	
	Fresh catalyst	Spent catalyst
C	--	22.9
H	--	1.4
N	--	0.6
S	--	6.7
Ni	2.7	1.8
Mo	15.9	6.6
Fe	--	0.1
Ti	--	0.1
Ca	--	0.002
Na	--	2.0
Surface area (m^2/g)	152	22.3
Medium pore diameter (Å)	96	--
Median pore volume (mL/g)	0.38	<0.05

Table 9

Metal Distribution in the Feed and Spent Catalyst
(Weight of Fresh Catalyst, 300 g; Calculated Weight
of Spent Catalyst, 450 g)

	Amount of metals (g)		
	In the feed (1)	Deposited on catalyst (2)	In the product (1) - (2)
Iron	6.6	0.5	6.1
Sodium	8.6	9.0	--
Titanium	5.2	0.5	4.7

FIGURE 1
VARIATION OF SRC CONVERSION WITH TIME

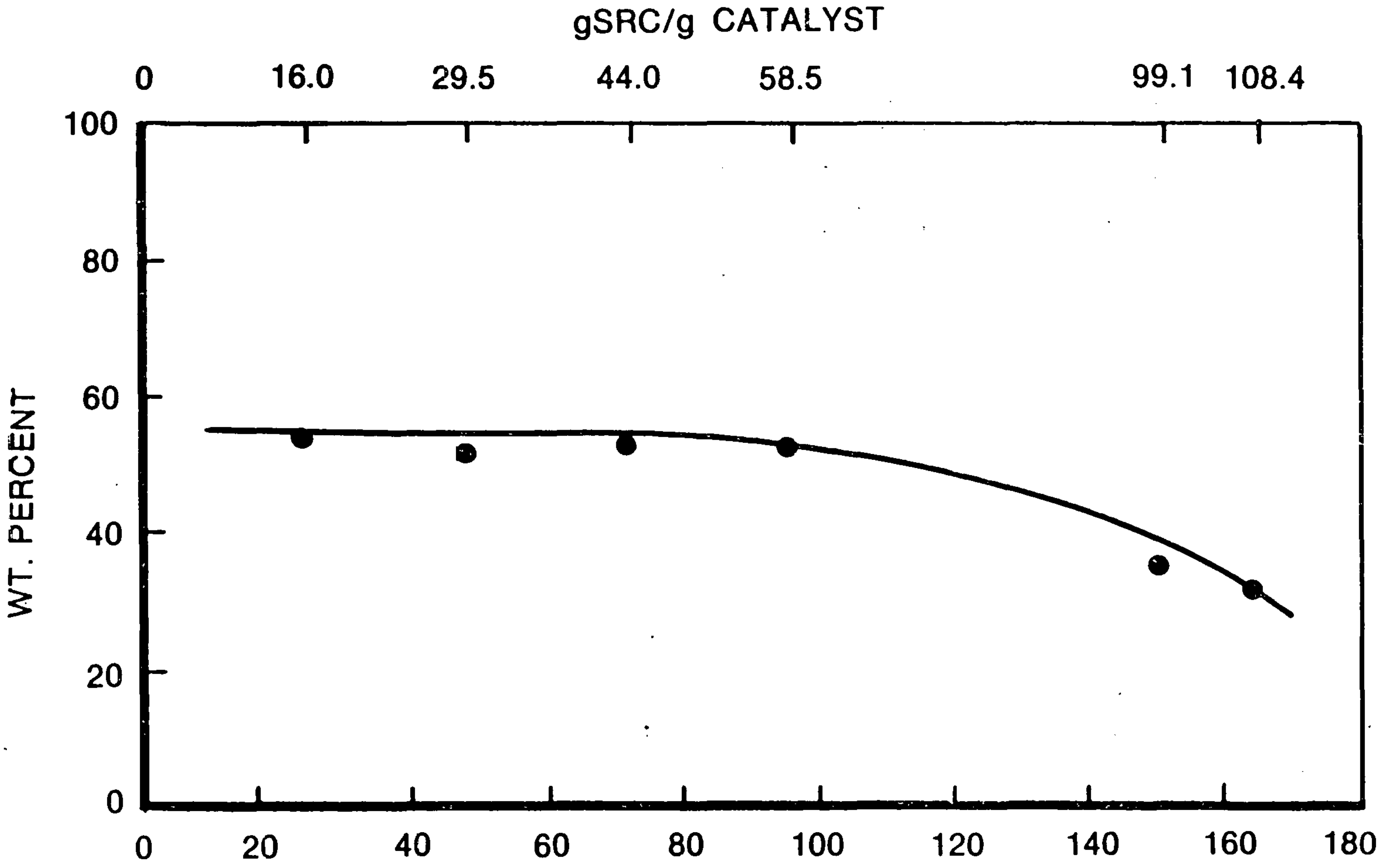


FIGURE 2
VARIATION OF PRODUCTION OF OILS AND HYDROCARBON GAS WITH TIME

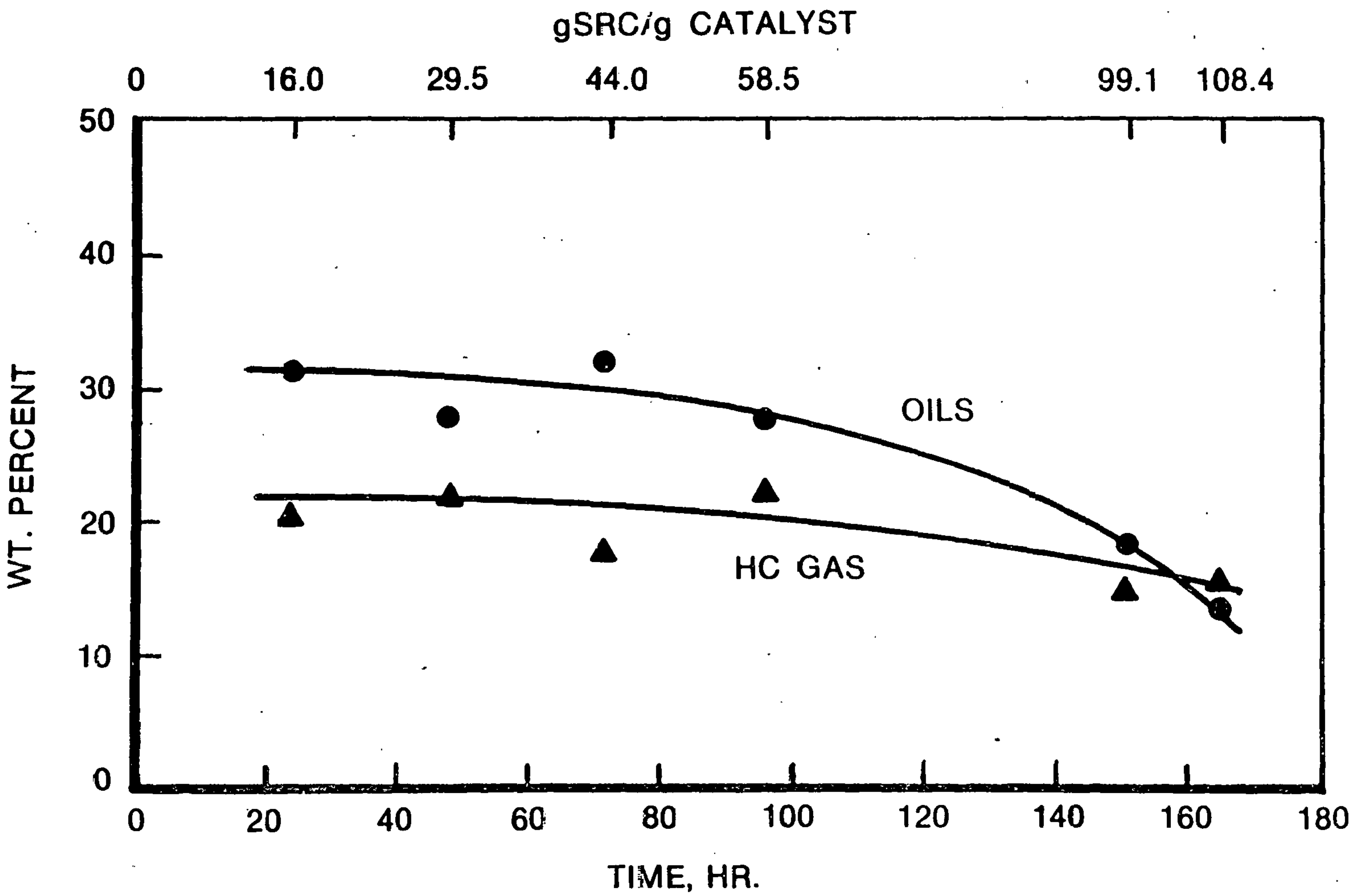


FIGURE 3
VARIATION OF H_2 CONSUMPTION WITH TIME

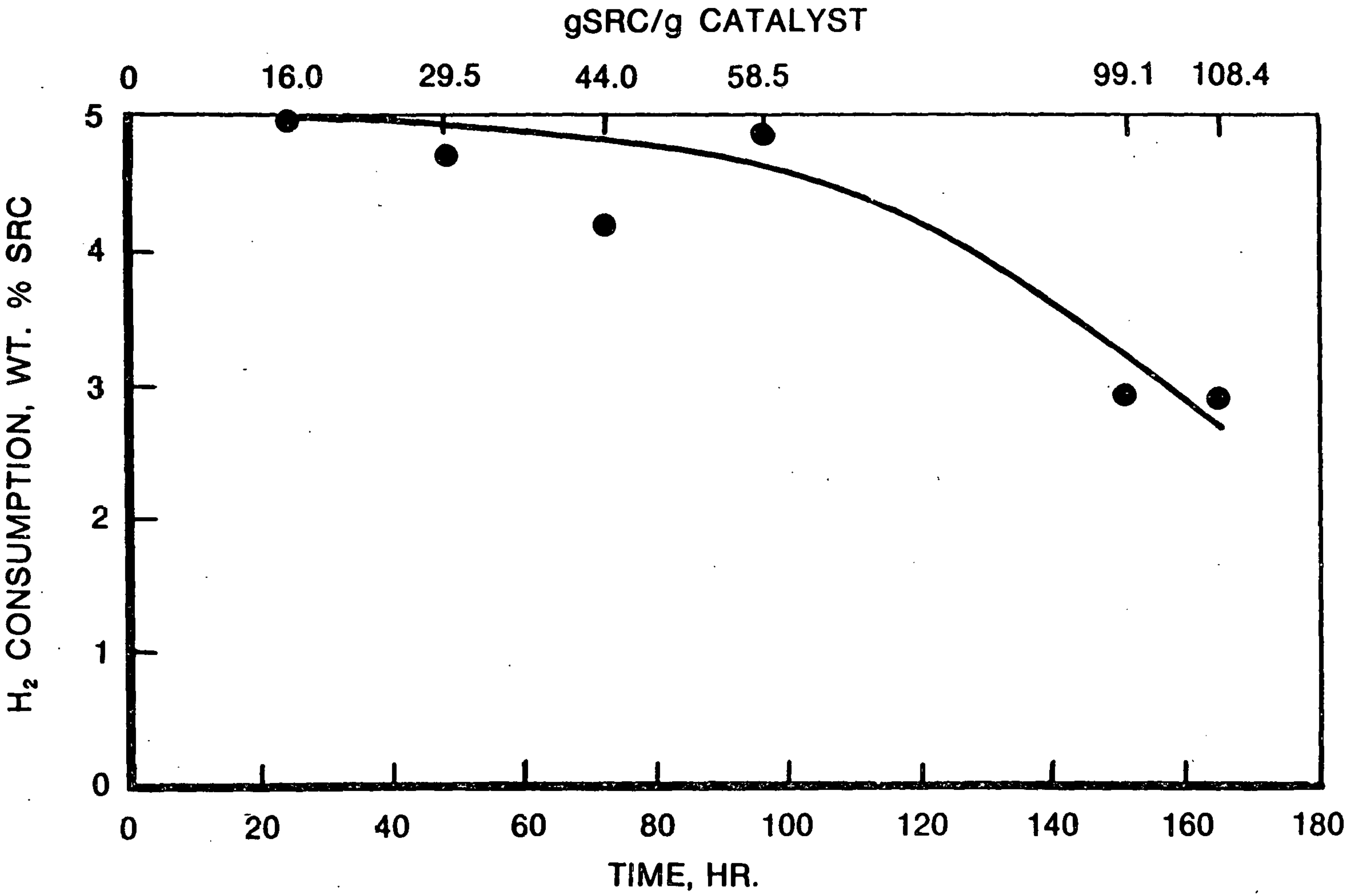


FIGURE 4
VARIATION OF HETEROATOMS REMOVAL WITH TIME

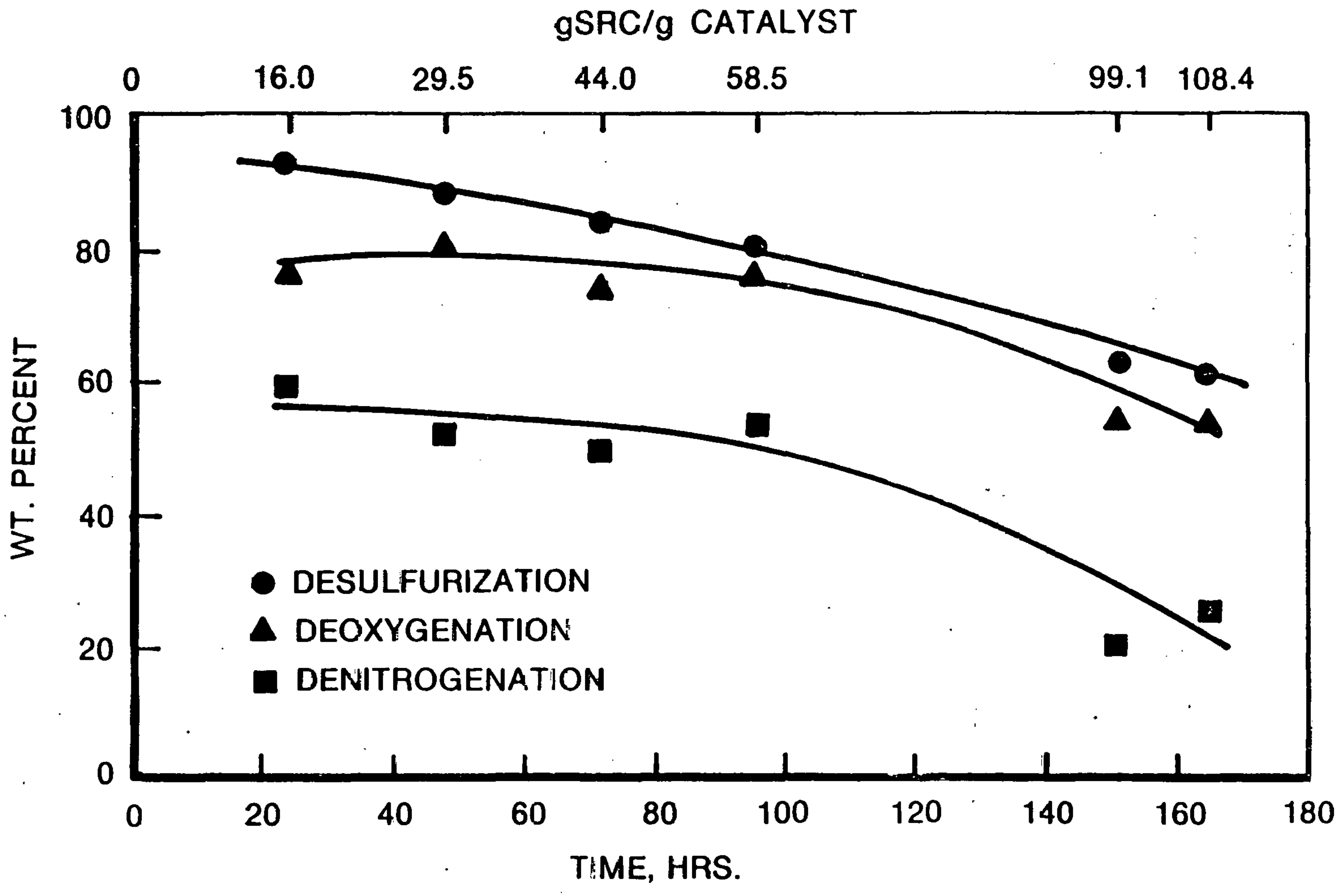
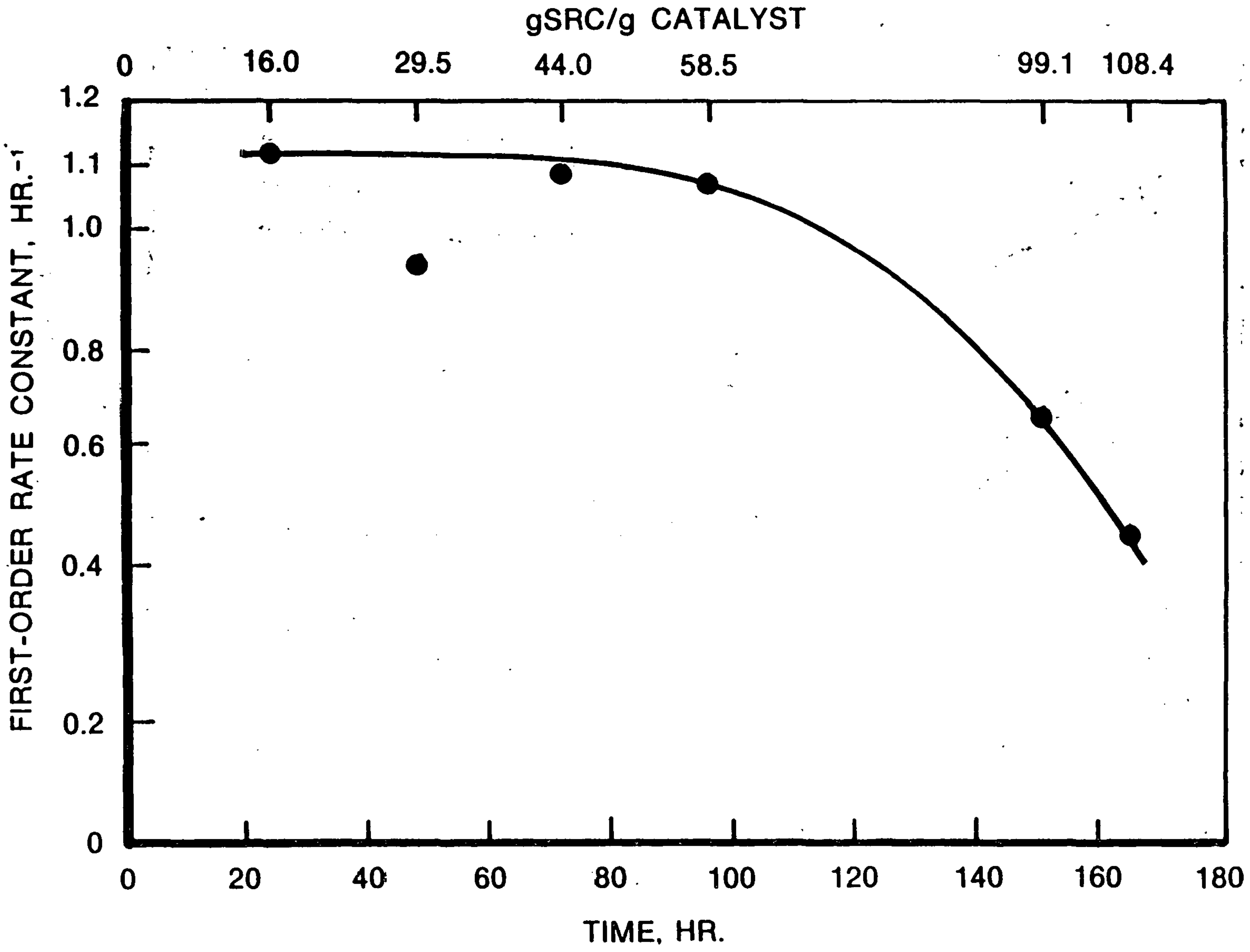


FIGURE 5
VARIATION OF FIRST-ORDER RATE CONSTANT FOR SRC CONVERSION WITH TIME



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The following three articles summarize research performed by ICRC at the SRC pilot plant in Ft. Lewis, Washington during 1980 and 1981. These reports had been withheld from publication pending patent clearance from The Gulf Companies. Clearance to publish has now been obtained.

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ICRC FIRED HEATER TEST PROGRAM

T. W. Thew*
and R. M. Thorogood*

From June 1 to August 8, 1980, experiments were conducted at the Ft. Lewis SRC Pilot Plant to establish a heat-transfer and pressure-drop data base in support of the design of the SRC-I Demonstration Plant slurry fired heater. The fired heater heats a complex three-phase (i.e., coal, solvent, and gas) non-Newtonian mixture to reaction temperatures of 760-800°F, which permits dissolution of the coal and initiates conversion to SRC. The fired heater at Ft. Lewis features a 2-in.-diameter, obround coil.

The series of 54 base-line (solvent only or solvent plus gas) and slurry runs (slurry only or slurry plus gas) examined a range of rates to allow for:

- Calibration of instrumentation
- Comparison of slurry flow and heat transfer with conventional single- and two-phase systems of known physical properties
- Correlation of heat-transfer and pressure-drop characteristics of the three-phase system for scale-up to demonstration plant design.

The primary objective of the experimental program was to gain insight into the apparent gel formation zone of the coal slurry where low heat-transfer coefficients are experienced, thereby increasing the

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*International Coal Refining Co. (ICRC).

possibility of coke formation due to high slurry film temperatures. To determine the allowable heat fluxes for the demonstration plant, it is necessary to correlate the heat-transfer characteristics of the three-phase Ft. Lewis system.

This report describes the experimental program at Ft. Lewis and provides a preliminary evaluation of the data obtained.

Two important results have been achieved:

- The solvent-only, base-line runs assessed the reliability/accuracy of the instrumentation, resulting in improved tube-wall temperature measurements by revision of the thermocouples.

- Reliable data have been obtained for the SRC-I system at demonstration plant conditions.

Future reports will reduce and correlate the data, and work will continue at Ft. Lewis on a 1-1/2-in. diameter coil.

PROGRAM OVERVIEW

During two periods of the summer of 1980, June and July/August, ICRC conducted fired-heater tests. There were two brief shutdowns when instrumentation problems were addressed and resolved.

Table 1 summarizes the number and types of experiments completed in each period. Base-line runs were performed without coal; either a single-phase system (solvent only) or a two-phase system (solvent plus gas) was examined. Slurry runs, either alone or with gas, were performed with coal. When evaluating trends and interpreting data, we treated coal slurry as a single-phase fluid of solids suspended in a liquid.

The 54 runs completed in this program provided an extensive source of data, most of which are discussed here. (Each type of run in the experimental matrix is identified in Table 2.)

Operating conditions are summarized in Table 3. Specifications reflect the demonstration plant design operating conditions at the time of the tests. Though the program was confined to generating fired heater data, the design temperature and pressure for the dissolver were used to obtain information on solvent yield and quality. Because of this, the fired-heater outlet temperature was not an independent variable for the slurry runs; it fell within the range 750-770°F.

Equipment

The fired heater used to conduct the ICRC program, preheater B, is one of two available at Ft. Lewis. Designed by Heat Research Corporation, preheater B features an obround coil (i.e., rounded rectangle), which was selected because the burner placement and coil shape are more representative of larger furnaces, which is not true of the helical design of preheater A (Ackerman, 1979). Preheater B (hereafter the fired heater) was installed in November 1979, commissioned the same month while in the SRC-II mode with a series of coal survey runs, and has been operating since then in both the SRC-I and SRC-II modes, generating design data for the demonstration plant.

Figure 1, a top view of the fired heater, shows the obround coil shape (sometimes referred to as a racetrack) and the six burners. Two distinct zones are apparent: double-fired and single-fired regions defined by the position of the coil relative to the burners. The coil is a 2-in., Schedule-160, 321-stainless steel pipe, 467-ft long, wound through 13-3/4 turns.

Instrumentation

Instrumentation on the 13 3/4 turns of the coil allowed measurement of differential pressure (dP), fluid temperature, and tube-wall temperature as a function of position along the tube length.

Pressure-Drop Profile. The profiles were developed from a series of seven dP cells, each spanning two turns of the coil. Hydrogen-purged pressure taps allowed measurement of differential pressure, provided the flow was balanced. Common taps at the inlet and outlet were linked to another dP cell to give an overall reading. Thus, the sum of the seven individual readings was checked by the overall reading.

Fluid Temperatures. Temperatures were monitored on alternate, odd-numbered turns at the southwest elbows of the coil. A thermowell penetrated the tube wall at the bend through an elbolet, and a thermocouple measured the bulk fluid temperature in the center of the pipe. Including the inlet and outlet points, a total of nine fluid temperatures were used to generate the profile.

Tube-Wall Temperatures. These temperatures were measured with skin thermocouples. Numerous problems with the original instrumentation (see Figure 2) resulted in successive "generations" of thermocouples, each of which addressed the credibility and comprehensiveness of the instruments. The following section discusses in detail the development of the thermocouples.

RESOLUTION OF SKIN THERMOCOUPLE PROBLEMS

In October 1979, an internal report was issued describing the instrumentation on the soon-to-be commissioned preheater B. At a meeting on December 13, 1979 to elucidate design data needs at Ft. Lewis, ICRC discussed two concerns regarding the skin thermocouple arrangement described in the report. First, ICRC felt that more information regarding heat-flux variations was needed than could be obtained with the 13 first-generation thermocouples confined to the west end, or single-fired zone. Second, the use of protective insulation as a radiant shield to prolong thermocouple life was questioned.

As a result of this meeting, a second-generation thermocouple was installed without insulation so that circumferential heat-flux variations could be discerned. However, rather large temperature discrepancies between the first and second generations compelled installing a third generation. Typically, a heat-transfer coefficient from the first generation was two to three times larger than the second- or third-generation coefficient, based on the tube wall-to-bulk fluid temperature difference. These results applied only to positions in the coil downstream of the apparent gel region where high slurry viscosities are experienced.

Because data were still inconsistent, ICRC requested more accurate instrumentation before starting the fired-heater program, so that a fourth generation of 54 skin thermocouples was installed in April. The thermocouple sheath was inserted into a small tube that had been welded to the tube wall (Figure 3). Thermal contact was limited to the tip of the thermocouple.

However, after the runs started in June, analysis of their temperature profiles showed that the thermal contact of these thermocouples was not sufficient to give an accurate reading. There was extensive temperature scatter, attributable to variable contact resistance between the tip of the sheath and the tube wall. Also, the readings were extraordinarily high, which could have resulted from a fin effect induced by the extended weld material and tube. The data were so inaccurate that the temperature profiles from the June runs using fourth-generation thermocouples are not included in Appendix 1 to prevent the transfer of unreliable data, although some profiles will be referred to in the discussion that follows.

Solvent-only Run B-41 typifies the problems encountered. It was conceived as a calibration run to develop correction factors for each of the thermocouples. Theoretical analysis of the variable-contact resistance concept prompted this solvent-only run in which a single-phase fluid with known physical properties was used to back-calculate the skin temperature. However, the attempt to correct previous data with this run was unsuccessful. Even with a sound theoretical correction, accuracies could never be verified. Also, the correction factors were heat-flux dependent, which would be difficult to apply with slurry runs.

Because of the uncertainty of the data, the pilot plant was shut down in early July to modify the instrumentation. The shutdown was agreed to in a meeting where predicted results were presented to show the inaccuracy of the data. Data from Run B-41 (Figure 4) are compared with a predicted skin-temperature profile in Figure 5. The "error band" incorporates a ±30% uncertainty in the inside heat transfer coefficient ($\underline{h}_i$), attributed to uncertainty in the Dittus-Boelter heat-transfer equation and to additional uncertainty in solvent viscosity. Figure 5 also includes the skin-temperature profile generated from the first generation of thermocouples. Although the discrepancy between the first and fourth generations is readily apparent, both monitor temperature on the flame side at the west end of the coil. Results illustrate problems of accuracy, since neither the first- nor the fourth-generation thermocouples fall within the ±30% range, and of consistency due to an ideal linear profile prediction. The latter is critical to the slurry runs, because swelling and coal dissolution create highly nonlinear profiles.

During the plant shutdown the fourth-generation thermocouples were modified to a fifth generation. The insert tube was truncated to minimize the fin effect, and silver solder was placed in the annulus as a thermally conductive medium (Figure 6). Seven such thermocouples were installed in the west end of the coil on the flame side. The results were encouraging (Figure 7). The observed temperature profile compares favorably with the predicted profile, and the scatter was eliminated. The slight "error" at the inlet of the coil may be attributed to a legitimate hot zone where radiant heat would be most intense.

During the shutdown, a test section of alternative thermocouples (Figure 8) was installed to compare readings at one location. The ICRC thermocouple design reflects the engineering judgment that, to obtain an accurate reading, the thermocouple junction should be fully immersed in metal and the sheath should have enough surface contact to allow for heat dissipation conducted along the sheath. This was achieved by completely welding a 1/8-in. tube along the longitudinal axis and inserting the thermocouple into this tube (Figure 9). This ICRC thermocouple was tested with solvent-only runs; it gave accurate readings when compared with the predicted profile of Figure 5. Thus, a second shutdown was requested following these solvent-only tests so that a full complement of fifth-generation and ICRC thermocouples could be installed to complete the profiles. A total of 13 ICRC thermocouples (1 per turn) were placed toward the flame in the double-fired zone on the south side of the coil, and 20 fifth-generation thermocouples were installed on the west side--13 toward the flame (1 per turn) and 7 toward the wall (1 per 2 turns).

The ICRC and fifth-generation thermocouples provided all the credible heat-transfer data generated during July/August. Figures 10 and 11 compare respectively the profiles of the ICRC thermocouples (double-fired) and the fifth generation (single-fired) for the B-41R run, which started the second period of operation. (Runs from July/ August that repeat earlier runs from June are designated by an R.) Both runs show essentially identical and credible profiles. These data correspond to a solvent-only run; the predicted heat-transfer coefficients for turbulent flow confirm the measured wall temperatures and support data from complex two-phase and three-phase systems examined in the remainder of the program.

The evolution of tube-wall temperature measurement in the fired heater is summarized in Table 4 (types of installation and position are identified for each generation).

HEAT-TRANSFER SUMMARY

After skin-thermocouple problems were resolved, pilot plant operation returned to the original ICRC fired-heater program. Few base-line runs were repeated, because June results indicated very low pressure drops, typically less than 10 pounds per square inch (psi) for one- and two-phase systems.

Figure 12 shows the temperature profile for Run B-22R, a solvent-plus-gas run. A slight nonlinearity in fluid temperature is observed at turn 9, which is judged to be thermocouple error for this nonreacting system. The 20°F bias suggested by linear regression through surrounding points was also observed in Run B-41R (Figures 10 and 11), a single-phase system. This error will be corrected when the data are analyzed.

Introducing slurry feed produced a readily identifiable peak temperature-difference region, whose magnitude is a function of gas rate. Figure 13 compares temperature profiles for SL-41R, SL-43R, and SL-45R slurry-plus-gas runs at an average slurry rate of 10,960 lb/hr over a 148-1,222 lb/hr range of gas rates. The figure clearly shows a predominant peak at the low gas rate of Run SL-41R. This peak region appears earlier in the coil, suggesting a time/temperature pattern, because gas rate affects slurry-phase residence time. Data shown in

Figure 13 indicate a possible gel region in the 460-630°F zone for the total data range.

Three slurry-only runs examined slurry behavior under heat-transfer conditions in the absence of hydrogen. The results were similar to the slurry-plus-gas runs. The profile for Run SL-73 (Figure 14) shows an immediate peak upon entry into the furnace at a slurry rate of 11,120 lb/hr. Several interesting phenomena were observed. First, the profile suggests two distinct peaks; one between 400 and 580°F and a smaller one between 580 and 660°F. Evidence for this dual-peak phenomenon exists in all three slurry-only runs. Also, correction of the fluid-temperature profile for the 20°F error at turn 9 suggests endothermic behavior previously observed at Wilsonville and in calorimetric experiments.

Unusually high local skin temperatures are usually interpreted as evidence of coke formation. For this reason, the solvent-only runs were repeated at intervals to monitor coke deposition over time. No evidence of coke was observed during the ICRC fired-heater tests.

Two observations are noteworthy. First, after the ICRC program, X-ray examination revealed insignificant quantities (less than 1/16 in.) of coke in the upper turns of the coil on the pipe. However, because X-ray examination is limited to a horizontal view, the pipe curvature obscures the picture and restricts resolution of fine, and perhaps uniform, films of coke. Usually it is impossible to distinguish coke deposition from a residue. Second, experience with preheater B at Ft. Lewis has shown that significant coke deposition (about 1/2 in. over a few months) was limited to the bottom of the pipe in the upper portion of the coil. Because the thermocouples are placed on the sides and top of the tube, it is doubtful that reduced heat transfer would be shown in the temperature profile. An important observation from the fired-heater tests was that, over the 9-week period, no significant quantities of coke were found.

This discussion was limited to identifying trends and interpreting heat-transfer data generated at Ft. Lewis to support the SRC-I Demonstration Plant design. No attempt was made to reduce these results to actual heat-transfer coefficients for correlation purposes; data correlation is in progress and will be presented later.

PRESSURE-DROP SUMMARY

Pressure drop across the fired heater was measured in three ways:

- As the difference between absolute pressure readings
- As the overall pressure drop (differential pressure (dP) cell)
- As the sum of individual pressure drops (dP cells)

After skin thermocouple problems were resolved, an interesting discrepancy was noticed in pressure drops of slurry runs that had to be duplicated. On the average, the differential pressures (dP) in the repeated SL-40 series (SL-40R) at maximum slurry rates were 44% less than those in analogous runs completed in June. Figure 15 compares the pressure drops as a function of total gas through the fired heater of corresponding series (SL-40's, SL-40R's) of slurry runs. An identical trend was observed for the SL-50 and SL-50R series at a nominal 5,500 lb/hr: a 34% mean reduction in pressure drop (Figure 16).

This difference in measured pressure drop was significant enough to warrant entirely repeating eight slurry runs (SL-41 through -45, SL-53 through -55) previously completed in June. Careful review of the data revealed two possible sources for the differences: slurry-mix system residence time and coal particle size distribution.

However, the data show comparable slurry blend tank (SBT) residence times for both the original and repeat series:

Run series	Slurry blend tank residence time (min)
SL-40's	12.9
SL-40R's	12.8
SL-50's	25.0
SL-50R's	26.9

Even if significant numerical differences existed between the corresponding series, evidence in a special high-temperature, slurry-mix study (see the following article in this report) does not support residence time as a significant factor in fired-heater pressure drop.

For a 38.5 wt % coal slurry, a modest 12% pressure-drop increase was observed over the range of 3-13 min SBT residence time, after which no increase was evident up to 23 min.

However, coal particle size distribution presents a significant difference. During June, erratic control of the coal pulverizer resulted in a fine grind considerably different from the demonstration plant specification of 70% through 200-mesh coal feed, whereas the repeat runs of July/August were characterized by consistent, specification coal grind (see Figure 17). Additional evidence that slurry viscosity increases with the fineness of particle size distribution suggests that coal particle distribution was responsible for the pressure-drop differences.

Another interesting phenomenon displayed in Figures 15 and 16 is the presence of a pressure-drop minimum as a function of gas rate. Although readily apparent in the SL-40 series, the function is less obvious in the SL-40R and SL-50R series and is not discernible in the incomplete SL-50 series. Interest in this phenomenon prompted the execution of a mini-series (m) of runs in an attempt to further define its characteristics. Pressure drops of Runs SL-46 (full repeat of SL-44R) and SL-41m, SL-42m, and SL-43m are also included in Figure 15 (dotted line). These data were generated over a discrete time period when all other variables were held constant except the gas rate (i.e., day-to-day random variations in coal grind, recycle solvent, etc., were minimized).

The mini-runs showed a very smooth transition through a minimum at 340 lb/hr as the gas rate was adjusted for SL-41m conditions. Most of the data indicate that the precise location of the minimum depends upon the sum of all effects induced by other parameters such as coal grind, residence time, and solvent/coal ratio.

Figure 18 provides another view of the pressure-drop data of the SL-40R (maximum slurry rates) and SL-50R series (50% turndown rates). The two curves are essentially parallel, reflecting an average pressure-drop reduction of 34% at half-rates. Also included in this figure are the results of the SL-60 series spanning a range of 5,500-10,900 lb/hr slurry for a fixed nominal gas rate of 300 lb/hr. Except for one anomalous point, the trend is generally as expected: pressure drop increases as slurry rate increases. The small difference in pressure drop between 5,530 lb/hr (SL-52R) and 6,860 lb/hr (SL-62) slurry may be scatter.

Another interesting pressure-drop phenomenon was seen with the slurry-only runs (i.e., no gas) of the SL-70 series at slurry rates of 5,500, 8,000, and 10,900 lb/hr. Pressure drops corresponding to each slurry rate are plotted on the ordinate axis of Figure 18. Extrapolations to zero gas rate at the two higher slurry rates of this series are consistent with the other data and support the concept of a pressure-drop minimum. However, at the lowest slurry rate a discontinuity appears; the pressure drop falls from 38 to 30 psid for a corresponding reduction in gas rate from 132 to 9 lb/hr, respectively. Though the magnitude of these changes is not great, careful treatment of the data is warranted due to limited understanding of flow-related phenomena. Discussion of this will be continued in the section on Residence Time.

For a final look at pressure-drop trends, the overall pressure drop was plotted as a function of slurry rate at gas rates of 0 and 300 lb/hr (SL-70 and SL-60 series, respectively) (Figure 19). The data parallel results shown in Figure 18: a lower pressure drop is observed at the higher gas rate at all points except the lowest slurry rate.

Individual and overall pressure-drop profiles revealed differences among various series of runs. For example, the individual profiles for Runs SL-43 and SL-43R show that the earlier run (SL-43) exhibited a peak pressure-drop region upstream of that identified with the later run (Figure 20); the difference can be attributed to coal particle size distribution. This trend, consistent with the conclusion regarding the magnitude of the overall pressure drop discussed earlier, is displayed in Table 5, which compares the magnitudes and positions of the peak pressure drops of corresponding runs during June and July/August. Two exceptions occur at the highest gas rates of the SL-45, -45R and SL-55, -55R pairs; the more uniform profile may be attributed to the dominating influence of gas rate as well as slurry-related phenomena.

For the July/August slurry runs, the peak pressure drop tended to occur further upstream as the gas rate increased (Figures 21 and 22). However, for the fixed-gas, variable-slurry runs of the SL-60 series (Figure 23), a uniform peak pressure-drop region was identified at turn 10 in all cases. Comparing temperature profiles with pressure-drop profiles shows that the peak temperature-difference region precedes the peak pressure-drop region in all slurry runs.

FIRED-HEATER RESIDENCE TIME

On August 12, 1980, a series of fired-heater radioactive tracer tests was conducted to determine gas- and slurry-phase residence times in the fired heater by injecting gas and liquid radioactive materials upstream and monitoring the time-delayed response downstream. Twelve experiments were conducted covering a range of gas and slurry rates consistent with those of the fired-heater program.

The experimental procedure involved lining out the plant at known rates for each injection. For the gas-phase injection, argon-41 (1.8 hr half-life) was injected into a sample point several feet upstream of the inlet to the fired heater by placing one to three vials of the material in a vessel connected in series to 3,600 psi N_2. About 0.25 L of inert gas and tracer was fed to the system through this inlet. For the slurry-phase injection, the procedure was identical, except that 1-bromophenanthrene (36-hr half-life) was the liquid tracer. A detector mounted 1.5 ft upstream of the fired heater signaled the injection, while a detector 33 ft downstream of the fired heater outlet indicated the response.

Because of the difference in diameter between the 2-in. coil and the 3-in. transfer line (both Schedule 160), all computed residence times were adjusted volumetrically. All values in this report reflect an 85.4% correction to the measured time to address residence time exclusively in the fired heater (the 2 ft of upstream piping is negligible). Also, it is assumed that the slurry phase is indeed a single-phase system; i.e., the solids and liquid travel at the same velocity.

Injection and response signals were fed to a digital data storage device connected to a plotter. A typical plot is shown in Figure 24. The peak on the right indicates the point of injection; the peak on the left shows the response. The distance between peaks determines the residence time between detectors for a chart speed of 20 cm/sec. Though the distance between peaks can be determined in many ways based on the geometry of the signals, our analytical method assumes an instantaneous injection followed by the response signal for which the following distances are defined:

- Miniumum--based upon initial response
- Mean--based upon centroid
- Maximum--based upon final detection of signal

Table 6 summarizes the results of the 12 radioactive tracer tests. Computed slurry- and gas-phase residence times reflect the correction of the mean values (distance between injection signal and centroid of response curve) to give the actual fired-heater residence time. During this series of experiments, an unplanned development proved helpful in confirming data. When injection changed from liquid to gas, residual liquid tracer in the injection port produced a second peak, displaced in time and identical with the peak previously generated. Data in Table 6 show that secondary (residual) liquid injection residence times in tests RT-3, -5, and -7 respectively correspond to times for the planned liquid injections in tests RT-4, -6, and -8. Thus, this "method" simultaneously determined residence times for two phases.

Residence times of the slurry phase were plotted against the total gas rate, for slurry rates of 5,540, 9,070, and 10,960 lb/hr, respectively (Figures 25-27). Each graph clearly shows that, as the gas rate increases, the residence time of the slurry phase decreases, or the linear velocity of the slurry increases. On the average, the data show that as the gas rate doubled the slurry residence time decreased 26%. Each plot also shows the boundaries of the computed residence times determined by the maxima, minima, and means defined in Figure 24.

A composite of the mean values from Figures 25-27 (Figure 28) shows that, as the slurry rate increases for a given gas rate, the residence time decreases. Extrapolation of these slurry-phase data suggests that at very high gas rates the slurry-phase residence time approaches the gas-phase residence time; that is, the slurry phase is entrained by the gas phase at very high gas-to-slurry volumetric ratios and travels at the same velocity.

However, at very low gas rates, evidence of nonlinear behavior exists, which would correspond to flow regime transitions. Figure 28 includes calculated residence times for the slurry-only runs (no gas present). These values were computed using a mean slurry density of 60 lb/ft^3 over a 350-760°F temperature rise in the fired heater. Linear extrapolation of the observed data does not predict the residence time if the coil cross-sectional area is occupied only by slurry.

Results presented in Figure 28 also suggest that the gas-phase residence time curve is independent of slurry rate for the rates examined in these tests. A single line has been drawn through all three pairs of data points to show this. At very high volumetric ratios, the gas-phase residence time would probably be a function of the gas rate only. However, at very low rates we infer that the gas bubbles would travel at the velocity of the slurry and have the same residence time. If so, the composite gas-phase curve of Figure 28 would diverge to each of the slurry-only residence times shown in the abscissa as plotted in Figure 29. Again, a flow regime transition is apparently a function of gas-to-slurry ratio.

This possibility was addressed by plotting the gas-phase data using gas rate as a parameter (Figure 30). The gas-phase residence time appears to be a very constant function of slurry rate, which is why the points overlapped in Figure 28. However, extrapolation of the observed data to calculated values without slurry flow gives rise to a situation analogous to the all-slurry, no-gas conditions just discussed. A transition is again evident; as the gas volume decreases and slurry increases, the residence time will decrease correspondingly. The apparent insensitivity of gas-phase residence time to slurry rate should be fully reconciled when employing correlations using slip/no slip conditions.

For the range of conditions examined in the radioactive tracer tests, the following conclusions have been reached:

1. Slurry-phase residence time varied between 1.2 and 2.2 min.
2. Gas-phase residence time varied between 0.5 and 0.9 min.
3. Slurry residence time is a function of both slurry and gas rates.
4. Gas-phase residence time is only weakly dependent upon the slurry rate.
5. Extrapolation of results of both phases to high gas rates is consistent with expected trends.
6. Extrapolation of the results from both phases to low gas rates is not linear. This would be consistent with transitions between flow regimes at low gas rates.
7. To compare residence-time results with specific fired-heater runs, the following chart is useful:

Run no.	Slurry rate (lb/hr)	Associated tests
RT-1 through RT-4	9,070	HT series
RT-5 through RT-8	10,960	SL-40's
RT-9 through RT-12	5,540	SL-50's

CONCLUSIONS

The ICRC fired-heater test program conducted at Ft. Lewis in 1980 provided a comprehensive data base for the design of the demonstration plant fired heater. Some preliminary conclusions derive from these tests:

1. Solvent-only base-line tests provided a very strong foundation to assess the accuracy of the instrumentation. Following the shutdowns to improve tube-wall temperature measurement techniques, these runs were repeated periodically throughout the program as a means of calibration and coke determination. We are highly confident in the accuracy of the data obtained.

2. Slurry runs were completed over a broad range of slurry and gas rates in a matrix of experiments. Trends in both heat-transfer characteristics and pressure-drop phenomena clearly point toward time/temperature and shear rate dependence.

3. Residence-time studies in the fired heater show clearly that slurry-phase residence time depends upon gas rate.

4. No coking was observed in this test series. This is consistent with the assumption that maintaining inside wall temperature below 850°F minimizes coke formation.

5. Pressure drop increases as the coal particle size decreases.

This report thoroughly documents the experimental program by reviewing its history and evaluating trends in the data. No attempt has been made to reduce and correlate the data.

This program was completed in direct support of the demonstration plant design effort. Future work, which includes an experimental program on a 1 1/2-in.-diameter coil at Ft. Lewis, will be conducted to verify the design and to expand the data base.

REFERENCES

Ackerman, C. D. 1979. Coal liquefaction preheater studies presented at Department of Energy Project Review Meeting, Oak Ridge, Tenn., by Pittsburgh & Midway Coal Mining Co., Ft. Lewis, Wash. March 21, 1979.

Thew, T. W. 1983. High-temperature slurry mix test. Following article in this report.

Table 1

ICRC Fired Heater Test Program Experimental Summary

		No. of experiments	
Name	Type of experiment	June	July/August
Base line	Solvent only	1	6
	Solvent plus gas	14	4
Slurry	Slurry only	0	3
	Slurry plus gas	8	18
Total		23	31

Table 2

Experimental Run Identification[a]

Run sequence	Type of experiment	Conditions
B-11 to B-15	Solvent plus gas	Variable gas rate, fixed full solvent rate, isothermal
B-21 to B-25	Solvent plus gas	Variable gas rate, fixed full solvent rate, heated
B-31 to B-35	Solvent plus gas	Variable gas rate, fixed half-solvent rate, heated
B-41 to B-45	Solvent only	Variable solvent rate, heated
SL-41 to SL-46	Slurry plus gas	Variable gas rate, fixed full slurry rate, heated
SL-51 to SL-55	Slurry plus gas	Variable gas rate, fixed half-slurry rate, heated
SL-61 to SL-65	Slurry plus gas	Variable slurry rate, fixed gas rate, heated
SL-71 to SL-73	Slurry only	Variable slurry rate, heated

[a] Several runs of the July/August period repeated those of the June period and are designated by an R in the label (e.g., SL-42R).

Table 3

General Conditions for Fired Heater Program

Slurry Blend Tank temperature	350°F
Coal	
Type	Kentucky #9, #14
Nominal grind	70% -200 mesh
Ash	10.5%
Solvent boiling range	450-850°F
Hydrogen-rich gas purity	86 mol %
Coal concentration	38.5 wt %
Fired Heater outlet temperature	760°F
Dissolver	
Temperature	825°F
Pressure	2,000 psig

Table 4

Summary of Thermocouple Generations

Generation	Date	No. of thermocouples	Type of installation	Position
1	November 1979	13	Sheath attached to pad; pad welded to tube wall and then insulated	West end, toward flame, 1 per turn
2	February 1980	13	Sheath embedded in half-tube tack-welded to tube wall along 3-in. length	4 clusters of 4 mounted circumferentially, turns 7 and 11, east and north sides (16); 1 on turn 11, west end; 1 at outlet on north side
3	March 1980	3	Sheath embedded in weld material	1 at outlet on north side; 1 at turn 7 on north side; 1 at turn 11 on west side
4	April 1980	34	Sheath inserted vertically into tube that has been welded to tube wall	20 on west side (13 toward flame; 7 toward wall); 14 on north side on alternate turns (7 toward flame, 7 on top of tube)
5	July 1980	20	Sheath inserted vertically into tube; tube is truncated at weld and annulus was filled with silver solder	20 on west side (13 toward flame, 1 per turn; 7 toward wall, alternate turns)
ICRC	July 1980	13	Sheath embedded in 1/8-in. tube fully welded to tube wall along 6-in. length	13 on south side, 1 per turn, toward flame

Table 5

Peak Pressure-Drop Comparison

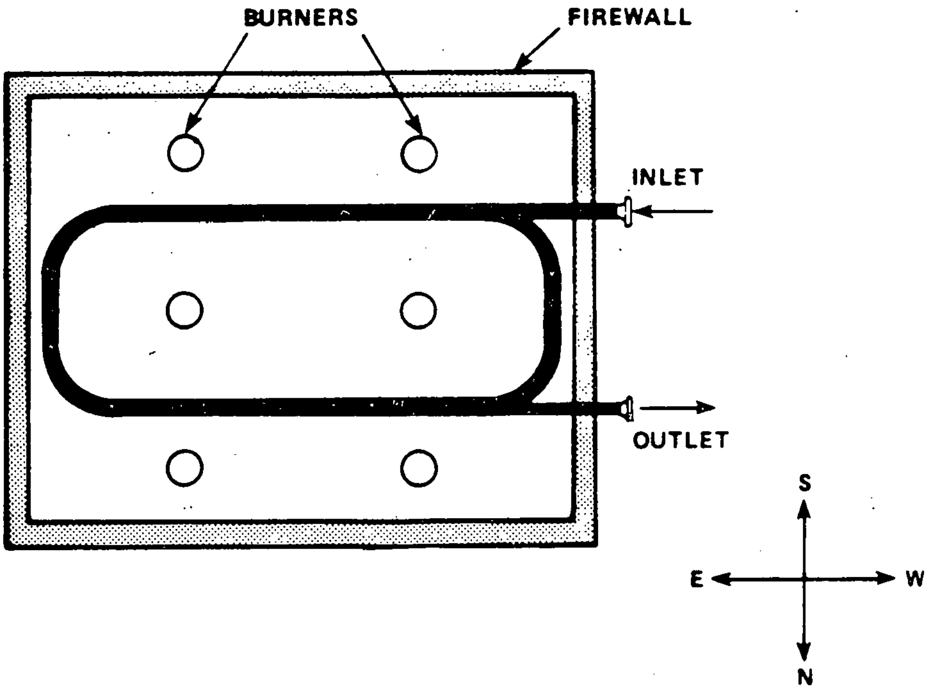
Run no.	Peak delta P (psid)	Coil turn no.
SL-41	41	8
SL-41R	21	10
SL-42	29	6
SL-42R	12	10
SL-43	27	6
SL-43R	16	8
SL-44	23	6
SL-44R	14	8
SL-45	23	10
SL-45R	18	6
SL-53	19	8
SL-53R	11	10
SL-54	14	8
SL-54R	11	10
SL-55	25	Outlet
SL-55R	12	Outlet

Table 6

Results of Radioactive Tracer Tests

Test no.	Gas rate (lb/hr)	Slurry rate (lb/hr)	Type of injection	Mean residence time (min)
RT-1	227	9,080	Gas	0.88
RT-2	224	9,070	Liquid	1.83
RT-3	574	9,050	Gas Liquid	0.57 1.23
RT-4	569	9,070	Liquid	1.24
RT-5	537	10,960	Gas Liquid	0.57 1.20
RT-6	539	10,960	Liquid	1.19
RT-7	269	10,970	Gas Liquid	0.77 1.49
RT-8	270	10,960	Liquid	1.50
RT-9	264	5,600	Liquid	2.18
RT-10	266	5,550	Gas	0.79
RT-11	532	5,490	Gas	0.51
RT-12	534	5,530	Liquid	1.62

Figure 1
Fired Heater, Top View



SOURCE:

PITTSBURGH & MIDWAY COAL MINING CO., FT. LEWIS, WASH., 1980.

Figure 2
Fired Heater Coil Isometric

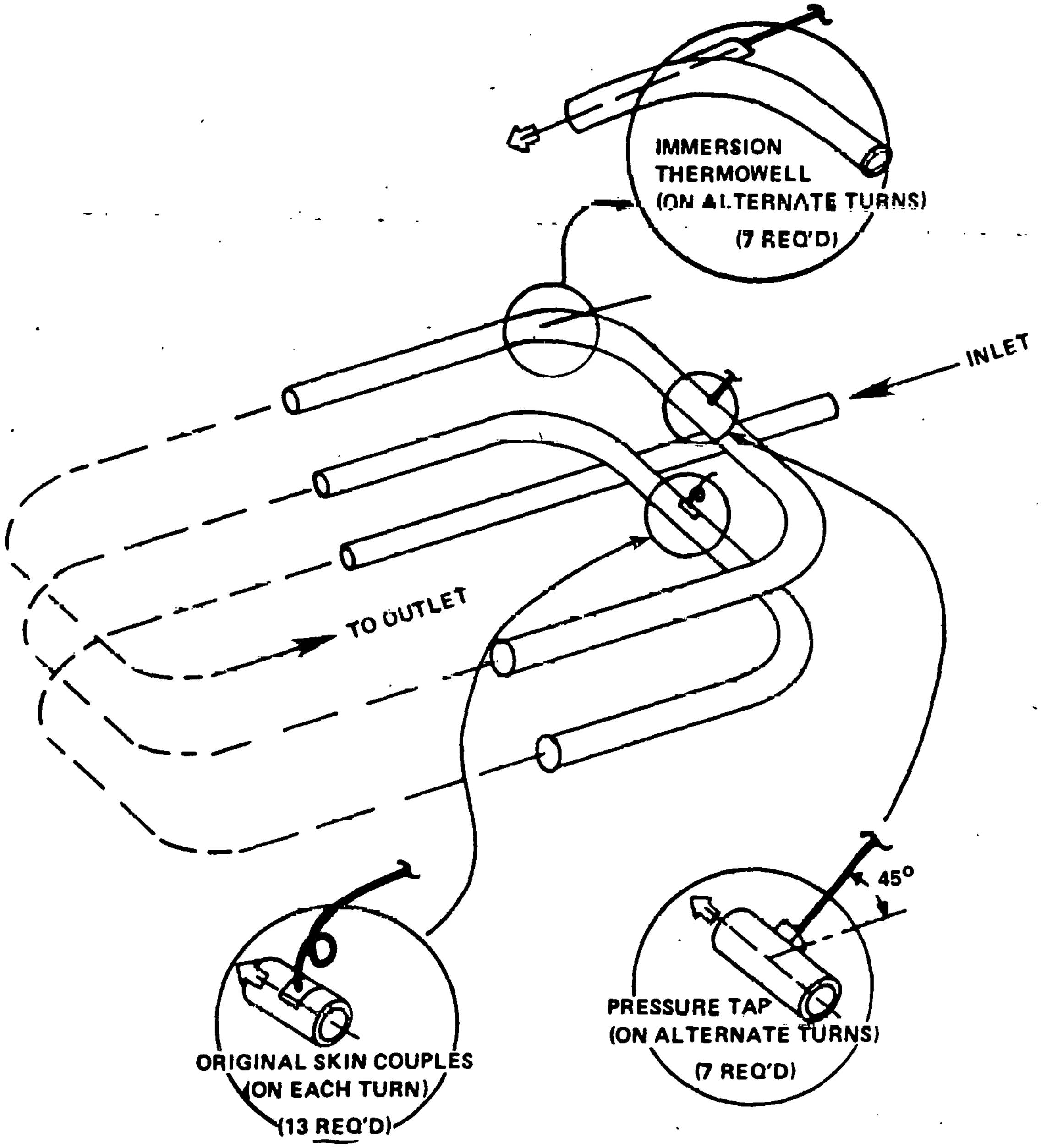


Figure 4
Original Solvent-Only Run Temperature Profile
Run B-41
(June 7, 1980)

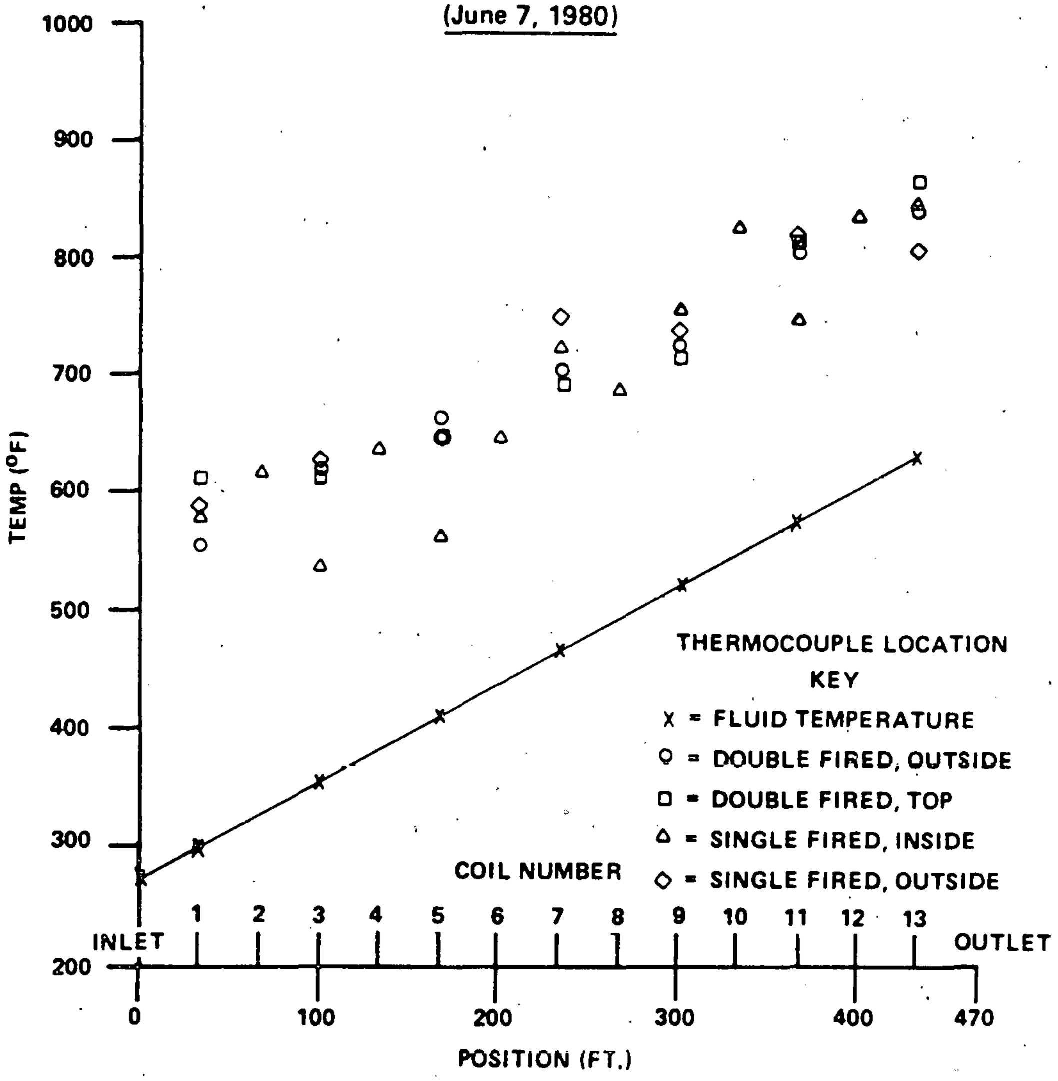
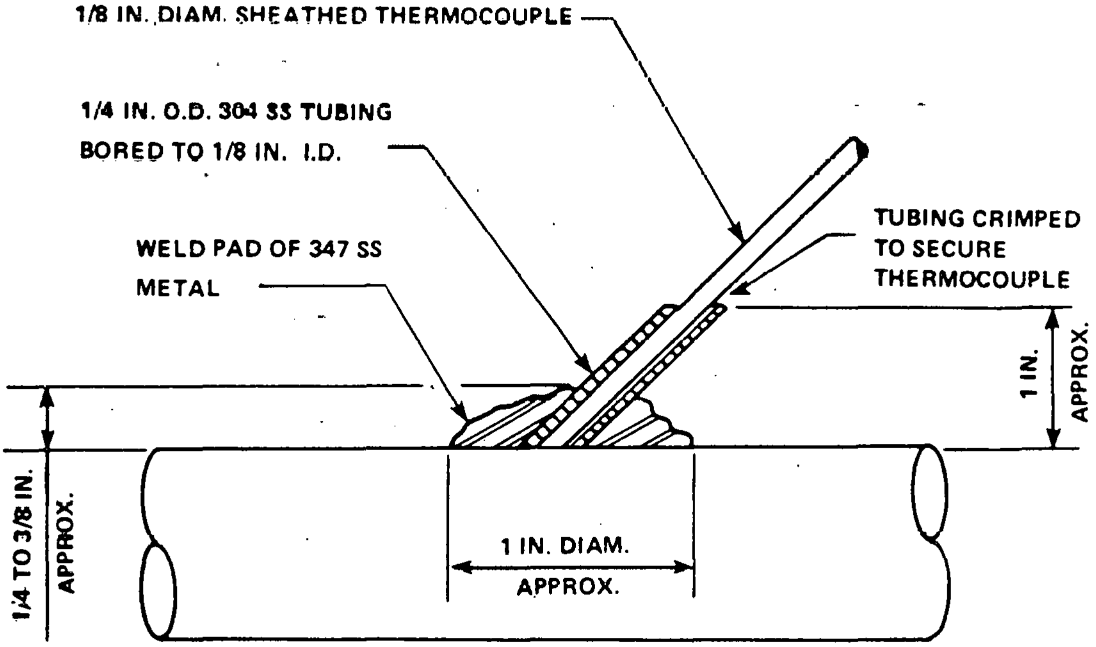


Figure 3
Fourth-Generation Method of Installation



NO.	ZONE	POSITION
13	SINGLE-FIRED	FLAME SIDE (INSIDE)
7	SINGLE-FIRED	WALL SIDE (OUTSIDE)
7	DOUBLE-FIRED	FLAME SIDE (INSIDE)
7	DOUBLE-FIRED	TOP
34		

SOURCE:

DIMENSIONS OBTAINED FROM S.L. O'TOOLE, PITTSBURGH & MIDWAY COAL MINING CO., FT. LEWIS, WASH., 1980.

Figure 5
Predicted Temperature Profile
for Solvent-Only Run B-41

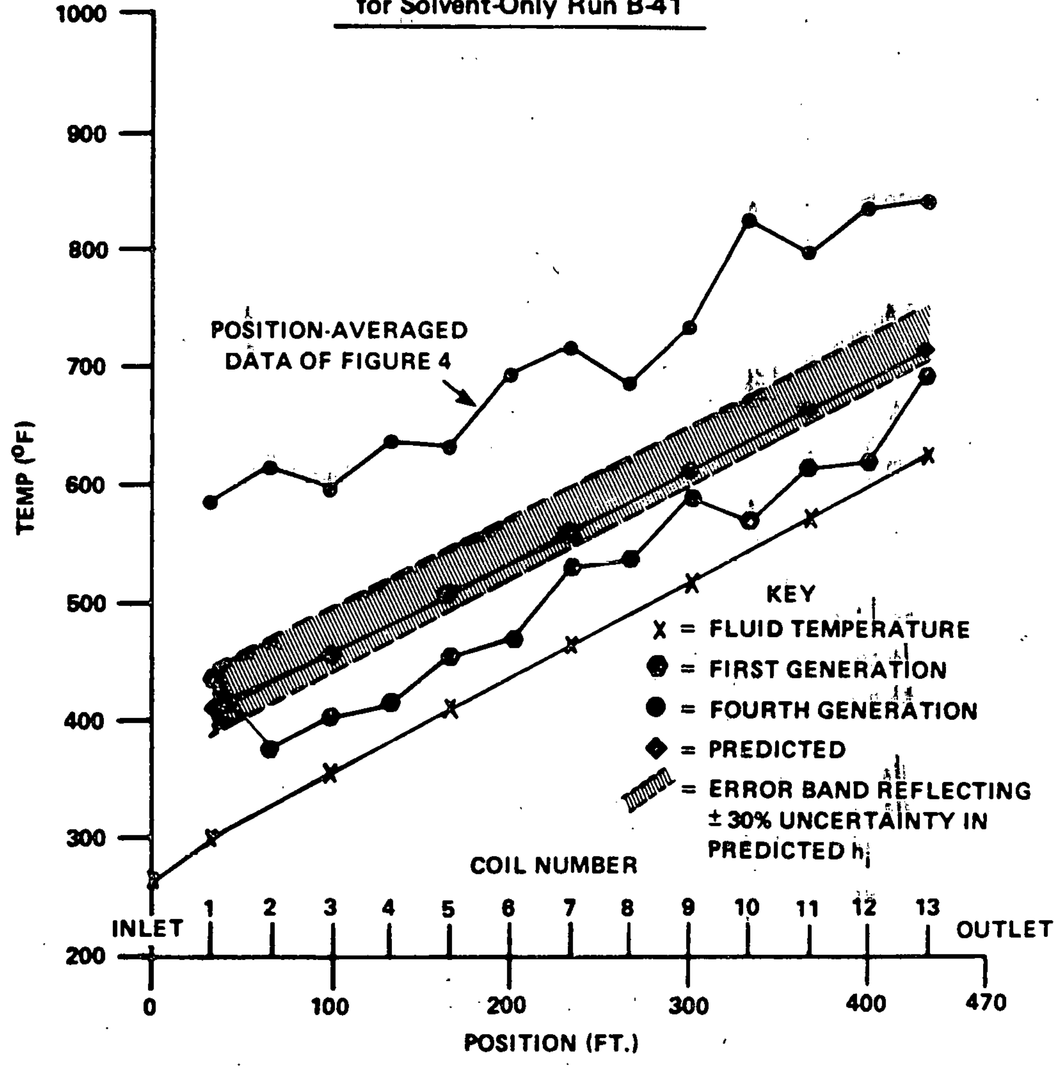
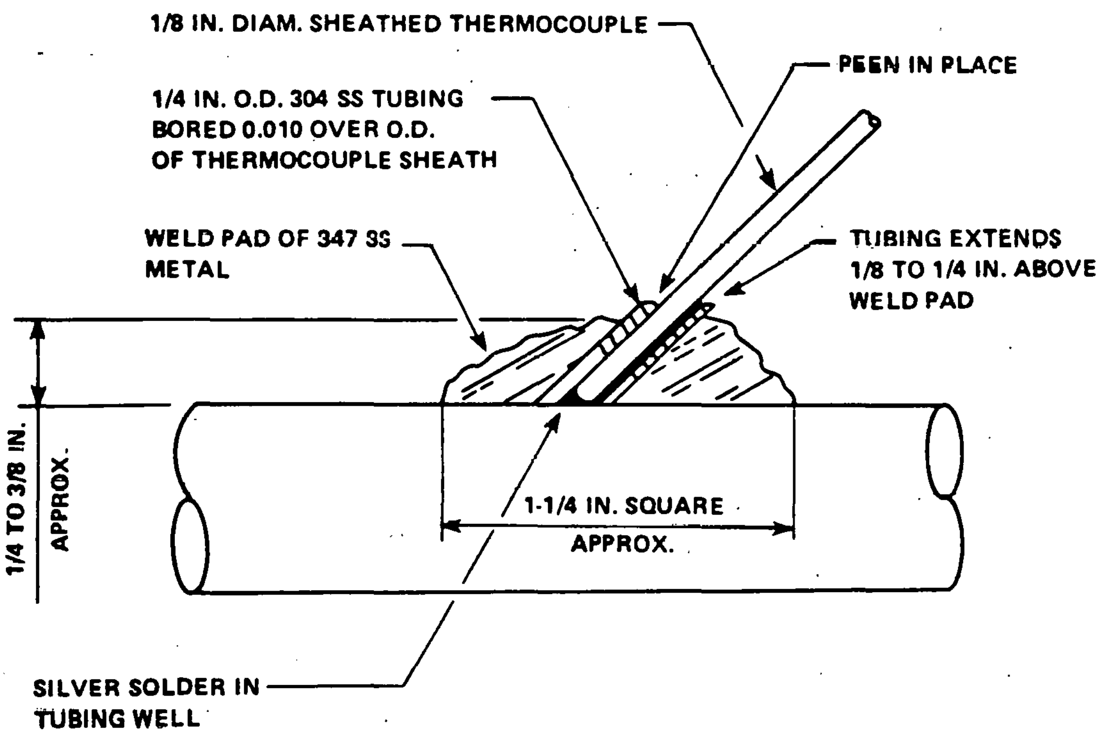


Figure 6
Fifth-Generation Method of Installation



SOURCE:

DIMENSIONS OBTAINED FROM S.L. O'TOOLE, PITTSBURGH & MIDWAY COAL MINING CO., FT. LEWIS, WASH., 1980.

Figure 7
Observed Temperature Profile
for Solvent-Only Run B-42
(July 10, 1980)

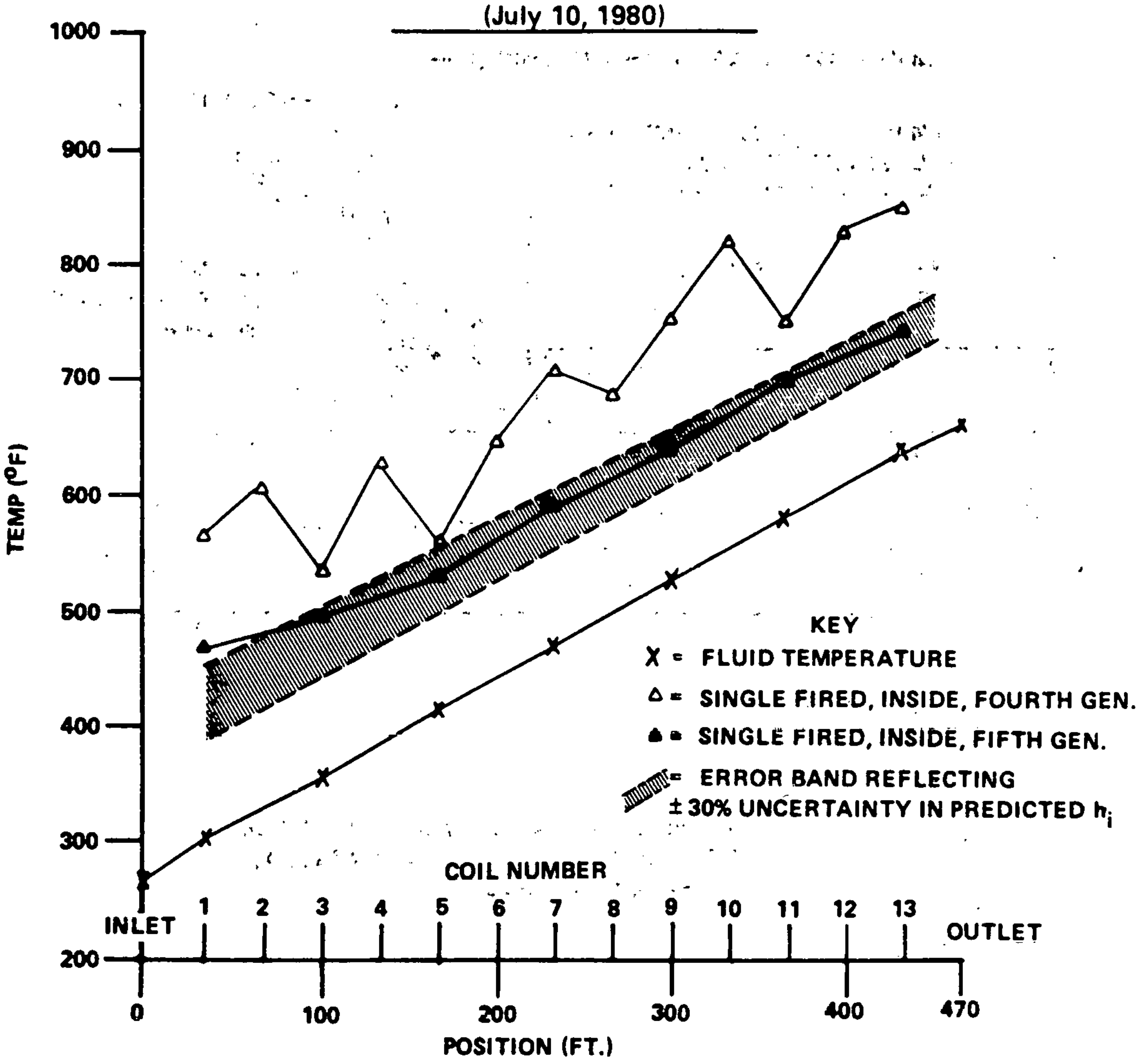
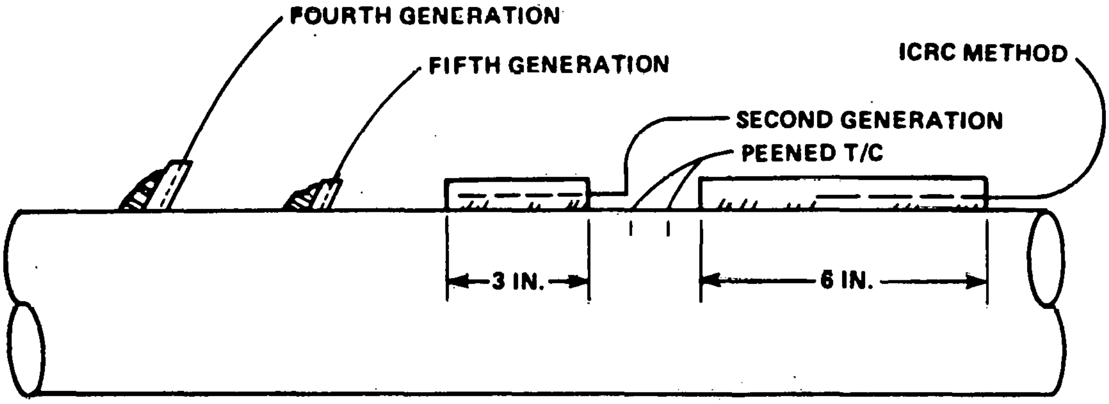


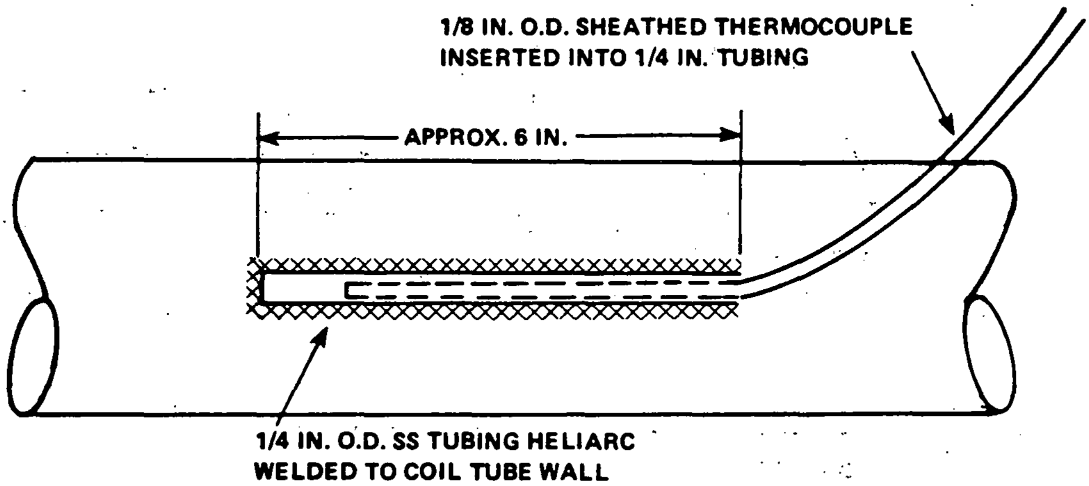
Figure 8
Thermocouple Test Section



SOURCE:

DIMENSIONS OBTAINED FROM S.L. O'TOOLE,
PITTSBURGH & MIDWAY COAL MINING CO.,
FT. LEWIS, WASH., 1980.

Figure 9
ICRC Method of Attachment



SOURCE:
DIMENSIONS OBTAINED FROM S.L. O'TOOLE,
PITTSBURGH & MIDWAY COAL MINING CO.,
FT. LEWIS, WASH., 1980.

Figure 10
Observed Temperature Profile
for Solvent-Only Run B-41R
(July 23, 1980)

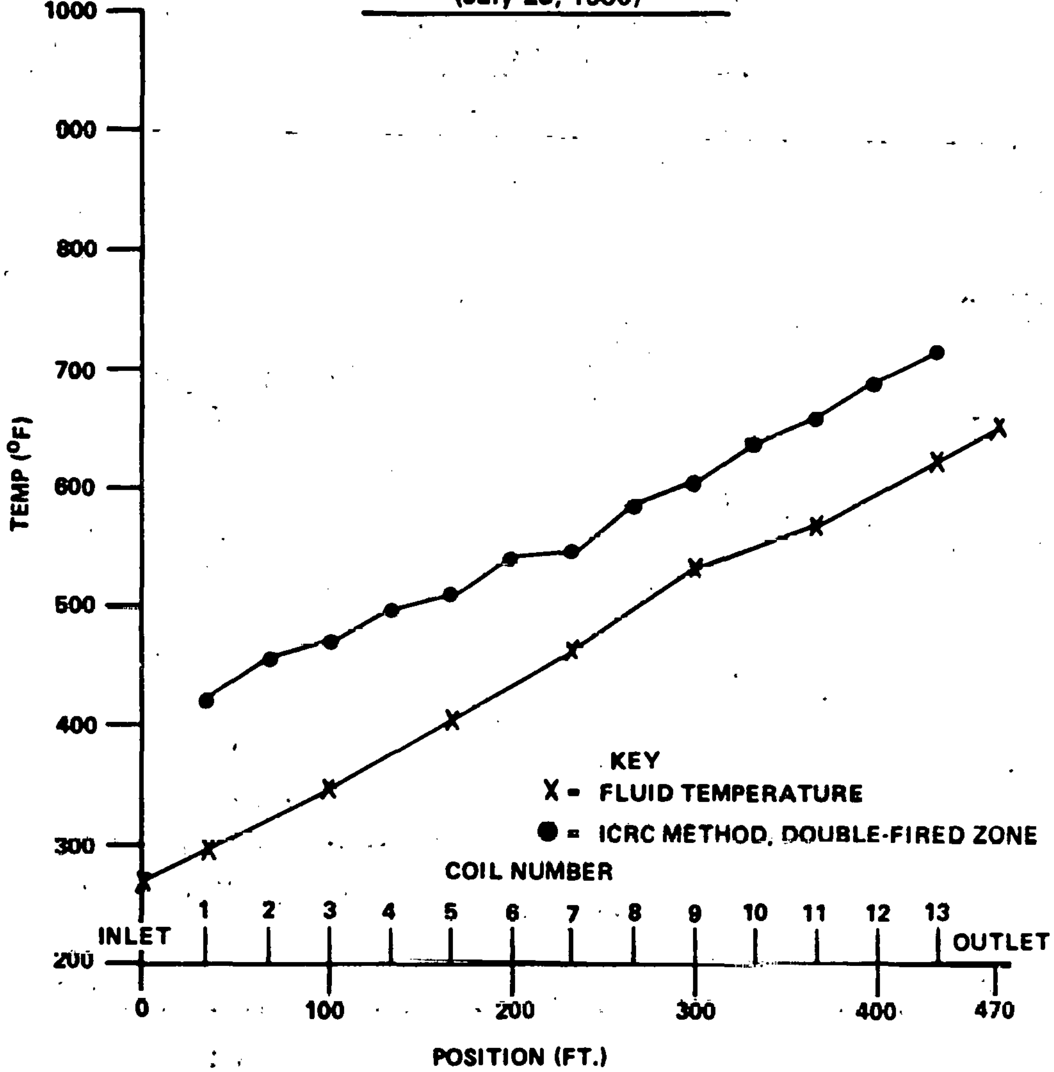


Figure 11
Observed Temperature Profile
for Solvent-Only Run B-41R
(July 23, 1980)

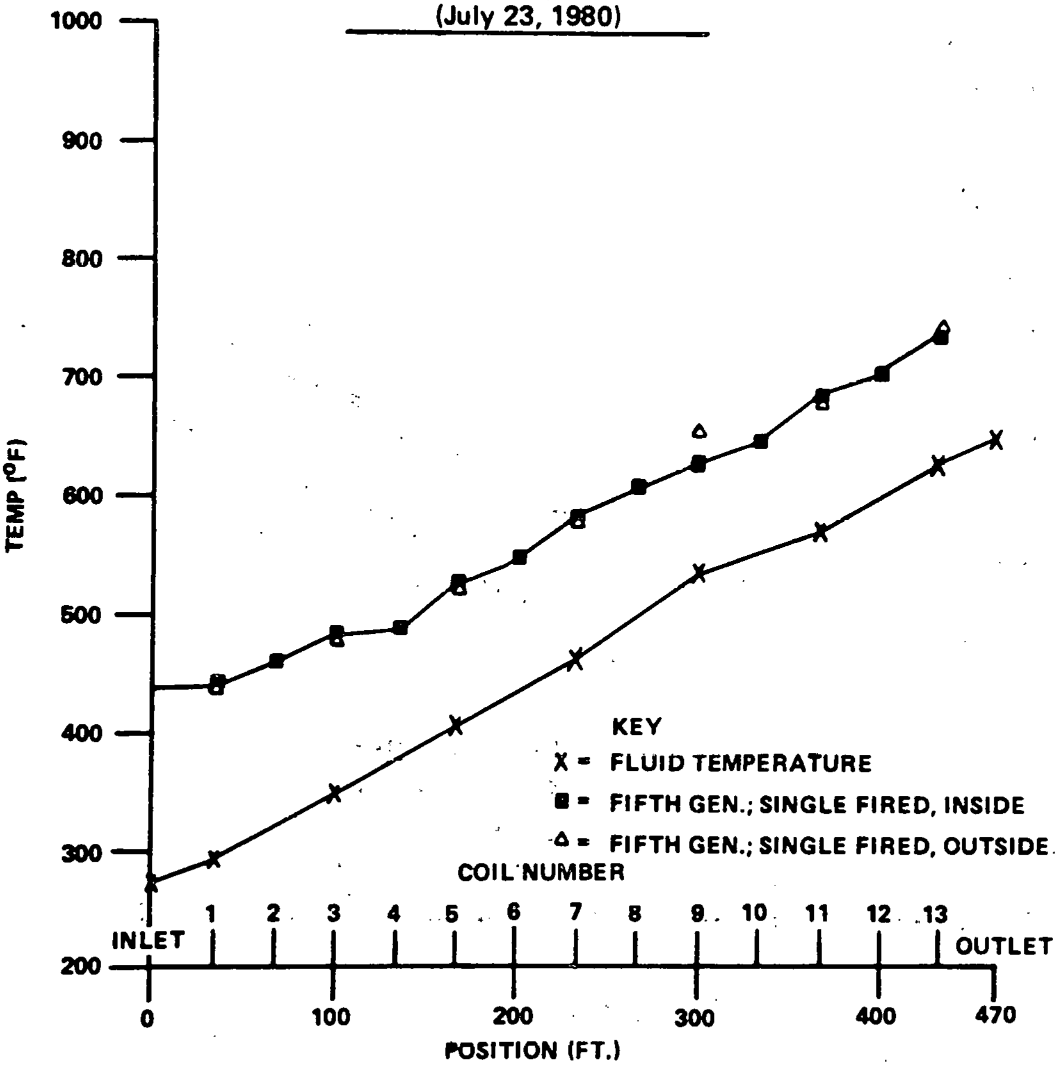


Figure 12
Observed Temperature Profile
for Solvent-Plus-Gas Run B-22R
(July 24, 1980)

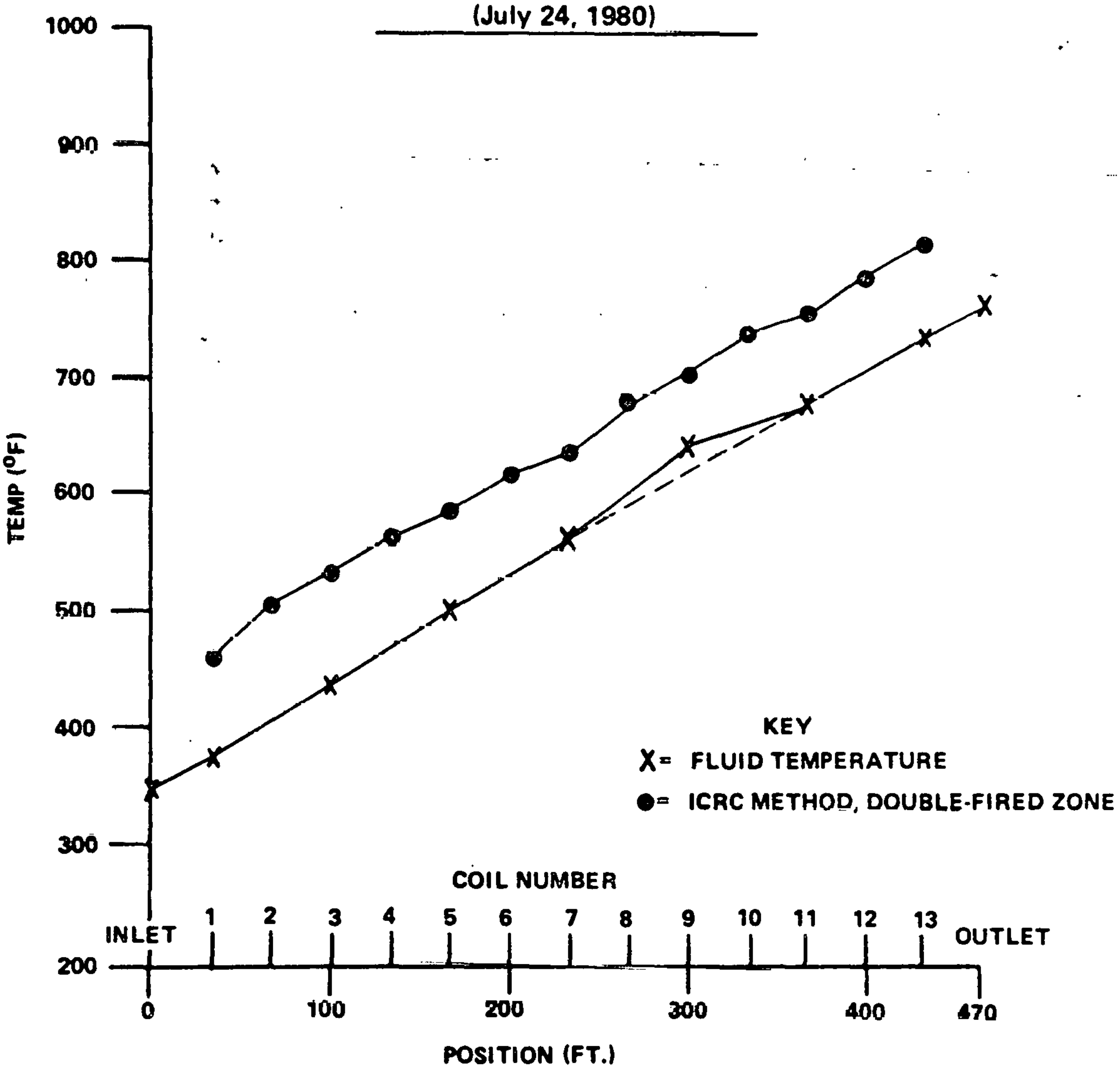


Figure 13
Observed Temperature Profiles of
Slurry-Plus-Gas Runs
SL-41R, SL-43R, and SL-45R

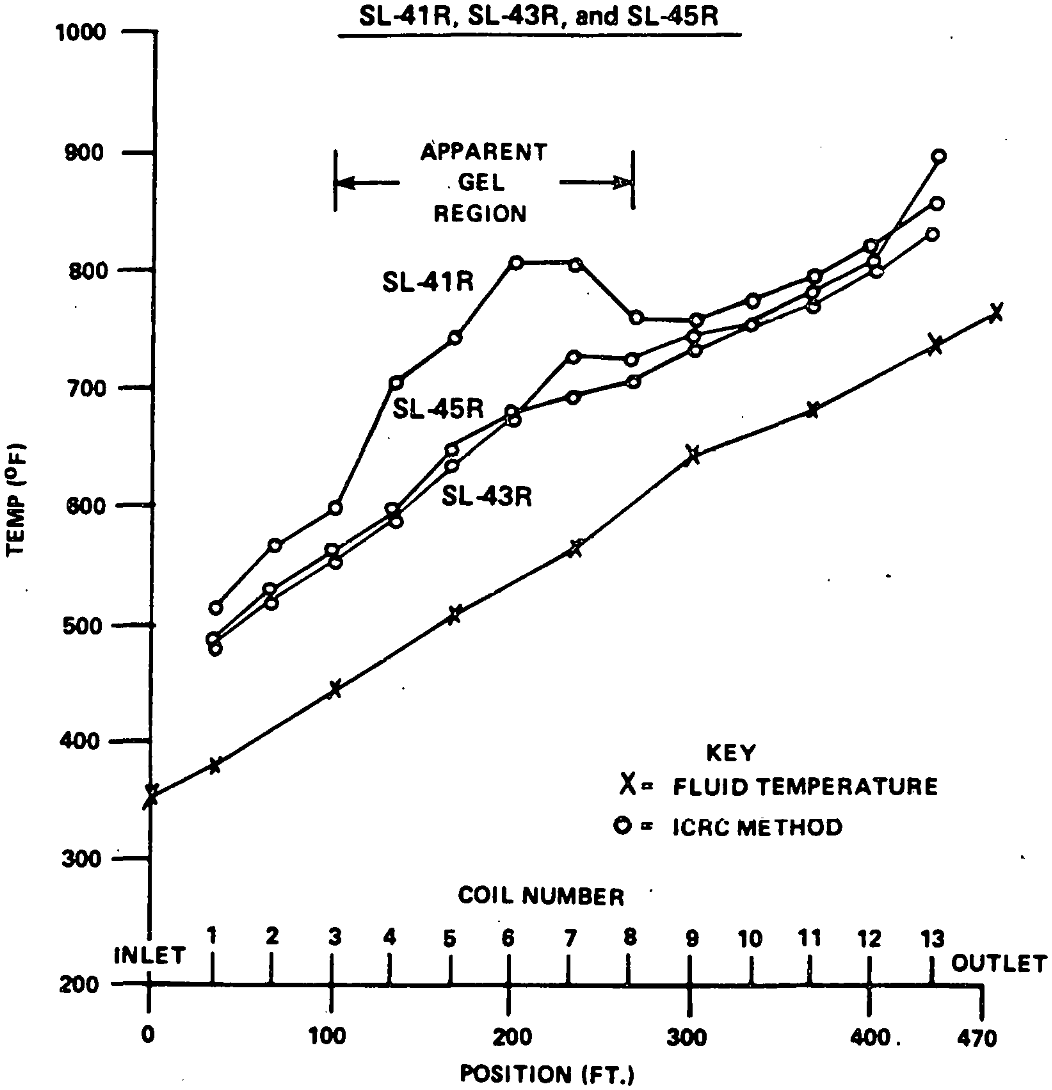


Figure 14
Observed Temperature Profile
for Slurry-Only Run SL-73
(August 6, 1980)

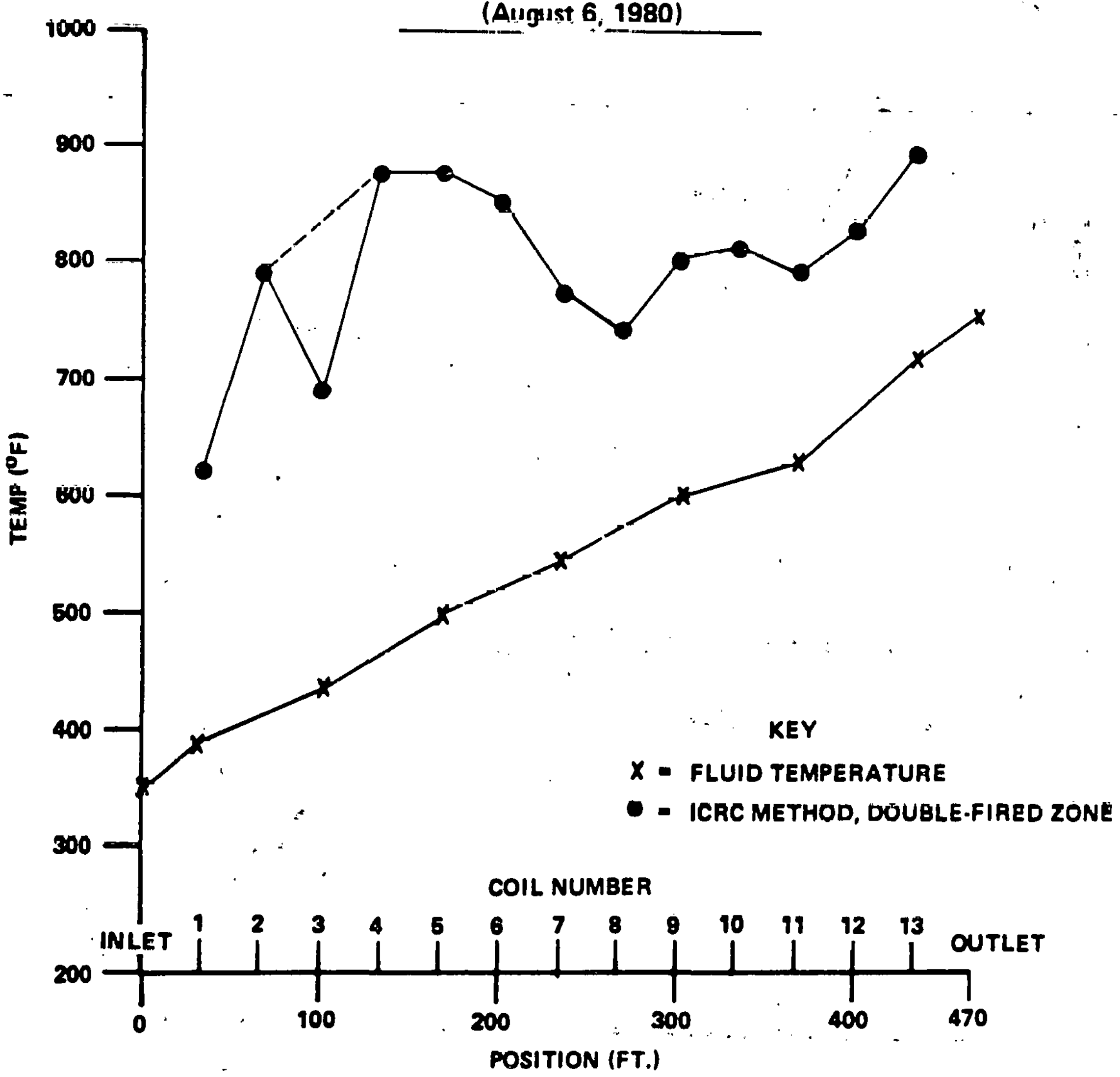


Figure 15
Comparison of Overall Pressure Drops at Maximum Slurry Rates
(SL-40, SL-40R Series)

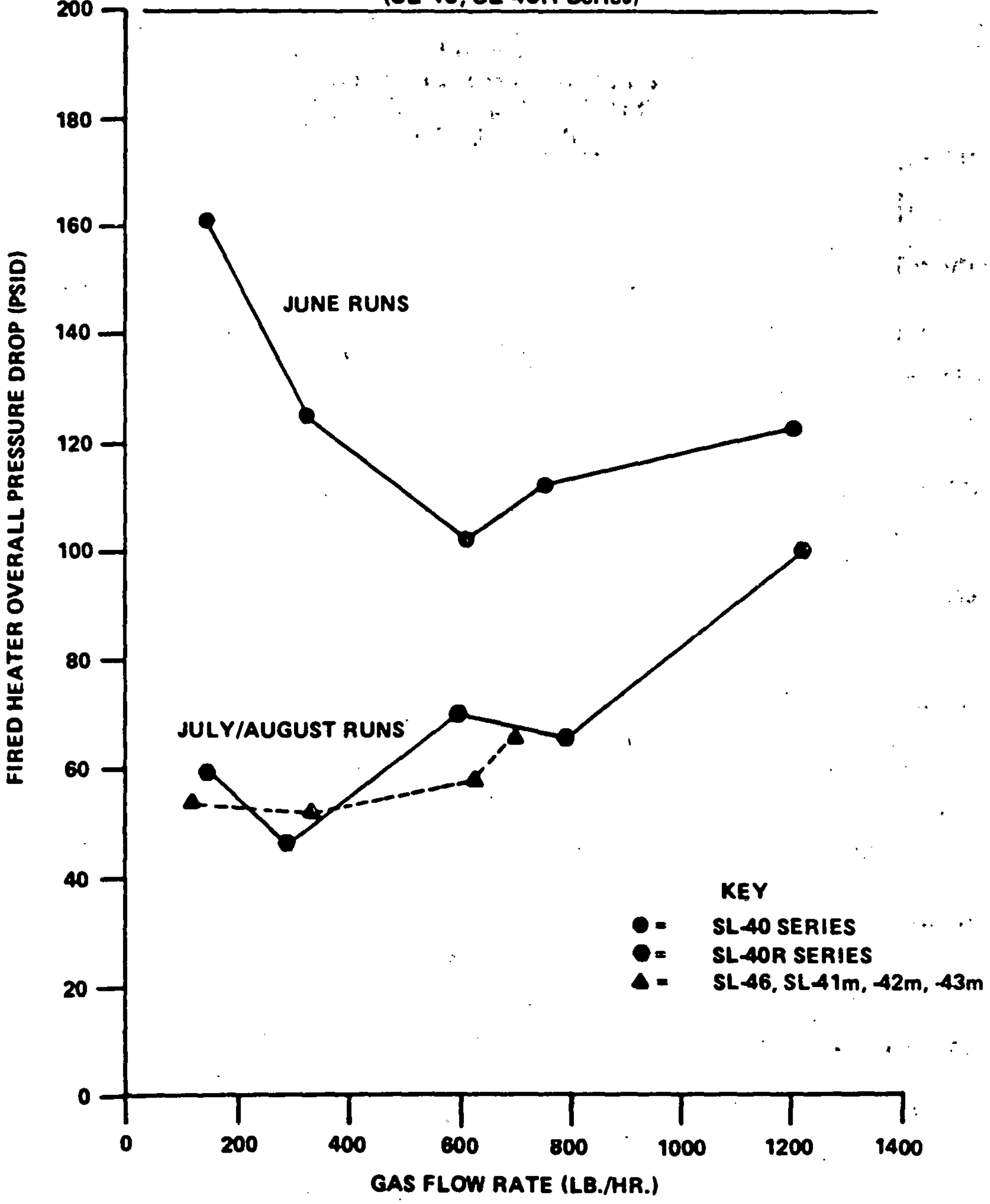


Figure 16
Comparison of Overall Pressure Drops at 50% Turndown Rates

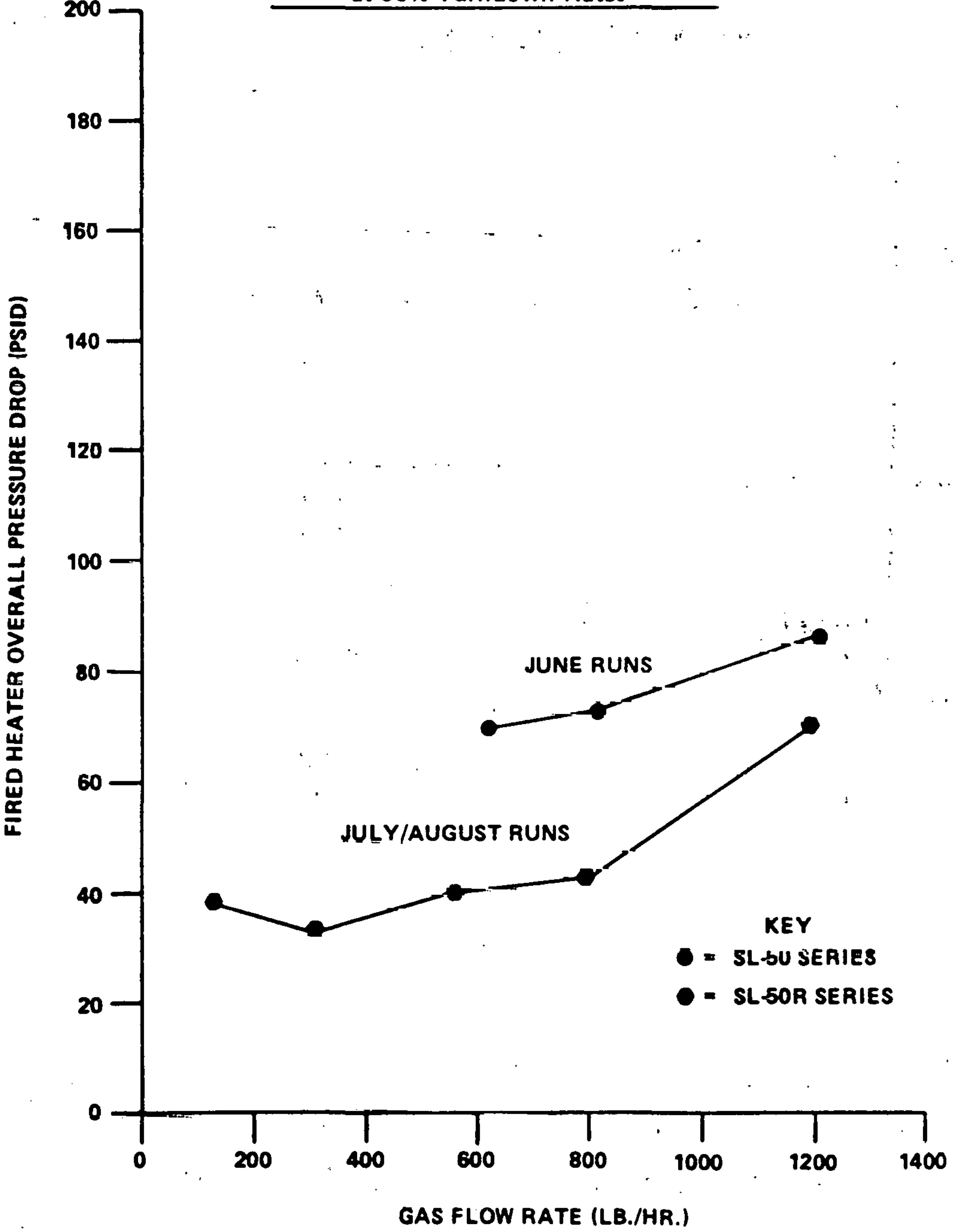


Figure 17
Nominal Coal Grind Comparison

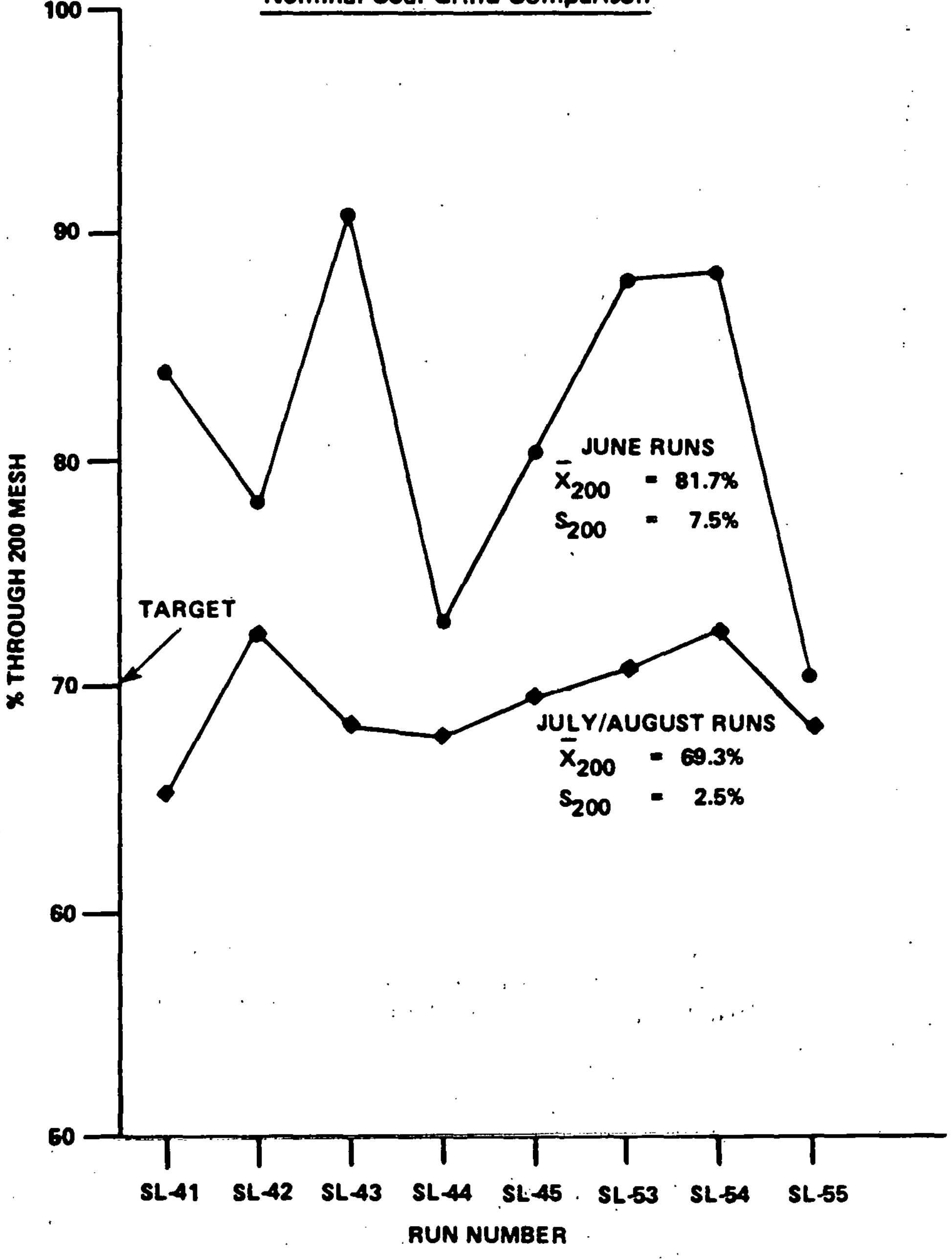


Figure 18
Overall Pressure Drop of Slurry Runs Completed in July/August Period

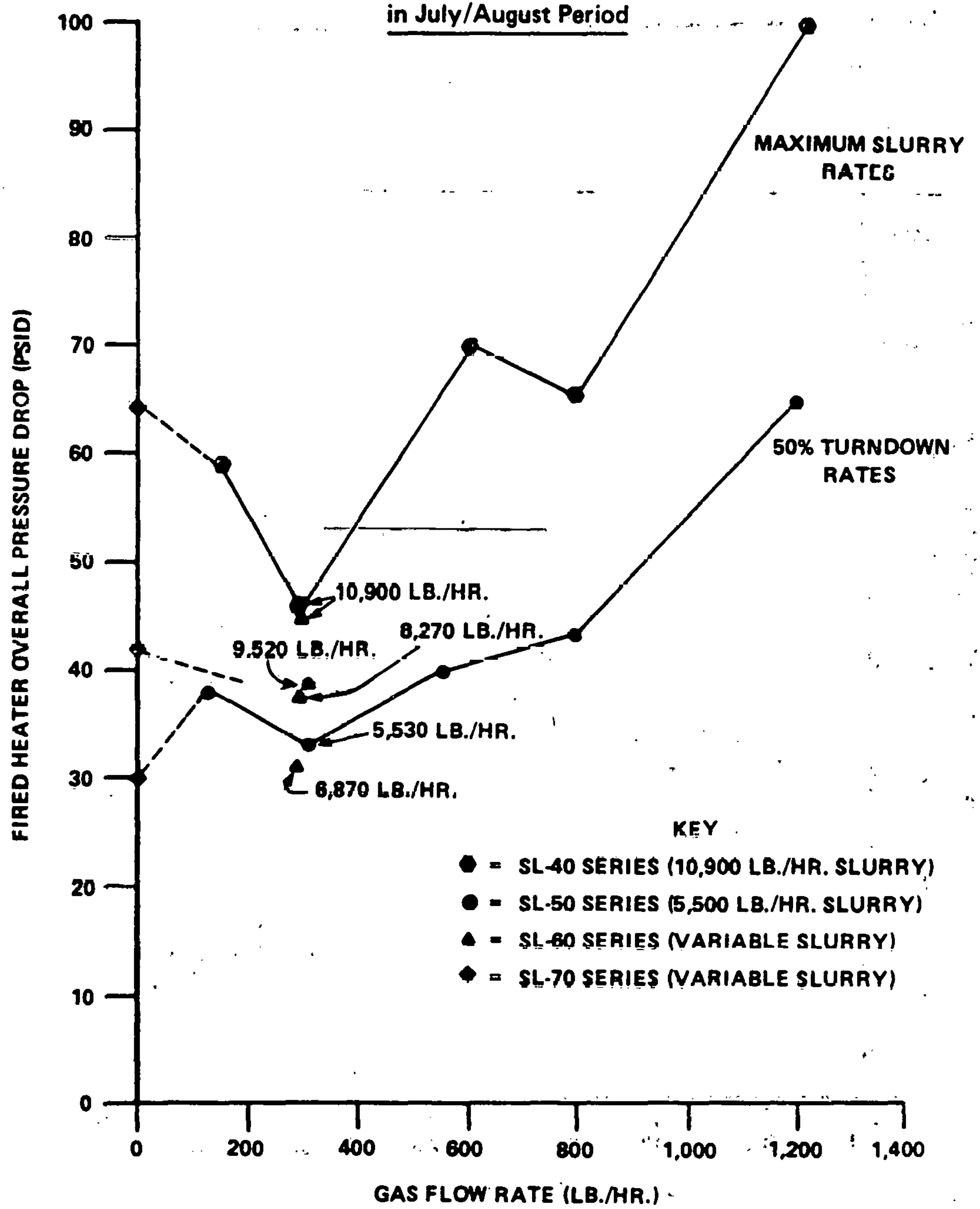


Figure 19
Fired Heater Overall Pressure Drop as Function of Slurry Rate

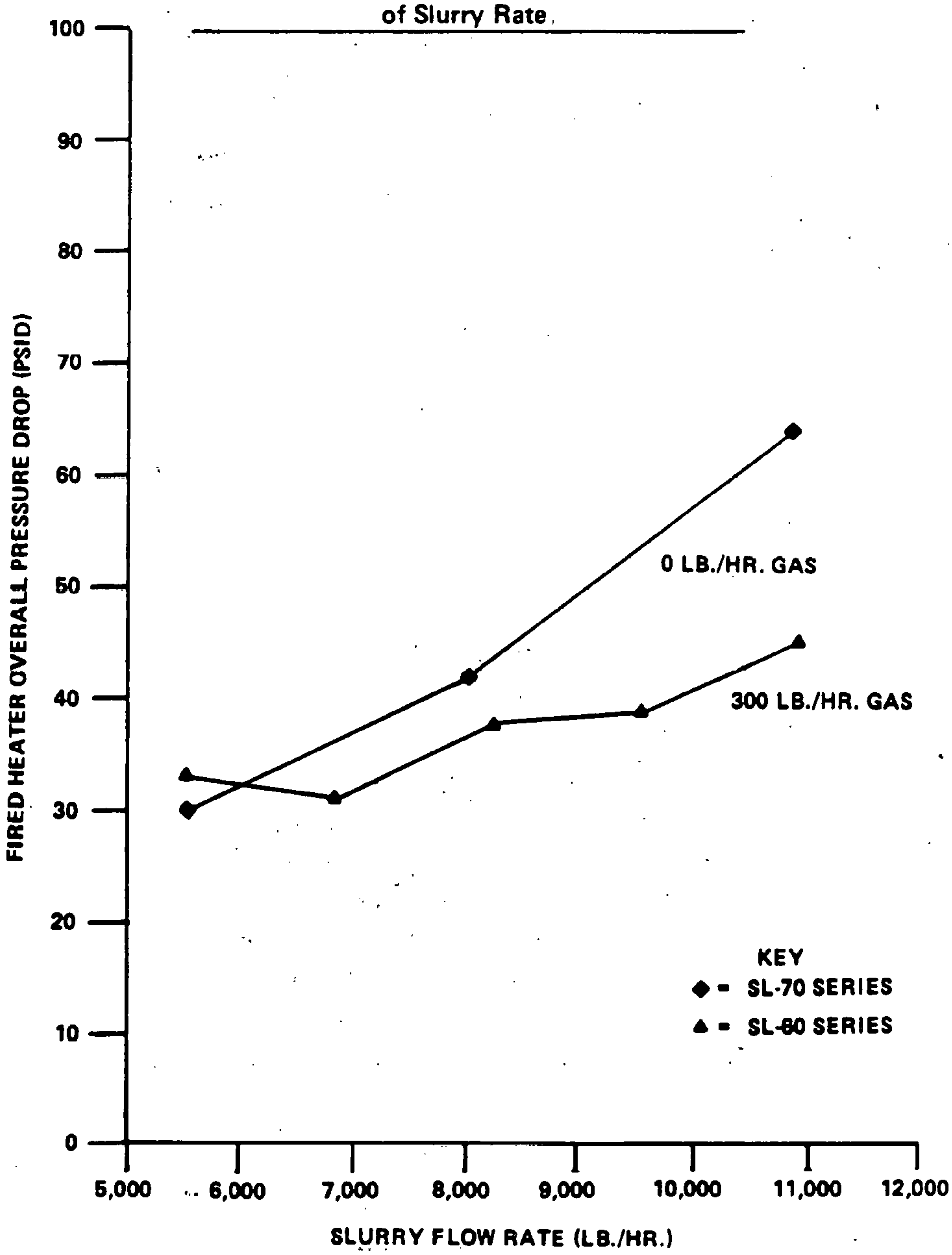


Figure 20
Pressure-Drop Profile Comparison of Runs SL-43 and SL-43R

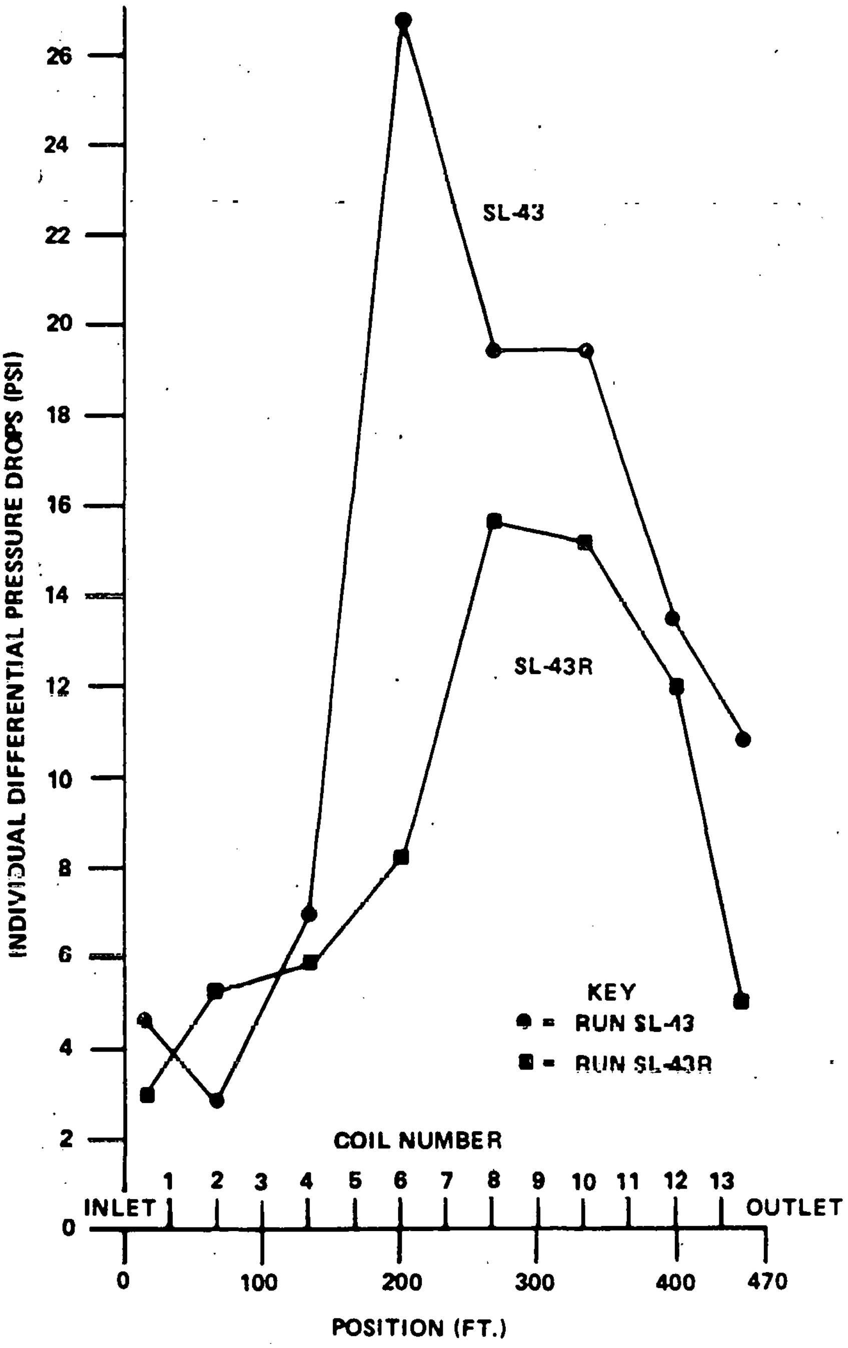
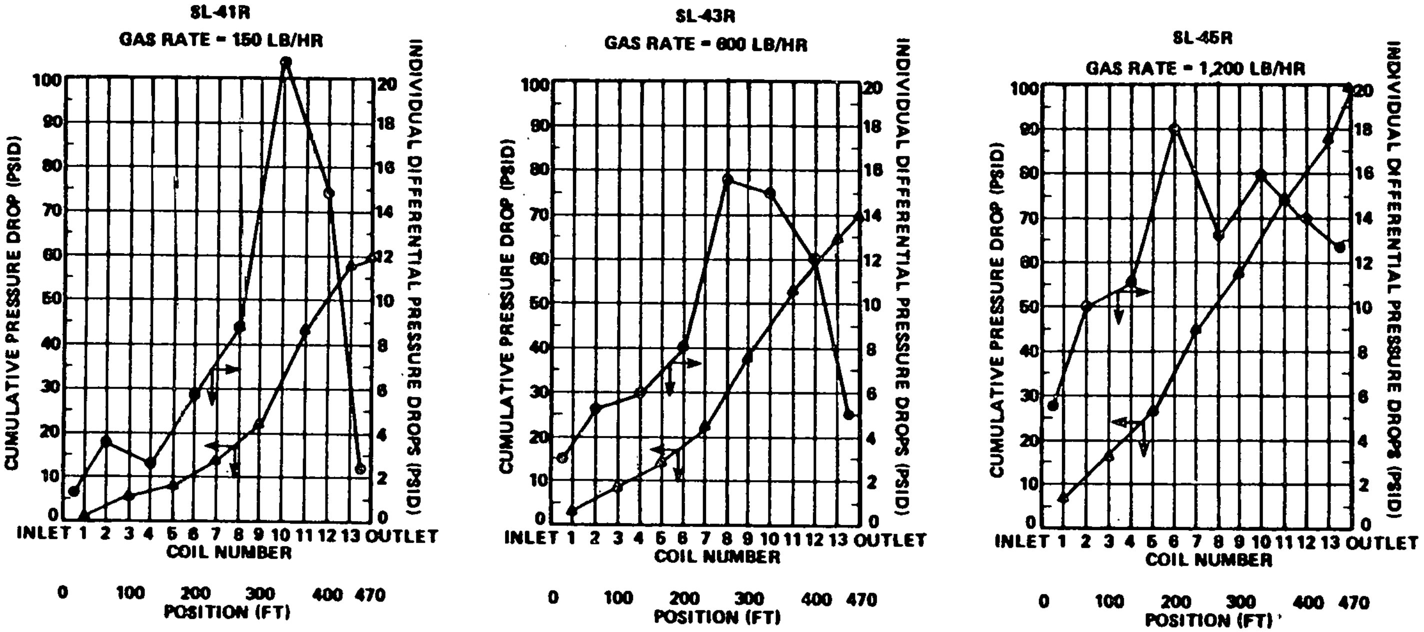


Figure 21
Comparison of Overall Pressure-Drop Profiles
at Maximum Slurry Rates



● – INDIVIDUAL DIFFERENTIAL PRESSURE CELL MEASUREMENT

▲ – CUMULATIVE PRESSURE DROP

Figure 22
Comparison of Overall Pressure-Drop Profiles
at 50% Turndown Rates

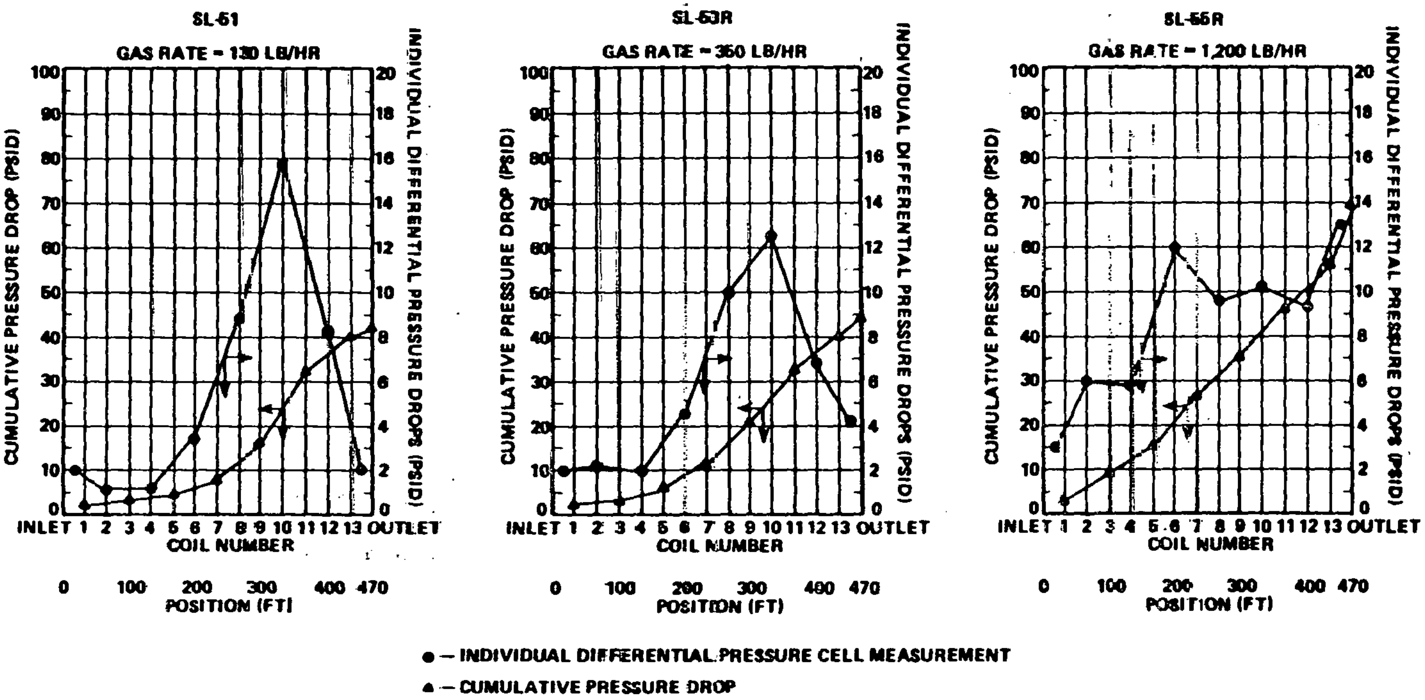


Figure 23a
Comparison of Pressure-Drop Profiles
for Variable Slurry Rates

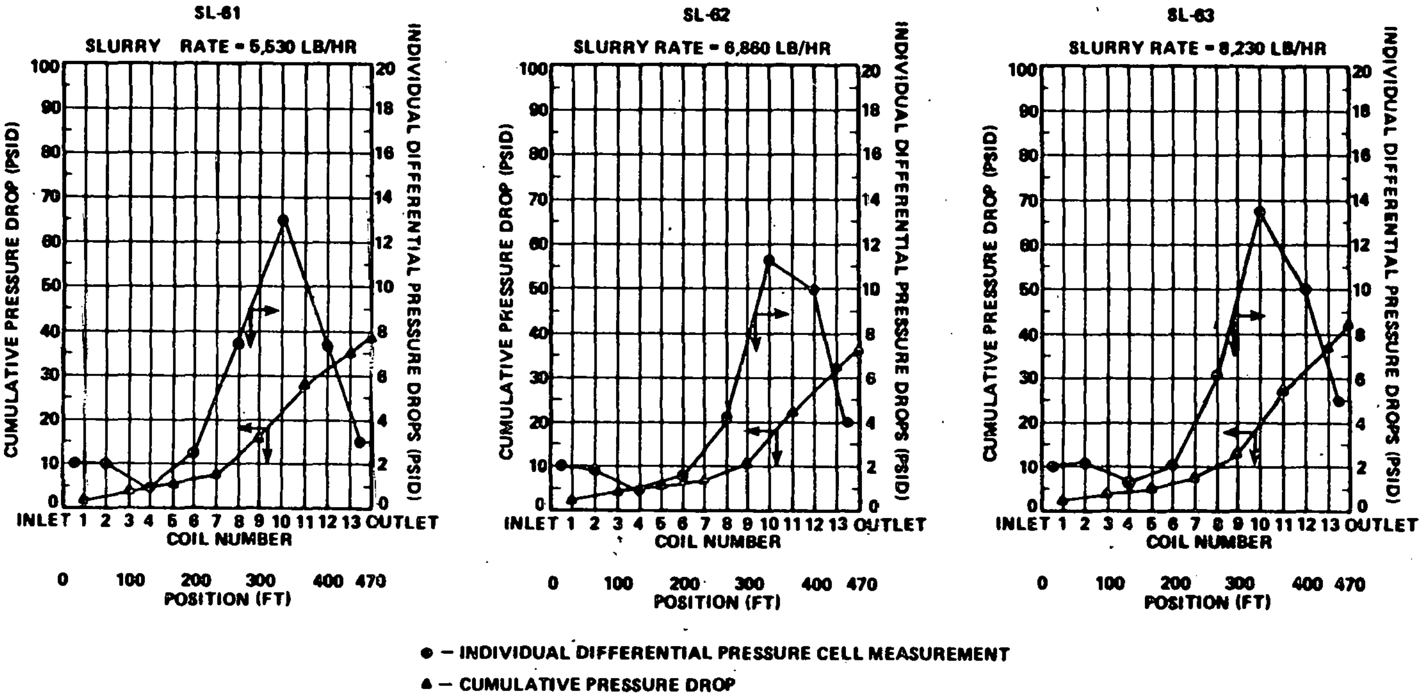


Figure 23b
Comparison of Pressure-Drop Profile
for Variable Slurry Rate

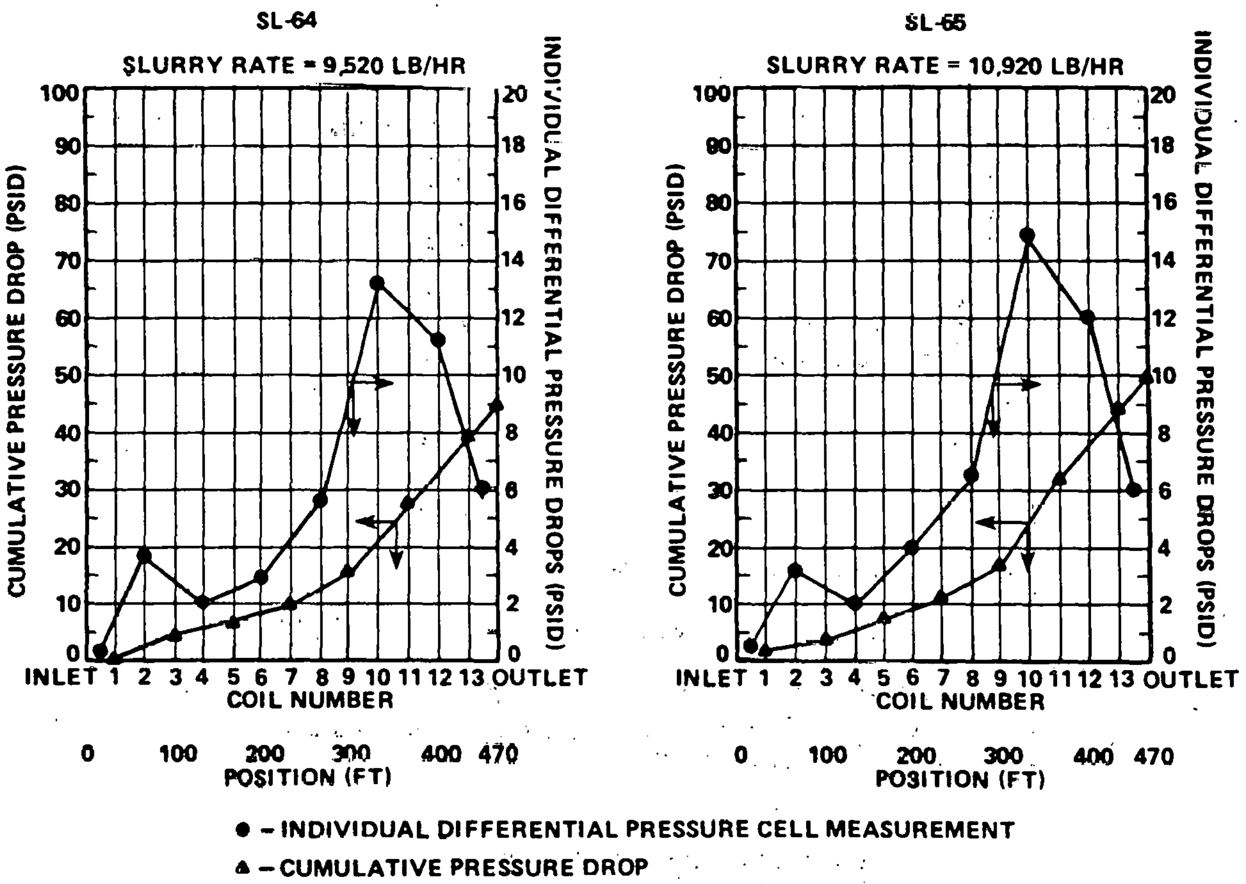
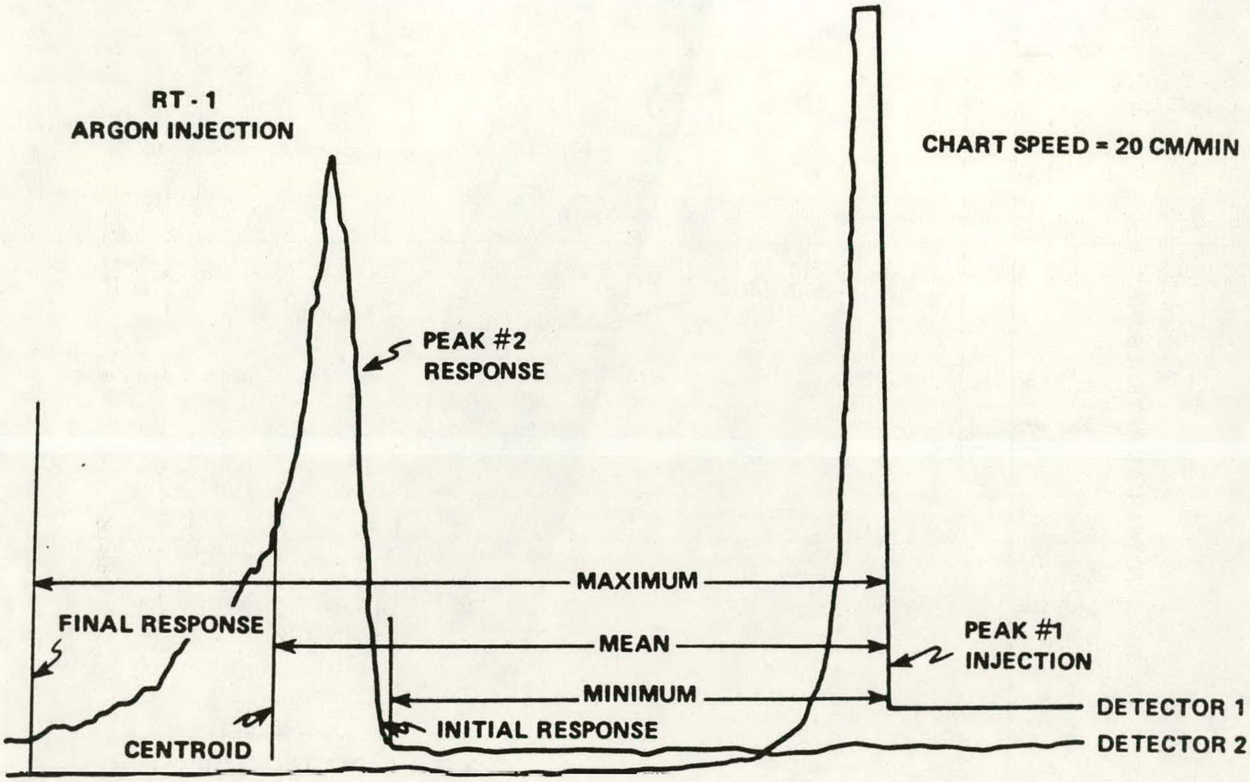


Figure 24
Representative Radioactive Tracer Plot



SOURCE:

OBTAINED FROM C. D. ACKERMAN,
PITTSBURGH & MIDWAY COAL MINING CO.,
FT. LEWIS, WASH., 1980.

Figure 25
Fired Heater Residence Time
50% Turndown Rates

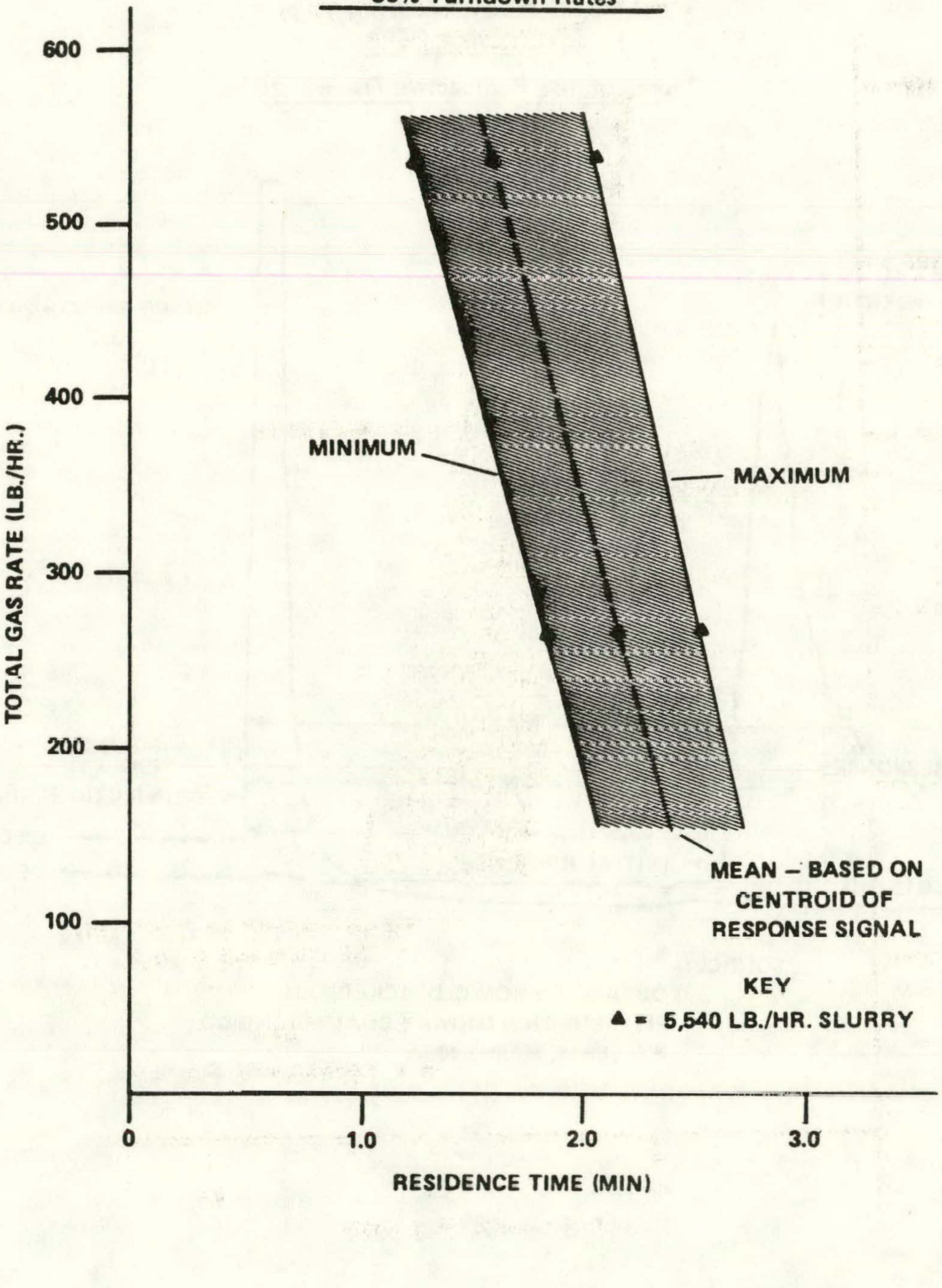


Figure 26
Fired Heater Slurry Residence Time
Intermediate Rates

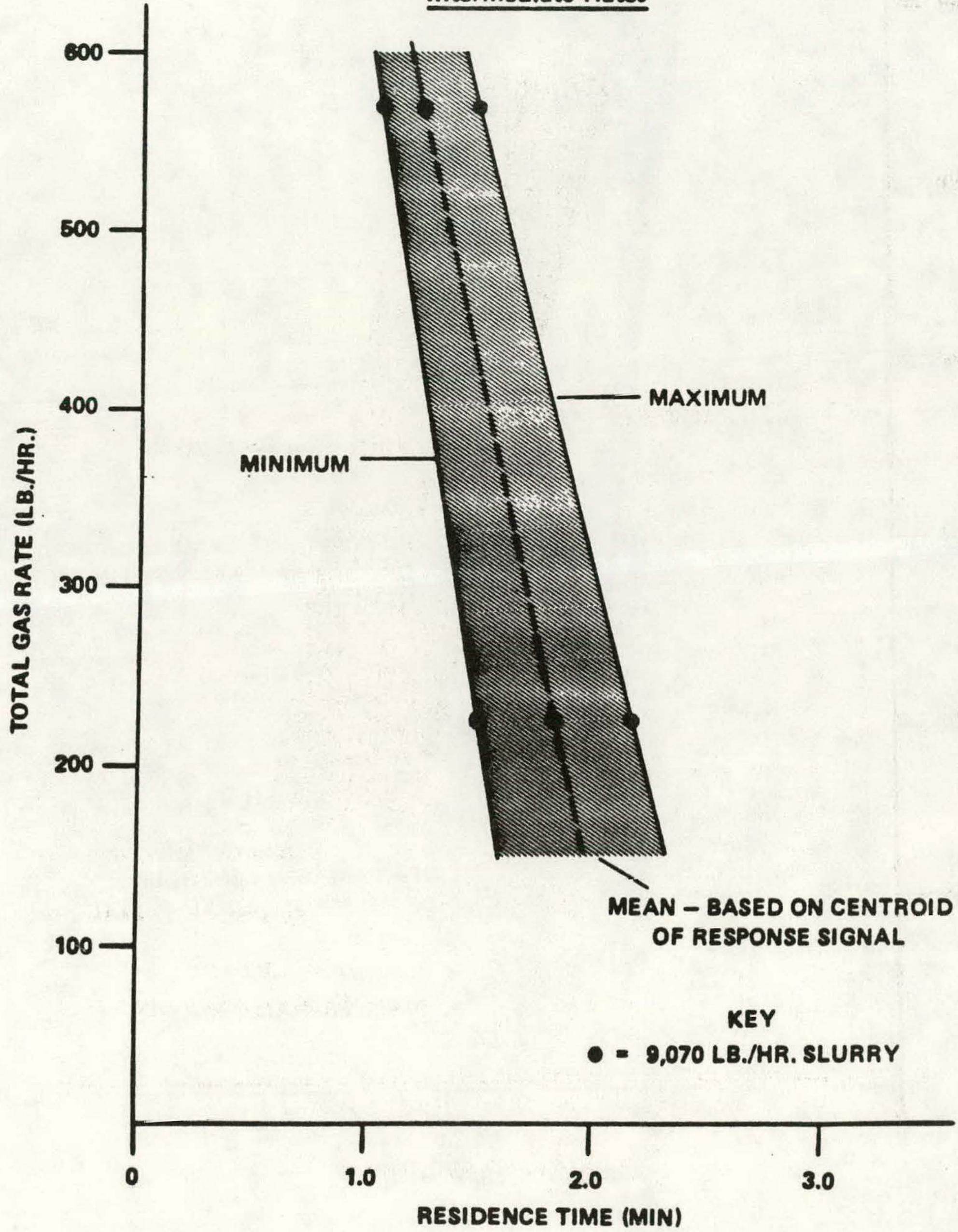


Figure 27
Fired Heater Slurry Residence Time
Maximum Slurry Rates

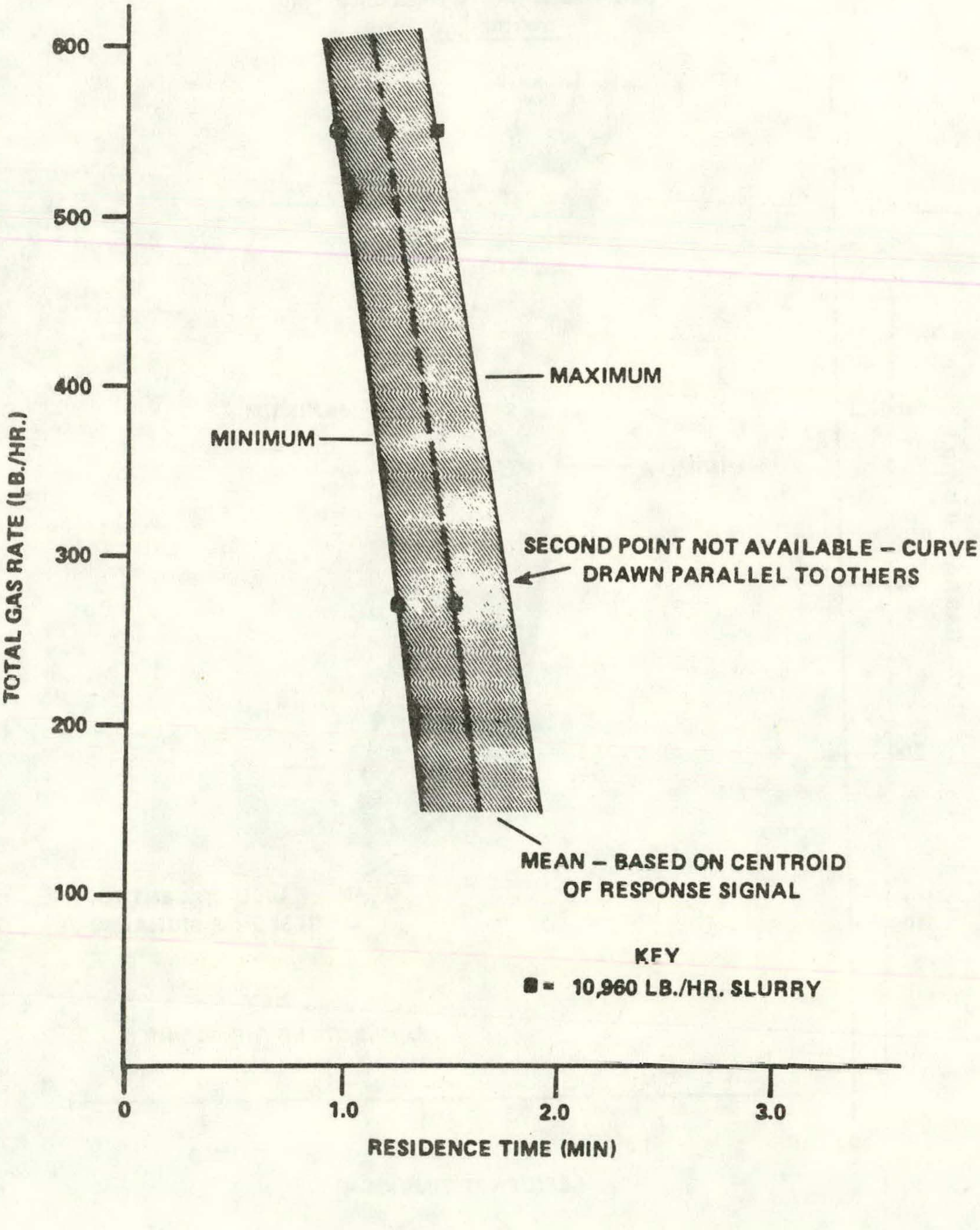


Figure 28
Fired Heater Residence Time
Slurry-Phase Extrapolation to Slurry-Only Runs

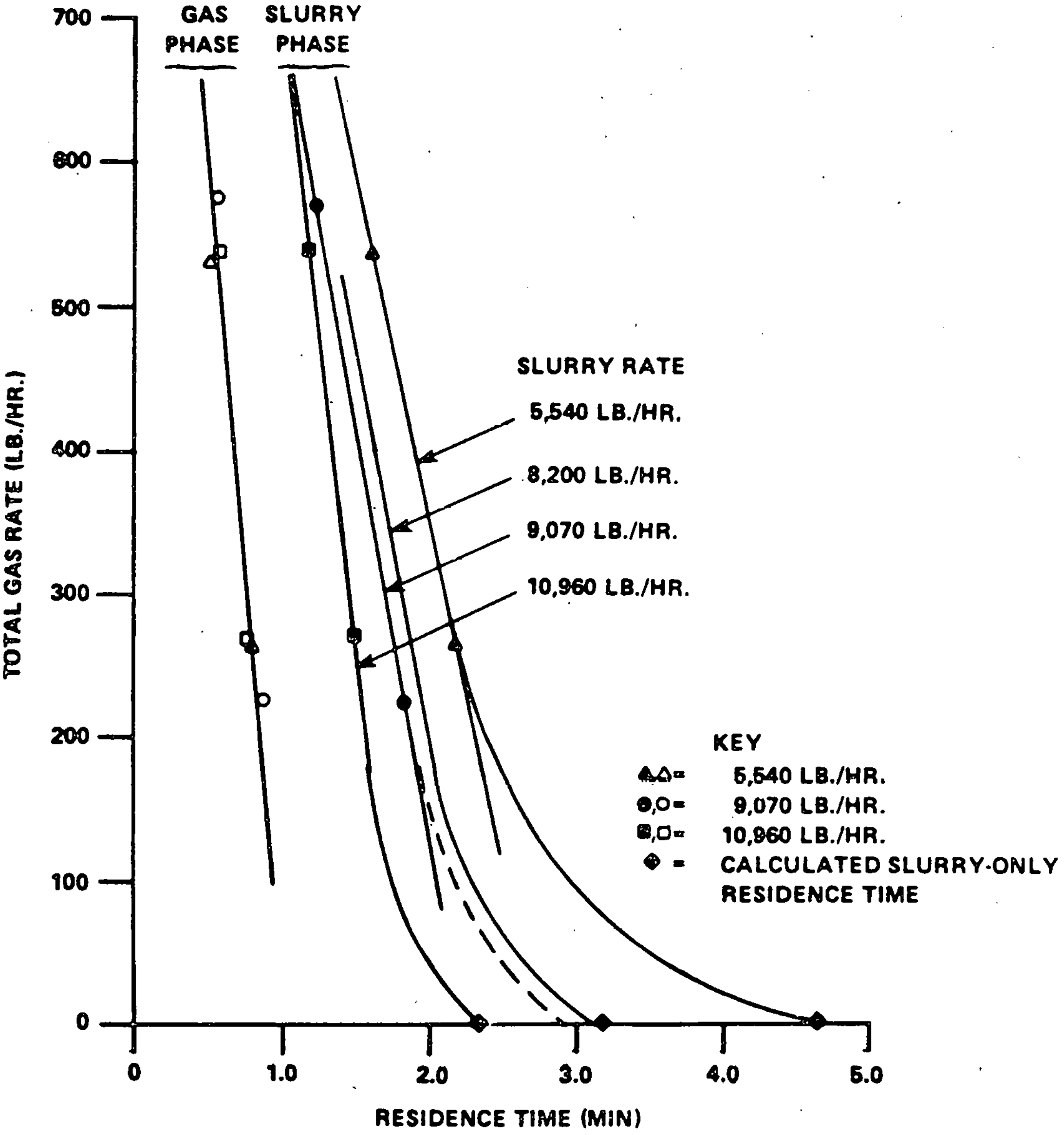


Figure 29
Fired Heater Residence Time
Gas-Phase Extrapolation to Slurry-Only Runs

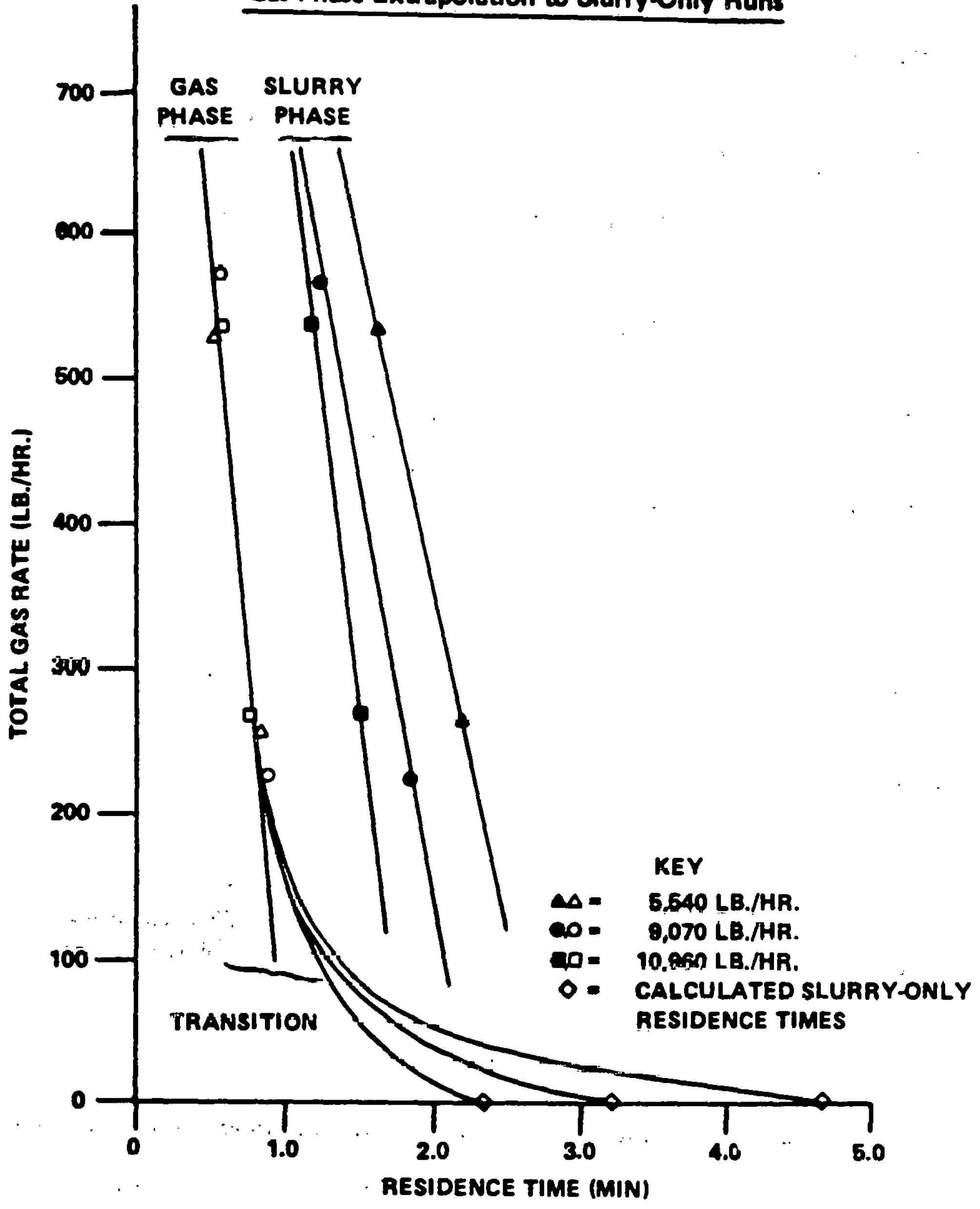
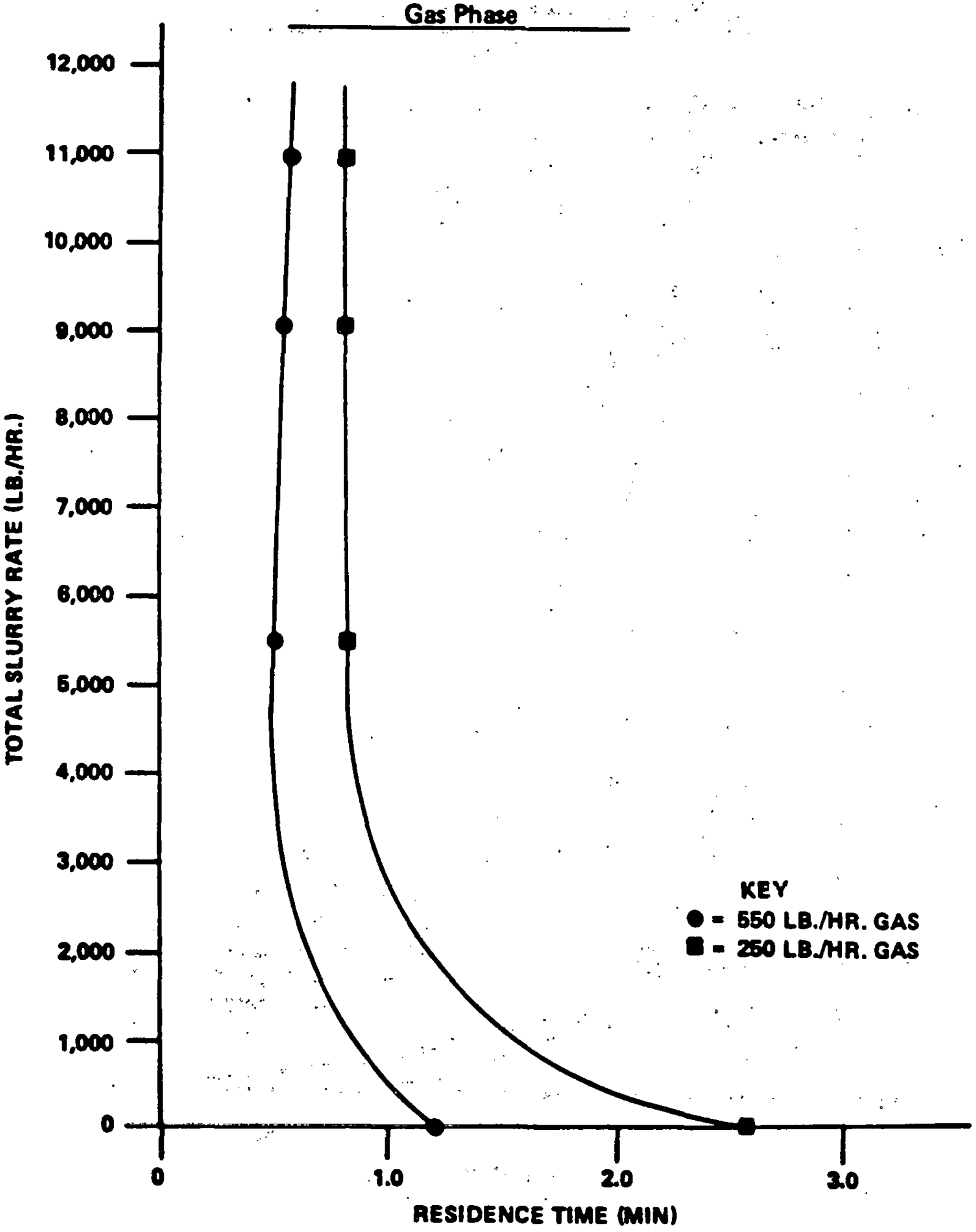


Figure 30
Fired Heater Residence Time
Gas Phase



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HIGH-TEMPERATURE SLURRY MIX TEST

T. W. Thew*

In August 1980, mixing tests at the Ft. Lewis Pilot Plant were conducted to determine the feasibility of preparing a 38.5 wt % slurry from feed coal and process solvent at temperatures of 350°F and higher. The Phase I SRC-I Demonstration Plant design has specified a blend tank temperature of 350°F. Blending at this temperature reduces the cooling required for the recycle process solvent, thus improving thermal efficiency.

In four experiments at temperatures ranging from 350 to 420°F, no operability problems were encountered in spite of a viscosity increase from 30 to 100 centipoise (cP). Thus, increasing the slurry mixing temperature up to 420°F should not significantly affect equipment design and provides for further potential thermal efficiency improvements.

In addition, a special study determining the effects of coal concentration and residence time has expanded the matrix of data required to understand and design the slurry mix system.

INTRODUCTION

This experimental program was formulated in support of the demonstration plant design. It was completed in August 1980, at the Ft. Lewis SRC Pilot Plant to determine the operability of the slurry mix system at elevated temperatures of 350-420°F while in the SRC-I mode of operation. The Phase 0 design specification for slurry blending was 280°F, which was then revised to 350°F in Phase I. The design also permits operation at up to 420°F, which further enhances the thermal efficiency of the process. In conjunction with the High-Temperature

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*International Coal Refining Co. (ICRC).

Program, a special study was completed to examine effects of variations in coal concentration and residence time upon slurry mixing at 350°F.

The Slurry Mix System

Figure 1 provides a flow schematic for the slurry mix system in service during this program. Dry coal feed is introduced into the top of the vortex mixer (23.5 in. i.d., 79 gal vol) via a varidrive auger (screw conveyer). Recirculated slurry is fed tangentially below the surface. Approximately 500 lb/hr of seal flush solvent is fed through a coal dust scrubber mounted above the tank. The combined feeds of coal and slurry are mixed at high speed (~1,540 rpm) with a three-blade dual impeller. The blended slurry passes via an overflow line to the slurry blend tank (SBT) which acts as a holdup tank for residence time. This vessel (5 ft i.d., 1,030 gal vol) is agitated at a rate of 112 rpm with a single four-paddle impeller. Additional data on these two vessels are provided in Table 1.

The slurry is gravity-fed through successive lengths of 4-, 6-, 4-, and 3-in. piping and valves to the 3-in. suction nozzle of the fully lined Lawrence or slurry circulation pump. The rate of discharge can vary between 80 and 150 gpm to provide sufficient velocity through the header of the charge pump to prevent settling of solids. Feed forward from the circulation pump is split between net feed to the reaction area via the charge pumps (20%) and recirculation to the vortex mixer (80%). The high rate of circulation effectively wets the dry coal feed.

Program Overview

Four high-temperature, slurry-mix tests were completed at temperatures (T_{SBT}) of 349, 377, 396, and 421°F, respectively (Table 2). The experimental program was originally conceived as a series of material balance runs. However, time constraints limited the duration of each experiment such that the program was completed in six days. Thus, the opportunity to generate yield data was never realized, and the tests were limited to demonstrating operability, alone.

HT-11 through HT-14 were run with a nominal 38.5 wt % coal slurry at an average throughput of 9,000 lb/hr. Recycle solvent was refractionated to an initial boiling point (IBP) of 500°F to deter excessive solvent vapor losses in the mix system. Figure 2 provides distillation curves for solvent averaged over the entire High-Temperature Program.

In addition to the four high-temperature runs, a series of 12, 2-hr experiments examining the effects of coal concentration and residence time were completed at a T_{SBT} of 350°F. This special study developed a matrix of data for parameters critical to slurry mixing and for the fired-heater program, which also specified a T_{SBT} of 350°F.

RESULTS AND DISCUSSION

Rheology

In preparation for the High-Temperature Program, laboratory rheological data were generated to predict high slurry viscosity problems. As expected, results of the rotary viscometer study indicated an increase in viscosity for a 38.5 wt % slurry with both temperature and time. However, a dramatic increase occurred at a temperature of 420°F, indicating a potential operating constraint in pilot plant tests.

Viscosities at very low residence times (10 min) were generally higher than other sources of data have indicated, and differences can be partly explained by the methods in which data are obtained. The rotary viscometer is open to the atmosphere, which permits solvent losses, resulting in a thicker paste. Also, shear rates in the circulation loop exceed the laboratory-calculated value of 25 sec^{-1}.

An on-line viscometer was used periodically through the program. Flow was directed from the circulation pump discharge to a 5-ft-long capillary viscometer via a slip stream. Viscosity data were generated in the 500-5,000 sec^{-1} shear rate range with two parallel capillaries, 0.305- and 0.5-in. i.d., respectively. In general, the measured viscosities fell within a range of 26-90 cP. The data indicated an initial reduction in viscosity followed by an increasing value as a function of temperature. The temperature at the viscometer was typically 20°F below that of the slurry blend tank because of thermal losses. No attempt to extrapolate the data to the SBT temperature has been made.

High Temperature Runs

Comprehensive run summary data provided in Table 3 are presented in flow sequence from the slurry mix system (i.e., vortex mixer - slurry blend tank - circulation pump - charge pump) to the reaction area (i.e., fired heater - dissolver). The nominal 1,000 lb/hr of seal flush was divided between the circulation pump, charge pump, and coal dust scrubber of the vortex mixer. Coal concentrations are computed for the net feed to the fired heater. In reality, the concentration may be slightly higher in the mix system since seal flush to the charge pump may be excluded; however, the difference is negligible.

The recycle solvent temperature indicates the heat input required to maintain the desired blend tank temperature. Figure 3 compares the recycle solvent temperature for each run to a parity curve for slurry blend tank temperature (T_{SBT}). The nonlinear behavior of the recycle solvent temperature plot is due to redistribution of seal flush and hot recycle solvent to permit a lower recycle solvent temperature. For a constant seal flush rate of 1,000 lb/hr (i.e., HT-11, -12, and -13), the solvent temperature would be a linear function of the desired blend tank temperature based upon a calculated mixing temperature of coal and solvent. The nonlinear deviation of Run HT-13 may indicate solvent losses through vaporization, especially since, at a temperature of 523°F, the IBP has been exceeded.

It was considered important to evaluate the effect of residence time as well as mixing temperature. Ideally, to determine slurry-mix system residence time, a boundary would be drawn around the equipment items of Figure 1 with the net input equal to the net feed forward under steady-state conditions. However, with the complexity of the piping and an unreliable level transmitter in the vortex mixer, the volume of the system is limited to those areas wherein accuracy can be maintained. As a result, residence time is computed on two volumetric bases:

(1) Volume of SBT indicated by level transmitter
(2) Volume of SBT and suction piping to circulation pump

Both bases allow for identification of trends in the data, but the latter is more representative of real time. Also, given the relative sizes of the two vessels, the volumetric contribution of the vortex mixer is considered negligible. The bottom tap of the level indicator is located 10 in. above the bottom of the vessel. The level indication relates the mass of fluid in only a portion of the vessel. Calculation of the volume of the tank below the tap and the suction piping yields an incremental volume of 95 gal, which, for levels maintained during the high-temperature tests, represents almost 50% of the defined slurry-mix system volume. Thus, as a general rule, residence times computed by method (2) are 50% higher than those of (1) and are closer to real time values as well. Review of the data of Table 3 reveals that an average slurry-mix system residence time of 16.8 min was realized versus a nominal 15 min specified.

A higher viscosity fluid is indicated by performance of the fired heater in which the measured pressure drop shows a dramatic increase at an SBT temperature of 421°F. Figure 4 shows a reasonably linear and constant pressure drop versus T_{SBT} curve through 400°F, followed by a 33% increase to 76.0 psid at 421°F. Figure 5 reveals that the higher pressure drop of Run HT-14 is broader than HT-11 in that the high pressure drop gradient extends over a greater portion of the coil. (Profiles of HT-11, HT-12, and FT-13 were very similar.)

Heat-transfer characteristics as manifested in the temperature profiles are displayed in Figure 6. The gel region, identified by the peak in the tube wall temperature relative to a linear fluid temperature, is displaced upstream as the slurry blend tank temperature increases. The most dramatic difference, however, again appears in Run HT-14 in which the gel is evident at the inlet of the fired heater, though in a much less distinguishable peak. As expected, the higher slurry blend tank temperature corresponds to a higher fired heater inlet temperature, which, for a fixed outlet temperature, implies a reduction in duty. Time-temperature dependence of slurry viscosity helps explain the phenomenon observed in Figure 6. For a constant residence time in the slurry blend tank, the coal would swell and pass into solution sooner as the temperature increases. Thus, the gel region is displaced upstream on entering the fired heater where heat transfer is very rapid. Discussion of the special study will clarify this.

Special Study: Coal Concentration and Residence Time

Over a 2-day period near the close of the High-Temperature Program, a special study examined the effects of coal concentration and residence time. Twelve, 2-hr experiments developed a matrix of data over ranges of 38.0-45.3 wt % coal concentration and 3.5-24.6 min residence time. The short duration of each run limited the data acquisition compared to that summarized for the high-temperature runs; thus, most of the interpretation will be based upon the observed fired heater performance.

Table 4 summarizes each of runs HT-15-1 through HT-15-12. Each sequence of four runs examines the effect of residence time in the blend tank for a constant coal concentration, and therefore, slurry rate. Coal concentration, examined at levels of 38.0, 41.2, and 44.8 wt %, was increased by increasing the dry coal feed to the mix system. This variance in throughput must be considered in interpreting the data. Also, unlike the determination of slurry residence time for the HT-11 through HT-14 runs, the residence times calculated for this special study are based upon the level maintained in the slurry blend tank itself.

During the High-Temperature Program, fired heater performance monitoring revealed several effects. First, with regard to pressure drop, Figure 7 plots the data as a function of slurry blend tank residence time for each of three coal concentrations, and, therefore, slurry rates. While relatively little sensitivity to residence time is observed, a qualitative trend is apparent. There is a 7-10% increase in pressure drop as residence increases to approximately 12 min, after which the values are constant. Valid comparison of residence time data with that of the four high-temperature runs should be limited to slurry blend tank residence time values. It should be noted that point A is not consistent with other points of the intermediate slurry runs, because a lower solvent rate was realized. The anomalous behavior of point B (Run HT-15-9) is explained by the fact that the plant was not operating at the steady-state conditions characterizing all other runs. However, this run does provide a useful dynamic study as Figure 8 illustrates.

The last of four intermediate coal concentration runs (HT-15-5, 41.2 wt %) was completed at 1600 on August 15, 1980. This low residence time study led directly to the analogous run (HT-15-9) at the higher coal concentration of 45.3 wt %. Line-out for the prescribed conditions was limited to 1 hr with a very rapid incremental increase in coal rate. Thus, although rates were stabilized by 1700, thereby allowing the start of that run, the temperature-time plot of Figure 8 clearly shows that steady-state conditions were not achieved until 1900, at the conclusion of the 2-hr run. The 700-lb increment of coal acted as a heat sink for which thermal recovery required over 2 hr. The consequences of this nonsteady-state condition are observed in the fired heater pressure drop plot also shown as a function of time in Figure 8. A very stable 70 psid pressure drop had been realized for Run HT-15-5. When the transition to HT-15-9 conditions was under way, the pressure drop was reduced to a value below 50 psid (actually as low as 33 psid where data are available for this interval). Observation of the trend clearly shows an increase in pressure drop with time until a steady-state value greater than 90 psid is reached. This occurs at 1900, concluding the run. Thus, over the 2-hr period, the computerized average delta P was only 80 psid. Following this run, only level changes were made to vary the residence time, i.e., the plant remained at steady-state temperatures and flows.

Neither the total pressure drop nor the shape of the differential pressure profile varies significantly with residence time for a constant coal concentration. However, as a function of coal concentration (and slurry rate) for a constant residence time in the mix system, the pressure drop not only increases in magnitude, but also displays a greater pressure drop over a larger portion of the coil length.

Figure 9 compares the profiles of HT-15-1 and HT-15-5, wherein the concentration increased from 38.0 to 41.2 wt %, respectively, while the slurry blend tank residence time was nominally 4 min. Comparison of analogous runs reveals the same trend, for it is evident that a greater pressure drop is observed in the middle of the coil, corresponding to the 480-600°F range in fluid temperature. Since rates do vary between series of runs showing coal concentration differences, residence time effects in the fired heater must be considered in interpreting this data.

Several trends analogous to those observed in the high-temperature runs are apparent in the temperature profiles. For a constant SBT residence time, the gel region is displaced upstream as the coal concentration increases. (The gel region is discussed in the preceding article in this report, "ICRC Fired Heater Test Program.") Figure 10 compares the temperature profiles of Runs HT-15-2, HT-15-6, and HT-15-10, corresponding to coal concentrations of 38.0, 41.2, and 45.0 wt %, respectively, and having an average SBT residence time of 8.7 min. The trend apparent in this figure was also observed in comparing remaining profiles with similar residence times. Also evident is a distinct change in the character of the gel region. The rather sharp, narrow peak of HT-15-10 was observed in all of the high concentration experiments. The position and shape of the gel region are speculated to be functions of rate (i.e., residence time) and coal concentration, although the precise relationship is not well understood.

CONCLUSIONS

Results of high-temperature, slurry-mix studies revealed that 38.5 wt % slurry from feed coal and process solvent can be prepared successfully at temperatures ranging from 350 to 420°F. Although all data indicate an increase in slurry viscosity with increase in temperature over this range, the measured values of 30-100 cP are consistent with other sources of slurry viscosity data and do not hinder design efforts. Results of these studies not only showed that equipment design should not be significantly affected by an increase in the slurry mixing temperature, but also identified potential thermal efficiency improvements.

A special study examined the effects of both residence time and coal concentration on slurry mixing (at a blend tank temperature of 350°F), as well as fired heater performance. Residence time seemed less important than anticipated over the 3- to 25-min range, but any effect may be enhanced at temperatures greater than 350°F, as the laboratory rheology study concludes. Increases in coal concentration revealed a trend toward upstream displacement of the gel region, as well as greater overall pressure drops.

Table 1

Vessel Geometry

Specification	Vortex Mix Tank	Slurry Blend Tank
Inside diameter (in.)	23.5	60
Height (ft, tan-tan)	3.5	7.0
Volume (gal)	78.9	1028
Material	CS[a]	CS
Agitator		
Number of impellers on shaft	2	1
Number of blades on impeller	3	4
Pitch	1.5	1
Impeller diameter (in.)	7	14.3
Shaft diameter (in.)	1.9	2.5
Position of blades (in., from bottom of vessel)	9	11.8

[a] carbon steel

Table 2

High Temperature Slurry Mix Tests

Run no.	Date	Duration (hours)	Blend tank temperature (°F)
HT-11	9-10 Aug 80	24	349
HT-12	11-12 Aug 80	24	377
HT-13	13-14 Aug 80	24	396
HT-14	16-17 Aug 80	12	421

Table 3

High Temperature Run Summary

Run no. Date	HT-11 9, 10 Aug 80	HT-12 11, 12 Aug 80	HT-13 13, 14 Aug 80	HT-14 16,17 Aug 80
MF coal rate (lb/hr)	3,464	3,451	3,437	3,449
Total solvent (lb/hr)	5,633	5,642	5,485	5,455
Total slurry (lb/hr)	9,097	9,093	8,922	8,904
Coal concentration (wt %)	38.1	38.0	38.5	38.7
Total gas (lb/hr)	218	238	264	257
(scfh)	16,750	17,590	19,600	19,050
Recycle solvent temperature (°F)	424	470	523	541
Vortex mixer temperature (°F)	352	380	406	431
Slurry blend tank				
Temperature (°F)	349	377	396	421
Residence time (min)	13.7	10.3	10.8	10.4
Slurry mix system				
Residence time (min)	19.1	15.7	16.3	15.9
Circulation pump				
Recirculation rate (gpm)	117	112	80	99
Fired heater				
Overall pressure drop (psid)	58.2	55.4	58.8	76.0

Table 4a

Special Study Run Summary

HT-15-1 through HT-15-4

Run no. Date	HT-15-1 14 Aug 80	HT-15-2 14 Aug 80	HT-15-3 14 Aug 80	HT-15-4 14 Aug 80
MF coal rate (lb/hr)	3,460	3,460	3,460	3,460
Total solvent (lb/hr)	5,650	5,650	5,655	5,655
Total slurry (lb/hr)	9,110	9,110	9,115	9,115
Coal concentration (wt %)	38.0	38.0	38.0	38.0
Total gas (lb/hr)	253	257	257	258
(scfh)	19,520	19,160	19,320	19,010
Recycle solvent temperature (°F)	430	429	429	429
Vortex mixer temperature (°F)	359	357	356	356
Slurry blend tank				
Temperature (°F)	353	351	349	350
Residence time (min)	3.5	7.8	13.3	23.1
Circulation pump				
Recirculation rate (gpm)	80	80	80	80
Fired heater				
Overall pressure drop (psid)	50.5	54.0	56.0	56.0

Table 4b

Special Study Run Summary

HT-15-5 through HT-15-8

Run no. Date	HT-15-5 15 Aug 80	HT-15-6 15 Aug 80	HT-15-7 15 Aug 80	HT-15-8 15 Aug 80
MF coal rate (lb/hr)	3,960	3,960	3,960	3,960
Total solvent (lb/hr)	5,640	5,640	5,650	5,445
Total slurry (lb/hr)	9,600	9,600	9,610	9,410
Coal concentration (wt %)	41.2	41.2	41.2	42.1
Total gas (lb/hr)	251	253	250	246
(scfh)	18,640	18,790	18,570	18,270
Recycle solvent temperature (°F)	444	440	440	435
Vortex mixer temperature (°F)	359	355	357	356
Slurry blend tank				
Temperature (°F)	352	348	350	349
Residence time (min)	3.9	9.0	14.3	24.6
Circulation pump				
Recirculation rate (gpm)	80	80	80	80
Fired heater				
Overall pressure drop (psid)	74.5	79.0	79.0	73.5

Table 4c

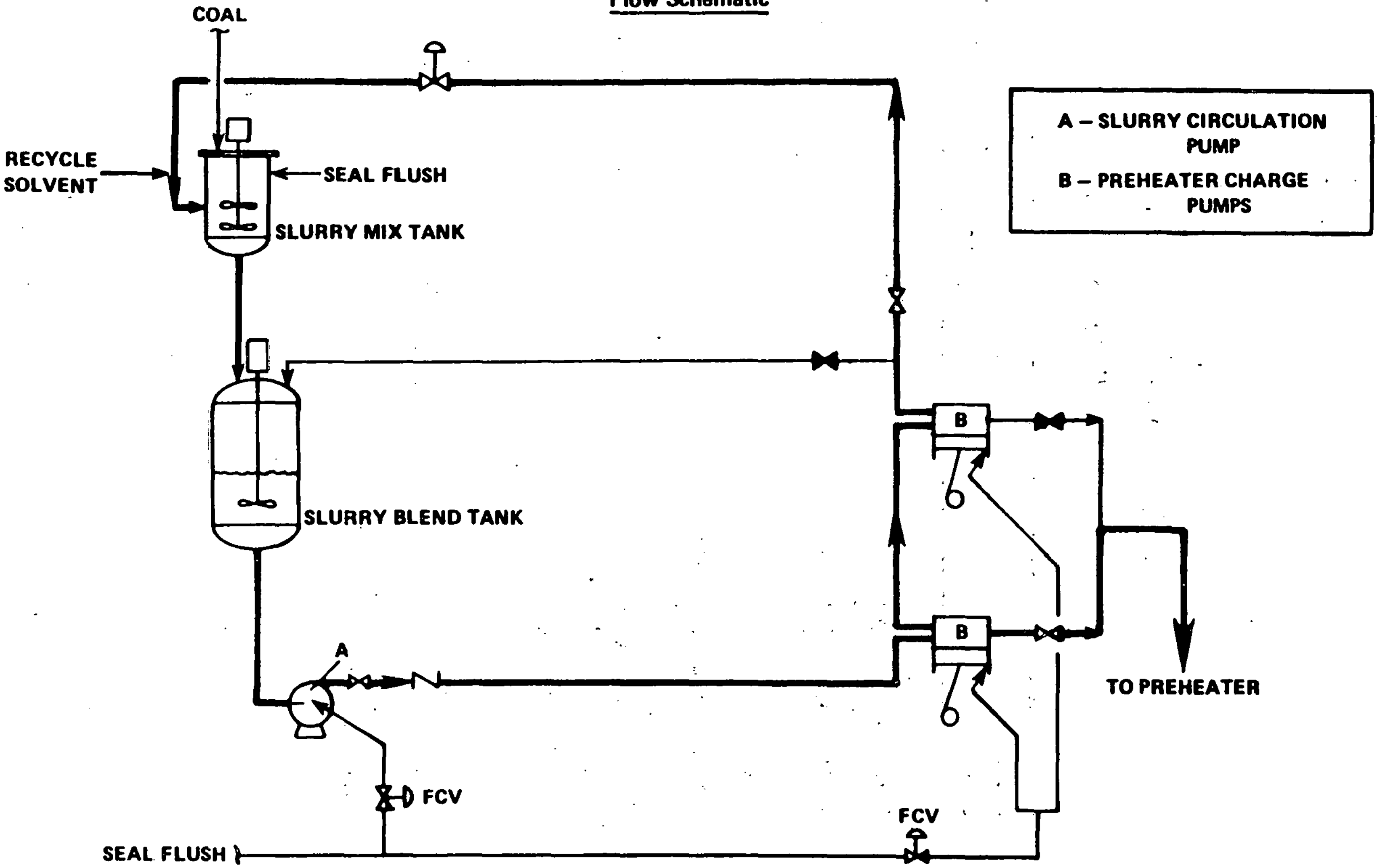
Special Study Run Summary

HT-15-9 through HT-15-12

Run no. Date	HT-15-9[a] 15 Aug 80	HT-15-10 15 Aug 80	HT-15-11 16 Aug 80	HT-15-12 16 Aug 80
MF coal rate (lb/hr)	4,660	4,600	4,510	4,570
Total solvent (lb/hr)	5,630	5,630	5,670	5,655
Total slurry (lb/hr)	10,290	10,230	10,180	10,225
Coal concentration (wt %)	45.3	45.0	44.3	44.7
Total gas (lb/hr)	241	238	231	240
(scfh)	17,900	17,680	17,160	17,830
Recycle solvent temperature (°F)	442	445	444	444
Vortex mixer temperature (°F)	335	357	359	361
Slurry blend tank				
Temperature (°F)	347	348	352	353
Residence time (min)	4.6	9.2	14.8	24.1
Circulation pump				
Recirculation rate (gpm)	80	115	115	115
Fired heater				
Overall pressure drop (psid)	80.0	106.0	105.0	105.0

[a] Results from this run are suspect because temperatures had not attained equilibrium.

Figure 1
Slurry Mix System
Flow Schematic



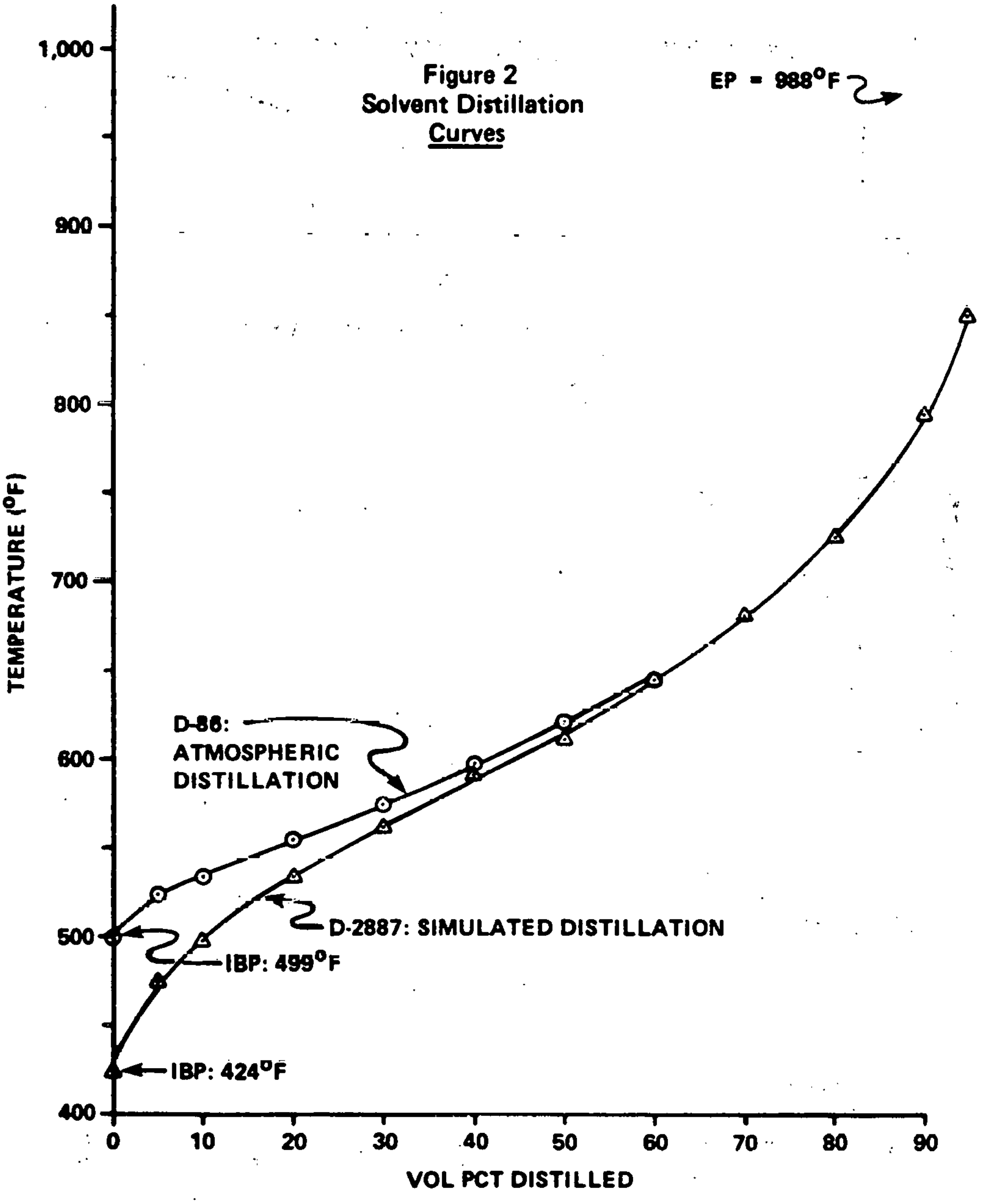


Figure 2
Solvent Distillation
Curves

Figure 3
Recycle Solvent
Temperature vs. Slurry Blend Tank Temperature

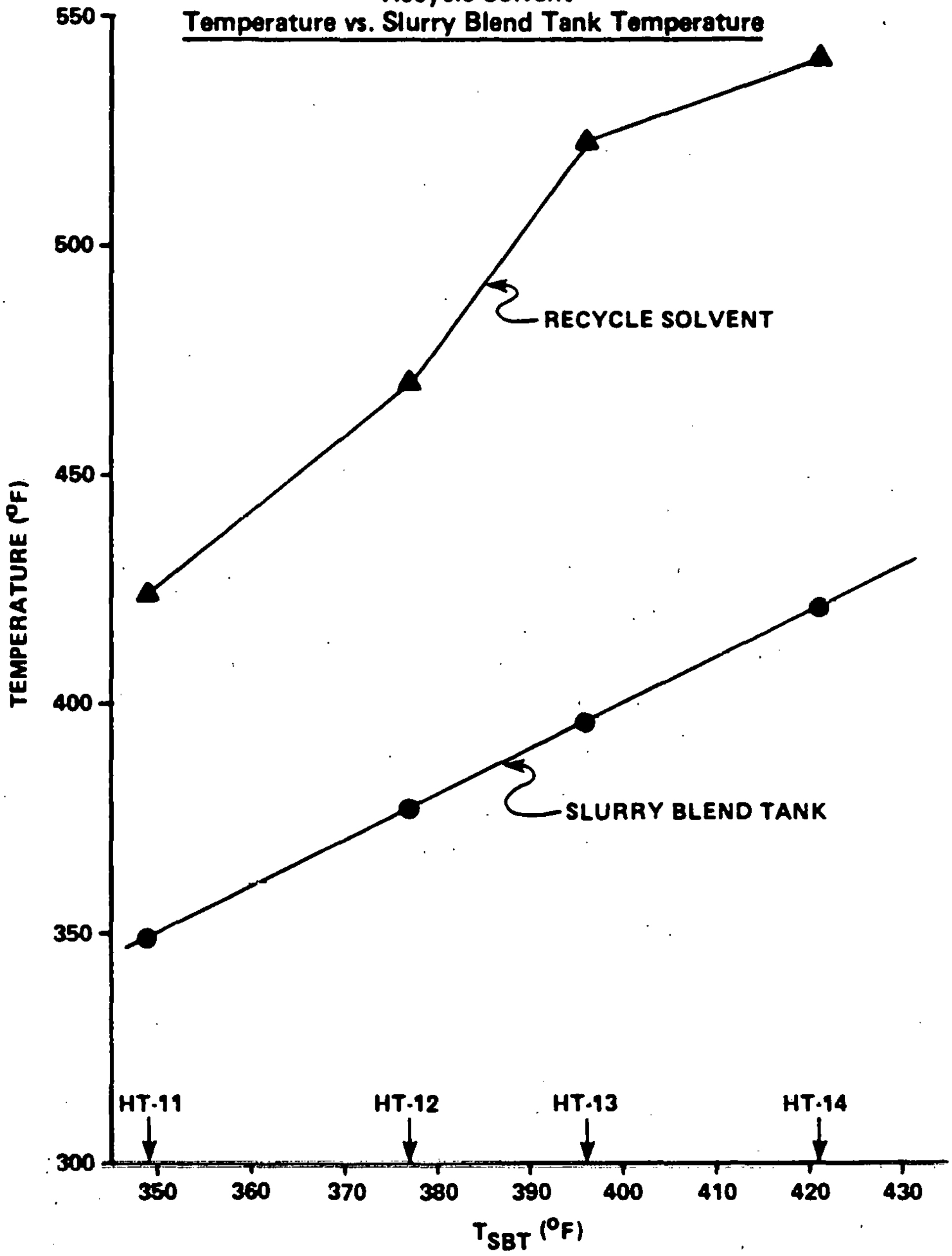


Figure 4
Fired Heater
Pressure Drop

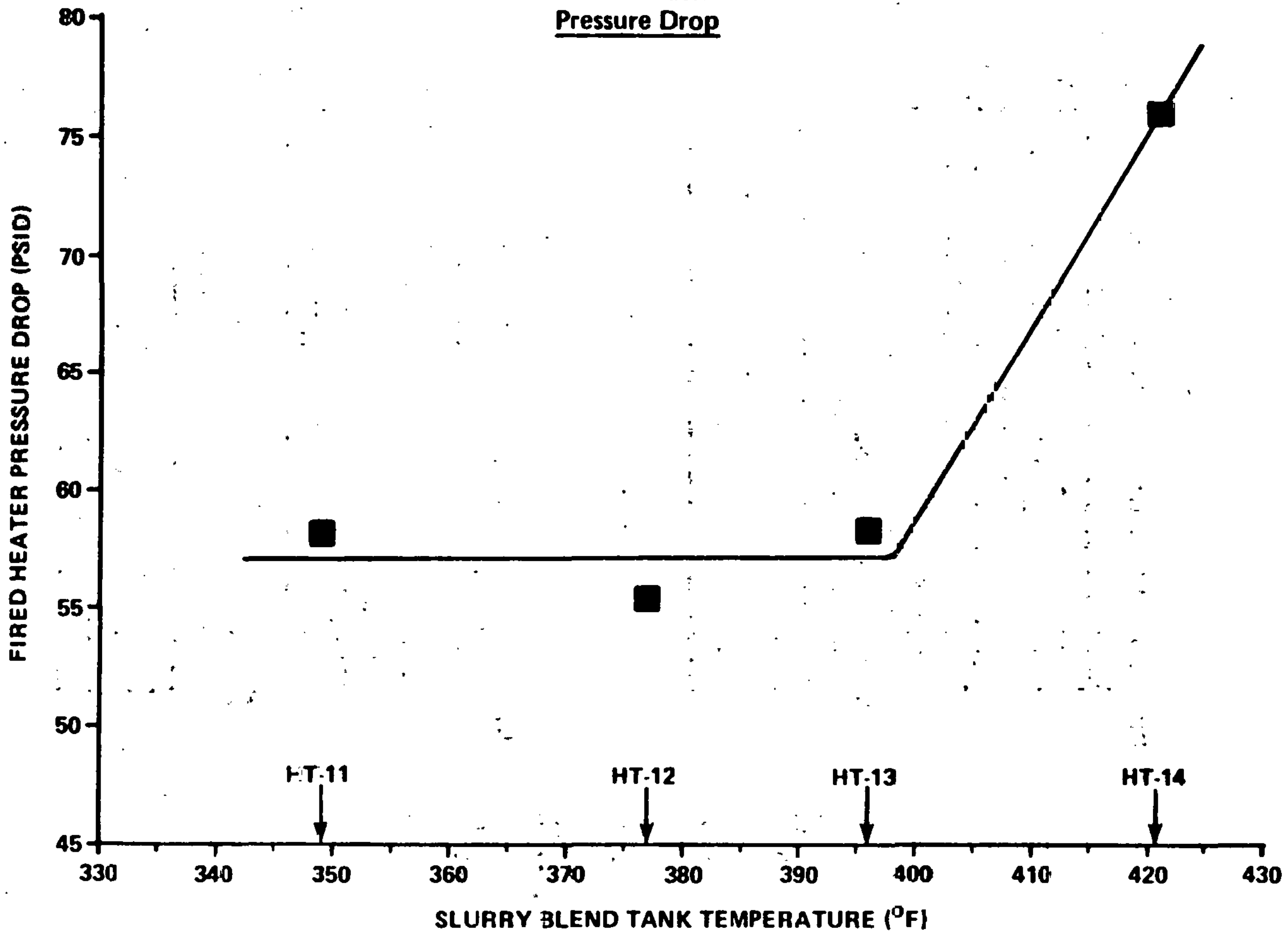


Figure 5
Fired Heater Pressure Drop Profiles
As Function of Blend Tank Temperature

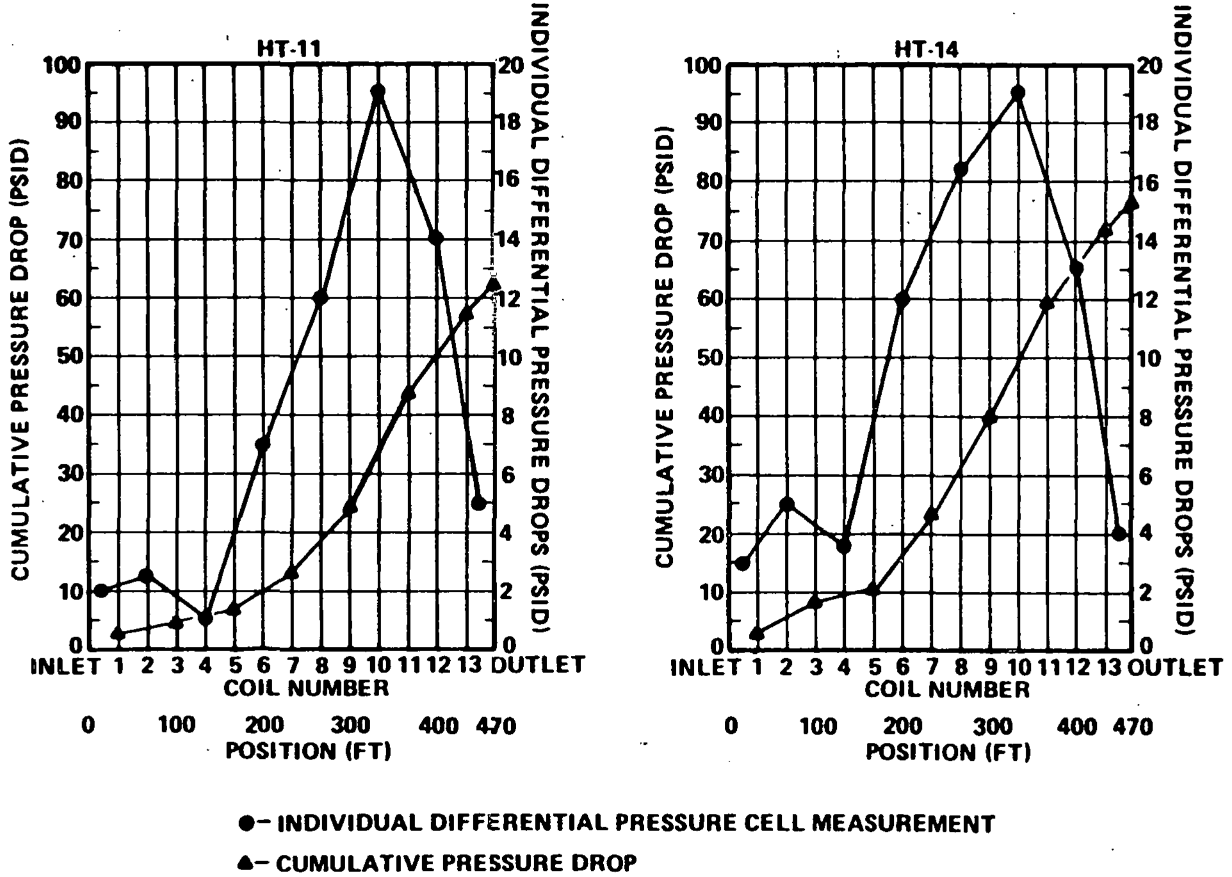


Figure 6
Comparison of Fired Heater
Temperature Profiles

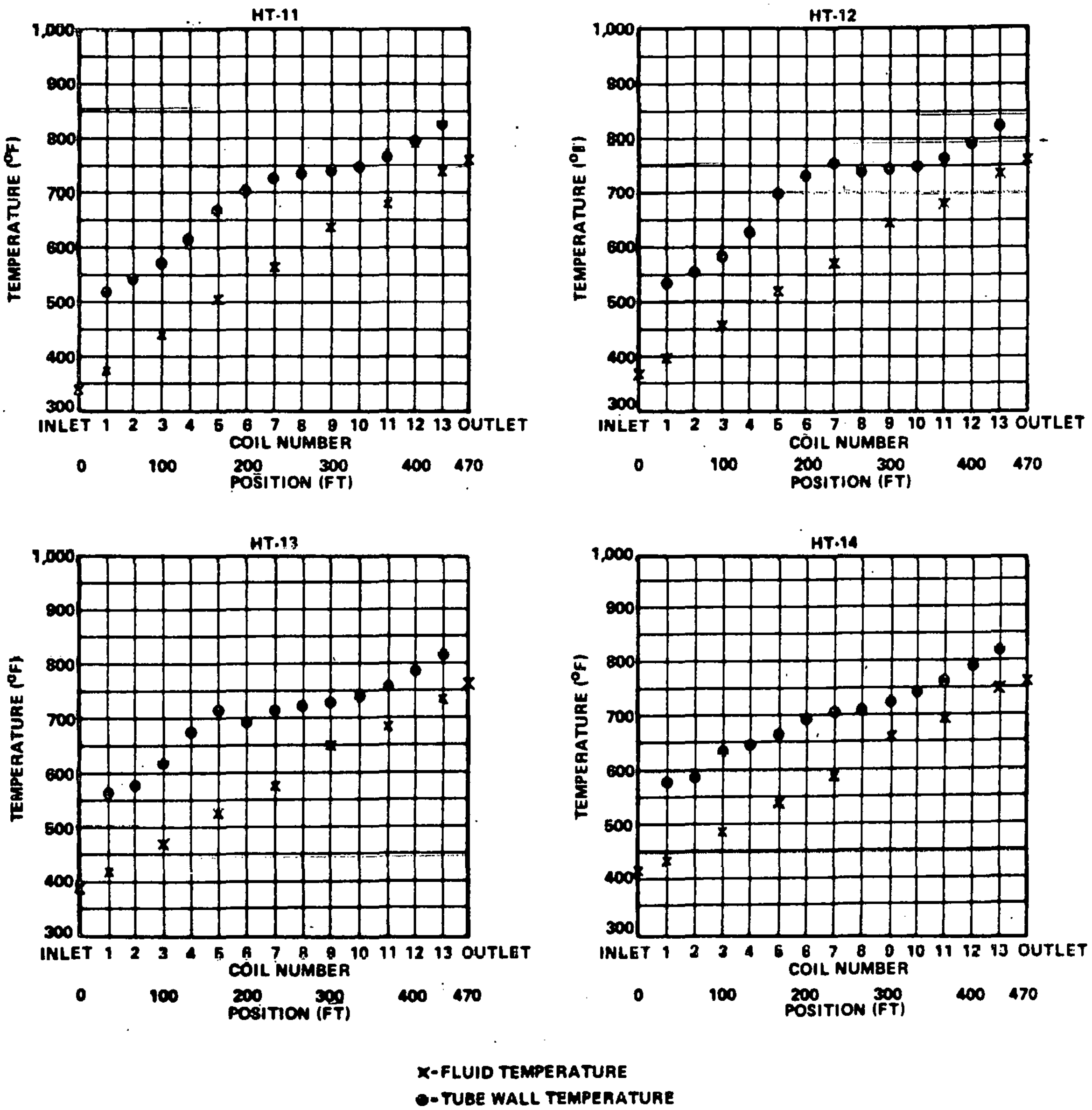


Figure 7
Special Study:
Fired Heater Pressure Drop

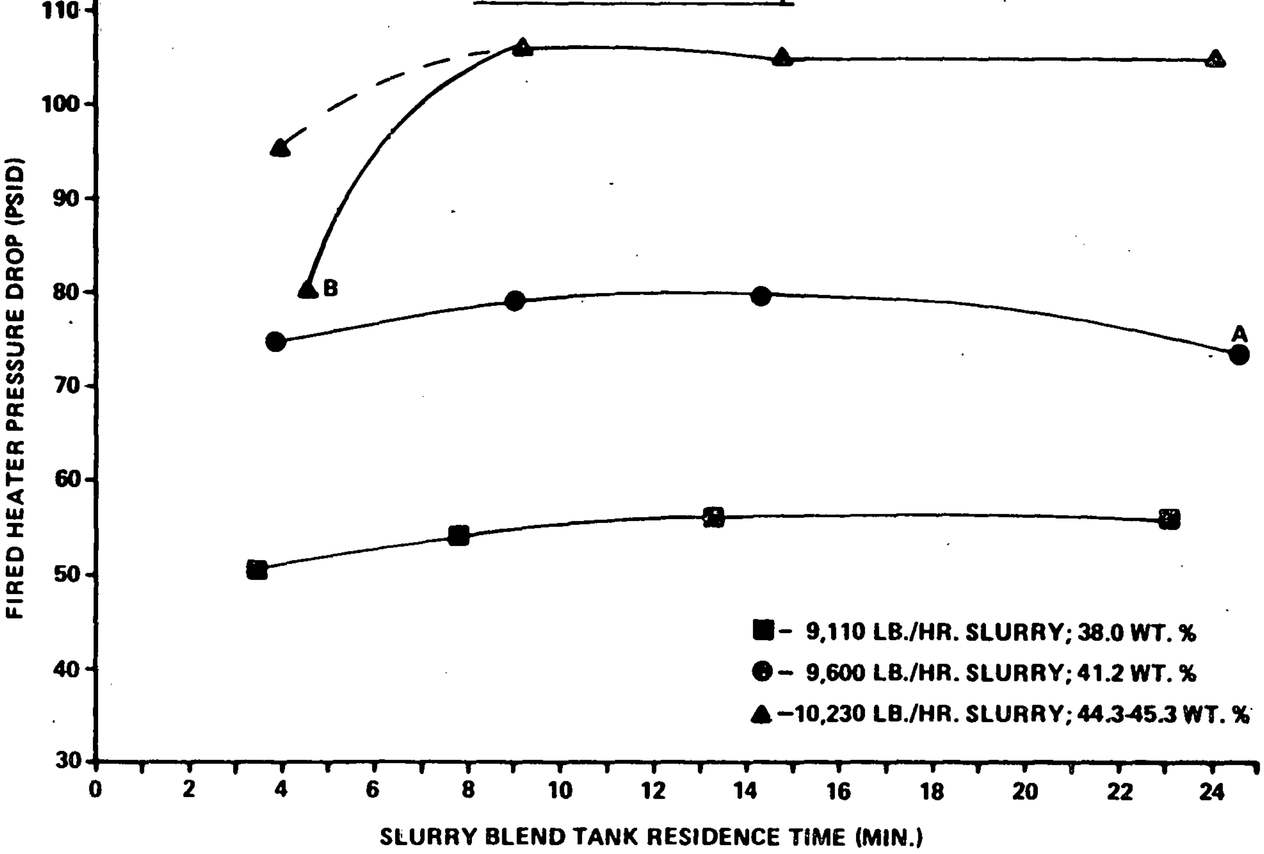


Figure 8
Dynamic Behavior of Run
HT-15-9

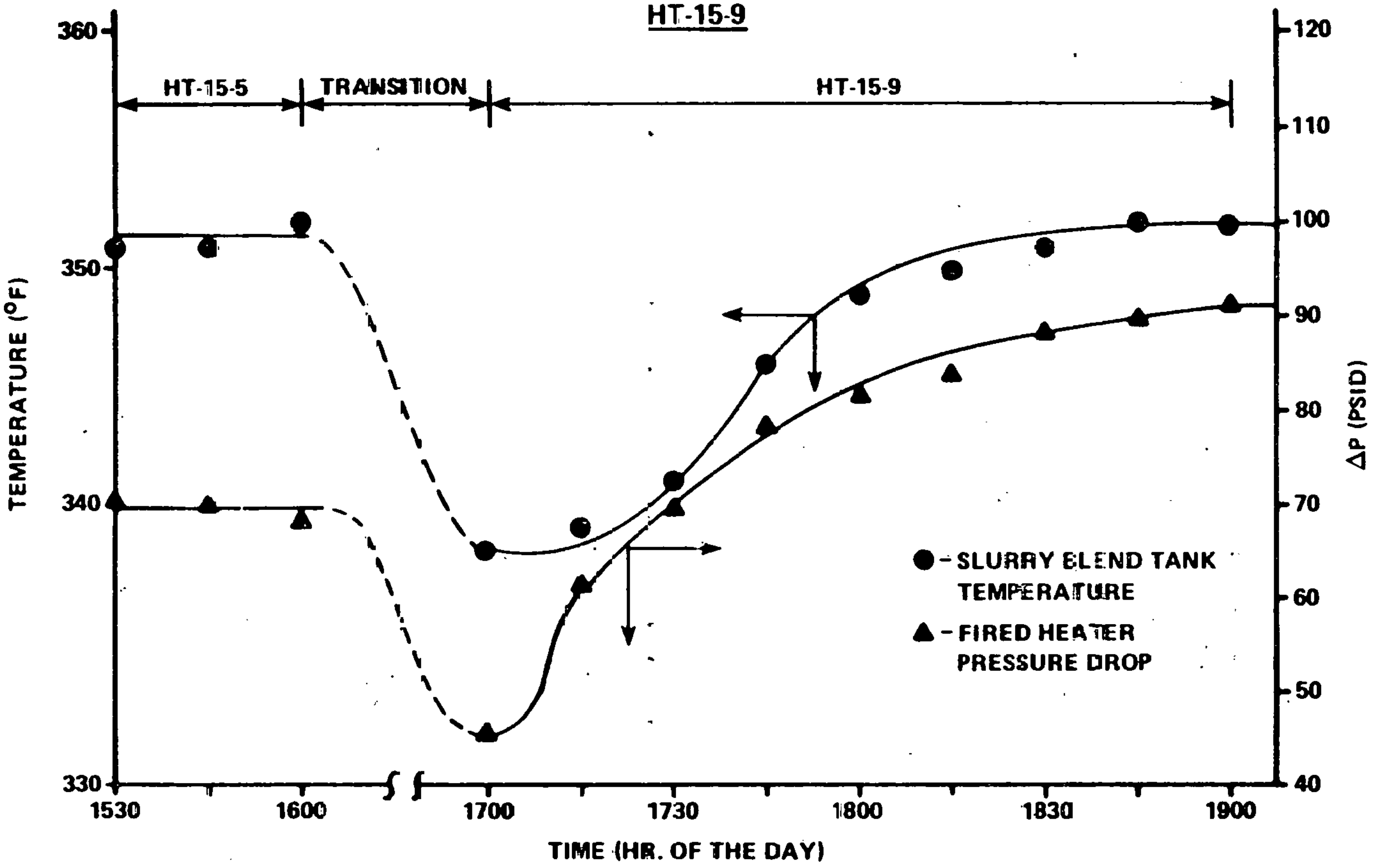
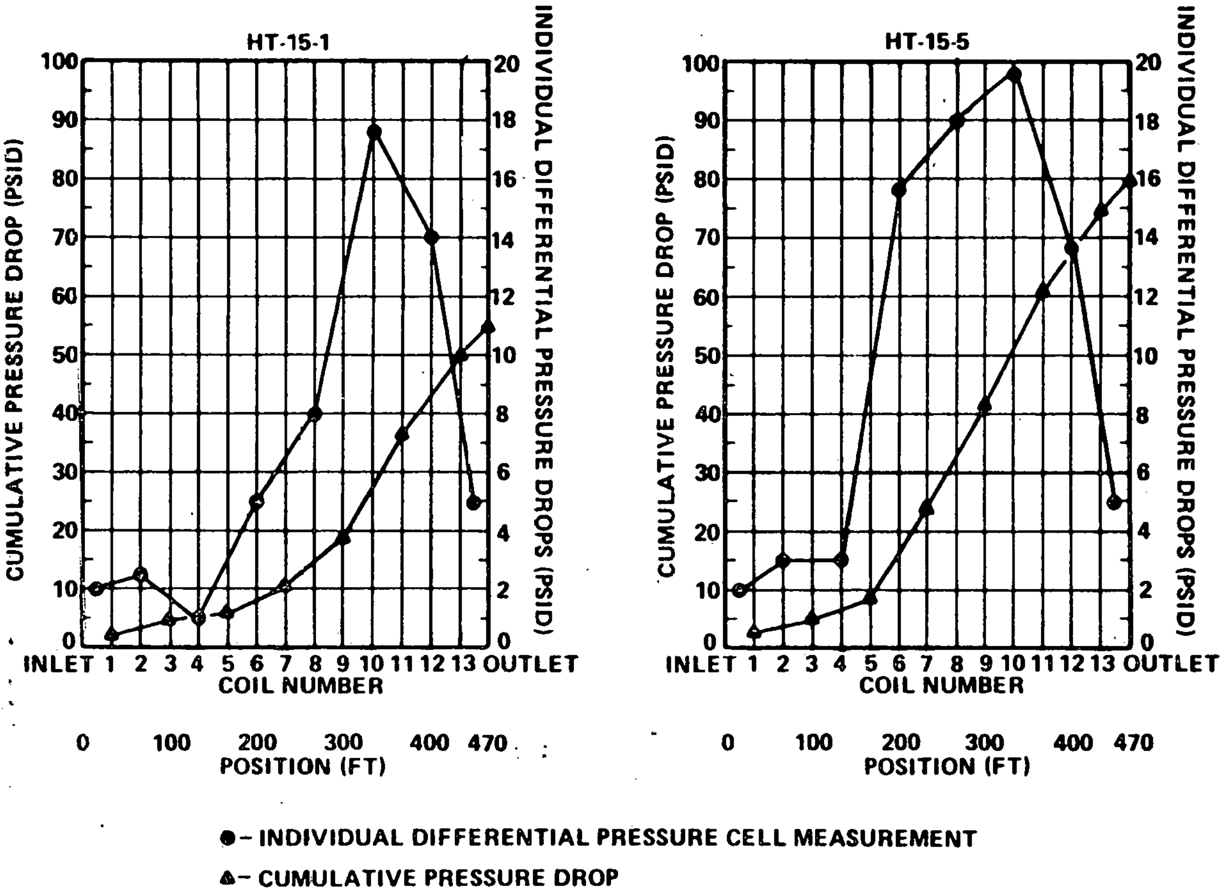


Figure 9
Fired Heater Pressure Drop Profiles As
Function of Coal
Concentration



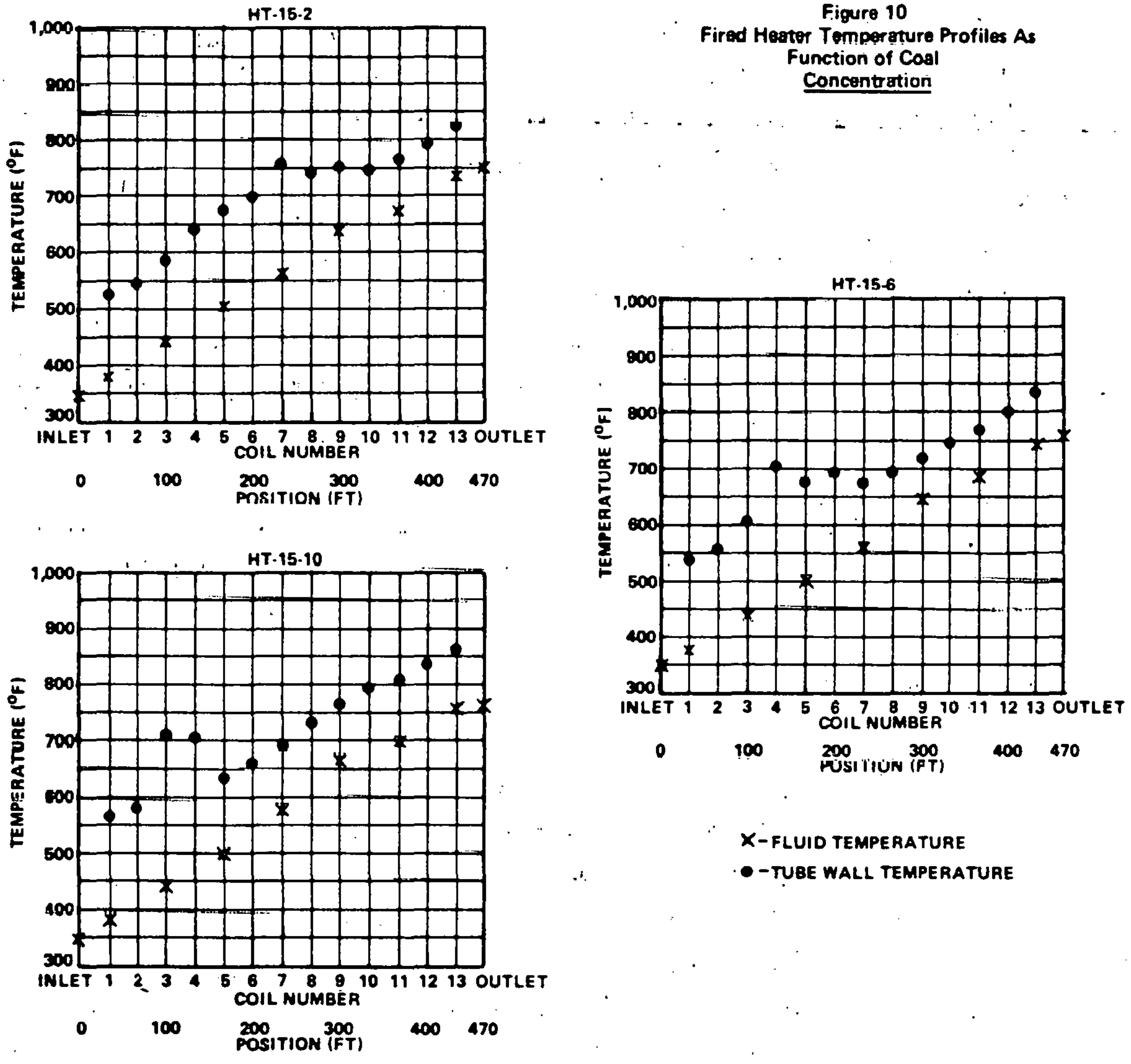


Figure 10
Fired Heater Temperature Profiles As Function of Coal Concentration

COAL FEED SLURRY RHEOLOGY

T. W. Thew*

Rheological characterization of coal feed slurry was undertaken in conjunction with SRC-I slurry mix tests at the Ft. Lewis SRC Pilot Plant in January 1981. Results show that the shear-dependent fluid behaves like a Bingham plastic, as reflected by a linear dependence of shear stress upon shear rate and a finite (>0) yield stress at zero shear. Viscosities increased with increasing temperature over the range of 285-420°F, but were independent of residence time over the range of 14-41 minutes. Shear rates spanned a range of 9.3-186 $\sec^{-1}$, and though non-Newtonian behavior was very evident in this interval, extrapolation to higher shear rates indicates coal feed slurry is essentially Newtonian.

These studies indicate that, for design purposes, a viscosity of 95 cP at temperatures up to 420°F should be specified for the coal feed slurry.**

INTRODUCTION

An SRC-I slurry mixing test program was completed at the Ft. Lewis SRC Pilot Plant over a 9-day period in January 1981. The overall purpose of the program was to define the makedown characteristics of coal with recycle solvent at temperatures and residence times consistent with the Phase I design basis of the demonstration plant. As part of this experimental program, coal feed slurry rheology was characterized to determine the effects of these parameters upon slurry viscosity.

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*International Coal Refining Company.

**See section "Application to Demonstration Plant Design."

EQUIPMENT AND PROCEDURE

Hot slurry samples were collected directly from the transfer line downstream of the slurry blend tank into a sample cylinder at temperature in a Brookfield viscometer with a #21 spindle (see Scheme I for the test set-up). Viscosity of each slurry sample was then determined at five shear rates ranging from 9.3-186 sec^{-1}. General conditions for the feed slurry are summarized in Table 1.

Of a total of 34 samples taken and analyzed, 26 were judged to be representative of steady-state conditions. These data were then grouped according to discrete values of residence time (θ) and temperature (T), as shown in Table 2, resulting in eight curves representing viscosity vs. shear rate.

DISCUSSION

Measured viscosities show increasing dependence upon shear rate with increasing temperature for a constant residence time over the range of 9.3-186 sec^{-1}. Figures 1, 2, and 3 illustrate this trend at residence times of 14, 27, and 41 minutes, respectively. The non-Newtonian behavior is enhanced at the higher temperatures in each family of curves, as evidenced by the increasing non-linearity. However, these same data plotted as a function of residence time for a constant temperature indicate that slurry viscosity is only weakly dependent upon residence time (Figures 4 and 5); at 354°F, the curves virtually overlap, and, though more discrete at 400°F, differences are no greater than error in measurement.

Shear stress may be obtained from the viscosity and shear rate data plotted in Figures 1, 2, and 3, using the relationship:

$$\mu_{APP} = \frac{\tau}{S} \qquad (1)$$

where μ_{APP} is measured viscosity, S is shear rate, and τ is shear stress. Figures 6, 7, and 8 are plots of shear stress as a function of shear rate on linear coordinates, corresponding to Figures 1, 2, and 3. The results show a linear relationship between τ and S, but with a y-intercept. This indicates a Bingham plastic behavior of the slurry with a residual shear stress at zero shear rate, which is equal to the y-intercept.

Each of Figures 6, 7, and 8 examines the effect of temperature at a constant residence time. Within each family of curves, as the temperature increases, both the slope and the y-intercept increase. (As expected, there is little difference in the plots for various residence times at a constant temperature, since the data were generated from Figures 1, 2, and 3.)

Extrapolation of the linear plots of shear stress-shear rate from low shear rates (i.e., 9.3 $\sec^{-1}$) to a shear rate of zero provides the basis for concluding that coal feed slurry exhibits Bingham plastic behavior. The following linear equation defines this behavior:

$$\tau = \tau_o + \eta_{pl} \cdot S \tag{2}$$

where τ_o, the yield stress, is given by the y-intercept of the plots in Figures 6, 7, and 8, and η_{pl}, the plastic viscosity, is the slope. Values of τ_o and η_{pl}, obtained by regression analysis, are presented in Table 2. Both parameters increase with increasing temperature at each residence time. Because a theoretical relationship with temperature is not known, several correlations were developed to provide the best fit of the data; the following were selected:

$$\tau_o = 2.330 - 6.13 \times 10^{-3}\, T + 4.04 \times 10^{-6}\, T^2 \tag{3}$$

$$\eta_{pl} = 0.0465 - 1.234 \times 10^{-4}\, T + 8.247 \times 10^{-8}\, T^2 \tag{4}$$

where T is in units of degrees Rankine, τ_o is in lb_f/ft^2, and η_{pl} is in $lb_f\text{-}sec/ft^2$. These equations should not be used to extrapolate outside the temperature range of 285-425°F, since it is known that other physical phenomena, such as coal swelling, may be occurring. Figures 9 and 10 plot the values of τ_o and η_{pl} as a function of temperature.

Combining equations 1 and 2 leads to an expression for the apparent viscosity based upon the Bingham plastic model:

$$\mu_{APP} = (\frac{\tau_o}{S} + \eta_{Pl})\ (4.79 \times 10^4) \qquad (5)$$

where the factor, 4.79×10^4, converts the units of viscosity from $lb_f\text{-sec/ft}^2$ to centipoise (cP). When the empirical relationships defined in equations 3 and 4 are used to generate values of τ_o and η_{Pl}, respectively, equation 5 reduces to a relatively simple function of shear rate and temperature to define the apparent viscosity of the coal feed slurry.

Figure 11 plots apparent viscosity vs. shear rate at various temperatures from the smoothed data of equations 3, 4, and 5. As expected, highly non-Newtonian behavior is evident by the shear-thinning behavior at low shear rates. The asymptotic approach at very low shear rates is characteristic of Bingham plastic materials, since a finite yield stress is required to initiate flow. Mathematically, this indicates that the slope of the viscosity curve is infinite at a shear rate of zero, since the shear stress-shear rate curves do not pass through the origin. At high shear rates, the apparent viscosity becomes independent of shear and thus can be treated as a Newtonian fluid.

APPLICATION TO THE DEMONSTRATION PLANT DESIGN

This study has identified the non-Newtonian characteristics of coal feed slurry over the temperature range relevant to the slurry mix system design. Potential problems are associated with low shear rates, which may occur during an unplanned shutdown or when there is a distribution of shear, such as in the slurry blend tank. At shear rates above approximately 300 sec^{-1}, the data indicate essentially Newtonian behavior, and viscosity is a function of temperature alone.

Based upon this study, a maximum apparent viscosity value of 95 cP is recommended to design the pumps and piping system downstream of the slurry mix tanks. (This recommendation was made in 1980. In the Baseline Design, Catalytic used 23 cP based on low-temperature Wilsonville data and Newtonian behavior assumptions; see SRC-I Baseline Design Technical Data Book, February 1984, DOE/OR/03054-23.) This corresponds to the maximum viscosity determined at temperatures up to 420°F for shear rates above 300 sec^{-1}.

Table 1

General Conditions for Slurry Mixing Tests

Coal type	Ky #9 & 14
Grind	70 ± 1% through 200 mesh
Solvent IBP (°F)	430
Solvent/coal ratio	(1.60 ± 0.03)/1
Temperature (°F)	285-425
Residence time (min)	14-41

Table 2

Bingham Plastic Parameters

Curve[a]	T (°F)	θ (min)	τ_o ($lb_f/ft^2 \times 10^{-2}$)	η_{Pl} ($lb_f\text{-sec}/ft^2 \times 10^{-4}$)
1	351	13	1.18	7.44
2	400	14	5.41	13.9
3	285	26	0.76	3.06
4	352	27	1.48	5.13
5	397	27	4.01	12.2
6	359	41	1.73	4.67
7	399	41	5.50	15.3
8	425	41	6.17	16.7

[a] As depicted in Figures 1 through 8.

Scheme I

Slurry Mix System Flow Schematic

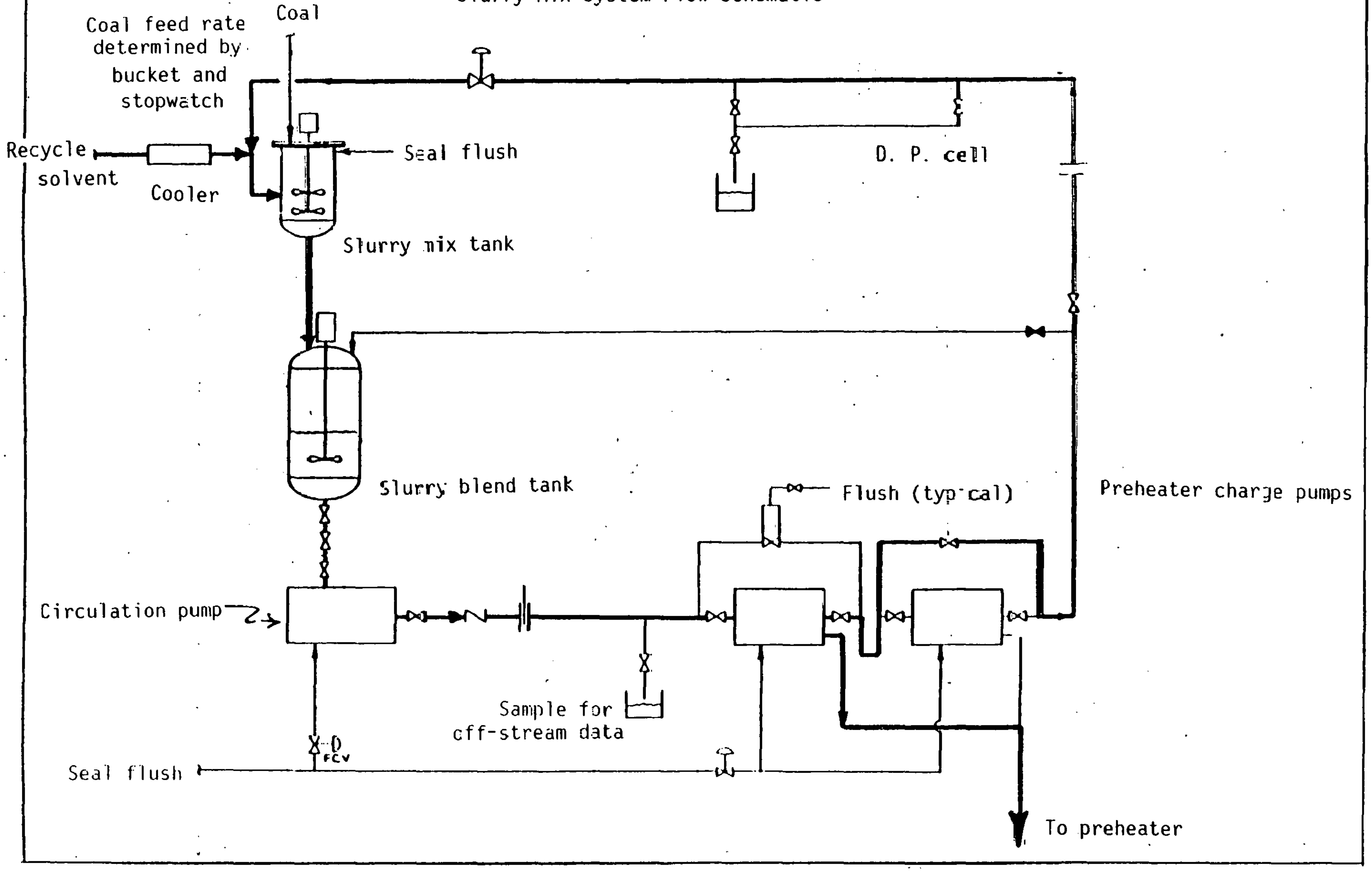


Figure 1
Viscosity vs. Shear Rate with Varying Temperature and Constant Low Residence Time (θ = 14 min)

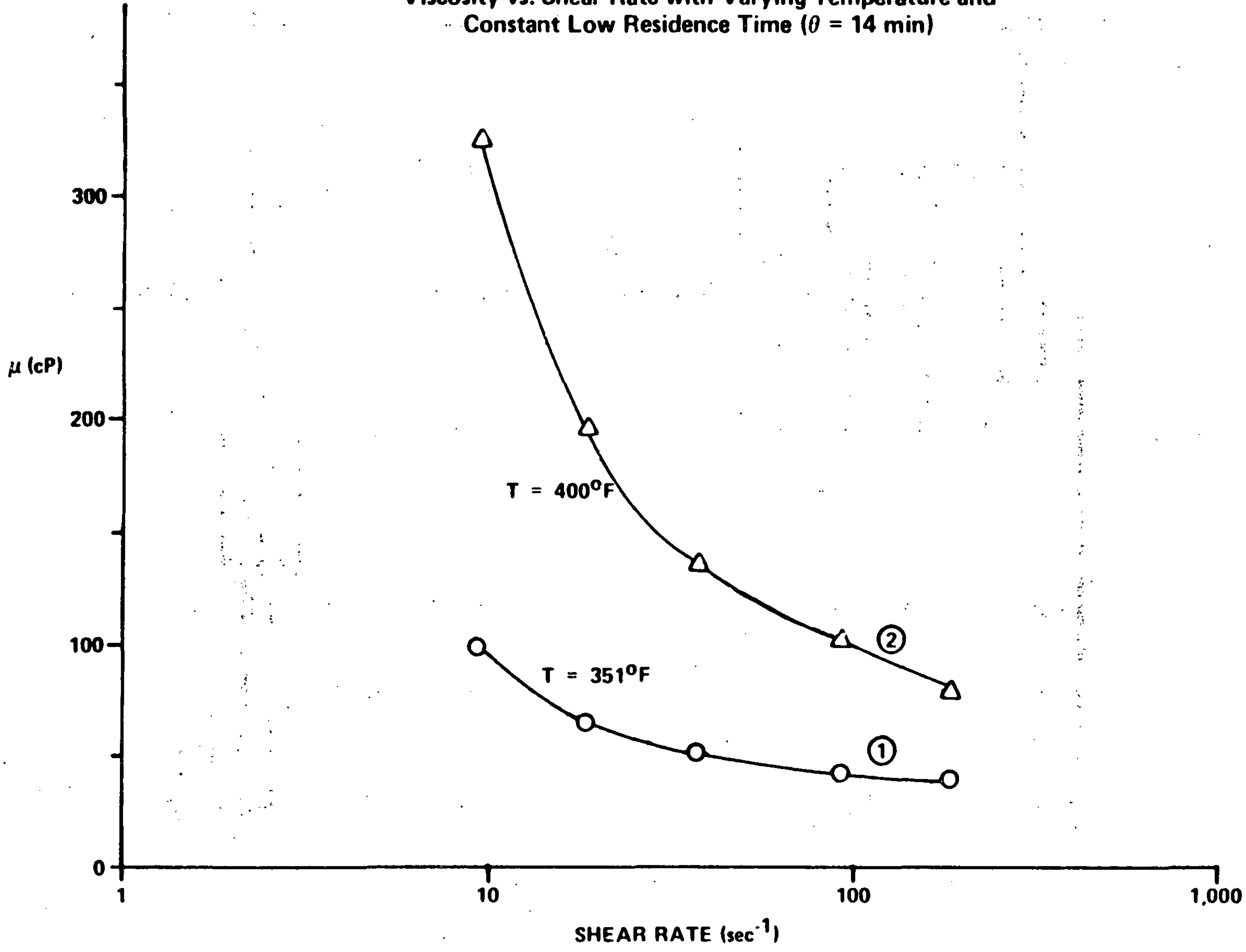


Figure 2

Viscosity vs. Shear Rate with Varying Temperature and Constant Intermediate Residence Time (θ = 27 min)

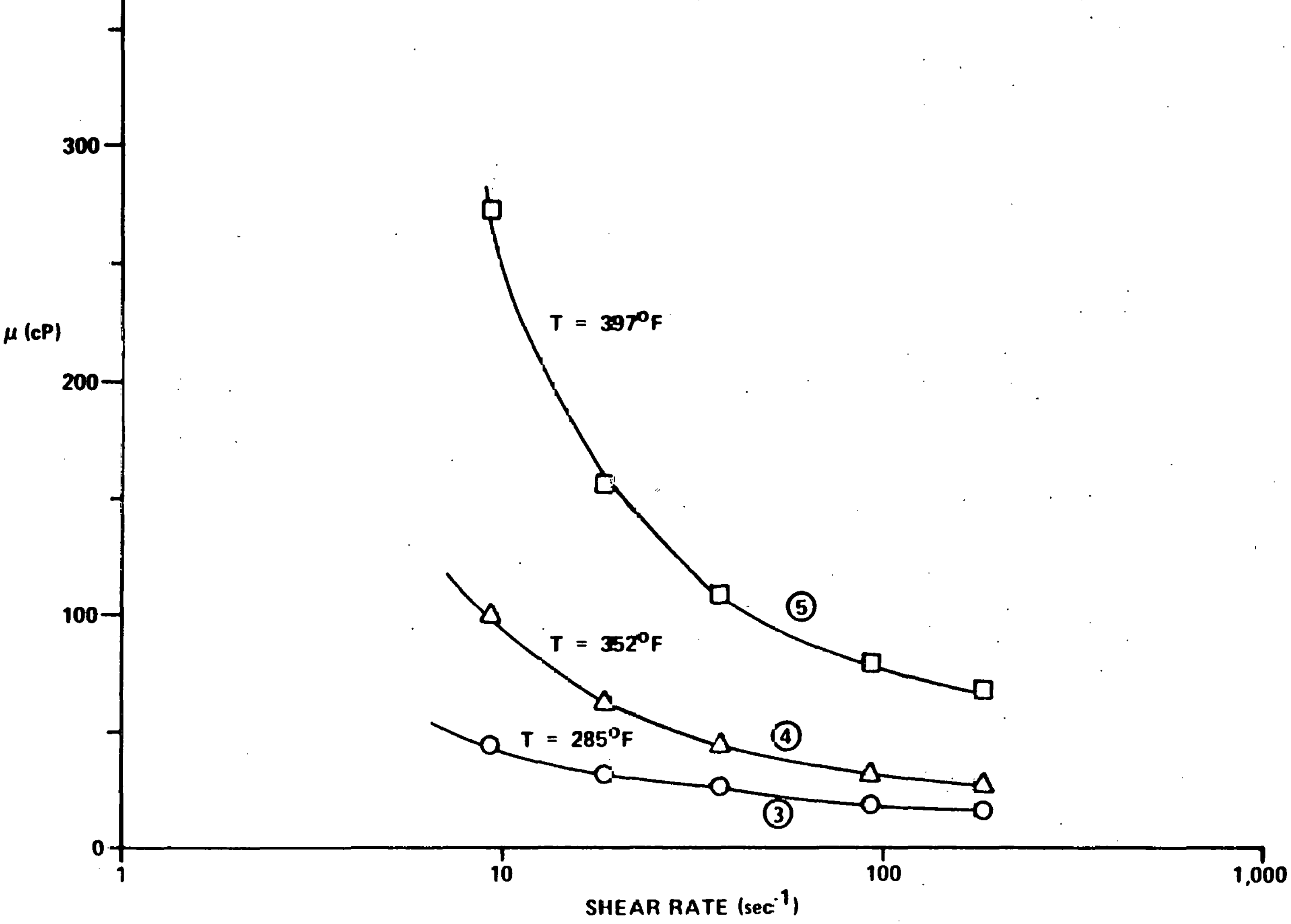


Figure 3
Viscosity vs. Shear Rate with Varying Temperature and Constant High Residence Time (θ = 41 min)

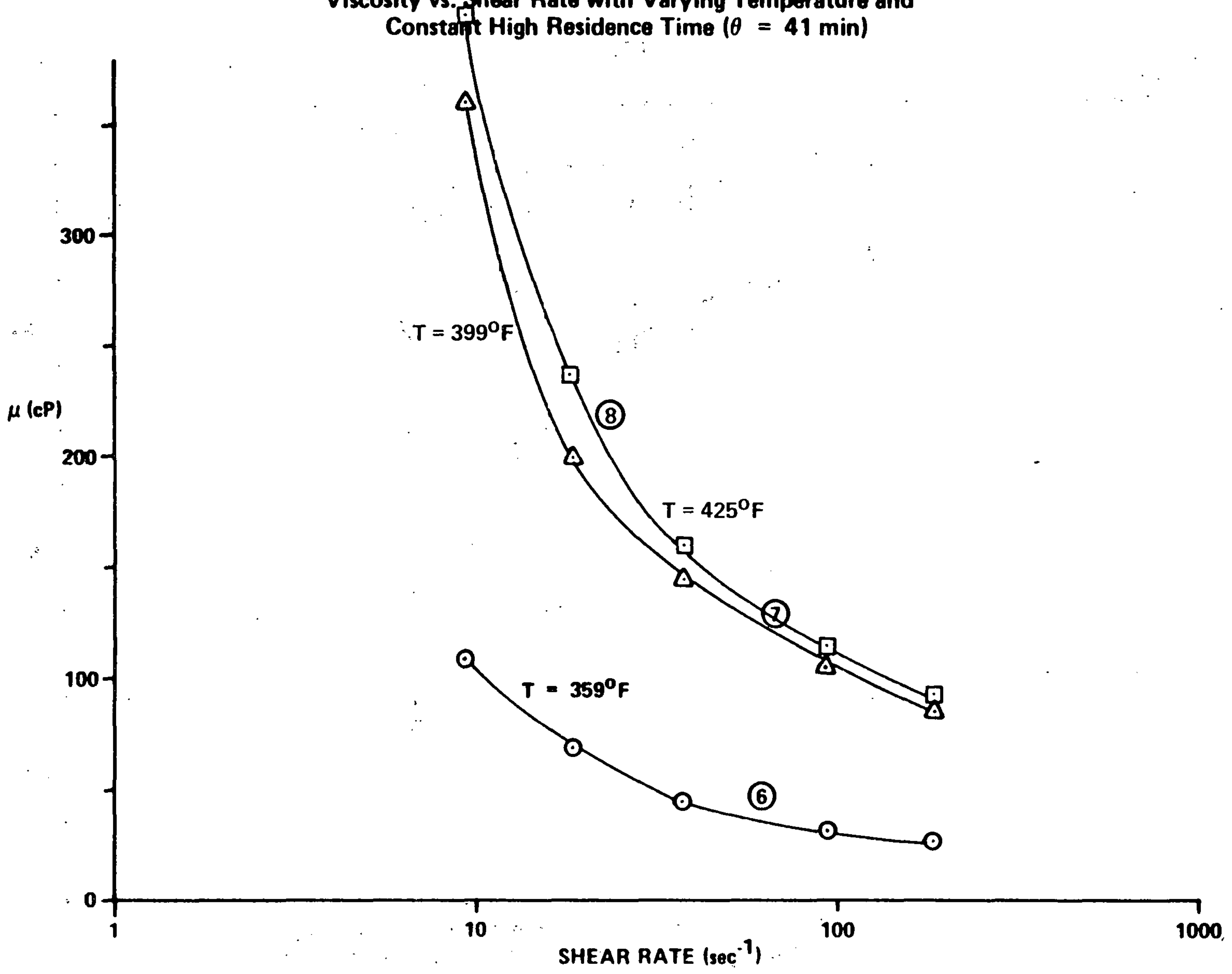


Figure 4
Viscosity vs. Shear Rate with Varying Residence Time and Constant Low Temperature (T = 354°F Approximate Average)

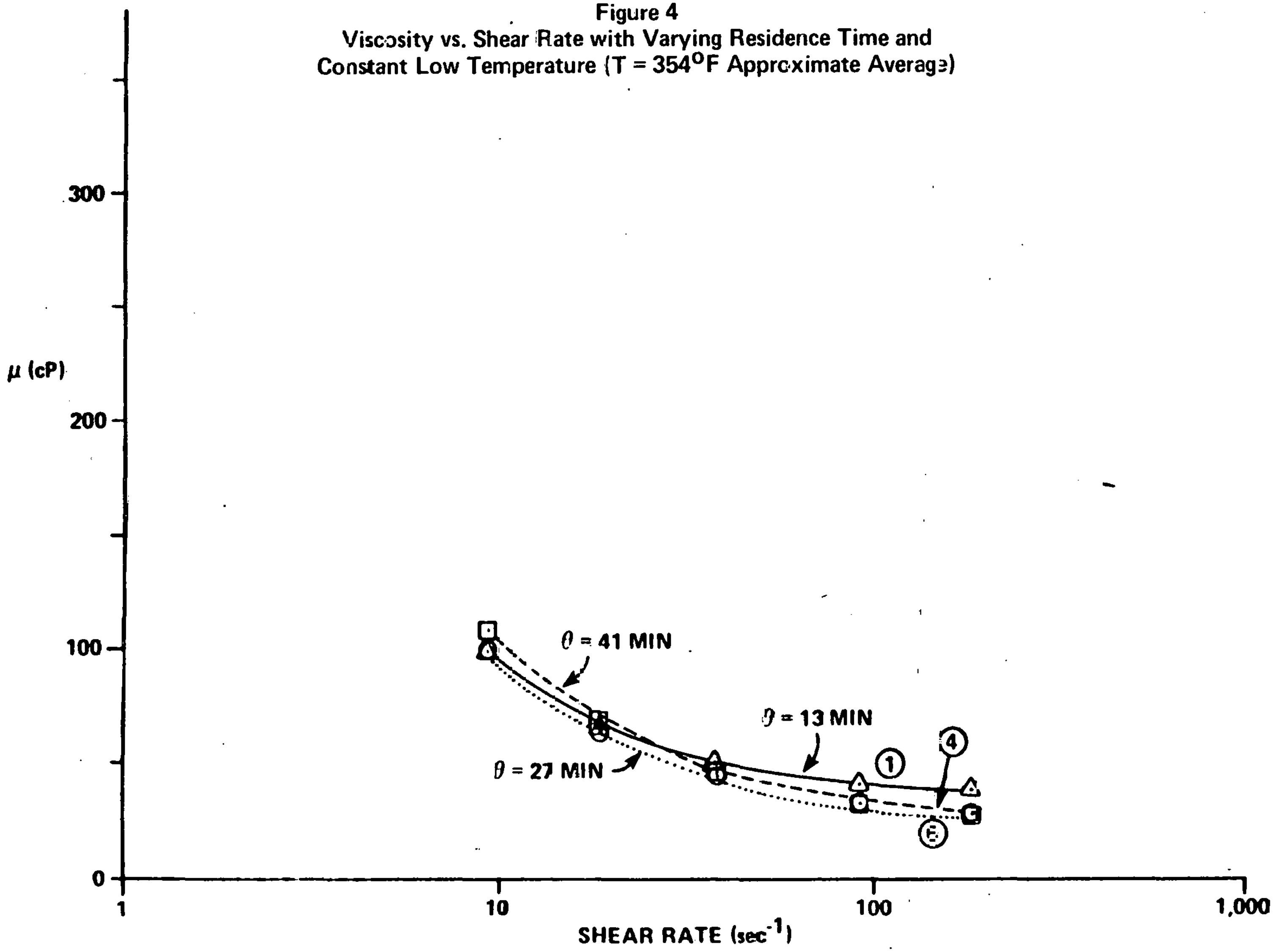


Figure 5
Viscosity vs. Shear Rate with Varying Residence Time and
Constant Intermediate Temperature (T = 400°F Approximate Average)

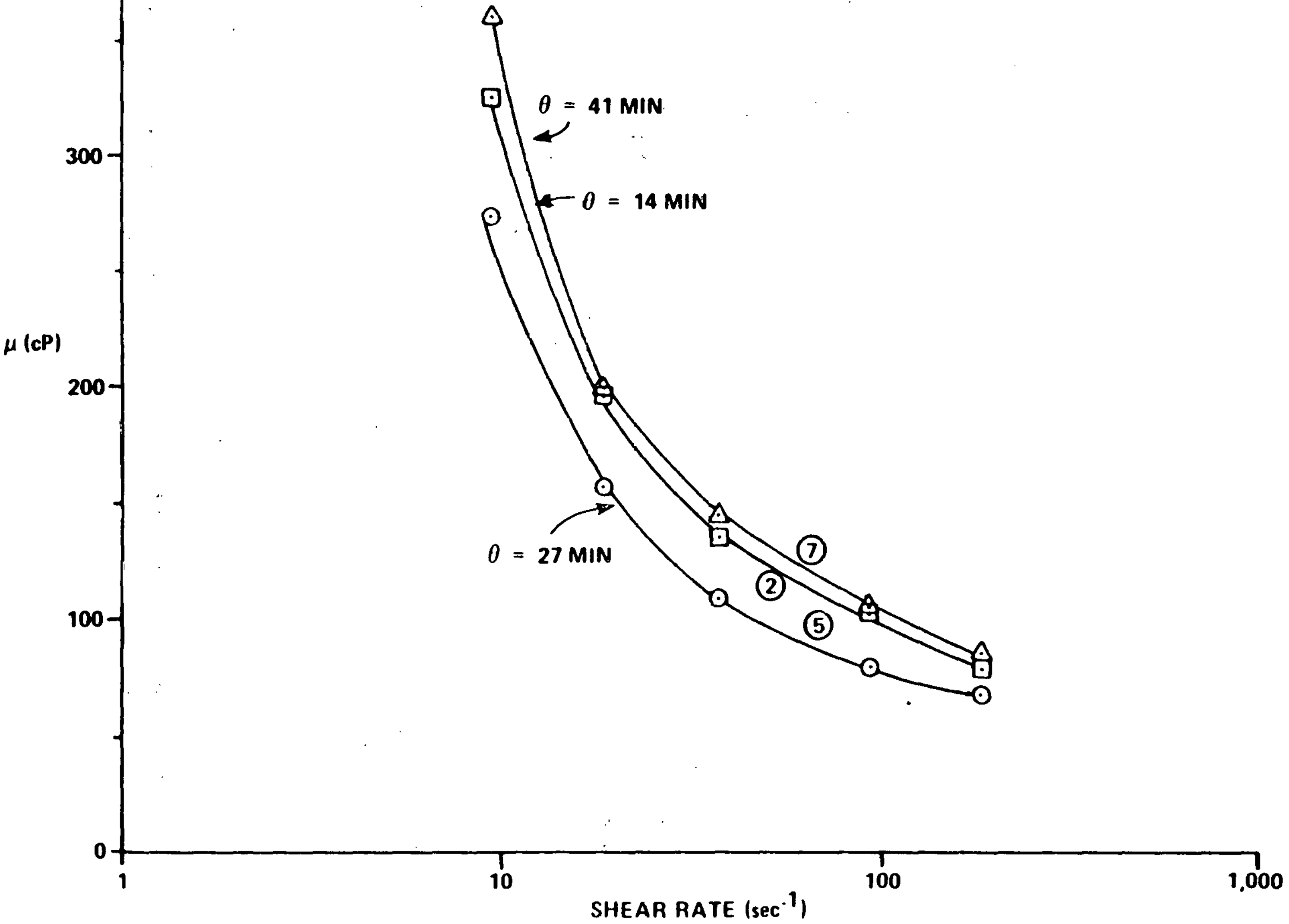


Figure 6
Shear Stress vs. Shear Rate with Varying Temperature and Constant Low Residence Time (θ = 14 min)

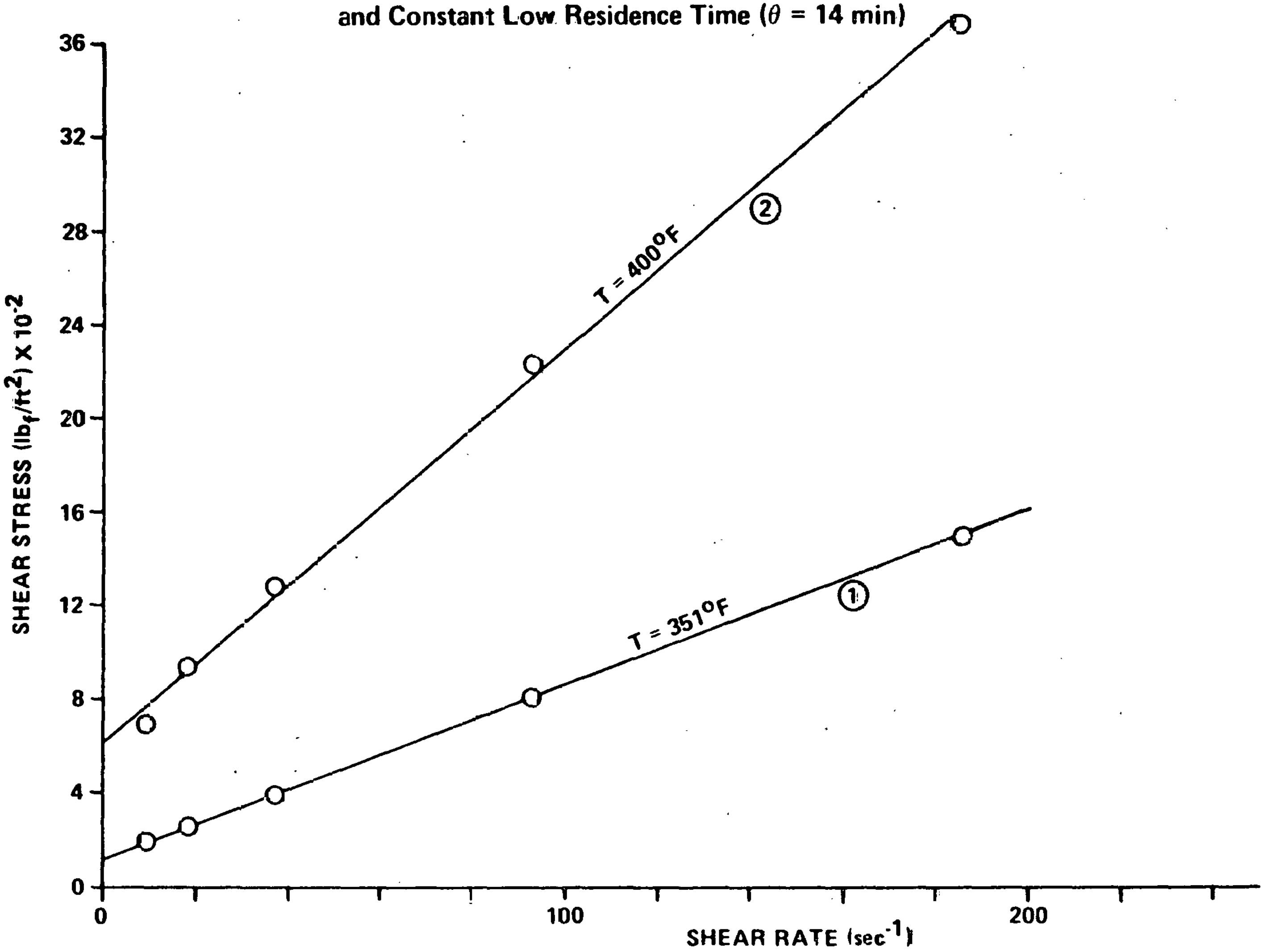


Figure 7
Shear Stress vs. Shear Rate with Varying Temperature
and Constant Intermediate Residence Time (θ = 27 min)

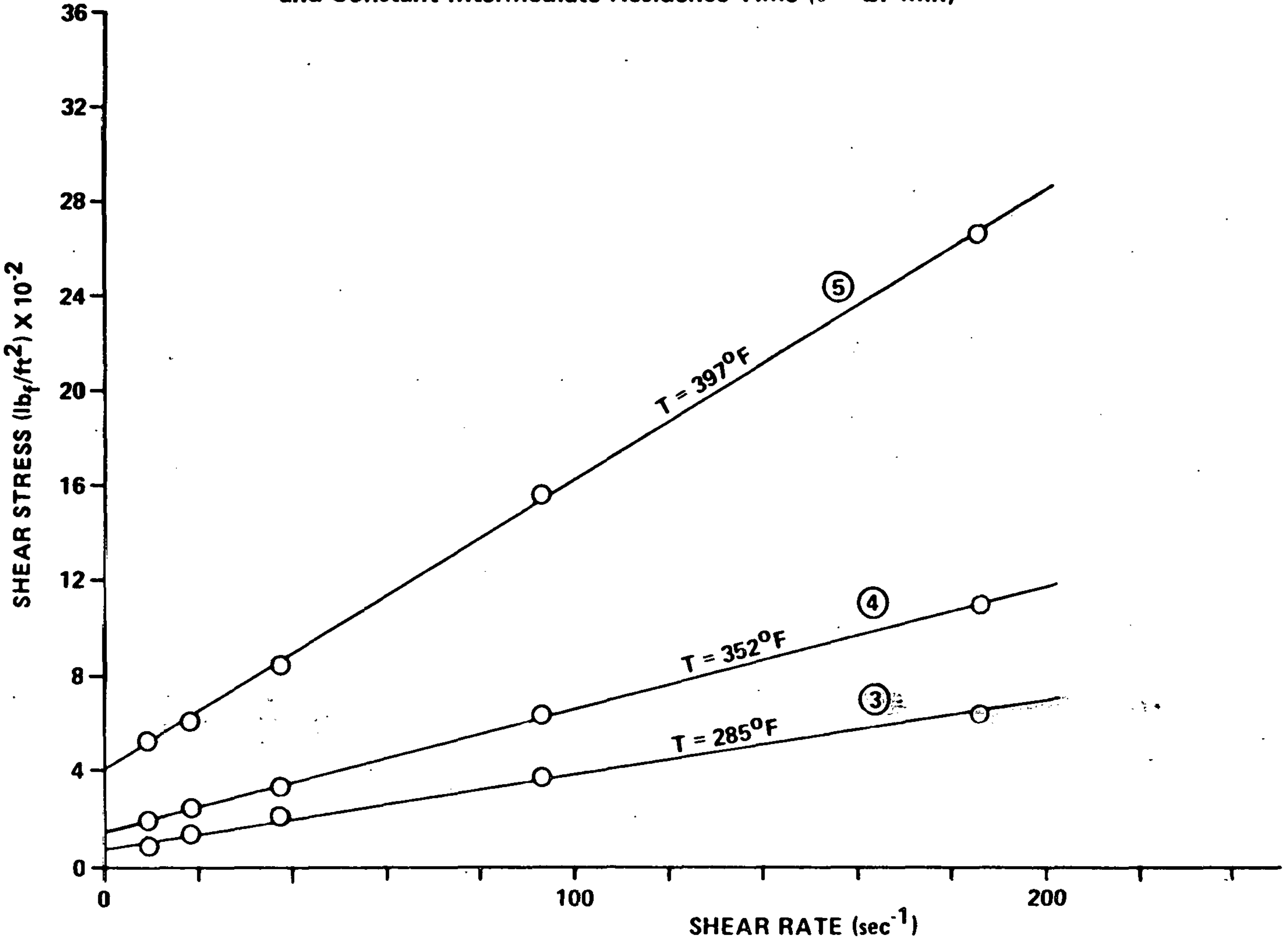


Figure 8
Shear Stress vs. Shear Rate with Varying Temperature and Constant High Residence Time (θ = 41 min)

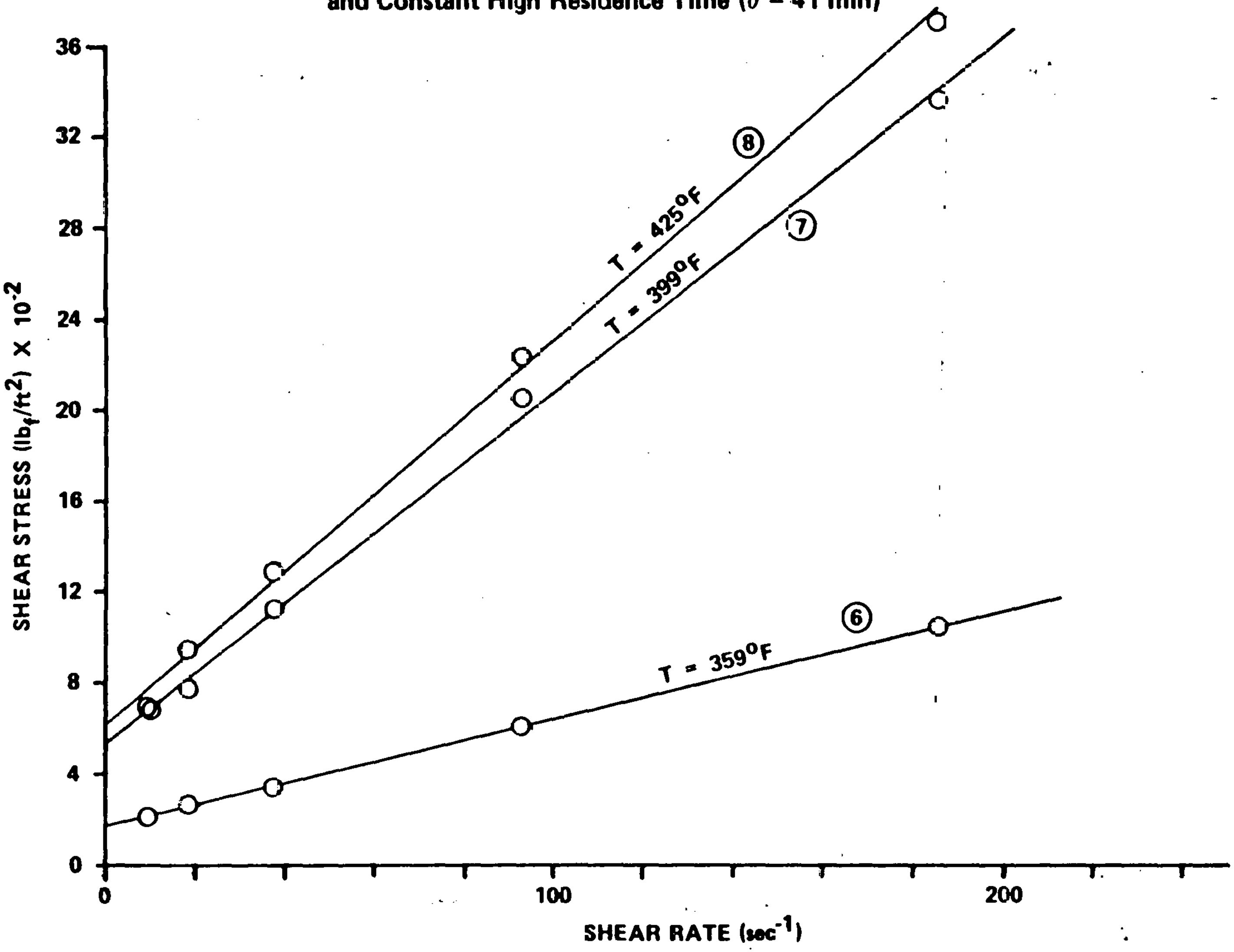


Figure 9
Plastic Viscosity vs. Temperature

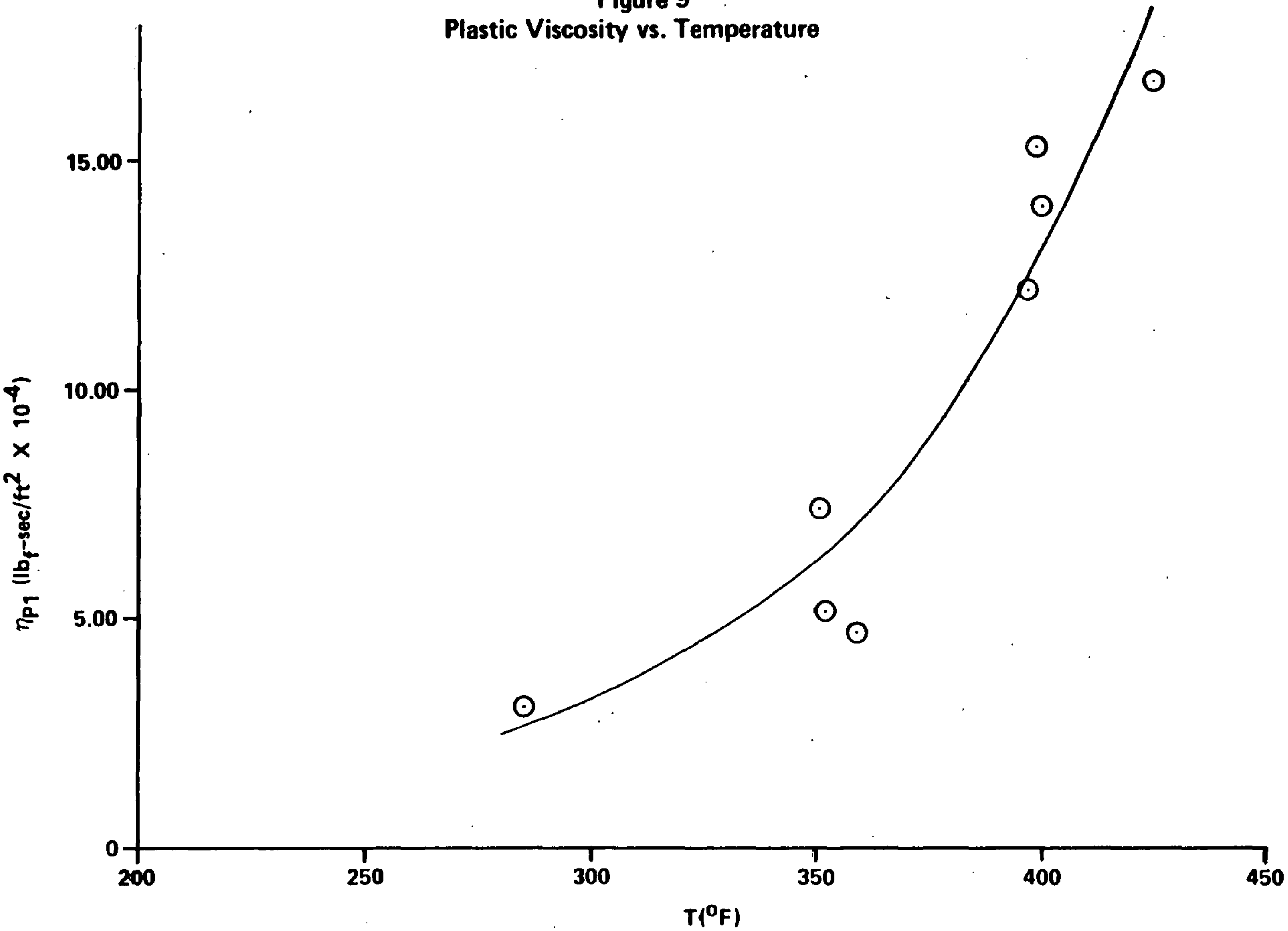


Figure 10
Yield Stress vs. Temperature

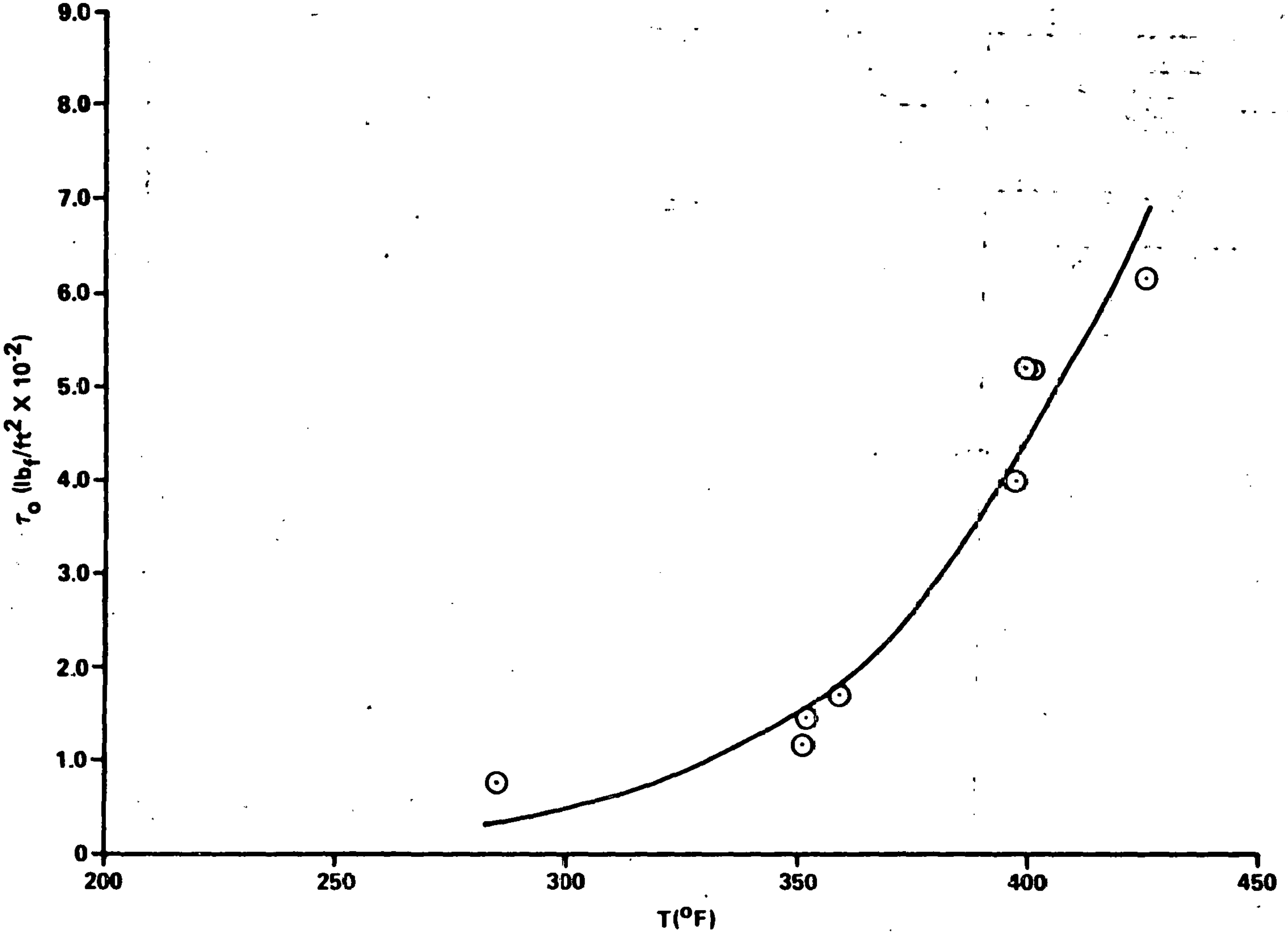
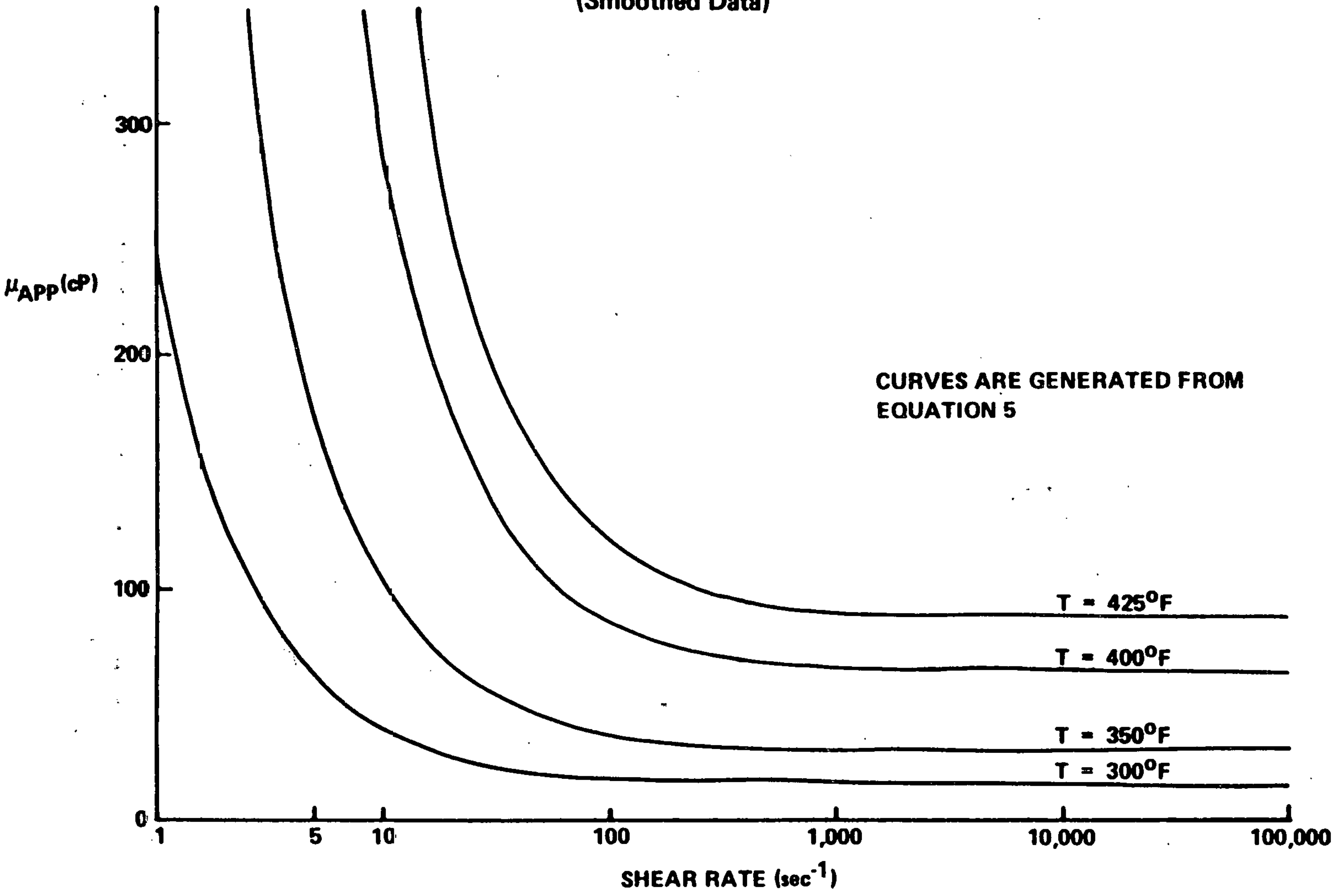


Figure 11
Viscosity vs. Shear Rate
(Smoothed Data)



CURVES ARE GENERATED FROM EQUATION 5

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The following two reports, "Chemical Cleaning of Coal: The Catalytic Heap Leaching Process" and "Chemical and Physical Cleaning of Coal. Part I. An Initial Review of the Literature on the Chemical Cleaning of Coal," were written in 1976 by Air Products and Chemicals, Inc. They are being published in this Technical Report as part of the "Transfer of Existing Technology" activity under Modification 31 to the SRC-I Contract at DOE's request, because of their historical significance to the SRC-I Project.

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CHEMICAL CLEANING OF COAL: THE CATALYTIC HEAP LEACHING PROCESS

J. B. Wetherington*

An evaluation of the proposed Catalytic Heap Leaching Process (CHLP) for pyrite removal from coal has been completed. Two conclusions were reached based upon a literature investigation and laboratory work designed to answer specific questions crucial to the process. First, based upon a review of the literature and discussions with persons knowledgeable in the field, no evidence has been found to support the theory that bacteria catalyze the actual oxidation of pyrite. It is well established that bacteria do catalyze the oxidation of ferrous ion to ferric ion, the latter being the primary oxidizing agent of pyrite in the proposed process. Therefore, there is no requirement for bacterial growth in the coal stack. Secondly, based upon laboratory work, substantial pyrite removal cannot be achieved using 3/4- to 3/64-in. coal at ambient temperature in five days. Only at elevated temperatures (>100°C) and for small-particle coal can the design specifications of 95% pyrite removal be met. These findings are supported by the literature.

In the author's opinion, the answers obtained are sufficiently definitive to preclude further laboratory work on this specific process.

THE PROCESS

The CHLP, as proposed by F. K. Kodras of Catalytic, Inc., Charlotte, N.C., consists of the treatment of large stacks of coal (10,000 tons per day) with an aqueous solution of ferric sulfate. Pyrite present in the coal is converted to ferrous ion, sulfate ion, and

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*Corporate Research, Air Products and Chemicals, Inc., Trexlertown, Pa.

elemental sulfur. Acid and additional ferrous ion are also products. The process would produce an environmentally acceptable boiler fuel when applied to coals low in organic sulfur. Relevant chemical reactions are given below:

$$FES_{2(c)} + 14Fe^{3+}_{(aq)} + 8H_2O \rightarrow 15Fe^{2+}_{(aq)} + 16H^{+}_{(aq)} + 2SO_4^{2-} \quad (1)$$

$$FeS_{2(c)} + 2Fe^{3+}_{(aq)} \rightarrow 3Fe^{2+}_{(aq)} + 2S_{(c)} \quad (2)$$

$$S_{(c)} + 6Fe^{3+}_{(aq)} + 4H_2O \rightarrow 6Fe^{2+}_{(aq)} + 8H^{+}_{(aq)} + SO_4^{2-}{}_{(aq)} \quad (3)$$

$$4Fe^{2+}_{(aq)} + O_2 + 4H^{+}_{(aq)} \rightarrow 4Fe^{3+}_{(aq)} + 2H_2O \quad (4)$$

The leaching would be carried out above ground at near-ambient conditions in the presence of iron-oxidizing bacteria. As proposed, the bacteria would serve to catalyze both the ferric ion oxidation of pyrite and the regeneration of ferric ion from the ferrous state. Conditions promoting bacterial growth would be maintained in the coal stack and in a separate bed where most of the ferric ion regeneration would take place. Included in the proposed system are provisions for coal-handling, bacterial life support, and waste treatment.

The design and economic considerations of the process are based on 95% pyrite removal from 3/4- to 3/64-in. lump coal containing approximately 3.5% pyritic sulfur and 0.7% organic sulfur following a five-day leach period. The proposed process is said to be derived from a combination of technologies from the TRW Meyers Process, heap leaching of ores, and acid mine drainage.

QUESTIONS

Evaluation of the process included consideration of the following questions.

(1) Do iron-oxidizing bacteria directly catalyze the oxidation of pyrite, or do they do so indirectly by catalyzing the regeneration of ferric ion, thereby maintaining a high concentration of the primary oxidizing agent for pyrite?

A literature review and discussions with those knowledgeable in the field produced no evidence to support the direct catalysis mechanism. It was felt that, for the purpose of further evaluation, this mechanism should no longer be considered.

Following a meeting with F. D. Kodras on 23 March 1976, three other questions crucial in assessment of the viability of the process were posed (W. A. Hart to D. G. Manly, 26 March 1976):

(2) Can coal 3/4-in. x 3/64-in. be successfully leached with an aqueous ferric sulfate solution to remove 95% of the pyritic sulfur present?

(3) Under the most favorable conditions of temperature and ferric ion concentration, what is the rate of pyritic sulfur removal?

(4) Can the spent leachant be regenerated practically by a biological oxidation process?

An answer to question 4 was required only if the answers to questions 2 and 3 were favorable to the proposed process. A set of laboratory experiments designed to answer these questions was begun, results of which are reported in the following section.

The question of patentability of the process has been discussed in a memo from R. L. Brewer to F. D. Kodras, 3 May 1976.

EXPERIMENTAL

Leaching experiments were conducted on Illinois #6 and Kentucky #9-14 (mixture) coals having the ultimate analyses shown in Table 1. Fifty grams of each coal, as received, was added to 3.0 L of 1.0 N Fe^{3+} (sulfate) solution and stirred at ambient conditions for 6 days. A

Table 1

Ultimate Analyses of Coals (as Received) Used for Ferric Sulfate Leaching Experiments

Coal	Illinois #6	Kentucky #9-14
Sample no	2120-31-5	2120-31-8
Size	-7/16 in., +5/16 in.	-80, +100
Carbon (wt %)	65.42	67.20
Hydrogen (wt %)	4.78	4.60
Nitrogen (wt %)	1.28	1.35
Oxygen (wt %)	12.19	11.39
Sulfur, total (wt %)	4.34[a]	3.38[b]
Sulfur,[c] pyritic (wt %)	0.95	1.63
Sulfur,[c] sulfate (wt %)	0.14	0.55
Sulfur,[c] organic (wt %)	3.25	1.20
Ash (wt %)	10.09	11.03
Total (wt %)	98.10	98.95
Moisture (wt %)	5.12	3.40

[a] ASTM D 3177, Eschka method, Galbraith Labs.
[b] ASTM D 3177, Bomb method, Linwood (APCI) Labs.
[c] ASTM D 2492, Linwood (APCI) Labs.

second set of 50.0 g of each coal was refluxed for 6 days in 3.0 L of 1.0 N Fe^{3+} (sulfate) solution.**

The amount of Fe^{3+} ion added represents a 9.6- and 5.6-fold excess of that required for complete pyrite removal based upon the stoichiometry of equation 1 for the Illinois #6 and Kentucky #9-14 coals, respectively.

Yellow crystals subsequently shown to be elemental sulfur were observed in the condensers of the two reactions undergoing reflux, confirming a contribution from equation 2.

At the end of the leaching period, a modified version of the acid wash/toluene extraction procedure described by TRW was employed. The reaction mixtures were filtered, and the treated coals were washed with 50 mL of distilled water and then slurried with 2 L of 1.0 N sulfuric acid at 70-80°C for 2 hr. Following filtration, the treated coals were again slurried with 2 L of 1.0 N sulfuric acid at 70-80°C for 2 hr, filtered, and allowed to stand overnight in 2 L of distilled water. The following day, the mixture was heated at 70-80°C for 2 hr and filtered.

The wet coal was then transferred to a 1-L round bottomed flask equipped with a magnetic stirrer and Dean-Stark trap. Four hundred milliliters of toluene was added and the mixture was refluxed. After 125 mL of toluene and toluene/water azeotrope was removed by distillation, the mixture was filtered and the coals were washed with 100-150 mL of toluene. The treated coals were dried for 16 hr at 110°C. In this procedure, toluene serves to remove water from the coal and as a solvent wash to remove from the coal any elemental sulfur formed in the leaching. Ultimate analyses of the starting coals and treated products from the four leaching experiments described above are shown in Table 2. Changes in coal composition due to leaching are shown in Table 3.

The following may be concluded from these data:

(1) Only at reflux temperature or for small-particle coal is there appreciable pyrite removal in five days. Only at reflux tem-

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**199.94 g of $Fe_2(SO_4)_3 \cdot nH_2O$ and distilled water to make 3.0 L of solution. The resulting solution is somewhat less than 1.0 N in Fe^{3+}.

Table 2

Ultimate Analyses of Coal (Dry Basis) before and after Ferric Sulfate Leaching

Coal	Illinois #6	Illinois #6	Kentucky #9-14	Kentucky #9-14
Size	-7/16 in., +5/16 in.	-7/16 in., +5/16 in.	-80, +100	-80, +100
Conditions	Ambient	Reflux	Ambient	Reflux
<u>Starting coal</u>				
Sample no.	2120-31-5	2120-31-5	2120-31-8	2120-31-8
Mass (g)	47.44	47.44	48.30	48.30
Carbon (wt %)	68.95	68.95	69.56	69.56
Hydrogen (wt %)	4.43	4.43	4.37	4.37
Nitrogen (wt %)	1.35	1.35	1.40	1.40
Oxygen (wt %)	8.05	8.05	8.67	8.67
Sulfur (wt %)	4.57[a]	4.57[a]	3.50[b]	3.50[b]
Sulfur,[c] pyritic (wt %)	1.00	1.00	1.69	1.69
Sulfur,[c] sulfate (wt %)	0.15	0.15	0.57	0.57
Sulfur,[c] organic (wt %)	3.42	3.42	1.24	1.24
Ash (wt %)	10.63	10.63	11.42	11.42
Total (wt %)	97.98	97.98	98.92	98.92
<u>Treated Coal</u>				
Sample no.	2120-32-2	2120-32-1	2120-32-4	2120-32-3
Mass (g)	44.8	45.3	43.4	46.1
Carbon (wt %)	70.23	71.16	73.39	65.54
Hydrogen (wt %)	4.75	4.83	4.65	4.12
Nitrogen (wt %)	1.28	1.23	1.65	1.36
Oxygen (wt %)	10.58	9.90	9.06	12.84
Sulfur[b] (wt %)	4.49	4.17	2.29	1.70
Sulfur,[c] pyritic (wt %)	1.10	0.72	0.93	0.08
Sulfur,[c] sulfate (wt %)	0.18	0.25	0.20	0.34
Sulfur,[c] organic (wt %)	3.21	3.20	1.16	1.28
Ash (wt %)	9.22	7.93	9.27	16.52
Total (wt %)	100.55	99.22	100.31	102.08

[a] ASTM D 3177, Eschka method, Galbraith Labs.
[b] ASTM D 3177, Bomb method, Linwood (APCI) Labs.
[c] ASTM D 2492, Linwood (APCI) Labs.

Table 3

Observed Changes in Coal Samples following a Six-Day Leaching in 1.0 N Fe^{3+} (Sulfate) Solution

Coal	Illinois #6	Illinois #6	Kentucky #9-14	Kentucky #9-14
Size	-7/16 in., +5/16 in.	-7/16 in., +5/16 in.	-80, +100	-80, +100
Conditions	Ambient	Reflux	Ambient	Reflux
<u>% Change</u>				
Mass	-5.56	-4.51	-10.14	-4.55
Carbon	1.86	3.21	5.51	-5.78
Hydrogen	7.22	9.03	6.41	-5.72
Nitrogen	-5.19	-8.89	17.86	-2.86
Oxygen	31.43	22.98	4.50	48.10
Sulfur	-1.75	-8.75	-34.57	-51.43
Sulfur, pyritic	10.00	-28.00	-44.97	-95.27
Sulfur, sulfate	20.00	66.67	-64.91	-40.35
Sulfur, organic	-6.14	6.43	-6.45	3.23
Ash	-13.26	-25.40	-18.83	44.66

peratures and for small-particle coal is the process design specification of 95% pyrite removal in five days achieved.

(2) Organic sulfur content is essentially unchanged by the leaching.

(3) The final sulfate sulfur content appears to be a function of the washing procedure.

(4) The dramatic increase in the ash content of the small-particle coal treated at reflux temperature was found to be due to precipitation of $Fe_2O_3 \cdot H_2O$ in the coal particles. An appreciable amount of the carbon content of this coal was lost under these conditions.

(5) The ash content of the coals generally decreased, with the exception of the small-particle coal treated at reflux temperature, as noted in (4) above.

(6) The oxygen contents of the coals were generally increased by the leaching.

CHEMICAL AND PHYSCIAL CLEANING OF COAL. PART I. AN INITIAL REVIEW OF THE LITERATURE ON THE CHEMICAL CLEANING OF COAL

R. R. Conrad, W. A. Hart, and
J. B. Wetherington*

INTRODUCTION

At the request of personnel in the Energy Systems Department, Corporate Research has carried out a comprehensive literature search on the chemical cleaning of coal covering the period 1927 to 1972. An initial review and evaluation of abstracts located during the search are complete. The results of the review are summarized in this paper.

Sulfur and other pollutants may be removed from coal before, during, or after the combustion process. Solvent refining, for example, removes sulfur and ash from the coal before combustion. Gasification of coal with intermediate removal of ash and sulfur values from the low-Btu gas is effectively cleaning during combustion. Flue-gas desulfurization removes sulfur values from the waste gases after combustion. Air Products has considered aspects of each of the above processes.

APCI is now interested in extending its knowledge into the areas of chemical and physical cleaning of coal, because these areas may provide a simpler, less expensive route to the production of clean boiler fuels.

Although considerable research is needed, the possibility of developing a viable proprietary process to remove ash and pyritic sulfur from coal seems to be good. However, it is unlikely that organic sulfur and nitrogen can be removed without extensive degradation of the coal. Therefore, the complete removal of all ash, sulfur, and nitrogen from the coal by simple chemical processing will be a difficult task requiring a greater research effort with less chance of success.

DISCUSSION

Scope of Literature Review and Survey

The literature reviewed in this paper covers the chemical cleaning of coal with various reagents for the period 1927 to 1972. References pertaining to the removal of nitrogen, sulfur, and ash from coal were sought for inclusion in this preliminary review. Although not specifically reviewed in this report, references appearing since 1972 have also been collected.

Pyrite and other minerals can be removed from coal by physical cleaning processes. These processes seek to remove pyrite and/or ash from coal using separation techniques based on differences in physical properties such as density and magnetic susceptibility. Most coal currently produced is subjected to at least some physical beneficiation to remove rock and metal scraps. The part of this literature review dealing with the physical cleaning of coal will be issued at a later date.

This survey of the literature did not directly include the chemistry of pyrite. This is a notable omission since the chemistry of pyrite is critical to its removal from coal. In addition, biochemical processes for coal cleaning have not been explicitly reviewed in this survey. Both of these topics will be reviewed in a subsequent report.

There are processes for chemically cleaning coal which are currently under development such as the Ledgemont process, the Meyers process, and the Batelle Hydrothermal Coal Process. Most of the information on these processes has been made available since 1972. Consequently, these processes are given little attention in this report. E. M. Phillips of MID is assembling a report on these and other specific processes for the cleaning of coal which will include the available information on chemistry, engineering, and economics.

Finally, it should be noted that most reagents which attack ash and/or pyrite also will react with the organic matter in the coal. Therefore, the action of chemical reagents on the organic matter of the coal is included as an important part of this review.

Outline of the Literature Review

Reagents which have been reported in the literature for the removal of sulfur, ash, and nitrogen from coal are outlined below. The same organization is used in the literature review in the next section of this report and in the list of abstracts and references in the final section of this paper.

A. Treatment with Oxidants

    1. Oxygen

        a. The Reaction of Oxygen with the Organic Components of Coal

            1) Oxidation of Dry Coal

            2) Oxidation of Coal in Slurries of Alkali Hydroxide

        b. Removal of Pyrite and Sulfur with Oxygen

    2. Hydrogen Peroxide

    3. Potassium Permanganate

4. Halogens
    a. Chlorination of Coal
    b. Bromination of Coal
5. Acids
    a. Treatment of Coal with Nitric Acid
    b. Treatment of Coal with Sulfuric Acid
6. Catalysts

B. Acids for Treating Coal
    1. Phosphoric Acid
    2. Hydrochloric and Hydrofluoric Acid

C. Bases for Treating Coal
    1. Sodium and Potassium Hydroxides
    2. Other Bases

D. Miscellaneous Reagents and Reactions

TREATMENT WITH OXIDANTS

Oxygen

Many references in the literature are concerned with the treatment of coal with molecular oxygen. Exclusive of papers on combustion, many papers discuss the oxidation of the organic portion of coal. A smaller number of references treat the removal of sulfur or pyrite from coal.

The Reaction of Oxygen with the Organic Components of Coal

Molecular oxygen reacts readily with the organic matter in coal. Much of the work on the interaction of coal and oxygen uses air as the reactant. Workers have studied many aspects of oxidation with oxygen including the kinetics and mechanisms of oxidation, the effects of oxidation on coking, and oxidation to yield polycarboxylic acids. Oxidation has been studied using dry coal and slurries of coal in aqueous alkali.

Oxidation of Dry Coal. At lower temperatures, coals of lower rank react readily with oxygen and increase in mass. Below about 250°C the aliphatic groups in coal are oxidized to yield -OH, $>$C=O, or -COOH groups. These reactions lead to changes in the nature of the coal such that caking is reduced or eliminated on subsequent heating of the oxidized coal. In extreme cases, the dry oxidation of some coals results in the formation of significant amounts of benzene polycarboxylic acids.

At or above 300°C, oxidation with molecular oxygen results in a decrease in the mass of coal concurrent with evolution of carbon monoxide and carbon dioxide.

Oxidation of Coal in Slurries of Alkali Hydroxide. The oxidation of the organic matter of coal can be carried out using oxygen in slurries of aqueous sodium or potassium hydroxides. Such reactions have been studied as a route to the formation of aryl polycarboxylic acids.

Removal of Pyrite and Sulfur with Oxygen

Oxidation with oxygen has been studied as a means of removing sulfur or pyrite from coal. Most of the references in this category concern the treatment of coal with air or air and steam at about 350 to 400°C to remove sulfur. Although the amount of sulfur in the coal can be reduced, the properties of the coal are significantly altered. Volatile matter is lost and some organic matter is oxidized. Furthermore, the ash content of the coal is not substantially altered.

Studies have been conducted on pyrite removal from coal by oxidation in an aqueous suspension. The Meyers and Ledgemont processes, which use oxygen to remove pyrite from coal, will be described in detail in a forthcoming engineering study by E. M. Phillips.

Hydrogen Peroxide

In comparison with papers on oxidation of coal with oxygen, there are relatively few treating the oxidation of coal with hydrogen peroxide. As in the case of oxygen, most papers refer to oxidation of the organic matter. Hydrogen peroxide oxidizes the organic portion of the coal to form polar groups, presumably -COOH. The solubility of the coal in aqueous sodium hydroxide is increased. However, the coking behavior of the coal is not significantly affected as in the cases of treatment with other oxidants and reagents.

A single reference states that hydrogen peroxide can be used to remove pyritic sulfur from the coal without substantially altering the coking characteristics of the coal.

It is of great importance to note that certain components of coal, notably iron compounds, cause catalytic decomposition of hydrogen peroxide.

Potassium Permanganate

Potassium permanganate has been studied as an oxidant for the organic matter in coal. Oxidation with permanganate occurs first on reactive peripheral groups yielding carbon dioxide and oxalic acid with little attack on the aromatic nuclei of the coal. Oxidations with permanganate are generally carried only to manganate ion by precipitation with barium ion rather than allowing the reactions to yield manganese dioxide.

Nitrogen in the coal can be recovered as ammonia and nitrate by exhaustive oxidation of coal with alkaline potassium permanganate.

Alkaline solutions of potassium permanganate are also used for hydrogenative degradation reactions of coal.

Halogens

Coal has been oxidized with both chlorine and bromine. Reactions with chlorine have been studied more thoroughly.

Chlorination of Coal

Chlorination of coal has been carried out dry and in aqueous and organic suspensions. The interaction of chlorine with both the organic and mineral matter of the coal has been studied.

The organic part of the coal is readily chlorinated with the uptake of substantial amounts of chlorine. Hydrogen chloride is formed, and the organic material is cleaved into the fundamental fused-ring systems believed to be a part of the basic structure of coal. The caking properties and elemental composition of the organic matter are severely altered. At higher temperatures and more rigorous conditions, coal can be burned in chlorine to yield carbon tetrachloride as a principal product.

The removal of ash by chlorination has also been studied. Dry chlorination of coal at about 1400°C removes iron, silicon, and titanium as volatile chlorides, but the coal is substantially altered by the process. Chlorination with aqueous chlorine is reported to remove most of the inorganic sulfur from the coal. However, as noted above, chlorine also reacts with the organic matter in the coal.

An increased rate of chlorine uptake by coal has been observed by ultrasonic stimulation of the reaction mixture.

Bromination of Coal

Bromination of the organic matter of coal has been studied using, principally, bromine in a solution of carbon tetrachloride. The uptake of bromine can be significant. No comments on the ability of bromine to remove ash were located.

Bromination is also influenced by ultrasonic stimulation.

Acids

One obvious means of removing ash from coal is to treat the coal with acid. However, both nitric and sulfuric acids act as oxidants as well as acids. Such side reactions complicate the use of these acids for chemical cleaning of coal.

Treatment of Coal with Nitric Acid

Most of the work on the reaction of nitric acid with coal has been related to the oxidation and nitration of the organic matter to gain insights regarding the structure of coal and to form humic and nitrohumic acids. Some work has been done in an effort to remove pyrite and sulfur from coal.

The reaction of nitric acid with coal results in oxidation and nitration of the organic matter. Carboxylic acid and nitro groups are introduced into the coal structure, and carbon dioxide and nitrogen oxides are formed as by-products. Rigorous treatment with nitric acid can result in a substantial degradation of the organic coal structure to aryl subunits. On treatment with nitric acid, coal can yield humic and nitrohumic acids.

Several articles discuss the removal of sulfur from the coal. Pyrite and other components of the ash can be dissolved with nitric acid. As with other oxidants, the sulfur in pyrite is oxidized to both elemental sulfur and sulfate. The extent of removal of the pyritic sulfur depends on the rank and nature of the coal and can be nearly complete. However, during the reaction to remove pyrite, nitrogen is incorporated into the coal and the chemical nature of the coal is changed.

The literature implies that nitrogen oxides formed in the treatment of coal with nitric acid can be recycled. However, no references described the use of nitrogen oxides to specifically attack pyrite.

Treatment of Coal with Sulfuric Acid

The references in the literature relate to the interaction of sulfuric acid with the organic matter in the coal.

Depending on the concentration of the acid and the reaction conditions, sulfuric acid can sulfonate, oxidize, and/or hydrolyze coal. The residue from the treatment with more concentrated acid has a decreased content of carbon, hydrogen, and ash, about the same concentration of nitrogen, and an increased content of sulfur and, presumably, oxygen. Oxidation results in the formation of -OH and -COOH groups in the coal and carbon dioxide. Sulfonation results in the addition of sulfonic acid groups. The ion exchange capacity of coal can be increased by treating with sulfuric acid.

Treatment of coal with sulfuric acid results in a decrease in the content of volatile matter and a loss of caking properties of the coal.

None of the references are directly concerned with the removal of pollutants using sulfuric acid as the treating reagent.

Catalysts

There is little information in the literature covered during this survey which deals with the catalytic oxidation of pyrite from coal. The only example is the Meyers process in which iron (III) ion is used to oxidize pyrite to sulfur and sulfate. Iron (II) ion which is formed during the reaction can be reoxidized to iron (III) and recycled. Other ions or molecular species might be used in a similar way.

ACIDS FOR TREATING COAL

The use of acids is inherently appealing for the removal of ash from coal. The use of either nitric or sulfuric acids, which act primarily as oxidants, is discussed above. Phosphoric acid and hydrohalic acids are discussed in this section.

Phosphoric Acid

Little information is available on the treatment of coal with phosphoric acid. It is known that treating coal with phosphoric acid decreases or eliminates the caking of the coal on heating.

Hydrochloric and Hydrofluoric Acids

Hydrochloric and/or hydrofluoric acids have been used to remove ash from coal. Though some of the work has been done for the analysis of coal, treatment with hydrohalic acid has been considered as the basis for industrial deashing of coal. Up to about 70 percent of the ash can be removed from brown coal using diluted hydrochloric acid. Hydrofluoric acid removes ash more effectively than hydrochloric acid, since hydrofluoric acid dissolves silicates. The degree of removal of pyrite and sulfur is not stated. Most of the references on the treating of coal with hydrohalic acids are old ones.

BASES FOR TREATING COAL

There are a fair number of references in the literature concerning the reaction of bases with coal. As usual, the papers treat the interaction of bases with both the organic and mineral portions of the coal. However, some papers treat the removal of sulfur and/or pyritic sulfur specifically.

Sodium and Potassium Hydroxides

Sodium and potassium hydroxides interact with both the organic and inorganic matter in the coal. These hydroxides have been reacted with coal separately and together, as melts, as aqueous solutions, and as organic solutions.

The mode of attack of aqueous solutions of alkali metal hydroxides on coal is primarily hydrolytic. Autoclaving coal with aqueous sodium hydroxide dissolves substantial amounts of the coal. Lower rank coals are more readily dissolved than higher rank coals.

Alcoholic potassium hydroxide, with or without pyridine, can be used to dissolve coal. Lower rank coals are, again, more readily dissolved.

Treatment of coal with alkali hydroxides removes ash from coal. Treating coal with aqueous sodium hydroxide at about 200°C is reported to remove ash but, curiously, not sulfur. Treatment of coal with molten caustic at 250°C is reported to remove pyrite. The pyrite is converted to polysulfides, sulfides, and sulfur. Intermittent introduction of steam to the reaction mixture aids the reaction. A similar reference reports the desulfurization of coal with a melt of these and other bases at temperatures up to 800°C. Molten hydroxides, oxides, or carbonates are said to remove sulfur from coal, coke, and oil. One strange reference states that sulfur can be removed from coal by milling with water, oil, and sodium hydroxide (or salts of weak acids).

Other Bases

Brown coal reacts with aqueous or gaseous ammonia with incorporation of nitrogen into the product.

Molten salt systems containing strong bases such as oxide or carbonate remove pyrite from coal.

Coals, treated with solutions of sodium carbonate or sodium chloride and dried, are reported to evolve less sulfur dioxide when burned.

MISCELLANEOUS REAGENTS AND REACTIONS

The references in this category may not fit well in other categories or may deserve additional emphasis because of the technique used.

A few references deal with the photochlorination of coal.

Phosphoric acid, hexafluorophosphoric acid, and o-chlorophenol are reported to extract organic sulfur from coal. This is doubtful.

The electrolysis of coal is reported to yield alkali soluble organic materials.

Refluxing an aqueous slurry of Raney nickel with coal is reported to remove sulfur. The mechanism is not reported.

A slightly different approach to the removal of pyrite involves converting the surface or the bulk of the pyrite to another material and carrying out a separation based on the change in properties. In one case, microwaves are used to heat the pyrite converting it to pyrrhotite. The material can then be separated more effectively by a magnetic process. Alternately, the coal is heated in steam and oxygen converting the surface of the pyrite to pyrrhotite. Again, magnetic separation is more effective with the treated coal.

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