FE-496-T13
Distribution Category UC-90d

SOLVENT REFINED COAL (SRC) PROCESS

DEVELOPMENT OF A PROCESS FOR PRODUCING AN ASHLESS, LOW-SULFUR FUEL FROM COAL

VOLUME III - Pilot Plant Development Work

Part 3 - Startup and Operation of the SRC Pilot Plant

RESEARCH AND DEVELOPMENT REPORT NO. 53

INTERIM REPORT NO. 22

Period of June 1974 - April 1977

The Pittsburg & Midway Coal Mining Co.
Merriam, Kansas 66202

May, 1977

PREPARED FOR THE UNITED STATES
ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION

Under Contract No. E(49-18)-496

NOTICE

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Price: Paper Copy $
Microfiche $

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ABSTRACT

This report summarizes the progress of the Fort Lewis, Washington Solvent Refined Coal (SRC) Pilot Plant during plant commissioning and the first two and one-half years of operation -- from June 1974 through April 15, 1977. The Pilot Plant is operated by The Pittsburg & Midway Coal Mining Co. for the Energy Research and Development Administration.

I. SUMMARY

A. Operational Phases

This report covers the activity at the Ft. Lewis SRC Pilot Plant over about a three year period from the time the various sections of the plant were accepted from the construction contractor in early 1974 until mid-April, 1977. Several different phases of operation were conducted during this period and each is discussed briefly below. The performance and problems encountered during this entire period of operation are reported in detail in subsequent sections.

1. Plant Commissioning

As sections of the plant were completed by the construction contractor between February, 1974 and September, 1974 and known deficiencies were corrected, P&M accepted them and proceeded with the commissioning operations which are discussed in Section II.

2. Preliminary Integrated Plant Operation

From October, 1974 through February, 1975, integrated operation of the entire plant was accomplished.

3. SRC-I Material Balance Runs

From February, 1975 to early July, 1975 the plant was operated with the filters bypassed so that reliable material balance data at various dissolver operating conditions could be attained in the minimum amount of time. The results of these tests are reported in Section IX.

4. SRC-II (Slurry Recycle) Operation

Following a short plant shutdown to make tie-ins to equipment which had been installed for preliminary slurry recycle or SRC-II operation, the plant was operated relatively smoothly in this mode from mid-July, 1975 to early September, 1975. The results of material balance runs completed during this period are reported in Section IX.

5. SRC-I Production Run

The period from early September, 1975 to the end of November, 1976 was devoted to a production run of solid SRC, primarily to produce 3,000 tons for a large scale burning test scheduled to be conducted under the sponsorship of ERDA in May, 1977, at Georgia Power Company's Plant Mitchell, near Albany, Georgia. Georgia Power Company is a unit of the Southern Company. The first 700 tons shipped analyzed 0.34 wt. % ash and 0.73 wt. % sulfur. The last 2300 tons shipped analyzed 0.16 wt % ash and 0.74 wt. % sulfur.

Many problems were encountered and solved during this time. Priority during this period was on production which limited the amount of research data obtained. However, due to the urgency to produce SRC as rapidly as possible, solving of process and mechanical problems could not be postponed but had to be approached in the same manner as similar problems in a commercial production facility. As a result, a great deal of progress was made toward defining process and mechanical problems and finding acceptable solutions to them. A major portion of the problems discussed in Section III were experienced during this period.

6. Filter Studies

December, 1976 and January, 1977 were spent testing the effect of several variables on filter performance. The result of this work is reported in Section IX.

7. SRC-II Modifications

During the period from February 1, 1977 to April 15, 1977 major modifications were completed to allow extensive investigations, over a fairly wide range of conditions, in the SRC-II or slurry recycle mode. These modifications are described in Section VII.

B. Major Progress and Accomplishments

During the period covered by this report, major progress was made in the further development of the SRC process on a pilot plant scale. Most of the advancements can be directly utilized in the design of future larger plants and thus major steps have been made toward the commercialization of both the SRC-I and the SRC-II processes. Among the accomplishments are:

1. Substantially improved the coal slurry high pressure pump operation. Increased period between maintenance repairs from less than one day to several months.

2. Demonstrated feasibility of decreasing solvent to coal ratio from the design of 3:1 to 1.25:1.

3. Demonstrated that the plant can be operated at design capacity utilizing only one-half the preheater coil area. This was accomplished by a combination of the reduction in solvent to coal ratio and the discovery that the process endothermic heat of reaction is substantially less than anticipated.

4. Demonstrated that satisfactory SRC-I results can be obtained at coal space rates as high as 75 lbs/hr-ft^3 by performing successful tests using one-fourth of the dissolver capacity provided.

5. Defined areas of serious erosion and made major progress in solving the problems by equipment and materials modifications.

6. Defined several areas of unexpected corrosion and collected data on causes and on improved materials of construction.

7. After making many engineering and mechanical improvements, have established that high pressure, high temperature, rotary precoat filters have the potential of performing acceptably for separation of mineral residue in commercial plants. However, if used, a substantial number of operators will be required and considerable maintenance downtime should be expected.

8. Made many improvements to the mineral residue drying system but its low capacity makes its commercial applicability questionable. Alternate methods of drying should be investigated.

9. Solved the serious and recurring coking problem in the vacuum flash system by major changes to the flow scheme and equipment in the entire distillation area of the plant.

10. Established a comprehensive health protection program.

11. SRC-I process studies have confirmed that solvent balance can be attained and have more clearly defined optimum conditions.

12. SRC-II process studies have confirmed previously obtained bench scale data, demonstrated the desirability of and the plant modifications required for a more comprehensive test program.

13. Greatly improved centrifugal pump performance by a comprehensive in-plant pump seal research and development program. However, erosion of centrifugal pumps handling unfiltered coal solutions remains a serious problem.

14. Identified areas of the process where unexpected dust and fume problems occur and greatly reduced the levels of contamination by the installation of recovery equipment.

15. Produced 3,000 tons of solid SRC meeting power plant fuel specifications for use in a large combustion test and supplied hundreds of other samples of SRC and intermediate streams for testing by others.

II. PLANT COMMISSIONING

A. Introduction

When sections of the plant were completed by the construction contractors, preliminary testing, such as electrical and instrument loop tests, pressure tests and mechanical checkout was performed and deficiencies corrected. The sections then were accepted from the contractor. During this time a detailed plan for commissioning and startup was undertaken. This plan included spare parts procurement.

As sections of the plant were accepted from the construction contractor, final testing and checkout was performed and the units placed in operation in a specific order.

B. Plant Staffing and Training

The SRC Pilot Plant organization was established initially during the latter phases of plant construction with five major departments-Operations, Engineering and Maintenance, Laboratory, Process Evaluation and Services. All departments reported directly to the Plant Manager. Key technical and administrative personnel were recruited six to nine months prior to acceptance of the first sections of the pilot plant and added to the staff over a three to six month period. Several were hired from outside sources. However, other segments of P & M's parent, the Gulf Oil Corporation, were relied on heavily as a source for experienced administrative, technical, supervisory and management personnel. In addition, they provided a substantial number of maintenance craftsmen and lead operators for the permanent complement. Further, eight experienced lead operators, two process engineers and numerous maintenance specialists (in crafts such as rotary equipment, process instrumentation, chromatography, etc.) from other Divisions of Gulf were assigned on a temporary basis for periods of two weeks to nine months during the actual plant startup. Many of the individuals assigned temporarily had previous experience in plant startup which proved to be quite valuable and contributed significantly to the overall startup effort.

The remainder of the plant staff was hired locally over a period of approximately three to six months with additions geared to mesh with the commissioning schedule. The total plant staff ranged from 150 to 170 during the plant startup.

The entire operating hourly staff, as well as selected laboratory technicians and mechanics, were divided into three groups and given training on process fundamentals, plant equipment and the SRC process by a department of the Singer Company which specializes in training of industrial personnel. The training sessions were held over a four month period and were subdivided into two sessions- one on fundamentals and the other on the plant and SRC process. Each was of five week's duration. Concurrent with operator training, the plant technical and supervisory staff was involved in planning, scheduling and preparation of detailed operating and maintenance procedures. As the operators completed their training schedule, they were phased into plant inspection, testing, and startup.

C. Procedures and General Plan

A general operating procedures manual and specific area or unit operating manuals were prepared. Each manual contained, at a minimum, the following procedures and information specific to each area, unit, or piece of equipment:

- Emergency Procedures
- Safety Procedures
- Fire Prevention
- Precommissioning Procedures
- Operating Procedures
- Instrument Lists with Control Settings
- Heat Tracing Schedule
- Freeze Protection Procedures
- Data Sheets

Additionally, equipment and lubrication manuals were assembled and made available to plant personnel. These contained detailed manufacturers' data, drawings, descriptions, procedures and information. They were subdivided by operating area. These manuals were distributed to the various departments and groups for their use during startup and subsequent plant operations.

The overall outline and plan for plant commissioning and startup consisted of numerous types of testing, checking and run-in and is presented in summary form below.

- Basic information such as P & I Diagrams, construction, electrical and instrument drawings, data sheets and vendor literature regarding design, performance, maintenance, operation, testing and code documents were collected and made readily available to all Engineering, Operations and Maintenance personnel.

- A final process piping, instrument, and mechanical check was completed by checking P & I D's against plant facilities and checking lines, equipment and instruments for process suitability, utility requirements, specification and code compliance.

- Electrical systems were tested and checked out against wiring diagrams for overload protection, electrical classification grounding and lockout devices. Simulated or actual conditional tests were made.

- The final instrument check, in summary included checks for condition, proper installation, set points, calibration, alarms, shutdowns, relief settings, operator action, proper response and operation.

- Mechanical inspection and run-in included inspection for proper installation, removal of debris, dryout if necessary, lubrication, adjustment, and a complete preparation for operation, followed by actual operation and final adjustment whenever possible. All vessels and tanks were opened, inspected and cleaned. Specific procedures and manufacturer's recommendations, including service if appropriate, were utilized for each piece of equipment.

- A thorough cleaning and flushing of all lines, vessels, and equipment was undertaken next. Any special chemical cleaning was performed during this step. All blinds were removed and units were made ready for operation by charging catalysts and chemicals and installing filter elements and temporary start-up devices such as pump strainers, etc.

- Final pressure and leak tests were performed after mechanical inspection and, with the exception of alterations which were tested to conform to the applicable code, these tests were generally limited to making reasonably certain that flanges, fittings, packing, etc. were properly made up before being exposed to operating conditions. Tests were made at the highest normal operating pressure or 90% of the relief device setting whichever was greater.

- The last step in the precommissioning procedure involved draining, drying out where applicable and thoroughly inerting any section of the plant in which process or combustible material would be handled.

Checklists and detailed procedures were prepared and utilized for each individual unit or processing step for precommissioning and startup. The units were, at this point in time, ready for actual startup and operation in the case of utility sections or start-up with the intent of full plant integration with water and nitrogen in the case of process areas.

D. Total Plant Integration

When all plant utility sections were fully operational, a full-scale integrated operation of the plant and process areas using demineralized water and nitrogen was performed. Normal operating pressures, levels, and flow rates were established. In certain areas, heat was applied to bring up temperature levels and test specific pieces of equipment. The purpose of this full scale plant integration was to accomplish the following:

- Test each piece of equipment under flow conditions.
- Identify, tag and repair any leaks or mechanical problems which were most easily accomplished when using water and nitrogen.
- Test all instrumentation for actual function and check as many alarms and protective devices as possible during actual flow conditions.
- Provide useful training for all plant personnel.

E. Commissioning Schedule

To minimize constant repetition of dates, an overall plant acceptance and startup schedule is presented in Table 1. The discussion on commissioning that follows will be specific to each individual utility or process section of the SRC Pilot Plant.

F. Commissioning of Plant Utility Sections

1. Plant Air and Instrument Air System

Prior to acceptance by P & M, the construction contractor had used the air system for instrumentation checkout. A temporary raw water connection had been installed for cooling since the cooling tower had not yet been commissioned. Both compressors, the air dryer, and all instrumentation were checked by P & M after acceptance. The system was then returned to service. The only problem of note was that of excessive vibration of the compressor discharge piping. This was corrected by the installation of additional pipe supports.

2. Fuel Gas System

After acceptance, P & M immediately started preparations for placing the fuel gas header in service. Plug valves were lubricated, block valves were closed, and blinds were installed in branch lines to all units except the boilers. Orifice plates were removed and dirt was blown from the main header and the boiler fuel lines with compressed air. The orifice plates were then replaced. The lines were then purged with nitrogen, pressured to 50 psig, and checked for leaks.

Table 1

SRC Pilot Plant Commissioning Schedule

AREA	DESCRIPTION	ACCEPTANCE BY P&M	STARTUP
09.4	Plant and Instrument Air System	02/04/74	02/09/74
All	Fuel Gas System	02/12/74	02/19/74
09.4	Boiler Plant	02/04/74	02/22/74
09.1	Waste Treatment -- Graver	02/25/74	02/26/74
	-- Incinerator	02/25/74	05/28/74
	-- Degremont	02/10/75	02/11/75
09.3	Cooling Water System	03/06/74	03/12/74
09.5	Inert Gas Generator	03/18/74	03/27/74
09.5	Inert Gas Generator*	04/19/74	04/20/74
09.3	Flare System	06/11/74	06/22/74
09.2	Tank Farm	04/01/74	07/10/74
04	Seal Flush System	04/30/74	08/23/74
09.8	Dowtherm System	04/23/74	06/10/74
04	Flush Solvent System	06/07/74	08/25/74
04	Solvent Recovery System	06/07/74	08/25/74
08.1	Product Solidification-Sandvik Belt	08/14/74	09/29/75
	-- Prilling Tower	08/20/74	not operated
08.2	Product Storage	08/14/74	09/29/74
09.5	Hydrogen Generator	06/17/74	06/30/74
09.5	Desulfurization -- DEA Unit	06/28/74	09/13/74
09.5	Desulfurization -- Stretford Unit	06/28/74	09/29/74
05	Gas Recovery & Recompression	06/20/74	06/25/74
01	Coal Receiving & Preparation	05/08/74	05/10/74
02	Slurry Preheating & Dissolving	07/03/74	09/13/74
03	Mineral Separation	06/14/74	09/12/74
09.0	Dry Chemical Storage	06/14/74	06/14/74

* Returned to construction contractor for correction of deficiencies.

Leak repairs and pressure testing were completed. Nitrogen pressure on the header was vented through the vent line between the two automatic shutoff valves at each boiler, the upstream valve having been opened by means of a temporary electrical connection. The line was then pressured to 25 psig with natural gas and vented to one psig to purge the nitrogen from the line. A gas detector at the vents on the boilerhouse roof showed that the purge was complete. The fuel gas header was then pressured to full pressure ready for service. Initially, the pressure was 46 psig but was later adjusted to 50 psig. The manual gas valves at the boilers were locked in the closed position while awaiting startup of the boilers.

3. Waste Treatment

The Graver system (surge reservoir, waste disposal treater, sand filters, and charcoal filters) and the incinerator were flushed with water and the filters were backwashed.

Inability to control the level in the waste disposal treater, loss of suction from the surge reservoir, and acid addition malfunctions were the first problems experienced with the Graver system. Those faults were corrected by relocating the level control valve on the waste disposal treater, adding a foot valve to the floating suction line in the surge reservoir, and installing a vacuum breaker upstream of the pH meter.

Startup of the incinerator was under the supervision of the manufacturer's representative. Problems were experienced with a faulty flame scanner, refractory obstructing the pilot burner, high amperage drawn by the air blower moter, flow switch malfunctions, and plugging of lines.

Several mechanical problems were experienced during startup of the Degremont system (air flotation and biological treatment) but they were easily corrected. A lengthy bacteria culture growing scheme was recommended by the vendor, but it was rejected in favor of the direct addition of a freeze-dried bacteria concentrate. These organisms multiplied rapidly and all projected waste water standards were being met only 18 days after acceptance of the unit.

4. Cooling Water System

Two days were used to check the circulating pumps, clean the tower basin, flush backward and finally flush forward through the piping and exchangers. After the system was filled with water and circulation was established, the Betz preliminary treatment program was started. First, the system was precleaned for 24 hours with a precleaning solution consisting of 340 pounds of Betz 403, 36 pounds of Betz 407 and 40,000 gallons of water. After the 24 hour precleaning operating, the system was drained, refilled with water and circulation was established again.

Next, the system was prefilmed with 300 pounds of Betz 807 in 40,000 gallons of water. After circulation of the prefilming solution for 6 hours, the tower blowdown was opened for 20 hours and then corrosion inhibitor addition started.

Only two of the three cooling water circulation pumps are in service at any one time. Therefore, 1/4 inch holes were drilled in the pump discharge check valves to allow a small quantity of water to flow backward through the pump for protection during freezing weather. The cooling water return sump pump in Area 01 was replaced since the original pump would not develop enough head to return water to the cooling tower.

All of the pressure safety valves on the cooling water side of heat exchangers were replaced. In most situations, the exchanger was rated for 100 psig and the valves were set to relieve at 125 psig. Safety valves were added to some exchangers on the nitrogen and hydrogen units that did not have safety relief protection.

5. Inert Gas Generator

After acceptance, the inert gas unit vessel internals were inspected and cleaned. Also, maintenance checked the rotating equipment and instrumentation. The unit was charged with water, heated to 180°F and 100 pounds of tri-sodium phosphate solution was heated to 215°F to remove grease, oil and dirt from the system. The cleaning solution was drained after a 24 hour boil-out.

The unit was then thoroughly flushed with demineralized water to clear out all traces of tri-sodium phosphate. Next, the distributor pans in the vessels were inspected, cleaned and welded into position. It was necessary to modify the burner throat to increase the firing capacity.

The unit was charged with MEA solution and the burner was fired. Four days later the unit was shut down and turned back to the construction contractor for repair of defects. When corrections were completed, the unit was started again. After fuel gas trim valve piping was modified, unit operation lined out at 65-70% of design gas production rate. The deficiency in the inert gas production rate was due to an undersized combustion blower. Several modifications were made to establish unit performance at 100% of design. Initially, the burner throat had a high pressure drop so the throat was drilled from 3 7/8" to a 6" diameter. After the burner modification, the unit operated at only 65-70% of design rate.

Finally, a larger combustion blower was installed and the gas production rate finally reached 100% of design.

Worthington representatives were in attendance for run-in of the inert gas compressors and the CO_2 compressor. The vendor for the unit was consulted on numerous occasions for startup advice for the main unit. They were also contacted to determine corrections for faulty equipment and design deficiencies.

Initially, large quantities of entrained MEA solution in the gas to the inert gas compressors prohibited compressor operation. The compressor suction was modified by adding a knockout drum and demister pad on top of the absorber. Also, a knockout pot was installed at the compressor suction. These modifications removed sufficient carryover to allow safe compressor operation.

The original separator downstream of the flue gas cooler was not large enough to efficiently remove entrained water droplets and the resulting carryover collected in the CO_2 Absorber, diluting the MEA solution. The problem was resolved by replacing the original separator with a larger separator. A knockout pot was installed at the CO_2 compressor suction since the original piping had no provisions for removal of entrained water.

Moisture condensing in the sample lines to the combustibles and trace oxygen analyzers rendered the analyzers useless. Sample coolers with knockout pots were installed to "dry" the sample streams. Also, a pump was installed to boost the pressure on the trace oxygen analyzer stream so the analyzer would function properly.

During initial unit operation, the unit would shake, vibrate and occasionally flame-out when an inert gas compressor unloaded. Vent valve PCV-12 was too insensitive to pressure changes created when one of the compressors unloaded, and apparently the weight of the valve stem made proper operation impossible at the low operating pressure of the inert gas unit. The valve was pulled, reinstalled with the valve stem under the diaphragm and reset. This reorientation of PCV-12 corrected the problem.

Due to improper switching and drying, the inert gas dryers became water saturated. New solenoids were installed in the dryer switching system and the air pressure to the actuating cylinder was increased from 40 to 100 psig. The dryers performed properly after the dessicant was dried with dry cylinder nitrogen.

An electrical trip was installed to shut down the inert gas compressors when the MEA stripper reboiler fuel gas flow stopped. Originally, the low compressor suction pressure switch would eventually shut the compressors down if all the stripper instrumentation worked properly. It was felt the direct shutdown would minimize chances of drawing air into the inert gas header and minimize chances of drawing a vacuum on the absorber.

6. Flare System

P & M accepted the flare system and immediately began preparing to place it in service. Maintenance workers checked all instruments and the flare knockout scavenger pump. Plug valves in fuel gas and slurry service were lubricated. Connections for future safety valves in the hydrogen compression area were installed. Piping for the inert gas purge to the flare header was modified to relocate the pressure reducing valve upstream of the purge rotameter. All lines connected to the flare header were valved off and tagged. The flare knockout drum and the flare stack knockout were flushed with water. Manways were opened for vessel inspection and then replaced.

The system was then pressure tested with inert gas at 25 psig. After removing a leaking bypass valve around the safety valve on the vacuum flash drum and replacing it with blind flanges, a satisfactory pressure test was obtained. The blinds which had been installed for pressure testing were removed, and an inert gas purge was started from Area 02 through the header, knockout drum, and flare stack. The fuel gas and steam lines to the flare stack were commissioned, and the pilot burners were lit. The steam flow was set at 500 lbs./hr., the inert gas purge was set at 10 SCFM, and the fuel gas purge was set at about 150-200 SCFH.

The flare system was placed in service with no difficulty other than finding and repairing the leaks in the system; however, a few problems and deficiencies have been noted and should be considered in the design of future plants.

- Early in 1974, the Washington State boiler inspector pointed out several deficiencies which have since been corrected:
    - Non-code safety valves where coded valves are required.
    - Safety valves set at pressures above the design pressure of the vessels.
    - Vessels which had no safety valve where safety valves are required.
- Several safety valves were welded into the piping, making it difficult and hazardous to remove them for inspection and repair.

- Prior to acceptance by P & M, almost all of the safety valves leaked around the bonnets or adjusting nuts. Most of these were repaired in the field, but six valves had to be returned to the factory for repair.

- Calculations showed the possibility of excessively high ground level temperatures under certain conditions of flaring. The flare under these conditions would be a danger to the cooling tower. This condition was corrected by the installation of a 25 foot extension to the flare stack during a plant shutdown in December, 1974.

- Steam flow to the flare is controlled by adjusting a pressure reducing valve at the base of the flare stack. The manufacturer specifies a minimum flow of 500 lbs./hr.; however, under severe flaring conditions, this should be increased to a maximum of about 1000 lbs./hr. Under these conditions it would be hazardous to approach the stack to adjust the steam flow. This problem could be solved by installing a remote loading station for the steam valve.

7. Hydrogen Unit

Five modifications were made to the unit before it was accepted by P & M. First, raw water was piped to the shift converter quench system to provide emergency quenching in the event of a condensate quench failure. Second, a low pressure cooling water system was provided for the noncoded primary reformer coolers. A 14 psig pressure reducing valve was installed in the cooling water supply line, and a sump pump was installed to return the cooling water to the plant cooling water system. Third, an automatic nitrogen purge was installed to purge the reformer on low pressure. This would prevent overheating of the reformer tubes and catalyst in the event of a power failure or other problems. Fourth, an inert gas purge was provided for the shift converter independent of the rest of the unit. Fifth, the electrical controls were modified to require a firebox purge by the pilot air blower as part of the firing sequence.

Prior to startup, the original reformer catalyst charge was dumped because the manufacturer had supplied the wrong catalyst. A new charge of catalyst was loaded, but subsequent laboratory tests showed that heating this new catalyst to operating tempature produced spalling which resulted in 8-10% fines. The catalyst was dumped, sent out to be heat treated, rescreened and recharged.

Extra precautions were taken during catalyst reloading to ensure proper tube loading. Each tube was vibrated to ensure that all tubes were packed uniformly. After the tubes were loaded, the pressure drop across each tube was measured with the same amount of air flow through each tube. Catalyst was added or removed as required until the pressure drop across each tube was within 5% of the mean.

Several problems were encountered in firing the reformer burners for refractory dryout. It was necessary to remove the pilot adapters to get the pilot flames to burn inside the firebox instead of flashing to the outside. The ignition system would light only 9 of the 28 pilots. This system has never worked satisfactorily, and it has been abandoned in favor of manually lighting the pilots with a propane torch. The flame failure system, with each of the 28 burners having a Fireye capable of shutting down the entire unit, also has never functioned properly. To make the unit operable, the flame failure system was eliminated. Without this protection, an operator must stay with the unit whenever the firebox temperature is below the gas ignition temperature.

The refractory was dried out using the pilot burners to raise the firebox temperature to 800°F over a period of 30 hours. The main burners were then lit and the temperature was gradually increased to 1950°F to complete the dryout.

Steam and process gas flows were then started to the reformer. The high temperature shift catalyst (Girdler G-3A) was charged, the shift converter was purged with inert gas, and the activation of the shift catalyst was started. The plan was to raise the catalyst temperature 150°F/hr. with the reformer gas until a temperature of 650°F was reached. Unfortunately, the gas temperature at the outlet of the primary reformer coolers could not be controlled at the desired 1000°F without boiling occurring on the water side of the coolers. In addition, even with no backpressure on the unit, the reformer would produce at only 60% of the design rate. The unit was shut down for modifications to improve the gas production rate and temperature control.

Undersized steam piping was replaced with larger pipe, and control valves were changed to permit increased flows of both steam and process gas. The primary reformer coolers were modified to allow proper temperature control without boiling the cooling water.

After modification, the reformer was brought on line, and activation of the high temperature shift catalyst was started again. The catalyst was heated to 650°F at 150°F/hr and held at 650°F until the off-gas contained only a trace of H_2S. Then the temperature was increased to 750°F for 2 hours to remove any residual sulfur.

Before charging the low temperature shift catalyst, the operation of the reformer, high temperature shift converter, and shift converter quench system was checked. The need for modification of the quench system was apparent, and the methane content of the reformed gas indicated a possible carbon laydown on the reformer catalyst. In an effort to remove the suspected carbon laydown, only steam was fed to the reformer with a firebox temperature of 1950°F. When process gas flow was resumed, the reformed gas still contained over 5% methane.

Since both low and high temperature shift catalysts are contained in the same vessel, it was necessary to oxidize the high temperature shift catalyst with air and steam before shutting down to make the required modifications and to charge the low temperature shift catalyst.

A smaller condensate quench valve at the high temperature shift inlet improved control in the "manual" mode, but operation in the "auto" mode has never been satisfactory. An external quench pot was installed between the high temperature shift outlet and the low temperature shift inlet. This provided good temperature control and eliminated the problem of flooding the high temperature shift catalyst. The low temperature shift converter was charged with catalyst (Girdler G-66RS), and, since no activation was required, it was brought on line with the rest of the unit.

The complete hydrogen unit was brought on line in September, 1974; however, the unit was not fully lined out at design conditions until the noncoded desulfurizer vessels were replaced with coded vessels in February, 1975. Prior to that, the unit would not operate at design rate and deliver gas to the suction of the syn-gas compressor at 10 psig. The noncoded desulfurizers had limited the outlet pressure to 7 psig.

In December, 1974, the reformer catalyst supplied by the vendor was dumped, and six of the ten tubes were recharged with Girdler G-90B reformer catalyst. With this catalyst, the unit produced reformed gas at full design rate with less than 1% methane leakage with the firebox temperature at 1900°F.

A few additional modifications were made during the commissioning and initial operation of the hydrogen unit. The reformer inlet flexible hose connections were modified to eliminate the low spots where condensate collected and sealed off the tubes. The reformer fuel gas control valve, TIC-90, was modified to provide throttling control rather than open/closed operations. This reduced the fluctuations in the natural gas flow to the desulfurizers and to the inert gas stripper. Thermocouples were installed in the outlet of two of the reformer tubes. It would be advisable to have a thermocouple in the outlet of each tube. Then the operation of each tube could be monitored and problems, such as tube blockage, could be discovered before they became serious. A steam super-heater was installed to provide 500°F steam for regeneration of the desulfurizers. Originally, plant steam heated the desulfurizer beds only to about 250°F during regeneration. A bed temperature of 350-450°F was recommended by Girdler.

The syn-gas compressor was commissioned and run-in under the supervision of a Worthington representative.

Girdler representatives were consulted about operation with G-90B reformer catalyst, G-3A high temperature shift catalyst, G-66RS low temperature shift catalyst, and G-32J desulfurizer charcoal. Girdler also provided supervision for loading the high temperature shift catalyst.

The hydrogen unit vendor was consulted by telephone and at the plant site. They provided startup and operation advice and supervision, and they were asked to correct design and equipment deficiencies.

8. Dowtherm System

Commissioning of the Dowtherm system basically followed a three step procedure which included:

- Heater refractory dryout.
- Chemical cleaning of the piping system (oil and millscale removal.)
- Filling system with Dowtherm A.

A Foster Wheeler representative was on site for the heater dryout. Considerable difficulty was encountered in firing the North American Burner System, however. After two days of attempts, the burner was lit.

The heater arch temperature was increased to 500°F and held for 24 hours at which time it was increased to 750°F. After 24 additional hours at 750°F, attempts to raise the arch to 1000°F were unsuccessful due to high tube skin temperatures. The recommended steam sweep was increased six-fold, but to no avail.

The dryout interval was extended an additional 24 hours, per Foster Wheeler, to compensate for the lower temperature. Additionally, an attempt was made to set the burner linkage for optimum air/fuel ratio, but excessive air leakage resulted in unreliable results.

Chemical cleaning of the piping system consisted of a preboil oil removal using low chloride caustic materials and mill-scale removal using alkaline chelates. The system was water flushed followed with chemical cleaning. The piping was reflushed with water and dried with inert gas. A residue was discovered plugging the heater orifice runs which was composed mainly of chelate and iron. The system was reflushed with water and redried with inert gas.

Dowtherm A was charged to the system using a truck to make the transfer from a rail tanker to the system surge tanks. Numerous leaks were repaired and the circulating Dowtherm was heated to 300°F to boil off trace quantities of water. This was completed and the heater was fired intermittently to maintain system temperature.

During the month of July, 1974, the heater was fired as required to maintain a temperature of 240-300°F for water boil-up of the solvent recovery area. The heater continued to pose firing difficulties, resulting in a contact with North American Burner Co. who advised that the wrong pilot assembly had been supplied with the burner unit. The correct unit was obtained and installed by P & M Maintenance.

The Dowtherm system was heated to operating temperature to provide heat for solvent fractionation. A severe leakage problem developed in the Dowtherm and solvent recovery areas at valve bonnets and packings. Circulation was established through the mineral separation area to identify leak problems. The unit was shut down, cooled and drained to a tank truck for repair of leaks. It was refilled, circulated and reheated to operating temperature. Leaks continued and remain as a moderate to minor problem.

9. Tank Farm

After acceptance of the tank farm, all the manways were removed and the tanks and all the connecting lines were flushed with water in preparation for storage of raw solvent. The first solvent, FS-120, was received by rail and startup solvent was pumped to the process area for fractionation on August 21, 1974. Naphtha from the fractionation section was first transferred to the tank farm on November 10.

A few minor changes were made to the tank farm during commissioning or shortly thereafter. They are:

- Installation of a pressure regulator on the inert gas supply line.
- Installation of a valve in the purge gas system so the inert gas would sweep over the contents of the tanks.

- Installation of drains on several lines.
- Installation of walkways over the tank farm dike.

Several undesirable features exist in the tank farm. Some of these are:

- No sewer line to drain the water from the naphtha tank.
- Suction lines installed such that the bottom two feet of each tank is not available as working volume.
- Absence of a sewer system for the tank field pumps.
- Flow totalizers which have never worked correctly.

10. Seal Flush System

After acceptance of the seal flush system, a differential pressure control and alarm system was installed prior to the chemical cleaning of the system. A solution of trisodium phosphate, sodium hydroxide, sodium metasilicate, and a surfactant was circulated for four hours at 180°F. The system was drained and refilled with a solution of chelating agent and filming amine. This was heated to 250°F, circulated for six hours and drained. The system was then flushed with water and purged with inert gas.

11. Flush Solvent System

The flush solvent system was thoroughly flushed with water and the flush solvent accumulator was opened and inspected. Maintenance checked the heaters, cooler, pumps, and all instruments. The system was then filled with solvent and placed in service.

12. Desulfurization Units

After precommissioning checks of rotating equipment and instruments were completed, the DEA sulfur removal unit was thoroughly flushed with raw water to remove any loose scale or debris. Normal operating levels were then established, circulation begun and the unit brought up to normal operating temperatures. The system then was dumped to the sewer and recharged with a 0.5% solution of DEA in demineralized water to remove any residual oils and to establish a rust preventive film. The solution was circulated and again brought up to operating temperatures for eight hours and again dumped. The unit then was charged with a 30% solution of DEA in demineralized water. Circulation was established and held by maintaining 200 psig of inert gas pressure on the DEA absorber. Normal operating conditions were maintained until the introduction of sour gas.

No significant operating problems were encountered during the precommissioning and initial operating phase. However, several modifications were made by P & M in order to improve operation and dependability. The main changes are listed below:

- Valves were installed to bypass the unit in the event of a process upset or when maintenance is required. A valve and connection to the flare also was installed so that dissolver off-gases could be routed to the flare. This line is used on all startups and shutdowns.
- Differential pressure recorders were installed on the absorber and stripper to detect flooding of the columns.
- A bypass was installed around the absorber high pressure level control valve.
- Tie-in points for chemical inhibitor addition were installed.
- A fresh DEA storage system was installed.
- A reclaim system for collecting drips and returning them to the system was installed.

The Stretford sulfur recovery system was thoroughly flushed with raw water to remove any scale or loose debris. A sodium carbonate-water solution then was circulated at 100°F to remove any foaming agents from the system. This solution was dumped and the unit charged with operating solution. During this cleaning and charging operation, instrumentation was checked out.

13. Boiler Plant

The boiler plant includes the demineralizer, deionized water tank, deaerator, "A" and "B" boiler and the plant steam system.

Initial operation of the demineralizer was begun and after two initial regeneration cycles, water of acceptable quality was produced and routed to the deionized water tank. Some problems were encountered with the timer and solenoid valves during the regeneration cycle and both were corrected. The unit was fed at rates ranging from 50 to 75 gpm to check the throughput and effluent quality. Both met design sepcifications.

Maintenance checks, run-ins and flushing were carried out on the deaerator, boiler feed water pumps and boilers. After test firing the boilers, 150 lbs. of Betz Preclean 346 was added to each steam drum and the boil out procedure begun. After a 24 hour boil out period the boilers were depressured, drained and inspected for any remaining mill scale or oil. The boilers were then reheated, pressured and the double pressure safety valves set for 210 and 215 psig each.

Subsequently, the deaerator was brought in service and the boilers opened into the steam header. All vents and bleeds were left open until a strong flow of steam was seen and all lines were hot. The bleeds were then closed and the header brought up to 200 psig over a several hour period. The only problem encountered was a 3" line in the tank farm that had an improper expansion loop support. This was corrected by installing a proper "shoe" in the pipe rack.

Each boiler was brought up to the design rate of 35,000 lbs./hr at 200 psig header pressure. After testing, the boiler was fired to meet plant steam requirements.

G. Commissioning of Coal Processing Section

1. Coal Receiving and Preparation

After acceptance of the coal preparation area, conveyor belts and bucket elevators were trained and the coal pulverizing and slurrying section was commissioned. All equipment was checked and lubricated. The dehumidifier hotwell was cleaned and water circulation was established around the dehumidification loop. The inert gas preheater was adjusted and fired under low load conditions. Next, the inert gas blower was commissioned and air was circulated through the gas loop.

The slurry circulating pumps and preheater charge pumps were commissioned with water. The charge pumps were used to flush the preheater and dissolving area piping and to provide 2000 psig water for the slurry preheating and dissolving area hydrostatic test.

Six hundred tons of coal were unloaded and stored with no problems. Twenty-nine tons of coal were pulverized and slurried with water. The coal-water slurry was pumped through the slurry preheater. Coal feed rates varied from 2000 to 4000 lb/hr., and no significant problems were encountered. After the test run, the piping was flushed with water.

The only problem encountered during this initial operation was that of controlling the storage bin discharge flow rate, and this was solved by modifying the discharge chutes.

During the last week of July and the first two weeks of August, the slurry circulating pumps and preheater charge pumps operated with water during the plant water integration run. The valves in both preheater charge pumps eroded and had to be replaced. The water was drained from the area piping and equipment and in September, the slurry section of Area 01 was brought on line for plant integration with solvent.

2. Slurry Preheating and Dissolving - Area 02

The slurry preheater was fired for refractory dryout in July. The heater temperature was increased 50°F each hour with 24-hour hold periods at 500°F, 750°F, and 1000°F. The dryout was delayed by minor problems but was completed satisfactorily.

During the plant integration step, all of the control instruments were checked and repaired as required. Water and nitrogen were circulated throughout the plant to test for positive control of all flows, temperatures, pressures and levels.

Circulation of cold process solvent and nitrogen throughout the plant was started in mid-September. Foaming problems were experienced in the slurry blending and preheating and dissolving areas while circulating cold solvent. Addition of Betz Foamtrol 128B was started which solved this problem.

Pressures and temperatures of the circulating solvent and nitrogen were gradually increased to approximately normal operating levels, and several leaks and thermal expansion problems were solved in the piping system. Hydrogen was added to the slurry preheating and dissolving area to replace the circulating nitrogen and system testing continued at approximately normal reaction conditions. Coal was introduced to the slurry blending area late in September in the first attempt to process coal. This initial attempt at coal processing was not successful due to failure of both high pressure slurry pumps to maintain adequate pumping rates. Erosion of the pump valves and failure of the check valves in the solvent flush piping of the slurry pumps were found to be the causes of this problem.

3. Gas Recovery and Recompression - Area 05

Prior to initial operation, all alarms and shutdowns were checked out on both hydrogen compressors. The valves were pulled and each compressor ran unloaded for about an hour. The valves were reinstalled and the fresh hydrogen compressor was run-in with an inert gas suction pressure of 55 to 75 psig and a discharge pressure of 800 psig.

The recycle hydrogen compressor was only able to reach a 300 psig differential pressure before the packing burned up. The compressor was shut down for packing replacement under the supervision of a vendor representative. The packing was replaced and on startup, a special run-in procedure was followed. The compressor operated at 800 psig suction pressure and 1500 psig discharge pressure for several days and the packing blew out again. The piston rod was pulled and inspected. Scoring of the rod was determined to be the possible cause of the packing problem. Since the spare rod had not been received, the defective one was reinserted and the compressor was operated at reduced discharge pressure until the new rod was received and installed. Operation then was satisfactory.

4. Mineral Separation - Area 03

After acceptance, all mechanical equipment was inspected and serviced by the Maintenance Department. Vessels were opened for inspection and all piping was water flushed or blown with air. Water circulation was established through the unit, all possible instrumentation was checked, and the filter gas compressors were run in with a vendor representative in attendance. The mineral residue dryer was fired and refractory was cured under the supervision of the vendor representative.

While on water circulation, several precoating tests were conducted to test equipment operation in the precoat/filtration loops, develop functional operating procedures, and train operators prior to introduction of solvent into the unit. Initially, cellulose was used as a basecoat medium since material handling equipment had not been completed for handling asbestos. Initial precoats were unevenly deposited and lengthy in application.

The area was integrated on water circulation with the remainder of the process areas and attention was turned to integrating the filters with the mineral residue drying system. Of prime concern was a suitable method for withdrawing the cake from the filters to the dryer.

Filter "B" cake removal system was modified to include a closed circulation loop from the discharge of the original Sier-Bath screw pump back to the cake hopper with a downstream level control valve controlling flow to the dryer. The filters were precoated numerous times in order to provide feed material to evaluate the system. It was impossible, however, to keep packing in the pump. As soon as cake entered the packing gland area of the pump, the packing would fail.

Filter "A" system for cake removal was modified by removing the pump and installing a level control valve to the dryer.

This system was tested and performed satisfactorily until it plugged at approximately 60% solids concentration. Filter "B" was subsequently modified to utilize an identical system.

The unit was shut down and purged with inert gas to remove water. Cold process solvent was introduced into the unit in mid-September 1974.

5. Solvent Recovery - Area 04

The fractionation section was accepted and all the control valves, orifice plates and target meters were pulled for inspection. The entire system was flushed with water to remove any debris. Before proceeding further with startup, the following modifications were made:

- Tell-tale valves were installed on the light ends column feed tank.
- A Gunnite-hexsteel lining was added to the vacuum flash drum.
- Action of several control valves were changed from air-to-open to air-to-close.
- Several drain valves were added.
- Corrosion probes or connections were installed.
- Sight glasses on two vessels were rotated for easier viewing.
- Two temporary tanks were added to hold the naphthalene rich light ends column overhead.
- A level gauge glass was added to the bottom of the oil-water separator.

The reboilers then were filled with water and the Dowtherm system was checked by "boiling up" the columns. The vacuum flash preheater was "dried out", under the direction of the vendor representative. The entire plant was integrated with water. The vacuum system was pressure tested and due to plugging of the jets, a vacuum was not established until plugging was corrected.

During August, solvent was transferred to the process solvent accumulator in Area 04. From there, it was pumped to the seal flush accumulator and the seal flush system was placed in service. The fractionation system then was heated to separate the startup solvent consisting of a blend of 52% Gulf carbon black feedstock 120, 32% Koppers coal car distillate blend and 16% Koppers Creosote Oil No. 1 into light ends, wash solvent and process solvent cuts. Solvent levels were established in feed tanks and reboilers. Toluene was added to the reflux drums to prevent plugging of the overhead systems with naphthalenes during startup. As the columns were coming up to temperature and the temperature of the Dowtherm system approached 700°F, most of the valves in Dowtherm service developed leaks at the bonnets. This forced a shutdown of the columns to replace valve gaskets.

The fractionation section was started up again, but was shut down due to the vacuum bottoms pump loosing its packing. The solvent cuts were not as good as desired and were pumped back to the tank farm and reblended. The solvents were refractionated but the area was required to shut down again when the light ends column feed exchanger blew a gasket and the flange above the light ends column reboiler developed a serious leak. The columns were placed back in operation and process solvent was supplied to the other sections of the plant in September.

III. PROCESS AND MECHANICAL PERFORMANCE

A. General

1. SRC-I Material Balance Runs

The first successful coal processing began on October 3, 1974. Many mechanical problems were encountered during the early months of operation but by February, 1975 sufficiently dependable operation had been achieved, with the exception of the mineral separation area, to allow the beginning of material balance runs under various operating conditions in the dissolver area. When performing these material balance runs, the mineral separation area was bypassed in order to maintain more steady state and dependable operation and to eliminate the large inventory of material in the mineral separation area which would have required that runs be of much longer duration to obtain acceptable accuracy. Eleven SRC-I material balance runs were completed between February and July, 1975. The results are reported in Section IX.

2. SRC-II (Slurry Recycle) Operation

The plant was shut down for a brief period in early July, 1975 to make tie-ins to equipment which had been installed for preliminary slurry recycle or SRC-II operation. The plant was operated in the slurry recycle mode from mid-July to early September, 1975 and several material balance runs were completed. The results are included in Section IX. As a result of the success of these preliminary runs and the continued development of the SRC-II process at P&M's Merriam, Kansas Research Laboratory, the decision was made at that time to begin the design of a more comprehensive plant modification so that a thorough confirmation of bench scale data for the SRC-II process could be undertaken.

3. Production Run of Solid SRC

The period from early September, 1975 through November, 1976 was devoted to a production run, with its prime objective being the production of 3,000 tons of solid SRC for a large scale combustion test. Numerous mechanical and operational problems were encountered during this period but major progress was made in solving them. The main problems consisted of:

a. Frequent Coking of the Vacuum Flash Preheater Coil

This was corrected by modifying the flow scheme of the solvent recovery equipment to remove the more volatile components from the feed to the vacuum flash preheater, combined with installing a vacuum flash drum to preheater recycle system which provided greater velocity through the preheater coil and which also made possible its operation at a lower temperature.

b. Corrosion and Fouling in the Solvent Recovery System

This was corrected by replacing much of the original carbon steel with Type 316 stainless steel. Replacements were installed periodically throughout the production run as failures occurred. Additional changes were made after the end of the production run so that very little corrosion problems, with resultant downtime, are expected in future operations.

c. Mechanical Failure of Filter Components

A multitude of mechanical failures plagued the filters for many months. A large number of modifications were made which gradually improved their mechanical dependability so that during the last four months of the production run, only relatively minor production losses were due to the filters.

d. Mechanical Problems and Coking of the Mineral Residue Dryer

Many mechanical problems were encountered and improvements made. Additional progress is needed. Operating methods were developed which alleviated the coke formation but it remains a very serious problem.

Table 2 summarizes the production, shutdown and outage data from the production run. Selected data from this table are plotted in Figures 1, 2 and 3. Figure 1 shows that:

- The vacuum flash preheater coking problem was solved early in the production run.
- The corrosion and fouling in the distillation towers were major factors in downtime and resultant production loss.
- The filters were the cause of much downtime during the first ten months but only a minor problem during the last four months. The data during December, 1975 and the first three months of 1976 is deceptive. It indicates that the filters were causing little difficulty. This is incorrect. They were not causing plant shutdowns because so many were being caused by the solvent recovery columns. During these shutdowns, repairs were made on the filters. Consequently, they were being repaired without being "charged" for a plant shutdown. Had they not been repaired during these periods, they would have caused subsequent shutdowns. In addition to the downtime caused by the filters during the first ten months of the production run, operation of the plant at reduced rates during this period primarily was due to the filters. However, as a result of mechanical and operational improvements, the filters had the capability of handling full plant capacity a major part of the time during the last four months of the run.

- The mineral residue dryer had an increasingly adverse effect on production toward the end of the run. The only reason it did not cause more shutdowns during the early part of the run was that it got a "free ride". Several times when the dryer was shut down, a plant shutdown was avoided by accumulating filter cake leg material (dryer feed) in surge tanks. Generally the dryer was operated a considerable amount of time during plant shutdowns, processing accumulated feedstock which allowed it to "catch up". Improved filter operation transferred the production bottleneck to the mineral residue dryer during the last four months of the run. Long term production data and short term tests indicate that its capacity is equivalent to only 30 to 35 tons per day of coal feed. The mineral residue dryer would be the production bottleneck if another production run were undertaken.

Figures 2 and 3 show the marked improvement which was made in plant onstream performance and SRC production rate when more dependable filter operation was attained. However, little additional improvement in production rate would be possible because of the limited capacity of the mineral residue dryer and the requirement for periodic decoking.

4. Filtration Tests

Several tests were completed in December, 1976 and January, 1977 to determine the effect of several filtration variables. These tests are discussed and the results included in Section IX. At the conclusion of these tests, the plant was shut down for making modifications to allow operation of the entire plant in an SRC-II mode and to complete repairs and inspection of the existing plant.

B. Slurry Preparation

1. General

The slurry preparation area involves solids handling, grinding, drying, mixing and pumping of slurry as shown schematically by Figure 4. All of these are physical operations and the technology is not new or unique. Only four plant shutdowns could be attributed to slurry preparation area malfunctions. Because the slurry preparation area has caused few shutdowns and seldom influences the shutdown length, it would have to be rated much higher in availability than the whole plant. Problems usually result in a loss of coal feed. Most coal feed interruptions are resolved in less than one hour. The normal, short term coal feed interruption usually does not seriously disturb plant operation.

The preparation area capacity is limited to about 6,000 lb./hr. of coal feed. 6,000 lb./hr. of raw coal feed is near the maximum drying and conveying capabilities of the pulverizer gas

TABLE 2

PRODUCTION, SHUTDOWN & OUTAGE DATA FROM PRODUCTION RUN

	1975			1976											TOTAL
	OCT	NOV	DEC	JAN	FEB	MAR	APR	MAY	JUNE	JULY	AUG	SEP	OCT	NOV	
DAYS DOWN FOR:															
Decoking Vacuum Flash Preheater	3	6	9	0	0	0	0	0	0	0	0	0	0	0	18
Solvent Recovery System	0	0	5	15	0	17	3	0	0	4	4	3	8	4	63
Filters	7	6	1	2	0	0	6	4	11	5	0	5	0	0	47
Mineral Residue Dryer	0	0	0	4	0	0	2	0	3	0	5	0	5	3	22
Miscellaneous	7	1	4	0	3	0	0	0	3	4	0	3	0	0	25
TOTAL	17	13	19	21	3	17	11	4	17	13	9	11	13	7	175
ONSTREAM DAYS(1)	14	17	12	10	26	14	19	27	13	18	22	19	18	23	252
NO OUTAGE DAYS(2)	7	4	4	0	15	7	6	21	6	3	7	10	17	13	120
NO. OF TIMES FILTER HEADS REMOVED															
"A" Filter	2	0	3	3	1	1	4	1	6	4	4	3	1	0	33
"B" Filter	6	2	0	0	3	1	2	1	3	1	0	2	1	1	23
TOTAL	8	2	3	3	4	2	6	2	9	5	4	5	2	1	56
SRC PRODUCTION															
Per Month	100	100	100	90	391	280	150	443	143	233	215	300	500	484	3529
Per Stream Day	7.1	5.9	8.3	9.0	15.0	20.0	7.9	16.4	11.0	12.9	9.8	15.8	27.8	21.0	
Per Calendar Day	3.2	3.3	3.3	3.0	13.5	9.0	5.0	14.3	4.8	7.5	7.0	10.0	16.1	16.1	

(1) Days in which coal is fed but not necessarily the entire 24 hours.
(2) Days in which coal is fed the entire 24 hours.

CAUSES FOR PLANT SHUTDOWNS DURING PRODUCTION RUN

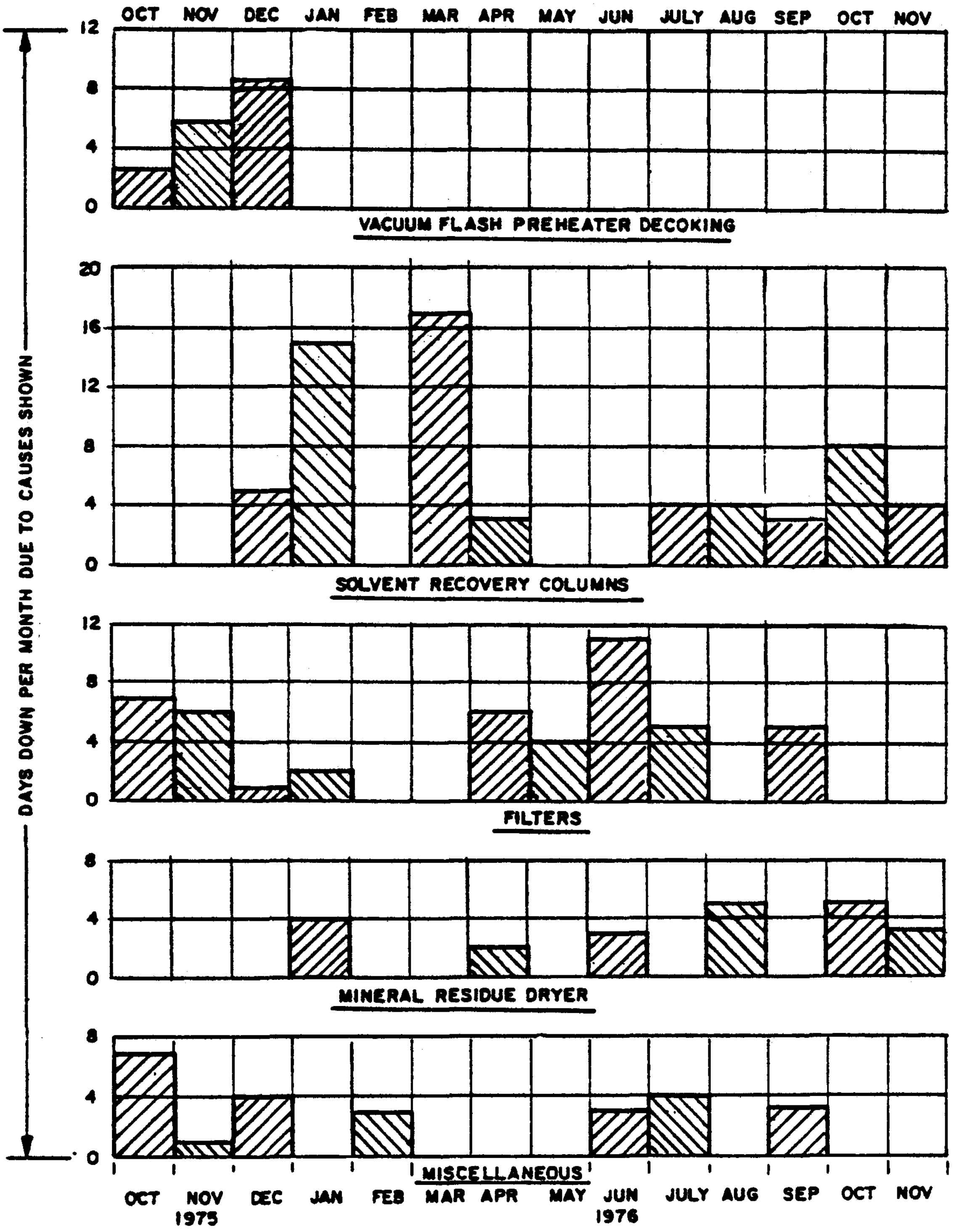
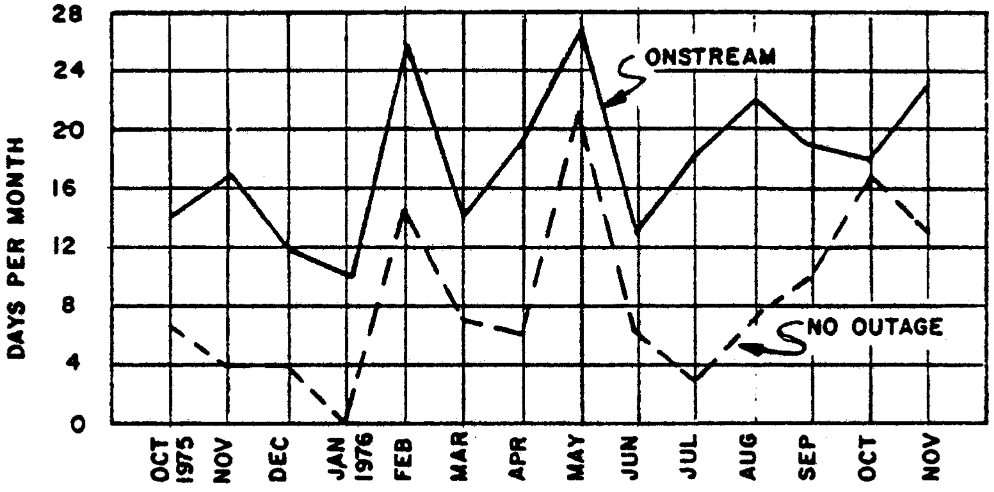
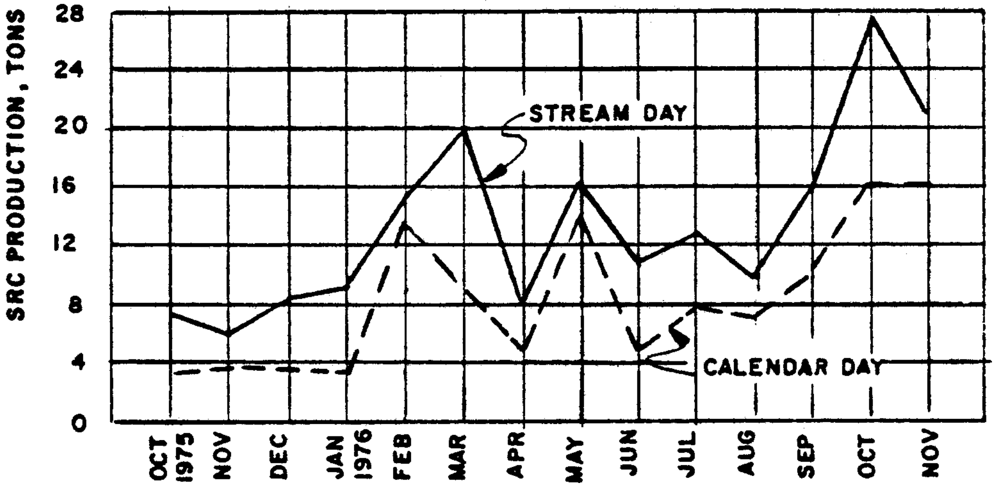


FIGURE I



PLANT
ON STREAM PERFORMANCE
DURING PRODUCTION RUN



PRODUCTION PERFORMANCE
DURING PRODUCTION RUN

FIGURE 2

MONTHLY SRC PRODUCTION DURING PRODUCTION RUN

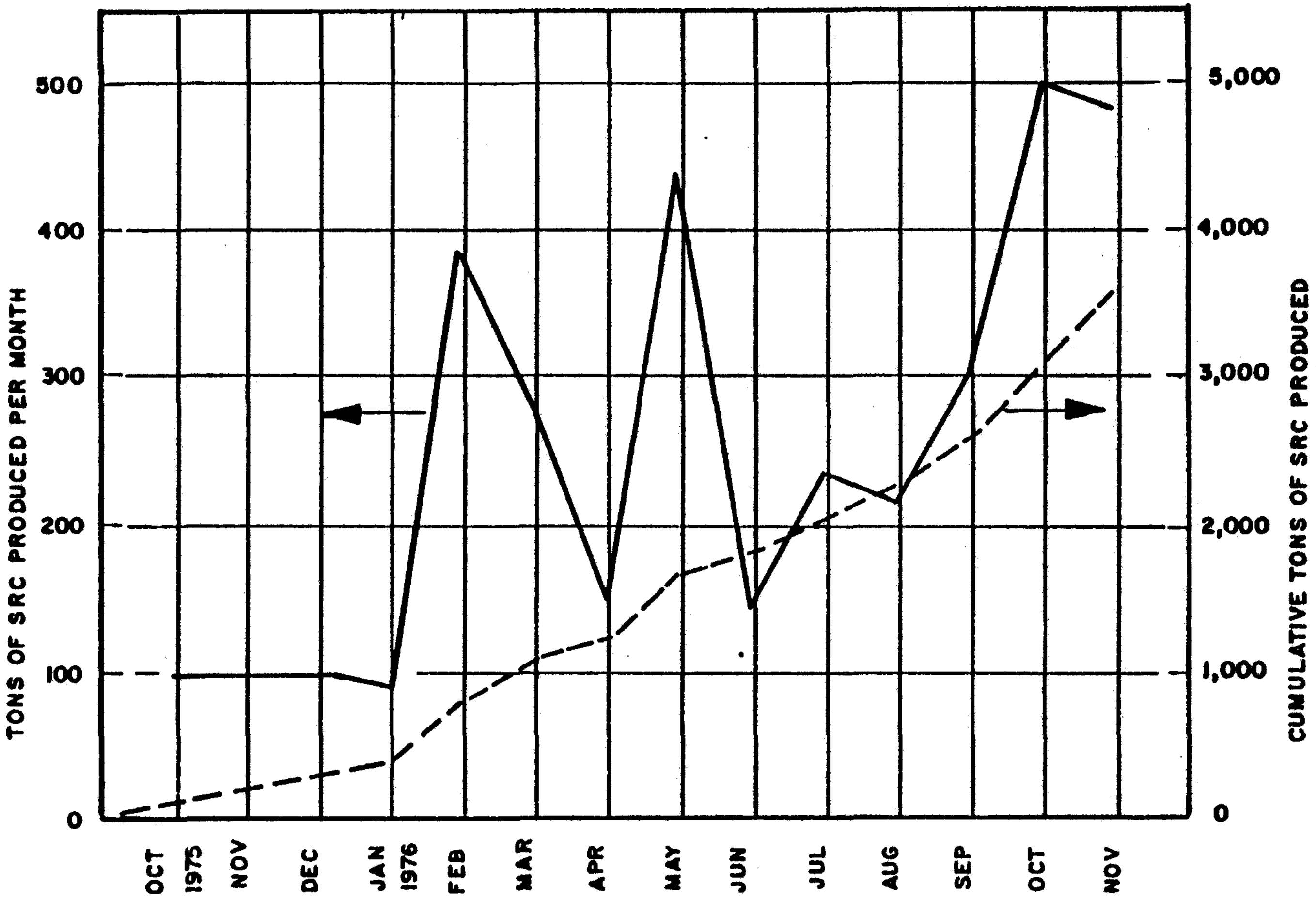


FIGURE 3

AREA 01 COAL RECEIVING AND PREPARATION FLOW SCHEME

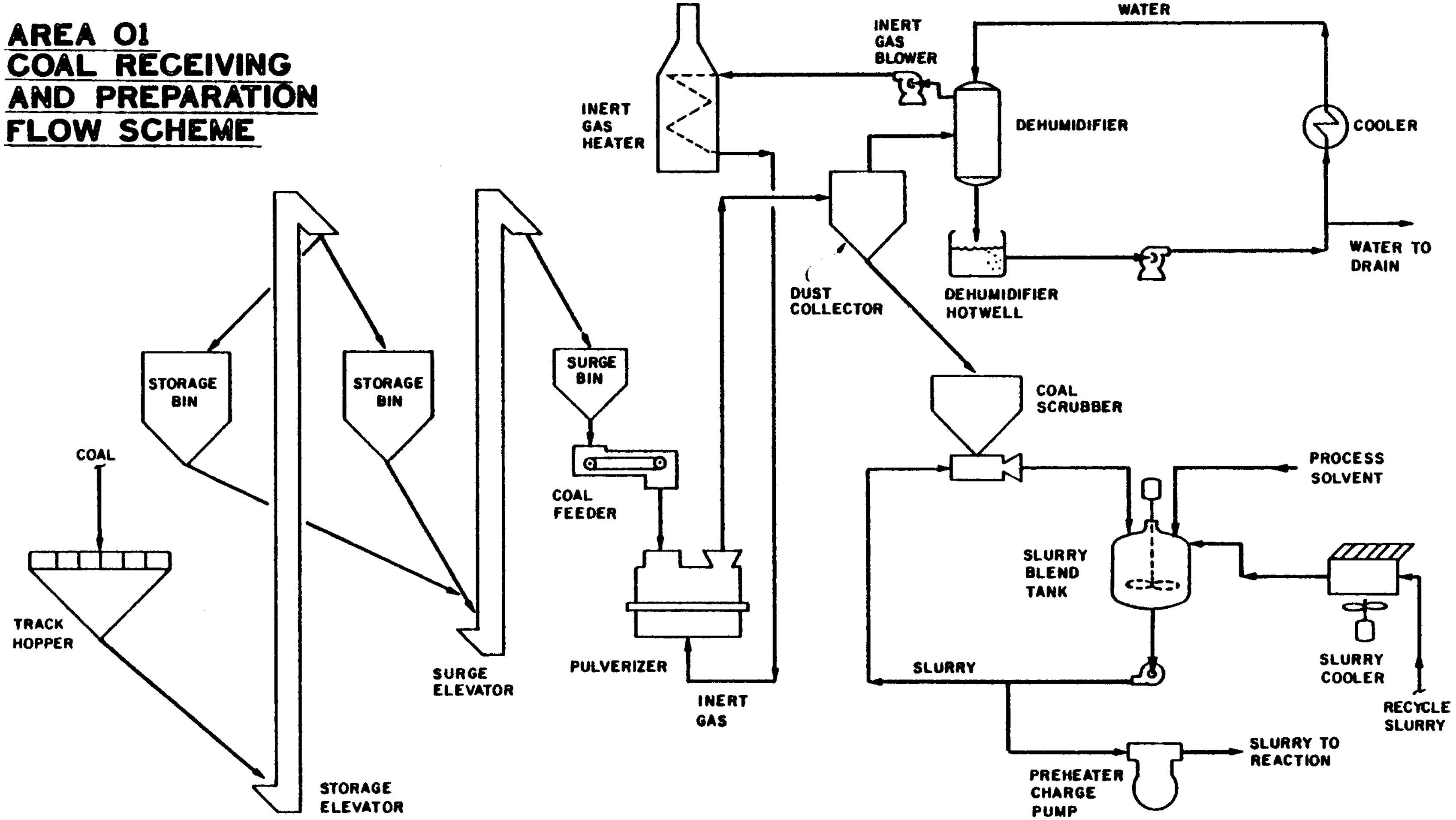


FIGURE 4

loop. A maximum slurry solids concentration has not been established. In SRC-I operation, the coal concentration has normally been about 40%, but concentrations up to 45% have been handled successfully. Upsets have created slurries of extremely high, but unknown, solids concentrations. These high coal concentration slurries have not caused mechanical failures in the slurry preparation area but have caused excessively high pressure drops which could have caused plugging.

2. Process and Mechanical Problems

a. Coal Receiving and Storage

Movement of rail cars was a big problem during early operation. The original installation provided a car puller to pull the cars downhill and handbrakes were the only means of stopping cars. By pulling cars downhill, it was very easy to get them moving but stopping rail cars on a downhill grade was risky and difficult. Since maintenance of railway rolling stock is poor, many cars have poor or totally nonfunctional brakes. Two derailments and several near-misses led to the lease of a Trackmobil to move cars.

Hauling wet coal several thousands of miles by rail results in well packed material. The packed coal bridges in the car and is impossible to remove without physical assistance. The problem was eventually solved by purchase and use of a car shaker.

The coal storage bins provide a natural chimney and coal fires have been noted. Bin fires have been rare when coal was stored less than a month. Plant problems have created instances of coal being stored several months, which almost guarantees a coal fire. Injecting carbon dioxide into the bin normally snuffs the fire sufficiently to allow dumping the coal. A 12-ton liquid CO_2 system was installed for this purpose. A commercial SRC plant will have outside coal storage and with proper rotation and handling, fires will be minimized. At least fires in an outside stockpile are accessible and can be controlled.

Chutes in the system have plugged and eroded. Installation of liners or constructing chutes from tough materials will prolong chute life. Wet, fine coal has a tendency to adhere to chute surfaces, eventually bridging and plugging the chute. An installation with large chutes will provide a higher ratio of cross sectional area to circumference, which will reduce plugging tendencies.

Several holes developed in the coal chute from the surge bin elevator to the surge bin due to abrasion. It was replaced with a polyethylene lined steel chute.

The pulverizer rotary airlock has presented several difficulties. Wet coal fines tend to pack between the vanes and plug the airlock but this will not be as severe with larger scale equipment. Numerous five and ten minute coal feed interruptions have been encountered when coal or hard objects jam the airlock. Very little can be done to prevent a rotary airlock from occasionally jamming in this type of service. Finally, dust blowing back through the airlock and through the packing have been problems. Blow back can be minimized if an airlock is provided with adjustable vane tips and the vane tip gap is properly maintained. The shaft packing must be purged with clean gas or the packing will wear out in minutes. To increase rotary valve packing, casing and rotor tip service life, a new heavy duty valve with purged packing, hard chrome plated bore and end plate and Stellited rotor tips for abrasion resistance was installed.

b. Coal Pulverizing

Three of the four plant shutdowns attributable to the slurry preparation area involved coal pulverization. Two shutdowns were side effects from plugging in the coal dust scrubber and one shutdown was caused by failure of the pulverizer. During one scrubber plug, the inert gas blower was turned off but, due to a malfunction, the inert gas heater kept firing. When the inert gas blower was turned on again, an extremely hot gas stream melted several Nomex felt bags in the pulverized coal dust collector. The bags had to be replaced. During another scrubber plug, the pulverized coal dust collector filled with coal. When the inert gas blower was turned off, the fluidized coal dust flowed back into the pulverizer, inert gas heater and blower. Operation was resumed after the equipment was cleaned out.

The pulverizer has been very reliable and has been a low maintenance item. The pulverizer internals failed after two years and caused a plant shutdown. Other than this failure and a short coal feed interruption to remove a tramp belt which caused internal jamming, the pulverizer operation has been faultless.

Other problems in this section have been short term or the type of problem that can be postponed until shutdown. Failures such as a bad bearing or worn coupling can be corrected by stopping coal feed and making repairs. Problems such as carryover of dehumidifier water into the inert gas blower generally can be tolerated until a plant shutdown. The dehumidifier demister pad has a tendency to partially plug with rust, which results in water carryover to the blower. On one occasion, water carryover caused severe vibration in the blower and overhaul, including installation of a new impeller, bearings and case suction disc, was required. Since that time, the dehumidifier demister pad is periodically checked and replaced as necessary and no further blower problems have occurred.

c. Coal Slurrying and Pumping

Plugging in the coal dust scrubber (mixer) has been the biggest single problem in this section. If plugs are not detected and taken care of shortly after they occur, pulverized coal accumulates in the baghouse. Undetected scrubber plugs have caused two of the plant shutdowns mentioned in the coal pulverizing section. While unplugging the scrubber, slurries of excessively high coal concentration have been produced. These slurries of high coal concentration have caused near-miss slurry preheater plugs. A level detector has been installed in the scrubber inlet chute to detect plugs before the problem becomes severe. Scrubber hopper wash nozzle malfunctions have caused most of the plugs. Nozzle plugging or an improper spray pattern results in a heavy mud that covers the scrubber eductor throat. In initial SRC-II operation, insufficient clean solvent was available for hopper wall washing. Eliminating the wall wash eliminated scrubber plugs but reduced the capacity by about 70%. Then coal piled up due to insufficient capacity.

Mixing coal into slurry with the present coal dust scrubber is not just an operating problem. Large quantities of slurry have to be pumped through the eductor to provide a motive force. Similar systems for large installations would be expensive. Vessels equipped with high speed agitators probably would do a better job of slurrying coal at much lower capital and operating costs.

Initially the centrifugal slurry circulating pumps were plagued with poor packing life. The packing was replaced with mechanical seals supplied with seal flush. Both pumps now are equipped with Chesterton 880 single seals with a tungsten carbide rotary face against a ceramic stationary face. Operation is vastly improved compared to the original packing and mechanical sealing arrangements. Of the initial Chesterton 880 seals installed, one functioned eight months and the other seven months, compared to the hours and days of operation experienced with packing and other seals. After 2-1/2 years of operation in slurry service, the pumps still function satisfactorily. Erosion has not been a problem because the wetted parts are constructed of Ni Hard. Although the Morris type JC, Ni Hard slurry pumps have provided excellent service, 300°F and 100 psi temperture and pressure rating severely limit their application.

Initially the triplex, positive displacement preheater charge pumps were troubled with short valve life and short packing life. The valves originally recommended by the manufacturer were spring loaded hemispheres. Valves were in service from a few hours to four days before severe erosion made replacement necessary. At the suggestion of I.G.T., chrome spheres hardened to Rockwell C 61-63, were substituted for the spring loaded hemispheres. The spherical valve life is measured in years, rather than days. Short packing life stemmed from the failure of the packing flush check valves. When the checks fail, slurry short circuits from a discharging cylinder to a cylinder on a suction stroke which causes packing failure and loss of pumping capability. Frequent inspections of the packing flush check valves have essentially eliminated these problems.

The preheater charge pumps have caused only one plant shutdown. In the early stages of plant operation, the valves failed on one pump and the safety lifted and would not reseat on the other pump. When slurries of extremely high coal concentration have been produced by accident, the pumps make loud knocking noises but they have never failed to pump the material.

Normal care of the preheater charge pumps has evolved to repacking every two to four weeks. Normal procedure is to replace the pump plungers when the packing is replaced. The worn plungers are rebuilt with nickel-chrome-boron and ground and polished to a 10 micro finish. Coal tends to settle in the pump suction chambers and the suction must be cleaned every month or two. On the infrequent occasions a pump discharge safety valve lifts, the valve must be replaced. The safety valves almost never reseat.

d. Standby Capability

The original SRC-II modifications provided the capability to circulate solvent or slurry between the dissolver and the slurry preparation areas. This capability enables the two areas to start up, shut down and stand by for coal feed independently of the other plant areas. In the SRC-I mode of operation, these areas have seldom dictated the length of shutdowns. Countless hours of production time have been saved by having the two areas at operating conditions when other areas become ready to process feedstock.

C. Preheating and Dissolving

1. General

Figure 5 is a simplified flow sheet of the preheating and dissolving area. The preheating and dissolving section of the plant has operated very reliably in comparison to other plant

AREA 02 & 05
PREHEATING - DISSOLVING
GAS RECOVERY & RECOMPRESSION
FLOW SCHEME

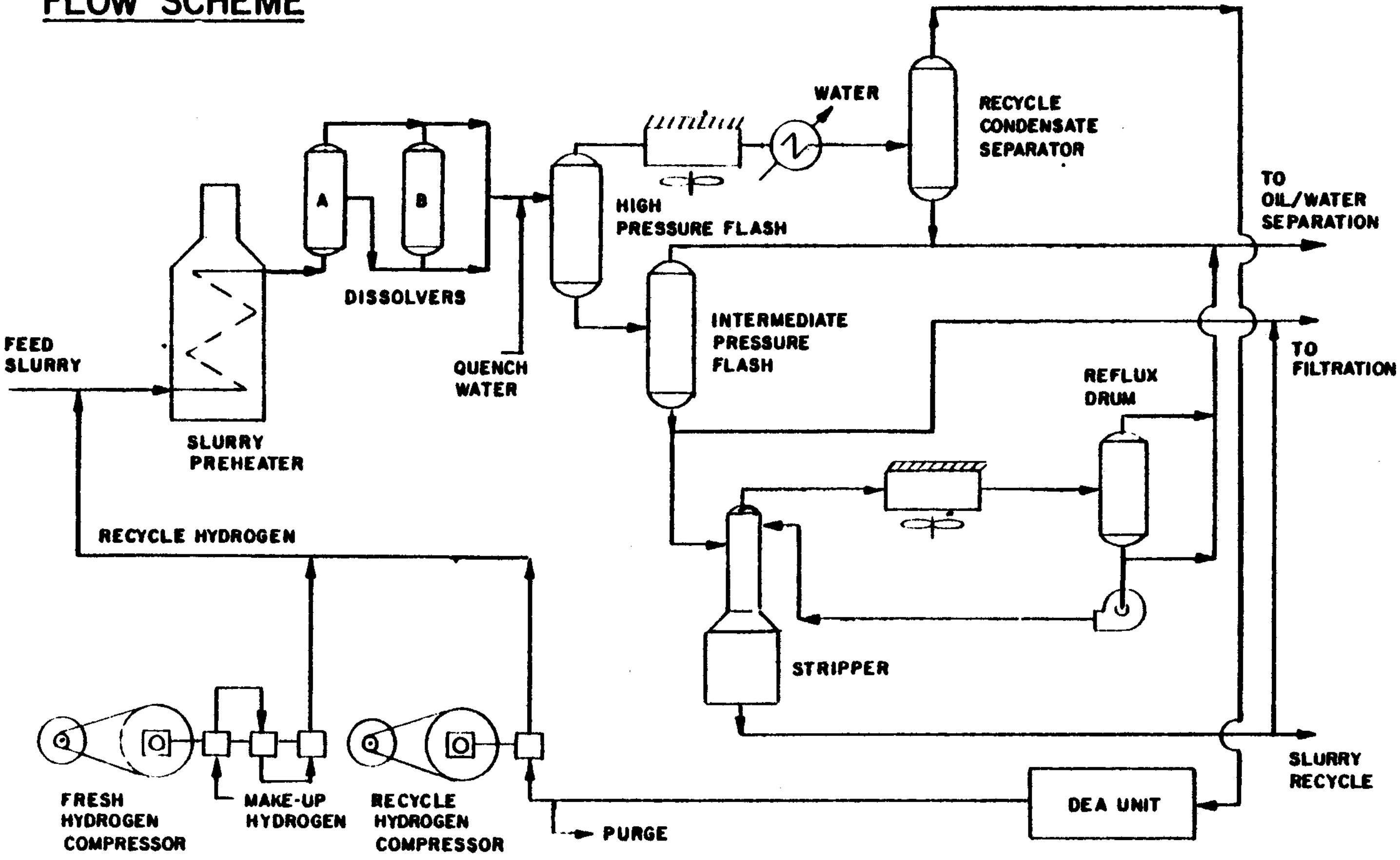


FIGURE 5

areas. This reliability resulted from conscientious attention to details during the design phase, use of adequate materials of construction and the similarity of this area to the core of this company's operating technology. During the entire operating period, this section has forced a general plant shutdown only three times. Only once did repairs take more than a day. One particular component, the high pressure flash vessel level control valve, has a definite failure frequency which is shorter than the failure frequency of the remainder of the plant. Consequently, the trim on this valve has been consistently replaced at regular intervals during plant shutdowns. Except for this particular letdown valve part, no other portion of the preheating and dissolving section is repaired during shutdowns. Therefore, a parallel valve was installed and this section of the plant now has an onstream factor that approaches 100%. The upper capacity of this area is not known since other bottlenecks are reached in other parts of the plant before the preheating and dissolving section reaches its maximum capacity. The Process Evaluation section of this report, Section IX, describes the effects of flow rates and other variables on the hydrogenation of coal.

2. Process and Mechanical Problems

a. Weephole Leak From the "A" Dissolver Bottom Nozzle

Shortly after plant startup, the weephole on the lined bottom nozzle of "A" dissolver began to drip fluid. The weephole was installed at this nozzle to monitor the integrity of the cladding of the dissolver. The volume of leakage was so small that the crack was thought to be very small. An internal inspection verified a small crack in a sleeve which joins the cladding to the nozzle. A conclusive theory of what caused this crack has not been advanced. The Corrosion-Erosion section of this report, Section IV, discusses this failure in more detail.

b. Dissolver Product Air Cooler

The air cooler on the dissolver outlet stream failed after a few days of operation due to excessive and nonuniform thermal stresses. This diagnosis is too general to suffice as a description of the exact mechanism of failure. The failure was discovered when some of the tubes began to leak. After the air cooler was taken out of service, it was carefully inspected. Some tubes were buckled, some were plugged and some were pulled out of the tubesheets. Some showed evidence of overheating while others showed evidence of underheating. One theory of the mechanism of failure is that the three phase flow components separated, with the solids settling out in the bottom tubes, and with a greater portion of the gas going through the top row of tubes. Both the tubes with settled solids and the tubes containing more than their share of gas would have had a lower temperature than the middle tubes. These colder tubes restrained the cooler from expanding evenly, thereby resulting in their being pulled to failure. This theory is fine except for some bothersome contradictory evidence. There were some overheated tubes where all should have been underheated. There were plugged tubes where gas should have been flowing according to the theory. Another theory is that the tubes that would first receive the dissolver product were overstressed by the high temperature in relation to the rest of the tubes of the cooler. A water quench was installed to replace the failed cooler. This method of cooling has worked well but water separation devices in downstream portions of the plant have been overloaded as a result. The use of quench water in a commercial plant could not be tolerated due to its inefficiency. The design of a satisfactory cooler is an engineering challenge. Testing of different types of coolers in the pilot plant would be desirable. The large amount of water injected also has complicated reaction area studies. Section IX of this report will deal with the inaccuracies introduced into reaction variable studies by the water quench.

c. Level Control Valves

The trim and sometimes the bodies of the level control valves which control slurry flow have been worn out at an alarming rate. The control valve that has experienced the most wear has been the level control valve on the high pressure flash vessel. Originally, both the high pressure flash vessel level control valve and the intermediate pressure level control valve were Fisher globe DBQ valves with microform trim. Even before the plant had started up, new Fisher angle globe DBAQ valves were ordered as replacements. By the time the new angle valves arrived, the trim in each of the level control valves had been worn enough to require replacement several times. There was also evidence of valve body wear. The angle valves were modified before installation. The outlet port was machined just enough to allow a liner to be installed. The liner consisted of a tungsten carbide lined tube which was welded to the seat ring of the valve. The tube was actually an oil field choke and was long enough so that it extended slightly beyond the outlet flange, thereby protecting the valve body from erosion. These liners have eliminated erosion in the valve bodies. The valve trim has continued to be a routine replacement item. As previously stated, the need to replace the trim in the high pressure flash level control valves has not been significant in keeping the plant from operating. When the plant is shut down for other reasons, the trim of these two valves is routinely checked. Usually the plug of the high pressure flash vessel level control valve is found to be eroded. Rather than risk a future shutdown due to failure to replace a somewhat eroded plug, a new plug is installed. There has been only one instance when it was necessary to replace a set of trim in the high pressure area level control valves because of lack of adequate control.

Another mode in which these control valves have failed is through deterioration of the solder which holds the tungsten carbide plug to the stainless steel stem. At the temperatures experienced in the high pressure flash bottoms stream, the solder is near its melt point. After a control tip fell off due to the high temperature, a specially designed plug which had been attached by heat-shrinking was successfully used.

The trim erosion in both slurry level control valves may be due to oversized valves. Tungsten carbide is corroded by ammonia, which is a byproduct of the coal hydrogenation reaction. There is indication that this type of corrosion has taken place.

The solution of the control valve wear problem is well within the capabilities of current technology as far as larger volumes of flow are concerned. As control valve size increases, there is more opportunity for design improvements such as hard liners, expansion chambers and sacrificial wear surfaces.

The use of parallel control valves, with double blocks and bleeds, would be justified in commercial plants.

d. Flow Meters

The original rotometers, target meters and turbine meters installed in slurry service performed unsatisfactorily. Erosion resulted in failure of the rotometer and target meters. Extraneous solids prevented satisfactory use of turbine meters. Venturi meters were substituted for these meters. However, the flows are too small for the smallest sized standard meter but acceptable accuracy is achieved provided the taps are kept clear with a gas purge. Larger flows would benefit the operation of venturi meters.

e. Plugging Problems and the Emergency Flush System

The plugging of lines that occur in this area has been confined to slurry solidification due to shutting down with insufficient flush out or by solids deposition from the slurry upon shutdown. Sometimes the plugs have been homogeneous solids which were simply recently solidified liquids, while at other times the plugs were a packed accumulation of fine solids. These plugs were removed by high-pressure hydroblasting.

During normal operation, a number of near-miss plugging incidents have been avoided with an emergency flush of the heater-dissolver system. The near-misses result from instrument failure in the coal preparation area. When the coal concentration exceeds 45%, the pressure drop across the preheater coil increases quickly. When the phenomenom is recognized, emergency flush is injected into the slurry preheater to wash out the potential plug. A complete plugging of the preheater has never occurred and there have been no indications of coke buildup inside the coil.

A catacomb-like coke deposit was found in the lower portion of the dissolver during the first inspection after twenty months of plant operation. A more thorough discussion of this deposit and its effects is included in Section IX.

f. Closure Problems

The seal rings on the Grayloc high pressure closures have developed small leaks due to excessive bending moments on the closures. These leaks were stopped when the cause of the moment was eliminated or the moment counteracted on both sides of the closure. Other leaks which could not be stopped by tightening the flange bolts were traced to scratches on the polished seal ring which may be due to stress corrosion. The leaks occurred long after startup, which indicated they were not due to improper handling of the seal rings before or during installation.

g. Pressure Control Problems

Other than one attempt to operate with two dissolvers, all plant operation has been with one-half or one dissolver. The reaction area was subjected to violent, 500 psi pressure swings when operation with two dissolvers was attempted. After several hours of extremely unstable operation, single dissolver operation was resumed. A deficiency in available hydrogen may provide one explanation for the unstable operation. Hydrogen starved reactions may have cycled in activity, causing the unit pressure to swing.

h. Ammonium Bicarbonate in the Stripper Flare Lines

The safety valve for the slurry recycle stripper was found to be packed with crystals which laboratory analysis revealed to be ammonium bicarbonate. The fact that the vent gases from the reaction area chemically combine to form solids will need to be considered when designing the safety vent systems in future facilities. Heat tracing of the safety valve inlet pipe and the safety valve itself was the solution at this facility.

3. Change For Heat Flux Study

Early operation of the slurry preheater coil revealed that the process heat load, and thus the heat flux, was substantially less than that anticipated. Because of this and successful operation with a lower than design solvent-to-coal ratio, the bottom half of the coil was insulated with a resultant heat flux of approximately 8,000 BTU/hr-ft^2 based on inside area. After the bottom half of the coil was insulated, the pressure drop through the preheater decreased about 35% and the tube to process fluid temperature differential increased. Apparently the higher flux decreases the time slurry is in a highly viscous gel state which decreases the pressure drop and increases the heat transfer rate.

4. Corrosion-Erosion

A great deal of corrosion-erosion data have been collected for this area and is discussed in the Corrosion-Erosion section of this report.

5. Initial Slurry Recycle Modifications and Operation

The slurry preheating and dissolving section of the plant was modified in the summer of 1975 to allow for preliminary slurry recycle operation. Briefly, a new stripper for the intermediate flash vessel bottoms stream with related pumps, instrumentation, and piping was installed. This modification allows for recycling part of the new stripper bottoms stream back to the slurry preparation area for use as liquid for slurrying of fresh coal. The remainder of the new stripper bottom stream flows to the solvent recovery area of the plant. The plant was operated in the initial slurry recycle mode for only about six weeks due to the urgency of beginning the production of 3,000 tons of solid SRC for a large scale combustion test. The results of this preliminary slurry recycle operation were encouraging and, as a result, plans were initiated to make more elaborate modifications to permit future operation of the entire plant in the slurry recycle or SRC-II mode.

During the initial slurry recycle operation, the trim of the slurry control valves eroded more rapidly than expected. The pumps that pressure the non-recycled slurry to the next processing section of the plant also eroded very quickly. The pressure in the downstream area was lowered to allow slurry flow without the use of a pump.

In spite of the higher viscosities associated with the slurry recycle mode, the preheating and dissolving area did not become plugged by the viscous high melt point slurry. Reaction conditions were made more severe very slowly and very cautiously to avoid plugging.

The capability for recycling of slurry from the dissolver to the slurry preparation area was very beneficial during the production of the 3,000 tons of solid SRC since the front end of the plant could be maintained on standby, at operating temperature and pressure, while repairs were being made to downstream equipment. As a result, the time required to get the plant back into production after repairs was greatly reduced.

D. Filtration

1. General

Figure 6 is a simplified flowsheet of the mineral separation area, which includes filtration and mineral residue drying. As discussed previously in this section, many process and mechanical problems have been encountered with the two pressure, precoat, rotary filters but much progress has been made toward their solution. As tabulated in Table 2, internal maintenance and modifications necessitated the removal of the head from "A" filter thirty-three times and from "B" filter twenty-three times during the fourteen month production run. The removal of the head is a time consuming job. It requires removal of about seventy-five 2-1/2" nuts before the five-ton head can be removed. About sixteen hours time is required to cool the filter before the head can be removed and another eight hours is required to heat the filter after the head is reinstalled. Commercial filters should have facilities to make rapid cooldown and heatup possible.

Figures 7 and 8 show the operational availability and time in operation for "A" and "B" filter by months during the production run.

2. Process Performance

Several factors affecting the operational or process performance of the filters are discussed below.

a. Sluicing/Screen Blinding

As a result of previous work, screens originally used were 20 X 250 double dutch twill. In an attempt to reduce problems with screen fouling, these were changed to a 24 X 110 single dutch weave. These screens will retain 74 and 138 micron particles, respectively. It would be desirable to evaluate a 60 X 60 square mesh screen, which is in inventory. This screen has a retention of 234 microns. The sluicing procedure has been modified but it still does little more than clean the vat walls. It is ineffective for the sludge which has built up in the bottom of the vat. Attempts are made to reduce this buildup by circulating a high volume of feed slurry through the filter and back to the filter feed flash vessel. This procedure decreases the buildup but does not eliminate it. As designed, the vat is an effective settling drum. While the agitator is effective, it also serves only to minimize

FILTRATION & MINERAL RESIDUE DRYING AREA 03

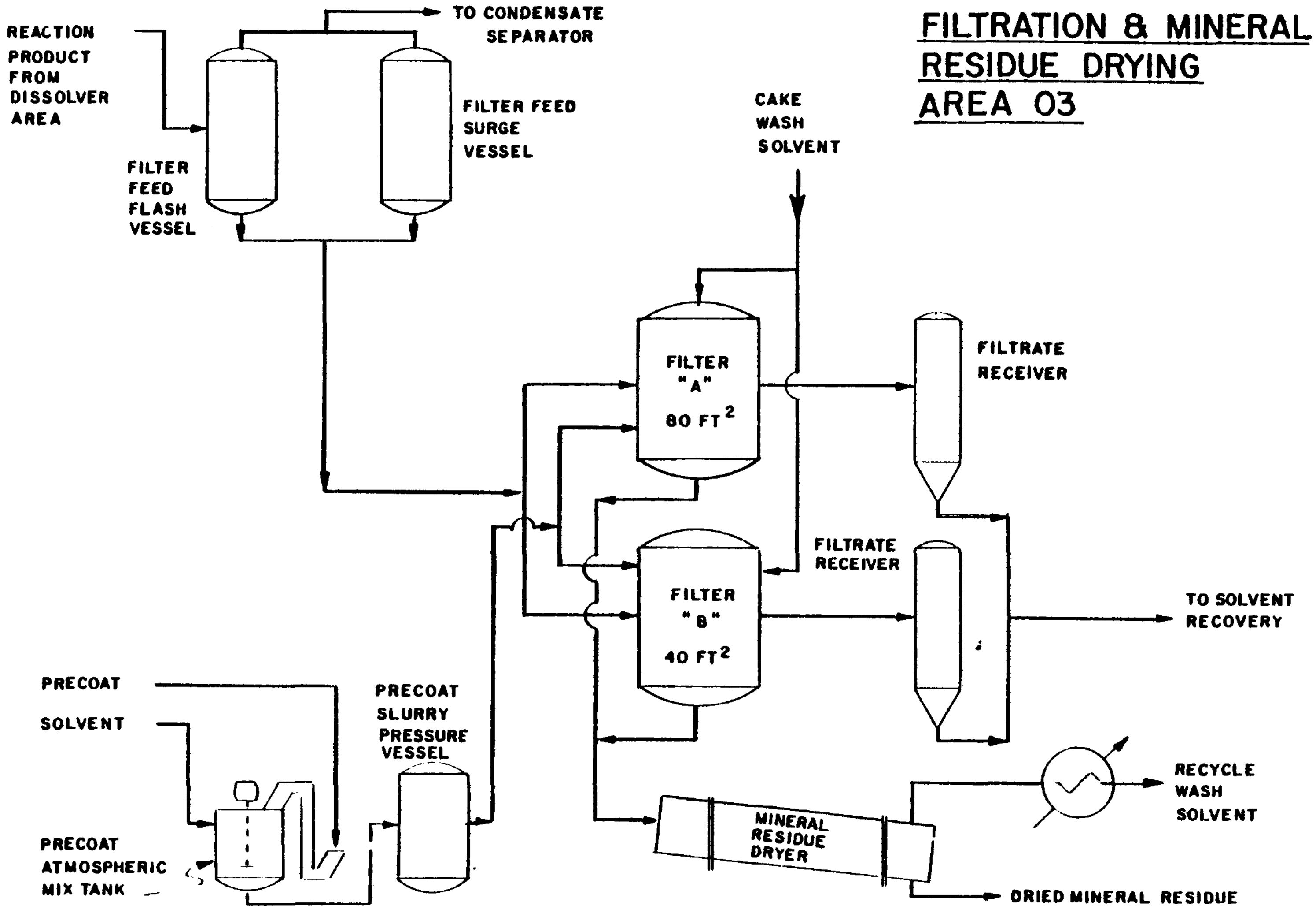


FIGURE 6

OPERATIONAL HISTORY OF "A" FILTER DURING PRODUCTION RUN

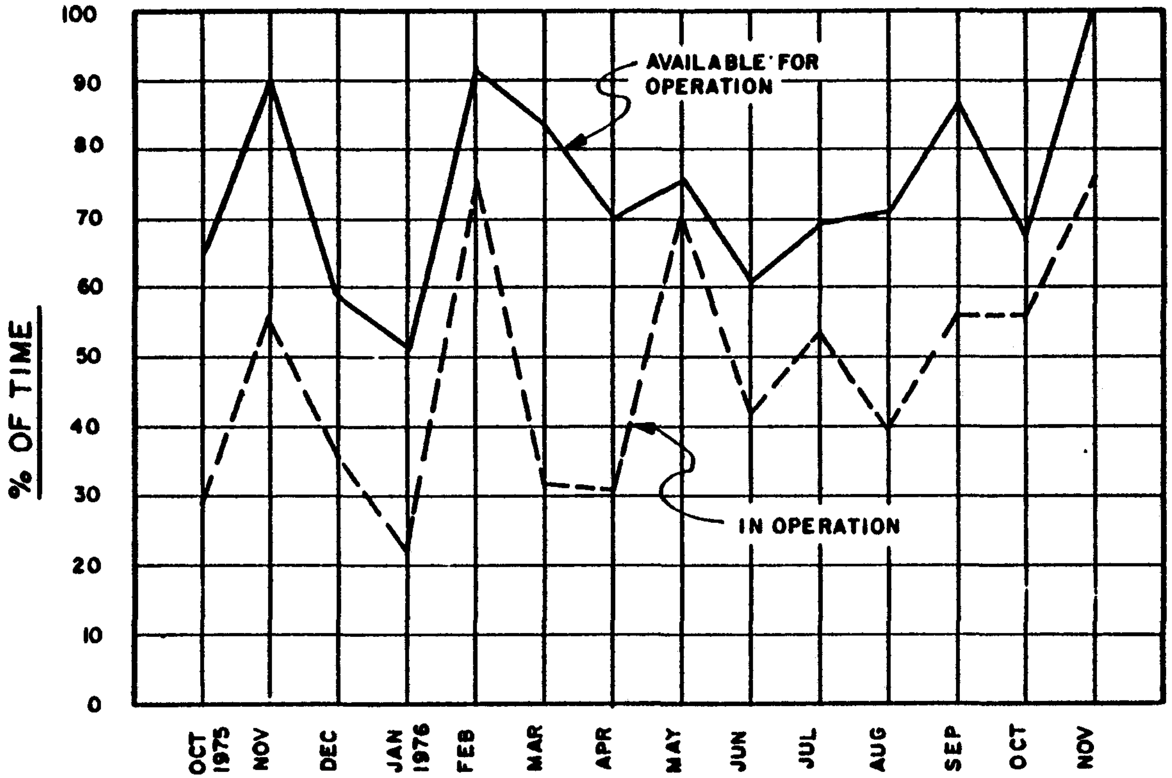


FIGURE 7

OPERATIONAL HISTORY OF "B" FILTER DURING PRODUCTION RUN

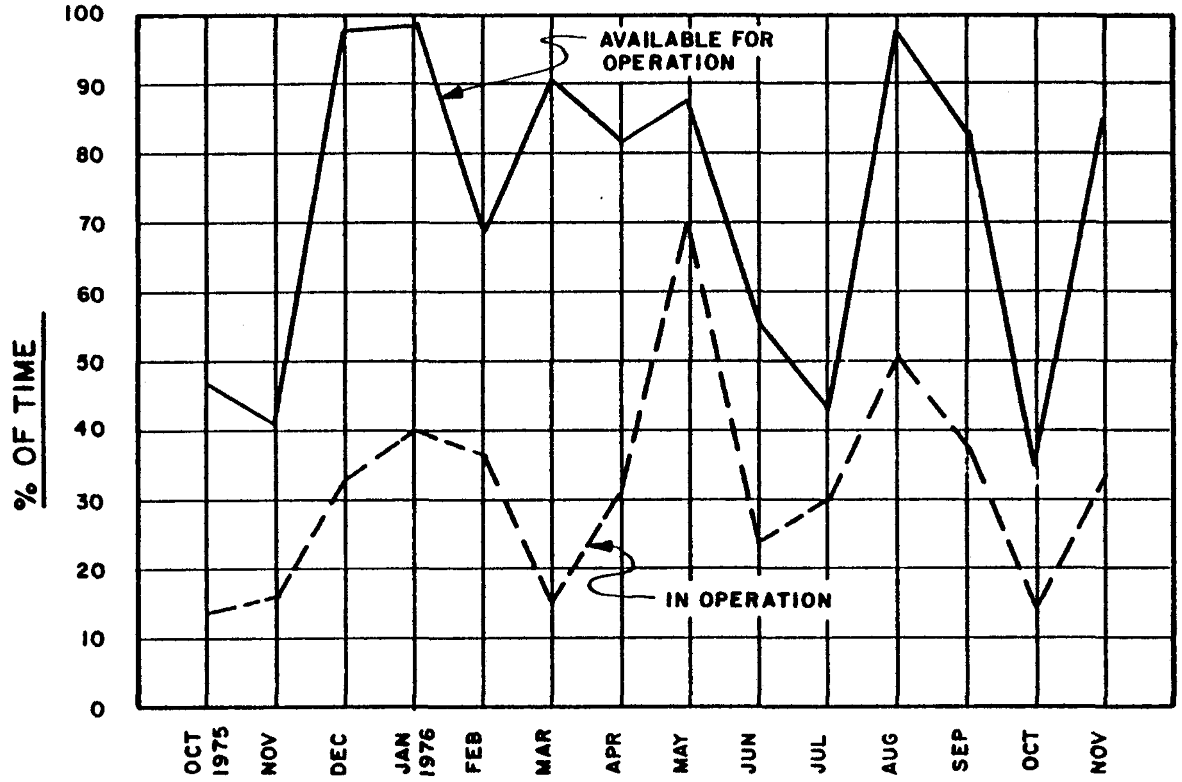


FIGURE 8

and not prevent sludge accumulation. Modifications to improve a second generation filter could include:

- Redesign of the vat to remove dead areas, as well as modifying the circulation loop through the filter to prevent settling.
- An effective vat sluicing system.
- High pressure screen sluicing. The screens are presently hydroblasted when they foul. This could be performed in situ with a clean solvent.
- With the above, it is possible that the agitator could be eliminated.

b. Drum Speed

The filter, as supplied, had provisions for drum speed control of from 1/5 to 1 RPM. Since increased rotational speed increased filtration rate, it soon became obvious that the full filtration rate potential was not being attained. The drum speed control was then changed to a range of 1 to 3 RPM and subsequently to 1 to 6 RPM. The Process Evaluation section of this report presents recent results of filtration parameter tests including not only drum speed, but also knife advance, pressure differential and other variables.

c. Doctor Knife

Filtration rates are highly elastic to not only knife advance rate, but also to the sharpness and knife stability as well. It is very important to maintain the knife in a sharp condition. It should be noted that there is considerable disagreement among the scholars of filter operation as to how the knife should be installed with respect to the beveled edge. The filters were received with the knife installed with the bevel facing down and this has not been changed. There is a school of thought that suggests that the bevel should be facing up. This should be investigated during subsequent filter test runs. At knife advance rates below 1 mil/min, the resulting filtration rate tends to be unstable. This could be a function of the knife advance stability as well as a cake penetration/blinding phenomena.

d. Differential Pressure

The filter pressure differential is not a natural one (e.g. - controlled by cake resistance). Instead, it is artifically maintained by two pressure control loops. The high side pressure is maintained by controlling the quantity of gas from the recirculating compressors to the filters -- excess gas is bypassed to the compressor suction. The low side pressure was originally maintained by venting excess gas to the flare but has been modified to include makeup of inert gas to the system when deficient (split range control loop). To improve the quality of filtration test data, a second set of gas flow transmitters have been installed in order to measure low gas flows which are characteristic of the filtration mode of operation. The filters have been operated at differential pressures ranging from 20 to 45 psi. These results are presented in the Process Evaluation section.

e. Precoating

The original precoating method consisted of establishing a hot solvent circulation loop through the filter, followed by injection of an asbestos basecoat on the downstream side of the filter, using a blowpot. Following deposition of the basecoat, precoat (diatomite) was injected from the precoat atmospheric mix tank to obtain approximately a 2% slurry at the filter screen. Pressure differential buildup was rapid. Drum speed was at maximum (1 RPM). Basecoat, as well as precoat deposition, was uneven, rough and tight. During these early operations, the Sier-Bath precoat pumps proved to be unusable due to packing leaks which could not be corrected.

A new basecoat mix and injection system, atmospheric mix tank and diaphragm pump was installed to control basecoat injection. The original Sier-Bath pumps were replaced with a piston pump and a centrifugal pump. The precoat procedure was revised to provide for beginning with an empty filter, followed by controlled injection of basecoat, precoat and circulating precoat slurry (from the precoat slurry pressure vessel) to result in an approximately 2 percent slurry contacting the screen initially. When the basecoat was depleted, the precoat was increased to hold an approximate 3% concentration. Pressure differential buildup was gradual. The result was a more permeable cake with increased rates. Precoating time was reduced to 3 to 4 hours.

Instrumentation for flow controlling the circulating slurry stream has been incorporated to better control slurry concentration. To reduce the quantity of solvent processed in the solvent recovery area, filtered coal solution was substituted for process solvent in the precoat procedure. The increased resistance from the more viscous filtrate was offset by increasing the precoat slurry concentrations to the 3% - 5% range.

A procedure was developed to recoat over the precoat heel following a filtration run, rather than sluicing the heel followed by precoating, as is normally done. Recoating is desirable from several points of view:

- Filter Aid Economy

  15 to 20 percent of each precoat is sluiced prior to reprecoating.

- Reduction in Screen Blinding

  A major source of screen fouling results from ash contamination of the filter screen at the beginning of the precoat operation (source of the ash is from solids that have settled out in the filter vat or piping dead legs). By recoating, the number of occasions that the bare screen is exposed to fouling is reduced.

- Reduction in Precoating Downtime

  Recoating should involve less time than sluicing/precoating, after the operating personnel have become familiar with the new procedure.

The recoating procedure was moderately successful in that three recoats were performed before sluicing was necessary. However, it did require more time (due to the learning curve phenoma) and the resulting cakes were not as smooth, which did adversely affect rates. Due to the urgency of obtaining maximum production rates during the 3,000 ton production run, further recoating experimentation was postponed.

Asbestos has been eliminated from use while precoating on the dutch twill screen. It is currently utilized for precoating over the spiral wound Johnson Screen which is more difficult to maintain a cake on. Elimination of asbestos on the Johnson Screen precoat will be attempted in the future. It is desirable to eliminate asbestos from a health and hygiene standpoint.

Refinement of precoating procedures has been minimal due to the production run. Now that it has been completed, it is hoped that this work can be resumed by fitting it into future filtration test run evaluations. Much work remains to be done on the effects of precoat types, slurry concentrations, drum speeds, etc., on filtration rates. It is believed that much of the auxiliary equipment used at this facility for precoating could be eliminated in a second generation facility.

f. Precoat Grade

The precoat grade refers to the particle size classification of the filter aid. Until now, only diatomites have been utilized as a precoat. Plans include a comparative evaluation of Perlites. Grades utilized have included the following, all of which are flux calcined:

	Relative Flow Ratio*	Mean Particle Size* (Micron)
Dicalite Speed Plus	700	8.8- 9.4
Dicalite Speedex	1030	10.0-12.0
Dicalite 4200	1800	18.0-20.0
Celite	545	20.0-22.0 (Dicalite Equivalent)

* From Vendor Literature

The above data would lead one to believe that the coarser grades should result in higher rates. The flow rate data, however, was derived using water. In actual practice, the coarser grades result in lower flow rates due to surface penetration blinding. A scanning electron micrograph of the filter feed material has indicated that as high as 85% of the feed slurry particles are of a size less than one micron. Use of the coarse grades require a greater knife advance to maintain rates. As a result, Speedex grade was used for the vast majority of filtration.

g. Cake Wash

Shortly after filter clarity was established for the first time, the use of cake wash was initiated. The wash solvent utilized is a process derived solvent with a boiling point range of 380-480°F. The use of the wash solvent both increased and stabilized filtration rates. It serves several functions:

- It displaces the higher boiling range solvents and SRC from the mineral residue cake. Its lower boiling point makes possible its separation from the mineral residue in the downstream mineral residue dryer. It is recovered for re-use.

- It increases rates by reducing the imbibed cake liquor viscosity. This rate function displays an elasticity, within limits, to both wash quantity and temperature. The quantity of cake wash used is normally in the range of 15-20% of the filtrate.

Much work remains to be performed on the effects of cake wash on filtration rate, as well as high boiling point solvent displacement.

h. Vat Level

As a result of a mechanical constraint, the vat level has never been operated greater than approximately one-third of drum submergence (diameter). Due to the internal trunnion bearings, and a concern for adequate lubrication, the maximum allowable level has been established at two inches below the bearings. Even for short term experiments it would be necessary to maintain the level somewhat below 40% diameter, since the unfiltered coal solution will overflow into the cake auger at 41%. Filtration rate is responsive to changes in level. Tests have been conducted which include this variable, the results of which are included in the Process Evaluation section.

i. Slurry Concentration and Temperature

Little effort has been expended on exploring the effects of feedstock composition and temperature. Whenever possible, the filtration temperature has been maintained at 500°F. The sight glasses have a maximum limit of 500°F. During intervals when heat exchange control has been lost due to erosion in the exchanger or piping loop, the filter temperature decreased to the 350-400°F range and considerable filtration rate losses were experienced. It is planned to replace the sight glasses with metal blinds in some limited future testing in order to perform high temperature (600°F) evaluations.

The slurry concentration has never been purposely varied for rate effect determination. During several runs on slurry recycle in 1975 (higher slurry concentration), it appeared that, while gross rates were diminished, the residue removal rate was little affected.

j. Filter Gas Temperature

The filter gas temperature has normally been maintained at 500°F. In early 1976, however, the ability to use "A" filter gas heater was lost due to a head gasket failure. No negative rate effects were experienced due to the lower gas temperature (200-350°F range). As a result, "A" filter continues to operate in this fashion. The only detrimental effect experienced has been a tendency for the precoats to dry out and drop off of the screen. This has been countered by utilizing cake wash sprays during the precoat cycle to maintain a saturated cake.

3. Mechanical Problems

A discussion of the many mechanical problems associated with the filters and the corrective action taken is discussed below.

a. Screens

Maintaining physical integrity of the filter screen has been a continual problem but much progress has been made. The outer screen serves to hold the precoat on the filter drum. The screen hole size must be uniformly small enough that the precoat does not pass through the screen and leave a spot which is unprecoated. Unprecoated spots allow unfiltered coal solution to pass into the downstream operations. The type of screen is of little importance as far as filtration rates are concerned as long as the screen is not blinded. The overwhelming resistance to filtrate flow results from the very thin mineral residue cake layer which forms as the first increment of filtrate passes through the precoat.

The original design called for the outer wire cloth to be caulked into each of the sixteen compartments. Caulking grooves were provided around the perimeter of each compartment. The design intent was to secure the wire cloth in the same manner as fabric cloth since the hold-down scheme which was provided was identical to the traditional fabric cloth drum filters. The method was unsatisfactory because the wire cloth would tear as it was being formed to fit the caulking grooves. The caulking around the perimeter of each of the sixteen filter compartments was abandoned for caulking only around the outer edges of the drum itself. The caulking groove was widened and the wire cloth was held in with lead wool. Winding wire was spiraled around the drum as had been originally specified. This screen attachment method was the first one found that did not result immediately in an off-specification filtrate due to screen holes. However, this lead caulking method was far from satisfactory. Other methods were considered or tried. The wire cloth manufacturer recommended that the caulking groove be widened further and made much shallower. They recommended that high-tensile round wire strapping be used to bind the wire cloth onto the drum with white asbestos tape as a gasket between the drum and the wire cloth. This method was never tried due to the difficulties of remaking the caulking groove and the complexity of installation. The screens were the cause of most filter outages while the lead caulking method was being used. Leaks and tears at the caulking points were a source of many shutdowns.

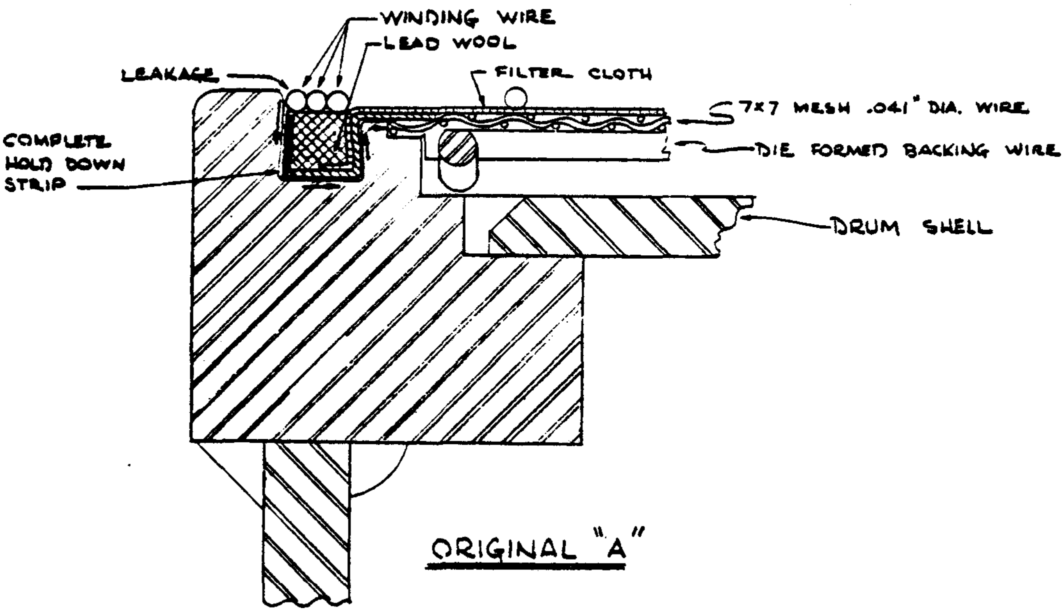
The wire cloth which was chosen was 24 X 110 Dutch weave because of its ruggedness and ability to withstand the punishment of the lead caulking method. Other secondary reasons were the better resistance to blinding of the Dutch weave as compared to other square mesh weaves and its ready availability.

Upon removal of an old screen in September, 1975, previously unnoticeable holes were detected in the "hold down" area. It was noticed that leakage was occurring under the edge of the hold down strip and eroding the screen. The holddown strips were modified by splitting them longitudinally and using only the inner half. The screen was caulked into the edges using solid lead packing rather than lead wool as in the past. This combination of only a half of a holddown strip and solid lead packing resulted in clear filtrate on both filters. It is postulated that the removal of the outside portion of the holddown strip allows the lead caulking to seal directly against the drum. This direct sealing eliminates flow beneath the holddown strip and up against the under side of the screen which was eroding it. The solid lead seems to "slip" into the holddown slot with less screen pulling than lead wool, thus eliminating small tears which, while undetectable at the time of installation, rapidly become worse and result in poor clarity. This modification proved to be beneficial. Figure 9 depicts this improved method of screen attachment.

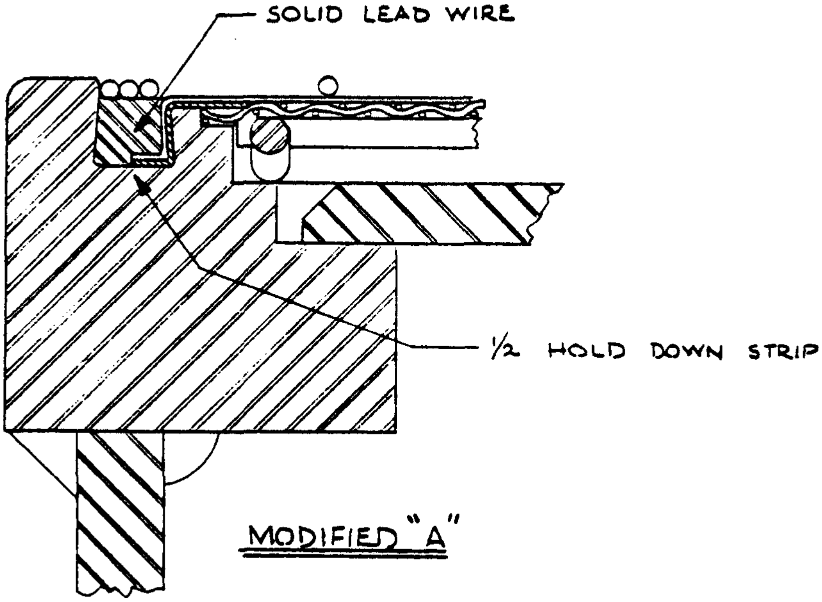
Since two problems associated with the screens have been leaks and blinding, a search for an alternate outer screen was initiated. A slotted screen consisting of multiple trapezoidal bars (wires) arranged parallel to one another was chosen for its ruggedness, long-life and great potential of resistance to blinding. It was supplied by the Johnson Division of Universal Oil Products Co. This screen costs about five times as much as a comparable wire cloth. The early experience with the slotted screen showed that it required more precise operational procedures. If a crack develops in the precoat, the crack will seal with the Dutch weave because the screen at the crack becomes blinded, whereas the slotted screen does not blind so the cracks cannot seal with mineral residue. The surface of the slotted screen is smoother, allowing a precoat patch encircled by cracks to fall off more easily. With careful operation, the slotted screen works satisfactorily. It is far more rugged than conventional screens.

b. Filter Lubrication

Since the supports for the filter drum and the agitator are inside the filter case, the lubrication of these bearing surfaces is a difficult problem. The lubricant must retain its properties at 500°F. The initial bearing surfaces had molybdenum disulfide powder applied to them. The original



ORIGINAL "A"



MODIFIED "A"

MODIFICATION TO ROTARY DRUM FILTER SCREEN HOLDING RING

FIGURE 9

lubricant was Dubois grease. Since the Dubois grease deteriorated with time, it was replaced with Mobil Hi-Temp. About the same time the Mobil Hi-Temp was substituted for the Debois, problems with seizure of bearing surfaces began. Although not known until later, the reason was that the molybdenum disulfide film had disappeared. When Dubois was substituted for Mobil, the seizures did not stop. A high temperature synthetic grease, Extemp 9901, was tried next. It worked well but was very expensive and its high viscosity created difficulties with the performance of the automatic lubricator. Next, Dow Corning Molykote 41 was tried because of its lower price and easier handling characteristics. The Molykote 41 worked well. Both the Extemp 9901 and the Molykote 41 contain solid lubricants. Externally located bearings would greatly minimize lubrication problems.

c. Filter Knife

The filter knife deserves a great deal of attention. Although the knife appears to be a simple part of the filter, a misdesigned knife can have a very detrimental effect on the performance of a filter. The knife supports must be rigid, so as not to fail or even noticeably flex under the severest cutting conditions. Knife flexing causes cake smearing which results in decreased filtration rates. The original Stellite blades were found to be too flexible. A composite blade of carbon steel, with a D-2 tool steel edge, proved satisfactory. A 17-4 PH blade was abraded away very quickly. A small Stellite blade inserted on the edge of a 17-4 PH holder worked satisfactorily. For best cutting, the angle of the knife to the drum should be 20° to 30°.

The angle of the knife from the horizontal was increased from 47° to 60° in an attempt to decrease the sticking of of the cake to the knife and, hopefully, to eliminate the need to wash the cake off of the knife. However, even at the 60° angle, the cake sticks to the knife and requires that a cake wash down be used.

d. Cake Removal Auger

The original auger used to transport the cake from the cake hopper to the discharge piping was a carbon steel solid screw operating at 60 rpm. The mineral residue balled-up on the solid scroll. The literature related that the auger should be a ribbon-screw and operate at 20-30 rpm and that polished surfaces (stainless steel) help to prevent sticking. A stainless steel ribbon-screw auger was installed and operated at 30 rpm in both filters. This resulted in much better performance.

e. Cake Hopper Level Control

The original device to control the level of the mineral residue in the cake hopper was a Sier-Bath pump which had been wired to run backward to let the pressure down to the pressure of the mineral residue dryer. The pumps were intended to work like a rotary airlock. These pumps did not work as pressure letdown devices because the seals leaked profusely and the rate could not be controlled. The pumps were replaced with double Camflex control valves installed in series. The valves operated on a timed sequence in lockhopper style. This scheme worked reasonably well. The major problem was erosion of the plug of the Camflex valves. Later, a Willis multiple orifice valve was installed to replace both Camflex control valves. The multiple orifice valve throttled the cake from the cake hopper to control the level. These valves have shown little sign of wear in the considerable period (18 months) that they have operated. The cake is normally 50% solids and the pressure reduction is approximately 115 psi.

Although it would be beneficial to deliver as dry a cake as possible to the mineral residue dryer, it always has been necessary to use wash to remove the cake from the knife and to add additional wash solvent to slurry the cake for removal from the filter and transfer to the mineral residue dryer.

f. Stuffing Boxes, Rotary Joints and Rotary Valves

The main drive packing gland had to be remade because the gland follower was undersized.

Two types of rotary valves for filtrate removal were supplied with the filters -- a fixed rotary valve and a spring loaded, bellows floating valve. The fixed rotary valve generally performed unsatisfactorily because of the difficulty of obtaining proper alignment between the valve faces. Initially the floating valve failed mechanically. The support pins or dogs were too small to resist the twisting moment. It worked satisfactorily after the support pins were enlarged to adequately compensate for the torque to which the valve is exposed.

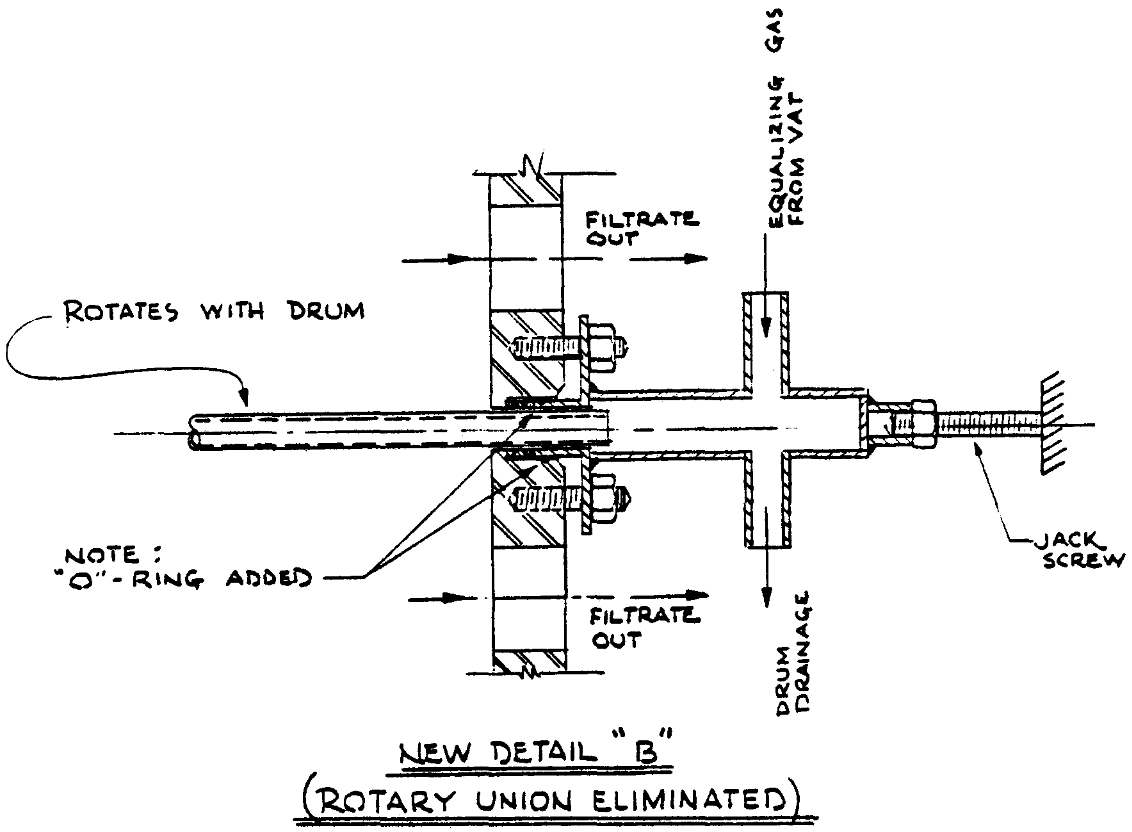
Since the seal rings on the knife support rods did not provide adequate sealing, they were replaced with stuffing boxes.

During the first year of operation, frequent leaks occurred which were associated with the rotary unions and stuffing box. These are part of the internal drum pressure equalizing system. This original design consisted of a 1" rotating pipe extending through a stuffing box to the exterior of the filter. This pipe was connected via a rotary union to a stationary 1" pipe leading to the filter vat. In this manner the interior of the drum was maintained at vat pressure. Due to these recurring leaks, the pressure equalizer line was modified. The rotating pipe was cut off just outside the filter. A chamber, into which the rotating pipe discharged, was welded to the outside flange of the stuffing box packing gland. The equalizing line was connected to this chamber. The need for the troublesome rotary union was eliminated. A sketch of this modification is shown by Figure 10.

g. Sight Glasses

The sight glasses provided with the filters are poorly located. Due to the location of the glasses, only the middle of the cake can be seen on the larger filter. This is unfortunate since the "funnels" in the precoat which occur when a screen leak occurs cannot be observed during operation of the large filter. The ability to observe the entire cake surface during operation is of utmost importance and should be provided when larger filters are manufactured. Many breakdowns in the knife, screen, agitator and auger parts of the filter could have been prevented and better diagnosis of the cause of poor filtrate clarity would have been possible if the sight glasses had been so mounted as to permit better viewing. The exact mechanism of failure could be more quickly determined if its failure could be observed rather than attempting to reconstruct what happened after the head of the filter has been removed.

The sight glasses supplied with the filters consisted of two layers of 3/4-inch glass with 75 psig inert gas between the glasses. Sealing was accomplished by compressing a flat gasket which created high compressive stress around the outer portion of the glass but none in the center. On numerous occasions, one glass cracked and a replacement was installed as soon as possible. In November, 1974, both glasses in one port failed simultaneously and a large amount of solvent and gas was released violently into the filter building. All sight ports then were blinded with 3/4" steel plate until an improved type of glass could be obtained. Safety glass assemblies manufactured by PresSure Products Co. were selected as having what was believed to be the best safety features available. This assembly consists of two glasses, each with a 300 psig design rating with a safety factor of 10. They are secured in a stuffing box and are sealed by compression of packing around the outside circumference of the glass. This type of seal imparts a uniform compressive stress throughout the entire glass. This stress



MODIFICATION OF VALVE END ASSEMBLY, ROTARY DRUM FILTER

FIGURE 10

is sufficiently great so that the glass is retained in its holder even if shattered while at operating pressure. It cannot be emphasized too strongly that sight glasses on larger filters should be of the stuffing box type. Due to a 500°F maximum working temperature for these sight glass assemblies, all filtration work has been performed at a temperature of 500°F or less. In the future, the sight glasses may be replaced temporarily with steel plates in order to perform filtration tests at higher temperatures. However, precoating, operation and sluicing are very difficult without the ability to observe the filter drum surface.

h. Agitator

The agitator is a key part of the filter and its importance is not always realized. The agitator is supported from the trunnions of the filter drum. The original agitator was so designed that it twisted easily and bound on the trunnions. The agitator sides were reinforced to prevent the twisting. At the same time, the inlet baffle on the bottom inlet of the filter housing was removed and a baffle was attached to the bottom of the agitator. Numerous failures of the agitator drive linkages have occurred and many different schemes have been tried to give longer life. None of these methods can be deemed to be completely successful. The best strategy for future filters would be to eliminate the requirement for a mechanical agitator.

i. Screen Blinding

If a bare filter screen is precoated with a stream containing mineral residue, the screen will be blinded in proportion to the percentage of mineral residue in the precoat stream. The mineral residue can contaminate the precoat by an operator error or, more frequently, from build-ups of solids in stagnant zones inside the filter. The stagnant zones must be designed out of the system. The operation of the filters has evolved so that if the agitator linkage is discovered to have come loose, the filter is shut down immediately to save the screen from being blinded.

j. Nozzle Problems

Several nozzle headers are inside the filter. Originally there were three cake wash nozzle headers, one sluice header, one knife wash header and one sight glass wash header. One cake wash header was deleted to provide a vat wash down header. This wash down has been only marginally successful. The sight glass wash header was ineffective and was converted to a cake hopper spray header. Each header is connected to the filter housing with a flexible hose. The flexible hoses have developed leaks, especially on the high pressure sluice line which is so oriented that the hose must be physically restrained to prevent it from expanding and contacting the rotating filter drum.

When solids inadvertently contaminate the wash solvent or the process solvent, the respective nozzles become eroded.

The knife wash nozzles are especially susceptible to becoming plugged since they are operated in an on-off fashion. Drips left in the nozzles chemically react and solidify at the filter temperature. The wash solvent heaters also coke up, producing solids to plug the nozzles downstream. Aiming and cleaning of the nozzles during each filter outage has been found to be very important for good filter operation.

k. Instrumentation

The pressure control loops were changed to give better differential pressure control for filtration. Electronic instruments now measure knife advance rate and amount of travel. These signals were routed to the computer to increase the accuracy of filter studies. A new wash solvent flow loop was installed to measure the wash solvent flow to the cake wash separately from that used for knife wash and cake slurrying. Board mounted recording ammeters were installed on both filter drive motors to monitor the requirement of lubrication and as a troubleshooting aid.

l. Bearings

The main filter bearings are located inside the filter housing in a very hostile environment. The seals originally supplied disintegrated due to temperature and solvent effects. The lips placed on the bearings to hold the seals were greatly undersized so initially the seals simply fell out. After the seal groove was remachined, the seals stayed in place but were quickly chemically and/or thermally destroyed. The bearings are secured with drilled and tapped machine screws. The drilling and tapping of the bearing shells and the housing were done in one operation without prints. When replacement bearings were installed, the countersunk holes of the new bearings did not line up exactly in angle or location with the tapped holes in the housing which resulted in the new bearings being installed with a slight twist which subsequently caused binding. The binding problem was solved when new bearings were installed by repeating the original shop method. One set of factory replacement bearings turned out to be undersized by 0.010 inch. The manufacturer specified a bearing clearance of 0.007 inch. After this clearance was increased to 0.030 inch and the twisting eliminated, the bearing binding problem was eliminated. The bearings wear excessively compared to industrial bearings. The best solution to bearing problems would be to place the bearings on the outside of the filter or, if the bearing must be on the inside, overlay the shaft with Stellite and provide a Stellite bearing surface.

4. Process and Mechanical Performance, Auxiliary Equipment

a. Vessels and Mixers

The precoat atmospheric mix tank has always been inadequate to mix concentrated (15%) filter aid slurry in an efficient and timely manner. Originally, the mixing system used an elevator to dump filter aid into the baffled tank which included a centermounted turbine agitator. Whenever the tank level increased above the incoming dilution solvent, wetting of the filter aid became extremely difficult. It was necessary to use wooden paddles to manually assist in wetting the agglomerated filter aid. Modifications included removal of the tank baffles, addition of a second turbine blade and off-setting the agitator. While the resulting vortex action did improve mixing, it is still necessary to manually assist in the mixing operation.

Considerations for new facilities might include the use of gravimetric feeders, mixing cones and in-line static mixers. In this way, most of the present atmospheric mix system, as well as parts of the pressure system, could probably be eliminated.

The filter feed surge vessel has experienced numerous problems as a result of its use pattern. All reclaimable solvents, slurries and sludges are returned to the process via this vessel. In October, 1975, the bottom portion was distorted due to excessive temperature caused by one or a combination of the following:

- Improper location of the thermostat controlling a 60,000 watt external surface mounted electric heater (1140 watts per square foot).
- Insulating sludge deposits on the internal shell resulting from a broken agitator shaft.

The 15/16" thick tank wall was "bulged" approximately 1-1/2" in the lower 3 feet where the heater was installed. The deformed areas were replaced with new metal in the field by the original vessel vendor. The electric heater was scrapped and replaced by a P&M designed, locally purchased, Dowtherm heated exchanger. An existing centrifugal pump was used to provide circulation. The pump suction was modified to draw from the vessel bottom rather than the side to provide constant purging of solids. The vessel was reinsulated and returned to service after the ASME code inspector certified the repairs.

b. Heat Exchangers

The primary difficulties experienced with exchangers in the filtration area pertain to erosion, fouling and coke formation.

The filter feed flash vessel heater and the precoat slurry pressure vessel heater were originally fixed tube, shell and tube heat exchangers. The tubes were carbon steel. Carbon steel corrodes when subjected to high temperature coal-derived liquids. After a leak developed on the filter feed flash heater, both heaters were replaced with locally manufactured heavy-wall double-pipe heaters. Double pipe was used, instead of stainless steel tubes in a shell and tube exchanger, because of speed of delivery of the double-pipe components.

Corrosion-erosion problems have occurred in all of these double-pipe heat exchangers. The Corrosion-Erosion Section of this report details the failure mechanisms more thoroughly.

The filter solvent supply exchangers have experienced considerable fouling problems. These units utilize Dowtherm to heat wash solvent (480°F endpoint). Wash solvent tends to coke readily.

The problem has been aggravated by loss of many of the tube fins during mechanical cleaning which results in subsequent overheating and, thus, more rapid coking. Problems were also experienced with these exchangers relative to frequently blowing process side flange gaskets. This problem was corrected by the development of modified operating procedures.

c. Insulation Caused Fires

The process-derived oil will ignite spontaneously if it soaks into calcium silicate insulation. There is a combination of wicking and catalytic effect from this insulation. Numerous such insulation caused fires have been experienced throughout the plant. The insulation on the filter feed surge vessel was removed and replaced with a non-permeable insulation called Foamglas (PPG Industries).

d. Floors

The ground level of several areas of the plant were originally provided with a smooth concrete finish. This finish, combined with recurring oil spills and the use of rubber footwear, caused a very slippery condition. These floors have been roughed-up with a special machine. Although housekeeping will be more difficult with the rougher surface, a serious safety hazard has been minimized.

e. Compressor Knockout Drums

The filter gas compressors experienced a rash of failures. Liquid in the feed gas was diagnosed as the problem. The carbon steel mist pads in both filter gas compressor knockout drums were found to have been corroded away. They were replaced with stainless steel mist pads.

f. Pumps

Many of the pumps in the filtration area handle slurries. Mechanical seal failure and erosion have been serious problems. Detailed discussions are provided in paragraph III.Q, "Pumps".

E. Mineral Residue Drying

1. General

As a result of substantial progress being made in solving major problems associated with the filters and solvent recovery system, the mineral residue dryer has emerged as the production "bottleneck" of the pilot plant. The major problem is associated with coking and resultant capacity loss. Both short term controlled test data and longer term production results indicate that the dryer has a capacity, when clean, equivalent to only about 35 tons per day of coal feed. This, coupled with four or five days downtime during each month for decoking, seal repair, vent line unplugging or patching, means that the main production limitation in the future will be the mineral residue dryer.

The operational history of the mineral residue dryer during the long production run is shown by Figure 11.

2. Process Performance and Problems

The mineral residue drying system is, without a doubt, the most difficult unit to operate within the plant. The primary reason is that the vast majority of the instrument control loops must be operated in a manual mode to optimize the dryer output.

A second factor that complicates the operation relates to the lack of any positive feed flow indication. The operator is basically in the dark on feed changes. If the feed tank level decreases he is unsure as to whether the flow to the dryer has increased or whether the flow from the filter to the feed tank has decreased -- the feed tank is relatively small (approximately 350 gallons between the level taps).

Feed composition is another complicating issue. Of course the dryer performs better at higher slurry concentrations but to minimize filter discharge problems, attempts are made to maintain a 45-50% concentration to the dryer. This ranges, however, from 30-60% which markedly affects the unit performance in an adverse manner.

OPERATIONAL HISTORY OF MINERAL RESIDUE DRYER DURING PRODUCTION RUN

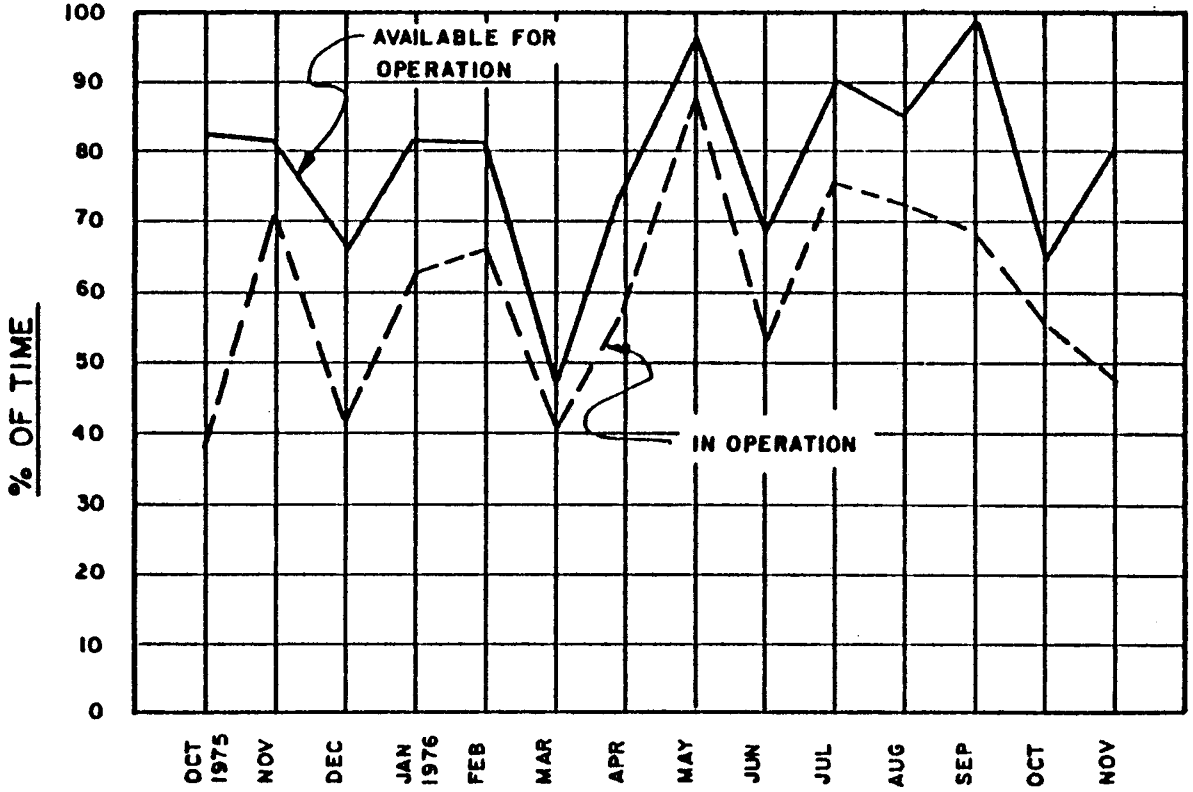


FIGURE II

Difficulties occur if the feed to the dryer contains oils which boil above the dryer's operating temperature. Wet product, coking and a plugged vapor line usually results. There are two main ways in which the dryer can be fed high boiling range material. First, if the level in the filter is raised too high, unfiltered coal solution overflows into the cake hopper and then to the mineral residue dryer. Secondly, upsets in the solvent recovery section can result in high boiling point material contaminates in the wash solvent stream.

Due to the large thermal inertia of the mineral residue dryer, startups are very slow since only a step change of feed rate to the dryer sometimes causes off-specification material before the temperature controls respond to deliver a greater amount of heat. When the dryer becomes coked inside, the thermal inertia increases.

A large feed surge volume was installed to minimize composition fluctuations, but it was used only briefly due to pipe plugging problems. Also, the lower temperature of the feed from the large surge vessel reduced considerably the dryer capacity. For long term, high capacity operation, the source of the plugging problem needs to be eliminated and a preheater installed on the dryer feed line.

Much of the mineral residue dryer operation has been at reduced rates. Due to the urgency to produce SRC toward the 3,000 ton burning test requirement, the dryer was kept in operation, even at reduced rates, as long as practicable, before shutting down for decoking, repairs, etc.

The dryer normally produces a material very low in residual solvent. Its pyridine insoluble content usually is around 97%. If the solvent content increases, a shutdown usually results due to chute plugging.

3. Mechanical Problems

a. Coking in the Dryer

Coke formation has been, and still is, the major problem associated with mineral residue drying. The coke is very hard and strong due to the aggregate effect of its mineral residue content. It has, at times, built up to a thickness as great as 12 inches. When coke formation is detected or suspected, the dryer operating temperatures are reduced. This allows continued operation at reduced rates but frequently results in other problems, such as plugged chutes, and usually results only in the postponement of the shutdown required for decoking. In an effort to decrease coke formation and build up, 1100 feet of heavy log chain was draped throughout the dryer parallel to its main axis, anchored at intervals of about 6 feet. The hope was that the movement of the chain as the dryer rolled would retard coke formation by scouring the heat transfer surface and would physically remove any coke which did form. It was also felt that the large mass of chain would provide appreciable conductive heat transfer area. This modification served to increase run time to about three weeks betwen shutdowns for decoking. However, chain support pieces and chain links broke off periodically and stuck in the dryer discharge chute, necessitating partial dryer shutdowns to remove the broken pieces. A steel I-beam of dryer length was installed to replace the chains to eliminate the scrap iron problem and to improve run length time. A newly installed I-beam rolls at first and cleans the dryer but, with time, the edges of the I-beam become dull and the beam slides. Coke formation results. The I-beam and the shackle which retains the I-beam in the dryer have been corroded.

b. Mineral Residue Dryer Seals

The dryer feed and discharge breech seals have been replaced several times. Maintaining acceptable alignment and flatness of the mating surfaces is difficult. Frequent maintenance is required to prevent excessive vapor leaks from these seals.

c. Condensation, Vent Line and Eductor Plugging

The dryer gas and slurry flow originally was co-current. This direction of gas flow aggravated wet product and chute plugging problems. The gas flow was switched to countercurrent. It is desirable to have the dryer vent line as short as possible and to have the vapor flow leaving the dryer at the highest practicable temperature to minimize condensation in the line and resultant plugging. Consequently a snorkel was inserted into the dryer to vent the gas from inside the drying zone.

The original installation included a cyclone separator and a six-inch vent line. The cyclone separator did not work because of the low velocities of the gas, the small particle size of the mineral residue dust and plugging due to mud formation resulting from condensation. The six-inch vent line was vastly oversized. The vapor velocity was too low, which allowed mineral residue dust to settle out. It was replaced with a two-inch line which worked well until its elbows began to erode. The two-inch vent line was replaced with a three-inch heavy wall line.

As mentioned above, condensation in the vent line will cause plugging. The vapor is not superheated, but near its dew point when it enters the pipe. Liquid on the walls collect dust from the vent gas and eventually build up to plug the line. To prevent condensation, the line must be kept hotter than the dew point of the dryer vapor. The dryer vent line was made the most heavily electrically heat traced line in the entire plant. Whenever the dryer feed contains high boiling range materials, a rash of vent line plugs usually occurs.

The eductor, which removes the vapor from the dryer and into the circulating stream of solvent for condensation, has plugged on two occasions. The cause was determined to be air leakage into the eductor which caused the dust to burn, leaving a hard-packed reddish ash. The air leak was stopped by welding up the old six-inch vent line nozzle. Wash down nozzles were installed to continuously wash down the sides of the eductor. One or both of these two changes were effective since plugging has not occurred since they were made.

d. Dryer Condensate Pump

The dryer condensate pump provides the motive force for the solvent flow through the eductor which induces the vapors to flow out of the dryer. The net positive suction head available on this pump was found to be marginal. When the normal working level of condensate in the drum was raised to provide more net positive suction head and to keep the pump from cavitating, the danger of liquid carryover from the vessel was increased. The Lamson blower, which removes noncondensables from the dryer condensate drum, was inundated at times with oil carryover. Inspection of the dryer condensate drum revealed that the mist pad was corroded, plugged and displaced. A new stainless steel mist pad was installed. With the new pad, the Lamson blower has run reasonably well but it is oversized and operates near or at its surge point.

A spare dryer condensate pump was installed after it was discovered that the original impeller had been eroded away by the mineral residue in the condensate. The new pump has a lower net positive suction head requirement than the original pump.

e. Dryer Flare Tie-In

The emergency vent from the dryer originally was routed to the atmosphere. Since a catastrophic fire could have resulted from this arrangement, the emergency vent was connected to the flare. The emergency vent valve is a pneumatically operated, air-to-close, butterfly valve. It is opened by a 3-way solenoid valve actuated by a high pressure switch. This emergency setup worked fine when the vent was routed to the atmosphere but tying the vent line into the flare header inadvertently created a serious hazard when the dryer was decommissioned for maintenance.

The flare header normally operates at a slightly negative pressure. However, once while the dryer was down for maintenance, the flare header pressure became positive and H_2S gas backflowed from the vent header into the dryer system. A maintenance man was overcome, fell and was seriously injured. A locked block valve was immediately installed in the emergency vent line. The valve is chained open during operation and chained shut when the dryer is shut down.

f. Dried Mineral Residue Handling

The outlet of the dryer breech originally consisted of a rotary air lock which fed the cooler. This air lock was constantly being plugged. The air lock was moved to the discharge chute of the cooler and a dogleg removed from the dryer discharge chute. A large slide valve was installed in the chute between the dryer and the cooler so that the cooler could be maintained without shutting down the dryer. The rotary air lock on the rear of the cooler was replaced with a lock hopper consisting of two automated, pneumatically operated 6" slide valves with a spool piece between the valves.

Dried mineral residue is potentially pyrophoric. A fire erupted and engulfed the dried mineral residue silo the first time unloading was attempted. Very quick and correct action by personnel prevented a potentially catastrophic fire. The use of the silo was terminated. Mineral residue then was stored in air tight 55-gallon drums to minimize the fire hazard. A special drum handling system, complete with a weigh scale, rollers, retractable dust hood and dust collector, was installed to protect personnel from the hazardous mineral residue dust.

g. Miscellaneous

Miscellaneous comments regarding the operation and performance of the mineral residue dryer are listed below.

- Rod-out devices were installed on the dryer discharge breech, the dryer discharge chute and the cooler discharge chute.
- It was found that coke sometimes can be spalled off of the dryer walls by cooling the dryer quickly and reheating it quickly.
- The thermocouple on the outlet of the dryer which is supposed to rest in the residue bed has a tendency to bend out of the bed and give false readings. This

creates serious problems since this is the control point for the drying section of the dryer.

- A small section of the dryer shell was distorted due either to excessively high temperature as the result of an excessively deep ring of insulating coke or to a malfunctioning burner control.

F. Solvent Recovery

1. General

A large number of unexpected operational and mechanical problems have been encountered during the operation of the solvent recovery area. Satisfactory solutions have been developed for them. However, many of these solutions were not immediately obvious but evolved through a series of modest improvements combined with some "giant leaps".

The original design provided for the flow shown by Figure 12. This system provided, as a first step, the separation of SRC from the more volatile solvents in the vacuum flash drum. The SRC flowed from the bottom of the drum to the Sandvik cooling belt for solidification. The overheads were condensed and then fed to the light ends column where a light oil or naphtha product was collected overhead. The bottoms flowed to the wash solvent column where it was separated into an overhead product (wash solvent) and a bottoms product (process solvent). Operation in this mode provided good separation into specification cuts of SRC, process solvent, wash solvent and light oil. Regrettably, though, coking of the vacuum flash preheater coil occurred frequently. There were strong indications that this coking was related to the more volatile components in the feed. Consequently, in December, 1975, the solvent recovery section was modified as shown by Figure 13. The packing was removed from the light ends column and the column converted to an atmospheric preflash with its underflow serving as feed to the vacuum flash preheater. The overheads from the modified light ends column were partially condensed and combined with the condensed overheads from the vacuum flash drum. This combined stream was fed to the wash solvent column where it was separated into wash solvent and process solvent. The non-condensed vapors from the top of the modified light ends column flowed to a small newly fabricated stripper tower for recovery of a light oil product. This mode of operation contributed greatly toward eliminating the coking problem in the vacuum flash preheater. However, it was not possible to produce light oil and wash solvent in the desired boiling point ranges. Each was contaminated in substantial amounts with the other.

ORIGINAL SOLVENT RECOVERY FLOW SCHEME AREA 04

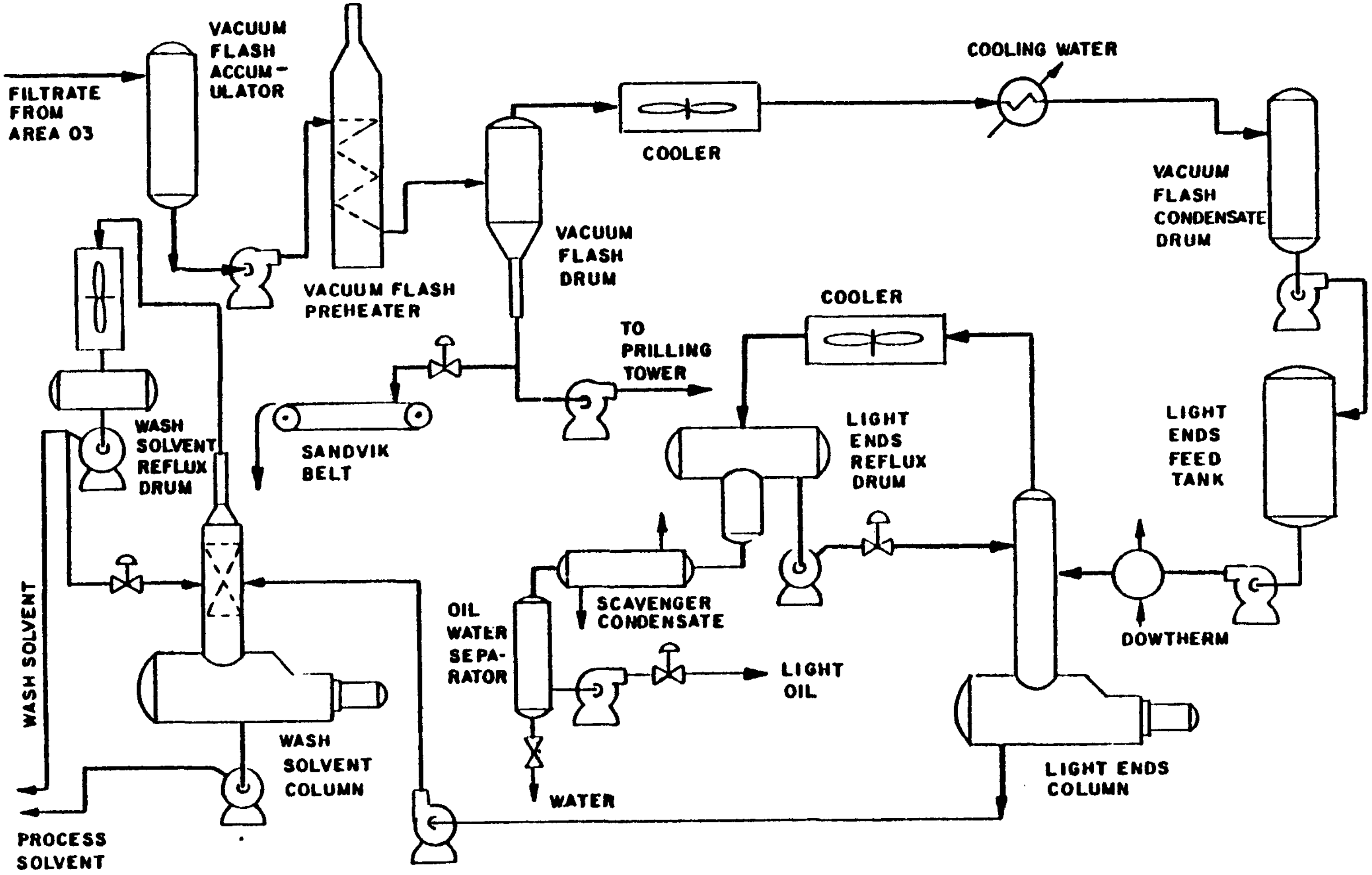


FIGURE 12

MODIFIED SOLVENT RECOVERY FLOW SCHEME AREA 04

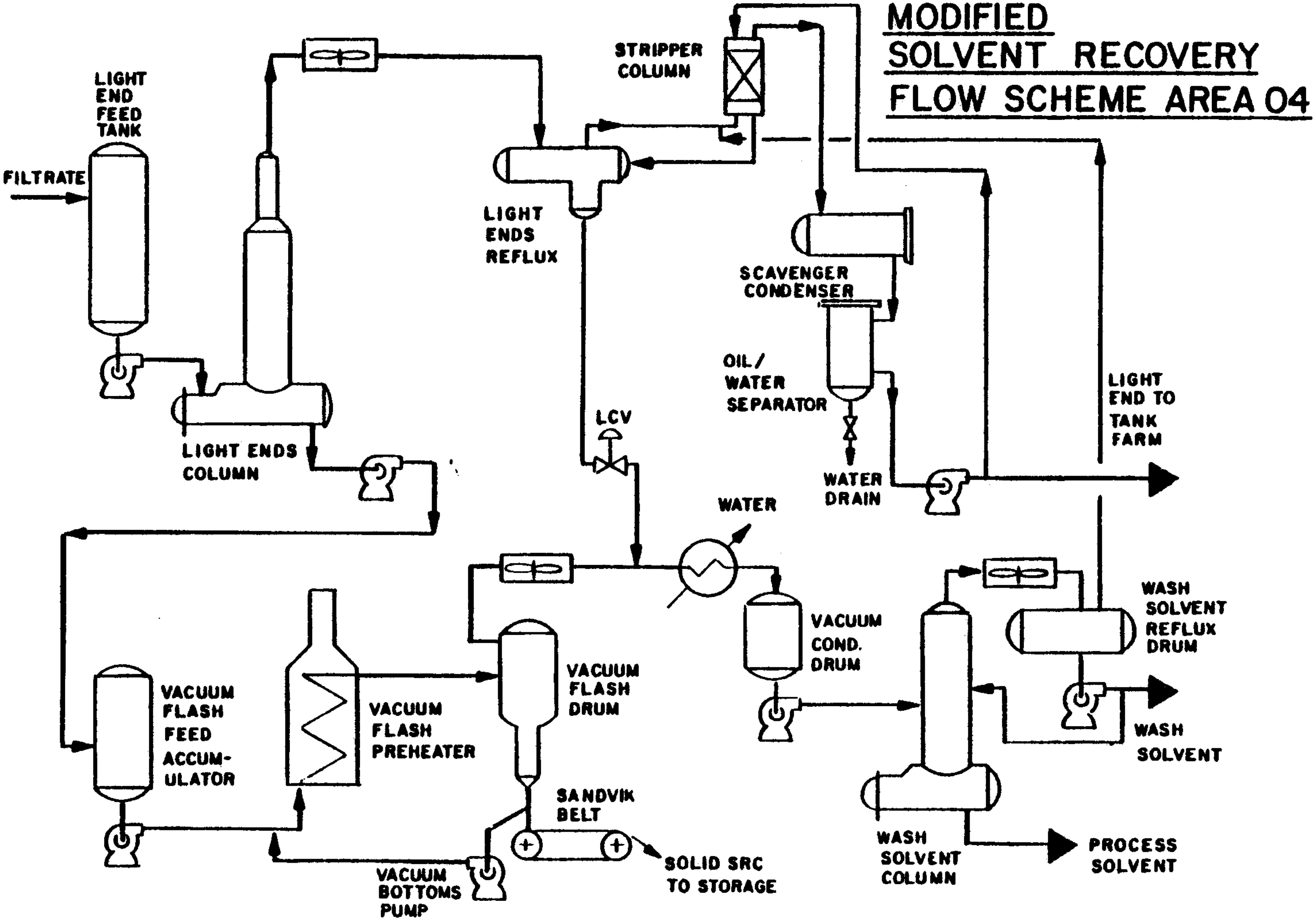


FIGURE 13

2. Process and Mechanical Performance

The process performance and mechanical performance of this section of the plant have been closely related and interwoven. An improvement in one generally was beneficial to the other and, in some cases, improvement in both was required in order to solve serious problems. Consequently, both process and mechanical performance will be covered when various portions of the solvent recovery system are discussed below.

a. Vacuum Flash System

During the original mode of operation of the solvent recovery system, frequent shutdowns were required to decoke the vacuum flash preheater coil. During a decoking operation in the fall of 1975 the coil became plugged with spalled coke which collected at thermowells. It became necessary to cut the coil in several locations and mechanically clean it by hydroblasting and the use of a pneumatic "tube rattler". It was observed that most of the coke occurred in the upper part of the coil (flow is from bottom to top) and that it primarily was deposited on the bottom half of the helical coil cross-section. Inspection of the long run of piping between the preheater and the vacuum flash drum revealed a large amount of coke deposits on the bottom half of horizontal piping and essentially none in vertical runs. This strongly indicated that two phase flow was occurring in the upper part of the helical coil and in the downstream piping, that the viscous liquid phase was settling to the bottom and moving relatively slowly so that residence time was sufficient for coking to occur and that the vapors were moving along the upper part of the coil tubing and horizontal piping. Apparently the reason for no coke formation in the vertical runs of piping was that the liquid phase was pumped upward in slugs, much the same as the flow in a vertical tube evaporator.

In order to prevent two phase flow with resultant coking, the following corrective actions were taken:

- The light oil and much of the wash solvent was removed from the feed to the vacuum flash preheater by conversion of the light ends column to an atmospheric preflash tower as described above.
- A system was installed to circulate vacuum flash drum bottoms (SRC) back to the inlet of the vacuum flash preheater coil. This made it possible to:
    - Maintain a higher flow rate (and thus lower residence time in the coil) during normal operation.

- Maintain design flow rates through the coil during startups, shutdowns and periods of reduced feed availability.

- Decrease the vapor pressure of the material flowing through the coil by the addition of large quantities of high boiling SRC.

- Utilize the sensible heat of the recycled SRC to provide heat of vaporization of the reduced percentage of volatile material flowing through the pressure control valve into the vacuum flash drum. This made it possible to reduce the normal outlet temperature of the coil from 800°F to about 690°F.

- Operate with a higher back pressure on the coil to minimize two phase flow.

The development of the SRC recycle system described above, to one which performed satisfactorily, was evolutionary. The original system utilized a Sier-Bath pump to pump the molten SRC to the Sandvik belt and to circulate back to the vacuum flash preheater during startup and shutdown. However, these pumps were removed because of seal leakage and their inability to handle the less viscous startup and shutdown flows which had been diluted with flush oil. Preliminary, hastily constructed, electrically heated replacement circulating systems were only marginally successful. Success was not achieved until a carefully designed system consisting of dual, high temperature centrifugal pumps adequately powered, and Dowtherm jacketed piping of large enough size to minimize pressure drop where installed. New Dowtherm jacketed piping was installed between the bottom of the vacuum flash drum and the Sandvik belt which allowed the SRC to flow by gravity.

A cone and a wear plate of refractory were installed in the vacuum flash drum during July, 1974 in an attempt to streamline the elliptical bottom head and provide erosion resistance. During October, it was discovered that spalling refractory was plugging the bottom piping. The refractory was removed and steel wear plates were installed.

The original piping scheme incorporated a 2-1/2 inch Fisher EC straight-through letdown valve located in the line between the vacuum flash preheater and the vacuum flash drum. It was located about 40 feet upstream of the drum. Severe erosion was experienced in the valve body and the downstream piping elbows. The valve was replaced with a 3" Fisher EAC angle valve installed on the inlet nozzle of the drum. A liner was inserted in the discharge side of the valve. The liner extended to the center of the drum where the flow was directed downward through a hard faced 6" elbow and impinged on a 12" hard faced pipe cap. The liner, elbow and cap all were worn through within 15 days. During this time the liquid stream contained high concentrations of mineral residue due to operational and mechanical problems with the filters. Several other modifications were tried but with limited success. The problem was ultimately solved by:

- Installing parallel angle valves.

- Installing new short 8" nozzles in the drum wall and utilizing a specially designed 8" to 3" adapter flange for mounting the angle valves. The 8" nozzle was jacketed with 10" pipe and a vacuum gage installed to monitor the pressure in the jacket. If the 8" nozzle wears through, it can be detected by a vacuum developing in the jacket. However, operation can continue until a subsequent plant shutdown for other reasons.

- Installing a special tungsten carbide valve seat with an integral extension nozzle in each pressure control valve.

The original design of the vacuum flash heater coil provided support for alternate coil turns with "U" clamps to a vertical post. After several months of operation, the unsupported coils relaxed to the point that they rested on the supported coil below. Thus, part of each unsupported coil was shielded from the radiant heat. After consulting Foster Wheeler, the manufacturer, the coils were respaced and those coils which were originally unsupported were set on 3 inch by 4 inch by 1-1/2 inch 316SS blocks. The blocks were welded to the support posts and the coils were supported by the blocks.

During early operation of the plant, a vacuum bottoms pump discharge line ruptured when liquid trapped between a downstream plug and a closed block valve was overheated. The heat tracing circuit on this section of line consisted of four heater cables in series with a design watt density of 35 watts/foot. Investigation after the rupture disclosed that two of the cables provided 98 watts/foot due to an installation error. The line ruptured when the trapped liquid was heated to a temperature of approximately 1000°F by the high watt density heater cable. Properly installed heater cable would not have subjected the trapped liquid to more than 550°F and the piping design strength would have been adequate. P&M's electrical department recalculated all 606 circuits of heat tracing installed and found 10 to be questionably high in wattage. Necessary corrections were made. This problem is discussed to point out the extreme care which must be exercised with the design, construction and check-out of systems utilizing electrical heating. Where possible, such systems should be replaced in future plants with jacketed assemblies utilizing a high temperature liquid as a heat source.

b. Distillation System

Severe corrosion has been experienced in the light ends and wash solvent columns and associated equipment. Replacement of carbon steel tubes with stainless steel was required on reboilers and condensers and the original carbon steel wash solvent column shell was replaced with stainless steel. A detailed description of these problems is included in the Corrosion-Erosion section of this report.

The solvent recovery reboilers are kettle type heat exchangers with Dowtherm on the tube side. The reboilers' process temperatures are maintained around 600°F. Sludge buildup on the process side, accompanied by a lack of heat transfer, has required mechanical cleaning (hydroblasting with high pressure water) on five occasions. The wash solvent reboiler is the worse offender. A reboiler of a different design with the process in the tubes would function better.

Many tubes in the vacuum flash drum overheads condenser have been plugged from carryover of SRC. The demister pad was removed from the drum during early operation because of plugging. Flooding of the wash solvent column has occurred due to the plugging of downcomers by carbonaceous material which also is believed to be SRC which was entrained in the vacuum flash drum overheads.

The light ends feed exchanger is a fixed tube sheet bundle with Dowtherm flow through the tubes. Process liquids enter at the bottom of the exchanger at 150°F and exit from the top of the exchanger partially vaporized at 500°F. This exchanger was oversized for normal operations. Plugging and coking quickly reduced its duty, requiring chemical cleaning during May, 1975. (Mechanical cleaning was impossible due to its construction). Heat transfer capacity was not recovered and a second chemical cleaning was attempted during November, 1975. Little increase in heat transfer duty was noted. The process temperature differential had deteriorated to about 150°F, when a tube leak occurred in December, 1975. The exchanger has been blocked in and bypassed since that time, resulting in the light ends column being heat limited. Where possible, coal derived solvents subject to coking should be on the tube side of future heat exchangers.

Pressure on both solvent recovery towers has varied widely since startup. The main reasons have been:

- Inability to separate water from the feed.

- Common overhead vapor line.
- Inconsistent feed.
- Control valves fighting each other.

The common overhead vapor lines are being separated, separate pressure control valves are being installed on each tower and the pressure makeup/pressure relief system is being modified. In addition, a new oil-water separator is being installed. These modifications should improve tower operation.

Shortly after original plant startup, additional piping was installed to allow the solvent recovery system to be placed on total recycle or a flywheel mode of operation. This results in much time saving when upsets occur in other parts of the plant. The towers could be placed on "Flywheel" at operating conditions and then activated in a short period of time.

The seal flush system as originally installed consisted of an accumulator, supply pumps, feed lines, one large filter, double mechanical seals on pumps and return lines to the accumulator. Filter element replacements were required quite often resulting in solids being pumped to the mechanical seals. Seal failures became commonplace. To correct this bypassing problem, five-micron filters were installed upstream of each user. Seal life improved but the individual filters plugged. The seal flush medium was changed from process solvent to wash solvent, which contained less solids. Again, additional seal life was obtained, but excessive wash solvent leaked to the process contributing to coking of the vacuum flash preheater. The individual seal flush filters were removed, 0.050 inch restrictive orifices were installed, process solvent became the medium and single seals replaced the double seals. Seal replacement in the solvent recovery area is now infrequent, but the restrictive orifices tend to enlarge which exerts a heavier load on the fractionating columns and requires the seal flush accumulator to be filled often.

c. Oil-Water Separation

The original system has never performed satisfactorilly while using automatic instrument control loops. It depended on differential pressure interface level control for separation of the heavy oil, water and light oil. The small separators and similarity of specific gravities (typically .02-.04 difference between each phase) necessitated Dp cells with very low range spans and very high suppression. The resultant sensitivity, coupled with changing compositions and system pressure fluctuations, resulted in unreliable operation.

With the exception of a minimal amount of heavy oil which is manually blown down to the solvent recovery area, there is no phase separation in the two separators. Flow passes through the separators to the recycle process water tank (RPWT) for phase separation.

The original system was modified in the following manner:

- A small pump with an elevated suction was added to remove the light oil phase in the RPWT. Initial control of interface level was again by a DP cell with an expanded range. It was again a failure, however. A conductivity cell was later incorporated to provide for interface control, but it was also unreliable. Control was established through the use of a level glass and manual operation of the oil flow control valve.
- The separators were heat traced and insulated to aid in separation. Also, when possible, naphtha from the solvent recovery area is recycled to the RPWT to decrease the gravity of the light oil phase.

The system was modified further during the shutdown to convert the plant to SRC-II operation. Three phase separation in the RPWT was provided. Capacitance probes will be utilized for interface level control.

Primary health/environmental protection considerations with the system relate to the rich concentrations of hydrogen sulfide in the streams. The water phase typically contains 1-2% sulfur, while concentrations of H_2S in the air at the discharge of sampling taps normally is in excess of 100 ppm. When designing new facilities, attention should be given to pump seal and packing selection, disposal of leakages and blowdowns, adequate ventilation and provision for respirators/fresh air masks for personnel use while sampling.

G. Product Solidification - Area 08.1

After the initial operation, several revisions were required. Fumes generated by the hot product made it impossible for operators to work near the cooling belt. A shroud covering the hot end of the belt was installed. Steam eductors withdrew the fumes and discharged them to a stack which was installed alongside the prilling tower. A tar-like deposit soon coated the prilling tower and adjacent equipment. A third generation fume cure, an Aircology Centi-Clean centrifugal separator, has been installed and is being evaluated. Initial results are promising, but filter plugging problems must be corrected.

No provisions were made in the original design to collect dust generated at the discharge end of the cooling belt. A bag house and a collection system were installed and the dust problem has been reduced but not eliminated.

The goffering assembly at the end of the Sandvik belt, which was designed to cut the product into regular shaped squares, never functioned properly. The unit was composed of a roller-shaped knife and a paddle wheel-shaped knife section. When the unit was set at a location where the product was soft enough for the paddle wheel knife to function, the roller knife plugged with product. When the assembly was set at a location where the roller knife functioned properly, the paddle wheel knife would catch and bind on the stiff material. A finger crusher operated by the goffering assembly power train was installed at the belt discharge and effectively prevented conveyor and bucket elevator plugging due to oversized product. When low ash SRC production became consistent, the finger crusher was no longer needed due to the friable nature of the product.

A deflector was installed over the waterside (backside) of the cooling belt to prevent SRC spills from falling onto the water side of the belt and forming a thermal resistant film.

A spring loaded brass scraper blade was mounted on the cooling belt discharge. The blade helps "peel off" product that tends to stick to the belt.

At times, the cooling conveyor does not totally solidify the SRC. Extra cooling is obtained by blowing air onto the top of the product stream. This has a disadvantage of blowing product into the cooling water return trough leading to pump suction and results in plugging of the pump suction and contamination of the cooling tower. The returning particle laden cooling water plugs the spray nozzles underneath the cooling belt reducing the heat removal capacity. A filter was installed on the inlet cooling water, but the elements were soon removed due to rapid plugging. Raw water was substituted for cooling water, thus ending the vicious circle.

A backward sloping "V" shaped, heated SRC feed distributor was installed just above the cooling belt. This results in a wider band of SRC and a reduced product thickness. This installation helped circumvent the SRC's poor thermal conductivity and increased the capacity of the belt.

The vacuum flash drum level control valve and associated line through which the molten SRC flows on its way to the Sandvik belt, originally plugged frequently with solidified SRC. Electrical heat tracing was replaced with a Dowtherm system which keeps the product in the liquid state. Plugs that form are easily removed by pumping flush solvent into the vacuum flash drum bottoms line and onto the belt. The belt is cleaned by a steam wand before operation resumes.

The edges of the cooling belt have been damaged on several occasions due to the failure of the centering device to keep the belt tracking properly. Idlers have been installed to hold the belt edges level with the middle of the actuating and limit switch rollers. This keeps the belt from dropping below the limit switch when upset conditions expand the belt. Periodic adjustments by the instrument department are required to keep the system functioning properly.

An alternate solution to the problem would eliminate the centering device by installing a "V" shaped guide attached to the belt underside. The guide would run in sheaves attached to the feed and discharge end pulleys. This system would mechanically hold the belt in place.

The original cooling belt was joined by a specialist from the manufacturer using a heliarc welding procedure. Heliarc fastening of a replacement belt was attempted by local personnel, but cracks on both sides of the weld occurred after limited service. Since that time, rivets have been used to connect the belt. The rivets pull out occasionally but are easily replaced.

In early operations, SRC ash specifications were seldom met due to filtration problems. When "on spec" product was being collected, ash breakthrough frequently occurred. Day bins were installed to prevent contamination of accumulated product in the storage silos. When a day bin was full, laboratory analyses were obtained before routing SRC to the product bins. This system produced heavy dusting and resulted in plugging diverters, malfunctioning conveyors and spillage of product. The day bins were removed when the dependability of the filters improved. The frequency of filtrate analyses was increased to guard against the production of high ash SRC.

In an effort to reduce dusty conditions of the product SRC, two alternate flow schemes have been tried. First, a "taffy" product was produced by allowing semi-solidified product from the cooling belt to collect and agglomerate. The dusty environment during production was about eliminated (more fumes were present), but the completely cooled material had essentially the same dusting properties as the flakes.

Casting was attempted next. An instrumented casting mold, five feet diameter and four feet high, was filled with liquid SRC at 580°F. Seven days were required before the center material cooled below 300°F. Thermal conductivity of SRC will be addressed further in the Process Development section.

The trial casting produced a less friable material and enlarged casting facilities were erected. The new facility consisted of two carbon steel pits with dimensions of 12 ft. by 12 ft. by 14 inches. The pits were filled from the vacuum bottoms circulation system and had a semi-effective fume removal apparatus. Problems with line plugging, Dowtherm leaks and valve binding resulted. The castings required about 36 hours to cool before mining could begin with a front end loader. The casting material tended to adhere to the metal surfaces which complicated removal. Dust was produced during the mining operation, but was considerably less than that produced with the flaked product. Subsequent handling considerably reduced the slab size and generated more fine particles and dust.

The prilling tower has not been placed in service. Appreciable quantities of hot hydrocarbon fumes and liquid product carry-over and solid SRC buildup on the tower walls are expected should the unit be operated.

H. Storage and Shipping - Area 08.2

A very serious dust problem developed when the first SRC production was routed to the storage building. It required the installation, on a crash basis, of a dust collection and bag house system. Later, when loading of rail cars for shipment began, another serious dust problem was encountered. A semi-effective water spray system was installed temporarily while a more effective system could be obtained. A permanent system, supplied by Johnson-March and utilizing Johnson-March Compound MR wetting agent in water, was installed and substantially alleviated the dust problem. However, improvement still would be desirable.

The original slide valves beneath the storage bins malfunctioned often. The problem was solved by replacing the worm gear mechanism with a direct threaded driver. A catwalk was installed to allow access to these valves.

The soft rubber diverter plows on the product conveyor above each storage bin required replacement after limited service. Metal plows were tried but rapid belt wear occurred. A soft rubber plow with a metal backing plate has operated successfully. Product spillage at the discharge point continues to be a problem. A tripper belt assembly would appear to be a superior system.

Two large masses of SRC, each of about 200 tons, have fused in the storage bins. The elevated temperature of the product entering the bins is believed to be the predominant factor affecting this agglomeration. Pressure and heat absorption by the green storage bins are thought to be a contributing cause.

Substantial loss of product was experienced enroute when the initial cars were shipped to Georgia as part of the 3,000 ton requirement for a large scale combustion test. When the second lot of cars were loaded, several methods were used to investigate their effect on dusting during loading and subsequent unloading and loss in transit. These included the Johnson-March spray, a water only spray, crusting agents and the use of a chip net (sawdust retention net). The tests indicated that the most desirable method was the use of the Johnson-March MR wetting agent during loading followed by crusting the top surface of the car prior to shipment with a latex crusting agent, Dowell M166. This method was followed when loading and preparing cars for subsequent shipments.

I. Waste Water Treatment - Area 09.1

1. General

The waste water treatment system is shown schematically by Figure 14. It was designed to process 150 gallons per minute (gpm). Treating rates during the winter months average 100 gpm, due to the nearly constant rainfall, and 50 gpm during summer months.

Waste treatment has been presented the challenge of not only treating process water and storm sewer water, but slugs of MEA, DEA, ammonia, sodium hydroxide, sulfuric acid, Stretford solution, the full range of aromatic coal solvents and pulverized coal.

The State of Washington has proposed the following discharge limits based on a flow rate of 150 gpm:

pH range	6.0 - 8.5
BOD	40 mg/l
COD	150 mg/l
Total suspended solids	50 mg/l
Phenol	0.5 mg/l
Extractable Oil	10 mg/l

The treating units have done an outstanding job in removing potential contaminates with the only exception being periods when the biological unit is experiencing an infrequent upset. Typical waste water treatment effluent analysis ranges are as follows:

	Surge Reservoir	Clarifier Effluent	Flottazur Flotation Unit	Bio-Unit Effluent	Plant Effluent
pH	5.0-9.0	6.2-6.8	6.2-6.8	6.2-7.4	6.2-7.4
BOD, mg/l	-	-	135-350	10-110	4-23
COD, mg/l	1000-9600	650-5000	500-4000	20-250	5-75
TSS, mg/l	90-400	50-300	30-200	20-300	0-20
Phenol, mg/l	30-1500	25-1100	10-1000	0.1-1.0	0.0-0.4
Extractable Oil, mg/l	10-250	6-150	4-30	0-4	0-3

WASTE WATER TREATMENT SYSTEM

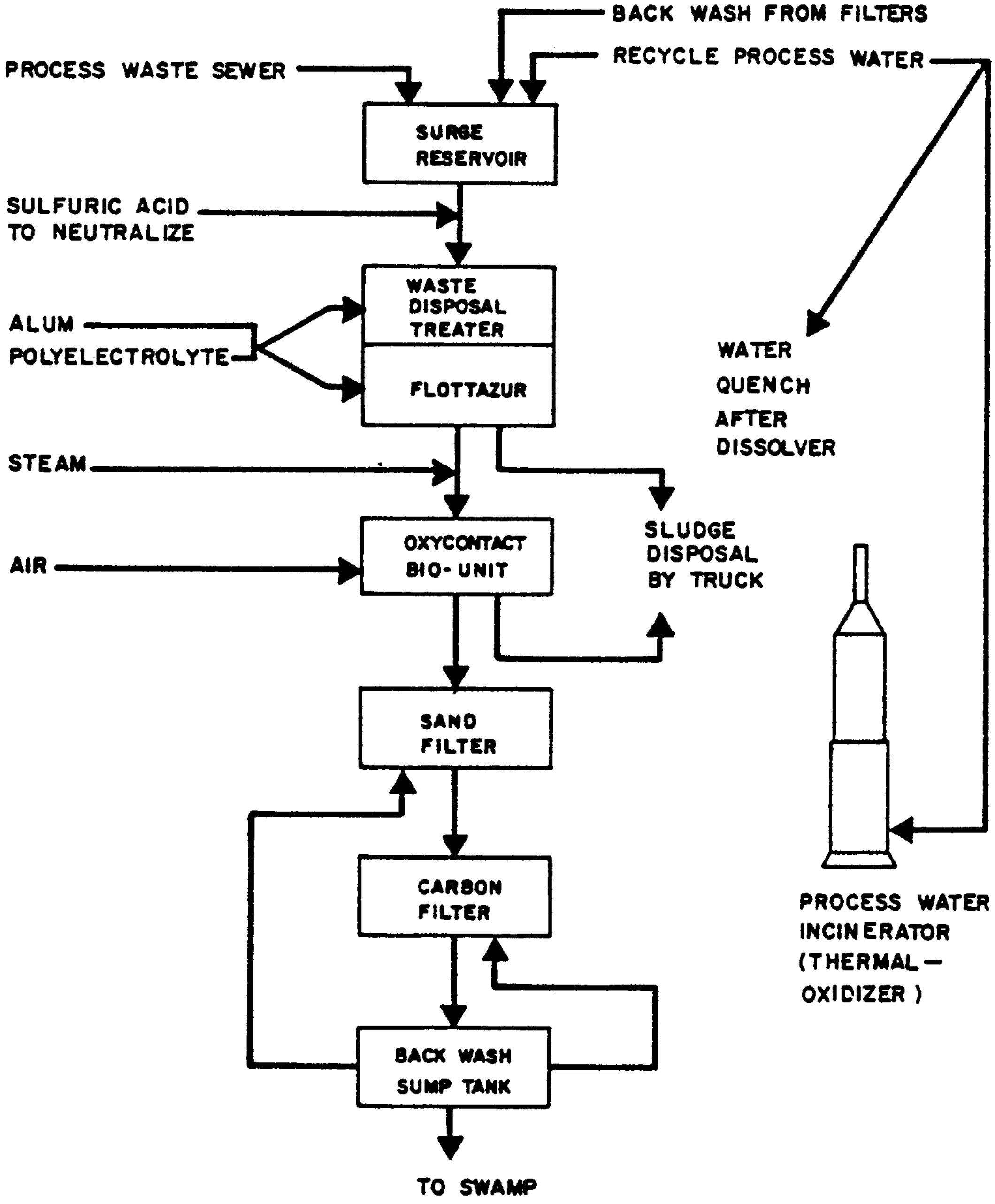


FIGURE 14

As can be seen by comparing proposed limits to actual discharge, the treating variables in the effluent are only 25% to 50% of the proposed limits.

Each of the processing units have experienced process and mechanical problems.

2. Clarifier

This unit uses alum, polyelectrolyte and circulation of settled solids to precipitate contaminates. Slugs of oil and pH swings tend to upset the unit. The draft tube, the variable speed drive and the level control device have required replacement or modification. The chemical feed pump problems have been partially corrected by sparing with the flotation unit pumps. The solids in the clarifier are pumped to a waste tank for outside disposal.

3. Flotation Unit

In this unit small air bubbles effervesce from a pressurized circulating water stream, attach themselves to oil and suspended particles and lift them to the top of the vessel for removal by skimming. The duty on this unit is increased when the clarifier is upset. The level switches and the level control valves on the pressure retention tank have required significant maintenance. The grid cell on the surface of the unit has been removed, resulting in better scum removal.

4. Biological Unit

The feed to the biological unit originally was by gravity flow. Accumulation of particles inside the transfer line resulted in flow limitations and direct discharge of untreated water to the plant effluent. The installation of a pump overcame this problem. Chemical nutrient injection systems (ammonia and phosphoric acid) have required considerable repairs.

The "bio-mass" for the biological unit was grown from a commercially prepared vacuum dried bacterial preparation. This method greatly reduced the time normally required for a unit to equilibrate. The unit's bacteria have been killed on several occasions. (The suspected culprit is sulfur containing Stretford solution.) The symptoms of a "sick" unit are a chronic tendency to low pH and a brownish-yellow effluent. The low pH results from sulfur-oxidizing bacteria converting sulfur containing compounds into acids (a pH of 2 has been observed). The colored effluent is due to anaerobic decomposition of the bacteria. Addition of soda ash to maintain a pH above 5 during an upset allows the bulk of the bacteria to survive. Each time the unit is "shocked", additional vacuum dried bacteria are added. Periodically an antifoam agent is required to prevent excessive foam production.

The biological unit's extended aeration section air sparger has failed. Deterioration of a rubber sleeve joint is the suspected cause. Replacement lines have been installed until repairs can be completed.

The casings on the biological unit discharge pumps have corroded. The cause is the low pH generated by the bacteria. Addition of soda ash to control the pH appears to have corrected the problem.

5. Sand and Charcoal Filters

The filters have experienced few processing or maintenance problems. Frequent backwashing is required to remove dead bacteria when the biological unit is experiencing an upset. Filter media in the sand filters have been replaced once. "A" charcoal filter media has been replaced twice. Due to a loose retaining screen, "B" charcoal filter media has been replaced three times.

6. Thermal Oxidizer

The John Zink "Thermal Oxidizer" installation was a misapplication of this equipment. The unit was not designed to handle solids or significant amounts of hydrocarbons. When startup operations began, this became very evident. Spray nozzles in the quench cell and the venturi scrubber constantly plugged and the high temperature limit switch tripped the unit each time even small amounts of hydrocarbons were injected. Mechanical deficiencies have required excessive maintenance and caused the unit to be down more than half of the time. The need to operate this unit was eliminated when the biological unit, with its excess capacity, was installed.

The major deficiency of the waste water treatment facility is the lack of an oil-water separator. The composite specific gravity of coal derived solvents is greater than 1.0 (oil phase heavier than water). A substantial oil volume accumulates in the surge reservoir, and consistent feed cannot be routed to the treating units because agitation leads to the formation of a stable emulsion.

The pH meters require continuous cleaning. Operator confidence in these instruments has been reduced to the point that a check by the laboratory is needed before an indicated change is believed.

A substantial amount of solids and sludge collects in the treating units. Disposal has presented a problem. An on site "land farm" operation would be desirable if space were available.

J. Tank Farm - Area 09.2

The tank farm has operated as expected, but some processing problems have developed. The transfer line from the unloading pump plugged with raw purchased solvent. Heat tracing and steam addition would not free the line. Several ells were removed from the line to allow hydroblasting. The ells were replaced with crosses to allow easy access in the event the line plugged again.

One of the raw solvent tanks was partially collapsed when naphthalene plugged the pressure-vacuum valve. As the tank cooled, a vacuum was created which sucked in the sides. Filling the tank with water restored the tank to near original condition. Steam tracing of the pressure-vacuum valves has prevented the recurrence of this problem.

To allow maintenance work to proceed, material from the filtration area has been pumped at times to the heavy oil tanks. A heavy, tarry material has accumulated in these tanks which is extremely difficult to remove.

Three liquid phases can develop in the flare knockout drum which is located in the main process area (water phase sandwiched between a heavy hydrocarbon and a light hydrocarbon phase). A batch of this material was pumped to one of the heavy oil tanks which was being maintained at an elevated temperature. The steam which was generated resulted in splitting the tank at the roof seam. Oversized relief valves are being installed to prevent another similar occurrence.

The flow totalizers (Neptune - Type SS and ITT Barton-Model No. 382) have never operated properly. Excessive maintenance has been required, the gaskets have blown and the totalized volume is usually in error.

None of the tanks have adequate drains. Compounding this deficiency, the naphtha tank requires daily draining for water removal. The ground around the naphtha tank was quickly saturated with oil. The contaminated gravel has been replaced on several occasions to reduce the fire hazard. A study is being conducted to determine the best method to make the tank farm area impervious.

K. Hydrogen Plant - Area 09.5

After correcting many problems during commissioning, the hydrogen plant performed well during most of the subsequent operation of the SRC pilot plant. Only infrequent repairs were required and its process performance was adequate.

During the first five days of operation, the methane content of the reformer increased from 1% to 8%. Sulfur leakage through the desulfurizers with resultant poisoning of the reformer catalyst was the suspected cause. A new charge of activated charcoal was installed in the desulfurizers. Upon startup of the reformer, the process gas flow was held at 20% of design and the temperature was increased to 1600°F to drive sulfur from the catalyst. When the effluent gas analysis indicated that desulfurization was complete, process gas flow and temperature was returned to normal. Methane leakage was normal.

An electrical heated steam superheater was installed to heat the steam used for regeneration of the desulfurizers so the bed temperatures could be increased to 350°F. Formerly the maximum temperature which could be attained was 250°F, which resulted in inadequate regeneration and subsequent sulfur breakthrough.

In order to eliminate a fresh hydrogen compressor capacity limitation, the original non-coded desulfurizers were replaced with coded vessels which allowed the suction pressure of the compressor to be increased. This modification increased the compressor capacity and allowed the hydrogen plant to operate at 100% of design rate.

The reformer outlet piping and some associated with the shift converters required replacement due to thinning.

Although the normal mode of operation has been to produce hydrogen of 98 to 99% purity, the plant has been successfully tested in the synthesis gas mode, producing a gas having an approximate composition of 50% hydrogen and 50% carbon monoxide.

Several shutdowns were required to repair or replace the flexible spool pieces at the outlet of the reformer tubes.

During the shutdown in early 1977 for completion of SRC-II modifications, new catalyst was installed in the reformer, high temperature shift converter and low temperature shift converter. New activated charcoal was charged to the desulfurizers.

L. Boiler Plant

Both boilers have performed dependably throughout the entire operation of the SRC Pilot Plant. Only routine maintenance has been required. Periodic inspections by the Washington State boiler inspector revealed no significant defects, corrosion or deposits.

M. Desulfurization Units

The DEA sulfur removal system has operated routinely with only two significant problems:

- Foaming occurred early in its operation, probably due to residual oil which was not completely removed during the commissioning cleaning. It occurred several times during subsequent operations due to carryover oil from the high pressure separator. Replacement of the charcoal in the filters usually corrects the foaming problem.

- Vibration in the discharge piping of the high pressure charge pumps has been severe at times requiring repair of the pulsation dampeners. Because of this vibration, the piping has been X-rayed to ensure that no cracking has resulted.

Numerous and frequent problems have been encountered with the Stretford sulfur removal system. Many modifications were required before attaining in late 1976 what appears to be satisfactory operation. Details of modifications will not be discussed since this is a proprietary process.

N. Hydrogen Compression

No major problems have occurred with either the fresh or the recycle hydrogen compressor. Short shutdowns are periodically required for valve changes but this is normal for reciprocating compressors. Both machines were overhauled during 1976 as a preventive maintenance step.

A large recycle hydrogen compressor was installed during the shutdown for SRC-II modifications in early 1977. This additional capacity will be required for SRC-II operation. The extra recycle gas will be available due to the installation of a naphtha scrubbing system during this same period. This system will increase the purity of the recycle and purge gas and thus decrease the quantity of hydrogen purged.

O. Cooling Water System

Substantial corrosion and fouling have been experienced in the cooling water system. Several contributing factors are:

- Lack of adequate attention to day-to-day control due to concentration on the many problems encountered in the coal conversion equipment and process. When it was realized that a problem existed, much attention then was diverted toward solving it and preventing recurrence.

- An inadequate or improper control program recommendation by the water consultants. Several separate chemical addition programs were tried before one which appears to be adequate was found. This difficulty may be due, at least partly, to the fact that the area in which the SRC Pilot Plant is located is not heavily industrialized and, consequently, experience with water treating systems may be limited.

- Periodic leakage of coal solutions and oil into the system.

It is believed that the program now in use will be effective.

Automatic chemical feed and pH control systems were installed.

Due to the buildup of sludge through the system, it was thoroughly acid cleaned, neutralized and prefilmed during the general plant shutdown in early 1977.

P. Dowtherm System

In general, the operation of the Dowtherm System was routine, requiring only normal maintenance. Much of the routine maintenance consisted of correcting leaks of valves and fittings. A leak tight Dowtherm system is very difficult to attain.

The major problem associated with the Dowtherm system has been the failure of tubes in several Dowtherm heated exchangers due to corrosion and/or erosion on the coal solution side. Large quantities of Dowtherm loss into the process occurs rapidly with such a failure. A level recorder with a low level alarm was installed on the Dowtherm surge tank to give a warning of Dowtherm loss to the process.

Q. Pumps

The pumping of abrasive coal slurries at elevated temperatures is an area that needs considerable additional testing and equipment development, especially in those applications involving unfiltered coal solutions. A pump with long life and reliability handling abrasive slurry is needed.

Most pump mechanical problems at the Pilot Plant have been alleviated by training and educating operators and pump maintenance personnel on mechanical seal fundamentals and installation techniques, improving maintenance procedures, converting to single mechanical seals, controlling the seal environment, sparing of critical pumps and setting up an effective but expensive local pump rebuild program.

1. Pump Performance

A brief description is given below of the Pilot Plant experience with pumps in the following types of service: High pressure coal slurry charge, coal slurry recirculation, filtered coal solution and unfiltered coal solution.

a. High Pressure Coal Slurry Preheater Charge Pumps

These are Wilson-Snyder horizontal triplex plunger pumps, Model #63-10R, each designed to handle 35 gpm at pressures up to 3100 psig. The capacity of the pumps can be varied from 1/3 to full by means of variable speed motors. Mechanical problems experienced have been short check valve life due to erosion and poor packing and plunger life due to inadequate packing flush.

These pumps originally were installed with hemispherical suction and discharge valves. After several sets failed, each lasting approximately three days, elastomer soft-seated valves with special seats were installed. Upon startup, they failed after eight hours. At the advise of I.G.T., high chromium balls (200 chrome hardened to Rockwell C61-63, 2.750 ± .001" diameter) were installed. After two years, the valves on "A" charge pump exhibit .005" wear on the ball diameter and 1/16" gain on the break of the Stellite seat. "B" charge pump was rebuilt using the hardened chrome balls and tungsten carbide seats. There has been .003" wear on the ball diameter and no measurable gain on the break of the tungsten carbide seat. Normally "A" charge pump is used as the primary pump with "B" as the spare or backup pump. Therefore, a direct comparison between the amount of wear and materials cannot be made as the hours of operation for each pump has been substantially different.

Preheater charge pump packing and plunger life is dependent upon whether or not packing flush is used. Generally, packing fails due to recycle standby operation when packing seal flush to the pumps is blocked in. During continuous plant operations, packing failure is not a problem. Normal procedure is to replace the charge pump plungers when the packing is replaced. The worn plungers are rebuilt with nickel-chrome-boron and ground and polished to a 10 micro finish.

b. Coal Slurry Recirculation Pumps

These are centrifugal Morris Pumps, Model 1-1/2 JC-14 rated for 120 gpm at 150 TDH, operating at 1800 rpm. The casing and suction disc liner are made of flintmetal (Ni-Hard): ASTM A532, Type I, Grade 1 and the impeller is made of high chromium iron: ASTM A532, Type III, Grade 2.

Currently, both coal slurry recirculation pumps are equipped with Chesterton 880 single seals with a tungsten carbide rotary face against a ceramic stationary face. Operation is much improved compared to the original packing and mechanical sealing arrangements. Of the initial Chesterton 880 seals installed, one functioned eight months and the other seven months, compared to the hours and days of operation experienced with packing and other seals.

Initial modification on the first pump utilized a Durametallic single seal and plans were to test a Chesterton seal in the second for a comparison test. However, it became necessary to limit the process solvent which was used to flush the seal since the plant was being operated in the slurry recycle mode and efforts were being made to minimize the addition of distilled solvent to the system. This flush limitation, coupled with stuffing box cooling, resulted in premature failure of the Durametallic seal due to seal torque resulting from solidification in the seal. The Durametallic seal, which performed well when properly flushed, was replaced with a readily available Chesterton 770 seal. Steam was piped to the previously water cooled stuffing box to keep the flush solvent fluid. The Chesterton 770 seals were later replaced with the current type 880 seals which have a clog-free/self-cleaning design to minimize the area where particles in the fluid can hang up or clog the seal.

c. Filtered Coal Solution Pumps

Abnormal difficulties have been experienced with only one type of filtered coal solution service - the vacuum flash drum bottoms recirculation pumps. These are Goulds pumps, Model 3735, API 610, size 1 X 2 - 11A. Design flow is 25 gpm at 480 TDH, driven by a 50-hp motor at 3600 rpm. Material of construction is 11-13% Chrome-Iron alloy (350 BHN) A 351 GR CA-6NM.

The pumps recirculate liquid SRC at 600-700°F. (SRC solidifies around 350°F). Initially, the SRC contained ash because of the necessity to bypass the filters and some erosion was noticed, but as filtration became more reliable this no longer was a problem.

The main problem associated with these pumps has been mechanical seal failures.

The pumps were originally fitted with John Crane type 15 WT metal bellows single seals. High product dilution with low boiling range cold seal flush (wash solvent 380°-480°F) boiling range caused cavitation and motor overloading due to higher viscosity created by cooling of the liquid SRC being pumped. To eliminate product dilution, John Crane 9-9B double seals were installed but failed within a few days. As a result, the type 15 WT single seals again were installed and changes were made to the seal flush system. Wash solvent was replaced with hot process solvent (480°-900°F boiling range) which was preheated to 400°F to lessen liquid SRC cooling due to dilution and also cooling water flow to the stuffing boxes was discontinued. The modifications were successful, with the pump having only one seal failure in the ensuing four months.

d. Unfiltered Coal Solution Pumps

(1) Stripper Bottoms Pumps - Area 02

These are Dean Brothers Pumps, Model R434, AVS, 1 X 2 X 11-1/2, with a design flow rate of 60 gpm at

420 TDH operating at 3600 rpm. They pump 600°F coal slurry containing 10-12 weight per cent solids. The slurry has a specific gravity of 1.1 and a viscosity of 1240 SSU at pumping temperature. They have cast steel casings and cast iron impellers.

These pumps take suction from the bottom of the stripper column, recycling slurry to the 01 area for fresh coal slurrying. The first pump operated only 14 days before a hole developed in the casing at a point just downstream from the centerline discharge. The casing wear ring had been completely eroded out and had broken into pieces. Some of the pieces had lodged in the impeller. This problem was solved by eliminating the need for a pump in this service by increasing the pressure in the stripper column and pressuring the slurry back to the 01 area without the use of a pump. The spare pump, which has not been utilized, has been kept ready for emergency use.

(2) Filter Feed Pumps

These are Goulds, Model 3735, API 610, size 1 X 1-1/2 - 8, design flow rate 60 gpm (normal 16 gpm) at 198 TDH operating at 3600 rpm, pumping unfiltered coal solution (UFCS): 600°F, S.G. of 0.98, viscosity at pumping temperature of 1.3 cp, containing 6 to 9 weight % solids. The material of construction of the pumps is 11-13% Chrome-Iron alloy (350BHN) A 351, GR CA-6NM.

The pumps take suction off the bottom of the filter feed flash vessel, charging unfiltered coal solution to each of the precoat rotary drum filters.

Mechanical problems associated with the filter feed pumps have been mechanical seal failures and severe to moderate erosion in the area around the stationary casing wear ring and moderate erosion to the impeller balance holes and periphery.

Originally, the wear rings were press fit into the casing and secured in place with set screws. The heads of the screws created turbulence and, in turn, severe erosion which cut the casing away around the back side of the wear ring to the suction side. This problem was reduced by building the wear ring into the casing and coating the casing and wear ring areas with a tungsten carbide flame spray. Average pump life in this service has been three to four months before rebuilding is required. Average seal life is approximately 2 months.

(3) Filter Feed Flash Recirculation Pumps

This is a Goulds, Model 3735, API 610, size 3 X 4-11, with a normal flow of 200 gpm at 62' TDH, operating at 1800 rpm, pumping unfiltered coal solution: 600°F, S. G. of 0.98, viscosity at pumping temperatures of 1.3 cp, containing 6-9 weight % solids. Material of construction is 11-13% Chrome-Iron alloy (350BNH) A 351 GR CA-6NM.

This pump takes suction off the bottom of the filter feed flash vessel, pumping unfiltered coal solution in a closed loop through the recirculation exchanger back into the flash vessel.

Mechanical problems associated with this pump have been a few seal failures and moderate to light erosion in the casing wear ring area.

This has been the most reliable UFCS service pump. One must note that it operates at 1800 rpm, develops a low head and operates at 66% efficiency. Average seal life has been five months. The pump has been rebuilt three times in 2-1/2 years service.

(4) Filter Feed Surge Vessel Pump

This is a Goulds pump, Model 3735, API 610, size 1 X 2-11A with a normal flow of 60 gpm at 309' TDH, operating at 3600 rpm, pumping UFCS: 600°F, S.G. of 0.935, viscosity at pumping temperature of 1.3 cp, containing 6 to 9 weight % solids including diatomaceous earth precoat. Material of construction is 11-13% Chrome-Iron alloy (350 BHN) A 351 GR CA-6NM.

This pump takes suction off the bottom of the filter feed surge vessel. It charges unfiltered coal solution into the filter feed flash recirculation loop and recirculates the balance of the flow through a Dowtherm double-pipe heat exchanger back into the surge vessel. This has been the most severe service as far as erosion is concerned. This, at least partially, is due to the high head and operation at low efficiency and to the presence of highly erosive diatomaceous earth.

Mechanical problems have been seal failures and severe erosion. Average pump casing and stuffing box life has been only 30 days. Severe erosion occurs in the casing wear ring area, the casing face, the stuffing box cover and in the pump tangential discharge opening.

Figures 15 and 16 are photographs of the internals of one of the Goulds Model 3735 pumps after 33 days in this service.

(5) Filter Feed Surge Vessel Spare Pump

This pump is a spare to the pump described in the preceding paragraph. A Goulds 3196 and a Durco Mark II, Group II pump have been used. Each is discussed separately below.

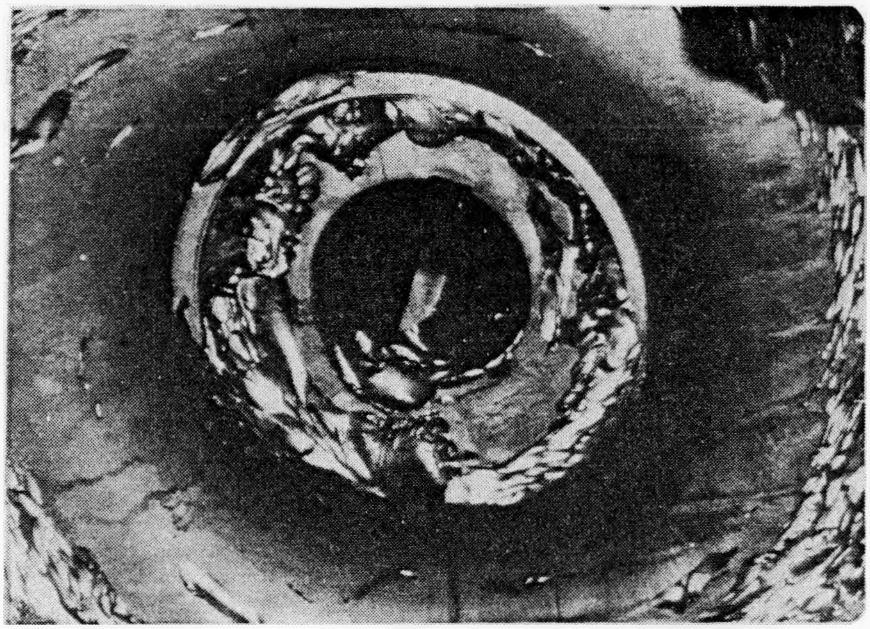
(a) Goulds, Model 3196 MT, AVS, 1 X 2-10, 60 gpm at 309' TDH, operating at 3600 rpm with 316 SS materials of construction. This pump has an open impeller with the wear surfaces being between the impeller and the casings. A hole eroded through the casing after approximately seven operating days. A new pump was installed only to be removed from service when the discharge flange began leaking. Inspection revealed severe internal casing and impeller erosion and that the centerline discharge had caused the flanges to wear because of the change in direction at that point. This pump was replaced with a Durco Mark II, Group II pump.

(b) Durco, Mark II, Group II, AVS, 2 X 1-10, 60 gpm at 316' TDH, operating at 3600 rpm with materials of construction being CD4MCU (BHN 260). Internals have been flame spray coated with tungsten carbide. This pump has a semi-closed impeller with the wear surfaces between the impeller and the flat stuffing box rear cover. After approximately ten operating days, a hole eroded through the stuffing box rear cover. Inspection revealed substantial wear on the impeller and casing. The pump was rebuilt using the same casing and a new impeller and stuffing box cover only to have a hole erode through the casing. A new pump was installed with a Stellite wear plate on the stuffing box cover. The pump operated 331 hours when a leak developed at the discharge flange. Figures 17 and 18 illustrate the absence of erosion on the Stellite wear plate but severe erosion on the unprotected casing and the semi-closed impeller.

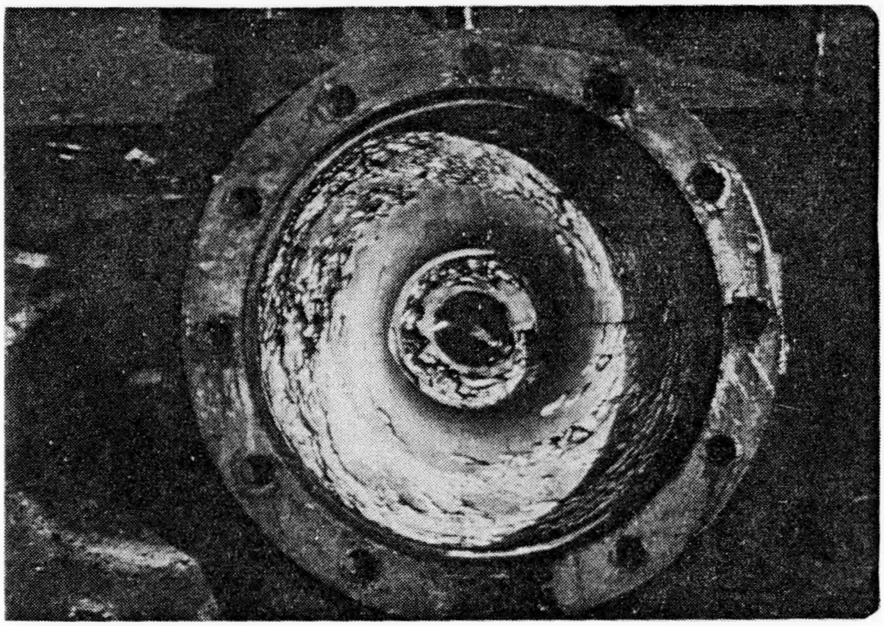
It is believed that, with improvements to hard surface the casing and impeller tips, the Durco pump life may be increased to equal the Goulds 3735 pumps in this service.

(6) Filter Precoat Pumps

These pumps circulate precoat slurry for the rotary drum filters. The slurry consists of process solvent with 5-10 weight % diatomaceous earth at 600°F, S.G.



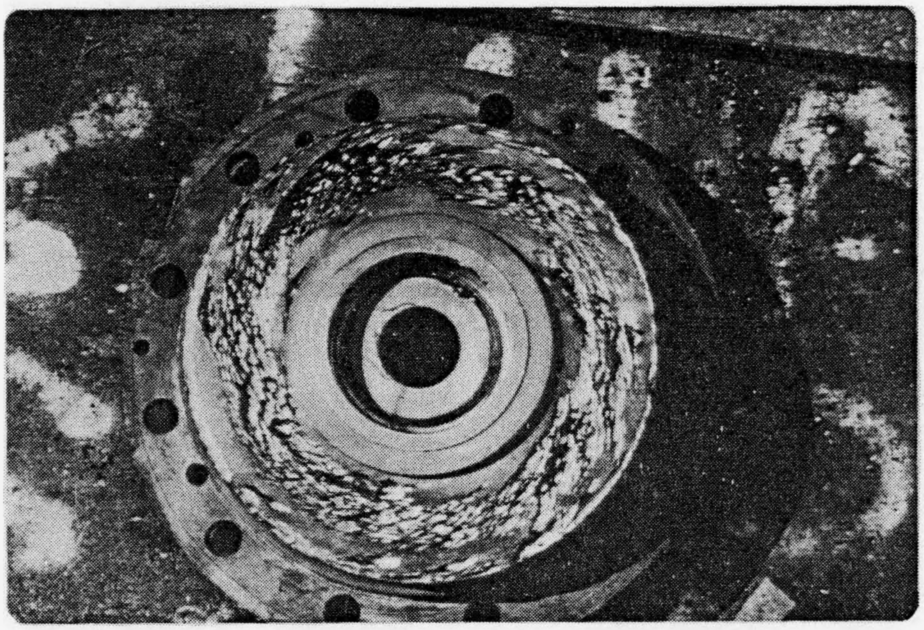
Casing Wear Ring



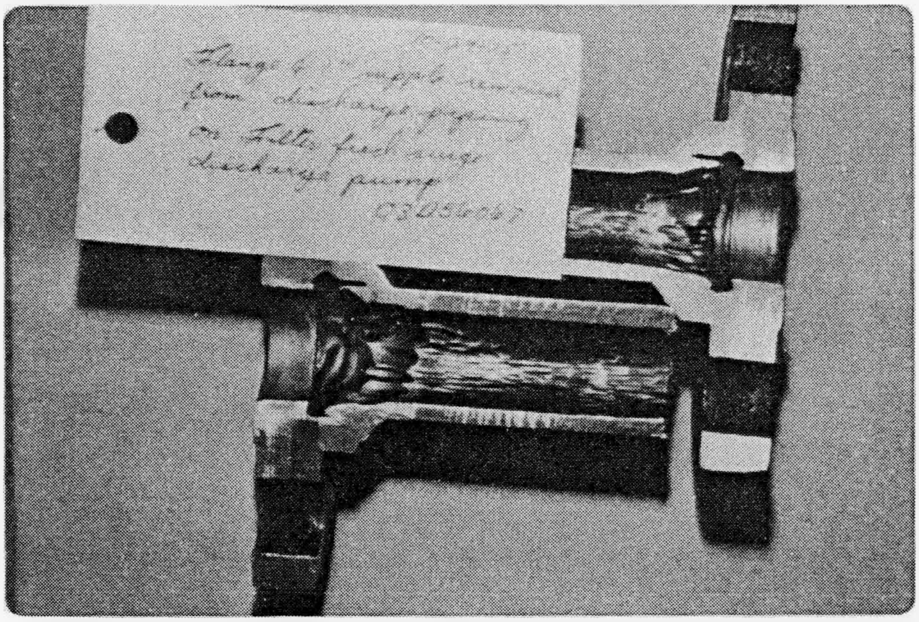
Casing Interior

Erosion of Internals of Filter Feed Surge Vessel Pump

Figure 15



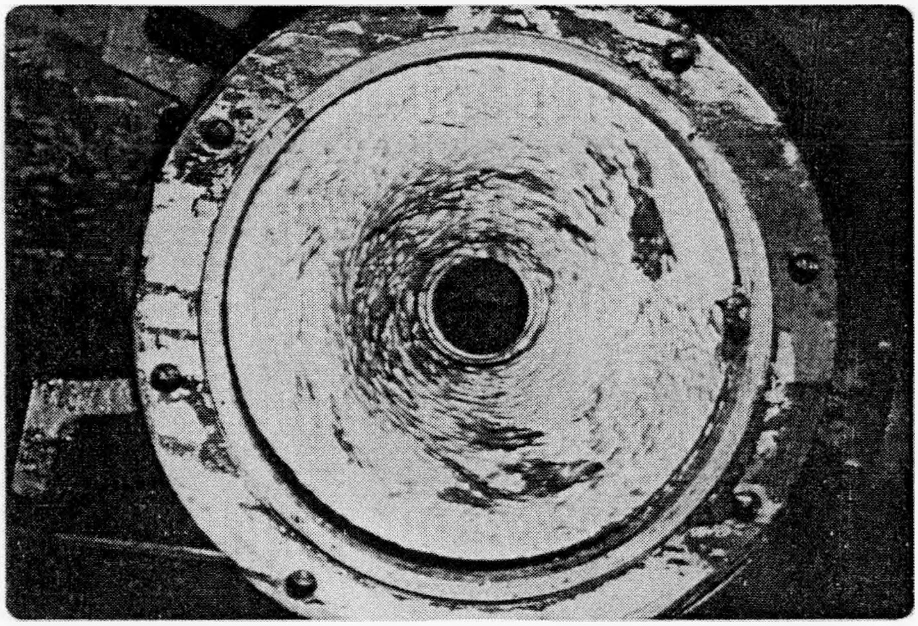
Rear Stuffing Box Cover



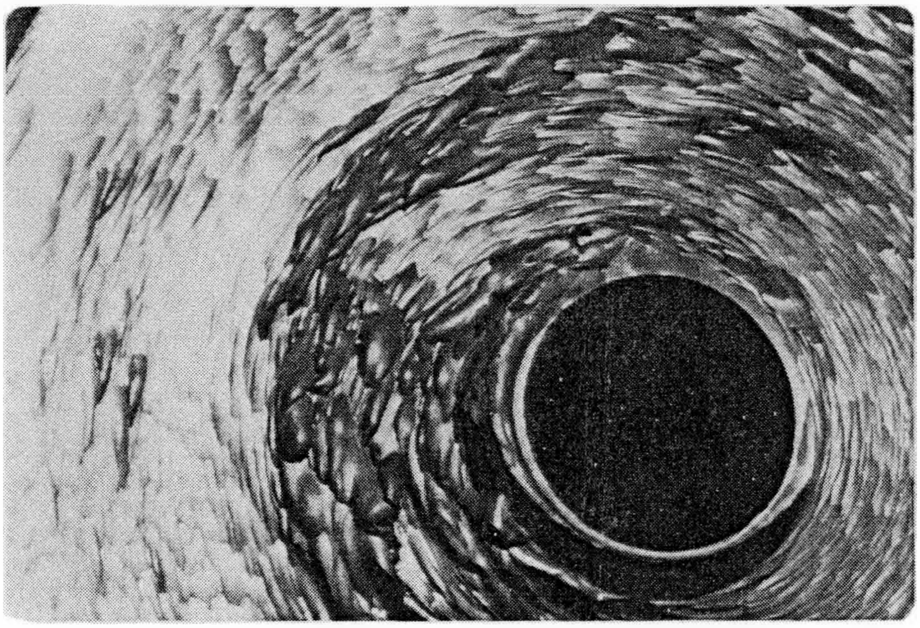
Discharge Piping Mating Flange

Erosion of Internals of Filter Feed Surge Vessel Pump

Figure 16



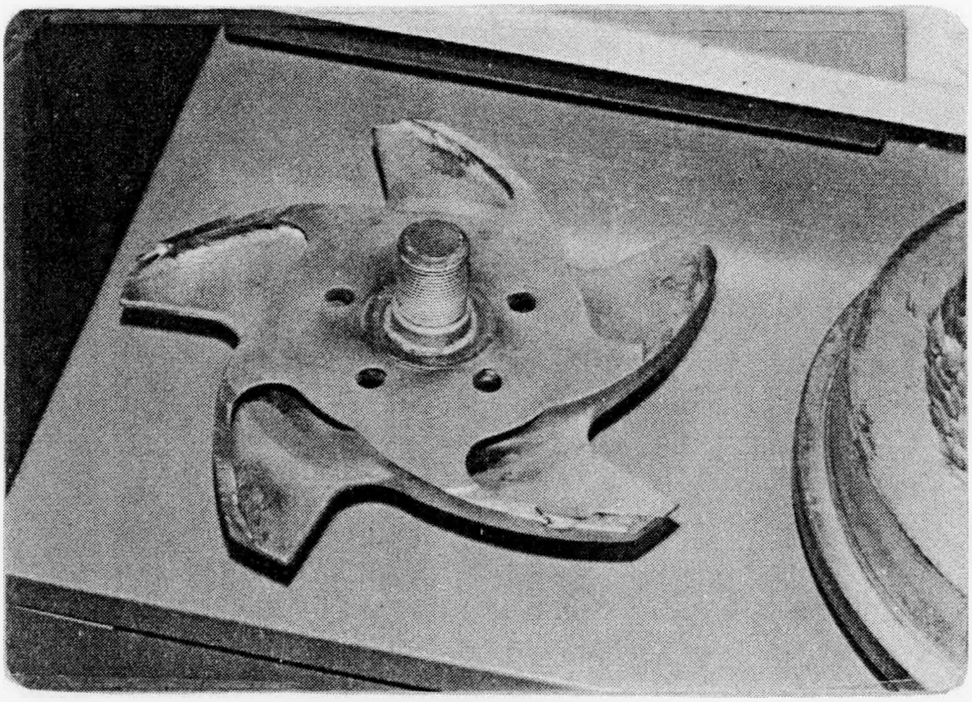
Casing



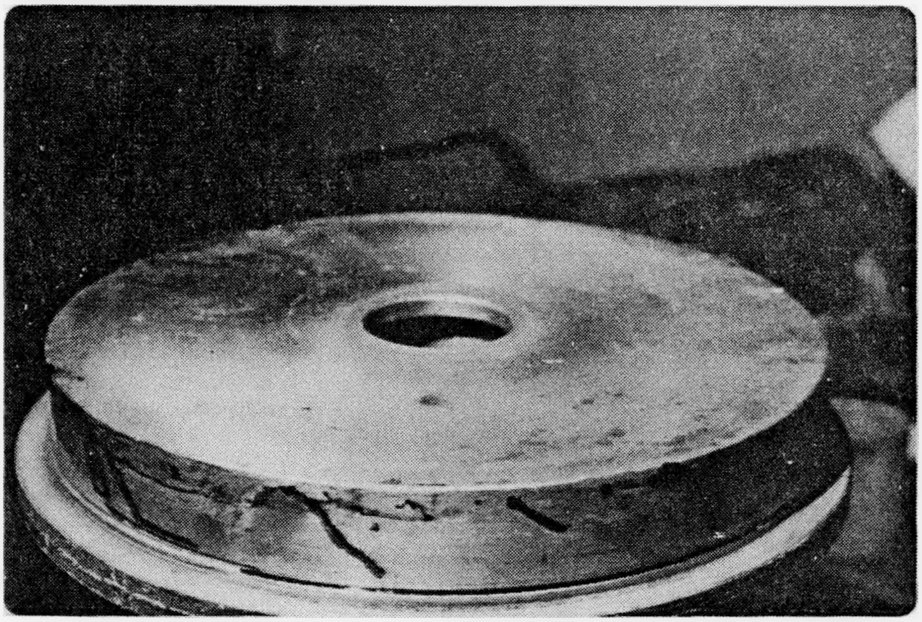
Casing Suction Area

Erosion of Internals of Filter Feed Surge Vessel Spare Pump

Figure 17



Semi-Closed Impeller



Stuffing Box Rear Cover with Stellite Wear Plate

Erosion of Internals of Filter Feed Surge Vessel Spare Pump

Figure 18

of 0.935, and a viscosity of 1.1 cp. Three types of pumps have been used. Each is discussed below.

(a) Sier-Bath pumps, rotary gear type, were installed as original equipment in this service, as well as several other services. All apparently were misapplications for this type of pump since it has been necessary to remove all of them from service and replace them with other types. Problems with the Sier-Bath pumps were rapid wear in the stuffing box area, excessive leakage and breakage of the brittle worm gears by the solids in the streams handled.

(b) A KSB Pump, Model CPK 300-Y size 3 X 2-6 replaced the Sier-Bath pumps in this service. It is rated for 150 gpm at 185' TDH, operating at 3600 rpm. It has a cast steel casing and a 316 SS impeller. The main problem associated with this pump has been casing, shaft and impeller damage due to the impeller nut coming loose on several occasions as a result of erosion of the nut and threads. This problem has been eliminated by welding the nut to the shaft. The pump has an enclosed impeller with wear rings on the casing side and repelling vanes on the backside. Operating at 3600 rpm and 60% efficiency, there has been only light casing and impeller erosion. However, operation with precoat is intermittent.

(c) A Durco Pump, Mark II, Group II, AVS, size 3 X 2-13, rated for 150 gpm at 177' TDH, operating at 1800 rpm and with materials of construction being CD4MCU (BHN 260) was installed as a spare in November, 1976 and has 482 hours of operation with no failures.

2. Pump Shaft Mechanical Seal Performance

Although the environment is severe in several parts of the SRC process, pump shaft sealing can be effectively accomplished with mechanical seals if proper seal selection, maintenance and operation is practiced.

Seals will leak when the seal either wears out or fails. A worn out seal is one whose carbon face has worn down to the metal. A failed seal is one that leaks for any reason other than worn out faces. Ninety percent of the seals removed from the pumps at the Pilot Plant leaked before the faces wore out and most failures were due to:

- Improper installation
- Excessive temperature

- Seal faces sticking open.

It is necessary to control or eliminate these main causes of failures.

Development of improved installation procedures through attendance at mechanical seal seminars, experience, and working closely with factory seal representatives has virtually eliminated seal failures due to improper installation.

Most seals have an elastomeric temperature sensitive member. Excessive temperature due to fouled stuffing box cooling water jackets, blocked in cooling water, or restricted pump flow have caused seal failures. Because of everchanging pumping conditions, most hot service (above 375°F) pumps now use John Crane 15WT all-metal bellows seals. This seal does not have a heat sensitive member.

Mechanical seals have moving parts that are designed to compensate for shaft movement to keep the sealing faces together. Dirt, abrasives and slurries tend to clog these sliding components, causing faces to separate and become scored by the particles. Likewise, fluids that set up and harden restrict movement of sliding components and cause faces to stick together. To give the mechanical seal as good a chance as possible of surviving, the seal environment in the pump stuffing box must be controlled. Replacing the few ounces of fluid in the stuffing box with a clean fluid will alleviate the problem of clogged and restricted moving seal components. In this way the seal faces will stay together and effect a non-leaking shaft seal.

Original equipment seals provided in the slurry pumps were John Crane 9-9B double seals. Dirty, abrasive slurry service is a misapplication of a double seal. Both Teflon and asbestos wedges were tried along with sealing face combinations of carbon vs. tungsten carbide and tungsten carbide vs. tungsten carbide with no appreciable gain in seal life. Many other changes have been made. However, improvement in pump mechanical seal life basically can be attributed to:

- Replacement of double seals with single seals
- Improved installation procedures
- Educating personnel on how a mechanical seal functions and the necessity for controlling its environment
- Use of a temperature independent seal.

There are combinations of three basic sealing problems associated with pumping solvent refined coal materials. The problems are listed below with the environmental controls used to handle them.

Problem	Environmental Control
• Dirty Fluid	Flush the stuffing box with clean process solvent by maintaining a positive flow of 3-7 gal/hr from the seal flush system through the stuffing box into the fluid being pumped. This positive flow keeps dirt and abrasives from getting into the seal area, clogging the seal and wearing of the seal faces.
• Fluid That Solidifies	Flush the stuffing box with clean process solvent (preheated for SRC) by maintaining a positive seal flush flow (3-7 GPH) during operation. Flush the pump cavity during shutdown and use a continuous steam quench on the gland to keep the seal faces clean and warm to prevent any foreign matter from solidifying.
• Fluids Greater Than 375°F	For clean liquids which do not solidify: Use cooling water in the stuffing box jacket with a dead-ended stuffing box and use a carbon throat restriction bushing to act as a thermal barrier between the fluid being pumped and the stuffing box to maintain 100-200°F. For a dirty product which solidifies: Maintain a positive seal flush flow (preheated for SRC) of 3-7 GPH in addition to the water cooled stuffing box, a carbon throat restriction bushing and a steam quench on the gland.

The key to seal survival is maintaining seal flush flow to the pump seal area.

In summary, pump shaft sealing of solvent refined coal process streams has been effected using single mechanical seals with an external, clean, process solvent seal flush and controlling stuffing box area temperature. Failure to control the seal environment will result in decreased seal life.

IV. CORROSION-EROSION TEST PROGRAM

The corrosion-erosion program is an open-ended plan of collecting as much data as feasible. Areas were initially selected for monitoring and, as experience indicated, the frequency and the complexity of procedures were modified. Initially about 190 locations were selected for ultrasonic thickness monitoring, several corrosion probes and several sets of metal corrosion samples were installed. All were later expanded in number. In addition, later use was made of dye-checking for cracks, special metallographic (microscopic) examinations, a few X-rays, sectioning, measuring, chemical and physical testing. The various tests used are briefly decribed in Table 3.

No corrosion inhibitors have been used in the process streams in the plant except in the DEA system for hydrogen recycle gas scrubbing. Various inhibitor systems have been used in the sulfur plant, steam, cooling water and other utilities but none are discussed here.

The corrosion-erosion program and results will be discussed by operating areas, except that control valves will be treated as a group. Pump corrosion-erosion is discussed in paragraph III.Q, entitled "Pumps".

A. Coal Preparation and Slurrying - Area 01

Coal receiving and preparation through the grinding operation is very similar to that experienced in many locations. Thus no detailed report of this area is included here. Some problems of erosion of chutes and rotary feeders was encountered. Moderate corrosion was encountered in the water system for handling and condensing water driven from the coal during heated pulverization and from conveyors above coal storage bins where coal occasionally smoldered.

Moderate erosion was encountered in the coal slurry circulating (centrifugal) pumps and severe erosion occurred in the original check valves on the high-pressure plunger (slurry preheater charge) pumps.

B. Slurry Preheating and Dissolving - Area 02

Most of the equipment and piping in this area is of high-alloy steel, thus more reason exists to monitor more locations for problems and to determine if higher or lower alloys are indicated. Voluminous data have been recorded. A summary of conclusions and typical data by function or location is given below.

1. Slurry Preheater Coil

The slurry preheater coil is 1690 feet of 3-inch Incoloy 800 tubing with an I.D. of 2.63 in. and an O.D. of 3.45 in. Gas

Table 3

Corrosion-Erosion Program Tests Utilized

Ultrasonic beam reflectance thickness gaging.

Direct-beam used:

Digital meter - Krautkramer Model DM-1B

Transducer DM-1-2-2-2-2065, 1/2 inch, 2.25 MHz for cold service with glycerin couplant. (For hot service used KMH4 transducer with DGM couplant.

With viewing screen contractor used:

KrautKramer USMZTM

Transducer long 1" diameter, 2.25 MHz.
Also long 1/2" diameter, KM8, 2.25 MHz

For shear wave testing used:

Transducer STR-70° 1/4" diameter, 2.25 MHz

Calibrated gain control check 200% for 3/16" flat

Reference level 100% of screen height

All cold tests, contractor used oil couplant.

Corrosimeter probe equipment:

Meter: Magna Model CK-2

Probes: Magna Model 2233 with Insert No. 22077-T4

Magna Model 3043 with Insert No. 30516 - 316 SS.

Corrosion samples were all 2" X 2" metal squares, all with a weld across one side of center, center mounted. In low-temperature (aqueous) service Teflon insulators were used. Otherwise all samples were held with stainless bolts and spacers.

Dye-checking procedure

Microscopic procedures

plus slurry pass upward through the coil which is about a 10 foot diameter helix. There are ells (about 18 inch radius) at the bottom and top of the helix. The pipes enter and leave the furnace shell vertically. These ells showed maximum erosion rates by ultrasonic thickness gaging of 30 to 90 mils in the first few months of intermittent operation. Since then operations and flows have become generally smoother. The outlet elbow has lost about 0.085± .020 inch wall thickness in a narrow area (perhaps a groove) in 19 additional months. No significant losses have been found in the large radius coils during that period. Typical velocity in the outlet of the coil is about 4 feet per second at design conditions: 13,000 lb/hr slurry feed rate, 30,000 SCFH gas recycle, 750°F and 1500 psig.

2. Dissolvers

Dissolver "A" was used during all operations while Dissolver "B" was used a very short time. An inspection of "A" was carried out September 5 and 6, 1976, about 19 months of operation since the first inspection. About 18 cubic feet of high-ash deposits were removed. Two nozzle liner-breather ring welds were found to be cracked. The bottom inlet nozzle had a small crack discernable with dye-checking. The center side outlet liner-breather ring weld had a wide crack extending about 90° (about 3 inches) around the periphery. Subsequent inspection in February, 1977 showed that the crack had enlarged to about 200°. It appeared that these cracked welds had poor penetration. No repairs were attempted. The weep holes indicated leaks in these nozzles for 1-1/2 to 2 years. Repeated direct and shear-wave ultrasonic inspections indicate no cracks or serious deterioration of the base metal (SA-336-F22) in these two nozzles. Direct wave reflectance from the end of the center nozzle indicates there may be 3/8 inch deep pits in the center of the area behind the cracked liner. The breather ring and sleeve of each nozzle is SA-240-T347. The dissolvers are otherwise lined with a 2-pass weld overlay. The first pass is 3/32" of 309L S.S. and the second pass is 3/32" of 347 S.S., but analyzes as follows (in 3 analyses):

Carbon, wt. %	0.076	0.047	0.069
Chromium	16.55	15.88	16.00
Nickel	8.29	7.50	7.94
Columbium + Tantalum	0.21	0.28	0.30
Manganese	1.74	1.88	1.76
Phosphorus	0.016	0.016	0.016
Silicon	0.46	0.47	0.47
Sulfur	0.019	0.012	n.d.

Dye-checking spots on the leaking breather rings and of segments of the top cover revealed no cracks, except as noted above. Metallographic examinations of the cover and the body weld overlay revealed no cracks in February 1977. However, a visual inspection revealed a localized attack of approximately 0.030 inches deep in several areas. The attack primarily was in areas where the weld pass stopped and started again.

3. High Pressure Separator

The high pressure separator is constructed of SA-387-D and is clad with 304 SS. Nozzle liners and breather rings are 304 SS. Normal operating temperature is 600°F. It was inspected September 4, 1976 and no cracks were found in the cladding using dye penetrant. However, during the February, 1977 inspection, dye penetrant revealed extensive cracking in the breather ring and the cladding. The metallographic inspection revealed, in addition to the cracking, large pits and carbon clumps in the dished section of the cladding. Possibly most of the cracking and pitting was present during the September inspection but was not detected due to insufficient cleaning.

4. Intermediate Pressure Separator

The intermediate pressure separator also operated at about 600°F. However, a September, 1976 inspection revealed extensive cracking in the 304 SS cladding. The inspection revealed large intergranular (probably polythionic acid induced) cracks and small transgranular (probably chloride induced) cracks in the clad plate area of the dished head. The top center outlet nozzle liner had a few intergranular and some small transgranular cracks. The microscopic examination revealed small grain size compared to that in the cladding on the dished head. A metallographic inspection of the vessel shell revealed no cracking. However, a dye penetrant inspection did detect a few cracks in the upper section of the shell. The bottom dish and breather ring were shear wave ultrasonic and dye checked. No cracks were revealed. Thus it appears that the vapor space environment of this vessel was more corrosive than the liquid phase or that the extra stresses and heat treatments of hot forming the clad plate into a dish sensitized the 304 SS. Ultrasonic thickness gaging clearly showed that the cracks had not penetrated into the base metal (SA-204-C to SA-264). The vessel was returned to service with more frequent reinspections and replacement of the cover planned. During shutdowns the sides of the two hot separators are kept warm by electric tracing but not the tops. Thus moisture reflux may accentuate polythionic acid corrosion of the covers.

5. Recycle Condensate Separator

The recycle condensate separator normally ran at 60° to 100°F with water plus light solvent condensates. The pH of the water usually is 8.0 to 8.5. The inspection of this vessel showed intergranular cracking partly through the 304 SS cladding of the top dish that was less severe than that in the high pressure and intermediate pressure separator covers. No transgranular cracking was found. The bottom dish showed no indication of cracks with dye checking.

6. Piping and Block Valves

The carbon steel piping upstream of the preheater has experienced little erosion or corrosion loss. This may be due to low velocity (3-4 ft/sec) slurry movement. A 0.90 inch deep groove was worn in the tapered wall of stainless steel rotameter (FT87) in this stream in 30 days of operation. The carbon steel venturi meter replacement is still serviceable after two years of service. Throat loss has been only 0.020 inches in that time.

The pipe used immediately upstream and downstream of the dissolvers is 4 inch schedule XX (double extra heavy) 347 SS tubing. Typical velocity in the tubing is about 3 feet per second. Early operations apparently caused wear at a few bends of as much as 0.090 inch loss in the worst case. The wear then stopped in all monitored locations. A February, 1977 inspection revealed partly plugged lines upstream and downstream of the dissolver. Where they could be measured, the openings were 20 to 25 percent of the tubing internal area. Thus the deposits were able to withstand at least two weeks of operation at 12 to 15 feet per second overall (3 phase) velocity. The deposits contained high ash (20-58 wt.%). No wear was detected in areas of pipe exposed to these narrowed channels.

Significant erosion occurred in the pipe and block valves downstream of LCV-166A and LCV-175A (the liquid let down valves after the high pressure and intermediate pressure separators). For example, the 1-inch Schedule 160 304-L stainless tubing immediately downstream of LCV-175A lost 0.050 to 0.129 inch from the internal walls in seven months. This severe erosion has necessitated several replacements of manifold block valves and piping. Both globe and gate valves with Stellite trim have been unsatisfactory. Special ball valves for high temperature, high pressure service recently have been installed. At this time, no data is available relative to their service life. Valve life still remains an unresolved problem.

7. Corrosion Rack Data

Table 4 summarizes the corrosion rack data collected in the slurry preheater and dissolver area utilizing unstressed 2 inch by 2 inch samples.

8. Control Valves

Of 129 control valves installed in the plant initially, trim changes or repairs were logged on 20 valves at least once. Of these repairs, some were not adequately documented or were not otherwise useful (such as accidental damage) for study. Table 5 summarizes the maintenance experience on 10 valves in seven locations. All but one of these are in slurry service. LCV-172, on recycle condensate separator liquid level control, apparently had primarily a corrosion problem of the tungsten carbide trim. Corrosion resistant binder in the carbide trim cured this problem.

LCV-37 on filter feed overflow was selected to represent several valves in similar service with similar wear rates. The first filter cake letdown valve on filter "A" consisted of two valves in series in cyclic operation as noted in the table. LCV-161A was a Fisher V-ball valve with a Mason-Neilan Camflex valve (LCV-161B) downstream. The high maintenance on these valves of 15 days trim life led to a trial of a single Willis valve, LCV-161. Life is not yet established on this valve or its trim but it has been in service over 190 days with no significant wear.

LCV-166B as shown in the table is the original Fisher globe valve used for high pressure separator slurry letdown. After the initial plant operations, this valve was replaced by the angle valve LCV-166A. LCV-166B has since been used intermittently for bypass service while LCV-166A is being serviced. The globe body type appears to be too short lived for continuous service in this location.

Likewise PCV-216 is the original globe valve in service on the vacuum flash drum inlet. Initial plant operations with unfiltered product resulted in high wear rates (4 days wear out time per body). It was replaced by PCV-216A, an angle valve in downflow configuration. As shown, this has worn very slowly during high ash operation.

The life of FCV-215, the flow control valve for the vacuum flash preheater, was increased from about three weeks to

Table 4

Slurry Preheater and Dissolver Area Corrosion Rack Data

Location	"A" Dissolver	Recycle Condensate Separator
Days installed	710	730
Days of operation on coal		
Slurry phase*	117	-
Gas Phase*	377	494
Operating Temp. Range, °F		
Slurry Phase	840 - 870	-
Gas Phase	360 - 660	60 - 100
Corrosion Rate, Mils/Yr.		
304 SS	-	0.0
Monel 400	-	0.1
90/10 Copper-Nickel	-	3.1
410 SS	-	0.1
321 SS	1.3	-
310 SS	0.5	-
Incoloy 800	2.0	-
304 SS Aluminized	0.3	-

* The samples in the dissolver were sometimes in the slurry phase and sometimes in the gas phase

TABLE 5

SUMMARY OF EROSION HISTORY
OF TYPICAL CONTROL VALVES IN SEVERE SERVICE

LOOP IDENTIFICATION	LCV-37	LCV-161	LCV-161A	LCV-166A	LCV-166B	LCV-172	LCV-175	FCV-215	PCV-216	PCV-216A
Duty	Filter A Feed Overflow	Filter A Cake Letdown	Filter A Cake Letdown	High Pressure Separator Level	(Bypass) (Service)	Recy. Cond. Sep. Btms	I.P. Flash Drum Level	Vac. Flash Preheater Feed	Vac. Flash Drum Inlet	Vac. Flash Drum Inlet
Fluid, % Solids	4-7	30-50	30-50	5-7	5-7	0	5-7	6-8	6-8	6-15
Type of Solids:	(Usually) 95-98% Min. Res. (Can be) 2-5% Filter Aid			Mineral Residue	Mineral Residue	None	Mineral Residue	Mineral Residue	Mineral Residue	Mineral Residue
Temperature °F	440-490	350-450	350-450	580-650	580-650	60-100	580-650	300-400	750-650	750-650
Δ P PSI	75	120	120	900-1000	900-1000	1450	420-520	40-80	140-175	140
P_d PSIA	65	20	20	500-600	500-600	65	90	175	2-5	2-5
Flow Rate, GPM	24	2	2	12-16	12-16	1-2 Lt.Oil;1 wtr	12-16	10-20	10-20	10-20
Valve Mfgr.	Fisher	Willis	Fisher**	Fisher	Fisher	Fisher	Fisher	Fisher	Fisher	Fisher
Catalog Number	657-EC	M-21IT	650-U	657-DBAQ	657-DBQ	657-DBQ	657-DBAQ	657-EC	657-EC	657-FAC
Body Size, in.	1	2	1	1	1	1	1	1	2 1/2	3
Body Style	Flanged Globe	Flanged Angle	Flanged Insert	Angle	Globe	Globe	Angle	Globe	Globe	Angle
Body Materials	Carbon Steel	Carbon Steel	Carbon Steel	Stainless	Stainless	Stainless	Stainless	Carbon Steel	Cr-Mo	Carbon Steel
Orifice Size, In.	1 5/16	(2) 1/2	1	1/2	1/2	1/4	1/2	3/4	2 7/8	2 1/4
Trim Type	Plug	Rotating Disc	Vee-Ball	Microform	Microform	Microform	Microform	3 Flute	Plug	Plug
Cage Material	Alloy 6	None	None	Stellite	Stellite	Stellite	Stellite	Stellite; W-C	Stellite	Stellite
Plug Material	316 SS with	W-C	Stainless	W-C Tip	W-C Tip	W-C Tip	W-C Tip	Stellite; W-C	Stainless	W-C Tip
Seat Material	Alloy 6 faces	W-C	Stainless	W-C Sleeve	W-C Insert	W-C Insert	W-C Sleeve	Stellite; W-C	Stellite Insert	W-C Sleeve
Trim Life, Days*	24 (Avg.)	190+ (no wear)	15	25 (Avg.)	3-10	24-290+	170	22/200+	10-12	90
Body Life, Days*	250	190+	200+	448+	50-60	500+	448+	500+	4	400+
Notes:			Cyclic Service	Downflow	Upflow	Cobalt Binder Corroded	Downflow			Downflow

W-C is an abbreviation for cemented tungsten carbide.

**Used in series with a Mason-Neilan Camflex Model 35-35200, Cv=0.5, Stainless steel ball and seat.

*Days life in approximate days in service with normal fluid.

more than 200 days (still not worn out) by replacing the Stellite trim with tungsten carbide. Tungsten carbide is required for high ash service even at low differential pressures.

Note that LCV-166A and PCV-216A show long body life (total life not determined) but trim life is still limited to about 25 and 90 days respectively. Larger valves used commercially could be expected to have a longer trim life with the same design. There is room for improvement in the valve designs, in the use of auxiliary restrictors to reduce valve erosion, or in improved trim materials. The key design modifications in LCV-166, 175 and PCV-216 have been:

- The use of angle valves.
- The use of downward flow along the axis of the plug through the orifice.
- The use of tungsten carbide tubes (chokes) beyond the orifice to conduct the high-speed stream beyond the valve body into an enlarged pipe as a deceleration chamber.

C. Filtration and Mineral Residue Drying - Area 03

Most of the corrosion/erosion problems in the filtration area have been with heat exchangers, pumps and the mineral residue dryer.

1. Pumps

The erosion/corrosion of the pumps is discussed in paragraph III.Q, entitled "Pumps".

2. Heat Exchangers

Initially it was thought that the failure of the original filter feed flash recirculation and precoat slurry shell and tube exchanger was the result of only erosion. However, in light of the corrosion patterns in the replacement double pipe exchangers and the Dowtherm jacketed piping in the fractionation area, the corrosion contribution seems to be very significant.

As shown in Figure 19, the corrosion rate is higher in the hotter straight sections of the countercurrent double-pipe exchanger than in the non-jacketed return elbows. If erosion were the only mechanism of metal loss, one would expect the metal loss to be the opposite of what was observed.

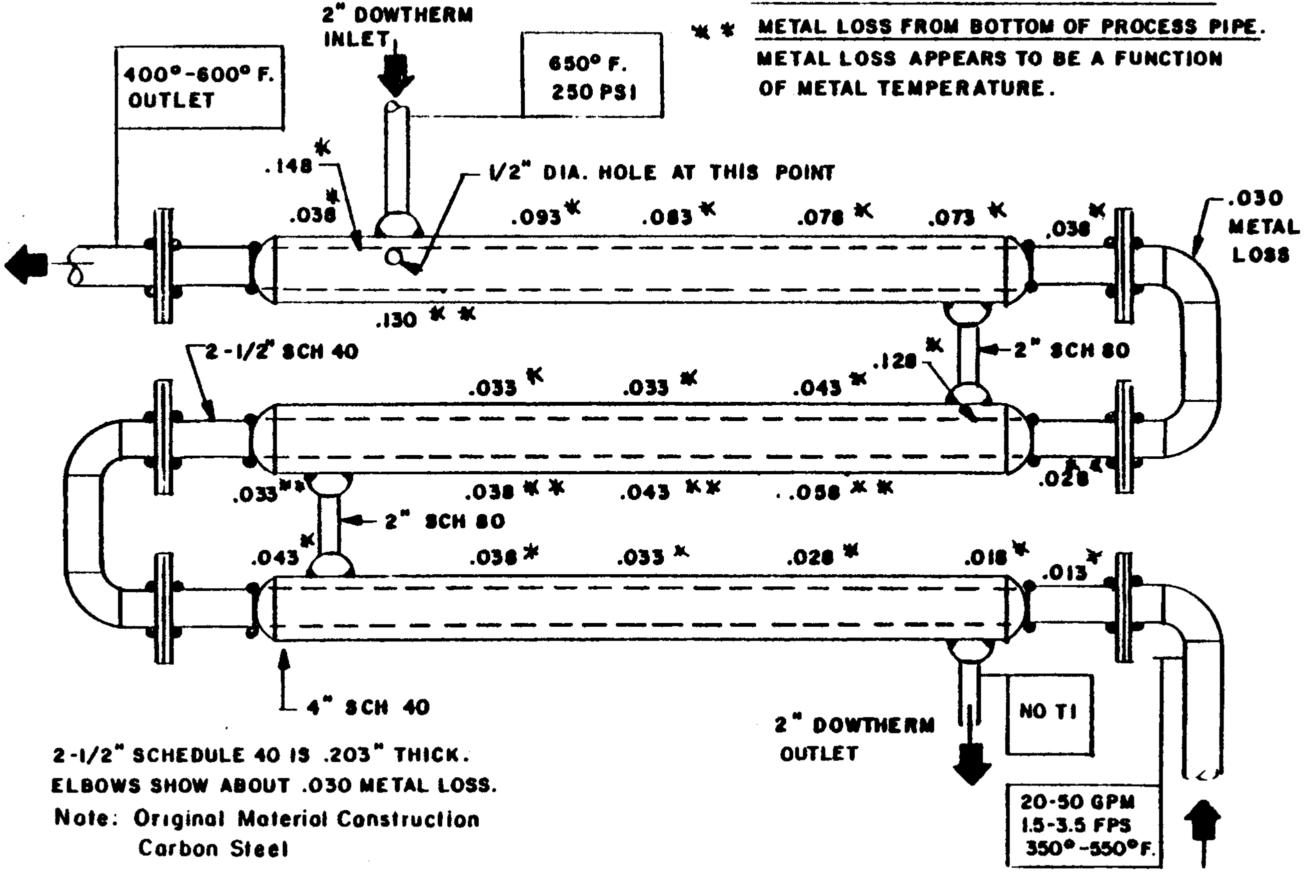
A corrosion pattern similar to the one shown in Figure 19 was also observed in the filter feed flash and precoat slurry recirculation double-pipe exchangers which replaced the original shell and tube exchangers. The effect of erosion is demonstrated by higher rates of metal loss downstream from the welds.

CORROSION - EROSION
OF
FILTER FEED SURGE VESSEL RECIRCULATION EXCHANGER

* METAL LOSS FROM TOP OF PROCESS PIPE

** METAL LOSS FROM BOTTOM OF PROCESS PIPE.

METAL LOSS APPEARS TO BE A FUNCTION OF METAL TEMPERATURE.



2-1/2" SCHEDULE 40 IS .203" THICK.

ELBOWS SHOW ABOUT .030 METAL LOSS.

Note: Original Material Construction
Carbon Steel

FIGURE 19

Usually the advantages of a stringer bead outweigh the disadvantages. However, in the case of erosive/corrosive slurry piping, a smooth pipe interior is desirable.

The erosion/corrosion problems with the heat exchangers in the filtration area have not been solved. Since it will be necessary to replace the present double-pipe exchangers in the near future, the installation of various materials will provide useful information.

3. Mineral Residue Dryer

In order for the mineral residue dryer to operate properly, the vapor velocity and the temperature in the vent line must be high. A detailed discussion of the mechanical problems with the vent line is discussed in paragraph III.E.3.c. Unfortunately the severe process condition causes erosion/corrosion of the vent line. Since the line is maintained at 700°F with electrical heat tracing, the corrosion may be significant. During the next failure of the vent line, a 316 SS elbow will be installed. The performance of the 316 SS will provide insight as to whether the problem should be solved with a large corrosion allowance on the carbon steel or with corrosion resistant materials.

4. Filter Feed Flash Vessel

There has been no significant corrosion in the vessel. A summary of corrosion rack data is shown in Table 6 . Corrosion rates as small as 10 mpy are hard to verify with field measurement techniques. Most measurements are made with ultrasonic equipment that has an accuracy of ± .005. As a result, longer time periods are necessary to detect low corrosion rates.

D. Solvent Recovery - Area 04

The corrosion/erosion problems have been quite severe in the solvent recovery area. Failure of the reboilers, air coolers, fractionation components, piping, and the tower walls have caused several major shutdowns. Since high corrosion rates have been observed during extended periods of solids free operation, corrosion rather than erosion is probably the main mechanism of metal loss. A common compound or group of compounds that would explain the corrosiveness has not been determined. However, the solvents do have the common characteristics of being noncorrosive to carbon steel at temperatures below 300°F and quite corrosive to carbon steel above 500°F. The corrosion experience with the main pieces of equipment is discussed separately below.

Table 6

Filter Feed Flash Vessel Corrosion
Rack Summary (Mils Per Year)

Rack No:	174
Date:	
Installed	02/07/75
Removed	02/04/77
Alloys:	
Carbon Steel	10.6
9 Cr-1Mo	.2
Type 410	.1
Type 304	0.0

1. Wash Solvent Column System

The wash solvent column (WSC) has been plagued with corrosion failures of the reboiler tubes, fractionation tray assemblies and of the column. Other auxiliary equipment such as the overhead vapor cooler and vapor line have also failed. In most cases the use of 316 SS has reduced the corrosion to an acceptable level. However, in some cases 316 SS may be marginal and the use of alloys such as Hastelloy G or Incoloy 825 may be necessary.

In the case of the wash solvent reboiler bundle, the original carbon steel tubes were found to have an exceptionally high corrosion rate. The original 12 BWG carbon steel tubes failed only after a few months of service. As a result of the smooth and corrosion products free tube surface, the severely corroded tube appeared to be in new condition. A summary of additional material experience with the wash solvent reboiler bundle is shown in Table 7 . The data were obtained by retubing the bundle with several different types of alloy tubes and placing the bundle in service. As one can see from examination of the table, the 316 SS was attacked with some light pitting while the 304 and 321 SS had a significant general attack in addition to pitting. There was no noticeable attack on the Hastelloy G or Incoloy 825.

The original carbon steel wash solvent column failed as a result of extensive corrosion. Inspection of the failed carbon steel wash solvent column revealed the following:

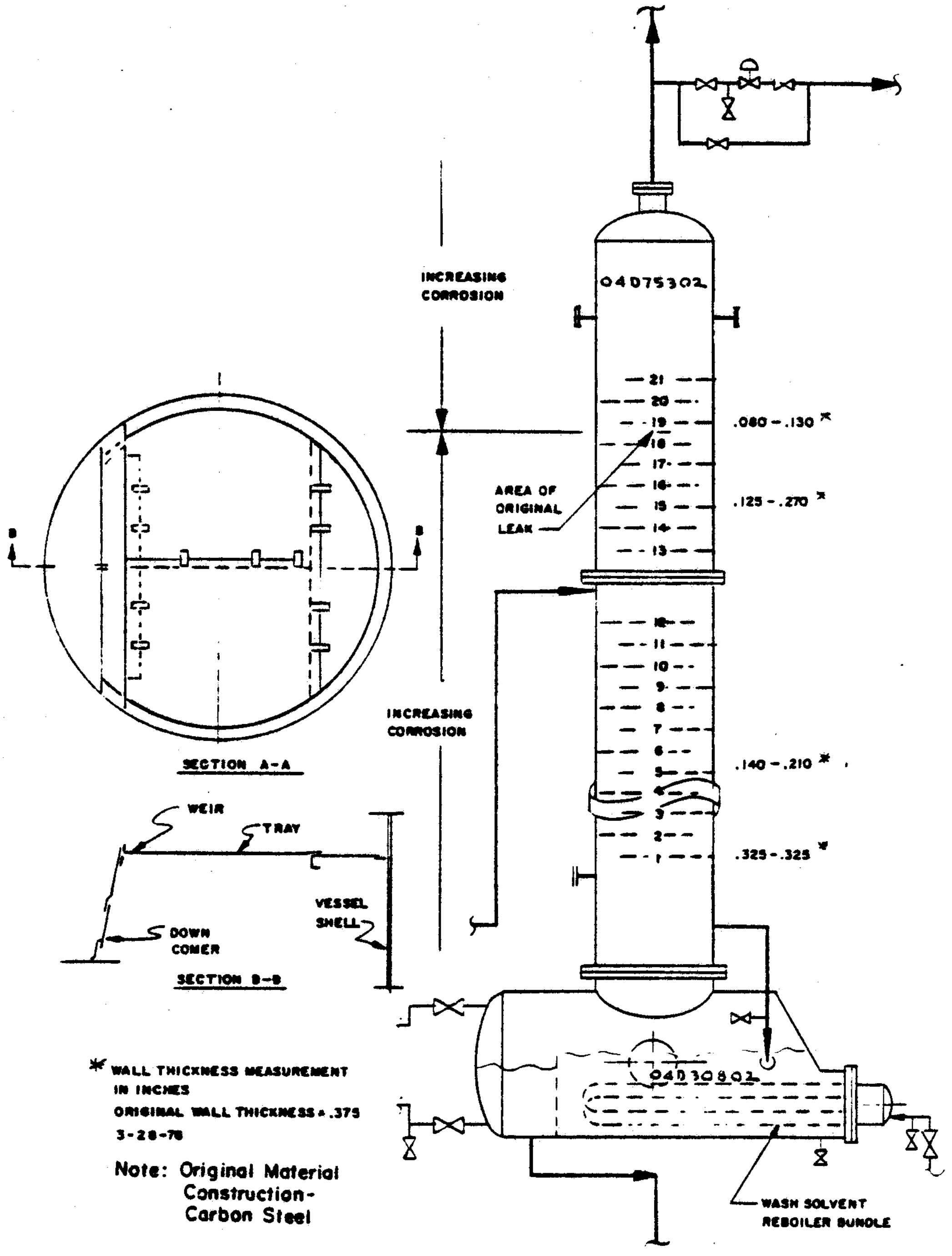
- Nearly all of the tray support rings and downcomer support rings and downcomer supports from tray 12 to 21 (see Figure 20) were nearly, if not completely, corroded.
- The corrosion increased in severity from tray 12 to 19.
- Areas of liquid phase contact were corroded significantly more than those in contact with the vapor phase. Where the liquid had a higher velocity and/or turbulence the carbon steel was severely attacked. For example, the carbon steel shell was cratered at the corner of the tray weir and the downcomer support bar.
- In the upper part of the column, above tray 21, where the corrosion was less severe, the vessel had grooves and rivulets down the shell of the column. For example, both a nozzle and thermowell had rivulets down the side of the column. Apparently, the vapor condensed on the intrusion and ran down the vessel well. In the case of the thermowell, the rivulets appeared as perfectly machined "V" grooves.

Table 7

Summary of Material Experience for the Wash Solvent Column Reboiler Tube Bundle

Material	Original O. D. 4-23-76 (inches)	O. D. 3-18-77 (inches)	Metal Loss (O. D.) (inches)
304	1.004	.984	.020
321	1.000	.990	.010
316	.950	.950*	.000*
Hastelloy G	.950	.950	.000
Incoloy 825	1.001	1.001	.000

*Some light pitting



* WALL THICKNESS MEASUREMENT IN INCHES
ORIGINAL WALL THICKNESS = .375
3-28-78

Note: Original Material Construction- Carbon Steel

CORROSION IN WASH SOLVENT COLUMN

FIGURE 20

- Sometimes the corroded surfaces were so smooth and clean that measurements were necessary to detect attack. Even in the less corroded areas, there appeared to be no corrosion products present.

- The 304 SS tray valves and the tray were thinning at the points of contact.

The carbon steel column was replaced with 316 L SS and there has been no measurable metal loss since. Even in the areas of the downcomers, where there was a high rate of corrosion of the carbon steel, there is no measurable metal loss.

A summary of the corrosion rack data for the WSC is shown in Table 8. In some cases the 316 SS is showing corrosion rates as high as 49.3 mpy, which is not consistent with experience. The difference in corrosion rates may be explained by variations in the different heat treatments of 316 SS. A high corrosion rate for 410 SS is shown which is consistent with the early failure of the 410 SS fractionation trays.

There have been three complete sets of fractionation trays in the WSC. The original set was 410 SS and the replacements have been 304 SS and 316 SS respectively. Even though there has been a significant improvement in material performance with each successive upgrading of material, the 316 SS still has some shortcomings.

The original 410 SS Nutter Engineering trays were almost completely corroded after approximately a year's operation. Of the 21 trays and downcomers, only a few showed any signs of ever existing. Since the original installation, separate sets of 304 SS and 316 SS trays have been installed.

Neither the 304 SS nor the 316 SS trays have performed satisfactorily. In the areas on the trays where there are moving parts, the metal loss is extremely high. For example, the clip ends of the 20-gage valves were completely corroded in two months of operation. Not only the valves wore, but the 14-gage tray mating surface wears at an equal rate. As shown in Figure 21, the corrosion rate is lower in areas beyond the valves, but the rate is still very high for only two months of operation. The 316 SS has performed superior to the 304 SS. However, the corrosion rate is unacceptably high. As shown in Table 8, the 316 SS usually has about half the corrosion rate of 304 SS.

In order to simulate actual operating conditions and to evaluate different materials, plans have been made to install trays and valves fabricated from 316 SS, Hastelloy C, Hastelloy G, Incoloy 825, and possibly Incoloy 800.

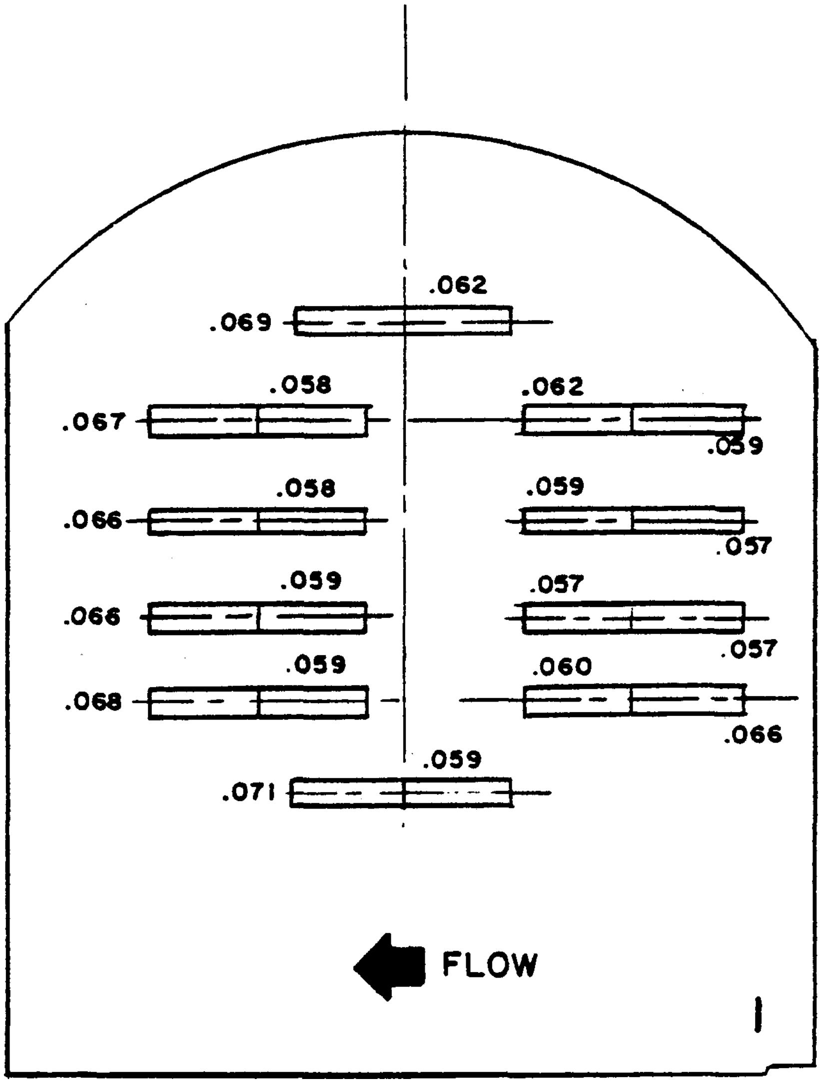
Table 8

Wash Solvent Column (WSC) Corrosion Rack Summary (Mils Per Year)

Rack No:	180	181	182	196	197	199	205	207
Location:	Top Manway	Mid Manway	Reboiler	Tray 21	Reboiler	Tray 12	Tray 21	Tray 12
Date:								
Installed	05/06/75	05/06/75	05/06/75	07/17/76	04/22/76	07/17/76	10/20/76	10/20/76
Removed	12/16/75	12/16/75	12/16/75	10/20/76	01/27/77	10/20/76	01/27/77	01/27/77
Alloys:								
Carbon Steel	61.5	74.8	47.5	663	24.7	443	183.0	*
5Cr-1/2Mo	61.2	90.6	127.3					
9Cr-1Mo	1.8	56.0	104.3					
Type 410	1.0	74.9	98.8	370	31.0	185	61.5	*
Type 304				75	.2	48		
Type 316				38	.1	7	1.8	49.3
Type 321	0.2	0.3	0.2	33	.1	28	7.3	36.7
Incoloy 800	0.1	0.3	0.2	14	0.0	4	1.8	6.1
26Cr-1Mo				142	0.0	83		
29Cr-4Mo				27	8.2	1	29.2	

*Coupons were completely corroded

CORROSION OF
WASH SOLVENT COLUMN TRAY



NOTE : MATERIAL
CONSTRUCTION
316 SS

ORIGINAL THICKNESS .083
INSTALLED 10-11-76
INSPECTED 3-7-77

FIGURE 21

2. Light Ends Column System

The corrosion rate in the light ends column has not been as severe as that in the wash solvent column. However, the reboiler bundle, tower internals, and the reboiler shell have failed.

After less than six months operating time, the carbon steel Norton tower internals were replaced with 316 SS due to extensive corrosion. The new 316 SS internals have shown no noticeable attack.

As in the case of the wash solvent reboiler tube bundle, the original carbon steel tubes failed. The light ends column reboiler bundle actually failed before the wash solvent tube bundle. The smooth, corrosion products free tube surface was the same as discussed for the wash solvent bundle. Experimental tubes were also installed in the bundle for evaluation and the results are shown in Table 9 . As one can see from examination of the table, all of the materials tested except 304 SS and 321 SS were satisfactory.

In the upper, cooler section of the column, the corrosion rate has been at an acceptable level (see Figure 22). However, the column has been operated most of the time without a feed exchanger due to plugging of that exchanger. Since the installation of a new light ends column feed exchanger probably will increase the temperature in the upper section of the column, the corrosion rate should also increase.

The lower section of the column and the reboiler shell have a considerably higher corrosion rate as shown in Figure 22. The reboiler shell actually had to be replaced with 316 L SS as a result of excessive metal loss. Since there is excessive thinning of the lower section of the column, the bottom section of the column probably will have to be replaced also.

As in the case of the wash solvent column, corrosion racks have been installed in the light ends column. A summary of the results is shown in Table 10. As one can see, the wash solvent column has a considerably higher corrosion rate than the light ends column.

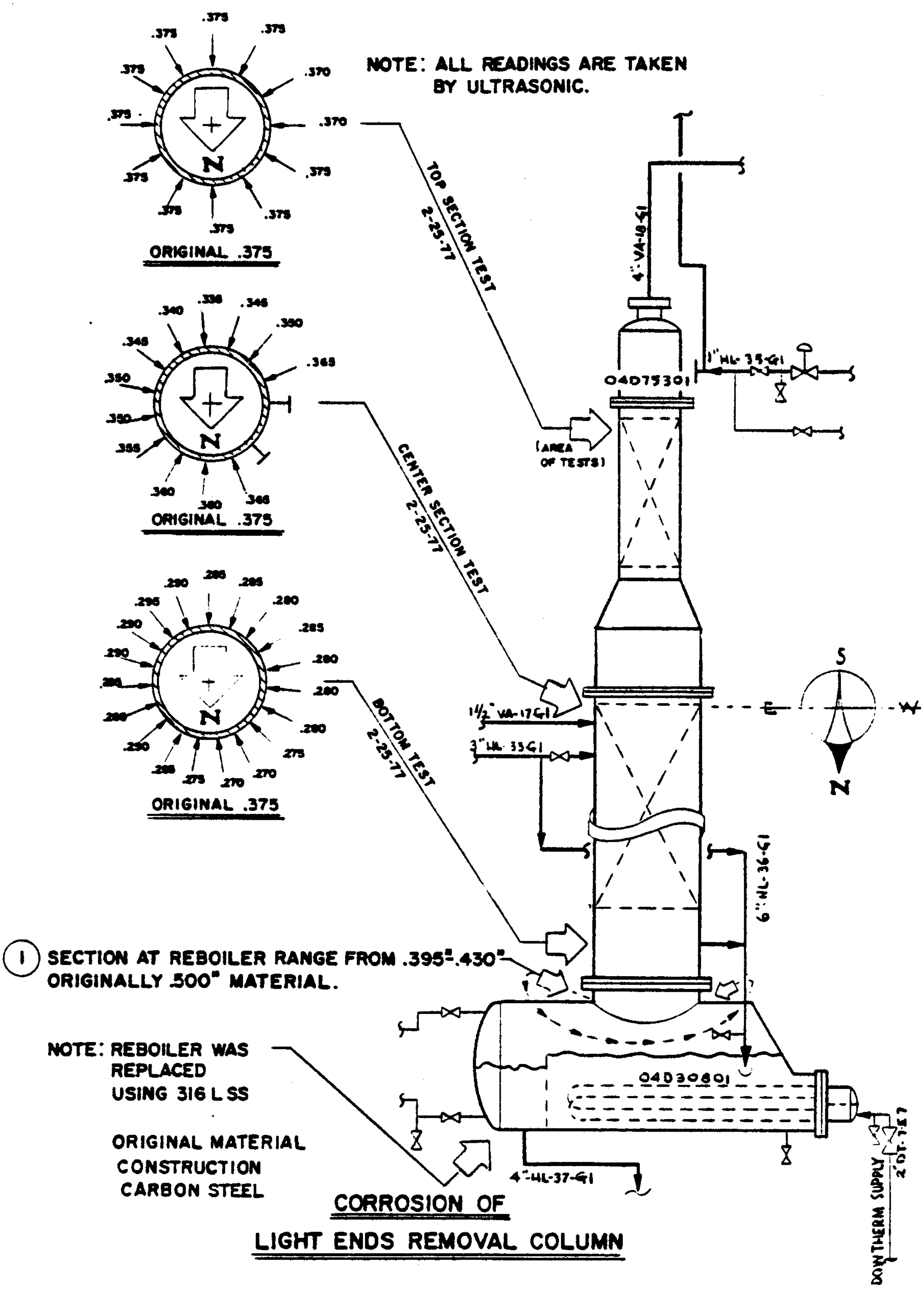
3. Overhead Vapor Coolers

Originally, the vapor coolers had aluminum finned carbon steel tubes. However, it was necessary to replace the light ends, wash solvent, and vacuum flash overhead vapor coolers with 316 SS aluminum finned tubes. The relative corrosiveness of the vapors was consistent with the experience in the columns. Wash solvent, process solvent, and the light ends were of decreasing corrosivity. The effect of the temperatures was also quite

Table 9

Summary of Material Experience For The Light Ends Column Reboiler Tube Bundle

Material	Original O. D. 4-23-76 (inches)	O. D. 2-1-77 (inches)	Metal Loss (O. D.) (inches)
304	1.004	.970	.034
321	1.002	.970	.032
316	.950	.950	.000
Hastelloy G	.950	.950	.000
Incoloy 825	1.001	1.001	.000



CORROSION OF

LIGHT ENDS REMOVAL COLUMN

FIGURE 22

Table 10

Light Ends Column (LEC) Corrosion Rack Summary (Mils Per Year)

Rack No:	177 Reflux Dist.	178 Reboiler	195 Reboiler
Date:			
Installed	05/06/75	05/06/75	04/22/76
Removed	01/02/76	12/16/75	01/27/77
Alloys:			
Carbon Steel	1.7	22.3	86.2
5Cr-1/2Mo	0.8	24.8	
9Cr-1Mo	0.4	17.4	
Type 410	0.2	29.5	.1
Type 304			0.0
Type 316			0.0
Type 321	0.1	2.1	.1
Incoloy 800	0.1	27.1	0.0
29Cr-4Mo			0.0

apparent in the coolers, since the corrosion was much higher in the first three feet of the exchangers. Beyond three feet, there was little corrosion.

4. Vacuum Flash Preheater

The original 7 chrome-1/2 molybdenum preheater coil has little if any erosion/corrosion (see Figure 23). However, the 1-1/4 chrome-1/2 molybdenum outlet line (CS-4) has had considerable metal loss and was replaced.

5. Vacuum Flash Preheater Outlet Line

Since the 1-1/4 chrome-1/2 molybdenum preheater outlet line has operated with and without solids present, the predominant mode of failure (erosion or corrosion) is unknown. Corrosion is definitely present, since the metal loss decreases with decreasing temperatures as shown in Figure 24. However, the increased metal loss at the elbows implies erosion. The installation of a 316 SS section of pipe into the new 1-1/4 chrome-1/2 molybdenum pipe should provide insight into material selection and mode of failure.

6. Vacuum Bottoms Recirculation Line

As shown in Figure 25, the corrosion rate increases as the process temperature increases. The jacketed piping is essentially a countercurrent double pipe exchanger.

Since this line has been in operation for less than a year, the corrosion rate is in excess of 100 mils per year. In the near future, this line will be replaced with 316 SS and/or other materials. A similar Dowtherm jacketed line, CP-26, is also corroded and needs replacing.

7. Overhead Vapor Lines

The corrosion in the wash solvent, light ends, and vacuum flash overhead vapor lines has been significant, but because of the large corrosion allowance, the vapor lines have not failed as frequently. During the replacement of the wash solvent column with 316 L SS, the WSC overhead vapor line was also replaced with 316 L SS due to excessive metal loss. The vacuum flash overhead vapor line has also shown some metal loss but the metal loss has not exceeded the 1/8" corrosion allowance. As in the case of the upper part of the light ends column, the LEC overhead vapor line has not shown much metal loss.

8. Vacuum Flash Condensate Drum

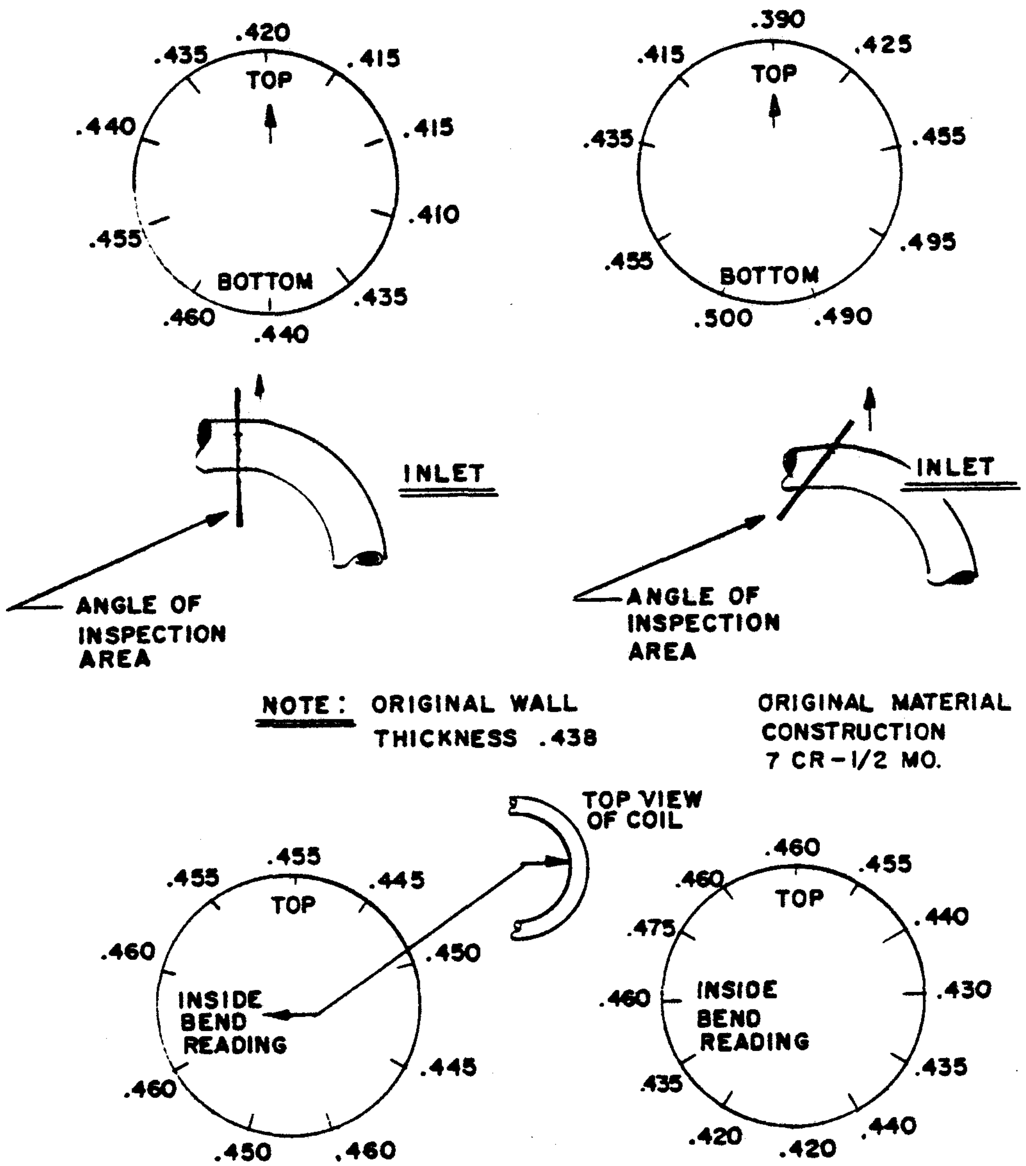
The effect of temperature on corrosion rate is further reinforced by the actual experience and by the corrosion rack data in the vacuum flash condensate drum. The same hot material that is very corrosive to the vacuum flash air cooler, is noncorrosive when cold, as shown in Table 11.

9. Fractionation Summary

The most severe corrosion problems have been in the light end and wash solvent fractionation towers. Field experience has shown that 316 SS will reduce the corrosion rate to less than 10 mils per year with the exception of the wash solvent fractionation trays. The moving parts in the trays are the most susceptible to high corrosion rates. However, short term experience has shown the 316 SS tray in general has an unacceptable rate. Plans have been made to install trays fabricated from alloys such as Incoloy 825, Hastelloy G, Hastelloy C, 316 SS and possibly Incoloy 800.

A corrosion rate that is dependent on temperature is demonstrated by the increasing corrosion rate along the countercurrent Dowtherm jacketed vacuum bottoms recirculation piping. Another example of the temperature effect was shown on the corrosion pattern of the discharge piping of the vacuum flash preheater.

VACUUM FLASH PREHEATER COIL CORROSION-EROSION READINGS



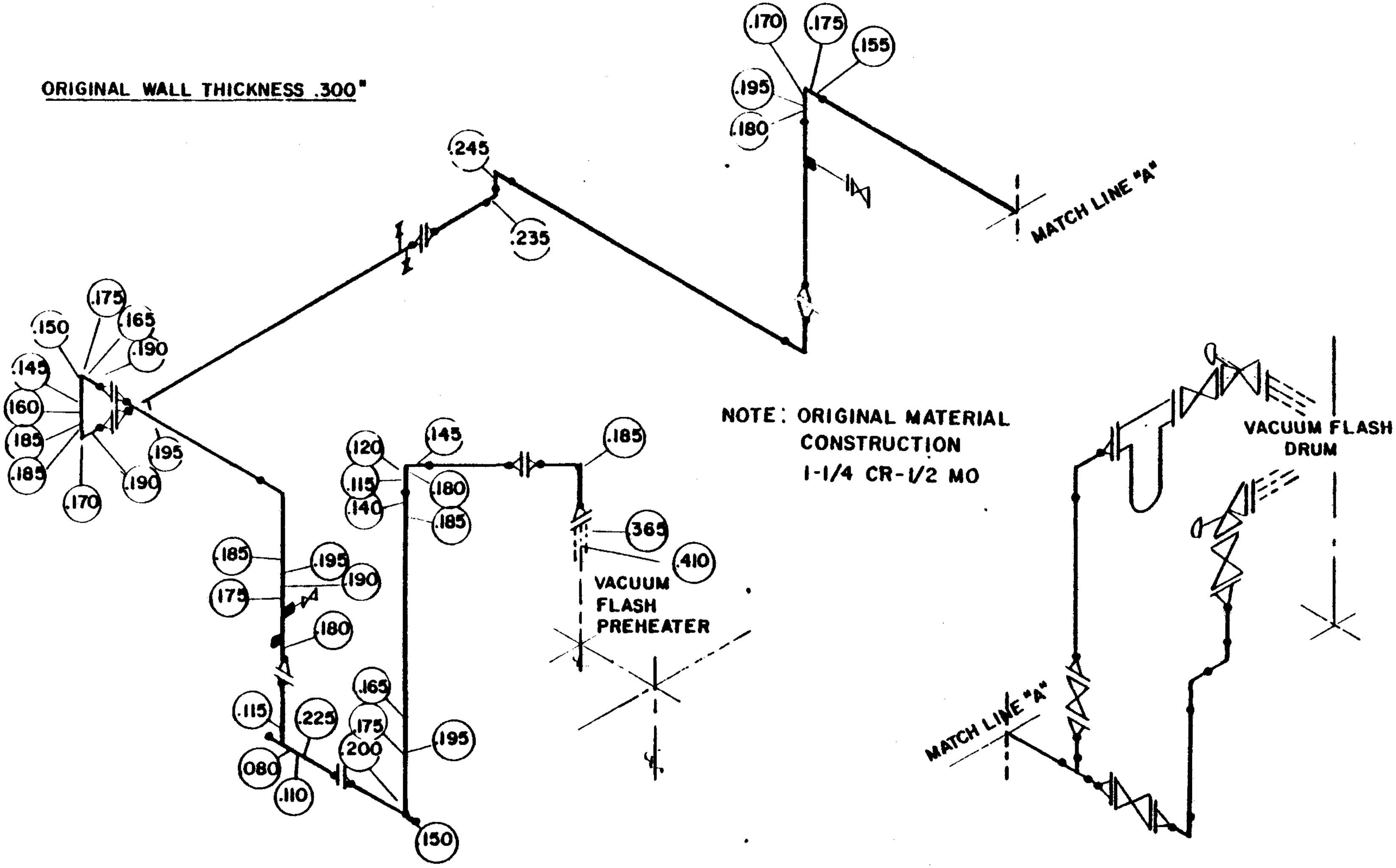
NOTE: ORIGINAL WALL THICKNESS .438

ORIGINAL MATERIAL CONSTRUCTION
7 CR-1/2 MO.

FACING WEST-10 TH. COIL FROM BOTTOM-DOWNSTREAM OF WELD

FACING SOUTH-17 TH. COIL FROM BOTTOM-DOWNSTREAM OF WELD

FIGURE 23



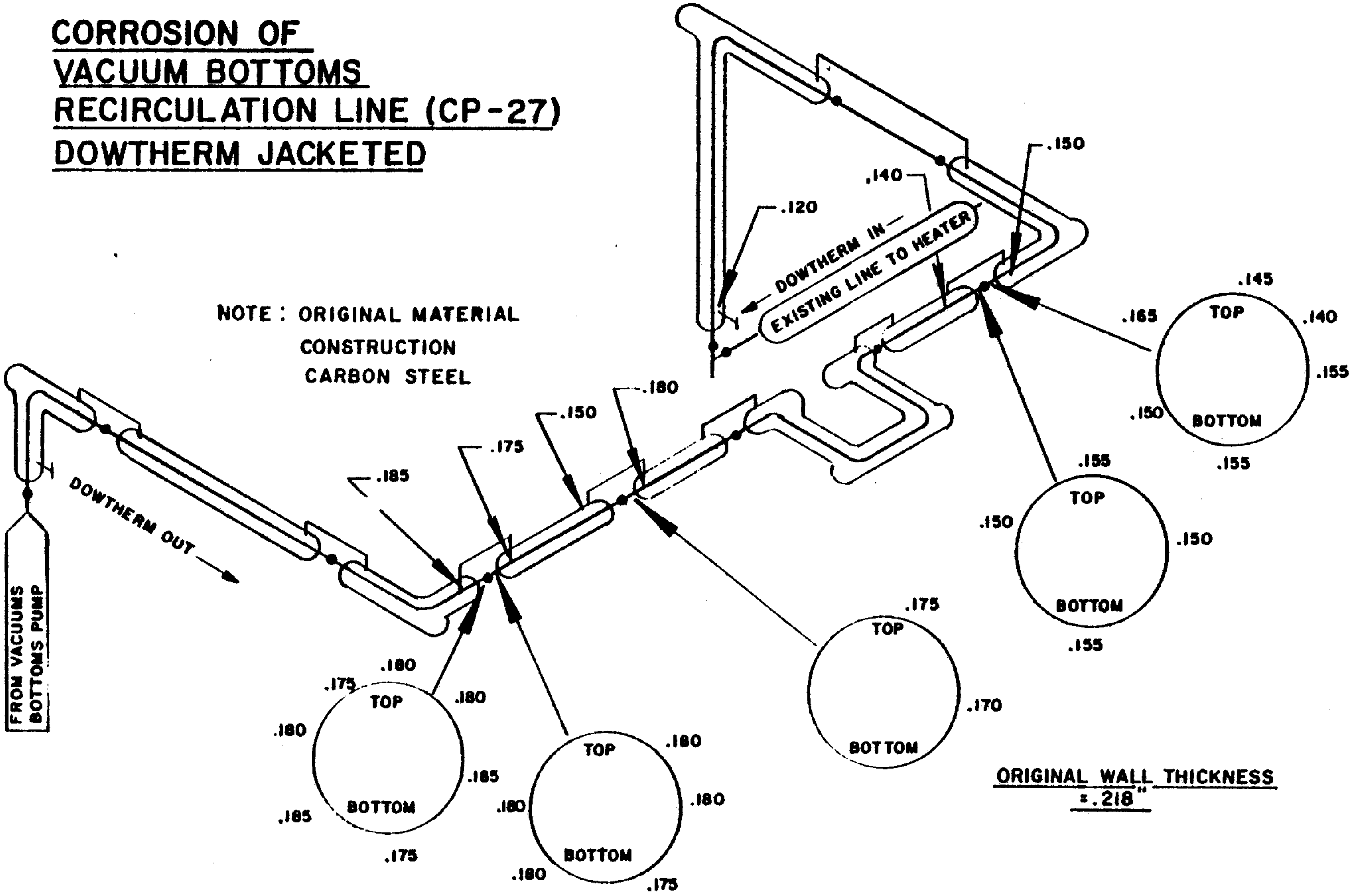
ORIGINAL WALL THICKNESS .300"

NOTE: ORIGINAL MATERIAL CONSTRUCTION 1-1/4 CR-1/2 MO

CORROSION-EROSION OF LINE FROM VACUUM FLASH PREHEATER TO DRUM (3" CS-4-A)

FIGURE 24

CORROSION OF VACUUM BOTTOMS RECIRCULATION LINE (CP-27) DOWTHERM JACKETED



NOTE : ORIGINAL MATERIAL CONSTRUCTION CARBON STEEL

ORIGINAL WALL THICKNESS = .218"

FIGURE 25

Table 11

Vacuum Flash Condensate Drum

Corrosion Rack Summary

(Mils per year)

Rack No.	176
Date:	
Installed	12-20-74
Removed	02-03-77
Alloys:	
Type 304	0.0
Monel	0.0
90/10 Copper-Nickel	0.2
Type 405	0.0

V. PLANT ANALYTICAL LABORATORY

A. Introduction

The plant laboratory was designed to furnish all of the direct analytical services for the plant except for those few on-the-spot analyses performed by the boiler house and waste treatment operators and on-line monitoring analytical instruments. It functions both as a quality control laboratory and as an analytical research unit.

In its quality control mode, the laboratory was equipped and staffed to do the routine day-to-day process control analyses required to monitor the various process streams and products. A sampling schedule is shown in Table 12.

The main analytical methods employed were direct or modified American Society for Testing and Materials (ASTM) standards on Coal and Coke; Gaseous Fuels; Petroleum Products - fuels, solvents, oils; Bituminous Materials - tars; Wood Preservatives - creosotes; and Water. Other speciality procedures utilized have come from the manufacturers of supplied plant units, petroleum companies, authoritative agencies, other coal conversion study groups and plants, as well as those developed at P & M's research laboratories at Merriam, Kansas. Some selected samples have been sent out to commercial laboratories for comparative purposes and for analyses the plant laboratory was not equipped to do. Table 13 is a listing of standard analytical methods which are used.

As an analytical research unit, the initial studies of the laboratory was procedure evaluation and development as needed as the plant came on stream. As the various plant units were started up, many analyses were required. Once the units had been commissioned, the number and frequency of analyses required declined to a minimum number of routine tests needed to monitor the unit.

B. Laboratory Areas and Description

The plant laboratory unit is located in a wing of the administration building occupying a 42 ft. x 68 ft. space divided into nine areas, not including the chemists' office. The areas are designated as: sample receiving, general supplies with personnel lockers and change area, instrument, solvent storage, research studies, main general laboratory, shipment sample retain, pressurized sample vessel (bomb) let-down and sample preparation, loading dock with solvent drum and gas cylinder storage. The steam boiler, air compressor, vacuum pump and heated air make-up furnace to supply specialty laboratory needs are housed in the administration building's mechanical equipment room.

C. Special Studies and Projects

The plant laboratory was involved in obtaining the majority of the results presented in the Process Evaluation section of this report. In addition to this, as an analytical services unit, the lab initiated various procedure evaluation and development studies as well as special assigned projects. Some of the more major endeavors are presented below, not necessarily in order of importance.

1. Comparison of Sulfur Determination Methods

Since one of the main objectives of the SRC process is to reduce the sulfur in coals, the source, course and fate of sulfur throughout the plant was desired. As many samples of differing matrices and sulfur concentrations were involved, a versatile-expedient-accurate method was needed. A review and comparison studies of various methods showed that ASTM D-1552, High-Temperature (LECO) Induction Furnace method, was applicable when modified to run coal derived products. It was found imperative that reference materials, as close as possible to the sample in matrix and sulfur concentration, must be used and the nitrogen interference eliminated to obtain the accuracy desired.

2. Feasibility Comparison of Pyridine Versus Cresol as a Solvent in Insolubles Tests

Results showed, on the average, cresol dissolves one percent (relative) less materials than pyridine. Observations during the tests also revealed that because of the colorless nature of pyridine, thoroughly washed insoluble materials can be readily distinguished from those that need further treatment. Lower boiling point of pyridine is also advantageous for use as a solvent, as evaporation will ensure no trace of pyridine remaining in the insoluble materials. Both solvents are toxic and have offensive odors, yet pyridine is many times more soluble in water than cresol. Thus, a person can wash off pyridine spills on his skin more readily with soap and water, but may suffer chemical and heat burn from a hot cresol spill.

3. Comparison of Fusion Point (Gradient Bar) and Cube-in-Air Softening Point (ASTM D-2319)

A special study was conducted to compare data obtained from our fusion point of SRC by gradient bar method and ASTM D-2319 Softening Point (cube-in-air) in an attempt to determine a relationship between these two tests. Results from the samples selected indicate that the softening point by ASTM D-2319 is generally 70-90°F. higher than fusion point by gradient bar. Softening points could not be determined for samples whose fusion points were above 325°F. as sample preparation would cause cracking and coking of the samples.

4. Analysis of "Oil" in Water by Infrared Spectroscopy

Trace level of "oil" in waste water is hard to detect by solvent-extraction APHA methods; however, it is necessary to check the actual oil concentration in waste water to meet environmental standards. Several methods for trace analyses were evaluated, and a procedure was developed by using infrared spectroscopy to detect the trace amount of oil in waste water. This involves extraction of the oil from water with carbon tetrachloride and the extract is scanned on the infrared spectrophotometer at 2930 cm-1. Absorbance is converted to ppm oil according to a graph using plant solvents as standards.

5. Heating Value of SRC

The heating value of coal can be obtained directly by bomb calorimetry or indirectly by calculation from the elemental values of an ultimate analysis. The usual formula employed in calculating the heat of combustion of coal is that of Dulong:

$$\text{BTU/lb.} = 14{,}544C + 62{,}028 \times \left(H - \frac{O}{8}\right) + 4{,}050S$$

where C, H, O and S are the decimal percents of carbon, hydrogen, oxygen, and sulfur found on the ultimate analysis made on the "as received" coal.

Comparison analyses on plant SRC and laboratory produced vacuum bottoms have shown that the Dulong formula yields heating values about 2.3% higher on the average than those obtained by calorimetry. Thus, a calculated value of 16,000 BTU/lb. by Dulong formula would be high by about 370 units. This difference may be caused by the different bond energies in SRC and coal.

6. Cyanide as a By-Product of the SRC Process

It has been suggested that the cyanide (CN^-) ion may be produced in the SRC process, and the stream of greatest concern in terms of its CN^- content is the plant effluent water. Also, the largest potential source of cyanide in this stream would be recycle process water, which makes up about 1 part in 200 to 1,000 parts of the plant effluent due to dilution of various blowdown, wash and rain waters. By a variety of methods, recycle process water was found to contain 2.9-to-7.8 ppm CN^-, and by dilution effect alone, the concentration would be reduced to 5-to-25 ppb in the plant effluent. By addition of a known amount of CN^- (as KCN) into plant effluent water, it was determined that the plant effluent water contained far below the safety limit of 100 ppb.

The work to date indicates that cyanide is a by-product of the SRC process, but that its concentration in the plant effluent water stream is safely below toxic level.

7. Relationship between Fusion Point and Solvent Entrainment in SRC

Data from this study indicate that the fusion point of SRC is inversely proportional to residual solvent. One test showed that an SRC having a fusion point of 350°F. would yield an average 12% of its weight as solvent to give an SRC of 400°F. fusion point. However, studies with several plant produced SRC's failed to show a consistent relationship. This indicates that there may be factors other than solvent entrainment which determine the fusion point of solvent refined coal.

8. Dust Suppression for Solvent Refined Coal

A method was devised in the laboratory to test the relative dustiness of samples of solvent refined coal. A sample is allowed to fall through a specially designed, enclosed baffled cabinet. The air in the cabinet is sampled and the amount of dust per volume of air is determined. Tests conducted in this manner indicate that SRC from the storage bins is substantially dustier than SRC from the belt. It appears also that SRC which has been dried under moderate heat will be significantly dustier than undried.

It was also shown that water containing a wetting agent suppresses the dust by 30%.

The addition of process-derived solvents and petroleum-derived oil exhibits great dust suppression properties in the 0.4-to-1% range. The resultant material, however, is somewhat sticky.

9. Autoignition Temperature of Plant Solvents

Studies of the autoignition temperature of plant solvents were run by ASTM D-2155 as operations had experienced several small fires which appeared to start spontaneously from solvent soaked insulation.

The autoignition temperature of wash solvent was determined to be about 900°F. and process solvent was about 1000°F. It now appears that the solvents that are absorbed on the insulation are changed with time (i.e. oxidized), perhaps by catalytic action of the insulation, so that their autoignition temperature is lowered. It was shown that if aged solvent-soaked insulation were removed when it was above 400°F., it would spontaneously ignite.

10. Pyrophoric Nature of Mineral Residue

It had been observed that when hot mineral residue was discharged from the dryer, it would spark and glow. This oxidation would continue if left exposed to air, leaving a red-brown ash with an evolvement of sulfur dioxide.

It was determined that mineral residue does not have true pyrophoric properties if it is cooled and in small quantities when first exposed to air. However, if it is above 400°F. when first exposed to air, it will begin to oxidize. Also, it is possible that in very large piles with moisture (e.g. rain) present, spontaneous heat reaction may occur. The main agent to cause this heat is the reactive, micro-sized ferrous sulfide produced from the pyrites in the coal.

11. High Temperature Viscometer

In order to determine the viscosities of SRC, filtered coal solutions, and other coal derived solutions at plant temperatures, the laboratory, cooperating with the process development department, constructed a custom high temperature capillary viscometer that measured viscosities up to 600°F. The interesting conclusion is that the slopes of all of the obtained viscosities were the same with respect to temperature for solvent refined coal solutions (i.e. $\frac{\Delta \text{ viscosity}}{\Delta \text{ temp.}}$ for process solvent = $\frac{\Delta \text{viscosity}}{\Delta \text{temp.}}$ for SRC). Our extrapolated results show that the viscosity of SRC at 600°F. would be 40 centistokes and 20 million centistokes at 340°F., assuming approximately a 325°F. fusion point material.

12. Determination of Dowtherm in Process Solvent

Due to leaks of Dowtherm heat transfer medium into the process solvent stream, a method was needed for its determination as it is a diluent to the system. It was found that the biphenyl oxide portion of Dowtherm gave an IR absorbance at 1240 cm^{-1} which is not seen in process solvent. An infrared method was developed in which 4% and/or 10% solution of the contaminated process solvent are prepared in carbon disulfide. These solutions are scanned between 1300 and 1100 cm^{-1} in 0.2 mm salt cells using carbon disulfide in the reference beam for comparison to a graph made from standard reference solutions.

13. Flow Characteristics of Laboratory Prepared SRC-II Vacuum Bottoms

During the SRC-II mode of operation, the filters will be bypassed and the stripped coal solution going to the flash evaporator will contain coal minerals. Thus, the vacuum residual going to the Sandvik belt will contain about 45% mineral residue. As mineral residue contains, on the average, 67% ash, this vacuum residual stream will contain about 30% ash. To recover the maximum amount of liquid product (i.e. solvents), the fusion point of the residual should be around 300-350°F. Since the product line is heated to around 600°F. by Dowtherm, the residual must flow at a temperature somewhat below 600°F. To study the flow characteristics of this stream, simulated materials were made in the laboratory. From the data obtained, it was concluded that during SRC-II startup, ash contents of 30% in the vacuum bottoms should be approached very slowly as problems of low fluidity at 600°F. may be encountered at about 27% ash in materials with fusion points of 350°F.

14. Naphthenic Acid in Plant Solvents

It had been proposed that the high corrosion rate in the wash solvent column may be due to the presence of "naphthenic acid" in the feedstock. According to the ASTM Metal Handbook, volume 1, 8th ed. p. 596, naphthenic acids are "mono and bicyclic carboxylic acids in a wide range of molecular weights and boiling between 350-to-650°F." Tests were conducted according to methods listed in ASTM D-644 with some modification. Results indicating no carboxylic acid of any kind were found, and that naphthenic acid, as defined by ASTM, are not present in sufficient quantity to cause corrosion. Some exploratory studies have shown that the fraction of wash solvent boiling at 445-510°F. was the most corrosive. However, comparative tests using cresylic acids show them not to be corrosive. Therefore, the corrosive agents are neither naphthenic nor cresylic acids and are yet unknown.

Table 12

Sample Schedule and Frequency

1. Routine Samples

Sample	Frequency	Analysis Required
Feed Coal	Every 4 hours for daily composite	Moisture, Ash
Ground Coal	Every 4 hours for daily composite	Moisture, Particle Size, Carbon, Hydrogen, Nitrogen, Sulfur, Ash. Sulfur Forms on Weekly Composite
Slurry Blend	Every 24 hours	Ash, Solvent to Coal Ratio
High Pressure Flash Drum Liquid (Unfiltered Coal Solution)	Every 24 hours	Compositional Analysis: Pressure Filtration, Blackness, Pyridine Insolubles, Infrared Ratio (IRR) Ash, Carbon, Hydrogen, Nitrogen, Sulfur Oxygen, Fractionation by Vacuum Distillation, Gas Analysis, Molecular Weight of Gas
Recycle Condensate	Every 24 hours	Water %, Oil %, Gas Analysis, Boiling Range w/w
Stripper Overhead	Every 12 hours	Boiling Range v/v
Recycle Process Water, Oil Phase	Every 24 hours	Specific Gravity, Boiling Range v/v, Water %
Recycle Process Water	Every 24 hours	Phenol, Nitrogen, Sulfur
Filter A Feed	Every 8 hours	Pyridine Insolubles, Boiling Range v/v

1. Routine Samples (Continued)

Sample	Frequency	Analysis Required
Filter B Feed	Every 8 hours	Pyridine Insolubles, Boiling Range v/v
Filter A Filtrate	Every 2 hours	Pyridine Insolubles, Specific Gravity, Viscosity
Filter B Filtrate	Every 2 hours	Pyridine Insolubles, Specific Gravity, Viscosity
Mineral Residue Dryer Condensate	Every 24 hours	Pyridine Insolubles, Boiling Range v/v
Mineral Residue	Every 8 hours	Pyridine Insolubles, Ash. Daily test for Carbon Hydrogen, Nitrogen, Sulfur
Filter A Cake Leg	Every 8 hours	Pyridine Insolubles
Filter B Cake Leg	Every 8 hours	Pyridine Insolubles
Solvent Refined Coal	Every hour	Fusion Point, Ash, Sulfur. Daily-test for Carbon, Hydrogen, Nitrogen, Sulfur
Light Oil	Every 8 hours	Boiling Range v/v/
Wash Solvent	Every 8 hours	Boiling Range v/v
Process Solvent	Every 8 hours	Boiling Range v/v
Plant Effluent	Daily Composite	Phenol, Oil, pH, Total Suspended Solids, Chemical Oxygen Demand, Biological Oxygen Demand

1. Routine Samples (Continued)

Sample	Frequency	Analysis Required
Back Wash Tank Effluent	Every 24 hours	Phenol, Oil, Total Suspended Solids, pH, Chemical Oxygen Demand, Biological Oxygen Demand
Graver Feed	Twice weekly	Phenol, pH, Alum Demand, Chemical Oxygen Demand, Total Suspended Solid
Graver Effluent	Twice weekly	Phenol, pH, Total Suspended Solid, Chemical Oxygen Demand
Bio-Unit Feed	Every 24 hours	Phenol, pH, Total Suspended Solid, Chemical Oxygen Demand, Biological Oxygen Demand
Bio-Unit Aerator	Twice weekly	pH, Total Suspended Solid
Bio-Unit Clarifier	Twice weekly	Dissolved Oxygen
Bio-Unit Effluent	Every 24 hours	Phenol, pH, Total Suspended Solid, Chemical Oxygen Demand, Biological Oxygen Demand
Sand Filter Effluent	Weekly	Phenol, pH, Total Suspended Solid, Chemical Oxygen Demand
Carbon Filter Effluent	Weekly	Phenol, pH, Total Suspended Solid, Chemical Oxygen Demand
Waste Effluent Lagoon Water Composite	Weekly	Phenol, pH, Oil, Total Suspended Solid, Chemical Oxygen Demand

1. Routine Samples (Continued)

Sample	Frequency	Analysis Required
Cooling Water	Every 24 hours	Zinc, Phosphate, Iron, pH, n-Conductivity
Demineralized Water	Every 8 hours	Silica
Deaerator Feed	Every 12 hours	Silica
Boiler Feed	Every 12 hours	Silica
Boiler A Effluent	Every 24 hours	Silica, Iron
Boiler B Effluent	Every 24 hours	Silica, Iron
Inert Gas Generator Flue	Weekly	Gas Analysis
Nitrogen Product Gas	Weekly	Gas Analysis
Lean MEA	Every 48 hours	Carbon Dioxide
Rich MEA	Every 48 hours	Carbon Dioxide, Vanadium, Monoethanolamine
Hydrogen Product Gas	On request	Gas Analysis
Lean DEA	Every 48 hours	Hydrogen Sulfide, Carbon Dioxide, Petromeen-52 (Corrosion Inhibitor)
Rich DEA	Every 48 hours	Foaming Tendency, Petromeen-52, Carbon Dioxide, Hydrogen Sulfide, Diethanolamine

Sample Schedule and Frequency

2. Inventory Samples - The following samples are taken once a week on Monday at 1400 hours.

Sample	Analysis Required
Recycle Condensate	Water, Oil, Boiling Range w/w
Oil from Recycle Process Water	Boiling Range v/v
Filter Feed Surge Vessel Liquid	Boiling Range by Vacuum Distillation
Vapor Surge Drum A	Boiling Range v/v
Vapor Surge Drum B	Boiling Range v/v
Filter Gas Scrubber A	Boiling Range v/v
Mineral Residue Dryer Condensate	Boiling Range v/v
Filter Feed Flash Liquid	Boiling Range by Vacuum Distillation
Vacuum Flash Feed Accumulator	Boiling Range by Vacuum Distillation
Light Oil	Boiling Range v/v, Carbon, Hydrogen, Nitrogen, Sulfur
Light End Column Feed Tank	Boiling Range by Vacuum Distillation
Process Solvent Accumulator	Boiling Range by Vacuum Distillation, Carbon, Hydrogen, Sulfur, Nitrogen
Wash Solvent Accumulator	Boiling Range v/v, Carbon, Hydrogen, Nitrogen, Sulfur

2. Inventory Samples (Continued)

Sample	Analysis Required
Flash Knock-Out Drum, Oil	Boiling Range v/v, Water %, Oil %
Hot-Well, Oil	Boiling Range v/v
Precoat Slurry Pressure Vessel	Pyridine Insoluble
Precoat Atmosphere Mix Tank	Pyridine Insoluble
Collection Tank at Flare Knock-Out Drum	Pyridine Insoluble, Boiling Range by Vacuum Distillation

3. Material Balance Samples

\- These samples should be taken daily during any material balance run, at 1400 hours. Also, be taken at the starting and ending hour of all material balance runs.

Filter Feed Surge Vessel	Boiling Range by Vacuum Distillation
Filter Feed Flash Vessel	Boiling Range by Vacuum Distillation
Vacuum Flash Feed Accumulator	Boiling Range by Vacuum Distillation
Vacuum Flash Condensate	Boiling Range v/v
Light Oil Product	Boiling Range v/v, Carbon, Hydrogen, Nitrogen, Sulfur

3. Material Balance Samples (Continued)

Sample	Analysis Required
Light Ends Column Feed Tank	Boiling Range v/v
Process Solvent Accumulator	Boiling Range v/v, Carbon, Hydrogen, Nitrogen, Sulfur
Flash Knock-Out Drum, Oil	Boiling Range v/v, Water %, Oil %
Hot Well Oil Tank	Boiling Range v/v

Table 13

Standard Analytical Methods

1. Analysis of Coal and Coke Ash
   Method: ASTM D-2795 .

2. Ammoniacal Nitrogen in Recycle Process Water, Determination of
   Method: AOAC Method 2.055
   Note: Ammoniacal Nitrogen is determined by a modified Kjeldahl method.

3. Ash in Coal, Determination of
   Method: P & M F117-76
   Note: Reference to ASTM D-271 and ASTM D-3174 .

4. Ash in Solvent Refined Coal, Determination of
   Method: P & M F148-76
   Note: Rapid Ashing method using custom made tube-furnace, with nitrogen and oxygen atmospheres to vaporize and quantitatively ash the sample.

5. Benzene Insoluble of Coal Tar Distillate and Related Products, Determination of Method: ASTM D-2317.

6. Biological Oxygen Demand, Determination of
   Method: P & M F110-75
   Note: Modified from APHA Standard Methods, 13th. ed., p. 219 (1971) Air incubator or water bath for bottles are not used.

7. Blackness of Coal Tar Distillate by Infrared Spectrophotometry
   Method: P & M F131-76
   Note: References:
   OCR R&D Report No. 53, Interim Report #6, page 39.
   OCR R&D Report No. 53, Interim Report #7, pages 62 and 63.
   OCR R&D Report No. 53, Interim Report #8, page 35.
   Absorbance per gram of coal solution per 100 ml. measured in 1/2" cell at 550 nanometers.

8. Boiling Range of Coal Tar Distillate by Distillation v/v and w/w
   Method: P & M F116-75, F132-75
   Note: Modified from ASTM D-86. Corning No. 4900 Saybolt Flask is used, condenser chamber temperature modified.

9. Boiling Range of Coal Tar Distillate by Vacuum Distillation v/v
Method: P & M F130-75
Note: Modified from ASTM D-1160.

10. BTU Heating Value, Determination of
Method: Dulong Formula
Note: BTU/pound = 14,544C + 62,028 $(H - \frac{0}{8})$ + 4050S

Where the elements are in decimal **percents** found on ultimate analysis made on "as received" basis.

11. Carbon and Hydrogen in Coal, SRC, and Related Products, Determination of. Method: P & M F113-76
Note: A weighed portion of sample is vaporized and burned quantitatively in the presence of oxygen in a combustion tube containing copper oxide. The carbon is converted to carbon dioxide and the hydrogen to water. These compounds are collected quantitatively on Ascarite and magnesium perchlorate respectively. The carbon and hydrogen contents of the sample are determined gravimetrically by appropriate calculation. Instrument used is Hallikainen Macrocombustion Apparatus, made by Totco Division, Baker Oil Tools, Inc., Glendale, CA 91201.

12. Infrared Ratio (IRR) in Coal Tar Distillate by Infrared Spectrophotometry
Method: P & M F134-76
Note: References:
OCR R&D Report, Interim Report No. 8, pages 39-42. By the general techniques of quantitative analysis as presented in ASTM E 168, a baseline is drawn from the vicinity of 3800 cm^{-1} to the vicinity of 2000^{-1}. The absorbance of the bands at 2920 and 3050 cm^{-1} are measured, and the ratio Absorbance of the banks at 2900 cm^{-1}/Absorbance at 3050 cm^{-1} is calculated.

13. Carbon Dioxide Loading in Monoethanolamine Solution
Method: Sample acidified and volume of released CO_2 measured.

14. Chlorine, Total, in Waste Water, Determination of
Method: HACH DPD Method (HACH Chemical Company, Ames, Iowa)
Note: Modified from APHA Standard Methods, 13th ed., p. 129 (1971).

15. Chlorine, Free, in Waste Water, Determination of
    Method: HACH DPD Method
    Note: Modified from Standard Methods, 13th ed., p. 129 (1971).

16. Chemical Oxygen Demand, Determination of
    Method: P & M F111-76
    Note: Adapted from Standard Methods, 13th ed., p. 220 (1971).

17. Cloud Point in Coal Tar Distillate, Determination of
    Method: ASTM D-2500 .

18. Coking Value of Tar and Pitch
    Method: ASTM D-2416 .

19. Compositional Analysis of Unfiltered Coal Solution by Vacuum Distillation
    Method: P & M F128-76
    Note: Reference:
        OCR R&D Report, Interim Report No. 8, pages 27A, 28.
        OCR R&D Report, Interim Report No. 7, pages 82-85.
        Specially designed apparatus is used to distill the coal solution under 0.5 mm to 1.0 mm Hg of Pressure.

20. Conradson Carbon of Solvent Refined Coal
    Method: P & M F101-75
    Note: Adapted from ASTM D-189/2416 using a one gram sample.

21. Conductivity of Aqueous Solution, Determination of
    Method: P & M F135-76
    Note: Adapted from ASTM D-1125.

22. Cyanide in Waste Water, Determination of
    Method: P & M F136-76
    Note: Orion Specific Ion Electrode Method:
        Several additions of a known cyanide solution are made to an unknown sample containing the complex Ag $(CN)_2$. The free cyanide in the known addition standard depresses the level of dissociated silver ion from the comples, and the silver electrode is used to record the loss of silver. The potential is recorded after each addition on special semi-antilog graph paper called Gran's Plot paper. The data will fall on a straight line which will intercept the horizontal axis of the paper at a point corresponding to the concentration of cyanide in the sample.

23. Density, Appearance, Determination of
    Method: ASTM D-167.

24. Density, True, Determination of
    Method: ASTM D-167.

25. Dew Point in Instrument Air, Determination of
    Method: ASTM D-1142
    Note: Lindberg Dew Point Instrument, Model 59910 is used. May be obtained from Lindberg, Division of Sola Basic Industries, Watertown, Wisconsin 43094.

26. Diethanolamine Concentration in Aqueous Solution, Determination of
    Method: P & M F119-75
    Note: Sample is added into standard sulfuric acid solution and excess acid is back titrated to determine the amount of diethanolamine present.

27. Dissolved Oxygen in Bio-Unit, Determination of
    Method: APHA Standard Methods, 13th Edition, 218F, (1971).

28. Dowtherm in Coal Tar Distillate by Infared Spectrophotometer, Determination of. Method: P & M F137-76
    Note: Sample is dissolved in Carbon Disulfide, and scanned between 1250 cm^{-1} and 1220 cm^{-1}, and the absorption at 1240 cm^{-1} is determined for comparison to a standard graph.

29. Filter-Aid Percent in Filter Cake, Determination of
    Method: P & M F138-76
    Note: An iron dilution method where % iron in filter cake ash and % iron in raw coal ash are determined and % filter aid is calculated from the variance.

30. Foaming Tendency of DEA Solution, Determination of
    Method: P & M F105-75
    Note: Foam height caused by air sparged into solution. Adapted from "Gas Conditioning Fact Book" by Dow Chemical, page 339 (1962).

31. Foaming Tendency of MEA Solution, Determination of
    Method: P & M F104-75
    Note: Foam height caused by air sparged into solution. Adapted from "Gas Conditioning Fact Book: by Dow Chemical, page 399 (1962).

32. Fusion Point of Solvent Refined Coal by Gradient Bar

    Method: P & M F120-76

    Note: Melting Point Apparatus Series 3821 - Dennis Type, made by Parr Instrument Company is used. Finely ground sample is sprinkled on the gradient bar, then brushed off lightly after 30 seconds. Temperature measurement is taken at the point where fusion started.

33. Gas Analysis by Gas Chromatography

    Method: P & M F127-76

    Note: Two stainless steel columns of 4 meters with 1/4" O.D. are used; one containing Molecular Sieve 5A 60/100 mesh, and one containing Porasil A 100/150 mesh. Hewlett Packard Gas Chromatograph Model No. 5710A is used with argon carrier gas.

34. Hydrogen & Carbon in Coal, SRC, and Related Products, Determination of. Method: P & M F133-76

    Note: A weighed portion of sample is vaporized and burned quantitatively in the presence of oxygen in a combustion tube containing copper oxide. The carbon is converted to carbon dioxide and the hydrogen to water; these compounds are collected quantitatively on Ascarite and magnesium perchlorate respectively. The carbon and hydrogen contents of the sample are determined gravimetrically by appropriate calculation. Instrument used is Hallikainen Macrocombustion Apparatus, made by Totco Division, Baker Oil Tools, Inc., Glendale, CA 91201.

35. Hydrogen Sulfide in Diethylanolamine Solution, Determination of

    Method: P & M F126-76

    Note: Sample is added into 50 ml. of chilled 10% HCl solution, excess 0.1 normal iodine solution is added and back titrated with sodium thiosulfate. Reference: L. Meties, "Handbook of Analytical Chemistry," McGraw Hill, page 3-70, Method No. 85.

36. Hydrogen Sulfide in Gases, Determination of

    Method: P & M F140-76

    Note: Adapted from ASTM D-2385 and ASTM D-1071. Gas sample is absorbed in Cadmium Sulfate solution, volume absorbed is measured by an orsat apparatus.

37. Iron Carbonyl, Determination of

    Method: P & M F139-76

    Note: The iron pentacarbonyl is extracted from the air sample with a solution of iodine in acetic acid. After reduction of the iodine with sodium sulfite, the iron is reduced to the ferrous state with hydroxylamine hydrochloride. The solution is buffered with sodium acetate and reacted with o-phenanthroline. The intensity of the iron-phenanthroline complex is measured in a color comparator.

38. Iron in Coal, Determination of
    Method: P & M F115-76
    Note: Reference: Kolthoff, Sandell, Mechan, Bruckenstein, "Quantitative Chemical Analysis," 4th ed., pages 829, 832, 833, 839. The McMillan Company, New York, 1971.
    Frank J. Welcher, ed., "Standard Methods of Chemical Analysis" 5th ed., Vol. IIa, p. 827. Van Nostrand Reinhold Company, New York, 1963.

39. Iron in Diethanolamine Solution, Determination of
    Method: P & M F107-76
    Note: Modified APHA Standard Methods, 13th ed., 124A (1971).

40. Iron in Monoethanolamine Solution, Determination of
    Method: P & M F106-76
    Note: Modified APHA Standard Methods, 13th ed., 124A (1971).

41. Iron in Cooling Water, Determination of
    Method: HACH - 1, 10 - Phenanthroline Method
    Note: Modified APHA Standard Methods, 13th ed., 189 (1971).

42. Iron in Waste Water, Determination of
    Method: HACH - 1, 10 - Phenanthroline Method
    Note: Modified APHA Standard Methods, 13th ed., 189 (1971).

43. Mercaptan in Gases, Determination of
    Method: Detector Tubes, as per supplier instruction.
        Manufacturer: Dragerwerk-AG-Lubeck
        Supplied by: Safety & Supply Co., Seattle, WA .

44. Moisture in Coal, Determination of
    Method: ASTM D-271.

45. Molecular Weight of Gases, Determination of
    Method: P & M F125-76
    Note: Reference:
        OCR R&D Report, Interim Report No. 8, 30, (1974)
        Molecular weight is determined by the gas density method.

46. Monoethanolamine Concentration in Aqueous Solution, Determination of. Method: P & M F125-76
Note: Sample is added into standard sulfuric acid solution, and excess acid is back titrated to determine the amount of monoethanolamine present.

47. Nickel Tetracarbonyl in Air, Determination of
Method: P & M F141-76
Note: Nickel tetracarbonyl is extracted from atmosphere with dilute hydrochloric acid. The extract is made basic with ammonium hydroxide and sodium hydroxide and reacted with alpha-furildioxime. The nickel-alpha-furildioxime chelate is extracted with chloroform and the color intensity of the extract is measured in a color comparator. The color comparator reading is converted to the concentration of nickel by means of a previously established calibration curve. Nickel tetracarbonyl concentration as volume percent is obtained by appropriate calculation.

48. Nitrogen in Coal Tar Distillate and Related Products, Determination of. Method: P & M F112-75
Note: Kjeldahl-Gunning Method, ASTM D-271, ASTM D-3179
Organic nitrogen is converted to ammonium salts by destructive digestion of the sample with a hot catalyzed mixture of sulfuric acid and potassium sulfate. The ammonium salts are subsequently converted to ammonia by the addition of sodium hydroxide, and the ammonia is recovered by distillation into a boric acid solution. The recovered ammonia is then titrated with a standard sulfuric acid solution, and the percent nitrogen in the original sample is calculated.

49. Oil in Water, by Infrared Spectrophotometry
Method: P & M F142-76
Note: Reference: Perkin-Elmer Infrared Bulletin No. 11, (Perkin Elmer, Norwalk, Conn. 06856)
Sample is extracted with carbon tetrachloride with a 50 fold concentration increase, an absorption path length of 1 cm. The region from 3300 cm^{-1} is scanned. The absorbance of the C-H band is measured from the base line of the peak to the maximum absorbance, at about 2930 cm^{-1}. Plant solvents used as "oil" references.

50. Oil in Water, by Petroleum Ether
Method: APHA Standard Methods, 13th ed., 137 (1971).
Note: Sample is extracted with petroleum ether, and filtered through a phase-separating filter paper into a small boiling

flask. Content of the flask is evaporated to dryness and the increase in weight is calculated as oil.

51. Particle Size of Ground Coal, by Sieve Analysis
    Method: P & M F143-76
    Note: References: ASTM D-410, ASTM D-431
    Allen Bradley Sonic Sifter, Model L3-PF is used, obtainable from Fisher Scientific Company, Pittsburgh, PA 15219.

52. Pentane Insolubles in Process Solvents and Related Products
    Method: ASTM D-893
    Note: Procedure A is used.

53. Petromeen 52 Concentration in DEA Solution, Determination of
    Method: According to manufacturer's suggestion.
    Note: Manufacturer: Betz, Trevose, PA 19047.

54. pH in Water
    Method: APHA Standard Methods, 13th ed., 144 (1971).

55. Phenol in Waste Water, Determination of
    Method: HACH 4-Aminoantipyrine Method
    Note: Adapted from APHA Standard Methods, 13th ed., 504 (1971).

56. Phosphate, Total, in Water, Determination of
    Method: APHA Standard Methods, 13th ed., 223 (1971).

57. Phosphate, Ortho, in Water, Determination of
    Method: HACH Ascorbic Acid Method
    Note: APHA Standard Methods, 13th ed., 532 (1971).

58. Porosity of Coal, Determination of
    Method: ASTM D-167.

59. Pour Point in Coal Tar Distillate, Determination of
    Method: ASTM D-97.

60. Proximate Analysis of Coal
    Method: ASTM D-3172.

61. Pressure Filtration of Unfiltered Coal Solution for Compositional Analysis
    Method: P & M F144-76
    Note: Custom designed and made filters are used. Filtration is at 260°F. and 90 psi of nitrogen.

62. Pyridine Insolubles in Coal Tar Distillation and Related Products.
    Method: P & M F129-76
    Note: A weighed sample is heated with pyridine and then filtered. The filter cake is rinsed with pyridine, followed with acetone. The filter cake is dried and then weighed.

63. Quinoline Insolubles in Coal Tar Distillation and Related Products
    Method: ASTM D-2318.

64. Silica in Deionized Water, Determination of
    Method: HACH Heteropoly Blue Method
    Note: APHA Standard Methods, 13th ed., 306 (1971).

65. Solvent-to-Coal Ratio in Coal Slurry in SRC Process
    Method: P & M F145-76
    Note: Percent of ash in Coal Slurry (C.S.) and percent of ash in feed coal is obtained: $(\%\ \text{Ash in Coal} \div \%\ \text{Ash in C.S.})^{-1}$.

66. Specific Gravity of Coal, SRC, and Other Solids
    Method: ASTM D-71, D-792.

67. Specific Gravity of Coal Tar Distillate, Determination of
    Method: ASTM D-1298.

68. Sulfur as Sulfate by Gravimetric Method, Determination of
    Method: ASTM D-129.

69. Sulfur Forms in Coal, Determination of
    Method: ASTM D-2492.

70. Sulfur Forms in Mineral Residue, Determination of
    Method: P & M F123-76
    Note: Sulfide sulfur is determined by suspending a sample in water in a flask which is joined in a closed system to a pair of absorbing solutions in series. The sample suspension is then made strongly acid and boiled to expel the hydrogen sulfide formed. The hydrogen sulfide is carried by a nitrogen purge into the absorber vessels where it is captured as zinc sulfide. The absorber solutions are combined and the sulfide determined by iodmetry. Sulfate sulfur and pyritic sulfur are determined according to ASTM D-2492.

71. Sulfur in Hydrocarbon, Total, Determination by LECO High Frequency Combustion/Titration Procedure
    Method: P & M F121-76
    Note: Adapted from manufacturer's suggested procedure. Sample is burned in a stream of oxygen in a high frequency induction

furnace at a sufficiently high temperature to convert approximately 95% of the sulfur to sulfur dioxide. The combustion products are passed into the absorber of an automatic titrator containing an acid solution of potassium iodide and starch indicator. A blue color is developed initially by addition of a small amount of standard potassium iodate solution. As the combustion proceeds, sulfur dioxide reduces the iodine, which bleaches the blue color. More iodate is then added automatically to maintain the original color. The sulfur content is calculated from the volume of potassium iodate consumed.

72. Total Dissolved Solid in Water, Determination of
Method: P & M F146-76
Note: Adapted from APHA Standard Methods, 13th ed., 224 (1971). Dissolved matter may be obtained by difference between the residue on evaporation and total suspended matter or by evaporating a filtered sample as directed for residue on evaporation.

73. Suspended Solid in Waste Water, Total, Determination of
Method: APHA Standard Methods, 13th ed., 224 (1971).

74. Ultimate Analysis of Coal
Method: See Determination of Carbon, Hydrogen and Nitrogen.

75. Vacuum Distillation of Unfiltered Coal Solution for Compositional Analyses
Method: P & M F128-76
Note: Reference:
OCR R&D Interim Report No 7, pages 82-85
OCR R&D Interim Report No. 8, pages 27A, 28
Specially designed apparatus is used to distill the coal solution under 0.5 mm to 1 mm Hg of pressure.

76. Vanadium in MEA Solution, Determination of
Method: P & M F146-76
Note: This method, adapted from Benfield Report R-119, page 3, used hydrazine to reduce pentavalent vanadium to tetravalent vanadium. The characteristic "vanadium blue" is produced by acidifying the solution. Absorbance at 785 mm is then proportional to vanadium concentration.

77. Viscosity of Coal Tar Distillate, Determination of
Method: ASTM D-2515
Note: Procedure for reverse flow viscometers for transparent and opaque liquids .

78. Volatiles in SRC, Determination of
    Method: P & M F101-77
    Note: 100-Coking value (ASTM-D-189/2416 using one gram sample) equals volatiles.

79. Water, Trace, Determination of
    Method: Karl-Fisher Titration
    Note: Automatic titrator used.

80. Water, Percent, Determination of
    Method: ASTM D-95.

81. Zinc in Cooling Water, Determination of
    Method: HACH ZincoVer V Zinc Reagent Method
    Note: APHA Standard Methods, 13th ed., 363 (1971).

VI. PROCESS DEVELOPMENT

A. General

The process development group principally performed the following categories of work:

- Testing of small-scale equipment on slip-stream samples from the plant in order to evaluate possible process improvements.
- Development of special analytical or performance data using small-scale equipment.
- Performing various process engineering tasks for improved plant operation, for overcoming plant operating problems or for major plant modification.

The process engineering tasks mentioned above are reported in other sections of this report so they will not be discussed here. The slip-stream and small-scale testing is discussed below.

B. Study of Hot Solvent-Coal Mixing

Previous work indicated that problems would be encountered when coal and solvent or recycle slurry were mixed at elevated temperatures. A small-scale test apparatus was assembled and tests completed to determine the effects of operating variables. Samples of plant pulverized coal, process solvent and synthesized slurry recycle were used. From these studies, an improved method of mixing coal with hot recycle slurry was designed and will be used for SRC-II work in the plant.

Studies were performed on both hot and cold slurries, sometimes in open vessels to study mixing action. Most of the study was performed in a continuous stirred tank system with cold coal feed, heated solvent feed, a circulating heater, a capillary viscometer and a vibrating probe viscometer. The stirred tank was 8 inches in diameter with a coned bottom. Depth and feed rate were varied to control retention time, usually from 10 to 20 minutes. Figure 26 summarizes the operating variables effect on viscosity of various solvents and slurries, all from Kentucky coal.

The swelling or gel formation tendency is most dependent on temperature for both SRC-I and SRC-II feed mixtures. The SRC-II feed slurry is much more viscous than that for SRC-I.

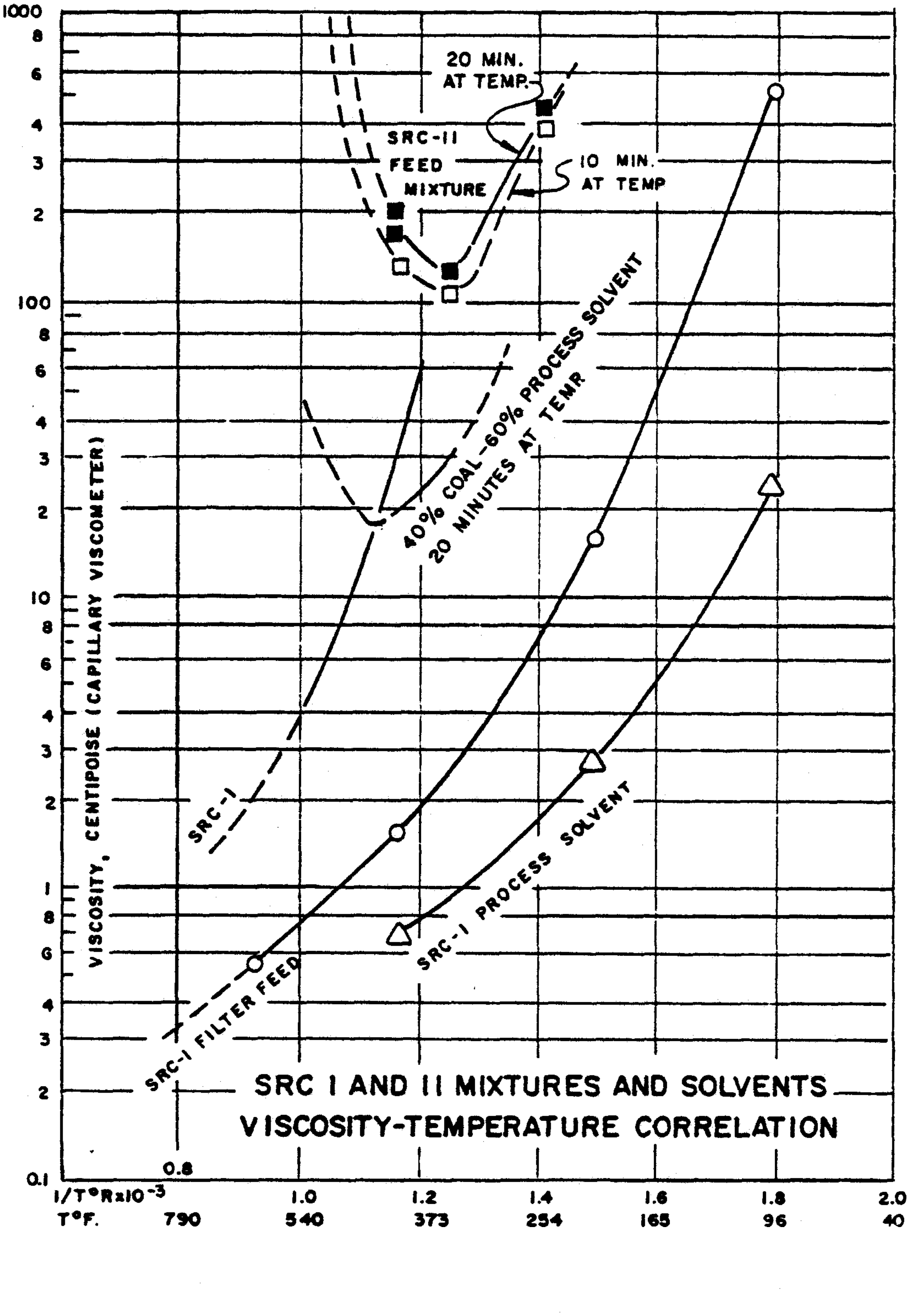

SRC I AND II MIXTURES AND SOLVENTS
VISCOSITY-TEMPERATURE CORRELATION

FIGURE 26

The effect of time at temperature is not well defined but is significant. Note, too, the minimum viscosity for SRC-I feed mixture occurs at about 450°F. while that for SRC-II is lower, at about 340°F. Shear rate can change the viscosity of the coal slurries by one or two orders of magnitude. That is, the slurries act pseudo-plastic. The slurry measurements were all made in the shear range of 700-1500 seconds $^{-1}$. At shears of 70-100 seconds $^{-1}$, the viscosity would approximately double.

Tests were run at ambient conditions with varying tank and mixer sizes to determine scaleup factors. While incomplete, these tests indicated that a vortex is the most practical method (of the several surveyed) of wetting the coal. An unbaffled tank with dual propellors was designed for the SRC-II modifications to the pilot plant. The horsepower measurements in this system will permit accurate scaleup of mixer and tank.

C. Tests on Naphtha Scrubbing of Recycle Hydrogen

A bench-scale gas scrubbing system was set up and operated on gas streams from the plant DEA scrubber inlet and outlet. Two naphtha stocks were used for scrubbing, the plant light oil from SRC-II tests of August, 1975, and light oil with 40% wash solvent. Their molecular weights were 106 and 130 respectively. The absorber could be operated as a single stage or using 60 inches of 1/4 inch porcelain Berl saddles in a 0.742" I.D. column. The increase in hydrogen purity ranged from 8.2% with the single stage absorber and heavy naphtha to 16.4% with the multistage column and light naphtha. Hydrogen recovery ranged from 91.3-to-95.9% of that charged. The gas liquid ratio was varied from 7.4-to-11.8 SCF/gal. of circulating naphtha. The operating pressure was 980-1025 psig and temperature was 56-68°F. K-values were estimated for the single-stage runs as tabulated in Table 14.

From the data generated, specifications were written for the packed naphtha absorber installed in the plant for SRC-II operations.

Table 14

K-Values Estimated for Single Absorption Runs
Naphtha Absorption of Recycle Condensate Separator Gas

Run No.	4-9-76-A	4-9-76-B	5-10-76	5-11-76
Naphtha Mol. Wt.	106	106	106	130
Temperature: ^{o}F.	65	63	56	63
Pressure: PSIG	1025	1025	980	980
K-Values $^{(a)}$				
H_2	37.0	36.0	40.0	70.5
N_2	17.6	17.6	16.5	26.1
C_1	5.38	5.43	7.1	14.7
CO_2	11.0	7.83	1.6	3.1
C_3	0.12	0.11	0.12	0.36
n-C_4	0.32	0.18	---	0.38
H_2S	0.15	0.29	0.10	0.21

(a) $K = \frac{\text{Mole Fraction in Product Gas}}{\text{Mole Fraction in Rich Oil}}$

D. Hydroclone Tests

Equipment and instruments were installed to permit evaluation of several small hydroclones on a slip-stream filter feed. Feed rate and overflow rate were metered by chrome-plated eccentric orifice plates which were calibrated each day of operation. The overflow-underflow split was controlled by throttling the underflow with a differential pressure cell and a 1/4 inch ball valve with a proportioning actuator. Two types of hydroclones were tested: the first was a standard Doxie-5 made by Dorr-Oliver, Inc. It was a stainless-steel forging, into which are bored six clone tubes, each 10 millimeters in diameter, with appropriate inlet, bottom and vortex fittings. The second was a hydroclone on loan from the Cresap CSF pilot plant and reported in "Pilot Scale Development of the CSF Process" July 1, 1968-December 31, 1970, R & D Report No. 39, Vol. IV, Book 3. It had a 2-inch barrel and replaceable nozzles made of porcelain. The nozzles used were: feed 0.404" I.D., overflow 0.250" I.D., and underflow 0.161" I.D.

The best data for each of the two hydroclones are summarized in Table 15. The ash particles in the feed were approximately 50 wt.% smaller than 3 microns by electron microscope analysis. The calculated efficiency for either clone was much poorer than was actually obtained. By inference, agglomeration was significant and was apparently greater (or agglomerates were broken up less) in the larger clone since it was nearly as efficient as the small diameter clones.

Table 15

Summarized Hydroclone Data

Hydroclone Used	Cresap 2"		Doxie - 5		
Temperature, °F	365	365	401	400	414
Feed Viscosity, Cp*	0.93	0.93	0.76	--	0.86
Inlet Pressure, Psig.	129	128	117	118	128
Overflow Pressure, Psig	48	48	69	69	80
Underflow Pressure, Psig	--	--	69	78.5	103
Feed Rate, GPM	4.6	4.6	5.16	5.16	4.67
Overflow, Wt.% of Feed	50.8	73.3	49.8	73.9	85.4
Underflow, Wt.% of Feed	49.2	26.7	50.2	26.1	14.6
Solids Distribution, Wt.% of Feed Solids:					
To Overflow	30.4	32.2	44.2	51.7	41.4
To Underflow	79.7	51.0	60.2	46.7	43.3
Unaccountable	-10.1	16.8	-4.4	1.6	15.3
Feeds Solids Content, Wt.%	6.0	6.5	7.0	7.0	6.7

* Viscosity of feed measured by Dynatrol Type CL-10DVT-12 (Range 1-100Cp) made by Automation Products, Inc.

There was no further work done on the hydroclones after these tests. It was decided that no combination of stages would provide SRC solid product with less than 0.16 wt.% ash, and that a dry enough cake could not be obtained.

E. Mineral Residue Drying Tests

Alternate methods of drying wet filter cake were reviewed when the plant dryer was having initial and seemingly insurmountable operating problems. Most alternatives could not be tested readily or at reasonable cost. The Strong-Scott Manufacturing Company made available a laboratory size (12" I.D. x 36" long) Torus Disc dryer for experimentation. This dryer is a trough-shaped vessel with a rotor inside and a tight cover (in the model tested). The rotor has bearings and seals at each end of the trough and eight hollow discs, each with plows to mix and transport solids. Dowtherm was circulated through the hollow shaft, through the hollow discs and then through the shell jacket. The Dowtherm was temperature controlled to provide the heat to vaporize solvent from the wet solids. Drying (sweep) inert gas was passed upward in the nine spaces between each disc and the ends.

Initial tests with wet cake from the plant filters indicated that mud formation quickly plugged the dryer. Experiments showed that when the solvent content of the feed was lowered from 60 wt.% to 15-25% by back-mixing with dried solids, the Torus Disc dryer was operable.

Several runs were made with pre-mixed feed pellets of about 20% solvent content. Approximately 90-95% of the solvent in the wet cake boiled below 500°F., with the remainder in the 500-800°F. boiling point range. Table 16 summarizes the operating conditions for the best of these runs:

Table 16

Operating Conditions for Torus Disc
Drying Tests of Simulated Backmixed Feed

Feed Temperature, °F	60
Total Feed Rate, lb./hr.	320
Inert Gas Rate, SCFH	300
Inlet Dowtherm, °F	525
Drier Product Outlet, °F	475
Feed Solvent Content, Wt.%	22.3
Rotor Speed, RPM	24
Dried Product, Wt.% Solvent	4
Calculated U, BUT/hr.-ft.^{2}-°F.	32

The solvent in the dried product was essentially constant over a range of feed rates and the drier temperature was limited to prevent coke formation. A drier product and/or higher feed rates may have been attained had there been no heavy (process) solvent in the feed. Most runs were terminated because of plugging in the vapor line.

Some of the unresolved operating problems at the end of the test, when the equipment had to be returned to the supplier, were:

- Excessive dust carryover. Recovery equipment will be necessary and difficult to operate.
- The feed must be mixed with dry product to overcome the sticking problem. Due to the pyrophoricity of dried mineral residue, the mixing must be conducted in an essentially oxygen-free environment.
- Solids transport was unsatisfactory or at least limiting in the model tested.

Design modifications are necessary to prevent solids accumulation in the feed end since at higher rates the vapor outlet line was covered with solids.

The advantages of the dryer tested are:

- Improved heat transfer (assuming no fouling of the scraped surfaces).
- Better temperature control and greater heat economy.
- No local overheating or solvent degradation.
- Easier maintenance since only small rotary seals are needed compared to those on a rotary drum dryer.

The manufacturer advised and consulted during these tests.

F. Experimental SRC Product Solidification

Experimental studies of SRC cooling and solidification indicate that the SRC properties dominate this step except when small-sized pieces are made. A demonstration test was performed several years ago on a pilot-plant prilling tower with technical success (samples were prepared). Recent tests on a French proprietary (Perlomatic) pilot plant failed. The product made strings which stuck to the apparatus instead of making pellets.

Tests at the Ft. Lewis pilot plant consisted of pours of 300°F. melt point SRC into molds of various sizes. Three of these were instrumented with thermocouples connected to a multipoint recorder.

The sizes were - 12 x 12 x 1.25 inch, 9 x 9 x 0.5 feet, and 5 feet diameter x 4 feet high.

Estimates of values were fairly consistent for the tests for thermal diffusivity (0.0065 ft.^{2}/hr.) and thermal conductivity (0.1 BTU/hr.-ft.^{2}-°F.) in the 400-500°F. range using the Gurney-Lyrie solution for transient cooling. Later measurements were made for Cp in the temperature range of 300-700°F. Heat capacity followed the equation $Cp = 0.4105 + 2.100 \times 10^{-4} T$, where T is in degrees F and Cp units are BTU/lb.-°F.

Hydrocarbon fog was emitted especially during the larger pours. Appropriate collection devices are required to control this fog.

Drop tests were made on approximately one-inch pieces of SRC cast by various procedures to attempt to quantify friability. The test was similar to ASTM D 440-49 "Drop Shatter Test for Coal." The test results showed that the thicker castings (with slower cooling) broke markedly less than thin rapidly-cooled pieces.

Later operations in the plant produced two rail-carloads of thick castings, utilizing a 12 ft. x 12 ft. x 14 in. casting pit.

VII. PLANT ADDITIONS

As the startup and operation of the Pilot Plant progressed, several miscellaneous additions were required in order to improve plant operation, safety, health protection and efficiency of personnel. In addition, modifications were completed to allow a thorough investigation of the SRC-II mode of operation.

A. Miscellaneous

Miscellaneous additions included the installation of firewater booster pumps; emergency electrical generators; adequate change house facilities; several temporary-type office, storage and maintenance buildings; an improved laboratory ventilation system; dust recovery equipment; a liquid carbon dioxide system for blanketing gas in the coal storage silos and an expanded computer system.

B. SRC-II Modifications

In order to provide the capability of operation in the SRC-II mode over a fairly wide range of conditions, the following additions and modifications were completed. A more detailed discussion of each is given in paragraph III.C.5.

1. Slurry Preparation - Area 01

Installed an additional slurry recirculation pump and an agitated coal slurry mixing tank with vent condenser for operation at temperatures up to 450°F.

2. Dissolver - Area 02

Provided hydrogen quench connections inside the dissolver for temperature control, modified the stripper internals and installed a new oil-water separator.

Installed a stripper column to strip out the light oil and wash solvent. Part of the bottoms of the stripper is recycled, through a cooler, for slurrying of coal and the remainder flows to the solvent recovery area.

3. Solvent Recovery - Area 04

Installed a new, smaller vacuum flash drum with a Dowtherm heated feed preheater and associated piping. The smaller drum was installed so that data can be obtained at vapor velocities anticipated for commercial design when operating in the SRC-II mode. This same vessel will be used as an atmospheric flash when operating in the SRC-I mode. As a result, the light ends column, wash solvent column and the original vacuum flash systems have been returned to the flow scheme provided in the original plant design. It had been necessary to convert the light ends column to an atmospheric flash in order to prevent recurring coking of the vacuum flash preheater.

Modified the recycle process water tank to provide improved oil-water separation and installed a higher capacity recycle water pump.

4. Gas Treating and Compression - Area 05

Modified the fresh hydrogen compressors to increase their capacity by about 20%.

Installed a naphtha scrubbing system to remove hydrocarbon impurities from the recycle hydrogen stream so less would be purged and more would be available for recycle.

Installed a larger recycle hydrogen compressor.

VIII. INDUSTRIAL HYGIENE AND ENVIRONMENTAL PROGRAMS

There are six industrial hygiene and environmental programs at or associated with the Ft. Lewis SRC Pilot Plant: worker medical, worker industrial hygiene, in-plant monitoring, surrounding area monitoring, laboratory studies with test animals and trace element distributions. Each of these programs is discussed below.

A. Worker Medical Program

In order to establish a base line and determine physical fitness for SRC employment, a pre-employment examination is given to each new employee. This pre-employment physical consists of a medical history, audiometric test, visual examination, complete blood count, SMA 16, urinalysis, PA and lateral chest X-Ray, pulmonary function test, and examination by a doctor with emphasis on noting any skin abnormalities. This same examination is given each year to any employee whose job requires work in the process area.

Employee occupationally connected non-serious medical needs are attended to by one of the two plant nurses. Any medical problem requiring the services of a physician are referred to one of the many local physicians, depending on the nature of the need. The majority of plant personnel are first-aid certified. This proves to be an asset in emergencies when the plant nurses are not available, such as on night shifts, weekends and holidays. The Plant Supervisor on shift is responsible for first-aid treatment and medical referral during these times.

B. Worker Industrial Hygiene Program

All new employees are given an Audio/Visual orientation, which emphasizes what is being done to protect the employees from potentially dangerous substances and what the employee, in turn, must do to protect himself. After the Audio/Visual presentation, they are given the SRC Health Protection manual to read.

Each process area employee is issued rubber boots, leather high-top safety shoes, two weeks' supply of work uniforms and an equal supply of underclothing and socks, a hard hat, work coats, skin emollient, barrier creams, is assigned a locker in the locker room (clean area) and a stall in the change house (dirty area).

In order to protect the worker and to prevent carrying of coal derived contaminants off of the plant site, special procedures have been developed for process area workers. Figure 27 is a sketch of shower and change facilities and the paths followed when going to and coming from work.

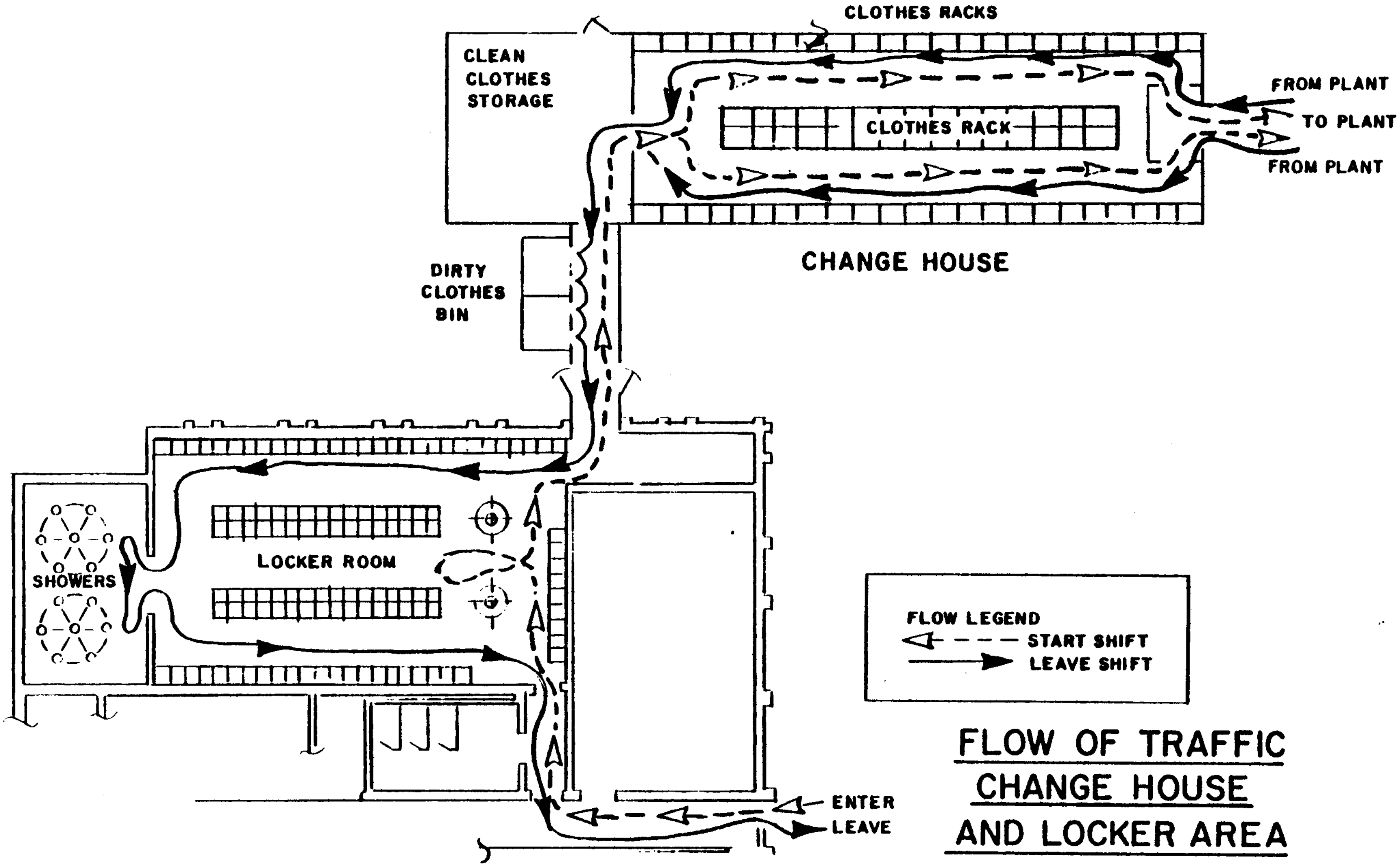
Upon coming to work, they go directly to the locker room and change into a clean uniform which includes underwear, shirt, pants and socks. They then proceed to the change house in shower shoes. In the change house, the employees don work footwear, headgear and rain suit if needed. They then proceed to work, and the reverse procedure is applied at the end of their work shift. On the way to the shower, dirty underclothing is left in a mesh bag in the change house (dirty-area) and the dirty uniform is dropped off in a dirty-clothes bin. The soiled clothing is laundered by a commercial laundry and cleaner. The employees are encouraged to thoroughly wash all coal tar from their body. Skin examinations with a U.V. light are periodically given by the plant nurse to determine how well the employees are cleaning themselves. Places characteristically missed in showering are facial folds, under the chin, behind the ears, scalp, under the finger nails and under jewelry. The majority of employees wish to take a post-shift shower, however, there are a dissident few that prefer to leave the plant area without showering, thus making necessary special supervision to ensure compliance with the program.

Those using the supplied skin barriers find it easier to wash the coal tar off. Any employee experiencing a spill of coal solution solvents or chemicals during a shift is encouraged to shower and change clothes as soon as possible.

All shower room necessities, clothes change and protective gear are provided without expense to the employees.

Employees entering vessels or undertaking exceptionally dirty jobs are encouraged to wear rain suits or coveralls over their uniforms and to wear appropriate gloves. Impervious gloves providing good grip and wrist protection are a favorite with the employees. Neoprene appears to hold up to the attacks of solvent better than other materials.

To date, there has been no diagnosed cases of occupationally connected skin cancer cases. However, there have been multiple cases of solvent spills on employees that have resulted in



FLOW OF TRAFFIC CHANGE HOUSE AND LOCKER AREA

FIGURE 27

contact dermatitis, the severity being directly proportional to the length of time the solvent remains in contact with the skin prior to washing.

C. In-Plant Monitoring Program

1. Summary

Many chemicals and products derived from coal are potentially hazardous. Protection of workers against occupational disease is of utmost concern to the plant management. The major objectives of the in-plant monitoring program is to evaluate the chemical and physical hazards to workers, thus helping to prevent and control job-related diseases; and to assist in the assessment of environmental impacts of plant operation. Information obtained from this monitoring program can be extrapolated and beneficially utilized in the design and operation of future demonstration and commercial-sized SRC facilities.

During 1976, the first year of this three-year comprehensive in-plant industrial hygiene monitoring program, major efforts were spent on designing the program, preparations both in the field and in the laboratory to set the air samplings in motion, training of a field technician and a laboratory technician, selection and purchase of field sampling equipment and laboratory analytical instruments, intensive data gathering and comprehensive data review.

Brief descriptions of the major studies accomplished during 1976 are given below.

- Noise Survey - conducted plant noise survey, occupational noise exposure study and environmental noise impact survey. Results indicate that the SRC Pilot Plant is a rather quiet plant with a minimum occupational noise overexposure problem and minimum noise impact on the surrounding community.

- Airborne Organic Vapors - 90 organic vapor samples were collected and results indicate that in-plant concentrations of organic vapors and hydrocarbon gases were essentially less than 0.1 ppm. Calculations show that the total hydrocarbon emission from SRC Pilot Plant operations is small, and its impact on community air quality is insignificant.

- Asbestos Survey - the only known source of asbestos is the filter aid material "Fibra-Flo." Results of several surveys show that occupational exposures to asbestos have been brought under control by the installation of an asbestos handling glove box and by stringent work practices and respiratory protection. Later, air sampling results showed less than 0.1 fiber per ml asbestos concentration.

- Free Silica and Mineral Residue Dust - several types of calcined diatomaceous earth containing 50% by weight of free silica were used as filter aid in the filter preparation building. Dust concentrations as high as 10 mg/m^3 have been monitored. The situation is controlled by a mandatory respirator program. The mineral residue contains an average of 4.5% free silica as α-quartz. Because of the large quantities of mineral residue to be handled, a severe occupational free silica exposure problem would be expected for commercial SRC facilities unless proper engineering precautions are taken.

- Hydrogen Sulfide Study - potential H_2S release sources in the Pilot Plant were identified. Thirty-four H_2S samples showed H_2S concentration mostly below 0.1 ppm, indicating an insignificant chronic H_2S exposure problem. H_2S should not present a chronic occupational exposure problem for future commercial-sized SRC facilities. However, potentially hazardous exposure can occur during process or equipment malfunctions.

- Sulfur Dioxide Study - potential SO_2 release sources in the Pilot Plant have been identified. Forty-two SO_2 samples collected showed less than 0.1 ppm SO_2, indicating no occupational exposure problem. It is predicted that SO_2 will not present a serious threat to the health and safety of plant workers in future commercial-sized SRC operations.

- Phenols Study - phenolic compounds exist in large quantities in essentially all liquid streams. However 61 samples showed virtually no phenols in the ambient air. It is concluded that phenolic compounds will present serious occupational skin contact hazards in SRC processing, but present no inhalation hazard.

- Carbon Monoxide Study - results of plant CO survey show that the major CO hazard in the SRC Pilot Plant was related to the use of plant inert gas which contained 1.5% CO. For a full-sized SRC facility, CO should be much less in the inert gas because of better combustion control and better inert gas generation equipment. In all probability, the nitrogen stream from the air separation plant, provided to produce oxygen for the gasifier used for hydrogen production, would be used for inerting.

Following are descriptions of industrial hygiene monitoring studies still in progress:

- Suspended Particulates - over one hundred suspended particulate samples have been collected with high volume air samplers and personal air sampling pumps. They will be analyzed for mass, benzene soluble fraction and specific PNAH.
- Settleable Particulates - monthly samples of settleable particulate samples have been collected. They will be analyzed for mass, benzene soluble fraction, PNAH and trace metals.
- Welding Fumes Study - this is not a study of metal fumes from welding operations, but a study of welder's exposure to coal tar and liquids when cutting or welding contaminated parts.
- Trace Metal Analysis - airborne particulate materials have been collected from area O1 (coal dust), 03 (mineral residue dust) and 08 (SRC product dust). These samples will be analyzed for trace metals by Washington State University.

2. Noise Surveys

a. Plant Noise Survey

Several plant noise surveys were conducted with a calibrated sound level meter (Scott Model 452 and GR Model 1565A). The major noise areas were identified as follows:

(1) Coal receiving and preparation: The major noise producing equipment are the pulverizer (90-to-95 dBA), preheater charge pump (95-to-100 dBA), gravimetric feeder, vibrator on the roof, and steam release noise.

(2) Wastewater treatment area: The major noise sources are the pumps by the holding tank and balancing tank, and especially the pump by the bio-unit, which produced up to 100 dBA at 3 feet distance. It has since been shielded.

(3) Dowtherm heater: Noise was 90-to-95 dBA under the Dowtherm heater and 90-to-100 dBA around the Dowtherm pump.

(4) Hydrogen generation unit: Noise was 95-to-100 dBA at the center of the Stretford unit and 90-to-95 dBA at the center of the hydrogen unit.

(5) Mineral separation area: Major noise sources are several pumps, steam releases, and the "knocker" on the mineral residue dryer. A large portion of the mineral separation area is in excess of 90 dBA.

(6) Solvent recovery area: Most noises are produced by the many steam release valves and pumps. A good portion of the first floor solvent recovery area is in excess of 90 dBA.

(7) Product solidification area: Noise levels exceed 90 dBA in the northern half of the Sandvik belt building. This is definitely one area that workers could be over-exposed to noise.

(8) Boiler house: Noise inside the boiler house ranges from 85-to-95 dBA.

In general, the Solvent Refined Coal Pilot Plant is judged to be a relatively quiet plant.

b. Occupational Noise Exposure Study

The measurement of employee noise exposures was facilitated by a noise dosimeter survey. A noise dosimeter is a device worn by a worker and automatically takes into account the fluctuation and time variations of worker noise exposure and integrates the intensity of measured sound with the duration of exposure. The resulting dosage, expressed as a percent of the OSHA permissible 8-hour dosage, is measured by a separate readout device. Noise dosimetry surveys at the SRC Pilot Plant were conducted with both DuPont audio dosimeters and a Bendix audio dosimeter.

There were 36 man-days surveyed during the period from May to November, 1976. The survey indicated that thirty-two persons received noise exposures less than 50% of the OSHA permissible value. Three persons received exposures between 50% and 100% of the permissible limit, and one person received noise exposure in excess of 100%.

In accordance with company practise, all of those employees exposed to noise levels in excess of 50% of the OSHA permissible limit will receive audiometry testing. High noise areas within the SRC Pilot Plant are marked by warning signs and wearing of hearing protective devices (ear muff or ear plugs) are required in these areas.

c. Noise Prediction for Demonstration and Commercial-Sized SRC Facilities

Without full knowledge of the process equipment selection and layout, it is difficult to quantitatively predict what will happen to the plant noise levels when the 50 ton-per-day pilot plant is scaled up to a demonstration plant or commercial-sized facility. Most likely, the noise levels will be increased due to the larger size of pumps, compressors, burners, heaters, fans and pulverizers to be used. Noise from steam releases will be even more severe. Piping and valves, which present no problem now, may become a significant factor in plant noise. The product solidification process using a Sandvik belt presents a moderate occupational noise exposure problem at the Pilot Plant. This noise problem may become intolerable when the same equipment and process is scaled-up, unless proper noise suppression is provided. It is recommended that for future SRC demonstration and commercial-sized facilities, the design engineer should consult a noise control acoustic engineering expert with regard to the proper selection, specification and layout of process equipment and with regard to noise abatement techniques and control devices.

d. Environmental Noise Survey

The SRC Pilot Plant is located in a forested area of North Ft. Lewis. There is no neighboring residential property, Results of this study showed workers at the Pilot Plant were exposed to very small amounts of organic gases and solvent vapors. The concentrations and exposure durations were such that occupational hazards from inhalation of airborne organic vapors were minimal.

d. Community Air Pollution Impact from Hydrocarbon Releases

A certain quantity of hydrocarbons is released to the atmosphere due to operation of the Pilot Plant. Although no direct ambient air sampling for hydrocarbon gases was conducted in the communities around the Pilot Plant, large quantity of in-plant data of airborne hydrocarbon gases and organic solvent vapors have been acquired. Through reasonable data extrapolation, an estimation of the impact on community air quality from this hydrocarbon emission source can be derived.

In-plant air sampling results showed that workplace hydrocarbon concentrations were, in general, less than 0.1 ppm. If the in-plant hydrocarbon concentration is less than 0.1 ppm, it is reasonable to assume that the concentration beyond the plant fence-line should be at least ten times less than the in-plant levels, due to the effect or normal air dilution and dispersion.

The most significant point hydrocarbon emission source inside the Pilot Plant is the surge reservoir, which collects all of the plant process wastewaters (containing oil, solvents, coal liquids, etc.) prior to their being fed to the biological waste treatment unit. The reservoir is ten feet deep, sixty feet in diameter and has a holding capacity of 216,000 gallons of wastewater.

A calculation of the rate of hydrocarbon emission from the surge reservoir, based on the average hydrocarbon concentration of 5.6 ppm measured during five sampling occasions and on an estimated upward air movement velocity of 5 feet per minute, shows that the rate of total hydrocarbon emission from the surge reservoir is 31 pounds per day.

A figure released from a U.S. Public Health Service book on air pollution, "Rapid Survey Technique for Estimating Community Air Pollution Emissions," U.S. Public Health Service Publication No. 999-AP-29, October, 1966, stated nor is there any commercial activities in the immediate neighborhood. Therefore, environmental and community noise complaints are unlikely to occur. The following environmental noise survey was conducted mainly for the sake of record and for the reference of site selection for future SRC facilities.

Noise level readings were taken at the fenceline around the SRC Pilot Plant on October 26, 1976, and again on February 16, 1977. The survey results indicate that, in general, the fenceline noise levels varied between lower 60's to mid 70's. Noise levels were slightly higher when the plant was in operation as compared to shutdown time. A few noise level readings taken at locations far away from the plant process area (i.e., outside the plant noise influence zone) showed the average ambient background noise was 56 dBA, mainly due to traffic noise from the interstate freeway.

3. Airborne Organic Vapors Surveys Completed

a. Air Sampling Technique

The sampling of airborne hydrocarbon gases and organic solvent vapors was facilitated by drawing air through glass tubes packed with 20/40 mesh activated coconut shell charcoal at a flow rate of approximately 0.2 liters per minute. Both personal air samples and area air samples were collected from various locations in the Pilot Plant.

b. Analysis of Airborne Organic Vapors

The analytical procedure used was essentially that designated by NIOSH P and CAM 127, except that a longer column was utilized which provided better resolution of the hydrocarbon species of interest.

c. Results and Discussions

Approximately 90 air samples were collected during the period from March through October 1976. The data showed that, in general, the in-plant hydrocarbon concentrations were less than 0.1 ppm (as benzene). A few higher concentrations were collected during maintenance time when vessels or tanks were opened for repair, or during unusual plant operating conditions.

that 524 pounds of hydrocarbons are emitted to the atmosphere when 1,000 gallons of gasoline are consumed by an average automobile. The emission of 31 pounds of hydrocarbons per day from the surge reservoir is, therefore, equivalent to the consumption of 59 gallons of gasoline by an automobile. To express the impact in another manner, the daily hydrocarbon emission from the reservoir is less than the hydrocarbon emissions from driving one standard automobile 1,000 miles.

In summary, it is concluded that the total hydrocarbon emission from the Pilot Plant operation is small, and its impact on community air quality is insignificant.

4. Asbestos Survey Completed

Airborne samples are collected adjacent to the breathing zone on 37-mm cellulose ester filters with calibrated personal sampling pumps using the procedure recommended by NIOSH criteria document on Occupational Exposure to Asbestos.

The only known source of asbestos in the Pilot Plant is from the base coat material, which contains approximately 4%-to-7% of asbestos fiber. Handling of asbestos material is confined within the filter preparation building and is a batch operation.

In the beginning, the base coat material was handled in the open. Air sampling results showed employees were sometimes exposed to asbestos concentrations in excess of the OSHA permissible standard. A special glove-box was designed and base coat then was handled and mixed strictly within this box. Very stringent control and work practices now are followed when handling asbestos. All employees are required to wear suitable respirators when working with asbestos.

Results of asbestos surveys show that high asbestos concentrations were encountered when the asbestos isolation glove box was not employed. When the box was used and the stringent material handling practices followed, the asbestos concentrations encountered during asbestos mixing work were very low, almost equivalent to the asbestos concentrations found in the ambient outdoor clean air samples.

Occupational asbestos exposure has been brought under control at the SRC Pilot Plant through material isolation, work practices and respiratory protection devices.

Recently, the National Institute for Occupational Safety and Health (NIOSH) has proposed an asbestos exposure guideline which required workers' time-weighted average asbestos exposure not to exceed 0.1 fiber per ml. of air.

5. Free Silica and Mineral Residue Dust Surveys Completed

Free silica exposure at the SRC Pilot Plant can be originated from two major material sources. They are the free silica contents in the coal feed stock and the free silica contents in the diatomaceous earth filter aid. The percentage of free silica in the coal varies with the origin of the coal. Kentucky coal contains roughly 2 percent free silica. Occupational exposures to coal dusts at the Pilot Plant occur mainly in and around the coal receiving and preparation building. A considerable cloud of coal dust is produced during dumping of coal from railroad cars to the receiving hopper. But the duration of dust exposure is short and respiratory protective dust masks are worn by workers during this rather infrequent operation.

Crude diatomaceous earth is essentially amorphous silica and contains less than 5 percent of quartz and only traces of cristobalite and tridymite. However, after being processed by high-temperature calcining, the cristobalite content may be as high as 60%. The filter aid materials used at the Pilot Plant are all calcined diatomaceous earth. According to the manufacturer, all contain approximately 50% of crystalline silica in the form of cristobalite.

Occupational exposures to calcined diatomaceous earth occur mainly inside the filter preparation building. Batch handling and mixing of filter aid materials last less than two hours and occur at a frequency of once every day or two. During this short duration, total dust concentration inside the filter preparation building can be as high as 10 mg/m^3. However, all workers are required to wear dust masks during filter aid preparation.

Free silica contents from both the coal feed stock and the filter aid materials all end up in the mineral residue. The mineral residue contains essentially no asbestos fiber, and an average of 4.5% free silica as α-quartz.

At the Pilot Plant, the mineral residue dusts are discharged from the mineral residue dryer through a chute and slide gate into a steel drum lined inside with a plastic bag. The filled drums are sealed and hauled away from the Pilot Plant and disposed of by a chemical wastes disposal contractor. A minor dusty situation exists during filling of the drums, but occupational exposure to free silica contents of mineral residue is not severe, and the problem is controlled through wearing of a respirator. Proper engineering of the process equipment and ventilation design are essential in controlling the free silica exposure problem in future large plants.

6. Hydrogen Sulfide Study Completed

Hydrogen sulfide occurs in many gas streams in the SRC process. Some of the places where H_2S can be released to the atmosphere are the sulfur recovery plant, the gas recovery and recompression area, the solvent recovery area, the mineral separation area and places where plant sour wastewater is present. Since H_2S has a very low odor threshold value (detectable by most people at 0.005 ppm), minor fugitive leaks from valves, flanges, vessels and compressor seals may be accountable for the rotton egg odor often present at several process units.

Thirty-four (34) air samples were collected from various plant locations for H_2S analysis. The sense of smell was used as a guide for selecting the sampling spots. The majority of the air samples showed very minimal or non-detectable H_2S concentrations. The few samples that showed slightly elevated H_2S concentrations can all be traced to definite sources of H_2S release. For example, two samples collected near the recirculating process water pump showed 0.37 and 0.26 ppm H_2S respectively, indicating H_2S leakage from the pump which handles sour water.

The threshold limit value for H_2S is 10 ppm. Results of this air sampling study indicate that, under normal plant operating conditions, the H_2S concentrations in all workplaces were well within the occupational exposure standard. It can be concluded that workers' chronical exposure to H_2S were insignificant in the Pilot Plant.

This, however, does not preclude the occurrences of injuries caused by H2S through acute intoxication in extraordinary events. In fact, prevention of acute H_2S poisoning cannot be over-emphasized in the SRC process. Every person involved in the operation should be informed periodically of the characteristics of H_2S gas, its dangers, and the safe procedures to be used when it is encountered.

7. Sulfur Dioxide Study Completed

Forty-two (42) sulfur dioxide air samples were collected from various areas in the Pilot Plant. Except for two samples, they all showed concentrations of 0.04 ppm or less. The two high samples were collected from the preheater and dissolver area while maintenance workers were fixing a flange on the intermediate pressure separator. Apparently the gas leaking from the flange contained small quantities of SO_2 and this resulted in the detection of the relatively elevated concentrations of 0.38 ppm and 0.50 ppm. However, since the threshold limit value for SO_2 is 5 ppm, it is quite evident these two readings are well within the allowable occupational exposure limit.

The chance of toxic exposure to SO_2 at the SRC Pilot Plant is extremely remote. Based upon the above SO_2 air sampling study results, it is predicted that SO_2 will not present a serious threat to the health and safety of plant workers in future commercial-sized SRC operations.

8. Phenols Study Completed

Phenol and phenolic compounds are present in many hydrocarbon and aqueous streams in the SRC process. For example, an SRC light oil contains about 17% by weight of phenols, and the phenol content in the combined plant wastewater stream can reach 1,000 ppm level before biological treatment.

Toxic exposure to phenol and cresols at the Pilot Plant has been limited to accidental skin contact. In April 1975, a worker suffered a case of skin burn of the hand (redness, vesiculation and ulceration of the skin) when he was moving a barrel containing coal derived solvent and some spilled on his hand. Subsequent analysis of the liquid showed it contained 6.7% phenol, 4.4% o-cresol, 13.4% m-cresol, and 3.5% p-cresol.

Vapors of phenolic compounds can be readily absorbed into the pulmonary circulation. The current threshold limits for phenol and cresols are both 5 ppm. The purpose of this phenols study was to determine whether vapors of phenolic compounds were present in significant quantities to impair the health of SRC process workers. A total of 61 air samples were collected for phenol analysis. All air samples showed less than 0.04 ppm of phenol and less than 0.01 ppm of the three types of cresols. Xylenols, ethylphenols and other high phenols were not detected. It is apparent that vapors of phenolic compounds are not present in significant quantities in the plant air to cause any adverse health effect. Inhalation hazards of phenols can be virtually neglected, but it is imperative that the prevention of skin contact with phenols be greatly emphasized.

Based on the results of this air sampling study and barring drastic changes in the SRC process, one should be able to predict that phenolic compounds should present no inhalation hazard in future commercial-size SRC facility.

9. Carbon Monoxide Surveys Completed

Carbon monoxide is present in various gaseous streams in the SRC process. It is normally confined in vessels and pipelines, but leaks from flanges, valves and compressor seals could result in CO release to the workplace atmosphere. A major source of CO is the plant inert gas system which supplies dry inert gas to various parts of the plant, including the dry pulverizer. Ideally, the inert gas contains 88% nitrogen with the remainder CO_2. However, up to 1.5% (15,000 ppm) of CO can be present in the inert gas due to incomplete combustion at the inert gas generator.

CO is a highly dangerous gas due to its lack of odor, taste and other warning properties. CO exerts its harmful effect by reducing the oxygen-carrying capacity of the blood. OSHA standard for CO exposure is 50 ppm. Lengthy exposure at 500-to-1,000 ppm CO can cause dizziness and coma, and death can result in a few minutes' exposure at 10,000 ppm.

A plant CO survey was carried out in May and June 1976 with Drager brand CO chemical detector tubes (models certified by NIOSH). The lower measuring range for the Drager CH25601 tube is 5 ppm CO, which is more than adequate to search for and identify locations of potential CO hazards. One minor and one major trouble spots were identified by the CO survey, and the high CO concentrations at both locations were caused by the release of plant inert gas. Equipment and operating procedures were modified to minimize the potential hazard.

10. Suspended Particulates Study in Progress

a. Sampling and Analytical Methods

Airborne suspended particulates were collected using high volume sampler and 8" x 10" fiberglass filter for area monitoring and 37 mm silver membrane filter in field monitor cassette for personal monitoring. The collected samples were analyzed for the following parameters:

(1) Total Mass Concentration: Based on gravimetric weighing of the filter before and after particulates collection.

(2) Benzene Soluble Fraction: The particulate material on the filters is extracted by benzene, using the continuous cycling process of a Soxhlet extractor. After extraction, the solution is filtered through the original filters. The weight loss of the filters is considered to be the amount of benzene soluble fraction. Detection limit of this method is 0.1 mg of benzene soluble material.

(3) Polynuclear Aromatics (PNA): The PNA compounds are extracted into the benzene soluble fraction. After the addition of an internal standard, the extract is evaporated to approximately 30μl and a portion is injected into a gas chromatograph. The PNA compounds and the internal standard are trapped as they elute from the chromatograph. The ultraviolet absorption spectra of the trapped compounds provide a quantitative measurement. This is method PSM-1016.

(4) Benzo (a) Pyrene and Other Specific PNA. Selected samples are further analyzed by thin layer chromatography (TLC) and fluorometric determination per PSM-1017.

b. Preliminary Results

High volume samples were analyzed for total mass concentration, benzene soluble fraction and PNA. Table 17 summarizes results of a set of high-volume suspended particulates samples collected during May 1976. Because the quantity of particulates caught on the filter was usually small for personal silver membrane filter samples,

Table 17

Analyses of Airborne Suspended Particulates

Area Hi-Vol Samples, May 6-27, 1976

ND: not detected
TR: trace

FIELD SAMPLE NUMBER	SAMPLE VOLUME m^3	TOTAL PARTICULATE mg m^{-3}	BENZENE SOLUBLE mg m^{-3}	TOTAL PNAH μg m^{-3}	ANTHRCENE μg m^{-3}	FLUORANTHENE μg m^{-3}	PYRENE μg m^{-3}	CHRYSENE μg m^{-3}	BENZ(a) ANTHRACENE μg m^{-3}	BENZ(a) PYRENE μg m^{-3}
76030586	Blank	--	--	--	--	--	--	--	--	--
76030582	260	0.71	0.59	5.4	1.1	0.73	0.92	0.08	0.3	0.3
76030585	Blank	--	--	--	--	--	--	--	--	--
76030580	513	0.052	0.039	0.25	ND	ND	ND	ND	TR	ND
76030578	480	0.090	0.024	ND	ND	ND	ND	ND	ND	ND
76030579	357	0.098	0.051	ND	ND	ND	ND	ND	ND	ND
76030584	Blank	--	--	--	--	--	--	--	--	--
76030581	479	0.060	0.044	0.2	ND	ND	ND	ND	ND	ND
76030570	446	0.032	0.030	0.5	0.02	ND	ND	ND	ND	ND
76030577	Blank	--	--	--	--	--	--	--	--	--
76030591	207	0.27	0.013	ND	ND	ND	ND	ND	ND	ND
76030576	Blank	--	--	--	--	--	--	--	--	--
76030569	319	0.39	0.20	1.3	0.38	0.13	0.28	ND	0.03	ND
76030575	Blank	--	--	--	--	--	--	--	--	--
76030573	189	3.4	0.041	0.5	0.05	ND	ND	ND	ND	ND
76030568	145	3.0	0.42	17.	3.1	2.1	2.7	0.28	0.62	0.97
76030571	1930	0.058	0.0013	ND	ND	ND	ND	ND	ND	ND
76030603	2080	0.010	0.0090	20.	0.07	<0.07	<0.07	ND	ND	ND
76030602	1900	0.17	0.11	1.8	0.49	0.19	0.24	0.02	0.05	0.04
76030605	Blank	--	--	--	--	--	--	--	--	--
76030600	2660	0.010	0.0012	ND	ND	ND	ND	ND	ND	ND
76030606	Blank	--	--	--	--	--	--	--	--	--
76030559	2510	0.049	0.0023	<0.04	ND	ND	ND	ND	ND	ND
76030346	355	0.21	0.15	2.0	0.085	ND	0.42	ND	TR[2]	ND
76030562	496	0.15	0.050	0.40	<0.02	<0.02	<0.02	ND	<0.02	ND
76030565	Blank	--	--	--	--	--	--	--	--	--
76030564	1680	0.016	0.0065	0.06	ND	ND	ND	ND	ND	ND
76030566	Blank	--	--	--	--	--	--	--	--	--
76030561	445	0.55	0.075	0.2	0.13	ND	ND	ND	ND	ND
76030563	198	0.89	0.63	4.	1.1	0.56	0.76	ND	0.20	TR
76030567	Blank	--	--	--	--	--	--	--	--	--
76030560	496	0.48	0.44	2.	0.4	0.2	0.3	ND	0.1	0.1
76030583	225	2.4	1.3	26.	4.	3.1	3.8	ND	1.1	1.2

data obtained was limited to total mass concentration and benzene soluble fraction.

Analytical data so far showed fairly inconsistent and scattered results in terms of the ratio between benzene soluble fraction vs. the total mass concentration for particulates from the same plant areas. However, the laboratory analytical techniques are getting better and more refined, and later data should show better reproducibility.

11. Dustfall Survey in Progress

Large settleable particulate matter, which becomes suspended in the atmosphere by wind forces or mechanical means, and other particulate matter cleansed from the atmosphere by rain or agglomeration, is measured using an open-mouth container exposed for a period of approximately one month. Following collection, the settleable dust samples can be analyzed in many ways to suit the needs of specific situations. The gross measurement of total mass concentration is normally undertaken and expressed as grams of dustfall per square meter of area per month duration. Further physical analysis can be performed on the insoluble fraction of dustfall, e.g. particle size distribution and specific gravity. Chemical analysis can be performed to determine compounds of specific interest.

Six dustfall buckets were utilized in the SRC Pilot Plant in-plant dustfall survey. Monthly dustfall samples have been analyzed for total weight concentration and benzene soluble fraction. The results are rather inconsistent and additional sampling and analytical work are required in order to establish any trend or pattern of dustfall results in relation to the sampling location and plant operating conditions.

12. Welding Fumes Study in Progress

This is not a study of metal fumes from welding operations but a study of welder's exposure to fumes of coal tar and liquids when cutting and welding pipes and vessels contaminated with process materials. Several samples have been collected with silver membrane filters and high volume filters during routine maintenance work. Additional sampling will be conducted in the future, especially during periods of high maintenance or construction activity.

13. Trace Metal Analysis Study in Progress

High-Vol samples of airborne suspended particulates have been collected from Area O1 (mainly raw coal dusts), Area O3 (mainly mineral residue dusts) and Area O8 (mainly SRC product dusts). These samples will be analyzed for trace metals by Washington State University personnel.

14. Skin Contamination Study in Progress

Occupational skin cancer is a potentially major threat in coal conversion industry. Results of animal experiments and past experience from a coal hydrogenation process plant indicated that excessive and prolonged skin contact with coal tar and liquids can cause dermatitis and skin cancer. A skin wipe study is a part of this comprehensive industrial hygiene monitoring program. The study consists of wiping workers' skin (hands and/or face) with a standardized wiping pad moistened with a suitable non-toxic solvent. The used pads will be analyzed for benzene solubles and specific polynuclear aromatic hydrocarbons to obtain indication of coal liquid contamination

A few trial skin wipe tests were conducted on SRC plant workers during the past year. The wipes used were "Alcohol Prep" which is a commercially available sterilization gauze pad soaked in 70% isopropyl alcohol, and individually wrapped in a neat aluminum foil package. Preliminary analytical results showed demonstrable differences in terms of benzene solubles fractions between "contaminated" samples (taken before shower and hand washing) and "clean" samples (taken after shower and hand washing). However, quantitative analysis of polynuclear aromatics was unsuccessful. Several kinds of non-toxic solvents will be tried and the analytical technique will be refined to make this study successful.

D. Surrounding Area Monitoring Program

1. Summary

The purpose of this program is to determine if any changes have occurred in the air, water and foliage environment since the baseline study which was conducted in 1973.

Data accumulated to date indicate no environmental degradation of the surrounding area due to the operation of the SRC Pilot Plant.

2. Air Quality Study

a. Sulfur Dioxide (SO_2) Monitoring

The sulfur dioxide monitoring was performed with a fixed station located along the south edge of the property just south of the product storage area. This area was selected because it is located downwind of the main potential SO_2 producing areas. The field station consisted of an equipment trailer housing wind speed and direction instrumentation and a Meloy SA 185-2 sulfur analyzer. The equipment became operative the latter part of August 1976 and has operated constantly since then, except for occasional equipment failures.

Based on data obtained to date, the maximum ambient value of SO_2 emitted from the plant, as recorded from the station, is .2 ppm over a 1-hour average, with the majority of the values below .05 ppm. The average of all readings taken is below .01 ppm. Table 18 shows these results as compared with the local standards.

Table 18

Sulfur Dioxide Levels vs. Local Standards

	SRC Pilot Plant Station	Puget Sound Air Pollution Control Agency Standard
Highest noted SO_2 levels	.2 ppm 1-hour average	.25 ppm 1-hour average
Average values on north wind days (station downwind of plant)	.05 ppm	.1 ppm 24-hour average
Average per sample period	.01 ppm	.02 annual average

It should be noted that this station is located within the plant boundary and the air pollution standards generally apply to a representative site away from the plant at a reasonable receptor site. Therefore, the possibility of exceeding the local standards at an off-site location is unlikely unless a plant upset condition would occur.

b. Hydrogen Sulfide (H_2S)

Although H_2S is emitted at various vent locations, it appears to be dispersed since zero concentrations were recorded, and the odor of H_2S was never observed at the equipment trailer. During the monitoring period, no detectable levels above .01 ppm H_2S were recorded.

c. Suspended Particulates

An 8-station sampling network was established in which three stations were located near the south edge of the plant, near areas where dust might be generated. Another five stations were located up to one mile away from the plant. Three of these stations were situated at the same locations as the 1973 baseline sampling stations.

An analysis of the data which has been obtained indicates that the levels of particulates at the surrounding area stations are essentially the same as the 1973 baseline results. Stations near the plant showed levels that were higher on certain days than the outlying stations. The main reason for these somewhat higher results is that the three stations are located near a gravel road which runs just south of the plant and sample stations. This road can generate sufficient dust to produce abnormally high values at these stations. An examination of the filters shows a predominately brown, with some black, color which is more representative of road dust than of coal dust.

In summary, the dust loadings close to and at some distance from the facility are essentially the same as those in 1973, and within the Puget Sound Air Pollution Control Agency Regulations. The suspended particulates standard is:

Annual Geometric Mean - 60 micrograms/meter^3

24-hour average - 150 micrograms/meter^3

It is planned to maintain an air quality surveillance program to detect any significant changes.

3. Airborne Hydrocarbons

Several samples were obtained to determine airborne hydrocarbon concentrations. The results of all of these tests show only trace quantities.

4. Heavy Metals

A total of sixteen air and water samples were obtained for determination of heavy metal content. Analyses of these samples are not complete.

5. Foliage Study

The objective of this investigation was to determine whether or not the dominant vegetative cover in the vicinity of the SRC Pilot Plant showed any visible evidence of damage attributable to pollutants originating from the plant.

A preliminary survey of the SRC Pilot Plant vicinity was conducted to determine the identity of dominant vegetative cover types, the location of unusual or potential problem areas and prevailing wind patterns. Following the preliminary survey, four areas of similar vegetation and representative of local conditions were chosen as sampling sites. Three of these sites are within one-half mile of the plant and are leeward of prevailing north-east and south-west winds. The fourth site is approximately one mile west of the plant and not in the path of prevailing winds. This site is considered to be a control location.

Foliage collected from the sample sites during the test program exhibited no symptoms attributable to sulfur dioxide, hydrocarbons or particulate matter injury. Foliage at all sites possessed similar growth characteristics. Injury symptoms attributable to insects or disease appeared uniformly throughout the sample areas.

A visit was made to the U.S. Army greenhouse and garden area near the Pilot Plant. The manager of the facility stated that he had not detected any evidence of damage to the greenhouse plants.

It can be concluded that no visible injury to dominant vegetation in the vicinity of the SRC Pilot Plant occurred due to the operation of the plant.

E. Laboratory Studies with Test Animals

A laboratory animal test program is underway to evaluate the toxicological effect of those products and intermediate streams of the SRC process which are considered to be potentially carcinogenic or teratogenic. The program consists of four parts, each of which is described briefly below.

1. Acute and Subacute Studies

The purpose of this part is to establish the dosage levels to be used in subsequent parts of the program.. This part is not yet complete.

2. Skin Painting

This is a two-year program to study the chronic effect of long-term, repetitive skin exposure.

3. Inhalation Study

This is a two-year study of the chronic effect of long-term, repetitive inhalation.

4. Teratogenic Study

This is about a three-month study to determine the teratogenic (fetus damage) effects, if any.

The toxicity test program using test animals is summarized in Table 19.

F. Trace Metals Distribution Study

A trace metals study is in progress on SRC-I by the Nuclear Radiation Center, Washington State University, under a subcontract. The objective of the study is to provide a complete analysis of the trace elements in the process, from the coal feed through the by-products to the final product. Such data will then allow the calculation of an approximate metals balance throughout the process. The treated effluent water is also being subjected to a similar study in order to provide information on typical trace metal concentrations for environmental considerations. Nuclear radiation and atomic absorption techniques are being utilized to provide data on approximately forty different elements. Preliminary materials balance calculations on two equilibrium sets of samples indicate good accountability with the bulk of the metals appearing to concentrate in the mineral residue. Similar studies will be done for the SRC-II operation and for different coal feeds.

Table 19

Summary of Toxicity Program Using Test Animals

Material	Acute	Acute Inhalation	Subacute Dermal	Carcinogenesis	Chronic Inhalation	Teratogenesis	Analytical
Process Solvent	X	X	X	X	X	X	X
Coal Slurry	X		X	X			X
Filter Feed	X			X		X	X
Dry Mineral Residue	X				X	X	X
Wet Mineral Residue	X			X		X	X
Light Oil	X	X		X		X	X
Wash Solvent	X	X	X	X	X	X	X
SRC Dust	X		X	X	X	X	X
Pulverized Coal	X					X	

IX. PROCESS EVALUATION

A. General Discussion and Concepts

1. Introduction

After successful and reliable plant operation was established and with the development of an extensive data acquisition system, process yield studies of the SRC I process using Western Kentucky coal were successfully completed. These studies defined the primary effects of operating variables on product yields in pilot scale equipment. Conditions were found where a positive yield of recycle solvent is produced, therein confirming the viability of the SRC-I process.

SRC II studies proved the basic operability in this mode and demonstrated the potential of the process to substantially increase liquid product yields. Based on the results of these studies, recommendations were made for process and mechanical modifications prior to additional pilot plant testing.

Numerous special studies have been conducted to advance the state of the art of solvent refined coal processing. Among these are studies of:

- Dissolver thermodynamic characteristics and solids deposits.
- The operation of rotary precoat filters to define the primary and interactive effects of operating parameters.
- The physical handling of solvent refined coal.
- Properties of feedstocks, products and intermediate streams.

2. Evolution of Material Balance Equipment and Techniques

Preparation of mass balance equipment, including plant instrumentation and the data acquisition system, began before initial plant startup and continued throughout the early startup period.

First priority was programming of the plant data acquisition computer system to calculate mass flowrates for all major plant streams. Programs were created which use current gas chromatographic data, temperature, pressure and physical property information to calculate hourly mass flowrates for some fifty-five plant streams. These programs were placed on-line in mid 1974 and formed the basis of subsequent mass balance work.

Material balance checking was initiated during periods in which water was circulated through the plant. These balances revealed many instrumentation malfunctions which were corrected in the field. In addition, some comprehensive problems with mass balance instrumentation were isolated and solved by engineering changes.

During the initial plant operating periods in late 1974, mass balance calculations continued, but results were erratic and inconsistent. A program of revisions to improve this situation was implemented, including tracing of flowmeter impulse lines, revision of transmitter spans and replacement of defective instruments. Level transmitters and flow sensing elements were calibrated and an extensive meter proving program utilizing timed flow into measured vessels was used to further improve measurements accuracy.

The results of the meter proving program indicated that measurement accuracy was poor, due largely to the magnitude of the viscosity corrections required at normal plant flow rates (typically in low Reynolds Number regions). As a result of these findings, on-line computer meter coefficient calculations were modified to include viscosity correction calculations and also to include adjustment factors generated from the meter proving data.

In spite of the many changes which were made to improve mass balance data, calculations made during plant operation in early 1975 revealed that the required instrumentation accuracy still did not exist to determine the effects of process variables with sufficient precision. At first it appeared that this was largely due to the relatively unstable operating conditions which resulted from mechanical difficulties. However, as plant operating stability improved, it became apparent that the combined effects of several factors made total reliance on plant feed and product instrumentation inadequate for accurate on-line mass balance determinations. The major problems encountered were:

- The large capacitance existing in the plant system due to the presence of very large liquid holding capacity in relation to plant throughput.
- Inability to measure several important byproduct streams accurately, notably water and mineral residue yield.
- Inaccuracy of flow measurement caused by inadequate physical property knowledge and large correction factors resulting from low Reynolds number flow regimes.

As a result of these problems, a new approach to predictive yield calculations was undertaken. This method involved selection of mass balance envelopes, using plant laboratory analyses and selected process flowrate measurements to calculate process yields. Study of the possibilities of this new method resulted in the selection of three basic mass balance envelopes, which were labeled Methods 1, 2 and 3. Figures 28, 29 and 30 are pictorial representations of these mass balance envelopes, which are described in more detail below.

METHOD NO.1 MATERIAL BALANCE ENVELOPE

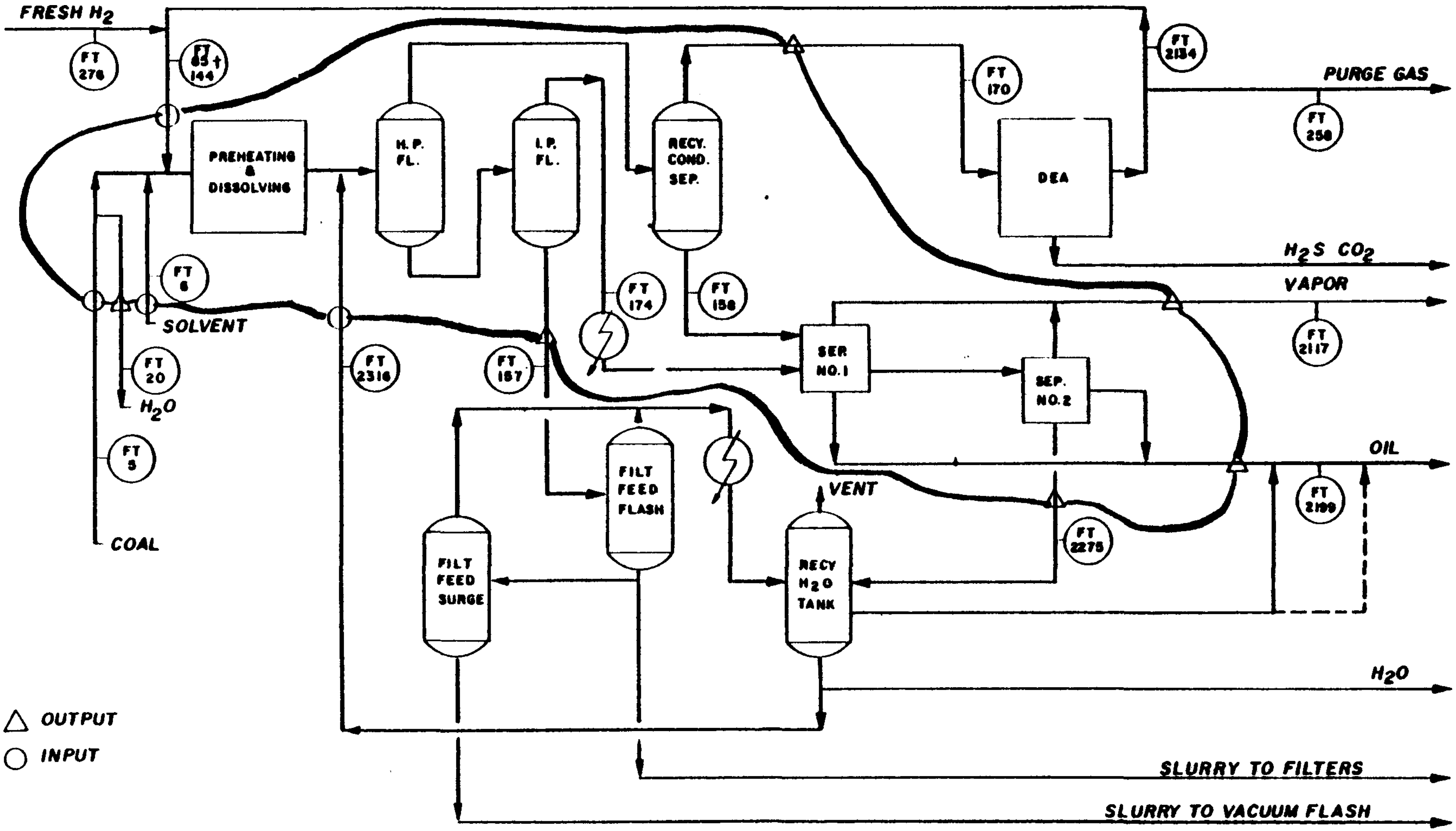


FIGURE 28

METHOD NO. 2 MATERIAL BALANCE ENVELOPE

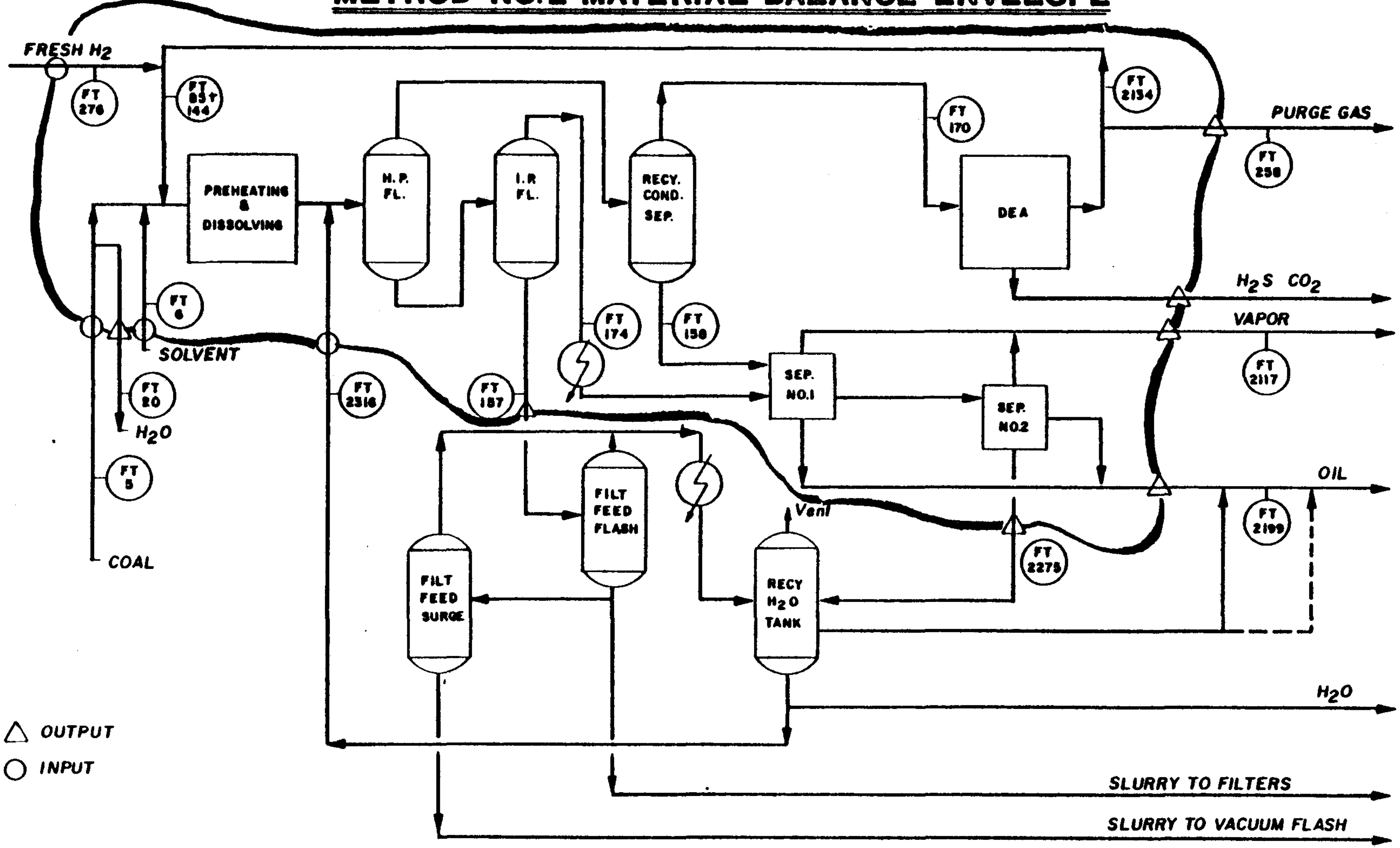
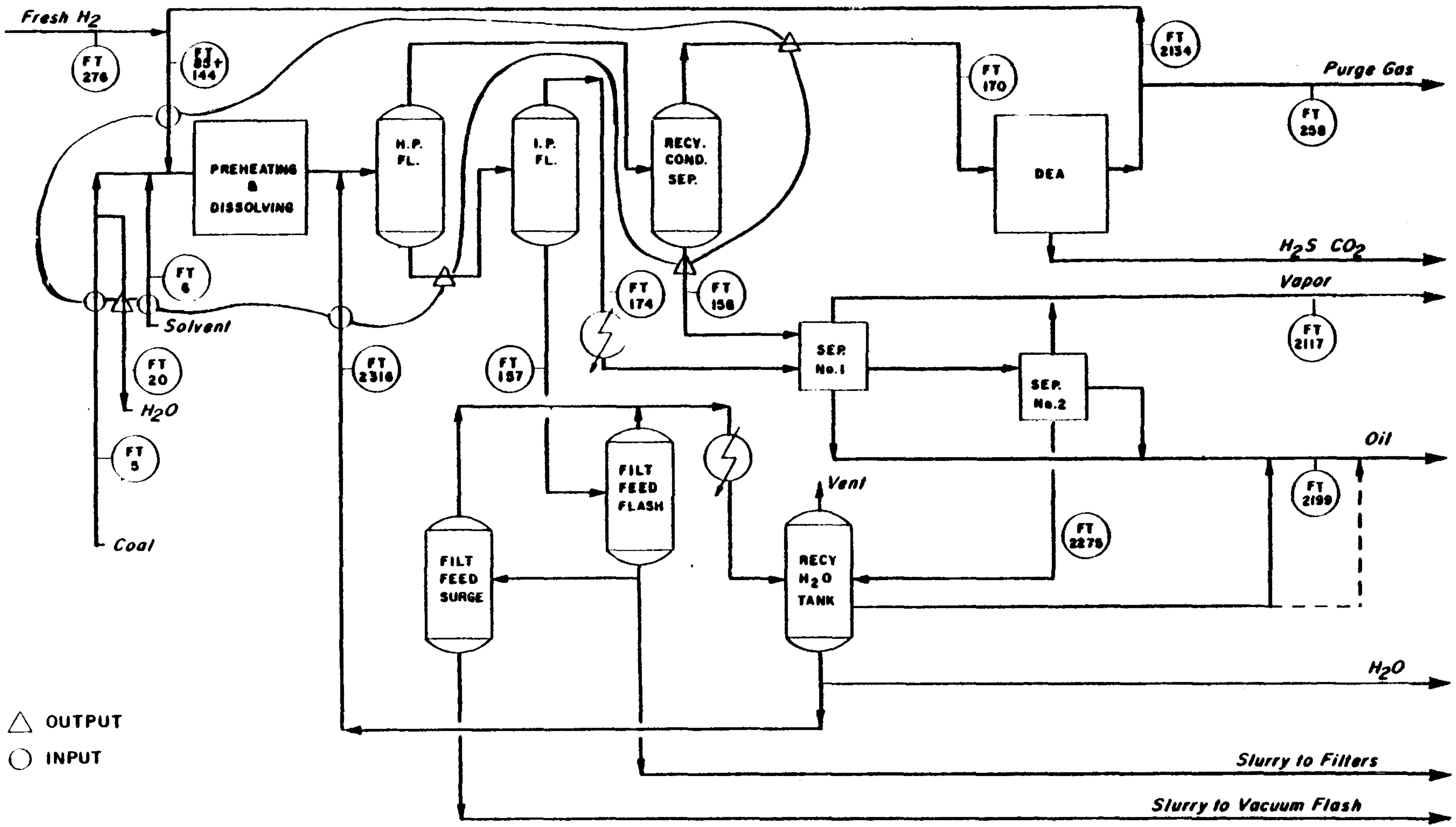


FIGURE 29

METHOD NO. 3 MATERIAL BALANCE ENVELOPE



△ OUTPUT

○ INPUT

FIGURE 30

Method 1

The Method 1 balance envelope is drawn to include inputs of coal, solvent and gas to the reaction area. Against these are balanced outputs of filter feed, oil/water separator products and effluent gas from the reaction area. This method has the advantage of being dependent on relatively few flow rate measurements, but has the disadvantage of being dependent on good oil-water separation and accurate sampling of the filter feed stream.

Method 2

The Method 2 balance is essentially the same as the Method 1 envelope except that it balances the flows of fresh make-up gas and purge gas into and out of the recycle gas loop. This method is subject the the same limitations as Method 1.

Method 3

The Method 3 balance involves a very small envelope drawn around the plant reaction area. Included in the balance are flowrates of coal, solvent and gas into the reaction area, and three output streams -- high pressure flash drum bottoms and recycle condensate separator overhead and bottoms. Because of the small number of streams involved, this method is the least dependent on flow measurements. As a consequence, however, it is highly dependent on accurate analyses of all streams involved.

During the early months of 1975, effort was focused on improving plant yield calculations based on the Method 1, 2 and 3 balances outlined above. Improvements made included adjustment of instrument ranges to ensure maximum accuracy, tuning of control loops to smooth widely varying flowrates, addition of new sampling connections to permit additional stream composition determinations and replacement of defective instrumentation.

With the aid of specially developed computer programs, comparison studies were made between material balance Methods 1, 2 and 3. In general, results showed encouraging accuracy for products excluding hydrocarbon liquids and water. Yield determinations for light oil, wash solvent, process solvent and water continued to be poor, however. The main reasons for this were found to be:

- Failure of the oil-water separators to adequately separate oil and water due to the addition of a large volume of quench water in the reaction area.
- Poor reproducibility of water and solvent analyses of flash vessel liquids.

To improve upon determinations of liquid yields, yet another mass balance technique was developed. In this method, liquid yields were accumulated in process vessels during extended mass balance periods. The accumulated inventory was then measured and sampled to determine its components. This method proved to be an acceptable technique for the measurement of liquid yields.

Based on the foregoing results, an overall mass balance technique was selected, involving a combination of the methods chosen to produce the most accurate results. This general mass balance method is outlined in some detail below.

The process is brought up to specified operating conditions and given sufficient time to achieve equilibrium. A mass balance run is then started, and continued for approximately 3-4 days. During the test period the filtration area and other plant areas not required are bypassed in order to minimize the accumulation of material in intermediate process vessels.

At the completion of the mass balance run, liquid hydrocarbon yields are determined from level change measurements made in each process vessel and tank during the run period. Level changes are then converted to equivalent mass accumulations of each boiling range liquid (light oil, wash solvent, and process solvent) using laboratory analyses and tank geometry.

Gas yields are determined from on-line chromatographic and flow rate measurements, using the Method 1,2 and 3 balances described previously. The results of the three methods are normally in substantial agreement and provide a method of cross-checking the gas yields.

Solvent Refined Coal yields may be calculated in several ways. The primary method uses data from an on-line product flowmeter (gravimetric feeder). In addition to this measurement, during many mass balance periods the total SRC product was accumulated and weighed to provide a check on the flowmeter measurement. A final calculation method uses a forced ash balance between feed coal and SRC product to predict SRC yield.

Mineral residue is not normally produced during mass balance periods since the filtration area is bypassed during the runs. A mineral residue yield is therefore calculated using unfiltered coal solution sample results obtained by the plant laboratory.

The process yield of water normally cannot be accurately measured due to the large volume of water which is injected into the reaction effluent as quench. This results in oil-water separation problems which mask water yield. As a result, process water yield is normally calculated by forced oxygen balance based on input elemental analyses of coal, solvent and gas.

The material balance techniques presented above were used to provide process yield information during eleven SRC I material balances and five SRC II runs. The results of these runs are presented in the following sections of the report.

B. SRC I Process Yield Studies

1. Scope of Work

Eleven material balance tests were made with the pilot plant operating in the SRC I mode. These tests were run during the period of March 1975 to October 1975. The purposes of the material balance studies were: (1) to obtain quantitative process yields, (2) to determine the interactive effects of various operating conditions on these yields and (3) to define operating conditions which optimize the SRC I process from both a process and mechanical standpoint.

Two principle process variables were studied; (1) coal space rate expressed as pounds per hour of coal feed per cubic foot of reactor volume and (2) dissolver outlet temperature. To a lesser extent, the hydrogen to coal mass feed rate ratio and dissolver pressure were investigated. The effects of coal type, gas feed composition, preheater outlet temperature and solvent/coal feed ratio were not studied in this series of experiments.

Coal space rates ranging from 30 to 100 lb/hr ft^3 were run at five different target levels: 30, 50, 75, 90 and 100 lb/hr ft^3. These rates correspond to calculated nominal reactor residence times of 0.92, 0.52, 0.35, 0.31 and 0.27 hours respectively.

The dissolver outlet temperature was varied from 844°F to 870°F. It was believed that relatively small changes in the dissolver temperature (5°-10°F) would generate substantially greater levels of reactivity resulting from the temperature dependency of the reaction rate constants. Temperatures above 870°F were excluded from the study because of possible high exothermic heats of reaction which might cause a runaway reaction or coking of the dissolver. Dissolver temperature conditions below 830°-840°F were not believed to be severe enough at the conditions studied to yield substantial conversion.

A list of the most important conditions studied in the eleven SRC I material balances is shown in Table 20. Five coal space rates (30, 50, 75, 90, & 100 lb/hr ft^3), five temperatures (840°, 845°, 850°, 860°, & 870°F), and three pressures (1500, 1250, & 1000 psig) were studied. The major emphasis of the program was toward higher coal space rates (75 - 100 lb/hr ft^3) to minimize the capital cost of future plant reaction areas and a study of reactor temperature effects.

Coal from the P&M Colonial Mine in Hopkins County, Kentucky, was used in each of the experimental runs. The composition of the coal was approximately 70% Kentucky number 14 seam and 30% Kentucky number 9 seam. The coal was washed and screened to 1 1/4 inches prior to delivery to the pilot plant.

TABLE 20

SRC-I Material Balance Run Conditions

Run Number	Coal Space Rate lb/hr ft^3	Dissolver Pressure psig	Dissolver Outlet Temperature °F
2	30	1500	850
1	50	1500	850
10	75	1000	840
11	75	1250	850
4,8	75	1500	845
3	75	1500	870
9	90	1500	860
5,7	100	1500	850
6	100	1500	870

The purity of the hydrogen feed stream to the reaction area varied from 91 mole % to 97 mole %. The corresponding hydrogen input flow rates varied from 148 lb/hr to 211 lb/hr. The hydrogen feed, expressed as a ratio of thousands of standard cubic feet per ton of moisture free coal, varied from 13.2 to 21.5. This variation resulted primarily from maintaining maximum hydrogen throughput while changing the coal feed rate.

The pilot plant was first operated at design solvent/coal feed ratios of 2.0-2.5. During the first few months of operation, this ratio was gradually decreased to approximately 1.6 (38.5% coal concentration). The ability to process coal at these higher concentrations was an obvious asset to the process economics. As a result, material balances at high solvent/coal ratios became unattractive as compared with the lower solvent/coal ratios. Solvent/coal ratios below 1.6 were not investigated during the first year of plant operation to avoid possible operating problems resulting from increased slurry preheater pressure drops. High pressure drops were thought to indicate high slurry viscosities and increased possibilities of plugging or coking the preheater.

2. Procedure

Prior to a material balance period, flow from the reaction and pressure letdown area of the plant was diverted to the solvent recovery area, thus avoiding flow through the filtration area. This procedure simplified operation of the plant and minimized accumulation of material in intermediate process vessels. In this arrangement, the flow of unfiltered coal solution (UFCS) was routed directly from the flash vessels to an accumulator in the filtration area, through the vacuum flash preheater and into the vacuum flash drum. The SRC product , containing ash and unreacted coal , was then solidified on the Sandvik cooling belt for sampling and analysis. Process solvent was recovered from the distillation area and reslurried with fresh coal feed.

Data for process yield calculations were obtained from liquid level changes of plant vessels and accumulators, on-line gas flow-rate and chromatographic measurements, and laboratory analyses of various product samples. This information was used to calculate average product compositions, an overall process yield material balance and an overall plant elemental balance for each material balance period, as described in paragraph A of this section.

A standardized technique for calculating process yields was not developed prior to material balance run number five. Earlier material balances were calculated in several different ways because of limited sample data, inaccurate instrumentation readings and computer data acquisition problems. Minor adjustments were also made to other material balances when the circumstances dictated. The following paragraphs describe the general procedure used to calculate the process yields.

Gas yields were calculated from flow meters and chromatographic data. The previously described method three gas balance was used to calculate the gas yields. Gas balance methods one and two were used to check results from method number three.

Level changes, recorded by field instrumentation on process vessels, were used to calculate total liquid yield. The liquid yield was further categorized as light oil, wash solvent, or process solvent, as defined by the atmospheric boiling ranges: light oil, 160°-380°F; wash solvent, 380°-480°F; and process solvent, 480°-850°F. Samples of plant distilled liquids were redistilled in the laboratory and analyzed for elemental composition.

Solvent Refined Coal yields were calculated on a forced ash balance with the feed coal, using laboratory analyses of the weight percent ash in the coal and SRC. These procedures were checked in later runs by weighing truck loads of the product. Results from these techniques were found to be in close agreement.

A forced oxygen balance was used to calculate a preliminary water yield. The water yield and SRC yield were later readjusted to eliminate gains or losses in the total mass balance.

Process yields and conditions from the eleven SRC I material balances are shown in Table 21. These results may differ slightly from those reported earlier due to correction of several minor computational errors, and recalculations to unify assumptions and bases. The yields and elemental balances shown in the table are presented as a best estimate. Further adjustments to the data have been attempted, but have not been found to clarify the results.

Product yields per cubic foot of reactor volume for these eleven material balance runs are shown in Table 22. These figures are useful in measuring the effectiveness of the dissolver; i.e., high conversions per unit volume indicate high reactor efficiency. The total liquid solvent yield is shown as the sum of light oil plus wash solvent plus process solvent. The following sections discuss each of the eleven balance runs in greater detail.

3. Material Balance Runs 1-11

Tables 27 through 37 present reaction condition summaries for these material balance runs. The conditions which are shown are averages of data collected during the material balance periods.

Elemental balances for these runs are shown in Tables 38 through 48. These balances were calculated on a forced total mass balance basis.

TABLE 21

SRC-I MATERIAL BALANCE YIELD SUMMARY
FOR
KENTUCKY COAL

CONDITIONS	RUN 1	RUN 2	RUN 3	RUN 4	RUN 5	RUN 6	RUN 7	RUN 8	RUN 9	RUN 10	RUN 11
Coal Space Rate (lb/hr ft^3) (M.F. Coal Basis)	49.5	29.0	75.2	77.9	97.6	98.5	98.8	79.7	91.0	72.8	73.7
S/C Ratio (Dehumidified Coal Basis)	1.66	1.57	1.65	1.57	1.60	1.59	1.59	1.55	1.54	1.61	1.56
H2 Feed, MSCF/TON M.F. Coal	14.0	21.3	18.2	17.7	14.2	13.4	13.2	17.0	14.9	21.5	20.8
Dissolver Pressure, PSIG	1528	1489	1496	1499	1488	1483	1471	1484	1478	1027	1261
Dissolver Outlet Temperature, °F	851	848	870	844	849	870	850	846	859	842	848
YIELDS (% M.F. COAL)											
H2 Consumed	2.5	2.7	2.1	2.0	2.0	2.0	1.9	2.4	2.1	3.6	2.9
C1	2.2	2.3	2.7	1.5	1.3	2.2	1.4	1.4	2.1	2.1	2.3
C2	1.3	1.5	1.6	0.9	0.8	1.3	1.0	1.0	1.2	1.1	1.3
C3	1.1	1.2	1.6	0.9	0.6	1.0	0.9	0.9	1.1	0.9	1.1
C4	0.8	0.5	2.0	0.4	0.2	0.4	0.4	0.4	0.5	0.5	0.7
CO	0.1	0.2	0.1	0.0	0.0	0.1	0.0	0.0	0.1	-0.2	-0.1
CO2	1.2	1.1	1.2	1.0	0.9	1.1	1.0	0.8	1.0	1.2	1.3
H2S	2.0	1.3	2.6	1.1	1.1	1.3	1.8	1.4	1.3	1.8	1.6
H2O	4.9	5.3	7.2	4.8	5.6	5.4	5.5	5.0	5.0	6.3	7.0
Light Oil, 160° to 380°F	6.6	10.2	15.8	4.3	2.6	4.1	3.7	5.1	5.1	1.5	5.6
Wash Solvent, 380° to 480°F	8.0	11.4	-3.2	5.1	7.2	5.2	6.3	4.0	5.1	6.6	2.4
Process Solvent, 480° to 850°F	0.1	-3.9	-4.0	2.9	-1.2	-2.6	-1.2	4.4	-0.2	-1.2	1.2
SRC	58.3	57.5	58.6	64.0	67.3	65.9	65.6	63.0	64.2	65.4	61.1
Ash	9.3	9.2	8.8	9.3	9.3	10.1	9.8	9.6	9.7	11.3	11.3
Unreacted Coal	6.6	4.8	7.1	5.8	6.3	6.5	5.7	5.4	5.9	6.3	6.1
TOTAL	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
Total Gas (excluding H_2)	8.7	8.1	11.8	5.8	4.9	7.4	6.5	5.9	7.3	7.4	8.4
Total Liquid (excluding H_2O)	14.7	17.7	8.2	12.3	8.6	6.7	8.8	13.5	10.0	6.9	9.1

TABLE 22

SRC-I MATERIAL BALANCE RUNS
CALCULATED YIELDS PER CUBIC FOOT OF REACTOR VOLUME
FOR
KENTUCKY COAL

Space Rates (lb/hr ft^3 of reactor volume)	Material Balance Run Number										
	1	2	3	4	5	6	7	8	9	10	11
Feeds											
M.F. Coal	49.5	29.0	75.2	77.9	97.6	98.5	98.8	79.7	91.0	77.8	73.7
Solvent	82.2	45.5	124.1	122.3	156.2	156.6	157.1	123.5	140.1	117.2	115.0
Slurry	131.7	74.5	199.3	200.2	253.8	255.1	255.9	203.2	231.1	190.0	188.7
Products											
Light Oil	3.2	3.0	12.1	3.3	2.5	4.1	3.6	4.0	4.6	1.1	4.1
Wash Solvent	3.9	3.3	-2.3	4.0	7.0	5.1	6.2	3.2	4.6	4.8	1.7
Process Solvent	.1	-1.1	-3.0	2.3	-1.2	-2.5	-1.2	3.5	-.2	-.9	.9
SRC (less Ash & Unreacted Coal)	28.8	16.7	45.0	47.2	65.6	64.9	64.8	50.2	58.4	56.8	45.0
TOTAL LIQUID (excluding SRC)	7.2	5.2	6.8	9.6	8.3	6.7	8.6	10.7	9.0	5.0	6.7

Tables 49 through 59 summarize the averaged laboratory results. The analyses are averages of several plant samples which were taken during each material balance period. The oxygen analyses were determined by difference. These tables also contain the analysis of the high pressure separator slurry sample, sample number 204. Since this sample is taken at operating conditions (600°F and 1500 psig), considerable gas is recovered when the sample is reduced to standard conditions. This sample is used primarily for estimating the relative amounts of SRC and unreacted coal.

Tables 60 through 70 list gas balance calculations for the material balance runs. These results were computed from averaged on-line chromatographic analyses and gas flow rates.

Plots of the slurry preheater temperature and pressure profiles for the eleven SRC-I material balance runs are shown in Figures 35 through 45.

Early material balance runs established that a desirable process yield structure could be obtained at higher space rates and lower residence times than design. Runs five and seven were duplicate runs at the highest space rate (100 lb/hr ft^3) and runs four and eight were duplicate runs at a moderate space rate (75 lb/hr ft^3). A comparison of these duplicate runs indicates good reproducibility which lends confidence to the interpretation of the yield trends in other runs. Furthermore, runs 4 and 8 conclusively demonstrate that the process can run in solvent balance. Comparison of runs three, five and six provides some insight into the combined effect of space rate and dissolver temperature. Runs ten and eleven elucidate the effect of dissolver pressure so as to determine a practical lower boundary for this operating variable.

4. Statistical Analysis of SRC I Material Balance Yield Data

Both linear regression and multiple linear regression were used in an effort to develop a model that would predict yields based on the material balance yield data listed in Table 21.

The yields of the various SRC products shown in Table 21 are the weight percent yield based on moisture free coal feed. Along with these yields, some pertinent operating conditions are also shown. As previously discussed, the operating variables which were of primary concern in these runs were coal space rate, dissolver outlet temperature and disssolver pressure.

The following independent variables were used in simple linear regression models to determine if there was a significant relationship between the chosen variable and the yields of various components.

VARIABLE	UNITS	DEFINITION
• Coal Space Rate (CSR)	lbs/hr ft^3	Moisture free coal feed per unit of dissolver volume.
• Total Hourly Space Rate (THSR)	lbs/hr ft^3	Coal slurry feed (Dehumidified coal plus solvent per unit of dissolver volume).
• Process Solvent Yield	% Moisture free coal	Yield of process solvent (on a moisture free coal basis).
• Hydrogen Consumption	% Moisture free coal	Consumption of hydrogen (on a moisture free coal basis).
• Dissolver Outlet Temperature (T)	°F	
• Inverse Dissolver Outlet Temperature (1/T)	1/°F	
• Inverse Total Hourly Space Rate (1/THSR)	ft^3 hr/lb	

Of the variables listed, total hourly space rate and dissolver outlet temperature were found to be the most significant in modeling the various component yields. Therefore, these variables were combined into a multiple linear regression model to predict yields of several components. Other combinations of the variables listed above were tried, but none of the models were as consistent in predicting product yields as the model which used dissolver outlet temperature and total hourly space rate.

The models obtained for several components using these two independent variables are shown in Table 23 and the dependent variables used in the correlation are shown in Table 24. Liquid yields for runs 1, 2 and 3 were not used to develop these models because of several problems (lack of adequate sample analysis and insufficient tank level data) which made the liquid yields for these runs less accurate than for later runs. As a result, the models for light oil, wash solvent and process solvent were based on the results from runs 4 through 9. The data developed in runs 10 and 11 also were not used in development of the models shown in Table 23 due to the different operating pressures involved in these runs.

TABLE 23

MULTIPLE LINEAR REGRESSION MODELS FOR SRC-I

1500 psig - Kentucky Coal - 1.6/1 Solvent to Coal Ratio

T = Dissolver Outlet Temperature, °F   THSR = Total Hourly Space Rate, lbs/hr ft^3

YIELD REGRESSION MODELS

(Yield as Percent Moisture Free Coal Feed)

Hydrogen:	- 3.685	$+ 3.919 \times 10^{-3}$ (THSR)	$+ 7.952 \times 10^{-4}$ (T)
Methane:	- 38.088	$- 5.782 \times 10^{-3}$ (THSR)	$+ 4.917 \times 10^{-2}$ (T)
Ethane:	- 18.185	$- 3.396 \times 10^{-3}$ (THSR)	$+ 2.350 \times 10^{-2}$ (T)
Propane:	- 16.870	$- 2.803 \times 10^{-3}$ (THSR)	$+ 2.165 \times 10^{-2}$ (T)
Butane:	- 31.396	$- 3.324 \times 10^{-3}$ (THSR)	$+ 3.830 \times 10^{-2}$ (T)
Carbon Dioxide:	- 6.999	$- 1.304 \times 10^{-3}$ (THSR)	$+ 9.723 \times 10^{-3}$ (T)
Light Oil:	- 41.767	$- 3.616 \times 10^{-2}$ (THSR)	$+ 6.410 \times 10^{-2}$ (T)
Wash Solvent:	58.670	$+ 4.609 \times 10^{-2}$ (THSR)	$- 7.544 \times 10^{-2}$ (T)
Process Solvent:	80.702	$- 8.211 \times 10^{-2}$ (THSR)	$- 7.088 \times 10^{-2}$ (T)
SRC:	135.130	$+ 5.456 \times 10^{-2}$ (THSR)	$- 9.793 \times 10^{-2}$ (T)
Total C_1-C_4:	-103.465	$- 1.520 \times 10^{-2}$ (THSR)	$+13.039 \times 10^{-2}$ (T)
Total Liquid:	97.617	$- 7.217 \times 10^{-2}$ (THSR)	$- 8.224 \times 10^{-2}$ (T)

TABLE 24

SRC-I DEPENDENT VARIABLES USED IN REGRESSION ANALYSIS

Independent Variables			Dependent Variables Yield, Percent Moisture Free Coal											
Run #	THSR	T	H_2	CH_4	C_2H_6	C_3H_8	C_4H_{10}	Total HC Gas	CO_2	LO	WS	PS	Total Liquid	SRC
1	136.6	851	-2.5	2.2	1.3	1.1	0.8	5.4	1.2	---	---	---	---	58.3
2	77.9	848	-2.7	2.3	1.5	1.2	0.5	5.5	1.1	---	---	---	---	57.5
3	208.2	870	-2.1	2.7	1.6	1.6	2.0	7.9	1.2	---	---	---	---	58.6
4	209.7	844	-2.0	1.5	0.9	0.9	0.4	3.7	1.0	4.3	5.1	2.9	12.3	64.0
5	264.1	849	-2.0	1.3	0.8	0.6	0.2	2.9	0.9	2.6	7.2	-1.2	8.6	67.3
6	264.9	870	-2.0	2.2	1.3	1.0	0.4	4.9	1.1	4.1	5.2	-2.6	6.7	65.9
7	264.9	850	-1.9	1.4	1.0	0.9	0.4	3.7	1.0	3.7	6.3	-1.2	8.8	65.6
8	210.3	846	-2.4	1.4	1.0	0.9	0.4	3.7	0.8	5.1	4.0	4.4	13.5	63.0
9	239.4	859	-2.1	2.1	1.2	1.1	0.5	4.9	1.0	5.1	5.1	-0.2	10.0	64.2

In Table 25, the actual yields and those predicted by the corresponding models are shown. Visual analysis of these results indicate good predictions from most of the models. To obtain a better understanding of the utility of the models, several statistical tests were performed.

The adequacy of each model was tested by a standard one-way analysis of variance in which the total sum of squares, error sum of squares and sum of squares due to regression were computed. From these values it was possible to calculate a value for the overall F statistic. These values are shown in Table 26. The coefficient of multiple determination (a measure of the total variance explained by the model) was also calculated and is shown in Table 26 as the "percent variance explained by the model".

The overall F statistics calculated for these models indicate that the hydrogen, methane, ethane, propane, carbon dioxide, process solvent and SRC models can be accepted at the 0.05 level of significance and the wash solvent model can be accepted at the 0.10 level of significance. Only the butane and light oil models are not accepted at the 0.10 level. Evaluation of the coefficient of multiple determination for each model suggests that, with the exception of the butane model, the models explain more than half of the variance of the data set. These two parameters (the coefficient of multiple determination and the overall F statistic calculated from the analysis of variance for the model) indicate that the two independent variables chosen for these models can be used to predict yield levels.

It is important to note that the yields of carbon monoxide, water, mineral residue and unreacted coal were not included in the modeling study. The reasons for the deletion of these yields are listed below:

• Carbon monoxide -	The yield (or consumption) of this component was found to be less than ± 0.3 percent of moisture free coal. This may be due to the presence of carbon dioxide, hydrogen and water, in the reaction area, resulting in a water-gas shift reaction. This would be expected primarily in the dissolver where chemical equilibrium favors the formation of carbon dioxide and hydrogen from carbon monoxide and water.
• Water	In calculating material balances, the water yield was determined by an elemental oxygen balance. The oxygen fraction in the coal feed, liquid products (light oil, wash solvent and process solvent) and SRC has been

TABLE 25

MODEL PREDICTIONS VS. ACTUAL MEASURED COMPONENT YIELDS

(EXPRESSED IN PERCENT OF M.F. COAL FEED)

1500 PSIG - Kentucky Coal - -1.6/1 Solvent to Coal Ratio

	-HYDROGEN-		-METHANE-		-ETHANE-		-PROPANE-		-BUTANE-		-C_1-C_4 Gas-	
Run No.	Predicted	Actual	Predicted	Actual	Predicted	Actual	Predicted	Actual	Predicted	Actual	Predicted	Actual
1	- 2.47	-2.5	2.17	2.2	1.35	1.3	1.17	1.1	0.74	0.8	5.52	5.4
2	- 2.71	-2.7	2.37	2.3	1.48	1.5	1.27	1.2	0.82	0.5	5.92	5.5
3	- 2.18	-2.1	2.69	2.8	1.55	1.6	1.38	1.6	1.23	2.0	6.81	7.9
4	- 2.19	-2.0	1.41	1.5	0.94	0.9	0.81	0.9	0.23	0.4	3.40	3.7
5	- 1.97	-2.0	1.34	1.3	0.87	0.8	0.77	0.6	0.24	0.2	3.22	2.9
6	- 1.96	-2.0	2.37	2.2	1.36	1.3	1.22	1.0	1.04	0.4	5.95	4.9
7	- 1.97	-1.9	1.38	1.4	0.89	1.0	0.79	0.9	0.28	0.4	3.34	3.7
8	- 2.19	-2.4	1.50	1.4	0.98	1.0	0.85	0.9	0.31	0.4	3.65	3.7
9	- 2.06	-2.1	1.97	2.1	1.19	1.2	1.05	1.1	0.71	0.5	4.90	4.9

	-CO2-		-LT. OIL-		-WASH SOLV.-		-PROC. SOLV.-		TOTAL LIQUIDS		-SRC-	
Run No.	Predicted	Actual	Predicted	Actual	Predicted	Actual	Predicted	Actual	Predicted	Actual	Predicted	Actual
1	1.10	1.2	*	*	*	*	*	*	*	*	59.24	58.3
2	1.14	1.1	*	*	*	*	*	*	*	*	56.33	57.6
3	1.19	1.2	*	*	*	*	*	*	*	*	61.29	59.8
4	0.93	1.0	4.75	4.3	4.66	5.1	3.66	2.9	13.07	12.3	63.91	64.0
5	0.91	0.9	3.10	2.6	6.79	7.2	-1.16	-1.2	8.74	8.6	66.39	67.3
6	1.11	1.1	4.42	4.1	5.25	5.2	-2.71	-2.6	6.95	6.7	64.33	65.9
7	0.92	1.0	3.14	3.7	6.76	6.3	-1.80	-1.2	8.60	8.8	66.34	65.6
8	0.95	0.8	4.86	5.1	4.54	4.0	3.47	4.4	12.86	13.5	63.75	63.0
9	1.04	1.0	4.64	5.1	4.90	5.1	0.15	-0.2	9.70	10.0	64.07	64.2

TABLE 26

STATISTICAL ANALYSIS OF MODEL ADEQUACY

1500 psig - Kentucky Coal - 1.6/1 Solvent to Coal Ratio

Component	Percent Variance Explained by Model*	Overall Calculated F	Level of Significance	Partial F Due to T	Level of Significance	Partial F Due to THSR	Level of Significance
Hydrogen	83.99	15.73	95	0.03	< 90	28.07	95
Methane	96.35	79.00	95	128.49	95	73.71	95
Ethane	95.40	62.07	95	86.32	95	74.80	95
Propane	73.42	8.29	95	12.62	95	8.78	95
Butane	48.27	2.80	< 90	5.21	90	1.63	< 90
Carbon Dioxide	65.21	5.62	95	8.34	95	6.22	95
Light Oil	74.14	4.30	< 90	3.57	< 90	8.49	90
Wash Solvent	85.51	8.75	90	6.32	90	17.66	95
Process Solvent	95.65	32.98	95	3.13	< 90	31.46	95
SRC	90.79	29.57	95	4.59	< 90	59.11	95
Total C_1-C_4 Gas	74.89	8.94	95	13.98	95	9.04	95
Total Liquid	96.18	37.76	95	5.54	90	31.92	95

* Percent Variance Explained by Model = R^2, the coefficient of multiple determination x 100

	calculated by difference in the elemental analysis of each of these materials. It is probable that the water yield based on these oxygen approximations will not be very accurate. For that reason, water yields were not modeled.
• Mineral Residue	The yield of this material would have been most strongly affected by the composition of the coal during the material balance runs. In addition, at the conditions studied, coal conversion was essentially complete, resulting in a fairly constant level of unreacted coal during the runs. At such high conversion levels, the unreacted coal yield was due more to the petrographic nature of the coal (the presence of inert carbonaceous matter such as inertinites and fusinites), than the reaction conditions.

After the models were developed, an attempt was made to determine which independent variable affected the product yields most strongly. To do this, a stepwise regression procedure was used whereby the partial F statistics for both independent variables were calculated for each model. The values of these partial F statistics are listed in Table 26. These data show that the dissolver outlet temperature appears to have the strongest effect on the gas yields, except for hydrogen. The total hourly space rate, which has an indirect relationship to dissolver residence time, seems to have the strongest effect on the yields of liquids (light oil, wash solvent, process solvent) and SRC and hydrogen consumption.

Further analysis of the partial F statistics shown in Table 26 indicates that the effect of temperature on the rate of hydrogen consumption and the formation of liquid products in not significant at the 0.05 level of significance. For all of the hydrocarbon gases except butane, the effects of both variables are not significant at the 0.05 level, however, the effect of temperature seems to be greater than that of space rate.

Analysis of the models listed in Table 23 results in some other observations which are of interest. The temperature had a positive effect on hydrocarbon gas yields. Thus, high dissolver temperatures result in higher gas yields than lower temperatures, as expected. The effect of space rate on hydrocarbon gas yield would also be expected, since the gas yields show a negative relationship to space rate, i.e. as the space rate increases, the gas rate decreases. This same negative relationship of space rate is true for light oil and process solvent. The wash solvent and SRC models show positive relationships between space rate and product yield. This result is reasonable since at lower space rates (higher reactor residence times) more of the asphaltene material would be reacted to gaseous and solvent products.

Analysis of the effects of temperature on liquid yields implies that the yields of process solvent and wash solvent decrease with increasing temperature, while the light oil yield increases as temperature increases.

The coefficients in the model for hydrogen consumption indicate a very small effect of temperature (as shown in the analysis of partical F statistics in Table 26) and a negative effect of space rate. The negative coefficient of space rate indicates that hydrogen consumption decreases with increasing space rate.

Additional correlation models for total liquids (light oil, wash solvent and process solvent) and total C_1-C_4 hydrocarbon gas are shown in Table 23, and the statistical analysis of these models is included in Table 26. In addition, the models developed for total liquid, total gas and SRC yield plus the model developed for hydrogen consumption are shown along with the actual data used in model formulation in Figures 31 through 34.

5. Conclusions

Significant conclusions, based upon the data from the eleven material balance runs in the SRC I experimental program are listed below:

- Higher coal space rates (lower reactor residence times) increase the SRC yield, decrease the total gas yield and decrease the total solvent yield.
- Increased dissolver temperatures decrease the SRC yield, increase the total gas yield and decrease the total solvent yield.
- Lower dissolver pressure promotes increased gas yields and reduces total solvent yields.
- The reaction may have been hydrogen limited.
- The water yields may not be representative of actual yields since they were calculated on a forced oxygen balance.
- The individual solvent yields (light oil, wash solvent and process solvent) were difficult to determine because of poor plant distillations and analysis, particularly during the first three material balance tests.

SRC I GAS YIELD vs SPACE RATE AND TEMPERATURE

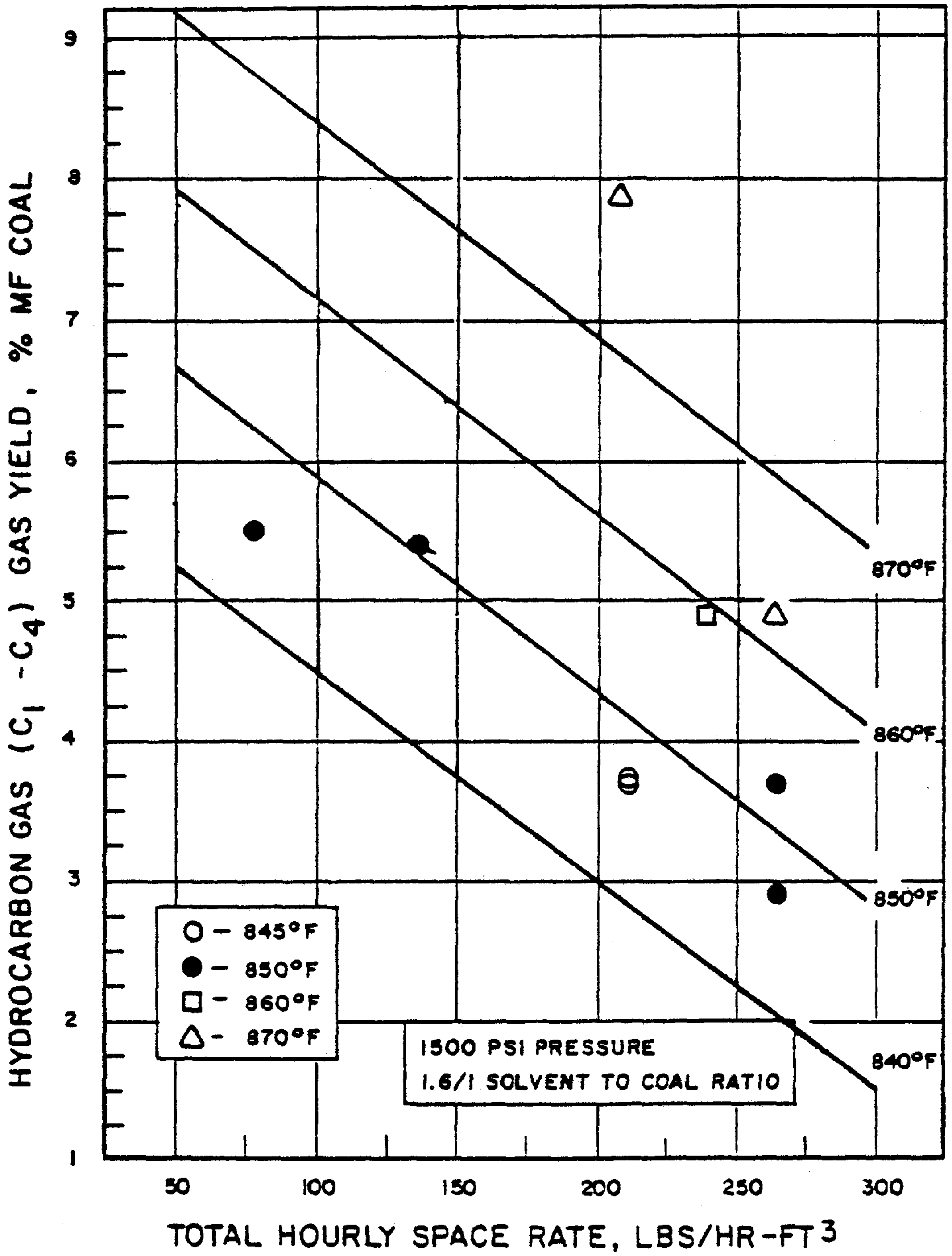


FIGURE 31

MODEL OF LIQUID YIELD AS A FUNCTION OF DISSOLVER OUTLET TEMPERATURE AND TOTAL HOURLY SPACE RATE

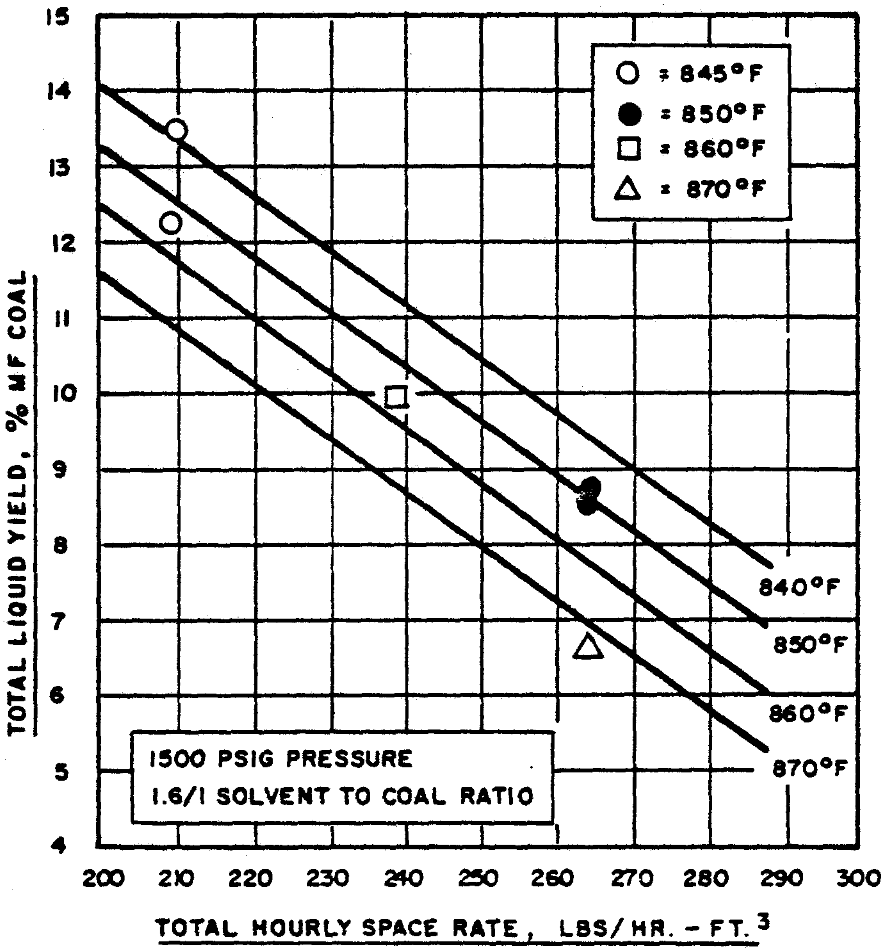


FIGURE 32

MODEL OF SRC YIELD AS A FUNCTION OF DISSOLVER OUTLET TEMPERATURE AND TOTAL HOURLY SPACE RATE

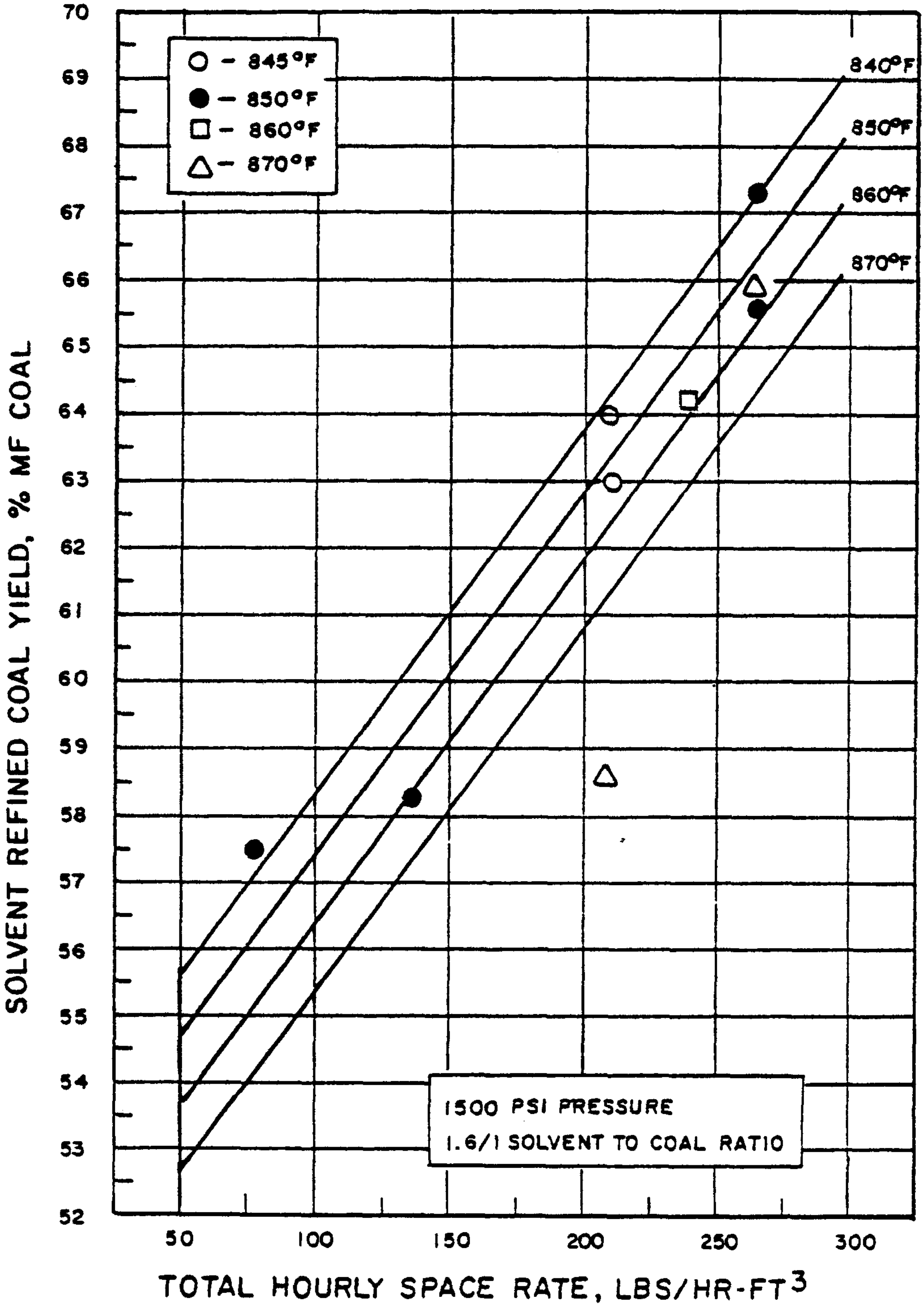


FIGURE 33

MODEL OF HYDROGEN CONSUMPTION AS A FUNCTION OF DISSOLVER OUTLET TEMPERATURE AND TOTAL HOURLY SPACE RATE

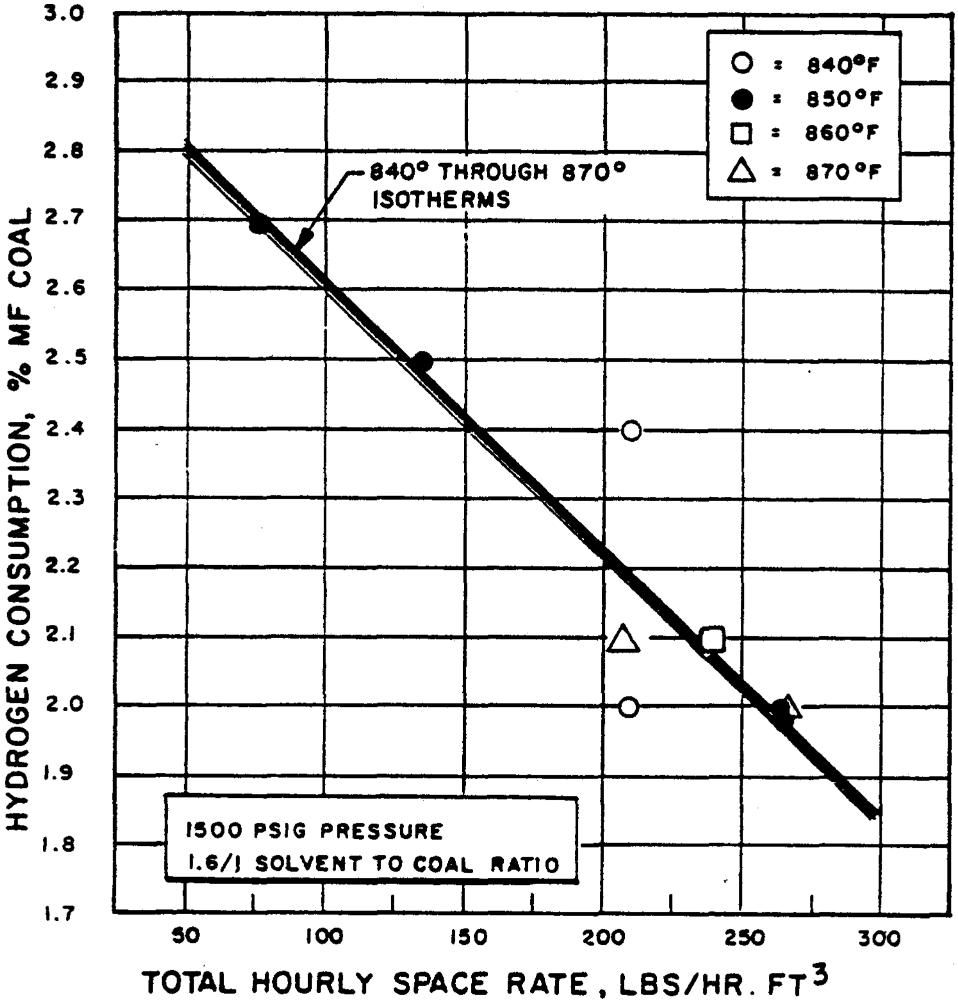


FIGURE 34

- The large imbalances calculated for hydrogen and sulfur in the elemental balances suggests some systematic error in the yield measurements.

- The effects of changing preheater outlet temperature or solvent/coal ratio are unknown.

- No substantial effect was noted when the reactor volume was reduced from 90 ft^3 to 50 ft^3; e.g. effects of reactor geometry (90 vs 50 ft^3) were not discernible.

- Increasing the heat flux during preheating reduced the pressure drop through the preheater.

- The majority of the pressure drop through the preheater occurs between tube turn 30 and 40. The pressure drops averaged about 50-60 psi.

- A substantial increase in dissolver temperature was recorded in the first four feet of length for each material balance run.

- The hydrogen consumption remained relatively constant and was not greatly affected by coal space rate or dissolver temperature.

SRC-I
SLURRY PREHEATER TEMPERATURE & PRESSURE PROFILE
MATERIAL BALANCE RUN NUMBER I

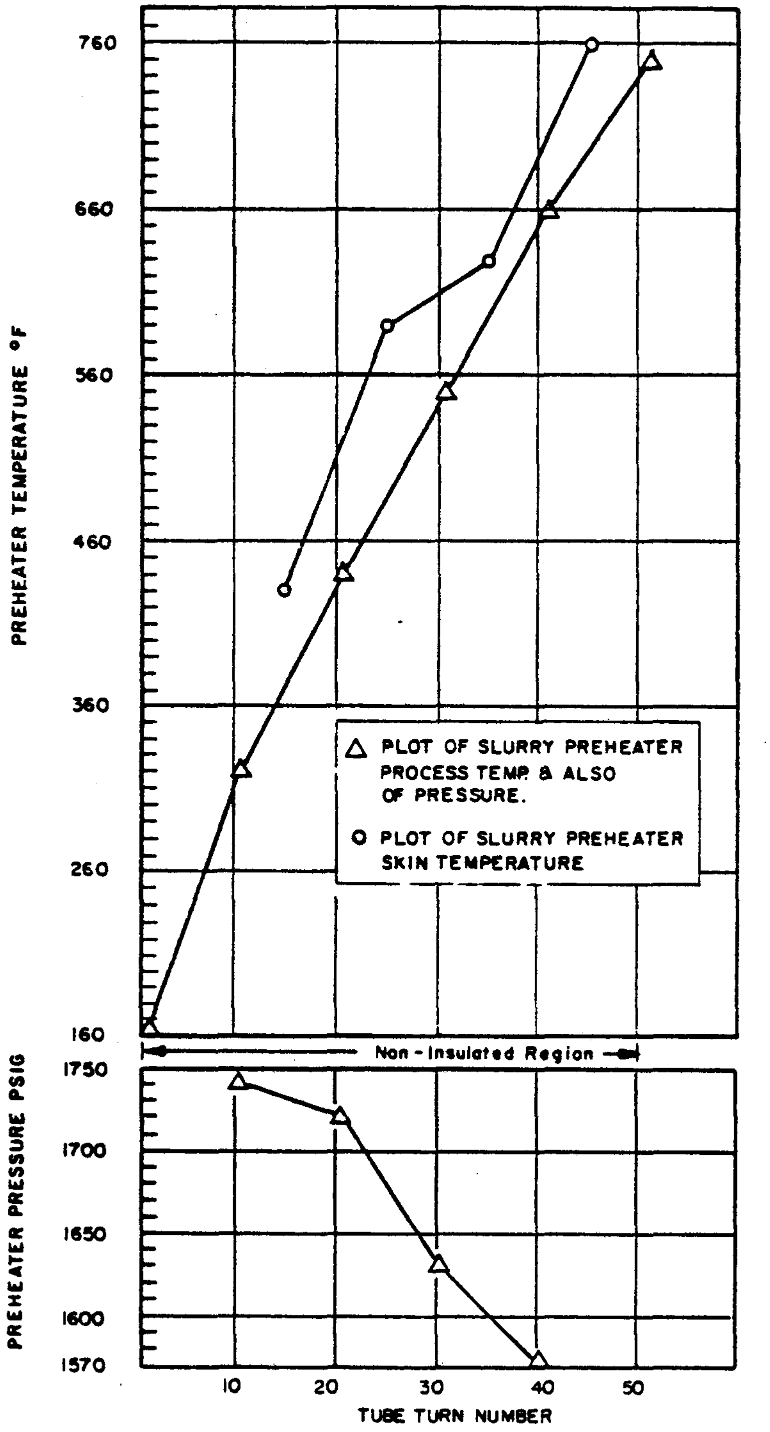


FIGURE 35

SRC-I
SLURRY PREHEATER TEMPERATURE & PRESSURE PROFILE
MATERIAL BALANCE RUN NUMBER 2

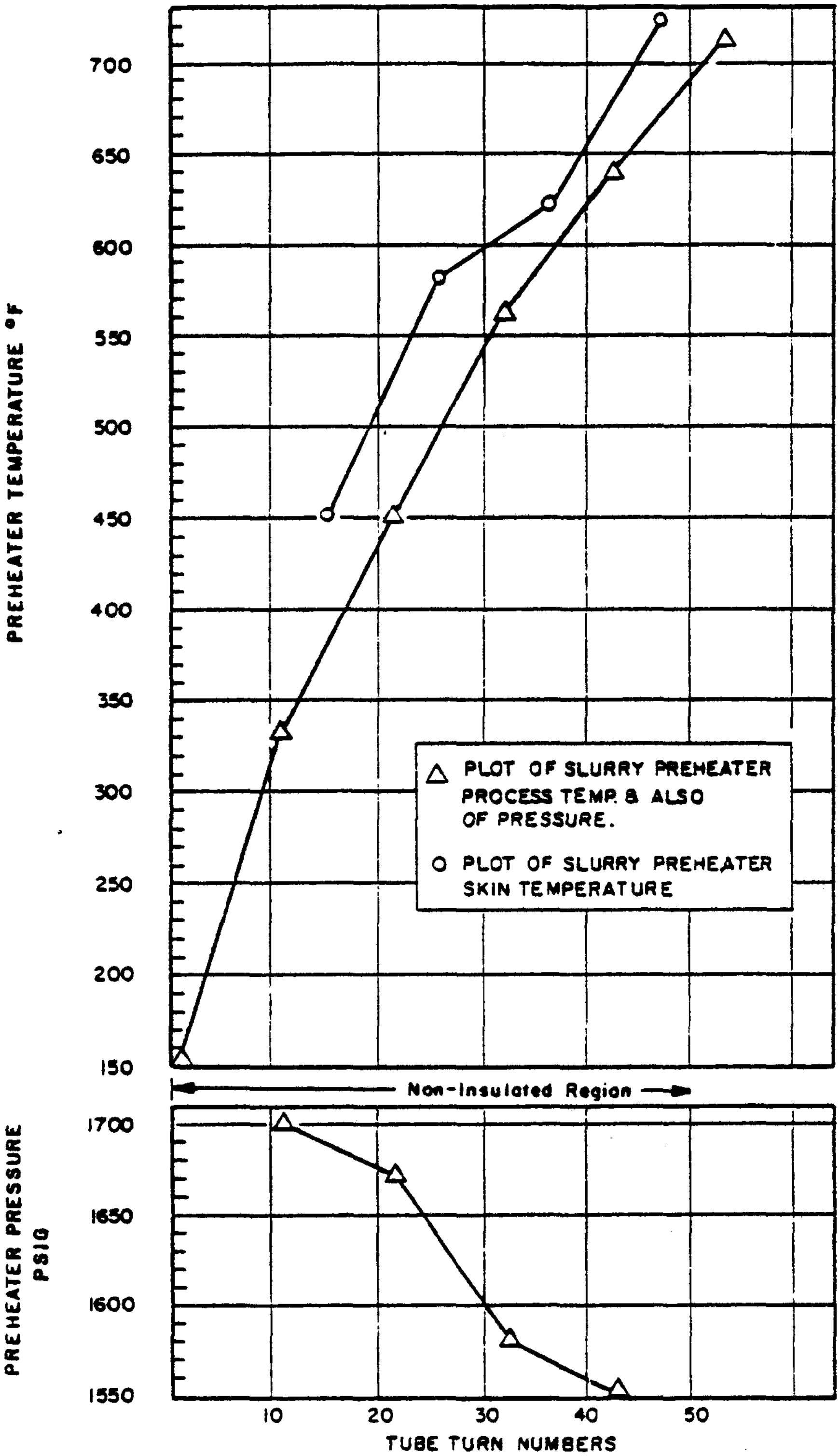
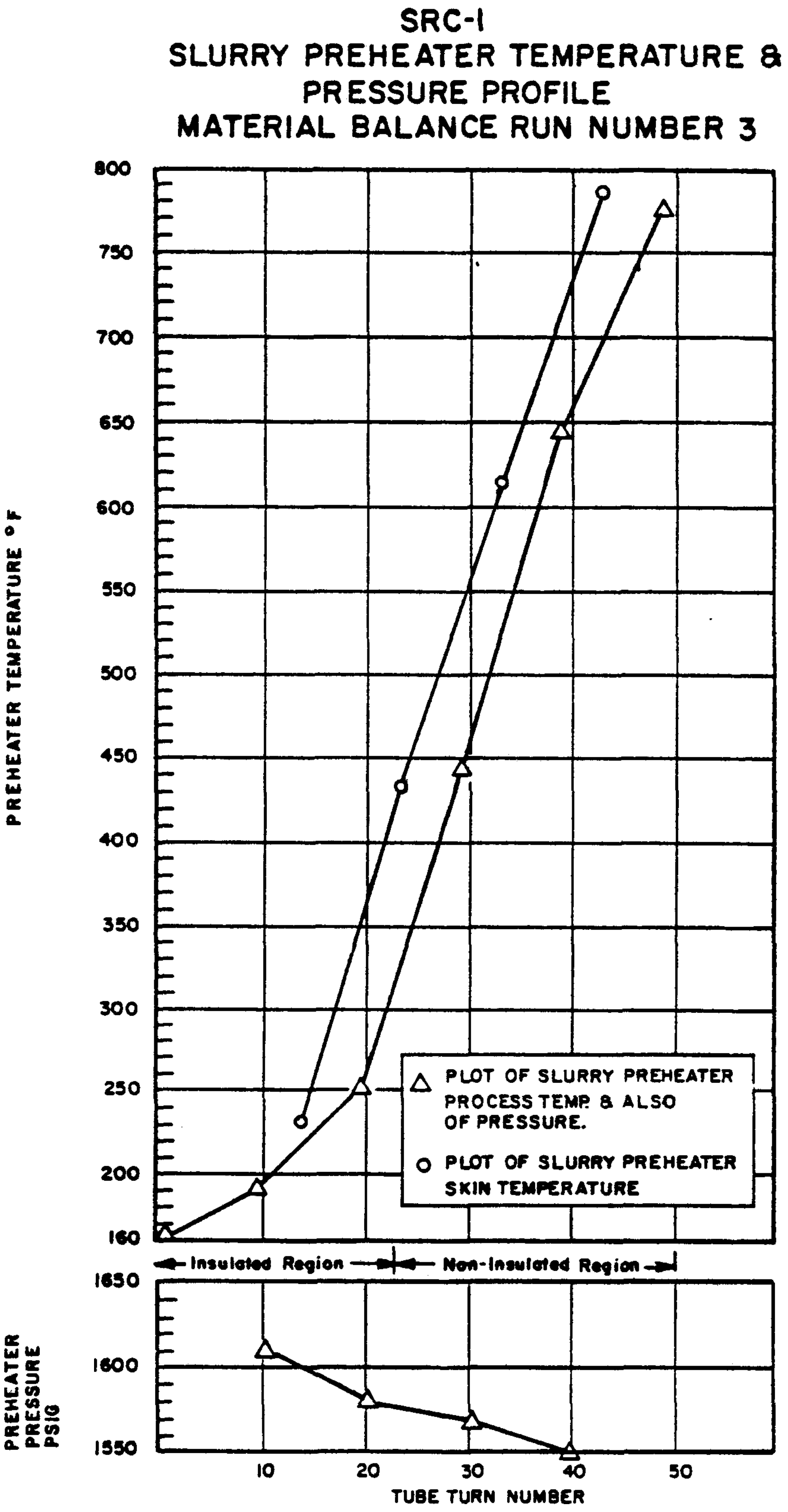


FIGURE 36



SRC-I
SLURRY PREHEATER TEMPERATURE &
PRESSURE PROFILE
MATERIAL BALANCE RUN NUMBER 3

FIGURE 37

SRC-I
SLURRY PREHEATER TEMPERATURE & PRESSURE PROFILE
MATERIAL BALANCE RUN NUMBER 4

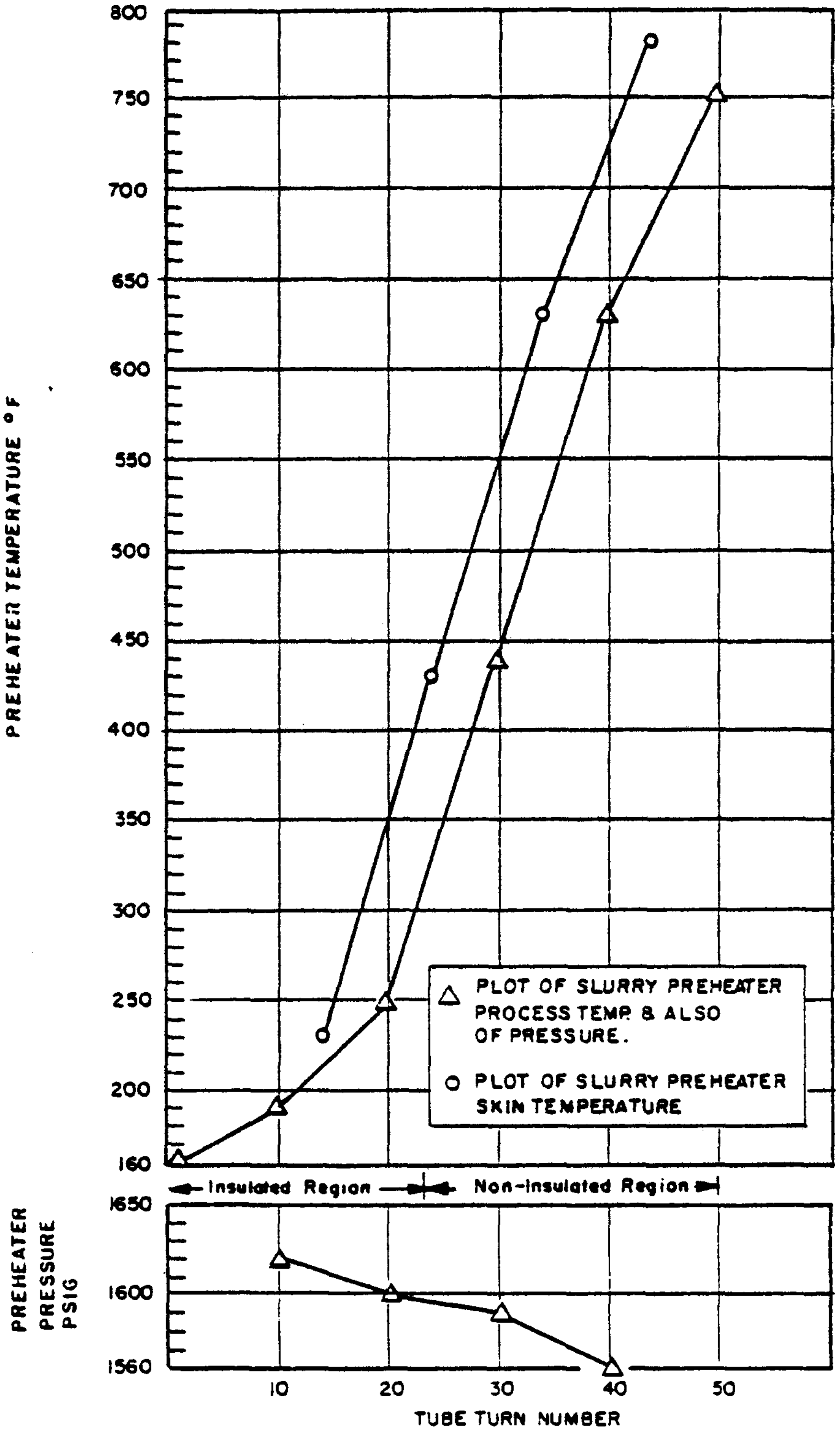


FIGURE 38

SRC-I
SLURRY PREHEATER TEMPERATURE &
PRESSURE PROFILE
MATERIAL BALANCE RUN NUMBER 5

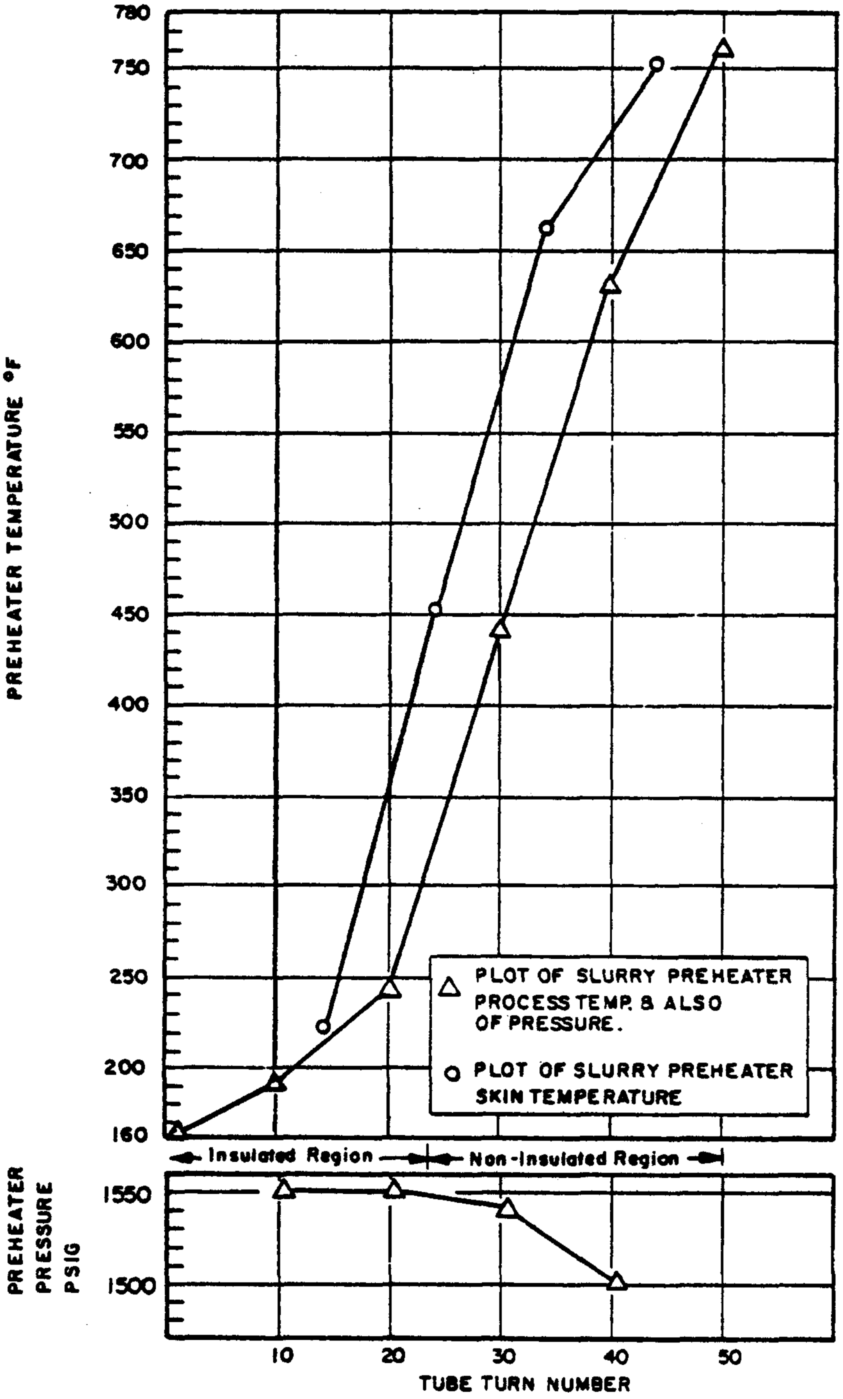


FIGURE 39

SRC-I
SLURRY PREHEATER TEMPERATURE &
PRESSURE PROFILE
MATERIAL BALANCE RUN NUMBER 6

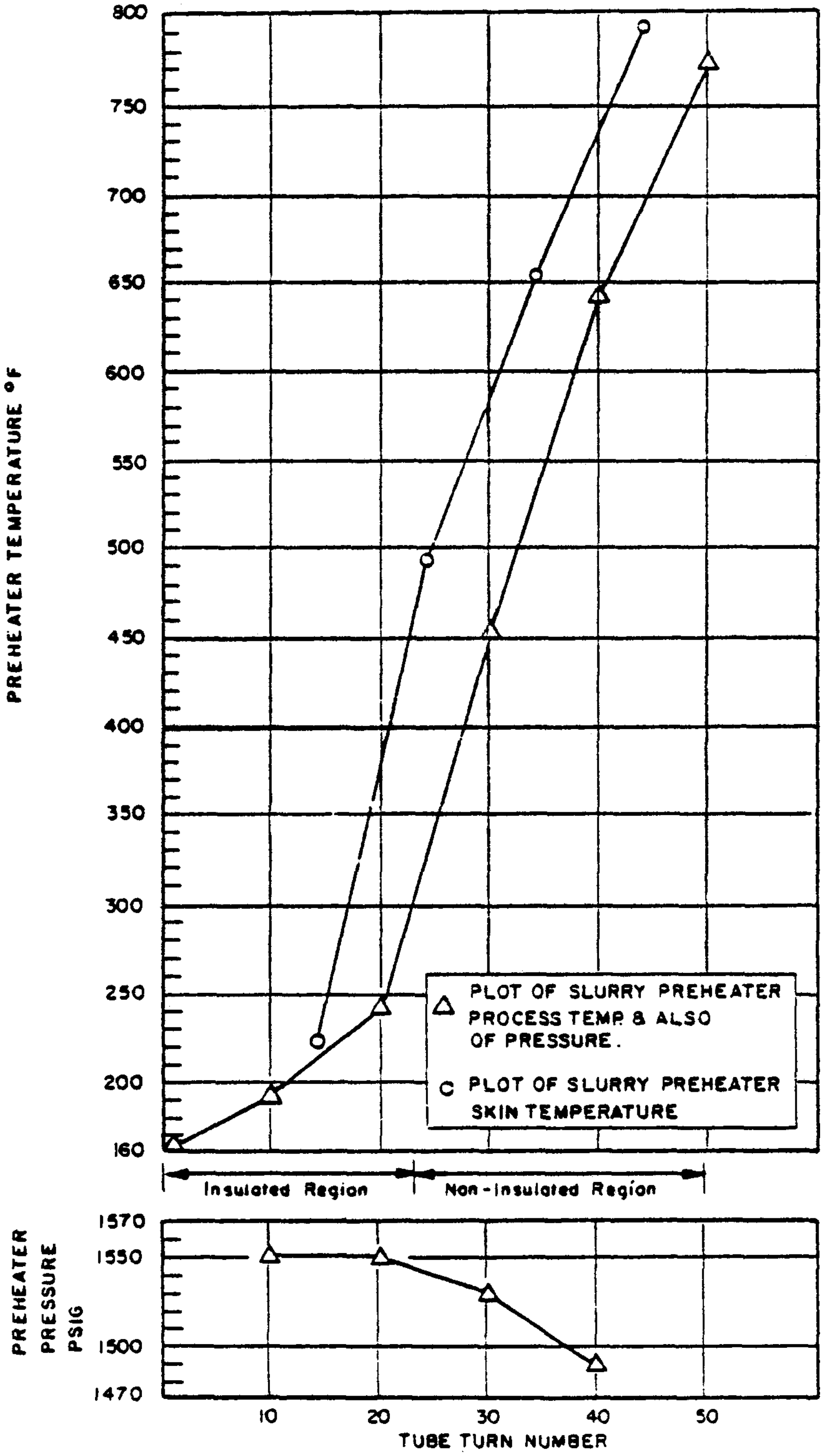


FIGURE 40

SRC-I
SLURRY PREHEATER TEMPERATURE & PRESSURE PROFILE
MATERIAL BALANCE RUN NUMBER 7

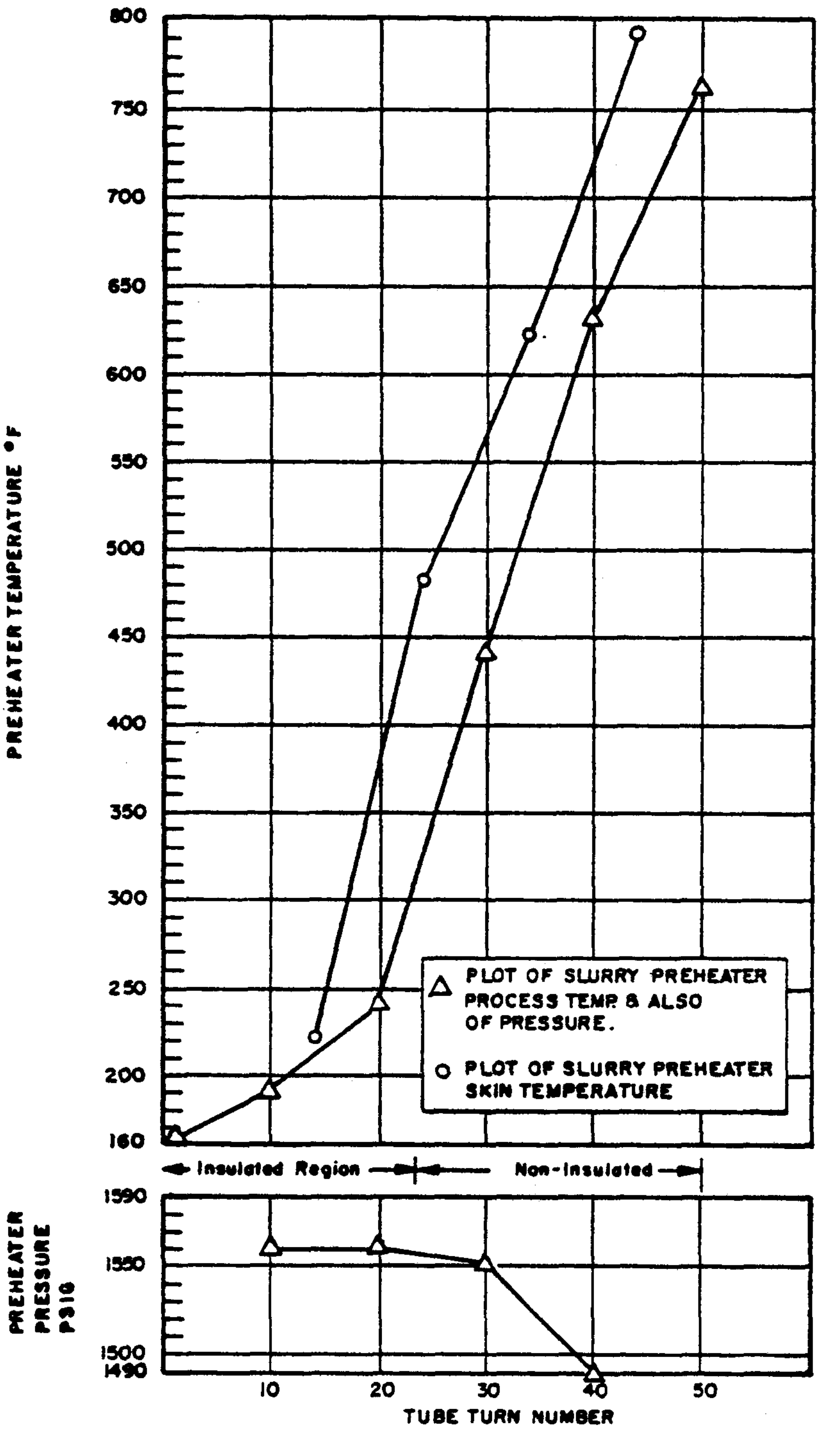


FIGURE 41

SRC-I
SLURRY PREHEATER TEMPERATURE & PRESSURE PROFILE
MATERIAL BALANCE RUN NUMBER 8

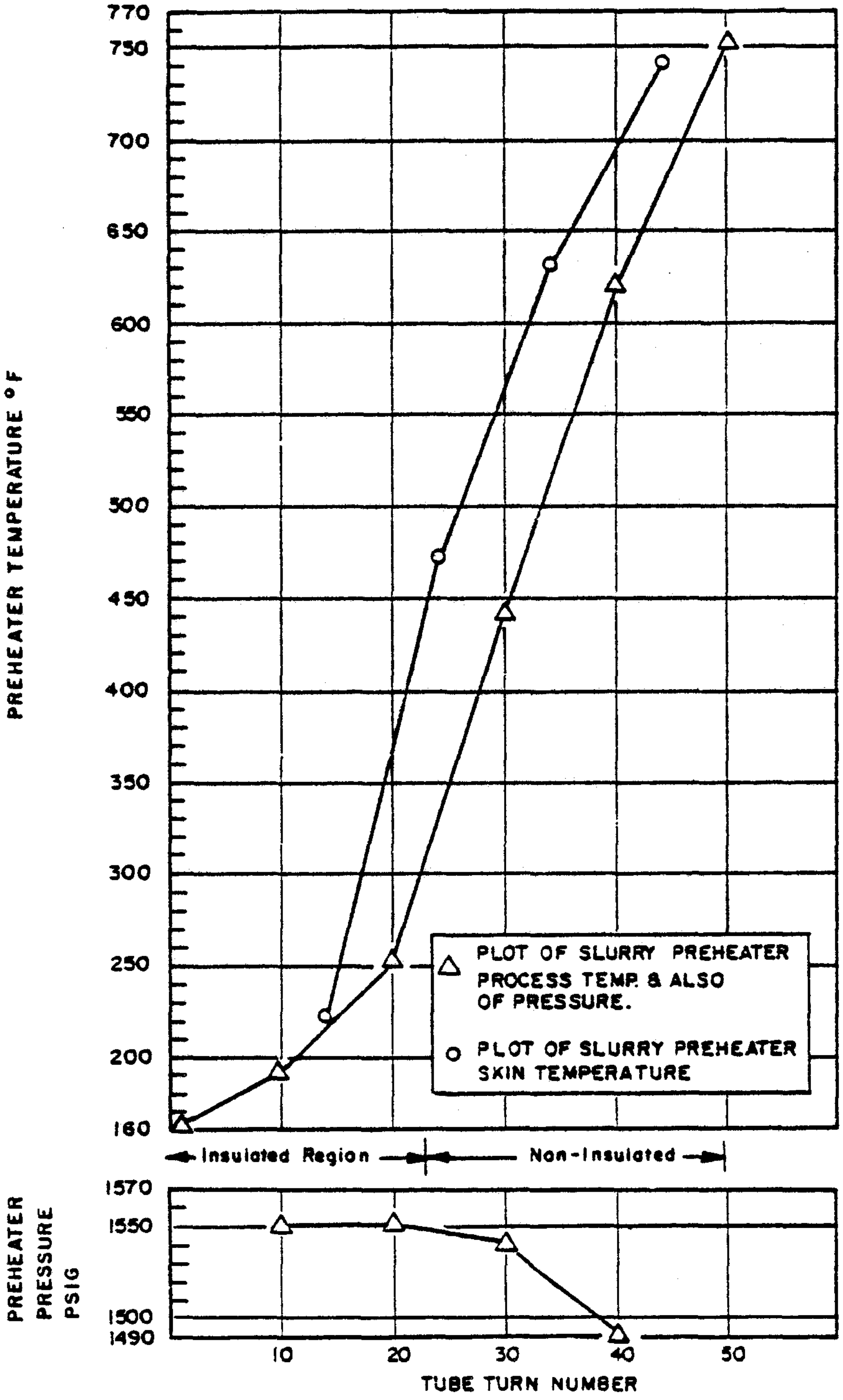
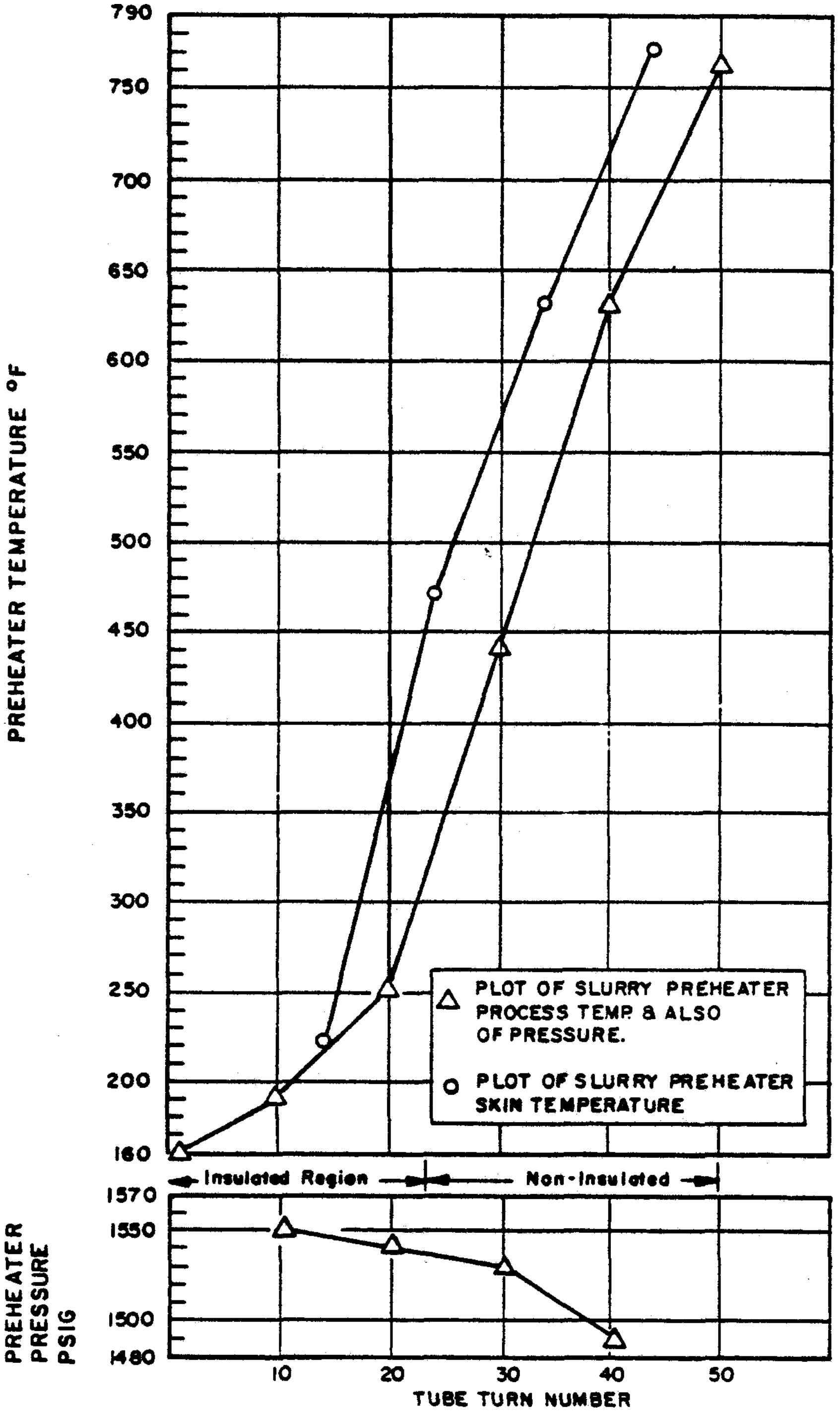


FIGURE 42



SRC-I
SLURRY PREHEATER TEMPERATURE &
PRESSURE PROFILE
MATERIAL BALANCE RUN NUMBER 9

FIGURE 43

SRC-I
SLURRY PREHEATER TEMPERATURE &
PRESSURE PROFILE
MATERIAL BALANCE RUN NUMBER 10

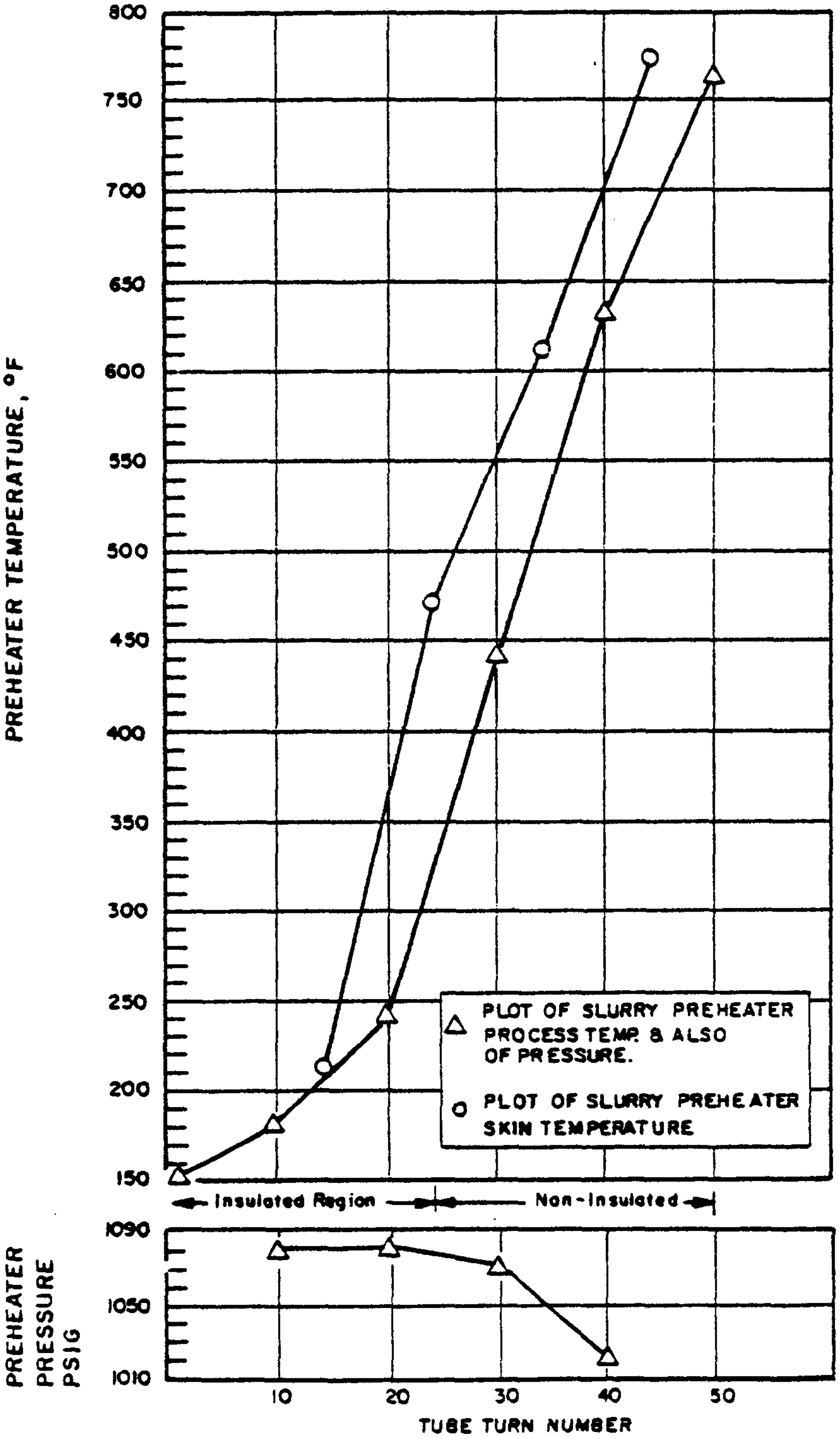


FIGURE 44

SRC-I
SLURRY PREHEATER TEMPERATURE & PRESSURE PROFILE
MATERIAL BALANCE RUN NUMBER II

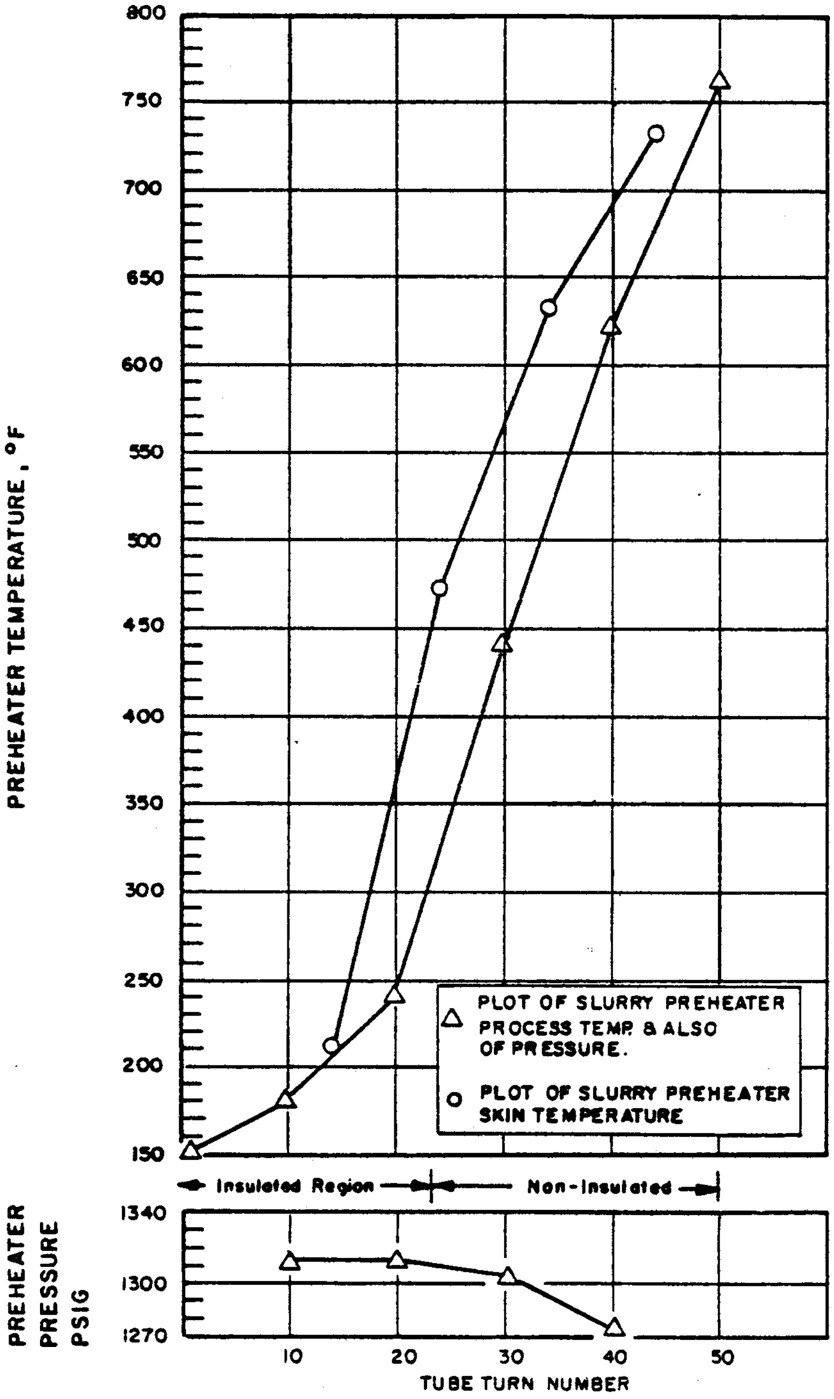


FIGURE 45

TABLE 27
SRC-I
MATERIAL BALANCE RUN NO. 1

Reaction Conditions Summary

3/7/75	1700 HOURS	TO	3/7/75	2200 HOURS
3/8/75	100 HOURS	TO	3/9/75	100 HOURS

RUN CONDITIONS

RAW COAL FEED	5042 #/HR
WATER REMOVED FROM COAL	419 #/HR
NET DEHUMIDIFIED COAL FEED	4624 #/HR
MOISTURE FREE COAL FEED	4551 #/HR
SOLVENT FEED	7665 #/HR
SLURRY FEED	12289 #/HR
SOLVENT TO DEHUM. COAL RATIO	1.66
HYDROGEN-RICH GAS FEED	281 #/HR
GAS FEED PURITY	94.8 MOL. PCT. H_2
HYDROGEN FEED	172.8 #/HR
H2 FEED RATIO, M.F. COAL BASIS	14001 SCF H2/TON COAL
SLURRY PREHEATER H2INLET PARTIAL PRESSURE	1461 PSIG
HYDROGEN CONSUMPTION	114 #/HR
HYDROGEN CONSUMPTION, M.F. COAL BASIS	2.5 WT. % OF COAL
HYDROGEN CONSUMPTION, M.F. COAL BASIS	9 MSCF/TON COAL
SLURRY PREHEATER OUTLET TEMP.	752 DEG F
DISSOLVER A PRESSURE	1528 PSIG
DISSOLVER OUTLET TEMPERATURE	851 DEG F
AVERAGE DISSOLVER TEMP	842 DEG F

DISSOLVER TEMPERATURE PROFILE:

Feet From Inlet	0'	4'	8'	12'	16'	20'	24'	28'
Degrees F.	747	828	835	839	843	847	849	851

NO. OF DISSOLVERS	1

TABLE 28

SRC-I

MATERIAL BALANCE RUN NO. 2

Reaction Conditions Summary

4/8/75 0400 HOURS TO 4/11/75 0400 HOURS

RUN CONDITIONS

RAW COAL FEED--2896 #/HR
WATER REMOVED FROM COAL---------------------------------- 193 #/HR
NET DEHUMIDIFIED COAL FEED------------------------------2703 #/HR
MOISTURE FREE COAL FEED---------------------------------2666 #/HR
SOLVENT FEED--4272 #/HR
SLURRY FEED---6975 #/HR
SOLVENT TO DEHUM. COAL RATIO--------------------------- 1.58
HYDROGEN-RICH GAS FEED----------------------------------195 #/HR
GAS FEED PURITY-- 97.1 MOL. PCT. H_2
HYDROGEN FEED-- 148 #/HR
H2 FEED RATIO, M.F. COAL BASIS------------------------20911 SCF H2/TON COAL
SLURRY PREHEATER INLET PRESSURE-------------------------1685 PSIG
HYDROGEN CONSUMPTION----------------------------------- 73.3 #/HR
HYDROGEN CONSUMPTION, M.F. COAL BASIS------------------ 2.7 WT. % OF COAL
HYDROGEN CONSUMPTION, M.F. COAL BASIS------------------ 13.8 MSCF/TON COAL
SLURRY PREHEATER OUTLET TEMP.---------------------------705 DEG F
DISSOLVER A PRESSURE-----------------------------------1489 DEG F
DISSOLVER OUTLET TEMPERATURE--------------------------- 848 DEG F
AVERAGE DISSOLVER TEMP.-------------------------------- 837 DEG F
DISSOLVER TEMPERATURE PROFILE:

Feet From Inlet	0'	4'	8'	12'	16'	20'	24'	28'
Degrees F.	685	823	829	834	838	842	844	848

NO. OF DISSOLVERS------------------------------------ 1

TABLE 29
SRC-I
MATERIAL BALANCE RUN NO. 3
Reaction Conditions Summary

6-9-75 0200 HOURS TO 6-10-75 0600 HOURS

RUN CONDITIONS

RAW COAL FEED	4112.	#/HR
WATER REMOVED FROM COAL	245.	#/HR
NET DEHUMIDIFIED COAL FEED	3867.	#/HR
MOISTURE FREE COAL FEED	3821.	#/HR
SOLVENT FEED	6378.	#/HR
SLURRY FEED	10245.	#/HR
SOLVENT TO DEHUM. COAL RATIO	1.65	
HYDROGEN-RICH GAS FEED	332.	#/HR
GAS FEED PURITY	93.5	MOL. PCT. H_2
HYDROGEN FEED	185.	#/HR
H_2 FEED RATIO, M. F. COAL BASIS	18227.	SCF H_2/TON COAL
SLURRY PREHEATER INLET PRESSURE	1594.	PSIG
HYDROGEN CONSUMPTION	80.2	#/HR
HYDROGEN CONSUMPTION, M. F. COAL BASIS	2.1	WT. % OF COAL
HYDROGEN CONSUMPTION, M. F. COAL BASIS	7.9	MSCF/TON COAL
SLURRY PREHEATER OUTLET TEMPERATURE	766.	DEG F
DISSOLVER A PRESSURE	1496.	PSIG
DISSOLVER OUTLET TEMPERATURE	870.	DEG F
AVERAGE DISSOLVER TEMPERATURE	863.	DEG F

DISSOLVER TEMPERATURE PROFILE:

Feet From Inlet	0'	4'	8'	12'
Degrees F.	762	857	862	870

NO. OF DISSOLVERS	1/2

TABLE 30
SRC-I
MATERIAL BALANCE RUN NO. 4
Reaction Conditions Summary

6-12-75 1000 HOURS TO 6-14-75 1800 HOURS

RUN CONDITIONS

RAW COAL FEED	4184.	#/HR
WATER REMOVED FROM COAL	167.	#/HR
NET DEHUMIDIFIED COAL FEED	4018.	#/HR
MOISTURE FREE COAL FEED	3959.	#/HR
SOLVENT FEED	6310.	#/HR
SLURRY FEED	10328.	#/HR
SOLVENT TO DEHUM. COAL RATIO	1.57	
HYDROGEN-RICH GAS FEED	326.	#/HR
GAS FEED PURITY	93.8	MOL. PCT. H_2
HYDROGEN FEED	186.	#/HR
H_2 FEED RATIO, M. F. COAL BASIS	17705.	SCF H_2/TON COAL
SLURRY PREHEATER INLET PRESSURE	1604.	PSIG
HYDROGEN CONSUMPTION	80.	#HR
HYDROGEN CONSUMPTION, M. F. COAL BASIS	2.0	WT. % OF COAL
HYDROGEN CONSUMPTION, M. F. COAL BASIS	7.6	MSCF/TON COAL
SLURRY PREHEATER OUTLET TEMPERATURE	750.	DEG F
DISSOLVER A PRESSURE	1499.	PSIG
DISSOLVER OUTLET TEMPERATURE	844.	DEG F
AVERAGE DISSOLVER TEMPERATURE	833.	DEG F

DISSOLVER TEMPERATURE PROFILE:

Feet From Inlet	0'	4'	8'	12'
Degrees F.	743	825	829	844

NO. OF DISSOLVERS----------------------------------1/2

TABLE 31

SRC-I
MATERIAL BALANCE RUN NO. 5

Reaction Conditions Summary

6-25-75 0100 HOURS TO 6-27-75 1400 HOURS

RUN CONDITIONS

RAW COAL FEED	5211.	#/HR
WATER REMOVED FROM COAL	200.	#/HR
NET DEHUMIDIFIED COAL FEED	5011	#/HR
MOISTURE FREE COAL FEED	4956.	#/HR
SOLVENT FEED	8039.	#/HR
SLURRY FEED	13050.	#/HR
SOLVENT TO DEHUM. COAL RATIO	1.60	
HYDROGEN-RICH GAS FEED	368.	#/HR
GAS FEED PURITY	92.	MOL. PCT. H_2
HYDROGEN FEED	186.	#/HR
H_2 FEED RATIO, M. F. COAL BASIS	14160.	SCF H_2/TON COAL
SLURRY PREHEATER INLET PRESSURE	1557.	PSIG
HYDROGEN CONSUMPTION	99.6	#/HR
HYDROGEN CONSUMPTION, M. F. COAL BASIS	2.0	WT. % OF COAL
HYDROGEN CONSUMPTION, M. F. COAL BASIS	7.6	MSCF/TON COAL
SLURRY PREHEATER OUTLET TEMPERATURE	763.	DEG F
DISSOLVER A PRESSURE	1488.	PSIG
DISSOLVER OUTLET TEMPERATURE	849.	DEG F
AVERAGE DISSOLVER TEMPERATURE	843.	DEG F

DISSOLVER TEMPERATURE PROFILE:

Feet from Inlet	0'	4'	8'	12'
Degrees F.	754	837	842	849

NO. OF DISSOLVERS------------------------------1/2

TABLE 32

SRC-I
MATERIAL BALANCE RUN NO. 6

Reaction Conditions Summary

6-29-75 0100 HOURS — 7-2-75 0600 HOURS

<u>RUN CONDITIONS</u>

RAW COAL FEED	5220.	#/HR
WATER REMOVED FROM COAL	171.	#/HR
NET DEHUMIDIFIED COAL FEED	5049.	#/HR
MOISTURE FREE COAL FEED	5002.	#/HR
SOLVENT FEED	8032.	#/HR
SLURRY FEED	13081.	#/HR
SOLVENT TO DEHUM. COAL RATIO	1.59	
HYDROGEN-RICH GAS FEED	377.	#/HR
GAS FEED PURITY	90.7	MOL. PCT. H_2
HYDROGEN FEED	178.	#/HR
H_2 FEED RATIO, M. F. COAL BASIS	13375.	SCF H_2/TON COAL
SLURRY PREHEATER INLET PRESSURE	1559.	PSIG
HYDROGEN CONSUMPTION	100.9	#/HR
HYDROGEN CONSUMPTION, M. F. COAL BASIS	2.0	WT. % OF COAL
HYDROGEN CONSUMPTION, M. F. COAL BASIS	7.6	MSCF/TON COAL
SLURRY PREHEATER OUTLET TEMPERATURE	774.	DEG F
DISSOLVER A PRESSURE	1483.	PSIG
DISSOLVER OUTLET TEMPERATURE	870.	DEG F
AVERAGE DISSOLVER TEMPERATURE	864.	DEG F

DISSOLVER TEMPERATURE PROFILE:

Feet from Inlet	0'	4'	8'	12'
Degrees F.	765	858	863	870

NO. OF DISSOLVERS----------------------------------1/2

TABLE 33

SRC-I

MATERIAL BALANCE RUN NO. 7

Reaction Conditions Summary

7-3-75 1200 HOURS | 7-7-75 0600 HOURS

RUN CONDITIONS

RAW COAL FEED	5211.	#/HR
WATER REMOVED FROM COAL	157.	#/HR
NET DEHUMIDIFIED COAL FEED	5055.	#/HR
MOISTURE FREE COAL FEED	5020.	#/HR
SOLVENT FEED	8042.	#/HR
SLURRY FEED	13097.	#/HR
SOLVENT TO DEHUM. COAL RATIO	1.59	
HYDROGEN-RICH GAS FEED	360.	#/HR
GAS FEED PURITY	91.4	MOL. PCT. H_2
HYDROGEN FEED	175.	#/HR
H_2 FEED RATIO, M. F. COAL BASIS	13153.	SCF H_2/TON COAL
SLURRY PREHEATER INLET PRESSURE	1579.	PSIG
HYDROGEN CONSUMPTION	97.4	#/HR
HYDROGEN CONSUMPTION, M. F. COAL BASIS	1.9	WT. % OF COAL
HYDROGEN CONSUMPTION, M. F. COAL BASIS	7.3	MSCF/TON COAL
SLURRY PREHEATER OUTLET TEMPERATURE	758.	DEG F
DISSOLVER A PRESSURE	1471.	PSIG
DISSOLVER OUTLET TEMPERATURE	850.	DEG F
AVERAGE DISSOLVER TEMPERATURE	845.	DEG F

Feet From Inlet	0'	4'	8'	12'
Degrees F.	748	841	845	850

NO. OF DISSOLVERS	1/2

TABLE 34

SRC-I
MATERIAL BALANCE RUN NO. 8

Reaction Conditions Summary

7-12-75 0200 HOURS 7-14-75 0600 HOURS

RUN CONDITIONS

RAW COAL FEED	4202.	#/HR
WATER REMOVED FROM COAL	136.	#/HR
NET DEHUMIDIFIED COAL FEED	4066.	#/HR
MOISTURE FREE COAL FEED	4050.	#/HR
SOLVENT FEED	6306.	#/HR
SLURRY FEED	10371.	#/HR
SOLVENT TO DEHUM. COAL RATIO	1.55	
HYDROGEN-RICH GAS FEED	312.	#/HR
GAS FEED PURITY	94.1	MOL. PCT. H_2
HYDROGEN FEED	182.	#/HR
H_2 FEED RATIO, M. F. COAL BASIS	16951.	SCF H_2/TON COAL
SLURRY PREHEATER INLET PRESSURE	1560.	PSIG
HYDROGEN CONSUMPTION	95.7	#/HR
HYDROGEN CONSUMPTION, M. F. COAL BASIS	2.4	WT. % OF COAL
HYDROGEN CONSUMPTION, M. F. COAL BASIS	8.9	MSCF/TON COAL
SLURRY PREHEATER OUTLET TEMPERATURE	749.	DEG F
DISSOLVER A PRESSURE	1484.	PSIG
DISSOLVER OUTLET TEMPERATURE	846.	DEG F
AVERAGE DISSOLVER TEMPERATURE	837.	DEG F

DISSOLVER TEMPERATURE PROFILE:

Feet From Inlet	0'	4'	8'	12'
Degrees F.	739	831	834	846

NO. OF DISSOLVERS ------------------------------ 1/2

TABLE 35

SRC-I

MATERIAL BALANCE RUN NO. 9

Reaction Conditions Summary

7-15-75 0200 HOURS TO 8-18-75 900 HOURS

RUN CONDITIONS

RAW COAL FEED	4818.	#/HR
WATER REMOVED FROM COAL	161.	#/HR
NET DEHUMIDIFIED COAL FEED	4657.	#/HR
MOISTURE FREE COAL FEED	4625.	#/HR
SOLVENT FEED	7158.	#/HR
SLURRY FEED	11815.	#/HR
SOLVENT TO DEHUM. COAL RATIO	1.54	
HYDROGEN-RICH GAS FEED	345.	#/HR
GAS FEED PURITY	92.5	MOL. PCT. H_2
HYDROGEN FEED	183.	#/HR
H_2 FEED RATIO, M. F. COAL BASIS	14876.	SCF H_2 TON COAL
SLURRY PREHEATER INLET PRESSURE	1559.	PSIG
HYDROGEN CONSUMPTION	97.7	#/HR
HYDROGEN CONSUMPTION, M. F. COAL BASIS	2.1	WT. % OF COAL
HYDROGEN CONSUMPTION, M. F. COAL BASIS	7.9	MSCF/TON COAL
SLURRY PREHEATER OUTLET TEMP.	758.	DEG F
DISSOLVER A PRESSURE	1478.	PSIG
DISSOLVER OUTLET TEMPERATURE	859.	DEG F
AVERAGE DISSOLVER TEMPERATURE	855.	DEG F

DISSOLVER TEMPERATURE PROFILE:

Feet From Inlet	0'	4'	8'	12'
Degrees F.	749	851	855	859

NO. OF DISSOLVERS 1/2

TABLE 36

SRC-I
MATERIAL BALANCE RUN NO. 10

Reaction Conditions Summary

10-8-75 1900 HOURS TO 10-11-75 0800 HOURS

RUN CONDITIONS

RAW COAL FEED	3908.	#/HR
WATER REMOVED FROM COAL	173.	#/HR
NET DEHUMIDIFIED COAL FEED	3735.	#/HR
MOISTURE FREE COAL FEED	3699.	#/HR
SOLVENT FEED	6032.	#/HR
SLURRY FEED	9767.	#/HR
SOLVENT TO DEHUM. COAL RATIO	1.61	
HYDROGEN-RICH GAS FEED	315.	#/HR
GAS FEED PURITY	96.0	MOL. PCT. H_2
HYDROGEN FEED	211.	#/HR
H_2 FEED RATIO, M. F. COAL BASIS	21467.	SCF H_2/TON COAL
SLURRY PREHEATER INLET PRESSURE	1086.	PSIG
HYDROGEN CONSUMPTION	133.2	#/HR
HYDROGEN CONSUMPTION, M. F. COAL BASIS	3.6	WT. % OF COAL
HYDROGEN CONSUMPTION, M. F. COAL BASIS	13.6	MSCF/TON COAL
SLURRY PREHEATER OUTLET TEMPERATURE	765.	DEG F
DISSOLVER A PRESSURE	1027.	PSIG
DISSOLVER OUTLET TEMPERATURE	842.	DEG F
AVERAGE DISSOLVER TEMPERATURE	838.	DEG F

DISSOLVER TEMPERATURE PROFILE:

Feet From Inlet	0'	4'	8'	12'
Degrees F.	744	833	838	842

NO. OF DISSOLVERS ---------- 1/2

TABLE 37

SRC-I

MATERIAL BALANCE RUN NO. 11

Reaction Conditions Summary

10-14-75 1400 HOURS TO 10-17-75 1400 HOURS

RUN CONDITIONS

RAW COAL FEED	4041.	#/HR
WATER REMOVED FROM COAL	257.	#/HR
NET DEHUMIDIFIED COAL FEED	3784.	#/HR
MOISTURE FREE COAL FEED	3745.	#/HR
SOLVENT FEED	5999.	#/HR
SLURRY FEED	9783.	#/HR
SOLVENT TO DEHUM. COAL RATIO	1.59	
HYDROGEN-RICH GAS FEED	354.	#/HR
GAS FEED PURITY	94.2	MOL. PCT. H_2
HYDROGEN FEED	207.	#/HR
H_2 FEED RATIO, M. F. COAL BASIS	20837.	SCF H_2/TON COAL
SLURRY PREHEATER INLET PRESSURE	1313.	PSIG
HYDROGEN CONSUMPTION	108.6	#/HR
HYDROGEN CONSUMPTION, M. F. COAL BASIS	2.9	WT. % OF COAL
HYDROGEN CONSUMPTION, M. F. COAL BASIS	10.9	MSCF/TON COAL
SLURRY PREHEATER OUTLET TEMPERATURE	756.	DEG F
DISSOLVER A PRESSURE	1261.	PSIG
DISSOLVER OUTLET TEMPERATURE	848.	DEG F
AVERAGE DISSOLVER TEMPERATURE	844.	DEG F

DISSOLVER TEMPERATURE PROFILE:

Feet From Inlet	0'	4'	8'	12'
Degrees F.	738	841	844	848

NO. OF DISSOLVERS-----1/2

TABLE 38
SRC-I
MATERIAL BALANCE RUN NO.1

ELEMENTAL BALANCES

INPUT (POUNDS PER HOUR)

	C	H	N	S	O	ASH	TOTAL
M.F. Coal	3292.6	239.5	125.3	73.0	379.6	440.6	4550.6
Inert Gas	8.0	0.2	0.0	149.3	20.7	0	178.2
H2 Rich Gas	74.6	193.1	0.3	9.3	3.9	0	281.2
Total In	3375.2	432.8	125.6	231.6	404.2	440.6	5010.0

OUTPUT (POUNDS PER HOUR)

	C	H	N	S	O	ASH	TOTAL
Gas	297.4	137.0	85.4	158.7	68.4	0	746.9
Water	0	25.0	0	0	200.2	0	225.2
Lt. Oil	243.9	36.2	1.1	1.0	11.8	0	294.0
Wash Solvent	308.2	31.9	1.1	2.3	18.5	0	362.0
Process Solvent	5.4	0.4	0	0.1	0.1	0	6.0
SRC*	2587.6	167.0	23.1	58.9	115.6	423.7	3375.9
Total Out	3442.5	397.5	110.7	221.0	414.6	423.7	5010.0
OUTPUT MINUS INPUT	67.3	-35.3	-14.9	-10.6	+10.4	-16.9	0
ERROR AS % OF INPUT	2.0	-8.2	-11.9	-4.6	2.6	-3.8	0

* Includes Ash & Insoluble Organic Matter.

TABLE 39
SRC-I
MATERIAL BALANCE RUN NO. 2
ELEMENTAL BALANCES

INPUT (POUNDS PER HOUR)

	C	H	N	S	O	ASH	TOTAL
M. F. Coal	1939.5	141.0	40.3	92.5	214.9	237.8	2666.0
H2 Rich Gas	30.7	157.3	6.2	0.1	0.6	0	194.9
Total In	1970.2	298.3	46.5	92.6	215.5	237.8	2860.9

OUTPUT (POUNDS PER HOUR)

	C	H	N	S	O	ASH	TOTAL
Gas	155.6	117.4	6.8	31.9	25.6	0	337.3
Water	0	15.7	0	0	125.6	0	141.3
Lt. Oil	226.6	27.4	1.7	0.7	15.0	0	271.4
Wash Solvent	261.2	26.3	2.1	0.6	13.9	0	304.1
Process Solvent	-92.8	-7.5	-1.5	-0.6	-1.5	0	-103.9
SRC *	1456.1	94.2	35.9	43.4	36.9	244.2	1910.7
Total Out	2006.7	273.5	45.0	76.0	215.5	244.2	2860.9
OUTPUT MINUS INPUT	36.5	-24.8	-1.5	-16.6	0	6.4	0
ERROR AS % OF INPUT	1.85	-8.3	-3.2	-17.9	0	2.7	0

* Includes Ash & Insoluble Organic Matter.

TABLE 40
SRC-I
MATERIAL BALANCE RUN NO. 3

ELEMENTAL BALANCES

INPUT (POUNDS PER HOUR)

	C	H	N	S	O	ASH	TOTAL
M. F. Coal	2719.8	200.6	58.5	118.1	379.4	344.7	3821.1
H_2 Rich Gas	96.9	210.6	9.8	0.0	14.4	0.0	331.7
Total In	2816.7	411.2	68.3	118.1	393.8	344.7	4152.8

OUTPUT (POUNDS PER HOUR)

	C	H	N	S	O	ASH	TOTAL
Gas	315.3	191.2	10.4	46.9	49.9	0.0	613.7
Water	0.0	31.2	0.0	0.0	249.6	0.0	280.8
Lt. Oil	510.9	63.1	3.0	2.8	39.2	0.0	619.0
Wash Solvent	-97.5	-10.7	-0.6	-0.3	-8.6	0.0	-117.7
Process Solvent	-129.8	-11.1	-1.3	-0.7	-7.4	0.0	-150.3
SRC*	2230.7	132.3	55.2	52.0	92.4	344.7	2907.3
Total Out	2829.6	396.0	66.7	100.7	415.1	344.7	4152.8
OUTPUT MINUS INPUT, LBS	12.9	-15.2	-1.6	-17.4	21.3	0.0	0.0
ERROR AS % OF INPUT	0.5	-3.7	-2.3	-14.7	5.4	0.0	0.0

* Includes Ash & Insoluble Organic Matter.

TABLE 41

MATERIAL BALANCE RUN NO. 4

ELEMENTAL BALANCES

INPUT (POUNDS PER HOUR)

	C	H	N	S	O	ASH	TOTAL
M. F. Coal	2858.3	206.3	61.0	139.4	324.2	369.8	3959.0
H_2 Rich Gas	90.9	209.0	10.3	0	16.2	0	326.4
Total In	2949.2	415.3	71.3	139.4	340.4	369.8	4285.4

OUTPUT (POUNDS PER HOUR)

	C	H	N	S	O	ASH	TOTAL
Gas	214.0	161.9	10.4	42.3	44.1	0	472.7
Water	0	21.1	0	0	167.6	0	188.7
Lt. Oil	137.9	17.1	0.9	0.9	12.8	0	169.6
Wash Solvent	172.7	17.5	1.0	0.6	10.4	0	202.2
Process Solvent	102.5	8.4	1.1	0.7	3.2	0	115.9
SRC *	2382.0	149.6	58.5	68.7	107.7	369.8	3136.3
Total Out	3009.1	375.6	71.9	113.2	345.8	369.8	4285.4
OUTPUT MINUS INPUT, LBS	59.9	-39.7	0.6	-26.2	5.4	0	0
ERROR AS % OF INPUT	2.0	-9.6	0.8	-18.8	1.6	0	0

* Includes Ash & Insoluble Organic Matter.

TABLE 42
SRC-I
MATERIAL BALANCE RUN NO. 5

ELEMENTAL BALANCES

INPUT (POUNDS PER HOUR)

	C	H	N	S	O	ASH	TOTAL
M. F. Coal	3515.3	252.3	79.3	154.1	500.1	455.0	4956.1
H_2 Rich Gas	119.7	217.6	12.7	0	18.4	0	368.4
Total In	3635.0	469.9	92.0	154.1	518.5	455.0	5324.5

OUTPUT (POUNDS PER HOUR)

	C	H	N	S	O	ASH	TOTAL
Gas	243.5	152.1	12.7	49.2	49.8	0	507.3
Water	0	31.5	0	0	252.0	0	283.5
Lt. Oil	105.8	14.0	0.5	0.8	6.6	0	127.7
Wash Solvent	293.3	30.6	1.8	0.9	30.8	0	357.4
Process Solvent	-53.9	-4.5	-0.6	-0.4	-1.6	0	-60.6
SRC*	3148.2	198.7	74.8	88.4	136.4	462.7	4109.2
Total Out	3736.9	422.4	89.2	138.9	474.0	462.7	5324.5
OUTPUT MINUS INPUT, LBS	101.9	-47.5	-2.8	-15.2	-44.5	7.7	0
ERROR AS % OF INPUT	2.8	-10.1	-3.0	-9.9	-8.6	1.7	0

* Includes Ash & Insoluble Organic Matter.

TABLE 43
SRC-I
MATERIAL BALANCE RUN NO. 6

ELEMENTAL BALANCES

INPUT (POUNDS PER HOUR)

	C	H	N	S	O	ASH	TOTAL
M. F. Coal	3598.7	258.5	78.7	182.2	398.8	485.1	5002.0
H_2 Rich Gas	134.8	213.8	11.4	0	16.9	0	376.9
Total In	3733.5	472.3	90.1	182.2	415.7	485.1	5378.9

<u>OUTPUT</u> (POUNDS PER HOUR)

	C	H	N	S	O	ASH	TOTAL
Gas	339.2	168.8	12.9	63.4	60.0	0	644.3
Water	0	29.6	0	0	236.8	0	266.4
Lt. Oil	172.0	23.8	0.7	1.2	8.6	0	206.3
Wash Solvent	220.0	23.3	1.2	0.7	15.3	0	260.5
Process Solvent	-113.0	-9.5	-1.2	-0.7	-4.2	0	-128.6
SRC*	3163.2	194.1	76.4	91.3	101.6	503.4	4130.0
Total Out	3781.4	430.1	90.0	155.9	418.1	503.4	5378.9
OUTPUT MINUS INPUT, LBS	47.9	-42.2	-0.1	-26.3	2.4	18.3	0
ERROR AS % OF INPUT	1.3	-8.93	-0.1	-14.4	0.6	3.8	0

* Includes Ash & Insoluble Organic Matter.

TABLE 44
SRC-I
MATERIAL BALANCE RUN NO. 7

ELEMENTAL BALANCES

INPUT (POUNDS PER HOUR)

	C	H	N	S	O	ASH	TOTAL
M. F. Coal	3606.4	270.1	79.8	197.8	391.6	474.4	5020.1
H_2 Rich Gas	122.4	207.4	12.0	0.7	17.9	-	360.4
Total In	3728.8	477.5	91.8	198.5	409.5	474.4	5380.5

OUTPUT (POUNDS PER HOUR)

	C	H	N	S	O	ASH	TOTAL
Gas	282.3	155.4	13.8	85.6	51.4	0	588.5
Water	0	30.9	0	0	247.5	0	278.4
Lt. Oil	153.5	21.7	0.7	1.0	7.8	0	184.7
Wash Solvent	215.4	27.9	1.4	0.9	21.5	0	317.1
Process Solvent	-52.2	-4.3	-0.5	-0.3	-1.6	0	-58.9
SRC*	3087.2	202.7	72.9	107.9	106.2	493.8	4070.7
Total Out	3736.2	434.3	88.3	195.1	432.8	493.8	5380.5
OUTPUT MINUS INPUT, LBS	7.4	-43.2	-3.5	-3.4	23.3	19.4	0
ERROR AS % OF INPUT	0.2	-9.0	-3.8	-1.7	5.7	4.1	0

* Includes Ash & Insoluble Organic Matter.

TABLE 45
SRC-I
MATERIAL BALANCE RUN NO. 8

ELEMENTAL BALANCES

INPUT (POUNDS PER HOUR)

	C	H	N	S	O	ASH	TOTAL
M. F. Coal	2894.5	205.3	63.6	146.2	345.1	395.3	4050.0
H_2 Rich Gas	85.7	205.0	10.0	0.0	10.9	0	311.6
Total In	2980.2	410.3	73.6	146.2	356.0	395.3	4361.6

OUTPUT (POUNDS PER HOUR)

	C	H	N	S	O	ASH	TOTAL
Gas	211.7	144.2	9.6	54.8	34.3	0	454.6
Water	0	23.0	0	0	184.2	0	207.2
Lt. Oil	170.7	24.0	0.8	1.2	7.6	0	204.3
Wash Solvent	136.3	14.5	0.8	0.6	9.0	0	161.2
Process Solvent	158.5	13.4	1.7	0.9	4.5	0	179.0
SRC *	2368.3	149.6	59.3	73.5	115.2	389.4	3155.3
Total Out	3045.5	368.7	72.2	131.0	354.8	389.4	4361.6
OUTPUT MINUS INPUT, LBS	65.3	-41.6	-1.4	-15.2	-1.2	-5.9	0
ERROR AS % OF INPUT	2.2	-10.1	-1.9	-10.4	-0.3	-1.5	0

* Includes Ash & Insoluble Organic Matter.

TABLE 46
SRC-I
MATERIAL BALANCE RUN NO. 9

ELEMENTAL BALANCES

INPUT (POUNDS PER HOUR)

	C	H	N	S	O	ASH	TOTAL
M. F. Coal	3349.8	241.0	72.6	172.5	358.0	431.1	4625.0
H_2 Rich Gas	112.9	213.4	3.5	0	11.2	0	345.0
Total In	3462.7	454.4	80.1	172.5	369.2	431.1	4970.0

OUTPUT (POUNDS PER HOUR)

	C	H	N	S	O	ASH	TOTAL
Gas	307.2	168.0	8.2	56.0	49.1	0	588.5
Water	0	25.9	0	0	207.0	0	232.9
Lt. Oil	190.7	26.7	0.8	0.9	14.8	0	233.9
Wash Solvent	195.5	20.8	1.4	0.6	15.8	0	234.1
Process Solvent	-8.3	-0.7	-0.1	0.0	-0.3	0	-9.4
SRC*	2808.6	175.6	71.2	84.1	101.1	449.4	3600.0
Total Out	3493.7	416.3	81.5	141.6	387.5	449.4	4970.0
OUTPUT MINUS INPUT, LBS	31.0	-38.1	1.4	-30.9	18.3	18.3	0
ERROR AS % OF INPUT	0.9	-8.4	1.7	-17.9	5.0	4.2	0

* Includes Ash & Insoluble Organic Matter.

TABLE 47
SRC-I
MATERIAL BALANCE RUN NO. 10

ELEMENTAL BALANCES

INPUT (POUNDS PER HOUR)

	C	H	N	S	O	ASH	TOTAL
M. F. Coal	2614.0	189.0	54.0	159.0	295.0	388.0	3699.0
H_2 Rich Gas	59.0	232.2	9.8	0.3	22.7	0	324.0
Total In	2673.0	421.2	63.8	159.3	317.7	388.0	4023.0

OUTPUT (POUNDS PER HOUR)

	C	H	N	S	O	ASH	TOTAL
Gas	201.8	132.0	12.5	62.5	50.6	0	459.4
Water	0	25.8	0	0	204.4	0	230.2
Lt. Oil	45.9	7.0	0.2	0.2	1.1	0	54.4
Wash Solvent	207.3	22.0	1.3	0.5	13.0	0	244.1
Process Solvent	-39.6	-3.4	-0.4	-0.2	-1.4	0	-45.0
SRC*	2299.4	145.4	55.4	86.9	73.3	419.5	3079.9
Total Out	2714.8	328.8	69.0	149.9	341.0	419.5	4023.0
OUTPUT MINUS INPUT, LBS	41.8	-92.4	5.2	-9.4	23.3	31.5	0
ERROR AS % OF INPUT	1.6	-21.9	8.2	-5.9	7.3	8.1	0

* Includes Ash & Insoluble Organic Matter.

TABLE 48
SRC-I
MATERIAL BALANCE RUN NO. 11

ELEMENTAL BALANCES

INPUT (POUNDS PER HOUR)

	C	H	N	S	O	ASH	TOTAL
M. F. Coal	2645.0	191.0	58.0	142.0	319.0	390.0	3745.0
H_2 Rich Gas	90.3	228.1	11.0	0.5	24.4	0	354.3
Total In	2735.3	419.1	69.0	142.5	343.4	390.0	4099.3

OUTPUT (POUNDS PER HOUR)

	C	H	N	S	O	ASH	TOTAL
Gas	258.9	163.3	10.3	58.1	59.0	0	549.6
Water	0	29.3	0	0	232.5	0	261.8
Lt. Oil	175.8	25.2	1.7	1.0	5.5	0	209.2
Wash Solvent	74.5	8.0	1.3	0.2	4.6	0	88.6
Process Solvent	40.1	3.6	0.4	0.2	1.6	0	45.9
SRC*	2175.2	141.6	51.8	83.3	67.7	424.6	2944.2
Total Out	2724.5	371.0	65.5	142.8	370.9	424.6	4099.3
OUTPUT MINUS INPUT, LBS	-10.8	-48.1	-3.5	0.3	27.5	34.6	0
ERROR AS % OF INPUT	-0.4	-11.5	-5.1	0.2	8.0	8.9	0

* Includes Ash & Insoluble Organic Matter.

TABLE 49

SRC-I

MATERIAL BALANCE RUN NUMBER ONE

AVERAGED LABORATORY RESULTS

HIGH PRESSURE SEPARATOR BOTTOMS SAMPLE-POINT NO. 204

SLURRY ANALYSIS

% Pyridine Insolubles	5.1
% Ash in P. I. = 59.2	
% H_2O	3.0
% Light Oil	2.2
% Wash Solvent	0.5
% Process Solvent	63.0
% SRC	26.2
TOTAL	100.0
Liquid Collected, grams	504.6

FLASHED GAS ANALYSIS

Vol. %	
H_2	47.0
N_2	7.4
C_1	21.0
CO	0.6
C_2	5.3
CO_2	7.6
C_3	4.7
C_4	0.8
H_2S	5.6
TOTAL	100.0

Volume of Gas Recovered, liters 3.1

ELEMENTAL ANALYSES OF PLANT PRODUCED PRODUCTS

WEIGHT PERCENTS

TEST	MOISTURE FREE COAL FEED	LIGHT OIL POINT 408	WASH SOLVENT POINT 410	PROCESS SOLVENT POINT 411	SRC (FILTRATION SECTION BY PASSED)	AVERAGE LABORATORY PRODUCED SRC
C	72.35	82.97	85.14	88.79	72.96	-----
H	5.26	12.32	8.81	7.04	4.65	-----
N	1.61	0.35	0.63	0.99	1.77	-----
S	2.75	0.36	0.30	0.74	3.30	0.87
O	8.35	4.00	5.12	2.44	5.32	-----
Ash	9.68	-----	-----	-----	12.0	-----
TOTAL	100.0	100.0	100.0	100.0	100.0	

TABLE 50

SRC-I

MATERIAL BALANCE RUN NUMBER TWO

AVERAGED LABORATORY RESULTS

HIGH PRESSURE SEPARATOR BOTTOMS SAMPLE-POINT NO. 204

SLURRY ANALYSIS	
% Pyridine Insolubles	4.9
% Ash in P. I. = 64.7	
% H_2O	2.6
% Light Oil	1.1
% Wash Solvent	1.6
% Process Solvent	62.5
% SRC	27.3
TOTAL	100.0
Liquid Collected, grams	734.7

FLASHED GAS ANALYSIS	
Vol. %	
H_2	56.5
N_2	2.9
C_1	20.9
CO	0.4
C_2	8.0
CO_2	3.8
C_3	3.4
C_4	0.4
H_2S	3.7
TOTAL	100.0
Volume of Gas Recovered, liters	3.1

ELEMENTAL ANALYSES OF PLANT PRODUCED PRODUCTS

WEIGHT PERCENTS

TEST	MOISTURE FREE COAL FEED	LIGHT OIL POINT 408	WASH SOLVENT POINT 410	PROCESS SOLVENT POINT 411	SRC (FILTRATION SECTION BY PASSED)	AVERAGE LABORATORY PRODUCED SRC
C	72.75	83.52	85.91	89.38	76.21	-----
H	5.29	10.09	8.64	7.19	4.93	-----
N	1.51	0.62	0.69	1.34	1.88	-----
S	3.47	0.24	0.21	0.60	2.27	0.57
O	8.06	5.53	4.55	1.49	1.93	-----
Ash	8.92	-----	-----	-----	12.78	-----
TOTAL	100.0	100.0	100.0	100.0	100.0	

TABLE 31

SRC-I

MATERIAL BALANCE RUN NUMBER THREE

AVERAGED LABORATORY RESULTS

HIGH PRESSURE SEPARATOR BOTTOMS SAMPLE-POINT NO. 204

SLURRY ANALYSIS	
% Pyridine Insolubles	6.68
% Ash in P. I. = 57.55	
% H_2O	2.73
% Light Oil	1.08
% Wash Solvent	3.78
% Process Solvent	57.16
% SRC	28.57
TOTAL	100.0
Liquid Collected, grams	942.7

FLASHED GAS ANALYSIS	
Vol. %	
H_2	63.22
N_2	3.11
C_1	19.68
CO	0.10
C_2	4.54
CO_2	3.28
C_3	1.85
C_4	0.83
H_2S	3.39
TOTAL	100.0
Volume of Gas Recovered, liters	2.95

ELEMENTAL ANALYSES OF PLANT PRODUCED PRODUCTS

WEIGHT PERCENTS

TEST	MOISTURE FREE COAL FEED	LIGHT OIL POINT 408	WASH SOLVENT POINT 410	PROCESS SOLVENT POINT 411	SRC (FILTRATION SECTION BY PASSED)	AVERAGE LABORATORY PRODUCED SRC
C	71.20	82.53	82.80	86.39	76.72	88.42
H	5.24	10.20	9.06	7.38	4.55	5.22
N	1.53	0.48	0.54	0.88	1.90	2.18
S	3.09	0.45	0.29	0.44	1.79	0.72
O	9.92	6.34	7.31	4.91	3.18	3.35
Ash	8.02	--	--	--	11.86	0.11
TOTAL	100.0	100.0	100.0	100.0	100.0	

TABLE 52
SRC-I
MATERIAL BALANCE RUN NUMBER FOUR
AVERAGED LABORATORY RESULTS

SLURRY ANALYSIS	
% Pyridine Insolubles	6.31
% Ash in P. I. = 62.28	
% H_2O	1.40
% Light Oil	0.59
% Wash Solvent	1.09
% Process Solvent	60.06
% SRC	30.55
TOTAL	100.0
Liquid Collected, grams	890.2

FLASHED GAS ANALYSIS	
Vol. %	
H_2	73.04
N_2	0.14
C_1	12.99
CO	1.43
C_2	4.33
CO_2	2.72
C_3	2.23
C_4	1.14
H_2S	1.98
TOTAL	100.0
Volume of Gas Recovered, liters	3.85

ELEMENTAL ANALYSES OF PLANT PRODUCED PRODUCTS
WEIGHT PERCENTS

TEST	MOISTURE FREE COAL FEED	LIGHT OIL POINT 408	WASH SOLVENT POINT 410	PROCESS SOLVENT POINT 411	SRC (FILTRATION SECTION BY PASSED)	AVERAGE LABORATORY PRODUCED SRC
C	72.20	81.24	85.44	88.475	75.95	----
H	5.21	10.12	8.64	7.24	4.77	----
N	1.54	0.52	0.51	0.93	1.865	----
S	3.52	0.53	0.29	0.605	2.19	0.85
O	8.19	7.59	5.12	2.75	3.435	----
Ash	9.34	----	----	----	11.79	----
TOTAL	100.0	100.0	100.0	100.0	100.0	

TABLE 53

SRC-I

MATERIAL BALANCE RUN NUMBER FIVE

AVERAGED LABORATORY RESULTS

HIGH PRESSURE SEPARATOR BOTTOMS SAMPLE-POINT NO. 204

SLURRY ANALYSIS	
% Pyridine Insolubles	6.80
% Ash in P. I. = 60.87	
% H_2O	1.7
% Light Oil	0.7
% Wash Solvent	1.4
% Process Solvent	59.0
% SRC	30.4
TOTAL	100.0
Liquid Collected, grams	948.1

FLASHED GAS ANALYSIS	
Vol. %	
H_2	74.4
N_2	0.1
C_1	15.7
CO	0.3
C_2	3.9
CO_2	2.5
C_3	1.8
C_4	0.3
H_2S	1.0
TOTAL	100.0
Volume of Gas Recovered, liters	3.2

ELEMENTAL ANALYSES OF PLANT PRODUCED PRODUCTS

WEIGHT PERCENTS

TEST	MOISTURE FREE COAL FEED	LIGHT OIL POINT 408	WASH SOLVENT POINT 410	PROCESS SOLVENT POINT 411	SRC (FILTRATION SECTION BY PASSED)	AVERAGE LABORATORY PRODUCED SRC
C	70.93	82.84	82.04	88.26	76.61	----
H	5.09	10.95	8.56	7.43	4.84	----
N	1.60	0.39	0.51	0.97	1.82	----
S	3.11	0.66	0.26	0.70	2.15	0.88
O	10.09	5.16	8.63	2.64	3.32	----
Ash	9.18	----	----	----	11.26	----
TOTAL	100.0	100.0	100.0	100.0	100.0	

TABLE 54

SRC-I

MATERIAL BALANCE RUN NUMBER SIX

AVERAGED LABORATORY RESULTS

HIGH PRESSURE SEPARATOR BOTTOMS SAMPLE-POINT NO. 204

SLURRY ANALYSIS

% Pyridine Insolubles	8.58
% Ash in P. I. = 60.26	
% H_2O	2.25
% Light Oil	0.98
% Wash Solvent	2.58
% Process Solvent	57.75
% SRC	27.81
TOTAL	100.0
Liquid Collected, grams	848.3

FLASHED GAS ANALYSIS

Vol. %	
H_2	61.53
N_2	0.41
C_1	19.36
CO	0.15
C_2	6.76
CO_2	3.82
C_3	3.89
C_4	1.28
H_2S	2.80
TOTAL	100.0

Volume of Gas Recovered, liters 3.25

ELEMENTAL ANALYSES OF PLANT PRODUCED PRODUCTS

WEIGHT PERCENTS

TEST	MOISTURE FREE COAL FEED	LIGHT OIL POINT 408	WASH SOLVENT POINT 410	PROCESS SOLVENT POINT 411	SRC (FILTRATION SECTION BY PASSED)	AVERAGE LABORATORY PRODUCED SRC
C	71.94	83.35	84.46	87.37	76.59	----
H	5.17	11.56	8.94	7.43	4.70	----
N	1.57	0.35	0.46	0.97	1.85	----
S	3.64	0.58	0.26	0.75	2.21	0.78
O	7.98	4.16	5.88	3.48	2.46	----
Ash	9.70	----	----	----	12.19	----
TOTAL	100.0	100.0	100.0	100.0	100.0	

TABLE 55
SRC-I
MATERIAL BALANCE RUN NUMBER SEVEN
AVERAGED LABORATORY RESULTS

HIGH PRESSURE SEPARATOR BOTTOMS SAMPLE-POINT NO. 204

SLURRY ANALYSIS

% Pyridine Insolubles	7.05
% Ash in P. I. = 63.43	
% H_2O	3.35
% Light Oil	0.73
% Wash Solvent	2.67
% Process Solvent	58.68
% SRC	27.52
TOTAL	100.0
Liquid Collected, grams	860.7

FLASHED GAS ANALYSIS

Vol. %	
H_2	53.81
N_2	1.16
C_1	21.17
CO	0.31
C_2	7.92
CO_2	5.01
C_3	4.25
C_4	1.33
H_2S	5.04
TOTAL	100.0

Volume of Gas Recovered, liters 3.23

ELEMENTAL ANALYSES OF PLANT PRODUCED PRODUCTS
WEIGHT PERCENTS

TEST	MOISTURE FREE COAL FEED	LIGHT OIL POINT 408	WASH SOLVENT POINT 416	PROCESS SOLVENT POINT 414	SRC (FILTRATION SECTION BY PASSED)	AVERAGE LABORATORY PRODUCED SRC
C	71.84	83.14	83.68	88.47	75.84	----
H	5.38	11.74	8.81	7.27	4.98	----
N	1.59	0.37	0.45	0.92	1.79	----
S	3.94	0.52	0.22	0.58	2.65	0.77
O	7.80	4.23	6.79	2.76	2.61	----
Ash	9.45	----	----	----	12.13	0.16
TOTAL	100.0	100.0	100.0	100.0	100.0	

TABLE 56
SRC-I
MATERIAL BALANCE RUN NUMBER EIGHT
AVERAGED LABORATORY RESULTS

HIGH PRESSURE SEPARATOR BOTTOMS SAMPLE-POINT NO. 204

SLURRY ANALYSIS

% Pyridine Insolubles	8.16
% Ash in P. I. = 64.55	
% H_2O	2.39
% Light Oil	0.73
% Wash Solvent	2.58
% Process Solvent	58.60
% SRC	27.54
TOTAL	100.0
Liquid Collected, grams	845.0

FLASHED GAS ANALYSIS

Vol. %	
H_2	62.15
N_2	1.24
C_1	16.82
CO	0.19
C_2	6.31
CO_2	3.75
C_3	3.22
C_4	2.14
H_2S	4.18
TOTAL	100.0

Volume of Gas Recovered, liters 3.71

ELEMENTAL ANALYSES OF PLANT PRODUCED PRODUCTS
WEIGHT PERCENTS

TEST	MOISTURE FREE COAL FEED	LIGHT OIL POINT 408	WASH SOLVENT POINT 410	PROCESS SOLVENT POINT 411	SRC (FILTRATION SECTION BY PASSED)	AVERAGE LABORATORY PRODUCED SRC
C	71.47	83.57	84.57	88.53	75.06	----
H	5.07	11.75	9.01	7.49	4.74	----
N	1.57	0.39	0.50	0.95	1.88	----
S	3.61	0.58	0.35	0.49	2.33	0.66
O	8.52	3.71	5.57	2.54	3.65	----
Ash	9.76	----	----	----	12.34	----
TOTAL	100.0	100.0	100.0	100.0	100.0	

TABLE 57

SRC-I

MATERIAL BALANCE RUN NUMBER NINE

AVERAGED LABORATORY RESULTS

HIGH PRESSURE SEPARATOR BOTTOMS SAMPLE-POINT NO. 204

SLURRY ANALYSIS

% Pyridine Insolubles	8.55
% Ash in P. I. = 62.32	
% H_2O	2.73
% Light Oil	0.89
% Wash Solvent	5.71
% Process Solvent	55.66
% SRC	26.46
TOTAL	100.0
Liquid Collected, grams	857.5

FLASHED GAS ANALYSIS

Vol. %	
H_2	47.44
N_2	2.01
C_1	22.79
CO	0.24
C_2	9.65
CO_2	5.19
C_3	4.97
C_4	1.95
H_2S	5.76
TOTAL	100.0

Volume of Gas Recovered, liters 3.36

ELEMENTAL ANALYSES OF PLANT PRDUCED PRODUCTS

WEIGHT PERCENTS

TEST	MOISTURE FREE COAL FEED	LIGHT OIL POINT 408	WASH SOLVENT POINT 410	PROCESS SOLVENT POINT 411	SRC (FILTRATION SECTION BY PASSED)	AVERAGE LABORATORY PRODUCED SRC
C	72.42	81.57	83.47	87.45	76.11	----
H	5.21	11.40	8.90	7.69	4.76	----
N	1.58	0.35	0.59	0.67	1.93	----
S	3.73	0.39	0.27	0.52	2.35	0.70
O	7.74	6.29	6.77	3.37	1.46	----
Ash	9.32	----	----	----	13.39	0.11
TOTAL	100.0	100.0	100.0	100.0	100.0	

TABLE 58
SRC-I
MATERIAL BALANCE RUN NUMBER TEN
AVERAGED LABORATORY RESULTS

HIGH PRESSURE SEPARATOR BOTTOMS SAMPLE-POINT NO. 204

SLURRY ANALYSIS	
% Pyridine Insolubles	7.95
% Ash in P. I. = 64.28	
% H_2O	2.53
% Light Oil	0.81
% Wash Solvent	11.95
% Process Solvent	51.89
% SRC	24.87
TOTAL	100.0
Liquid Collected, grams	470.3

FLASHED GAS ANALYSIS	
Vol. %	
H_2	53.82
N_2	4.58
C_1	18.04
CO	2.18
C_2	5.38
CO_2	6.18
C_3	5.71
C_4	1.26
H_2S	2.85
TOTAL	100.0
Volume of Gas Recovered, liters	1.18

ELEMENTAL ANAYLSES OF PLANT PRODUCED PRODUCTS
WEIGHT PERCENTS

TEST	MOISTURE FREE COAL FEED	LIGHT OIL POINT 408	WASH SOLVENT POINT 410	PROCESS SOLVENT POINT 411	SRC (FILTRATION SECTION BY PASSED)	AVERAGE LABORATORY PRODUCED SRC
C	70.68	84.46	84.94	88.01	74.66	----
H	5.12	12.80	9.02	7.57	4.72	----
N	1.46	0.28	0.52	0.86	1.80	----
S	4.29	0.40	0.20	0.42	2.82	0.75
O	7.97	2.06	5.32	3.14	2.38	----
Ash	10.48	----	----	----	13.62	----
TOTAL	100.0	100.0	100.0	100.0	100.0	

TABLE 59
SRC-I
MATERIAL BALANCE RUN NUMBER ELEVEN
AVERAGED LABORATORY RESULTS

HIGH PRESSURE SEPARATOR BOTTOMS SAMPLE-POINT NO. 204

SLURRY ANALYSIS

% Pyridine Insolubles	7.46
% Ash in P. I. = 64.82	
% H_2O	1.44
% Light Oil	0.89
% Wash Solvent	10.93
% Process Solvent	53.51
% SRC	25.27
TOTAL	100.0
Liquid Collected, grams	454.5

FLASHED GAS ANALYSIS

Vol. %

H_2	72.14
N_2	0.45
C_1	14.36
CO	0.44
C_2	3.41
CO_2	3.91
C_3	1.97
C_4	0.56
H_2S	2.76
TOTAL	100.0

Volume of Gas Recovered, liters 1.36

ELEMENTAL ANALYSES OF PLANT PRODUCED PRODUCTS
WEIGHT PERCENTS

TEST	MOISTURE FREE COAL FEED	LIGHT OIL POINT 408	WASH SOLVENT POINT 410	PROCESS SOLVENT POINT 411	SRC (FILTRATION SECTION BY PASSED)	AVERAGE LABORATORY PRODUCED SRC
C	70.61	84.02	84.11	87.49	73.88	----
H	5.10	12.06	9.05	7.81	4.81	----
N	1.55	0.79	1.42	0.82	1.76	----
S	3.80	0.49	0.21	0.39	2.83	0.74
O	8.52	2.64	5.21	3.49	2.30	----
Ash	10.42	----	----	----	14.42	----
TOTAL	100.0	100.0	100.0	100.0	100.0	

TABLE 60

SRC-I

MATERIAL BALANCE RUN NO. 1

GAS BALANCE CALCULATIONS

	FT 276 FRESH H2			FT 258 05 VENT GAS			FT 2117 03 SEP. GAS			FT 170 RCY CN SEP OH		
	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR
H2	99.19	88.8	162.8	67.71	15.8	52.1	7.86	0.5	1.0	63.06	12.0	57.3
N2	0.29	3.6	6.6	0.82	2.7	8.8	60.38	55.4	109.7	0.75	2.0	9.4
C1	0.08	0.6	1.1	21.08	39.0	128.9	0.60	0.3	0.6	19.86	30.0	143.3
CO	0.22	2.7	4.9	1.03	3.3	11.0	0.16	0.1	0.3	0.95	2.5	12.0
C2	0.09	1.2	2.2	5.97	20.7	68.4	1.83	1.8	3.6	5.73	16.2	77.5
CO2	0.02	0.3	0.6	0.01	0.1	0.2	8.62	12.4	24.6	2.56	10.6	50.7
C3	0.05	0.9	1.6	2.60	13.3	43.8	4.60	6.6	13.2	2.50	10.4	49.5
IC4	0.02	0.6	1.1	0.13	0.9	3.0	0.73	1.4	2.8	0.13	0.7	3.3
NC4	0.02	0.6	1.1	0.54	3.6	11.9	3.63	6.9	13.7	0.52	2.8	13.5
H2S	0.00	0.1	0.1	0.07	0.3	0.9	10.40	11.6	23.0	3.91	12.5	59.9
C5+	0.02	0.7	1.3	0.05	0.4	1.3	1.19	2.9	5.8	0.05	0.3	1.7
TOTAL			183.4			330.3			198.3			478.1
MOL. WT.			2.26			8.66			30.56			10.63
SCFH			30830.45			14468.23			2460.38			17070.51

	FT 2134 RECYCLE GAS			FT 144 & FT 85 PREHEATER FEED			FT 2107 LT. OIL/H2O SEPT.		
	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR
H2	67.71	15.8	62.8	94.80	61.4	172.8	5.8	0.4	0.3
N2	0.82	2.7	10.6	0.37	3.3	9.3	62.2	55.9	39.6
C1	21.08	39.0	155.3	3.15	16.2	45.6	0.8	0.4	0.3
CO	1.03	3.3	13.3	0.25	2.2	6.2	0.2	0.1	0.1
C2	5.97	20.7	82.4	0.87	8.3	23.5	1.7	1.7	1.2
CO2	0.01	0.1	0.2	0.02	0.2	0.6	9.0	12.7	9.0
C3	2.60	13.3	52.7	0.44	6.3	17.6	4.6	6.5	4.6
IC4	0.13	0.9	3.6	0.02	0.4	1.2	0.8	1.4	1.0
NC4	0.54	3.6	14.3	0.05	0.9	2.5	3.8	7.1	5.0
H2S	0.07	0.3	1.0	0.01	0.1	0.3	10.1	11.0	7.8
C5+	0.05	0.4	1.6	0.02	0.5	1.5	1.2	2.8	2.0
TOTAL			397.8			281.1			70.9
MOL. WT.			8.66			3.12			31.20
SCFH			17432.55			34241.49			861.25

TABLE 61
SRC-I
MATERIAL BALANCE RUN NO. 2
GAS BALANCE CALCULATIONS

	FT 276 FRESH H2			FT 258 05 VENT GAS			FT 2117 03 SEP. GAS			FT 170 RCY CN SEP OH		
	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR
H2	98.79	89.6	138.5	76.55	21.8	46.6	56.36	7.3	5.1	67.70	14.1	39.8
N2	0.26	3.3	5.1	0.62	2.4	5.2	0.10	0.2	0.1	0.64	1.8	5.2
C1	0.93	6.7	10.3	14.83	33.6	71.7	10.78	11.1	7.8	17.17	28.4	80.1
CO	0.00	0.0	0.0	0.50	2.0	4.2	0.22	0.4	0.3	0.60	1.7	4.9
C2	0.00	0.0	0.0	4.23	18.0	38.4	8.54	16.5	11.5	5.02	15.6	43.9
CO2	0.02	0.3	0.5	0.04	0.3	0.5	2.88	8.1	5.7	2.18	9.9	28.0
C3	0.00	0.0	0.0	2.39	14.9	31.8	7.51	21.2	14.9	2.65	12.0	34.0
IC4	0.00	0.0	0.0	0.13	1.1	2.2	0.57	2.1	1.3	0.15	0.9	2.5
NC4	0.00	0.0	0.0	0.55	4.5	9.6	2.31	8.6	6.0	0.61	3.7	10.4
H2S	0.00	0.1	0.1	0.03	0.1	0.3	10.33	22.6	15.3	3.17	11.2	31.5
C5+	0.00	0.0	0.0	0.13	1.4	2.9	0.41	2.0	1.4	0.09	0.7	2.0
TOTAL			154.5			213.4			70.1			282.3
MOL.WT			2.23			7.08			15.61			9.70
SCFH			26337.16			11439.29			1701.58			11040.70

	FT 2134 RECYCLE GAS			FT 144 & FT 85 PREHEATER FEED			FT 2107 LT. OIL/H2O SEPT.		
	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR
H2	76.55	21.8	38.7	97.10	76.1	148.3	2.97	0.2	0.0
N2	0.62	2.4	4.3	0.29	3.2	6.2	73.56	66.7	0.7
C1	14.83	33.6	59.5	2.03	12.6	24.6	0.26	0.1	0.0
CO	0.50	2.0	3.5	0.02	0.2	0.4	0.22	0.2	0.0
C2	4.23	18.0	31.8	0.34	3.9	7.7	0.86	0.8	0.0
CO2	0.04	0.3	0.5	0.01	0.3	0.5	11.36	16.2	0.2
C3	2.39	14.9	26.4	0.18	3.0	5.9	2.87	4.1	0.0
IC4	0.13	1.1	1.9	0.00	0.0	0.0	0.31	0.6	0.0
NC4	0.55	4.5	8.0	0.03	0.6	1.1	2.15	4.0	0.0
H2S	0.03	0.1	0.2	0.00	0.0	0.1	4.62	5.1	0.1
C5+	0.13	1.4	2.4	0.00	0.0	0.0	0.83	2.0	0.0
TOTAL			177.2			194.8			1.0
MOL. WT.			7.08			2.58			30.92
SCFH			9492.81			28702.06			12.35

TABLE 62
SRC-I
MATERIAL BALANCE RUN NO. 3
GAS BALANCE CALCULATIONS

	FT 276 FRESH H2			FT 258 05 VENT GAS			FT 2117 03 SEP. GAS			FT 170 RCY CN SEP OH		
	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR
H2	98.86	86.6	146.7	76.26	22.1	64.3	59.38	8.2	6.7	74.21	19.1	101.4
N2	0.29	3.6	6.1	0.53	2.1	6.2	0.48	0.9	0.8	0.51	1.8	9.6
C1	0.11	0.8	1.3	15.22	35.0	102.0	10.87	11.9	9.8	14.65	29.9	159.0
CO	0.70	8.6	14.5	1.47	5.9	17.2	0.68	1.3	1.1	1.42	5.1	26.9
C2	0.00	0.0	0.0	3.72	16.0	46.7	7.46	15.3	12.6	3.61	13.8	73.5
CO2	0.02	0.5	0.8	0.07	0.5	1.3	3.19	9.5	7.9	1.50	7.3	38.7
C3	0.00	0.0	0.0	2.19	13.9	40.3	7.66	23.0	18.9	2.18	12.2	65.1
IC4	0.00	0.0	0.0	0.10	0.8	2.5	0.58	2.3	1.9	0.10	0.7	3.8
NC4	0.00	0.0	0.0	0.42	3.5	10.3	2.38	9.4	7.7	0.41	3.0	16.0
H2S	0.00	0.0	0.0	0.00	0.0	0.0	6.88	16.0	13.1	1.59	6.9	36.7
C5+	0.00	0.0	0.0	0.01	0.1	0.4	0.44	2.2	1.8	0.01	0.1	0.7
TOTAL			169.4			291.2			82.3			531.4
MOL. WT.			2.31			6.97			14.69			7.86
SCFH			27876.86			15851.59			2124.93			25669.91

	FT 2134 RECYCLE GAS			FT 144 & FT 85 PREHEATER FEED			FT 2107 LT. OIL / H2O SEPT.		
	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR
H2	76.26	22.1	59.4	93.48	55.9	185.4	2.29	0.1	0.1
N2	0.53	2.1	5.7	0.35	2.9	9.8	56.46	47.0	25.2
C1	15.22	35.0	94.1	3.83	18.2	60.3	0.79	0.4	0.2
CO	1.47	5.9	15.9	0.92	7.6	25.2	0.06	0.0	0.0
C2	3.72	16.0	43.1	0.84	7.5	24.8	3.24	2.9	1.6
CO2	0.07	0.5	1.2	0.02	0.2	0.8	11.26	14.7	7.9
C3	2.19	13.9	37.2	0.49	6.3	21.1	8.86	11.6	6.2
IC4	0.10	0.8	2.3	0.00	0.0	0.0	1.03	1.8	1.0
NC4	0.42	3.5	9.5	0.08	1.3	4.3	5.23	9.0	4.9
H2S	0.00	0.0	0.0	0.00	0.0	0.0	9.47	9.6	5.2
C5+	0.01	0.1	0.4	0.00	0.0	0.0	1.31	2.9	1.6
TOTAL			268.8			331.7			53.9
MOL. WT.			6.97			3.38			33.69
SCFH			14632.13			37253.81			605.63

TABLE 63

SRC-I

MATERIAL BALANCE RUN NO 4

GAS BALANCE CALCULATIONS

	FT 276 FRESH H2			FT 258 05 VENT GAS			FT 2117 03 SEP. GAS			FT 170 RCY CN SEP OH		
	MOL%	WT %	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR
H2	93.67	85.3	139.9	81.93	28.2	55.2	70.64	13.2	7.4	79.77	23.8	102.9
N2	0.28	3.3	5.5	0.58	2.8	5.4	0.10	0.3	0.1	0.56	2.3	10.0
C1	0.29	2.0	3.3	11.09	30.3	59.3	8.68	12.9	7.2	10.58	25.1	108.4
CO	0.73	8.7	14.3	1.51	7.2	14.1	0.85	2.2	1.2	1.49	6.2	26.6
C2	0.00	0.0	0.0	2.68	13.8	26.9	4.66	12.9	7.3	2.55	11.3	49.1
CO2	0.03	0.6	1.0	0.06	0.4	0.9	2.00	8.1	4.6	1.29	8.4	36.2
C3	0.00	0.0	0.0	1.71	12.8	25.1	4.77	19.4	10.9	1.61	10.5	45.5
IC4	0.00	0.0	0.0	0.08	0.8	1.6	0.28	1.5	0.9	0.08	0.7	3.0
NC4	0.00	0.0	0.0	0.35	3.5	6.8	1.45	7.8	4.4	0.37	3.2	13.7
H2S	0.00	0.0	0.0	0.00	0.0	0.0	6.30	19.8	11.1	1.69	8.5	36.7
C5+	0.00	0.0	0.0	0.01	0.1	0.2	0.27	1.9	1.0	0.01	0.1	0.4
TOTAL			164.0			195.5			56.1			432.5
MOL.WT.			2.34			5.86			10.82			6.77
SCFH			26642.14			12649.26			1966.16			24240.80

	FT2134 RECYCLE GAS			FT 144 & FT 85 PREHEATER FEED			FT 2107 LT. OIL/H2O SEPT.		
	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR
H2	81.93	28.2	54.7	93.83	57.1	186.5	5.59	0.4	0.2
N2	0.58	2.8	5.4	0.37	3.2	10.3	62.49	55.8	25.6
C1	11.09	30.3	58.7	3.52	17.0	55.6	0.87	0.4	0.2
CO	1.51	7.2	14.0	0.99	8.3	27.2	0.07	0.1	0.0
C2	2.68	13.8	26.7	0.73	6.6	21.7	1.61	1.5	0.7
CO2	0.06	0.4	0.9	0.02	0.3	0.9	10.58	14.8	6.8
C3	1.71	12.8	24.8	0.45	6.0	19.5	4.64	6.5	3.0
IC4	0.08	0.8	1.6	0.00	0.0	0.0	0.57	1.1	0.5
NC4	0.35	3.5	6.7	0.08	1.4	4.7	3.68	6.8	3.1
H2S	0.00	0.0	0.0	0.00	0.0	0.0	3.37	9.3	4.3
C5+	0.01	0.1	0.1	0.00	0.0	0.0	1.35	3.2	1.5
TOTAL			193.6			326.4			45.9
MOL. WT			5.86			3.32			31.36
SCFH			12537.68			37345.50			555.26

TABLE 64

SRC-I

MATERIAL BALANCE RUN NO. 5

GAS BALANCE CALCULATIONS

	FT 276 FRESH H2			FT 258 05 VENT GAS			FT 2117 03 SEP. GAS			FT 170 RCY CN SEP OH		
	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR
H2	98.37	83.2	137.9	75.82	21.9	50.9	69.50	12.9	10.2	72.98	18.1	84.2
N2	0.33	3.9	6.4	0.77	3.1	7.2	0.08	0.2	0.2	0.72	2.5	11.5
C1	0.51	3.4	5.7	15.30	35.1	81.6	10.51	15.5	12.3	14.72	28.9	134.8
CO	0.75	8.8	14.6	1.80	7.2	16.8	0.64	1.6	1.3	1.74	6.0	27.8
C2	0.00	0.0	0.0	3.94	17.0	39.4	4.53	12.6	9.9	3.80	14.0	65.2
CO2	0.04	0.8	1.3	0.08	0.5	1.2	2.25	9.1	7.2	1.70	9.2	42.8
C3	0.00	0.0	0.0	1.89	12.0	27.8	4.54	18.5	14.6	1.79	9.7	45.1
IC4	0.00	0.0	0.0	0.07	0.6	1.4	0.29	1.5	1.2	0.07	0.5	2.3
NC4	0.00	0.0	0.0	0.29	2.4	5.6	1.34	7.2	5.7	0.28	2.0	9.1
H2S	0.00	0.0	0.0	0.00	0.0	0.0	6.07	19.1	15.1	2.20	9.2	42.8
C5+	0.00	0.0	0.0	0.01	0.2	0.4	0.24	1.7	1.3	0.02	0.1	0.7
TOTAL			165.9			232.3			78.0			466.3
MOL. WT.			2.39			6.99			10.85			8.16
SCFH			26327.16			12612.82			2761.20			21669.09

	FT 2134 RECYCLE GAS			FT 144 & FT 85 PREHEATER FEED			FT 2107 LT. OIL/H2O SEPT.		
	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR
H2	75.82	21.9	61.1	91.96	50.7	186.8	5.17	0.3	0.2
N2	0.77	3.1	8.6	0.45	3.5	12.7	59.98	52.6	25.1
C1	15.30	35.1	98.0	4.81	21.1	77.5	0.69	0.3	0.2
CO	1.80	7.2	20.2	1.08	8.3	30.4	0.18	0.2	0.1
C2	3.94	17.0	47.3	1.10	9.0	33.2	2.41	2.3	1.1
CO2	0.08	0.5	1.4	0.03	0.4	1.4	8.22	11.3	5.4
C3	1.89	12.0	33.3	0.51	6.1	22.6	7.21	9.9	4.7
IC4	0:07	0.6	1.7	0.00	0.0	0.0	8.96	1.7	0.8
NC4	0.29	2.4	6.8	0.06	1.0	3.6	4.83	8.8	4.2
H2S	0.00	0.0	0.0	0.00	0.0	0.0	9.19	9.8	4.7
C5+	0.01	0.2	0.4	0.00	0.0	0.0	1.15	2.7	1.3
TOTAL			278.8			368.2			47.8
MOL. WT			6.99			3.66			31.96
SCFH			15146.76			38152.88			566.27

TABLE 65

SRC-I

MATERIAL BALANCE RUN NO. 6

GAS BALANCE CALCULATIONS

	FT 276 FRESH H2			FT 258 05 VENT GAS			FT 2117 03 SEP. GAS			FT 170 RCY CN SEP OH		
	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR
H2	98.52	84.0	143.5	67.59	16.0	52.1	58.04	8.0	9.4	64.55	13.2	74.1
N2	0.32	3.8	6.5	0.71	2.4	7.6	0.10	0.2	0.2	0.67	1.9	10.7
C1	0.37	2.5	4.3	21.68	40.9	132.7	12.38	13.5	15.9	20.68	33.7	188.5
CO	0.73	8.7	14.8	1.70	5.6	18.2	0.39	0.7	0.9	1.62	4.6	25.7
C2	0.00	0.0	0.0	5.28	18.7	60.6	7.62	15.6	18.3	5.10	15.6	87.1
CO2	0.06	1.0	1.8	0.15	0.8	2.4	3.00	9.0	10.6	2.05	9.1	51.2
C3	0.00	0.0	0.0	2.40	12.4	40.4	7.53	22.6	26.6	2.26	10.1	56.7
IC4	0.00	0.0	0.0	0.10	0.7	2.1	0.48	1.9	2.3	0.09	0.5	3.0
NC4	0.00	0.0	0.0	0.34	2.4	7.6	2.07	8.2	9.6	0.33	1.9	10.8
H2S	0.00	0.0	0.0	0.03	0.1	0.4	8.05	18.7	22.0	2.63	9.1	51.0
C5+	0.00	0.0	0.0	0.02	0.2	0.6	0.35	1.8	2.1	0.02	0.2	1.0
TOTAL			170.9			324.7			117.9			559.8
MOL.WT			2.37			8.51			14.71			9.85
SCFH			27364.16			14485.52			3036.59			21557.78

	FT 2134 RECYCLE GAS			FT 144 & FT 85 PREHEATER FEED			FT 2107 LT.OIL/H2O SEPT.		
	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR
H2	67.59	16.0	67.6	90.71	47.2	178.1	2.71	0.2	0.1
N2	0.71	2.4	9.9	0.42	3.0	11.4	57.28	47.8	25.0
C1	21.68	40.9	172.2	5.88	24.3	91.6	0.48	0.2	0.1
CO	1.70	5.6	23.6	1.00	7.2	27.1	0.11	0.1	0.0
C2	5.28	18.7	78.6	1.32	10.2	38.3	3.31	3.0	1.5
CO2	0.15	0.8	3.2	0.04	0.5	1.9	8.38	11.0	5.7
C3	2.40	12.4	52.3	0.57	6.5	24.6	9.61	12.6	6.6
IC4	0.10	0.7	2.8	0.00	0.0	0.0	1.24	2.1	1.1
NC4	0.34	2.4	9.9	0.07	1.0	3.7	5.87	10.2	5.3
H2S	0.03	0.1	0.5	0.00	0.0	0.0	9.65	9.8	5.1
C5+	0.02	0.2	0.8	0.00	0.0	0.0	1.35	3.0	1.6
TOTAL			421.4			376.9			52.1
MOL. WT.			8.51			3.88			33.57
SCFH			18789.77			36881.18			590.21

TABLE 66

SRC-I

MATERIAL BALANCE RUN NO. 7

GAS BALANCE CALCULATIONS

	FT 276 FRESH H2			FT 258 05 VENT GAS			FT 2117 03 SEP. GAS			FT 170 RCY CN SEP OH		
	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR
H2	98.54	84.0	138.7	73.11	19.7	44.6	57.34	8.1	8.3	69.90	15.9	75.4
N2	0.32	3.8	6.2	0.78	2.9	6.6	0.09	0.2	0.2	0.74	2.3	11.0
C1	0.36	2.4	4.0	17.43	37.3	84.5	14.94	16.8	17.2	16.47	29.8	141.0
CO	0.71	8.4	13.9	1.71	6.4	14.4	0.64	1.3	1.3	1.60	5.1	24.0
C2	0.00	0.0	0.0	4.36	17.5	39.6	6.21	13.1	13.4	4.17	14.2	67.0
CO2	0.07	1.4	2.2	0.11	0.7	1.5	2.78	8.6	8.8	1.78	8.8	41.7
C3	0.00	0.0	0.0	2.08	12.2	27.7	6.24	19.3	19.7	1.95	9.7	45.9
IC4	0.00	0.0	0.0	0.08	0.6	1.4	0.37	1.5	1.6	0.07	0.5	2.3
NC4	0.00	0.0	0.0	0.32	2.5	5.6	1.82	7.4	7.6	0.30	2.0	9.9
H2S	0.00	0.0	0.0	0.01	0.0	0.1	9.25	22.1	22.6	2.99	11.5	54.4
C5+	0.00	0.0	0.0	0.01	0.1	0.3	0.31	1.6	1.7	0.02	0.1	0.7
TOTAL			165.0			226.3			102.4			473.3
MOL. WT.			2.37			7.49			14.26			8.86
SCFH			26450.96			11468.14			719.28			20255.15

	FT 2134 RECYCLE GAS			FT 144 & FT 85 PREHEATER FEED			FT 2107 LT. OIL/H2O SEPT.		
	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR
H2	73.11	19.7	62.6	91.37	48.8	175.7	4.03	0.3	0.1
N2	0.78	2.9	9.3	0.45	3.3	12.0	62.08	54.0	26.9
C1	17.43	37.3	118.4	5.23	22.1	79.8	0.55	0.3	0.1
CO	1.71	6.4	20.2	1.01	7.5	27.0	0.05	0.0	0.0
C2	4.36	17.5	55.6	1.21	9.6	34.6	1.93	1.8	0.9
CO2	0.11	0.7	2.1	0.08	0.9	3.4	8.54	11.7	5.8
C3	2.08	12.2	38.8	0.55	6.4	23.2	6.49	8.9	4.4
IC4	0.08	0.6	1.9	0.00	0.0	0.2	0.81	1.5	0.7
NC4	0.32	2.5	7.9	0.07	1.1	3.8	4.77	8.6	4.3
H2S	0.01	0.0	0.2	0.02	0.2	0.7	9.45	10.0	5.0
C5+	0.01	0.1	0.5	0.00	0.0	0.0	1.30	3.0	1.5
TOTAL			317.5			360.4			49.7
MOL. WT.			7.49			3.79			32.22
SCFH			16077.45			36131.69			587.11

TABLE 67

SRC-I

MATERIAL BALANCE RUN NO. 8

GAS BALANCE CALCULATIONS

	FT 276 FRESH H2			FT 258 05 VENT GAS			FT 2117 03 SEP. GAS			FT 170 RCY CN SEP OH		
	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR
H2	99.23	90.3	139.4	80.45	26.7	50.6	66.87	11.6	8.0	77.71	22.0	84.1
N2	0.28	3.5	5.5	0.61	2.8	5.3	0.10	0.2	0.2	0.58	2.3	8.7
C1	0.06	0.4	0.7	12.30	32.5	61.4	11.01	15.2	10.5	11.94	26.9	102.6
CO	0.39	5.0	7.7	1.29	5.9	11.3	0.41	1.0	0.7	1.25	4.9	18.8
C2	0.00	0.0	0.0	3.27	16.20	30.6	5.02	13.0	8.9	3.20	13.5	51.6
CO2	0.03	0.6	1.0	0.08	0.6	1.1	1.83	6.9	4.8	1.13	7.0	26.5
C3	0.00	0.0	0.0	1.65	12.0	22.7	5.14	19.4	13.4	1.59	9.8	37.6
IC4	0.00	0.0	0.0	0.06	0.6	1.1	0.29	1.4	1.0	0.06	0.4	1.7
NC4	0.00	0.0	0.0	0.27	2.6	4.9	1.51	7.5	5.2	0.26	2.1	8.1
H2S	0.00	0.0	0.0	0.00	0.0	0.0	7.56	22.1	15.3	2.27	10.9	41.4
C5+	0.00	0.1	0.2	0.01	0.1	0.2	0.25	1.6	1.1	0.01	0.1	0.5
TOTAL			154.5			189.2			69.1			381.6
MOL. WT.			2.22			6.08			11.65			7.12
SCFH			26388.55			11810.38			2246.21			20320.22

	FT 2134 RECYCLE GAS			FT 144 & FT 85 PREHEATER FEED			FT 2107 LT. OIL/H2O SEPT.		
	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR
H2	80.45	26.7	53.9	94.10	58.6	182.7	4.47	0.3	0.1
N2	0.61	2.8	5.7	0.37	3.2	10.0	63.47	56.4	27.3
C1	12.30	32.5	65.4	3.49	17.2	53.7	0.46	0.2	0.1
CO	1.29	5.9	12.0	0.66	5.7	17.9	0.05	0.0	0.0
C2	3.27	16.2	32.6	0.88	8.1	25.3	1.89	1.8	0.9
CO2	0.08	0.6	1.2	0.02	0.3	1.0	9.04	12.6	6.1
C3	1.65	12.0	24.1	0.43	5.8	18.0	5.66	7.9	3.9
IC4	0.06	0.6	1.1	0.00	0.0	0.0	0.68	1.3	0.6
NC4	0.27	2.6	5.2	0.05	1.0	3.0	3.77	6.9	3.4
H2S	0.00	0.0	0.0	0.00	0.0	0.0	9.58	10.3	5.0
C5+	0.01	0.1	0.2	0.00	0.0	0.0	0.94	2.2	1.1
TOTAL			201.4			311.6			48.7
MOL. WT.			6.08			3.24			31.56
SCFH			12578.89			36479.25			586.29

TABLE 68

SRC-I

MATERIAL BALANCE RUN NO. 9

GAS BALANCE CALCULATIONS

	FT 276 FRESH H2			FT 258 05 VENT GAS			FT 2117 03 SEP. GAS			FT 170 RCY CN SEP OH		
	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR
H2	98.47	85.6	145.8	72.96	19.4	54.4	55.32	7.6	6.8	70.41	16.4	83.0
N2	0.23	2.8	4.8	0.41	1.5	4.2	0.09	0.2	0.2	0.39	1.2	6.3
C1	0.83	5.7	9.7	17.83	37.6	105.6	16.48	17.9	16.1	17.14	31.6	160.4
CO	0.42	5.0	8.6	1.44	5.3	14.9	0.48	0.9	0.8	1.38	4.5	22.6
C2	0.01	0.1	0.2	4.17	16.5	46.3	6.79	13.8	12.4	4.00	13.8	70.2
CO2	0.04	0.7	1.2	0.11	0.6	1.8	2.55	7.6	6.8	1.62	8.2	41.7
C3	0.00	0.0	0.0	2.48	14.4	40.4	7.02	20.9	18.8	2.35	11.9	60.4
IC4	0.00	0.0	0.0	0.11	0.8	2.3	0.43	1.7	1.5	0.11	0.7	3.6
NC4	0.00	0.0	0.0	0.46	3.5	9.9	2.09	8.2	7.4	0.46	3.0	15.5
H2S	0.00	0.0	0.0	0.00	0.0	0.1	8.38	19.3	17.4	2.13	8.8	42.3
C5+	0.00	0.0	0.0	0.03	0.3	0.7	0.37	1.9	1.7	0.03	0.2	1.2
TOTAL			170.3			280.6			89.9			507.2
MOL. WT.			2.32			7.60			14.78			8.69
SCFH			26820.92			14013.27			2306.16			22144.44

	FT 2134 RECYCLE GAS			FT 144 & FT 85 PREHEATER FEED			FT 2107 LT. OIL/H2O SEPT.		
	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR
H2	72.96	19.4	64.4	92.50	53.1	183.1	1.19	0.1	0.1
N2	0.41	1.5	5.0	0.27	2.2	7.5	72.87	64.1	46.6
C1	17.83	37.6	125.0	4.94	22.5	77.6	0.19	0.1	0.1
CO	1.44	5.3	17.7	0.66	5.2	18.0	0.03	0.0	0.0
C2	4.17	16.5	54.8	0.95	8.1	28.0	1.23	1.2	0.8
CO2	0.11	0.6	2.1	0.03	0.4	1.4	8.51	11.8	8.6
C3	2.48	14.4	46.8	0.56	7.1	24.4	4.40	6.1	4.4
IC4	0.11	0.8	2.7	0.00	0.0	0.1	0.55	1.0	0.7
NC4	0.46	3.5	11.7	0.08	1.4	4.8	3.40	6.2	4.5
H2S	0.00	0.0	0.1	0.00	0.0	0.0	6.67	7.1	5.2
C5+	0.03	0.3	0.9	0.00	0.0	0.0	0.97	2.3	1.7
TOTAL			331.2			344.9			72.7
MOL. WT.			7.60			3.52			31.83
SCFH			16584.83			37185.04			866.15

TABLE 69

SRC-I

MATERIAL BALANCE RUN NO. 10

GAS BALANCE CALCULATIONS

	FT 276 FRESH H2			FT 258 05 VENT GAS			FT 2117 03 SEP. GAS			FT 170 RCY CN SEP OH		
	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR
H2	98.63	83.6	140.8	79.53	24.5	47.9	25.74	2.3	2.1	78.05	22.0	86.2
N2	0.28	3.3	5.5	0.51	2.2	4.3	55.72	67.9	63.4	0.50	2.0	7.7
C1	0.00	0.0	0.0	11.47	28.1	54.9	2.42	1.7	1.6	11.22	25.2	98.4
CO	1.04	12.2	20.5	1.91	8.2	16.0	0.61	0.7	0.7	1.84	7.2	28.1
C2	0.00	0.0	0.0	3.03	13.9	27.2	2.14	2.3	2.6	2.99	12.5	49.1
CO2	0.05	0.9	1.5	0.85	5.7	11.1	7.43	14.2	13.3	1.64	10.1	39.3
C3	0.00	0.0	0.0	1.29	8.7	17.0	1.55	3.0	2.8	1.27	7.8	30.5
IC4	0.00	0.0	0.0	0.06	0.6	1.1	0.08	0.2	0.2	0.06	0.5	2.0
NC4	0.00	0.0	0.0	0.32	2.8	5.5	0.62	1.6	1.5	0.31	2.5	9.9
H2S	0.00	0.0	0.0	1.02	5.3	10.4	3.59	5.3	5.0	2.12	10.1	39.4
C5+	0.00	0.0	0.0	0.01	0.1	0.2	0.12	0.4	0.4	0.01	0.1	0.5
TOTAL			168.3			195.6			93.6			391.1
MOL. WT.			2.38			6.55			23.00			7.16
SCFH			26821.85			11326.56			1541.08			20746.24

	FT 2134 RECYCLE GAS			FT 144 & FT 85 PREHEATER FEED			FT 2475 I.G. TO STR			FT 2107 LT. OIL/H2O SEPT		
	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR
H2	79.53	24.5	29.8	96.03	67.0	210.9	0.17	0.0	0.0	2.16	0.1	0.1
N2	0.51	2.2	2.6	0.32	3.1	9.8	89.24	0.0	0.0	86.07	82.5	44.1
C1	11.47	28.1	34.1	1.74	9.6	30.3	0.00	0.0	0.0	0.06	0.0	0.0
CO	1.91	8.2	9.9	1.21	11.7	36.8	1.21	0.0	0.0	0.52	0.5	0.3
C2	3.03	13.9	16.9	0.44	4.6	14.5	0.07	0.0	0.0	0.30	0.3	0.2
CO2	0.85	5.7	6.9	0.05	0.7	2.3	9.17	0.0	0.0	9.83	14.8	7.9
C3	1.29	8.7	10.5	0.17	2.6	8.1	0.08	0.0	0.0	0.29	0.4	0.2
IC4	0.06	0.6	0.7	0.00	0.0	0.0	0.06	0.0	0.0	0.04	0.1	0.0
NC4	0.32	2.8	3.4	0.03	0.6	2.4	0.00	0.0	0.0	0.26	0.5	0.3
H2S	1.02	5.3	6.5	0.01	0.1	0.3	0.00	0.0	0.0	0.41	0.5	0.3
C5+	0.01	0.1	0.1	0.00	0.0	0.0	0.00	0.0	0.0	0.07	0.2	0.1
TOTAL			121.4			315.4			0.00			53.5
MOL. WT			6.55			2.90			29.47			29.22
SCFH			7037.06			41262.07			0.00			694.24

TABLE 70
SRC-I
MATERIAL BALANCE RUN NO. 11
GAS BALANCE CALCULATIONS

	FT 276 FRESH H2			FT 258 05 VENT GAS			FT 2117 03 SEP. GAS			FT 170 RCY CN SEP OH		
	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR
H2	98.53	82.6	172.8	80.16	26.6	50.1	46.81	5.4	5.9	77.72	22.1	102.1
N2	0.29	3.4	7.1	0.57	2.6	5.0	25.28	40.7	43.9	0.55	2.2	10.0
C1	0.00	0.0	0.0	12.37	32.6	61.5	6.31	5.8	6.3	11.92	27.0	124.4
CO	1.13	13.4	27.5	2.11	9.7	18.3	0.84	1.3	1.4	2.04	8.0	37.1
C2	0.00	0.0	0.0	3.01	14.9	28.1	6.68	8.1	8.7	2.99	12.7	58.5
CO2	0.04	0.8	1.7	0.04	0.3	0.5	4.55	11.0	11.9	1.52	9.4	43.4
C3	0.00	0.0	0.0	1.29	9.4	17.7	3.42	8.7	9.3	1.19	7.4	34.2
IC4	0.00	0.0	0.0	0.06	0.5	1.0	0.24	0.8	0.9	0.06	0.5	2.2
NC4	0.00	0.0	0.0	0.30	2.9	5.4	1.36	4.5	4.9	0.30	2.4	11.2
H2S	0.00	0.0	0.0	0.08	0.5	0.9	6.47	12.7	13.6	1.72	8.3	38.2
C5+	0.00	0.0	0.0	0.00	0.0	0.0	0.23	1.0	1.1	0.00	0.0	0.1
TOTAL			209.1			188.5			107.9			461.4
MOL. WT.			2.41			6.08			17.42			7.09
SCFH			32950.04			11751.30			2349.23			24683.38

	FT 2134 RECYCLE GAS			FT 144 & FT 85 PREHEATER FEED			FT 2475 I.G. TO STR			FT 2107 LT. OIL/H2O SEPT.		
	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR
H2	80.16	26.6	47.9	94.21	58.5	207.3	0.04	0.0	0.0	0.00	0.0	0.0
N2	0.57	2.6	4.7	0.36	3.1	11.0	84.51	0.0	0.0	89.00	84.1	41.9
C1	12.37	32.6	58.7	2.96	14.6	51.7	0.00	0.0	0.0	0.01	0.0	0.0
CO	2.11	9.7	17.5	1.40	12.0	42.7	1.47	0.0	0.0	0.39	0.4	0.2
C2	3.01	14.9	26.8	0.69	6.4	22.5	0.07	0.0	0.0	0.34	0.3	0.2
CO2	0.04	0.3	0.5	0.04	0.5	1.9	13.78	0.0	0.0	10.19	15.1	7.5
C3	1.29	9.4	16.9	0.28	3.7	13.2	0.08	0.0	0.0	0.07	0.1	0.0
IC4	0.06	0.5	1.0	0.00	0.0	0.0	0.06	0.0	0.0	0.00	0.0	0.0
NC4	0.30	2.9	5.1	0.05	1.0	3.4	0.00	0.0	0.0	0.00	0.0	0.0
H2S	0.08	0.5	0.8	0.01	0.2	0.5	0.00	0.0	0.0	0.0	0.0	0.0
C5+	0.00	0.0	0.0	0.00	0.0	0.0	0.00	0.0	0.0	0.0	0.0	0.0
TOTAL			179.9			354.2			0.00			49.8
MOL			6.08			3.25			30.25			29.67
SCFH			11226.60			41332.76			0.00			637.32

C. SRC II Material Balance Studies

1. Background

During the months of July and August 1975, five additional material balance tests were made. These tests, referred to as SR1-SR5, were conducted with the plant operating in the SRC II mode. In this mode a portion of the product slurry (UFCS) is recycled, mixed with fresh coal and solvent and then pumped back into the dissolver. The objective of the SRC II operation was to achieve additional conversion of solvent refined coal and unreacted material into distillate products.

Process yields from SRC I operation typically averaged 6% - 8% gas, 5% - 6% water, 7% - 12% total solvent, and 60% - 65% SRC. The SRC II process was developed as a means of reducing the yield of SRC and thus increasing the distillate yield. The obvious advantage of such a process was to develop a liquid product which could be transported and used in existing liquid fuel systems.

A schematic flow diagram for slurry recycle is shown in Figure 46. During an SRC II experiment, flow was diverted from the bottom of the intermediate pressure separator in area 02 to the slurry stripper. Here an inert gas stripping medium was used to remove a substantial portion of the light solvent range material. Part of the flow from the bottoms of the stripper was then diverted back to area 01 for coal slurry preparation.

With the introduction of slurry recycle, it became necessary to determine the mean residence time for the coal solution under reaction conditions in order to estimate the effect of residence time on reaction rate. A plug flow model was developed for flow through the dissolver and the estimated volume of liquid in the dissolver was calculated based upon the effective volume of gas in the dissolver. An estimated single pass slurry residence time (E) was then calculated, using the volumetric flow rate of the liquid and the liquid volume in the dissolver. The model was later tested experimentally using a temperature tracer technique and predicted and experimental results were in close agreement.

Since in slurry recycle operation a portion of the reactor effluent is returned to the dissolver, the effective slurry residence time is increased. The amount of this increase is proportional to the fraction of material recycled. This average residence time (θ) is defined as $\theta = \frac{1}{1-(R/F)} \times \bar{t}$ where R = recycle feed rate, F = total feed rate, and $\bar{t}$ = single pass residence time. The above equation suggests that at higher recycle ratios (R/F) the average residence time increases greatly over the single pass (SRC 1) residence time. The calculated single pass and average residence time for the five material balances are shown in Table 71.

SLURRY RECYCLE FLOW DIAGRAM

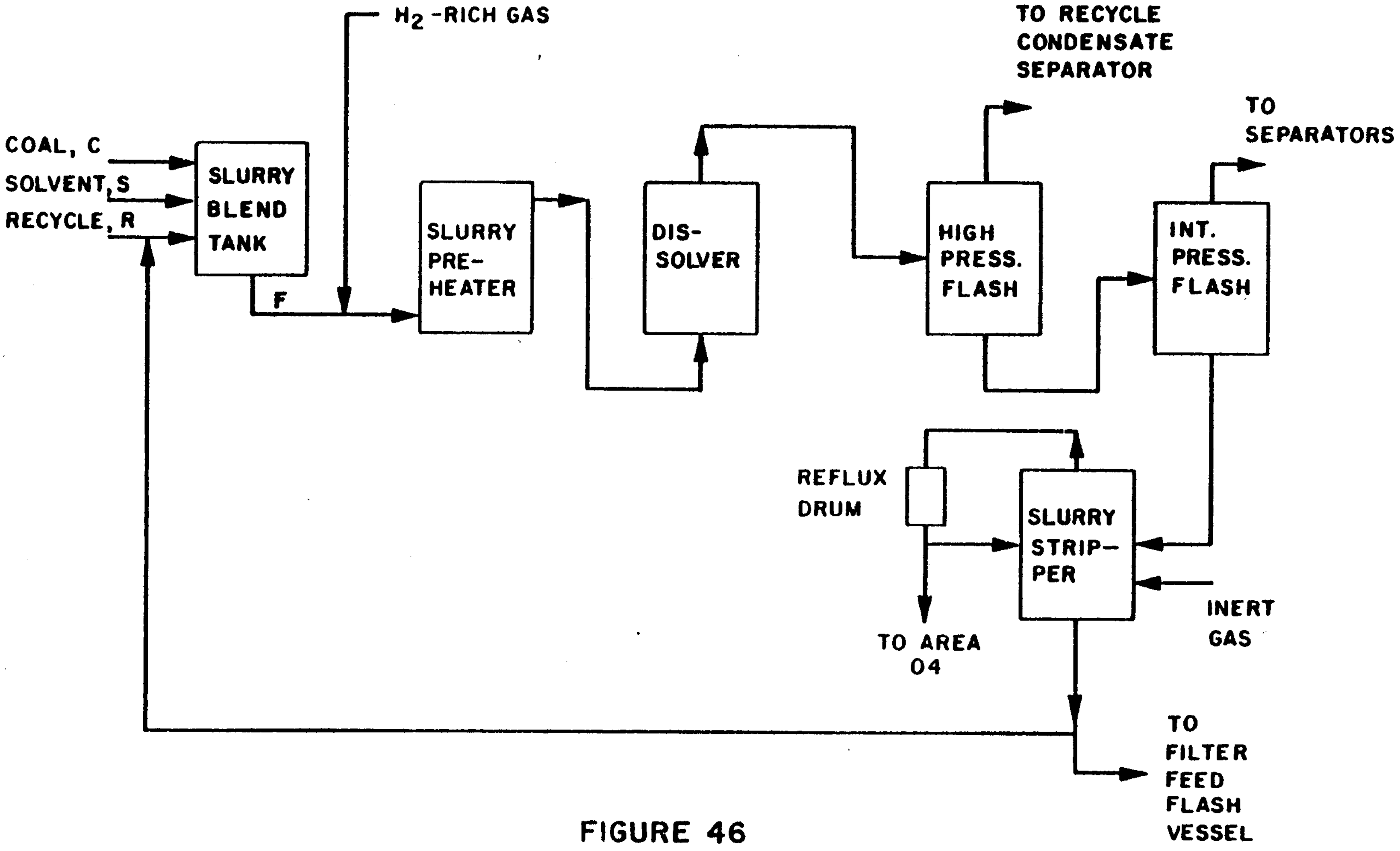


FIGURE 46

TABLE 71

SLURRY RECYCLE DISSOLVER FEED CONDITIONS

Slurry Feed Composition (%)	SR-1	SR-2	SR-3	SR-4	SR-5
Coal	19	19	20	17	15
Solvent	18	17	7	5	3
Recycle Slurry	63	64	73	78	82
TOTAL	100	100	100	100	100
Dissolver Space Rates (lb/hr ft^3)					
Coal	36.6	39.9	21.9	31.5	26.4
Total Slurry	190	216	111	189	181
Dissolver Slurry Residence Times (Minutes)					
Nominal Single Pass, ($\bar{t}$)	16.3	14.5	27.8	16.7	17.3
Average, (θ)	43	40	107	80	96

2. Procedure

Material balance calculation procedures were modified for slurry recycle operation as described below.

A preliminary gas yield was determined by the method two gas balance, based on data from on-line gas flow meters and chromatographic analyses. Total solvent yields were calculated from tank inventory changes during the test period. Laboratory distillation analysis was then used to determine the composition (light oil, wash solvent, & process solvent) in each vessel. The SRC yield was calculated by a forced ash balance based on the ash in the coal and the ash in the plant produced SRC. Finally, a preliminary water yield was estimated by forcing an elemental oxygen balance between the input and output streams. Results from these raw yields showed total mass imbalances of nearly 10% and elemental hydrogen imbalances of 15% by weight based on the input feed.

In an attempt to report all results on an equivalent basis, a technique to simultaneously normalize the hydrogen and total mass was developed. The normalization procedure produced product yields that were balanced for total mass and elemental hydrogen. The technique also resulted in acceptable elemental carbon balances (0 - 5% error). A possible source of error was the inability to accurately measure the product gas flow rate. The objective of this first series of slurry recycle material balance runs was to gain scoping type process yield information for future work, while proving the basic operability of the SRC II process. As a result, the yields obtained from these tests should be used qualitatively, rather than as accurate yield determinations. Table 72 contains the calculated yields for the five SRC II material balances.

The operating conditions for the first slurry recycle material balances were chosen to minimize potential mechanical problems caused by high concentrations of viscous or unconverted material. The first slurry recycle material balance was made at a low recycle slurry/feed ratio of 0.62. The proportion of recycle in the feed slurry was gradually increased from the initial 0.62 recycle ratio to 0.82 in the last SRC II material balance. The other primary operating conditions which were studied included: (1) reactor pressure, which was varied from 1478 psig to 1883 psig, (2) dissolver temperature, which was held at three levels - 850°, 860°, and 870°F, (3) the fractionated solvent concentration in the slurry feed which decreased from 18% to 3%, and (4) the coal space rate which varied from approximately 20 to 40 lb/hr ft^3. The average operating conditions for the five slurry recycle experiments are shown in Table 72.

TABLE 72

SLURRY RECYCLE MATERIAL BALANCE
PROCESS YIELD SUMMARY
FOR
KENTUCKY COAL

	Run SR-1	Run SR-2	Run SR-3	Run SR-4	Run SR-5
Conditions					
M.F. Coal Feed Rate, #/Hr	1861	2027	2017	1598	1342
M.F. Coal Space Rate, #/Hr/Ft^3	36.6	39.9	21.9	31.5	26.4
Fractionated Solvent Feed, #/Hr	1779	1885	700	453	310
Recycle Slurry Feed, #/Hr	6024	7040	7493	7531	7554
Recycle to Total Feed Ratio (R/F)	0.62	0.64	0.74	0.79	0.82
Gas Feed, #/Hr	281	295	293	266	249.8
Dissolver Pressure, PSIG	1478	1765	1878	1883	1887
Dissolver Outlet Temperature, °F	849	871	861	862	851
Duration of Run, Hours	60	10	65	30	66
Yields (% M.F. Coal)					
H_2	-2.0	-3.4	-3.7	-3.5	-3.8
C_1	4.4	5.4	7.1	5.9	6.3
C_2	2.4	3.0	3.5	3.8	3.9
C_3	2.3	2.8	5.0	3.2	3.9
C_4	0.8	1.0	1.8	1.2	1.5
CO	0.3	0.3	0.3	0.3	0.1
CO_2	1.1	1.0	3.2	-0.1	1.0
H_2S	1.3	1.1	1.7	1.2	2.2
Water	6.6	6.0	5.4	6.3	4.9
Light Oil	-1.7	8.9	6.4	7.7	5.5
Wash Solvent	11.9	4.4	1.8	7.4	12.7
Process Solvent	-1.2	14.8	19.9	13.6	12.1
SRC	58.9	40.5	34.0	37.2	39.0
Ash	9.1	9.2	9.2	10.6	7.7
Unreacted Coal	5.8	5.0	4.4	5.2	3.0
TOTAL	100.0	100.0	100.0	100.0	100.0
% Total Gas (excluding H_2)	12.6	14.6	22.6	15.5	18.9
Total Liquid (excluding H_2O)	9.0	28.1	28.1	28.7	30.3

3. Results

A reaction condition summary for slurry recycle material balance run numbers SR-1 through SR-5 are shown in Tables 73 through 77. The conditions are averages of data collected during the material balance periods.

The results of the elemental balances for slurry recycle material balance run numbers SR-1 through SR-5 are shown in Tables 78 through 82. These results were calculated by simultaneous forced balance of hydrogen and total mass.

Tables 83 through 87 contain the results of the laboratory analyses of the high pressure separator bottoms and the plant produced products for material balance run numbers SR-1 through SR-5. The elemental analyses shown in these tables were based on limited sample data and may not accurately represent the actual product compositions. The oxygen elementals were calculated by difference.

The average gas compositions, computed from on-line chromatography and gas flow rate measurements, for material balance runs number SR-1 through SR-5 are shown in Tables 88 through 92. Slurry preheater temperature and pressure profiles for these runs are shown in Figures 49 through 53.

The process yield results obtained for material balance runs SR-1 through SR-5 were not as accurate as those obtained for SRC I material balance runs one through eleven. The principle reasons for the greater degree of uncertainty in the SRC II process yields are listed below:

- Many upsets in coal feed rates were experienced due to the inability of the eductor type coal slurrying system to perform adequately with the substantially lower fresh solvent feed. This, along with loss of measurement of coal moisture removal, resulted in uncertainty in the moisture free coal feed rate measurement.
- The variation of composition in the reaction area resulting from interruptions in coal feed seriously affected the steady state reaction conditions but the yields were calculated assuming steady state.
- Variations in indeterminate liquid composition in the plant (i.e., pipelines, dissolver vessel, heaters, etc.) caused by coal interruptions could have been more significant to yield calculations than normal due to the lower than normal coal feed rates.
- Gas balance calculations were complicated when the monitoring of one gas stream composition and the flow rate of another gas stream failed for portions of the material balance periods. In addition, two leaking pressure safety valves may have resulted in loss of material from the

reaction area.

- SRC yields were calculated by forced ash balance. As a check, SRC yield predictions were made from high pressure flash drum samples. The average yields from this method were not always in agreement with the forced ash balance. A determination of SRC yield by product weighing was made for run number three, and the resulting yields were found to be still lower by this method. These problems were due to difficulty of defining and determining ash in the SRC product.
- As the percent recycle is increased at constant total feed rate, the average residence time in the dissolver increases exponentially. The resulting lag time may require longer run periods than those provided in order to assure stabilized run conditions.
- The material balance run periods were too short (particularly in the case of run number SR-2) to achieve a high degree of accuracy.
- The process solvent feed rate was not accurately known, particularly for material balance number SR-3.

4. Conclusions

Significant conclusions based upon the limited data available from the five material balance runs are listed below:

- The SRC II process is fundamentally operable in pilot scale equipment and yields substantially less SRC product and more liquid product than the SRC I process.
- The SRC yield decreases with an increasing recycle ratio (R/F).
- The hydrogen consumption increases with an increasing recycle ratio (R/F).
- Gas yields increase as R/F increases.
- The elemental sulfur concentration in the SRC product and in the liquid products was lower in the slurry recycle process.

Due to the requirement to produce 3,000 tons of solid SRC for a large scale combusion test, it was necessary to terminate SRC-II testing. The tests which were performed were adversly affected by equipment limitations. However, the data obtained was encouraging and a study was initiated to determine the plant modifications which were needed so that the successful bench scale SRC-II experimented program performed at P&M's Merriam, Kansas Laboratory, under ERDA sponsorship, could be confirmed at the Fort Lewis Pilot Plant. The equipment limitations and the modifications which were made are discussed in the following paragraphs. A more comprehensive SRC-II test program is planned for 1977.

5. Pilot Plant SRC II Design Considerations

During the SRC II Material Balance Runs SR-1 through SR-5, it became evident that there were several plant limitations which prevented thorough investigation of the SRC II process. In this section of the report, the major plant modifications which were deemed necessary to provide flexibility to run a successful slurry recycle program are discussed. A brief analysis of the allowable operating parameters are discussed.

a. Coal Mixing System

The coal injection system in the plant during SRC I operation consisted of an eductor in which dry pulverized coal was mixed with process solvent or the slurry blend tank contents. During the period when the SRC II runs were made, the fluid passing through the eductor was changed from solvent to recycle slurry, which oftentimes consisted of 50% process solvent, 35% SRC and 15% solids (pyridine insolubles). Since the slurry recycle process eliminates or reduces the amount of fractionated solvent mixed with the coal, this solvent was no longer available for use in the eductor. The recycle slurry (stripper bottoms from the reaction area) was known to have a much higher viscosity than process solvent, and this resulted in a high pressure drop through the eductor piping and, as a result, less flow. The end result was that coal did not mix well with either the recycle slurry or the blend tank recirculation stream. Numerous coal outages occurred which made plant lineout impossible. These coal outages were also at least partially responsible for the widely fluctuating reaction exotherms observed at the time.

Through constant operator observation of the eductor system, it was possible to reduce the number of coal outages to two per shift, but this was still considered to be too frequent for two reasons. First, constant observation of the eductor was impractical and, second, any coal outages would result in a disturbance of conditions in the dissolver, which would cause control problems. To eliminate the problem of stoppages in the coal feed, a mix tank system in which dry,pulverized coal was mechanically mixed with slurry in a small mix tank was devised and tested on a bench scale. The results of those tests were very promising and are discussed in the Process Development section of this report. The mix tank system, in which a high speed agitator creates a vortex in the slurry, and coal is added into the vortex, was chosen as the preferred method of mixing coal and slurry in the SRC II process.

b. Seal Flush System

The recirculation pumps and high pressure charge pumps in the coal mixing and charging area all require seal flush when in operation. During the SRC II material balance runs, the amount of process solvent used as seal flush was not measured, so it had to be estimated. As the amount of process solvent blended with the coal was decreased, the amount of solvent used as seal flush became a very sizeable fraction of the total process solvent added to the process. In the final stages of SRC II operation, no process solvent was blended with the coal, but solvent was still added as seal flush. The seal flush rate at that time was estimated to be about 300 lbs/hour. The coal feed rate was about 1300 lbs/hr, resulting in a ratio of seal flush addition to coal feed of 25%. The inaccuracy in the seal flush flowrate determination and the high ratio of it to the coal feed made it difficult to determine solvent yields.

To eliminate this problem, a seal flush metering system was designed that provided the seal flush from within the reaction area. It will consist of an internal recycle loop which will not be included in the material balances calculated utilizing the inventory material balance technique. To obtain the internal recycle loop for seal flush, a side draw was added to the slurry recycle stripper. This side draw will be located in the stripper column at a position where the boiling range of the liquid is sufficiently high to substitute as seal flush.

A computer program was developed to simulate the flash vessel operations downstream of the dissolver. K-values from the Grayson-Streed Modification of the Chao-Seader Correlation[1] were used to predict composition and flowrates of the liquid and vapor streams from the high pressure flash vessel, the intermediate pressure flash vessel, and the recycle condensate separator. The simulations indicated that a substantial quantity of process solvent (more than the predicted yield) flashed in the high pressure flash vessel, and was carried over to the recycle condensate separator. Since the recycle slurry stripper was expected to remove enough process solvent to supply seal flush, and a shortage of solvent in the stripper bottoms would result in a very viscous recycle slurry (the fractions of vacuum bottoms would increase as process solvent was removed), it became apparent that it would be advantageous to reinject the flashed oil into the stripper. The stripper was redesigned so that the flashed oil could be injected above the seal flush side trap. This material will be injected hot and will provide reflux and seal flush.

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[1] Grayson, H. G., and Streed, C. W., "Vapor-Liquid Equilibira for High Temperature, High Pressure Hydrogen-Hydrocarbon Systems", Proc. 6th World Petrol Congr., Frankfort/Mein VII Paper 20-PD7, 233-245 (1963)

c. Water Separation

Many attempts have been made to develop on-line material balances in the reaction area. These balances have been plagued by problems in predicting liquid yields. Since the introduction of the water quench system for cooling the dissolver effluent, it has been impossible to determine the amounts of water and oil in the liquid stream from the recycle condensate separator. This was due to the fact that the oil/water separators which process that stream were not designed to handle the substantial quantity of water introduced into the system as quench. The oil-water separation was allowed to occur in the recycle water tank, which is used to separate emulsions from several areas of the plant.

To correct this problem, and to provide a water-free stream for reinjection into the slurry recycle stripper (see above discussion), the installation of an adequate oil/water separator was undertaken. The separator should provide accurate data for the flow of light oils, water, heavy oils, and flashed hydrocarbon gases. That information should greatly benefit the development of an adequate on-line mass balance for predicting process yields. Such a balance will minimize or eliminate the long line-out times required for material balances when the inventory changes in the tanks is used to determine yields.

d. Dissolver Temperature Control

Up to the present time, there has been no adequate system for controlling dissolver temperatures. Most studies of reaction conditions have used the dissolver outlet temperature as the important dissolver temperature. A temperature profile has always existed in the dissolver, with temperatures increasing from the inlet to the outlet. The dissolver is also a dynamic system, with occasional variations in the overall exotherm when feed conditions are unchanged. This deviation from an isothermal system and the instability of the temperature make temperature control very difficult during mass balance runs and result in deviations in product yields during the runs.

In the past, the dissolver outlet temperature has been controlled solely by controlling the preheater outlet temperature. The preheater outlet temperature is controlled by changing the firing rate in the slurry preheater. This system results in a lag time of 15-30 minutes between changes in the preheater firing and the subsequent change in the dissolver temperature.

During SRC I operation, it was possible to control the dissolver temperature adequately by using the control scheme discussed above. However, during the SRC II operations in August 1975, greater exotherms between the dissolver inlets and outlets were experienced and these exotherms were erratic. The fluctuations might have been due to several reasons but, nevertheless, they did exist and resulted in changes in product distribution which affected the coal mixing step and may have been the cause of coal outages. It is known that coal outages result in a rapid drop in exotherm.

To provide adequate temperature control in the dissolver, a hydrogen quench system was provided. This type of system was used successfully to control reactor temperatures in Germany in World War II, when coal liquefaction was used to produce liquid fuel. The system consists of injecting cold hydrogen into the dissolver to cool the high temperature hydrogenation reaction. The use of hydrogen has a double barreled effect in that not only does it quench the reaction but it also serves to increase the hydrogen partial pressure in the dissolver, which may assist in achieving higher conversions to liquid products at less severe conditions than would be necessary with lower partial pressures. The use of hydrogen as a quench stream instead of an inert substance was chosen because of the double barreled effect.

Unfortunately, the type of flow conditions in the dissolver are unknown (i.e. plug flow, or backmixed or a combination of the two). The dissolver temperature profile suggests that a high amount of backmixing does occur because the temperature increases very rapidly at the bottom of the dissolver and less rapidly in upper sections. A graph of a typical profile during SRC II operation in the past is shown in Figure 47. The half-dissolver profile suggests a substantial amount of backmixing, however, the full dissolver profile is of particular interest. It suggests two distinct reaction zones, one in each half of the dissolver. (A single dissolver was used for both of these profiles)

Many models have been proposed for the Ft. Lewis dissolver flow pattern. Some of these are relatively simple, tending toward pure backmixed or pure plug flow, but some are complex, suggesting flow patterns such as ebullating flow and fluidized flow. The design of the quench system attempted to take all of these flow patterns into consideration. The resultant design provides three quench locations -- one just above the dissolver inlet, one below the half-dissolver outlet and one above the half-dissolver outlet. The combination of these three locations should provide enough flexibility to control dissolver temperatures.

DISSOLVER TEMPERATURE PROFILES DURING SLURRY RECYCLE OPERATION

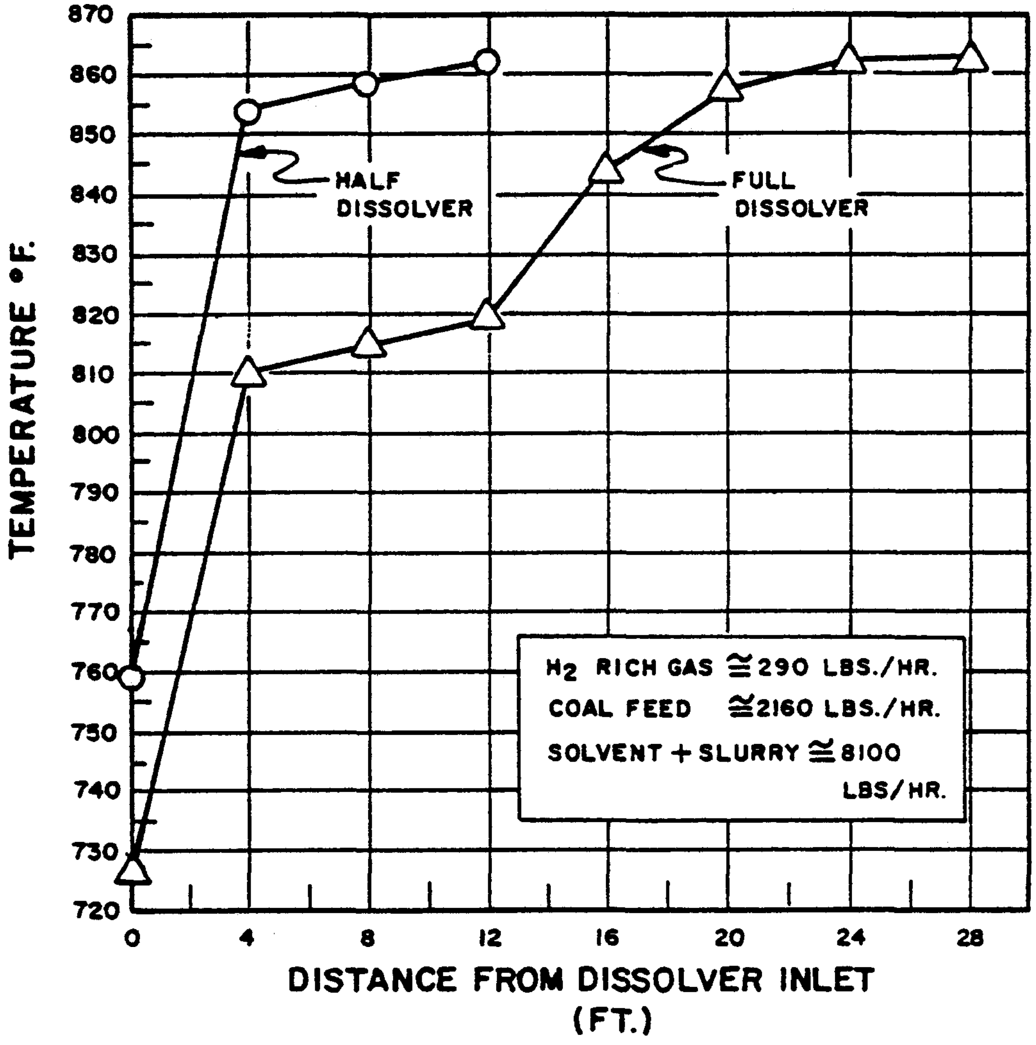


FIGURE 47

The considerations which led to the choice of three quench locations have also indicated the need for a study on dissolver flow patterns. Hopefully, resources will be available in the future to conduct tracer studies on dissolver flow that will aid in the development of a flow model for a large diameter commercial-sized reactor.

e. Recycle Hydrogen Recompression

With the addition of the hydrogen quench system, a serious problem in producing and compressing of sufficient quantities of hydrogen-rich gas results. The Fort Lewis plant recycle hydrogen compressor has a capacity of 35-40 MSCFH of hydrogen rich gas. Converting this to actual cubic feet per hour at ambient conditions results in a gas rate of about 300 ACFH. Most of this gas is required to maintain sufficiently high velocities in the preheater coil. The remainder would not be enough to provide sufficient capacity for hydrogen quench in the dissolver. It also was predicted that the SRC II process would yield the highest amounts of liquids at high hydrogen partial pressure and an increase in hydrogen consumption would result. Consideration of all of these matters led to the conclusion that increased recycle gas compression was necessary. The installation of a second recycle gas compressor was initiated so that the compression capacity in the plant would be increased to more than 100 MSCFH. The increased recirculation rate at that capacity would have resulted in the build-up of substantial amounts of light hydrocarbons (methane, ethane, propane, butane) in the recycle gas because the recycle gas treatment system, a DEA absorption column, does not have the capability to remove these compounds. Buildup of hydrocarbons would have resulted in a loss in hydrogen partial pressure in the recycle gas - exactly the opposite effect of that which was desired. Therefore, a naphtha absorption system was installed. Using plant produced naphtha which will be fairly selective in removing hydrocarbons, it is felt that recycle gas purities of at least 90% can be achieved with a minimal loss of hydrogen. The lack of accurate K-value data for such a system prevented a detailed analysis of such a column, but an approximating computer program and a field test on a bench scale unit during SRC I operations (reported in the Process Development Section of this report) provided the data needed for design.

f. Potential Operating Parameters

After the initial plant modification design was started, a project was initiated to estimate parameters for operation within the proposed plant modification. Several parameters were identified and are discussed below. A series of background experiments were conducted in the Merriam laboratory based on these considerations.

(1) Slurry Blend Tank Solids Concentrations

The maximum allowable concentration of solids in the slurry blend tank was chosen as 48%. This value was estimated as the pumping limitation of the slurry recirculation and high pressure charge pumps. Since tests have never been made on the maximum concentration of coal slurry solids that these pumps could handle, the choice of 48% was rather arbitrary, but it was necessary to limit the amount of recycle at high coal concentrations in the feed slurry.

The solids concentration term includes feed slurry solids due to the coal feed and the pyridine insoluble fraction of the recycle slurry. Consider, for instance, that at coal concentrations of 40% in the feed slurry, only 8% of the feed slurry can consist of pyridine insolubles. Since these pyridine insolubles exist in a ratio with process solvent and vacuum bottoms in the stripper bottoms, and this ratio often would result in more than the maximum 8% solids contribution of pyridine insolubles in the blend tank, it is necessary to add process solvent to the slurry to dilute the feed slurry. Taking this example one step further, if a 48% coal concentration in the feed slurry were desired, no stripper bottoms could be recycled because no additional solids could be tolerated in the blend tank. As a result, only process solvent could be blended with the coal - the SRC I process.

The conclusion from this example is that at high coal concentrations in the feed slurry, if only recycle stripper bottoms were used with the coal in formulating the feed slurry, the solids constraint would be exceeded. Therefore, the coal is mixed with a combination of recycle stripper bottoms and process solvent. On the other hand, when low coal concentrations in the blend tank are desired, say 20%, there is not enough solid material in the recycle slurry to achieve a 48% solids concentration. To eliminate this would have required installation of an ash enrichment system on the recycle slurry stream, which is well beyond the scope of the plant modifications. Instead, it was decided that if low coal concentrations are desired, lower solids concentrations in the feed will be accepted.

(2) Nominal Dissolver Residence Time

Since residence time is believed to be one of the most important variables in coal hydrogenation reactions, it was felt that a variable that would be unbiased by other operating conditions needed to be specified. In the past, coal space rate was used as an indication of residence time. However, with the use of coal concentration in the feed slurry as a correlating parameter, the meaningfulness of the coal space rate becomes clouded. (The previous plant operating conditions have nearly always been maintained at a coal concentration of 38%, or a solvent to coal ratio of 1.6/1.) To fit this need, a change to a nominal residence time will be made in SRC II. The nominal residence time is related to the space rate of the feed slurry (coal plus solvent and stripper bottoms). The nominal residence time is expected to be an indication of relative slurry residence time in the dissolver.

Based on a minimum flow velocity of 1 ft/sec through the preheater, a minimum slurry feed rate of 6000 lbs/hr was calculated. At that rate, the maximum nominal residence time in the dissolver would be 1.1 hour for a full dissolver volume or about 1/2 hour for a half dissolver volume. Tests in this range of conditions were specified and run in the Merriam laboratory.

(3) Coal Concentration

An upper limit of slurry feed based on a coal concentration of 40% was specified due to the solids limitation contraint. A lower limit of 20% was also proposed, since long lineout times are necessary to reach steady state when low coal concentrations are used.

The three limitations described above were used to construct a map of potential SRC II operating parameters. This map is shown in Figure 48. The purpose of the map was to spatially show conditions at which plant operation is feasible, and to develop a background experimental program for use in the Merriam Laboratory. This map has been continually updated as new information has been made available, and will be of use in future SRC II operation.

At a 30% coal concentration and a 48% solids level in the feed slurry, it has been estimated that no solvent need be added to the slurry. This has been confirmed by tests at the Merriam laboratory. Therefore, it can be concluded that at coal concentrations of less than 30% in the feed slurry, the solids concentration will be less than 48%, meanwhile, at coal concentrations of more than 30%, process solvent must be added to the feed to prevent the solids level from exceeding 48%. Note that by further increasing the process solvent feed, the SRC I process is approached. Therefore, the SRC II process differs from the SRC I process in that a solids containing stripped

MAP OF POTENTIAL SRC-II OPERATING PARAMETERS

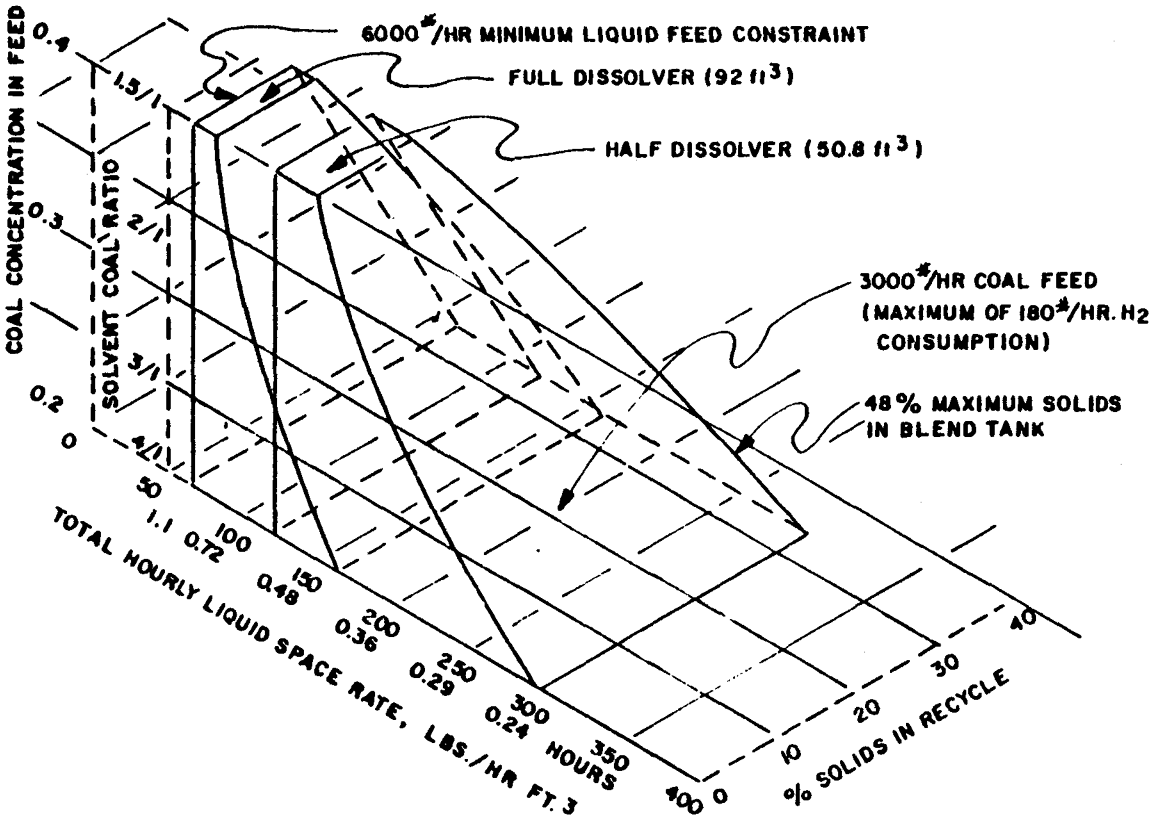


FIGURE 48

reactor effluent is utilized (with process solvent if necessary) to formulate the feed slurry.

SRC II
SLURRY PREHEATER TEMPERATURE & PRESSURE PROFILE
MATERIAL BALANCE RUN NUMBER SR-1

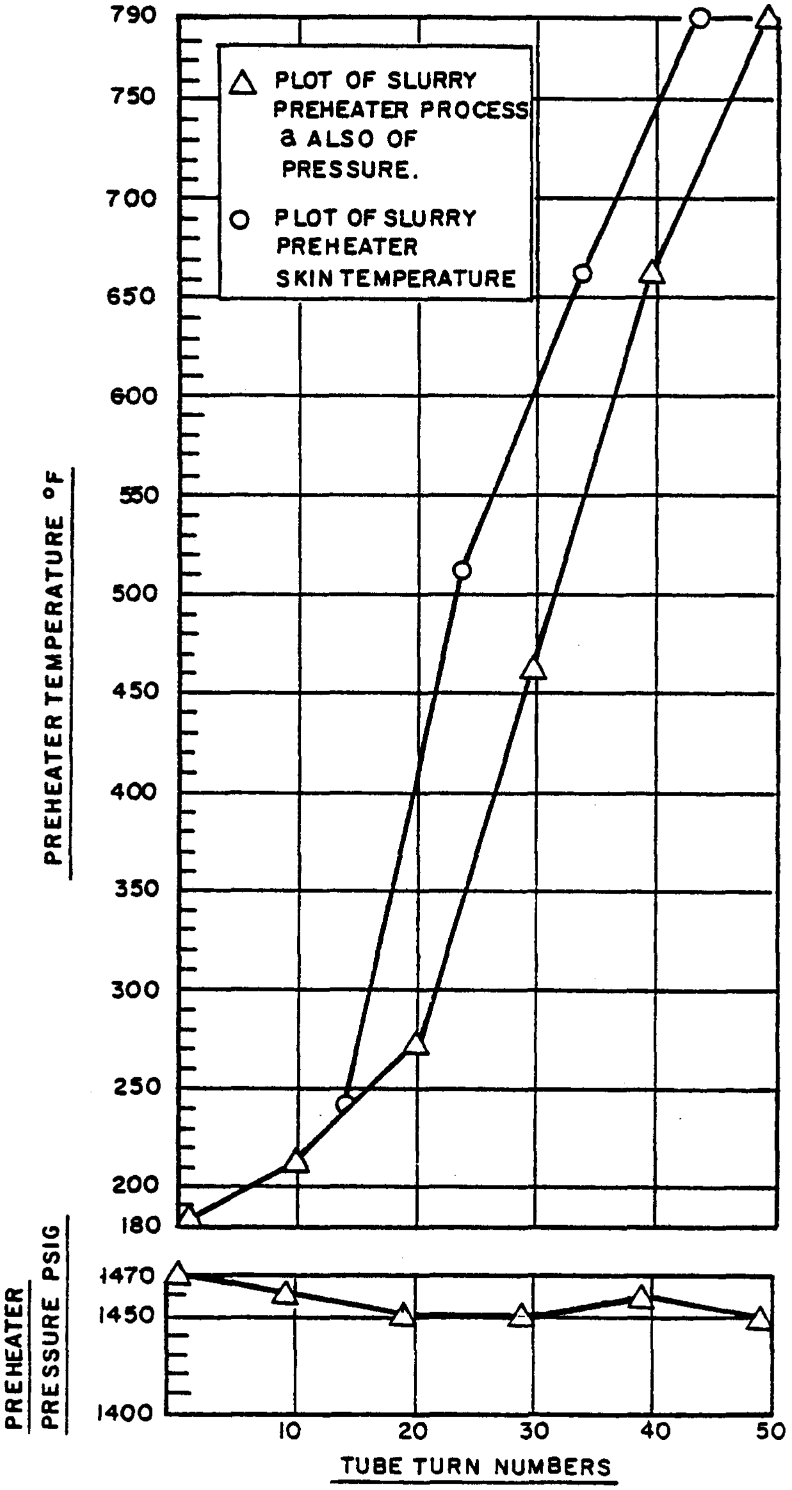


FIGURE 49

SRC II
SLURRY PREHEATER TEMPERATURE &
PRESSURE PROFILE
MATERIAL BALANCE RUN NUMBER SR-2

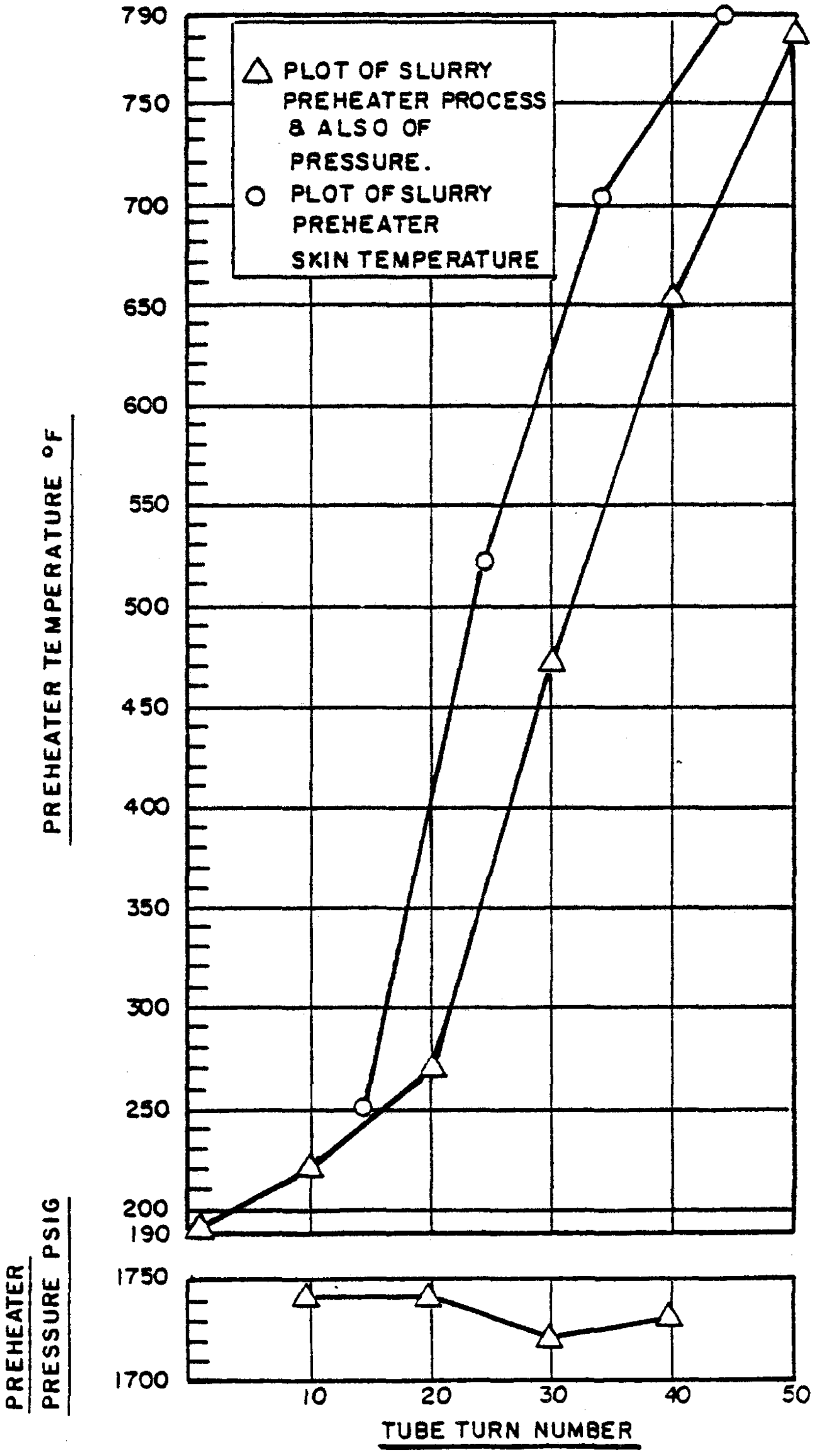


FIGURE 50

SRC II
SLURRY PREHEATER TEMPERATURE & PRESSURE PROFILE
MATERIAL BALANCE RUN NUMBER SR-3

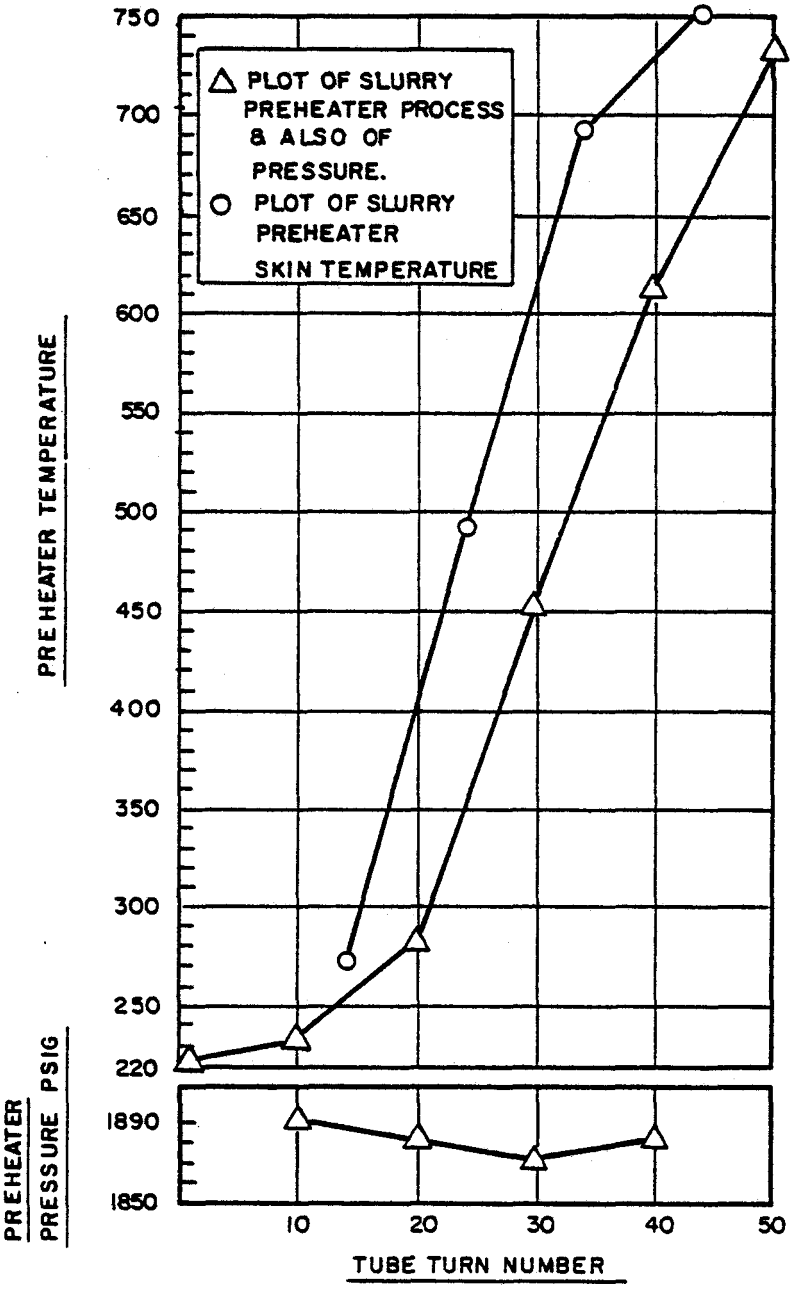


FIGURE 51

SRC II
SLURRY PREHEATER TEMPERATURE & PRESSURE PROFILE
MATERIAL BALANCE RUN NUMBER SR-4

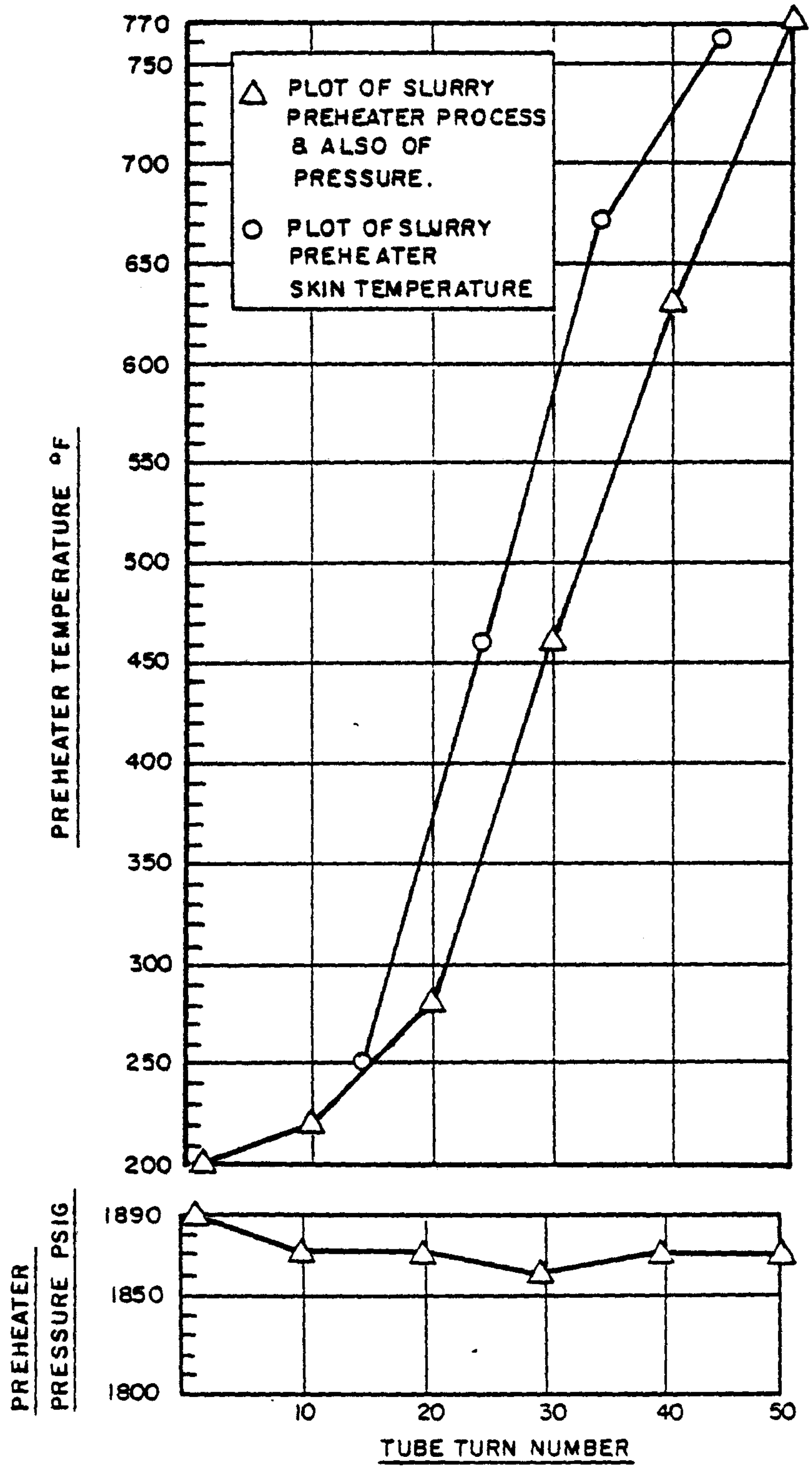


FIGURE 52

SRC II
SLURRY PREHEATER TEMPERATURE & PRESSURE PROFILE
MATERIAL BALANCE RUN NUMBER SR-5

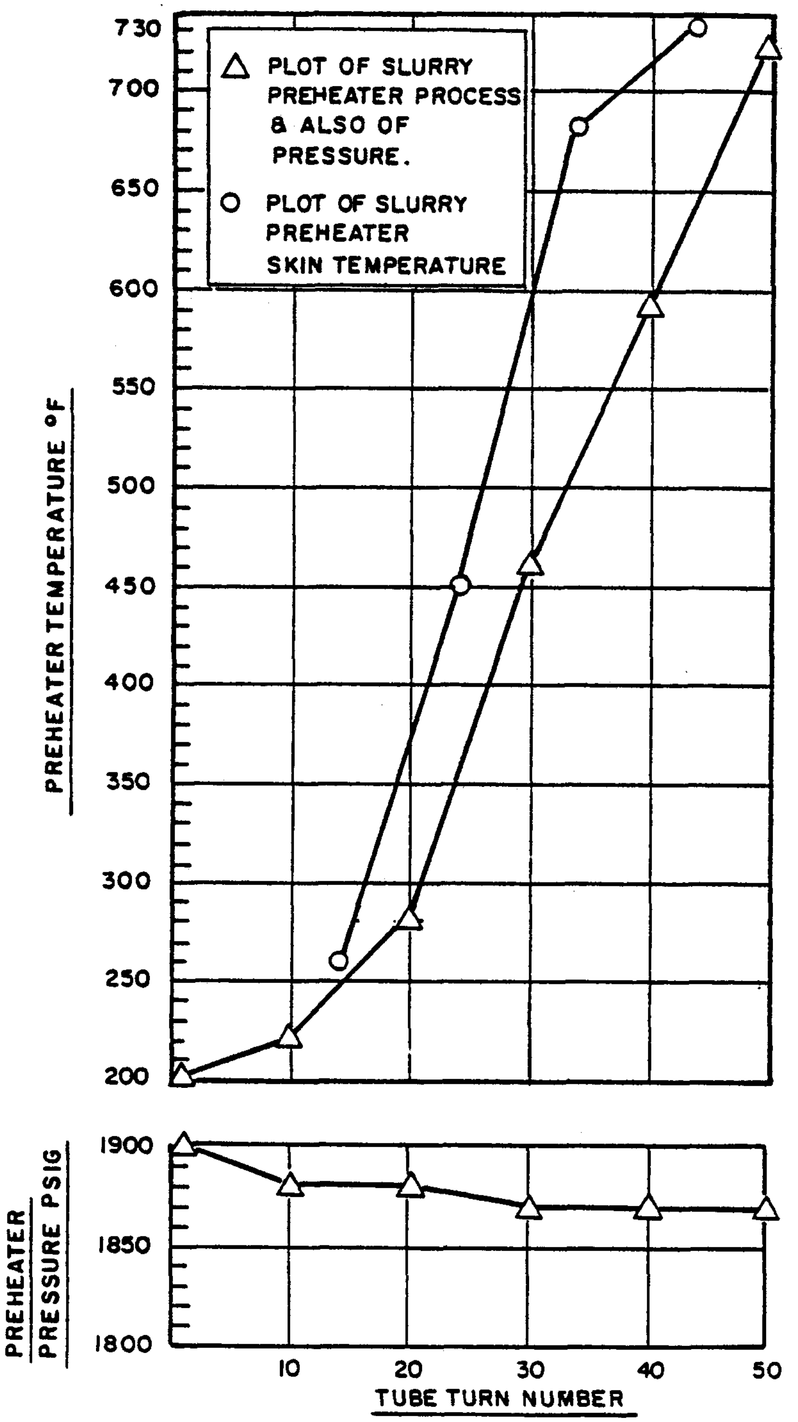


FIGURE 53

TABLE 73

SRC II
MATERIAL BALANCE RUN NO. SR-1

Reaction Conditions Summary

7-25-75 2200 HOURS TO 7-28-75 1000 HOURS

RUN CONDITIONS

RAW COAL FEED, #/HR--1963
WATER REMOVED FROM COAL, #/HR------------------------------------76
NET DEHUMIDIFIED COAL FEED, (C), #/HR------------------------1887
MOISTURE FREE COAL FEED, #/HR--------------------------------1861
FRACTIONATED SOLVENT FEED, (S), #/HR-------------------------1779
RECYCLE SLURRY FEED, (R), #/HR-------------------------------6024
SLURRY FEED TO PREHEATER, (F)=(R+S+C), #/HR------------------9690
COAL CONCENTRATION IN FEED, (C)/(F)--------------------------0.20
RECYCLE SLURRY CONCENTRATION IN FEED, (R)/(F)----------------0.62
FRACTIONATED SOLVENT CONCENTRATION IN FEED, (S)/(F)----------0.18
HYDROGEN-RICH GAS FEED, #/HR----------------------------------281
GAS FEED PURITY, MOL. PCT. H_2-------------------------------95.9
HYDROGEN FEED, #/HR---190
H_2 FEED RATIO, M.F. COAL BASIS, SCF H_2/TON COAL------------38455
SLURRY PREHEATER DIFFERENTIAL PRESSURE, PSI------------------17.3
HYDROGEN CONSUMPTION, #/HR-----------------------------------37.5
HYDROGEN CONSUMPTION, M.F. COAL BASIS, WT. % OF COAL---------2.0
HYDROGEN CONSUMPTION, M.F. COAL BASIS, MSCF/TON COAL---------7.6
SLURRY PREHEATER OUTLET TEMP., DEG F--------------------------789
DISSOLVER A PRESSURE, PSIG-----------------------------------1478
DISSOLVER OUTLET TEMPERATURE, DEG F---------------------------849
AVERAGE DISSOLVER TEMP., DEG F--------------------------------846
DISSOLVER TEMPERATURE PROFILE:

Feet From Inlet	0'	4'	8'	12'
Degrees F.	777	842	846	849

NUMBER OF DISSOLVERS---1/2

TABLE 74

SRC II
MATERIAL BALANCE RUN NO. SR-2

Reaction Conditions Summary

8-1-75 2000 HOURS TO 8-2-75 0600 HOURS

RUN CONDITIONS

RAW COAL FEED, #/HR	2178
WATER REMOVED FROM COAL, #/HR	140
NET DEHUMIDIFIED COAL FEED, (C), #/HR	2038
MOISTURE FREE COAL FEED, #/HR	2027
FRACTIONATED SOLVENT FEED, (S), #/HR	1885
RECYCLE SLURRY FEED, (R), #/HR	7040
SLURRY FEED TO PREHEATER, (F)=(R+S+C), #/HR	10963
COAL CONCENTRATION IN FEED, (C)/(F)	0.19
RECYCLE SLURRY CONCENTRATION IN FEED, (R)/(F)	0.64
FRACTIONATED SOLVENT CONCENTRATION IN FEED, (S)/(F)	0.17
HYDROGEN-RICH GAS FEED, #/HR	295
GAS FEED PURITY, MOL. PCT. H_2	94.9
HYDROGEN FEED, #/HR	185
H_2 FEED RATIO, M.F. COAL BASIS, SCF H_2/TON COAL	34315
SLURRY PREHEATER DIFFERENTIAL PRESSURE, PSI	17.9
HYDROGEN CONSUMPTION, #/HR	69.1
HYDROGEN CONSUMPTION, M.F. COAL BASIS, WT. % OF COAL	3.4
HYDROGEN CONSUMPTION, M.F. COAL BASIS, MSCF/TON COAL	12.9
SLURRY PREHEATER OUTLET TEMP., DEG F	783
DISSOLVER A PRESSURE, PSIG	1750
DISSOLVER OUTLET TEMPERATURE, DEG F	871
AVERAGE DISSOLVER TEMP., DEG F	865

DISSOLVER TEMPERATURE PROFILE:

Feet from Inlet	0'	4'	8'	12'
Degrees F.	775	857	866	871

NUMBER OF DISSOLVERS	1/2

TABLE 75

SRC II
MATERIAL BALANCE RUN NO. SR-3

Reaction Conditions Summary

8-15-75 2100 HOURS TO 8-18-75 1400 HOURS

RUN CONDITIONS

RAW COAL FEED, #/HR	2136
WATER REMOVED FROM COAL, #/HR	111
NET DEHUMIDIFIED COAL FEED, (C), #/HR	2025
MOISTURE FREE COAL FEED, #/HR	2017
FRACTIONATED SOLVENT FEED, (S), #/HR	700
RECYCLE SLURRY FEED, (R), #/HR	7493
SLURRY FEED TO PREHEATER, (F)=(R+S+C), #/HR	10218
COAL CONCENTRATION IN FEED, (C)/(F)	0.20
RECYCLE SLURRY CONCENTRATION IN FEED, (R)/(F)	0.73
FRACTIONATED SOLVENT CONCENTRATION IN FEED, (S)/(F)	0.07
HYDROGEN-RICH GAS FEED, #/HR	293
GAS FEED PURITY, MOL. PCT. H_2	95.2
HYDROGEN FEED, #/HR	186
H_2 FEED RATIO, M.F. COAL BASIS, SCF H_2/TON COAL	34709
SLURRY PREHEATER DIFFERENTIAL PRESSURE, PSI	34.2
HYDROGEN CONSUMPTION, #/HR	73.7
HYDROGEN CONSUMPTION, M.F. COAL BASIS, WT. % OF COAL	3.7
HYDROGEN CONSUMPTION, M.F. COAL BASIS, MSCF/TON COAL	13.8
SLURRY PREHEATER OUTLET TEMP., DEG F	727
DISSOLVER A PRESSURE, PSIG	1878
DISSOLVER OUTLET TEMPERATURE, DEG F	861
AVERAGE DISSOLVER TEMP., DEG F	836

DISSOLVER TEMPERATURE PROFILE:

Feet from Inlet	0'	4'	8'	12'	16'	20'	24'	28'
Degrees F.	726	805	812	817	842	855	861	861

NUMBER OF DISSOLVERS	1

TABLE 76

SRC II
MATERIAL BALANCE RUN NO. SR-4

Reaction Conditions Summary

8-24-75 1100 HOURS TO 8-25-75 1700 HOURS

RUN CONDITIONS

RAW COAL FEED, #/HR	1709
WATER REMOVED FROM COAL, #/HR	92
NET DEHUMIDIFIED COAL FEED, (C), #/HR	1617
MOISTURE FREE COAL FEED, #/HR	1598
FRACTIONATED SOLVENT FEED, (S), #/HR	453
RECYCLE SLURRY FEED, (R), #/HR	7531
SLURRY FEED TO PREHEATER, (F)=(R+S+C), #/HR	9601
COAL CONCENTRATION IN FEED, (C)/(F)	0.17
RECYCLE SLURRY CONCENTRATION IN FEED, (R)/(F)	0.78
FRACTIONATED SOLVENT CONCENTRATION IN FEED, (S)/(F)	0.05
HYDROGEN-RICH GAS FEED, #/HR	266
GAS FEED PURITY, MOL. PCT. H_2	95.8
HYDROGEN FEED, #/HR	170
H_2 FEED RATIO, M.F. COAL BASIS, SCF H_2/TON COAL	40129
SLURRY PREHEATER DIFFERENTIAL PRESSURE, PSI	19.5
HYDROGEN CONSUMPTION, #/HR	56.2
HYDROGEN CONSUMPTION, M.F. COAL BASIS, WT. % OF COAL	3.5
HYDROGEN CONSUMPTION, M.F. COAL BASIS, MSCF/TON COAL	13.3
SLURRY PREHEATER OUTLET TEMP., DEG F	767
DISSOLVER A PRESSURE, PSIG	1883
DISSOLVER OUTLET TEMPERATURE, DEG F	862
AVERAGE DISSOLVER TEMP., DEG F	859

DISSOLVER TEMPERATURE PROFILE:

Feet from Inlet	0'	4'	8'	12'
Degrees F.	751	855	860	862

NUMBER OF DISSOLVERS	1/2

TABLE 77

SRC II
MATERIAL BALANCE RUN NO. SR-5

Reaction Conditions Summary

8-26-75 1200 HOURS TO 8-29-75 600 HOURS

RUN CONDITIONS

RAW COAL FEED, #/HR	1435
WATER REMOVED FROM COAL, #/HR	75
NET DEHUMIDIFIED COAL FEED, (C), #/HR	1360
MOISTURE FREE COAL FEED, #/HR	1342
FRACTIONATED SOLVENT FEED, (S), #/HR	310
RECYCLE SLURRY FEED, (R), #/HR	7554
SLURRY FEED TO PREHEATER, (F)=(R+S+C), #/HR	9224
COAL CONCENTRATION IN FEED, (C)/(F)	0.15
RECYCLE SLURRY CONCENTRATION IN FEED, (R)/(F)	0.82
FRACTIONATED SOLVENT CONCENTRATION IN FEED, (S)/(F)	0.03
HYDROGEN-RICH GAS FEED, #/HR	250
GAS FEED PURITY, MOL. PCT. H_2	97.4
HYDROGEN FEED, #/HR	191
H_2 FEED RATIO, M.F. COAL BASIS, SCF H_2/TON COAL	53332
SLURRY PREHEATER DIFFERENTIAL PRESSURE, PSI	34.2
HYDROGEN CONSUMPTION, #/HR	51.3
HYDROGEN CONSUMPTION, M.F. COAL BASIS, WT. % OF COAL	3.8
HYDROGEN CONSUMPTION, M.F. COAL BASIS, MSCF/TON COAL	14.4
SLURRY PREHEATER OUTLET TEMP., DEG F	722
DISSOLVER A PRESSURE, PSIG	1877
DISSOLVER OUTLET TEMPERATURE, DEG F	851
AVERAGE DISSOLVER TEMP., DEG F	848

DISSOLVER TEMPERATURE PROFILE:

Feet from Inlet	0'	4'	8'	12'
Degrees F.	704	844	848	851

NUMBER OF DISSOLVERS	1/2

TABLE 78

SRC II
MATERIAL BALANCE RUN NO. SR-1

ELEMENTAL BALANCES

INPUT (POUNDS PER HOUR)

	C	H	N	S	O	ASH	TOTAL
M.F. Coal	1330.3	95.7	28.7	60.5	166.4	179.4	1861.0
H2 Rich Gas	60.5	206.1	0	0	9.8	0	276.4
Total In	1390.8	301.8	28.7	60.5	176.2	179.4	2137.4

OUTPUT (POUNDS PER HOUR)

	C	H	N	S	O	ASH	TOTAL
Gas	212.4	209.8	0	22.3	27.8	0	472.3
Water	0	13.8	0	0	109.8	0	123.6
Lt. Oil	-27.0	-3.8	-0.1	-0.1	-1.1	0	-32.1
Wash Solvent	188.0	19.6	0.9	0.4	12.7	0	221.6
Process Solvent	-19.4	-1.6	-0.2	-0.1	-0.5	0	-21.8
SRC (Total)	1059.4	64.0	26.0	26.0	28.3	170.1	1373.8
Total Out	1413.4	301.8	26.6	48.5	177.0	170.1	2137.4
OUTPUT MINUS INPUT	22.6	0	-2.1	-12.0	0.8	-9.3	0
ERROR AS % OF INPUT	1.6	0	-7.3	-19.8	0.5	-5.2	0

TABLE 79

SRC II
MATERIAL BALANCE RUN NO. SR-2

ELEMENTAL BALANCES

INPUT (POUNDS PER HOUR)

	C	H	N	S	O	ASH	TOTAL
M.F. Coal	1439.8	102.0	31.4	92.2	164.0	197.6	2027.0
H2 Rich Gas	80.0	203.2	0	0	10.2	0	293.4
Total In	1519.8	305.2	31.4	92.2	174.2	197.6	2320.4

OUTPUT (POUNDS PER HOUR)

	C	H	N	S	O	ASH	TOTAL
Gas	281.0	189.0	0	21.5	29.9	0	521.4
Water	0	13.7	0	0	108.5	0	122.2
Lt. Oil	149.7	21.0	0.8	0.6	7.7	0	179.8
Wash Solvent	74.5	7.8	0.5	0.2	5.2	0	88.2
Process Solvent	267.9	22.5	2.7	1.3	6.2	0	300.6
SRC (Total)	826.1	51.2	19.7	12.5	12.4	186.3	1108.2
Total Out	1599.2	305.2	23.7	36.1	169.9	186.3	2320.4
OUTPUT MINUS INPUT	79.4	0	-7.7	-56.1	-4.3	-11.3	0
ERROR AS % OF INPUT	5.2	0	-24.5	-60.8	-2.5	-5.7	0

TABLE 80

SRC II
MATERIAL BALANCE RUN NO. SR-3

ELEMENTAL BALANCES

INPUT (POUNDS PER HOUR)

	C	H	N	S	O	ASH	TOTAL
M.F. Coal	1446.6	104.5	32.1	78.3	171.5	184.2	2017.2
H2 Rich Gas	72.6	204.8	0	0	11.3	0	288.7
Total In	1519.2	309.3	32.1	78.3	182.8	184.2	2305.9

OUTPUT (POUNDS PER HOUR)

	C	H	N	S	O	ASH	TOTAL
Gas	367.6	208.3	0	31.5	60.7	0	668.1
Water	0	12.3	0	0	96.8	0	109.1
Lt. Oil	106.6	14.9	0.6	0.3	5.6	0	128.0
Wash Solvent	31.2	3.2	0.3	0.1	2.1	0	36.9
Process Solvent	356.8	29.4	4.2	1.6	9.4	0	401.4
SRC (Total)	687.3	41.2	16.8	3.0	27.7	186.4	962.4
Total Out	1549.5	309.3	21.9	36.5	202.3	186.4	2305.9
OUTPUT MINUS INPUT	30.3	0	-10.2	-41.8	19.5	2.2	0
ERROR AS % OF INPUT	2.0	0	-31.8	-53.4	10.7	1.2	0

TABLE 81

SRC II
MATERIAL BALANCE RUN NO. SR-4

ELEMENTAL BALANCES

INPUT (POUNDS PER HOUR)

	C	H	N	S	O	ASH	TOTAL
M.F. Coal	1163.7	81.8	24.9	69.4	112.5	146.1	1598.4
H2 Rich Gas	56.4	183.2	0	0	22.7	0	262.3
Total In	1220.1	265.0	24.9	69.4	135.2	146.1	1860.7

OUTPUT (POUNDS PER HOUR)

	C	H	N	S	O	ASH	TOTAL
Gas	234.3	176.6	0	18.2	24.7	0	453.8
Water	0	11.2	0	0	89.9	0	101.1
Lt. Oil	103.2	14.7	0.5	0.2	3.8	0	122.4
Wash Solvent	100.4	10.5	0.8	0	6.1	0	117.8
Process Solvent	194.7	16.3	2.2	0.7	3.8	0	217.7
SRC (Total)	594.9	35.7	13.5	2.7	31.6	169.5	847.9
Total Out	1227.5	265.0	17.0	21.8	159.9	169.5	1860.7
OUTPUT MINUS INPUT	7.4	0	-7.9	-47.6	24.7	23.4	0
ERROR AS % OF INPUT	0.6	0	-31.7	-68.6	18.3	16.0	0

TABLE 82

SRC II
MATERIAL BALANCE RUN NO. SR-5

ELEMENTAL BALANCES

INPUT (POUNDS PER HOUR)

	C	H	N	S	O	ASH	TOTAL
M.F. Coal	961.6	68.0	19.0	65.0	111.4	117.0	1342.0
H2 Rich Gas	36.1	198.8	0	0	10.5	0	245.4
Total In	997.7	266.8	19.0	65.0	121.9	117.0	1587.4

OUTPUT (POUNDS PER HOUR)

	C	H	N	S	O	ASH	TOTAL
Gas	204.6	194.2	0	27.5	21.6	0	447.9
Water	0	7.4	0	0	58.9	0	66.3
Lt. Oil	61.5	9.1	0.4	0.2	2.0	0	73.2
Wash Solvent	143.6	14.8	1.2	0.2	10.5	0	170.3
Process Solvent	144.9	12.0	1.7	0.5	3.6	0	162.7
SRC (Total)	477.2	29.3	10.9	27.7	18.6	103.3	667.0
Total Out	1031.8	266.8	14.2	56.1	115.2	103.3	1587.4
OUTPUT MINUS INPUT	34.1	0	-4.8	-8.9	-6.7	-13.7	0
ERROR AS % OF INPUT	3.4	0	-25.3	-13.7	-5.5	-11.7	0

TABLE 83

SRC II

MATERIAL BALANCE RUN NO. SR-1

AVERAGED LABORATORY RESULTS

HIGH PRESSURE SEPARATOR BOTTOMS SAMPLE-POINT NO. 204

SLURRY ANALYSIS	
% Pyridine Insolubles	8.2
% Ash in P. I. = 61.1	
% H_2O	1.4
% Light Oil	0.5
% Wash Solvent	4.3
% Process Solvent	55.4
% SRC	30.2
TOTAL	100.0

Liquid Collected, grams: 836.2

FLASHED GAS ANALYSIS	
Vol. %	
H_2	47.0
N_2	7.4
C_1	21.0
CO	0.6
C_2	5.3
CO_2	7.6
C_3	4.7
C_4	0.8
H_2S	5.6
TOTAL	100.0

Volume of Gas Recovered, liters: 3.1

ELEMENTAL ANALYSES OF PLANT PRODUCED PRODUCTS

WEIGHT PERCENTS

TEST	MOISTURE FREE COAL FEED	LIGHT OIL POINT 408	WASH SOLVENT POINT 410	PROCESS SOLVENT POINT 411	SRC (FILTRATION SECTION BYPASSED)	AVERAGE LABORATORY PRODUCED SRC
C	71.49	83.69	84.80	89.11	77.12	----
H	5.14	11.94	8.87	7.26	4.66	----
N	1.54	0.34	0.44	0.91	1.89	----
S	3.25	0.40	0.16	0.37	1.89	0.61
O	8.94	3.63	5.73	2.35		----
Ash	9.64	----	----	----	12.38	----
TOTAL	100.0	100.0	100.0	100.0	97.9	----

TABLE 84

SRC II

MATERIAL BALANCE RUN NO. SR-2

AVERAGED LABORATORY RESULTS

HIGH PRESSURE SEPARATOR BOTTOMS SAMPLE-POINT NO. 204

SLURRY ANALYSIS

% Pyridine Insolubles	11.6
% Ash in P. I. = 65.0	
% H_2O	0.8
% Light Oil	0.8
% Wash Solvent	4.9
% Process Solvent	54.2
% SRC	27.7
TOTAL	100.0
Liquid Collected, grams:	465.0

FLASHED GAS ANALYSIS

Vol. %	
H_2	62.9
N_2	0.
C_1	21.0
CO	0.1
C_2	6.9
CO_2	2.5
C_3	5.1
C_4	0.8
H_2S	0.7
TOTAL	102.8

Volume of Gas Recovered, liters: 3.0

ELEMENTAL ANALYSES OF PLANT PRODUCED PRODUCTS

WEIGHT PERCENTS

TEST	MOISTURE FREE FEED	LIGHT OIL POINT 408	WASH SOLVENT POINT 410	PROCESS SOLVENT POINT 411	SRC (FILTRATION SECTION BYPASSED)	AVERAGE LABORATORY PRODUCED SRC
C	71.03	83.25	84.46	89.07	74.30	88.34
H	5.03	11.69	8.85	7.50	4.61	5.34
N	1.55	0.41	0.56	0.91	1.83	2.31
S	4.55	0.34	0.22	0.45	2.88	0.55
O	8.09	4.31	5.91	2.07	----	3.46
Ash	9.75	----	----	----	15.55	----
TOTAL	100.0	100.0	100.0	100.0	99.2	100.0

TABLE 85

SRC II

MATERIAL BALANCE RUN NO. SR-3

AVERAGED LABORATORY RESULTS

HIGH PRESSURE SEPARATOR BOTTOMS SAMPLE-POINT NO. 204

SLURRY ANALYSIS

% Pyridine Insolubles	18.4
% Ash in P. I. = 67.7	
% H_2O	0.9
% Light Oil	0.5
% Wash Solvent	2.9
% Process Solvent	38.2
% SRC	39.1
TOTAL	100.0
Liquid Collected, grams:	244.4

FLASHED GAS ANALYSIS

Vol. %	
H_2	49.8
N_2	3.1
C_1	26.0
CO	0.1
C_2	10.9
CO_2	2.5
C_3	5.2
C_4	1.9
H_2S	0.6
TOTAL	100.1

Volume of Gas Recovered, liters: 2.06

ELEMENTAL ANALYSES OF PLANT PRODUCED PRODUCTS

WEIGHT PERCENTS

TEST	MOISTURE FREE FEED	LIGHT OIL POINT 408	WASH SOLVENT POINT 410	PROCESS SOLVENT POINT 411	SRC (FILTRATION SECTION BYPASSED)	AVERAGE LABORATORY PRODUCED SRC
C	71.72	88.33	84.57	88.89	71.41	89.61
H	5.18	11.65	8.83	7.33	4.28	5.34
N	1.59	0.44	0.69	1.05	1.74	2.09
S	3.88	0.23	0.13	0.38	3.25	0.40
O	8.50	4.85	5.78	2.35	----	2.35
Ash	9.13	----	----	----	19.37	0.21
TOTAL	100.0	100.0	100.0	100.0	100.1	100.0

TABLE 86

SRC II

MATERIAL BALANCE RUN NO. SR-4

AVERAGED LABORATORY RESULTS

HIGH PRESSURE SEPARATOR BOTTOMS SAMPLE-POINT NO. 204

SLURRY ANALYSIS

% Pyridine Insolubles	15.4
% Ash in P. I. = 67.0	
% H_2O	0.7
% Light Oil	0.5
% Wash Solvent	2.9
% Process Solvent	43.3
% SRC	37.2
TOTAL	100.0

Liquid Collected, grams: 346.0

FLASHED GAS ANALYSIS

Vol. %	
H_2	74.0
N_2	2.4
C_1	15.1
CO	0.0
C_2	4.6
CO_2	0.9
C_3	1.9
C_4	0.8
H_2S	0.3
TOTAL	100.0

Volume of Gas
Recovered, liters: 2.26

ELEMENTAL ANALYSES OF PLANT PRODUCED PRODUCTS

WEIGHT PERCENTS

TEST	MOISTURE FREE FEED	LIGHT OIL POINT 408	WASH SOLVENT POINT 410	PROCESS SOLVENT POINT 411	SRC (FILTRATION SECTION BYPASSED	AVERAGE LABORATORY PRODUCED SRC
C	72.80	84.19	85.18	89.42	70.15	89.46
H	5.12	12.06	8.88	7.47	4.21	5.34
N	1.56	0.39	0.66	1.02	1.59	2.29
S	4.34	0.20	0.03	0.32	4.05	0.41
O	7.04	3.16	5.25	1.77	----	2.38
Ash	9.14	----	----	----	20.00	0.12
TOTAL	100.0	100.0	100.0	100.0	100.0	100.0

TABLE 87

SRC II

MATERIAL BALANCE RUN NO. SR-5

AVERAGED LABORATORY RESULTS

HIGH PRESSURE SEPARATOR BOTTOMS SAMPLE-POINT NO. 204

SLURRY ANALYSIS

% Pyridine Insolubles	17.7
% Ash in P. I. = 71.4	
% H_2O	0.7
% Light Oil	0.6
% Wash Solvent	3.2
% Process Solvent	40.9
% SRC	36.9
TOTAL	100.0

Liquid Collected, grams: 511.2

FLASHED GAS ANALYSIS

Vol. %	
H_2	73.4
N_2	1.9
C_1	13.4
CO	0.1
C_2	5.4
CO_2	1.5
C_3	2.5
C_4	1.0
H_2S	0.8
TOTAL	100.0

Volume of Gas Recovered, liters: 1.94

ELEMENTAL ANALYSES OF PLANT PRODUCED PRODUCTS

WEIGHT PERCENTS

TEST	MOISTURE FREE FEED	LIGHT OIL POINT 408	WASH SOLVENT POINT 410	PROCESS SOLVENT POINT 411	SRC (FILTRATION SECTION BYPASSED)	AVERAGE LABORATORY PRODUCED SRC
C	71.64	84.12	84.35	89.01	71.54	89.14
H	5.07	12.47	8.72	7.40	4.39	5.63
N	1.42	0.44	0.71	1.05	1.64	2.16
S	4.85	0.22	0.08	0.30	4.16	0.53
O	8.30	2.75	6.14	2.24	----	2.39
Ash	8.72	----	----	----	19.11	0.15
TOTAL	100.0	100.0	100.0	100.0	100.8	100.0

TABLE 88

SRC II
MATERIAL BALANCE RUN NO. SR-1

GAS STREAM COMPOSITIONS

	FT 276 FRESH H2			FT 258 05 VENT GAS			FT 2117 03 SEP. GAS			FT 170 RCY CN SEP OH		
	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR
H2	98.63	86.3	138.1	84.67	32.7	65.6	42.64	4.8	10.9	83.35	29.6	97.1
N2	0.14	1.7	2.7	0.20	1.1	2.2	39.14	60.7	138.2	0.20	1.0	3.3
C1	0.64	4.5	7.1	9.85	30.2	60.6	4.93	4.4	10.0	9.94	28.0	92.0
CO	0.55	6.7	10.7	1.05	5.6	11.2	0.23	0.4	0.8	1.03	5.1	16.6
C2	0.00	0.0	0.0	2.29	13.2	26.5	2.09	3.5	7.9	2.32	12.3	40.3
CO2	0.04	0.7	1.1	0.09	0.7	1.4	5.82	14.2	32.3	0.54	4.2	13.7
C3	0.00	0.0	0.0	1.49	12.6	25.3	2.13	5.2	11.8	1.31	11.7	38.4
IC4	0.00	0.0	0.0	0.05	0.6	1.1	0.09	0.3	0.6	0.05	0.5	1.7
NC4	0.00	0.1	0.1	0.29	3.2	6.4	0.62	2.0	4.5	0.29	2.9	9.7
H2S	0.00	0.0	0.0	0.01	0.1	0.2	2.19	4.1	9.4	0.77	4.6	15.1
C5+	0.00	0.1	0.1	0.00	0.1	0.1	0.11	0.4	1.0	0.00	0.1	0.2
TOTAL			159.9			200.6			227.4			328.1
MOL. WT.			2.31			3.23			18.05			5.69
SCFH			26302.30			14562.27			4781.60			21889.71

	FT 2134 RECYCLE GAS			FT 144 & FT 85 PREHEATER FEED			FT 2107 LT. OIL/H2O SEPT.		
	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR
H2	84.67	32.7	49.8	95.32	64.8	184.3	1.92	0.1	0.1
N2	0.20	1.1	1.7	0.16	1.5	4.2	74.93	67.1	38.3
C1	9.85	30.2	46.0	2.94	15.9	45.1	0.07	0.0	0.0
CO	1.05	5.6	8.5	0.62	5.9	16.7	0.04	0.0	0.0
C2	2.29	13.2	20.1	0.52	5.3	15.1	0.85	0.8	0.5
CO2	0.09	0.7	1.1	0.04	0.6	1.6	11.21	15.8	9.0
C3	0.49	12.6	19.2	0.34	5.0	14.2	3.15	4.4	2.3
IC4	0.05	0.6	0.9	0.00	0.0	0.0	0.33	0.6	0.4
NC4	0.29	3.2	4.9	0.05	1.0	2.8	2.44	4.5	2.6
H2S	0.01	0.1	0.1	0.01	0.1	0.3	4.20	4.6	2.6
C5+	0.00	0.1	0.1	0.00	0.0	0.0	0.84	2.0	1.2
TOTAL			152.4			284.3			57.2
MOL. WT.			5.23			2.97			31.31
SCFH			11049.96			36315.43			692.18

TABLE 89

SRC II
MATERIAL BALANCE RUN NO. SR-2

GAS STREAM COMPOSITIONS

	FT 276 FRESH H2			FT 258 05 VENT GAS			FT 170 RCY CN SEP OH			FT 2134 RECYCLE GAS		
	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR
H2	98.81	87.5	146.1	78.98	24.9	62.7	77.71	22.8	89.9	78.98	24.9	47.6
N2	0.12	1.5	2.5	0.20	0.9	2.2	0.19	0.8	3.1	0.20	0.9	1.6
C1	0.45	3.2	5.3	13.71	34.4	86.4	13.63	31.8	125.2	13.71	34.4	65.6
CO	0.59	7.2	12.0	1.19	5.2	13.1	1.18	4.8	18.9	1.19	5.2	10.0
C2	0.00	0.0	0.0	3.33	15.6	39.3	3.33	14.6	57.3	3.33	15.6	29.9
CO2	0.03	0.6	1.0	0.09	0.6	1.6	0.62	4.0	15.6	0.09	0.6	1.2
C3	0.00	0.0	0.0	1.99	13.7	34.4	1.96	12.6	49.5	1.99	13.7	26.2
IC4	0.00	0.0	0.0	0.08	0.7	1.8	0.08	0.7	2.6	0.08	0.7	1.4
NC4	0.00	0.0	0.0	0.42	3.8	9.7	0.42	3.5	13.9	0.42	3.8	7.3
H2S	0.00	0.0	0.0	0.00	0.0	0.0	0.87	4.3	17.0	0.00	0.0	0.0
C5+	0.00	0.0	0.0	0.01	0.1	0.3	0.01	0.1	0.6	0.01	0.1	0.3
TOTAL			166.9			251.5			393.6			191.1
MOL. WT.			2.28			6.40			6.87			6.40
SCFH			27778.18			14909.41			21730.21			11325.08

	FT 144 & FT 85 PREHEATER FEED			FT 2117 03 SEP. GAS			FT 2107 LT. OIL/H2O SEPT.		
	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR
H2	94.91	62.7	185.1	49.00	6.6	15.0	3.11	0.2	0.1
N2	0.14	1.2	3.7	47.67	89.4	201.8	70.10	62.2	34.7
C1	3.23	16.9	50.0	2.89	3.1	7.0	0.02	0.0	0.0
CO	0.61	5.6	16.6	0.38	0.7	1.6	0.17	0.2	0.1
C2	0.64	6.3	18.6	0.02	0.0	0.1	1.20	1.1	0.6
CO2	0.03	0.4	1.1	0.02	0.1	0.1	10.97	15.3	8.5
C3	0.38	5.5	16.2	0.01	0.0	0.0	4.44	6.2	3.5
IC4	0.00	0.0	0.0	0.00	0.0	0.0	0.45	0.8	0.5
NC4	0.07	1.3	3.7	0.00	0.0	0.0	3.19	5.9	3.3
H2S	0.00	0.0	0.0	0.01	0.0	0.0	5.43	5.9	3.3
C5+	0.00	0.0	0.0	0.00	0.0	0.0	0.91	2.2	1.2
TOTAL			295.0			225.79			55.78
MOL. WT.			3.06			14.94			31.56
SCFH			36643.65			5734.55			670.78

TABLE 90

SRC II
MATERIAL BALANCE RUN NO. SR-3

GAS STREAM COMPOSITIONS

	FT 276 FRESH H2			FT 258 05 VENT GAS			FT 170 RCY CN SEP OH			FT 2134 RECYCLE GAS		
	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR
H2	98.97	88.4	158.1	77.51	23.2	57.4	76.79	21.9	74.8	77.51	23.2	52.7
N2	0.13	1.7	3.0	0.24	1.0	2.4	0.23	0.9	3.1	0.24	1.0	2.2
C1	0.30	2.1	3.8	14.65	34.9	86.2	14.33	32.5	110.8	14.65	34.9	79.1
CO	0.54	6.7	12.0	1.17	4.9	12.1	1.16	4.6	15.6	1.17	4.9	11.1
C2	0.00	0.0	0.0	3.51	15.7	38.7	3.40	14.5	49.3	3.51	15.7	35.6
CO2	0.06	1.1	1.9	0.11	0.7	1.8	0.64	4.0	13.6	0.11	0.7	1.7
C3	0.00	0.0	0.0	2.22	14.5	35.8	2.16	13.5	46.0	2.22	14.5	32.9
IC4	0.00	0.0	0.0	0.08	0.7	1.8	0.08	0.7	2.3	0.08	0.7	1.6
NC4	0.00	0.0	0.0	0.50	4.3	10.6	0.49	4.0	13.7	0.50	4.3	9.8
H2S	0.00	0.0	0.0	0.00	0.0	0.0	0.71	3.4	11.7	0.00	0.0	0.0
C5+	0.00	0.0	0.0	0.01	0.1	0.2	0.01	0.1	0.2	0.01	0.1	0.2
TOTAL			178.8			247.0			341.1			226.9
MOL. WT.			2.26			6.74			7.07			6.74
SCFH			30007.52			13918.62			18302.92			12776.72

	FT 144 & FT 85 PREHEATER FEED			FT 2117 03 SEP. GAS			FT 2107 LT. OIL/H2O SEPT.		
	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR
H2	95.18	63.6	186.3	35.62	3.4	11.5	0.05	0.0	0.0
N2	0.15	1.4	4.1	35.40	47.5	158.6	62.09	56.8	32.1
C1	2.96	15.7	46.0	6.33	4.9	16.2	10.99	5.8	3.3
CO	0.63	5.9	17.2	0.33	0.4	1.5	3.65	3.3	1.9
C2	0.59	5.9	17.2	4.54	6.5	21.8	4.28	4.2	2.4
CO2	0.05	0.7	2.0	6.46	13.6	45.5	10.73	15.4	8.7
C3	0.37	5.4	15.9	4.93	10.4	34.8	2.50	3.6	2.0
IC4	0.00	0.0	0.0	0.28	0.8	2.6	2.91	5.5	3.1
NC4	0.07	1.4	4.1	1.63	4.5	15.1	2.76	5.2	3.0
H2S	0.00	0.0	0.0	4.18	6.8	22.8	0.01	0.0	0.0
C5+	0.00	0.0	0.0	0.30	1.1	3.6	0.04	0.1	0.1
TOTAL			292.8			334.04			56.48
MOL. WT.			3.02			20.89			30.62
SCFH			36776.61			6068.58			699.93

TABLE 91

SRC II
MATERIAL BALANCE RUN NO. SR-4

GAS STREAM COMPOSITIONS

	FT 276 FRESH H2			FT 258 05 VENT GAS			FT 2117 03 SEP. GAS			FT 170 RCY CN SEP OH		
	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR
H2	98.50	83.0	152.6	83.02	28.7	66.3	32.91	3.1	9.4	82.54	28.5	81.8
N2	0.15	1.8	3.2	0.22	1.1	2.5	47.26	61.6	188.2	0.22	1.0	3.0
C1	0.49	3.3	6.0	10.50	28.8	66.6	2.80	2.1	6.4	10.58	29.0	83.2
CO	0.58	6.8	12.5	1.07	5.2	11.9	0.18	0.2	0.7	1.08	5.2	14.9
C2	0.00	0.0	0.0	2.12	10.9	25.2	2.52	3.5	10.8	2.50	12.9	36.9
CO2	0.28	5.1	9.3	0.07	0.6	1.3	7.42	15.2	46.4	0.52	3.9	11.1
C3	0.00	0.0	0.0	2.10	15.8	36.6	3.29	6.7	20.6	1.60	12.1	34.6
IC4	0.00	0.1	0.1	0.46	4.6	10.7	0.15	0.4	1.2	0.06	0.6	1.9
NC4	0.00	0.0	0.0	0.32	3.2	7.4	1.16	3.1	9.6	0.36	3.6	10.3
H2S	0.00	0.0	0.0	0.05	0.3	0.7	2.18	3.4	10.5	0.53	3.1	8.9
C5+	0.00	0.0	0.0	0.06	0.8	1.8	0.15	0.5	1.6	0.00	0.0	0.0
TOTAL			183.7			231.0			305.4			286.6
MOL. WT.			2.40			5.84			21.49			5.84
SCFH			29099.76			13003.60			5394.20			18608.57

	FT 2134 RECYCLE GAS			FT 144 & FT 85 PREHEATER FEED			FT 2107 LT. OIL/H2O SEPT.		
	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR
H2	83.02	28.7	38.4	95.85	64.1	170.7	1.46	0.1	0.1
N2	0.22	1.1	1.4	0.16	1.4	3.8	73.94	64.8	40.1
C1	10.50	28.8	38.5	2.13	11.3	30.1	0.01	0.0	0.0
CO	1.07	5.2	6.9	0.63	5.8	15.6	0.27	0.2	0.1
C2	2.12	10.9	14.6	0.29	2.9	7.6	0.84	0.8	0.5
CO2	0.07	0.6	0.7	0.49	7.1	19.0	10.60	14.6	9.0
C3	2.10	15.8	21.2	0.34	4.9	13.1	4.42	6.1	3.8
IC4	0.46	4.6	6.2	0.09	1.7	4.6	0.45	0.8	0.5
NC4	0.32	3.2	4.3	0.03	0.6	1.6	3.86	7.0	4.3
H2S	0.05	0.3	0.4	0.00	0.0	0.0	3.31	3.5	2.2
C5+	0.06	0.8	1.0	0.00	0.0	0.0	0.83	1.9	1.2
TOTAL			133.6			266.1			61.84
MOL. WT.			5.84			3.02			31.95
SCFH			8683.59			33451.64			734.58

TABLE 92

SRC II
MATERIAL BALANCE RUN NO. SR-5

GAS STREAM COMPOSITIONS

	FT 276 FRESH H2			FT 258 05 VENT GAS			FT 2117 03 SEP. GAS			FT 170 RCY CN SEP OH		
	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR
H2	98.64	86.4	160.6	87.64	37.5	82.4	35.65	3.4	10.6	85.99	33.4	98.8
N2	0.14	1.7	3.2	0.20	1.2	2.6	46.42	62.0	191.3	0.20	1.1	3.2
C1	0.59	4.1	7.6	7.50	25.5	56.0	1.05	0.5	2.5	7.95	24.5	72.5
CO	0.61	7.5	13.9	0.88	5.2	11.5	0.28	0.4	1.2	0.89	4.8	14.2
C2	0.00	0.0	0.0	1.91	12.2	26.7	2.03	2.9	9.0	2.01	11.6	34.4
CO2	0.02	0.3	0.6	0.11	1.0	2.2	7.35	15.4	47.6	0.34	2.9	8.6
C3	0.00	0.0	0.0	1.32	12.4	27.1	3.05	6.4	19.7	1.38	11.7	34.6
IC4	0.00	0.0	0.0	0.04	0.5	1.1	0.14	0.4	1.2	0.05	0.5	1.6
NC4	0.00	0.0	0.0	0.32	4.0	8.8	1.21	3.4	10.4	0.55	3.9	11.5
H2S	0.00	0.0	0.0	0.08	0.6	1.2	2.68	4.4	13.4	0.84	5.5	16.4
C5+	0.00	0.0	0.0	0.00	0.0	0.0	0.15	0.5	1.7	0.00	0.0	0.0
TOTAL			185.9			219.6			308.6			295.8
MOL. WT.			2.31			4.72			20.97			5.20
SCFH			30586.14			17673.42			5582.17			21594.31

	FT 2134 RECYCLE GAS			FT 144 & FT 85 PREHEATER FEED			FT 2107 LT. OIL/H2O SEPT.		
	MOL%	WT%	#/HR	MOL%	WT%	#/HR	MOL%	WT%	#/HR
H2	87.64	37.5	22.3	97.37	76.3	190.5	1.02	0.1	0.0
N2	0.20	1.2	0.7	0.15	1.7	4.1	70.68	60.2	38.6
C1	7.50	25.5	15.2	1.44	8.9	22.3	0.04	0.0	0.0
CO	0.88	5.2	3.1	0.65	7.1	17.7	0.11	0.1	0.1
C2	1.91	12.2	7.2	0.17	2.0	5.1	0.75	0.7	0.4
CO2	0.11	1.0	0.6	0.01	0.3	0.6	10.66	14.3	9.1
C3	1.32	12.4	7.3	0.13	2.2	5.4	4.62	6.2	4.0
IC4	0.04	0.5	0.3	0.05	1.1	2.6	0.60	1.1	0.7
NC4	0.32	4.0	2.4	0.02	0.5	1.2	5.21	9.2	5.9
H2S	0.08	0.6	0.3	0.00	0.0	0.0	4.94	5.1	3.3
C5+	0.00	0.0	0.0	0.00	0.0	0.1	1.37	3.1	2.0
TOTAL			59.4			249.6			64.1
MOL. WT.			4.72			2.58			32.90
SCFH			4778.23			36752.70			739.63

D. High Rate Study

Process Evaluation work during the SRC I production run period involved the collection and interpretation of plant data obtained during plant operating periods. A study was made on the process effects of a high coal feed rate during the SRC I production run. During this study, coal feed rates in excess of plant design were achieved. The design coal rate of the plant is 50 tons per day, or about 4200 pounds per hour of raw coal. Tables 93 through 98 show operating data obtained during a period when design rates were exceeded. The purpose of presenting all of these conditions is to provide a fairly complete set of operating conditions obtained during high rate periods.

TABLE 93

SRC-I HIGH RATE OPERATION

SLURRY PREPARATION

Operating Period: 0000 10-7-76 through 2400 10-8-76

Raw Coal Feed, lbs/hr	4795
Water Removed From Coal, lbs/hr	341
Dehumidified Coal Feed, lbs/hr	4454
Moisture Free Coal Feed, lbs/hr	4427
Solvent Feed, lbs/hr	6674
Solvent to Dehumidified Coal Ratio	1.50
Slurry Blend Tank Temperature, °F	176
Inert Gas Temperature To Pulverizer, °F	450
Slurry Feed To Area 02, lbs/hr	11128

TABLE 94

SRC-I HIGH RATE OPERATION

PREHEATING & DISSOLVING

Slurry Feed From Area 01, lbs/hr	11128
Hydrogen-Rich Gas Feed, To Preheater, lbs/hr	426.7
Hydrogen-Rich Gas Feed To Instrument Purges, lbs/hr	85.3
Hydrogen-Rich Gas Feed To Preheater, SCFH	36083
Hydrogen Purity Of Feed Gas, mole%	88.1
Preheater Inlet Temperature, °F	161
Preheater Outlet Temperature, °F	750
Pressure Drop Through Preheater, psi	80.3
Dissolver Volume (1/2 Dissolver A), Ft^3	50.8
Dissolver Pressure, psig	1547
Dissolver Temperatures, °F	
Transfer Line 12 Ft Ahead Of Inlet	760
Inlet	761
4 Ft Above Inlet	831
8 Ft Above Inlet	838
12 Ft Above Inlet	851
Water Quench, lbs/hr	1267
High Pressure Flash (HPF) Temperature, °F	669
Recycle Condensate Separator (RCS) Temp., °F	104
Gas Flow From RCS, lbs/hr	614
Liquid Flow From RCS, lbs/hr	2895
Slurry Flow From HPF, lbs/hr	9398
Intermediate Pressure Flash Pressure, psig	621

TABLE 95

SRC-1 HIGH RATE OPERATION

FILTRATION

AREA 03	FILTER A	FILTER B
Filter Area, ft^2	80	40
Number of Cycles	1	2
Time Off Filtration, %	37.5	56.2
Time On Filtration, %	62.5	43.8
Total Hours On Filtration	30	21
Filtration Rate For Hours On Filtration, lbs/hr	10057	7377
Wash Solvent To Cake, lbs/hr	1615	1400
Net Filtrate, lbs/hr	8442	5977
Filtrate Rate, lbs/hr	106	149
Filter Temperature, °F	471	499
Drum Speed, RPM	2	2.5
Knife Advance, mil/min	1.62	2.6
Filter Differential Pressure, PSI	30	30
Wash Solvent To Cake Leg, lbs/hr	400	335

Precoat Composition:	Run 117-A-2	Run 128 B	Run 129 B
Speedex, lbs	1000	550	550
Celite 535, lbs	0	50	50

TABLE 96

SRC-I HIGH RATE OPERATION

MINERAL SEPARATION & DRYING

Filter Feed Flash (FFF) Temperature, °F	541
FFF Pressure, psig	83
Filter Feed Surge (FFS) Temperature, °F	511
FFS Pressure, psig	72
Mineral Residue (MR) Dryer Pressure, In H_2O	0.4
MR Dryer Preheat Zone Temperature, °F	814
MR Dryer Cylinder Skin Temperatures, °F	
#1	330
#2	960
#3	1100
#4	1210
#5	1270
MR Dryer Hold Zone Temperature, °F	660
MR Dryer Condensate Temperature, °F	87

TABLE 97

SRC-I HIGH RATE OPERATION

SOLVENT RECOVERY

Light Ends Column(LEC) Feed Tank Temperature, °F	372
LEC Feed Temperature, °F	362
LEC Reboiler Temperature, °F	570
LEC Feed, lbs/hr	13000
LEC Bottoms, lbs/hr	10443
Vacuum Flash Feed Accumulator Temperature, °F	521.2
Fresh Feed To V.F. Heater, lbs/hr	8700
Recycle Feed To V.F. Heater, lbs/hr	7820
Total Feed To V.F. Heater, lbs/hr	16520
Ratio Of Recycle To Fresh V.F. Feed	0.9
V.F. Heater Feed Temperature, °F	600
V.F. Heater Outlet Temperature, °F	684
V.F. Heater Outlet Pressure, psig	159
Pressure Drop Through V.F. Heater, psi	32.8
V.F. Drum Temperature, °F	586
V.F. Drum Pressure, psia	3.3
V.F. Condensate Drum Temperature	167
Wash Solvent Column (WSC) Reboiler Temperature, °F	493
WSC Reflux Drum Temperature, °F	280
WSC Overhead Temperature, °F	430
WSC Reflux, lbs/hr	4129
WSC Bottoms, lbs/hr	7750
Stripper Column Pressure, psig	11.3
SRC To Storage, lbs/hr	2500

TABLE 98

SRC-I HIGH RATE OPERATION

GAS TREATMENT AND RECOMPRESSION

DEA Column Feed, lbs/hr	614.2
DEA Outlet Gas to Recycle Compressor, lbs/hr	319
DEA **Outlet** Gas To Flare, lbs/hr	184
Fresh Feed Gas To Recycle Compressor, lbs/hr	276
Recycle Compressor Feed To Area 02, lbs/hr	512
Recycle Gas Composition, mole%	
H_2	88.1
N_2	0.4
CO	3.1
CO_2	0.0
C_1	6.6
C_2	1.2
C_3	0.4
C_4	0.1
C_5	0.1
H_2S	0.0

E. Special Studies

1. Reaction

a. Dissolver Solids Deposits

Two thorough inspections were made to determine the extent of coke deposits in the dissolver - one in September 1976 and one in February 1977. A previous visual inspection in May 1975 had shown considerable coke had accumulated in the dissolver. Since build-ups of significant quantities of coke could affect the reactor volume, measurement of internal dissolver temperatures or hyrodynamic and chemical reaction characteristics, it was necessary to gather information concerning the extent of these effects.

The first detailed inspection of the dissolver internals was made during a plant shutdown in September 1976. Visual observations revealed that, although little or no coke was present above the dissolver side draw off nozzle, a substantial quantity was present below the nozzle. Approximately 15 ft^3 of the bottom section of the dissolver (about 30% of the volume of that section) was filled with coke. This resulted in an apparent free volume of about 35 ft^3 for half-dissolver operation.

Diagrams of the coke deposit before and after hydroblasting and cleaning are shown in Figure 54. An annulus was observed in the coke deposit at the side outlet as expected. Surprisingly, however, the coke deposit had no large central core for fluid flow, containing instead numerous small flow channels.

Analyses of samples of dissolver coke are shown in Table 99. A comparison of the sample results indicates that the composition of the dissolver coke is not uniform.

The dissolver temperature profiles before and after the September shutdown are shown in Figure 55. These profiles show that the coke-free dissolver exhibited a much greater temperature exotherm than before hydroblasting. A profile obtained after the clean dissolver was operated for two weeks is also shown. This third profile indicates that, since the startup following the hydroblasting, a decrease in exotherm had occurred. However, the exotherm shown was greater (by about 20°F) than before the dissolver was hydroblasted. Additonal data collected from September 1976 to January 1977 verified the continually decreasing exotherm, as shown in Figure 56.

MAP OF DISSOLVER "A" COKE DEPOSITS

SEPTEMBER 1976

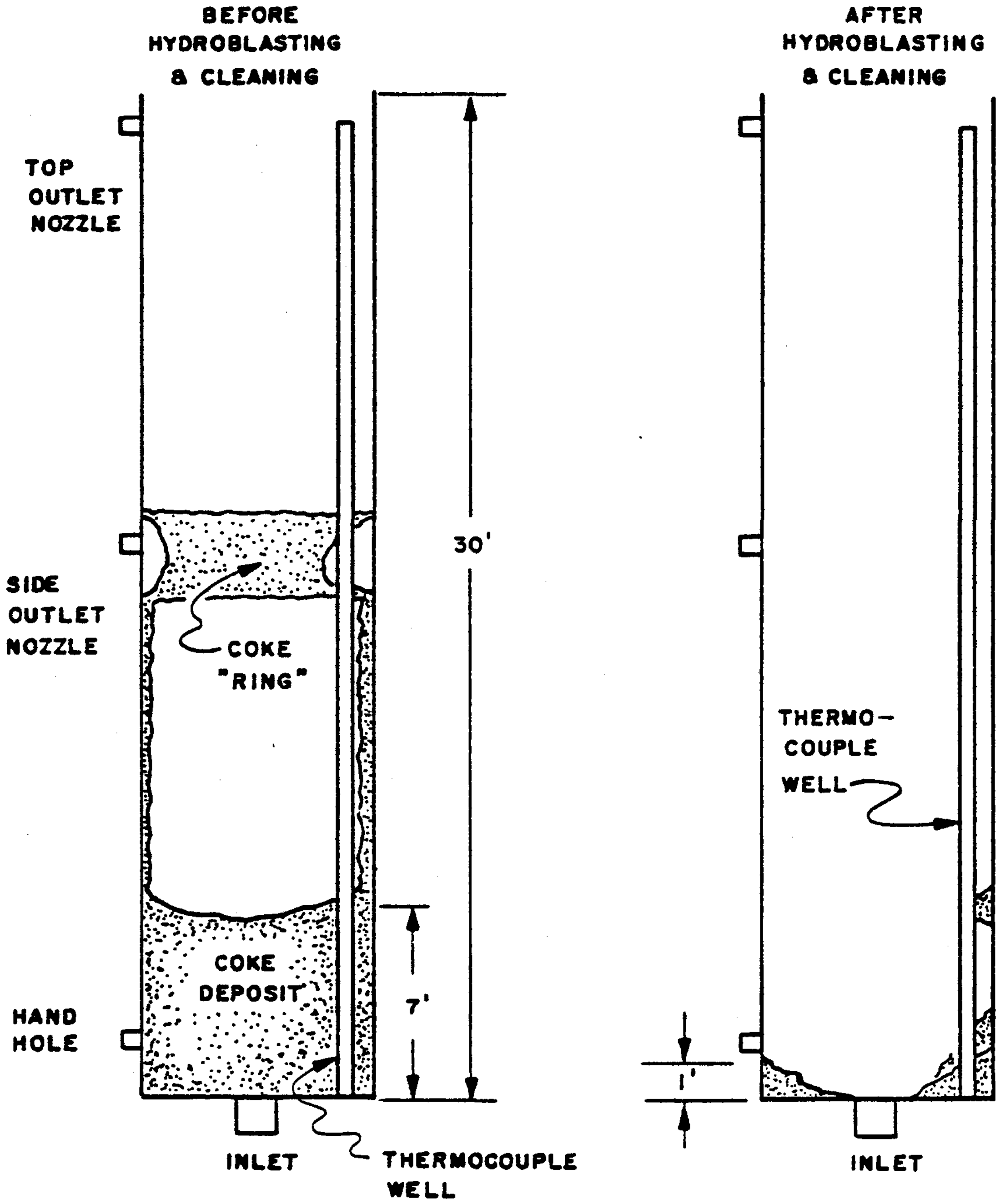


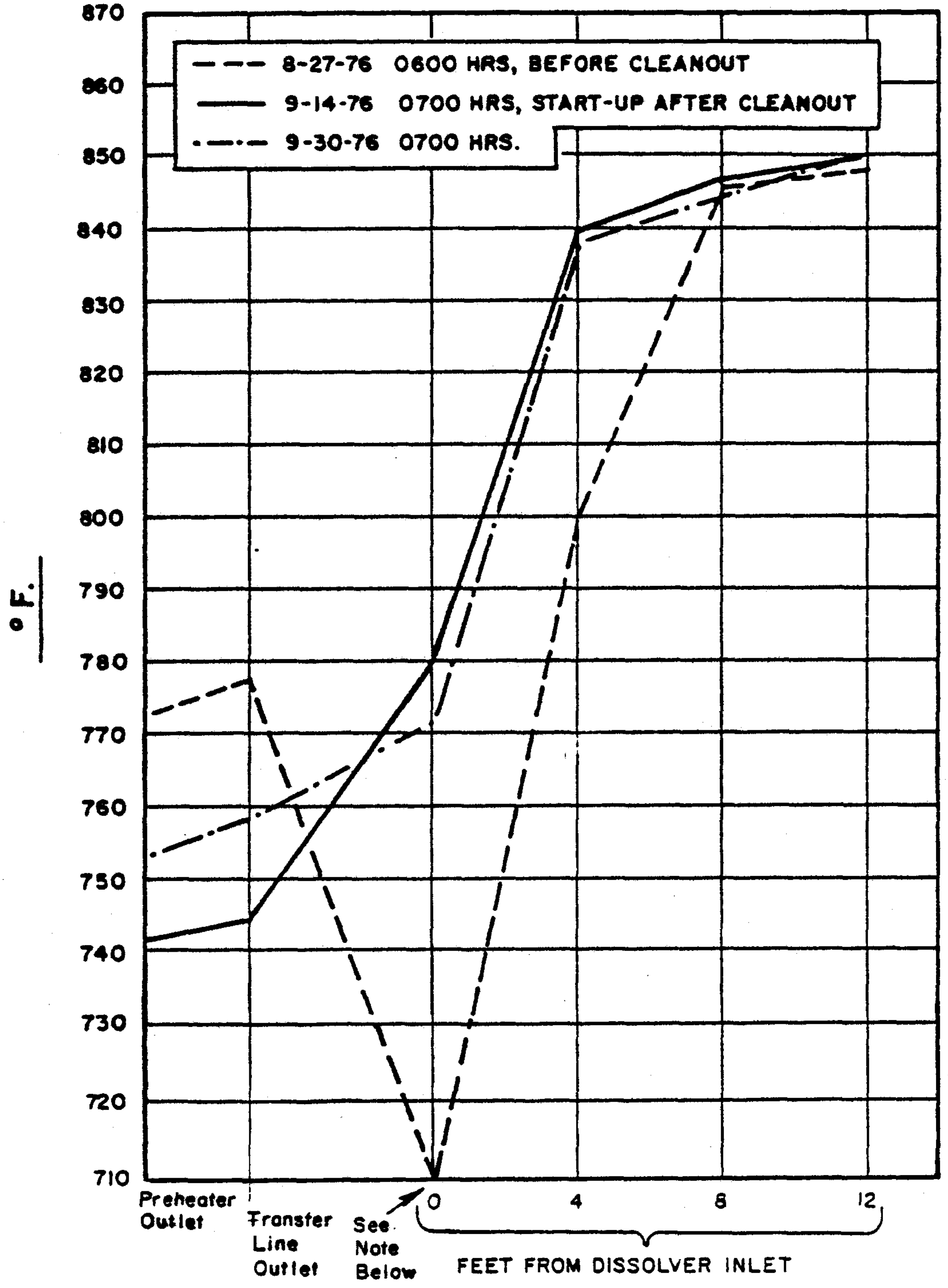
FIGURE 54

TABLE 99

DISSOLVER COKE ANALYSES

Elemental Analysis	September 1976 Coke Removed From Hole Core	September 1976 Bricklike Coke at Start of Hydroblasting	September 1976 Bricklike Coke after 48 Hours of Hydroblasting	February 1977 Transfer Line
C	36.2	49.7	53.1	50.7
H	3.8	2.3	1.9	2.2
N	0.7	0.7	1.0	0,6
S	8.3	2.7	5.7	7.9
Ash	23.2	45.4	41.3	42.3
FE	13.3	5.2	9.7	4.8

REACTION AREA TEMPERATURE PROFILES



NOTE: THERMOWELL AT 0 FEET APPARENTLY GIVES A LOWER THAN ACTUAL READING.

FIGURE 55

DISSOLVER TEMPERATURE PROFILE HISTORY
AT 3700 LBS/HR COAL FEED

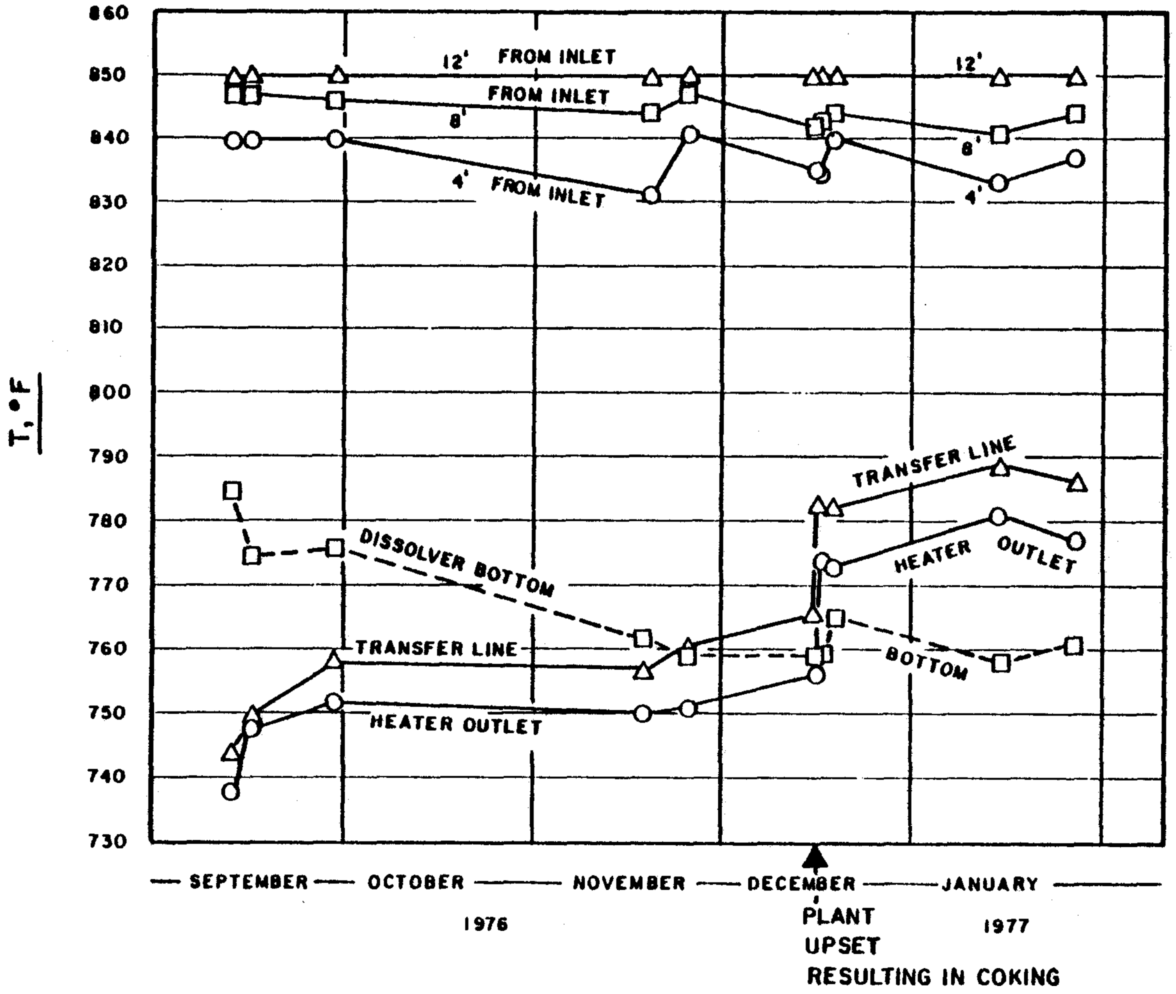


FIGURE 56

The dissolver was reinspected in February 1977 for expected coke deposits as predicted by the severely reduced temperature exotherm since the September 1976 cleaning. A measurement of the dissolver volume showed a loss of approximately 4 ft^3 due to coke formation in 50.8 ft^3 half-dissolver volume.

The coke deposit was found to be very hard and was not completely removed by the first hydroblasting. Laboratory analysis of a sample taken from the transfer line, which was found to have approximately 50% or more of the cross-section area blocked by coke deposits, is shown in Table 99. Analysis of this sample showed similarities with the previous dissolver samples.

The absence of a substantial quantity of coke appeared to refute the theory that the decrease in dissolver exotherm was due to the formation of coke deposits in the dissolver. Two other explanations were hypothesized to account for the decreased exotherm:

- Coke deposits were formed by the gradual buildup of small coke particles, which were removed during shutdown and flushing operations.
- The measured solvent-to-coal ratio was not the same as the actual ratio. (The apparent solvent-to-coal ratio in December and January, determined by plant instrumentation, was 1.5 - 1.6, however, the lab measured an average solvent-to-coal ratio of 1.24 in January).

b. Dissolver Temperature Studies

Several investigations of the dissolver temperature profile have been conducted in an effort to gain an understanding of the flow mechanisms in the reaction area. These studies have dealt with trying to define slurry residence times in the dissolver and transfer line upstream of the dissolver and in identifying typical dissolver exotherms. Some effort has been expended in identifying the changes in the dissolver which occur when coal is injected into the system.

During SRC-I operations, the dissolver outlet temperature was controlled by varying the heating conditions in the preheater. The lag time between a response in the preheater outlet temperature and the subsequent response in the dissolver outlet temperature was expected to be a significant portion of the total system lag time. To estimate the length of the lag, a series of step changes in the heater firing rate were made during a period when hydrogen and solvent were being recirculated in the system, but coal was not being fed. By eliminating the dynamic effects of the coal hydrogenation exotheric reactions, it was felt that a reasonable approximation to actual system lag times could be made. The preheater outlet, dissolver inlet and dissolver outlet temperatures were monitored during the step changes in preheater firing rate. The resultant temperature responses are shown in Figure 57. The initial decrease in preheater firing rate was sudden and resulted in a nearly instantaneous step change in preheater outlet temperature. This sudden drop in preheater outlet temperature resulted in subsequent decreases in the dissolver inlet and dissolver outlet temperatures. By suddenly increasing the heater firing rate, a rapid increase in heater outlet temperature began to occur. This sudden change resulted in the trough shown in Figure 57. The trough was subsequently followed by other troughs, first in the dissolver inlet and finally in the dissolver outlet. The dissolver inlet trough trailed the preheater outlet by about 18 minutes and the dissolver outlet trailed the preheater outlet by 25 minutes. The data shown in Figure 57 was obtained at solvent feed rates of about 8700 lbs/hr and hydrogen feed rates of about 170 lbs/hr, common conditions at low coal feed rates.

Aside from providing an indication of the lag times in the reaction area, the curves in Figure 57 also provide some insight into the flow mechanisms involved. The shape of the peaks gives the best indication of this. The preheater outlet curve has a very abrupt minimum and was chosen that way to approximate an impulse change in the system. By following that impulse through the system, and observing how the shape of the curve changes, it is possible to gain some insight into the degree of backmixing. Note that the dissolver inlet curve has approximately the same shape as the heater outlet. There is a slight spreading of the curve, which does indicate a small amount of backmixing but, overall, the close resemblance to the preheater outlet response suggests that flow in the transfer line between the preheater and dissolver is probably close to plug flow, even though three phase flow (gas, liquid, solid) is involved.

The curve in Figure 57 which represents the dissolver outlet temperature is considerably different in shape than the other two curves and this leads to a different interpretation of the flow regime in the dissolver. The absence of the sharp trough observed in the preheater outlet curve suggests

SRC I REACTION AREA TIME/TEMPERATURE PROFILE

AFTER STEP CHANGE IN HEATER TEMPERATURE

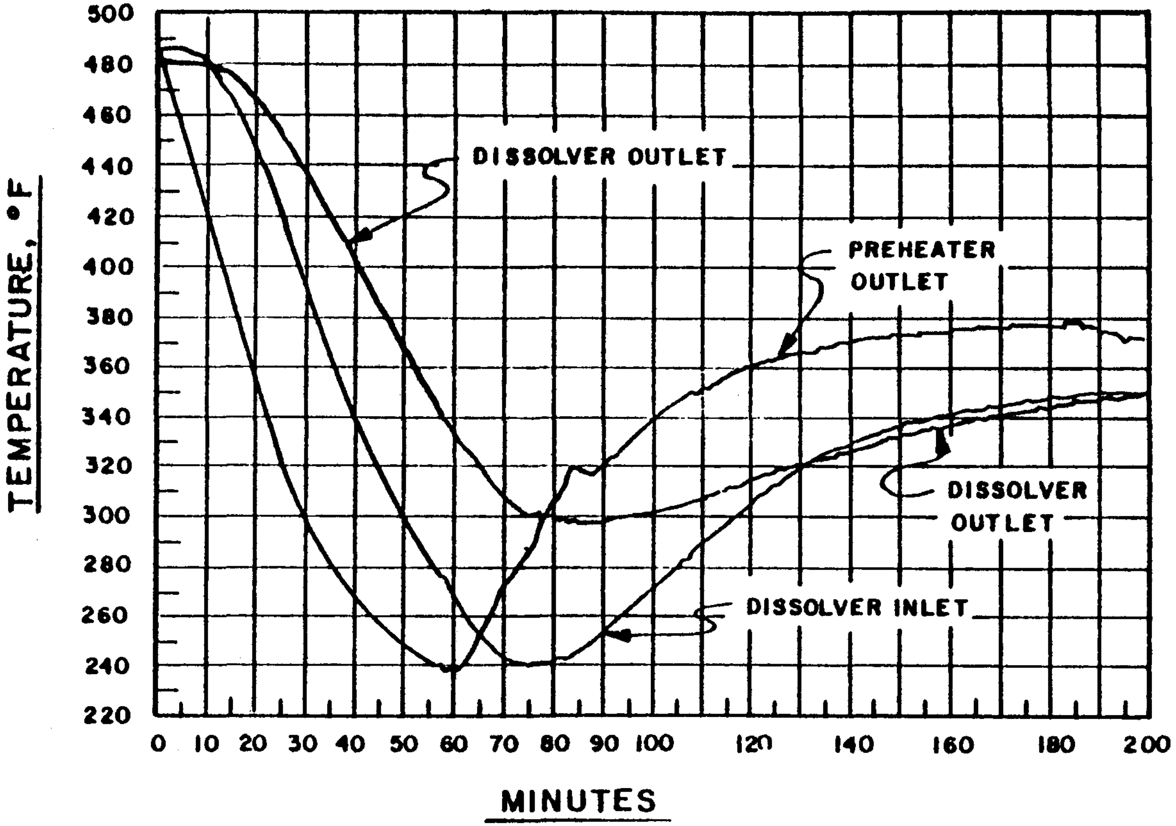


FIGURE 57

substantial backmixing in the dissolver. The rapidity with which the dissolver outlet responded (7 minutes after the inlet) is also of interest. The transfer line between the heater and dissolver has a volume of less than 20 ft^3 and responded to the temperature change in 18 minutes. In comparison, the dissolver, with a volume of about 50 ft^3, indicated a minimum outlet temperature only 7 minutes after the minimum inlet temperature. The dissolver outlet temperature curve in Figure 57 was very highly dumped, resulting in a much smaller trough than for the preheater outlet or dissolver inlet. All of this led to the conclusion that the dissolver was heavily backmixed and channelling of flow through the dissolver occurred. (The channelling would explain the short time required for the trough in the dissolver outlet to occur.)

Figure 58 shows the temperature response of the same three points (preheater outlet, dissolver inlet and dissolver outlet) during a period when the plant was running. This particular period was chosen because of the peaks and trough which were observed, and which were due to a brief coal outage, which was followed by coal injection. The trailing effect which was used to estimate lag time, as discussed above, is apparent, as is the dispersion and dampening of the dissolver outlet temperature curve.

An investigation was also made into the difference in the reaction area profile at moderate (3700 lbs/hr) and low (2700 lbs/hr) coal feed rates. This study was conducted over a several month period and corresponds to the same time frame shown in Figure 56. During that period, both moderate and low rates were run in the plant and conditions were held long enough to establish apparent equilibrium in the dissolver. Due to the sudden change in exotherm on December 15, 1976, which has been discussed above, the time span in Figure 56 was split into two periods and average profiles for the 3700 lbs/hr and 2700 lbs/hr coal feed rates were calculated. The results are presented in Table 100.

Both periods show that the overall exotherm between the heater outlet and dissolver outlet are greater at the low coal feed rate. Each period shows that in the case of low flow, while the dissolver inlet temperature is lower than in the moderate flow case, the temperature at the thermocouple located 4 ft. above the dissolver inlet is higher. This indicates a greater exotherm at the dissolver inlet in the low flow case. There are several reasons for this difference in exotherms between the low flow and moderate flow case: more sensible heat require-

SRC I REACTION AREA TIME/TEMPERATURE PROFILE

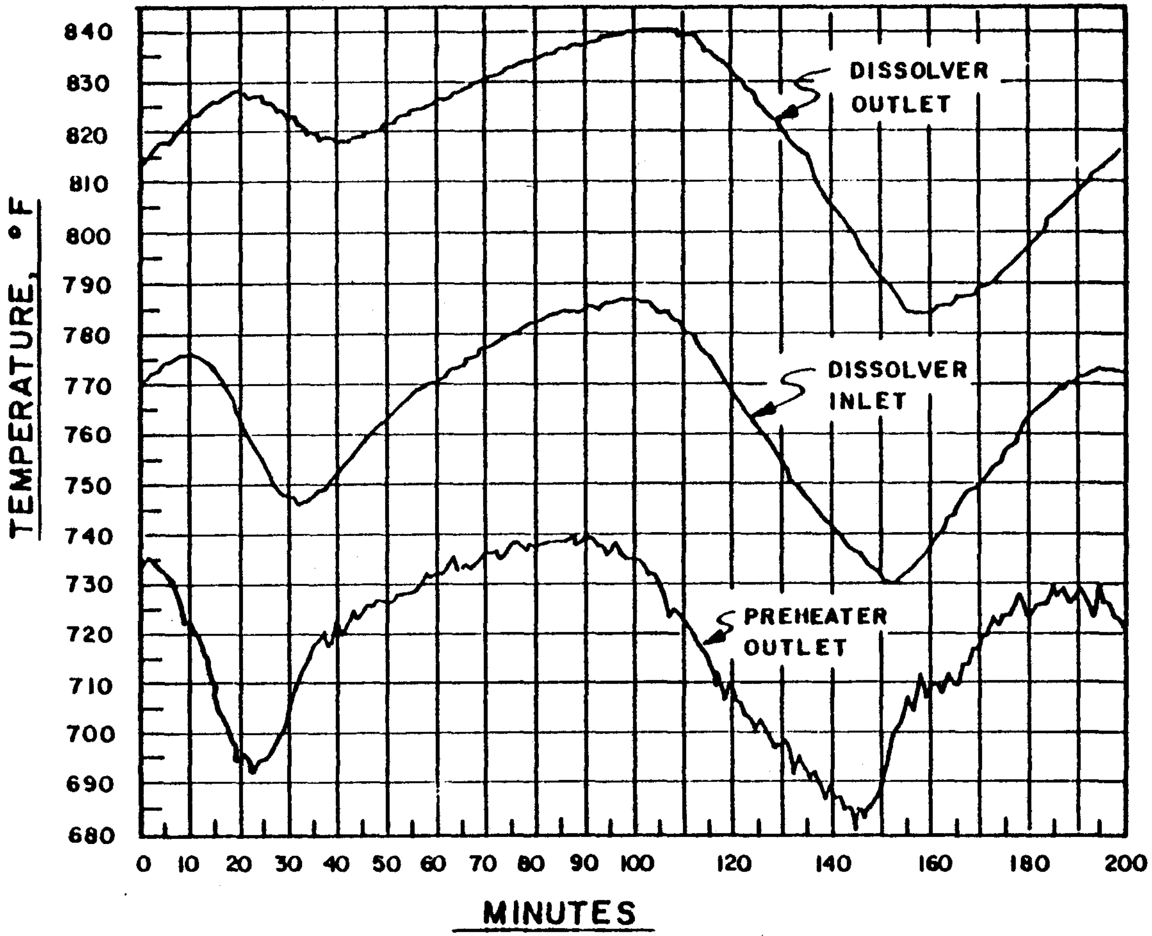


FIGURE 58

TABLE 100

REACTION AREA AVERAGE TEMPERATURE PROFILES

		Period I		Period II	
Coal Rate, lbs/hr		2700	3500	2700	3500
Temperatures, °F					
	TE 105	738	753	751	776
	TE 1542	745	761	760	784
	TE 1382	842	836	838	833
	TE 1382	847	844	845	843
	TE 1384	850	850	850	850

Period I includes data obtained from 11/4/76 through 2100 hrs. 12/14/76.

Period II includes data obtained from 0000 hrs. 12/15/76 through 1/26/77.

Thermocouple Locations:

TE 105:	Preheater Outlet
TE 1542:	Transfer Line upstream of Dissolver Inlet
TE 1382:	Dissolver, 4 ft from inlet
TE 1383:	Dissolver, 8 ft from inlet
TE 1384:	Dissolver, 12 ft from inlet

ments in the moderate flow case, decreased liquid residence time in the moderate flow case and a decreased hydrogen concentration in the moderate flow case (possible mass transfer limitations). The data presented here do not eliminate any of these possiblities but are useful in the sense that they indicate the dissolver inlet is a very crucial reaction and/or heat transfer area.

The data in Table 100 also show that, after the sudden change in exotherm on December 15, 1976, the temperature at lower levels in the dissolver were lower than in Period I. This indicates that more exothermic hydrogenation was occurring in the upper section of the dissolver, contributing to the total exotherm.

c. Effluent Heat Capacities

The bulk heat capacity of the dissolver effluent stream was determined by observing the overall temperature change between the dissolver and the high pressure flash drum and the quantity of water quench added to the dissolver effluent. Heat transfer was calculated as the change in enthalpy of saturated water (corrected for pressure) from 80°F to the high pressure flash drum temperature. The bulk heat capacity was then calculated from the following equation:

$$Cp = \frac{Q}{M\Delta T}$$

where:

Cp - Bulk heat capacity (BTU/lb °F)

Q = Heat Transfer (BTU/hr)

M = Dissolver effluent rate (lb/hr), calculated from feed rates.

ΔT = Difference in temperature in °F from the dissolver effluent to the high pressure flash drum (HPFD).

Bulk heat capacities were calculated from available data taken during SRC-I material balance runs 1 through 11 and are presented in Table 101. Heat capacities for slurry recycle material balances SR-1 through SR-5 were calculated in a similar manner and are presented in Table 102.

TABLE 101

CALCULATED DISSOLVER EFFLUENT HEAT CAPACITIES FOR SRC-I MATERIAL BALANCE RUNS

RUN NUMBER	MBR-1	MBR-2	MBR-3	MBR-4	MBR-5	MBR-6	MBR-7	MBR-8	MBR-9	MBR-10	MBR-11
Water Quench (lb/hr)	1589	842	1545	1326	1900	2052	1852	1394	1840	1720	1360
Coal Feed (lb/hr)	4624	2728	3867	4018	5011	5049	5055	4066	4657	3735	3784
Gas Feed (lb/hr)	281	195	332	326	368	377	360	312	345	315	354
Solvent Feed (lb/hr)	7665	4272	6378	6310	8039	8032	8042	6306	7158	6032	5999
Recycle (lb/hr)	0	0	0	0	0	0	0	0	0	0	0
Total, less water (lb/hr)	12570	7194	10577	10654	13418	13458	13457	10684	12160	10082	10137
System Pressure (psig)	1528	1489	1496	1498	1488	1482	1471	1484	1478	1027	1261
Temperature: Dissolver Effluent (°F)	851	848	870	844	849	870	850	846	859	842	848
HPFD (°F)	614	610	602	610	601	600	602	604	594	516	583
ΔT (°F)	237	238	268	234	248	270	248	242	265	326	265
ΔH Water (BTU/lb)	1122.8	1122.8	1122.8	1122.8	1122.8	1122.8	1122.8	1122.8	1137.9	1148	1163
Q (BTU/hr x 10^{-6})	1.784	0.9454	1.735	1.489	2.133	2.304	2.080	1.565	2.093	1.974	1.582
Cp (BTU/hr/°F)	0.60	0.55	0.61	0.60	0.64	0.63	0.62	0.61	0.65	0.60	0.59
ΔH_R (BTU/ft^3 H_2 reacted)	34.6	28.3	43.4	39.7	39.3	43.0	42.0	34.9	44.6	18.7	26.1

TABLE 102
DISSOLVER EFFLUENT HEAT CAPACITIES
FOR SRC-II MATERIAL BALANCE RUNS

RUN NUMBER	SR-1	SR-2	SR-3	SR-4	SR-5
Water Quench (lb/hr)	1407	1372	599	731	585
Coal Feed (lb/hr)	1832	2038	2025	1617	1353
Gas Feed (lb/hr)	284	295	293	231	250
Solvent Feed (lb/hr)	1779	1885	700	453	311
Recycle (lb/hr)	6007	7037	7473	7531	7554
Total, less water (lb/hr)	9902	11255	10491	9832	9468
System Pressure (psig)	1478	1765	1878	1883	1877
Temperature:					
Dissolver Effluent (°F)	849	871	861	862	851
HPFD (°F)	613	630	625	627	627
ΔT (°F)	236	241	236	235	224
ΔH Water (BTU/lb)	1148	1132	1092	1145	1145
Q (BTU/hr x 10^{-6})	1.615	1.553	0.653	0.798	0.638
Cp (BTU/hr/°F)	0.69	0.57	0.26	0.34	0.30
ΔH_R (BTU/ft^3 H_2 reacted)	39.0	43.9	23.3	25.5	29.1

d. Dissolver Heat of Reaction Calculations

By estimating the sensible heat necessary to raise the reaction products from the dissolver inlet temperature to the dissolver outlet temperature, the dissolver heat of reaction was calculated. Heats of reaction ranging from 50-60 Btu per cubic foot of hydrogen consumed were calculated using this method.

An alternative method of calculation yielded the heat of reaction data shown in Tables 101 and 102. The method used to calculate those values assumes that the effluent bulk heat capacity can be used as an approximation of the heat capacity of the inlet stream. Using this value for the bulk heat capacity, the slurry and gas feed rates, and the temperature difference between the preheater outlet and dissolver outlet, the heat of reaction was calculated as the overall change in enthalpy in the system, based on the following equation:

$$\Delta H_R = MCp\Delta T$$

This technique is subject to several sources of error, the most severe of which are the assumed bulk heat capacity and the hydrogen consumption. The values shown in Tables 101 and 102 are useful in that they do provide an indication of the overall heat of reaction in the SRC process.

e. Slurry Preheater

(1) Heat of Dissolution

From analyses of the combustion gases leaving the slurry preheater and estimates of convective and sensible heat transfer, the heat of reaction for coal dissolution in the SRC-I process for Kentucky coal has been calculated. An endothermic heat of reaction ranging from 150-250 Btu/lb. dry coal has been estimated.

(2) Pressure Drop Correlation

The pressure drop across the slurry preheater was closely monitored during plant operation at low solvent-to-coal ratios. Slurry feed rates varied between 6000 and 9000 lbs/hr, and the solvent-to-coal ratio varied between 1.2 and 1.7 during the test period. The relationship of solvent to coal ratio and slurry feed rate to pressure drop was studied and two statistical models were developed. Table 103 shows the models and the data used to develop them. The single most important variable was found to be solvent to coal ratio. Slurry feed rate did not produce a significant model when used alone as an independent variable, however, when used

TABLE 103

SRC-I SLURRY PREHEATER PRESSURE DROP DATA

1500 psig — Kentucky Coal

SOLVENT TO COAL RATIO	SLURRY FEED, LB/HR	ΔP, psi
1.21	7093.0	124.8
1.27	6552.3	108.8
1.30	6565.7	120.7
1.31	6665.4	99.5
1.35	6615.4	112.6
1.38	8482.9	114.5
1.41	8495.2	109.4
1.45	8806.0	105.9
1.66	6454.1	73.5
1.71	6364.2	54.0

MODELS

1) One Variable

$$\Delta P = 276.088 - 123.643 \text{ [S/C Ratio]}$$

(83.8% of variation explained by model)

2) Two Variables

$$\Delta P = 226.892 - 120.056 \text{ [S/C Ratio]} + 6.125 \times 10^{-3} \text{ [Feed]}$$

(91.2% of variation explained by model)

together with solvent to coal ratio, a statistically significant model resulted.

A plot of the correlation between pressure drop and solvent to coal ratio is shown in Figure 59 along with the data obtained during the study.

An additional extensive study, now underway, has been directed toward developing an overall pressure drop model based on the data from the Wilsonville, Alabama SRC pilot plant and the Ft. Lewis plant. That study will model the pressure drop as a function of coal concentration in the feed slurry, preheater inlet pressure, slurry feed rate, preheater diameter, coal slurry density, gas feed rate and average preheater temperature.

(3) Preheater Outlet Samples

Two samples of the preheater effluent were taken during the production run. Both samples were obtained when the plant was operating in a typical manner (preheater outlet of 750-790°F).

The first sample was analyzed for pyridine insoluble material, ash and blackness and was vacuum distilled in a manner similar to that used for unfiltered coal solution. Results of these analyses are shown in Table 104. For comparison, the analysis of an unfiltered coal solution sample (obtained at the liquid outlet of the high pressure flash drum) is also shown.

The data in Table 104 indicate that, at the preheater outlet, the process solvent, which was used to formulate the coal slurry, has become bonded to the coal matrix. This bond did not break even under vacuum distillation at 500°F, so it appears to be stable. Comparison with the unfiltered coal solution (UFCS) shows that in the dissolver, process solvent is released from the coal matrix. The data in Table 104 also show that most of the coal is readily converted to pyridine soluble material in the preheater at the conditions maintained during sampling. Therefore, the function of the dissolver in this case is to convert a pyridine soluble high molecular weight material to process solvent and SRC.

Analytical results obtained from the second sample are shown in Table 105. An attempt was made to determine the coal conversion at the preheater outlet, however the pyridine solubility test which is used to evaluate coal conversion resulted in widely scattered, non-reproducible results. Plugging of the filter paper

SLURRY PREHEATER PRESSURE DROP
VS
SOLVENT TO COAL RATIO
SRC-I - KENTUCKY COAL - 1500 PSIG

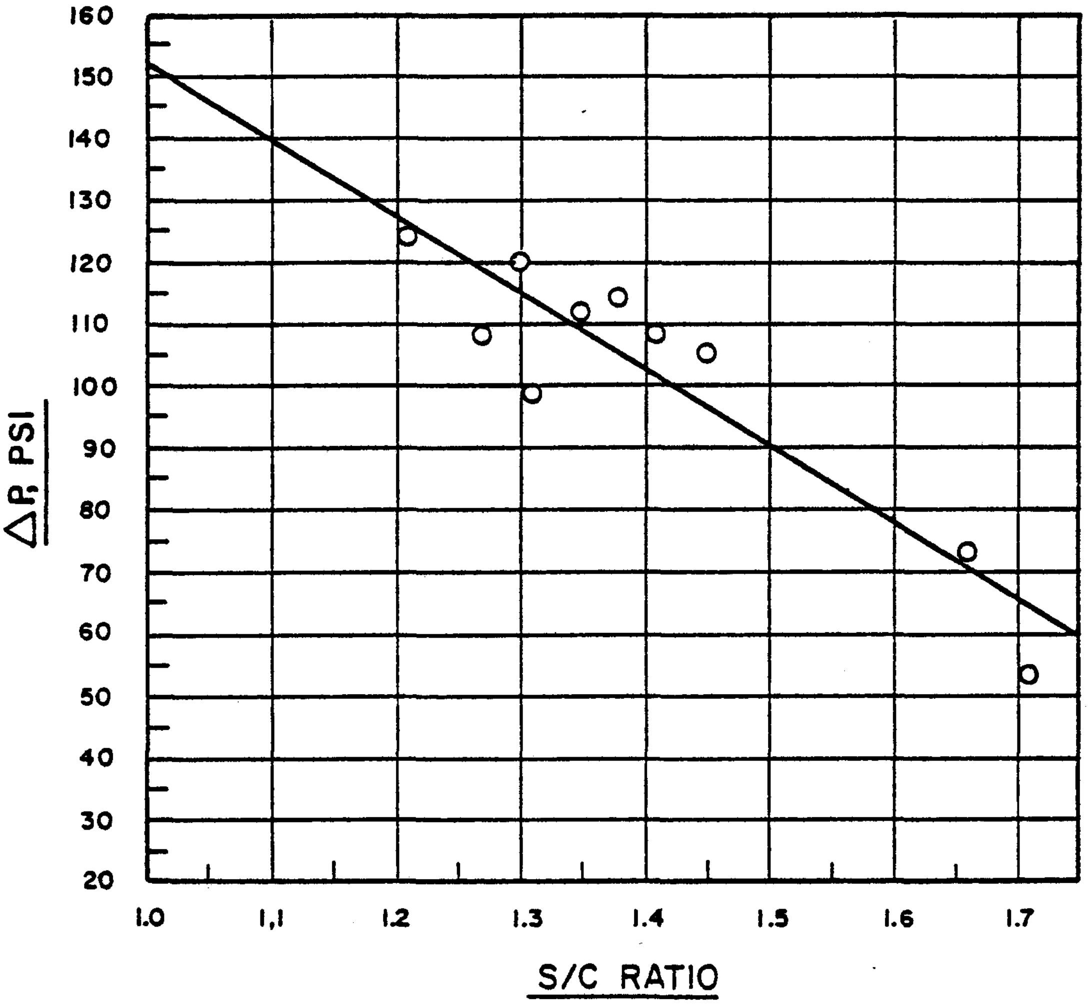


FIGURE 59

TABLE 104

SRC-I SLURRY PREHEATER SAMPLE INFORMATION

Kentucky Coal

Approximate Plant Operating Conditions

Raw Coal Feed	2600 lb/hr
Solvent Feed	4500 lb/hr
Solvent to Coal Ratio	1.8
Preheater Outlet Temperature	750°F
Preheater Pressure	1500 psig

Analysis

	Preheater Sample (5/6/76)	UFCS Sample (5/6/76)
Lt. Oil + Water	2.7	2.14
Wash Solvent	0	1.83
Process Solvent	21.1	59.35
Vacuum Bottoms	64.8	29.17
Ash	5.3	5.04
Unreacted Carbon	6.1	6.47
Melting Point	350°F	----
Pyridine Insolubles	11.39-13.36	7.51
Blackness	15.93	10.24

TABLE 105

SRC-I SLURRY PREHEATER OUTLET SAMPLE ANALYSIS

1500 psig Kentucky Coal

Feed Conditions: 1.7/1 Solvent to Coal Ratio

Sample taken at 1400 hrs., 8-24-76

PROXIMATE ANALYSIS

	Sample	Feed Coal
% Moisture	2.95	1.94
% Volatile Matter	65.85	37.15
% Ash	8.69	11.09
% Fixed Carbon	22.51	49.82

ULTIMATE ANALYSIS

	Sample	Feed Coal	Solvent
%C	80.93	70.15	88.88
%H	6.18	5.03	7.74
%N	1.23	1.58	0.93
%S	1.82	5.08	0.32
%O	2.05	6.99	2.13
%Ash	7.79	11.17	0

Distillation Analysis:		
	Light Oil	- 1.52%
	Process Solvent	- 40.53%
	Distillation Residue	- 57.95%

used in the pyridine solubility analysis seemed to be the cause of the non-reproducibility.

The appearance of the sample led to the postulation that it was heterogeneous. In fact, ash analysis results tended to verify this, with ash varying between 6.61% and 17.6% in the sample.

A mixture of one-half sample and one-half process solvent was vacuum distilled and the results are shown in Table 105. (The distillation results were adjusted to correct for the additional process solvent.) The initial feed slurry, with a 1.7 to 1 solvent to coal ratio (63% process solvent), had a higher process solvent concentration than the sample (40.5% process solvent). Again, this supports the conclusion that process solvent and coal interact to form an intermediate reaction product in the preheater. Analysis of the dissolver effluent indicates that this intermediate reacts in the dissolver, liberating process solvent and SRC.

The ultimate and proximate analysis of the sample reveal that the sample is in an intermediate stage between coal and process solvent, being more highly hydrogenated and containing a greater quantity of volatile matter than coal, but less than process solvent.

f. Coal Swelling Studies

Coal swelling was examined by submerging coarse lumps of raw coal in process solvent at a constant temperature and by observing the increase in weight versus time. The temperature range studied was 200 to 400°F. Essentially no increase in weight was observed for coal in process solvent at 200°F, but weight increases were measured for temperatures from 350°F to 400°F. This data is presented in Table 106.

Typically, a time delay is observed before a particle begins to gain weight. There is some correlation of this time delay with the heat history of the particle since those which were dehumidified at 230°F or 390°F exhibited longer delay times. The relationship of the minimum time delay to particle size is unknown.

A mathematical model has been proposed which describes the swelling behavior of these large particles and which may be applicable to smaller particles although this has not been tested. The model related total swelling time, t, in minutes, to unpenetrated particle diameter, d, in centimeters, by the following:

$$t = \frac{\rho_c D^2}{24 \rho_p b D_e} \left[1 - 3 \left(\frac{d}{D}\right)^2 + 2 \left(\frac{d}{D}\right)^3\right] + t_d$$

where: ρ_c and ρ_p are densities of coal and process solvent, respectively (gm/cc)
D is the initial particle diameter (cm)
b is the ratio of absorbed solvent to coal in a completely swollen particle = 2.86
D_e is the effective diffusivity (cm^2/sec)
t_d is the time delay, min., before swelling starts.

Complete swelling occurs when d = 0 where:

$$t = \frac{\rho_c D^2}{24 \rho_p b D_e} + t_D = t_s + t_D$$

The average diffusivity for twelve runs between 350° and 400°F is 9.65×10^{-7} cm^2/sec which for a 200 mesh (0.0074 cm) particle gives a net swelling time, t_s, of about 1 minute. Since no estimate of minimum delay time for such a particle is available, it cannot be stated which is controlling.

TABLE 106

SUMMARY OF DATA FOR KENTUCKY COAL SWELLING STUDY

	Initial Weight, w (gm)	Initial Coal Diameter, D (cm)	Temperature °F	Complete Swelling Time t_s (min) x 10^5	Time Delays t_d (min)	Effective Diffusivity, De (cm^2/sec) x 10^{-7}
Run 3	2.2205	1.4469	400	0.15547	12.86	25.6981
*Run 4	1.2508	1.1949	350	1.77141	63.47	1.5380
Run 5	1.2895	1.2071	390	2.80225	14.20	0.9922
Run 6	1.3394	1.2225	400	0.130895	25.99	21.7862
Run 7	2.4976	1.5047	380	1.10963	26.74	3.8934
Run 8	2.1358	1.4283	390	0.413023	32.80	9.0312
Run 9	1.1714	1.1691	390	0.128817	23.42	20.2458
**Run 10	1.4511	1.2256	390	0.54499	76.83	5.5200
***Run 11	1.1218	1.1524	370	0.718764	134.15	3.5254
***Run 12	1.2483	1.1942	370	0.815625	148.16	8.6211
***Run 13	1.7211	1.3291	370	0.268679	141.54	12.5456
***Run 14	1.3350	1.3350	370	1.406513	121.90	2.4179

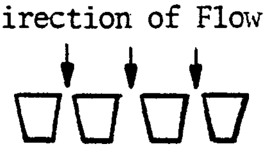
* Dehumidified for 40 min. at 390°F
** Dehumidified for 50 min. at 230°F
*** Dehumidified for 60 min. at 390°F

2. Mineral Separation

a. Rotary Filter Experimental Program

(1) Background

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



To date, no comparative tests have been made with 110 x 24 mesh Dutch weave screens, which have seen widespread use at Fort Lewis. The Johnson screen is an experimental screen that has been installed for evaluation of its operability, potential to eliminate or minimize screen blinding and to increase screen life.

Before describing the actual pilot plant program or the variables used in that program, it is necessary to develop an expression to explain the parameters which control filtration rate. In general, the filtration rate (F) was assumed to be affected by three resistance variables, expressed as:

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

Precoat resistance can be described as the flow resistance due to the filter aid itself. This term would be highly related to the viscosity of the filtrate and the pore size of the precoat.

Cake resistance results from formation of a layer of solids removed from the filter feed. In a typical filter feed slurry, the distribution of particle size shows a substantial particle number fraction in the submicron range (see section IX. F. 7). Such a fine material could form a cake with very small pore size, much smaller than that of the filter aid. The last resistance term, interface resistance, results from penetration of solid material into the precoat. The principle behind operation of the rotary precoat filter is to deposit solids and then shave off a thin layer of material each time the drum rotates. If the knife is sufficiently sharp, it is possible to shave off all the cake formed during the last revolution and a small increment of precoat. If there is no penetration of solids beyond the precoat which is removed by the knife, the only resistance to flow immediately after cutting the cake is the precoat resistance. In some instances this is not the case, since some solids have penetrated beyond the depth of the knife cut and, as a result, the pore size of the precoat layer is reduced.

It was recognized that it would not be possible to evaluate these resistance terms in the pilot plant because of operating limitations, and evaluation of the parameters was left to laboratory or bench scale experiments. The objectives in the Fort Lewis study were to observe overall effects of operating variables on the resistance parameters and to investigate optimization alternatives.

In all, twelve runs were completed during the test period. These runs were designed to investigate the effects of a single variable on filtration at a time. A factorial experimental design was not chosen due to the time constraints on the program limiting the choice of variables and the range within which those variables could be tested. Attempts were made to begin filtration optimization toward the end of the program, but plant operating problems prevented this .

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

- Precoat Application: Two precoat application procedures were followed. The standard P&M procedure was used as a base condition and the second procedure was used for comparison. It was expected that the two procedures would yield precoats which were packed to different degrees. At the start of the program, it was not known if a more tightly packed cake would hinder rates due to higher flow resistance than a less tightly packed cake, or if it would aid filtration by preventing solids penetration into the cake.

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

- Knife Advance Rates: Previous experience at Fort Lewis and elsewhere had shown that increases in knife advance rates in the range of 1-5 mil/min resulted in increased filtration rates. The units of mils (10^{-3} inches) per minute were chosen in this study to specify operating conditions. Most runs were at constant drum speed, which would result in comparable knife penetrations per drum revolution. When drum speed was changed, knife advance rates were expressed in units of mils per revolution, which provides information on the unit rate of knife penetration per revolution. Knife advance is a very important variable because it affects both the cake and interface resistance terms.

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

- Drum Speed: Drum speeds in the range of 1-6 RPM were available on Filter B. The higher drum speeds result in shorter drum submergence times on each cycle at a constant vat level. By minimizing submergence time, two effects are realized:
    - The time available for solids penetration into the precoat is minimized.
    - The thickness of cake built up over the precoat is decreased.
- Drum Submergence: Another variable which can be used is drum submergence, which can be varied between 9% diameter submerged to about 40% submerged. The upper limit on this range is due to the location of the top of the cake leg. At a drum submergence of more than about 40% diameter, the slurry in the filter overflows into the cake leg. At submergences much in excess of 30% diameter, the drum bearings are submerged in filter feed. Due to continual lubrication problems in the filter, a decision was made to conduct these tests at drum submergences of 30% diameter or less.
- Pressure Drop: The driving force which causes the filtrate to flow through the cake and precoat is pressure differential. Increased pressure differential in the filter can promote several effects, some of which occur simultaneously. First, higher pressure differentials provide more driving force to cause the filtrate to flow through the cake. Second, higher pressure differential may cause additional cake compacting, which would result in a precoat and cake with smaller pore sizes and, hence, lower relative flow rates. Third, due to the increased driving force causing higher filtrate rates, solids penetration into the precoat may increase, resulting in a higher level of interface resistance.

Alternatively, if the precoat is compacted, then smaller pore sizes might limit solids penetration.

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

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

The solids removal rate was calculated by computing the rate of filtered coal solution and using the average pyridine insolubles content of the filter feed for the run to estimate the quantity of feed solids removed. There were some minor variations in feed composition between runs. The volumetric rate of precoat removal was calculated by measuring the total distance of knife travel during an hour, and computing the corresponding volume which would have been shaved under these conditions. The rate of solids removal per unit volume of precoat removed was then calculated.

The last parameter used for comparison of filtration operating conditions was gas rate through the filter in units of pounds per hour. The gas rate was found to be a good measure of cake permeability and probably can be used as an estimate of relative flow rate.

(2) Experimental

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

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

The base conditions and a brief description of the variables investigated in each run are listed in Table 107. The base conditions were chosen to represent what was felt to be midrange operating conditions when the program started.

As discussed above, each run began with a fresh application of precoat. A simplified flow sheet

TABLE 107

FILTRATION TEST RUNS
FOR
KENTUCKY COAL UNFILTERED COAL SOLUTION

BASE CONDITIONS

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

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

REACTION CONDITIONS

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

representing the precoat loop is shown in Figure 60. Precoat application was preceded by introduction of a basecoat slurry (containing 10 lbs of a diatomite-asbestos fiber mixture) into the filter. The precoat filter aid was then charged into the filter through a piston-type positive displacement pump. The filter aid slurry was diluted to a concentration of approximately 5 wt% solids in the filter by addition of process solvent recirculation from the precoat slurry pressure vessel. Typically, this process would result in precoat application over a span of 3-4 hours. A more detailed description of precoat procedures is provided in paragraph III.D.2. of this report.

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

Figure 61 shows the schematic flow scheme which was followed during the tests. The filter was fed directly from the filter feed flash vessel (FFFV). The feed to the flash vessel consisted of undiluted unfiltered coal solution from the reaction area and overflow from the filter. (Overflow from the filter was provided for filter temperature control and to minimize settling of pyridine insolubles in the filter). It was felt that the best way of controlling the feed composition during these tests was to prevent contamination of the unfiltered coal solution and to feed only fresh material. Attempts were also made to limit feed composition variations by holding reaction conditions constant.

Figure 61 shows that wash solvent is used in the filter as cake wash, knife wash and auger wash. Of these three streams, the cake wash is the only one which should influence filtration rate. In all of these tests, the assumption was made that all of the cake wash passes through the precoat and is included in the filtration rate. Filtration rate was measured as the liquid flow out of the filtrate receiver. Any liquid carried out of

PRECOAT CYCLE FLOW SCHEME

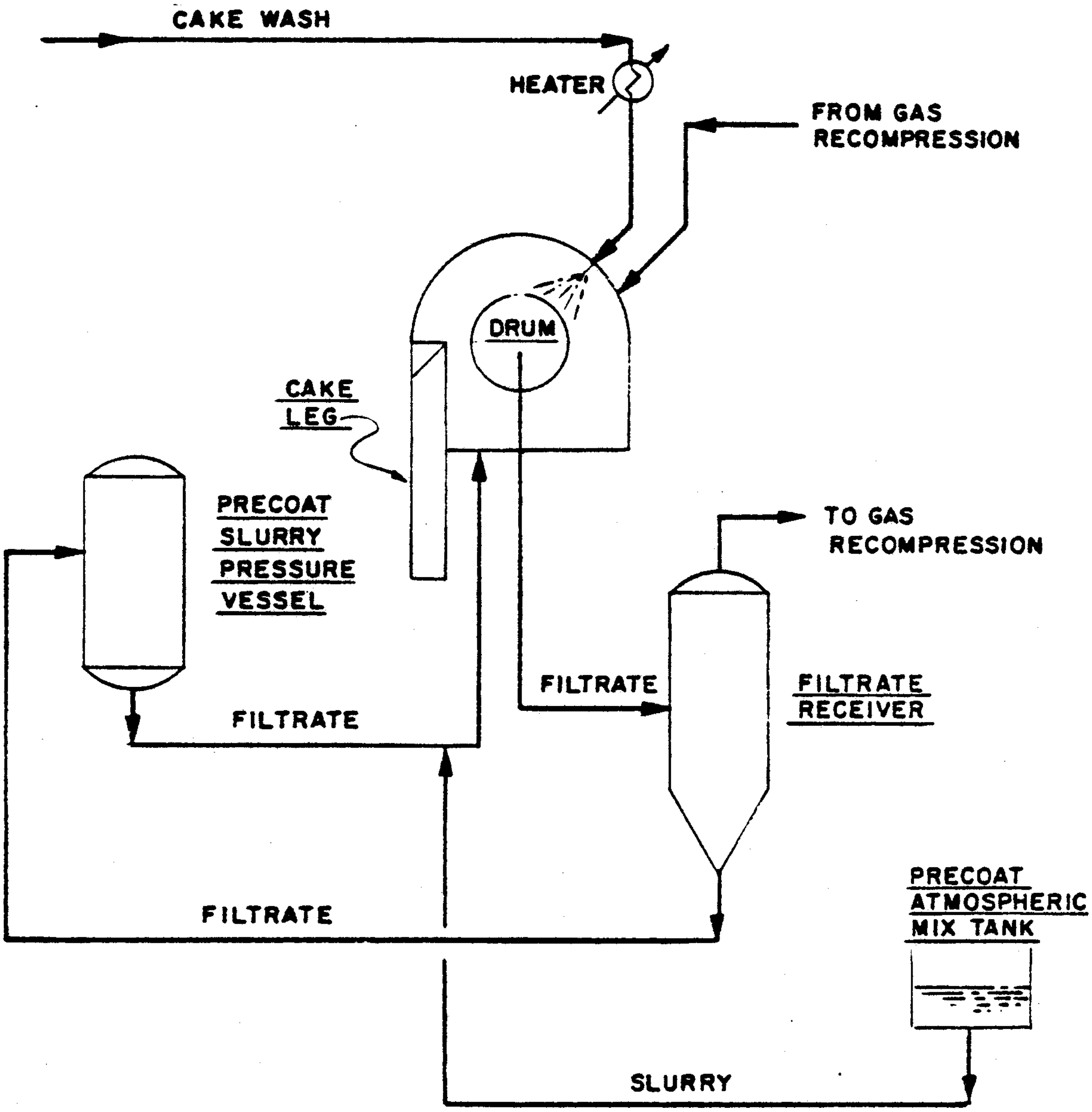


FIGURE 60

FILTRATION CYCLE FLOW SCHEME

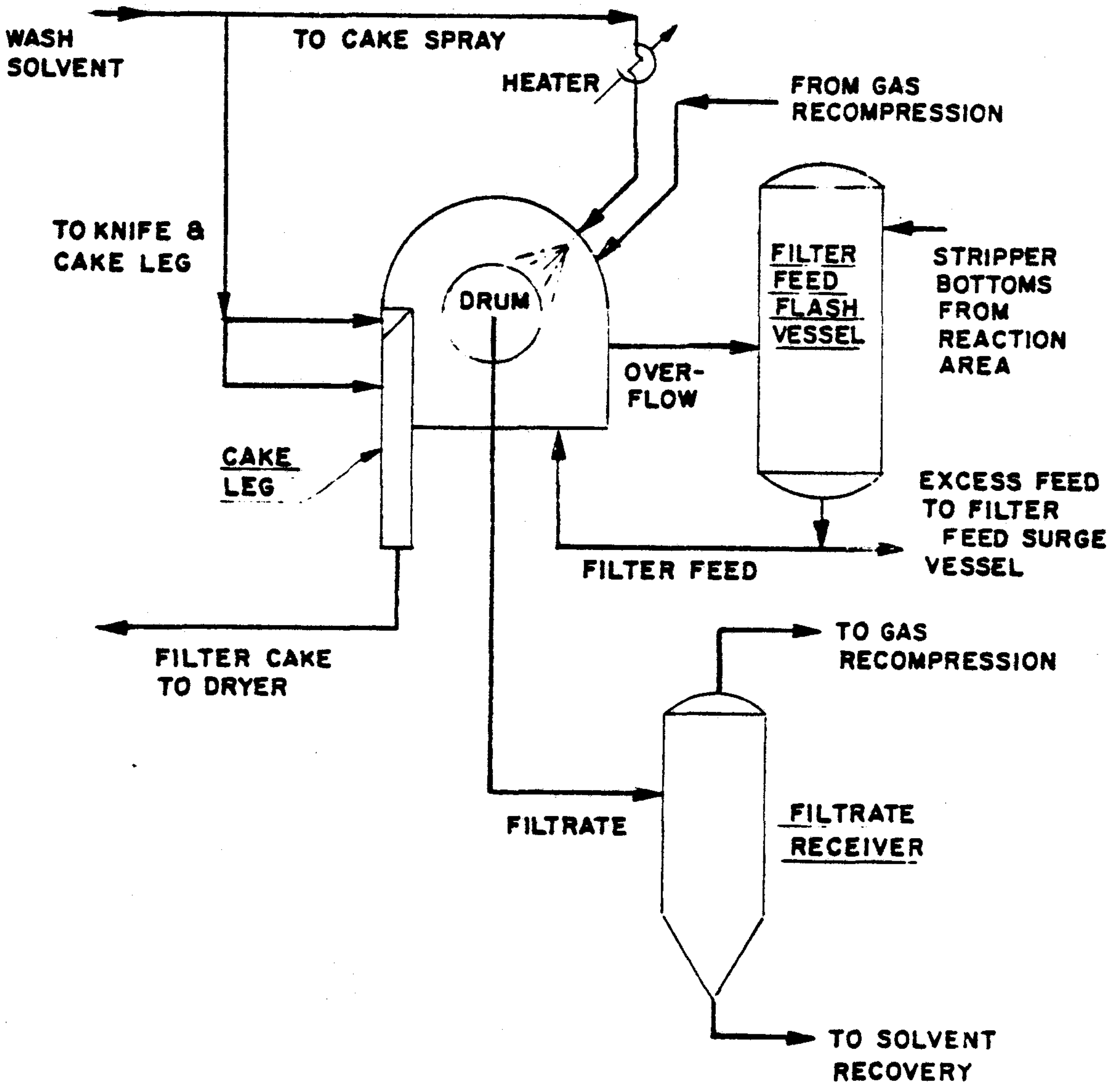


FIGURE 61

the filtrate receiver in the gas stream and condensed upstream of the compressors was reinjected into the gas stream recirculated into the filter.

Samples of the filtrate and filter feed were routinely analyzed to determine viscosity and the concentration of vacuum bottoms and solids in the sample.

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

(3) Results

Each run was split into several run periods during which operating conditions were held relatively constant. Tables 109 to 120 list the operating conditions and filtration data obtained during each run period. Figures 74 through 85 show average filtration rates, knife advance rate, drum speeds, drum submergence, cake wash rates, and gas flow rates for each hour or each run.

Unusual procedures, run objectives, and comments on each run are listed in the appropriate tables. Comparison of the data has revealed some interesting relationships, which will be discussed at length at this time.

- Precoat: The effects of precoat on filtration rate were never quantitatively determined during these runs. A comparison of the filtration rates which were obtained while operating at base conditions during each run has been made. The data, which are shown in Figure 62, indicate that for the last 5 runs in which a Speedex precoat was used, the filtration rate at the base conditions in the early stages of the run were comparable, and averaged 146 lb/hr ft^2 (including cake wash). The first five runs were not included in the analysis

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

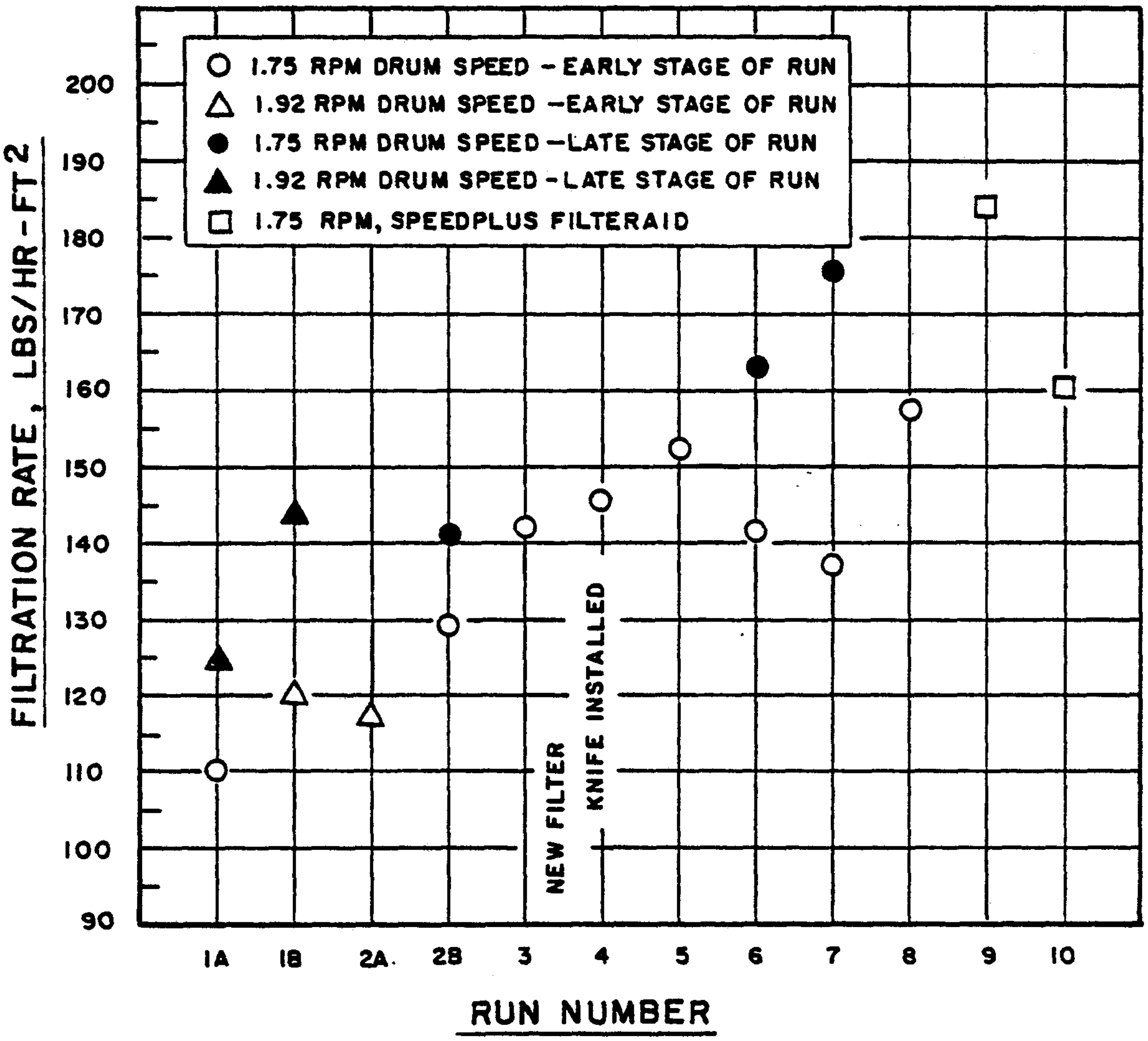


FIGURE 62

because prior to Run 4, a new knife was installed in the filter and filtration rates in subsequent runs seemed higher than in previous runs.

Figure 62 also shows data which were obtained at base conditions, but in the latter stages of some of the runs. These data show that filtration rates toward the end of a run tend to be higher than at the early stages of a run. The average filtration rates for base conditions in the runs in which Speedplus was used as a precoat material are also shown in Figure 62, and the rates appear to be higher than for Speedex precoat runs. However, the Speedplus data were obtained at later stages in the run than was normal for the Speedex runs. This fact makes comparison of the data for the different precoat materials difficult and no definitive conclusion is possible.

Precoat application can also be evaluated based on the degree to which the cake is packed. This "packing" effect can be compared between various runs by comparing the gas flow rate through the filter at the base conditions. Gas rates for all the runs at base conditions varied from 300 to 500 lbs/hr, indicating that there was some variation in precoat permeablility due to the precoat application. This difference between runs probably can be attributed to subtle differences in precoat application procedures.

Observation of the gas rate during the runs indicates that as the run progresses and the precoat thickness decreases, the gas rate increases, especially during the later stages of the filtration cycle. The increases in gas rate are probably due to decreased precoat resistance and may be a function of the residence time of the precoat in the vapor region of the filter. When the precoat thickness decreases sufficiently, there may be enough time to "blow the precoat dry" before the precoat reenters the slurry. By removing the liquid imbedded in the precoat, the resistance of the precoat to gas flow is decreased. Figure 63 shows gas flow rates during Run 1B versus precoat thickness. (All operating conditions were held constant during Run 1B) The data show an

GAS FLOW RATE vs KNIFE POSITION FOR RUN IB

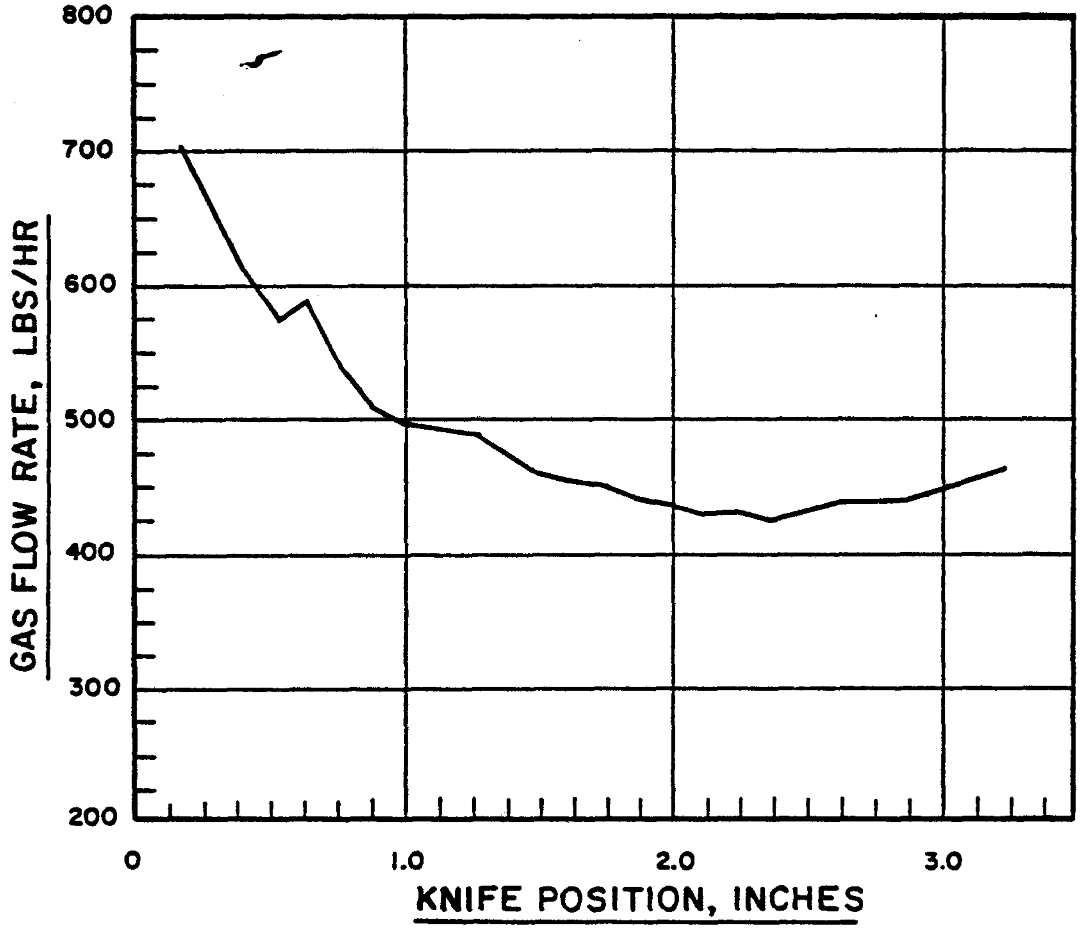


FIGURE 63

increase in gas flow rates in the latter stages of the run. This corresponds with a slight increase in filtration rate during Run 1B (see Figure 75). From this information, it seems probably that precoat thickness has an effect on filter performance. To simplify the results of later runs,. this effect was assumed to be negligible.

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

  Figure 64 is a plot of filtration rate as a function of knife advance rate for Runs 3 through 8 in which Speedex filter aid was used as precoat material. The data shown in Figure 64 were all obtained with all of the other operating variables in the precoat loop at the base condition levels. The curve drawn through the data indicates increasing filtration rates with increasing knife advance rates. This increase would be expected to continue until the knife advance rate is high enough to permit deep enough knife penetration to remove all of the filter cake (pyridine insolubles in the coal) and cake-precoat interface during each drum revolution. This principle of "diminishing returns" in filtration rate with increasing knife advance is represented in Figure 64 by the approach to an asymptotic value of filtration rate at high knife advance rates. Attempts to operate the filter at knife advance rates in excess of 4 mil/min were not made and, as a result, the point at which knife advance rate increases do not result in increased filtration rates has not been defined.

  To determine the efficiency of precoat usage in these tests, the rate of removal of the solids (pyridine insoluble matter) in the filter feed was estimated from the filtration rate and average solids concentration during each run. An hourly precoat usage, in units of cubic feet, was calculated from knife position data recorded

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

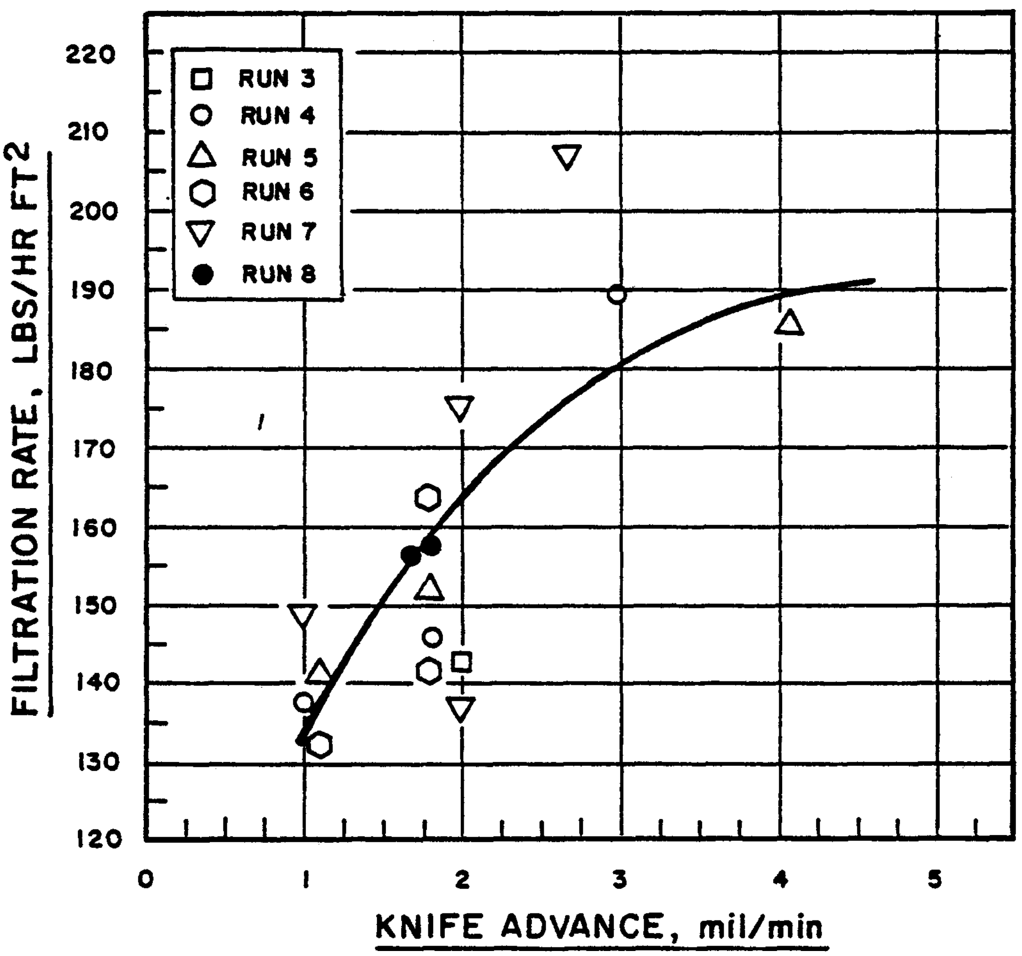


FIGURE 64

each hour. The ratio of solids removal to precoat usage was then computed for each run. This ratio, with units of lbs solids/cubic ft. precoat was defined as a measure of filter efficiency and was used in comparison of the data obtained for the various operating periods. A comparison was made of these filter efficiency data for the runs in which knife advance was varied. The results showed that the ratio of solids removed from the filter feed per unit of precoat volume decreased as knife advance increased. This implies that more precoat is consumed in producing the same quantity of filtrate at high knife advance rates than at low knife advance rates. However, at the low knife advance rates, the filtration rate is lower, requiring more filtration time to produce the comparable quantity of filtrate.

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

- Drum Submergence: Figures 65 and 66 show the effect of drum submergence on filtration rate. Figure 65 shows the filtration rate as a function of submergence. The data indicate that increased submergence resulted in higher rates. The fact that higher rates were achieved at higher submergence proves that the filter cake is not completely blinding the precoat and preventing additional filtration. Actually, the data show that precisely the opposite is true. By increasing the drum residence time in the slurry, additional filtrate will be produced. However, a trend toward a gradual approach to some maximum filtration rate seems apparent in Figure 65. Each additional unit of area results in a smaller gain in filtration rate at high submergence. As drum submergence is increased, the cake thickness will also increase due to longer drum residence time in the slurry. As the cake thickness increases, the cake resistance should also increase, resulting in a gradual decrease in instantaneous rate through the precoat. This phenomenon apparently caused the leveling off of filtration rate as seen in Figure 65.

FILTRATION RATE vs. % AREA SUBMERGED

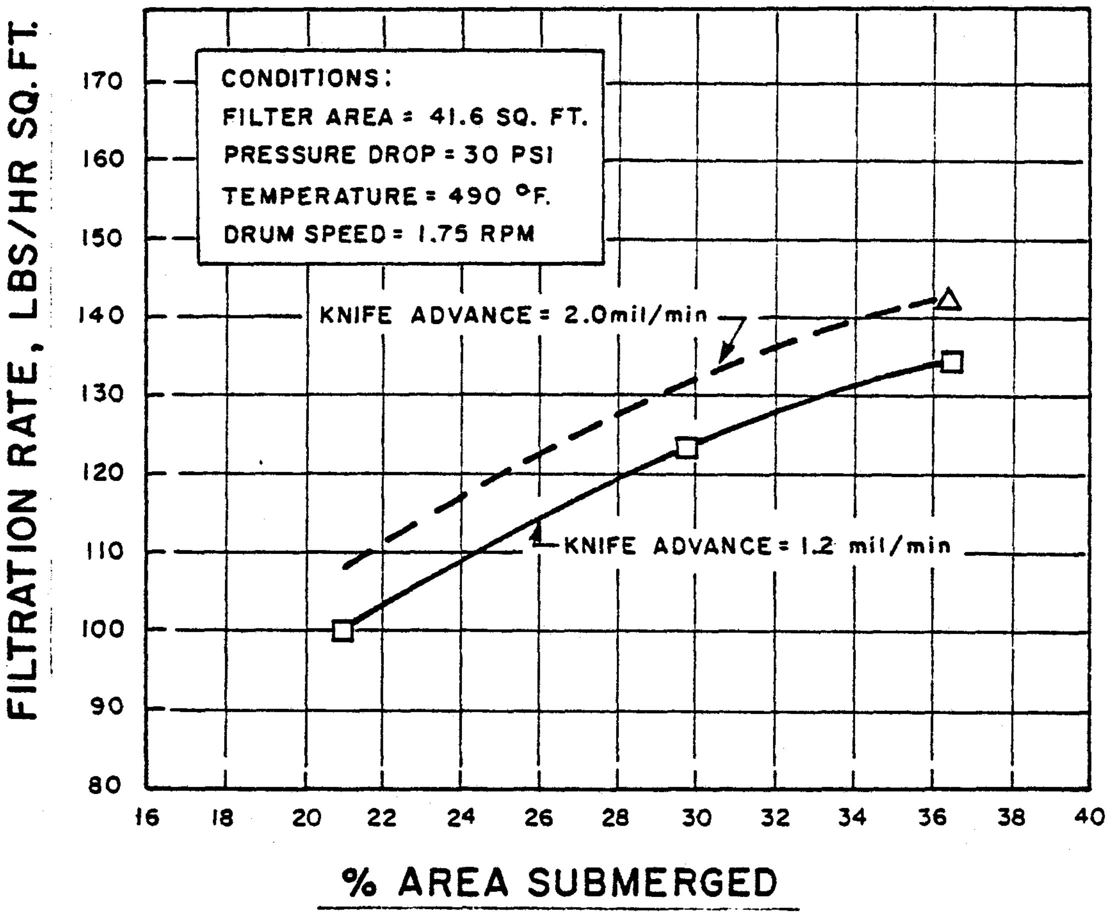


FIGURE 65

FILTRATION RATE/UNIT AREA SUBMERGED VS PERCENT AREA SUBMERGED FOR RUN 3

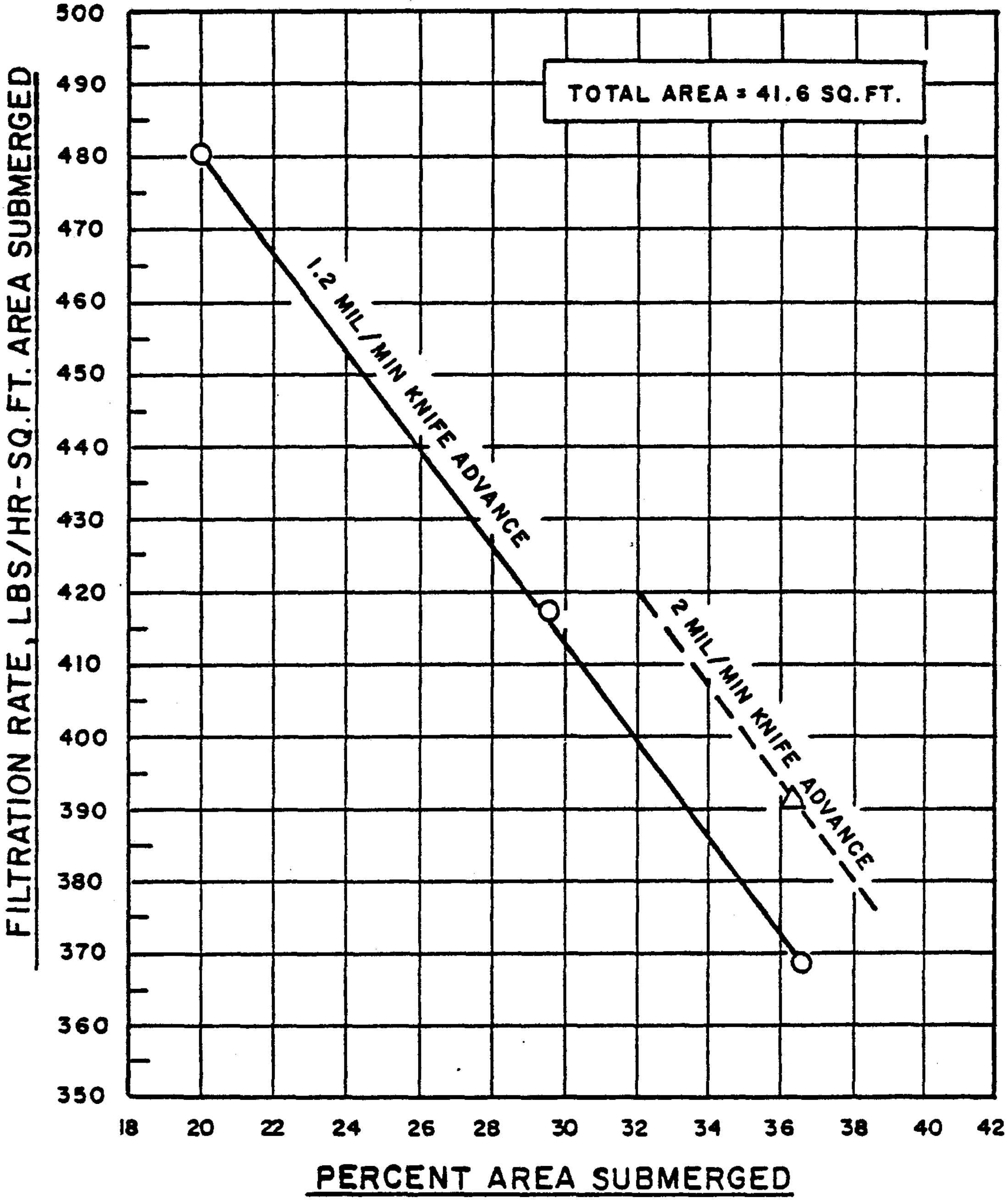


FIGURE 66

The filtration rate per unit of area submergence is another variable which can be used to describe the filter performance at various levels of submergence. Figure 66 shows that the rate per unit area submerged decreased with increased submergence (the same data set plotted in Figure 65 was used to generate Figure 66). This decrease in utility of submerged area with increased submergence can be explained by the same phenomenon described above in the discussion of Figure 65, namely, that the marginal increase in filtration rate decreases as knife advance increases.

Both Figures 65 and 66 show that increased knife advance rates result in higher filtration rates at higher submergence than low knife advance rates. Enough data at high knife advance were not generated during the submergence study to provide parametric curves of the effect of knife advance on filtration at various levels of submergence. Therefore, the filtration rates which might be expected at various levels of submergence and at 2 mil/min knife advance rate have been extrapolated and plotted as broken lines in Figure 65 and 66.

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

  The results of Run 4 are illustrated in Figure 67, in which filtration rate has been shown as a function of drum speed, with knife advance rate as a parameter. Figure 67 suggests two conclusions. First, at the knife advance rates studied, the highest drum speed resulted in the highest filtration rate. Second, at the drum speeds studied, the highest knife advance rate always resulted in the highest filtration rate. The obvious conclusion from this study is that to optimize filtration rates, maximum knife advance rates and drum speeds should be utilized.

FILTRATION RATE vs. DRUM SPEED

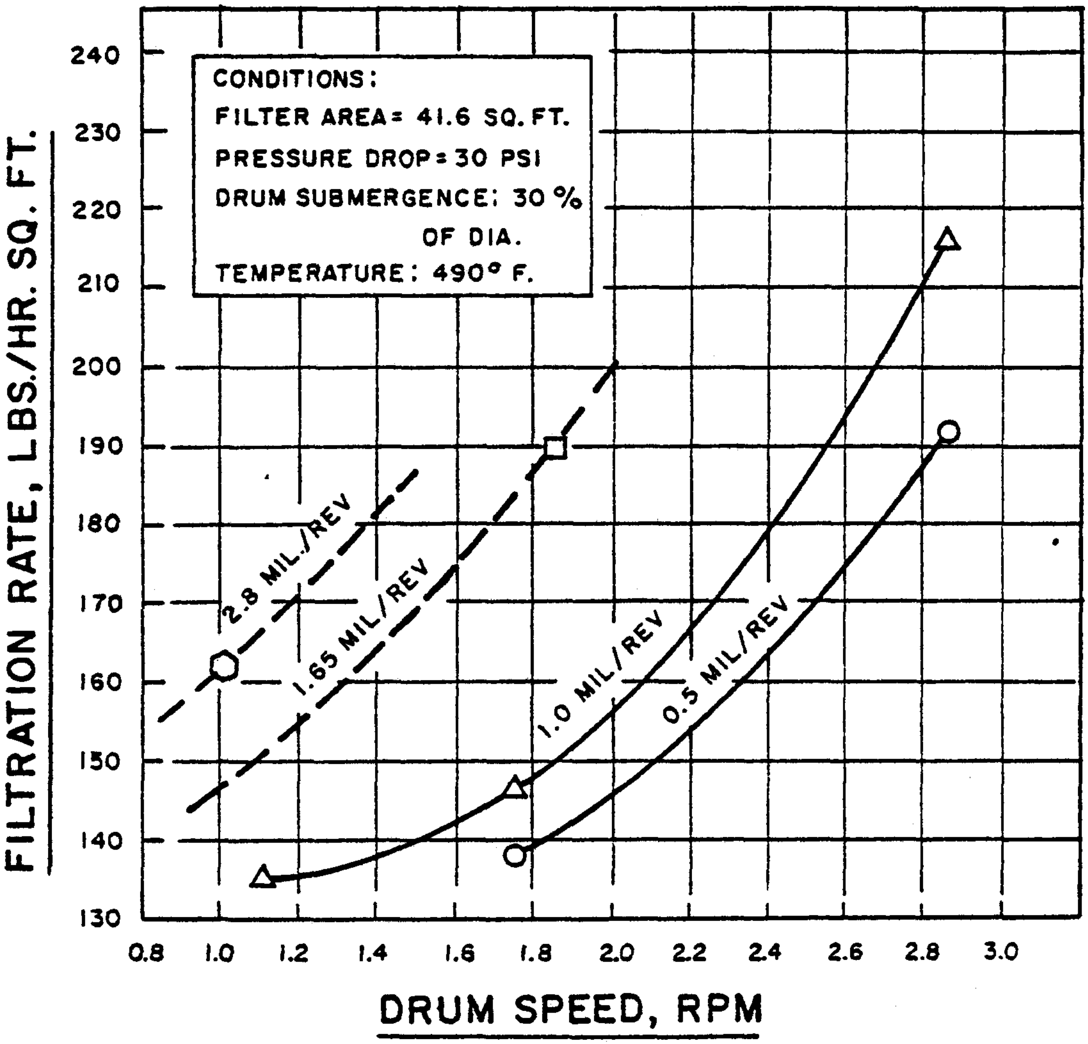


FIGURE 67

This same conclusion on the effects of knife advance rate and drum speed has been reached elsewhere using coal solutions in bench scale tests on a simulated rotary pressure precoat filter.(2)

Obviously, high knife advance rates will consume substantial amounts of precoat and, therefore, the efficiency of solids removal by the precoat must be evaluated. Solids removal per cubic foot of precoat usage were calculated for the data discussed above and are shown in Figure 68. The data suggest that solids removal rates were not a function of drum speed, but were a strong function of knife advance rate. As the knife advance rate increased, the solids removal efficiency decreased.

The fact that solids removal efficiency was independent of drum speed would suggest that it would be most advantageous to obtain the high filtration rates associated with high drum speeds. At the same time, the data in Figure 68 indicate that high knife advance rates result in a rapid drop in efficiency when rates increase above 1 mil/rev. Therefore, it might not be advantageous to maximize knife advance rate. It would be most useful to optimize the knife advance rate to produce a high filtration rate at a high solids removal efficiency. The data in Figure 68 imply that the optimum knife advance rate should be in the 0.5-1.0 mil/rev range, the range where the solids removal efficiency drops rapidly as knife advance is increased. Further work in this area is necessary to explore possible optimum conditions.

- Cake Wash Rate: A brief test was made to determine the effects of cake wash rate on filtration rates. The results are shown in Figure 69. In the figure, line A represents a least squares linear regression fit of the data. Line B has been drawn to illustrate the position and slope of a line which would indicate a gain in filtration rate equal to the gain in cake wash rate (recall

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(2) Smith, GRS, and Martin, Dr. P.C., "Filtration Process and Equipment Studies for Coal Liquefaction Processes," March, 1977 Progress Report to ERDA, FE-2007-31.

SOLIDS REMOVAL EFFICIENCY
VS
KNIFE ADVANCE FOR RUN 4

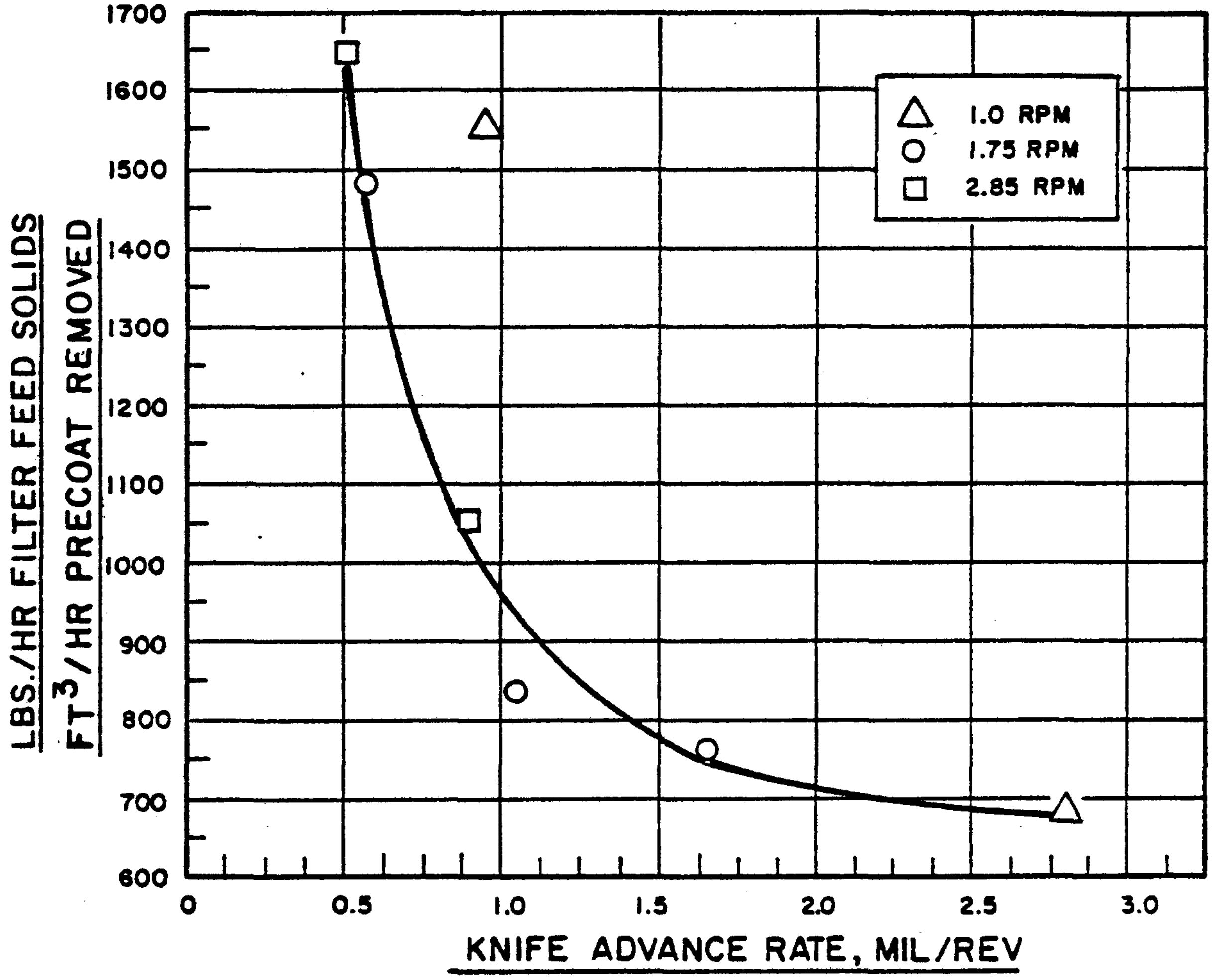


FIGURE 68

EFFECT OF WASH SOLVENT RATE ON FILTRATION RATE DURING RUN 7

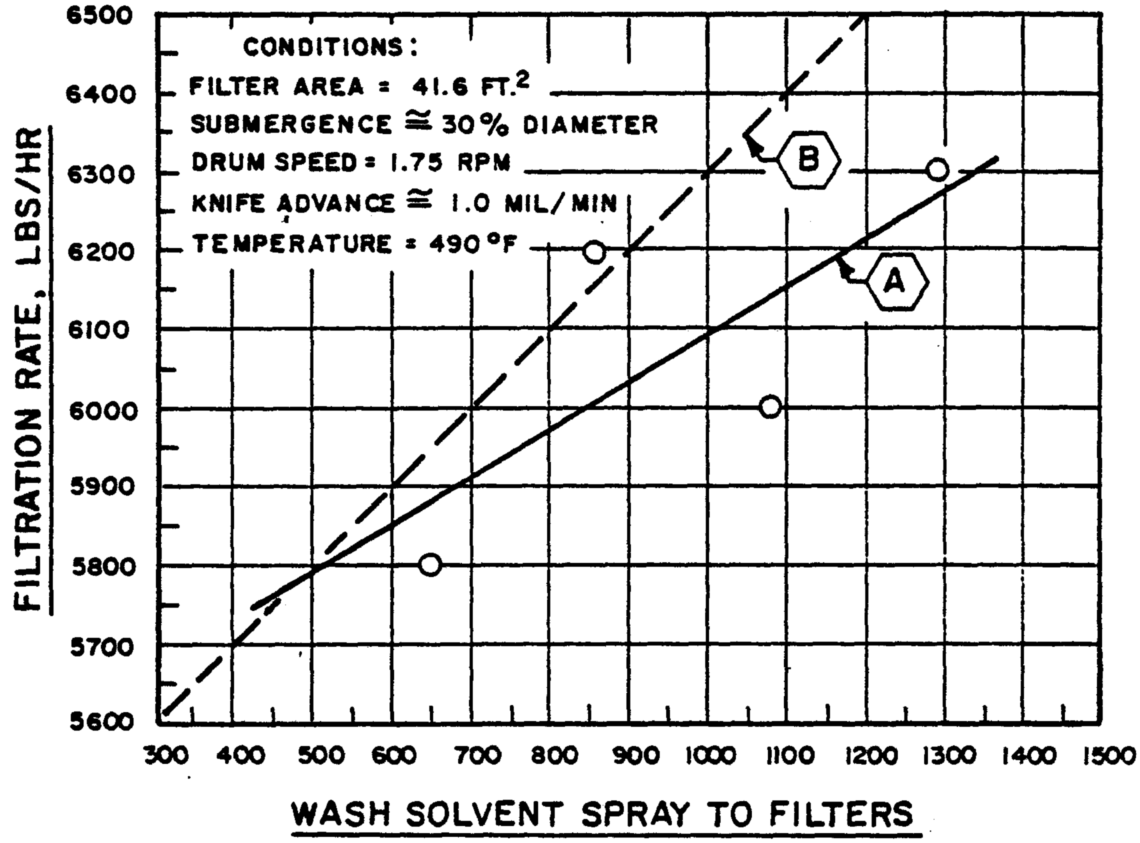


FIGURE 69

that the filtrate has been assumed to include the cake wash to the filter). The slope of line A is less than the slope of line B, suggesting that increasing the amount of cake wash in the range studied does not increase the true filtration rate. In fact, if the cake wash is subtracted from the filtrate, the actual filtration rate decreases as the cake wash increases. The results of this cake wash study indicate that more work needs to be done to determine the minimum amount of cake wash needed to adequately remove imbibed process solvent and SRC from the cake. If the quantity of cake wash can be reduced significantly without deleterious effects, as these data seem to suggest, the amount of solvent that has to be distilled in the fractionating towers can be reduced.

- Pressure Differential: In Run 5, the effects of pressure differential (ΔP) and knife advance rate on the filtration rate were investigated. The results are illustrated in Figure 70. Indications are that increasing the differential pressure from 30 to 40 psi acts to increase the filtration rate at any knife advance rate. In addition, the data in Run 5 indicated that knife advance rates resulted in a greater increase in filtration rate at 40 psi than at 30 psi.

  The fact that high knife advance rates seem to result in higher filtration rates at 40 psi than at 30 psi has been attributed to more efficient solids removal at the higher pressure, possibly due to the formation of a cleaner cake-precoat interface than at the lower pressure.

  The dramatic effect of knife advance rate at 30 and 40 psi pressure differential is shown in Figure 71. The increase in slope of the constant knife advance curves as the knife advance increases is evidence of the strong supplemental effect of knife advance on the filtration rate.

  The solids removal efficiency parameter was calculated for the data shown in Figures 70 and 71, and the results have been plotted in Figure 72. As with all other solids removal efficiency data at different knife advance rates, the implication from Figure 72 is that solids removal efficiency

FILTRATION RATE vs KNIFE ADVANCE RATE

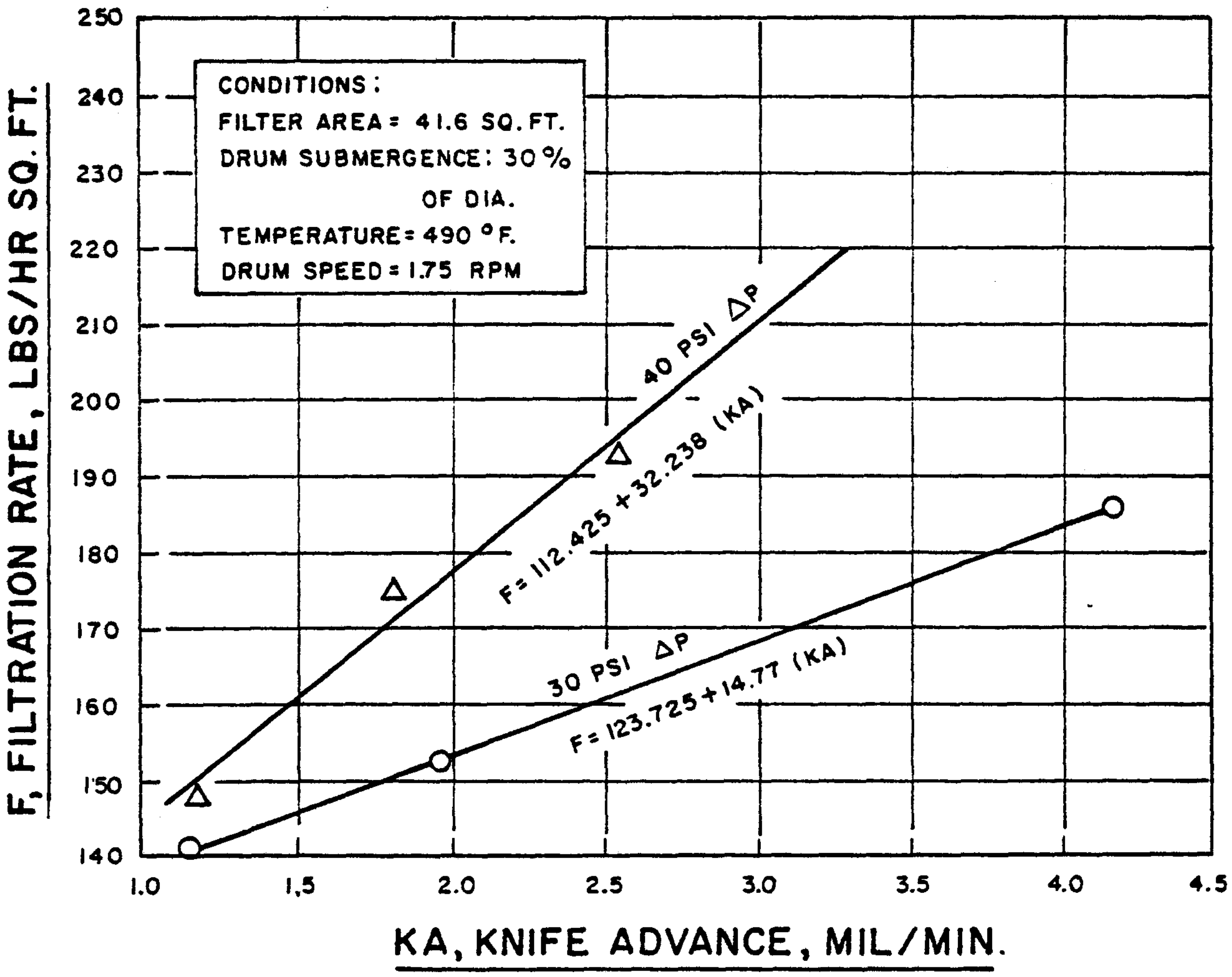


FIGURE 70

FILTRATION RATE vs. PRESSURE DIFFERENTIAL

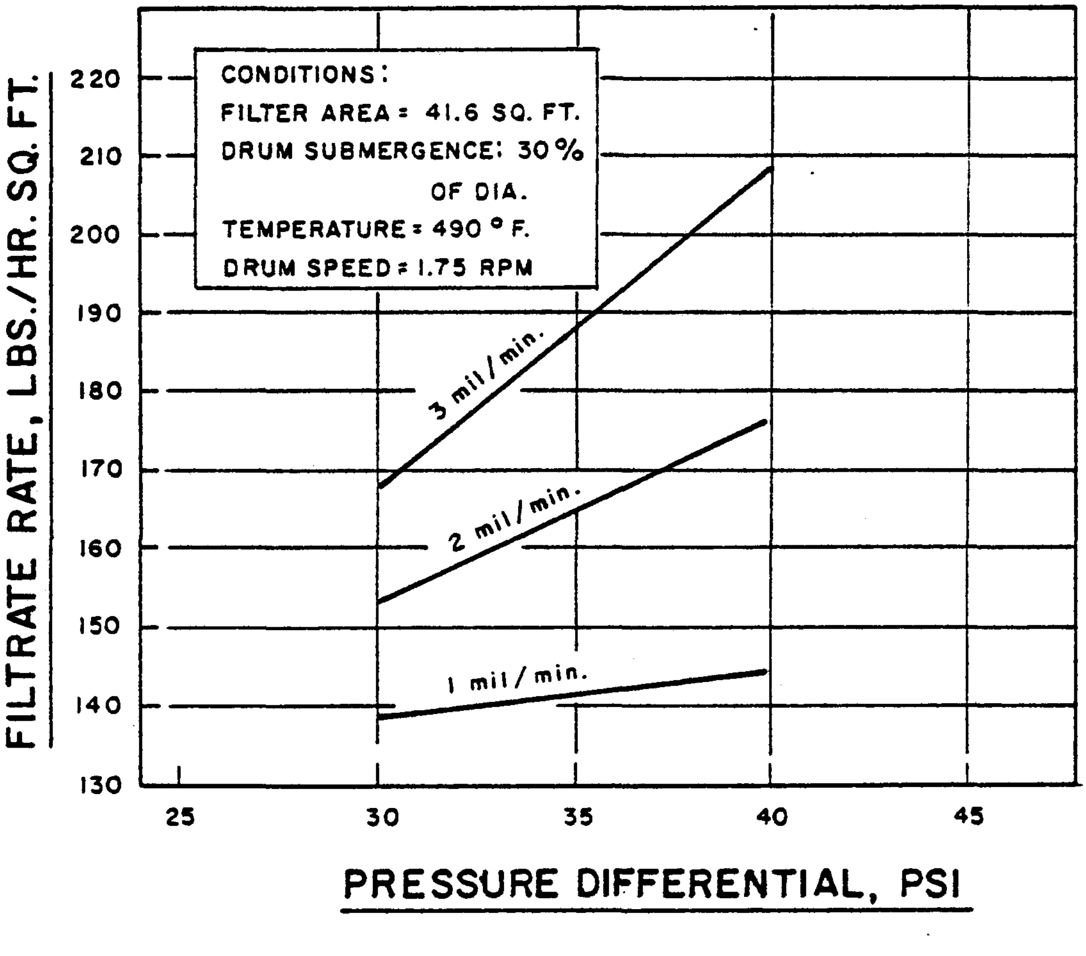


FIGURE 71

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

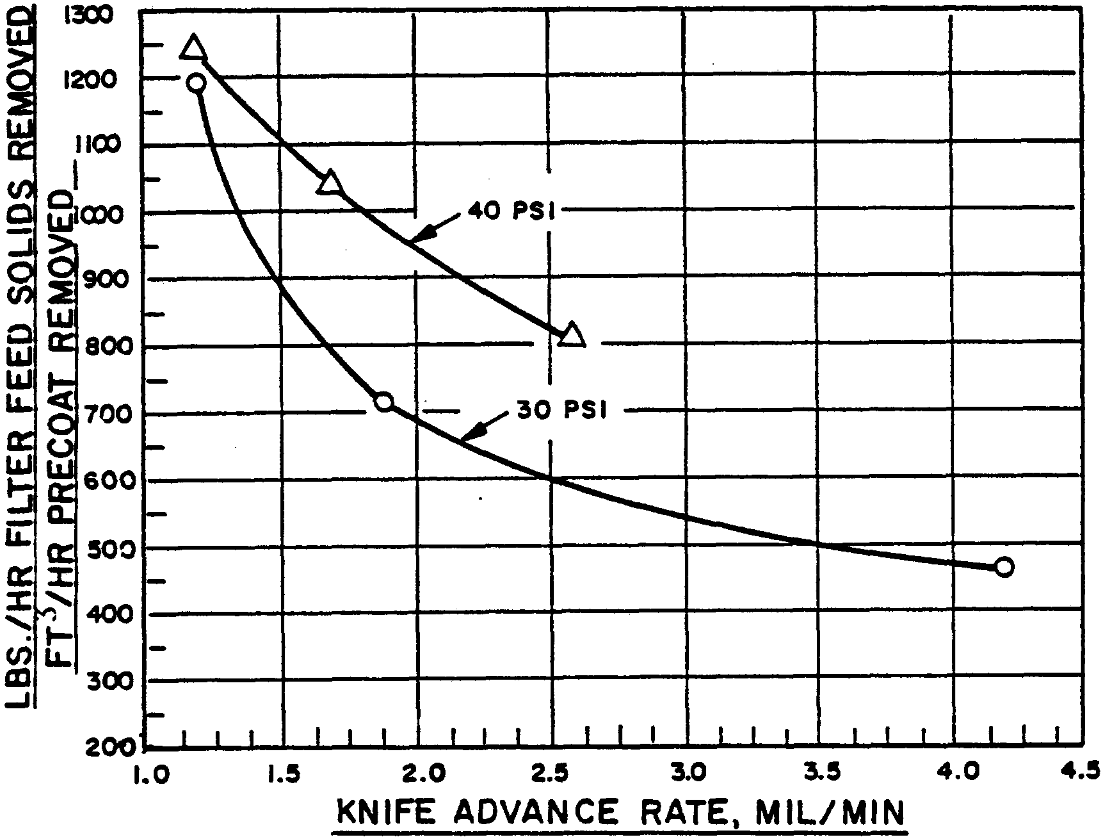


FIGURE 72

decreases as knife advance increases, and the decrease is rapid between 0.5 and 1.0 mil/rev. (The drum speed in Run 5 was 1.74 RPM) Figure 72 shows that there is a positive effect of pressure differential on solids removal efficiency. At the 40 psi differential, the efficiency of solids removal was much higher than at the 30 psi differential.

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

(4) Optimization

The data from all of the periods of all twelve runs were analyzed and estimated filter feed rates and solids removal efficiencies calculated for each period. These data were then classified into two groups: periods in which Speedex precoat was used and periods in which Speedplus precoat was used. These two groups were each broken into four subgroups according to knife advance rates, with the subgroups representing average knife advance rates of 0.5-1.5, 1.5-2.5, 2.5-3.5, and 3.5-5.0 mil/min. The data were then plotted to determine if any unusual correlations existed. Some interesting trends in the data were noted and are shown by least squares regression curves in Figure 73.

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

Based on the results of this program, conditions for an optimum run have been formulated. These conditions, which are listed in Table 108, assume that maximum filtration rate is important, but not the only objective, and that increasing the solids removal efficiency is an important objective.

FILTRATION vs. SOLIDS REMOVED EFFICIENCY

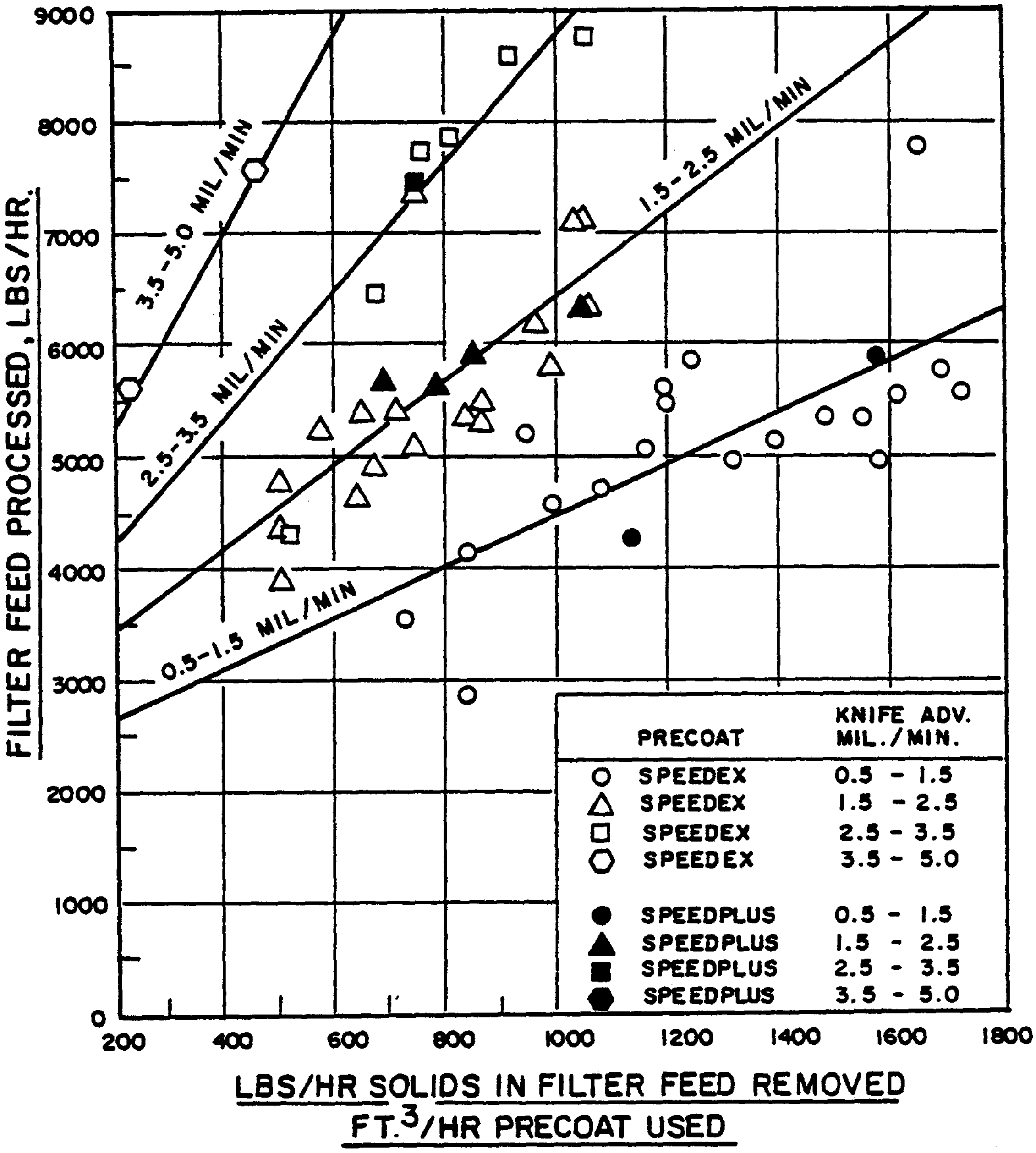


FIGURE 73

TABLE 108

PROPOSED OPTIMUM FILTRATION RUN

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

* Includes recycled overflow. Refer to Figure 61.

PLOT OF FILTER OPERATING DATA FOR RUN 1A

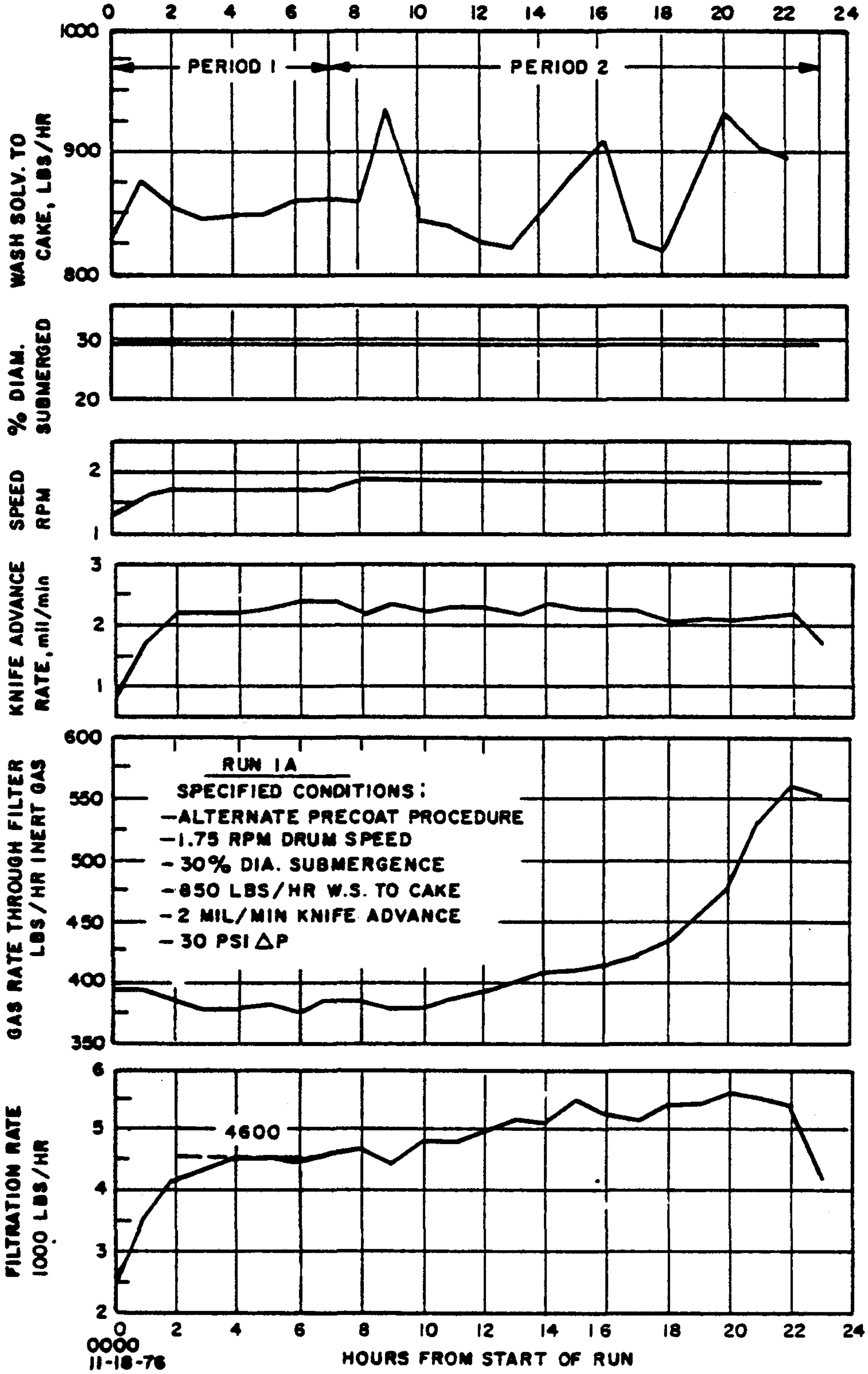


FIGURE 74

PLOT OF FILTER OPERATING DATA FOR RUN IB

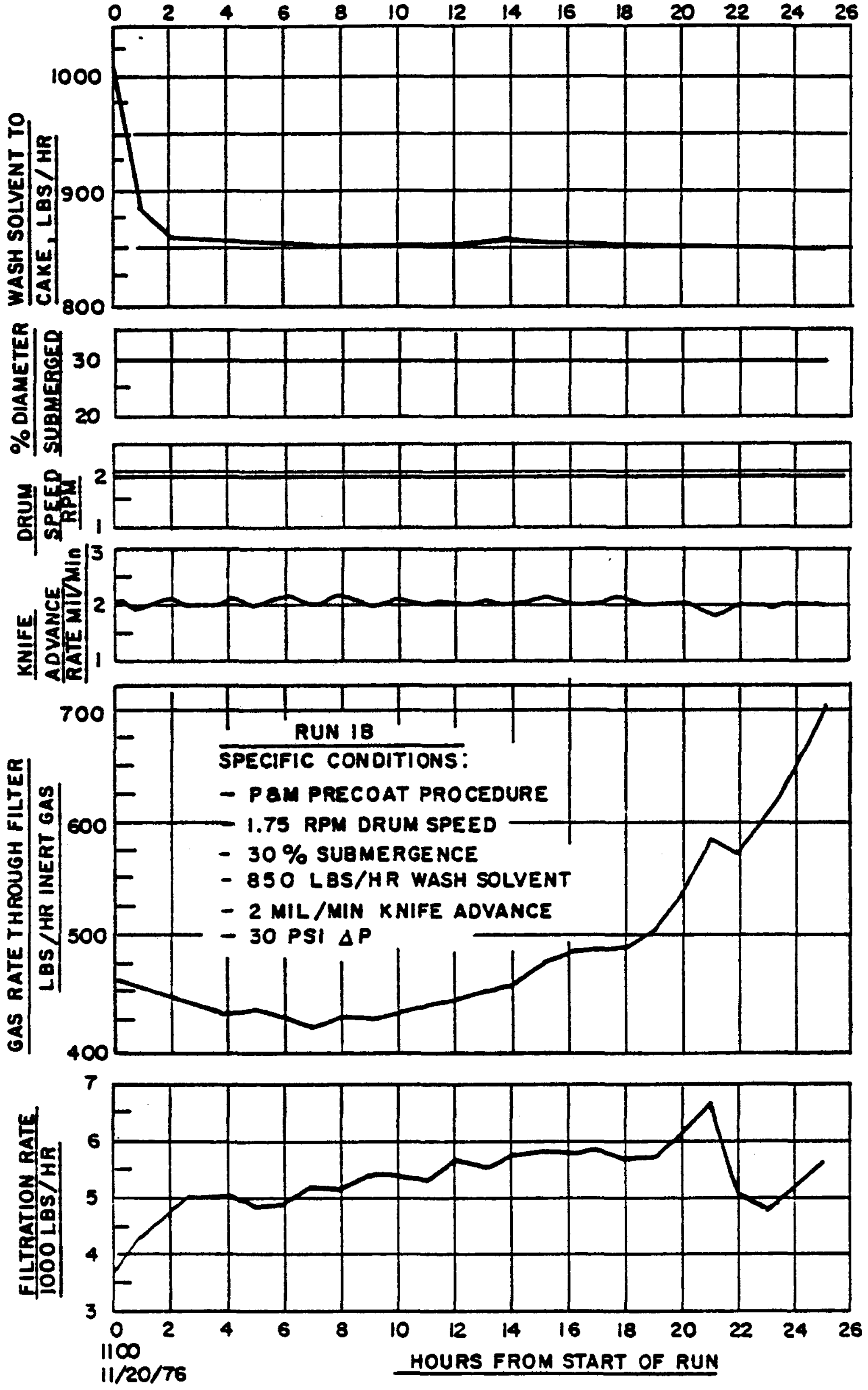


FIGURE 75

PLOT OF FILTER OPERATING DATA FOR RUN 2A

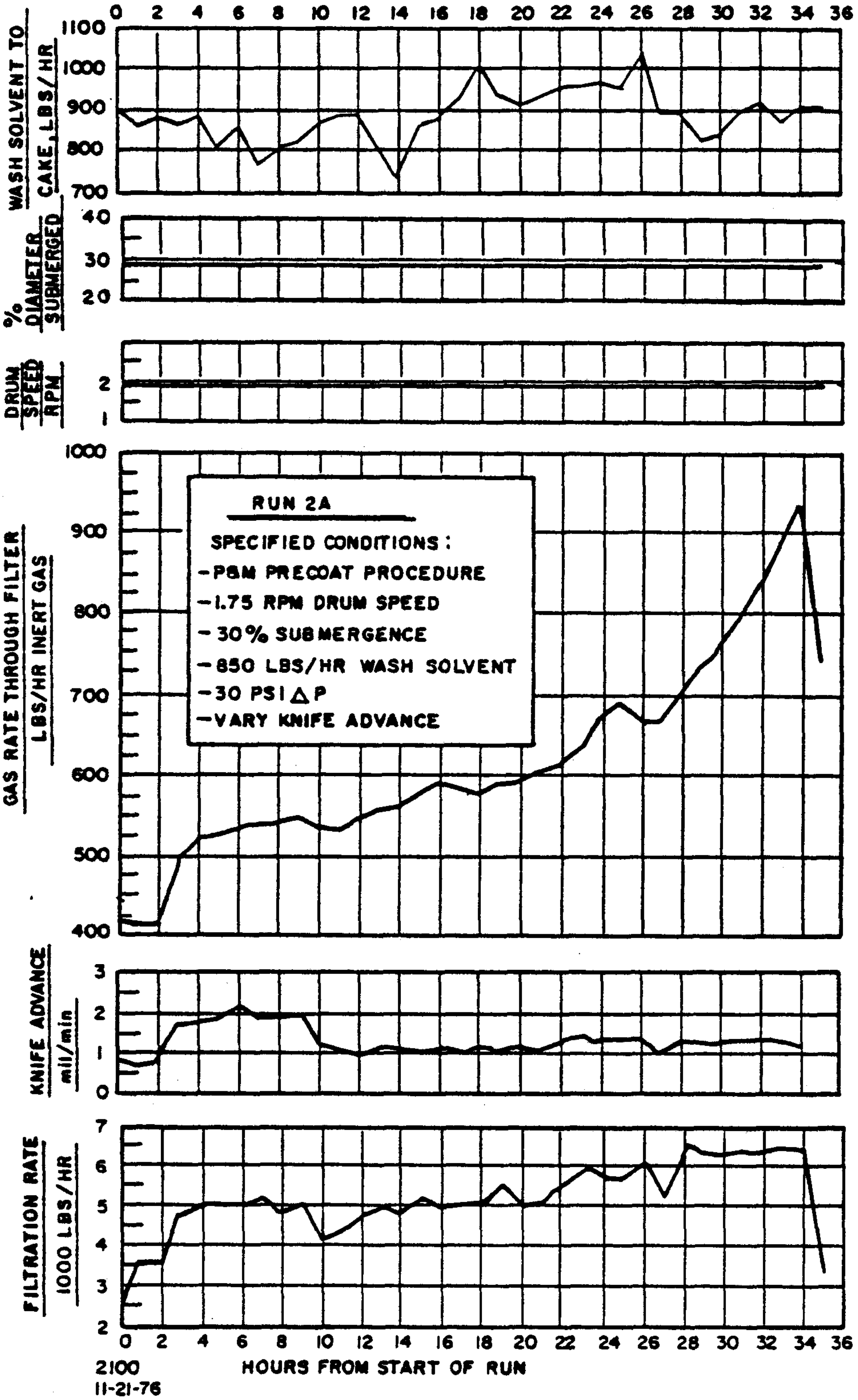


FIGURE 76

PLOT OF FILTER OPERATING DATA FOR RUN 2B

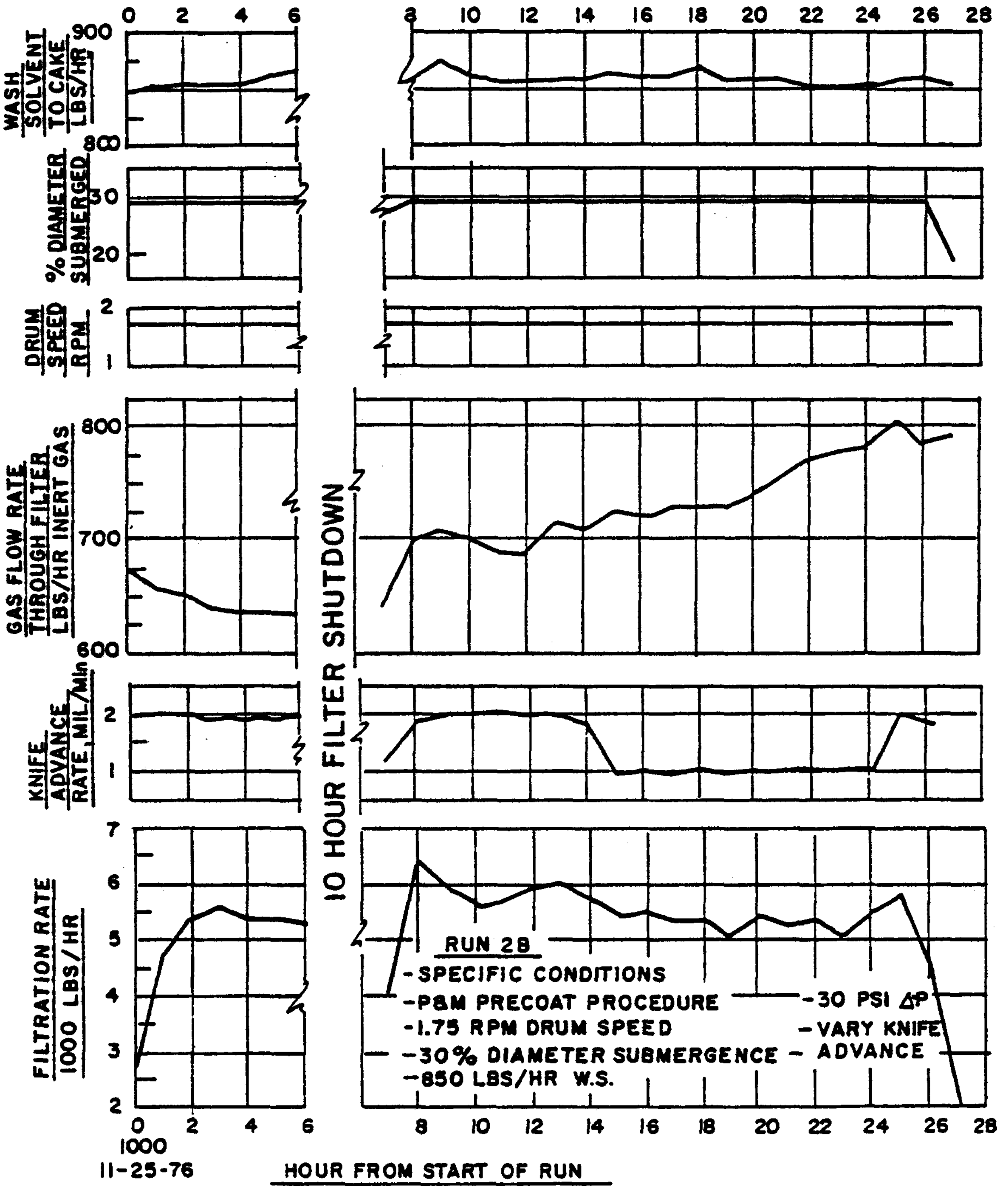
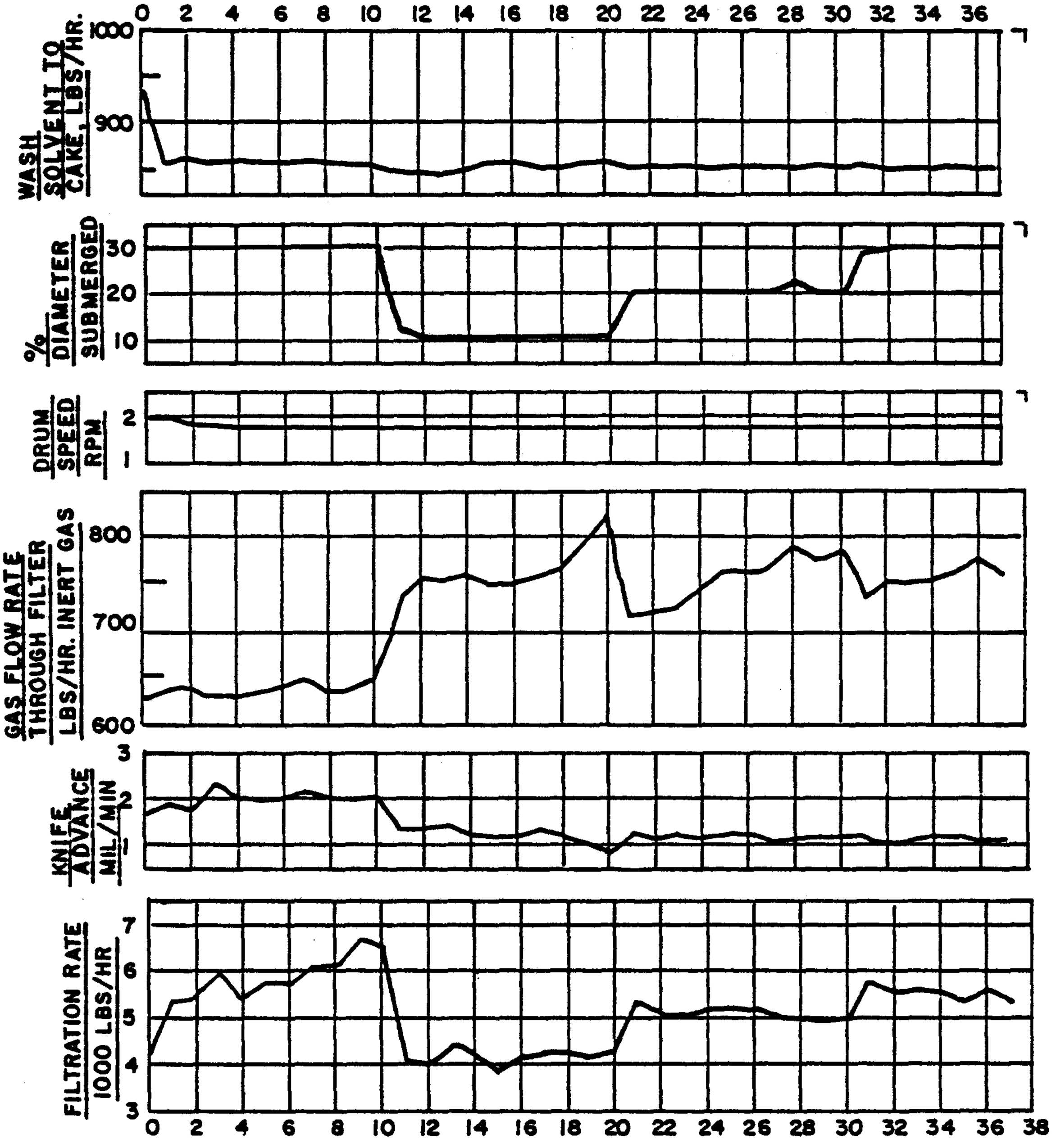


FIGURE 77

PLOT OF FILTER OPERATING DATA FOR RUN 3



RUN 3
SPECIFIED CONDITIONS

- P&M PRECOAT PROCEDURE
- 850 LBS/HR WASH SOLVENT
- VARY SUBMERGENCE
- 1.75 RPM DRUM SPEED
- 30 PSI ΔP
- VARY KNIFE ADVANCE

FIGURE 78

PLOT OF FILTER OPERATING DATA FOR RUN 4

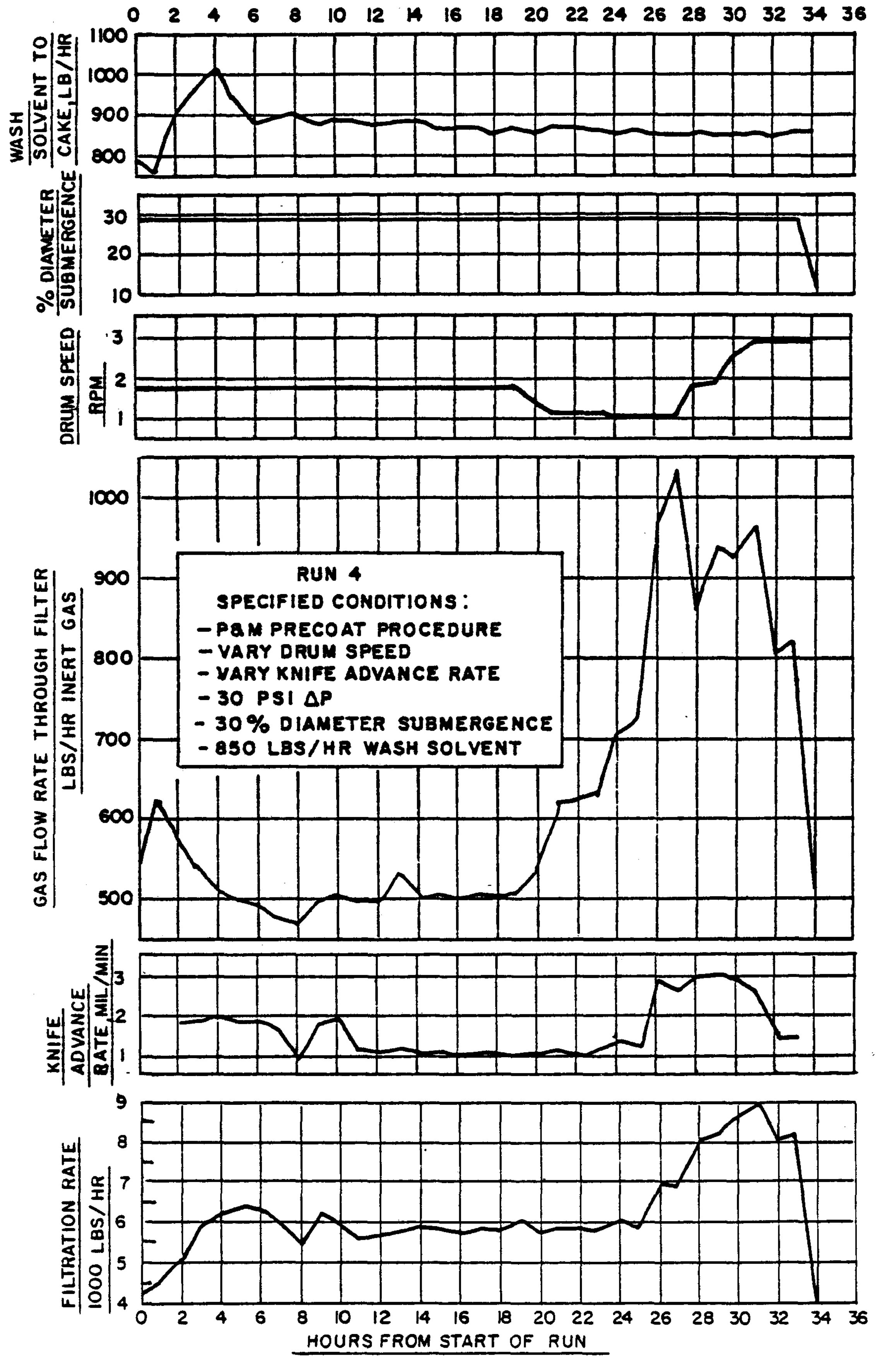
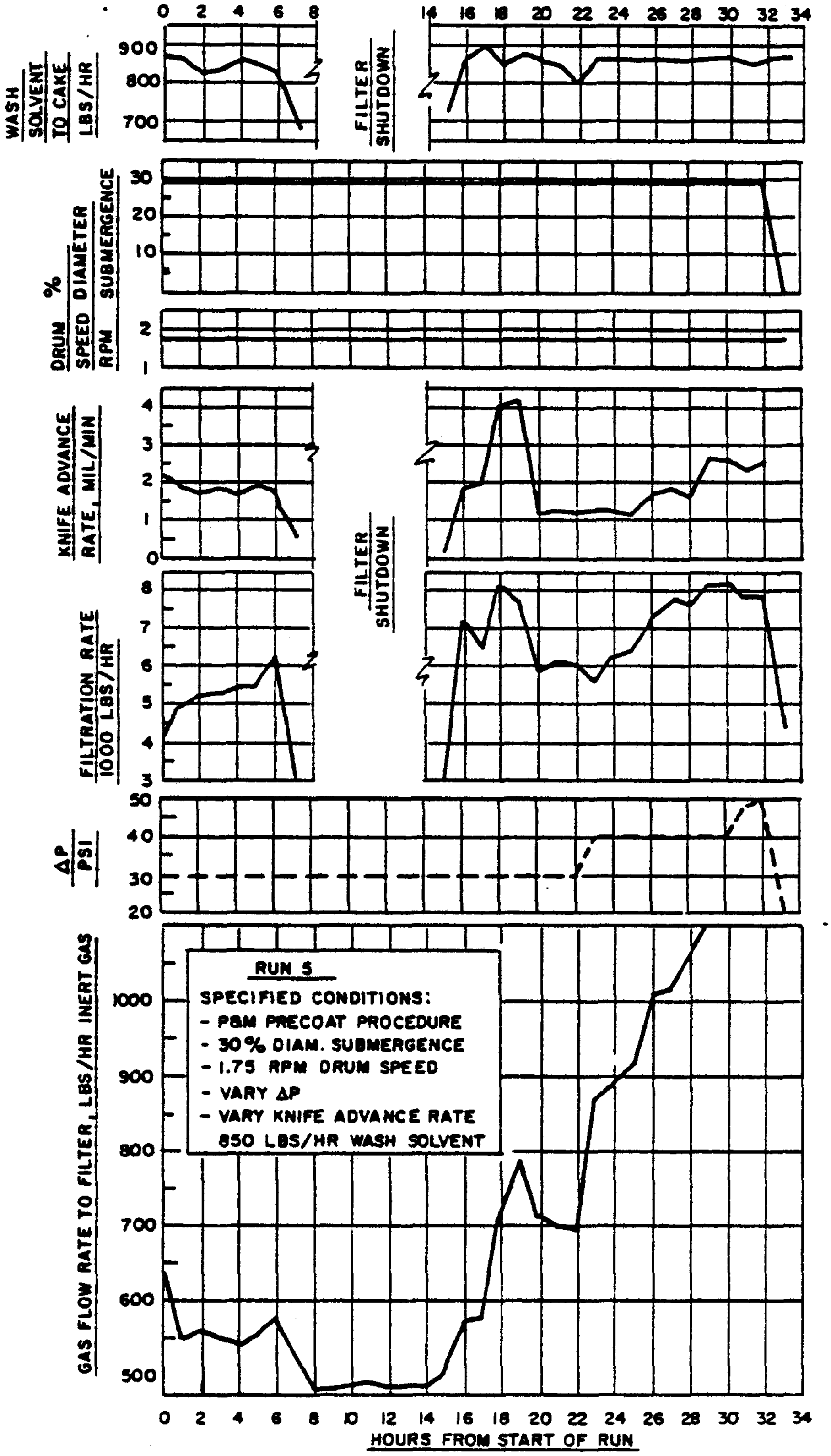


FIGURE 79

PLOT OF FILTER OPERATING DATA FOR RUN 5



RUN 5

SPECIFIED CONDITIONS:
- PBM PRECOAT PROCEDURE
- 30% DIAM. SUBMERGENCE
- 1.75 RPM DRUM SPEED
- VARY ΔP
- VARY KNIFE ADVANCE RATE
850 LBS/HR WASH SOLVENT

FIGURE 80

PLOT OF FILTER OPERATING DATA FOR RUN 6

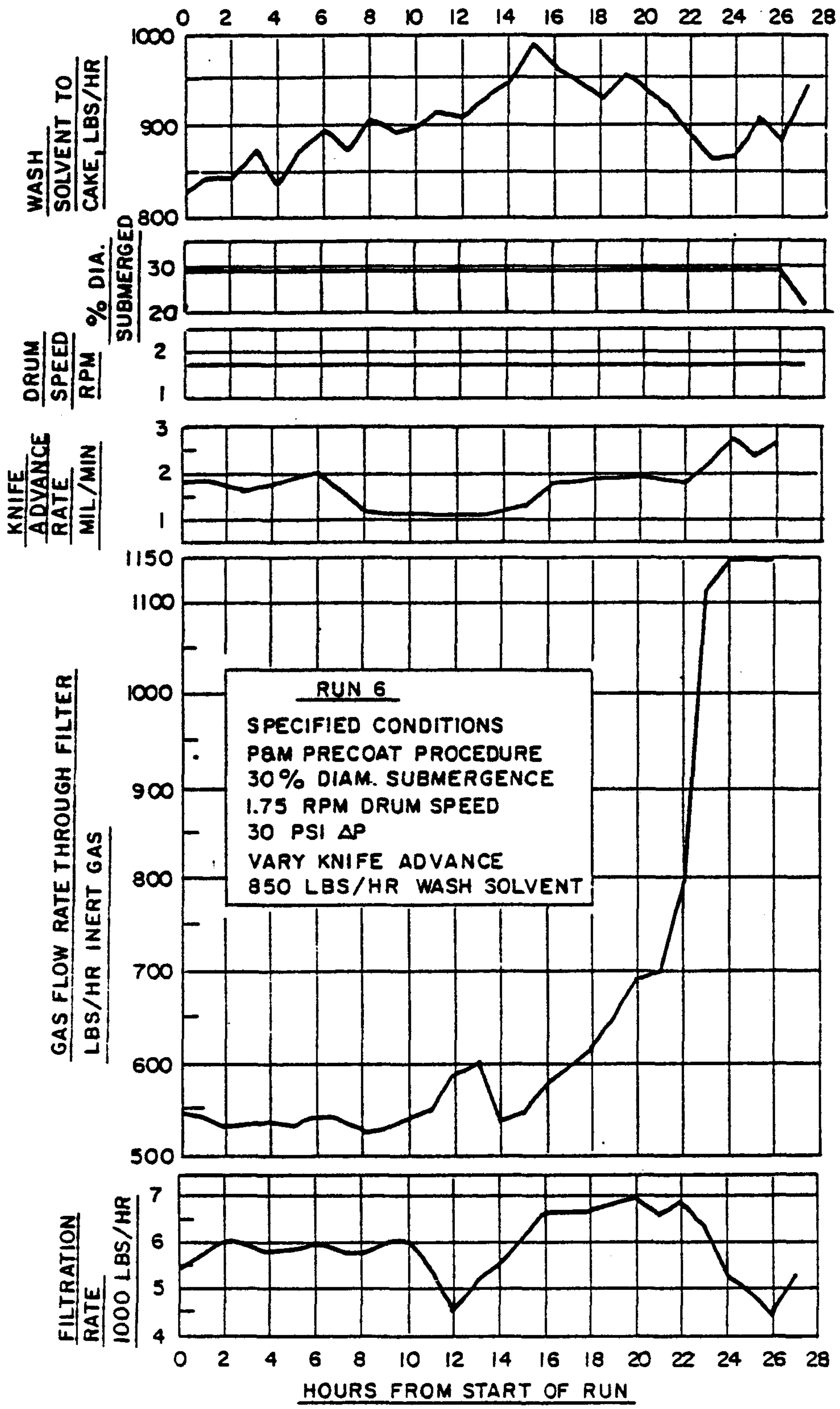


FIGURE 81

PLOT OF FILTER OPERATING DATA FOR RUN 7

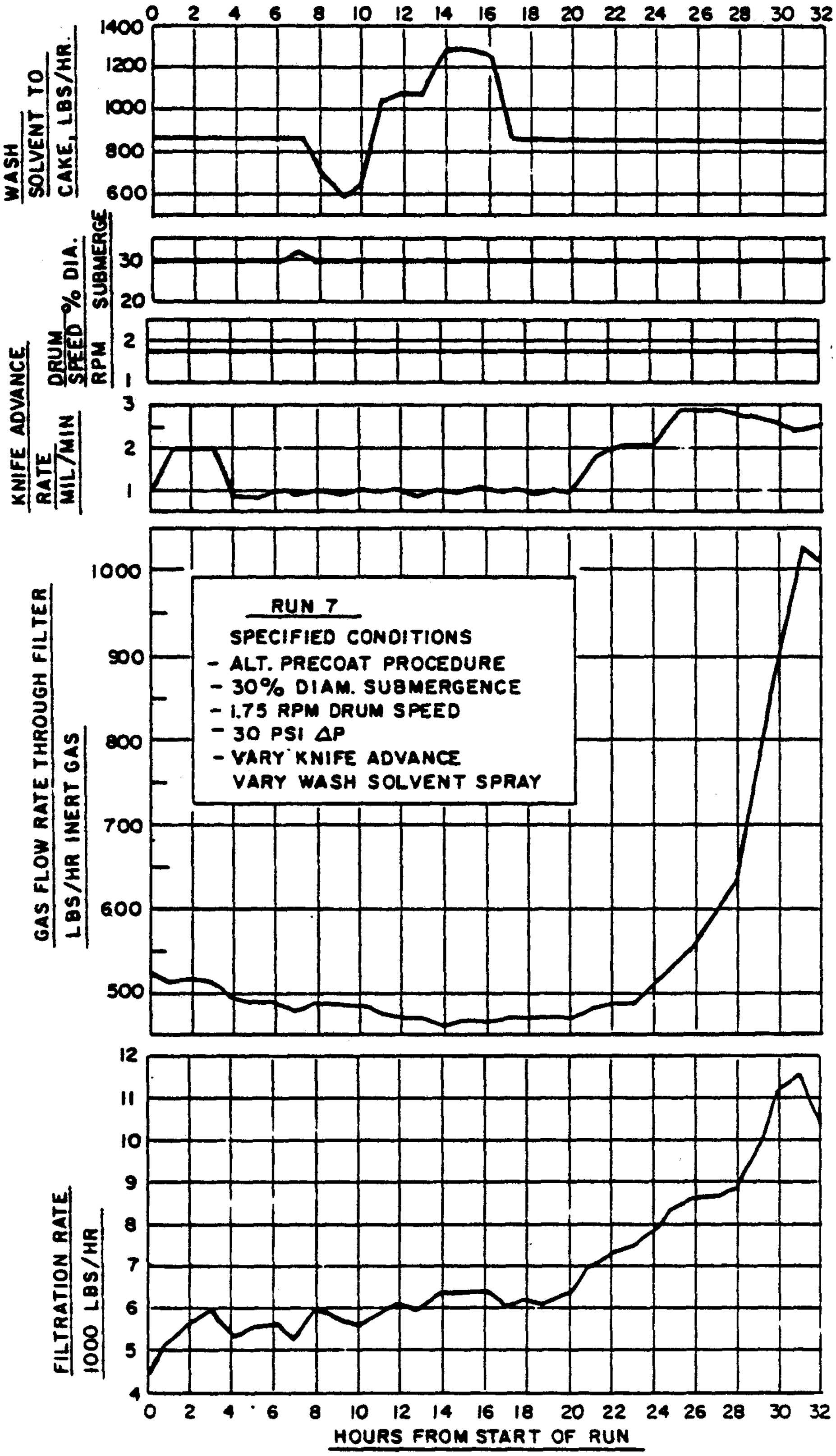


FIGURE 82

PLOT OF FILTER OPERATING DATA FOR RUN 8

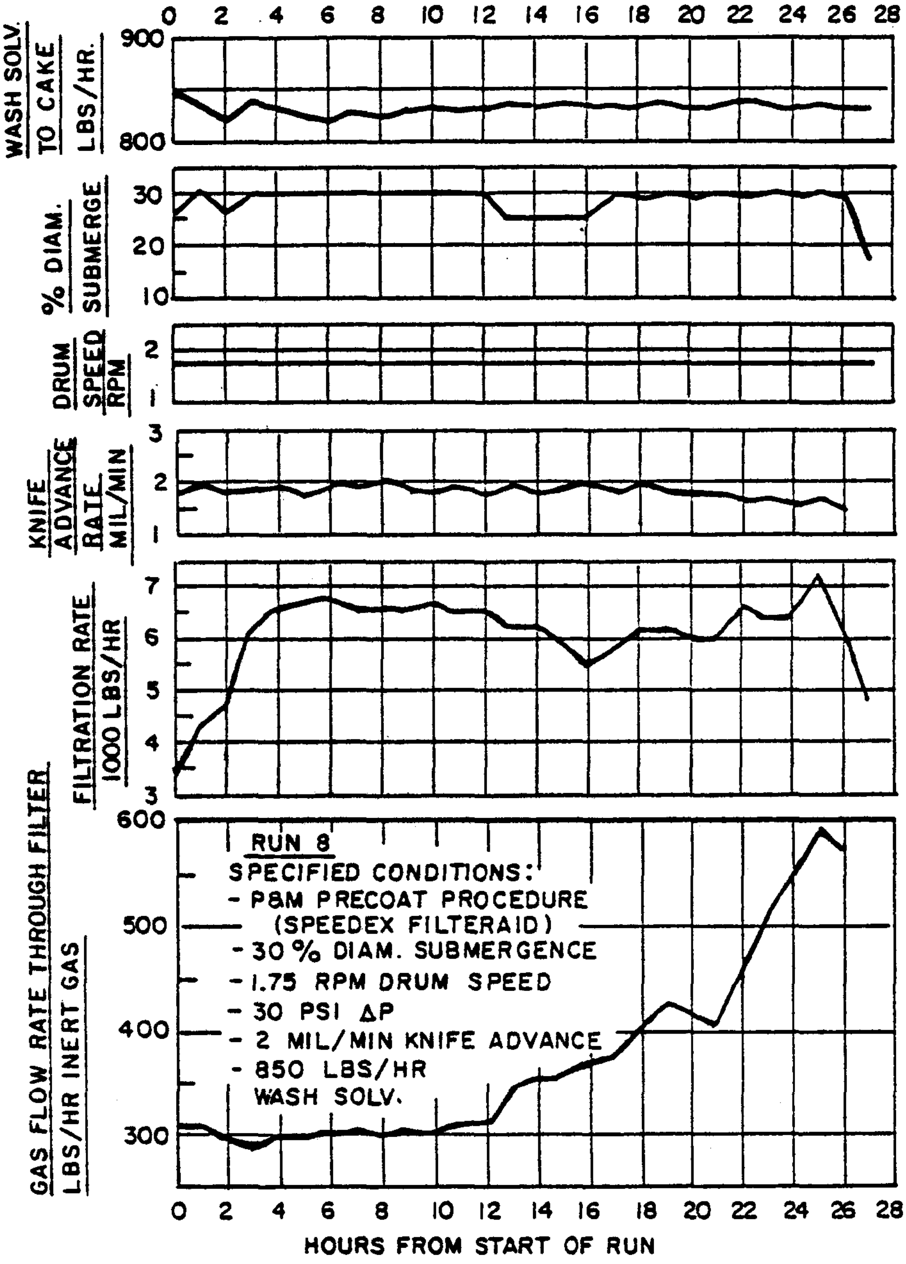


FIGURE 83

PLOT OF FILTER OPERATING DATA FOR RUN 9

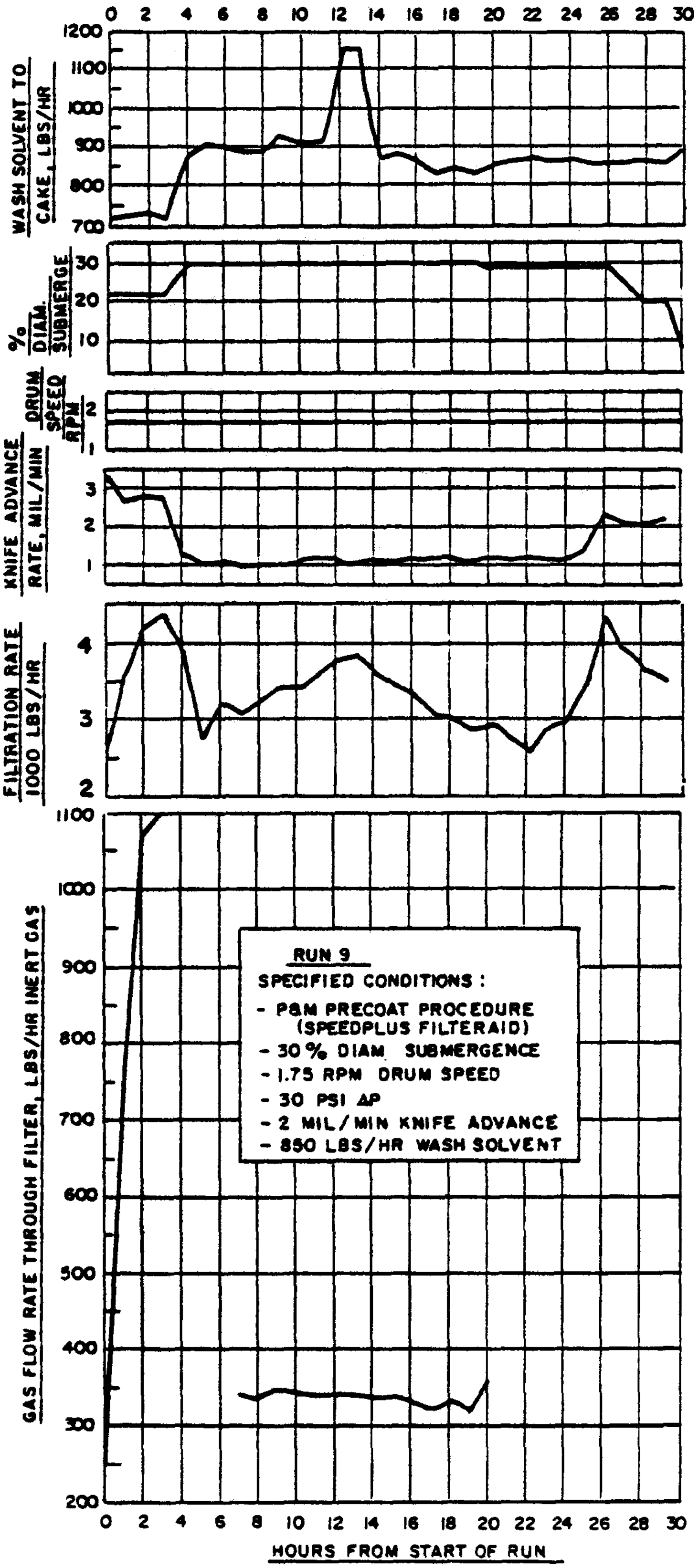
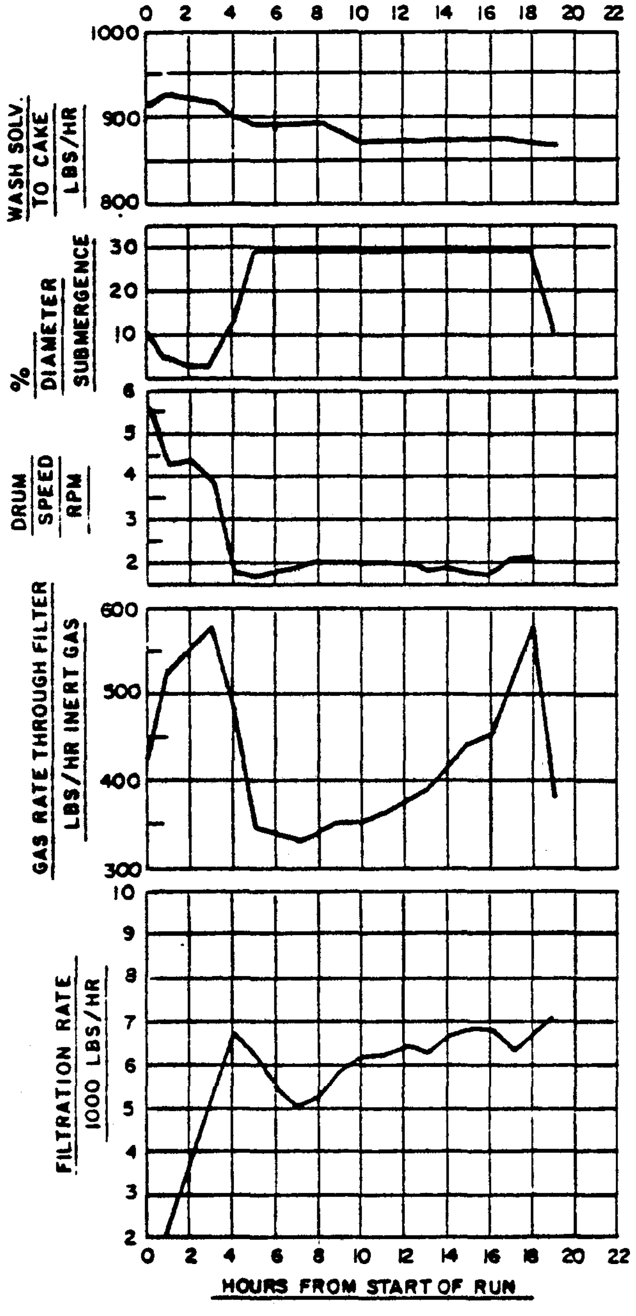


FIGURE 84

PLOT OF FILTER OPERATING DATA FOR RUN 10



RUN 10

SPECIFIED CONDITIONS:

- P&M PRECOAT PROCEDURE (SPEEDPLUS FILTERAID)
- 30 % DIAM. SUBMERGENCE
  1.75 RPM DRUM SPEED
- 30 PSI ΔP
- 2 MIL/MIN KNIFE ADVANCE RATE
- 850 LBS/HR WASH SOLVENT

FIGURE 85

TABLE 109

FILTER TEST RUN OPERATING CONDITIONS

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

CONSTANT CONDITIONS:

BASECOAT: 10 lbs 11-C

PRECOAT: 500 lbs Speedex

FILTER FEED TEMPERATURE: 520°F

FILTER TEMPERATURE: 480 - 485°F

SLURRY FEED RATE: 23000 lbs/hr

AVERAGE PERCENT PYRIDINE INSOLUBLES IN FILTER FEED: 7.6; 28% SRC

FILTER FEED VISCOSITY: ---

VARIABLE CONDITIONS & RESULTS:

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

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

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

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

TABLE 110

FILTER TEST RUN OPERATING CONDITIONS

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

CONSTANT CONDITIONS:

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

VARIABLE CONDITIONS & RESULTS:

	RUN PERIOD
	1
DURATION, HOURS	26
VAT PRESSURE, PSIG	116
Δ P, psi	29.8
DRUM SUBMERGENCE,	
Percent Diameter	29.6
Percent Area*	36.6
DRUM SPEED, RPM	1.92
KNIFE ADVANCE RATE,	
mil/min	2.0
mil/rev	1.0
CAKE WASH, LBS/HR	855
GAS RATE, LBS/HR	423-706
FILTRATE RATE, **LBS/HR	5000-6000
Percent P.I.	0.002
Percent Ash	---
Viscosity @ 100°, cSt	---

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

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

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

TABLE 111

FILTER TEST RUN OPERATING CONDITIONS

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

CONSTANT CONDITIONS:

BASECOAT: 10 lbs 11-C

PRECOAT: 500 lbs Speedex

FILTER FEED TEMPERATURE: 520°F

FILTER TEMPERATURE: 490°F

SLURRY FEED RATE: 26000 lbs/hr

AVERAGE PERCENT PYRIDINE INSOLUBLES IN FILTER FEED: 5.8; 18% SRC

FILTER FEED VISCOSITY: ---

VARIABLE CONDITIONS & RESULTS:

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

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

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

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

TABLE 112

FILTER TEST RUN OPERATING CONDITIONS

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

CONSTANT CONDITIONS:

BASECOAT: 10 lbs 11-C

PRECOAT: 500 lbs Speedex

FILTER FEED TEMPERATURE: 520 - 535°F

FILTER TEMPERATURE: 420 - 495°F

SLURRY FEED RATE: 26000 lbs/hr

AVERAGE PERCENT PYRIDINE INSOLUBLES IN FILTER FEED: 5.3; 21% SRC

FILTER FEED VISCOSITY: ---

VARIABLE CONDITIONS & RESULTS:

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

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

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

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

TABLE 113

FILTER TEST RUN OPERATING CONDITIONS

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

CONSTANT CONDITIONS:

BASECOAT: 10 lbs 11-C

PRECOAT: 500 lbs Speedex

FILTER FEED TEMPERATURE: 520°F

FILTER TEMPERATURE: 480 - 490°F

SLURRY FEED RATE: 26000 lbs/hr

AVERAGE PERCENT PYRIDINE INSOLUBLES IN FILTER FEED: 6.17; 22% SRC

FILTER FEED VISCOSITY: ---

VARIABLE CONDITIONS & RESULTS:

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

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

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

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

TABLE 114

FILTER TEST RUN OPERATING CONDITIONS

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

CONSTANT CONDITIONS.

BASECOAT: 10 lbs 11-C

PRECOAT: 500 lbs Speedex

FILTER FEED TEMPERATURE: 520°F

FILTER TEMPERATURE: 495°F

SLURRY FEED RATE: 25500 lbs/hr

AVERAGE PERCENT PYRIDINE INSOLUBLES IN FILTER FEED: 7.23; 20% SRC

FILTER FEED VISCOSITY: ---

VARIABLE CONDITIONS & RESULTS:

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

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

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

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

TABLE 115

FILTER TEST RUN OPERATING CONDITIONS

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

CONSTANT CONDITIONS:

BASECOAT: 10 lbs 11-C

PRECOAT: 500 lbs Speedex

FILTER FEED TEMPERATURE: 525°F

FILTER TEMPERATURE: 495°F

SLURRY FEED RATE: 14000 - 20000 lbs/hr

AVERAGE PERCENT PYRIDINE INSOLUBLES IN FILTER FEED: 6.27; 28% SRC

FILTER FEED VISCOSITY: ---

VARIABLE CONDITIONS & RESULTS:

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

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

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

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

TABLE 116

FILTER TEST RUN OPERATING CONDITIONS

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

CONSTANT CONDITIONS:

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

VARIABLE CONDITIONS & RESULTS:

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

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

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

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

TABLE 117

FILTER TEST RUN OPERATING CONDITIONS

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

CONSTANT CONDITIONS:

BASECOAT: 10 lbs 11-C

PRECOAT: 500 lbs Speedex

FILTER FEED TEMPERATURE: 525°F

FILTER TEMPERATURE: 495°F

SLURRY FEED RATE: 17000 - 25000 lbs/hr

AVERAGE PERCENT PYRIDINE INSOLUBLES IN FILTER FEED: 7.43; 20% SRC

FILTER FEED VISCOSITY: 16.47 cSt @ 210°F

VARIABLE CONDITIONS & RESULTS:

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

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

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

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

TABLE 118

FILTER TEST RUN OPERATING CONDITIONS

RUN 8 0700 HRS 1/13/77 to 0800 HRS 1/14/76

CONSTANT CONDITIONS:

BASECOAT: 10 lbs 11-C

PRECOAT: 500 lbs Speedex

FILTER FEED TEMPERATURE: 500°F

FILTER TEMPERATURE: 470 - 475°F

SLURRY FEED RATE: 23000 lbs/hr

AVERAGE PERCENT PYRIDINE INSOLUBLES IN FILTER FEED: 7.34; 23% SRC

FILTER FEED VISCOSITY: 31.3 @ 210°F

VARIABLE CONDITIONS & RESULTS:

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

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

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

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

TABLE 119

FILTER TEST RUN OPERATING CONDITIONS

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

CONSTANT CONDITIONS:

BASECOAT: 10 lbs 11-C

PRECOAT: 500 lbs Speedplus

FILTER FEED TEMPERATURE: 513°F

FILTER TEMPERATURE: 475 - 485°F

SLURRY FEED RATE: 26000 lbs/hr

AVERAGE PERCENT PYRIDINE INSOLUBLES IN FILTER FEED: 6.99; 24% SRC

FILTER FEED VISCOSITY: 19.8 @ 210°F

VARIABLE CONDITIONS & RESULTS:

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

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

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

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

TABLE 120

FILTER TEST RUN OPERATING CONDITIONS

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

CONSTANT CONDITIONS:

BASECOAT: 10 lbs 11-C

PRECOAT: 500 lbs Speedplus

FILTER FEED TEMPERATURE: 513°F

FILTER TEMPERATURE: 485°F

SLURRY FEED RATE: 27000 lbs/hr

AVERAGE PERCENT PYRIDINE INSOLUBLES IN FILTER FEED: 5.8

FILTER FEED VISCOSITY: 26.7 @ 210°F

VARIABLE CONDITIONS & RESULTS:

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

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

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

COMMENTS: The precoat application in this run resulted in a grooved and uneven precoat. As a result, 1 1/2 inches of precoat were removed prior to beginning the run. This resulted in the run being of rather short duration. The data obtained during lined out operation indicate that the filtration rates with Speedplus filter aid are comparable to those obtained with the most favorable Speedex precoats. The gas flow through the filter was noted to increase at the end of the run, as had been the case in the Speedex precoat runs.

b. Mineral Residue Dryer Heat Transfer Study

A 48 hour test run was conducted from December 14 to December 16, 1976, on the mineral residue dryer to evaluate its heat transfer capability. The dryer had been returned to service on December 9, after decoking by hydroblasting. Table 121 summarizes the findings of the test run. Average pyridine insolubles for dryer feed, product and condensate and the total production of dry mineral residue were used to calculate the total slurry feed by forcing a pyridine insoluble balance. An equivalent coal feed rate to the dissolver was calculated from the basis of the dryer feed. However, during this test, no attempts were made to maximize dryer feed rate.

Total heat input was calculated from flue gas rates. Heat loss to the flue gas was calculated from flue gas temperature using an estimate of flue gas heat capacity based on Orsat analysis. The heat transfer coefficient was calculated from the net heat transfer and from dryer dimensions and was found to be approximately 7.9 BTU/ft^2 hr °F.

TABLE 121

SUMMARY OF DATA FROM MINERAL RESIDUE DRYER HEAT TRANSFER STUDY

Average Pyridine Insolubles (Wt.%), Slurry Feed		44.7
Average Pyridine Insolubles (Wt.%), Dry Mineral Residue		98.7
Average Pyridine Insolubles (Wt.%), Dryer Condensate		3.9
Total Dry Mineral Residue Produced During 48 Hrs, Lbs		19,580
Calculated Total Slurry Feed For 48 Hrs, Lbs		45,420
Total Fuel Gas To Dryer, SCF		92,530
Total Heat Input to Dryer, BTU		9.580×10^7
Slurry Feed Temperature, °F		243
Dryer Outlet Temperature, °F		683
LMTD, °F		393
Flue Gas Discharge Temperature, °F		1000
Orsat Analysis Of Flue Gas:	O_2, (Vol %)	11.1
	N_2, (Vol %)	83.8
	CO, (Vol %)	0.1
	CO_2, (Vol %)	5.0
Calculated % Excess Air		109
Heat Loss to Flue Gas, %		49
Net Heat Supplied To Dryer, BTU/LB Wet Slurry		1075
Heat Transfer Coefficient*, $BTU/ft^2.hr.°F$		7.9
Calculated Coal Feed Rate, TPD		32.6

* Assuming ALL Heat Loss Is To Flue Gas

3. Product Solidification and Storage

a. Sandvik Belt Studies

Tests for specific cooling capacity and flaking and breaking tests were conducted by Sandvik Conveyor, Inc. on two samples of solvent refined Kentucky coal provided from the pilot plant at Fort Lewis.

Product	P&M No.	% Ash	Melting Point °F
Specification SRC	229-A	0.17	305
Unfiltered SRC	229-B	15.89	330

The test equipment was comprised of a stainless steel belt 18 inches wide with coolant spray applied from below for a distance of 18 feet. An air hood was used to blow ambient air on the product and water sprays on top of the product were used in part of these tests.

In these tests the hot feed material was formed on the belt in 1/8-inch and 1/4-inch thicknesses. For each case, retention time for the product to reach the desired discharge temperature was measured. The specific capacity for cooling (lb/hr ft^2) was calculated using the retention time, thickness, and a specific weight of 80 lb/ft^3.

It was determined that an average discharge temperature of 175°F was a practical maximum for reliable breaker operation. At higher temperatures central portions of thicker slabs were still soft and could jam the breaker.

Results are summarized in Table 122 and Figure 86. Cooling time is obviously shorter for the thinner product and when air hoods are used. The effect of thickness may be seen more clearly in Figure 86. Here, we also see that the unfiltered SRC with ash in the product cools slightly faster than the specification product. The differences between the two products are minor, however, compared to the differences due to product thickness. Maximum capacity with a particular size flaker will be obtained with the thinner products.

Samples of product were taken and run through a set of standard sieves using a mechanical shaker. The resulting particle size distribution was then plotted to obtain the value of sieve opening through which 80% by weight of the total sample would pass. These values are given in the last column of Table 122.

The data show that the specification SRC is slightly more brittle than the unfiltered SRC and forms smaller pieces. In addition, the thicker flake (1/4-inch) resulted in an average particle size more than twice as large as the 1/8-inch flake.

TABLE 122

SRC COOLING TEST RESULTS BY SANDVIK, INC.

1/8" THICK

Product	Tf (°F)	Tc (°F)	Td* (°F)	Air Hood and Water Sprays	Retention Time (min)	Specific Capacity (lb/hr/ft^2)***	80% Mesh Size** (inches)
"229-A"	450	60	175	no	1.17	38.3	.33
"A"	450	60	175	yes	.63	71.3	.29
"B"	450	60	175	no	.97	46.2	.56
"B"	450	60	175	yes	.48	92.7	-

1/4" THICK

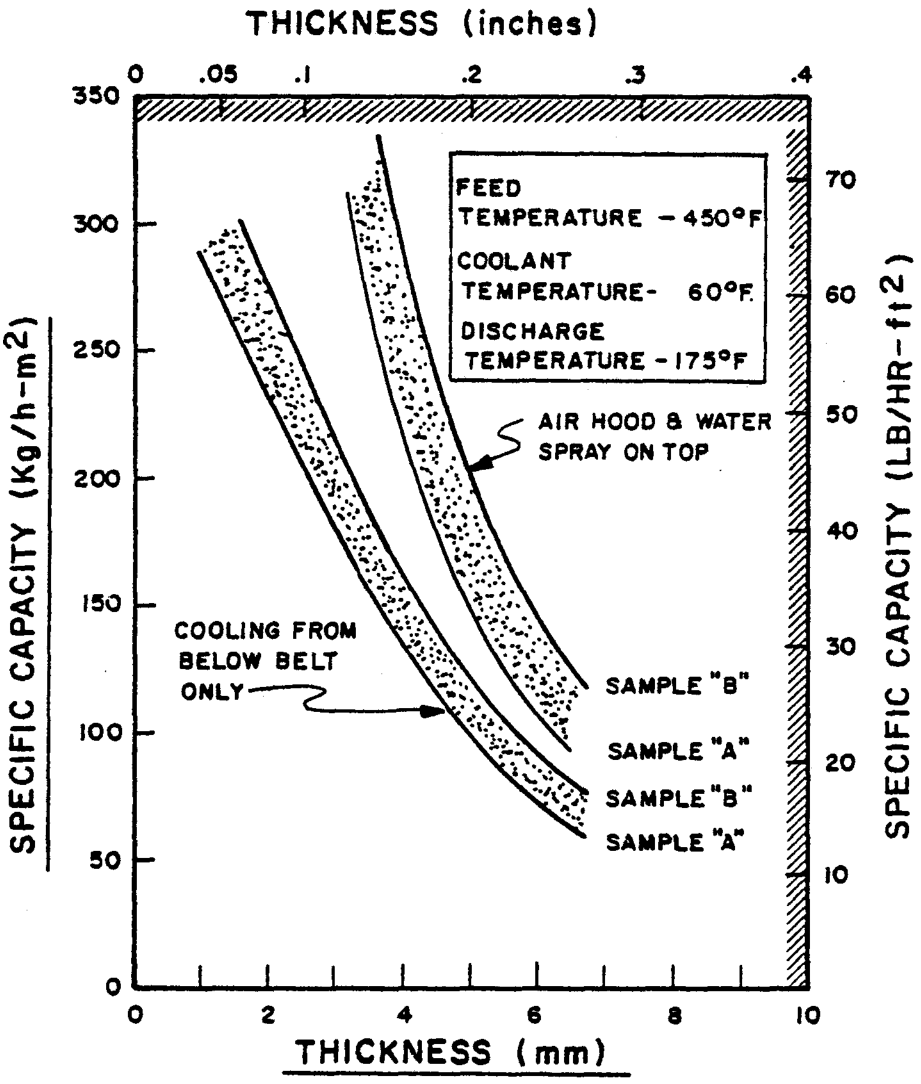
Product	Tf	Tc	Td*	Air Hood and Water Sprays	Retention Time (min)	Specific Capacity (lb/hr/ft^2)	80% Mesh Size (inches)**
"229-A"	450	60	175	no	5.65	15.8	.73
"A"	450	60	175	yes	3.73	24.5	-
"B"	450	60	175	no	4.65	19.2	-
"B"	450	60	175	yes	3.1	28.9	.63

* Measured by a thermometer after product was crushed
** 80% of particles pass thru this mesh opening
*** These values contain a 10% safety factor for scale-up

Product "A" is filtered SRC
Product "B" is unfiltered SRC

Tf = Product feed temperature
Tc = Coolant temperature
Td = Product discharge temperature

GRAPHICAL PRESENTATION OF SANDVIK TEST RESULTS



SAMPLE "A" IS FILTERED SRC
SAMPLE "B" IS UNFILTERED SRC

FIGURE 86

b. SRC Agglomeration Studies

An experimental study was completed which investigated the tendency of SRC flakes to "agglomerate" or clump into a solid mass during storage. This agglomeration of SRC has previously been observed in the Fort Lewis storage silos.

A small laboratory test apparatus was designed and constructed to simulate SRC storage at various conditions. A total of 68 experimental runs were made to determine the conditions which can lead to agglomeration. Typical specification SRC product with a fusion point of 320°F was selected for the study, and this material was classified to 7 or 8 mesh prior to its introduction into the test apparatus. Conditions of study ranged from room temperature to 275°F and from 15.6 to 217.4 psi.

Agglomerated SRC produced in the test apparatus appeared to be indentical to that previously seen in the pilot plant storage bins.

Experimental results showed that as SRC is subjected to increasing levels of temperature or pressure stress, it gradually changes to a partially agglomerated, fully agglomerated and finally a melted state. This is shown in Figure 87 where the results are plotted on temperature vs. pressure coordinates. The conditions required to produce agglomeration of SRC are shown as a shaded area on Figure 87.

The effect of time on SRC agglomeration was also evaluated in several tests. Those tests were conducted at temperatures ranging from 70°F to 275°F, and constant pressure (118.5 psi). Bulk densities were determined as a function of time at each temperature condition. The results, shown in Figure 88, indicate that agglomeration might be dependent on elapsed time. However, the apparent effect of time on agglomeration may not be significant due to a lower SRC initial temperature (ambient) than the desired test temperature and poor heat transfer and SRC heat conductivity. It is felt that if lags due to heat transfer and thermal conductivity were eliminated, the time effect on agglomeration might be negligible. An additional test run, not shown in Figure 88, confirmed a hypothesis that at room temperatures, the effect of time on agglomeration is negligible. In this test, a sample of SRC was stored at room temperature under a pressure of 118.5 psi. After approximately four months of storage, the SRC was examined and no signs of agglomeration were found.

Based on the foregoing experimental investigation, it appears that the following conclusions can be drawn regarding the agglomeration tendency of SRC:

- The tendency of stored SRC to agglomerate is related to its thermal history, storage temperature and storage pressure.
- Normally, SRC agglomeration should not be a problem at the Pilot Plant since the conditions which favor agglomeration are far beyond those found in the plant storage silos.
- To prevent problems of SRC agglomeration in future plants, the SRC product should be well fractionated to ensure separation of lighter fractions. The SRC should also be stored at conditions below those shown in the shaded region of Figure 87.
- The SRC agglomeration which was previously seen in storage bins at Fort Lewis may have been caused by unusual material such as poorly cooled or poorly fractionated SRC being placed in storage.
- The effect of time on agglomeration is negligible.

SRC AGGLOMERATION vs PRESSURE AND TEMPERATURE

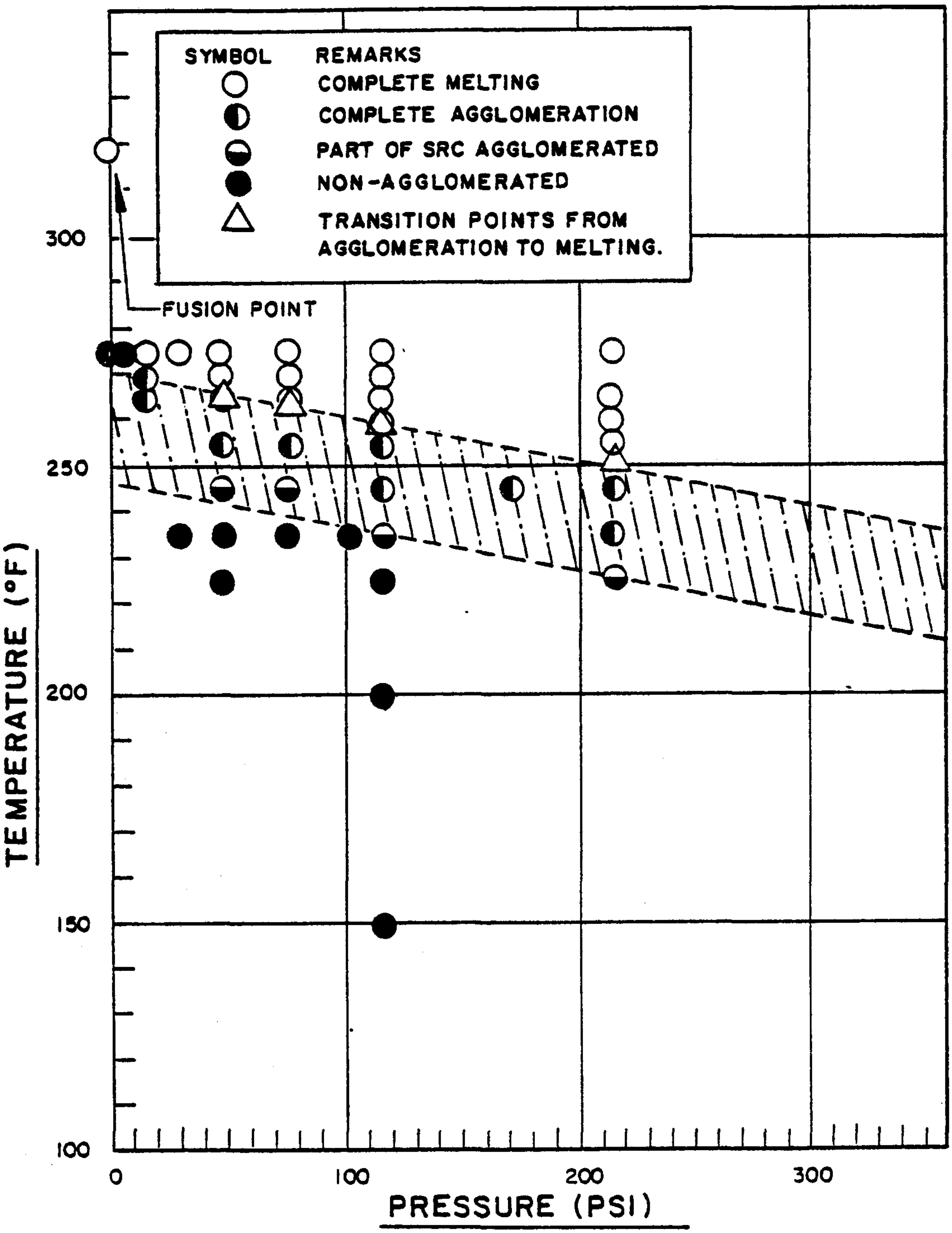


FIGURE 87

TIME EFFECT ON SRC AGGLOMERATION
AT 118.5 PSI

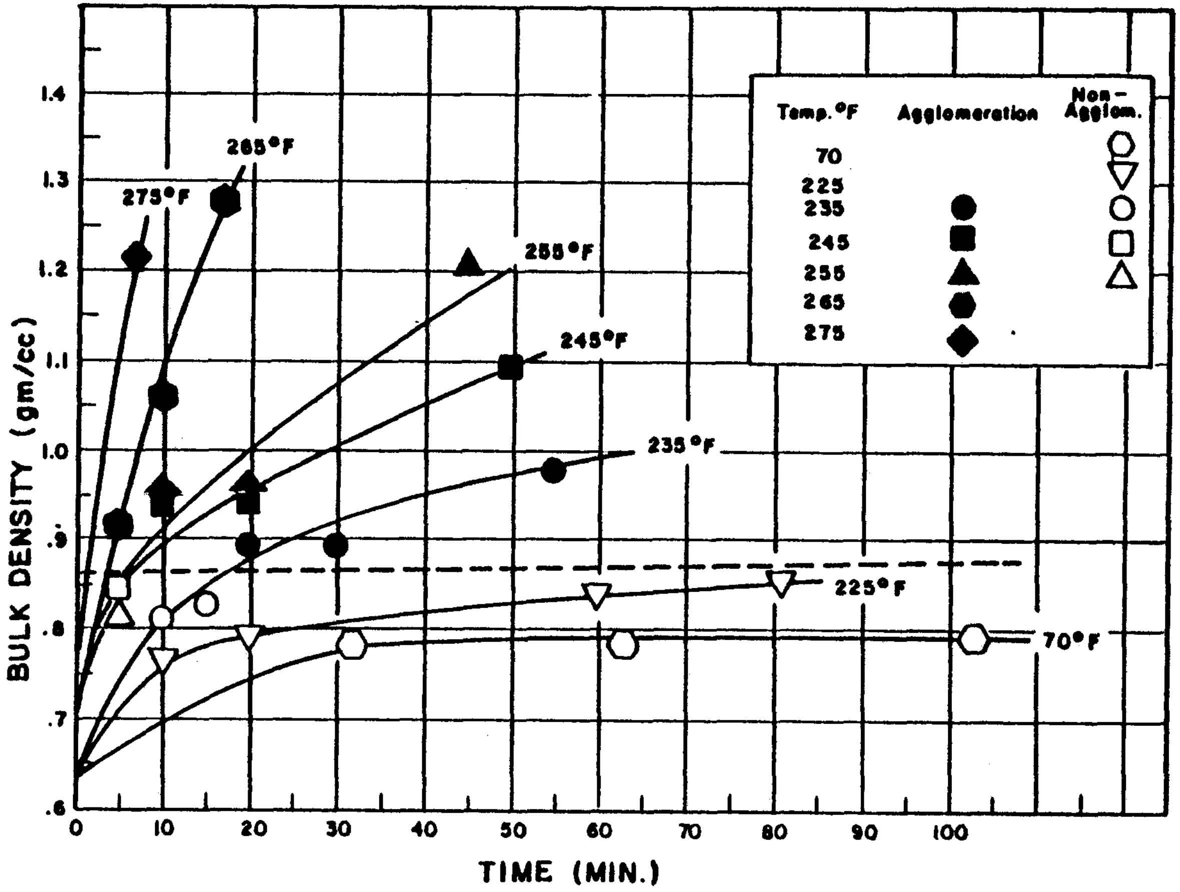


FIGURE 88

F. PHYSICAL AND CHEMICAL PROPERTIES

1. Coal Characterization

All of the coal which has been used at the Fort Lewis SRC Pilot Plant since startup has been from the P&M mine at Madisonville (Hopkins County), Kentucky. It is a blend of 70% Kentucky No. 14 seam coal and 30% Kentucky No. 9 seam coal, which are both classified high volatile A bituminous by ASTM D388. Early laboratory studies involved this feedstock, and it was this coal which was tested at the previous process development pilot plant described in OCR Research and Development Report No.9(3). With this background information, it was also chosen for initial studies at Fort Lewis.

Before loading for shipment, the coal is washed and screened to approximately 1 1/4" to 28 mesh size. This pretreatment removes about 50% of the ash and about 1% of the sulfur in the raw coal. Before processing, the coal is pulverized under hot inert gas which reduces the moisture content to about 1%. A typical sieve analysis of the pulverized coal is:

TABLE 123

Sieve Analysis of Pulverized Kentucky Coal

Sieve No. (ASTM E11)	Differential Wt. % Retained	Total Wt. % Passed
100	1.33	98.67
140	5.97	92.70
200	10.95	81.75
370	17.87	63.88
325	9.55	54.33
400	15.24	39.09
Minus 400	39.09	

Average proximate, ultimate, and mineral analyses of the coal processed in 1975 and 1976 are found in Tables 124 and 125. A summary of routine forms of sulfur analyses performed on weekly composites of feed coal are included in Table 126. Petrographic analysis of a composite of feed coal for 1976 is found in Table 127.

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(3) Dean Kloepper, et al, Solvent Processing of Coal to Produce a De-ashed Product, PB 167 809 NTIS.

2. Density and Specific Gravity

a. Density of Solids

Table 128 shows typical values of bulk density and true density for coal and solid SRC process products handled at the pilot plant. It should be remembered, however, that these values are subject to considerable variation. Bulk density of coal will usually vary with the run of the mine and with the degree of size reduction. Bulk density of SRC product shows variation according to the manner of solidification, as shown by the difference between the bulk density of Sandvik belt product and that of cast product (Table 128).

Low ash SRC product is brittle and excessive handling by elevators, conveyors and vibrators results in further size reduction and a corresponding increase in bulk density. The manner in which a container is filled has an unexpectedly large effect on apparent bulk density of SRC product. Shaking a freshly filled container will cause considerable reduction in the bulk volume of the SRC. The true density (ASTM D-71) of SRC product is generally less variable, except that a measureably higher value is found for SRC with an appreciable ash content (Table 128).

b. Specific Gravity of Liquids

Specific gravity (ASTM D-1298) versus temperature relationships for typical plant liquids are shown in Figure 89. Specific gravity of other liquids may be approximated using parallel lines if a specific gravity at one temperature is known. Specific gravity is also related to the average boiling point of the liquid. Table 129 lists distillation data along with specific gravity for selected plant liquids.

3. Distillation Data

Typical temperature versus volume percent distillate curves for plant solvent cuts and for startup solvent are shown in Figures 90,91,92, and 93. Analysis is by ASTM D-86 and a modification of ASTM D-1160(4). A nomograph relating reduced pressure distillation of process solvent to atmospheric pressure is provided in Figure 94. This was constructed by comparison of ASTM D-86 and vacuum distillations for a number of process solvent samples. Averaged distillation

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(4) P&M Procedure F103-75

data for eleven material balance runs on SRC-I and five runs on SRC-II are presented in Tables 130 through 135. In addition, average distillations for various plant liquids during the 1976 production run are included in Tables 136 and 137.

4. Heat Capacity

Figure 95 and Table 138 show heat capacity versus temperature relationships for wash solvent, process solvent, and SRC which were obtained using a differential scanning calorimeter. Measurements were not obtained above the ranges shown due to problems with increasing vapor pressure and thermal expansion, although it is believed the results may be extrapolated with reasonable confidence. SRC shows an inflection in the heat capacity curve in the region of its fusion point.

5. Viscosity

Process liquids exhibit essentially Newtonian behavior in the absence of solid matter. In general, viscosity versus temperature relationships for process liquids may be described by a family of parallel lines on standard viscosity paper (Figure 96). Viscosity at a given temperature can be related to volumetric average boiling point (VABP) for oils which are light enough to distill completely at atmospheric pressure. Very light naphtha, which contains a large saturated hydrocarbon fraction, shows a viscosity versus temperature relationship which deviates somewhat from that of the more aromatic oils (shown by the different slope of the light oil curve in Figure 96). Viscosity versus temperature for the initial plant startup solvent blend is shown in Figure 97.

The inclusion of solid matter in the fluid stream presents several complications to the determination of viscosity. Concentrated (approx. 50%) slurries of mineral residue in wash solvent have been shown to be pseudoplastic. Slurries of unreacted coal (about 40%) in process solvent at low temperature are Newtonian, but as temperature increases to around 300°F the coal begins to absorb solvent and swell. A viscosity minimum is usually observed near this temperature. As the temperature of the slurry increases further, the coal begins to react with the solvent and a gel point is reached causing an abrupt increase in viscosity. If temperature is increased further, pyrolysis and depolymerization have the effect of decreasing viscosity.

Many factors (such as temperature, pressure, time, coal concentration, coal composition, etc.) are known to have an effect on this sequence of events and therefore a clear description of fluid behavior is still lacking for coal/solvent slurries. SRC-II feed slurries are pseudoplastic below reaction temperatures and for a limited range of shear rates obey a power law flow model.

Pour points (ASTM D-97), a viscous property, are given in Table 139 along with cloud points (ASTM D-2500) for plant solvents.

6. Surface Tension

Surface tension of samples of light oil, wash solvent, and process solvent were determined at 25°C by the capillary height method using the following formula:

$$\sigma = \frac{rdgl}{2}$$

where: σ = surface tension (dynes/cm)

d = density of liquid (grams/cm^3)

g = gravitational constant (980 cm/sec^2)

l = height of liquid in capillary (cm)

r = radius of the capillary

The radius of the capillary was determined by back-calculating from capillary height measurements with liquids of known surface tension (acetone, n-propanol). Densities and measured surface tensions are presented in Table 140.

7. Particle Size

A sample of filter feed material (sample point 309, 10/2/76, 1720 hours) was analyzed for particle size distribution by Coulter counter and by computer assisted scanning electron microscopy (SEM). The Coulter method requires dispersion of the sample in an electrolyte solution (in this case, lithium chloride in methanol) which, for coal solution requires that the sample first be dispersed in pyridine. The inclusion of a small amount of pyridine in the electrolyte solution limits the sensitivity of the counter to particles above 0.79µm diameter. The SEM technique measures a smaller number of particles (in this case 2358) and requires solvent washing (pyridine) and mounting of a representative sample on a solid surface. Diameters are measured along four axes for each particle. The SEM technique is sensitive for particles from 0.06 µm to about 50 µm. Figure 98 shows a comparison of average diameter distributions for both methods. Table 141 compares differential number and volume percents determined by both methods. The reason for the considerable discrepancy between these two methods is unclear at this time. Figure 99 shows photomicrographs made by the SEM of the sample which was analyzed by this technique. The magnification is 2000 diameters.

8. Molecular Weight

Average molecular weights were determined by freezing point depression on selected plant solvent cuts shown in Table 142. Since the interpretation of molecular weights requires reference to boiling points, these data are also included. Molecular weight of SRC was not determined because a suitable solvent was not available.

9. Elemental and Mineral Analysis

Elemental analyses of various pilot plant materials are shown in Table 143. These analyses are all averaged over the same period to facilitate comparison. Mineral analysis of ash from solvent refined coal, mineral residue, and dissolver coke are found in Table 144. Of particular interest is the concentration of titanium in the SRC ash. This is apparently distributed in the coal solution as an organometallic solute (or at least as a very finely dispersed colloid), and being so, is unfilterable. The presence of iron, silicon, and aluminum in the SRC ash indicates that some particles of coal solids also escape filtration. Note that the mineral residue which was analyzed was contaminated with approximately 5% filter aid which is primarily silica.

10. Detailed Characterization

This section covers detailed chemical analyses which have been carried out on samples of SRC, process solvent, wash solvent, and light oil. Much of this analysis has been done by modifications of methods developed for petroleum. Numerous problems were encountered during the course of these studies due to the essentially different chemical nature of coal derived material. However, it is believed that analysis of these solvents according to standard procedures, while perhaps not achieving a precise characterization, at least will define reproducible classes of compounds whose exact nature may eventually be elucidated.

Samples of SRC, process solvent, and wash solvent were submitted for analysis by SARA (for saturates, aromatics, resins, and asphaltenes) chromatography(5). (Table 145) In this method, samples are fractionated to n-pentane soluble (maltenes), pentane insoluble-benzene soluble (asphaltene), and benzene insoluble components. The maltene fraction is further separated by countercurrent absorption on several stationary phases which absorb the resins (polar, nonhydrocarbons) and from which oils (hydrocarbons) are eluted. The succession of stationary phases for absorption of resins includes cationic, weak anionic (bicarbonate form), and strong anionic (hydroxyl form) exchange resins followed by iron chloride treated clay. The desorption of these subfractions by successively more polar solvents results in the basic, acidic I, acidic II, and neutral resin classifications shown in Table 145. The separation of resin fractions of wash solvents by this technique was not achieved due to the much higher concentrations of strongly absorbing substances. However, the wash solvents were extracted exhaustively with caustic and with acid and these fractions were analyzed by gas chromatography. These data are presented in Table 146. The separation of the oil fractions into saturates and aromatics was accomplished by high pressure chromatography on silica gel. With process solvents and wash solvents, a sufficient quantity of aromatic hydrocarbon fraction was isolated for mass spectrometric group type analysis which is summarized in Tables 147 and 148. Analysis of these same samples according to the usual control procedures is included as Table 149 to provide a frame of reference to plant conditions.

Characterizations of two separate light oil samples were conducted, one (No. 255, 11/10/75) emphasizing capillary gas chromatography and the other (No. 31, 4/16/75) using a combination of liquid chromatography and mass spectrometry. Sample No. 255 was extracted with caustic to yield a phenolic concentrate which was analyzed by capillary GC separately and an unextracted aliquot of the original sample was analyzed by capillary GC for volatile aromatics and saturates. These data are presented in Table 150. Table 151 summarizes other analyses performed on the original sample. Sample No. 31 was fractionated by preparative high pressure liquid chromatography into aromatic, saturate and polar fractions. The polar fraction was not recovered, but the aromatic and saturate fractions were analyzed by mass spectrometry. Table 152 summarizes

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(5) D.M. Jewell, et al., Ind. Eng. Chem. Fundam., Vol. 13, No. 3, 1974, pages 278-282.

these data and Table 153 shows other analyses performed on Sample No. 31.

11. Vapor-Liquid Equilibrium

An extended study to determine equilibrium K-values for components of the recycle condensate separator and the high pressure flash vessel systems resulted in the values shown in Table 154. The equilibrium K-values were calculated as the ratio of the mole fraction of a component in the vapor phase to the mole fraction of the same component in the liquid phase. These ratios were calculated from data obtained from on-line gas chromatographic analyses of the recycle condensate separator vapor stream, and laboratory analyses of high pressure liquid samples for liquid composition and for dissolved gases.

In this manner, it was possible to obtain equilibrium K-values for the components of the gas phase in the recycle condensate separator, and for all components in the high pressure flash vessel. The liquid phase in both vessels was split into pseudocomponents according to the boiling range fractions used in the plant. All material in the 0-380°F boiling range was designated light oil, the 380-480°F range was designated wash solvent, the 480-850°F range was designated vacuum bottoms.

Wide variations of the oil-water ratio analysis in the recycle condensate separator, and uncertainties in the carbon monoxide and heavy hydrocarbon (C_4) gas fractions resulted in considerable scatter in the calculated K-values. The inability to obtain representative samples, especially at the recycle condensate separator bottoms, seems to be the heart of this problem. As a solution, an efficient oil-water separator was designed and installed as part of the 1977 SRC II modification. Accurate flow metering and on-line gas chromatographic analysis of streams from this separator should greatly improve K-value measurements.

In the interim, a computer program was designed, based directly on the Grayson and Streed(6) correlation, and used with success, which indicates that petroleum hydrocarbon K-values are reasonable approximates for use at these flash conditions.

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(6) Grayson, H.G. and Streed, C.W., Proc. 6th World Petrol. Congress, Frankfurt/Mein, VII, paper 20, PD 7, 1963, ppg.233-245.

12. Comparison of Physical Properties During SRC-I and SRC-II

For liquid distillate streams, generalized colligative properties such as viscosity and density appear to be little different between SRC-I and SRC-II runs, as would be expected, since the cut points were the same in both. However, elemental analyses exhibit differences, as shown in Table 155, which compares oils produced just prior to and during SRC-II operation in July and August of 1975. The most notable differences shown in Table 155 are in the sulfur contents of the oils, especially light oil and wash solvent.

Feed slurries in SRC-II are more dense and more viscous than those in SRC-I and, as mentioned in section 5, are pseudoplastic. More data on this material will be found in subsequent reports.

Since only high ash SRC was produced in the plant during the 1975 SRC-II runs, changes in properties of SRC were observed by study of laboratory-produced vacuum bottoms. Laboratory vacuum bottoms properties have been shown to correlate closely with plant SRC when low ash SRC is produced . In Table 156, properties of vacuum bottoms from selected samples taken of the high pressure flash drum bottoms (sample point #204) are listed. It can be seen by inspection that vacuum bottoms from SRC-II are typically lower in sulfur, oxygen, and hydrogen-to-carbon atomic ratio than those from SRC-I. This suggests that the more severe hydrogenation conditions of SRC-II actually produce a more condensed residue.

TABLE 124

ANALYSIS OF KENTUCKY COAL* - PROCESSED AT TACOMA SRC PILOT PLANT DURING 1975

PROXIMATE ANALYSIS

	AS RECEIVED	DRY BASIS
% MOISTURE	1.78	----
% ASH	10.13	10.31
% VOLATILE	37.70	38.38
% FIXED CARBON	50.39	51.30
	100.00	100.00
HEAT OF COMBUSTION, BTU/#	12,700	12,929
% SULFUR	3.61	3.67
% TOTAL ALKALINITY AS Na_2O	----	0.24
SULFUR FORMS		
% PYRITIC S	2.05	2.09
% SULFATE S	0.18	0.18
% ORGANIC S	1.37	1.40
	3.60	3.67
WATER SOLUBLE ALKALIES		
% Na_2O	----	0.009
% K_2O	----	0.003

FUSION TEMPERATURE OF ASH	(REDUCING)	(OXIDIZING)
INITIAL DEFORMATION	1974°F	2177°F
SOFTENING (H=W)**	2145°F	2335°F
SOFTENING (H=1/2W)**	2231°F	2450°F
FLUID	2394°F	2522°F

**WHERE: W = CONE WIDTH
H = CONE HEIGHT

FREE SWELLING INDEX = 5 1/2

ULTIMATE ANALYSIS

	% WEIGHT AS RECEIVED	% WEIGHT DRY BASIS
MOISTURE	1.78	----
CARBON	70.54	71.82
HYDROGEN	4.93	5.02
NITROGEN	1.31	1.33
CHLORINE	0.04	.04
SULFUR	3.61	3.68
ASH	10.13	10.31
OXYGEN (DIFF.)	7.66	7.80
	100.00	100.00

MINERAL ANALYSIS	IGNITED BASIS
PHOS. PENTOXIDE, P_2O_5	0.22
SILICA, SiO_2	39.44
FERRIC OXIDE, F_2O_3	31.47
ALUMINA, Al_2O_3	20.71
TITANIA, TiO_3	0.90
LIME, CaO	2.13
MAGNESIA, MgO	0.69
SULFUR TRIOXIDE, SO_3	1.73
POTASSIUM OXIDE, K_2O	1.91
SODIUM OXIDE, Na_2O	0.40
UNDETERMINED	0.40
	100.00

SILICA VALUE = 49.98
T250 = 2235°F

* Not from composite samples. Averaged from 1975 samples tested by outside commercial laboratories.

TABLE 125

ANALYSIS OF KENTUCKY COAL* - PROCESSED AT TACOMA SRC PILOT PLANT DURING 1976

PROXIMATE ANALYSIS

	AS RECEIVED	DRY BASIS
% MOISTURE	2.30	------
% ASH	11.03	11.29
% VOLATILE	36.59	37.45
% FIXED CARBON	50.08	51.26
	100.00	100.00
HEAT OF COMBUSTION, BTU/#	12,430	12,722
% SULFUR	3.75	3.83
% TOTAL ALKALINITY AS Na_2O	----	0.19

SULFUR FORMS

	AS RECEIVED	DRY BASIS
% PYRITIC S	2.26	2.31
% SULFATE S	0.22	0.22
% ORGANIC S	1.27	1.30
	3.75	3.83

WATER SOLUBLE ALKALIES

	AS RECEIVED	DRY BASIS
% Na_2O	----	.002
% K_2O	----	.003

FREE SWELLING INDEX = 5.0

ULTIMATE ANALYSIS

	%WEIGHT AS RECEIVED	% WEIGHT DRY BASIS
MOISTURE	2.30	-----
CARBON	68.86	70.48
HYDROGEN	4.88	4.99
NITROGEN	1.19	1.22
CHLORINE	0.03	0.03
SULFUR	3.75	3.83
ASH	11.03	11.29
OXYGEN (DIFF.)	7.96	8.16
	100.00	100.00

MINERAL ANALYSIS

	IGNITED BASIS
PHOS. PENTOXIDE, P_2O_5	0.19
SILICA, SiO_2	41.08
FERRIC OXIDE, F_2O_3	29.25
ALUMINA, Al_2O_3	20.45
TITANIA, TiO_3	0.88
LIME, CaO	1.96
MAGNESIA, MgO	0.73
SULFUR TRIOXIDE, SO_3	2.40
POTASSIUM OXIDE, K_2O	2.00
SODIUM OXIDE, Na_2O	0.39
UNDETERMINED	0.67
	100.00

SILICA VALUE = 56.27
T250 2275

* Averaged from composite samples for period of 1/76 thru 7/76. Tested by outside commercial laboratories.

TABLE 126

SULFUR FORMS OF COMPOSITE FEED COAL SAMPLES, SRC PILOT PLANT

OBTAINED AT SAMPLE POINT 105

KENTUCKY COAL

DATE	MEAN VALUE % PYRITIC	% SULFATE	% ORGANIC	% TOTAL
1974				
May	1.99	0.05	1.29	3.33
1975				
March	1.60	0.06	1.90	3.56
April	1.72	0.10	1.59	3.41
July	2.10	0.10	1.70	3.90
August	2.30	0.18	1.46	3.94
September	2.37	0.19	1.57	4.13
October	2.58	0.27	1.39	4.24
November	2.18	0.09	1.71	4.18
December	1.70	0.21	1.65	3.56
1976				
January	1.98	0.11	1.42	3.51
February	2.48	0.15	1.73	4.36
March	2.06	0.10	1.96	4.12
April	2.27	0.46	1.42	4.15
May	1.82	0.42	1.40	3.64
June	1.76	0.14	1.46	3.36
July	2.24	0.26	1.38	3.88
August	2.50	0.30	1.25	4.06
September	2.24	0.27	1.59	4.10

TABLE 127

PETROGRAPHIC ANALYSIS

Composite Sample of Kentucky Coal - 1976

MACERAL	VOLUME PERCENT, MINERAL MATTER FREE
Vitrinoids	87.5
Exinoids	3.8
Semifusinoids	1.3
Fusinoids	1.7
Micrinoids	5.7
TOTAL	100.0

TABLE 128

BULK DENSITY AND TRUE DENSITY OF SOLIDS

	Bulk Density gm/cm^3	True Density gm/cm^3
RAW COAL	0.75	1.39
GROUND COAL	0.55	1.41
SRC		
At Sandvik Belt	0.53	----
In Storage Bin	0.61	----
Cast SRC in Railcar	0.90	----
High Ash SRC	----	1.36
Low Ash SRC	----	1.24
MINERAL RESIDUE	0.76	1.74

SPECIFIC GRAVITY vs TEMPERATURE FOR SRC LIQUIDS

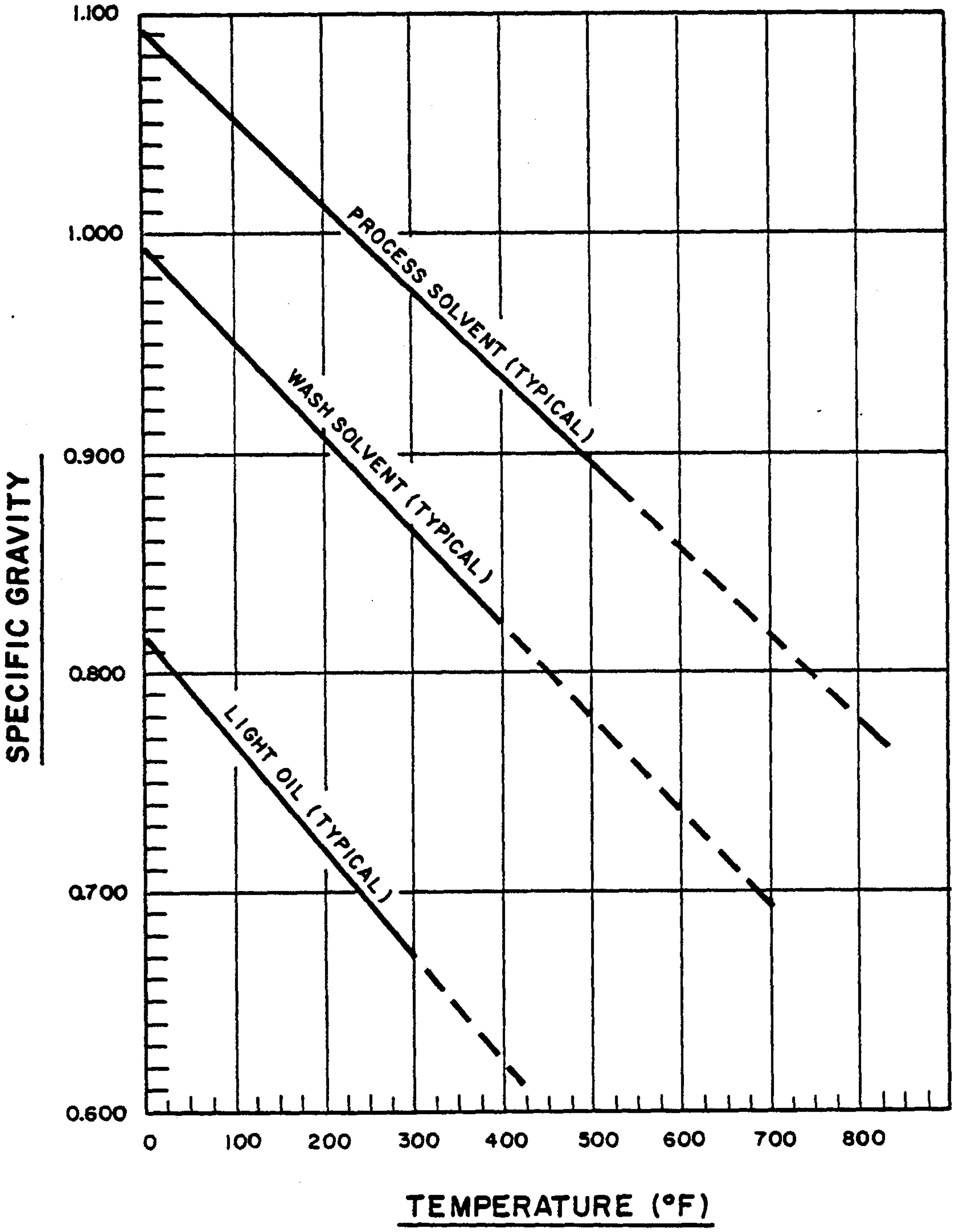


FIGURE 89

TABLE 129

SPECIFIC GRAVITY vs DISTILLATION FOR LIQUID STREAMS

SAMPLE	Light Oil	Wash Solvent	Process Solvent	Filter Feed (4.3% Ash)	Filtrate
Specific Gravity 60/60°F	0.846	0.976	1.056	1.158	1.101
ASTM Distillation	D86	D86	D1160*	D1160*	D1160*
Initial	120	253	-----	-----	-----
5%	151	311	495	515	433
10%	171	348	570	518	446
20%	219	368	532	551	476
30%	258	375	550	590	510
40%	300	384	573	628	548
50%	325	394	596	680	580
60%	348	403	618	758	619
70%	363	419	663	---	700
80%	380	450	702	---	840
90%	399	501	770	---	---
END	486	600	877	912	930
Recovery (Vol. %)	99	95	97	69.5	80

* Converted to atmospheric pressure using nomograph, Figure 94.

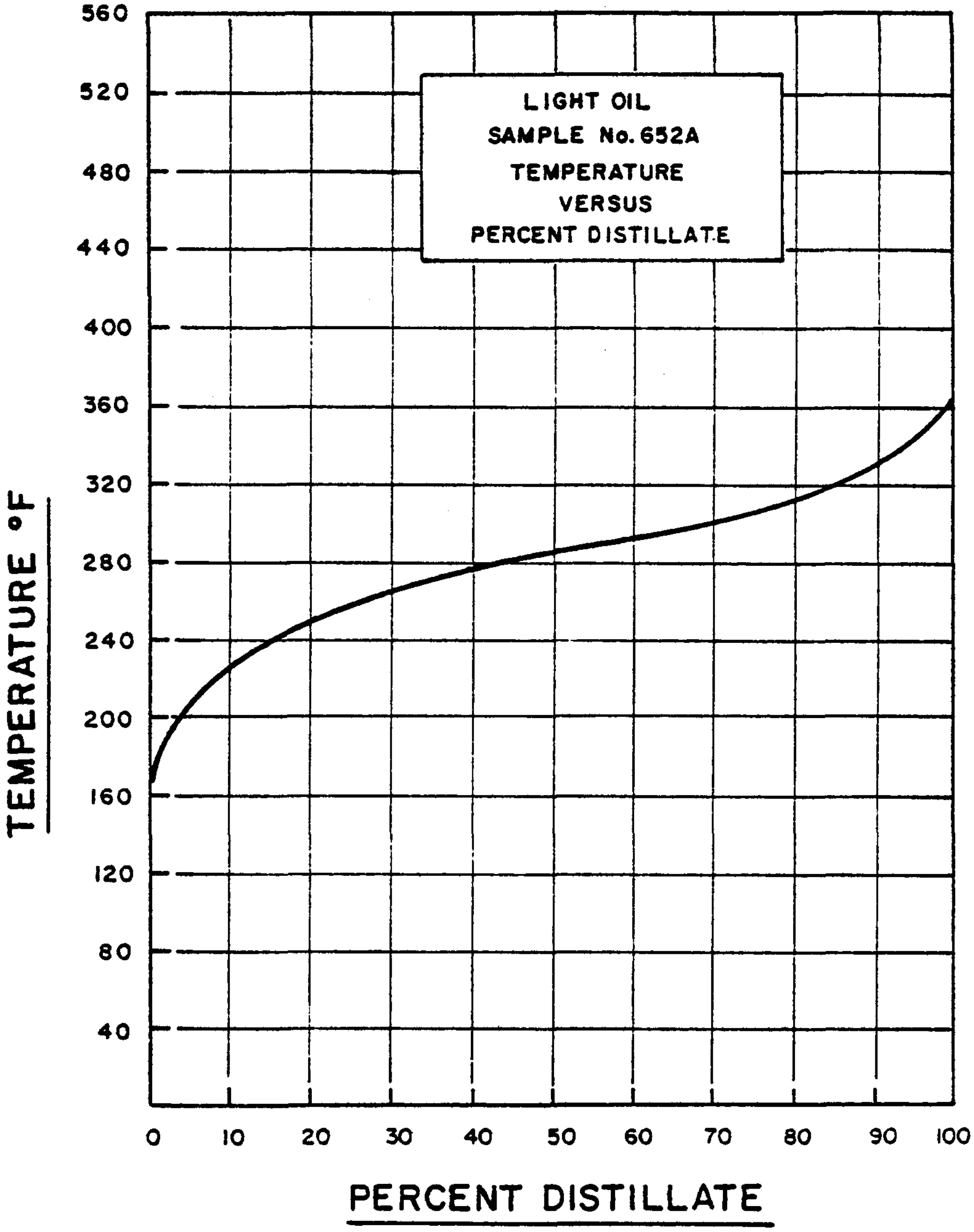

LIGHT OIL
SAMPLE No. 652A
TEMPERATURE
VERSUS
PERCENT DISTILLATE
TEMPERATURE °F
560
520
480
440
400
360
320
280
240
200
160
120
80
40
0
10
20
30
40
50
60
70
80
90
100
PERCENT DISTILLATE

FIGURE 90

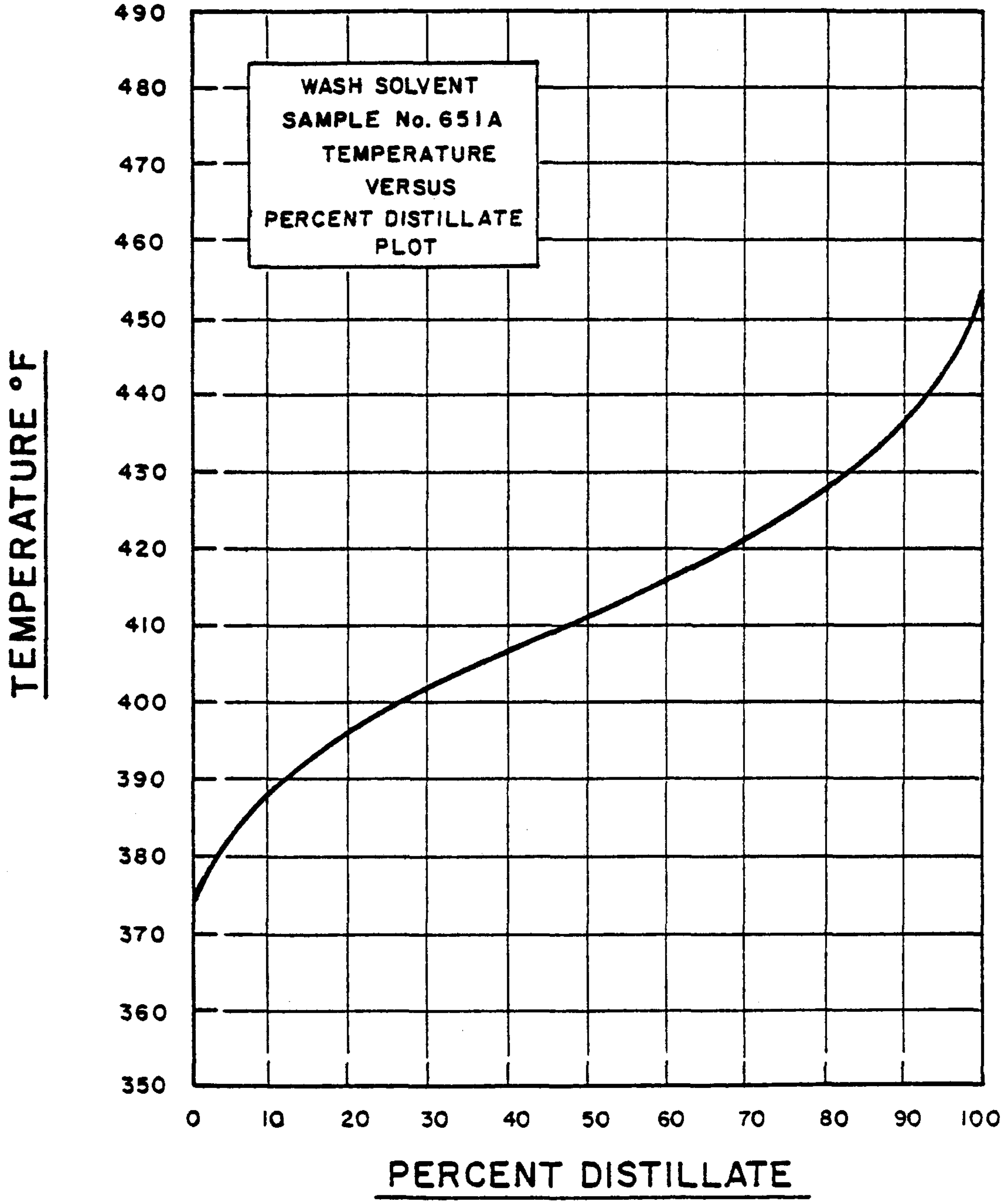
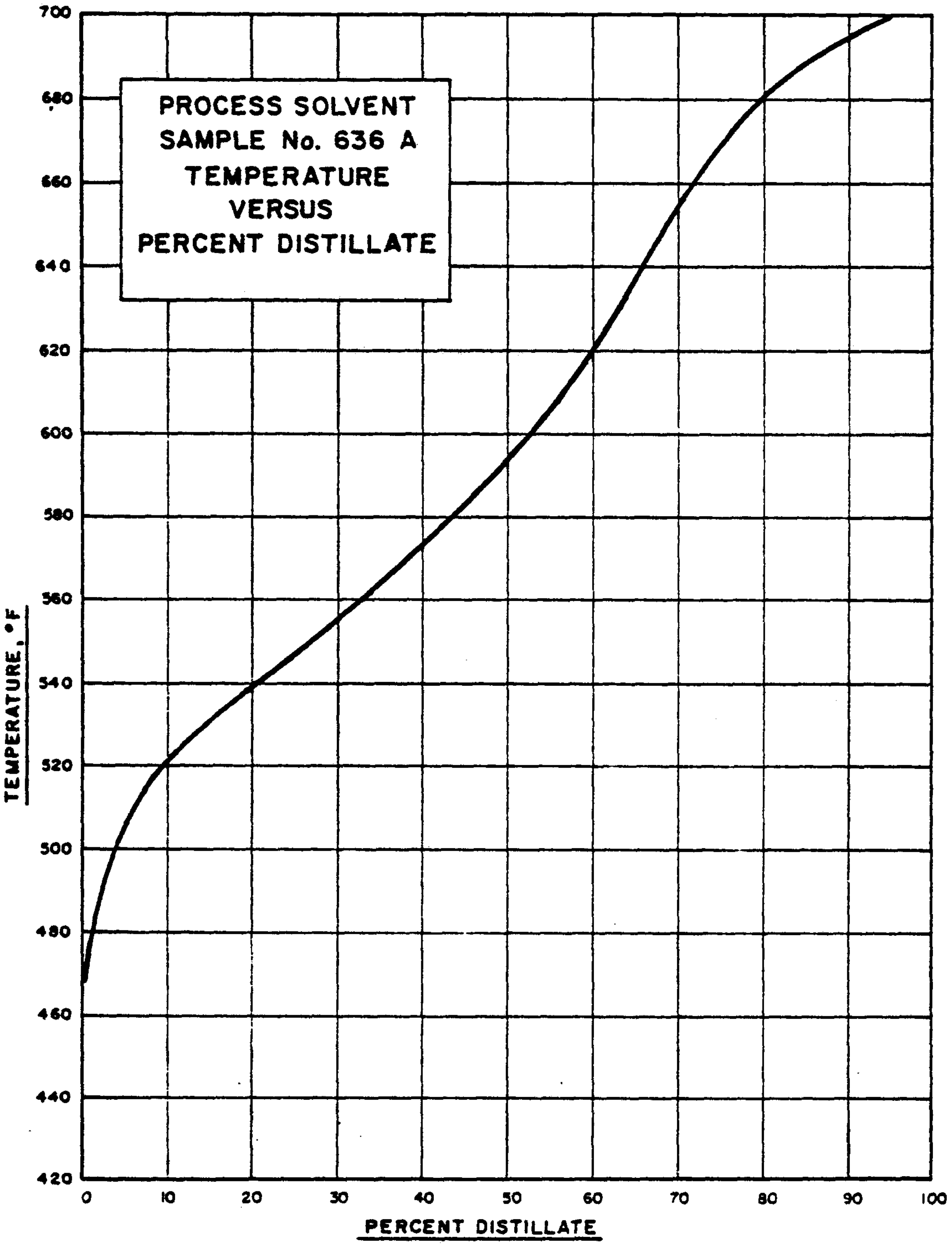

WASH SOLVENT
SAMPLE No. 651A
TEMPERATURE
VERSUS
PERCENT DISTILLATE
PLOT
TEMPERATURE °F
490
480
470
460
450
440
430
420
410
400
390
380
370
360
350
0
10
20
30
40
50
60
70
80
90
100
PERCENT DISTILLATE

FIGURE 91



ASTM D-1160 Distillation Converted To Atmospheric Pressure

FIGURE 92

INITIAL PLANT STARTUP SOLVENT

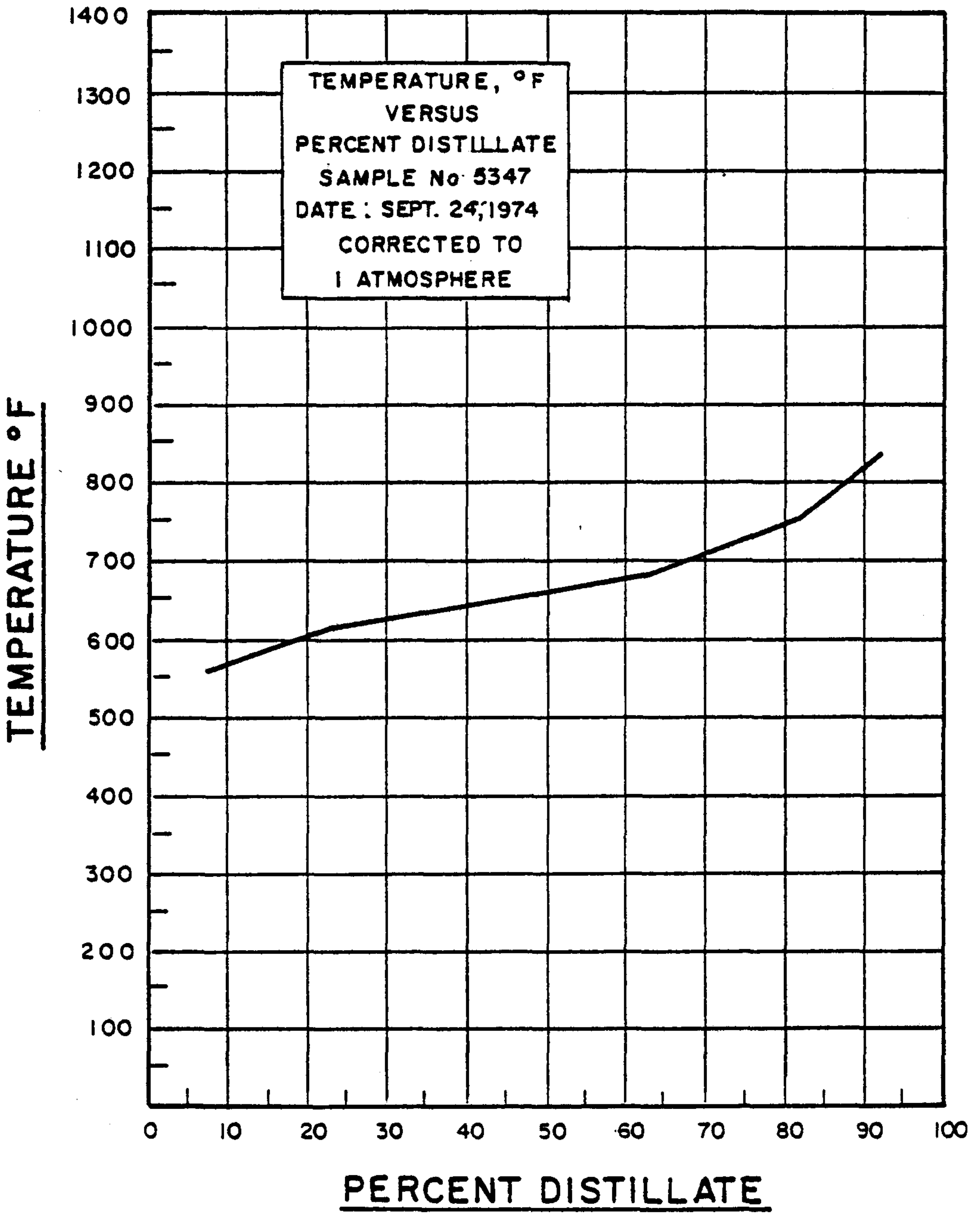


FIGURE 93

PROCESS SOLVENT

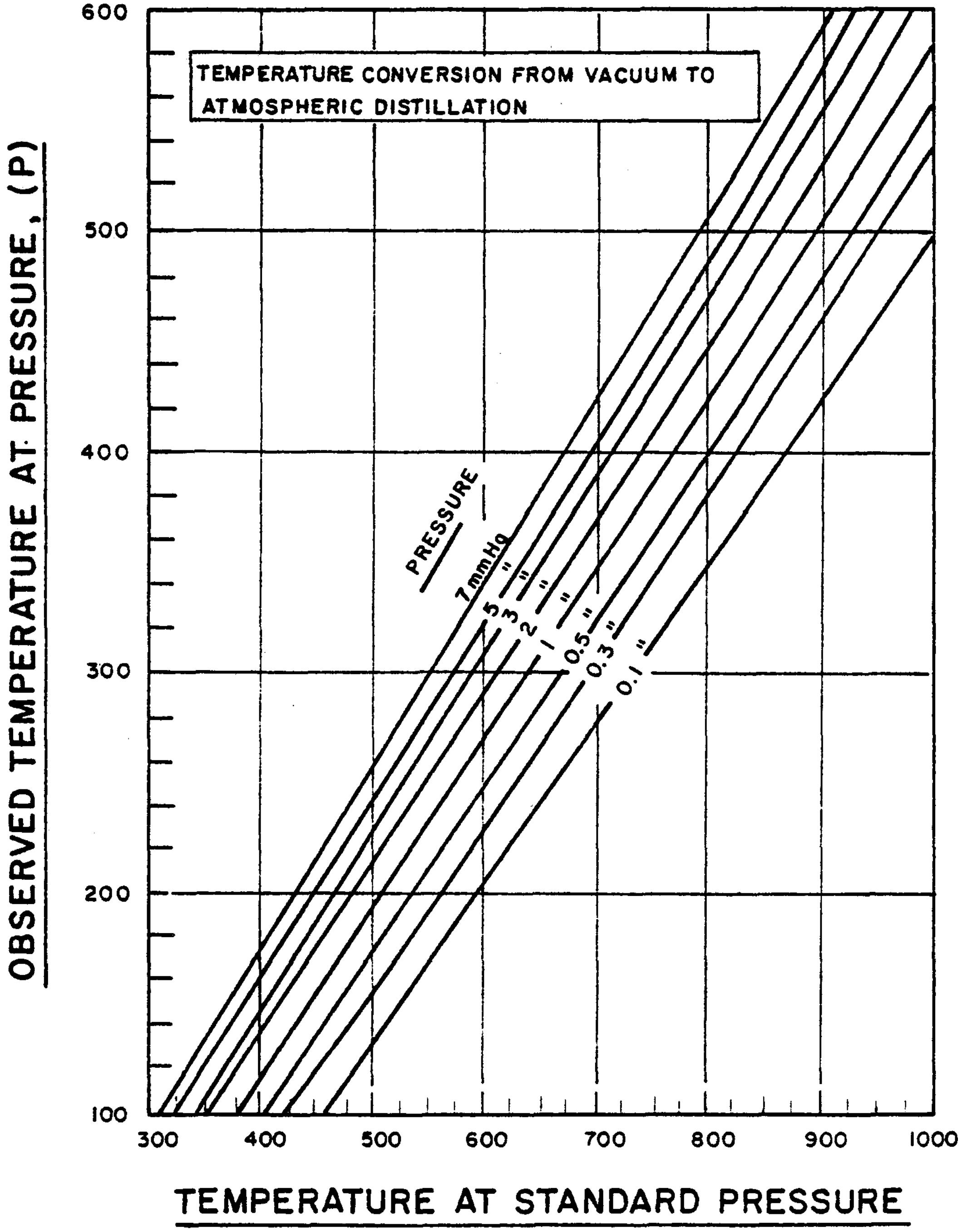


FIGURE 94

TABLE 130

SRC I MASS BALANCE RUNS 1-11

AVERAGE PRODUCT DISTILLATIONS

Light Oil	MBR #1	MBR #2	MBR #3	MBR #4	MBR #5	MBR #6	MBR #7	MBR #8	MBR #9	MBR #10	MBR #11	AVERAGE
Volume % Cuts												
IBP	184°F	161°F	151°F	162°F	152°F	150°F	137°F	149°F	138°F	119°F	136°F	150°F
5	222	187	189	193	182	201	192	205	186	146	168	189
10	324	259	222	237	216	233	220	227	212	172	190	228
20	340	292	261	276	276	262	246	256	244	202	218	261
30	345	312	286	290	296	283	260	269	263	220	249	279
40	351	326	299	302	311	296	272	285	280	239	268	294
50	356	341	310	309	324	311	288	299	293	250	285	306
60	360	347	325	318	335	323	290	308	307	264	298	316
70	366	355	328	323	345	332	309	320	319	282	313	327
80	372	363	337	331	355	343	320	331	331	304	330	338
90	384	375	349	344	373	359	337	364	347	345	351	357
95	397	390	369	358	395	377	353	368	351	372	363	372
EP	414	401	386	369	416	400	371	392	399	402	405	396
% Recovery	98	98	97	97	98	97	97	98	98	97	98	98
VABP	355	328	299	301	311	304	283	296	287	254	278	300
WABP	357	332	303	304	308	308	286	300	291	259	283	303
MeABP	353	323	292	296	296	298	277	288	279	242	267	292
CABP	354	326	296	298	301	303	281	293	284	251	273	296
Slope, °F/% = $\frac{90\%-10\%}{80}$	0.75	1.45	1.59	1.34	1.96	1.58	1.46	1.71	1.69	2.16	2.01	1.61
WATSON K	10.72	11.10	10.96	10.98	10.96	10.99	10.88	10.94	10.89	10.79	10.83	10.91

TABLE 131

SRC I MASS BALANCE RUNS 1-11

AVERAGE PRODUCT DISTILLATIONS

Wash Solvent	MRB #1	MBR #2	MBR #3	MBR #4	MBR #5	MBR #6	MBR #7	MBR #8	MBR #9	MBR #10	MRB #11	AVERAGE
Volume % Cuts												
IBP	360°F	383°F	369°F	353°F	367°F	327°F	369°F	367°F	378°F	339°F	362°F	361°F
5	268	402	382	376	394	393	387	389	388	374	374	384
10	382	408	391	384	398	397	391	393	393	384	384	391
20	408	413	396	391	403	400	398	397	397	392	392	399
30	412	417	401	396	408	406	403	403	401	397	397	404
40	417	421	405	400	411	408	408	407	405	403	402	408
50	421	425	409	405	416	411	413	411	409	407	407	412
60	425	430	413	409	421	415	418	416	413	413	412	417
70	430	436	419	415	426	420	424	422	418	420	418	423
80	439	444	427	424	434	425	431	430	425	429	425	430
90	450	455	439	437	447	436	443	441	437	442	438	442
95	460	463	453	453	460	446	453	453	449	458	454	455
EP	465	482	470	468	495	465	477	495	484	492	502	481
% Recovery	97	98	98	97	99	99	99	98	99	99	99	98
VAPB	419	428	412	407	419	414	415	414	412	410	409	414
WAPB	422	429	413	408	415	416	417	416	413	411	411	416
MeABP	416	426	410	405	411	412	415	412	410	408	406	412
CABP	417	427	411	406	413	413	414	413	411	409	408	413
Slope, °F/% = $\frac{90\%-10\%}{80}$	0.98	0.59	0.60	0.66	0.61	0.49	0.65	0.60	0.55	0.73	0.68	0.65
WATSON K	9.82	9.86	9.80	9.78	9.80	9.81	9.81	9.81	9.80	9.80	9.79	9.81

TABLE 132

SRC I MASS BALANCE RUNS 1-11

AVERAGE PRODUCT DISTILLATIONS

Process Solvent	MBR #1	MBR #2	MBR #3	MBR #4	MBR #5	MBR #6	MBR #7	MBR #8	MBR #9	MBR #10	MBR #11	AVERAGE
Volume % Cuts												
IBP	502°F	495°F	435°F	451°F	462°F	427°F	477°F	478°F	452°F	472°F	467°F	465°F
5	536	531	473	481	489	492	495	500	473	491	476	494
10	550	539	488	496	504	507	510	505	491	502	495	508
20	570	562	513	518	524	525	531	531	514	519	512	529
30	588	583	533	542	543	546	550	551	537	537	530	549
40	606	602	564	572	563	561	572	570	558	557	549	570
50	622	626	600	604	585	586	597	592	584	580	572	595
60	644	648	632	634	613	605	624	618	612	607	600	622
70	668	682	666	665	644	632	652	643	642	636	631	651
80	702	700	691	695	679	672	687	679	682	669	667	684
90	702	700	705	700	700	700	710	692	711	720	698	703
95	702	700	710	700	700	700	715	700	714	734	700	707
EP	702	705	716	705	702	702	721	702	716	744	700	710
% Recovery	80	78	85	86	87	88	88	89	93	90	89	87
VABP	626	608	598	601	595	594	604	597	593	595	585	600
WABP	629	611	603	606	597	596	608	601	597	600	590	603
MeABP	618	600	585	590	583	591	593	586	580	582	573	589
CABP	623	605	593	597	589	593	599	593	589	591	580	596
Slope, °F/% = $\frac{90\%-10\%}{80}$	1.90	2.01	2.71	2.55	2.45	2.41	2.50	2.34	2.75	2.73	2.54	2.44
WATSON K	9.56	9.51	9.47	9.48	9.46	9.48	9.49	9.47	9.45	9.46	9.43	9.48

TABLE 133

SLURRY RECYCLE RUNS 1-5

AVERAGE PRODUCT DISTILLATIONS

Light Oil	SR-1	SR-2	SR-3	SR-4	SR-5	Average
Volume % Cuts						
IBP	163°F	140°F	153°F	148°F	164°F	154°F
5	209	183	202	181	201	195
10	234	209	225	204	226	220
20	257	231	245	221	246	240
30	272	250	264	242	263	258
40	283	264	272	259	275	271
50	294	283	293	274	282	285
60	304	297	304	289	300	299
70	314	310	314	304	314	311
80	323	323	323	316	322	321
90	335	339	336	336	339	357
95	349	357	348	359	362	355
EP	376	392	371	404	374	383
% Recovery	98	98	99	98	98	98
VABP	290	278	286	272	285	282
WABP	293	282	289	276	288	286
MeABP	285	270	281	264	278	276
CABP	287	275	284	269	282	279
Slope, °F/% = $\frac{90\%-10\%}{80}$	1.26	1.63	1.39	1.65	1.41	1.47
WATSON K	10.92	10.85	10.90	10.82	10.89	10.88
Sp. Gr. 60/60°F	0.83	0.825	0.836	0.817	0.798	0.82
Pressure-Atmospheric						

Elementals

Carbon	83.69	83.25	83.33	84.19	84.12	83.72
Hydrogen	11.94	11.69	11.65	12.06	12.47	11.96
Nitrogen	0.34	0.41	0.44	0.30	0.47	0.41
Sulfur	0.40	0.34	0.23	0.20	0.22	0.28
Ash	----	----	----	----	----	----

Viscosity

VIS. 77°F cSt	1.03	0.96	1.02	----	----	1.00
VIS. 100°F cSt	0.84	0.77	0.86	0.78	0.75	0.79

TABLE 134

SLURRY RECYCLE RUNS 1-5

AVERAGE PRODUCT DISTILLATIONS

Wash Solvent	SR-1	SR-2	SR-3	SR-4	SR-5	Average
Volume % Cuts						
IBP	377°F	381°F	376°F	354°F	375°F	373°F
5	390	394	386	387	384	388
10	394	399	393	393	389	394
20	399	403	398	399	394	399
30	402	406	402	403	399	402
40	406	408	407	407	403	406
50	409	412	411	411	408	410
60	413	416	412	417	414	414
70	418	421	422	423	419	421
80	424	427	430	431	428	428
90	436	439	447	444	441	441
95	448	449	458	457	454	453
EP	485	476	486	488	485	484
% Recovery	99	99	99	99	98	99
VABP	412	415	415	415	411	414
WABP	413	416	416	416	412	415
MeABP	410	413	412	413	408	411
CABP	411	414	414	414	409	412
Slope, °F = $\frac{90\%-10\%}{80}$	0.53	0.50	0.68	0.64	0.65	0.60
WATSON K	9.80	9.81	9.81	9.81	9.79	9.80
Sp. Gr. 60/60°F	0.97	0.98	0.97	0.97	0.97	0.97
Pressure-Atmospheric						
Elementals						
Carbon	84.80	84.46	84.57	85.18	84.35	84.67
Hydrogen	8.87	8.85	8.83	8.88	8.72	8.83
Nitrogen	0.44	0.56	0.69	0.66	0.71	0.61
Sulfur	0.16	0.22	0.13	0.03	0.08	0.12
Ash	----	----	----	----	----	----
Viscosity						
VIS. 77°F cSt	3.74	3.72	4.18	3.86	3.65	3.83
VIS. 100°F cSt	2.68	2.64	3.03	2.70	2.75	2.76
VIS. 210°F cSt	----	----	1.07	----	----	1.07

TABLE 135

SLURRY RECYCLE RUNS 1-5

AVERAGE PRODUCT DISTILLATIONS

Process Solvent	SR-1	SR-2	SR-3	SR-4	SR-5	Average
Volume % Cuts						
IBP	461°F	475°F	480°F	486°F	481°F	477°F
5	483	494	509	501	508	499
10	497	508	523	507	522	511
20	522	526	542	524	542	531
30	542	545	561	548	560	551
40	562	562	580	568	580	570
50	583	586	602	598	601	594
60	615	609	620	622	627	619
70	649	637	652	647	655	648
80	681	671	684	682	688	681
90	700	700	710	700	700	702
95	700	700	720	700	700	704
EP	700	700	724	700	702	705
% Recovery	84	83	90	84	84	85
VABP	594	595	610	600	608	601
WABP	599	590	615	602	613	604
MeABP	582	583	600	592	598	591
CABP	589	591	606	595	605	597
Slope, °F/% = $\frac{90\%-10\%}{80}$	2.54	2.40	2.34	2.41	2.23	2.38
WATSON K =	9.46	9.46	9.51	9.49	9.51	9.49
Sp. Gr. 60/60°F	1.070	1.069	1.077	1.070	1.068	1.071
Elementals						
Carbon	89.11	89.07	88.89	89.42	89.01	89.1
Hydrogen	7.26	7.50	7.33	7.47	7.40	7.39
Nitrogen	0.91	0.91	1.05	1.02	1.05	0.99
Sulfur	9.37	0.45	0.38	0.32	0.30	0.36
Ash	----	----	----	----	----	----
Viscosity						
VIS. 100°F cSt	15.90	15.51	22.64	18.74	19.69	18.50
VIS. 210°F cSt	2.20	2.18	3.34	2.57	2.66	2.59

TABLE 136

PRODUCTION RUN 4/5/76 - 11/29/76

AVERAGE PRODUCT DISTILLATIONS

Volume % Distilled	Light Oil	Wash Solvent	Process Solvent
IBP	166°F	266°F	---°F
5	199	353	420
10	230	371	440
20	277	379	442
30	305	386	460
40	326	392	480
50	344	398	510
60	362	404	540
70	377	411	565
80	382	421	605
90	437	436	697
95	490	452	750
EP	506	523	780
% Recovery	98	99	97
VABP	339	400	534
WABP	345	402	540
MeABP	323	398	505
CABP	333	399	527
Slope, °F% = $\frac{90\%-10\%}{80}$	2.59	0.81	3.21
WATSON K	11.1	9.76	9.22

TABLE 137

PRODUCTION RUN (4/6/76 - 11/29/76)

AVERAGE DISTILLATIONS

	Filter Feed Surge Vessel	Vac. Flash Feed Acc.	LT. Ends Feed Tank	Hot Well	Recycle Proc. Water Tk.-Oil	"A" Vapor Surge Drum	"A" Comp. Scrubber Knockout D.	Dryer Cond. Drum	Recycle Condensate Sep.
Sample Point	339	401	412	422	303	307	318	326	206
Distillation	D-1160	D-1160	D-1160	D-86	D-86	D-86	D-86	D-86	W/W
mm Hg	0.52	1.08	1.23	Atm.	Atm.	Atm.	Atm.	Atm.	Atm.
IBP	---	---	---	155°F	154°F	280°F	232°F	203°F	TO 380°F 14.7%
5%	135°F	148°F	135°F	187	198	360	331	350	
10%	149	167	144	201	315	368	357	367	---
20%	174	201	164	219	398	385	377	381	TO 480°F 48.9%
30%	207	229	196	229	423	393	388	390	
40%	235	262	215	249	441	400	400	398	---
50%	265	297	252	264	455	408	410	404	TO E.P. 31.0%
60%	302	335	293	280	472	417	424	416	
70%	362	404	334	301	491	429	440	432	---
80%	---	---	413	327	524	446	460	456	---
90%	---	---	---	373	580	481	500	524	---
95%	---	---	---	410	---	516	549	530	---
EP	475	496	506	419	590	567	579	585	642
% Recovery	75	77	83	98	94	99	98	96	96.8 2.2% Residue

HEAT CAPACITY vs TEMPERATURE

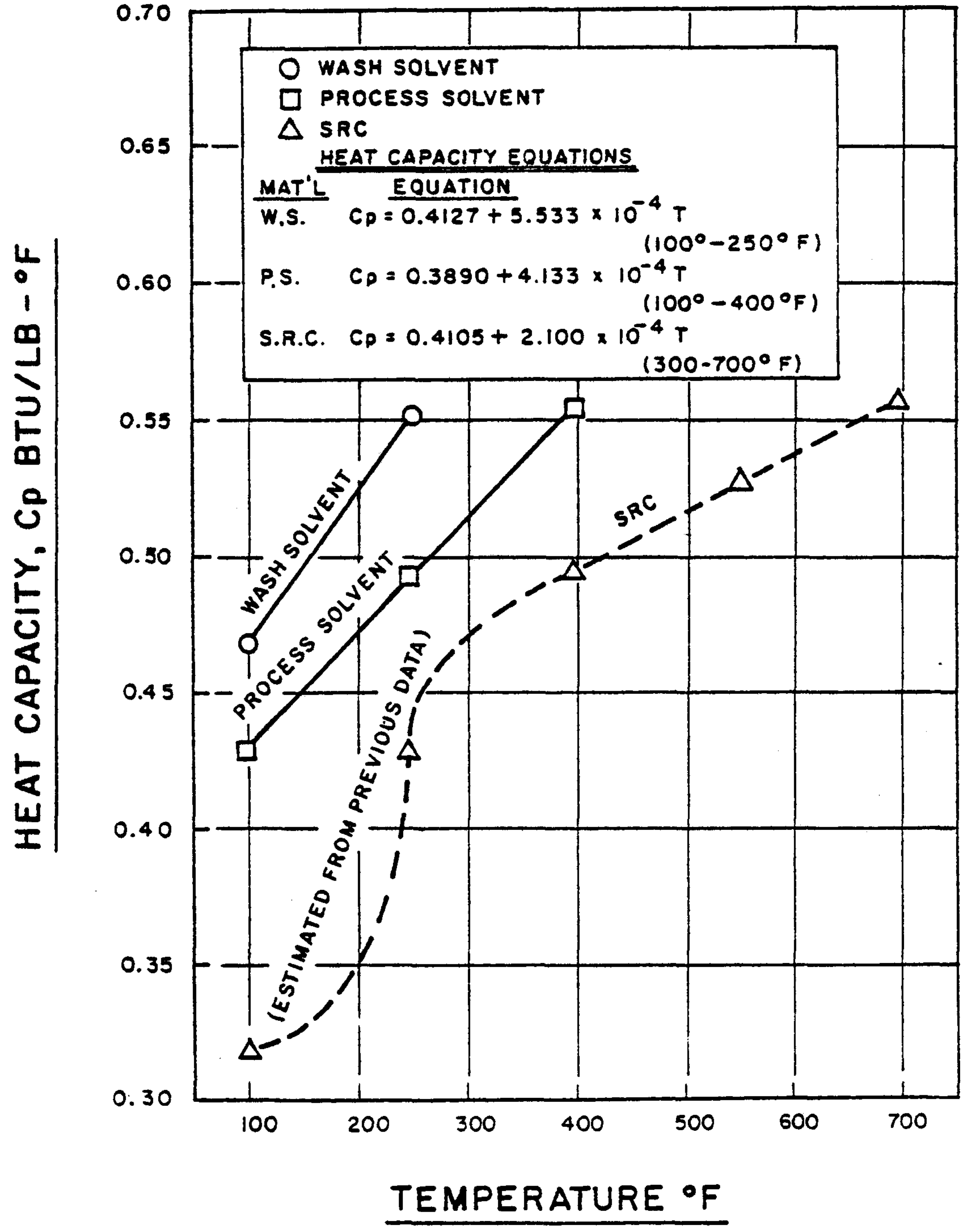


FIGURE 95

TABLE 138

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

Temp. °F	SRC	Process Solvent	Stripper Bottoms	Wash Solvent
100	0.319	0.430	--	0.468
250	0.429	0.493	--	0.551
400	0.494	0.554	--	--
450	--	--	0.566	--
550	0.527	--	--	--
600	--	--	--	--
700	0.557	--	--	--

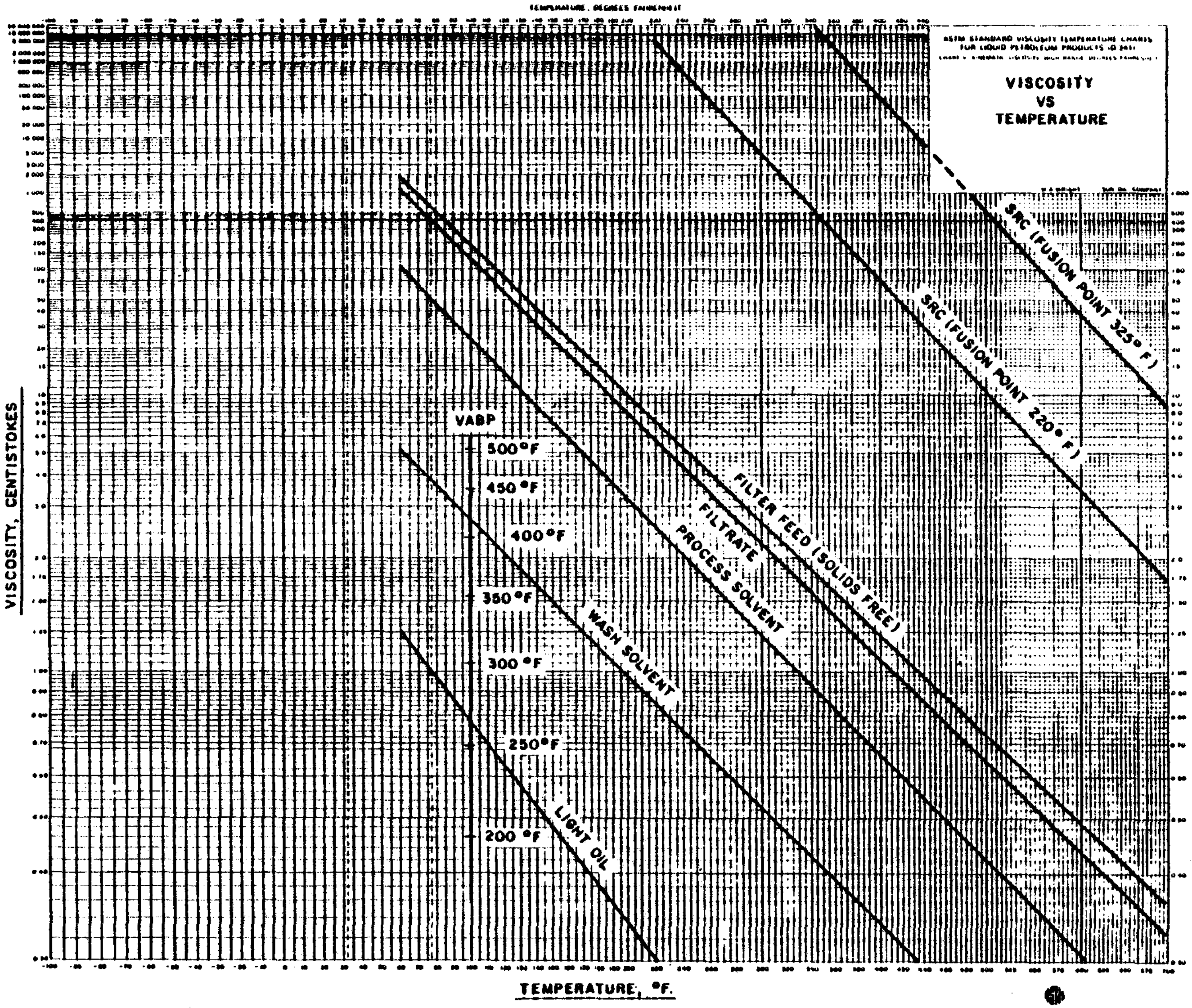

ASTM STANDARD VISCOSITY TEMPERATURE CHARTS
FOR LIQUID PETROLEUM PRODUCTS (D 341)
VISCOSITY
VS
TEMPERATURE
SRC (FUSION POINT 325° F)
SRC (FUSION POINT 220° F)
FILTER FEED (SOLIDS FREE)
FILTRATE
PROCESS SOLVENT
WASH SOLVENT
LIGHT OIL
VABP
500°F
450°F
400°F
350°F
300°F
250°F
200°F
VISCOSITY, CENTISTOKES
TEMPERATURE, °F.
KINEMATIC VISCOSITY, CENTISTOKES
TEMPERATURE, DEGREES FAHRENHEIT

FIGURE 96

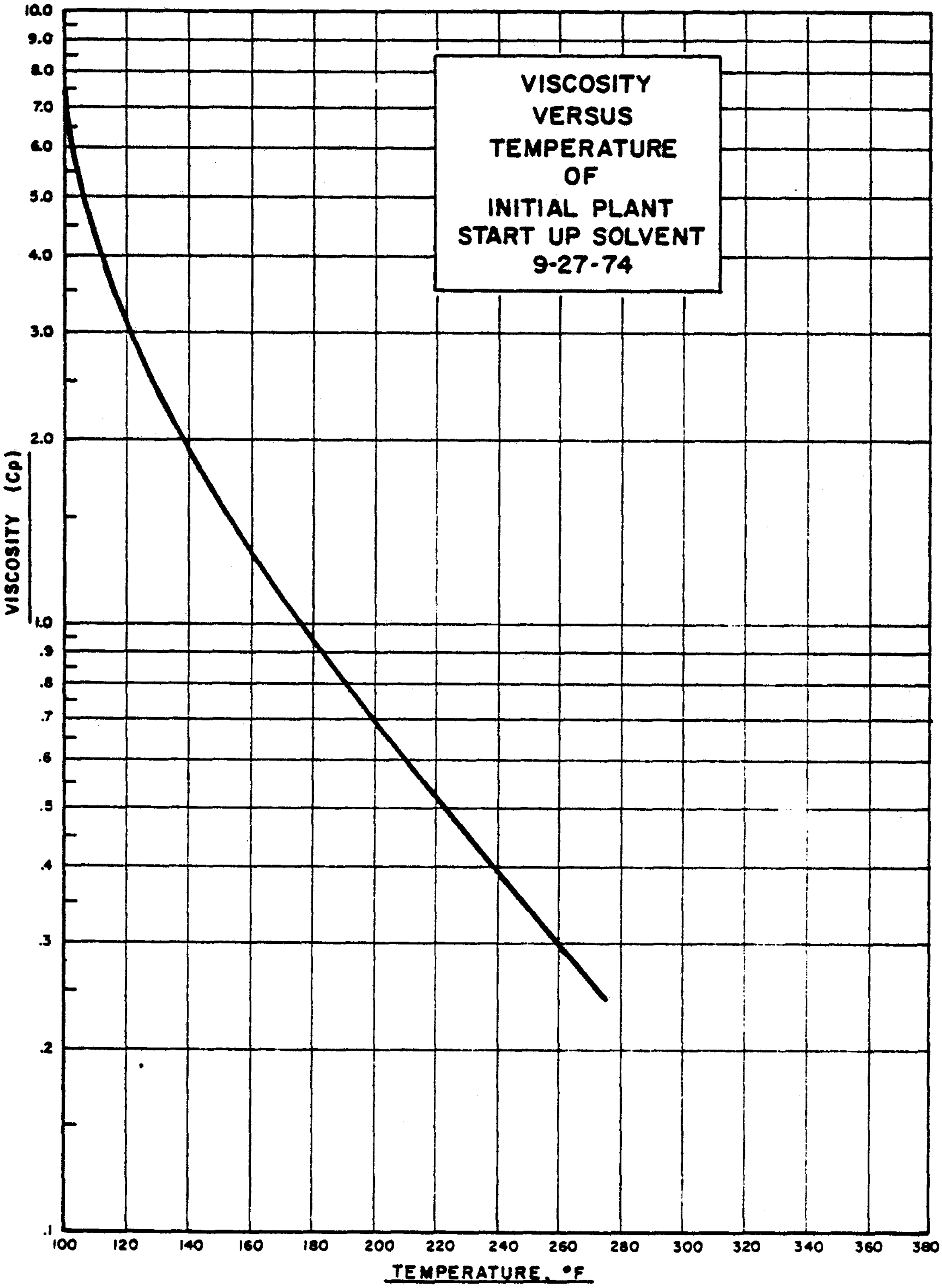

VISCOSITY
VERSUS
TEMPERATURE
OF
INITIAL PLANT
START UP SOLVENT
9-27-74
VISCOSITY (Cp)
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
.9
.8
.7
.6
.5
.4
.3
.2
.1
100
120
140
160
180
200
220
240
260
280
300
320
340
360
380
TEMPERATURE, °F

FIGURE 97

TABLE 139

CLOUD POINT AND POUR POINT OF SRC PLANT SOLVENTS

SOLVENTS	BOILING RANGE °F	CLOUD POINT °F	POUR POINT °F
Light Oil	Ambient to 380	- 84	- 95
Wash Solvent	380 to 480	2	- 30
Process Solvent	480 to 850	52	25

TABLE 140

SURFACE TENSION

Sample	Density (25°C) (gm/cm^3)	Surface Tension (25°C) (dynes/cm)
Process Solvent #12143 (3-10-75)	1.080	38.7
Wash Solvent #12144 (3-10-75)	0.970	27.8
Light Oil #12121 (3-9-75)	0.898	27.7

AVERAGE PARTICLE DIAMETER DISTRIBUTION

FOR FILTER FEED SAMPLED 10/2/76, 1720 HOURS (SAMPLE POINT No. 309)

COMPARISON OF COULTER AND SEM TECHNIQUES

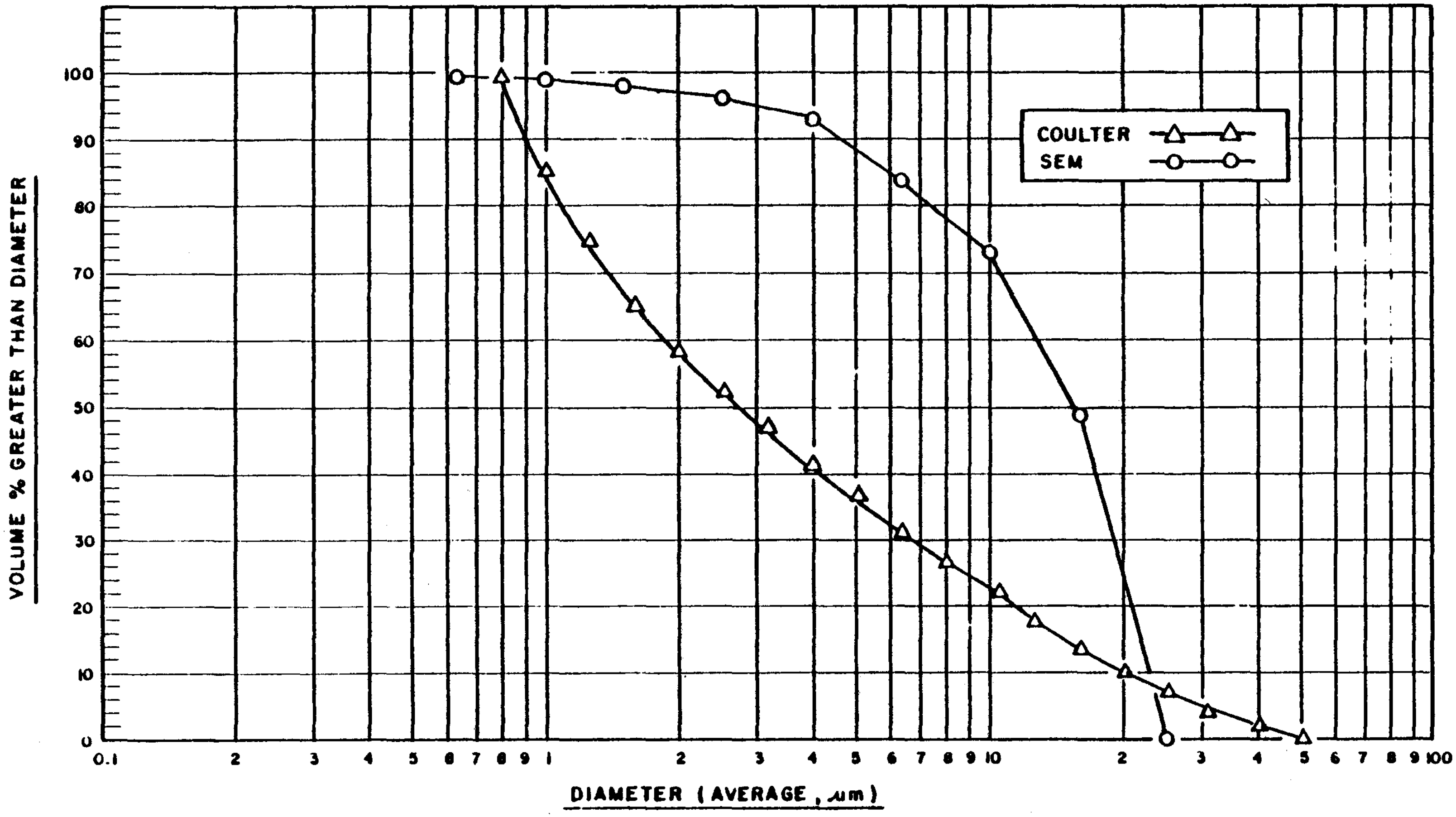


FIGURE 98

TABLE 141

COMPARISON OF PARTICLE SIZE ANALYSIS BY COULTER COUNTER[a] AND SCANNING ELECTRON MICROSCOPE[b] TECHNIQUES

Sample: Filter Feed (Sample Point 309), 10/2/76, 1720 hrs

Diameter	Number %		Volume %	
µm	Coulter	SEM	Coulter	SEM
< 0.10		1.23		< 0.01
0.10 - 0.16		8.10		< 0.01
0.16 - 0.25		19.17		0.01
0.25 - 0.4		23.96		0.06
0.4 - 0.63		19.51		0.18
0.63 - 1	60.48 (0.79 - 1 µm)	14.12	14.2 (0.79 - 1 µm)	0.51
1 - 1.6	32.80	7.85	20.3	1.15
1.6 - 2.5	5.19	3.22	12.8	1.85
2.5 - 4	1.21	1.31	11.0	3.01
4 - 6.3	0.25	0.98	10.0	8.89
6.3 - 10	0.06	0.30	9.0	10.72
10 - 16	0.01	0.17	8.7	24.87
16 - 25	< 0.01	0.08	6.8	48.75
25 - 40			5.0	
40 - 64			2.1	
> 64			0.1	
TOTAL	100.00	100.00	100.00	100.00

a. Analysis by Coulter Electronics, Inc., Fine Particle Lab
b. Analysis by Gulf Science and Technology Co.

SCANNING ELECTRON MICROGRAPHS OF REACTED COAL MINERAL MATTER

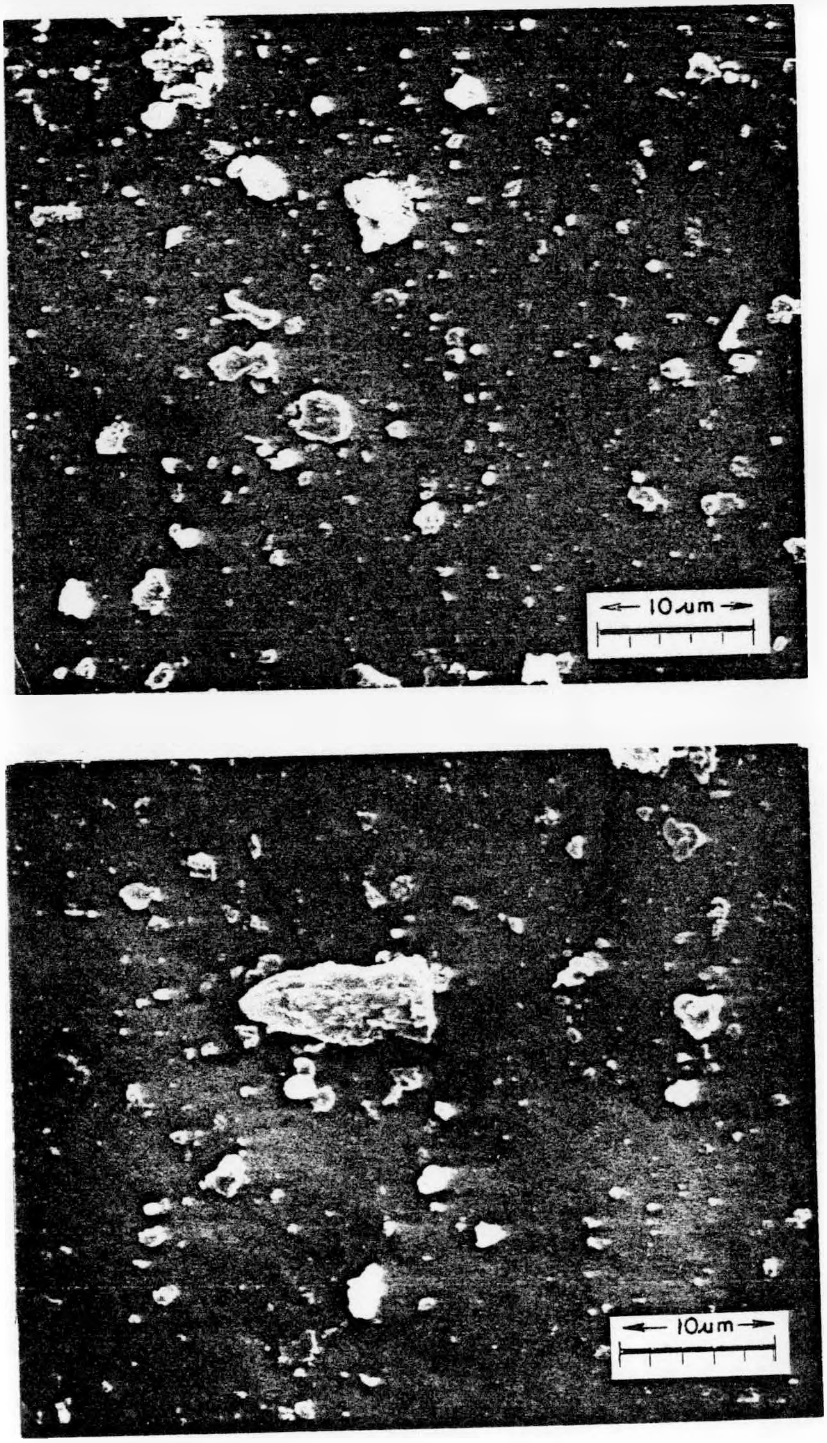


FIGURE 99

TABLE 142

ASTM D-86 DISTILLATION AND MOLECULAR WEIGHT DATA ON PLANT SOLVENTS

Sample Type	Light Oil	Light Oil	Light Oil	Wash Solvent	Wash Solvent	Wash Solvent	Process Solvent	Process Solvent
Date Sampled	1-27-76	8-15-75	2-15-76	7-9-75	4-4-75	2-15-76	4-2-75	2-21-76
Sample Number	Shipment #310	Shipment #318	Shipment #339	Shipment #126	Shipment #319	Shipment #341	Shipment #320	#27512
D-86								
Initial (°F)	122	148	125	376	371	368	501	488
5%	150	208	157	390	404	382	520	510
20%	203	258	202	400	412	392	557	530
40%	239	290	239	408	418	401	598	564
60%	268	315	275	417	424	412	641	634
80%	309	329	314	431	434	425	701	700
95%	359	366	368	459	459	461	---	---
End Point	375	397	392	475	476	468	701	700
Recovery	98	98	97	97	98	98	80	80
Mean Molecular Weight	109*	124*	107*	139*	147*	135*	211**	209**
Average Molecular Weight	113			140			210	

* Determined by freezing point depression (Beckman) using benzene.

** Determined by freezing point depression (Beckman) using phenol.

TABLE 143

ELEMENTAL COMPOSITION
(Samples: Nov., 1975 - Dec., 1976)

Wt. %	Raw Coal	Ground Coal	SRC	Mineral Residue (Dry)
Carbon	67.70	70.57	87.24	32.30
Hydrogen	5.09	5.09	5.77	1.89
Nitrogen	1.33	1.54	2.15	0.60
Sulfur	3.92	3.84	0.75	7.74
Oxygen	5.29	7.62	3.93	---
Ash	9.87	9.89	0.16	59.32
Water	6.80	1.45	---	---
Fusion Point	---	---	324	---
Pyridine Insolubles	---	---	---	86.96

	Light Oil	Wash Solvent	Process Solvent
Carbon	84.29	84.81	88.93
Hydrogen	11.06	8.65	7.28
Nitrogen	0.36	0.55	0.94
Sulfur	0.44	0.22	0.46
Oxygen	3.85	5.77	2.39

TABLE 144

MINERAL ANALYSIS OF SELECTED SOLIDS

Sample	Ash from Mineral Residue[1]	Ash from Solvent Refined Coal	Ash from Dissolver Coke
Date	10/14/76	4/20/76	9/14/76
Mineral Analysis			
Phosphorous Pentoxide, P_2O_5	0.18 wt %	0.07 wt %	0.22 wt %
Silica, SiO_2	47.38	13.69	35.85
Ferric Oxide, Fe_2O_3	24.22	28.42	27.79
Alumina, Al_2O_3	21.06	11.13	16.46
Titania, TiO_2	0.68	32.00	2.80
Manganese oxide Mn_4O_4	*	1.11	*
Zinc oxide, ZnO	*	0.50	*
Lime, CaO	1.50	5.14	4.27
Magnesia, MgO	0.60	0.73	0.60
Sulfur trioxide, SO_3	1.27	0.62	4.63
Potassium oxide, K_2O	2.08	0.53	1.02
Sodium oxide, Na_2O	0.77	0.63	0.49
Undetermined	0.26	5.43	0.87
Total	100.00	100.00	100.00
Total Ash Content of Sample	58 %	20 %	45 %

*Not determined

[1] Mineral Residue is contaminated with approximately 5% Filter aid.

TABLE 145

PROXIMATE CHARACTERIZATION OF OILS BY PREPARATIVE LIQUID CHROMATOGRAPHY

Sample (a)	SRC #373-R	Process Solv. #371-R	Process Solv. #427-R	Process Solv. #539-R	Wash Solv. #375-R	Wash Solv. #428-R	Wash Solv. #538-R
Date	3/7/76	3/7/76	5/18/76	8/27/76	3/7/76	5/18/76	8/27/76
All wt. % of sample							
Benzene Insolubles$^{(b)}$	39.73	8.22 Total	11.31 Total	2.90 Total	0.0	0.0	0.0
Asphaltenes$^{(b)}$	48.00				0.0	0.0	0.0
Maltenes$^{(b)}$	12.27	91.78	88.69	97.10	100.0	100.0	100.0
Resins$^{(b)}$	10.96	36.04	29.30	37.50	52.67	65.47	86.37
Very Weak Bases	0.80	4.70	7.13	4.30	(f)	(f)	(f)
Weak Bases	0.78	1.90	0.33	2.22	---	---	---
Strong Bases	2.74	8.23	6.09	12.00	---	---	---
Very Weak Acids I	0.12	5.07	9.13	7.95	---	---	---
Weak Acids I	0.09	9.78	2.29	2.39	---	---	---
Strong Acids I	2.41	1.91	1.35	1.58	---	---	---
Very Weak Acids II	0.18	0.12	0.19	3.00	---	---	---
Weak Acids II	0.37	0.24	0.17	0.32	---	---	---
Strong Acids II	2.89	2.04	2.10	1.72	---	---	---
Neutral Resins	0.58	2.06	0.52	2.02	---	---	---
Oils$^{(b)}$	1.31	55.74	59.39	59.60	47.33	34.53	13.63
Saturates$^{(c)}$	0.16	2.87	3.56	3.10	11.32	4.83	10.70
N-Paraffins$^{(d)}$		0.69	1.58	0.90	5.22	2.28	5.68
Cycloaklanes$^{(d)}$		1.22	1.88	1.69	5.89	2.49	4.86
Others$^{(d)}$		0.96	0.10	0.51	0.21	0.06	0.16
Aromatic$^{(c)}$	1.15	52.87*	55.83*	56.50*	36.01*	29.70*	2.93*
Mono -$^{(e)}$	0.06	5.25	0.88	3.13	---	---	---
Di -$^{(e)}$	0.07	19.74	18.67	9.35	---	---	---
Tri -$^{(e)}$	0.32	16.68	16.32	24.00	---	---	---
Polynuclear -$^{(e)}$	0.70	11.20	19.96	20.00	---	---	---

(a) Further analyses of these samples provided in Tables 146 through 149.
(b) Isolated and determined by "SARA" chromatographic analysis, D.M. Jewell, et. al., Ind. Eng. Chem. Fundam., Vol. 13, No. 3, 1974, ppg. 278-282.
(c) Isolated and determined by chromatography on silica gel.
(d) Determined by mass spectrometry.
(e) Determined by charge transfer chromatography.
(f) Unable to separate resin fraction further because of large concentrations of strongly absorbing substances.
* For mass spectrometric analysis of aromatic fractions, see Tables 147 and 148.

TABLE 146

CHARACTERIZATION OF ACID AND BASIC EXTRACTS OF WASH SOLVENT BY GAS CHROMATOGRAPHY*

Analysis By Gulf Science And Technology Co.

Sample	375-R	428-R	538-R
Date	3/7/76	5/18/76	8/27/76
Material extracted with NaOH	12.50 Wt.%	49.18 Wt.%	40.00 Wt.%
Phenol	3.70	6.88	5.66
o-Cresol	1.45	3.76	3.10
m-Cresol	2.42	8.13	6.38
p-Cresol	1.35	4.31	3.22
o-Ethyl cresol	0.29	1.21	0.94
2,3-Xylenol	0.02	0.44	0.39
2,4-Xylenol	0.41	2.21	1.53
2,5-Xylenol	0.36	2.19	1.74
2,6-Xylenol	0.09	0.75	0.51
3,5-Xylenol	0.35	3.09	2.36
p-Ethyl phenol	0.15	2.15	1.51
Isopropyl phenol	0.34	3.84	2.37
Unknown	1.55	10.22	10.29
Material extracted with HCl	6.49	3.26	4.01
Pyridine	0.01	trace	trace
2-Picoline	0.04	0.07	0.05
3-Picoline	0.08	0.06	0.21
Lutidines	0.29	0.37	0.26
Aniline	0.29	0.18	0.19
Toluidines	0.55	0.53	0.89
Xylidines	1.10	1.15	0.63
Quinoline	0.68	0.18	0.09
Unknown	3.45	0.72	1.79

*For further analyses of these samples, see Tables 145, 146 and 149.

TABLE 147

MASS SPECTROMETRIC AND NMR ANALYSIS OF AROMATIC HYDROCARBON FRACTION* OF PROCESS SOLVENTS

All values are wt % of Sample

Sample	371-R	427-R	539-R
Date	3/7/76	5/18/76	8/27/76
Mass Spectrometry			
Decahydrobenzchrysene	0.46	0.06	0.23
Octahydrobenzchrysene	0.48	0.06	0.34
Hexahydrobenzchrysene	0.37	0.06	0.28
Tetrahydrobenzchrysene	0.32	0.06	0.28
Benzochrysene	0.27	0.06	0.23
Decahydrobenzpyrene	0.53	0.17	0.45
Octahydrobenzpyrene	1.38	0.61	1.13
Hexahydrobenzpyrene	1.06	0.56	0.96
Tetrahydrobenzpyrene	0.32	0.17	0.34
Dihydrobenzpyrene	0.42	0.22	0.40
Benzopyrene	0.69	0.28	0.57
Dodecahydrochrysene	0.53	0.39	0.56
Octahydrochrysene	1.48	0.61	0.79
Hexahydrochrysene	0.48	0.11	0.23
Chrysene	0.27	0.17	0.28
Decahydropyrene	2.01	1.40	1.75
Hexahydropyrene	5.45	3.52	4.12
Tetrahydrofluoranthrene	1.48	0.89	1.19
Pyrene-Fluoranthrene	1.96	2.07	3.22
Dihydropyrene-Tetrahydrochrysene	4.29	3.07	4.35
Octahydrophenanthrene	0.27	1.56	1.41
Hexahydrophenanthrene	1.38	1.51	1.24
Tetrahydrophenanthrene	14.71	13.48	12.57
Phenanthrene	6.08	6.48	8.93
Fluorene-Dihydorphenanthrene	2.86	4.24	3.96
Acenaphthene-Biphenyl	0	4.13	1.64
Tetralins	0.43	1.12	0.40
Tetrahydroacenaphthene	0.52	0.84	0.40
Naphthalene	2.03	7.93	4.24
TOTAL	52.87	55.83	56.50
NMR			
Hydroaromatic Carbon	7.26	6.87	5.30
Transferrable Hydrogen	0.29	0.29	0.22

* For further analyses of these samples: See Tables 145 and 149

TABLE 148

MASS SPECTROMETRIC AND NMR ANALYSIS OF AROMATIC HYDROCARBON FRACTION* OF WASH SOLVENTS

All Values are wt% of Sample

Sample	375-R	428-R	538-R
Date	3/7/76	5/18/76	8/27/76
Mass Spectrometry			
Hexahydropyrenes	0.04	0.0	0.0
Dihydropyrenes	0.04	0.0	trace
Octahydroanthracenes	0.07	0.0	0.0
Tetrahydroanthracenes	0.14	0.0	0.01
Fluorenes/Dihydroanthracenes	0.07	0.06	0.01
Hexahydrophenanthrene	0.04	0.0	0.0
Phenanthrene	0.04	0.0	0.01
Acenaphthenes/Biphenyls	1.37	0.92	0.08
Tetrahydroacenaphthenes	1.22	0.89	0.12
Tetralins	16.85	18.89	1.32
Naphthalenes	13.04	8.70	1.19
Benzenes	3.09	0.24	0.19
TOTAL	36.01	29.70	2.93
NMR			
Hydroaromatic Carbon	14.50	13.10	12.82

*For further analyses of these samples: See Tables 145, 146 and 149.

TABLE 149

PROCESS CONTROL ANALYSES FOR SOLVENT SAMPLES
See Tables 145 through 148

Sample	SRC #373-R	Process Solv. #371-R	Process Solv. #427-R	Process Solv. #539-R	Wash Solv. #375-R	Wash Solv. #428-R	Wash Solv. #538-R
Date	3/7/76	3/7/76	5/18/76	8/27/76	3/7/76	5/18/76	8/27/76
Distillation		D1160*	D1160*	D1160*	D86	D86	D86
IBP		-	-	-	311	324	316
5% (Vol.)		495	501	495	355	363	360
10%		505	513	502	372	374	372
20%		513	529	520	386	387	383
30%		535	549	544	393	396	389
40%		556	575	570	400	399	393
50%		586	598	598	408	405	398
60%		619	632	626	414	411	404
70%		643	668	662	422	418	410
80%		708	720	720	432	425	418
90%		826	803	810	444	438	428
95%		-	890	-	454	450	438
EP		913	890	850	474	470	458
Recovery : %		94	95	94	99	99	99
Carbon : Wt %	87.41	88.15	88.55	88.88	84.45	84.83	84.29
Hydrogen : Wt %	5.89	7.66	7.65	7.74	9.09	9.11	9.69
Nitrogen : Wt %	2.11	0.99	0.98	0.93	0.55	0.47	0.55
Sulfur : Wt %	0.90	0.44	0.50	0.32	0.27	0.20	0.31
Oxygen, by difference : Wt %	3.55	2.76	2.32	2.13	5.64	5.39	5.16
Specific Gravity		1.067	1.070	1.073	0.963	0.966	0.969
% Dowtherm A Contaminant		4.8	3.16	4.35	-	-	-
Fusion Point : °F	331						
Ash : Wt %	0.14						

* Converted to atmospheric pressure

TABLE 150

ANALYSIS OF LIGHT OIL NO. 255. (11/10/75) BY CAPILLARY GAS CHROMATOGRAPHY

C_8 AND LIGHTER

Component	Vol.%
Isobutane	0.12
n-Butane	1.18
Isopentane	0.69
N-Pentane	1.95
Cyclopentane	1.17
2,3-Dimethylbutane	0.12
2-Methylpentane	0.88
3-Methylpentane	0.63
n-Hexane	2.28
Methylcyclopentane	2.81
2,2-Dimethylpentane	0.03
Benzene	0.28
2,4 Dimethylpentane	0.03
2,2,3-Trimethylbutane	0.41
Cyclohexane	2.62
3,3-Dimethylpentane	0.84
1,1-Dimethylcyclopentane	0.08
2-Methylhexane	0.40
2,3-Dimethylpentane	0.19
1,cis-3-Dimethylcyclopentane	0.44
3-Methylhexane	0.54
1,trans-3-Dimethylcyclopentane	0.40
1,trans-2-Dimethylcyclopentane	0.65
3-Ethylpentane	0.17
n-Heptane	2.09
1,cis-2-Dimethylcyclopentane	0.35
Methylcyclohexane	2.69
1,1,3-Trimethylcyclopentane	0.05
Ethylcyclopentane	1.22
2,5-Dimethylhexane	0.04
2,4-Dimethylhexane	0.02
2,2,3-Trimethylpentane	
1,trans-2,cis-4-Trimethylcyclopentane	0.16
Toluene	1.41
1,trans-2,cis-3-Trimethylcyclopentane	0.54
3,3-Dimethylhexane	0.01
2,3,4-Trimethylpentane	0.01
1,1,2-Trimethylcyclopentane	0.48
2,3,3-Trimethylpentane	0.01
2-Methyl-3-ethylpentane	0.15
2,3-Dimethylhexane	
1,cis-2,trans-4-Trimethylcyclopentane	0.04
1,cis-2,trans-3-Trimethylcyclopentane	0.04
2-Methylheptane	0.65
3-Methyl-3-ethylpentane	
3,4-Dimethylhexane	
4-Methylheptane	0.31
3-Ethylhexane	0.03
3-Methylheptane	0.38
1,cis-3-Dimethylcyclohexane	0.47
1,trans-4-Dimethylcyclohexane	
1-Methyl-trans-3-ethylcyclopentane	1.25
1-Methyl-trans-2-ethylcyclopentane	
1-Methyl-1-ethylcyclopentane	
1,cis-2,cis-3-Trimethylcyclopentane	0.23
1,trans-2-Dimethylcyclohexane	
1,trans-3-Dimethylcyclohexane	0.26
1,cis-4-Dimethylcyclohexane	
n-Octane	1.96
1-Methyl-cis-2-ethylcyclopentane	0.16
1-cis-2-Dimethylcyclohexane	0.09
Ethylcyclohexane	1.39
Ethylbenzene	1.01
1,4-Dimethylbenzene (para-Xylene)	0.37
1,3-Dimethylbenzene (meta-Xylene)	0.89
1,2-Dimethylbenzene (ortha-Xylene)	0.75
TOTAL	38.43

PHENOLS

Component	Wt. %
Phenol	9.85
O-Cresol	0.54
P-Cresol	0.52
M-Cresol	1.40
O-Ethyl Phenol	0.02
2,4 Dimethyl Phenol	0.03
2,5 Dimethyl Phenol	0.03
2,3 Dimethyl Phenol	0.03
P-Ethyl Phenol	0.03
2,3,6 Trimethyl Phenol	0.06
3,5 Dimethyl Phenol	0.01
TOTAL	12.52

AROMATICS

Component	Vol.%
Benzene	0.24
Toluene	1.65
Ethyl benzene	0.49
P-Xylene	0.39
M-Xylene	0.77
O-Xylene	0.64
Isopropylbenzene	0.06
n-Propylbenzene	0.18
1-Methyl-4-ethylbenzene	0.23
1-Methyl-3-ethylbenzene	0.63
1,3,5 Trimethylbenzene	0.13
Isobytylbenzene	0.27
Sec-Butylbenzene	0.02
1-Methyl-3-isopropylbenzene	0.51
1-2,4-Trimethylbenzene	
1-Methyl-2-ethylbenzene	
1-Methyl-2-isopropylbenzene	0.07
α Methyl Styrene	
Neo-Pentylbenzene	0.01
1,2,3-Trimethylbenzene	0.10
1,3-Diethylbenzene	
1-Methyl-3-n-propylbenzene	
1-Methyl-4-isopropylbenzene	
1-Methyl-4-n-propylbenzene	0.27
n-Butylbenzene	
1,4-Diethylbenzene	0.10
1-Ethylpropylbenzene	
1,2-Diethylbenzene	0.09
1-Methyl-2-n-propylbenzene	
Indane	0.58
1-Methyl-4-tert-butylbenzene	0.13
1,3-Dimethyl-4-ethylbenzene	0.08
(2-Methylbutyl) benzene	0.11
Indene	0.44
1,2,3,4-Tetramethylbenzene	0.67
Naphthalene	0.74
TOTAL	9.50

TABLE 151

OTHER ANALYSES OF LIGHT OIL NO. 255 (11/10/75)

See Table 150

Carbon		Wt. %	84.61
Hydrogen		Wt. %	12.30
Oxygen	Neutron Activation	Wt. %	2.23
Nitrogen	G-811	Wt. %	0.27
Sulfur	D-2622	Wt. %	0.55
Mercaptan Sulfur	GRM 1154	Wt. %	0.098
Hydrogen Sulfide	GRM 681	Wt. %	0.040
Carbonyl Sulfide	G-1527	Wt. %	0.008
Basic Nitrogen	GRM 1152	Wt. %	0.188
Water	Gas Chromatography	Wt. %	0.01
Organic Carbonyls	Infrared Spectroscopy	Wt. % CO	0.70
Saturates	ASTM D-1319	Vol.%	64.0
Olefins	ASTM D-1319	Vol.%	9.5
Aromatics	ASTM D-1319	Vol.%	26.5

TABLE 152

ANALYSIS OF LIGHT OIL NO. 31 (4/16/75) BY MASS SPECTROMETRY

Fraction Type	Group Type		Wt % of Sample
Aromatics			
	Benzopyrenes		0.0
	Chrysenes		0.0
	Decahydropyrenes		0.0
	Hexahydropyrenes		0.0
	Hexahydrofluoranthrenes		0.0
	Pyrenes/Fluoranthrenes		0.1
	Dihydropyrenes		0.1
	Octahydroanthracenes		0.0
	Hexahydrophenanthrene		0.0
	Tetrahydroanthracenes		0.8
	Phenanthrenes		0.5
	Fluorenes/Dihydroanthracenes		0.4
	Acenaphthenes/Biphenyls		0.4
	Tetralins		15.6
	Tetrahydroacenaphthenes		1.1
	Naphthalenes		8.6
	Benzenes		12.1
	Naphthol		0.6
	Dihydroxybenzenes		1.2
	Phenol		17.4
		TOTAL	58.7
Saturates			
	Paraffins		11.0
	Non condensed cycloparaffins		18.9
	Condensed dicycloparaffins		1.5
	Condensed tricycloparaffins		0.3
	Alkylbenzenes		0.2
		TOTAL	31.9

TABLE 153

ANALYSIS FOR FUEL VALUE OF LIGHT OIL NO. 31 4/16/76

See Table 152

	Result
Gravity, °API	27.0
Carbon, Wt., %	83.27
Hydrogen, Wt., %	10.54
Oxygen, Wt., %	5.0
Nitrogen, Wt., %	0.53
Sulfur, Wt., %	0.45
Hydrocarbon Analysis, FIA, Vol., %	
Aromatics	57.0
Olefins	3.0
Saturates	40.00
Total Acid No.	0.49
Total Base No.	5.2
Aniline Point, °C	15.0
Bromine Number	63.0
Ash Oxide, Wt., %	0.0003
Copper Strip, 3 Hrs. @ 122°F	4.0
Copper Dish Gum, mg./100 ml.	15,600
Existent Gum, mg/100 ml.	935
Oxidation Stability, D 525, min.	1,440[a]
Insoluble in pentane, Wt., %	0.03
Insoluble in naphtha, Wt., %	0.05
Vapor Pressure, PSI	2.40
Distillation D86, 760 MM Hg	
10% Distilled at, °F	208
20	278
30	331
40	343
50	363
60	376
70	388
80	405
90	432
95	496
End Point	581
Recovered, %	96.8
Residue, %	1.2
Loss, %	2.0

[a] 40 Lb. drop in pressure in 24 hours.

TABLE 154
MEAN EXPERIMENTAL K-VALUES

	RECYCLE CONDENSATE SEPARATOR (100°F)			HIGH PRESSURE FLASH (600°F)		
	1025 PSIG	1250 PSIG	1500 PSIG	1025 PSIG	1250 PSIG	1500 PSIG
H_2	50.11	71.83	64.85	64.83	34.28	18.40
N_2	0.82	5.07	4.64	42.71	18.96	35.26
CH_4	27.04	12.12	10.53	44.24	29.89	15.43
CO	38.95	33.25	15.89	15.96	162.33	163.61
C_2H_6	7.70	3.32	2.93	79.80	35.46	22.59
CO_2	-----	6.07	13.13	51.76	16.44	10.14
C_3H_8	2.99	1.12	0.94	130.37	31.79	21.01
iC_4H_{10}	0.23	0.40	0.19	-----	51.43	19.01
nC_4H_{10}	1.01	0.66	0.51	30.46	48.15	28.57
H_2S	1.55	1.06	1.16	46.17	88.08	18.19
H_2O				0.60	1.03	1.03
Light Oil				4.04	1.68	1.24
Wash Solvent				0.30	0.27	0.21
Process Solvent				0.10	0.07	0.07
Vaccum Bottoms				0.01	0.01	0.01

TABLE 155

ELEMENTAL ANALYSIS OF TYPICAL PLANT SOLVENTS PRODUCED IN SRC-1 AND SRC-II

	Light Oil		Wash Solvent		Process Solvent	
	SRC-I	SRC-II	SRC-I	SRC-II	SRC-I	SRC-II
% Carbon	83.1	84.0	84.9	85.0	88.4	88.5
% Hydrogen	11.1	11.5	9.0	8.7	7.4	7.4
% Nitrogen	0.4	0.4	0.5	0.6	0.9	1.0
% Sulfur	0.5	0.2	0.3	0.1	0.5	0.4
% Oxygen (by difference)	4.9	3.9	5.3	5.6	2.8	2.6

TABLE 156

COMPARISON OF SRC-I AND SRC-II LABORATORY VACUUM BOTTOMS

Vacuum Bottoms from 204 #	Date Sampled	Time Sampled	Mode of Operation	% C	% H	% N	% S	% O by diff.	% Ash	Fusion Point (°F)	H/C Atomic Ratio	BTU/lb.	Blackness*	% Benzene Insoluble ASTM D-2317	Remarks
10980	3/ 1/75	2330	SRC-I	87.47	5.48	2.18	0.52	4.07	0.28	390	0.747	15,825	57.9	29.42	Plant produced SRC
10353	3/ 8/75	2200	SRC-I	86.81	5.74	2.22	0.87	5.46	0.08	355	0.788	15,885	----	-----	Material Bal. Run #1
10373	4/10/75	2330	SRC-I	87.45	5.74	2.27	0.56	3.85	0.13	275	0.771	16,005	40.7	21.02	MBR #2
17619	6/10/75	1700	SRC-I	88.73	5.12	2.16	0.63	3.25	0.11	430	0.688	15,855	78.3	54.02	MBR #3
17371	6/13/75	2020	SRC-I	86.91	5.64	2.13	0.80	4.72	0.17	375	0.773	15,805	54.0	36.40	MBR #4
19405	6/26/75	1800	SRC-I	86.98	5.43	2.14	0.82	4.48	0.15	420	0.744	15,805	65.4	45.17	MBR #5
19386	7/ 1/75	1630	SRC-I	87.03	5.36	2.26	0.69	4.57	0.07	365	0.734	15,705	57.9	-----	MBR #6
19361	7/ 6/75	0230	SRC-I	86.84	5.41	2.25	0.66	4.64	0.20	430	0.742	15,655	68.2	42.17	MBR #7
18	7/13/75	0830	SRC-I	86.65	5.60	2.14	0.65	4.86	0.10	360	0.770	15,725	51.3	-----	MBR #8
20259	7/17/75	0001	SRC-I	87.22	5.34	2.33	0.73	4.21	0.17	400	0.730	15,700	62.9	-----	MBR #9
24190	6/16/76	1600	SRC-II	89.07	5.26	2.37	0.40	2.37	0.06	300	0.704	15,050	59.6	24.07	Full Dissolver
24136	8/17/75	1000	SRC-II	89.81	5.57	2.22	0.36	1.98	0.06	310	0.739	16,380	57.0	20.84	Full Dissolver
24141	8/18/75	1400	SRC-II	89.88	5.37	2.30	0.39	1.79	0.27	310	0.712	16,415	61.9	23.45	Full Dissolver
23390	8/21/75	1800	SRC-II	90.54	5.17	2.33	0.31	1.59	0.06	335	0.680	16,265	70.2	28.73	Half Dissolver
24031	8/23/75	1800	SRC-II	89.24	5.25	2.13	0.37	2.98	0.03	310	0.701	16,125	58.2	28.22	Half Dissolver
24111	8/26/75	1800	SRC-II	89.99	5.59	2.21	0.40	2.21	0.04	295	0.740	16,380	49.3	16.83	Half Dissolver

* Energy Research & Development Adm. R&D Report No. 53, Contract E (49-18)-496. Interim Report No. 7, September 1975-Volume II, Part 2, pg. 62.

G. Data Acquisition System

1. System Development

Development of the plant data acquisition system began early in 1974, following vendor selection of The Foxboro Company's Fox 2/30 computer system. The system included 24K words of core memory for program processing, 980K words of drum storage for fast memory access, analog to digital converters to process 288 plant process variables, a high speed paper tape reader and punch for off-line storage, two KSR35 teletypes and an IBM Selectric typer for reporting data.

The first priority in system development was programming of the computer to handle the basic data logging and to service the 3 on-line process chromatographs.

It was decided that the hourly average of each process variable would be saved in computer memory. In addition, the high and low value observed each hour, the standard deviation and the calculated average flowrate for each flowmeter was saved. The molecular weight and average composition for the 12 process chromatograph streams were also stored in the computer.

Logic flow charts and Fortran coding forms were prepared prior to arrival of the computer in April, 1974. Several programs were written and debugged to perform needed utility functions, such as zeroing data files, taking points on and off scan, setting the current date and resetting failure bits when peripheral devices were repaired. The "IMPAC Data Base", a system of Foxboro programs, was prepared and entered into the library.

During July and August, the computer-process interface was checked by imposing signals on the field mounted instruments and reading these through the computer. This procedure revealed some problems, and the appropriate changes were made. When the basic data gathering program, which was to run once every 30 seconds, was first written using the Foxboro library routings, the program required 25 seconds of execution time. An alternate approach was developed to read the values directly from the measurement table (scaled 0-4000 digital counts) and to then convert this data to engineering units with a user range file. The resulting system ran in less than one second.

A meter coefficient program was completed and used for the calculation of 55 flows each hour using average meter differential pressure, temperature, pressure and physical property data. A series of 16 programs to scan and record chromatographs came on line. An hourly operating log of average values of all measurements made during the previous hour, high and low readings observed, and standard deviation values for all points was placed in service. Also included was each hour's averaged chromatographic data. This log was 19 pages long and revolved around a large paper rack printing one line of data each hour on each page. Operators changed the paper loop each day at midnight, thus producing a daily 24 hour log of data.

The next set of programs installed provided maintenance and operations assistance. A program designed to produce a "scan" or trend log of up to 6 operating parameters at an operator specified scan rate was installed in September, 1974. A companion to this program was the "alarm" program, which monitored 25 process variables and printed an alarm message upon detection of a measurement which deviated from a specified high or low limit. A program called "Interspec Tester" was written to provide instrument maintenance personnel with a tool for obtaining the current value of any loop in the computer system. A companion program was later provided to operations personnel for obtaining a current process value by entering the loop number desired.

In the fall of 1974, attention turned mainly to programs that would assist in calculating material balances. It was first hoped that an hour by hour overall plant material balance and area balance could be obtained; however, process fluctuations made this impossible. Therefore, a program was completed to allow data to be averaged over longer periods of time and punched on paper tape for permanent storage. Another program was written to allow data produced in the SRC Pilot Plant laboratory to be entered into the computer. This lab data, coupled with plant process data, was used to produce a summary report of key operating parameters. A procedure was developed to average the previous day's data together, enter lab data supplied and run a summary report of the previous day's data. To facilitate the averaging of data over longer periods of time, the days of storage of process data were increased from 3 to 7.

In December, 1974, most of the basic software had been installed in the computer and more attention was given to process flowmeters and flowrate calculations. Flowmeter proving runs were begun, and results indicated that measurement accuracy was poor due to the large viscosity corrections required at normal plant flowrates. As a result, on-line computer meter coefficient calculations were modified to include viscosity correction calculations and also to include adjustment factors generated from the meter proving data.

In January, 1975, a program was placed on-line to produce a plot of the temperature and pressure profiles in the Area 02 preheater. The operator scan program was also modified to time-average values for scan periods of one minute or more; and a program to calculate process gas stream component information from averaged chromatographic and flowrate data was created. Programs were also designed to calculate reaction area material balance data around mass balance envelopes. These results were reported with the daily summary report.

A problem which always existed in the computer system concerned the system terminal and operators' terminal. The system software caused access to these terminals to be mutually exclusive. To help relieve this problem, a series of programs were written to produce the last chromatographic analysis for each stream upon the activation of a switch. This interface to the computer system was through digital contact closures, and was not affected by the use of any other terminal.

In the spring of 1975, plans were made for a new mode of plant operation called SRC-II. New equipment was installed in the plant and several new instruments were connected to the computer. In order to accomodate these changes, most of the programs in the system required modification. This work began in June and was completed in July, 1975. A great deal of knowledge about the Foxboro system software as well as better programming techniques had been acquired by this time, and the opportunity was taken to greatly improve the system, making future program changes easier, and also reducing the amount of drum storage required. At this time, a new program was added to the system to monitor plant instrumentation, allowing the computer to spot instrument problems which may require maintenance.

In August, 1975, a Foxboro Company representative installed an automatic restart function in the computer system. This modification ensures that if the computer shuts down for a short duration, the system automatically restarts and resumes data logging.

In the fall of 1975, work was dedicated mainly to software modifications designed to take less drum storage, thus making more room for additional programs. A program was added to increase the capacity of data averaging from days to several months by reading in off-line data from paper tape. A "Trend Log" program was added to bring hourly averages of up to seven points for a user specified data and time within the previous seven days. Finally, a program was written which computes equilibrium flash calculations. The program uses estimated equilibrium K-values derived from literature sources and plant experimental data, and performs a double linear interpolation to determine the K-value at any specified temperature and pressure.

In November of 1975, a model LA36 Decwriter was rented on a trial basis, thus evaluating a new type of peripheral device. (The merits of the Decwriter will be discussed later in the report.) Several programs were revised during January, 1976, to take advantage of the Decwriter's greater printing speed and 132 characters per line. A set of reports which previously took 40 minutes to run on the KSR35 teletype took 15 minutes on the Decwriter.

In the spring of 1976, primary software development was complete and attention now was turned to refining the system for both accuracy of measurement and optimization of program storage.

Two new programs were provided using the "Flash" subroutine to perform trial and error equilibrium flash calculations. Both of the programs simulate the SRC-II process, and were used to estimate stream compositions and flowrates for future slurry recycle testing. Several refinements were also made to the basic data collection system. The beginning and ending tank level measurements were expanded to allow one decimal place accuracy, and the out of range values were saved on an hourly basis, rather than daily totals as before. These revisions allowed some changes in the hourly average log to be made. If a process variable exceeded the set range of the field transmitter at any time during the hour, an "X" was printed alongside the value to indicate instrument service was required and that the values printed may be in question.

During the month of May, a major revision was made to the program which calculates flowrates based on plant process readings. The program was improved by adding a specific gravity correlation developed from laboratory data. For concentric orifice meters located in pipes of 1.5 inches I.D. or less, the number of iterations used for calculating "K", the coefficient of discharge, was increased from one to five. Further, for pipe sizes greater than 1.5 inches, a quadratic formula was utilized, which makes the calculation of "K" mathematically more precise. Several other refinements were made in the methods for calculating both liquid and gas flows, which should provide more accuracy as well as making future program modifications easier.

In September, 1976, new peripheral equipment for the Fox 2/30 computer system was installed. The equipment included 3 model LA36 Decwriters and a model 2200 Tally Corporation lineprinter. Software revisions were made to 21 different programs to take advantage of the reporting speed of the new equipment. At the same time, a Foxboro software option called "Fortran Run Time Package" was added to the system. When user programs were developed using the new package, the resulting program storage requirement was reduced 40 to 60%. A total of 48 programs were recompiled using the "Fortran Run Time Package", resulting in an overall saving of 15% of computer storage space. The additional room was utilized for plant process data storage, expanding this to 9 days.

When the major peripheral changes were completed, attention was again turned to the availability and reliability of data. When plant process data is collected by the computer system, a series of 5 programs must manipulate the data before it is ready for publication. To ensure that the data was properly processed, a status word was assigned each hourly file and as each program completed its assigned task, a corresponding bit was set. Any program accessing the data files checked the status word, and if the bits had not been properly set, a warning was printed and the data not used. In addition, a program was written to report the hourly value of the standard deviation for each process variable requested.

When a great deal of emphasis was placed on filtration area data, a series of program changes were made to provide better data for filtration studies. A program was designed to read the filter knife position once every 10 minutes, calculate and store the hourly knife advance rate, and report the average rate for the previous 20 minutes when requested. A new program was provided to accumulate all filtration data into a single report for each filter. The report was run routinely on the previous day's data or over any period desired by the user.

A procedure was implemented to provide documentation for all programs in the data acquisition system. The documentation includes a specification sheet (title, file number, mode of execution, etc.), a logic flow chart, a general description and purpose, a set of operation instructions and a listing of the Fortran program statements.

As part of a major revision for plant operation in the SRC-II mode, some 20 to 30 new instrument loops were to be read into the computer. The present system was already full, so a major revision of hardware and software was required. Two additional Analog Input Modules were installed to expand the process variable inputs to 336, requiring the installation of new system software. Since every user program had to be modified and recompiled, the computer system was taken out of service the second week in February, 1977, and a whole new data base was built. An operator console CRT was furnished with the new system which made some programs no longer necessary. The size of core memory was expanded by 4K words to allow larger programs to execute, and a magnetic tape unit will provide offline storage that can be quickly and easily retrieved.

The data collection system was placed back on-line March 15, 1977. Several modifications were made to the programs to improve performance. The system now maintains a cross reference table of date and time versus file number to allow fast access to a process data file when a specific date and hour is requested by the user. A program was added to calculate the feed from the coal surge bin via the new load cells on the bin and to compare the results with coal feed rate obtained from the gravimetric feeder (FTS). In order to evaluate the effect of erosion and corrosion in certain vessels and transfer lines throughout the plant, test racks of various materials have been installed. The data acquisition system logs the hours of operation at various flow and temperature ranges for each test point.

The new magnetic tape unit was a very important addition to the system. A program has been installed to dump all of the hourly process data to this device. This includes hourly averages for all 336 process variables, detected instrument malfunctions, all tank levels at the beginning of each hour, the calculated hourly flowrates for each flowmeter and hourly averaged chromatographic data for 12 streams. This will allow data to be retrieved and used again at any time in the future. In addition, the entire operating system is stored on tape and the system can be reloaded in 4 minutes. (The same process took 4 hours with the old paper tape system.)

2. Present Data Acquisition System

The functions of the computer system can be divided into two main categories. The first of these is a "Real Time" system which allows programs to be controlled by an internal clock, operating the system on a continuous basis without operator intervention. This system also allows interaction between programs through the use of system subroutines. A second system involves batch programs, which calculate and produce reports in the background, without interfering with the "Real Time" data collection system.

The highest priority of programs in the system are those which collect process measurements of temperature, flowrate, pressure and level, convert these to engineering units, and store them in bulk storage. A filing system for this data consists of 146 data files, allowing 6 days of bulk storage plus 2 extra working files. When the last file number is reached, the system automatically starts over at the first file number.

A data base for each process variable was built using The Foxboro Co. "IMPAC" system. Points can be specified to scan at 1, 10 or 30 second intervals. The "IMPAC" system automatically scans each point at the periods specified and stores these values in digital counts in a core measurement table. A program then picks up the 336 process variable measurements once every 30 seconds and stores the sum of the counts and the total number of readings in the designated process data storage file. A test is also made on each measurement to indicate instrumentation malfunctions which are indicated on the daily report.

At the beginning of each hour, a set of level readings are recorded and, at the end of each hour, the measurement sums are averaged, omitting any erroneous readings. Then, using a set of ranges maintained in computer memory, the measurement averages are converted to engineering units. Level readings are also converted to engineering units and statistical data are developed. A program called "Flow Calc" then converts raw flowmeter signals to mass flowrates using sophisticated equations involving current temperature and pressure information, data supplied by on-line chromatographs and laboratory determinations of physical properties. This is a large complex program that was sectioned to allow it to fit into the core transit area. A large portion of the program input data is also stored in data files which makes calculation changes possible without recompiling the program.

A series of programs were designed to service the three on-line chromatographs. The chromatographs communicate their needs through a series of contact closures supplied at the start of analysis, during stream identification and when the timing gate has been closed indicating that the maximum peak value for a component has been reached. A start of analysis program sets up an index file to guide future readings to their correct storage location. The stream identification is read and stored to indicate which stream is being analyzed. When the chromatograph has completed the reading of a component, a timing gate closes, triggering a program which immediately retrieves the value, scales it according to the range stored in the computer, reads and updates the index file, and places the value in the process data storage file. At the end of each hour a program converts the peak height values to component mole percents. The total amount of material recovered is tested and if the accountability is not between 95-105%, the analysis is discarded. Remaining analyses are normalized to 100%, averaged, sorted into stream order and stored in the process data storage files.

A second group of programs was designed to retrieve process data, perform calculations and print reports. The following is a list of reports that are published daily:

- Daily logs of hourly averages, beginning and ending levels and chromatographic analyses.
- Twenty-four hour tank level changes in inches and pounds.
- Twenty-four hour averaged chromatographic analyses.
- Slurry preheater plots of tube skin and process temperatures vs. the tube turn number, and pressure vs. the tube turn number.
- Gas balance calculations for gas flows reported as mole %, weight %, and calculated component flowrates in pounds per hour.
- Out of range reports for each variable measured.
- Summaries of 24 hour operating periods using data supplied by the laboratory and process measurements.
- A report on the hours of operation on each of the test racks in the corrosion/erosion test program.

A third classification of programs was written to provide plant operator assistance. A program to produce a scan or trend log of up to 12 process variables at a user specified scan period provides a continuing look at any operating parameters desired. A companion program allows the operator to retrieve the current value of any measurement. Both of these programs use the same "Flow Calc" program previously described to calculate flowrates in pounds/hour. Other programs include a slurry preheater temperature and pressure plot and a trend log of hourly averages for any part of the previous 6 days. A switch box allows the operator to call the last chromatographic analysis of any stream. By setting a series of switches according to a code provided, the operator may also obtain the current value of any loop without the use of the terminal keyboard.

A final series of batch programs was designed for engineering assistance. One program performs orifice plate sizing and coefficient calculations on demand. Also, three programs provide various vapor-liquid equilibrium calculations. In addition, several other programs were written to collect test data on various pieces of equipment and to test the reliability of various flow meters.

In general, nearly all of the computer programs used in this system were developed by P&M computer personnel. To date, approximately 115 programs have been completed, representing approximately 10,000 lines of Fortran coding, plus several assembler language subroutines.

3. Reliability

This section will deal with maintenance of the system hardware. The system central processor is a Digital Equipment Corporation model PDP 11/15. It is a mini-computer with a unibus design which allows only one instruction to be processed at a time. This is somewhat limiting in terms of program execution, but is sufficient for our purposes. It has generally been reliable but a few shutdowns have occurred due to component failures.

The interspec rack is the interface between process and computer, and performs analog to digital conversion. It had several malfunctions when first installed, but caused no trouble after a few weeks of operation.

The KSR35 teletypes were dependable peripheral devices for input and output. However, they were slow (10 characters per second) and noisy. To speed output capability, in November, 1975, a new model LA36 Decwriter was leased on a trial basis. This device prints at 30 characters per second, is very quiet, and since it is primarily electronic with few mechanical parts, it requires practically no maintenance. Three new Decwriters were added to the system in September, 1976, to replace the KSR35 teletypes.

The IBM Selectric typers were a source of continual trouble. They were an output device only, operating at 14 characters per second. The typer printed one character at a time and required several mechanical contact closures between each print cycle. The contact closures were constantly going out of adjustment and causing the typer to fail. A back-up typer was added on January 6, 1975, and a third typer was donated by The Foxboro Company at a later date. Even with 3 typers it was sometimes difficult to keep one in working order.

A model 2200 Tally Lineprinter was added to the system in September, 1976, to replace the Selectric typers. The lineprinter prints 200 lines per minute and has few moving parts. Despite a few small problems, it has proven very reliable.

A 980K word sealed disk is the on-line data storage device. When the original computer system was leased, The Foxboro Company quoted a mean time between drum failures of 2 years. However, mean time between failures experienced has been 9 months.

The paper tape reader, used to enter off-line data, has performed well. Occasional cleaning and adjustment of the optics is all that has been required.

The paper tape punch is used to store data off-line. The original punch lasted 2 years with no problems, but since that time 5 replacements have been required.

The new magnetic tape unit should eliminate most of the problems of off-line paper tape data storage; however, the unit has not been on-line long enough to properly evaluate it.

The system in general has performed well. A composite reliability report covering the period April 1975 to March 1977 is shown in Table 157.

TABLE 157

COMPUTER SYSTEM RELIABILITY
FOR
4/75 THRU 3/77

EQUIPMENT TYPE	% AVAILABILITY
INPUT DEVICES;	
A.) Operators Console CRT	100.0
B.) Tape Reader	99.9
C.) Decwriters (Avg. of 3)	99.8
D.) Magnetic Tape	----
E.) Process/computer Interface	99.1
F.) Teletypes (Avg. of 2)	99.8
OUTPUT DEVICES:	
A.) Tape Punch	96.5
B.) Decwriters (Avg. of 3)	98.7
C.) Line Printer	95.3
D.) Magnetic Tape	----
E.) Operators Console CRT	74.2
F.) Selectric Typers (Avg. of 3)	91.1
G.) Teletypes (Avg. of 2)	99.8
CENTRAL PROCESSOR	97.3
OVERALL SYSTEM	91.2

H. Sample Shipment Program

1. Scope

Since the inception of the SRC pilot plant operation, over 700 samples ranging in size from a few grams to over 3,000 tons have been sent to persons and research organizations all over the United States and to various foreign countries. Among these shipments are samples of coal and solvent refined coal, various feedstocks and intermediate streams, waste products and corrosion test samples. Prior to shipment, the Plant Laboratory confirms that the sample meets minimum specifications, if applicable, and retains a portion of the sample indefinitely for future reference.

2. Analysis of SRC shipments from Production Run

As described in Sections I.A.5 and III.A.3, the pilot plant was operated during the period from September 1975 to November 1976 for the purpose of producing 3000 tons of specification SRC to be used as fuel for a large scale burn test at Georgia Power Company's Plant Mitchell.

A summary of the control tests performed on a composite of SRC from each rail car is included herein as Table 158.

TABLE 158

SRC SHIPMENTS
FOR
SOUTHERN SERVICES BURNING TESTS

CAR NO.	TACOMA NET WEIGHT (TONS)	% ASH	% SULFUR	FUSION PT. °F	BTU/LB
BN 522106	85.2	0.35	0.68	350	--
BN 522171	71.3	0.31	0.66	265	15,975
BN 522549	83.9	0.54	0.70	275	15,840
NP 73279	85.8	0.30	0.87	285	16,030
BN 523121	50.4	0.48	0.71	300	16,135
NP 73116	84.9	0.46	0.81	350	15,810
UP 39003	76.9	0.10	0.86	315	16,282
UP 39004	76.7	0.26	0.63	300	15,890
UP 39005	73.1	0.29	0.65	300	15,920
UP 39006	80.1	0.16	0.65	305	16,065
UP 18396	70.2	0.14	0.69	345	15,850
UP 18579	59.9	0.14	0.70	355	15,785
UP 18481	64.1	0.14	0.57	325	15,970
UP 18595	62.1	0.17	0.60	320	15,923
UP 18598	66.2	0.16	0.64	350	15,855
UP 18609	60.6	0.14	0.67	325	15,977
UP 18711	60.4	0.17	0.71	325	15,870
UP 38979	74.3	0.17	0.77	345	15,783
UP 18155	53.4	0.15	0.91	340	16,262
UP 36772	75.2	0.14	0.87	325	16,312
UP 37082	79.9	0.16	0.87	326	16,079
UP 39299	79.6	0.11	0.68	335	16,173
BN 522459	72.3	0.25	0.74	350	15,732
BN 522112	78.5	0.17	0.83	350	15,975
NP 73279	38.5	0.17	0.85	335	15,699
CBQ 160850	75.6	0.16	0.83	330	15,644
NP 73548	90.2	0.14	0.83	345	15,966
CBQ 160599	71.9	0.16	0.77	360	15,758
BN 522496	79.7	0.17	0.73	355	15,939
BN 522171	80.6	0.16	0.72	353	15,805
WP 10138	58.7	0.12	0.63	345	15,875
WP 10123	63.4	0.14	0.56	347	15,846
WP 10101	61.4	0.16	0.64	335	15,063
WP 10235	59.8	0.12	0.61	340	15,987
WP 10151	62.2	0.09	0.77	310	15,924
WP 10110	72.1	0.15	0.78	330	15,924
WP 10247	79.3	0.20	0.76	350	15,871
WP 10280	62.6	0.10	0.92	335	15,892
BN 522142	99.9	0.16	0.65	289	16,101
CBQ 160834	73.5	0.16	0.71	315	15,800
NP 73625	89.8	0.09	0.82	330	16,010