

Solvent-Refined Coal (SRC) Process
Operation of Solvent-Refined Coal Pilot Plant,
Wilsonville, Alabama

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NOMENCLATURE

ABBREVIATIONS

avg	average
AWS	acetone-washed solids
BP	boiling point
BR	boiling range
btm(s)	bottom, bottoms
Btu	British thermal unit(s)
°C	degrees Celsius (Centigrade)
cc	cubic centimeter(s)
CF	capacity factor
cfm	cubic feet per minute
CI	cresol insoluble(s)
cm/sec ²	centimeters per second per second
col	column
comp	composite
conc	concentrate, concentrated, concentration
cons	consumed
conv	conversion
corr	corrected
cp	centipoise
CSD	Critical Solvent Deashing
cum	cumulative
cyc	cycle(s)
dia	diameter
DOE	United States Department of Energy
EP	end point
EPRI	Electric Power Research Institute
est	estimated
Eq	equation
°F	degrees Fahrenheit
Fe/S	iron to sulfur atomic ratio
filt	filtered, filtrate, filtration
fps	feet per second
Frac	fractionation
ft	foot (feet)
ft ⁻¹	reciprocal foot (feet)
ft/lb _m ₂	foot (feet) per pound mass
ft/sec ²	feet per second per second
ft ²	square foot (feet)
ft ³	cubic foot (feet)
ft-lb	foot-pound(s)
g	acceleration due to gravity (32.2 ft/sec ²)
gal	gallon(s)
gm	gram(s)
gph	gallons per hour
gpm	gallons per minute
H/C	hydrogen-to-carbon atomic ratio
HP	high pressure
hp	horsepower

hr	hour(s)
hr ⁻¹	reciprocal hour(s)
HVB	High Volatile Bituminous
IBP	initial boiling point
ID	inside diameter
in.	inch(es)
k	vapor-liquid equilibrium constant
KM	Kerr-McGee Corporation
lab	laboratory
L/D	length-to-diameter ratio
lb	pound(s)
lb-ft ²	pound feet (of torque)
lb/ft ²	pounds per square foot
lb/ft ³	pounds per cubic foot
lb/hr	pounds per hour
lb/hr-ft ²	pounds per hour per square foot
lb/hr-ft ³	pounds per hour per cubic foot
LP	low pressure
LSRC	light Solvent Refined Coal
lt	light
lt org	light organic
M	thousand(s)
mA	milliampere(s)
MAF	moisture and ash-free
MASF	moisture, ash, and solvent-free
max	maximum
MB	material balance
MCIF	moisture and cresol-insolubles-free
MF	moisture-free
mg	milligrams
mg/l	milligrams per liter
mid	middle
min	minimum, minutes
ml	milliliter(s)
mm	millimeter(s)
MM	million(s)
moist	moisture
mol %	mole per cent
mol wt	molecular weight
MP	melting point
MW	molecular weight
NA	not available
ND	not detected
NMR	nuclear magnetic resonance
no.	number
OD	outside diameter
OF	overflow
OH	overhead
OR	oxygen removed
org	organic(s)
OSR	organic sulfur removal
ovhd	overhead
P	pressure

PA	preasphaltene(s)
$p(H_2)$	hydrogen partial pressure
ppm	parts per million
prod	product, produced
psi	pounds per square inch
psia	pounds per square inch absolute
psig	pounds per square inch gauge
qtr	quarter
r	correlation coefficient
$^{\circ}R$	degrees Rankine
ref.	reference
R_m	filter medium resistance
rpm	revolutions per minute
scf	standard cubic foot (feet) at 525 $^{\circ}R$ and one atmosphere pressure
scfh	standard cubic feet per hour
scfm	standard cubic feet per minute
sec	second
sec^{-1}	reciprocal seconds
SG	specific gravity
SLS	solid-liquid separation
SN	sample number
solv	solvent
sp gr	specific gravity
spec	specification
SRC	solvent refined coal
SRT	short residence time
SS	stainless steel
std	standard
TPB	true boiling point
temp	temperature
THF	tetrahydrofuran
TI	terphenyl insoluble(s)
tpd	tons per day
tr	trace
UC	unreacted coal
UCC	undissolved carbon compounds
USF	United States Filter Corporation
vac	vacuum
visc	viscosity
vol	volume
vs	versus
wt	weight

SYMBOLS

α	average specific cake resistance
@	at
ΔH_R	heat of reaction
Δt	temperature differential
ΔP	pressure differential
<	less than
>	greater than
\approx	about, approximately equal to
Σ	sigma (sum)

EQUIPMENT DESIGNATIONS

B	boiler, heater
C	compressor
D	dryer
DE	density element
DIT	density indicator transmitter
DP	differential pressure
DPCV	differential pressure control valve
DPSH	differential pressure switch
DPT	differential pressure transmitter
E	exchanger
F	filter
FIC	flow indicator and controller
FR	flow recorder
FV	flow valve
GC	gas chromatograph
GLC	gas/liquid chromatograph
HV	hand valve
LSH	high level switch
LSL	low level switch
K	product cooler or flare
LV	letdown valve
LS	level switch
P	pump
PSH	high pressure switch
PSV	pressure safety valve
PV	pressure valve
R	reactor
SV	safety valve
T	tower; timer
TC	thermocouple
TR	temperature recorder
V	vessel
XV	filter cycle program operated valve

COAL IDENTITY

Coals processed during 1979 are listed below, together with the nomenclature used in this report for identification:

- o Period: 1 through 29 January

Company:	Pyro Mining Company
State:	Kentucky
Mine:	Pyro
Seam:	No. 6 and 11
Abbreviated identity:	Ky 6 and 11

- o Period: 1 February to 20 May

Company:	Pyro Mining Company
State:	Kentucky
Mine:	Pyro
Seam:	No. 9
Abbreviated identity:	Ky 9 (Pyro)

- o Period: 20 May through 31 December

Company:	Lafayette Coal Company
State:	Kentucky
Mine:	Lafayette
Seam:	No. 9
Abbreviated identity:	Ky 9 (Lafayette)

ABSTRACT

Operating conditions and test results obtained at the six-ton-per-day Solvent Refined Coal (SRC-I) pilot plant in Wilsonville, Alabama, during 1979 are summarized. The plant was operated for a total of 284 days, or 78% of the year. Three coals were processed: Kentucky 6 and 11 and Kentucky 9 from the Pyro mine, and Kentucky 9 from the Lafayette mine. The coal space rate ranged between 25 and 50 lb/hr-ft³, dissolver conditions ranged between 1,700 and 2,100 psig at 825°F, and gas feed rates ranged from 6,000 to 12,000 scfh. Conversion to cresol soluble organics ranged from 90 to 95 wt % of the MAF coal, while hydrogen consumption ranged from 1.3 to 2.7 wt % and SRC yield ranged from 45 to 75 wt % of the MAF coal. Mineral separation techniques studied included Critical Solvent Deashing (CSD) and vertical leaf filtration. SRC recoveries at the CSD unit ranged from 65 to 85 wt % of SRC in the CSD feed. Filtration rates of 4.4 gal/hr-ft² of filter area were demonstrated with precoat. However, operation without precoat was unsuccessful.

I. INTRODUCTION

In March 1972, the Southern Company and the Edison Electric Institute (EEI) jointly began an investigation of the solvent refining process for making low-sulfur and low-ash solid fuel from coal. A six-ton-per-day solvent refined coal (SRC) pilot plant was designed and constructed at Wilsonville, Alabama by Catalytic, Inc. Catalytic also operates the facility under the management of Southern Company Services, Inc., a unit of the Southern Company.

Since April 1973, the Electric Power Research Institute (EPRI) has performed the functions of utility industry project supervision initiated by EEI. The United States Energy Research and Development Administration (ERDA), now Department of Energy (DOE), has been a co-sponsor since 1976.

Operation with coal began in January 1974. This report presents a summary of activities during 1979 with emphasis on work completed during the fourth quarter. Earlier work has been documented in preceding monthly, quarterly, and annual Technical Progress Reports.

The principal objectives of the 1979 Wilsonville SRC program were:

- o To evaluate promising solid-liquid separation (SLS) processes, including

Kerr-McGee Critical Solvent Deashing

- improve performance
- establish the effects of pilot plant and CSD operating conditions on yield and performance
- establish a data base for process scale-up: heat transfer, solids settling rates, solvent losses, and solvent stripping requirements

USF Vertical-Leaf Filter

- investigate performance in both precoat and non-precoat modes
- o To provide technical support for a planned 6,000 tpd SRC-I demonstration plant, with emphasis on determination and control of long-term stability of the process, yield structures, hydrogen consumption, process solvent balance, and product specifications

- To establish improvements in process equipment and operations
- To evaluate potential improvements in the SRC process, including:
 - CSD third stage solvent and LSRC recovery
 - process variables
 - equipment requirements

To fulfill these objectives, a series of runs was planned in which the following parameters were varied for evaluation:

- Dissolver volume
- Dissolver pressure
- Coal feed rate
- Feed gas rate (to preheater and bypass)
- Dissolver solids withdrawal rate
- Dissolver product cooler bypass

The processing of Kentucky 6 and 11 coal from the Pyro Mining Company's Pyro Mine, begun in September 1978, was concluded on 29 January 1979. Processing of Kentucky 9 (which is the design basis coal for the SRC-1 demonstration plant) from the Pyro Mine was begun on 1 February. A change to Kentucky 9 coal from the Lafayette Coal Company's Lafayette Mine was made on 20 May, and the Lafayette coal was used for the remainder of the year.

Runs 155 through 194 were completed during 1979. These have been documented as follows:

<u>Runs</u>	<u>Wilsonville Technical Report</u>
155-161	First quarter: January-March 1979
161-155	Second quarter: April-June 1979
166-171	Third quarter: July-September 1979
172-194	Annual Report for 1979

II. SUMMARY

A. PROCESS DESCRIPTION

In the Solvent Refined Coal (SRC-I) process, pulverized coal is slurried with a process-derived recirculating solvent. Hydrogen is added to the slurry which is heated and fed to a dissolver. Effluent from the dissolver is flashed and the gas is separated and scrubbed to remove hydrogen sulfide and carbon dioxide. Makeup hydrogen is added and the scrubbed gas is recycled.

Effective solids removal is critical to SRC product quality. During 1979, two systems were evaluated for separating undissolved coal solids from SRC. One of these utilized pressure-leaf filtration and the other employed the Kerr-McGee Critical Solvent Deashing (CSD) unit.

In the filtration mode, solids are filtered from the SRC solution, and the filtrate is distilled to separate the solvent and SRC fractions. In the CSD operating mode, the unfiltered SRC slurry is distilled to separate the solvent, and the ash-laden SRC fraction is extracted with a deashing solvent at conditions near the critical temperature and pressure of that solvent. The SRC and light SRC (LSRC) are removed from the heavy ash concentrate as separate phases. The deashing solvent is recovered from each phase and recycled. A portion of the LSRC may be used for coal slurry preparation.

After deashing by either method, the SRC is solidified by cooling. Recovered solvent is fractionated to separate the liquids boiling below 450°F from the solvent. When filtration is used for solids separation, a wash solvent boiling at 350-450°F is also recovered and recycled for filter cake washing. When the CSD unit is operating, wash solvent is not recycled.

B. OPERATING HIGHLIGHTS

The major objectives of the 1979 pilot plant program were fulfilled, and several projects of importance were begun. Forty runs (155 through 194) were completed using coals from three different sources. From 1 to 29 January, a mixture of Kentucky 6 and 11 from the Pyro mine was processed; and from 1 February to 20 May, Kentucky 9 coal from the Pyro mine was processed. Lafayette coal testing began in the second quarter during Run 163. Runs 172 through 194 were made during the fourth quarter, all with Kentucky 9 coal from the Lafayette mine. The SRC plant was in operation for 78% of the year, the best annual on-stream factor to date.

There were two noteworthy coal-related problems during the year. The first problem was extensive corrosion in the solvent recovery system. From inspection of corrosion coupons, it appeared that corrosion began during Pyro coal processing and continued during the Lafayette coal tests. The corrosion rate appears to be strongly related to the chloride content of the coal. In any case, when measured, the corrosion rate was as high as 6,000 mils/year in the fractionator. Adding sodium carbonate to the slurry feed at a rate of 1.1 wt % of coal prevented significant additional corrosion as measured by resistance-type corrosion probes installed during the year.

The other significant coal-related problem encountered during 1979 was a high slurry preheater coking rate experienced with Lafayette coal. Primarily as a result of operating conditions at the pilot plant, this coal produced a solvent of low quality, which might have contributed to the high coking rate. It was necessary to decoke B102 Slurry Preheater three times within a six-month period while operating with this coal. One noteworthy achievement was the demonstration of high temperature operation of the high pressure separator, called the hot flash mode of operation. This is a modification of the normal procedure for dissolver slurry cooling, flashing, and reheating for solvent recovery. In hot flash operation, dissolver slurry cooling and subsequent reheating for solvent recovery are minimized or eliminated. Substantial energy savings may result from this mode of operation.

The CSD unit was operated in every month except November and December. For the year, 388 tons of material from T102 Vacuum Column and 7.8 tons of bottoms from an H-Coal vacuum still were processed. Continuous operation was logged during several extended periods. Plugging, particularly in the ash recovery portion of the unit, caused several interruptions. Deashing solvent losses were high (about 5 to 10% of the CSD feed). Both problems, plugging and deashing solvent losses, are being studied.

The United States Filter Corporation (USF) vertical-leaf filter was operated during the fourth quarter. The cresol-insolubles in the filtrate were very low when screen integrity was good. Screen failures, however, resulting principally from improper fabrication, were common. Mechanically, the USF filter performed much better than the Funda filter which had been tested at Wilsonville for four years. Future operations with solution-annealed screens should result in improved screen performance.

C. PROCESS ANALYSIS

1. Overview

Several significant technical contributions were made during 1979 to the understanding of the SRC process. These were as follows:

- o Sodium carbonate, when mixed in the coal slurry, was an effective agent for reducing corrosion in the SRC plant solvent recovery system. (See Run 166).
- o Sodium carbonate introduced into the coal slurry apparently contributed to high ash in the filtered SRC products. (See Runs 173-194).
- o Operation of the USF filter as configured at Wilsonville without precoat was not practical. (See Runs 191-193).
- o The preasphaltene content of the high-ash SRC was reduced significantly by improving the quality of the process solvent, all other factors being the same. SRC recovery in the CSD unit was also improved. (See Runs 167 and 171).
- o Reducing the coal space rate reduced the pre-asphaltene content in the high-ash SRC product and increased the SRC recovery in the CSD unit. However, hydrogen consumption increased with little significant improvement in process solvent quality. (See Runs 155, 156, 157, 159, 171, 172, and 190).
- o During the 1979 Wilsonville program, the process solvent quality was most strongly governed by the nature of the coal being processed. From best solvent to worst, the coals rank as follows: Pyro No. 9, Pyro No. 6 and 11, and Lafayette No. 9. (See Runs 156, 157 and 163).
- o Processing of Kentucky 9 coal from the Lafayette mine resulted in a lower SRC recovery in the CSD unit than when processing Kentucky 9 coal from the Pyro mine, all other factors being the same. (See Runs 163 and 166A).
- o Reducing the vacuum column bottoms temperature below 560°F had very little effect on SRC recovery in the CSD unit. (See Runs 167 and 168).
- o Unhydrogenated ("neat") anthracene oil was successfully used as a process solvent supplement. It significantly improved a solvent of poor quality. (See Runs 167-172).
- o Increasing the solvent content in the CSD feed only slightly increased the SRC recovery. (See Runs 171-172).

2. Summary of Individual Runs

Run 155, begun on 3 January, was made to study the effects of dissolver operating conditions on CSD unit performance. The conditions were: 450 lb/hr of Kentucky 6 and 11 (Pyro) coal at a slurry concentration of 38.5 wt %, a coal space rate of 50 lb/hr-ft³, a feed gas rate of 5,000 scfh at 85 mole % hydrogen purity, a dissolver temperature of 825°F and a pressure of 1,700 psig. There was no LSRC addition and no solids were withdrawn from the dissolver. Solvent quality as measured by microautoclave (short test) ranged in the low 60's. Coal conversion ranged from 92 to 94 wt % of MAF coal, and hydrogen consumption ranged from 1.4 to 1.9 wt % of MAF coal. This low hydrogen consumption resulted in a dissolver hydrogen partial pressure higher than the desired 900 psia. Reducing the feed gas rate to correct the hydrogen partial pressure, and increasing the slurry preheater outlet temperature to 866°F to offset an increase in the dissolver temperature, ultimately led to plugging of the slurry pre-heater on 11 January.

The preheater was decoked, and Run 156 began on 15 January. The operating conditions were the same as for Run 155, except that the feed gas rate was increased to 10,000 scfh and the coal space rate was reduced to 25 lb/hr-ft³ (100% of the dissolver volume was used). The solvent quality did not improve significantly during this run, indicating that the dissolver residence time has limited effect on solvent hydrogenation capability. Coal conversion was about the same as in Run 155, but hydrogen consumption was higher, ranging from 2.1 to 2.6 wt % of the MAF coal. The SRC yield and sulfur content were lower in this run than in Run 155. The CSD unit was operated for a total of 129 hours during Run 156, and the average recovery of SRC in the CSD feed by forced ash balance was 83%.

Run 157, which began on 1 February, was the first run in which Kentucky 9 coal from the Pyro mine was used. The conditions were otherwise identical to those for Run 156. Coal conversion, hydrogen consumption, sulfur removal, SRC yield, and CSD recovery were initially about the same in both runs; but, after a week of feeding Kentucky 9 coal, the solvent quality began to increase significantly. Due to operating problems with T102 Vacuum Column, traced to corrosion in the lower rectifying section, this run ended on 11 February.

Run 158 was a 16-hr short residence time (SRT) run. During this run, process solvent from excess solvent inventory was used for slurry preparation to avoid affecting the working process solvent. The run conditions were: 500 lb/hr coal feed in a 38.5% slurry, 3,400 scfh of feed gas at 93 mole % hydrogen purity, 2,400 psig preheater inlet, and 850°F pre-heater outlet. The dissolver was bypassed. The effective coal space rate was 125 lb/hr-ft³. Coal conversion to

cresol-soluble organics was 90 wt % of MAF coal. Consumption of gaseous hydrogen was 0.9 wt % of MAF coal, and SRC yield averaged 68 wt % of MAF coal. The reaction product was withdrawn from V110. Neither the flash tank, the solvent recovery system nor the CSD unit were operated.

Run 159 was started on 29 February using the same process solvent for slurry preparation as was used in Run 151. The run conditions were identical to those for Run 157 except that the coal space rate was increased to 50 lb/hr-ft³. The process solvent quality was excellent during this run, ranging in the mid-70's (% THF conversion, microautoclave short test). The yield of SRC was similar to that for Run 157, while the hydrogen consumption was much less, ranging from 1.2 to 2.0 wt % of MAF coal. CSD recovery was lower than in Run 157, averaging about 75% SRC in the CSD feed. The data suggest that decreasing the dissolver residence time reduces the quality (increases the sulfur content) of the SRC, although the hydrogen consumption is reduced.

Run 160 began on 14 March with conditions identical to those for Run 159, except that 75% of the dissolver volume was used and the coal feed rate was increased to 515 lb/hr. (The resulting coal space rate was 38 lb/hr-ft³). The process solvent quality remained high as did conversion to cresol-soluble organics. The CSD recovery improved at these conditions, averaging 81% during the last eight days of the run. Hydrogen consumption increased, ranging from 1.8 to 2.1 wt % of MAF coal during the last eight days. The preasphaltene content was lower in the CSD feed for Run 160 than for Run 159.

The first attempt at solids withdrawal from the dissolver occurred during Run 161, which began on 29 March. Solids were withdrawn at a rate equivalent to 5 to 10% of the slurry feed, and the run conditions were identical to those for Run 160. The process solvent quality was such that 71 to 73 wt % conversion to THF-soluble material was obtained consistently in the short microautoclave test. Conversion of coal to cresol-soluble organics remained high, ranging from 92 to 95 wt % of MAF coal. Sulfur removal and SRC yield were similar to those of Run 160. However, CSD recovery was much lower, averaging in the mid-70's during the 48-hr MB period and in the few days preceding it. The preasphaltene content of the CSD feed was higher in Run 161 than in Run 160, although the solvent extraction analyses for the SRC derived from the dissolver product slurry were similar in both runs.

Run 162 began on 12 April after a power failure had ended Run 161. The conditions were the same as those for Run 161 except that the dissolver pressure was increased from 1,700 to 2,100 psig. Solvent quality increased slightly during the run despite a decrease in the average boiling point. Hydrogen consumption increased from 1.5 to 1.7 wt % of MAF coal. CSD recovery increased significantly, from the mid-70's to the low-80's, with

a net increase of about 5 to 8 wt % of the feed. The SRC derived from the dissolver product slurry during Run 162 MB showed a lower preasphaltene content (16.1 wt %) than that for Run 161 MB (36.0 wt %). The decrease in preasphaltene in the CSD feed was even more dramatic: 8.7 wt % for Run 162 MB as compared to 26.1 wt % for Run 161 MB. This decrease can be explained by the T102 Vacuum Column bottoms temperature, which was lower (552°F) during Run 162 MB than that during Run 161 MB (573°F).

The conditions for Run 163, which started on 1 May, were similar to those for Run 161, except that part of the 10,000 scfh feed gas bypassed the slurry preheater. Operating in this manner allowed the slurry preheater coil outlet temperature to be increased from 795°F to 845°F. The dissolver outlet temperature was the same in both runs. Run 163 yielded less preasphaltene and more process solvent than Run 161. Solvent quality and hydrogen consumption were similar in both runs, but there was more sulfur removed from the SRC product in Run 163. The recovery of SRC in the feed to the CSD unit was slightly higher for Run 163, and the T102 bottoms temperature was lower.

On 20 May, the feed coal for Run 163 was changed from Pyro to Lafayette mine coal. (Both are from the Kentucky 9 seam.) The run was continued for ten more days without other process changes. After the coal change, the T102 bottoms temperature remained the same, but the recovery of SRC from the CSD unit decreased from about 80% to about 77%. Solvent quality also began to decrease, and the average yield of SRC increased from 68 to 74 wt % (by forced ash balance on the dissolver product slurry).

Run 164, begun on 2 June, was a short residence time (SRT) run in which the dissolver was bypassed. It was made at a lower gas rate and higher slurry preheater coil outlet temperature than Run 158. The run conditions were: 235 lb/hr coal feed rate, 38.5 wt % slurry concentration, 1,000 to 2,000 scfh feed gas at 85 mole % hydrogen purity, and preheater outlet conditions of 2,400 psig and 850-885°F. The CSD unit was operated and LSRC was added to the SRC plant feed slurry. Initially, the LSRC concentration in the slurry was 15 wt %, but on 3 June, it was increased to 25 wt %. A 24-hr material balance was completed on 4-5 June. Hydrogen consumption was low, averaging 1.4 wt % of MAF coal for the MB period. Coal conversion was also low, averaging 91 wt % of MAF coal. The recovery of SRC from the CSD unit feed averaged 75% for the MB period. Operation at these conditions with relatively poor quality solvent soon resulted in severe coking in the preheater coil. The high preheater outlet temperature contributed to the coking. The run was stopped on 5 June when the preheater coil plugged.

Run 165 was begun on 14 June to rehydrogenate the process solvent and to evaluate the modified USF filter. Run conditions were: coal feed of 450 lb/hr, 38.5 wt % slurry concentration, 10,000 scfh feed gas (85 mole % hydrogen), 75% dissolver in service (33 lb/hr-ft³ coal space rate), dissolver conditions of 825°F and 2,100 psig, and no solids withdrawal. LSRC was not added to the slurry feed. Ten cycles were completed on the USF filter before it was shut down due to an off-center hub gasket which caused high cresol-insolubles (CI) in the filtrate. An inspection of the filter revealed the potential for screen failure under the stress induced by the high-pressure sluice system. A new screen fabrication procedure was proposed as a result of this inspection. Run 165 ended on 17 June when the fuel oil supply at the plant was exhausted as the result of an independent truckers' strike.

Run 166 was begun on 21 June at the following conditions: 515 lb/hr coal feed (38.5 wt % concentration in slurry), 10,000 scfh feed gas (85 mole % hydrogen), LSRC added to the slurry at an approximate rate of 20 lb/hr, dissolver conditions of 825°F and 2,100 psig, and solids withdrawal at a rate equivalent to 6 to 10 wt % of the slurry feed. These conditions were similar to those of Run 162, except that Lafayette coal was used during Run 166. The conversion to cresol-soluble organics, SRC yield, hydrogen consumption and sulfur removal were similar in the two runs. The solvent quality increased from 66 wt % THF conversion (micro-autoclave, short test) to 76 wt %. After 19 days, the dissolver pressure was decreased to 1,700 psig, and shortly thereafter, the mode of dissolver product recovery was switched from normal to hot flash. At 1,700 psig, Run 166 continued for 24 more days. A 24-hr material balance was completed in the hot flash mode. At the end of the run, the SRC plant was returned to the normal flash mode and another 24-hr material balance was completed. The SRC recovery at the CSD unit did decrease when switching from normal to hot flash, but more data is needed before any firm conclusions can be drawn.

During Run 166, sodium carbonate was introduced into the slurry feed to reduce corrosion in the SRC plant solvent recovery system. The corrosion rate in the middle of the T105 Fractionation Column had been as high as 6,000 mils per year, on carbon steel, as measured by a resistance-type corrosion probe. The addition of sodium carbonate on 28 June at a rate of 40 lb/slurry charge (1.1 wt % of coal feed) quickly reduced the corrosion rate to a negligible level. Acid gas production seemed to decrease after sodium carbonate was added. However, this could be attributable to the use of Lafayette coal.

Run 167, begun on 19 August, was made to study the effects of varying the solvent content in the CSD feed on CSD performance. The following conditions were used: coal feed

rate of 450 lb/hr at 38.5% slurry concentration, 10,000 scfh of feed gas at 85 mole % hydrogen purity, 50% dissolver in service (50 lb/hr-ft³ coal space rate), LSRC addition at a rate of about 30 lb/hr, dissolver conditions of 2,100 psig at 825°F, and solids withdrawal from the dissolver at a rate equivalent to approximately 6 wt % of the slurry feed. The dissolver product separator was operated in the normal flash mode. The solvent quality was poor during the run, averaging 65 wt % conversion to THF soluble material in the micro-autoclave short test. The yield of SRC at the vacuum column bottoms ranged from 49 to 65 wt % of MAF coal. The hydrogen consumption was low, averaging 1.5 wt % of MAF coal. The temperature in the vacuum column bottoms was controlled at approximately 555°F to maintain a 12 wt % solvent content in the CSD feed. The apparent SRC recovery at this condition was 74 to 76 wt %.

Run 168, begun on 9 September, lasted for 79 hours. The only difference between this run and Run 167 was the solvent content in the CSD feed. The vacuum column bottoms temperature was adjusted to about 530°F in an attempt to produce vacuum bottoms having 20 wt % solvent. However, the maximum solvent content which could be produced was only 15 wt %. The SRC recovery in the CSD unit during Run 168 did improve slightly and averaged 78 to 79%.

Run 169, begun on 13 September, was conducted at the same conditions as Runs 167 and 168, except that the dissolver product separator was in the hot flash mode. Solvent quality remained low, causing concern that the operating conditions were unsuitable to sustain long-term operations. The run was interrupted for more than two days by a power failure, and was ultimately terminated on 17 September when the vacuum column preheater outlet plugged.

During Run 166, 168, and 169, the process solvent inventory had decreased to a quantity insufficient for plant startup. Consequently, during Run 170 which began on 19 September, 23,000 lb of Allied 24CB anthracene oil was added to the process solvent on a pound-for-pound basis. The run conditions were essentially the same as for Runs 167 and 168, except that solids were not withdrawn from the dissolver. The process solvent quality showed little increase during this 71-hour run. However, the quality of the solvent improved from 65 to 74 wt % conversion to THF soluble material during the first few days of the next run.

Run 171, begun on 22 September, differed from Run 167 only in the separator mode (hot flash versus normal for Run 167) and in the process solvent quality. The addition and subsequent hydrogenation of the anthracene oil in the previous run had improved the solvent quality markedly. At the same time, the specific gravity of the process solvent increased and the hydrogen content decreased. The solvent content in

the CSD feed was maintained at 9 to 10 wt % by controlling the vacuum column bottoms temperature at approximately 560°F. Two 24-hr material balances were completed. The recovery of SRC in the CSD unit ranged from 77 to 80 wt %, which was slightly higher than the 74 to 76 wt % calculated in Run 167. The preasphaltene contents of the SRC derived from the dissolver product slurry and also from the CSD feed were lower in Run 171 than in Run 167.

Run 172 was a short run started on 30 September at conditions identical to those for Run 171, except that the vacuum column bottoms temperature was decreased to 480-505°F in an effort to achieve 20 wt % solvent in the CSD feed. A 24-hr material balance was completed on 2 October with 24 wt % solvent in the CSD feed. The SRC recovery in the CSD unit was 82 wt %, which was slightly higher than that in Run 171. The relative percentages of asphaltene and preasphaltene in the CSD feed for Runs 171 and 172 were not significantly different. This result indicated that further reductions in the vacuum column bottoms below 560°F had little significant effect on CSD feed quality or SRC recovery in the CSD unit.

In Runs 173 through 194, the USF vertical-leaf filter was used for solids separation and the CSD unit was out of service for modifications. Run 190 MB was the only material balance completed during the filter runs.

Run 173 started on 2 October at the following conditions: coal feed rate of 180 lb/hr in a 23.5 wt % slurry, no LSRC addition, sodium carbonate addition of 40 lb/batch (1.1 wt % of coal feed), 10,000 scfh of 85 mole % hydrogen feed gas, dissolver conditions of 2,100 psig and 840°F, solids withdrawal from the dissolver at a rate equivalent to 6 wt % of slurry feed, and the dissolver product separator in the normal flash mode. These conditions remained the same through Run 180, except for feed rate changes to accommodate filtration requirements. Process solvent quality remained high during this series of runs, ranging in the mid-70's. Coal conversion to cresol solubles ranged from 92 to 95 wt % and hydrogen consumption ranged from 1.6 to 2.4 wt % of MAF coal. Problems with filter screen integrity plagued this series of runs. Numerous interruptions were required to repair or replace screens damaged by corrosion resulting from faulty fabrication procedures, or to reseat gaskets which had caused the screen assembly to bind during rotation. However, the sluicing system worked well. Precoat was used in all of these runs at a rate of 0.35 lb/hr-ft³ of filter area. For Run 179, the specific cake resistance was calculated to be about 1.5×10^{12} ft/lb_m. This was approximately 2.5 times the cake resistance measured for Indiana V coal and the Funda filter. Although determinations of cresol insolubles indicated that the filtrate had good clarities, the filtrates had high ash contents, ranging up to 0.2 wt %. SRC derived from these filtrates also had high ash, averaging 0.55 wt %.

It was suspected that the addition of sodium carbonate was increasing the ash contents of the filtrate and of the SRC, even though filtrate clarity was acceptable. Some sodium compounds, such as $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$, are soluble in process solvent. Consequently, a series of runs was initiated in which the sodium carbonate addition rate was reduced and the corrosion rate was carefully monitored. Run 181, the first of the series, was started on 30 October at essentially the same conditions as Runs 173-180, but with sodium carbonate addition reduced from 40 lb/slurry batch (1.1 wt % of coal feed) to 30 lb/slurry batch (0.83 wt %). No change in the corrosion rate was detected during this 142-hr run. At the beginning of Run 182, on 5 November, the sodium carbonate was further reduced to 20 lb/slurry batch, or 0.55 wt % of coal feed. No corrosion rate increases were noted in the solvent recovery system. By Run 182, the average ash in the SRC product from the solidifier had decreased from 0.55 wt % to 0.43 wt %, suggesting a connection between sodium carbonate addition and the observed filtrate of good clarity but high ash. Screen performance improved during Run 179 when the frequency of sluicing was reduced to once per 25 cycles at 1,000 psig sluice header pressure, and this enabled the longer Runs 181 and 182 to be completed.

Run 183 was started on 13 November at conditions identical to those of Run 182, except that sodium carbonate addition was further reduced to only 10 lb/slurry batch, or 0.27 wt % of coal feed. After 34 hours of operation at these conditions, the hydrogen partial pressure in the dissolver was reduced to approximately 1,100 psia to simulate the design conditions of a planned 6,000 tpd demonstration plant.

Run 184 was begun on 14 November with the dissolver pressure reduced to 1,700 psig and the feed gas rate reduced to 9,000 scfh. Both the conversion to cresol-soluble organics and the hydrogen consumption decreased during this and subsequent runs made at these dissolver conditions. Process solvent quality also decreased, from 75 to 69 wt % conversion to THF soluble material by the microautoclave short test. The decrease was from 67 to 60 wt % for the long microautoclave test. Run 184 was terminated when poor clarity filtrate was detected. Two of the three filter leaves were replaced.

Run 185 was started on 16 November at the same conditions as Run 184. During this 94-hr run, the average ash content in the SRC product was 0.21 wt %. No corrosion was detected in the solvent recovery system.

Run 186 was started on 20 November at the same conditions as Run 185, except that the feed gas rate was reduced from 9,000 to 8,000 scfh and the sodium carbonate addition was reduced to 5 lb/slurry batch, (0.14 wt % of coal feed). The average ash content in the SRC product for this 160-hr run

was 0.20 wt %. Although the specific cake resistance at constant pressure was similar in this run to that of Run 179, the cake was more compressible. No appreciable increase in corrosion rate was detected in the solvent recovery system during Run 186.

Runs 187 and 188 were short runs conducted under the same conditions as Run 186, except that the coal feed rate was reduced from 450 lb/hr to 390 lb/hr, which reduced the coal space rate from 50 to 44 lb/hr-ft³. The filter was operated without precoat for the first time and sluicing frequency was increased to once per cycle at 1,200 psig sluice header pressure. This seemed to increase the ash content in the filtrate; consequently, the filter was switched back to precoat operation for Run 188.

The feed gas was reduced from 8,000 to 6,000 scfh for Run 189, which began on 1 December, in an effort to operate the dissolver at a hydrogen partial pressure of 1,100 psia. Hydrogen consumption had been decreasing slightly in November, to a low of 1.5 wt % of MAF coal. As a result, the hydrogen partial pressure in the dissolver gas phase was higher than desired. The filter was operated without precoat. The first two cycles gave acceptable filtrate clarity, but a gasket leak caused excessive ash in the remaining cycles. Consequently, the run was stopped on 3 December.

Since August, the slurry preheater had been monitored closely to observe any evidence of coking. The most predominant trend was measured by the skin/fluid temperature probes at the hottest turns in the preheater coil. Based on these temperature readings, it was decided to coke the slurry preheater with steam and air.

While inspecting the dissolver, solids deposits were found which could have greatly reduced gas/slurry contacting efficiency.

Run 190 was started on 9 December. In late November, the corrosion rate in the solvent recovery system increased, so for this run the sodium carbonate addition was increased to 10 lb/slurry batch. The run conditions were as follows: coal feed rate of 450 lb/hr at 38.5 wt % slurry concentration, feed gas rate of 10,000 scfh at 85 mole % hydrogen purity, dissolver conditions of 2,100 psig and 825°F, and solids withdrawal at a rate equivalent to 6 wt % of the slurry feed. The dissolver volume in use was increased from 50 to 75%, reducing the coal space rate from 50 to 36 lb/hr-ft³. At the higher feed gas rate, pressure and dissolver volume, the hydrogen consumption increased from about 1.7 wt % to 2.5 wt % of MAF coal. The process solvent quality remained unchanged. The filter was operated satisfactorily in the precoat mode during this 140-hr run. The average filtration time at the end of the run was 75 minutes

for 4.4 gallons of filtrate per square foot of filter area. The average ash in the SRC product was 0.14 wt %. A 48-hr material balance period was completed. The preasphaltene and organic sulfur contents of the SRC derived from the dissolver product slurry were significantly lower than those obtained in Runs 171 or 172, both of which were made at a higher coal space rate.

Runs 191, 192, and 193 were all conducted at essentially the same reaction section conditions, with the exception of sodium carbonate addition and small feed adjustments necessary to compensate for varying filtration rates. The coal was fed at 500 lb/hr in a 38.5% slurry concentration, the recycle gas rate was 11,700 scfh at 85 mole % hydrogen purity, the dissolver pressure was 1,700 psig, the temperature of 825°F, and the space rate was 36 lb/hr-ft³. Solids were withdrawn from the dissolver at a rate equivalent to 6% of slurry feed. Sodium carbonate addition was 0.44% of coal feed for Run 191, and 0.55% for Runs 192 and 193. Hydrogen consumption averaged 2.2% of MAF coal feed for these runs, and conversion averaged 93.1% of MAF coal feed. The sulfur content in the reaction section extract was 0.99 wt %, and the solvent quality was fairly constant, averaging 69% conversion by the short method and 62% by the long method. The USF filter was run without precoat for the three runs. Filtration time increased during Run 191 from 95 minutes initially to 160 minutes at the end for 4.4 gallons of filtrate per square foot of filter area. Increasing the sluicing pressure to 1,500 psig did not improve the filtration rate. Filtration time for 4.4 gal/ft² increased from 70 minutes at the beginning of Run 192 to 210 minutes after only 10 cycles. When the sluicing pressure was increased to 1,500 psig, a high-ash filtrate resulted. Poor screen quality plagued filter operations during Run 193, and even using precoat did not result in sustained acceptable filtrate clarity.

Run 194 was in progress at the end of the year. The reaction conditions were the same as for Runs 191-193, except that sodium carbonate addition was increased to 1.1% of coal feed, or 40 lb/slurry batch.

III. PROCESS DESCRIPTION

A. COAL SLURRY PREPARATION

Figure 1 is a schematic flowsheet of the Wilsonville SRC pilot plant. Pulverized coal, 95% of which is smaller than 200 mesh (74 x 74 microns), is mixed with a process-generated solvent in V101A Slurry Blend Tank. The boiling range of this solvent is 450 to 900°F. The coal-solvent slurry is transferred to V101B Slurry Feed Tank where it is agitated and recirculated to maintain a uniform suspension. P102B Slurry Circulating Pump recirculates the slurry and also feeds P102A or B Slurry Preheater Feed Pump.

Feed gas, which consists of scrubbed recycle gas plus makeup hydrogen, is normally added to the coal slurry upstream of the preheater inlet. (Its composition is usually 85% hydrogen by volume).

The mixture of coal slurry and feed gas flow upward in B102 Slurry Preheater through a 600-ft long helical coil of 1.25-in. schedule 160, 316 SS pipe. The coil is heated by an oil burner located in the bottom of the preheater.

B. COAL DISSOLUTION

The coal slurry-and-gas mixture leaving the preheater flows upward through R101 Dissolver. The dissolver is 23-ft high and one foot in inside diameter, centrifugally cast of high-chrome stainless steel. It can be operated at 800 to 875°F, 1,400 to 2,500 psig, and can provide residence times of 15 to 60 minutes. Outlets are located at the 25, 50, 75, and 100% volume levels to permit changes in residence times. Effluent from the dissolver is cooled to 600-650°F by E102 Dissolver Product Cooler.

Improvements made to the dissolver include the installation of a distributor plate in the bottom (September 1977), a solids density monitoring system (November 1977), and a solids withdrawal system (February 1978). The solids withdrawal system may be operated continuously or intermittently to control solids accumulation. The solids are conveyed to V103 through a 3/4-in. line.

C. HIGH PRESSURE GAS AND SLURRY SEPARATION

The vapor and slurry phases are separated in V103 High Pressure Separator. Vapor from V103 is cooled to about 150°F by E103 High Pressure Separator Cooler and is then passed into V104 High Pressure Vent Separator. Water and organic compounds condensed in E103 are fed through LV430 Letdown Valve to V105 Solvent Decanter. Vapor from V104 includes unreacted hydrogen, light hydrocarbon gases, hydrogen sulfide, and carbon oxides.

D. FILTER FEED PREPARATION

Slurry from V103 is flashed through LV415A or B High Pressure Letdown Valve to V110 Flash Tank. The 115 psig vapor goes to E107 Flash Condenser. The V110 liquid phase flows to V111 Reclaim Tank which serves as a feed reservoir for the batch filtration system. Noncondensed vapors from E107 and vapors from V104 are vented from V105 to K110 Flare. Organic liquids from E107 and V104 are separated from the water phase in V105.

E. FILTRATION

A schematic flow diagram of the filtration system is shown in Figure 2. Undissolved solids are presently removed from the coal solution by F125 United States Filter Corporation (USF) Pressure-Leaf Filter. (Formerly, a Funda Pressure-Leaf Filter was used). The USF unit will accommodate four vertical, circular filter leaves with metal wire screens having a total effective filtration area of 87.2 ft². The filter operates at 480 to 580° F and 150 to 200 psig, with a maximum pressure drop of 100 psi between the slurry inlet and filtrate outlet. The leaves are cleaned by sluicing with wash solvent at high pressures.

The following batch filtration operations are automatically controlled by a programmer:

- precoating or no precoating
- filtration
- washing with sluice wash solvent
- cake blowing
- depressurization
- vacuum flash drying
- cake discharging
- repressurization
- sluicing (2,000 psig max)

High-boiling "process" solvent is used in precoat makeup and deposition. The washing step uses low boiling range (350 to 450° F) "wash" solvent. Nitrogen is used for cake blowing and repressurization.

F. MINERAL RESIDUE PROCESSING

After depressurization, the pressure in the filter is reduced to 1-2 psia for 10 minutes. Most of the residual wash solvent in the filter cake flashes to K115 Dryer Vent Gas Scrubber where it is condensed by a circulating stream of cold solvent.

The cake, now containing less than 5% residual solvent, is cooled and discharged into storage drums. The recovered solvent is pumped to V111 Reclaim Tank.

G. VACUUM FLASH SYSTEM

Filtered SRC solution flows to V120 Vacuum Preheater Surge Drum. P116A or B pumps the solution through B103 Vacuum Preheater to T102 Vacuum Column where it flashes as it enters. The primary function of T102 is to separate SRC from organic liquid boiling at temperatures up to 900°F. Liquid SRC is withdrawn from the bottom, solvent fractions from trays 3 to 8, and vapors from the overhead of T102. The overhead vapor then passes through a vacuum-jet precondenser and light organic condensate is pumped to V164 Feed Tank and then to T104. T102, three feet in diameter, contains valve trays, tunnel cap trapout trays, and Koch-Sulzer packing. Overhead pressure is maintained at 0.3 to 1.5 psia by a two-stage jet, K111 (with pre- and after-condensers). The column feed is heated in B103 to maintain a bottom temperature of approximately 600°F. A portion of the liquid SRC is recirculated and mixed with the material from V120.

H. PRODUCT SOLIDIFICATION

Liquid SRC from T102 is fed to the vibrating water-cooled trays of K125 Product Cooler. Two trays provide a total surface area of 30 ft². The SRC solidifies into brittle sheets which shatter into small fragments. The fragmented SRC is conveyed to storage.

I. CRITICAL SOLVENT DEASHING (CSD)

A schematic flow diagram of the CSD unit is shown in Figure 3. When this system is used as an alternative to filtration, the V111 material is diverted through B103 to T102. The bottoms concentrate from T102 is transferred to the CSD system, which separates the feed into ash concentrate (first stage), deashed SRC (second stage), and a light solvent-containing SRC, called light SRC (LSRC), which can be added to the recycle solvent (third stage). The separations are made using a proprietary deashing solvent operating in the region of its critical temperature and pressure. Under these conditions, most of the SRC is dissolved. Deashing is accomplished in two steps. The ash-laden feed, mixed with solvent at critical conditions, flows to the first-stage settler where the mineral matter and undissolved coal separate as a heavy phase which is withdrawn from the bottom of the settler. Solvent is stripped from the mass and the solids (ash) become a free-flowing powder which is discharged. The density of the first stage overhead is changed and it is then fed to the second stage. The ash-free SRC (heavy phase material) settles and is removed as the underflow. Solvent is stripped

for recycle. The second-stage overhead undergoes a third-stage separation into deashing solvent for recycle (overhead) and LSRC (underflow).

When the CSD unit is in operation, V131B becomes the process solvent surge tank, receiving the third-stage LSRC and the T105 bottoms fraction. V131A tank and the filter surge vessels are bypassed. In-process solvent inventory is approximately 50% less in the CSD operating mode than in the filtration mode.

J. GAS RECOVERY AND RECOMPRESSION

Vapor from V104 High Pressure Vent Separator contains 60 to 80% hydrogen by volume, plus hydrocarbon gases, hydrogen sulfide, and carbon dioxide. The hydrogen sulfide and carbon dioxide are removed by a dilute solution of caustic soda in T101 Hydrogen Scrubber. The exit gas from T101 is scrubbed with water in V106 Recycle Hydrogen Water Scrubber to remove entrained caustic. Excess scrubbed gas is vented to K110 Flare. C104 Fresh Hydrogen Compressor brings pure hydrogen from storage to C102 Hydrogen Recycle Compressor which boosts the feed gas stream to the inlet pressure of B102 Slurry Preheater.

K. LIGHT ORGANICS RECOVERY

Organic liquid from V105 Solvent Decanter and the condensed overhead from T102 are combined in V164 and fed to T104 Light Organics Recovery Column. Components boiling below 350°F are collected in V170 Light Organics Storage Tank. Liquids boiling at 350°F or higher are combined in V160 with material withdrawn from T102 trays 3 and 8.

T104, six inches in diameter, contains two 5-ft sections. The top section contains 1/4-in. "Pro-Pak" packing, and the bottom section is packed with 5/8-in. SS Pall rings. The bottom contains an internal reboiler coil which is heated by Dowtherm. Reboiler duty is 104,000 Btu/hr, and overhead condenser duty is 30,000 Btu/hr.

L. SOLVENT FRACTIONATION

The material from V160 is heated to 220°F by the T105 bottom product stream and is then fed to T105 Fractionation Column. It may enter at tray 10, 12, 14, or 16, depending upon the feed composition.

The T105 bottoms, having a boiling range of 450 to 900°F, is sent to V131 for use as recycle process solvent. The overhead has a boiling range of 350 to 450°F and is recycled to the filter section for use in filter cake washing.

T105 contains 20 valve trays. Heat is supplied by a Dowtherm reboiler, E173. The overhead vapor is condensed by a fan cooler, E170. The reboiler design duty is 3.3 MM Btu/hr and the overhead condenser duty is 2.9 MM Btu/hr.

M. SOLVENT STORAGE

The following solvent storage is provided:

- o V123 Process Solvent Storage, startup and makeup;
- o V131A Recovered Solvent Tank, recycle process solvent (boiling range 450 to 900°F);
- o V131B Process Solvent Surge Tank, recycle process solvent plus CSD third stage (light SRC);
- o V124B Wash Solvent Storage Tank (350 to 450°F boiling range solvent); and
- o V124A Light Solvent Storage Tank, light organic liquid and excess wash solvent as total organic liquid by-product (boiling range normally below 350°F).

IV. OPERATIONS AND RESULTS

A. RUN SUMMARY

The Solvent Refined Coal (SRC) pilot plant at Wilsonville, Alabama, was on-stream for 284 days, or 78% of the 1979 calendar year. The on-stream factor for the fourth quarter was 70.4%. Table 1 summarizes the pilot plant operating hours for the past six years.

The following coals were processed during 1979:

<u>Type of Coal</u>	<u>Mine</u>	<u>Period, 1979</u>	<u>No. of Runs</u>
Kentucky 6 and 11	Pyro	1-29 Jan	2
Kentucky 9	Pyro	29 Jan-20 May	6 (a)
Kentucky 9	Lafayette	20 May-31 Dec	31 (a)

(a) Does not include one run in which both Pyro and Lafayette coals were processed.

Accounts of Run 155 through the start of Run 161 are presented in the first quarterly report, Run 161 through the start of Run 166 in the second, Run 166 through 170 in the third, and Runs 171 through 194 in this report. Operating data are tabulated in each quarterly report. Conditions and results for Runs 171 through 194 are summarized in Tables 2 through 6 of this report. The ranges in operating conditions during the quarter were as follows:

Coal rate, MF lb/hr	150-530
Space rate, lb/hr-ft ³	32-51
Slurry concentration, wt % coal	34-38.4
Gas rate, scfh	6,287-11,162
R101 outlet pressure, psig	1,700 and 2,100
R101 temperature, °F	825
R101 volume in service, %	50 and 75
R101 solids withdrawal, equivalent % of total feed	5-7
Sodium carbonate, lb/batch of coal slurry	5-40

Operating conditions for the two material balance periods completed during the fourth quarter were:

	<u>Run 172</u>	<u>Run 190</u>
Coal rate, MF lb/hr	425	449
Space rate, lb/hr-ft ³	46.2	32.9
Slurry concentration, wt % coal	34.6	37.2
Gas rate, scfh	10,000	10,000
R101 outlet press, psig	2,100	2,100
R101 temperature, °F	824	822
R101 volume in service, %	50	75
Sodium carbonate, lb/batch of coal slurry	40	10

1. Filtration

During the fourth quarter, 595 cycles were completed by the United States Filter Corporation (USF) filter. The mechanical performance of the filter was satisfactory except for major problems with leaf construction. Screen failures caused poor filtrate clarities during some runs.

Specification SRC was produced with and without precoat, but rapid screen blinding made the non-precoat operation impractical. High-pressure sluicing was successfully used to clean the screens.

2. Distillation

The distillation section ran well. Up to 40 lb of sodium carbonate (a corrosion inhibitor) was added per batch of coal slurry in V101A, because corrosion probes indicated significant corrosion rates at lower dosages.

3. Kerr-McGee Critical Solvent Deashing (CSD)

The CSD unit was being modified during the fourth quarter. Before the shutdown, runs were completed using the following feed materials:

- A material balance with feed containing 20% solvent
- H-Coal vacuum bottoms
- Filtered material from the SRC plant
- Unfiltered material from the SRC plant.

B. COAL COMPOSITION AND SLURRY PREPARATION

Table 9 presents analyses of the Kentucky 9 (Lafayette mine) coal processed during the fourth quarter. Selected analytical data for composites of 1979 material balance tote bin feed coal are shown below. Data for the last quarter of

1978 are representative of the Kentucky 6 and 11 coal processed from September 1978 until 29 January 1979. (No material balance data for this coal were obtained in 1979.)

Period	4th Qtr, 1978	1st Qtr, '79	2nd Qtr, '79	2nd Qtr, '79	3rd Qtr, '79	4th Qtr, '79
Coal	Ky 6 & 11	Ky 9				
Mine	Pyro	Pyro	Pyro	Lafayette	Lafayette	Lafayette
Ash, wt %	9.28-9.36	10.4-11.2	12.9-14.8	8.6-8.7	8.4-8.8	8.7-9.3
Chlorine, wt %	0.25-0.29	0.20-0.28	0.28-0.45	0.25-0.29	0.18-0.27	0.19-0.24
Volatile, wt %	30.6-34.2	29.7-31.0	27.8-29.6	27.3-32.2	31.9-38.5	31.6-38.4
H/C ratio	0.86-0.87	0.83-0.85	0.82-0.88	0.85	0.85-0.88	0.85-0.86
Sulfur, wt %						
Pyritic	0.95-1.12	1.11-1.32	1.17-1.61	1.11-1.36	0.75-1.17	0.76-0.97
Sulfate	0.14-0.22	0.03-0.04	0.01-0.14	0.01-0.14	0.04-0.19	0.05-0.14
Sulfide	0.01-0.01	0.01-0.03	0.01-0.02	0.01-0.02	0.01-0.09	0.03-0.05
Organic	1.54-1.65	1.45-2.43	1.75-2.07	1.61-2.67	1.87-1.95	1.86-1.87
Titania, TiO ₂ , in ash, wt %	1.07-2.07	1.25-1.56	1.03-1.12	0.90-1.34	1.04-1.52	1.42-1.55

The coal slurry batches for 37 of the 40 runs during the year were prepared at a nominal 38.5% MF concentration. When the CSD unit was on-line, light SRC (LSRC) from the third stage was added to the V131B recycle solvent in varying amounts.

Spot coal slurry specific gravity data follow:

Coal	Kentucky 6 & 11		Kentucky 9	
Concentration, wt %	<u>38.5%</u>		<u>38.5%</u>	
Temperature, °F	60	150	60	150
Specific gravity				
1st quarter	1.116	1.096	1.116	1.104
2nd quarter	-	-	1.116	1.091
3rd quarter	-	-	1.095	1.065
4th quarter	-	-	1.135	1.096

Slurry viscosity data at 38.5% coal concentration were:

Temp °F	Shear rate sec ⁻¹	Ky 6 & 11 viscosity, cp Jan	Ky 9 viscosity, cp					
			Mar	May	June	July	Oct	Dec
80	20.4	200	278	205	150	93	355	210
	10.2	210	290	220	155	100	360	215
150	20.4	90	60	50	55	35	83	58
	10.2	105	65	60	60	40	90	65
200	20.4	53	40	33	45	23	58	33
	10.2	60	50	40	50	25	65	40

The slurry circulating and transfer pumps performed satisfactorily throughout the year. V101A mixer blades became loose several times during July and August. The mixer unit was completely overhauled in September.

C. PREHEATING AND DISSOLVING

1. B102 Slurry Preheater Performance

Table 11 contains typical performance data for the slurry preheater during the fourth quarter.

a. Coke Formation

It was found that the temperature differences between turn 35 skin and fluid as well as those between turn 37 skin and fluid generally provided reliable indications of coke buildup in the coil. Differential pressure measurements were less useful in predicting coke buildup, as can be seen from the following typical data collected since 11 August (when the furnace was last decoked):

Date	$\Delta t, {}^{\circ}\text{F}$		$\Delta p, \text{psi}$	
	Turn 35	Turn 37	Turns 23-31	Turns 31-37
22 Aug 79	26	29	13.2	4.2
4 Sept	21	36	6.0	6.0
12 Sept	25	45	13.5	6.3
1 Oct	37	45	12.0	6.0
18 Oct	32	52	-	-
14 Nov	36	52	13.5	7.5
15 Nov ⁽¹⁾	35	54	11.1	6.6
16 Nov	36	51	12.0	4.8
19 Nov	36	55	11.4	6.0
20 Nov	39	59	-	7.2
21 Nov	36	64	11.4	6.9
27 Nov	40	65	-	5.4
10 Jan 79 ⁽²⁾	67	64	-	-

(1) Dissolver pressure reduced from 2,100 to 1,700 psig.

(2) B102 plugged on 11 Jan 1979 (1,700 psig dissolver).

The differential pressure in the coil is affected by coke buildup, slurry viscosity, feed rate, and coil temperature. Because of the significant visco-thermal effects which occur in B102 before turn 31 (Ref. 1, 2), the pressure differential between turns 23 and 31 is generally erratic. Although the pressure differential between turns 31 and 35 is more steady, it is still not as reliable an indicator of coke accumulation as the temperature differentials.

In an analysis of all measurements of fluid and skin temperatures along the B102 coil, it was found that apparent heats of reactions and visco-thermal effects caused unpredictable shifts in the temperature differentials below turn 31. However, for turn 31 and above, the temperature differentials appeared to be less affected by these phenomena. Moreover, the temperature differentials at turns 35 and 37 usually increased with operating time following decoking. Although the heat flux was less in turns 35 and 37 than in the lower turns, they were among the hottest turns. A progressive increase in the temperature differentials at these

turns provides a strong indication of coke buildup. Based on the differential temperature at turn 37 on 11 January, immediately prior to coil plugging, it was decided that the high temperature differential readings at turn 37 in late November justified a shutdown to decoke. It is believed that turn 35 and turn 37 temperature differentials will be useful in predicting coil coking in future runs.

b. Preheater Temperature Profile

The B102 Slurry Preheater temperature profiles taken during Run 155, after Run 164 MB and during Run 166 indicated some coke formation in the areas between turns 15 and 27, turns 33 and 35 and around turn 7, respectively (Ref. 4, 5, 6). Coking was confirmed when the coils were cleaned, in these cases. The temperature profile and pressure drop data of other runs during the first three quarters indicated no significant coke formation in the coil.

Figure 6 is a plot of representative data from Table 11 for the fourth quarter. The temperature differential between the skin and fluid is shown as a function of coil fluid temperature. Each data point represents an average of 12 temperature readings over a 24-hr period.

Most of the available data fall reasonably near the trend line as shown. The four points which are above the trend line (in the 600°F fluid temperature range) were from turn 27 before the furnace was decoked in early December. The temperature differential at turn 27 had increased from about 45°F after decoking in August to over 100°F by the end of November. After decoking in December, the turn 27 temperature differential returned to the 45-55°F range. This behavior in the 600°F fluid temperature range suggests low temperature carbonization and consequent coke accumulation. However, due to unpredictable visco-thermal effects, it is difficult to interpret the temperature differential data below turn 31; i.e., at less than 700°F fluid temperature.

D. REACTION

1. Stability

a. Dissolver Solids Inventory

The solids withdrawal system for the R101 Dissolver was in operation during the entire fourth quarter, and the withdrawal rate was maintained at approximately 6 wt % of equivalent slurry feed. The nuclear density gauges on R101 were inoperable. Figure 5 presents a schematic view of the R101 Dissolver with its withdrawal system and the nuclear density gauges. The analyses of selected samples withdrawn during the quarter are shown in Table 12.

The SRC plant was shut down in early December and R101 was inspected. Deposits of solids were found above and below the inlet diffuser and near the product drawoff point. The dissolver had been operating at the 50% level with a coal space rate between 40 and 50 lb/hr-ft³. Table 13 contains the ultimate, proximate, sulfur, and mineral analyses for these deposits. Analyses of dissolver solids performed on-site produced the following results:

Sample	(a)	(b)	(c)	(d)
Identification No.	51390	51367	51392	51391
Amount removed, lb	2.5	2.5	15	5
UC, %	49.0	51.4	53.0	37.0
Ash, %	40.1	42.0	33.6	56.1
SRC, %	3.3	1.3	7.5	5.0
Solvent, % (diff)	7.6	5.3	5.9	1.9

- (a) Beneath the inlet distributor.
- (b) Above the distributor plate.
- (c) On the lower dissolver wall near the distributor.
- (d) On the dissolver wall near the 50% point (drawoff point).

The deposits showed a lower hydrogen-to-carbon ratio and a much higher ash content than the feed coal analyses shown in Table 9. The deposits were agglomerated into cakes, which prevented them from being purged from the dissolver during normal operations or during the subsequent solvent flushes and blowdowns.

b. Solvent Quality

Table 5 shows data for solvent quality from the fourth quarter material balance periods. In September 1979, the solvent quality changed dramatically a few days after 23,000 lb of anthracene oil was added. At that time, the recycle process solvent specific gravity increased from 0.88 \pm 0.010 to 1.033 \pm 0.010; the hydrogen-to-carbon atomic ratio decreased from 1.28 to 1.11; and the microautoclave conversion to THF soluble organics increased from 62 to 68% (based on equilibrium test conditions: 30 minutes reaction time and 2:1 solvent-to-reference coal ratio).

During the fourth quarter, solvent quality deteriorated steadily from 68.2% THF conversion in late September (Run 171 MB) to 63.5% in mid-December (Run 190 MB). The process solvent specific gravity also decreased from 1.033 for Run 171 MB to 1.016 for Run 190 MB, while the hydrogen-to-carbon ratio increased from 1.07 to 1.148 during the same period.

c. Coal Conversion

Coal conversion is defined as the percent of MAF coal converted to cresol-soluble organics. The yield of cresol-soluble organics is calculated daily by a forced ash balance for the reaction products at V110 Flash Tank. The daily conversion data are shown in Tables 2, 3 and 4 and conversion data for the material balance periods are shown in Table 5. Coal conversion averaged above 92% for all runs except Runs 186 through 189. For these runs, the combination of high coal space rate (45-50 lb/hr-ft³) and low dissolver pressure (1,700 psig) with moderate to poor solvent quality (60-65% THF conversion obtained by equilibrium test) contributed to the lower conversion. When conversion was low, the hydrogen consumption was also low, suggesting that operating severity was borderline for this coal.

2. Yields

Yields of SRC based on MAF coal feed were calculated daily by a forced ash balance based on the analysis of dissolved slurry in the V110 Flash Tank. These data are shown in Tables 2, 3 and 4. SRC yields were also calculated from product weights at either the K125 Product Cooler or the CSD unit. Yields calculated in this manner usually require more than 24 hours of data; consequently, the results reported in Tables 2, 3 and 4 represent a 2 to 3-day period when operations were reasonably steady. Although there was much scatter in the daily reported yields, there was a trend toward higher SRC yields whenever coal conversion was low. The forced ash SRC yields show this trend more clearly than do the product rate SRC yields.

Complete product yields (gases, liquids, solids) were calculated for MB periods only. The data for Runs 171, 172 and 190 MB's are shown in Table 6. Two methods of calculation are shown: The first (V110 Short Method) is based on the analysis of a composite dissolved slurry sample from V110, plus analyses and flows of all other streams entering or exiting the reaction section of the SRC plant. The dissolved slurry flow rate is determined by difference, or by forced overall material balance. The second (Process Method), uses the actual SRC product yields to calculate the flow of distillable liquids by forced overall material balance. The analysis of distillable liquids is the same in either method.

The procedure for producing an elementally balanced set of yields is described elsewhere (Ref. 3). Generally, for carbon and hydrogen, the difference between input and output is within 2 to 3%. The elemental analyses of the process solvent, the coal, and the distillate product have large effects on the carbon and hydrogen balances, and small errors in these analyses can greatly effect the elementally balanced hydrogen consumption.

Nitrogen, sulfur, and oxygen seldom balance closely. Consequently, there are likely to be large differences between unadjusted and elementally balanced yields of products which contain substantial amounts of any of these elements.

3. Heteroatom Removal

Oxygen, nitrogen, and sulfur removals can be estimated from elemental analyses of feed coal and SRC as follows:

$$\text{Heteroatom removal, \%} = 100 (A-B)/A \quad (1)$$

where:

A = heteroatom-to-carbon ratio in feed coal

B = heteroatom-to-carbon ratio in SRC

Heteroatom removal calculations for runs made during the fourth quarter are summarized in Table 28.

Total sulfur removal (TSR) can be calculated by the following equation based on the heating value of coal and SRC:

$$\text{TSR, \%} = \left[\frac{(A/C-B/D)}{A/C} \right] \times 100 \quad (2)$$

where:

A = Total sulfur in feed coal, wt %

B = Total sulfur in SRC, wt %

C = Heating value of coal, Btu/lb

D = Heating value of SRC, Btu/lb

The values of TSR calculated by equation (2) are summarized in Table 28.

The sulfur forms in feed coals and SRC products for runs during the fourth quarter follow:

Run	Sulfur Forms, wt %			
	Pyritic	Sulfate	Sulfide	Organic
<u>Feed Coal</u>				
171AB MB	1.09	0.05	0.03	1.99
172A MB	1.03	0.05	0.03	1.98
190AB MB	0.75	0.14	0.05	1.85
<u>CSD feed</u>				
171AB MB	0.09	0.11	0.37	1.12
172A MB	0.13	0.03	0.54	0.70
<u>KM-deashed SRC</u>				
171AB MB	<0.01	0.01	0.02	0.93
172A MB	<0.01	0.01	0.02	0.86
<u>K125 SRC</u>				
190AB MB	<0.01	<0.01	<0.01	0.91

E. FILTRATION

The United States Filter Corporation (USF) vertical-leaf filter processed reaction product slurry made in Runs 173 through 194. The filter has space for four leaves with two screens per leaf and a total screen area of 87.7 ft². Three leaves, with a total screen area of 65.8 ft² were used during these tests. The screens on the leaves were 24 x 110 Dutch weave, calendered to an 83 micron nominal retention size.

During the first part of the year, plates were installed in the front and rear heads, reducing the filter volume to 345 gallons. Equipment was installed for circulating Dowtherm-A through the head cavities to provide additional heat to the filter. A high-pressure sluice header with sluice arms and nozzles to spray between the leaves was also installed in the filter. A high-pressure sluice pump was added to supply wash solvent at pressures to 2,000 psig, flow rates to 34 gpm, and temperatures to 400°F. A precoat tank, V147, was installed so that the tank formerly used as the precoat tank could be used as a sluice receiver or as a wash solvent fill-and-drain tank.

Following is a summary of filtration operations for 1979:

Period, 1979	Run No.	No. of Cycles	Loading, lb/ft ²		Filtration Rate, gph/ft ² (b)		Filtrate Ash range, wt %	Average SRC Ash, wt %
			(a) Precoat	Wet Cake	Second day	Last day		
9-15 Oct	175-7	52	0.35	3.1	3.9	4.4	0.01-0.25	0.55
17-24 Oct	177-9	64	0.35	3.3	4.7	4.4	0.01-0.15	0.49
29 Oct-11 Nov	179-82	155	0.35	2.9	3.6	4.1	0.01-0.12	0.43
16-20 Nov	185	40	0.35	3.4	5.7	3.8	0.02-0.08	0.21
20-27 Nov	186	56	0.35	3.8	3.8	3.9	0.01-0.15	0.20
6-14 Dec	190	61	0.35	3.3	9.3	3.7	0.01-0.14	0.14
14-17 Dec	191	26	None	3.2	2.8	2.1	0.03-0.10	0.14

(a) Precoat was Dicalite Perlite 436.

(b) Rate during filtration.

The filtration cycle developed for precoat and no-precoat operation is as follows:

Filtration Cycle

<u>Operation</u>	Time, Minutes	
	<u>Precoat</u>	<u>No Precoat</u>
Fill with precoat	3	-
Precoat or recirculation	10	1
Remove excess precoat solvent	1	-
Precoat drain	2	-
Fill with filter feed	4	3
Drain filter feed	2	2
Fill with wash solvent	3	3
Wash cake, 3.3 lb solv/lb cake	27	42
Drain wash solvent	2	2
Cake blow	1	1
Depressure	1	1
Dry and break vacuum	9	9
Discharge	3	3
Repressure to 120 psig	3	3
Sluice at 1,200 psig @ 3 rpm	0.2 (each 25 cycles)	6 (each cycle)
Valve sequencing time	<u>2</u>	<u>2</u>
Total downtime	73	78
Filtration time	<u>72</u> (4.4 gal/ft ²)	<u>116</u> (3.8 gal/ft ²)
Total cycle time	145	194
Cycle rate, lb/hr-ft ²	15.2	9.7
Sodium carbonate, % of coal	0.25	0.38
Mass of filter feed, lb	2,703	2,283

During the precoat experiments, Dicalite Perlite 436 precoat was slurried with process solvent (containing 0.5 wt % solids) in mix tank V140. The cold slurry was pumped to the hot precoat tank V147 for precoating the filter. The process solvent was recirculated through the filter at 100 gpm (1.5 gpm/ft²) during the 10-minute precoat step. The pressure drop across the leaves and precoat at 100 gpm was normally 8 to 15 psi. If the dumping of ash from the previous cycle was incomplete, the residual ash would mix with the precoat and deposit on the leaves, resulting in a higher precoat differential pressure.

Cycle downtime was reduced by draining the filter through the 3-in. drain valve XV659. For precoat operation, it was found that sluicing during each cycle was not necessary. This reduced the downtime by 6 minutes.

V178 Light Solvent Storage Tank contained clean solvent used for sluicing and also for cake washing. To avoid contamination of V178, the sluice receiver tank V141 was used to fill and drain the filter of wash solvent. On cycles in which the filter was sluiced, the wash solvent and cake solids from V141 were used to wash the filter cake. On cycles without sluicing, the filter was filled from V141, then a fixed amount of solvent from V178 was pumped through the filter for washing the cake. (Since some filter feed remains in the filter prior to washing, some SRC accumulates in V141. Solids sluiced from the leaves may be present as 8-12% slurry in V141).

The filter cake was dried for 5-8 minutes at 22 in. of mercury vacuum during October and November. Wear on the steam jet venturi resulted in less vacuum (15-20 in. of mercury) in December.

Nitrogen is used during the drain steps to hold the cake or precoat in place and to repressurize the filter after vacuum drying. The nitrogen consumed was essentially that required to displace the liquid from the filter with little going through the cake. Nitrogen rates during various stages of the filtration cycle were:

	<u>scfm</u>	<u>scf (total)</u>
Precoat drain	104	220
Filter feed drain	104	220
Wash solvent drain	104	220
Cake blow	104	104
Pressure to filter from atm. to 130 psig	104	<u>218</u>
Total nitrogen/cycle		982

1. Filtrate Clarity

When Na_2CO_3 was mixed with feed coal to inhibit corrosion, the ash content of the filtrate was sometimes higher than the cresol-insoluble content, as shown below:

<u>Samples (SN)</u>		<u>% Ash</u>	<u>% Cresol Insolubles</u>
F-125-179-46C	(49680)	0.07	0.02
-32	(49557)	0.08	<0.01
-53A	(49689)	0.05	<0.01
-53B	(49690)	0.20	<0.01
F-125-180-2	(49745)	0.09	0.02
-4	(49754)	0.05	0.01
-9	(49782)	0.10	0.02
-10	(49783)	0.07	<0.01
-20	(49839)	0.09	<0.01
V-120	(49611)	0.07	0.016
	(49626)	0.07	0.015
	(49920)	0.06	-
	(49963)	0.10	-
	(50081)	0.09	-
	(50127)	0.09	-
V-110 filtrate	(49658)	0.06	-
B-103 feed	(49892)	0.06	0.03

This may have resulted from dissolved materials from ash in the process solvent or particulate ash fine enough to pass through lab filter papers used for determination of cresol-insolubles. Ignited ash from the filtrate produced a yellowish flame and a significant amount of the ash dissolved in deionized water. The ash solute was tested with 0.141-normal silver nitrate, 90% nitric acid and 0.01 molar barium chloride. The tests showed that sulfate ion (SO_4^{2-}) was present, but that neither sulfide (S^{2-}) nor chloride (Cl^-) was present.

Solubility and reaction studies of Na_2CO_3 , Perlite (precoat material), and $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$ (hydrated sodium sulfide) in process solvents were conducted in open beakers or in microautoclave bombs. The results are summarized below:

(1) Open beaker test (filtered hot)

<u>Sample</u>	<u>Vac dried</u> <u>Na₂CO₃</u>	<u>Vac dried</u> <u>Perlite</u>	<u>Na₂S•9H₂O</u>
Salt, gm	2.02	1.98	2.02
Process solvent, gm (ml)	240.7(250)	234.9(250)	247.8(250)
Temp, °F	292-325	310-360	282-294
Time, hr	2	2.3	1
Recovery of salt, gm (%)	1.95(96.7)	1.95(98.0)	0.53(26.8)
Solubility in process solvent, wt %	0.025	0.017	0.05 ^(a)
Ash % in			
Original salt	96.4	96.5	-
Treated salt	99.0	98.0	-
Process solvent	0.005	0.005	0.005
Filtrate	0.004	0.005	0.12

(a) Calculated on water-free basis.

(2) Tubing bomb test (filtered at room temperature)

<u>Sample</u>	<u>Vac dried</u> <u>Na₂CO₃</u>	<u>Na₂S•9H₂O</u>	<u>Vac dried</u> <u>Na₂CO₃</u>	<u>Vac dried</u> <u>Na₂CO₃</u>
Salt, gm	2.02	2.20	2.01	2.01
Process solvent, gm	12.61	12.54	12.66	11.65
Distilled water, gm	-	-	1.05	0.57
Temp, °F	760-770	760-770	750-760	750-760
Time, hr	0.5	0.5	0.5	0.5
Recovery of salt, gm (%)	2.00(98.9)	1.17(53.1)	-	2.04(101.5)
Ash in filtrate, %	<0.01	0.011	<0.01	<0.01
Elemental analysis of filtrate, %				
Sulfur	0.27	0.30	0.27	0.27
Nitrogen	1.84	1.52	1.65	-
Carbon	87.68	88.75	88.20	-
Hydrogen	8.84	9.08	8.91	-

It appears that $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$ is very soluble in process solvent, but Na_2CO_3 and Perlite are only slightly soluble.

SRC pilot plant data (in a hydrogen atmosphere) suggest that Na_2CO_3 reacts with nitrogen and sulfur compounds in the process solvent. It was observed that addition of Na_2CO_3 reduces NH_3 and H_2S production and decreases the nitrogen content of the process solvent.

Process solvent distillates produced after reaction with Na_2CO_3 , $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$, and H_2O in the tubing bomb test were analyzed by gas chromatography. The results are summarized below:

GC Peak (B.P.)	distillate	V131B								
		wt %	wt %	Δ %	wt %	wt %	Δ %	wt %	wt %	Δ %
(1) major peaks										
460°F (quinolinc)	2.38	2.34	-0.04		2.28	-0.10		2.16	-0.22	
465.8 (2-methylnaphthalene)	4.47	4.70	+0.23		5.77	+1.30	+29	3.90	-0.57	
492.0 (biphenyl)	7.44	7.65	+0.21		7.37	+0.33		8.12	+0.68	
505.1	3.21	3.29	+0.08		4.10	+0.89	+28	2.97	-0.24	
517.6	2.86	2.85	-0.01		2.96	+0.10		2.60	-0.26	
531.1 (acenaphthene)	3.59	3.71	+0.12		3.74	+0.15		3.56	-0.03	
538.6 (dibenzofuran)	3.64	3.80	+0.16		3.85	+0.21		3.49	-0.15	
568.4 (fluorene)	6.05	6.04	-0.01		6.36	+0.31		6.09	+0.04	
583.4	3.22	3.15	-0.07		4.10	+0.86	+28	3.16	-0.06	
643.0 (phenanthrene)	2.81	2.61	-0.20		2.96	+0.15		2.84	+0.03	
665.4 (9-methylanthracene)	3.71	3.47	-0.24		4.05	+0.34		3.77	+0.06	
721.9 (fluoranthene)	2.21	2.09	-0.12		1.88	-0.33	-15	1.98	-0.23	
740.0 (pyrene)	1.57	1.53	-0.04		1.92	+0.35	+22	1.89	+0.32	+20
(2) most changed peaks								4.74(2)		7
401.5	0.39	0.39	0.00		0.32	-0.07	-18	0.58	+0.19	+49
405.0 (tetralin)	1.99	2.05	+0.06		0.86	-1.13	-57	2.10	+0.11	
424.0 (naphthalene)	1.20	1.23	+0.03		0.70	-0.50	-41	1.18	-0.02	
435.1	0.82	0.92	+1.10		1.03	+0.21	+25	0.85	+0.03	
442.0	2.07	2.08	+0.01		1.01	-1.06	-51	2.03	-0.04	
602.5	1.59	-	-1.59	-100	-	-1.59	-100	-	-1.59	-100
703.8	2.30	1.96	-0.34	-15	1.52	-0.76	-34	1.52	-0.78	-34
total	57.52	55.86			57.18			54.79		
increase (+)		+1.00			+5.22			+1.46		+2.21
decrease (-)		-2.66			-5.56			-4.19		-4.27
net		-1.66			-0.34			-2.73		-2.06
(3) GC boiling fraction distribution, wt %										
100-350°F	0.0	0.03			0.24			0.23		
350-450	6.95	7.09			4.39			7.22		
450-500	21.90	22.63			22.00			22.51		
500-550	14.95	15.54			15.60			14.29		
550-650	24.75	24.62			26.21			24.43		
650-EP	31.45	30.09			31.55			31.32		

The data show that $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$ has a greater effect on compounds in the process solvent than does Na_2CO_3 . This is most clear from the peaks of 602.5 and 703.8°F BP.

In order to determine the effects on cresol-insolubles of fine particulate ash passing through a lab filter paper, two experiments were performed:

- Lab filtration tests were made with two filter papers (Whatman #42, good for particles larger than 2.5μ and Glass microfibre (GF/C), good for particles larger than 1.2μ); and
- Ash analyses of lab-filtered filtrates and residues.

Both experiments indicated that no significant amounts of particles larger than 1.2μ were present in the F125 filtrate and that 92-94% of the original ash was recovered in the lab-filtered filtrate. Further studies will be required to determine the effects of particles smaller than 1.2μ on the ash content of the F125 filtrate.

2. Filtration Data

Filtration tests performed include cake compressibility, cake washing, sluice nozzle and sluice pressure variations, and bench-scale filtrate clarity.

The filtration rate data for November and December are presented in Figures 7 and 8. The filtrate volumes and the filtration times were fitted to the empirical equation, $t/v = a_0 + a_1 v$, where t is time in seconds, and v is the filtrate volume in cubic feet.

The time required to filter 4.4 gallons of filtrate per square foot of filter area is shown in Figures 7 and 8. Theoretically, constants a_0 and a_1 are related to the filter medium resistance, R_m , and to the specific cake resistance, α , as follows:

$$R_m = \frac{a_0 A}{\mu} \frac{\Delta P}{g c}$$

$$\alpha = \frac{2a_1 A^2}{\mu c} \frac{\Delta P}{g c}$$

where

A = filtration area, ft^2

ΔP = filtration pressure drop, lb/ft^2

μ = viscosity of filtrate, $\text{lb}/\text{ft}\text{-sec}$

c = weight of solids per volume of filtrate, lb/ft^3

The viscosities of the filtrates are presented in Figures 9 and 10.

The coefficient a_0 varied considerably (see Figures 7 and 8). Actually, a_0 is significant only during no-precoat operation, as in Run 191. For the no-precoat tests, filter feed is circulated through the screen for 1-3 minutes to develop a small cake. Since the calculations are done without incorporating the volume of material filtered during the 1-3 minute period, the coefficient a_0 reflects the resistance of cake deposited in the circulation period as well as the resistance of the medium.

In December (see Figure 8), the filtration rate at the start of Run 190 was very high and stabilized at 3.77 gal/hr-ft² (70 minutes for 4.4 gallons of filtrate per square foot). During Run 191, the coefficient a_1 increased, suggesting a loss of effective filtration area. The screens were not clean when inspected. During Run 192, a wider angle spray nozzle, (Spraying Systems Co. 6510 tungsten carbide) was used. The coefficient a_1 increased rapidly, indicating further loss of screen area. Inspection revealed that the filter leaves were not clean and that the screen contained ridges of ash. The best sluicing results were obtained with Spraying Systems Co. 4006 TC nozzles at 1,200 to 1,500 psig.

For Runs 179 and 186, filtration cycles were run at 25, 50 and 90 psig. For both runs, the precoat loading was 0.35 lb/ft² of Dicalite Perlite 436. Filtration area was 65.8 ft², and viscosity was assumed to be 1 centipoise. The test results were as follows:

<u>Cycle</u>	<u>ΔP, psig</u>	<u>Temp, °F</u>	<u>$R_m \times 10^{-10}$, ft⁻¹</u>	<u>$\alpha \times 10^{-12}$, ft/lb_m</u>
179-34	90	542	3.4	2.02
-35	90	543	83	1.36
-36	48	541	40	1.34
-37	47	540	21	1.58
-38	25	533	26	1.17
-39	25	536	11	1.09
186- 9	90	551	55	1.73
-10	90	552	-1.5	1.99
-11	50	551	6	1.45
-12	50	551	5	1.17
-13	25	552	2.4	0.806
-14	25	550	0.2	0.845

A relationship between α and the cake pressure drop, ΔP , is given by:

$$\alpha = \alpha_0 \Delta P^S$$

where α_0 and S are empirical coefficients with S being the compressibility coefficient.

The cake from Run 186 was more compressible than the cake from Run 179.

$$\text{Run 179, } \alpha = 2.68 \times 10^{11} \Delta P^{0.444}$$

$$\text{Run 186, } \alpha = 1.08 \times 10^{11} \Delta P^{0.633}$$

Values of α are given in Figure 11, together with data obtained in prior years. The empirical constants, α_0 and S are plotted in Figure 12. The specific cake resistances at 1 psig (α_0) fall along the line of the 1978 data, but the compressibility coefficients were higher.

Cake washing tests were made for three filter cycles. Tank V141 was used to provide wash solvent for the tests. During regular operation, V141 is used as a fill-and-drain tank for the cake washing step. Since some filter feed remains in the filter prior to washing, some SRC accumulates in V141. The results from the washing tests indicate that, after washing with 3 lb of wash solvent per lb of discharged cake, no additional SRC could be removed by further washing with solvent from V141. This is illustrated below:

Cycle	181 - 30			181 - 31			186 - 12		
Temperature, °F	530 - 480			526			544 - 526		
During wash	483 - 456			485 - 454			509 - 472		
During drying									
Vacuum, In. Hg ^(a)				21.5			21		22
Elapsed time in wash step, min.	1	6	(b)	3	8	12	(b)	4	8
Amount of wash solvent, 1b wash solvent/lb filter cake	0.5	1.9		1.7	3.1	4.2	4.4	1.2	2.5
Analyses of wash filtrate									
CI, %	0.02	0.01	-	0.01	-	-	-	0.04	0.01
Ash, %	0.03	0.04	-	0.01	0.03	0.02	-	0.02	0.01
SRC, %	13.4	10.2	-	10.8	6.1	5.6	-	7.8	5.7
Δ SRC, % difference ^(c)	8.3	5.1	-	5.0	0.3	-0.2	-	4.9	2.8
Analyses of cake									
Solvent, %			10.2				9.8		5.2
SRC, %			-				4.7		2.0

(a) Vacuum at end of drying.

(b) End of wash.

(c) Percent difference between the percent SRC in the wash solvent pumped to the filter and the wash solvent out of the filter.

In Figure 13, the cake washing time (time required to pass 737 lb of wash solvent through the cake at 46-50 psi) is correlated with the product of cake weight and specific cake resistance. Cake washing time was higher after filtering at 90 psi than at 25 psi due to cake compression.

A filter cake sample from cycle 190-38 was used to determine the cake density. The surface of the sample was wetted by rubbing it under water. The cake was placed in various concentrations of salt water to determine its specific gravity.

Drying conditions for the filter cake and the boiling range distribution of the solvent distilled from the filter cake are presented in Tables 17, 18, and 19.

The 0.01 ft² bench filter was run during Run 182 to test the calendered 24 x 110 mesh screen without precoat. For this run, with 20 lb of sodium carbonate per tote bin of coal, the percent ash in the filtrate was high.

Filter at 500-550°F and 140 psig

Filtration time, minutes	Ash as-is in filtrate, %
0-3 + 11-16 (a)	0.19
3-11	0.14
16-32	0.13
32-47	0.17
37-62	0.12

(a) Filtrate for these two time intervals was inadvertently combined during test.

The cake was 3-in. thick at the end of the test.

Another 0.01 ft² bench filter test was made to determine if HF-75 double Dutch weave could be used as a no-precoat screen. The test was run during Run 188 with 5 lb of sodium carbonate per tote bin. The HF-75 retained filter feed solids and gave lower solids in the filtrate than was obtained during Run 182.

0.01 ft² filter at 480-530°F, 130 psig

Time, min	Filtrate collected, gm	Ash as-is, %
0-3	33.6	0.75
3-6	15.1	0.07
6-16	37.2	0.05
16-35	40.8	0.04

The filter cake collected during this test weighed 16.5 grams, was 7/8-in. thick, and was composed of 29.8 wt % ash, 60.3 wt % CI, and 14.6 wt % SRC.

A sample of filter feed from Run 182 was sent to Micron Data Laboratories for particle size analysis by X-ray sedimentation. A summary of their results follows:

Equivalent spherical diameter, microns	Cumulative mass finer than diameter indicated, %
40	99
32	98
25	94
20	88
10	64
7	51
5	39
3	17
2	8
1	1

3. Screen Performance

The mechanical performance of the USF filter has been good. The shaft seals on the conveyor, on the leaf shaft, and on the sluice shaft have withstood the operating conditions. The high-pressure sluice pump, spraying nozzles and sluice arm rotation assembly have also performed well. The most difficult remaining problem in the filtration program has been the development of leaves that will withstand processing conditions. Using solution-annealed screens to solve the corrosion problem looks promising. A summary of filter inspections, sluicing conditions and leaves used to-date

is presented in Table 32.

The 3-ply leaf is a diffusion-bonded laminate of calendered 24 x 110 Dutch weave on the outside and a 16-mesh drainage screen between the Dutch and the perforated plate. This 3-ply sandwich is laid on each side of the tubular slit screen at the leaf center. The tubular slit screen has an 8-mesh spacer screen on each side. The outer layers are spot-welded to metal rods in the tubular slit screen. The 2-ply leaf is the same as the 3-ply leaf except that the sandwich consists of the outer Dutch weave and a calendered 6-mesh diffusion-bonded together. The non-diffusion bonded leaves have the same three layers as the 3-ply leaf.

F. SOLVENT RECOVERY

The distillation systems are shown schematically in Figure 1. Reaction product is separated in T102 Vacuum Column into SRC and solvent (350-900°F boiling range). The solvent is fed into T105 Fractionation Column, which separates the feed into wash solvent (350-450°F boiling range) and process solvent (450-900°F boiling range). The T102 overhead is combined with material from V105 Solvent Decanter and fed to T104 Light Solvent Recovery Column. T104 separates the feed into an overhead fraction boiling from 100-350°F and a bottom fraction which is combined with T105 feed.

1. T102 Vacuum Column

T102, a 3-ft diameter column, contains three valve trays, a section containing Koch-Sulzer packing and two tunnel-cap trapout trays. Vacuum is provided by a two-stage jet vacuum system. The lower limit of pressure is 0.3 psia. The feed is heated in B103 before being flashed in the column.

The column separates SRC from liquids boiling up to 900°F. The top temperature is adjusted to keep the amount of light organics at less than 3% of the feed to T105 Solvent Fractionation Column.

Typical operating data for the column during Runs 171, 172 and 190 material balance periods are given in Table 20. Summary operating and performance data for the material balance runs during 1979 follow:

<u>Run</u>	<u>159</u>	<u>160</u>	<u>161</u>	<u>162</u>	<u>163</u>	<u>164</u>	<u>164A</u>
T102 pressure, psia	0.5	0.4	0.5	0.6	0.6	0.9	0.7
T102 bottom temperature, °F	580	580	577	551	557	566	559
Overhead temperature, °F	192	192	193	192	193	201	203
Overhead rate, lb/hr	14.0	27.2	137.0	110.7	65.8	53.0	215.0
Distillate in SRC							
@ 500°F, 0.1 mm Hg.	4.1	1.9	2.1	1.3	4.9	1.8	3.1
@ 600°F, 0.1 mm Hg.	6.6	14.7	6.5	6.1	10.3	8.9	12.6
Melting point of SRC, °F	311	329	320	300	307	303	352
% oil (SRC)	26.7	26.1	21.4(a)	26.2	26.5	20.3	22.9
% asphaltene (in SRC)	59.9	32.1	63.2(a)	39.4	32.4	47.9	31.9
% preasphaltene (in SRC)	13.4	41.8	15.4(a)	34.4	41.1	31.8	45.2

<u>Run</u>	<u>166C</u>	<u>167</u>	<u>168</u>	<u>171</u>	<u>172</u>	<u>190</u>
T102 pressure, psia	8.1	1.5	1.5	0.3	0.3	0.4
T102 bottom temperature, °F	554	557	532	561	497	596
Overhead temperature, °F	203	212	214	191	203	190
Overhead rate, lb/hr	92.9	130.0	189.0	153.0	170.0	47.2
Distillate in SRC						
@ 500°F, 0.1 mm Hg.	4.5	4.9	11.0	1.6	22.0	2.9
@ 600°F, 0.1 mm Hg.	11.2	12.5	14.1	9.25	24.0	4.8
Melting point of SRC, °F	337	332	327	337	193	>518
% oil (SRC)	21.2	27.2	24.3	24.0	32.7	31.2
% asphaltene (in SRC)	34.0	27.1	34.0	35.6	32.5	45.0
% preasphaltene (in SRC)	44.8	45.7	41.7	40.4	34.8	23.8

(a) SRC from CSD unit.

2. T105 Solvent Fractionation Column

T105 contains twenty valve trays. Heat is supplied by Dowtherm in a reboiler, E173. The overhead vapor is condensed by a fan cooler, E170. The column separates process solvent from wash solvent and light organic liquid present in the feed. Process solvent is obtained as the bottom product.

Feed to T105 consists of streams from trays 3 and 8 of T102 and T104 bottoms. The feed is heated by the T105 bottom stream in a double pipe heat exchanger, E174.

The hot feed enters tray 12. Overhead wash solvent is recycled to the filter system (when operating). Part of the bottom stream is vaporized in the reboiler and returned to the column. The bottoms product (after heat exchange with the column feed) is sent to V131B, to be recycled with the slurry feed to V101A. The solvent specifications are: less than 5% boiling below 450°F in the process solvent and 95% boiling below 450°F in the wash solvent.

T105 was down during material balances 159 through 164. The feed compositions for the remaining material balance runs during 1979 were:

Run	166A	166C	167	168	171	172	190
<u>Feed composition, wt %</u>							
IBP-350°F	1.0	0.4	Down	Down	0.8	0.8	4.2
350-450°F	8.0	4.4	Down	Down	8.0	10.1	34.5
450-EP°F	91.0	95.2	Down	Down	91.2	89.1	61.3
Filter on?	No	No	Down	Down	No	No	Yes

Overhead and bottom product specifications for other material balance runs during the year were:

Run	166A	166C	167	168	171	172	190
<u>Overhead Product</u>							
IBP-350°F	13.6	15.2	Down	Down	10.1	6.4	4.2
350-450°F	82.8	81.2	Down	Down	88.3	87.3	82.3
450-EP°F	3.6	3.6	Down	Down	1.6	6.3	13.5
<u>Bottom Product</u>							
IBP-350°F	0	0	Down	Down	0	0	0
350-450°F	4.1	3.4	Down	Down	2.9	2.9	0.8
450-EP°F	95.9	96.6	Down	Down	97.1	97.1	99.2

Data for Runs 171, 172 and 190 MB periods are given in Table 23. The wash and process solvent analyses and compositions are given in Table 27. The process solvent and wash solvent distillation curves are shown in Figures 15 through 21.

3. T104 Light Organics Recovery Column

T104 removes light organic liquid from heavier components. The feed is a mixture of T102 overheads, collected in the vacuum jet precondenser hot well and the V105 Solvent Decanter overflow stream.

Typical data for Runs 171, 172 and 190 are found in Table 21. The overhead stream specification is 10% boiling above 350°F. The overhead product is condensed and collected in V170. The bottom stream is sent to T105 feed. Distillation curves for light organic liquid are shown in Figures 22, 23 and 24. A summary of overhead and bottom stream compositions for T104 during 1979 follows:

Run	159	160	161C	162	163	164	
<u>Flow Rates</u>							
Feed	-	166	297	315	275	-	
Overhead	-	14	14	17	17	-	
<u>Overhead Composition, %</u>							
IBP-350°F	79.2	57.8	98.7	92.2	92.2	-	
350-450°F	20.8	42.2	1.3	7.8	7.8	-	
<u>Bottoms Composition, %</u>							
IBP-350°F	{ sent to TJ102 }	4.5	5.1	8.2	8.0	-	
350-450°F		34.7	38.1	32.5	38.8	-	
450-EP°F		60.8	56.8	59.3	53.2	-	
Run	166A	166C	167	168	171	172	190
<u>Flow Rates, lb/hr</u>							
Feed	520	225	258	355	369	406	165
Overhead	8	7	13	18	15	10	13
<u>Overhead Composition, %</u>							
IBP-350°F	93.9	100.0	100.0	96.9	91.4	92.6	98.9
350-450°F	6.1	0	0	3.1	8.6	7.4	1.1
<u>Bottoms Composition, %</u>							
IBP-350°F	0.9	1.5	7.2	3.4	1.1	1.0	6.6
350-350°F	9.5	8.9	17.9	14.5	15.1	13.5	28.3
350-450°F	89.6	89.6	74.9	82.1	83.8	85.5	67.1

4. Solvent Inventory

The solvent yields for Runs 171, 172 and 190 material balance periods are shown below:

<u>Run</u>	<u>171 MB</u>	<u>172 MB</u>	<u>190 MB</u>
Date, 1979	28 Sept	1 Oct	12-14 Dec
Yields, % MAF coal			
Process solvent	24.61 (16.45) ^(a)	16.15 (-10.60) ^(a)	22.48
Wash solvent	0.52	3.69	5.04
Light organic liquid	2.96	2.93	2.58

(a) Yields in parentheses are calculated with solvent left in SRC.

Other runs during the fourth quarter did not include a material balance period; thus, no accurate solvent yield data were available for these runs.

Total solvent inventories for the quarter were:

<u>Date, 1979</u>	<u>1 Oct</u>	<u>31 Dec</u>	<u>Change</u>	<u>Composition, (a) wt %</u>		
				<u>< 350°F</u>	<u>350-450°F</u>	<u>>450°F</u>
<u>Inventory, M 1b</u>						
Light organic liquid						
in tanks	19.2	2.5				
in drums	-	-				
shipment	-	41.9				
total	19.2	44.4	+25.2			
5.4	76.9	17.7				
Wash solvent						
in tanks	84.1	68.2				
in drums	-	-				
shipment	-	-				
total	84.1	68.2	-15.9			
5.2	81.1	13.7				
Process solvent						
in process	25.4	42.2				
in storage	27.3	39.8				
in drums	3.2	-				
shipment	-	1.6				
make-up	52.0	41.3				
total	107.9	126.5	+18.6			
0	2.1	97.9				

(a) Gas chromatograph analyses.

The composition of the light organic liquid is based on material from the V124A Light Organic Liquid Storage Tank. This liquid comes directly from V170, T104 overhead storage tank. The analyses of T104 overhead consistently show 100% boiling below 350°F.

The reaction section conditions and solvent yields for the material balance periods run with Kentucky 9 coal are presented below:

Run Date, 1979	159 MB 9 March	160 MB 25 March	161C MB 9 April	162A MB 26 April	163A MB 11 May
<u>Reaction Section Condition</u>					
Dissolver pressure, psig	1,700	1,700	1,700	2,100	1,700
Dissolver temp., °F	825	825	825	825	825
Dissolver volume, %	50	75	75	75	75
Space rate, lb/hr-ft ³	50	40	40	40	40
V110 asphaltene/preasphaltene ratio in SRC	1.57	0.99	0.95	3.21	1.68
Solids withdrawal?	No	No	Yes	Yes	Yes
<u>Solvent yields, % MAF coal</u>					
Process solvent	15.35 (11.65) (a)	18.80 (12.65) (a)	7.26 (1.24) (a)	17.29 (11.92) (a)	14.28 (7.91) (a)
Wash solvent	3.31	4.05	8.38	4.22	5.98
Light organic liquid	3.85	1.66	1.09	1.43	1.74
Run Date, 1979	164 4 June	166A 21 July	166C 2 Aug	167 9 Sept	168 12 Sept
<u>Reaction Section Condition</u>					
Dissolver pressure, psig	-	1,700	1,700	2,100	2,100
Dissolver temp., °F	-	825	825	825	825
Dissolver volume, %	-	75	75	50	50
Space rate, lb/hr-ft ³	-	38	38	50	50
V110 asphaltene/preasphaltene ratio in SRC	1.15	0.79	0.91	1.36	0.90
Solids withdrawal?	-	Yes	Yes	Yes	Yes
<u>Solvent yields, % MAF coal</u>					
Process solvent	15.46 (3.45) (a)	34.81 (24.83) (a)	28.05 (18.99) (a)	20.15 (8.84) (a)	25.77 (13.40) (a)
Wash solvent	-9.53	1.86	1.97	2.02	1.96
Light organic liquid	1.84	1.40	2.19	2.25	1.86
Run Date, 1979	171 28 Sept	172 1 Oct	190 12-14 Dec		
<u>Reaction Section Condition</u>					
Dissolver pressure, psig	2,100	2,100	2,100		
Dissolver temp., °F	825	825	825		
Dissolver volume, %	50	50	75		
Space rate, lb/hr-ft ³	50	50	34		
V110 asphaltene/preasphaltene ratio in SRC	1.46	1.36	1.89		
Solids withdrawal?	Yes	Yes	Yes		
<u>Solvent yields, % MAF coal</u>					
Process solvent	24.61 (16.48) (a)	16.15 (-10.60) (a)	22.48 (19.71) (a)		
Wash solvent	0.52	3.69	5.04		
Light organic liquid	2.96	2.93	2.58		

(a) Yields in parentheses calculated with solvent left in SRC.

5. Solvent Boiling Range Compositions

Typical process solvent boiling ranges and compositions for Kentucky 9 coal during the year were:

<u>Run</u> <u>Date, 1979</u> <u>Boiling Fraction, wt %</u>	<u>159</u> <u>9 March</u>	<u>160</u> <u>25 March</u>	<u>161</u> <u>9 April</u>	<u>162</u> <u>26 April</u>	<u>163</u> <u>11 May</u>	<u>164</u> <u>4 June</u>
IBP-450°F	3.0	3.4	4.0	6.1	5.6	31.1
450-550°F	34.5	24.9	30.7	37.1	33.9	35.3
550-650°F	30.5	29.8	29.1	27.5	27.5	16.8
650°F-EP	32.0	41.9	36.2	29.4	33.0	16.8
Carbon, %	88.7	88.3	87.0	86.2	86.7	85.1
Hydrogen, %	8.7	8.6	8.9	8.7	8.7	8.9
H/C atomic ratio	1.092	1.084	1.139	1.123	1.117	1.164
Specific gravity	1.033	1.07	1.02	1.01	1.00	1.00
T102 bottoms temp, °F	580	580	577	551	557	566
T102 pressure, psig	0.5	0.4	0.5	0.6	0.6	0.9
<u>Run</u> <u>Date, 1979</u> <u>Boiling Fraction, wt %</u>	<u>166A</u> <u>21 July</u>	<u>166C</u> <u>2 Aug</u>	<u>167</u> <u>9 Sept</u>	<u>168</u> <u>12 Sept</u>	<u>171</u> <u>25 Sept</u>	<u>172</u> <u>1 Oct</u>
IBP-450°F	4.6	3.2	5.6	4.4	5.9	3.0
450-550°F	46.7	48.0	45.1	45.9	35.9	42.5
550-650°F	28.2	29.9	25.1	25.0	27.4	27.7
650°F-EP	20.5	18.9	24.6	24.7	30.8	26.8
Carbon, %	87.1	87.1	87.2	86.6	88.6	88.4
Hydrogen, %	9.1	9.1	9.3	9.2	8.2	8.4
H/C atomic ratio	1.163	1.163	1.187	1.182	1.030	1.058
Specific gravity	1.00	1.00	1.00	1.00	1.03	1.02
T102 bottoms temp, °F	559	554	557	532	561	497
T102 pressure, psig	0.1	1.1	1.5	1.5	0.3	0.3
						596
						0.4

At similar T102 operating pressures and with continuous operation from run to run, as the T102 bottom temperature decreases, the percentage of 650°F-EP material decreases.

If the temperature is held constant and the pressure is decreased (vacuum is increased), the 650°F-EP solvent content of T102 bottoms increases.

<u>Run</u>	<u>171</u>	<u>172</u>	<u>162(a)</u>	<u>163(a)</u>
<u>Date, 1979</u>	<u>28 Sept</u>	<u>1 Oct</u>	<u>26 Apr</u>	<u>11 May</u>
T102 pressure, psia	0.3	0.3	0.6	0.6
T102 bottoms temp, °F	561	497	551	557
650°F-EP	30.8	26.8	29.4	33.0

(a) T105 down.

At higher temperatures and lower absolute pressures, more solvent would boil off the SRC fraction in T102 and become part of the process solvent.

<u>Run</u>	<u>159(a)</u>	<u>160(a)</u>	<u>166A</u>	<u>166C</u>
Date, 1979	9 March	25 March	21 July	7 Aug
T102 pressure, psia	0.5	0.4	0.7	1.1
T102 bottoms temp, °F	580	580	559	554
650°F-EP	32.0	41.9	20.5	18.9

(a) T105 down.

G. KERR-MCGEE CRITICAL SOLVENT DEASHING

Prior to the CSD unit shutdown in October for modifications, 388.1 tons of T102 bottoms and 7.8 tons of H-Coal vacuum still bottoms were processed. In the 1979 program, emphasis was placed on determining the effects of R101 Dissolver conditions and of T102 Vacuum Column conditions on SRC recovery in the CSD unit.

The effect of dissolver volume (coal space rate) was studied during Runs 156, 159 and 160 using dissolver volumes of 100%, 50%, and 75%, respectively. The corresponding SRC recoveries (average of forced ash balance and of normalized balance) were 85%, 72%, and 82%. The ratios of benzene solubles to preasphaltenes (benzene insoluble but cresol soluble) in the feed were 3.0, 2.0, and 2.6, respectively.

The effect of dissolver pressure on SRC recovery was also studied. Runs 159 (1,700 psig) and 171 (2,100 psig) resulted in average SRC recoveries of 72% and 79%, respectively. The ratio of benzene solubles to preasphaltenes in the feed were 2.0 and 2.6, respectively.

The effects on SRC recovery of SRC pilot plant hot and normal separator mode operations were studied during Run 166A. The recoveries were about the same for the hot mode (166A-B MB) and the normal mode (166A-C MB): 75.3% vs 72.5% by the forced ash balance method. However, some improvements are necessary in the equipment and operating technique before the full effects of hot mode operation can be determined.

Operability of the CSD unit was tested during Run 164, a short residence time (SRT) experiment. The dissolver was bypassed, the preheater temperature was increased, and up to 25% light SRC was added to the process solvent. However, the process solvent was depleted of hydrogen donors during the test due to the lack of hydrotreating before recycle and to the high preheater outlet temperature.

The effect of solvent content in the CSD feed on SRC recovery was studied during Runs 171 and 172, in which the CSD feed contained 9.3% and 24.0% solvent, respectively. The corresponding SRC recoveries in the CSD unit were 79% and 83%. The higher recovery of SRC at the higher solvent content in the CSD feed could be due to the lower temperature in T102 Vacuum Column, which decreased the degree of repolymerization.

Ash-containing H-Coal still bottoms obtained from Hydrocarbon Research were also tested for operability in the CSD unit. It was necessary to use an alternate deashing solvent (DAS) in which the H-Coal product was relatively insoluble in order to obtain suitable performance.

Several improvements were made to the CSD unit during 1979, and these have contributed to improved operation.

1. Process Stability

Occasional power outages, severe weather conditions, plugging of various lines, malfunctions in the heat transfer medium heater, and lack of available feed due to problems in the SRC plant have limited the continuity of runs in the CSD unit.

2. Feed Composition

It was found that SRC recovery in the CSD process is related to the preasphaltene content of the feed. A higher ratio of benzene solubles to preasphaltenes generally results in higher SRC recovery. The preasphaltene content of the feed depends on the SRC plant conditions. The feed compositions for Runs 156, 159 and 160, representing 100, 50, and 75% dissolver volumes in use, are presented in the following table:

Run Date, 1979 Source	156 19 January CSD feed SRC	160 25-26 March CSD feed SRC	159 10-11 March CSD feed SRC
<u>Composition, wt %^(a)</u>			
Oil	19.2	32.3	30.3
Asphaltene	56.0	39.8	36.3
Preasphaltene ^(b)	24.8	27.9	33.4
SRC benzene solubles/preasphaltene ratio	3.0	2.6	2.0
<u>SRC Recovery in CSD unit, wt %</u>			
by forced ash balance method	86.7	82.6	72.8
by normalized balance method	83.9	81.3	72.8
average of above	85.3	82.0	72.8
<u>SRC Plant Conditions</u>			
Coal space rate, lb/hr-ft ³	25	38	50
Dissolver volume, %	100	75	50
Coal type	Ky 6 & 11	Ky 9	Ky 9
Dissolver pressure, psig	1,700	1,700	1,700
Dissolver outlet temp, °F	825	825	825
Solvent THF conversion, % ^(c)	66.8	70.7	72.9

(a) Cresol solubles.

(b) Benzene insoluble but cresol soluble.

(c) Microautoclave short method.

The data indicate that increased reaction time yields less preasphaltenes in the CSD feed.

The CSD feed compositions for Runs 159 and 171, representing the pressure effects on the preasphaltene content of the feed, are presented below:

<u>Run</u>	<u>159</u>	<u>171A-B</u>
<u>Date, 1979</u>	<u>10-11 March</u>	<u>28-30 September</u>
<u>Source</u>	<u>CSD feed</u>	<u>CSD feed</u>
<u>SRC</u>	<u>SRC</u>	<u>SRC</u>
<u>Composition, wt %^(a)</u>		
Oil	30.3	29.1
Asphaltene	36.3	43.1
Preasphaltene ^(b)	33.4	27.8
SRC Benzene solubles/preasphaltene ratio	2.0	2.6
SRC Recovery in CSD unit, wt %		
by forced ash balance method	72.8	78.5
by normalized balance method	72.8	79.4
average of above	72.8	79.0
SRC Plant Conditions		
Coal space rate, lb/hr-ft ³	50	50
Dissolver volume, %	50	50
Coal type	Ky 9	Ky 9
Dissolver pressure, psig	1,700	2,100
Dissolver outlet temp, °F	825	825
Solvent THF conversion, % ^(c)	72.9	72.6

(a) Cresol solubles.

(b) Benzene insoluble but cresol soluble.

(c) Microautoclave short method.

Increasing the pressure reduces the preasphaltene content and consequently increases the SRC recovery in the CSD unit. There was no significant change in the CSD feed composition when operating the SRC pilot plant in either the hot flash mode (Run 166A-B MB, in which the high pressure air cooler was effectively bypassed) or in the normal mode (Run 166A-C MB, in which the dissolver effluent was cooled to about 625°F). The feed compositions were as follows:

<u>Run</u>	<u>166AB MB</u>	<u>166AC MB</u>
<u>Date, 1979</u>	<u>21 July</u>	<u>2-3 August</u>
<u>Feed composition^(a), wt %</u>		
Oil	28.8	27.2
Asphaltene	40.1	43.6
Preasphaltene ^(b)	31.1	29.2
CSD-SRC Recovery, % ^(c)	75.3	72.5

(a) Cresol soluble.

(b) Benzene insoluble but cresol soluble.

(c) By forced ash balance.

The relationship between SRC recovery and the preasphaltene content of the feed may also depend on such factors as preasphaltene compositions, solubilities of preasphaltene fractions, and preasphaltene conversion to UC.

Feeds other than those from the SRC process were also tested in the CSD unit. H-Coal derived feeds obtained either in the Fuel Oil mode or in the Syncrude mode were easily processed. However, their compositions (with the exception of ash content) are not available.

3. Ash Separation

Ash concentrate from the first stage underflow is discharged continuously from the ash processing system as a light powder. The percentage of the feed in the ash concentrate discharge and the ash content in the ash concentrate were as follows:

<u>Run</u>	<u>156</u>	<u>159BC MB</u>	<u>160BC MB</u>
Ash concentrate, % of feed	51.6	40.4	32.9
Ash in ash concentrate, %	47.0	29.8	38.1
<u>Run</u>	<u>161C MB</u>	<u>162A MB</u>	<u>163A MB</u>
Ash concentrate, % of feed	41.6	38.0	38.2
Ash in ash concentrate, %	38.4	44.0	42.1
			18.8
<u>Run</u>	<u>166AB MB</u>	<u>166AC MB</u>	<u>167C MB</u>
Ash concentrate, % of feed	40.2	45.9	40.4
Ash in ash concentrate, %	26.4	24.4	29.1
			30.5
<u>Run</u>	<u>171AB MB</u>	<u>172 MB</u>	
Ash concentrate, % of feed	34.6	32.0	
Ash in ash concentrate, %	29.8	31.3	

Ash content in an ash concentrate sample appears to depend strongly on the ash, UC, and preasphaltene contents of the CSD feed. The variables were correlated by linear regression analysis (of the form $Y = aX + b$). X and Y are tabulated below:

<u>Run</u>	<u>X</u> <u>% Ash/%</u> (UC + Preasphaltene) in Feed	<u>Y</u> <u>% Ash in Ash Concentrate</u>
156	0.554	47.00
159BC MB	0.360	29.80
160BC MB	0.441	38.10
161C MB	0.485	38.40
162A MB	1.061(?)	44.00
163A MB	0.606	42.10
164A MB	0.204	18.80
166AB MB	0.306	26.40
166AC MB	0.310	24.40
167C MB	0.287	29.10
168A MB	0.335	30.49
171AB MB	0.351	29.80
172 MB	0.368	31.30

The data are plotted in Figure 25. When the parameters $a = 64.5$ and $b = 7.32$ were used, a correlation of 0.952 between X and Y was obtained.

The compositions of the cresol-soluble fractions of the ash concentrate were:

<u>Run</u>	<u>156 MB</u>	<u>159BC-MB</u>	<u>160BC-MB</u>
<u>Composition, wt %</u>			
Oil	9.0	12.4	13.2
Asphaltene	13.4	10.8	18.4
Preasphaltene	77.6	76.8	68.4
<u>Run</u>	<u>161C MB</u>	<u>162A MB</u>	<u>163A MB</u>
<u>Composition, wt %</u>			
Oil	16.3	26.0	28.2
Asphaltene	14.8	25.2	19.3
Preasphaltene	68.9	48.8	52.5
<u>Run</u>	<u>166AB MB</u>	<u>166AC MB</u>	<u>167C MB</u>
<u>Composition, wt %</u>			
Oil	16.6	14.2	13.9
Asphaltene	25.9	8.4	11.0
Preasphaltene	57.5	77.4	75.1
<u>Run</u>	<u>171AB MB</u>	<u>172 MB</u>	
<u>Composition, wt %</u>			
Oil	17.8	18.3	
Asphaltene	15.0	14.4	
Preasphaltene	67.2	67.3	

Runs 162A MB and 163A MB produced ash concentrates with the lowest concentrations of preasphaltene, because the pre-asphaltene level in the CSD feed for these runs was as low as possible.

Ash concentrate analyses are presented in Table 26.

Results of cresol and quinoline extractions of the CSD ash concentrate and the CSD feed from Run 168 (see Table 27) show:

- o CI in the feed was equivalent to the QI.
- o QI in the ash concentrate was equivalent to UC in the feed.

About one-third of the CI in the ash concentrate appears to be recoverable by tar base extraction.

4. SRC Recovery

(a) On-stream Balance

SRC recovery can be calculated by the forced ash balance method as follows:

$$\text{SRC recovery, \%} = \left[\frac{100 - \frac{(\% \text{ ash in feed}) (100)}{\% \text{ ash in ash conc.}} } \right] : \left[\frac{100 - \frac{\% \text{ CI in feed}}{100}}{100} \right]$$

SRC recovery can also be calculated by the normalized material balance method as follows:

$$\text{SRC recovery \%} = \left[\frac{(\text{SRC in lb/hr} + \text{LSRC in lb/hr})}{100} \right] + \left[\frac{(\text{feed in lb/hr}) (100 - \text{CI in feed})}{100} \right]$$

SRC recoveries are tabulated below:

Run Date, 1979	156		159BC MB		160BC MB	
	19 Jan as-is	19 Jan solv-free	10-11 March as-is	10-11 March solv-free	25-26 March as-is	25-26 March solv-free
By forced ash balance	86.7	85.3	72.8	71.2	82.3	80.6
By normalized balance	83.9	84.8	73.0	69.5	83.2	81.8
Average	85.3	85.1	72.9	70.4	82.8	81.2

Run Date, 1979	161C MB		162A MB		163A MB		164A MB	
	9-10 April as-is	9-10 April solv-free	26 April as-is	26 April solv-free	11-12 May as-is	11-12 May solv-free	4-5 June as-is	4-5 June solv-free
By forced ash balance	75.9	73.7	80.6	78.9	78.9	76.7	77.5	74.9
By normalized balance	76.3	77.4	82.6	78.1	80.6	81.1	74.4	73.9
Average	76.1	75.6	81.6	78.5	79.8	78.9	76.0	74.4

Run Date, 1979	166AB MB		166AC MB		167C MB		168 MB	
	21 July as-is	21 July solv-free	2-3 August as-is	2-3 August solv-free	9 September as-is	9 September solv-free	12 September as-is	12 September solv-free
By forced ash balance	75.3	70.6	72.5	67.9	81.2	77.7	78.5	74.1
By normalized balance	75.2	79.2	69.4	67.9	74.5	76.1	76.7	75.4
Average	75.3	74.9	71.0	67.9	77.9	76.9	77.6	74.8

Run Date, 1979	171AH MB		172 MB	
	28 Sept as-is	28 Sept solv-free	1-2 Oct as-is	1-2 Oct solv-free
By forced ash balance	78.5	75.8	82.4	75.5
By normalized balance	79.3	80.3	81.2	86.2
Average	78.9	78.1	81.8	81.9

(b) Component Balances

The SRC recoveries calculated by computerized component balances were:

Run	SRC Recovery, % (a)		
	oil + asphaltene	preasphaltene	Total
159BC MB	93.51	25.62	69.47
160BC MB	91.36	53.14	79.67
161C MB	104.96	30.13	77.38
162A MB	81.94	50.52	77.92
163A MB	97.82	40.31	80.63
164A MB	88.28	40.65	73.74
166AB MB	100.46	42.85	79.19
166AC MB	84.26	36.00	67.78
167C MB	100.08	35.34	75.43
168 MB	99.24	31.74	75.23
171AB MB	86.30	65.93	79.93
172 MB	108.25	45.31	87.81

(a) Solvent-free and normalized balance using recovered SRC flow rates.

More than 80% of the oil and asphaltene in the CSD feed were recovered in the final SRC product. Recovery of preasphaltene in the CSD feed was only 26 to 66% (average 42%). This low recovery of preasphaltene seems to be mainly responsible for the low total SRC recovery (68 to 88%).

The solvent refined coal analyses are presented in Tables 7, 8 and 9.

The elemental balances for each run were:

Run	Element, percent error					
	Sulfur	Carbon	Hydrogen	Nitrogen	Oxygen	Ash
159BC MB	-1.96	0.27	0.57	4.04	-14.06	-0.64
160BC MB	-7.70	0.19	0.55	0.01	-11.49	2.00
161C MB	-4.55	-0.45	-0.14	-11.74	22.36	0.70
162A MB	3.74	0.05	1.08	4.56	-29.16	3.66
163A MB	6.89	-0.32	0.18	16.05	-2.06	1.98
164A MB	-5.51	0.14	0.56	2.38	-7.30	-7.72
166A MB	-18.77	0.62	-0.24	2.67	-79.12	-0.36
166AC MB	-8.89	0.18	0.28	28.17	-11.39	-5.89
167C MB	-8.63	0.64	0.92	3.98	-8.67	-15.23
168 MB	-2.89	0.26	0.50	3.97	-11.65	-4.72
171AB MB	1.27	0.08	0.18	14.36	-31.20	1.45
172 MB	-10.19	0.49	0.09	-9.91	-14.98	-4.90

The carbon and hydrogen balances are less subject to error than the sulfur and nitrogen balances. The oxygen balance shows the highest error because the oxygen contents were calculated by difference: % oxygen = 100 - %S - %C - %H - %N - %ash.

(c) Overall SRC Yields Based on MAF Coal

The data are tabulated below:

Run	159BC MB		160BC MB	
Date, 1979	10-11 March		25-26 March	
<u>Plant Conditions:</u>				
Coal feed rate, MF lb/hr	440		491	
Slurry conc, % MF coal	37.0		37.8	
Feed gas rate, scfh	10,000		10,000	
Dissolver volume, %	50		75	
Solids withdrawal	no		no	
Preheater temp, °F	802		782	
Solvent in CSD feed, %	4.5		7.1	
Solvent in CSD-SRC, %	6.6		8.5	
SRC yield (based on MAF coal), %	w/solv	solv-free	w/solv	solv-free
by unadjusted process method	62.6	58.8	68.3	62.2
in the front				
SRC recovery in the CSD unit	73.0	69.5	83.2	81.8

Run	161C MB	162A MB	163A MB	164A MB
Date, 1979	9-10 April	26 April	11-12 May	4-5 June
<u>Plant conditions:</u>				
Coal feed rate, MF lb/hr	498	494	498	230
Slurry conc, % MF coal	38.4	38.0	38.2	35.4
Feed gas rate, scfh	10,000	10,000	4,500 (bypass 5,000)	2,225
Dissolver volume, %	75	75	75	0
Solids withdrawal	yes	yes	yes	no
Preheater temp, °F	791	776	849	890
Solvent in CSD feed, %	6.5	6.1	7.2	8.9
Solvent in CSD-SRC, %	3.2	11.2	5.5	7.5
SRC yield (based on MAF coal), %	w/solv	solv-free	w/solv	solv-free
by unadjusted process method in front	71.2	65.1	64.0	57.7
SRC recovery in the CSD unit	76.3	77.4	80.6	81.1
			73.9	61.8
			74.4	73.9

Run	166AB MB	166AC MB	167C MB	168 MB
Date, 1979	21 July	2-3 August	9 September	12 September
<u>Plant conditions:</u>				
Coal feed rate, MF lb/hr	523	536	473	489
Slurry conc, % MF coal	40.1	37.5	37.8	37.2
Feed gas rate, scfh	10,689	10,138		
Dissolver volume, %	75	75	50	50
Solids withdrawal	yes	yes	yes	yes
Preheater temp, °F	788	790	782	779
Solvent in CSD feed, %	12.6	11.2	12.5	14.1
Solvent in CSD-SRC, %	5.1	5.5	10.0	10.7
SRC yield (based on MAF coal), %	w/solv	solv-free	w/solv	solv-free
by unadjusted process method in the front	59.8	49.8	69.4	58.0
SRC recovery in the CSD unit	75.2	79.2	61.3	52.2
			66.7	54.3
			76.7	75.4

Run	171AB MB		172 MB	
Date, 1979	28 Sept		1-2 Oct	
<u>Plant conditions:</u>				
Coal feed rate, MF lb/hr	453		458	
Slurry conc, % MF coal	36.2		36.4	
Feed gas rate, scfh	10,000		10,000	
Dissolver volume, %	50		50	
Solids withdrawal	yes		yes	
Preheater temp, °F	800		792	
Solvent in CSD feed, %	9.3		24.0	
Solvent in CSD-SRC, %	5.8		16.1	
SRC yield (based on MAF coal), %	w/solv	solv-free	w/solv	solv-free
by unadjusted process method in the front	66.4	58.3	89.7	63.0
SRC recovery in the CSD unit	79.3	80.3	81.2	88.2

The highest yields were obtained in Run 172 MB with high solvent (24%) in the CSD feed.

5. Light SRC (LSRC) Separation

The LSRC is a mixture of distillate process solvent and SRC which is obtained from the third stage of the CSD unit. Some or all of the LSRC produced is added to the recycled process solvent to improve its quality. The amounts of LSRC produced and recycled, together with the qualities of the process solvent for various runs during 1979 were:

Run	CSD feed, lb/hr	Process solvent in CSD feed, lb/hr	LSRC produced, lb/hr	LSRC added to process solvent, lb/hr	Process solvent in the LSRC, %	Process solvent quality, Microautoclave short test, THF conversion, %
159	326.5	14.7	23.0	23.0	35.3	71.0
160	283.7	20.1	12.9	9.1	34.2	73.6
161	331.7	21.6	25.3	0	33.3	74.3
162	293.5	17.9	21.6	10.8	28.0	74.9
163	311.3	22.4	32.0	16.0	25.0	67.9
164	297.0	26.4	99.8	110.5	14.5	66.4
166AB	281.9	35.5	43.0	22.0	29.3	66.7
166AC	246.9	27.7	46.0	23.0	36.6	67.0
167	338.8	42.4	30.4	30.4	35.3	66.4
168	259.2	36.6	52.9	28.0	34.8	68.9
171	317.2	29.3	56.2	22.0	22.0	71.7
172	254.6	61.1	67.3	22.0	31.6	72.3

Laboratory microautoclave studies (without gaseous hydrogen) have shown that LSRC (and especially its distillate and oil components) may improve short residence time coal conversion. These tests were described in detail in earlier reports (Ref. 1, 2). The significant data were:

Solvent:Coal ratio	Reaction Time, min	THF Conversion, %		
		Run 151 MB Process Solvent	50% Process Solvent +50% LSRC (151 MB)	50% Process Solvent +50% LSRC oil (a) (166AC MB)
8:1	10	63.9	76.2	
2:1	10	57.8	69.9	
		Run 166AC MB Process Solvent	50% Process Solvent +50% LSRC oil (a) (166AC MB)	50% Process Solvent +50% LSRC Asphaltenes (a) (166AC MB)
8:1	10	67.0	68.7	57.0
		Distillate (b) from Run 168 MB LSRC	Distillate (b) from Run 162 MB LSRC	
6:67	10	84.0	81.2	

(a) Oil and asphaltene fractions obtained by soxhlet extractions with pentane and benzene.
 (b) Distillate obtained at 600°F and 0.1 mm Hg.

6. Deashing Solvent Recovery

Studies indicated that more than 95% of the deashing solvent (DAS) losses were associated with the ash recovery system. Some improvements to this system were made during the third quarter to reduce the DAS losses. During Runs 171 and 172 MB, solvent losses to products averaged 1.65%, while the total losses were about 9% of the feed.

The distribution of the DAS losses was:

<u>Run</u>	<u>171 MB</u>		<u>172 MB</u>	
<u>Stream</u>	<u>lb/hr</u>	<u>% of CSD feed</u>	<u>lb/hr</u>	<u>% of CSD feed</u>
Ash concentrate	3.86	1.2	3.60	1.4
SRC	1.07	0.3	0.58	0.2
LSRC	0.11	0.03	0.20	0.08
Total	5.04	1.6	4.38	1.7
Solvent losses to the atmosphere	28.57	9.0	14.52	5.7

H. PRODUCT ANALYSES AND PROPERTIES

Studies were directed toward finding an effective means to control the qualities of SRC products and to evaluate the effects of process variables on plant performance. In these studies, feed coal and SRC product samples were analyzed as follows:

- Soxhlet extraction for composition of SRC products as oil, asphaltene, and preasphaltene.
- Viscosity for flow properties of SRC products.
- Microautoclave conversion and GC boiling point distribution determination for control of recycled process solvent quality.
- Ultimate and proximate analyses for carbon, hydrogen, nitrogen, sulfur, ash, oxygen (by difference), etc.
- Other pertinent tests: cresol-insoluble and SRC yields, specific gravities, fusion points, GC analyses of gaseous and liquid samples, etc.

1. Compositions of SRC Products

Compositions of SRC products such as V110 SRC, CSD feed, CSD SRC (free of unreacted coal and ash), CSD light SRC, and CSD ash concentrate were determined by conventional Soxhlet solvent extraction. Each sample was first extracted with benzene in the presence of Celite to separate the oil-asphaltene mixture from the benzene-insoluble portion.

After evaporating the benzene from the benzene-solubles, the mixture of oil and asphaltene was precipitated with pentane to separate the oils from the pentane-insoluble asphaltenes. Preasphaltenes and unreacted coal in the benzene-insoluble portion were then determined by cresol extraction. Analyses of the feed coal samples and results from solvent extraction of the SRC products for the fourth quarter are shown in Tables 9 and 10.

Solvent extraction data for SRC products from V110 and from the CSD unit on a cresol-soluble basis are tabulated below:

<u>Run Date, 1979</u>	159BC MB 10-11 March		160BC MB 25-26 March		161C MB 9-10 April	
Sample location	V110	CSD	V110	CSD	V110	CSD
Material	Lab-filt. SRC	Deashed SRC	Lab-filt. SRC	Deashed SRC	Lab-filt. SRC	Deashed SRC
<u>Composition, wt %</u>						
Oil	27.5	26.7(21.5)	27.7	28.3(21.4)	24.2	21.4(18.8)
Asphaltenes	44.3	60.0(64.3)	36.0	52.1(57.1)	37.0	63.3(65.4)
Preasphaltenes	28.2	13.3(14.2)	36.3	19.6(21.5)	38.8	15.3(15.8)
Solvent in SRC	-	6.6	-	8.7	-	3.2
<u>Sulfur, wt %</u>	0.85	0.99	0.84	1.25	0.89	0.98
<u>Fusion Point, °F</u>	311	311	365	293	365	336
<u>Run Date, 1979</u>	162A MB 26 April		163A MB 11-12 May		164A MB 4-5 June	
Sample location	V110	CSD	V110	CSD	V110	CSD
Material	Lab-filt. SRC	Deashed SRC	Lab-filt. SRC	Deashed SRC	Lab-filt. SRC	Deashed SRC
<u>Composition, wt %</u>						
Oil	33.9	32.8(24.3)	33.3	30.9(26.9)	24.8	20.3(13.8)
Asphaltenes	50.0	59.1(66.6)	41.8	53.0(56.1)	40.2	48.0(51.9)
Preasphaltenes	16.1	8.1(9.1)	24.9	16.1(17.0)	35.0	31.7(34.3)
Solvent in SRC	-	11.2	-	5.5	-	7.5
<u>Sulfur, wt %</u>	0.94	0.91	0.82	0.85	1.06	1.09
<u>Fusion Point, °F</u>	340	276	347	278	338	356

Run Date, 1979	166AB MB 21 July		166AC MB 2-3 August		167C MB 9 September	
Sample location	V110	CSD	V110	CSD	V110	CSD
Material	Lab-filt. SRC	Deashed SRC	Lab-filt. SRC	Deashed SRC	Lab-filt. SRC	Deashed SRC
<u>Composition, wt %</u>						
Oil	19.6	26.5(22.2)	20.8	21.8(17.3)	20.8	24.9(16.6)
Asphaltenes	35.6	49.8(52.7)	34.2	55.2(58.4)	45.6	57.6(63.3)
Preasphaltenes	44.8	23.7(25.1)	45.0	23.0(24.3)	33.6	18.1(20.1)
Solvent in SRC	-	5.5	-	5.5	-	10.0
<u>Sulfur, wt %</u>	0.92	1.00	0.94	0.94	1.03	1.04
<u>Fusion Point, °F</u>	-	329	-	329	437	279
Run Date, 1979	168AB MB 12 September		171AB MB 28-30 September		172 MB 1-2 October	
Sample location	V110	CSD	V110	CSD	V110	CSD
Material	Lab-filt. SRC	Deashed SRC	Lab-filt. SRC	Deashed SRC	Lab-filt. SRC	Deashed SRC
<u>Composition, wt %</u>						
Oil	23.0	33.7(25.8)	28.9	33.7(29.6)	24.6	31.4(18.2)
Asphaltenes	36.5	48.5(54.3)	42.2	35.3(37.5)	43.7	51.4(61.3)
Preasphaltenes	40.5	17.8(19.9)	28.9	31.0(32.9)	31.7	17.2(20.5)
Solvent in SRC	-	10.7	-	5.8	-	16.2
<u>Sulfur, wt %</u>	1.00	1.00	1.10	0.96	1.10	0.89
<u>Fusion Point, °F</u>	473	297	374	302	430	219

Note: Values in parentheses are based on solvent-free deashed SRC.

Preasphaltene contents as high as 34% were observed in the solvent-free CSD-deashed SRC samples from Runs 164A MB and 171AB MB, while in Run 162A MB, the preasphaltene content was only 9.1%. The CSD-deashed SRC samples contained 17-27% oils, 53-65% asphaltenes and 14-25% preasphaltenes.

Figures 26 and 27 show fusion point and oil content correlations of the V110 SRC samples and of the CSD-deashed SRC samples. Fusion points decreased with increasing oil contents in both SRC samples.

Effects of several process variables on the sulfur contents of the V110 lab-filtered SRC samples are tabulated below:

(1) Residence Time Effect

<u>Run</u>	<u>Dissolver Volume, %</u>	<u>Sulfur, wt %</u>	<u>Remarks</u>
167C MB	50	1.03	
168A MB	50	1.00	
171AB MB	50	1.10	
162A MB	75	0.94	significant

(2) Pressure Effect

<u>Run</u>	<u>Press, psig</u>	<u>Sulfur, wt %</u>	<u>Remarks</u>
166AC MB	1,751	0.94	
166AB MB	1,775	0.92	
162A MB	2,150	0.94	little

(3) Preheater Temperature Effect

<u>Run</u>	<u>Temp, °F</u>	<u>Sulfur, wt %</u>	<u>Remarks</u>
161C MB	791	0.89	
163A MB	849	0.82	little

(4) Solids Withdrawal

<u>Run</u>	<u>Withdrawal</u>	<u>Sulfur, wt %</u>	<u>Remarks</u>
160BC MB	No	0.84	
161C MB	Yes	0.89	little

Residence time apparently had the greatest effect on the sulfur contents of the V110 lab-filtered SRC samples. The effect of dissolver temperature was not investigated during the year.

Effects of process variables on the sulfur contents of the CSD-deashed SRC are summarized below:

(1) T102 Bottom Temperature Effect

<u>Run</u>	<u>Temperature, °F</u>	<u>Sulfur, wt %</u>	<u>Remarks</u>
172A MB	500	0.89	
171AB MB	560	0.96	little

(2) Hot Separator Mode (effect of residence time at elevated temperatures)

<u>Run</u>	<u>Minimized</u>	<u>Sulfur, wt %</u>	<u>Remarks</u>
166AC MB	No	0.94	
166AB MB	Yes	1.00	little
167C MB	No	1.03	
168A MB	No	1.00	
171AB MB	Yes	1.10	little

Only small effects on the sulfur contents of the CSD-deashed SRC were observed by varying the T102 bottom temperature or by using the hot separator mode.

2. Viscosity Changes in SRC Products

Viscosity data for CSD-deashed SRC, V110 lab-filtered filtrate, and recycle process solvent were obtained by using a Brookfield thermocell viscometer (Model 638) with a proportional temperature controller.

Three major factors affecting viscosity changes in SRC were observed:

- o Reaction conditions. These include temperature (constant for this study at 825°F), residence time (dissolver volume), preheater temperature, reaction pressure, and solids withdrawal rate.
- o Physical process variables. These include T102 distillation and CSD separator conditions which affect compositions of SRC products in terms of solvent, oil, asphaltene and preasphaltene.
- o Repolymerization of SRC Products. This is caused by T102 distillation bottom temperature.

The chemical process variables affect the viscosity data of the V110 lab-filtered filtrates, and the other major factors listed above affect the CSD-deashed SRC products.

Effects of process variables on the viscosities of the V110 lab-filtered filtrates are shown below:

(1) Residence Time Effect

<u>Run</u>	<u>Dissolver Volume, %</u>	<u>At 250°F</u> <u>Viscosity, cp</u>	<u>Remarks</u>
167C MB	50	17.5	significant
168A MB	50	17.0	"
162A MB	75	9.6	"

(2) Pressure Effect

<u>Run</u>	<u>Pressure, psig</u>	<u>At 250°F</u> <u>Viscosity, cp</u>	<u>Remarks</u>
166AC MB	1,751	10.0	insignificant
162A MB	2,150	9.6	"
167C MB	2,175	17.5	insignificant
168A MB	2,175	17.0	"
159BC MB	1,415	16.8	"

(3) Preheater Temperature Effect

<u>Run</u>	<u>Temperature, °F</u>	<u>At 250°F</u> <u>Viscosity, cp</u>	<u>Remarks</u>
161C MB	791	11.2	slight
163A MB	849	8.4	"

(4) Solids Withdrawal

<u>Run</u>	<u>Withdrawal</u>	<u>At 250°F</u> <u>Viscosity, cp</u>	<u>Remarks</u>
160BC MB	No	11.5	insignificant
161C MB	Yes	11.2	"
159BC MB	No	16.8	insignificant
167C MB	Yes	17.5	"
168A MB	Yes	17.0	"

Residence time (dissolver volume) apparently has the greatest effect on the viscosity of the V110 lab-filtered filtrate. (Viscosity is reduced with increasing residence time.) Increasing the preheater temperature also reduces the viscosity slightly.

Effects of variables on the viscosities of solvent-free CSD-deashed SRC were:

(1) T102 Bottom Temperature Effect

<u>Run</u>	<u>Temperature, °F</u>	<u>At 500°F</u> <u>Viscosity, cp</u>	<u>Remarks</u>
172 MB	500	1,700	significant
171AB MB	560-580	13,400	"

(2) Hot Separator Mode (effect of residence time at elevated temperatures)

<u>Run</u>	<u>Minimized</u>	<u>Viscosity, cp</u>	<u>Remarks</u>
166AC MB	No	5,800	slight
166AB MB	Yes	4,000	"
167C MB	No	6,500	significant
168 MB	No	6,600	"
171AB MB	Yes	13,400	"

Increasing the T102 bottoms temperature from 500 to 560°F greatly increased the viscosity of the CSD-deashed SRC (solvent-free) product. Inconsistent results were obtained in determining the effects of heat transfer rate at the E102 Dissolver Product Cooler.

The relationships of the viscosities of the solvent-free CSD-deashed SRC to their compositions in terms of oil, asphaltenes, and preasphaltenes were determined. Preasphaltene contents in the solvent-free CSD-deashed SRC had the closest correlation to the viscosity data. The following is a summary of these data, which have been classified into three groups:

<u>Classification</u>	<u>Run No.</u>	<u>Preasphaltene in CSD-SRC, %</u>	<u>Viscosity, (a) cp</u>
Group I: (i) 50% dissolver volume, P = 2,175-2,190 psig, solids withdrawal, T, °F: B102 outlet = 780-800 R101 outlet = 825	167C MB 168A MB 171AB MB (b)	20.1 19.9 32.9	6,500 6,600 13,400
(ii) P = 1,415 psig, no solids withdrawal	159BC MB	14.2	5,800
(iii) 75% dissolver volume	162A MB	9.1	4,300
(iv) T at T102 bottom = 500°F	172 MB (b)	20.5	1,700
Group II: (i) 75% dissolver volume, P = 1,705-1,775 psig, solids withdrawal, T, °F: B102 outlet = 780-900 R101 outlet = 825	161C MB 166AB MB (b) 166AC MB	15.8 25.1 24.3	3,700 4,000 5,800
(ii) No solids withdrawal	160BC MB	21.5	5,500
(iii) T, °F: B102 outlet = 850	163A MB	17.0	3,200
(iv) P = 2,150 psig	162A MB	9.1	4,300
Group III: (i) 0% dissolver volume, P = 2,400 psig, no solids withdrawal, T, °F: B102 outlet = 890	164A MB	34.3	N.A.

(a) Viscosity determined at T = 500°F and shear rate = 0.84 sec⁻¹.

(b) Minimizing heat transfer of the E102 Dissolver Product Cooler.

The data are plotted in Figures 28 (linear scale) and 29 (semi-log scale). Better correlations were obtained from the semi-log plot. These results confirm that the viscosities of the solvent-free CSD-deashed SRC are strongly dependent on their preasphaltene contents. This relationship may be described by the non-linear regression equation:

$$\log_{10} y = AX + C$$

where

y = viscosity, cp

X = preasphaltene content

A, C = constants

<u>Group</u>	<u>Equation</u>	<u>Correlation Coefficient</u>
Group I:	$\log_{10} y = 0.0202 X + 3.443$	$r = 0.987$
Group II:	$\log_{10} y = 0.0297 X + 3.061$	$r = 0.923$

Viscosities of the V110 lab-filtered filtrates are summarized below:

<u>Group</u>	<u>Run</u>	<u>SRC % in V110</u>	<u>% in SRC (% in V110)</u>			<u>Viscosity, cp</u>	
			<u>Oil</u>	<u>Asphaltene</u>	<u>Preasphaltene</u>	<u>at 200°F</u>	<u>at 250°F</u>
Group I	167C MB	30.7	20.8(6.4)	45.8(14.0)	33.6(10.3)	51	17.5
	168A MB	30.9	23.0(7.1)	36.5(11.3)	40.5(12.5)	40	17.0
	171A-B MB	35.2	28.9(10.2)	42.2(14.9)	28.9(10.2)	-	-
	159B-C MB	28.0	27.5(7.7)	44.3(12.4)	28.2(7.9)	74.5	16.8
	162A MB	27.2	33.9(9.2)	50.0(13.6)	16.1(4.4)	-	9.6
	172 MB	35.0	24.6(8.6)	43.7(15.3)	31.7(11.1)	-	-
Group II	161C MB	26.5	24.2(6.4)	37.0(9.8)	38.8(10.3)	31.5	11.2
	166A-B MB	40.0	19.6(7.8)	35.6(14.2)	44.8(17.9)	38.2	67.5
	166A-C MB	27.9	20.8(5.8)	34.2(9.5)	45.0(12.6)	33	10.0
	160B-C MB	25.8	27.7(7.1)	36.0(9.3)	36.3(9.4)	26	11.5
	163A MB	25.7	33.3(8.6)	41.8(10.7)	24.9(6.4)	21	8.4
	162A MB	27.2	33.9(9.2)	50.0(13.6)	16.1(4.4)	-	9.6
Group III	164A MB	39.2	24.8(9.7)	40.2(15.8)	35.0(13.7)	54.5	70.5

The data are plotted in Figures 30 (linear scale) and 31 (semi-log scale). Poor correlations were obtained in the semi-log plot. A regression analysis yielded the following equation:

<u>Group</u>	<u>Equation</u>	<u>Correlation Coefficient</u>
Group I:	insufficient data	
Group II:	$\log_{10} y = 0.0765 X + 0.305$	$r = 0.888$

The intercept of Group II represents a viscosity of 2 centipoise, which is very close to the actual viscosity of the process solvent (1.5-2.5 cp at 250°F).

The following is a summary of the viscosities of process solvents:

<u>Run</u>	<u>Viscosity of Process Solvent at 250°F, cp</u>
160BC	2.5
161C	2.0
162A	2.0
163A	1.8
166AB	1.8
166AC	1.8
167C	1.8
168	1.8
171AB	1.5
172	1.8

I. PRODUCT SOLIDIFICATION

Liquid SRC is normally fed to two vibrating water-cooled trays of the Rexnord product cooler at 600°F. Each of the two trays provides 15 ft² of surface. The SRC solidifies into brittle sheets 1/8 to 1/4 in. thick, which break into fragments when the tray vibrates.

During the quarter, two other methods of solidification were attempted:

- Using a dual-pipe heat exchanger

The dual pipe heat exchanger failed for several reasons: the velocity of the SRC in the pipe (0.2 ft/sec) was too slow, and the SRC began to solidify in the pipe itself; the Δt between the cooling medium (water) and the SRC was too great, causing a thin layer of SRC to form on the pipe wall which quickly thickened until the pipe plugged. Studies will continue.

- Using a steam-jet solidifier

The steam-jet solidifier (consisting of a one-inch steam jet siphon into which liquid SRC was flowing from the drum-out station) also plugged immediately upon steam contact with the SRC. The comparatively cool (300°F) steam probably cooled the SRC to its solidification point immediately.

V. MECHANICAL PERFORMANCE

During 1979, some major operating problems were found and several key improvements were made. The principal problem was excessive corrosion in T102 Vacuum Column and T105 Fractionation Column. Improvements included the operation of the LV415 High Pressure Letdown Valves for more than 3,000 hours and operation of P111B Filter Feed Pump for 1,500 hours without maintenance.

A. AGITATORS

The packing problems that had previously been associated with the plant agitators during filtration runs were greatly reduced after the drop-type oilers were removed and daily lubrication with a high-pressure service grease was begun.

B. COMPRESSORS

Both hydrogen compressors (C102 Hydrogen Recycle and C104 Fresh Hydrogen) operated during the year without maintenance. Valves were changed on 4 December to prevent future problems. These compressors have operated without major repairs since early 1978.

C. DRYERS

The Kahn Instrument Air Dryer caused several problems. The four-way valve to the dryer and the outlet checks were replaced with ball valves to prevent leakage during the regeneration cycle. Number 2 tower heaters failed in early November, when a thermostat malfunctioned. The water trap on the receiver tank failed, allowing the dryer to fill with water. A desiccant change was required to bring the dew point back to the -40°F specification. A Panametrics Dew Point Indicator was installed to monitor the performance of the dryer.

D. EXCHANGERS

There were no maintenance problems with heat exchangers during the fourth quarter.

E. FIRED HEATERS

The B102 Slurry Preheater was shut down for decoking on 4 December. When attempting to restart the plant on 8 December, a 316 SS pipe at the outlet of the heater was found to be leaking. The leaking line was sent to Oak Ridge National Laboratory (ORNL) for metallurgical analysis. The ORNL test results will be published when available.

In June, seven thermowells in the top coils of the heater were replaced, along with a section of the coil. These were sent to Air Products & Chemicals, Inc. for failure analysis, as two thermowells were leaking. The cause of the leakage was erosion of the thermowell tips (which project into the flowing slurry).

The B103 Vacuum Column Preheater burner was converted from No. 2 oil to propane gas on 26 December.

To reduce the problems with coke formation in the heater burners and to reduce fuel cost, engineering is in progress to convert as many plant heaters as possible to propane gas.

F. MINERAL RESIDUE SEPARATION

F125 United States Filter Corporation (USF) filter was ready for operation at the start of the fourth quarter. During the quarter, the filter was inspected when excessive ash appeared in the filtrate. Shutdowns occurred on 8, 24 and 26 October. To determine the causes of the high ash, the bands were removed from the outer edges of two of the 3-ply screens. It was found that the fabricator had cut stress-relief slots in the support channels, which allowed unfiltered material to bypass the screens. The slots were seal-welded and the bands were replaced. On 16 and 29 October, the filter shaft seized when a cake cut-off bearing became galled to the shaft.

On 12, 16 and 30 November, the filter screens were inspected. Each time, small pinholes were found near spot welds on the screens. The filter was restarted on 1 December after installing three new filter screens, but screen life continued to be short. The screens were removed and pinholes near the plug welds were repaired on 3, 8, 21 and 27 December. In each case, the pinholes were repaired with silver solder.

The high-pressure sluice system worked exceptionally well and did not present mechanical problems. Mechanically, the USF filter is much simpler and more reliable than the Funda filter.

The Kerr-McGee Critical Solvent Deashing (CSD) unit was shut down late in the year for major modifications. The key maintenance problem with the CSD unit was plugging of the first stage letdown system. Packing and plunger problems also occurred with the feed pumps. Engineering is underway to reduce maintenance in both areas.

G. PUMPS

The P111B Lawrence Filter Feed Pump was inspected after 1,500 hours of operation, and was in excellent condition.

Impellers on both P110A and B Reclaim Tank pumps plugged. This was caused by slugs of solids from the V111 Reclaim Tank.

A hole developed in the case discharge nozzle of the P143 Precoat Pump on 12 November. A new pump case was installed. On 27 November, a hole developed in the discharge nozzle of P111A Filter Feed Pump. This nozzle was repaired by welding.

A Dowtherm leak in the jacketed piping of the P119C and D Liquid Coal Pump circulation loop was located and the leaking section was isolated. A new section of line will be fabricated for installation during the next shutdown.

Failures of major process pumps were greatly reduced during the year by the extensive use of double mechanical seals, by anchoring pump suction and discharge as near the pump as possible and by very close attention to pump alignment. Most pumps were aligned cold and rechecked after heating to operating temperatures.

H. VALVES

LV415A High Pressure Letdown Valve was repaired on 30 October. The trim was badly worn and had failed after 3,220 hours of service.

LV415B High Pressure Letdown Valve trim was changed on 26 November. This trim had been operated for 3,320 hours.

Two attempts during the year to test a silicon carbide plug in the LV415A valve failed when the plug fractured.

A new letdown valve from Consolidated Control Valves was tested at the pilot plant. Earlier in the year, in letdown service, bench tests of the valve indicated leakage too great to enable control of the process flow. The valve was returned to the manufacturer for modification.

LV507 Level Control Valve for V110 and FV904 Flow Valve for B102 Vacuum Preheater continue to present plugging problems. Improved heat tracing is being installed in an effort to reduce the plugging.

The first stage letdown system at the CSD unit continues to plug. Engineering is underway to redesign the piping in this area.

I. REACTION SECTION

After repeated problems with the density meters on the R101 Dissolver, it was determined that there was a mistake in the design of the solid state components. Corrections are being made by the vendor.

The bottom of the R101 Dissolver was removed for inspection on 4 December while B102 was being decoked and again on 29 December due to a gasket leak. The bottom head on the dissolver usually leaks on startup if the plant has been depressured or allowed to cool. When this occurs, the bottom gasket requires replacement.

A hydraulic torque wrench was purchased early in the year to tighten the bottom head bolts. If the temperature in the dissolver is maintained above 400°F, the gasket does not leak on restart of the plant.

J. COLUMNS

T102 Vacuum Column was not inspected during the fourth quarter. Extensive repairs had been made to the column in February and in August.

Five electrical resistance corrosion probes were installed for monitoring corrosion rates inside the column.

The T105 Fractionation Column manways were opened on 26 December. Corrosion coupons were evaluated by a metallurgist from Oak Ridge National Laboratory. The following observations were made:

o Visual

- The circumferential crack noted in the middle manway cladding during previous inspections had become a large fissure. The bottom edge of the manway cladding extending into the column had become thin and jagged.
- The shiny, black scale removed through the middle manway was thin and brittle. It could be peeled easily from the 321 SS downcomer in large pieces, exposing a bright, gold-colored metal surface. However, peeling the scale from the 304 SS clad surface revealed a black powder underneath. The rough metal surface under the powder displayed a dull gold finish. The scale removed through the top manway was thick, gray, dull and pulpy, and flaked readily. The scale at the bottom of the column was grimy.
- Tray 11, had three badly corroded and dislodged ballast caps. The ballast cap section of the tray was removed for rebuilding and, in so doing, many more ballast caps fell out. Tray 12 was also inspected and found to be in very poor condition. Half

of the ballast cap assemblies had been dislodged. Those assemblies which had been intentionally disabled were in better condition than those which had not. Ballast cap components were in various stages of disintegration, ranging from extremely thin (<0.08-in.) to completely deteriorated. The condition of tray 13 appeared to be similar to that of tray 12.

Tray 1 looked as clean and substantial as new. Tray 20 was dirty, but intact.

o Ultrasonic

- The shell was tested at three external points in two areas above tray 9 which had experienced the greatest metal loss when observed in March. The average metal loss since then was 22 mils.
- Trays 1, 10, and 12 were tested. The results are listed in Table 29, along with those obtained previously.

o Corrosion Coupons

The Oak Ridge National Laboratory group cleaned and weighed all corrosion coupons. Results are listed in Table 30. The U-bend racks (corrosion specimens in tension) were cleaned and photographed. The E-Brite specimen in the middle manway U-bend rack had disintegrated prior to the previous inspection on 21 September 1979.

o Corrosion Probes

Data from electrical resistance corrosion probes, when compared to actual corrosion rates measured ultrasonically (See Table 31), show that the trays are corroding at twice the rate indicated by the corrosion probes. This suggests that corrosion is occurring on both sides of the trays.

The following conclusions can be drawn from the corrosion data obtained during 1979:

- o The high-nickel alloys had superior corrosion resistance.
- o The ballast caps had corroded at twice the rates of the trays on which they were assembled, even though all components were fabricated from 321 SS material.
- o Corrosion had occurred on both sides of the trays.

Corrosion coupon racks (including the substitutions indicated in Table 30), were reinstalled. Tray 12 was cleared of all debris, tray 11 was rebuilt and all manholes were closed. Corrosion probes at trays 9 and 15 were replaced with 316 SS and 304L SS specimens, respectively.

During December, sodium carbonate was added to the coal slurry fed to the SRC plant as a corrosion inhibitor at the following rates:

1-9 December	5 lb per batch of coal
10-14 December	10 lb per batch of coal
15-19 December	15 lb per batch of coal
20-26 December	20 lb per batch of coal
26-28 December	Column down
29-31 December	40 lb per batch of coal

When an increase in the corrosion rate was detected by monitoring the corrosion probes, the amount of sodium carbonate was increased until the corrosion was reduced to an acceptable level.

VI. PROJECTS

A. ACTIVE

1. Project 4142: New V204 Flush Solvent Tank

This project provides for a new V204 Flush Solvent Tank rated at 600°F and 200 psig. It is scheduled to be completed in conjunction with the hydrotreater installation (Project 4143).

2. Project 4143: Hydrotreater Installation

This project provides for the installation of a hydrogenation unit at the Wilsonville pilot plant. It will permit exploration of various modes of short residence time (SRT) operation while maintaining process solvent quality at low hydrogen consumption. It will also yield experimental data on production of liquid fuels from SRC-I using conventional hydropyrolysis catalysts. Construction work has begun in the areas of site preparation, underground piping and concrete foundations. Erection of structural steel is scheduled to begin in mid-February 1980. The target date for project completion is August 1980.

3. Project 4144: Critical Solvent Deashing Modifications

This project provides the following:

- Recovery of deashing solvent in a high-pressure, high-temperature recycle system,
- A new first stage to provide increased capacity for future process conditions, and
- Increased plant operating area to accommodate new and existing equipment.

Construction work is scheduled to be completed by mid-January 1980.

4. Project 4146: Laboratory, Warehouse and Locker Room

This project provides for the following:

- A controlled-atmosphere laboratory to house highly sophisticated electronic equipment,
- Increased warehouse and maintenance shop area, and
- Expanded locker room facilities.

Construction is scheduled to begin in February 1980 and to be completed by mid-April.

B. COMPLETED

1. Project 4117: Waste Water Treatment

Installation of the facility was completed in February.

2. Project 4122: New Ash Cooler

Installation was completed in May.

3. Project 4127: Lawrence Slurry Pump

Installation of the pump was completed in May.

4. Project 4128: Weigh Cells for V110 Flash Tank

Installation of the weigh cells was completed in January.

5. Project 4133: Solids Withdrawal System Modifications

Modifications were completed in March.

6. Project 4145: USF Filter Modifications

This project provided for the addition of tanks, pumps, and piping systems to provide the filter with a high-pressure sluice capability in the precoat and non-precoat modes of operation. This project was completed in September.

VII. CONCLUSIONS

In comparing Runs 168 through 172 and Runs 166AB and 166AC, it was concluded that improving the process solvent quality had a significant effect on the SRC plant performance. Most notably, yield of SRC (especially SRC oils) increased and yield of process solvent (boiling above 450°F) decreased.

In Critical Solvent Deashing (CSD) experiments, a strong correlation was found between the preasphaltene content of the feed and the recovery of SRC. It was also apparent that the quantity of process solvent in the CSD feed had very little effect on the SRC recovery. In addition, the hot flash mode of operation did not appear to improve recovery significantly. However, this system could not be tested to its full potential due to equipment limitations. Deashing solvent losses remained high, averaging 9% of the CSD feed. Approximately 95% of the losses were associated with the ash concentrate recovery system.

The CSD unit apparently performs quite well on feed from the bottoms stream of the H-Coal process. However, the deashing solvent used for this feed was different from that used for processing SRC plant bottoms.

Solids separation by the United States Filter Corporation (USF) vertical-leaf filter was affected by poor screen quality and by the presence of sodium carbonate, a corrosion inhibitor, in the coal slurry. Although the USF filter is simple and easy to maintain, the filter screens, as constructed, did not hold up well under processing conditions. When filtrate clarity specifications were achieved, as indicated by the quantity of cresol insolubles (CI) in the filtrate, it was found that the SRC product ash content remained significantly higher than the specified maximum of 0.16 wt %. As the rate of Na_2CO_3 addition was reduced, the ash content in the filtrate decreased. It was found that while sodium carbonate is not appreciably soluble in process solvent, sodium sulfide is. The ash from the filtrate or SRC contained significantly more sulfate than did the ash from the ash cake.

Operating the USF filter with precoat gave generally better results than operating without precoat. Total cycle times were shorter, sluicing was required less frequently, and screen blinding was less severe.

Considerable corrosion data were gathered from resistance probes, test specimens, and inspections of the solvent recovery section. It was found that corrosion was the most severe in the mid-section of the T105 Fractionation Column, and that some of the trays were nearly destroyed. Resistance type corrosion probes were found to be good indicators of corrosion activity, although in some instances actual cor-

rosion rates of column materials were more than double the rates indicated by the probes. High-nickel alloys were found to have the best corrosion resistance, with Hastelloy-C at the top of the list in most cases. High iron alloys showed poor corrosion resistance. Sodium carbonate was shown to be highly effective in reducing the rate of column corrosion, as measured by corrosion probes.

VIII. FUTURE PLANS

The objectives of 1980 calendar year Wilsonville program will be:

- o To evaluate improved solid-liquid separation processes.
- o To provide technical support for a planned SRC-I demonstration plant program.
- o To establish improvements in process equipment and operation.
- o To evaluate SRC process improvements.

The scope of work which encompasses these objectives includes:

- o United States Filter Corporation (USF) vertical leaf filter.

Testing initiated in the fourth quarter of 1979 will be continued during the first quarter of 1980. Emphasis will be placed on operation without precoat and on cycle optimization studies.
- o Critical Solvent Deashing.

The CSD unit will be restarted after modifications to the first stage separator and deashing solvent processing system are complete. Emphasis will be placed on establishing a new baseline for CSD performance, reducing deashing solvent losses, and on providing scale-up data for the demonstration plant design effort.
- o Industrial Filter & Pump Corporation (IF) Candle Filter.

Studies will be initiated with a skid-mounted unit. Emphasis will be placed on demonstrating operational feasibility using freshly dissolved coal slurry, on operation with carbonaceous body feed, and on screen blinding.
- o Evaluation of SRC Hydrotreating.

External hydrogenation of process solvent and selected SRC fractions will be used to enhance solvent quality and to improve sulfur removal. Variables to be studied include external hydrogenation conditions, and dissolver coal space rate,

temperature, and pressure. Emphasis will be placed on demonstrating short residence time (SRT) operation in which the SRC plant is used primarily to produce SRC with minimal hydrogen consumption and the hydrotreater is used to rehydrogenate solvent, reduce sulfur and optimize yields.

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APPENDIX A
OPERATING LOG

October 1979

1. Run 172, using Kentucky 9 coal in the hot separator mode, was in progress. B103 Vacuum Column Preheater was bypassed to produce a feed containing 20% process solvent for the Critical Solvent Deashing (CSD) unit. A 24-hr material balance was started at 0200 hr.
2. The material balance ended on schedule. The United States Filter Corporation (USF) vertical-leaf filter was heated and pressure-tested.
3. A leak at the USF filter door was repaired by hot-bolting. The CSD unit was on standby.
4. H-Coal runs were started in the CSD unit. The USF filter system was cooled for inspection. Coal feed was stopped at 1700 hr.
5. Filter screen damage (caused by the rubbing of a sluice arm) was discovered.
6. Roller guides for the filter leaves were added. The filter system was brought up to run conditions.
7. Three filter cycles were completed. Filtrate clarity was unsatisfactory, so the filter was cooled for inspection.
8. Inspection of the USF filter showed screen damage at a weld. One of the sluicing nozzles was also purged.
9. The USF filter completed two cycles with improved filtrate clarity. Coal feed was begun at 0230 hr.
10. A small leak was found on E102 Dissolver Product Cooler.
11. In general, the plant ran well.
12. Coal feed was reduced from 450 lb/hr to 350 lb/hr.
13. The H-Coal runs were completed in the CSD unit, after which filtered material from the SRC plant was fed to the CSD unit. Coal feed was increased to 450 lb/hr.
14. Unfiltered material from the SRC plant was fed to the CSD unit. The USF filter leaves could not be turned.

15. Coal feed was stopped at 1900 hr. The USF filter was cooled for inspection. The CSD unit was shut down for modifications.
16. Inspection of the USF filter revealed cake build-up on the sides around the roller guides and between the back leaf and the rear filter head. The leaf cut-off blades had become wedged on their stops causing binding of their hubs on the leaf shaft.
17. Replacement of the USF filter door gasket delayed heating the system. Coal feed was started at 1700 hr.
18. The USF filter was shut down to repair a door gasket leak. Coal feed was stopped at 1545 hr.
19. The USF filter was restored to service and coal feed was resumed at 1245 hr.
20. Coal feed was increased from 375 lb/hr to 450 lb/hr to provide feed for the USF filter.
21. In general, the plant ran well.
22. A cake compressibility test was run on the USF filter.
23. A gasket leak on a fuel oil filter shut down both B102 Slurry Preheater and B204 Dowtherm Heater.
24. The USF filter was cooled for inspection. A pinhole had developed near the edge of a filter screen. Coal feed was stopped at 1850 hr. The USF filter was returned to service.
25. The filtrate clarity worsened in the fourth cycle after sluicing. The filter was prepared for inspection.
26. Numerous holes were found in previously silver-soldered areas of two screens. The filter was reassembled with different screens. On the first filter cycle, the leaves would not turn so the filter was again shut down.
27. A gasket failure had caused a cutoff blade center hub to warp and bind on the leaf shaft.
28. The USF filter was returned to service. Coal feed was started at 0500 hr.
29. Sluicing of the USF filter after each cycle was discontinued.
30. Sodium carbonate addition to the coal slurry was reduced from 40 lb to 30 lb/batch. LCV415A was replaced after 3,220 hours of on-stream operation.
31. Specification SRC was produced. In general, the plant ran well.

November 1979

1. Run 181 was in progress on Kentucky 9 coal. The USF filter leaves would not turn, but sluicing on the next filter cycle freed the leaf drive motor.
2. Reprocessing of light organic liquid from V124A was started. Sluicing of the USF filter was done every 25 cycles.
3. In general, the plant ran well.
4. Settled solids were removed from the liquid waste sump.
5. Sodium carbonate addition to the coal slurry was reduced from 30 lb to 20 lb/batch.
6. The filter was sluiced twice due to high precoat differential pressure.
7. In general, the plant ran well.
8. Reprocessing of light organic liquid was completed.
9. Difficulties were experienced with ash discharge from the USF filter. The vibrator on the 10-in. dump chute from F125 to D102 would not start. A hammer was used to vibrate the chute.
10. Problems with the ash discharge from the USF filter continued. The filter was pressurized to 20 psig with nitrogen to aid in ash discharge.
11. The USF filter was cooled for inspection. Coal feed was stopped at 1100 hr.
12. Inspection of the USF filter showed two broken leaf cut-off blades. The 10-in. dump chute was cleaned. All solids were removed from V120.
13. The USF filter was returned to service. Coal feed began at 0700 hr. Sodium carbonate addition to the coal slurry was reduced from 20 lb to 10 lb/batch.
14. The pressure in R101 outlet was reduced from 2,100 psig to 1,700 psig. Recycle gas flow was reduced from 10,000 scfh to 8,500 scfh.
15. Specification SRC was produced. After sluicing, the USF filtrate solids content increased so the filter was cooled for inspection.
16. Two leaves of the USF filter had partially disintegrated. Coal feed was stopped at 0758 hr and was resumed at 2030 hr.

17. The USF filter was sluiced after 11 cycles due to a high precoat differential pressure.
18. Specification SRC was produced. Dowtherm content increased in the process solvent.
19. A Dowtherm leak was detected in the jacketed piping loop to K125.
20. Sodium carbonate addition to the coal slurry was reduced from 10 lb to 5 lb/batch. Recycle gas flow was reduced from 8,500 scfh to 8,000 scfh.
21. A cake compressibility test was run on the USF filter.
- 22 and 23. Specification SRC was produced.
- 24 and 25. In general, the plant ran well.
26. Specification SRC was produced.
27. Use of precoat in the USF filter operation was stopped temporarily. The 10-in. dump chute between F125 and D102 was cleaned.
28. Precoat was added to the USF filter to improve filtrate clarity.
29. The USF filter was cooled for inspection.
30. Inspection of the USF filter showed a partial leaf disintegration. Coal feed was stopped at 1400 hr.

December 1979

1. Run 189 was begun at 0130 hr with a recycle gas flow of 6,000 scfh. The USF filter was operated without precoat.
2. Filtrate clarities became increasingly poor so the filter was cooled for inspection. Recycle gas flow was increased to 8,000 scfh.
3. Inspection of the filter revealed a blown gasket between leaves. Coal feed was stopped at 0858 hr. The reaction section was depressured so that B102 Slurry Preheater could be decoked.
4. R101 was opened for inspection and cleaned. Leaks in E102 were repaired, V120 was cleaned, and B102 was decoked.
5. R101 was reassembled and the drawoff point was changed from the 50% to the 75% volume level.

6. The filter was returned to service. The reaction section was pressure-tested.
7. The reaction section was heated. The filter was not used because of a lack of feed.
8. Coal feed was started at 1600 hr and the filter was returned to service. Sodium carbonate addition to the coal slurry was increased from 5 to 10 lb/batch.
9. Filter operation was suspended until repairs to XV754 were completed.
10. In general, the plant ran well.
11. Specification SRC was produced.
12. A 24-hr material balance was started at 1200 hr. Specification SRC was produced.
13. The first 24-hr material balance was completed and a second material balance was started.
14. After the second material balance was completed, the R101 outlet pressure was reduced from 2,100 psig to 1,700 psig. Recycle gas flow was increased from 10,000 scfh to 11,500 scfh. Sodium carbonate addition was increased from 10 to 15 lb/batch of coal slurry. Specification SRC was produced.
15. The frequency of sluicing the USF filter was changed from each twenty-five cycles to each five cycles to improve filtration time.
16. Specification SRC was produced.
17. Filtration time remained high. Sluicing frequency was changed to each cycle without improvement. The filter was cooled for inspection. Coal feed was stopped at 1900 hr.
18. An inspection of the filter revealed small holes at the spot welds. Sluicing of the leaves had prevented blinding.
19. Coal feed was started at 0830 hr. Sodium carbonate addition was increased from 10 to 20 lb/batch of coal slurry.
20. Specification SRC was produced, but filtrate clarities became poor and the filter was shut down for inspection.

21. Coal feed was stopped at 0155 hr. Inspection of the filter revealed holes in one leaf. The leaf was replaced and the filter system was returned to service.
22. Coal feed was started at 1015 hr. Solids in the filtrate increased. Precoat was added to the filter to improve filtrate clarity.
23. Solids in the filtrate did not improve so the precoat time was changed from 10 to 20 minutes.
24. Solids in the filtrate continued to increase.
25. The filter was cooled for inspection. Coal feed was stopped at 1013 hr and the reaction section was depressured to permit relocation of electric supply lines.
26. The cooling tower was emptied and cleaned. B103 Vacuum Column Preheater burner was converted to burn propane as fuel. Inspection of the filter showed holes in one leaf.
27. Small leaks on E102 were repaired. Electrical power was off.
- 28 and 29. V120 was cleaned. R101 bottom flange started to leak on startup. The reaction section was depressured to repair the leak.
30. Coal feed was started at 1925 hr. When the filter system was returned to service, XV654 malfunctioned.
31. The filter was returned to service after the valve was repaired.

APPENDIX B
WASTEWATER TREATMENT SYSTEM

In February 1979, an activated sludge water treatment system was placed in operation at the Wilsonville pilot plant. Its purpose was to secure compliance with Alabama Water Improvement Commission (AWIC) and National Pollutant Discharge Elimination System (NPDES) regulations regarding wastewater discharge to Yellow Leaf Creek.

The following NPDES regulatory limits apply as of 26 July 1979:

<u>Component</u>	<u>Allowable Daily Average</u>	<u>Allowable Daily Maximum</u>
BOD, mg/l	30.0	60.0
Phenol, mg/l	0.25	0.5
Oil and Grease, mg/l	10.0	15.0
Total Suspended Solids, mg/l	30.0	60.0
Settleable Solids, ml/l	1.0	1.0
pH	6.0-9.0	6.0-9.0
Temperature, °F	95	100

The effluent treatment system is shown schematically in Figure B-3. A description of the study which culminated in the design of the Wilsonville effluent treatment system is contained in the report, "Treatment of Wastes Originating from a Coal Conversion Pilot Plant", which is included in this Appendix. Treatability data for the various biological bench-scale systems tested during the study are contained in Appendix B-1.

Results of wastewater treatment operations at Wilsonville are summarized as a series of trend charts in Appendix B-2. Operating problems during 1979 were minimal. As much as 29,000 gal/day of wastewater was treated during rainy periods or SRC plant upset conditions without significant deterioration in effluent quality. Removal of BOD has consistently been above 98% and removal of phenolics has exceeded 99.5%. The addition of activated carbon to improve phenolics removal was discontinued except during periods of organic shock loads. Under normal conditions, phenolics removal was satisfactory without carbon addition; furthermore, small particles of carbon tended to plug the sand filter.

Plugging of the final filter was caused by solids escaping over the clarifier weir. Filter performance was improved by increasing or decreasing the food-to-microorganism ratios in the aeration basins. The ratio adjustment was based on the BOD and COD of the influent to the activated sludge.

Given the stringent applicable regulatory limits, overall performance of the wastewater treatment system to date has been excellent. Few modifications were necessary and maintenance has been low.

TREATMENT OF WASTES ORIGINATING
FROM
A COAL CONVERSION PILOT PLANT

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(ABSTRACT)

With increasing need for low sulfur coal, the use of solvent refining techniques to reduce sulfur content will occupy an important place in the overall coal industry. At the present time only pilot facilities exist using this technology but prototypes are now under design and full-scale plants will shortly follow. Subsequently, the treatment of waste originating from these plants will be of major concern.

A treatability study for handling these wastes was conducted at a pilot facility operated by Catalytic, Inc. in Wilsonville, Alabama. The facility is sponsored by Southern Company Services and is funded by Electric Power Research Institute (EPRI) and the U.S. Department of Energy (DOE). As a result of this study a treatment scheme was developed and facilities were designed and constructed for treatment of the wastes.

In order to comply with stringent stream quality effluent requirements, treatment methods for removing hydrogen sulfide, substituted phenols and other organics were investigated. These included the use of hydrogen peroxide, activated carbon and biological treatment. Many variations of the biological treatment process were investigated such as completely mixed systems, two-stage systems and enhancement by addition of powdered activated carbon and mutant bacteria.

The study was conducted over a six-month period. The results, comparisons of the various systems, and the final solution will be discussed.

INTRODUCTION

Catalytic's Environmental Systems Division conducted a treatability study on the wastewaters originating from a solvent refined coal facility located in Wilsonville, Alabama. The facility is a pilot plant operated by Catalytic, Inc. and sponsored by Southern Company Services. The entire pilot plant project is funded by the Electric Power Research Institute (EPRI) and the U. S. Department of Energy (DOE). Although the pilot plant operation does not generate wastes representative of a demonstration or full scale facility, (one of the major sources of pollution in the pilot plant is a caustic scrubber blowdown which will be replaced by some sort of sulfur recovery in a larger installation), it does present some unique and difficult constraints for wastewater treatment.

The major part of the study was performed on-site at the Wilsonville plant during the period of February through April 1978. Some subsequent laboratory work was conducted at Catalytic's Environmental Laboratory in Linwood, PA. On the basis of this study a design for a full-scale wastewater treatment facility was developed. The treatment plant has been designed and constructed, and is now in operation.

INVESTIGATIVE PROGRAM

The first phase of the study involved an investigative program to determine the characteristics and treatment techniques that would be applicable to the wastewaters. Several different biological unit processes and treatment-train combinations were screened experimentally to establish feasibility. Then, bench-scale systems were operated to develop design data and determine effluent quality. Wastewater characterization was an inherent element of this effort. The characterization data derived during the study were used in conjunction with historical data available from the plant to establish the design waste load.

Wastewater Characterization

Two wastewater streams emanate from the process areas. The first comes from the Waste Caustic Sump (WCS) and contains process contact waters including the blowdown from a caustic scrubber which removes sulfur and organic impurities from the solvent in the process area. The second wastewater stream comes from the Liquid Waste Sump (LWS). The sump collects wastewaters generated from a continuous cooling tower blowdown, boiler blowdown, process vessels removing trace quantities of water from the process stream, heat trace drip legs, and the yard sump collecting runoff in the tank farm area. The WCS wastewater is a high strength waste containing caustic, sulfides, phenolics, high COD and high BOD. The LWS wastewater is a relatively low strength waste, generally containing less than 50 mg/l BOD and 1 mg/l of phenolics and sulfides.

Waste characteristics for the two waste streams and for the combined stream are shown in Table B-1. The combined stream data is after sulfide oxidation of the caustic stream had been accomplished. This combined stream was the feed to the biological unit.

Screening Test

With the waste stream characterized, the next step in the study was to conduct a number of screening tests. These tests are designed to provide a quick, inexpensive qualitative indication of the technical feasibility of various unit processes to determine the processes that were to be investigated further on a continuous basis.

The first series of screening tests conducted dealt with finding an economical method of oxidizing the sulfides present. Prior laboratory work had been performed at Wilsonville and in Catalytic's home laboratory. This work was an interim effort aimed primarily at peroxide oxidation of sulfides and phenolic compounds. The chemical doses involved were economically prohibitive. A method utilizing air as the oxidant was investigated and batch testing was performed using different catalysts, reaction times, and sulfide concentrations. The effect of temperature and pH were also evaluated. The data from the screening tests for developing an air oxidation method of oxidizing the sulfide can be found in Table B-2.

From these tests it was determined that 24-hour duration without pH adjustment, using 200 to 400 mg/l of manganese sulfate as a catalyst, would normally reduce sulfide concentrations to acceptable levels for biological treatment without the release of hydrogen sulfide. At times, however, this method did not succeed in reaching the desired levels of sulfides; and when this occurred, peroxide was used as a polishing step.

The next series of tests dealt with methods for removing organics from the waste streams. As alternatives to biological treatment of the wastewaters, activated carbon adsorption and distillation were investigated. While carbon adsorption proved to be successful at removing the phenolics and other organics present, the projected operating costs were extremely high, and consistent BOD removal to required levels was questionable. For this reason, further evaluation of the process was abandoned. Distillation of waste caustic was unsuccessful at removing the organics and was also abandoned as a possible treatment step.

Other methods utilizing chemical oxidation techniques were examined as possible alternatives to biological treatment. Ozonation was considered, but discarded because of economics. Chlorine oxidation was considered briefly, but was excluded from further consideration because of the possibility of forming chlorinated phenols and other toxic chlorinated organics.

Treatability-Testing

Sulfide Removal

The efficacy of hydrogen peroxide treatment for sulfide removal had been established in prior work and this technique was utilized initially to generate equalized wastes for evaluating biological treatment. Once the benchscale bio-units were established and acclimated, some preliminary operating parameters were developed. Following this acclimation/stabilization period and as time became available the screening work was done on alternative pretreatment technology as described in the previous section. On or about 28 March, the daily peroxide oxidation pretreatment of the waste caustic was changed to 24-hour continuous catalyzed air oxidation as determined from the screening tests. A vessel with a 5-gallon capacity was continuously fed raw caustic waste with

400 mg/l of manganese sulfate added. The diffused air was supplied at the rate of 9.4 cfh. Influent and effluent sulfide and pH were measured once a day. Effluent was collected in a holding vessel and batches that exceeded 20 mg/l of sulfide were "polished" with peroxide before neutralization. The oxygen uptake was measured daily and ranged between 8 and 10 mg/l per hour.

Sludge formed during the oxidation step remained in the aeration basin. This sludge was collected from the tank and measured. The quantity of sludge resulting from this step was approximately 35 ml per 5 gallons of 12 percent (dry solids sludge) concentration. No chemical analysis was made, but it was dark brown with small and discrete particles. Although the sludge blanket was fairly stable, it would flow even after several days of compaction.

Removal of Separable Organics

The characterization studies indicated that the waste caustic stream contained organics that would separate either by floating or settling. These organics could then be removed and either reprocessed or used as fuel. To simulate this removal, the waste caustic samples were allowed to settle for 30 minutes before and after sulfide removal. Separable organic volumes were estimated, and the wastewater was removed from the middle. From these tests, it was estimated that 5 ml of organics per 5 gallons of waste could be removed.

Neutralization

The waste caustic stream had, of course, a very high pH of 12 to 13. Before this was fed into the biological reactors, it was adjusted to a pH between 6 and 8, using concentrated sulfuric acid.

Equalization Storage

Due to the variability inherent in the pilot plant operation, it was decided to provide a minimum of a 10-day equalization volume. Also, knowing that the production facilities could cease operation for fairly long periods of time for maintenance or modifications, flow rates to the biological unit would be cut back to one-half the design rate providing a sufficient feed volume for sustaining the biological system for a shut down period lasting as long as 20 days. Historical data for the production facilities showed that operational shutdowns rarely exceeded this period of time.

In the continuous treatability testing, a 10-day equalization time was provided and, whenever the production facilities were shut down, the feed was cut back to the biological units until the production facilities resumed operation. From this it was possible to determine if any adverse effects would result to the systems either during a shutdown period or on resuming normal operations of the biological system.

Biological Treatment

Three types of biological systems were investigated during the study period: rotating biological contactors; aerated lagoons; conventional activated sludge. Table B-3 shows a comparison of the three types of biological systems tested.

The rotating biological contactor used in the study was a bench-scale unit consisting of four stages with four discs per stage. Each disc was 11 inches in diameter. After several weeks, this system was abandoned as not feasible since the biological film would not attach to the discs; and it was evident that this unit would not be applicable due to low BOD removal efficiencies.

Aerated lagoons and activated sludge were shown to be effective in removing BOD and phenol concentrations at Food to Microorganism (F/M) ratios below 0.25. F/M ratios in the biological system tested ranged from 0.07 up to about 0.4. Figure B-1 shows a plot of F/M ratio vs percent BOD removal. From this curve, it is evident that loading rates about 0.25 show a drastic drop off in efficiency.

Even though BOD removal efficiency was better than 96% for BOD and 98% for phenolics, stream effluent criteria were still not achieved, as shown in Table B-4. For this reason, additional testing was conducted to remove the 2 to 7 mg/l of phenolics remaining in the wastewater down to the 0.1 mg/l criteria. To do this, two types of activated sludge enhancement systems were tried. The first was to use bacterial enhancement as a means of removing residual phenol concentrations to the acceptable level. Bench scale evaluations were made in concentrations to the acceptable level. Bench scale evaluations were made in the Catalytic laboratory. No advantage was observed in phenol removals with the use of bacterial additives. It should be pointed out that bench scale evaluation of bacterial additives is difficult, except for a qualitative indication of its effectiveness.

Powdered activated carbon addition to the biological system for removal of the trace quantities of phenols still remaining in the wastewaters was then investigated. Table B-5 shows the results of this pilot study. The system tested had a total hydraulic retention time of 7 days. Each day, powdered activated carbon was added to the units. In all, four dosages of powdered carbon were investigated ranging from 0.3 g/l to 6.4 g/l. The lower dosages (0.3 to 1.2 g/l) approached the effluent limitation on phenols, but did not consistently meet this limit. The high dosage of 6.4 g/l would meet limitations but the practicality of using such a high dosage was questionable.

This lead to investigating the applicability of using a two-stage biological system, since the wastewater contained a large portion of readily biodegradable substrate as indicated by the conventional system tested. It was anticipated that the first stage of the system would remove the readily biodegradable organics, leaving the more difficult organics for the second stage. In such a system, two biological cultures develop, one to remove the easy-to-degrade organics, and the second to remove the hard-to-degrade organics such as substituted phenols. In a one-stage system, the bioculture tends to attack the more easily degradable organics first, leaving the more difficult. In the second stage of a two-stage system, a biological culture acclimated to the difficult-to-degrade organics will dominate.

Therefore, a two-stage system was set up with a total of 5 days retention time, 2½ days in each stage. Each stage had an internal sludge recycle so that only the effluent from the first stage passed on to the second stage. To insure phenol removal, powdered activated carbon was added to both stages. Figure B-2 shows a schematic sketch of the bench-scale system used.

The system was operated for three months and the results are shown in Table B-6. Powdered carbon was added to the first stage at a dosage of 0.4 g/l, and to the second stage at a dosage of 0.2 g/l. From the results, it is seen that the system can consistently produce an effluent that will meet the standard of less than 0.1 mg/l of phenolics.

PROCESS DESIGN

General Requirements

In addition to the difficulties associated with the waste compositions generated by coal conversion, there were several items affecting design of a treatment system that were unique to the pilot plant operation in contrast with a full-scale plant.

There is no desulfurization unit in the SRC plant. Instead, a caustic scrubber is used to remove sulfides, CO₂ and organics, and is one of the major sources of pollutants. The high levels of sulfides present in the waste because of this arrangement would not be present with a desulfurization unit. The wastewater volumes are relatively small (although concentrated) which allow using relatively large hydraulic capacities to gain flexibility and ease of operation in the treatment process. This SRC process uses widely varying coal types at different times. The process is constantly being modified, operations are changed, equipment is added, and there are also frequent periods when the unit is not operating and the wasteload is drastically reduced. The other major item is the stringent effluent requirements for discharge based on stream quality criteria. The receiving stream is a recreational water body, and effluent concentrations must be met regardless of the treatment removal efficiencies attained. This can require overall removals in excess of 99% for several parameters.

Thus, a flexible treatment system was needed, capable of advanced wastewater treatment. The system would have to respond to changes in wastewater characteristics and matrix, handle severe hydraulic variability, remain operational during process shut-downs and turn-arounds, and then respond with full treatment capability upon process start-up, and consistently meet the stringent effluent requirements. In addition, the system would have to handle spills and contaminated stormwater runoff.

Description of Facilities

Basis of Design

Based on the treatability work that was conducted, a system design was made for the treatment facility which consisted of oil removal, batch catalyzed air oxidation of the caustic waste at the raw pH to remove sulfide, a polishing step using hydrogen peroxide (if necessary), pH neutralization, equalization, combination with the liquid wastes in treating the combined stream in a package two-stage activated sludge bioreactor. The total system includes the capability to store spills, excessively contaminated storm water, or the effluent from the bio-systems for three days and recycle it back for treatment. A flow sheet showing the proposed system design is shown in Figure B-3.

Separable Organic Removal

The caustic waste contained quantities of separable organics, and prior to pretreatment, these should be removed. For this purpose a package carbon steel cil separator is provided. The unit is designed to handle the maximum flow rate of 20 gpm of caustic wastes, and remove the floatable and settleable organics present in the waste stream. From this unit, the caustic waste will flow by gravity to the pretreatment facilities.

Sulfide Removal

The sulfide removal facilities consist of a two-chambered concrete basin which receives the high pH caustic waste. Each chamber is 14.5 ft. by 16.0 ft. by 10.5 ft. deep and holds a minimum of 10,000 gallons. Diffused air at a rate of 250 SCFM per basin for mixing and sulfide oxidation is provided by two blowers, one for each chamber with cross-connected discharge piping to allow either blower to be a spare for the other. Granular manganese sulfate is added manually from bags. Hydrogen peroxide is provided at a 50% concentration from 55-gallon drums, when required.

The basin also serves as a batch neutralization unit. A 4600 gallon concentrated sulfuric acid tank is provided. The tank is above ground, and acid is added by gravity and controlled by manually-operated valves.

The sequence of operations for the sulfide removal facilities is to first add the manganese sulfate to the tank and fill the tank while aerating. When a chamber is filled, aeration is continued for 24 hours. The waste is then analyzed for sulfide concentration and, if below specified concentration, neutralized and transferred to the equalization/storage tank. If not below the specified sulfide concentration, peroxide is added and the chamber is aerated to mix. Sulfide concentration is then rechecked before neutralization and transfer.

It is necessary to remove sulfides before any neutralization and subsequent treatment because even the slightest reduction in pH causes hydrogen sulfide to be released from the wastewaters. Pretreatment with air reduces and, at times, eliminates the need for chemical conversion of sulfide, thus reducing chemical costs. The batch operation provides the most positive control assuring that there is no release of hydrogen sulfide, and that the waste is properly treated. Therefore, the vessels are sized so each one can handle the maximum daily waste caustic flow. If the air oxidation is inadequate on any given day, hydrogen peroxide can be added to finish converting the sulfide before neutralization.

Equalization/Storage

The equalization/storage tank is a 55,000-gallon 30 ft. by 30 ft. by 10.5 ft. deep, below grade concrete basin which serves several functions. It provides for concentration/equalization of pollutants, acceptance of hydraulic surges, settling and storage of the organic sludge generated in the pretreatment step, and sufficient wastewater storage capacity to continue feeding the bioplant during periods when the SRC plant is not operating. The tank receives the 100-gpm flow of pretreated neutralized wastewater, as well as the waters from the emergency holding basin, when necessary. The incoming waters discharge into a dispersion device below the liquid level away from the draw-off point to avoid disturbing the settled sludge. The draw-off point is above the sludge level (approximately 2.5 feet above the bottom of the basin) and protected by a baffle

to avoid picking up sludge. This arrangement provides at least a year's worth of sludge storage capacity. At present, 13 to 15 gallons per batch are generated (4,500 gallons per year). Sludge removal will be by a portable sludge suction pump. The sludge, at an estimated sludge solids concentration of 12%, will be dewatered with the biosludge. A good liquid level indicator is essential to keep track of the feed inventory in order to keep the biosystems running during the period when the SRC pilot plant is not. A variable flow pump constantly transfers a steady flow to the biounit.

The flow from the liquid waste sump is mixed with the equalized caustic waste just prior to biological treatment.

Biological Treatment

Due to the relatively small size of the biological treatment system required, a packaged treatment system of steel construction was installed. Figure B-4 shows a schematic sketch of the packaged system. This system consisted of two aeration tanks in series, each with a volume of 37,500 gallons: an intermediate hopper bottomed clarifier 8 ft. x 8 ft. square; a final clarifier 10 ft. in diameter equipped with a sludge removal mechanism; sand filtration facilities; and aerobic digester, and three 310 SCFM blowers for providing air to the aeration basins, aerobic digester and to the air lifts included in the package. The need for the sand filter became evident during our study when poor effluent solids quality was observed in our settling tests.

The stabilized sludge is combined with the sludge taken from the equalization/storage tank and dewatered using sandbeds. It is estimated that 50-100 gallons per day of sludge will be wasted from the biological system.

Since phenolic removal is very much dependent on temperature, both aeration tanks are below grade and covered for heat conservation. Should temperature levels become too low, provisions have been made to install heating elements in the tanks. In addition, as insurance for meeting the stringent phenol limitations, provisions for adding powdered activated carbon to the influent of both aeration tanks using a pump and eductor system is included in the design.

The wastewater is deficient in phosphorus needed for biological activity. Phosphoric acid will be used to supply this nutrient, and will be added to the first stage aeration basin.

A pH control system is provided for in the first stage to maintain pH between 6 and 8. Sulfuric acid from the sulfuric acid storage tank will be used for pH control and will be supplied using a metering pump.

Emergency Holding

A 50,000 gallon, below grade concrete emergency holding basin is provided in the system to prevent contaminated storm water, excess process wastes, spills and improperly treated effluent from being discharged. The basin has a small chamber at one end equipped with a V-notch weir for measuring and recording the final effluent flow before discharge. Should an upset in the treatment plant occur, a valve on the discharge line is shut and the flow will back up and overflow into the main basin. This basin also receives excessive rainwater from process areas and all overflows from the liquid waste sump. Pumping

facilities are provided at the basin to recycle the contents back to the treatment system. If the contents of the basin is clean storm water, these same pumps can discharge directly to the receiving stream. Analytical tests are made on the contents of the basin before any direct discharge is made.

CONCLUSIONS

Although there will surely be many differences between the pilot scale SRC operation and any future larger scale installations, many of the pollutants associated with coal conversion will be common to each in varying quantities and mixtures. These coal by-product pollutants such as polycyclic aromatics, substituted phenols, and sulfur compounds are unique and often difficult waste treatment problems. Coupled with this were the difficulties specifically associated with the pilot scale operation and also the stringent effluent requirements. In general, we were presented with a complex and unique set of parameters affecting feasibility assessment, design, and operation.

The study showed however, that the wastes originating from this pilot scale SRC process are biodegradable, and removals of between 95 and 98% of BOD and phenolics could be expected in conventional biological systems. Due to the stringent limitations for phenolics on the plant effluent, however, a two-stage biological treatment system was necessary. Pretreatment of the waste caustic stream before biological treatment using air with manganese sulfate as a catalyst, was shown to be effective in reducing sulfide concentration to acceptable levels without evolving H₂S. Overall system design required flexibility and adaptability while, at the same time, maintaining a high level of process removal efficiency.

TABLE B-1
WASTE CHARACTERISTICS

	Waste Caustic Sump (WCS)	Liquid Waste Sump (LWS)	Combined*
Flow - gpd	2000 - 8300	1900 - 15,000	7600 - 15,600
pH	9.8 - 13.5	3 - 11	5.5 - 8.3
BOD - mg/l	250 - 2500	7 - 23	360 - 1600
COD - mg/l	900 - 5500	----	800 - 2800
phenolics - mg/l	30 - 800	<1.0	120 - 500
Oil and Grease - mg/l	20 - 110	1 - 15	5 - 70
Sulfides - mg/l	70 - 3400	<1.0	<20
NH ₃ -N - mg/l	100 - 200	----	16 - 70

*Streams combined after catalyzed air oxidation and equalization of WCS

TABLE B-2
AIR OXIDATION OF SULFIDES

pH Adjusted to 8.5

Beaker No.	Chemical	Dosage mg/l	time 0 pH	S (mg/l)	60 min pH	S (mg/l)	120 min pH	S (mg/l)	1440 min pH	S (mg/l)
1	Blank	0	8.5	1456	9.2	1456	9.2	1232	9.2	13.4
2	KMnO ₄	30	8.5	1456	9.2	1288	9.2	1232	9.2	4.7
3	KMnO ₄	75	8.5	1456	9.3	1344	9.2	1176	9.2	1.34
4	MnSO ₄	40	8.5	1456	9.4	1176	9.3	952	9.3	0.45
5	MnSO ₄	100	8.5	1456	9.4	1120	9.3	896	9.3	0.3
6	MnSO ₄	200	8.5	1456	9.6	896	9.5	597	9.5	0.23

Raw Sample: pH 13.0; sulfide 2508 mg/l; phenol 250 mg/l

Raw pH

Beaker No.	Chemical	Dosage mg/l	time 0 pH	S (mg/l)	60 min pH	S (mg/l)	120 min pH	S (mg/l)	1440 min pH	S (mg/l)
7	Blank	0	13.1	3360	13.1	2464	13.1	2128	13.1	261
8	KMnO ₄	75	13.1	3360	13.3	2240	13.3	1736	13.3	72
9	MnSO ₄	200	13.1	3360	13.4	1904	13.4	1680	13.3	27

Continuous vs Batch vs Temperature

	System #1	System #2	System #3
Type	continuous	batch	batch
Detention time	24 hours	24 hours	20 hours
Chemical dose	8 g MnSO ₄ /5 gal	8 g MnSO ₄ /5 gal	8 g MnSO ₄ /5 gal
Temperature	ambient	ambient	80°F
Effluent/final sulfide concentration	153 mg/l	165 mg/l	146 mg/l

Raw Sample: Sulfide 3378 mg/l; pH 12.8

Notes: 1. All attempts at adjusting pH of raw sample liberated H₂S. Sample was adjusted under hood to 8.5 and aerated as described above.

2. Acidifying beaker #9 to pH 7.0 after 1440 min evolved no detectable H₂S odor.

TABLE B-3
COMPARISON OF BIOLOGICAL TREATMENT UNITS

<u>Parameter</u>	<u>Influent</u>	<u>EFFLUENT</u>		
		<u>Activated Sludge</u>	<u>Aerated Lagoon</u>	<u>Rotating Biological Contactor</u>
BOD (mg/l)	360-1600	4 - 77	36 - 100	250 - 500
COD (mg/l)	800-2800	100 - 1000	275 - 1000	450 - 2000
Phenolics (mg/l)	120-500	2.0 - 7.0	2.0 - 7.0	---
pH	5.5-8.3	7.8 - 8.2	6.6 - 7.8	7.4 - 8.0
Retention Time	---	6 days	10 days	----
MLVSS (mg/l)	---	700	600	----
% BOD Removed (mg/l)	---	90 - 99	92 - 98	30 - 70
% COD Removed	---	60 - 85	70 - 80	30 - 50
% Phenolics Removed	---	95 - 99	95 - 99	----
F/M Ratio	---	0.25	0.20	----

TABLE B-4
EFFLUENT CRITERIA

<u>Permit Requirements</u>	<u>Conventional Activated Sludge Effluent</u>
BOD mg/l	20
Phenolics mg/l	0.1
pH	6.0 - 9.0
Sulfide mg/l	0.5
Oil & Grease mg/l	< 10

TABLE B-5

POWDERED ACTIVATED CARBON ADDITION
TO ACTIVATED SLUDGE SYSTEM

<u>Carbon Dosage</u> g/l	INFLUENT			EFFLUENT		
	<u>BOD</u> mg/l	<u>COD</u> mg/l	<u>Phenolics</u> mg/l	<u>BOD</u> mg/l	<u>COD</u> mg/l	<u>Phenolics</u> mg/l
0.3 - 0.4	900	1400	180	9	90	0.20
1.2	1100	1900	180	12	100	0.25
6.4	1100	1900	170	< 5	110	< 0.1

Note: Activate sludge system operating at 7 day retention time with a 30 day sludge retention time.

TABLE B-6
TWO-STAGE ACTIVATED SLUDGE
WITH ACTIVATED CARBON ADDITION

Sample	Influent Phenol (mg/l)	Retention Time (Days)		Carbon Dosage (g/l)		* Eff Phenol (mg/l)	
		Stage 1	Stage 2	Stage 1	Stage 2	Stage 1	Stage 2
1	45	2½	2½	0.4	0.2	0.21	0.03
2	50	2½	2½	0.4	0.2	0.188	0.039
3	100	2½	2½	0.4	0.2	0.17	0.06
4	145	2½	2½	0.4	0.2	0.66	0.15
5	145	2½	2½	0.4	0.2	0.41	0.051
6	120	2½	2½	0.4	0.2	0.156	0.022

* Average Values

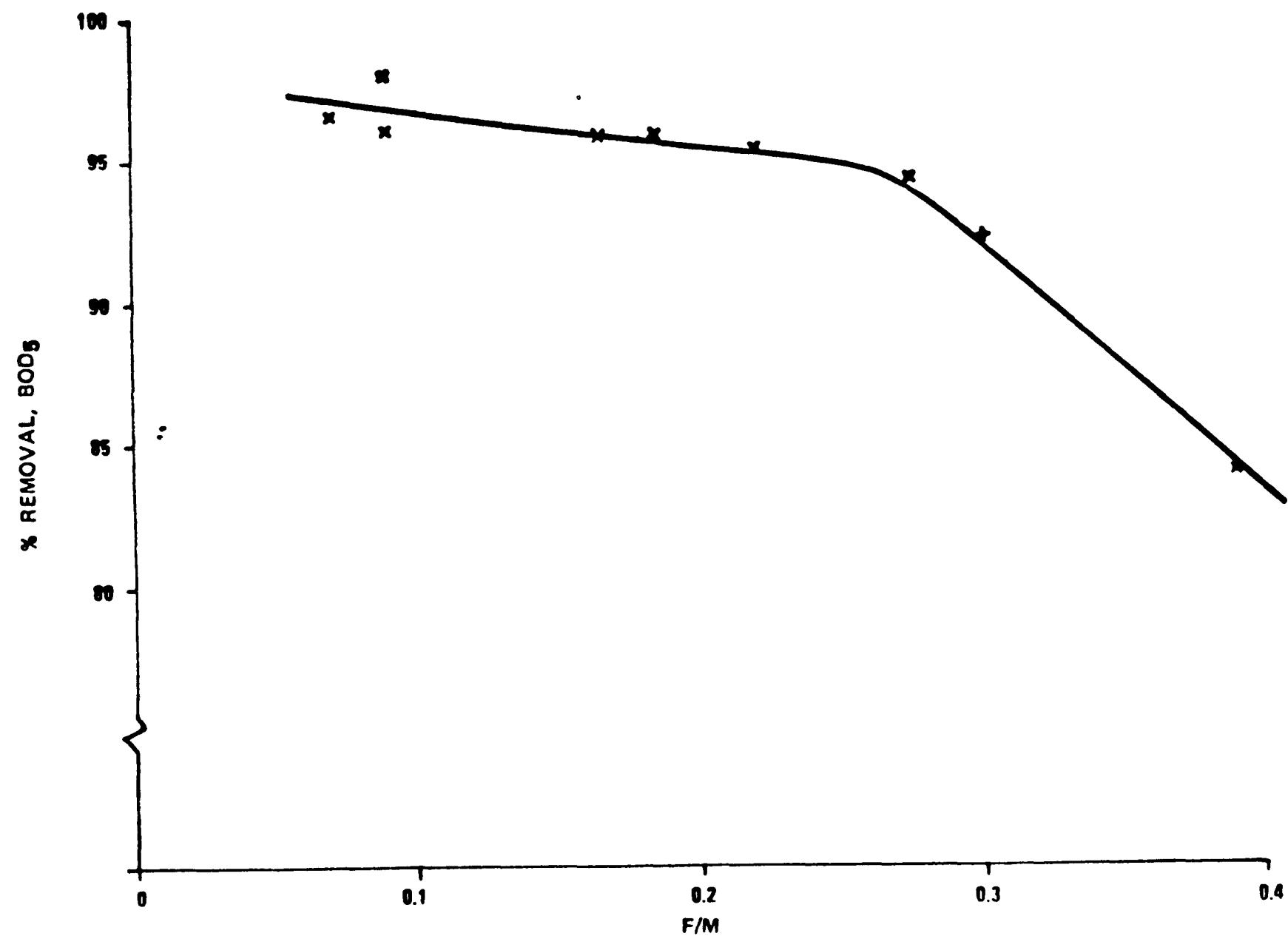


FIGURE B-1
% BOD₅ REMOVAL VS F/M RATIO

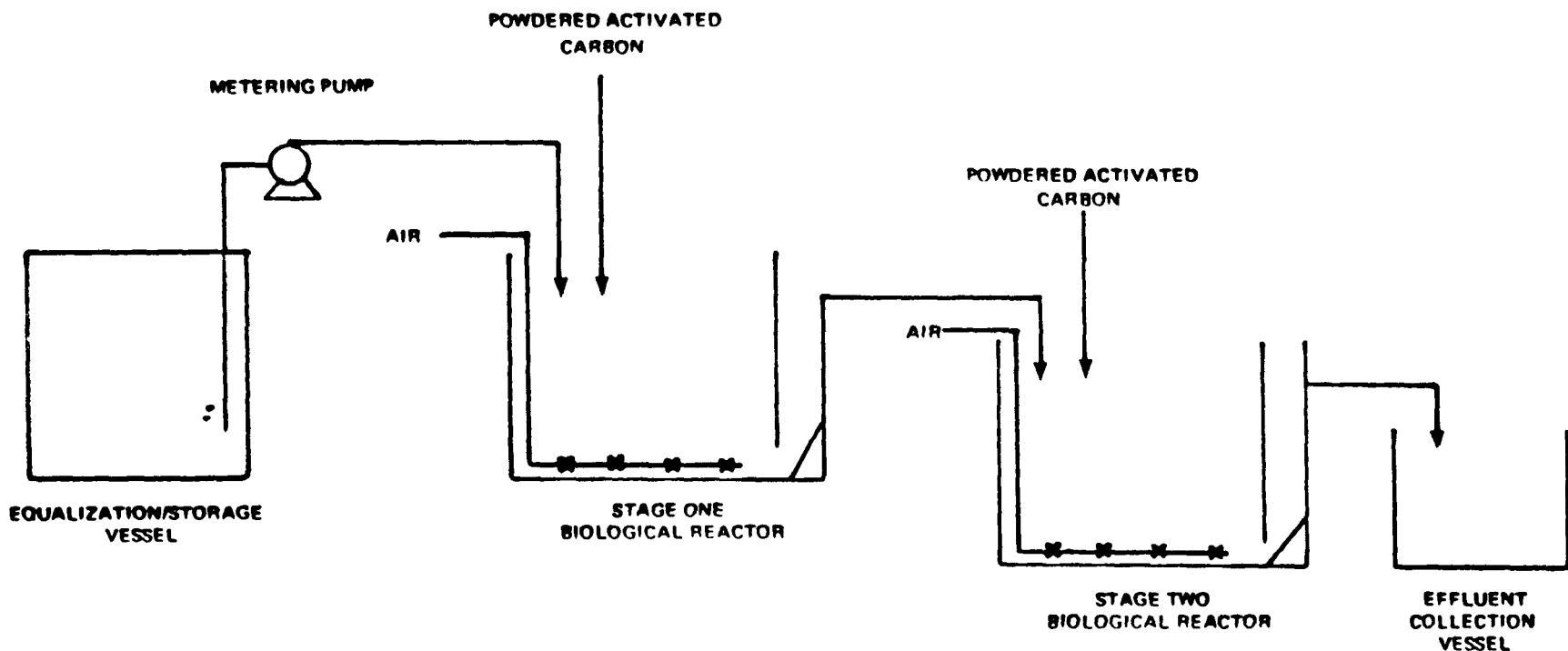


FIGURE B-2
TWO-STAGE BIOLOGICAL
BENCH-SCALE SYSTEM

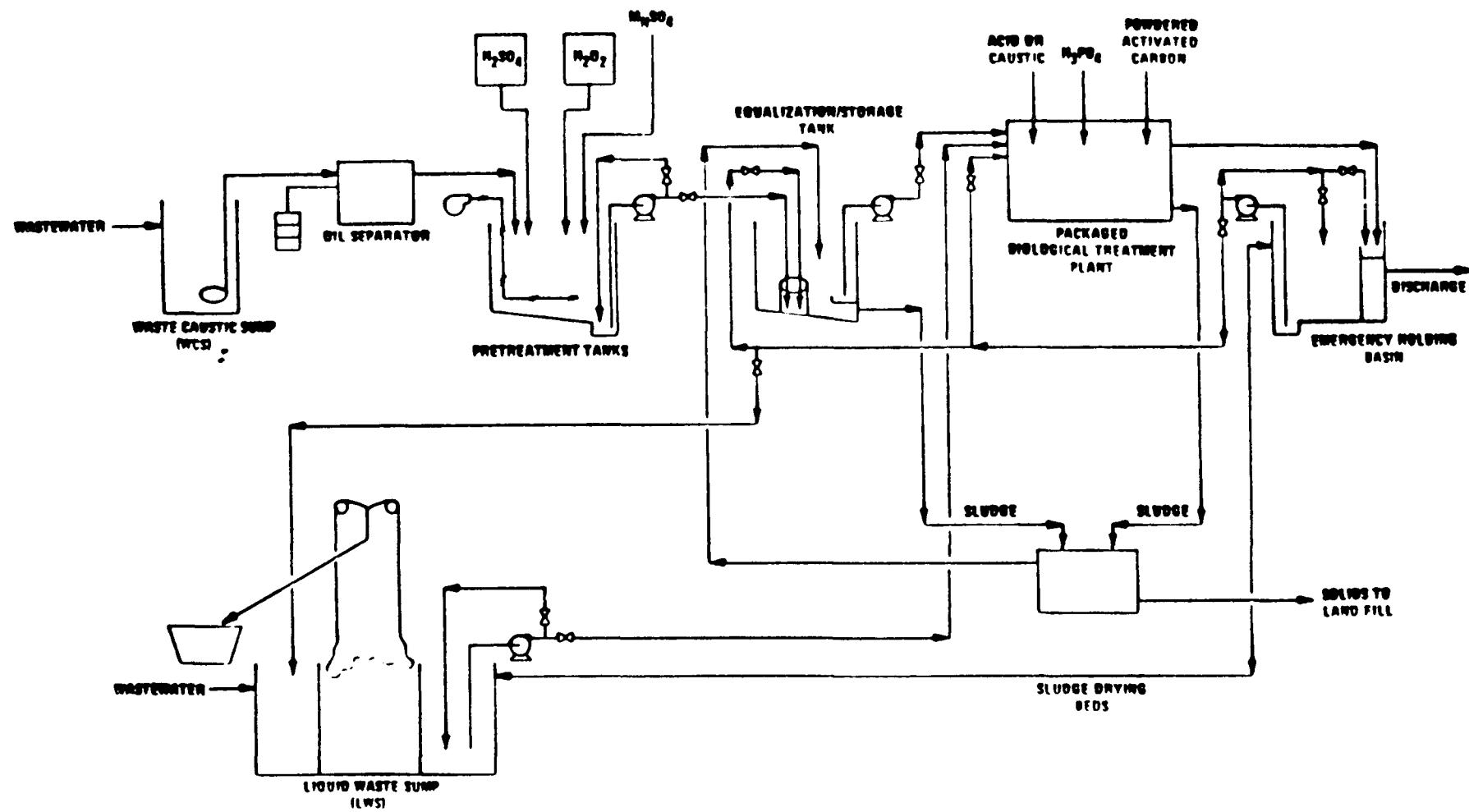


FIGURE B-3
WASTEWATER TREATMENT SYSTEM

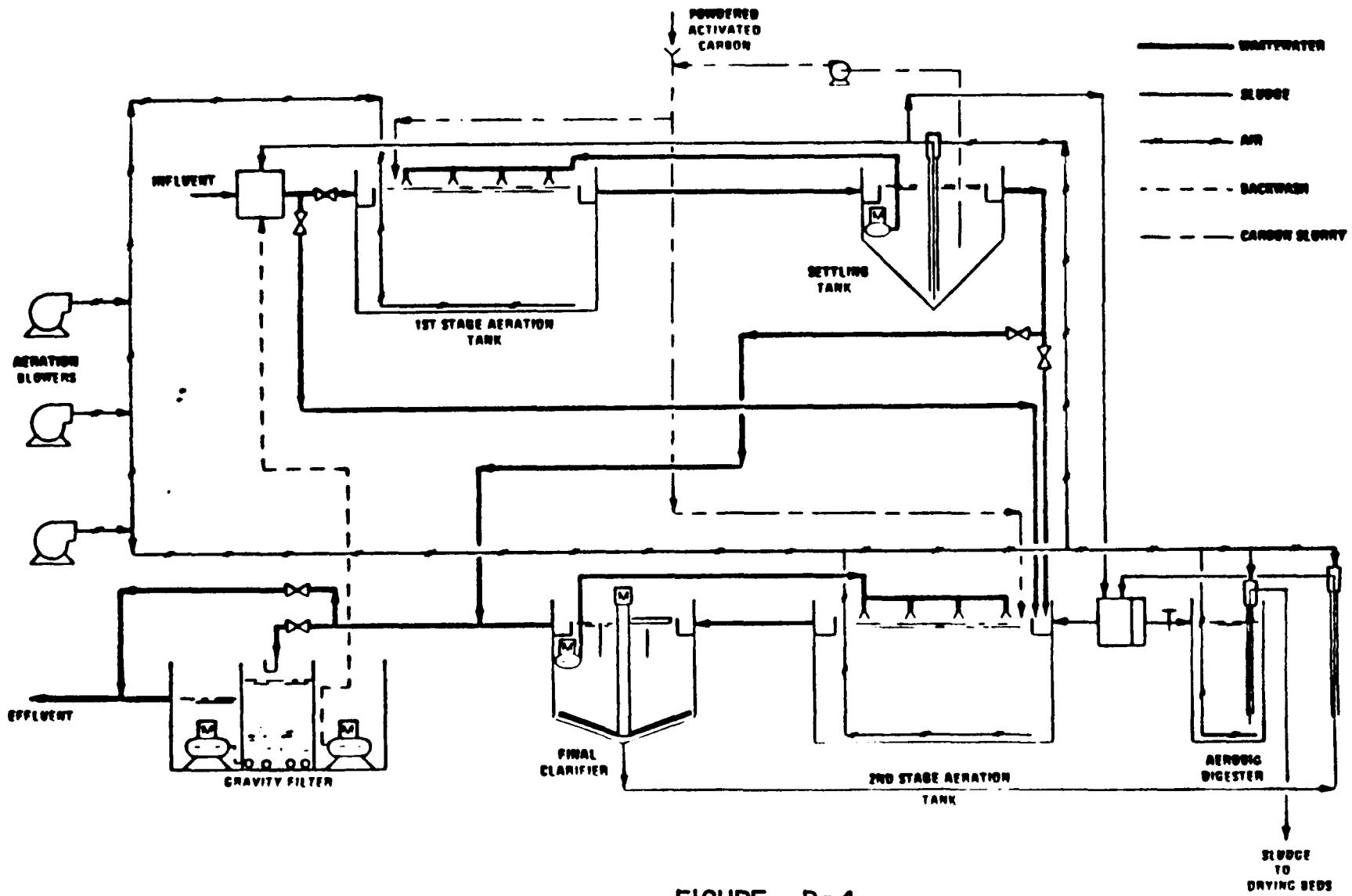


FIGURE B-4
PACKAGED BIOLOGICAL SYSTEM

APPENDIX B1
TREATABILITY TESTING: BENCH-SCALE BIOLOGICAL SYSTEMS

The following bench-scale biosystems were evaluated as described below in order to determine whether the waste caustic could be treated biologically, both by itself and in combination with the untreated, unequalized liquid waste. Data and observations were acquired to define operating parameters, develop design data and determine the projected effluent quality for meeting the AWIC discharge requirements.

System #1 - An activated sludge unit with 100% internal sludge recycle was fed undiluted equalized caustic waste. The feed rate was aimed at a 5-day hydraulic residence time. There was no sludge wasted from the reactor. However, there was continuous loss of suspended solids over the effluent weir. The system could not produce enough solids to maintain a reasonable F/M (Food/Microorganism) ratio under the described condition. The data are contained in Table B1-1.

System #2 - An aerated lagoon with an hydraulic detention time of 10 days was fed undiluted equalized caustic waste. The data are summarized in Table B1-2.

System #3 - A bench-scale rotating biological contacter was run at several different loadings using equalized caustic waste. After several weeks, it was abandoned as not feasible, since the biological film would not attach to the disc surface. The data are summarized in Table B1-3.

System #4 - System #1 was changed to combine waste beginning March 1st. The data are summarized in Table B1-4.

System #5 - An aerated lagoon with an hydraulic residence time of 4 days was fed combined waste. At an average F/M of less than 0.2, the average BOD removal was 96%. The data are summarized in Table B1-5.

System #6 - System #6 was a continuation of System #2. At an average F/M of approximately 0.2, the BOD₅ removal averaged 96%. The data are summarized in Table B1-6.

System #7 - System #7 was similar to System #5, except that 0.4 g powdered activated carbon per liter of influent per day was added to the aeration basin. The data are summarized in Table B1-7.

System #8 - An activated sludge system with a 6-day hydraulic residence time was fed combined waste. Sludge was wasted to maintain a 30-day sludge age. BOD removal averaged 96%. The data are summarized in Table B1-8.

System #9 - This was a system similar to System #8 with an average hydraulic residence time of 7 days. Powdered activated carbon was added in various doses to the aeration basin. Phenol removal averaged 99%, BOD₅ removal averaged in excess of 99%. Data are summarized in Table B1-9.

System #10 - An attempt was made to evaluate a commercially available biological supplement in a system similar to System #8. The data are contained in Table B1-10, however, the duration of the run was not sufficient to obtain any meaningful information due to the late arrival of the supplement sample.

System #11 - After leaving the field, a sample of combined waste was returned to Catalytic's Linwood, Pa. Laboratory. An activated sludge was run with a total hydraulic residence time of 5 days (2½ days per stage). There was 100% internal recycle of sludge on each stage. Each day 0.4 g of powdered carbon per liter of influent wastewater was added to the 1st stage and 0.2 g to the second stage. The sludge retention time was 15 days. The phenol data after system stabilization are shown in Table B1-11.

Settling - Two-liter settling tests were run during the study on several of the systems. The data are plotted in Figures B1-1 through B1-8. The sludge settled quickly and compacted, but supernatant clarity was not good. Supernatant suspended solids ranged from 100 to 400 mg/l.

Clarification - Jar tests were run to find coagulant aids that would improve effluent clarity. Inorganic coagulants such as alum, lime, and $FeCl_3$ were evaluated. Also, cationic, non-ionic, and anionic polymers from several manufacturers, both singly and in combination with other polymers and the inorganic salts, were tested. There were no promising results from this work.

Wilsonville SRC Pilot Plant
 Job #43080
 February 4, 1978 to February 28, 1978
 System #1

TABLE B1-1

BIO SYSTEM TREATABILITY
 Continuous Data

MIXED LIQUOR DATA												INFLUENT DATA							EFFLUENT DATA				
Date	Feed ml/day	pH	Temp °F	SS mg/l	VSS mg/l	DO mg/l	DO UPT mg/l/hr	COD mg/l	BOD ₅ mg/l	pH	TDS mg/l	TVDS mg/l	SS mg/l	VSS mg/l	COD mg/l	BOD ₅ mg/l	pH	SS mg/l	VSS mg/l				
4				56		10.3	3																
5	1920	7.8	64			8.7	10		7.1											7.8			
6	1440	7.7	64	552	464	10.0	22			7.6	21612									7.8			
7	1380	7.8	64	760	664	7.1	22	5117	7.9	28450									708	8.1			
8	1560	8.1	65	752	648	7.8	17	4605	7.9	25650									1023	8.1	172	144	
9	1500	8.0	65	516	458	8.2	34	4635	7.0	32350	4985	11	5					1136	7.9	238	154		
10	1680	8.1	65	596	516	8.0	22	5589	7.2	26488								1136	7.9	156	116		
11	1800	8.0	65	610	470	7.1	24	5273	2510	7.0								1543	155	8.0	200	132	
12	1620	8.3	71			6.8	24			7.1													
13	1500	8.4	73	700	588	6.5	36	5396	7.1	26760	1116	24	14					1822	8.2	358	280		
14	960	8.4	62	1058	446	6.8	26	5396	6.8	26262		145	26					1595		476	306		
15	960	7.3	64	564	438	6.8	18	5118	7.3									14	9	1628			
16																							
17																							
18																							
19																							
20																							
21	960	7.7	70	288	192	5.3	23	4902	7.7									66	20	1674		244	134
22	1920	7.6	68	682	466	5.6	23	4536	2070	7.6								144	58	1533	173	232	130
23	1800	7.4	71	796	564	5.4	22	5137	2370	7.3								80	18	1284	341	188	110
24	1860	7.5	70	690	490	1.1	42	4982	2216	7.1								140	32	1728	384	414	200
25	1020	7.5	74			6.0	36			7.0													
26	960	7.4	74			7.3	38			7.1													
27	1080	7.8	69	644	484	8.4	18	4339	7.2									188	38	1642		622	432
28	1080	7.0	70	596	374	6.9	18			7.2								150	40			414	254

Wilsonville SRC Pilot Plant

Job #43080

February 4, 1978 to February 28, 1978

System #2

TABLE B1-2

BIO SYSTEM TREATABILITY
Continuous Data

FLOW RATES				MIXED LIQUOR DATA						INFLUENT DATA						EFFLUENT DATA								
Date	Deten time	Feed ml/12min	Feed ml/day	pH	Temp °F	SS mg/l	VSS mg/l	DO mg/l	DO UPT mg/l/hr	COD mg/l	BOD ₅ mg/l	TDS mg/l	TVDS mg/l	SS mg/l	VSS mg/l	pH	Settled COD mg/l	BOD ₅ mg/l	pH	SS mg/l	VSS mg/l	total		
4	9.7	12.0	1440			56		10.3	6															
5	10.1	11.5	1380	7.8	64			9.0	13															
6	11.1	10.5	1260	7.4	64	1056	972	10.1	22															
7	10.6	11.0	1320	7.3	64	1080	1000	7.5	10	5117	21612													
8		9.0	1080	7.3	65	1020	904	7.7	12	4605	28450													
9	11.1	10.5	1260	7.0	65	980	850	6.8	13	4635	25650													
10	11.1	10.5	1260	7.0	65	1038	934	5.8	16	5589	32350	4985	11	5	7.9	433								
11	9.7	12.0	1440	7.1	65	1054	840	7.3	13	5273	26488													
12	10.1	11.5	1380	6.9	71			3.6	13															
13	10.6	11.0	1320	6.8	73	1030	924	4.3	26	5396	26760	1116	24	14	7.1	433								
14		6.0	720	6.9	62	1030	828	7.1	16	5396	26272													
15		8.5	1020	7.1	64	932	818	6.8	18	5118														
16																								
17																								
18																								
19																								
20																								
21	14.6	8.0	960	7.1	70	842	748	4.2	14	4902														
22	9.7	12.0	1440	6.9	68	952	814	4.8	12	4536	2070													
23	10.1	11.5	1380	6.9	70	896	796	5.0	13	5137	2370													
24	10.6	11.0	1320	6.9	69	816	722	6.8	18	4982	2216													
25	10.1	11.5	1380	6.9	74			6.3	24															
26	10.6	11.0	1320	6.9	74			6.2	22															
27	10.1	11.5	1380	6.8	68	940	810	8.9	18	4339														
28	10.6	11.0	1320	6.6	70	890	774	6.3	18															

Wilsonville SRC Pilot Plant

Job #43080

February 4, 1978 to February 28, 1978

System #3

TABLE B1-3

BIO SYSTEM TREATABILITY
Continuous Data

107

Date	FLOW RATES				INFLUENT DATA							1ST STAGE		2ND STAGE		3RD STAGE		4TH STAGE		CLARIFIED EFFLUENT DATA						
	Feed ml/day	Feed ml/12 min	Disk Speed rph	pH	Temp °F	SS mg/l	VSS mg/l	TDS mg/l	TVDS mg/l	COD mg/l	BOD ₅ mg/l	DO mg/l	COD mg/l	BOD ₅ mg/l	DO mg/l	COD mg/l	DO mg/l	COD mg/l	BOD ₅ mg/l	SS mg/l	DO mg/l					
4																										
5	3000	25	123	7.1	64								6.4	7.8												
6	2400	20	123	7.6	64								9.2	7.5												
7	5040	42	66	7.9	64								4.8	7.3	2362											
8	3360	28	66	7.9	65	11	5	25650		4605			3.6	8.0	2739											
9	3120	26	66	7.0	65	16	9	32350	4985	4635			1.2	7.8	2547											
10	3120	26	45	7.2	65	34	21	26488		5589			1.3	8.0	2614											
11	2880	24	45	7.0	65	406	115			5273	2510		1.9	7.8	2786	667		2408		2282	4.3	2250	508	200	<1	
12	3240	27	45	7.1	71								1.6	7.3												
13	3240	27	45	7.1	73	24	14	26760	1116	5396			1.4	7.1	3081			2862		2762	4.5	2721				
14	1920	16	45	6.8	62	145	26	26262		5396			1.4	7.9	3237			3097		3144	4.2	2502				
15	3120	26	45	7.3	64	14	9			5118			1.4	7.4	3530			3466		3434		2708		295		
16																										
17																										
18																										
19																										
20																										
21	2880	24	45	7.7	70	66	20			4902			2.1	7.7	3586			>3905		>3905			>3905			
22	3000	25	45	7.6	68	144	58			4536	2070			7.7	3680			4098		4150	5.4	4046	300	162		
23	3000	25	45	7.3	63	80	18			5137	2370		1.8	8.0	4022			4282		4412	.	4308	960	284	116	
24	5000	45	45	7.1	64	140	32			4982	2216		1.4	8.0	4188			4212		4262	4.2	4836	1130	720	184	
25	5760	48	45	7.0	72								1.4	8.0												
26	5880	49	45	7.1	73								1.3	8.0												
27	5760	48	45	7.2	69	188	38			4339			1.4	7.6	4498							4.8	4364		578	148
28	6000	50	45	7.2	70	150	40						1.3	7.4							4.5		416	164		

Wilsonville SRC Pilot Plant
 Job #43080
 March 1, 1978 to April 1, 1978
 System #4

TABLE B1-4

BIO SYSTEM TREATABILITY
 Continuous Data

FLOW RATES			MIXED LIQUOR DATA						INFLUENT DATA						EFFLUENT DATA										
Date	Time	Feed ml/12min	Feed ml/day	pH	Temp °F	SS mg/l	VSS mg/l	DO mg/l	DO UPT mg/l/hr	COD mg/l	BOD ₅ mg/l	Phenol mg/l	Oil & Grease mg/l	NH ₃ -N mg/l	SS mg/l	VSS mg/l	COD mg/l	BOD ₅ mg/l	Phenol mg/l	NH ₃ -N mg/l	O-PO ₄ mg/l	% re move	SS mg/l	VSS mg/l	
1		10.0	1200	7.1	74	674	434	7.1	20	2346	680	106		35	68	64	26	1571	205			680	494		
2		9.0	1080	7.1	71	638	408	8.7	16	1673	1260				64	2	1560	197	3.5			1058	702		
3																									
4																									
5																									
6	5.6	9.5	1140	7.4	70	414	208	8.4	20	2792		250					1098					618	218		
7	5.3	10.0	1200	7.1	71	568	412	7.3	21		960	250						202					79.0		
8	5.9	9.0	1080	7.3	76	356	202	8.4	14	1927	1560	200		19.2	89	206	38	891	222			89.0	368	394	
9	5.6	9.5	1140	7.2	71	356	202	8.2	18	1968	1420						60	34	864	54			87.1	268	152
10	6.3	8.5	1020	7.2	70	458	218	7.7	16								84	22					270	120	
11	6.7	8.0	960	7.3	73			8.4	16		1005								180					82.0	
12	6.3	8.5	1020	7.1	71	936	630	8.1	14								152	142					87.0		
13	6.3	8.5	1020	7.6	74	1550	1018	7.9	14	1266	1005						570	131					91.3	216	140
14	6.3	8.5	1020	7.6	74	800	534	7.6	12		1005	350					100	78	537	87	2.5		92.8		
15	5.9	9.0	1080	7.7	71	1050	688	8.7	14	1790	1215	180		69	70										
16																									
17	5.9	9.0	1080	7.4	71	1030	694	8.0	14	1430		110					330	186	461				176	100	
18										855								47					95.0		
19																									
20	5.9	9.0	1080	8.9	71	1246	820	0.3	12	1373		120					84	42	366				146	32	
21	5.3	10.0	1200	7.8	69	898	608	8.1	10								96	42					132	68	
22	5.3	10.0	1200	8.1	68	1188	950	8.0	12	1191		140					136	92	322				106	62	
23	10.0	1200	7.4	70	1012	738	9.2	10	1488	360						132	60	300	14			96.1	220	170	
24																									
25																									
26																									
27																									
28	5.0	5.6	72			7.6		4									70	28	357	26			97.3		
29	10.5	5.9	69	1210	858	7.3	6		1631	995							382	106	270	18			97.2	1056	730
30	10.0	4.1	76	1526	1074	7.0	4		2305	645							184	74	318	105	8.0		91.5	300	168
31	10.5	8.3	68	2020	1344	8.8	8		2854	1237	175												5.0	12.0	3.0
1	9.5	8.7	80	1534	972	6.8	8		150																

Wilsonville SRC Pilot Plant
 Job #43080
 March 1, 1978 to April 1, 1978
 System #5

TABLE B1-5

BIO SYSTEM TREATABILITY
 Continuous Data

Date	FLOW RATES			MIXED LIQUOR DATA						INFLUENT DATA						EFFLUENT DATA									
	Deten time	Feed ml/12min	Feed ml/day	pH	Temp °F	SS mg/l	VSS mg/l	DO mg/l	DO UPT mg/l/hr	COD mg/l	BOD ₅ mg/l	Phenol mg/l	Oil & Grease F/M	NH ₃ -N mg/l	SS mg/l	VSS mg/l	COD mg/l	BOD ₅ mg/l	Phenol mg/l	XBOD ₅ mg/l removed	NH ₃ -N mg/l	SS mg/l	VSS mg/l	total	
1	3.8	9.5	1140	6.7	72	3688	2964	6.8	16	2346	680	106	.06	35	68	54	26	735	24	89					
2	3.8	9.5	1140	6.9	71	3244	2608	8.9	20	1673	1260		.06			64	2	988	35	97					
3																									
4																									
5																									
6	3.3	11.0	1320	7.4	71	2600	2128	9.0	18	2792		250													
7	4.0	9.0	1080	6.9	70			8.2	18		960	250	.11												
8	3.8	9.5	1140	6.9	74	2982	2066	8.4	16	1927	1560	200	.20	19.2	89.0	206	38	466	55	73.0	96		2184	1384	
9	3.3	11.0	1320	6.7	69	2256	1874	8.4	16	1968	420		.07			60	34	448	27		94		1233	1024	
10	4.5	8.0	960	6.5	69	2016	1668	8.3	14							84	22	448					1296	1070	
11	4.5	8.0	960	7.1	71			8.0	12		1005		.14						35		96.5				
12	4.8	7.5	900	7.1	69	1928	1562	8.3	12								152	142							
13	4.0	8.5	1020	7.6	72	1678	1350	7.6	14	1266	1005		.18					340	53		95				
14	4.0	8.5	1020	7.6	75	2004	1570	7.8	16		1005	350	.15					50			95				
15	4.5	8.0	960	7.4	70	1830	1448	8.9	12	1790	1215	180	.19	69	70			327	51	2.5	96				
16												55													
17	3.8	9.5	1140	6.5	70	1612	1304	7.8	16	1430		110						330	186	346			640	502	
18			1100				1225				855		.18						42		95				
19																									
20	4.0	9.0	1080	8.4	70	1404	1134	0.4	10	1373		120						84	42	298			790	636	
21	4.0	9.0	1080	7.0	69	1270	998	8.1	8									96	42				986	804	
22	7.2	5.0	600	7.8	68	1218	952	7.8	10	1191		70						46	28	313			2192	1650	
23																		132	60	300	8		97.7		
24																									
25																									
26																									
27																									
28	5.5		4.3	72			8.0		4																
29	7.0		8.3	70		914	692	7.0	6	1631								70	28	662	51				
30	8.5		8.4	78		858	654	7.0	6	2305								382	106	627	54		564	424	
31	8.0		7.2	68		1116	892	6.4	11	2854		175						184	74	423	83	6.0			
1	8.5		5.8	80		1068	864	7.0	8		150									4.0			564	438	
																								12.0	

Wilsonville SNC Pilot Plant
 Job #43080
 March 1, 1978 to March 23, 1978
 System #6 (continuation of System #2)

TABLE B1-6

BIO SYSTEM TREATABILITY
 Continuous Data

110	FLOW RATES				MIXED LIQUOR DATA						INFLUENT DATA						EFFLUENT DATA						total				
	Date	Deten time	Feed ml/12min	Feed ml/day	Temp pH	SS mg/l	VSS mg/l	DO mg/l	DO UPT mg/l/hr	COD mg/l	BOD ₅ mg/l	Phenol mg/l	Oil & Grease mg/l	F/M mg/l	NH ₃ -N mg/l	SS mg/l	VSS mg/l	settled	COD mg/l	BOD ₅ mg/l	Phenol mg/l	XBOD removed	total	SS mg/l	VSS mg/l		
1	12.3	9.5	1140	6.6	72	876	750	6.3	18	4614	1380	246	106	.15	189	124	34	934	36	3.5	97	1074	850				
2	9.7	12.0	1440	6.6	71	892	740	9.0	18	4798	2280			.32	124	124	10	878	63		97	772	646				
3																											
4																											
5																											
6	11.7	10.0	1200	6.7	71	820	680	8.7	20	3712		500						815						1356	556		
7	10.1	11.5	1380	6.6	70			6.8	18		1260	500							45								
8	13.0	9.0	1080	7.0	74	988	666	8.7	16	3333	1940	450		27.6	.22	129	336	48	755	101	109.5	98		1458	884		
9	13.7	8.5	1020	6.7	69	726	640	8.4	16	3573	1800				.20		100	36	736	42				624	554		
10	27.8	4.2	504	7.1	69	708	570	7.8	14									122	58					1302	1076		
11				7.1	71			8.6	14	2070									52								
12	13.0	9.0	1080	7.0	69	768	612	7.9	14									90	42								
13	13.7	8.5	1020	6.9	72	728	592	7.8	16	3006	2010							649	161					92			
14	13.7	8.5	1020	6.8	75	772	650	7.6	16		1900	400							93					95	718	566	
15	13.0	9.0	1080	6.9	70	942	718	9.0	16	2465	1580	240		32	.20	118	130	38	669	84	4.9	95					
16											86																
17	12.3	9.5	1140	6.8	70	778	590	8.2	16	2410								162	34	731				650	524		
18										1520									48								
19																											
20	13.7	8.5	1020	8.5	70	728	616	0.6	10	2238		300						154	34	649				578	456		
21	13.7	8.5	1020	7.8	70	1162	872	8.7	10									92	34					708	526		
22	13.0	9.0	1080	7.0	68	756	632	7.3	10	2157		140						136	92	602				752	628		
23				7.0	71	722	608	8.8	6	2160								74	50	552				742	568		

Wilsonville SRC Pilot Plant
 Job #43080
 April 3, 1978 to April 13, 1978
 System #7

TABLE B1-7

BIO SYSTEM TREATABILITY
 Continuous Data

Date	Feed ml/12min	Flow ml/day	pH	MIXED LIQUOR DATA						INFLUENT DATA						EFFLUENT DATA						total SS mg/l	VSS mg/l				
				Temp °C	SS mg/l	VSS mg/l	DO mg/l	DO UPT mg/l/hr	COD mg/l	BOD ₅ mg/l	pH	Phenol mg/l	Sulfide mg/l	Chloride mg/l	F/M	SS mg/l	VSS mg/l	COD mg/l	BOD ₅ mg/l	Phenol mg/l	NH ₃ -N mg/l	O-PO ₄ mg/l	Sulfide mg/l	XBOD mg/l removed			
3	6.5	560	6.2	70	968	776	7.8	10.0	1140	755		120				0.08		89		3.0	13.2	8.0		1244	1094		
5	0	100	7.8	71	1046	682	7.6	6.0	1461	810						0	102	22	202	10				98.8	470	328	
6	8.0	810	7.7	74	896	688	7.0	8.0	826	360		120				0.06	68	22	179	16				95.6	818	636	
7																											
8																											
9																											
10	8.5	950	8.8	72		520	6.0	6.0								0.203											
11	9.0	560	8.7	72	684	514	7.3	6.0	1678	735		150	30.0	10	0.118	88	32	694	35	3.0			<0.1	95.2	690	560	
12	8.5	1060	9.4	72	644	442	6.3	6.0				720	7.4	140	8.3		0.254	30	24		33	5.0		<0.1	95.4	402	288
13	8.0	875	8.8	71	684	490	5.4	6.0	1349	1155	7.0	210	9.6	86	0.303	116	38	772	41	8.0			<0.1	96.4	430	306	

Wilsonville SRC Pilot Plant
Job #43080
April 3, 1978 to May 3, 1978
System #8

TABLE B1-8

BIO SYSTEM TREATABILITY Continuous Data

Illinoiville SRC Pilot Plant
Job #43080
April 3, 1978 to May 11, 1978
System #9

TABLE B1-9

BIO SYSTEM TREATABILITY
Continuous Data

Date Time	FLOW RATES			MIXED LIQUOR DATA						INFLUENT DATA						EFFLUENT DATA										
	Detention Time	Feed ml/12min	Eff vol ml/day	Temp °C	SS mg/l	VSS mg/l	DO mg/l	DO UPT mg/l/hr	COD mg/l	BOD ₅ mg/l	Phenol mg/l	Sulfide mg/l	Chloride mg/l	SS mg/l	VSS mg/l	COD mg/l	BOD ₅ mg/l	Phenol mg/l	Sulfide mg/l	NH ₃ -N mg/l	O-PO ₄ mg/l	ZPhenol mg/l	SS removed mg/l	VSS mg/l		
3	9.7	7.0	660	6.5	70	602	444	8.0	7.0	1140	120					77		0.6		8.4	8.0		54	50		
4	6.4	0	100	7.7	71			7.6	6.0	1461	810					107	22									
5	7.3	8.5	880	7.8	74	860	558	7.1	6.0									91	6				536	378		
6	9.2	8.0	695	7.8	70	1528	1130	6.0	6.0	826	360	120				.138	68	22	72	7	4			96.7	666	480
7	7.1	8.5	900	7.8	71	1496	1178	6.3	8.0																	
10					7.2	71		6.3	8.0																	
11	8	9.0	800	6.8	72	1496	1178	6.2	8.0	1678	735	150	30	100	.078	88	32	138	14	1.5 <0.1			99	134	78	
12	6.6	8.0	960	7.6	72	1626	1238	5.4	8.0		720	7.4	140	8.3		.126	30	24	15	2.0 <0.1			98.6	833	638	
13	7.4	8.0	865	7.8	71	2014	1542	6.3	6.0	1349	1155	7.0	210	9.6	86	.101	116	38	94	4	2.5 <0.1			98.8	118	38
14	6.9	9.0	930	8.3	72	1432	1090	6.3	8.0	1725	7.6	150	1.3			234	126	55			1.5 <0.1			99	792	592
15	8	8.5	800	9.1	75		1000	6.3	8.0		1125	8.3	150	4.3		.14		6		1.5 <0.1			99			
16	6.9	8.0	930	8.0	74		1000	6.0	8.0		765	7.6	150			.111		6		1.5			99			
17	6.8	7.5	940	6.9	74	1244	1008	6.3	8.0	1774	1035	7.4	150	13.7		.151	52	34	89	12	1.0 <0.1			99.3	110	46
18	7.4	7.5	865	8.5	75	1848	1479	5.9	8.0	1900		230	8.2			102	50	82			3.0 <0.1			98.7	164	46
19	7.4	7.5	860	8.7	74		1500	6.9	8.0	2454	1920	200			.091		112	7	2.0					99		
20	6.9	7.5	930	7.8	71	2074	1548	7.8	17		1185	130	8.4			.111	90	30		12					154	50
21	7.3	7.5	880	7.8	69	1576	1224	6.3	15		1105	8.3	120	13.8		.133	258	94		15	2.5 <0.1			97.9	154	56
24	7.5		850	8.2	68			6.0	14				8.4			6.3										
25	6.7	7.5	960	8.4	68	2206	1712	7.3	16		960	7.5		4.2		.091	438	120		11	3.0 <0.1	3.6	1.7		1596	1236
26	6.4	8.0	1000	8.4	66	2208	1752	7.2	17	1935			5.5				174	46	75						454	316
27	7.1	8.0	900	9.0	69	2116	1722	7.8	16	1893	1065	5.7				.087	264	104	98	8	2 <0.1	4.5	5		188	110
28	9.6	8.0	670	6.4	68	2144	1654	7.8	16			150				106	30			1.5 <0.1	1.4 >5.0	99	226	124		
29	6.4	8.5	1000	5.8	68			8.1	16																	
30	6.7	8.5	950	5.8	68	2794	2136	8.1	16		7.8	160	3.6				76	34			3.5 <0.1			97.8	274	158
1	5.3	8.5	1200	8.8	68	4104	2808	8.4	18	1909	7.0		<0.1				144	30	155		ND <0.1			100	168	48
2	6.6	8.5	950	6.6	67	2130	1698	9.2	16	1861		175	14.0				184	106	110		ND <0.1			100	250	220
3	12.1	8.5	530	7.0	68			9.8	14											ND						
4			600																	0.2						
5			950																	0.4						
6			560																	1.0						
7			490																	0.8						
10			1100																	3.0						
11			710																	0.5						

Wilsonville SRC Pilot Plant
Job #43080
April 21, 1978 to May 3, 1978
System #10

TABLE B1-10

BIO SYSTEM TREATABILITY Continuous Data

TABLE B1-11
System #11

Two stage activated carbon/activated sludge
2½ day detention time each stage
Design flow and phenol concentration of 150-200 mg/l

<u>Date</u>	<u>MLSS</u>	<u>MLVSS</u>	<u>Phenolics 1st stage</u>	<u>Phenolics 2nd stage eff.</u>
5/22	1076 mg/l	740 mg/l	.66 mg/l	.03 mg/l
5/23			<.13	.051
5/24	1326	870	.325	.066
5/25	1206	918	.35	.038
5/26	1716	1256	.33	.05

CATALYTIC, INC.
PHILADELPHIA, PENNSYLVANIA 19102

Figure B1-1
SETTLING OR THICKENING DATA SHEET

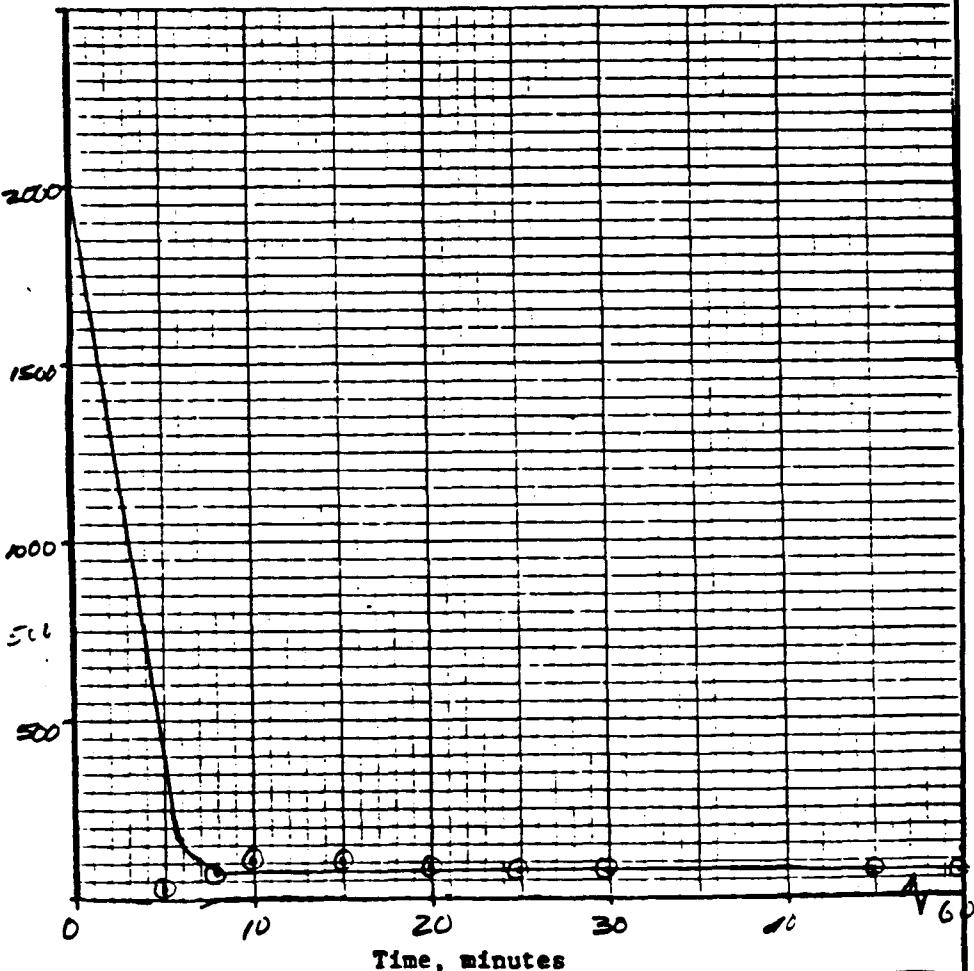
CONTRACT NO. 43080 UNIT NO. 1 SPECIFICATION NO. _____
 CLIENT: Southern Services SERVICE OF UNIT Activated Sludge
 LOCATION: Wilsonville, Ala. SRC Pilot Plant DATE: 24-2-78 BY: _____

PRELIMINARY DATA
 Detention Time: 5 days Solids Retention Time: no wasting
 MLSS - Initial (Co) 670 mg/l Supernatant 386 mg/l @ 30 MIN time.
 Initial Height (H₀) 2000 ml Final Height 80 ml.
 ASL

Curve

Time (min)	Ht. (ml)
1	0
2	200
3	60
4	100
5	100
6	90
7	80
8	60
9	50
10	40
11	
12	
13	
14	
15	
16	
17	
18	
19	
20	
21	
22	
23	
24	
25	
26	
27	
28	
29	
30	

Height to Interface, ml.



Data Interpretation

H_u = _____ t_u = _____
 H_c = _____ t_c = _____
 $U.A. = \frac{t_u}{C_0 H_0} =$ _____ $G(\text{flux}) = \frac{1}{U.A.} =$ _____

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Figure B1-2
SETTLING OR THICKENING DATA SHEET

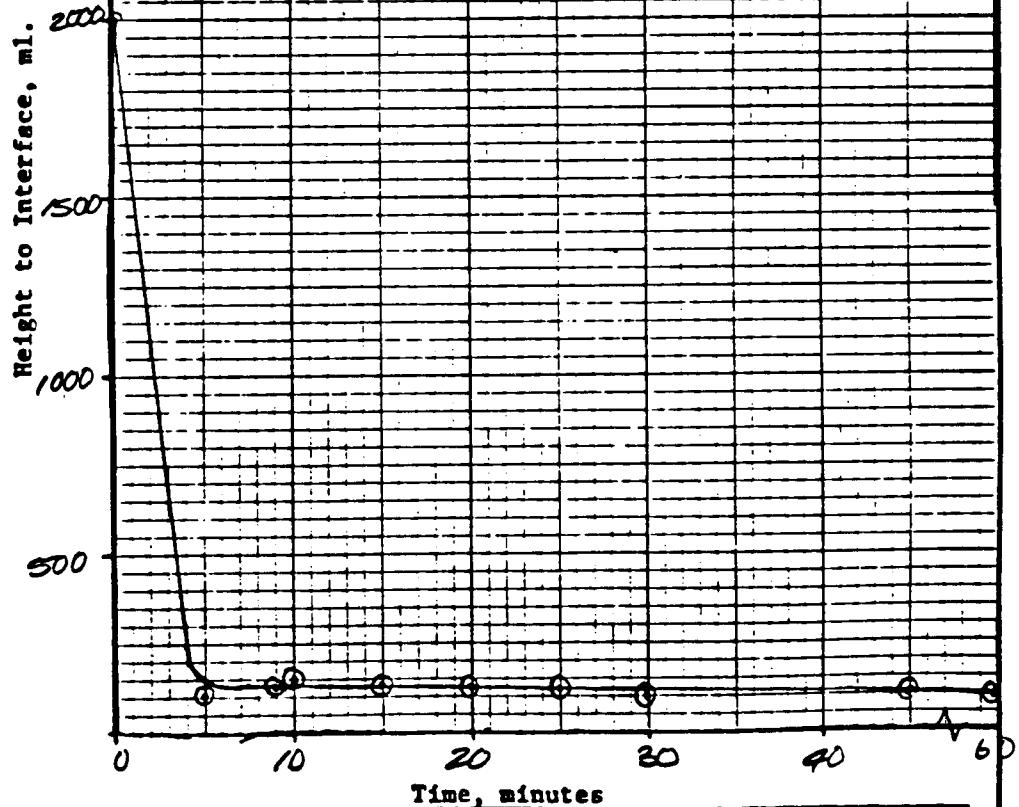
CONTRACT NO. 43080 UNIT NO. 2 SPECIFICATION NO.
 CLIENT: Southern Services SERVICE OF UNIT seated lagoon
 LOCATION: Wilsonville, Ala. SRC Pilot Plant
 DATE: 25-2-78 BY:

DETENTION TIME: 10 days PRELIMINARY DATA
 MLSS - Initial (Co) 816 mg/l Solids Retention Time: 2.99 mg/l ~~0.31 N.C. 1000~~
 Initial Height (Ho) 2000 ml Supernatant 50 ml.

ALA 10

Curve

Time (min)	Ht. (ml)
0	2000
5	100
6	140
10	150
15	100
20	130
25	120
30	100
45	100
60	100
11	
12	
13	
14	
15	
16	
17	
18	
19	
20	
21	
22	
23	
24	
25	
26	
27	
28	
29	
30	



Data Interpretation

E_u = _____ t_u = _____
 H_c = _____ t_c = _____

$U.A. = \frac{t_u}{C_0 H_0} =$ _____ $G(\text{flux}) = \frac{1}{U.A.} =$ _____

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Figure B1-3
SETTLING OR THICKENING DATA SHEET

CONTRACT NO. 43080 UNIT NO. 6 SPECIFICATION NO. _____
 CLIENT: Southern Services SERVICE OF UNIT aerated lagoon
 LOCATION: Wilsonville, Ala. SRC Pilot Plant
 DATE: 17-3-78 BY: _____

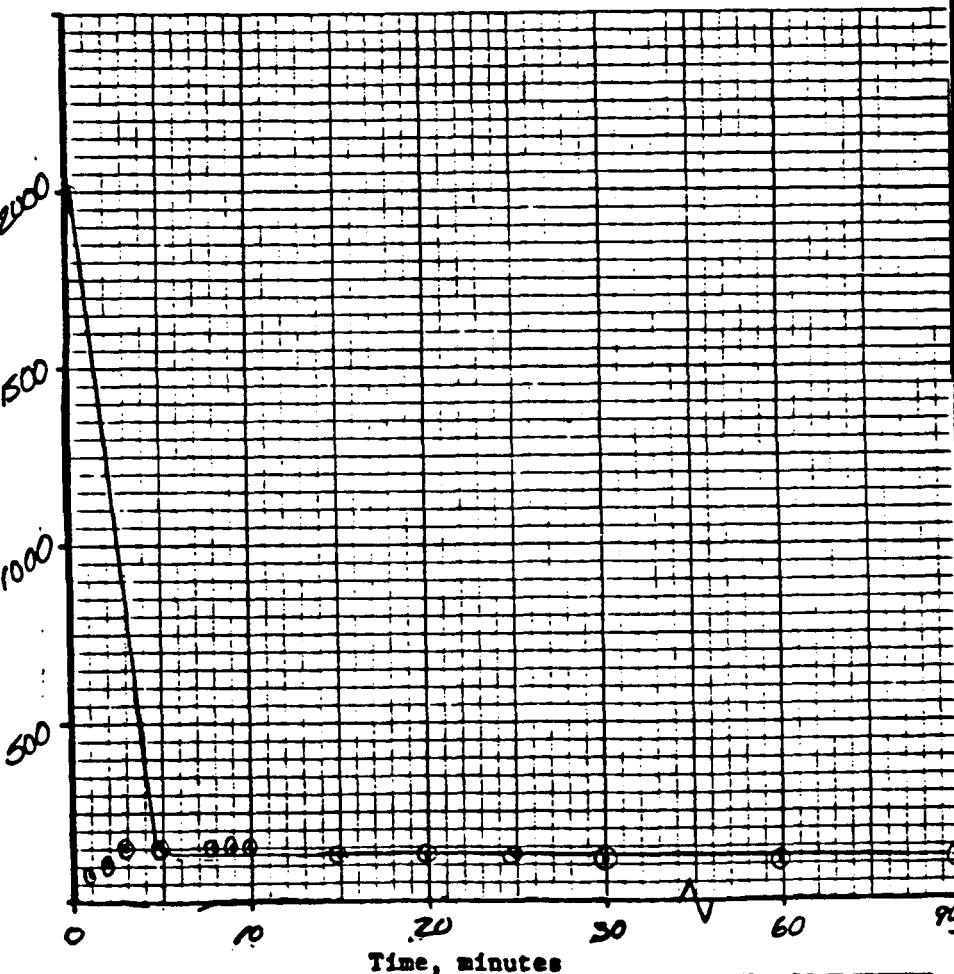
PRELIMINARY DATA
 Detention Time: 10 days Solids Retention Time: _____
 MLSS - Initial (C_0) 778 mg/l Supernatant 200 mg/l e 30 min hrs.
 Initial Height (H_0) 2000 ml Final Height 60 ml.

ALA 10

Curve

Time (min)	Ht. (ml)
1	2000
2	30
3	60
4	100
5	
6	100
7	
8	
9	100
10	100
11	100
12	100
13	60
14	80
15	60
16	60
17	60
18	
19	
20	
21	
22	
23	
24	
25	
26	
27	
28	
29	
30	

Height to Interface, ml.



Data Interpretation

H_u = _____ t_u = _____
 H_c = _____ t_c = _____

$U.A. = \frac{t_u}{C_0 H_0} =$ _____ $G(\text{flux}) = \frac{1}{U.A.} =$ _____

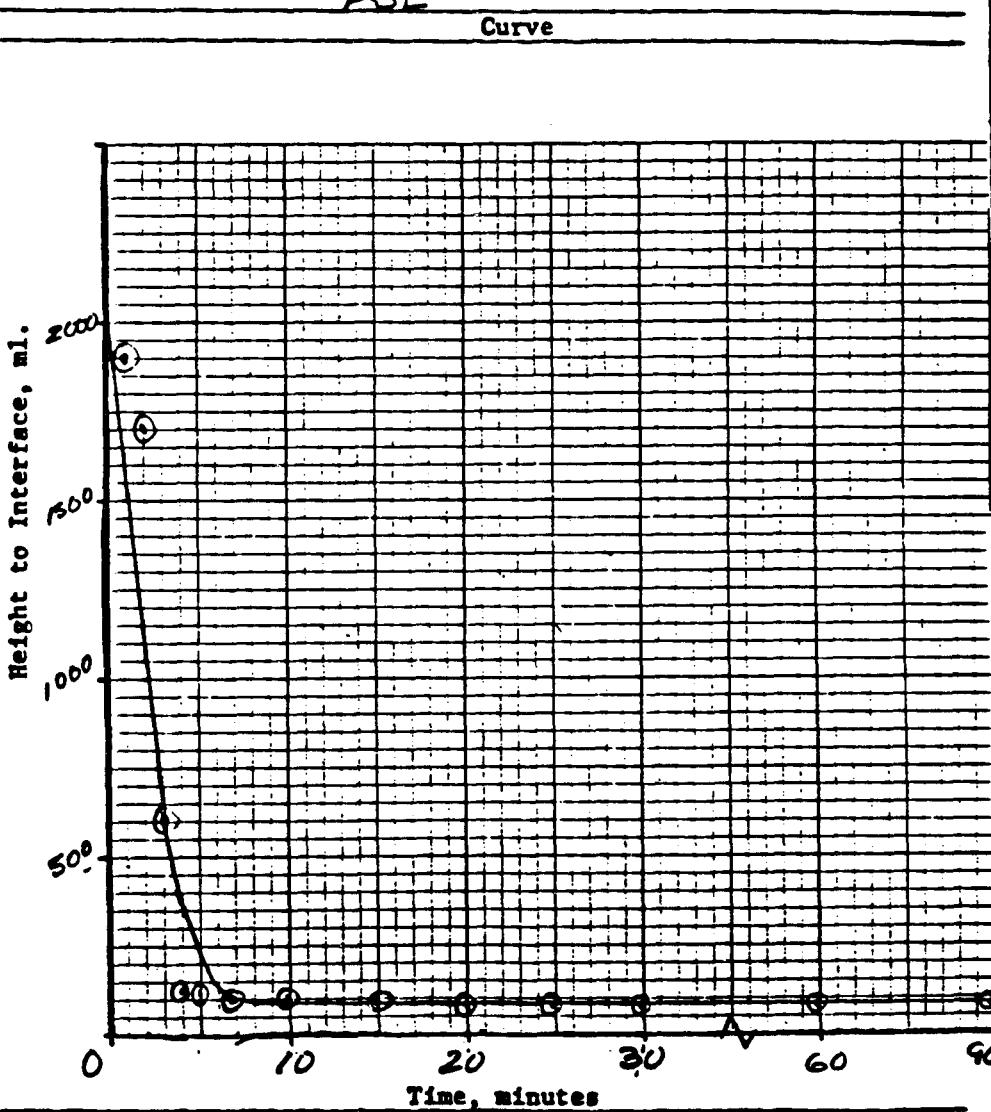
CATALYTIC, INC.
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Figure B1-4
SETTLING OR THICKENING DATA SHEET

CONTRACT NO. 43080 UNIT NO. 4 SPECIFICATION NO.
CLIENT: Southern Services SERVICE OF UNIT Activated Sludge
LOCATION: Wilsonville, Ala. SRC Pilot Plant DATE: 7-3-78 BY: _____

PRELIMINARY DATA
Detention Time: _____ Solids Retention Time: _____
MLSS - Initial (Co) 1030 mg/l Supernatant 100 PPM mg/l e 30 min hrs.
Initial Height (H₀) 2000 ml Final Height 80 ml.

Time (min)	Ht. (ml)
1	0
2	1900
3	1200
4	600
5	120
6	110
7	100
8	100
9	
10	
11	100
12	90
13	80
14	80
15	80
16	80
17	80
18	
19	
20	
21	
22	
23	
24	
25	
26	
27	
28	
29	
30	



Data Interpretation

$H_u =$ _____ $t_u =$ _____

$H_c =$ _____ $t_c =$ _____

$U.A. = \frac{t_u}{C_0 H_0} =$ _____ $G(\text{flux}) = \frac{1}{U.A.} =$ _____

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Figure B1-5
SETTLING OR THICKENING DATA SHEET

CONTRACT NO. 43080 UNIT NO. 5 SPECIFICATION NO. _____
CLIENT: Southern Services SERVICE OF UNIT aerated lagoon
LOCATION: Wilsonville, Ala. SRC Pilot Plant
DATE: 17-3-72 BY: _____

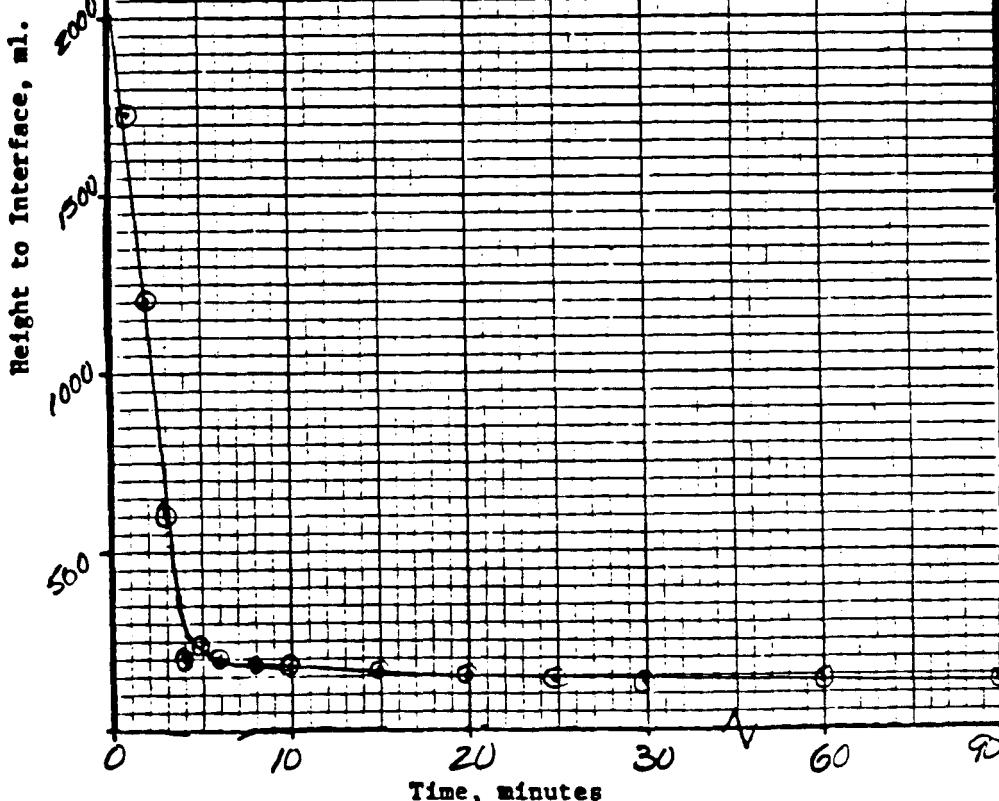
PRELIMINARY DATA

Detention Time: 4 days Solids Retention Time: _____
MLSS - Initial (C_0) 1612 mg/l Supernatant 132 mg/l 0.30 min hrs.
Initial Height (H_0) 2000 ml Final Height 190 ml.

A/A

Curve

Time (min)	Ht. (ml)
0	2000
1	1730
2	1200
3	600
4	
5	200
6	220
7	200
8	
9	160
10	175
11	15
12	160
13	70
14	150
15	25
16	140
17	60
18	100
19	
20	
21	
22	
23	
24	
25	
26	
27	
28	
29	
30	



Data Interpretation

$$H_u = \text{_____} \quad t_u = \text{_____}$$

$$H_c = \text{_____} \quad t_c = \text{_____}$$

$$U.A. = \frac{t_u}{C_0 H_0} = \text{_____}$$

$$G(\text{flux}) = \frac{1}{U.A.} = \text{_____}$$

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Figure B1-6
SETTLING OR THICKENING DATA SHEET

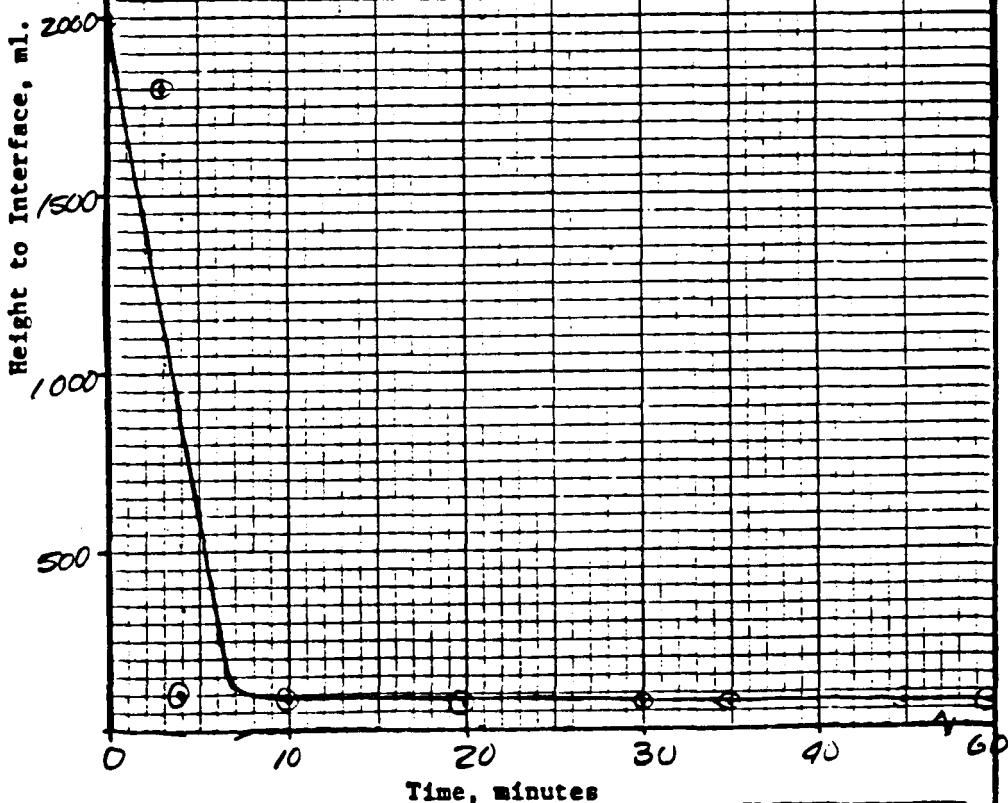
CONTRACT NO. 43080 UNIT NO. 9 SPECIFICATION NO.
 CLIENT: Southern Services SERVICE OF UNIT Activated Sludge
 LOCATION: Wilsonville, Ala. SRC Pilot Plant
 DATE: 15-4-78 BY:

DETENTION TIME: 7 days SOLIDS RETENTION TIME: 30 days
 MLSS - INITIAL (Co) mg/l SUPERNATANT 110 mg/l @ 30 MIN hrs.
 INITIAL HEIGHT (Ho) 2000 ml FINAL HEIGHT 75 ml.

ASL-C

Curve

Time (min)	Ht. (ml)
0	2000
3	1800
4	1000
5	100
10	90
15	80
20	80
25	75
30	75
45	75
60	75
12	
13	
14	
15	
16	
17	
18	
19	
20	
21	
22	
23	
24	
25	
26	
27	
28	
29	
30	



Data Interpretation

$H_u =$ $t_u =$
 $H_c =$ $t_c =$

$U.A. = \frac{t_u}{C_0 H_0} =$

$G(\text{flux}) = \frac{1}{U.A.} =$

CATALYTIC, INC.
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Figure B1-7
SETTLING OR THICKENING DATA SHEET

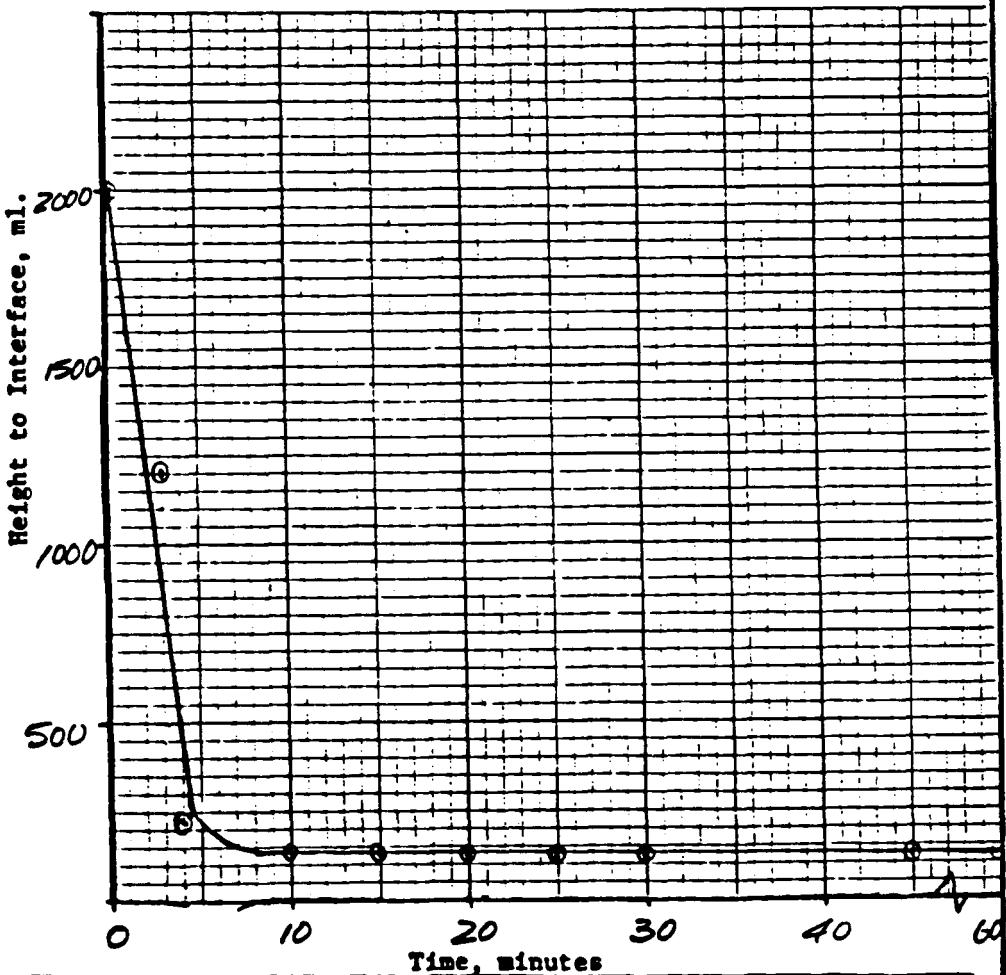
CONTRACT NO. 43080 UNIT NO. 8 SPECIFICATION NO. _____
CLIENT: Southern Services SERVICE OF UNIT Activated Sludge
LOCATION: Wilsonville, Ala. SRC Pilot Plant
DATE: 15-4-78 BY: _____

PRELIMINARY DATA

Detention Time: 6 days Solids Retention Time: 30 days
MLSS - Initial (Co) 1130 mg/l Supernatant 130 mg/l @ 30 min hrs.
Initial Height (H₀) 2000 ml Final Height 120 ml.

Curve

Time (min)	Ht. (ml)
0	2000
3	1200
9	230
10	100
13	150
20	120
25	120
30	120
45	120
60	120
11	
12	
13	
14	
15	
16	
17	
18	
19	
20	
21	
22	
23	
24	
25	
26	
27	
28	
29	
30	



Data Interpretation

H_U = _____
H_C = _____
t_U = _____
t_C = _____
U.A. = $\frac{t_U}{C_0 H_0}$ = _____ $C(\text{flux}) = \frac{1}{U.A.} =$ _____

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Figure B1-8
SETTLING OR THICKENING DATA SHEET

CONTRACT NO. 43080 UNIT NO. 8 SPECIFICATION NO. _____
 CLIENT: Southern Services SERVICE OF UNIT Activated Sludge
 LOCATION: Wilsonville, Ala. SRC Pilot Plant DATE: 2-5-78 BY: _____

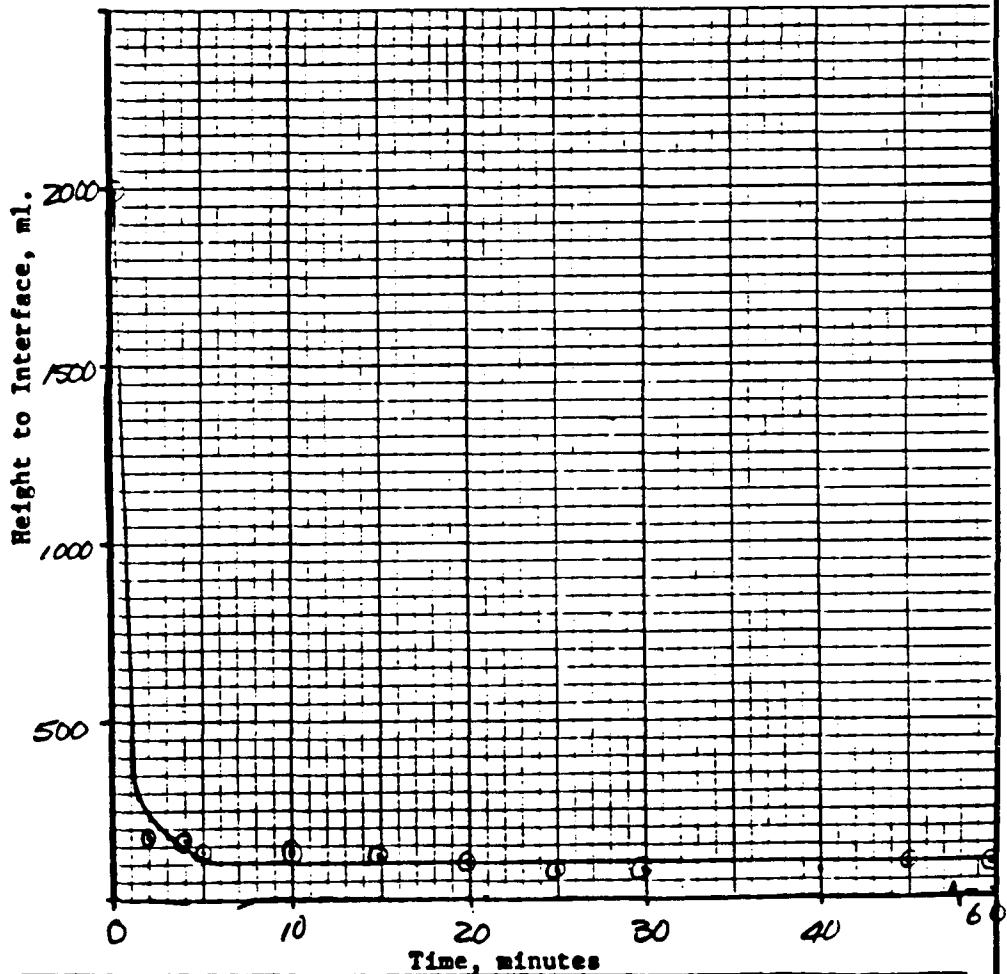
PRELIMINARY DATA

Detention Time: 6 days Solids Retention Time: _____
 MLSS - Initial (Co) 2130 mg/l Supernatant 194 mg/l ℓ 30 minutes.
 Initial Height (H₀) 2000 ml Final Height 80 ml.

ASLC

Curve

Time (min)	Ht. (ml)
0	2000
1	
2	180
3	
4	
5	160
6	140
7	120
8	100
9	
10	80
11	60
12	45
13	30
14	
15	
16	
17	
18	
19	
20	
21	
22	
23	
24	
25	
26	
27	
28	
29	
30	



Data Interpretation

$H_u =$ _____ $t_u =$ _____
 $H_c =$ _____ $t_c =$ _____

$U.A. = \frac{t_u}{C_0 H_0} =$ _____ $G(\text{flux}) = \frac{1}{U.A.} =$ _____

APPENDIX B2
EFFLUENT TREND CHARTS

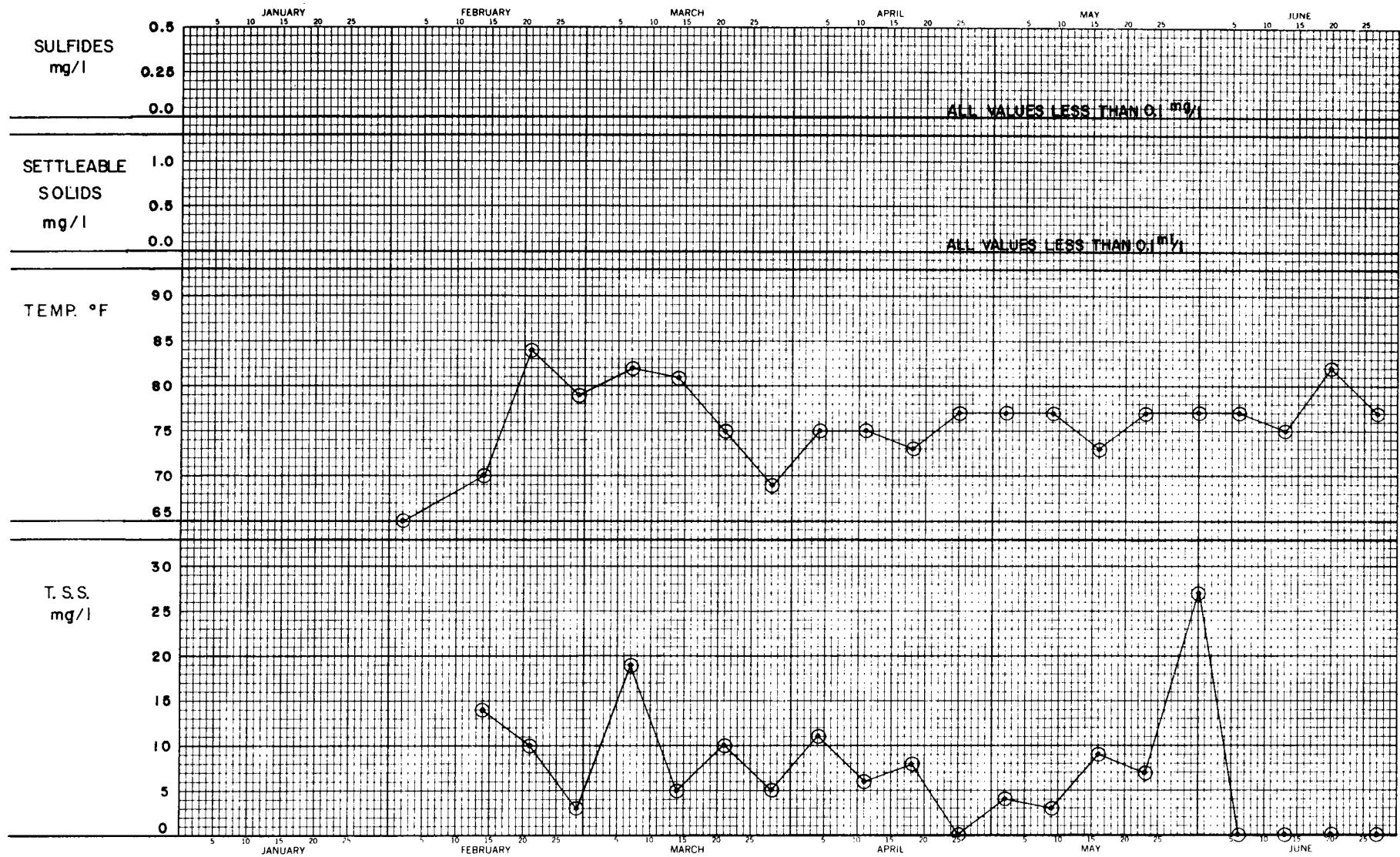


FIGURE B2-1. EFFLUENT TREND CHART: SOLIDS, TEMPERATURE AND SULFIDES - FEBRUARY - JUNE 1979

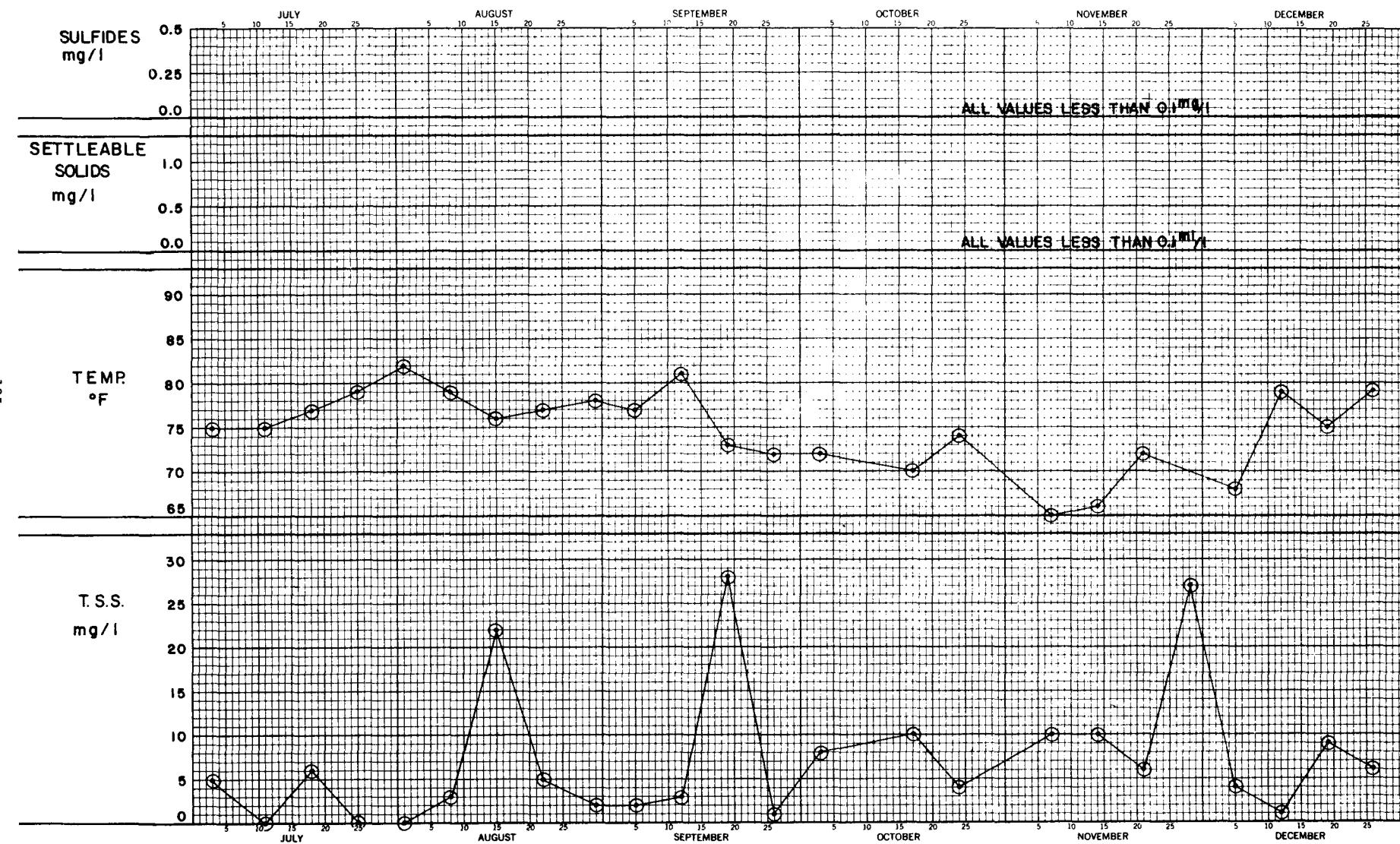


FIGURE B2-1. EFFLUENT TREND CHART: SOLIDS, TEMPERATURE AND SULFIDES- JULY- DECEMBER 1979

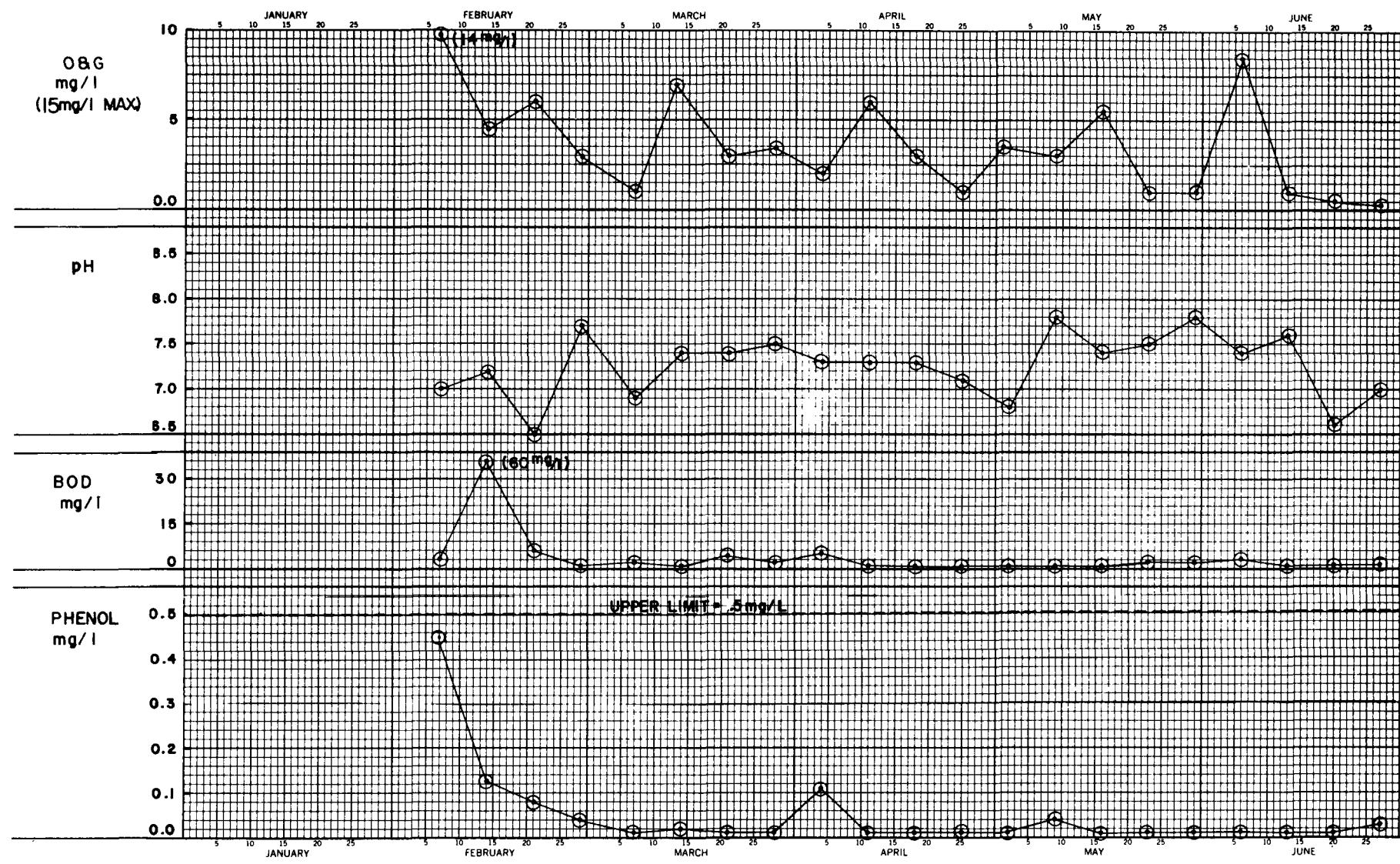


FIGURE B2-2.EFFLUENT TREND CHART: PHENOL, BOD, pH AND OIL & GREASE - FEBRUARY - JUNE 1979

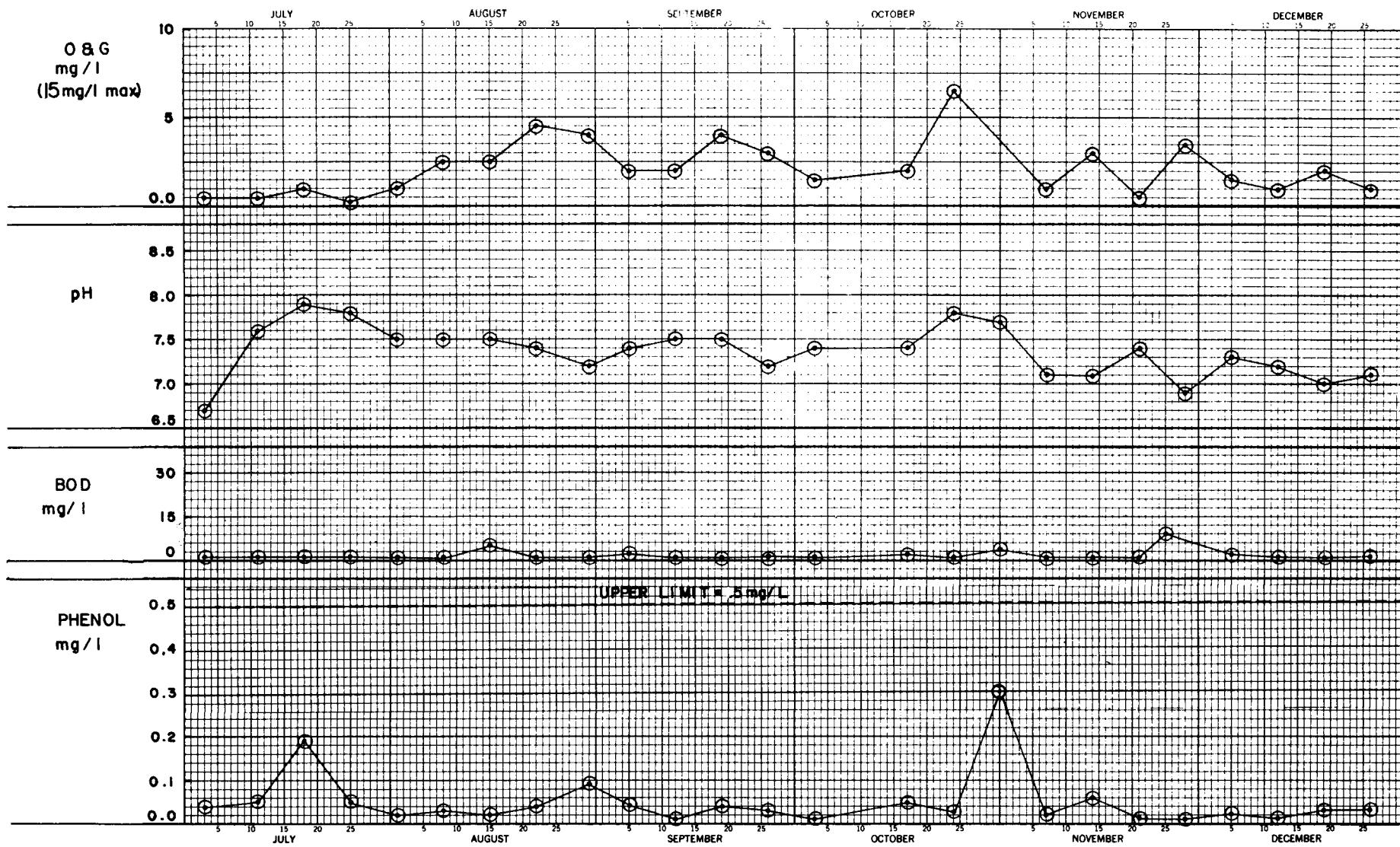


FIGURE B2- 2. EFFLUENT TREND CHART: PHENOL, BOD, pH AND OIL & GREASE- JULY - DECEMBER 1979

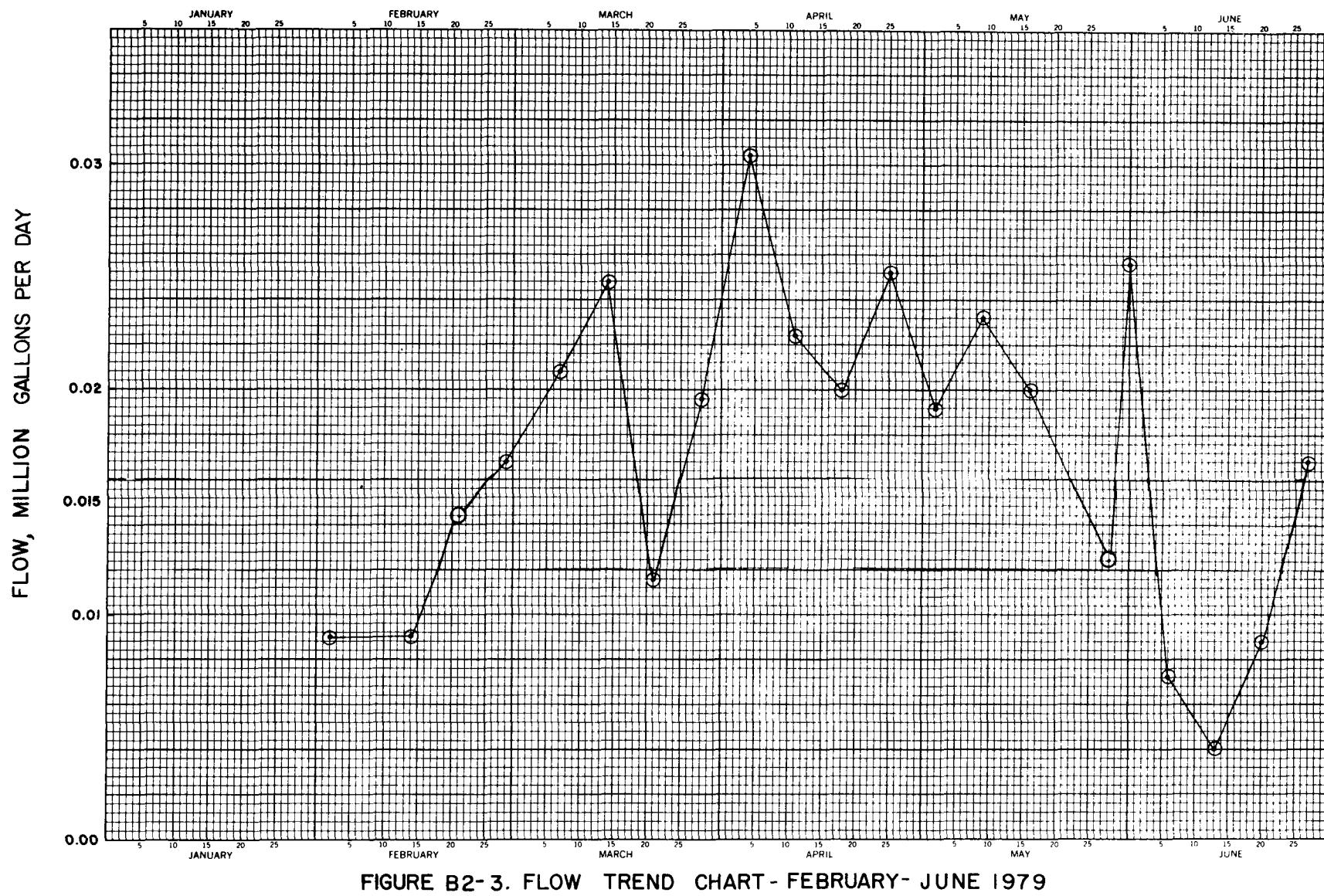


FIGURE B2-3. FLOW TREND CHART - FEBRUARY- JUNE 1979

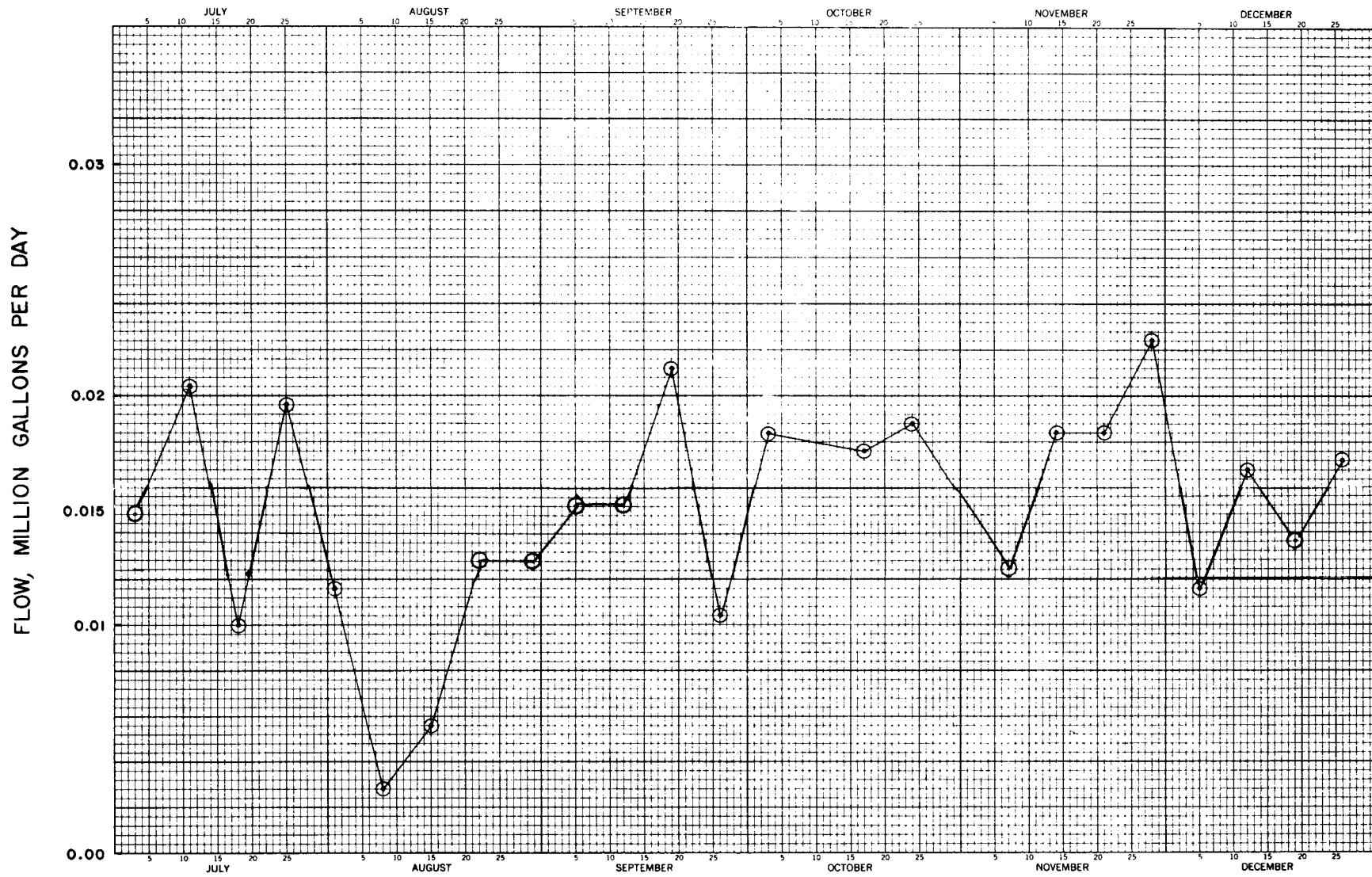


FIGURE B2-3. FLOW TREND CHART - JULY - DECEMBER 1979

Table 1
SRC Pilot Plant Operating Hours and Filter Cycles

Run	w/solv. & H ₂ hrs ^(a)	Reaction Section							Filtration section, cycles	Vacuum Flash section, hrs	
		5%(e)	10%(e)	with coal slurry and hydrogen, hr ^(b)	15%(e)	20%(c)	25%(c)	27%(c)	33%(c)	38.5%(c)	40%(c)
172(d)	27								27	0	27
173	37					37				4	37
174	26			26						0	26
175	73.5								72	22	71.5
176	36.5								37	16	36.5
177	54								54	16	54
178	28							23		12	28
179	144							126		57	144
180	68.2							62		25	61.2
181	141.8							142		65	141.8
182	134							134		63	134
183	41.5							34		18	41.5
184	39.5							40		12	42.0
185	105.9							93		40	93.9
186	160.1							160		56	160.1
187	24							24		8	24
188	52							52		17	52
189	58							54		16	58
190	149							140		61	149
191	83							79		26	83
192	66							41		17	63
193	84							72		37	94
194	32							27		7	32

(Table continued)

Table 1 (continued)
SRC Pilot Plant Operating Hours and Filter Cycles

Run	w/solv, & H ₂ hrs(a)	Reaction Section							Filtration section, cycles	Vacuum Flash section, hrs
		5%(c)	10%(c)	with coal slurry and hydrogen (b)	20%(c)	25%(c)	27%(c)	33%(c)		
<u>Report period</u>	1,665	0	0	26	0	37	0	0	1,493	0
<u>Prior 1979</u>	5,172	0	0	0	0	0	0	71	4,695	0
<u>Total 1979</u>	6,837	0	0	26	0	37	0	71	6,188	0
<u>Total 1978</u>	7,207	0	0	0	0	0	0	0	6,697	0
<u>Total 1977</u>	5,559	0	39	0	40	324	713	992	2,574	0
<u>Total 1976</u>	6,329	0	111	0	184	1,367	0	69	4,098	0
<u>Total 1975</u>	4,883	0	48	0	637	2,013	0	1,041	0	300
<u>Total 1974</u>	4,675	23	73	0	69	3,193 ^(e)	0	407	0	0
<u>Overall total to date</u>	35,490	23	271	26	930	6,934	713	2,580	19,557	300
									9,702	35,533

(a) On-stream hours with solvent and hydrogen through B102 Slurry Preheater and R101 Dissolver.

(b) On-stream hours with coal slurry and hydrogen through B102 Slurry Preheater and R101 Dissolver.

(c) Coal slurry concentration, MF basis.

(d) October portion of run.

(e) Includes 165 hours on 5-20% concentration during early runs.

Table 2
Operating Data Summary for October 1979
Kentucky 9 Coal

Date Oct 1979	Coal Feed, MF lb/hr	Coal Feed, hr	Slurry conc, MF coal, %	Feed Gas			Process solv(a), % conv	B102 Preheater					R101 Dissolver				
				H ₂ , %	to B102, scfh	total scfh		inlet press, psig	ΔP btm, psi	ΔP mid, psi	ΔP top, psi	outlet temp, °F	Temperature	Temperature	Temperature	Density	
								btm, °F	outlet, °F	btm, %	mid, %	top, %					
- Run 172 (Lafayette) Coal - cont.																	
1 2	458 3	24 3	36.2 37.9	84 86	9,910 10,230	9,910 10,230	72.3 75.5	2,184 2,180	11 10	14 14	6 5	792 800	811 814	823 824	- -	21 19	- -
- Run 173 (Lafayette) Coal -																	
2 3	190 173	21 16	23.6 23.0	86 85	10,230 10,190	10,230 10,190	75.5	2,203 2,169	3 2	7 6	1 1	821 819	814 812	824 820	- 12	18 19	- -
- Run 174 (Lafayette) Coal -																	
3 4	161 154	8 18	22.3 16.3	85 89	10,190 11,360	10,190 11,360		2,167 2,160	3 2	6 3	2 1	806 821	809 802	818 812	- -	20 13	- -
- Run 175 (Lafayette) Coal -																	
9 10 11 12	455 476 446 390	22 24 24 1.5	34.9 38.2 37.5 37.5	86 84 85 85	9,037 9,724 9,880 9,880	9,037 9,724 9,880 9,880	75.0 76.3 74.8 74.8	2,165 2,190 2,195 2,200	8 17 20 19	11 11 12 13	3 6 6 5	807 800 799 791	814 812 812 813	825 824 824 825	- 12 13 18	- 14 17 18	- - - -
- Run 176 (Lafayette) Coal -																	
12 13	361 363	22.5 14	37.1 37.2	84 85	9,790 9,790	9,790 9,790	74.8	2,200 2,202	18 19	13 12	3 4	796 794	813 813	826 826	15 13	19 21	- -
- Run 177 (Lafayette) Coal																	
13 14 15	424 460 482	10 24 20	37.7 37.7 37.9	85 85 85				2,200 2,224 2,220	19 19 17	15 15 12	5 8 7	795 794 798	813 812 812	826 825 824	14 11 11	18 17 17	- - -
- Run 178 (Lafayette) Coal -																	
17 18	696 400	6 16.8	37.5 37.2	- 85	- 10,170	- 10,170	76.9	2,200 2,202	16 14	16 12	2 6	793 795	831 810	848 825	- -	- -	- -

(Table continued)

Table 2 (continued)
 Operating Data Summary for October 1979
 Kentucky 9 Coal

Date Oct 1979	Coal Feed MF 1b/hr	Coal Feed hr	Slurry conc, MF coal, %	Feed Gas			Process solv (a), % conv	B102 Preheater					R101 Dissolver				
				to B102, scfh	total scfh	inlet psig		ΔP btm, psi	ΔP mid, psi	ΔP top, psi	outlet temp, °F	Temperature btm, °F	outlet, °F	Temperature btm, °F	mid, %	top, %	
- Run 179 (Lafayette) Coal -																	
19	384	10.3	35.5	84	9,740	9,740	68.2	2,193	14	19	3	789	810	827	20	20	
20	380	24	36.2	84	9,880	9,880		2,190	16	15	4	794	810	823	17	19	
21	391	24	37.1	85	10,070	10,070		2,190	13	15	6	797	812	824	10	19	
22	412	24	37.7	85	10,070	10,070	69.6	2,190	15	14	4	795	812	823	8	18	
23	408	24	36.0	85	10,090	10,090	78.3	2,200	16	11	11	799	812	823	8	15	
24	401	19.8	37.1	84	9,880	9,880	75.9	2,210	17	12	6	797	812	824	8	16	
- Run 180 (Lafayette) Coal -																	
28	374	18	37.2	85	10,010	10,010		2,185	5	9	3	810	817	826	-	-	
29	415	24	37.8	84	9,920	9,920	72.5	2,193	13	11	6	796	813	824	-	-	
30	417	20.2	37.4	85	10,030	10,030		2,194	15	11	7	796	814	824	-	-	
- Run 181 (Lafayette) Coal -																	
30	417	3.8	37.4	85	10,030	10,030		2,190	15	11	6	799	814	824	-	-	
31	392	24	37.3	85	9,730	9,730		2,190	15	11	6	795	814	824	-	-	

(Table continued)

Table 2 (continued)
 Operating Data Summary for October 1979
 Kentucky 9 Coal

Date Oct 1979	Performance				H ₂ con- sumed, % MAF	T102 Temperature btm, °F	T102 Vacuum Column top, °F	Column Press top, psia	T104 (e) 1t org +350 °F, wt%	T105 Frac Column (e)		
	Coal conv, % MAF	yield(b), % MAF coal	SRC yield(c), % MAF coal	sulfur(d), %						btm wt%	top wt%	wash solvent +450 °F, wt%
- Run 172 (Lafayette) Coal -												
1	92.7	70.5	63.0	1.1	1.9	497	203	0.3	5.5	4.2	8.4	6.7
2	92.5			1.13		505	203	0.3	8.7	3.6	6.0	5.5
- Run 173 (Lafayette) Coal -												
2	92.5				3.9	561	212	0.7	8.7	3.6	6.0	5.5
3	92.7			0.77	3.2	577	202	0.5	7.9	4.7	9.1	1.5
- Run 174 (Lafayette) Coal -												
3	92.7			0.77	3.2	577	202	0.5	7.9	4.7	9.1	1.5
4	94.7	46.7			2.8	497	141	-	0	1.3	6.4	7.2
- Run 175 (Lafayette) Coal -												
9	92.5				1.6	590	204	0.4	15.6	4.8	3.2	10.7
10	93.8	51.1	[56.9]	0.98	1.7	585	199	0.4	6.4	5.6	7.4	12.0
11	94.6	48.1		1.02	2.0	583	195	0.4	5.1	5.3	5.4	7.6
12	94.0	52.3		0.88	2.4	578	192	0.4	2.5	3.3	4.0	9.8
- Run 176 (Lafayette) Coal -												
12	94.0			0.88	2.4	594	191	0.4	2.5	3.3	4.0	9.8
13	97.1			0.87		601	191	0.4	0	7.9	30.3	1.9
- Run 177 (Lafayette) Coal -												
13	97.1			0.87		599	190	0.4	0	7.9	30.3	1.9
14	94.0	58.5	66.7	1.03	1.8	594	189	0.5	0	5.4	7.2	6.2
14	94.1	57.6		1.03	1.9	593	191	0.6	0	6.0	9.1	4.3
- Run 178 (Lafayette) Coal -												
17					560	208	0.4					
18	94.1				2.2	574	202	1.1	26.9	6.7	4.4	7.5

(Table continued)

Table 2 (continued)
 Operating Data Summary for October 1979
 Kentucky 9 Coal

Date Oct 1979	Coal conv % MAF	Performance			H ₂ con- sumed, % MAF	T102 Vacuum Temperature btm, °F	Column top, °F	Press top, psia	T104 (e)		T105 Frac Column (e)	
		yield ^(b) , % MAF coal	SRC yield ^(c) , % MAF coal	sulfur ^(d) , %					1t org +350°F, wt%	btm -450, wt%	wash solvent -350°F, +450°F, wt%	
- Run 179 (Lafayette) Coal -												
19	95.0					2.3	594	194	0.4	0.6		
20	94.9	50.1	[59.9]	0.89	2.1	596	194	0.4	14.9	5.7	11.1	4.3
21	92.7			0.98	1.7	599	193	0.4	16.8	7.0	4.0	20.0
22				0.96	1.8	602	194	0.4	47.7	4.2	3.5	9.2
23	93.3	60.6			1.9	597	194	-	13.4	5.6	3.4	10.5
24	94.3	54.2			1.9	587	193	0.5	41.6	3.7	1.8	12.1
- Run 180 (Lafayette) Coal -												
28					1.9	600	191	0.6				
29	93.2	62.4			1.05	2.0	594	193	0.5			
30	93.9	60.6			1.02	1.9	592	192	0.4	6.4	3.3	8.8
- Run 181 (Lafayette) Coal -												
30	93.9				1.02	1.9	586	192	0.5			
31	96.4				0.87	2.0	593	193	0.5			

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- (a) From V131, THF microautoclave conversion, short method.
- (b) Density gauges not operating properly.
- (c) Cresol-soluble, solvent-free T102 btm.
- (d) V110 lab analysis and forced ash balance method, adjusted for LSRC added to coal feed slurry.
- (e) From laboratory workup of V110 Flash Tank sample, distilled at 600°F, 0.1 mm Hg.

Table 3
Operating Data Summary for November 1979
Kentucky 9 Coal

Date Nov 1979	Coal Feed MF lb/hr	Coal Feed hr	Slurry conc MF coal, %	Feed Gas			Process solv (a) % conv	B102 Preheater					R101 Dissolver		
				H ₂ %	to B102 scfh	total scfh		inlet press, psig	ΔP btm, psi	ΔP mid, psi	ΔP top, psi	outlet temp, °F	Temperature	Density(f)	
													btm °F	outlet °F	btm, % mid, % top, %
-Run 181 (Lafayette) Coal -															
1	393	24	36.8	85	9,840	9,840	78.0	2,190	14	7	11	795	813	824	
2	393	24	37.0	85	9,870	9,870	79.6	2,198	15	8	11	798	814	823	
3	384	24	37.2	85	9,960	9,960		2,200	15	8	11	797	814	824	
4	403	24	36.8	85	10,260	10,260		2,200	16	9	11	788	815	825	
5	389	18	36.8	85	10,180	10,180	77.7	2,200	16	9	12	795	814	825	
- Run 182 (Lafayette) Coal -															
5	466	6	36.1	85	10,080	10,080	77.7	2,200	20	9	5	791	814	825	
6	428	24	37.2	85	10,150	10,150	78.0	2,200	17	9	11	795	814	825	
7	401	24	37.3	85	10,090	10,090	74.7	2,200	17	9	12	793	815	825	
8	378	24	37.0	85	10,050	10,050		2,200	16	9	12	795	814	824	
9	394	24	36.6	85	10,090	10,090	73.1	2,193	16	8	12	797	813	822	
10	410	24	36.9	85	10,100	10,100		2,190	16	9	12	803	816	826	
11	436	8	36.6	86	10,530	10,530		2,200	14	9	12	797	814	823	
- Run 183 (Lafayette) Coal -															
13	385	16	36.1	87			72.2	2,188	14	8	11	799	813	822	
14	436	17.5	37.1	85	10,320	10,320	74.8	2,200	17	10	12	801	814	823	
- Run 184 (Lafayette) Coal -															
14	441	6.5	38.0	85	9,110	9,110	74.8	1,810	19	11	11	802	813	822	
15	427	24	38.0	85	8,800	8,800	74.8	1,807	20	10	9	804	815	824	
16	403	8.9	38.1	86	8,950	8,950		1,800	16	9	8	802	815	824	
- Run 185 (Lafayette) Coal -															
16	327	2.5	38.1	86	8,950	8,950		1,800	16	9	8	802	815	824	
17	426	24	36.6	86	8,890	8,890		1,791	14	8	7	804	813	821	
18	462	24	35.7	85	8,620	8,620		1,802	14	7	10	804	815	824	
19	444	24	36.8	85	8,680	8,680		1,800	18	8	8	803	815	823	
20	446	18.9	36.5	85	8,620	8,620	69.6	1,795	21	7	8	802	816	825	

(Table continued)

Table 3 (continued)
 Operating Data Summary for November 1979
 Kentucky 9 Coal

Date Nov 1979	Coal Feed MF lb/hr	Coal Feed hr	Slurry conc MF coal, %	Feed Gas			Process solv (a), % conv	B102 Preheater					R101 Dissolver			
				H ₂ %	to B102, scfh	total, scfh		inlet press, psig	ΔP btm, psi	ΔP mid, psi	ΔP top, psi	temp, °F	Temperature btm, °F	outlet, °F	btm, %	mid, %
- Run 186 (Lafayette) Coal -																
20	438	5.1	36.4	88	8,800	8,800	69.6	1,780	13	6	13	783	798	812		
21	451	24	37.5	85	8,010	8,010	69.4	1,789	15	6	12	803	817	825		
22	440	24	36.5	85	7,970	7,970		1,791	15	6	13	799	815	823		
23	450	24	36.4	86	8,110	8,110		1,789	15	7	13		815	823		
24	442	24	37.3	85	8,200	8,200		1,792	18	7	9	807	817	823		
25	434	24	36.3	85	8,230	8,230		1,800	17	7	16	807	816	823		
26	430	24	36.8	86	8,480	8,480	65.8	1,809	-	9	14	801	815	824		
27	437	11	37.1	85	8,240	8,240		1,800	-	10	-	804	815	823		
- Run 187 (Lafayette) Coal -																
27	389	13	36.5	84	7,980	7,980	69.8	1,792	15	14	16	804	815	823		
28	385	11	36.4	85	8,050	8,050		1,800	17	7	10	802	816	823		
- Run 188 (Lafayette) Coal -																
28	386	13	36.7	85	8,180	8,180		1,800	17	8	11	802	816	823		
29	401	24	37.6	85	8,180	8,180		1,800	15	9	14	805	816	823		
30	405	15	37.8	84	8,090	8,090		1,806	15	11	13	806	817	824		

(Table continued)

Table 3 - continued
Operating Data Summary for November 1979

Date Nov 1979	Performance				H ₂ con- sumed, % MAF	T102 Vacuum Column			T104(e) 1t org +350°F, wt %	T102 Frac Column(e)		
	Coal conv, % MAF	yield(b), % MAF coal	SRC yield(c), % MAF coal	sulfur(d), %		Temperature btm, °F	top, °F	top, psia		btm -450°F, wt %	wash -350°F, wt %	solvent +450°F, wt %
- Run 181 (Lafayette) Coal - cont.												
1	94.0		61.1	0.93	2.1	598	193	0.4	49.9	6.0	3.1	5.5
2	94.0		46.6	0.99	1.8	595	193	0.5	49.8	2.8	4.4	5.5
3	93.7	[58.1]	57.6	0.90	1.8	598	193	0.4	1.3	3.4	3.2	4.8
4	94.3		48.8	0.90	1.7	595	192	0.4	14.6	4.3	4.2	4.1
5	94.0		57.3	0.89	1.9	593	193	0.4	29.8	5.7	4.7	3.7
- Run 182 (Lafayette) Coal -												
5	94.0			0.89	1.5	596	193	0.4	29.8	5.7	4.7	3.7
6	94.6	[]	89.5	1.13	1.7	596	193	0.4	38.7	1.9	5.3	4.2
7	93.7		58.5	0.97	1.9	600	192	0.4	6.0	3.3	3.9	4.1
8	95.1	[69.0]	48.6	1.29	2.0	601	190	0.4	48.0	4.6	0.9	9.4
9	93.8		67.1	0.96	2.0	602	190	0.4	11.8	5.2	1.7	11.3
10	92.8		69.9	0.90	1.9	600	190	0.4	27.9	2.2	2.2	6.5
11	92.6		67.7	0.96	1.9	603	190	0.4		6.0	1.4	6.4
- Run 183 (Lafayette) Coal -												
13						564	187	0.4	7.5			
14	92.5		74.4	0.93	1.7	594	190	0.4	43.1	3.7	1.9	12.6
- Run 184 (Lafayette) Coal -												
14	92.5			0.93	1.7	598	190	0.4	43.1	3.7	1.9	12.6
15	94.0		46.3	0.98	1.7	594	190	0.6	20.9	4.4	1.1	11.0
16	94.0		52.1	1.36	1.8	595	190	0.8	-	3.4	1.2	9.4
- Run 185 (Lafayette) Coal -												
16	94.0			1.36	1.8			0.8		3.4	1.2	9.4
17	92.2		88.8	1.26	1.6	591	190	0.4	15.9	3.0	1.2	18.5
18	92.1	[64.2]	82.9	1.05	1.5	587	190	0.4	27.5	2.5	1.0	15.6
19	91.8		74.9	-	1.6	590	190	0.8	19.3	2.5	1.6	12.5
20	92.5		84.7	0.91	1.7	590	190	0.8	0.0	1.5	2.1	27.3

(Table continued)

Table 3 - continued
Operating Data Summary for November 1979

Date Nov 1979	Coal conv, % MAF	Performance			H ₂ con- sumed, % MAF	T102 Vacuum Column			T104(e) lt org +350°F, wt %	T102 Frac Column(e)		
		yield(b), % MAF coal	SRC yield(c), % MAF coal	sulfur(d), %		Temperature btm, °F	top, °F	Press top, psia		btm -450°F, wt %	-350°F, wt %	wash solvent +450°F, wt %
- Run 186 (Lafayette) Coal -												
20	92.5			0.91	1.6	589	189	0.6	0.0	1.5	2.1	27.3
21	92.2		74.9	0.85	1.6	590	190	0.6	0.0	3.1	2.7	9.1
22	92.1	[52.3]	79.6	0.87	1.7	598	190	0.6	0.0	3.2	4.0	7.7
23	91.5		81.4	0.94	1.7	598	190	0.6	0.0	4.1	4.9	6.7
24	91.4	[67.6]	68.7	0.95	1.5	596	190	0.7	4.1	3.3	4.3	7.6
25	91.1		78.7	1.03	1.5	597	190	0.7	1.7	2.5	4.8	8.4
26	90.3	[]	69.7	1.16	1.7	596	189	0.7		2.0	3.8	9.9
27	91.4		77.9	1.09	1.6	595	189	0.8				
- Run 187 (Lafayette) Coal -												
27	91.4		77.9	1.09	2.0	596	189	0.8		5.1	4.2	14.8
28					1.8							
- Run 188 (Lafayette) Coal -												
28	90.7				1.5	593	190	0.9		8.9	13.4	3.2
29	92.8	63.6	62.4	1.02	1.1	593	190	0.9		1.6	3.3	5.4
30	-		77.4	0.90		562	192	0.8	0.2	3.3	3.4	6.6

(a) From V131B, THF microautoclave conversion, short method.
 (b) Density gauges not operating properly.
 (c) Cresol-soluble, solvent-free T102 btsms.
 (d) V110 lab analysis and forced ash balance method, adjusted for LSRC added to coal feed slurry.
 (e) From laboratory workup of V110 Flash Tank sample, distilled at 600°F, 0.1 mm Hg.

Table 4
Operating Data Summary for December 1979
Kentucky 9, Lafayette Mine Coal

Date Dec 1979	Coal Feed, MF lb/hr	Coal Feed, hr	Slurry conc., MF coal, %	Feed Gas			Process solvent ^(a) % conv	B102 Preheater					R101 Dissolver		
	H ₂ %	to B102, scfh	total scfh	inlet press, psig	ΔP btm, psi	ΔP mid, psi	ΔP top, psi	outlet temp, °F	Temperature btm, °F	outlet, °F	Temperature btm, °F	mid, % top, %	Density(b) mid, top, %		
- Run 189 (Lafayette) Coal -															
1	415	21.5	37.0	85	6,010	6,010		1,800	11	10	3	803	818	823	
2	448	24	37.2	85	6,010	6,010		1,800	13	13	4	800	816	820	
3	457	8.9	37.2	85	6,840	6,840	71.0	1,825	13	13	5	800	818	822	
- Run 190 (Lafayette) Coal -															
8	341	7.0	38.6	87	9,230	9,230		2,125	2	6	5	811	809	821	
9	438	24	38.1	83	9,430	9,430		2,148	2	17	6	803	811	823	
10	440	24	36.0	84	9,770	9,770	68.2	2,180	2	25	8	795	810	825	
11	471	24	36.6	85	10,000	10,000		2,128	2	25	8	790	811	825	
12	520	24	37.6	86	10,190	10,190	71.4	2,132	2	25	8	790	813	823	
13	478	24	37.1	85	10,010	10,010	72.1	2,132	23	6	8	791	816	823	
14	456	13	36.8	84	9,770	9,770	70.0	2,115	30	4	9	795	817	823	
- Run 191 (Lafayette) Coal -															
14	454	11	37.5	85	10,770	10,770		1,776	23	15	13	792	811	817	
15	491	24	38.1	85	11,110	11,110		1,737	24	10	13	813	818	823	
16	496	24	37.2	85	11,200	11,200		1,738	26	18	6	812	808	823	
17	469	20	37.1	86	11,570	11,570	66.8	1,940	30	14	5	811	817	821	
- Run 192 (Lafayette) Coal -															
19	464	14.5	37.8	82	9,370	9,370		1,724	22	20	6	808	820	825	
20	478	24	37.6	84	11,000	11,000	69.8	1,742	21	17	8	807	820	824	
21	451	2.9	38.0	86	11,340	11,340		1,740	18	16	9	807	821	825	
- Run 193 (Lafayette) Coal -															
22	504	12.7	38.7					1,722	21	19	9	812	820	824	
23	538	24	38.6	86	11,295	11,295		1,725	27	18	5	819	819	823	
24	507	24	38.9	86	11,315	11,315		1,725	24	22	5	818	819	822	
25	454	11.3	38.7	87	11,760	11,760									
- Run 194 (Lafayette) Coal -															
30	283	3.6	31.8					1,735	11	9	12	808	821	823	
31	429	23.8	35.5	86	11,310	11,310									

(Table continued)

Table 4 - continued
 Operating Data Summary for December 1979
 Kentucky 9, Lafayette Mine Coal

Date Dec 1979	Coal conv, % MAF	Performance			H ₂ con- sumed, % MAF	T102 Vacuum Column			T104(f) lt org +350°F, wt %	T105 Frac Column(f)		
		yield(c), % MAF coal	SRC yield(d), % MAF coal	sulfur(e), %		Temperature btm, °F	top, °F	Press top, psia		btm -450°F, wt %	wash solvent -350°F, wt %	+450°F, wt %
- Run 189 (Lafayette) Coal -												
1					1.7	582	190	0.6	4.1	1.2	6.8	8.2
2	91.6		64.5		1.6	585	190	1.1	0	2.2	3.4	7.7
3	90.4		76.4	1.20	1.7	571	193	0.8	-	2.0	3.9	7.5
- Run 190 (Lafayette) Coal -												
8					2.0	584	186	0.8	-	-	-	-
9	93.0	[67.0]	56.4	0.99	2.4	594	190	0.9	51.2	0.7	3.0	15.0
10	91.5	[67.0]			2.7	588	190	0.5	-	0.6	4.0	12.0
11	93.9	[67.0]	68.6	0.83	2.6	593	190	0.5	0.1	6.5	11.2	10.0
12	95.3		71.4	0.83	2.3	595	190	0.5	1.8	3.9	4.9	10.5
13	93.6		76.2	1.14	2.4	597	190	0.4	0.6	3.5	4.4	9.7
14	93.4		76.1	1.02	2.5	597	190	0.4	-	1.9	3.9	11.6
- Run 191 (Lafayette) Coal -												
14	93.4		76.1		2.3	592	190	0.4	-	1.9	3.9	11.6
15	94.6	[69.4]	51.3	0.88	2.1	592	190	0.3	0	4.7	3.5	4.4
16	93.1	[69.4]	73.5	0.93	2.1	593	190	0.3	-	1.9	3.6	8.1
17	91.8		79.6	1.06	2.1	595	190	0.6	1.0	1.5	3.6	10.7
- Run 192 (Lafayette) Coal -												
19					2.4	590	190	0.6	4.4	-	-	-
20	92.6		80.1	0.94	2.3	593	189	0.5	3.7	0.7	3.4	14.6
21	93.1		63.0	0.93	2.4	585	190	0.8	-	0.4	4.5	9.8
- Run 193 (Lafayette) Coal -												
22									-	2.7	3.8	8.7
23	93.9		66.4	0.82	2.1	590	184	0.6	4.0	0.6	4.0	5.8
24	92.9	67.7	68.9	0.83	2.1	588	184	0.6	1.3	0.4	3.9	11.7
25	92.8		73.9	0.92	2.3	589	184	0.6	0.9	0.4	3.4	11.1

(Table continued)

Table 4 - continued
 Operating Data Summary for December 1979
 Kentucky 9, Lafayette Mine Coal

Date Dec 1979	Coal conv. % MAF	Performance			H ₂ con- sumed, % MAF	T102 Vacuum Column			T104(e) lt org +350°F, wt %	T105 Frac Column(e)		
		yield(b), % MAF coal	yield(c), % MAF coal	sulfur(d), %		btm, °F	top, °F	top, psia		btm, -450°F, wt %	wash -350°F, wt %	solvent +450°F, wt %
- Run 194 (Lafayette) Coal -												
31	92.9				2.0	500	206	1.2	4.3	3.3	2.9	16.4

- (a) From V131B, THF microautoclave conversion, short method.
- (b) Density gauges not operating properly.
- (c) Cresol-soluble, solvent-free T102 btms.
- (d) V110 lab analysis and forced ash balance method, adjusted for LSRC added to coal feed slurry.
- (e) From laboratory workup of V110 Flash Tank sample, distilled at 600°F, 0.1 mm Hg.
- (f) GC.

Table 5
Conditions and Results Summary
Kentucky 9, Lafayette Mine Coal

Date, 1979	28 Sept 171	1 Oct 172	12-14 Dec 190
<u>Run</u>			
<u>Coal</u>			
Volatile matter, (Dry basis), %	38.53	38.36	31.57
H/C atomic ratio	0.79	0.78	0.76
<u>Microautoclave conversion, %</u>			
short run	-	-	-
long run	77.2	77.2	76.5
<u>Solvent</u>			
IBP- °F	369	405	424
EP, °F	908	905	883
% minus 450°F/% plus 650°F	5.9/30.8	3.0/26.8	0.8/28.5
Specific gravity	1.033	1.023	1.016
H/C atomic ratio	1.107	1.126	1.148
<u>Microautoclave conversion, %</u>			
short run	71.8	72.6	65.2
long run	68.2	67.6	63.5
<u>Operating conditions</u>			
<u>Feed</u>			
Coal feed rate, MF lb/hr	442	458	478
Concentration, % MF coal	36.1	36.2	37.1
Feed gas, Mscf/ton MF coal to B102	45.0	43.3	41.9
Hydrogen purity, mol %	85	84	85
LSRC (from CSD) recycle, lb/hr	22	22	0
E102 Dissolver Product Cooler bypass	Yes	Yes	No
Dissolver solids withdrawal (a)	Yes	Yes	Yes
<u>Reaction</u>			
Coal space rate, MF lb/hr-ft ³			
Cumulative(b)	33.7	35.0	27.4
Dissolver(c)	48.1	49.8	35.2
Temperature, °F			
Preheater outlet	800	800	791
Dissolver			
Bottom	814	811	816
Middle	-	-	-
Outlet	825	823	823
Pressure, psig	2.175	2,184	2,132
Hydrogen partial pressure, psia			
Preheater inlet	1,875	1,840	1,825
Dissolver outlet(d)	1,540	1,520	1,340
<u>Results</u>			
Conversion, % MAF coal	-	-	-
Preheater	92.0	-	93.6
Dissolver			

(Table continued)

Table 5 - continued
 Conditions and Results Summary
 Kentucky 9, Lafayette Mine Coal

Date, 1979
 Run

	28 Sept 171	1 Oct 172	12-14 Dec 190
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Results (continued)

Hydrogen consumption (e)			
% MF coal	1.7	1.8	2.1
Sulfur, % of SRC product	1.1	1.1	0.96
Yields, % MF coal (e)			
SRC (f)	53.2 (60.7)	57.6 (81.8)	51.2 (53.8)
Organic liquid (f)	25.7 (18.2)	20.8 (-3.6)	26.1 (23.6)
Gases			
C ₁ -C ₅	2.9	2.7	4.5
CO-CO ₂	0.4	1.0	0.5
H ₂ S	2.0	2.1	2.5
NH ₃	0.1	0.1	0.1
Water	1.6	1.0	4.3
Unreacted coal	7.4	7.0	4.4
Ash	8.4	9.5	8.5

(Table continued)

Table 5 -(continued)
 Conditions and Results Summary
 Kentucky 9, Lafayette Mine Coal

Run	171A-B MB		172A MB	
Feed Rate, lb/hr	317		255	
CSD Yields	% MF Coal (g)	% of Feed	% MF Coal (g)	% of Feed
SRC				
oil	10.6	13.2	6.5	6.4
asphaltene	13.5	16.8	22.2	21.8
preasphaltene	11.9	14.8	7.5	7.3
unreacted coal	0.2	0.2	0.1	0.1
ash	0.0	0.0	0.1	0.1
deashing solvent	0.3	0.3	0.2	0.2
solvent	2.2	2.8	7.0	6.9
Ash Concentrate				
oil	0.8	1.0	0.0	0.0
asphaltene	1.7	2.2	1.6	1.6
preasphaltene	7.9	9.8	7.5	7.4
unreacted coal	7.8	9.7	10.5	10.3
ash	8.3	10.3	9.9	9.7
deashing solvent	1.0	1.2	1.4	1.4
solvent	1.2	1.6	3.0	3.0
SRC Recovery ^(h)	79.9		87.8	
Distillate ⁽ⁱ⁾				
in SRC, wt %	5.8		16.1	
in Light SRC, wt %	22.0		31.7	
Deashing Solvent Loss, %				
total	4.2		3.0	
to products	0.6		0.7	

(a) 5-10% of the slurry feed.
 (b) Using 1.6 ft³ as preheater volume, plus 1.5 ft³ for full-dissolver transfer line, plus dissolver volume.
 (c) Using 9.197 ft³ as half-dissolver volume and 13.58 as three-quarter-dissolver volume.
 (d) Dissolver outlet hydrogen partial pressure (psia)

$$= (\text{Pressure at B102 inlet, psia}) \times \frac{\% \text{ H}_2 \text{ in B102 feed gas}}{100} \times \left[100 \times \frac{\text{H}_2 \text{ consumed, lb/hr}}{\text{H}_2 \text{ in B102 feed, lb/hr}} \times 100 \right] \times \frac{1}{100}$$

 (e) Based upon unadjusted yields, process method. SRC is on solvent-free basis (distillation in a flash @ 600°F, 0.1 mm Hg).
 (f) SRC at T102 Vacuum Distillation Column is based upon solvent-free basis (distillation @ 600°F and 0.1 mm Hg). As-is SRC basis is shown parenthesis.
 (g) Based on adjusted coal feed and Kerr-McGee feed (from T102) rates which produced elementally balanced material balances.
 (h) Based on net SRC in feed and SRC product stream.
 (i) At 600°F and 0.1 mm Hg.

Table 6
 Conditions and Results Summary
 Adjusted Yields
 Kentucky 9, Lafayette Mine Coal

Date, 1979
 Run

28 Sept
 171A MB

Material Balance Method Basis	Process Method		V110 Short Method	
	Unadjusted	Elementally balanced	Unadjusted	Elementally balanced
<u>Yields, % MF coal</u>				
<u>Gases</u>				
H ₂ S	1.97	1.96	1.95	1.81
CO ₂	0.34	0.33	0.34	0.31
CO	0.07	0.06	0.07	0.07
C ₁	1.25	1.20	1.25	1.25
C ₂	0.87	0.83	0.87	0.86
C ₃	0.54	0.52	0.54	0.53
C ₄ -5	0.25	0.24	0.25	0.24
NH ₃	0.13	0.26	0.13	0.24
Water	1.63	10.52	1.63	9.90
<u>Distillates</u>				
C ₅ -350°F	2.71	2.57	2.71	2.59
350-450°F	0.48	0.0	-0.05	-0.36
450°F-EP	22.48	16.71	-1.88	-5.48
<u>SRC (d)</u>				
Oil	9.77	9.49	21.53	21.00
Asphaltene	25.73	25.07	31.94	31.17
Benzene insoluble (a)	17.73	17.34	23.15	22.62
Ash	8.43	7.62	10.08	8.23
Unreacted coal	7.35	6.65	7.22	5.90
<u>Hydrogen consumption, % MF coal</u>	1.73	1.39	1.73	0.88
<u>Organic liquid yield (b)</u>				
<u>Distribution, % of total liquid product (c)</u>				
IBP-350°F	2.71	2.57	2.71	2.59
350-450°F	0.48	0	-0.05	-0.36
450-550°F	3.02	0.60	-3.56	-5.08
550-650°F	8.24	6.54	0.77	-0.36
650°F-EP	11.22	9.57	0.91	-0.04

(Table continued)

Table 6 - continued
 Conditions and Results Summary
 Adjusted Yields
 Kentucky 9, Lafayette Mine Coal

Date, 1979
 Run

1 Oct
 172A MB

Material Balance Method
 Basis

	Process Method		V110 Short Method	
	Unadjusted	Elementally balanced	Unadjusted	Elementally balanced
<u>Yields, % MF coal</u>				
Gases				
H ₂ S	2.12	1.71	2.12	1.60
CO ₂	0.94	0.75	0.94	0.69
CO	0.07	0.07	0.07	0.07
C ₁	1.17	1.16	1.17	1.20
C ₂	0.81	0.78	0.81	0.80
C ₃	0.52	0.49	0.52	0.50
C ₄ 5	0.24	0.23	0.24	0.23
NH ₃	0.13	0.21	0.13	0.20
Water	1.00	7.17	1.00	6.40
Distillates				
C ₅ -350°F	2.67	2.60	2.67	2.62
350-450°F	3.37	3.20	3.08	3.01
450°F-EP	14.73	12.71	-6.26	-6.37
SRC (d)				
Oil	8.00	7.77	19.19	18.75
Asphaltene	30.02	29.19	33.02	32.26
Benzene insoluble (a)	19.43	18.93	25.31	24.73
Ash	9.53	8.18	10.74	8.47
Unreacted coal	7.01	6.02	7.01	5.53
Hydrogen consumption, % MF coal	1.76	1.17	1.76	0.69
Organic liquid yield (b)				
Distribution, % of total liquid product (c)				
IBP-350°F	2.67	2.60	2.67	2.62
350-450°F	3.37	3.20	3.08	3.01
450-550°F	-10.13	-10.90	-15.65	-15.20
550-650°F	7.48	6.85	1.09	0.99
650°F-EP	17.38	16.76	8.30	7.84

(Table continued)

Table 6 - continued
 Conditions and Results Summary
 Adjusted Yields
 Kentucky 9, Lafayette Mine Coal

Date, 1979
 Run

12-14 Dec
 190A MB

Material Balance Method
 Basis

	Unadjusted	Elementally balanced	Unadjusted	Elementally balanced
<u>Yields, % MF coal</u>				
<u>Gases</u>				
H ₂ S	2.48	1.89	2.48	1.76
CO ₂	0.26	0.20	0.26	0.19
CO	0.21	0.17	0.21	0.17
C ₁	1.69	1.63	1.69	1.68
C ₂	1.20	1.16	1.20	1.19
C ₃	0.95	0.92	0.95	0.93
C _{4..5}	0.61	0.59	0.61	0.60
NH ₃	0.13	0.14	0.13	0.13
Water	4.33	5.93	4.33	5.40
<u>Distillates</u>				
C ₅ -350°F	2.33	2.27	2.33	2.31
350-450°F	4.53	4.49	3.89	3.90
450°F-EP	19.25	17.12	-5.45	-3.84
<u>SRC</u>				
Oil	5.93	5.90	22.83	22.63
Asphaltene	23.46	23.32	32.93	32.64
Benzene insoluble ^(a)	21.89	21.78	17.42	17.26
Ash	8.46	10.07	10.05	9.39
Unreacted coal	4.42	5.29	6.31	5.90
<u>Hydrogen consumption, % MF coal</u>	2.13	2.87	2.13	2.24
<u>Organic liquid yield^(b)</u>				
<u>Distribution, % of total liquid product^(c)</u>				
IBP-350°F	2.33	2.27	2.33	2.31
350-450°F	4.53	4.49	3.89	3.90
450-550°F	18.82	17.81	8.87	9.51
550-650°F	5.28	4.83	-0.77	-0.39
650°F-EP	-4.85	-5.52	-13.60	-12.96

(a) Benzene insoluble, cresol soluble.

(b) Liquid fractions by GC determination.

(c) A negative value indicates that the amount of that fraction decreased because part of that fraction was consumed in the process.

(d) Solvent-free basis (distilled to 600°F and 0.1 mm Hg).

Table 7
Coal Feed Summary
Kentucky 9, Lafayette Mine Coal

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Run	Feed Slurry Conc.,		Coal Feed, Pounds			Weighted Average, wt %		
	MF 1b/hr	% MF Coal	As-is	MF	MAF	Moist.	Ash	Sulf.
172 (a)	468	36.5	12,795	12,636	11,541	1.2	8.7	2.9
173	183	23.3	6,877	6,759	6,175	1.7	8.6	2.9
174	155	17.5	4,090	4,036	3,701	1.3	8.3	2.8
175	439	37.0	31,629	31,410	28,483	0.7	9.3	2.8
176	363	37.1	13,312	13,234	11,865	0.6	10.3	2.8
177	461	37.8	25,012	24,879	22,166	0.5	10.9	2.8
178	396	37.3	9,061	9,032	8,019	0.3	11.2	2.8
179	398	36.8	50,411	50,180	45,014	0.5	10.3	2.7
180	403	37.5	25,293	25,090	22,722	0.8	9.4	2.9
181	393	37.0	56,072	55,672	50,458	0.7	9.4	3.0
182	407	36.9	54,909	54,560	49,173	0.6	9.9	2.7
183	411	36.6	13,950	13,774	12,415	1.3	9.9	2.7
184	423	38.0	16,902	16,716	15,103	1.1	9.7	2.7
185	435	36.3	40,978	40,623	36,467	0.9	10.2	3.0
186	437	36.6	70,613	69,951	63,104	0.9	9.8	2.8
187	388	36.5	9,396	9,321	8,445	0.8	9.4	2.7
188	401	37.3	21,037	20,862	18,871	0.8	9.5	2.7
189	436	37.1	23,830	23,717	21,468	0.5	9.5	2.6

(Table continued)

Table 7 (continued)
 Coal Feed Summary
 Kentucky 9, Lafayette Mine Coal

Run	Feed Slurry Conc.,		Coal Feed, Pounds			Weighted Average, wt %		
	MF lb/hr	% MF Coal	As-is	MF	MAF	Moist	Ash	Sulf
190	460	36.9	64,944	64,335	58,095	0.9	9.7	2.6
191	480	37.5	38,339	37,956	34,219	1.0	9.9	2.5
192	470	37.6	19,598	19,446	17,521	0.8	9.9	2.5
193	509	38.8	36,949	36,612	33,174	0.9	9.4	2.7
194	429	35.3	11,901	11,747	10,547	1.3	10.2	2.7
Average	419	36.8				0.8	9.8	2.8
Total			657,898	652,548	588,756			

(a) October portion of run.

Table 8
SRC Production Summary

Run	Coal	MCIF, 1b/hr		MCIF yield, % MAF coal	Analysis, wt %					MP °F	SRC Pounds		MAF		MCIF	
		K125	KM		Solv	CI	Ash	S			K125	MF KM	K125	KM	K125	KM
172 ^(a)	Ky 9	108	209	74.2	18.5	15.7	9.6	-	305	3,434	6,678	3,108	6,042	2,909	5,651	
173	Ky 9	58		-	7.8	21.5	12.2	-	317	2,712		2,381		2,129		
174	Ky 9	65		45.5	9.3	22.9	13.5	1.5	330	2,183		1,888		1,683		
175	Ky 9	216		54.1	2.6	3.74	1.97	1.11	392	16,015		15,699		15,416		
176	Ky 9	182		56.0	3.2	0.75	0.52	0.93	423	6,700		6,665		6,650		
177	Ky 9	223	70	71.5	2.8	5.1	2.5	1.03	405	12,659	4,047	12,369	3,921	12,050	3,804	
178	Ky 9	285		80.9	1.8	0.93	0.76	0.89	410	6,550		6,500		6,489		
179	Ky 9	240		67.3	3.7	0.69	0.58	0.93	231	30,512		30,334		30,301		
180	Ky 9	209		57.2	4.8	0.70	0.46	0.95	522	13,083		13,023		12,991		
181	Ky 9	177		49.8	5.1	1.27	0.45	0.93	540	25,455		25,341		25,132		
182	Ky 9	296		80.8						40,645		40,472		39,708		
183	Ky 9	81		-						2,755		2,749		2,707		
184	Ky 9	208		54.5	4.4	4.28	0.97	0.97	337	8,594		8,511		8,226		
185	Ky 9	252		64.5	5.1	2.35	0.20	1.04	393	24,090		24,043		23,525		
186	Ky 9	250		63.4	4.8	5.54	0.18	1.01	419	42,369		42,291		40,022		
187	Ky 9	84		59.3	7.0	7.39	0.28	1.05	353	5,410		5,395		5,010		
188	Ky 9	347		-	6.5	6.11	0.27	1.05	427	19,227		19,175		18,052		
189	Ky 9	179		45.4	3.7	2.99	0.51	1.12	500	10,043		9,992		9,743		

(Table continued)

Table 8 (continued)
SRC Production Summary

Run	Coal	MCIF, lb/hr		MCIF yield, % MAF coal	Analysis, wt %				MP °F	SRC Pounds					
		K125	KM		Solv	CI	Ash	S		MF K125	MF KM	MAF K125	MAF KM	MCIF K125	MCIF KM
190	Ky 9	244		58.7	4.9	0.86	0.15	0.88	361	34,387		34,336		34,091	
191	Ky 9	276		63.7	5.7	2.79	0.12	1.00	378	22,438		22,412		21,813	
192	Ky 9	267		63.0	4.8	3.30	0.68	0.92	414	11,423		11,345		11,046	
193	Ky 9	416		-	4.9	4.80	2.50	0.99	416	31,500		30,710		29,985	
194	Ky 9	179		46.6	3.8	4.43	0.96	1.03	520	5,140		5,091		4,912	
Average		234	6	63.5	5.1	3.60	1.10	1.02	415						
Total										377,324	10,725	373,830	9,963	364,590	9,455

(a) October portion of run.

Table 9
Feed Coal Analyses
Kentucky 9, Lafayette Mine Coal

Date, 1979	28-30 Sept	1-2 Oct	12-14 Dec
Run	<u>171AB MB</u>	<u>172A MB</u>	<u>190AB MB</u>
<u>Proximate Analysis, wt %</u>			
Moisture	0.61	0.78	1.30
Ash	8.61	8.69	9.33
Volatile Matter	38.53	38.36	31.51
Fixed Carbon	52.25	52.17	57.86
<u>Ultimate Analysis, wt %</u>			
Carbon	72.90	72.49	72.60
Hydrogen	4.73	4.77	4.60
Nitrogen	1.30	1.39	1.42
Sulfur	3.16	3.09	2.79
Chlorine	0.18	0.19	0.24
Ash	8.66	8.76	9.45
Oxygen (by difference)	9.07	9.31	8.90
<u>Dry Heating Value, Btu/lb</u>	13,339	13,327	13,250
<u>Sulfur Forms, wt %</u>			
Pyritic	1.09	1.03	0.75
Sulfate	0.05	0.05	0.14
Sulfide	0.03	0.03	0.05
Organic	1.99	1.98	1.85
<u>Mineral Analysis, wt %</u>			
Phos. Pentoxide, P_2O_5	0.06	0.06	0.07
Silica, SiO_2	54.11	54.05	57.47
Ferric Oxide, Fe_2O_3	19.10	19.33	14.21
Alumina, Al_2O_3	20.01	19.60	20.63
Titania, TiO_2	1.52	1.55	1.42
Lime, CaO	0.91	0.86	1.25
Magnesia, MgO	1.03	1.05	0.91
Sulfur Trioxide, SO_3	0.87	0.72	0.75
Potassium Oxide, K_2O	2.01	2.06	2.00
Sodium Oxide, Na_2O	0.30	0.30	0.65
Undetermined	0.08	0.42	0.64

Table 10
Solvent Refined Coal Analyses
Kentucky 9, Lafayette Mine Coal

Date, 1979	28-30 September			1-2 October			12-14 December	
Run	171AB MB			172A MB			190AB MB	
Sample	CSD-Feed	CSD-SRC	V110(a)	CSD-Feed	CSD-SRC	V110(a)	K125	V110(a)
<u>Proximate Analysis, wt %</u>								
Volatile Matter	44.04	49.86	49.43	46.36	55.64	46.94	41.65	40.98
Fixed Carbon	45.46	50.05	50.57	44.27	43.67	53.06	58.14	59.02
Moisture	<0.01	<0.01	<0.01	<0.01	0.54	<0.01	<0.01	<0.01
Ash	10.50	0.08	0.00	9.37	0.15	0.00	0.21	0.00
<u>Ultimate Analysis, wt %</u>								
Carbon	77.92	87.61	87.67	79.34	87.21	86.58	87.30	87.08
Hydrogen	5.02	5.71	5.61	5.29	6.11	5.53	5.53	5.88
Nitrogen	1.84	1.58	1.96	1.01	1.46	1.88	2.03	1.91
Sulfur	1.69	0.96	1.10	1.40	0.89	1.10	0.91	0.90
Chlorine	0.17	0.0	0.00	0.17	0.00	0.00	0.07	0.08
Ash	10.5	0.08	0.00	9.37	0.15	0.00	0.21	0.00
Oxygen (by difference)	2.86	4.06	3.66	3.42	4.18	4.91	3.94	4.15
Heating Value, Btu/lb	14,245	15,865	15,720	14,440	15,978	15,647	15,757	15,784
<u>Sulfur Forms, wt %</u>								
Pyritic	0.09	<0.01	<0.01	0.13	<0.01	<0.01	<0.01	<0.01
Sulfate	0.11	0.01	0.07	0.03	0.01	<0.01	<0.01	<0.01
Sulfide	0.37	0.02	<0.01	0.54	0.02	0.02	<0.01	<0.01
Organic	1.12	0.93	1.03	0.70	0.86	1.08	0.91	0.90
Melting Point, °F	337	296	365	204	213	409	>518	311
<u>Distillate</u>								
at 500°F, wt %	1.6	2.5	-	22.0	7.7	-	2.9	-
vacuum, mm Hg	0.03	0.02	-	0.10	0.10	-	1.0	-
at 600°F, wt %	9.3	5.8	-	24.0	16.1	-	4.8	-
vacuum, mm Hg	0.13	0.01	-	0.10	0.05	-	0.05	-
<u>Solvent Fractionation Analysis, wt %</u>								
Oil(b)	24.0	33.5	28.9	32.7	31.2	24.6	15.7	31.2
Asphaltenes(c)	35.6	35.1	42.2	32.5	51.2	43.7	43.1	45.0
Benzene insoluble(d)	22.9	30.9	28.9	19.8	17.1	31.7	39.4	23.8
Cresol insoluble coal	7.0	0.4	0.0	5.6	0.3	0.0	1.6	0.0
Ash	10.5	0.1	0.0	9.4	0.2	0.0	0.2	0.0

(a) Laboratory filtered and vacuum distilled.

(b) Pentane soluble.

(c) Benzene soluble, pentane insoluble.

(d) Cresol soluble.

Table 11
Slurry Preheater Operating Data

Run	171	172	175	179	181	182
Date, 1979	29 Sept	1 Oct	10 Oct	21 Oct	31 Oct	7 Nov
B102 Feed, lb/hr						
gas	135	133	131	135	131	136
solvent	786	807	770	663	659	674
coal	446	458	476	391	392	401
Total Feed	1,367	1,398	1,377	1,189	1,182	1,211
B102 Fuel, gph	8.6	8.4	9.0	7.8	7.8	8.3
stack, °F	957	953	972	934	930	953
B102 Inlet, psig	2,185	2,184	2,190	2,190	2,190	2,200
B102 Outlet, psig	2,148	2,153	2,156	2,156	2,158	2,162
Total ΔP	37	31	34	34	32	38
ΔP btm	18	11	17	13	15	17
ΔP mid	14	14	11	15	11	9
ΔP top	5	6	6	6	6	12
B102 Inlet, °F	147	154	141	155	148	136
Turn 7, skin/fluid, °F	353/339	348/333	353/336	340/311	344/331	342/329
Δt	14	15	17	9	11	13
Turn 15, skin/fluid, °F	540/493	533/481	535/477	533/475	528/481	533/494
Δt	47	52	58	58	47	39
Turn 19, skin/fluid, °F	618/538	615/535	614/538	611/538	630/544	633/544
Δt	80	80	78	73	86	89
Turn 23, skin/fluid, °F	638/592	638/588	636/589	639/594	650/593	658/594
Δt	46	50	47	45	57	64
Turn 27, skin/fluid, °F	692/601	687/597	689/595	691/606	697/606	697/599
Δt	91	90	94	85	91	98
Turn 31, skin/fluid, °F	769/729	757/720	764/724	763/728	765/730	770/733
Δt	40	37	40	35	35	37
Turn 33, skin/fluid, °F	798/754	796/750	805/759	804/781	806/763	806/760
Δt	44	46	46	23	43	46
Turn 35, skin/fluid, °F	815/781	813/779	822/786	818/787	825/792	825/791
Δt	34	34	36	31	33	34
Turn 37, skin/fluid, °F	858/817	857/814	869/806	863/815	864/817	867/817
Δt	41	43	63	48	47	50
Coil outlet, °F	798	792	800	797	795	793

(Table continued)

Table 11 (continued)
Slurry Preheater Operating Data

Run	184	185	186	186	186	188
Date, 1979	15 Nov	18 Nov	21 Nov	23 Nov	26 Nov	29 Nov
B102 Feed, lb/hr						
gas	118	116	108	109	114	110
solvent	697	832	752	786	738	665
coal	427	462	451	450	430	401
Total Feed	1,242	1,410	1,311	1,345	1,282	1,176
B102 Fuel, gph						
stack, °F	8.8	8.9	8.2	8.3	8.4	9.0
	972	978	958	957	958	972
B102 Inlet, psig						
B102 Outlet, psig	1,807	1,802	1,789	1,789	1,809	1,800
Total ΔP	1,768	1,771	1,756	1,754	1,786	1,762
ΔP btm	39	31	33	35	23	38
ΔP mid	20	14	15	15	-	15
ΔP top	10	7	6	7	9	9
	9	10	12	13	14	14
B102 Inlet, °F						
Turn 7, skin/fluid	133	143	148	148	134	119
Δt	356/340	363/347	361/345	355/342	357/340	356/334
Turn 15, skin/fluid, °F	16	16	16	13	17	22
Δt	538/498	549/501	555/512	550/507	564/518	554/506
Turn 19, skin/fluid, °F	40	48	43	43	46	48
Δt	647/552	646/554	652/562	656/560	638/557	673/564
Turn 23, skin/fluid, °F	95	92	90	96	81	109
Δt	662/596	655/602	663/606	664/606	669/601	669/599
Turn 27, skin/fluid, °F	66	53	57	58	68	70
Δt	705/601	706/606	708/612	706/609	715/607	710/601
Turn 31, skin/fluid, °F	104	100	96	97	108	109
Δt	771/735	767/737	770/739	770/738	782/747	777/746
Turn 33, skin/fluid, °F	36	30	31	32	35	31
Δt	861/772	819/776	820/777	821/778	824/778	828/783
Turn 35, skin/fluid, °F	44	43	43	43	46	45
Δt	839/801	843/805	842/805	845/807	845/804	850/809
Turn 37, skin/fluid, °F	38	38	37	38	41	41
Δt	880/827	884/830	881/830	884/832	883/831	888/851
Coil outlet, °F	53	54	51	52	52	51
	804	804	803	-	801	805

15

(Table continued)

Table 11 (continued)
Slurry Preheater Operating Data

Run	189	190	190	191	193
Date, 1979	2 Dec	9 Dec	13 Dec	16 Dec	24 Dec
B102 Feed, lb/hr					
gas	81	127	135	151	152
solvent	756	712	810	837	796
coal	448	438	478	496	507
Total Feed	1,285	1,277	1,423	1,484	1,455
B102 Fuel, gph	8.7	9.5	9.0	11.3	10.9
stack, °F	963	944	921	1005	1008
B102 Inlet, psig	1,800	2,148	2,132	1,738	1,725
B102 Outlet, psig	1,770	2,123	2,095	1,688	1,675
Total ΔP	30	25	37	50	50
ΔP btm	13	2	23	26	27
ΔP mid	13	17	6	18	18
ΔP top	4	6	8	6	5
B102 Inlet, °F	129	125	143	131	137
Turn 7, skin/fluid, °F	367/340	342/339	346/331	341/315	364/339
Δt	27	3	15	26	25
Turn 15, skin/fluid, °F	557/515	518/471	530/481	511/461	535/476
Δt	42	47	49	50	49
Turn 19, skin/fluid, °F	670/561	613/548	659/562	640/553	673/565
Δt	109	65	97	87	108
Turn 23, skin/fluid, °F	666/612	662/607	660/608	684/614	672/616
Δt	54	55	52	70	56
Turn 27, skin/fluid, °F	708/602	704/647	695/651	704/669	707/663
Δt	106	57	44	35	44
Turn 31, skin/fluid, °F	772/744	759/729	748/719	765/726	777/735
Δt	28	30	39	39	42
Turn 33, skin/fluid, °F	827/782	792/759	776/744	796/763	808/769
Δt	45	33	32	33	39
Turn 35, skin/fluid, °F	848/807	818/781	805/769	830/799	836/796
Δt	41	37	36	31	40
Turn 37, skin/fluid, °F	884/833	842/810	835/803	866/832	879/837
Δt	51	32	32	34	42
Coil outlet, °F	800	803	791	812	819

Table 12
Dissolver Operating Profile

Run	171	179	179	190	193
Date, 1979	28 Sept	20 Oct	22 Oct	11 Dec	24 Dec
Volume in Use, %	50	50	50	75	75
Coal Space Rate, lb/hr-ft ³ (a)	50	42	46	35	37
B102 Feed, lb/hr					
Coal	453	380	412	471	507
Solvent	798	670	681	816	796
Gas	9,710	9,880	10,070	10,000	11,315
Soda Ash	5.0	4.2	4.6	1.3	2.8
Gas Hydrogen Purity, mole %	84	84	85	85	86
R101 Bottoms Withdrawal Rate, lb/hr	65	65	65	65	95
Withdrawal Sample Analysis, wt %					
UC	4.4	4.8	5.4	7.6	7.5
Ash	6.8	10.7	10.3	11.9	9.7
SRC	39.2	39.6	38.8	37.3	48.2
Solvent (by difference)	49.6	44.9	45.5	43.2	34.2
V110 Analysis, wt %					
UC	3.1	2.7	3.1	2.7	3.5
Ash	5.3	6.2	5.5	4.6	4.8
SRC	35.4	29.7	30.2	30.4	32.5
Solvent (by difference)	56.2	61.4	61.2	62.3	59.2
R101 Temperature Profile, °F					
Inlet	771	768	769	756	781
5% from bottom	812	811	812	811	819
10% from bottom	814	813	815	813	821
15% from bottom	815	815	817	813	819
20% from bottom	819	820	821	815	820
25% from bottom	821	821	822	815	820
50% from bottom	824	824	824	819	821
75% from bottom	-	-	-	825	823
R101 Outlet Pressure, psig	2,135	2,100	2,100	2,100	1,695

(a) Based on no bottoms withdrawal.

Table 13
 Reaction Solids Analyses (V144 Emergency Blowdown Tank)
 Kentucky 9, Lafayette Mine Coal

Date, 1979

3 December

Run 189

Run Source	R101(a)	R101(b)	R101(c)	R101(d)	V144/AWS
<u>Proximate Analysis, wt %</u>					
Moisture	2.04	4.37	3.00	1.70	1.10
Ash	34.71	30.27	43.71	47.25	47.31
Volatile Matter	11.80	8.91	8.19	7.76	10.92
Fixed carbon (by difference)	51.45	56.45	45.10	43.29	40.67
<u>Ultimate Analysis, wt %</u>					
Carbon	50.32	46.23	46.30	39.43	40.32
Hydrogen	2.55	2.20	2.06	1.78	2.17
Nitrogen	1.08	0.85	0.91	0.81	1.07
Sulfur	4.30	5.62	4.52	4.65	6.85
Chlorine	2.79	2.58	4.49	5.32	0.44
Ash	35.43	31.65	41.72	48.01	47.83
Oxygen (by difference)	3.53	10.87	-	-	1.32
<u>Sulfur Forms, wt %</u>					
Pyritic	0.40	0.71	0.53	0.81	0.45
Sulfate	0.23	0.16	0.20	0.14	0.40
Sulfide	2.95	4.31	3.10	3.33	0.79
Organic	0.72	0.45	0.69	0.37	5.21
<u>Mineral Analysis of Ash, wt %</u>					
Phos. Pentoxide, P_2O_5	0.51	0.30	0.37	0.29	0.27
Silica, SiO_2	29.99	27.42	33.84	27.47	45.11
Ferric oxide, Fe_2O_3	26.96	20.56	25.78	15.72	27.94
Alumina, Al_2O_3	11.21	9.03	11.58	11.36	15.20
Titania, TiO_2	2.70	5.33	2.64	10.97	1.69
Lime, CaO	3.31	2.57	2.89	3.57	2.80
Magnesia, MgO	0.65	0.55	0.50	0.77	0.83
Sulfur Trioxide, SO_3	9.89	8.56	9.59	10.46	1.52
Potassium Oxide, K_2O	1.17	0.61	1.41	1.03	1.83
Sodium Oxide, Na_2O	10.84	10.87	8.44	7.92	1.22
Undetermined	2.77	14.20	2.96	10.44	1.59

(a) Solids cake below inlet distributor.
 (b) Solids cake above distributor plate.
 (c) Solids cake wall above distributor.
 (d) Solids cake at 50% point (or on the dissolver wall near the drawoff point).

Table 14
 High Pressure Vent Separator Gas Analyses (V104)
 Kentucky 9, Lafayette Mine Coal

Date, 1979	28-30 Sept	1-2 Oct	12-14 Dec
Run	171AB MB	172A MB	190AB MB
<u>Component</u>	<u>Mol wt</u>	<u>Mole %</u>	<u>Mole %</u>
H ₂	2.02	78.6	80.7
N ₂	28.02	0.2	0.2
CO	28.00	0.6	0.6
CO ₂	44.00	0.2	0.3
H ₂ S	34.08	0.8	0.8
CH ₄	16.03	15.5	13.7
C ₂ H ₂	26.02	ND	ND
C ₂ H ₄	28.03	ND	ND
C ₂ H ₆	30.05	3.1	2.8
C ₃ H ₆	42.05	ND	ND
C ₃ H ₈	44.06	0.9	0.8
iC ₄ H ₁₀	58.08	ND	ND
nC ₄ H ₁₀	58.08	0.1	0.1
iC ₅ H ₁₂	72.15	ND	ND
nC ₅ H ₁₂	72.15	ND	ND
C ₆ H ₁₄	86.18	ND	ND

Table 15
Flare Gas Analyses
Kentucky 9, Lafayette Mine Coal

Date, 1979	28-30 Sept	1-2 Oct	12-14 Dec	
Run	171AB MB	172A MB	190AB MB	
Component	Mol wt	Mole %	Mole %	Mole %
H ₂	2.02	31.8	32.7	28.8
N ₂	28.02	57.5	56.4	59.7
CO	28.00	0.2	0.2	0.2
CO ₂	44.00	0.1	0.1	ND
H ₂ S	34.08	ND	ND	ND
CH ₄	16.03	6.3	6.3	6.8
C ₂ H ₂	26.02	ND	ND	ND
C ₂ H ₄	28.03	ND	ND	ND
C ₂ H ₆	30.05	2.3	2.5	2.5
C ₃ H ₆	42.05	ND	ND	ND
C ₃ H ₈	44.06	1.2	1.3	1.4
iC ₄ H ₁₀	58.08	0.1	0.1	0.1
nC ₄ H ₁₀	58.08	0.4	0.4	0.4
iC ₅ H ₁₂	72.15	ND	ND	ND
nC ₅ H ₁₂	72.15	0.1	ND	0.1
C ₆ H ₁₄	86.18	ND	ND	ND

Table 16
Low Pressure Flash (V110) Product Analyses
Kentucky 9, Lafayette Mine Coal

Date, 1979	28-30 September	1-2 October	12-14 December
Run	171AB MB	172A MB	190AB MB
<u>Composition, wt %</u>			
Cresol insoluble (CI)	7.8	8.2	7.0
Ash	4.6	4.9	4.3
SRC(a)	35.2	35.0	31.3
Distillate(b)	57.0	56.8	61.7
<u>Distillate Composition, wt %, by GC</u>			
IBP-350°F	0.0	0.0	0.1
350-450°F	2.1	1.3	2.5
450-550°F	26.5	25.4	39.1
550-650°F	30.0	30.1	23.8
650°F-EP	41.4	43.2	34.5
<u>Distillate, lab</u>			
<u>Ultimate Analysis, wt %</u>			
Carbon	88.34	88.22	87.52
Hydrogen	7.82	7.95	8.56
Nitrogen	0.86	0.86	0.46
Sulfur			0.30
Ash	<0.01		<0.01
Oxygen (by difference)			3.16
<u>SRC, lab</u>			
<u>Solvent Fractionation and</u>			
<u>Ultimate Analysis (c)</u>			
<u>Cresol Insoluble, lab</u>			
<u>Ultimate Analysis, wt %</u>			
Carbon	32.54	31.18	30.12
Hydrogen	1.51	1.42	1.41
Nitrogen	0.53	0.71	0.46
Sulfur	5.22	4.81	3.79
Ash	59.20	59.30	61.20
Oxygen (by difference)	1.00	2.58	3.02

(a) Distillation conditions: 600°F @ 0.1 mm Hg.

(b) Distillate = 100 - (CI + SRC)

(c) See Table 10.

Table 17
Daily Average Filtration Summary - October 1979

Date, Oct 1979	Run	No of cycles	Filtra- tion time, min	Cycle time, min	Wash solv, lb/cyc	Filtration Volume		Filtration Rate		Precoat, lb/cyc	Precoat range	ΔP, psi		
						To V120 gal*	From V112, gal*	gpm	gph/ft ²			Filtration range	avg	
		(a)												
3	173	2	17	90	500	313	409	18	17	23	3-10	10-50	50	
		(b)												
7	173	2	44	142	811	285	332	6.5	5.9	23	1-2	70-50	50	
		(c)												
9	175	4	35	128	846	205	279	5.9	5.3	23	4-12	80-49	49	
10	8	51	150	805	218	266	4.3	3.9	23	4-10	80-50	49		
11	9	58	145	832	214	260	3.7	3.4	23	4-9	48-50	49		
12(d)	175-6	11	49	131	754	218	259	4.4	4.0	23	4-9	48-50	49	
13	176-7	12	47	128	723	215	255	4.6	4.2	23	4-9	49-50	49	
14	177	5	43	136	677	209	249	4.9	4.4	23	4-15	49-50	49	
15	177	3	34	133	731	179	225	5.3	4.9	23	4-16	49-50	50	
		(e)												
17	177-8	5	46	127	668	209	260	4.6	4.2	23	4-10	49-50	49	
18	178	3	44	99	270	225	289	5.1	4.7	23	4-9	49-50	49	
		(f)												
19	178-9	11	41	127	701	214	257	5.2	4.7	23	4-13	49-50	49	
20	179	11	42	127	780	211	258	5.0	4.6	23	4-9	49-50	49	
21	179	11	47	132	828	211	252	4.5	4.1	23	4-9	49-50	49	
22	179	11	42	124	854	212	249	5.1	4.7	23	4-9	25-90	54	
23	179	10	43	129	834	209	287	4.9	4.4	23	4-7	49-51	51	
24	179	2	48	155	882	209	269	4.4	4.0	23	4-8	49-51	50	
		(g)												
25	179	4	44	123	800	207	259	4.7	4.3	23	4-12	38-51	50	
		(h)												
26	179	1	65	157	525	209	249	3.2	2.9	23	5-13	10-51	50	
		(i)												
28	179-80	9	51	138	790	211	276	4.2	3.8	23	4-9	30-50	50	
29	180	8	52	133	783	204	279	4.0	3.6	23	7-13	29-50	50	
30	180-1	11	41	120	804	186	216	4.6	4.2	23	6-11	40-100	50	
31	182	12	40	122	799	201	235	5.0	4.6	23	6-10	49-52	50	

(Table continued)

Table 17 (continued)
Daily Average Filtration Summary - October 1979

Date, Oct 1979	Run	Filter Cake					Filtrate solids, wt %	V110 LFSFE Sulfur, wt %	Filter feed		Filter Temperature		Sluice, 1b/cycle	Wash time, min
		% solv	% ash	% UC	% SRC	wt, lb			solids, wt %	SRC, wt %	Avg In, °F	Avg Out, °F		
	(a)													
3	173	-	-	-	-	36	0.1-0.3	-	-	-	538	548	740	2
	(b)													
7	173	-	-	-	-	36	0.1-1.3	-	-	-	542	535	1,002	11
	(c)													
9	175	-	-	-	-	160	0.01-0.07	-	4.6	15.8	543	542	1,040	12
10		16.9	53.5	28.6	1.0	199	0.01-0.25	0.98	6.0	23.3	525	536	1,014	15
11		15.4	54.0	28.1	2.5	223	0.01-0.14	1.02	8.1	22.4	527	540	981	22
12(d)	175-6	14.9	56.1	24.3	4.7	225	0.01-0.20	0.88	7.0	23.9	537	543	1,006	18
13	176-7	12.1	56.3	24.9	6.7	219	0.01-0.23	0.87	7.0	24.4	530	541	1,014	22
14	177	18.5	49.4	31.0	1.1	182	0.01	1.03	7.4	25.9	524	534	1,037	21
15	177	-	-	-	-	243	0.01	1.03	7.8	28.2	527	535	1,097	17
	(e)													
17	177-8	24.3	52.7	23.7	-	191	0.01	-	6.7	25.6	550	551	954	12
18	178	-	-	-	-	218	0.01	-	6.1	25.2	547	548	429	-
	(f)													
19	178-9	16.9	54.3	23.9	8.7	214	0.01	-	6.7	23.6	545	542	872	20
20	179	18.1	53.8	24.2	4.4	221	0.01-0.15	0.89	6.9	23.2	536	539	1,045	22
21	179	17.0	53.3	23.9	6.5	229	0.02-0.1	0.98	7.2	25.2	538	542	1,045	21
22	179	19.8	51.8	23.0	5.9	218	0.01-0.15	0.96	7.5	24.1	538	541	993	16
23	179	18.7	54.4	25.1	4.8	212	0.01-0.13	0.82	6.9	23.3	535	536	845	20
24	179	-	-	-	-	182	0.01-0.4	-	-	24.5	543	542	988	24
	(g)													
25	179	-	-	-	-	211	0.01-2.7	-	6.9	26.5	537	539	221	17
	(h)													
26	179	-	-	-	-	269	0.22	-	-	-	528	529	-	13
	(i)													
28	179-80	16.1	56.0	26.0	6.2	217	0.02-0.2	-	7.0	26.8	554	554	896	20
29	180	17.5	59.8	23.0	4.2	198	0.3-0.01	1.05	6.3	22.5	551	551	112	14
30	180-1	14.1	59.5	23.7	5.2	194	0.01	1.02	6.2	26.6	551	556	0	14
31	182	24.0	53.2	27.6	-	211	0.01	0.87	6.0	26.9	549	558	0	17

(Table continued)

Table 17 (continued)
Daily Average Filtration Summary - October 1979

Date, Oct 1979	Run	K125 SRC Analysis			Melting Point, °F	GC Analysis of Solvent in Filter Cake			Cake Drying Conditions	
		Ash, %	CI, %	Solv, %		IBP-350 %	350-450 %	450-550 %	Final Vacuum, In. Hg	Temp, °F
(a) 3	173	13.2	22.9	9.3	330					
(b) 7	173	-	-	-	-					
(c) 9	175	9.1	19.5	3.8	398					
10	175	3.2	4.9	4.0	-					
11	175	0.67	1.6	4.8	356					
12	175-6	0.61	0.87	3.4	423					
13	176-7	0.51	0.70	3.0	423					
14	177	2.3	3.5	3.0	410					
15	177	8.5	14.8	8.0	-					
(e)										
17	177-8	-	-	-	-	1.5	35.7	41.5	25	480-460
18	178	0.76	0.53	1.8	-	-	-	-		
(f)										
19	178-9	0.73	1.0	3.3	257	0.4	32.0	44.6	24	485-425
20	179	0.46	0.6	3.3	233	1.5	29.3	45.9	22	485-428
21	179	0.46	0.6	4.2	276	1.7	31.3	44.9	23	480-419
22	179	0.52	0.74	-	156	1.7	34.5	42.5	23	467-413
23	179	0.52	0.75	3.5	231	-	-	-		
24	179	0.48	0.73	3.6	231	-	-	-		
(g)										
25	179	-	-	-	-	-	-	-		
(h)										
26	179	-	-	-	-	-	-	-		
(i)										
28	179-80	1.85	-	-	-	0.04	38.2	28.0		
29	180	0.63	-	3.1	-	0.3	39.2	33.7		
30	180-1	0.52	-	8.0	493	0.1	41.0	37.8		
31	182	0.24	0.70	3.4	550	-	-	-		

* Converted from weight at 8.34 lb/gal.

(a) Three leaves, 65.8 ft², calendered 24 x 110, 3-ply construction, 1,500 psig sluicing pressure, Dicalite Perlite 436 as precoat.
 (b) Three leaves, 3-ply construction, repaired.
 (c) Three leaves, 65.8 ft², calendered 24 x 110, 2-ply construction.
 (d) Asbestos at 7.5% of precoat added for 8 cycles.
 (e) Leaf drive would not turn due to cut-off blade jamming on stop. Three leaves of 2-ply construction reinstalled.
 (f) Flexitallic head gasket leaking. Replaced with Raybestos gasket.
 (g) Poor filtrate clarity. Three leaves of 3-ply construction installed.
 (h) Poor filtrate clarity due to disintegration of 24 x 110 near silver-solder. Three leaves of 2-ply construction installed.
 (i) Hub gasket wedged between leaf cut-off blade hub guide and spacer rings. One leaf of 3-ply (CI) and two leaves of 2-ply (B3,B4) installed. Sluice pressure reduced to 1,000 psig.

Table 18
Daily Average Filtration Summary - November 1979

Date, Nov 1979	Run	No of cycles	Filtra- tion time, min	Cycle time, min	Wash solv, lb/cyc	Filtration Volume		Filtration Rate		Precoat 1b/cyc	Precoat range	ΔP, psi	
						To V120, gal*	From V112, gal*	gpm	gph/ft ²			Filtration range	avg
1	181	11	42	133	751	209	238	5.0	4.5	23	4-16	49-50	50
2	181	10	43	131	757	210	231	4.9	4.4	23	5-14	49-50	50
3	181	11	49	130	746	209	237	4.3	3.9	23	4-17	49-50	49
4	181	11	47	128	753	212	230	4.5	4.1	23	4-18	49-50	49
5	181-2 (a,b)	11	52	133	737	209	235	4.0	3.7	23	6-16	40-50	50
6	182	10	56	147	737	209	238	3.7	3.4	23	8-20	49-50	50
7	182	10	52	137	782	209	240	4.0	3.7	23	8-15	30-50	50
8	182	11	47	128	787	209	228	4.4	4.1	23	8-15	30-50	50
9	182	13	44	113	764	211	233	4.8	4.4	23	8-15	30-50	50
10	182	13	40	111	787	210	235	5.2	4.7	23	6-12	40-50	50
11	182	4	47	121	725	223	243	4.8	4.3	23	7-14	44-50	50
(c)													
13	183	9	30	90	737	209	233	7.0	6.4	23	11-20	48-50	50
14	183-4	13	36	99	737	210	230	5.8	5.3	23	10-13	48-50	50
15	184	8	35	95	774	233	253	6.6	6.1	23	6-12	49-50	50
(d)													
16	185	4	53	116	737	286	317	5.4	4.9	23	5-10	40-60	50
17	185	11	46	107	755	289	321	6.2	5.7	23	12-17	30-50	50
18	185	10	55	121	772	280	310	5.1	4.7	23	9-12	30-50	50
19	185	9	60	124	748	280	310	4.7	4.3	23	8-12	30-50	50
20	185-6	8	69	137	762	289	319	4.2	3.8	23	8-12	30-50	50
(b)													
21	186	10	54	124	757	273	302	5.0	4.6	23	7-12	30-90	58
22	186	9	60	121	748	290	320	4.8	4.4	23	7-12	25-50	44
23	186	8	61	122	737	289	319	4.8	4.3	23	7-12	30-50	50
24	186	8	52	116	768	281	316	5.4	4.9	23	9-13	30-50	50
25	186	9	56	121	737	280	315	5.0	4.6	23	10-14	30-50	50
26	186	8	68	139	737	289	322	4.2	3.9	23	10-15	30-50	50
27	186-7	8	57	120	805	272	320	4.8	4.4	5.8	-	0-60	50
28	187-8	9	58	120	743	289	331	5.0	4.6	23	12-16	30-50	50
29	188	7	43	105	737	290	-	6.8	6.2	23	12-16*	30-50	50
(e)													
30	188	3	43	98	731	289	348	6.7	6.1	0	-	0-49	46

(Table continued)

Table 18 (continued)
Daily Average Filtration Summary - November 1979

Date, Nov 1979	Run	Filter Cake				Filtrate solids, wt %	V110 LFSFE Sulfur, wt %	Filter feed		Filter Temperature		Sluice, lb/cycle	Wash time, min	
		% solv	% ash	% UC	% SRC			solids, wt %	SRC, wt %	Avg In, °F	Avg Out, °F			
1	181	22.4	54.6	28.9	6.1	180	0.01	0.93	6.1	26.6	550	561	72	22
2	181	16.1	55.4	32.6	3.4	195	0.01	0.99	6.5	28.5	537	549	0	20
3	181	12.2	65.0	33.0	5.9	205	0.01-0.03	0.90	6.2	28.2	533	542	0	15
4	181	21.8	54.1	28.8	5.5	192	0.01-0.02	0.90	6.3	27.4	532	541	91	14
5	181-2	12.6	54.0	27.5	5.8	193	0.01	0.89	6.5	29.7	534	543	0	20
(a,b)														
6	182	6.8	57.2	31.6	2.4	189	0.07-0.12	1.13	6.5	29.3	534	543	167	26
7	182	18.7	60.3	26.8	4.0	190	0.03-0.1	0.97	6.6	27.5	535	544	0	29
8	182	18.9	60.8	12.0	6.9	181	0.08-0.10	1.29	6.5	27.5	537	544	85	26
9	182	7.9	65.0	26.7	5.7	175	0.05-0.02	0.96	6.5	26.7	539	545	0	22
10	182	12.4	58.4	28.7	4.6	182	0.02-1.1	0.90	6.7	26.7	542	546	68	20
11	182	-	-	-	-	199	0.03-0.09	0.96	6.8	29.1	538	545	221	22
(c)														
13	183	11.3	58.2	29.7	3.8	185	0.03-0.04	-	6.1	22.5	542	548	0	16
14	183-4	4.0	68.1	25.2	3.5	174	0.04-0.06	0.93	6.2	26.7	543	554	0	17
15	184	1.4	65.3	27.8	4.1	173	0.02-2.8	0.98	6.4	28.5	544	550	123	14
(d)														
16	185	-	-	-	-	225	0.04-0.08	1.36	6.0	25.2	541	555	0	19
17	185	3.6	62.6	30.8	4.3	196	0.02-0.11	1.26	6.1	25.8	545	556	91	17
18	185	2.3	60.0	32.9	2.3	213	0.02-0.05	1.05	6.1	26.3	541	554	103	22
19	185	11.1	53.2	34.6	3.7	236	0.01-0.07	-	6.8	28.5	548	560	0	22
20	185-6	5.5	56.4	34.4	2.6	252	0.01-0.05	0.91	6.8	27.9	533	550	0	24
(b)														
21	186	5.2	59.1	34.1	2.0	229	0.01-0.1	0.85	6.8	30.5	538	550	108	22
22	186	9.2	56.1	31.1	1.6	250	0.04-0.7	0.87	6.8	28.8	535	549	0	18
23	186	-	-	-	-	263	0.01-0.16	0.94	7.3	28.9	532	548	0	19
24	186	-	-	-	-	257	0.02-0.08	0.95	7.1	29.2	534	547	123	17
25	186	7.3	51.6	37.7	2.8	262	0.04-0.08	1.03	7.1	29.2	533	547	0	19
26	186	7.1	50.9	37.5	2.4	277	0.02-0.18	1.16	7.0	30.9	530	547	125	22
27	186-7	5.6	31.3	62.5	0.8	208	0.2-0.5	1.09	7.6	29.8	532	544	459	20
28	187-8	3.7	50.8	42.9	3.7	243	0.05-0.8	1.08	6.8	31.2	532	548	80	18
29	188	8.1	50.7	38.9	6.0	218	0.05-0.09	1.02	7.0	28.6	535	547	95	15
(e)														
30	188	7.1	50.8	44.8	9.0	223	0.05-0.14	0.90	-	-	539	539	747	12

(Table continued)

Table 18(continued)
Daily Average Filtration Summary - November 1979

Date, Nov 1979	Run	K125 SRC Analysis			Melting Point, °F	GC Analysis of Solvent in Filter Cake			Cake Drying Conditions	
		Ash, %	CI, %	Solv, %		IBP-350 %	350-450 %	450-550 %	Final Vacuum, In. Hg	Temp, °F
1	181	0.51	1.3	7.3	550	1.6	44.0	33.4	21	473-409
2	181	0.31	1.2	2.8	550	1.4	42.1	34.1	21	471-417
3	191	0.50	1.4	5.5	476	0.7	25.4	33.3	22	448-379
4	181	0.81	1.4	4.4	-	0.9	31.0	35.9	22	451-396
5	181-2	0.26	1.3	7.3	572	1.1	30.2	32.4	22	472-413
(a,b)										
6	182	0.41	3.1	2.8	414	1.9	37.1	33.6	22	472-438
7	182	0.30	3.0	3.4	443	1.5	41.3	39.5	21	474-425
8	182	0.49	0.85	4.6	405	1.5	39.0	31.9	20	476-421
9	182	0.40	3.1	2.5	536	0.8	26.4	32.1	19	476-419
10	182	0.52	1.04	6.6	550	1.3	24.0	33.4	20	476-427
11	182	0.42	3.1	6.6	-					
(c)										
13	183	-	-	-	-	0.9	31.9	24.8		
14	183-4	0.21	1.74	2.9	370					
15	184	0.98	4.3	6.0	304					
(d)										
16	185	-	-	-	-					
17	185	0.27	1.96	5.3	348					
18	185	0.13	1.67	3.88	371					
19	185	0.19	4.36	5.71	352	0.8	40.8	33.2		
20	185-6	0.20	0.95	5.3	500					
(b)	186	0.16	5.1	6.3	388	0.1	56.7	30.4	19	500-464
22	186	0.13	6.1	4.4	383	0.2	58.5	28.3	22	497-456
23	186	0.18	4.0	4.7	424					
24	186	0.17	5.4	3.5	383					
25	186	0.20	4.9	4.9	456	0.1	46.8	27.0	20	496-456
26	186	0.26	7.5	4.7	397	0.3	56.4	26.7	20	505-465
27	186-7	0.22	-	-	-					
28	187-8	0.33	7.3	7.0	353	1.1	50.6	29.7	20	489-454
29	188	0.26	-	-	-	0.0	41.1	27.2	20	493-457
(e)										
30	188	0.26	4.4	6.1	500	1.11	32.7	27.4	20	492-470

* Converted from weight at 8.34 lb/gal.

(a) % ash in filtrate rather than % CI.

(b) Modified lab procedure for % solvent in cake.

(c) Hole in leaf B3, cut-off blades broken, changed to 2-ply and perforated sheet, B1, B2.

(d) Screens bad on leaves B1, B2; used leaves B4, C1, and 2-ply with perforated sheet.

(e) B4 hole in leaf from cut-off blade. Installed D1, D2, D3 with no diffusion bonding.

Table 19
Daily Average Filtration Summary - December 1979

Date, Dec 1979	Run	No of cycles	Filtra- tion time, min	Cycle time, min	Wash solv, lb/cyc	Filtration Volume		Filtration Rate		Precoat, lb/cyc	Precoat range	ΔP, psi		
						To V120, gal*	From V112, gal*	gpm	gph/ft ²			Filtration range	avg	
1	189	9	43	93	726	283	368	6.6	6.0	0	-	26-50	46	
2	189	7	45	96	716	289	371	6.4	5.9	0	-	40-45	42	
(a)	6	190	5	32	90	737	273	330	8.4	7.7	23	8-14	30-50	50
	8	190	3	24	76	770	289	358	12.2	11.1	23	5-14	30-50	50
	9	190	9	27	80	737	275	356	10.2	9.3	23	8-12	30-50	50
	10	190	8	55	118	737	280	317	5.1	4.6	23	8-12	30-50	50
	11	190	10	61	131	737	281	316	4.6	4.2	23	8-12	30-50	50
	12	AMB	10	69	142	737	276	309	4.0	3.7	23	8-12	30-50	50
	13	AMC	10	72	146	737	284	318	4.0	3.6	23	8-12	30-50	50
	14	BMB	9	51/90	121/156	737	259	302	4.0	3.7	23/0	7-11	30-50	50
	15	191	9	102	172	737	280	348	2.7	2.5	0	-	50-50	50
	16	191	8	113	186	768	262	336	2.3	2.1	0	-	50-50	50
	17	191	6	95	163	779	229	292	2.4	2.2	0	-	46-50	48
	19	192	8	103	169	748	261	323	2.5	2.3	0	-	46-50	46
	20	192	9	94	165	816	267	307	2.9	2.6	0	-	48-50	49
	21	193	2	93	185	1,026	239	253	2.6	2.4	0	-	48-50	49
	22	193	8	84	166	722	287	327	3.4	3.1	0/23	13-30	35-50	50
	23	193	9	70	145	737	292	322	4.2	3.8	23	12-22	30-50	50
	24	193	11	43	113	755	278	313	6.4	5.8	23	15-22	30-50	50
	25	193	7	35	100	778	299	325	8.6	7.8	23	13-24		50
	31	193	7	54	121	750	276	357	5.1	4.7	0	-	50-50	50

(Table continued)

Table 19 (continued)
Daily Average Filtration Summary - December 1979

Date, Dec 1979	Run	Filter Cake				Filtrate solids, wt %	V110 LFSFE Sulfur, wt %	Filter feed		Filter Temperature		Sluice, lb/cycle	Wash time, min			
		% solv	% ash	% UC	% SRC			solids, wt %	SRC, wt %	Avg In, °F	Avg Out, °F					
1	189	5.7	45.8	49.1	1.0	208	0.09-2.2	-	7.1	25.9	552	552	774	9		
2	189	-	-	-	-	182	0.6-1.3	1.2	6.7	27.6	552	552	718	8		
(a)	6	190	-	-	-	225	0.02-0.11	-	-	28.8	544	547	0	11		
	8	190	-	-	-	200	0.02-0.04	-	7.1	25.2	545	545	0	7		
	9	190	14.4	49.0	33.0	10.2	0.01-0.14	0.99	5.2	22.7	542	548	0	9		
	10	190	10.1	57.9	35.2	1.8	0.01-0.15	0.77	5.3	26.1	534	546	0	21		
	11	190	14.3	55.8	29.9	-	0.01-0.06	0.83	5.9	26.8	530	543	0	26		
	12	AMB	-	-	-	211	0.02-0.06	0.83	6.0	30.2	532	545	0	29		
	13	AMC	4.0	60.2	33.7	-	219	1.14	7.2	-	530	546	81	28		
	14	BMB	-	-	-	207	0.03-0.07	1.02	5.6	-	528	542	175	23/37		
	15	191	10.7	53.4	31.9	6.7	0.03-0.07	0.88	6.8	29.6	519	537	175	37		
	16	191	8.9	49.4	36.9	2.7	0.02-0.08	0.93	7.2	30.6	519	537	197	39		
	17	191	-	-	-	-	0.04-0.10	1.06	7.5	31.2	539	547	723	30		
	(b)	19	192	6.0	52.9	40.3	0.8	162	0.02-0.08	-	8.1	26.4	552	550	936	24
		20	192	-	-	-	-	165	0.05-2.0	0.94	8.9	27.9	548	552	1,023	32
(c)	21	193	-	-	-	-	214	0.07-0.2	0.93	7.8	29.7	509	522	811	53	
	22	193	14.5	50.2	31.8	2.0	231	0.04-0.7	-	7.2	27.7	531	543	644	35	
	23	193	8.7	56.7	33.3	1.2	211	0.04-1.5	0.82	6.5	29.5	540	556	85	25	
	24	193	14.0	54.3	28.3	2.4	160	1.6-2.5	0.83	7.0	31.4	540	555	0	15	
	25	193	-	-	38.6	-	175	1.6-2.4	0.92	7.5	30.6	543	555	126	11	
(d)	31	193	-	-	-	-	121	0.4-0.9	-	5.1	10.4	540	550	676	28	

(Table continued)

Table 19 (continued)
Daily Average Filtration Summary - December 1979

Date, Dec 1979	Run	K125 SRC Analysis			Melting Point, °F	GC Analysis of Solvent in Filter Cake			Cake Drying Conditions	
		Ash, %	CI, %	Solv, %		IBP-350 %	350-450 %	450-550 %	Final Vacuum, In. Hg	Temp, °F
1	189	0.38	2.9	3.7	-	1.0	32.8	29.4	22	500-474
2	189	-	-	-	500	-	-	43.4		
(a)										
6	190	-	-	-	-	1.5	40.0	36.8	16	480-448
8	190	-	-	-		1.1	31.5	34.9	15	493-459
9	190	0.23	1.4	4.2	-	1.6	46.6	34.3		
10	190	0.15	0.6	4.0	352					
11	190	0.15	0.6	4.0	352					
12	AMB	0.08	0.9	-	-					
13	AMB	0.21	0.9	-	-					
14	AMB	0.05	1.0	7.3	378	0.4	54.3	29.3	18	488-547
15	191	0.05	1.0	7.3	378	0.0	43.6	27.5	18	485-462
16	191	0.05	4.1	3.1	382	0.2	56.9	26.8	18	490-465
17	191	0.19	4.4	5.0	375					
(b)										
19	192	0.27	1.6	6.9	-	3.3	60.1	28.3	18	484-470
20	192	0.18	3.2	3.9	414					
(c)										
21	193	1.5	4.4	4.7	415					
22	193	1.5	4.4	4.7	415	0.0	60.5	28.8	19	484-456
23	193	1.0	2.3	4.4	389	0.0	59.4	28.2	20	482-448
24	193	2.5	5.6	4.2	430	0.8	59.1	28.2	20	484-454
25	193	4.7	6.6	6.5	428	0.0	37.0	31.9	19	487-447
(d)										
31	193	0.96	4.4	3.8	520					

* Converted from weight at 8.34 lb/gal.

(a) Gasket seal failure. Reinstalled leaves D1, D2, D3 (non-diffusion bonded leaves).
 (b) Screens torn at spot welds. To leaves C1, B3, and 2-ply with perforated sheet; using 6510 nozzles.
 (c) Screen disintegration on leaf B3. To leaves C1, D2, and 2-ply with perforated sheet.
 (d) Hole in leaf D2. Leaf D2 reinstalled. Using 4006 nozzles.

Table 20
Vacuum Column Operating Data
T102

Date, 1979	28 Sept	1 Oct	12-14 Dec
Run	171 A	172 A	190 A-B
<u>Operating Conditions</u>			
Pressure, psia	0.3	0.3	0.4
Flow rates, lb/hr			
Feed			
from V110	990.7	1,000.0	372.8
from T104 bottom	0	0	0
Overhead Light Solvent			
to T104 feed	153.0	170.0	47.4
Tray 1 reflux	4,500	4,200	7,400
Wash Solvent (Tray 3)			
to V178	To T105	To T105	To T105
Process Solvent (Tray 8)			
to V131B	To T105	To T105	To T105
Bottoms (SRC)			
to K125/CSD	359.1	465.6	253.6
to B103	>18,227	>18,227	>18,227
Temperature, °F			
Tray 1 (ovhd)	202	203	190
Tray 8	339	387	340
Packing reflux	180	181	175
B103 outlet	571	-out-	600
Bottom	561	499	597
Product Pump Power, amps	33	27	32
<u>Compositions, wt %</u>			
Tray 3			
IBP-350 °F	See T105	See T105	See T105
350-450 °F			
450°F-EP			
Tray 8			
IBP-350 °F	See T105	See T105	See T105
350-450 °F			
450°F-EP			

Table 21
T104 Light Organics Recovery Column Operating Data
Kentucky 9, Lafayette Mine Coal

Date, 1979	28 Sept	1 Oct	12-14 Dec
Run	171A	172A	190A-B
<u>Operating Conditions</u>			
Pressure, psig	9.5	9.4	11.0
Flow rates, lb/hr			
Feed			
from V105	216.0	235.7	117.5
from T102	153.0	170.0	47.4
Overhead			
Product	15.5	10.1	12.8
Reflux	0.0	0.0	0.0
Vent	0.0	0.0	0.0
Temperature, °F			
Top	207	206	208
Bottom	-	-	-
Feed	208	208	204
<u>Composition, wt %</u>			
Feed			
IBP-350°F	6.2	5.8	12.8
350-450°F	18.1	15.4	13.3
450°F-EP	75.7	78.8	73.9
Water in feed			
from V105	0.2	0.4	0.3
from T102	1.0	1.0	0.6
Overhead oil			
IBP-350°F	91.4	92.6	98.9
350-450°F	8.6	7.4	1.1
450°F-EP	0.0	0.0	0.0
Bottom			
IBP-350°F	1.1	1.0	6.6
350-450°F	15.1	13.5	26.3
450°F-EP	83.8	85.5	67.1
<u>Specific gravity</u>			
Overhead	0.784	0.775	0.738
Bottom	0.999	0.998	0.968
Feed	0.982	0.979	0.950

Table 22
Organic Liquid Product Analyses
Kentucky 9, Lafayette Mine Coal

Date, 1979	28 Sept	1 Oct	12-14 Dec
Run	171A	172A	190A-B
<u>Recycle solvent (V131B)</u>			
<u>Boiling fractions, wt %</u>			
(IBP, °F)	(369)	(405)	(424)
IBP-350°F	0.1	0.1	0.0
350-450°F	5.8	3.0	0.8
450-550°F	35.9	42.5	47.6
550-650°F	27.4	27.7	23.1
650°F-EP	30.8	26.7	28.5
(EP, °F)	(907)	(905)	(883)
<u>Specific Gravity</u>	1.032	1.023	1.016
<u>Ultimate Analysis, wt %</u>			
Carbon	88.6	88.4	88.0
Hydrogen	8.2	8.4	8.4
Nitrogen	0.6	0.4	0.4
Chlorine	-	-	-
Sulfur	0.4	0.4	0.4
Ash	0.0	0.0	0.0
Oxygen (by difference)	2.2	2.4	2.8
<u>Light Organic Liquid from T104 overhead</u>			
<u>Boiling fractions, wt %</u>			
(IBP-°F)	(132)	(142)	(146)
IBP-200°F	44.9	51.4	90.8
200-350°F	46.5	41.2	8.1
350-450°F	8.6	7.4	1.1
450°F- EP	0.0	0.0	0.0
(EP, °F)	(405)	(406)	(360)
<u>Specific Gravity</u>	0.784	0.775	0.738
<u>Ultimate Analysis, wt %</u>			
Carbon	86.1	85.0	84.2
Hydrogen	13.2	13.5	14.4
Nitrogen	0.4	0.9	0.9
Chlorine	-	-	-
Sulfur	0.3	0.4	0.4
Ash	<0.01	<0.01	0.0
Oxygen (by difference)	-	0.2	0.1

Table 23
Fractionation Column Operating Data
T105

Date, 1979	28 Sept	1 Oct	12-14 Dec
Run	171 MB	172 MB	190 MB
<u>Operating Conditions</u>			
<u>Pressure, psig</u>	5.3	5.5	4.5
<u>Rate, lb/hr</u>			
Feed	832.1	760	1,223.9
Overhead product	75.2	26.4	339.4
Reflux	1.64	2.05	2.79
Vent	0	0	0
Reboiler	95,597	95,701	107,586
<u>Temperature, °F</u>			
Top	383	399	402
Reflux	333	341	341
Middle	409	437	445
Feed	349	356	282
Reboiler	552	558	565
<u>Composition, wt %</u>			
<u>Overhead</u>			
Light organic liquid	10.1	6.4	4.2
Wash solvent	88.3	87.3	82.3
<u>Bottom</u>			
Light organic liquid	0	0	0
Wash solvent	2.9	2.9	0.8
<u>Feed</u>			
Light organic liquid	0.8	0.8	4.2
Wash solvent	8.0	10.2	34.5

Table 24
Recovered Solvent Analyses

Date, 1979	28 Sept	1 Oct	12-14 Dec
Run	171 A	172 A	190 A-B
<u>Recycle Solvent from T105 btrms</u>			
<u>Boiling Fractions, wt %</u>			
(IBP- °F)	(415)	(415)	(424)
IBP-350 °F	0	0	0
350-450 °F	2.9	2.9	0.8
450-550 °F	44.0	42.2	46.6
550-650 °F	26.6	28.1	22.9
650°F-EP	26.5	26.8	29.7
(EP, °F)	(872)	(892)	(883)
<u>Specific gravity</u>	1.032	(1.022)	1.030
<u>Ultimate Analysis, wt %</u>			
Carbon	88.8	88.5	88.1
Hydrogen	8.3	8.2	8.6
Nitrogen	0.6	0.8	0.2
Chlorine	-	-	-
Sulfur	0.2	0.4	0.4
Ash	0	0	0
Oxygen (by difference)	2.1	2.1	2.7
<u>Wash Solvent from T105 (ovhd)</u>			
<u>Boiling Fractions</u>			
(IBP- °F)	(195)	(198)	(251)
IBP-350 °F	10.1	6.4	4.2
350-450 °F	88.3	87.3	82.3
450-550 °F	1.6	6.3	13.5
550-650 °F	0	0	0
650°F-EP	0	0	0
(EP, °F)	(460)	(466)	(480)
<u>Specific gravity</u>	0.932	0.957	0.954
<u>Ultimate Analysis, wt %</u>			
Carbon	83.0	83.4	83.2
Hydrogen	9.9	9.3	9.7
Nitrogen	1.1	1.2	1.1
Chlorine	-	-	-
Sulfur	0.2	0.4	0.1
Ash	<0.01	0	0
Oxygen (by difference)	5.8	5.7	5.9

Table 25
Organic Liquid Analyses
Kentucky 9, Lafayette Mine Coal

Date, 1979	28 Sept	1 Oct	12-14 Dec
Run	171A	172A	190A-B
<u>Organic Liquid from V105</u>			
<u>Boiling fractions, wt %</u>			
(IBP, °F)	(146)	(249)	(139)
IBP-200°F	3.2	0.0	5.5
200-350°F	2.9	5.4	3.3
350-450°F	14.8	12.7	6.7
450°F-EP	79.1	81.9	84.5
(EP, °F)	(740)	(722)	(751)
<u>Specific Gravity</u>	0.981	0.981	0.958
<u>Ultimate Analysis, wt %</u>			
Carbon	88.3	87.6	86.9
Hydrogen	8.8	8.9	9.3
Nitrogen	1.5	1.3	1.3
Sulfur	0.4	0.4	0.2
Ash	0.0	0.0	0.0
Oxygen (by difference)	1.0	1.8	2.3
<u>Organic liquid from T102 ovhd</u>			
<u>Boiling fractions, wt %</u>			
(IBP, °F)	(281)	(281)	(231)
IBP-200°F	0.0	0.0	0.2
350-450°F	3.3	2.5	15.4
450°F-EP	64.7	60.1	75.6
(EP, °F)	32.0	37.4	8.8
	(507)	(511)	(492)
<u>Specific Gravity</u>	0.972	0.978	0.913

Table 26
Ash Concentrate Analyses (KM-CSD Unit)
Kentucky 9, Lafayette Mine Coal

Date, 1979	28-29 September	1-2 October
Run	<u>171A-B</u>	<u>172</u>
<u>Composition, wt %</u>		
Ash	28.8	29.9
Unreacted coal	27.2	31.8
Solvent refined coal	36.3	24.8
Solvent	4.3	9.1
Deashing solvent	3.4	4.4
<u>Solvent Extraction Analyses, wt %</u>		
Oil	10.6	10.5
Asphaltenes	6.1	4.9
Benzene insoluble (cresol soluble)	27.3	22.9
Cresol insoluble	27.2	31.8
Ash	28.8	29.9
<u>Ultimate Analysis, wt %</u>		
Carbon	59.24	56.92
Hydrogen	3.13	2.98
Nitrogen	1.16	1.07
Ash	29.80	31.30
Sulfur	3.02	3.11
Chlorine	0.57	-
Oxygen (by difference)	3.08	4.62

Table 27
Cresol and Quinoline Extraction of CSD Ash Concentrate and CSD Feed

Run No. and Date	166A-C MB (31 Aug 1979)				168 (11 Sept 1979)			
Sample	Ash Concentrate (SN97505)			Ash Concentrate (SN48256)		Feed (SN48257)		
Extraction solvent	cresol	quinoline	quinoline	cresol	quinoline	cresol	quinoline	quinoline
Extraction method			(ASTM) (a)		(ASTM) (a)			(ASTM) (a)
Solvent amount, ml	450	200	25	450	25	450	100	25
Sample amount, gm	10	1	0.5	10	0.5	10	1.0	0.6
Extraction temp, °F	210	260-275	160	210	160	210	~340	160
Extraction time, hr	1	1	1	1	1	1	1	1
Insolubles								
% of initial amount	61.2	40.9	39.3	61.7	50.0	17.7	18.2	20.1
Ash, %	24.1	24.1	24.1	30.2	30.2	10.3	10.3	10.3
Unreacted coal, %	37.1	16.8 (b)	15.2	31.5	19.8 (b)	7.4	7.9	9.8

(a) Modified ASTM method D-2318-66 (1971).

(b) Theoretical UC in ash concentrate equivalent to UC in feed.

Run 166 = (24.4/10.2) x 7.4 = 17.7% ~ 16.8.

Run 168 = (30.2/10.3) x 7.4 = 21.7% ~ 19.8.

Table 28
Heteroatom Removal

Run	Heteroatom	Weight Ratio of Heteroatom to Carbon				Heteroatom Removal, %				
		Feed Coal	V110	CSD Feed	SRC	V110	CSD Feed	K125	CSD Product	
171AB MB	Oxygen	0.124	0.042	0.037	-	0.046	66.1	70.2	-	62.9
	Nitrogen	0.018	0.022	0.024	-	0.018	-22.2	-33.3	-	0.0
	Sulfur	0.043	0.013	0.022	-	0.011	69.8	48.8	-	74.4
172A MB	Oxygen	0.128	0.057	0.043	-	0.048	55.5	66.4	-	62.5
	Nitrogen	0.019	0.022	0.013	-	0.017	-15.8	31.6	-	10.5
	Sulfur	0.043	0.013	0.018	-	0.010	69.8	58.1	-	76.7
190AB MB	Oxygen	0.123	0.048	-	0.045	-	61.0	-	63.4	-
	Nitrogen	0.020	0.022	-	0.023	-	-10.0	-	-15.0	-
	Sulfur	0.038	0.010	-	0.010	-	73.7	-	73.7	-

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Run	Coal	Heating Value, Btu/lb			Total Sulfur, wt %			Total Sulfur Removal, %			
		V110	K125	CSD Product	SRC	V110	K125	CSD Product	V110	K125	CSD Product
171AB MB	13,339	15,720	-	15,865	3.16	1.10	-	0.96	70.5	-	74.5
172A MB	13,327	15,647	-	15,978	3.09	1.10	-	0.89	69.7	-	76.0
190AB MB	13,250	15,784	15,757	-	2.79	0.90	0.91	-	72.9	72.6	-

Table 29
Ultrasonic Test Data

<u>T105</u> <u>Tray</u>	15 June 1979 Original <u>Thickness (in.)</u>	21 September 1979		28 December 1979	
		<u>Loss (mils)</u>	<u>% Loss</u>	<u>Loss (mils)</u>	<u>% Loss</u>
1	0.135	Not Measured		0-5	3.7
10	0.135	15	11	15-25	18.5
11	0.135	20-25	18.5	Not Measured	
12	0.135	15	11	25-35	25.9
13	0.135	15	11	Not Measured	
14	0.135	25	18.5	Not Measured	
15	0.135	15-20	14.8	Not Measured	

Table 30
T105 Corrosion Coupon Data

Coupon Material	Original Wt (grams)	21 September 1979			28 December 1979		
		Wt (grams)	% Loss	Substitutions	Wt (grams)	% Loss	Substitutions
Rod 2 (Top)							
Hast. C-276	65.5421				65.5420	0.0	
Hast. G	123.4158				123.4158	0.0	
Hast. G-3	120.3792				120.3786	0.0	
Titanium	10.5187				10.5189	Gain	
Haynes 20 mod	29.3836				29.3835	0.0	
2 RE 69	51.2049				51.7052	Gain	
317	116.0051				115.9621	0.0	
304	82.7504				82.7488	0.0	
410	109.8596				109.8666	Gain	Inconel 625
Monit	25.6237				25.6227	0.0	
SC-1	30.0074				30.0075	Gain	
CS	116.2165				116.0101	0.2	Incoloy 825
18CR-2MO	71.4475				71.4443	0.0	
26-15	92.2600				92.2586	0.0	
Nitronic 50	21.9140				21.9134	0.0	321 904L
Rod 1 (Middle)							
Hast. C-276	64.831	64.827	0.0		64.8285	0.0	
Hast. G	123.289	123.046	0.2		122.9289	0.3	
Hast. G-3	121.122	120.866	0.2		120.7876	0.3	
Titanium	10.485	9.914	5.4		9.9103	5.5	310
Haynes 20 mod	29.648	29.066	1.9		27.8618	6.0	
2 RE 69	51.736	48.941	5.4		43.5888	15.7	
317	115.963	111.181	4.1		107.8186	7.0	
304	82.568	76.043	7.9		70.8958	14.1	
410	110.203	97.680	11.4		92.0356	16.5	Monel 400
Monit	25.580	12.214	52.3		9.2129	64.0	904L
SC-1	29.990	11.800	60.6	Incoloy 825	88.982	5.0	
CS	116.253	91.148	21.6		84.5660	29.2	Inconel 625
18CR-20	72.838	48.748	33.1	321	60.0091	59.8561	0.3
26-15	93.179	61.405	34.1	317 LM	29.2931	27.2168	7.1
Nitronic 50	22.228	23.423	Gain		18.5248	16.7	Carp. 20 Cb-3
Rod 3 (Bottom)							
Hast. C-276	65.6859				65.6955	Gain	
Hast. G	124.5123				124.5156	Gain	
Hast. G-3	121.4985				121.5054	Gain	
Titanium	10.361				10.3658	Gain	
2 RE 69	51.9177				50.6752	23	
317	116.0293				116.0280	0.0	
304	87.4242				82.9716	5.1	
410	110.9877				100.0503	9.9	Haynes 20 mod
Monit	25.4985				25.5023	Gain	
SC-1	28.1933				28.1995	Gain	
CS	116.5459				107.6225	7.7	Inconel 625
18CR-2MO	73.3482				73.3541	Gain	
26-15	92.4328				92.3484	0.1	
Nitronic 50	22.4523				22.3895	2.8	Incoloy 825

Table 31
Corrosion Probe Data Analysis

T105 Probe	Original O.D. (in.)	21 September 1979			28 December 1979		
		Indicated Loss (mils) Per Surface	Adjusted ^(a) Indication (mils)	Actual Tray Loss (mils)	Indicated Loss (mils) Per Surface	Adjusted ^(a) Indication (mils)	Actual Tray Loss (mils)
#1 (CS)	0.0397	0.4	0.4	-	1.1	1.1	0-5 (321 tray) (1010 probe)
#9 (321SS)	0.0396	2.7	11.2	15 ^(b)	4.8	13.3	15-25 ^(b)
#15 (321SS)	0.0397	4.8	7.6	15-20	9.1	11.9	-

(a) Tray 9 had six more days at 515 mpy than did the probe. Tray 15 had three more days at 339 mpy than did the probe.

(b) Tray 10 data used. (Tray 9 not accessible).

Table 32
Screen Performance in U. S. Filter

	<u>Leaf Description</u>	<u>Run Days</u>	<u>Filter Inspection/Correction</u>	<u>Sluice</u>
		Precoat:	Dicalite Perlite 436	
1.	Run 173, 24 x 110 calendared 3-ply	1	Leaves rubbing on sluice arm. Rollers added to guide leaves.	1,500 psig each cycle 4006 nozzles
2.	Run 173 3-ply repaired	1	Acid paste damage	1,500 each cycle
3.	Runs 175-177 2-ply, No. B1, B2, B4	7	Erratic clarity, cut-off blade ring seized. Lengthened blade stops. Installed spacer rings between blade guide rings.	1,500 each cycle
4.	Runs 178-179 2-ply, No. B1, B2, B4	8	Erratic clarity, nothing wrong on external inspection.	1,500 each cycle
5.	Run 179 3-ply + rescreened 3-ply, No. C1	1	Acid paste or diffusion bonding sensitization.	1,500 each cycle
6.	Run 179 2-ply, No. B1, B2, B4	1	Spacing ring wedged.	1,000 each cycle
7.	Runs 179-182 3-ply, No. C1 2-ply, No. B3, B4	15	Poor initial clarity. Sluicing each cycle stopped. Holes in leaf periphery. Cut-off blades broken. Spacing rings were removed.	1,000 psig each 25 cycles
8.	Runs 183-184 2-ply w/perforated sheet 2-ply, No. B1, B2	3	Poor clarity. Screens on 2-ply leaves disintegrated in spots.	1,000 psig each 25 cycles
9.	Runs 185-188 3-ply, No. C1 2-ply w/perforated sheet 2-ply, No. B4	14	Hole in leaf No. B4 from cut-off.	1,000 psig each 25 cycles
10.	Runs 188-189 non-diffusion-bonded leaves, No. D1, D2, D3	3	Leaking gasket.	1,200 psig each cycle

(Table continued)

Table 32 (continued)
Screen Performance in U. S. Filter

	<u>Leaf Description</u>	<u>Run Days</u>	<u>Filter Inspection/Correction</u>	<u>Sluice</u>
		Precoat:	Dicalite Perlite 436	
11.	Run 190 Non-diffusion-bonded leaves, No. D1, D2, D3	8	Good precoat operation.	1,200 psig four times
	Run 191	3	No precoat operation. Holes in screens where spot welding bonded only five wires. Screen silver soldered.	1,500 psig seven times
12.	Run 192 3-ply, No. C1 2-ply w/perforated sheet, 2-ply, No. 3	2	Holes in B3. Diffusion-bonded sensitization.	600-1,500 each cycle with 6510 nozzle
	Run 193 3-ply, No. C1 2-ply w/perforated sheet non-bonded, No. D2	2	No precoat operation. Poor clarity.	1,000 psig first 8 cycles with 6610 nozzles
		3	Precoat operation. Hole in leaf No. D2, Leaf D2 repaired.	Not sluiced.

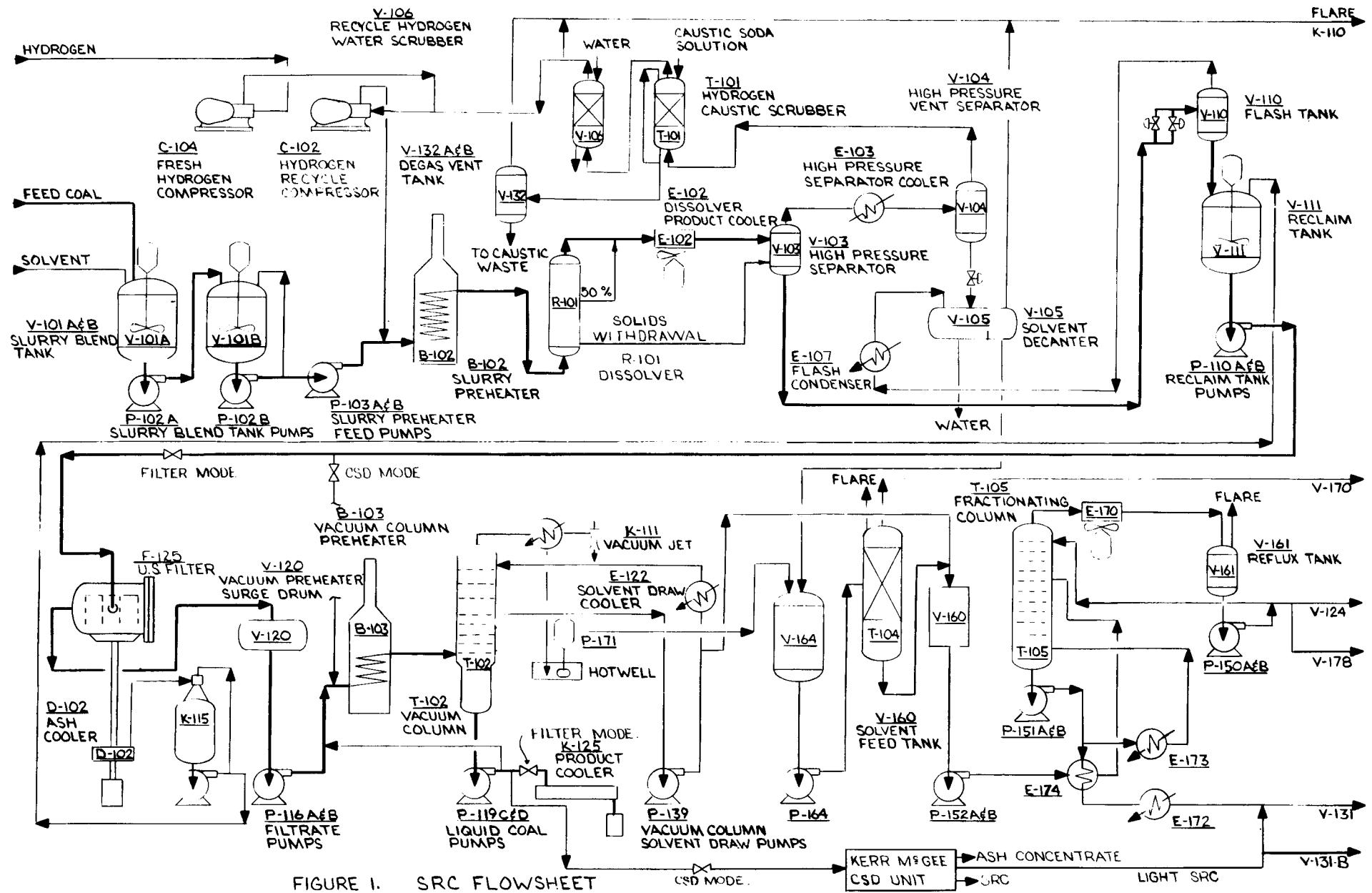
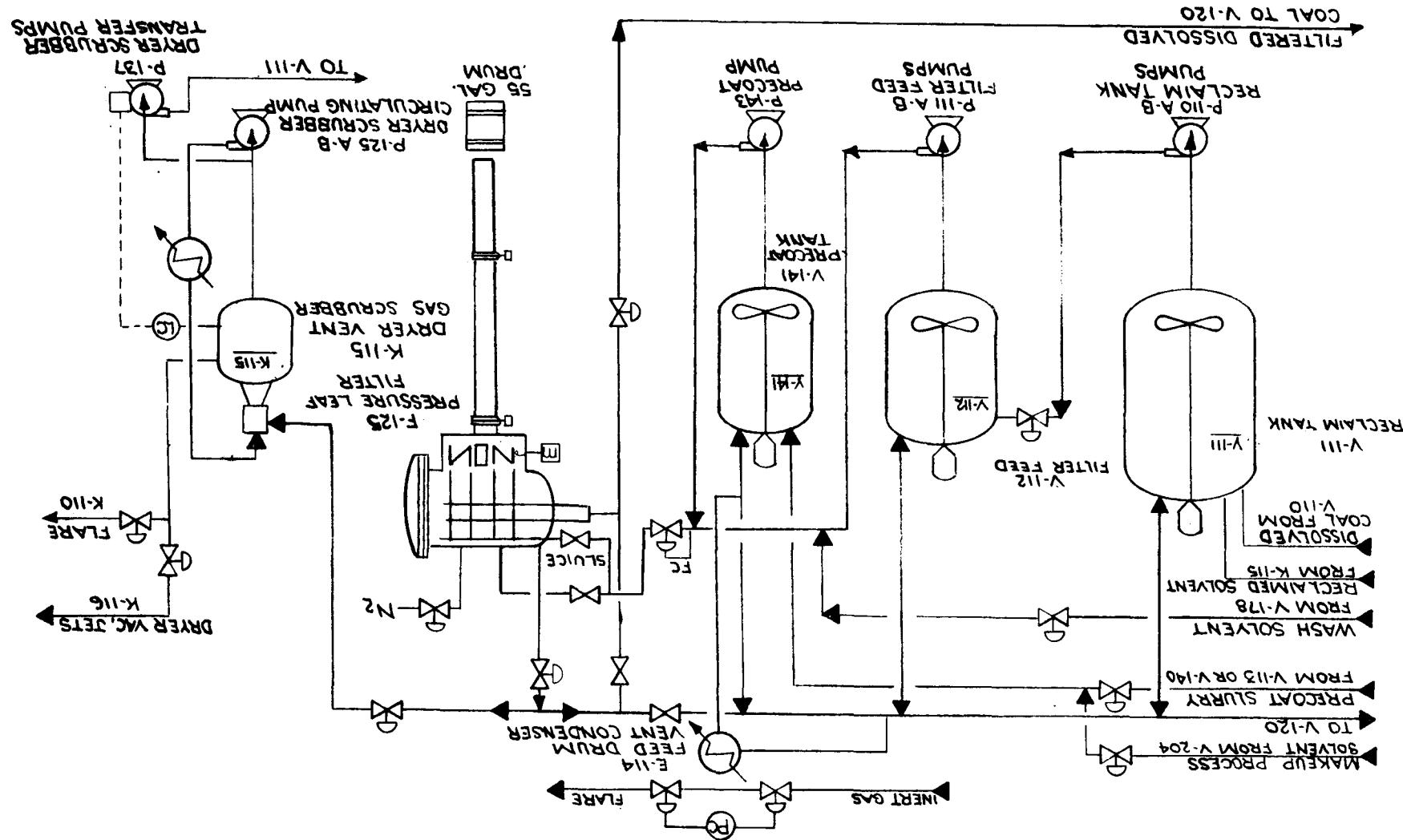


FIGURE 2. FILTRATION FLOWSHEET (U.S. FILTER)



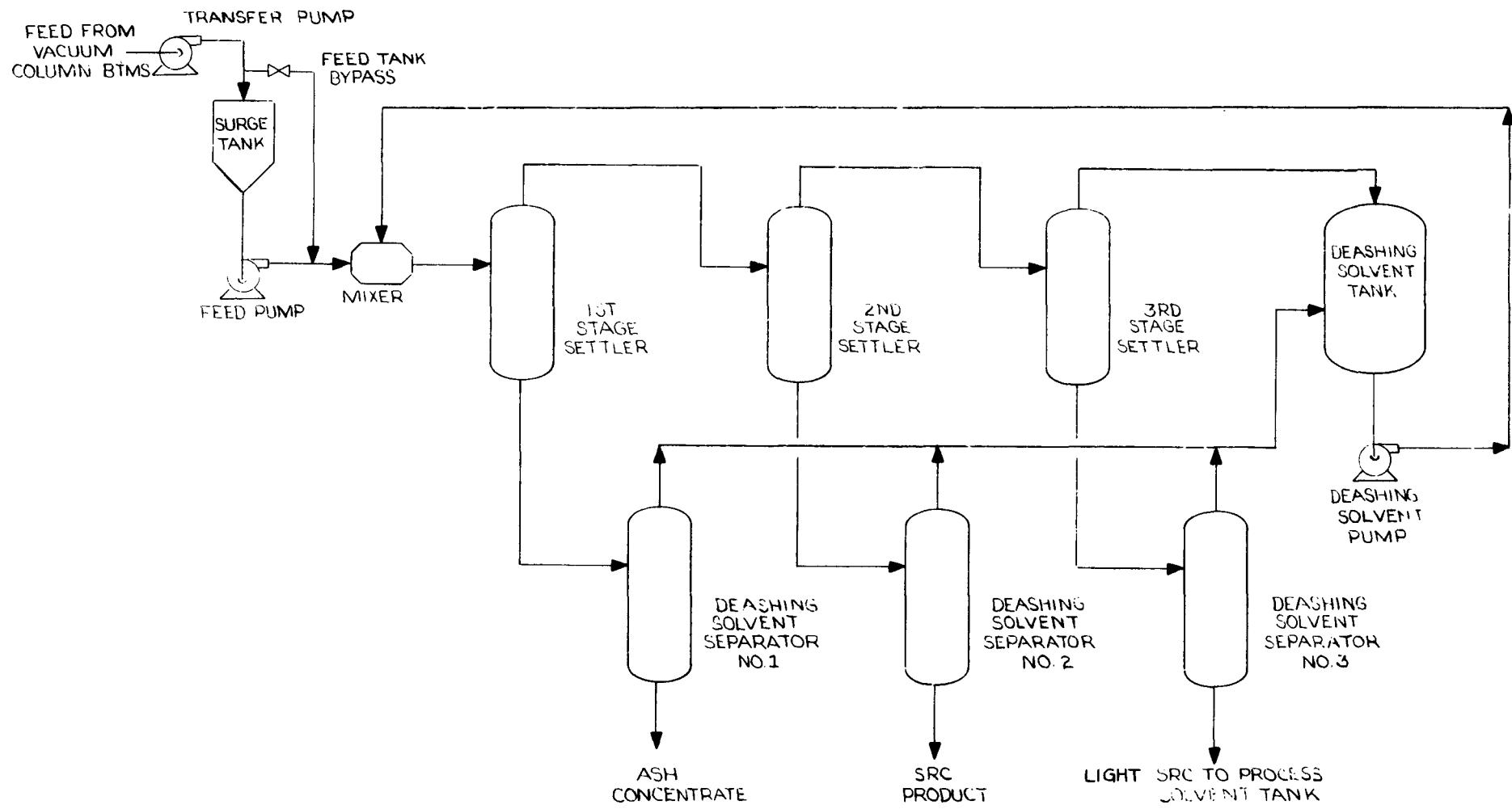


FIGURE 3. CRITICAL SOLVENT DEASHING PROCESS FLOW DIAGRAM

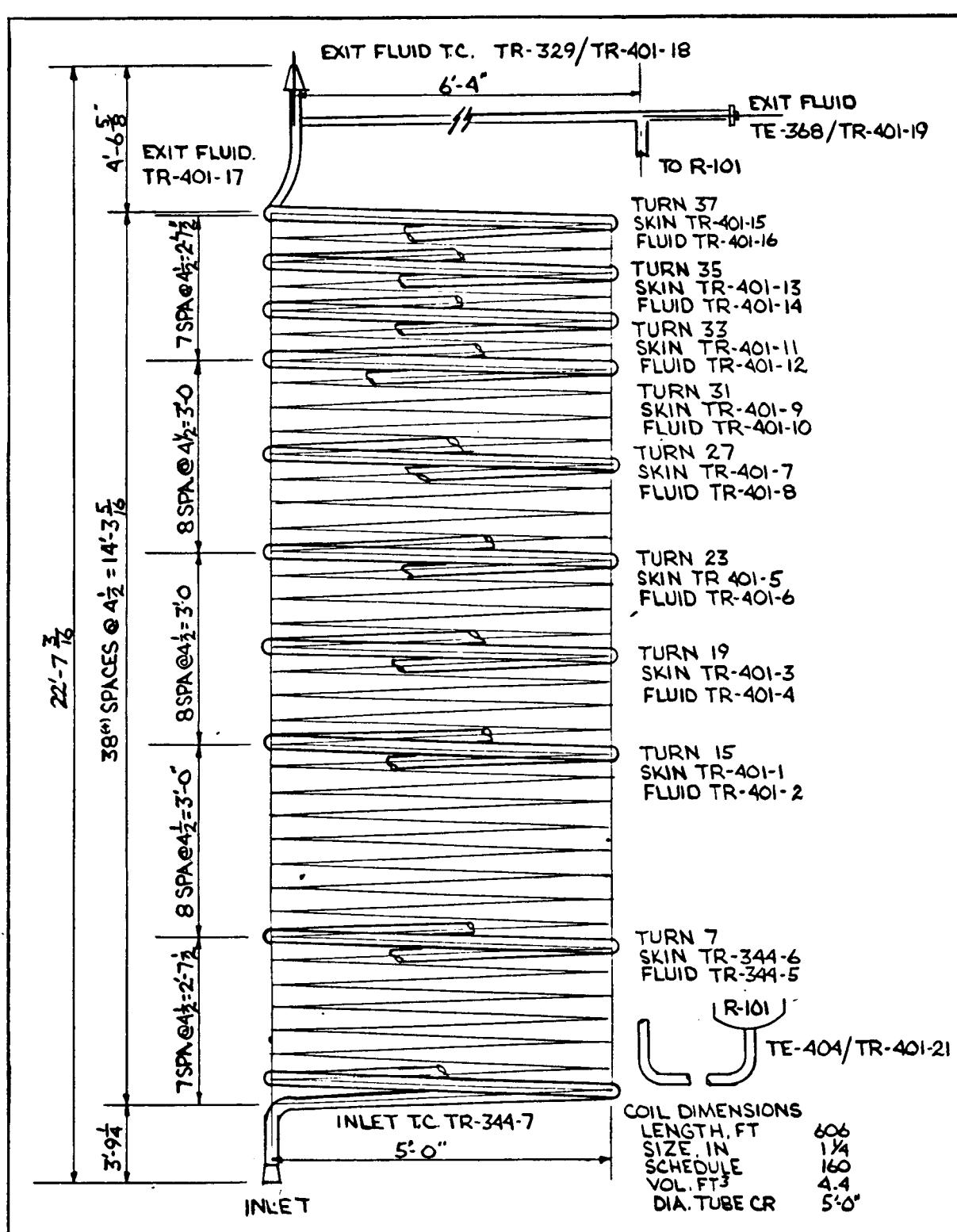


FIGURE 4. B-102 SLURRY PREHEATER THERMOCOUPLE LOCATIONS

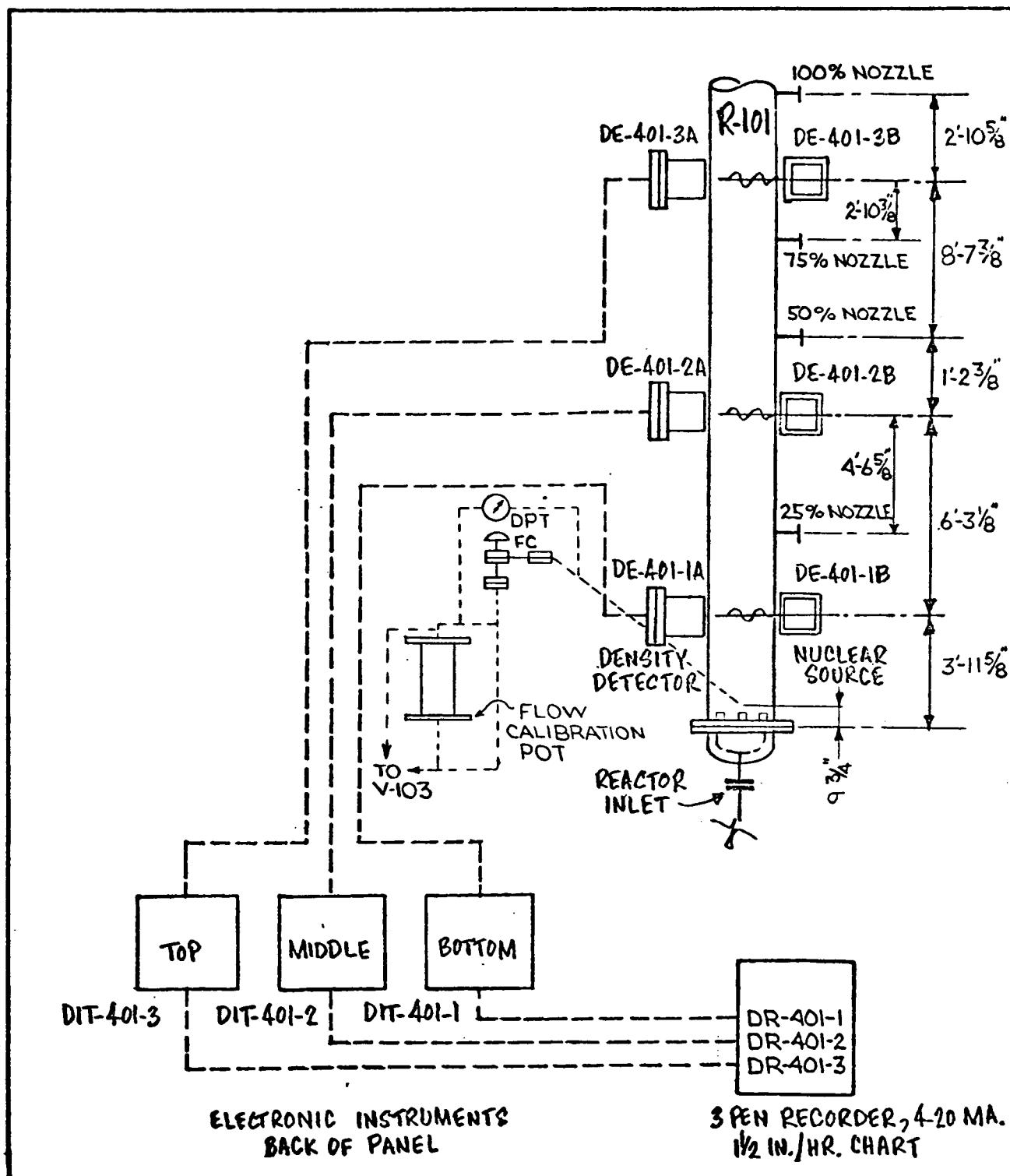


FIGURE 5. DISSOLVER SOLIDS MEASUREMENT AND CONTROL SYSTEM

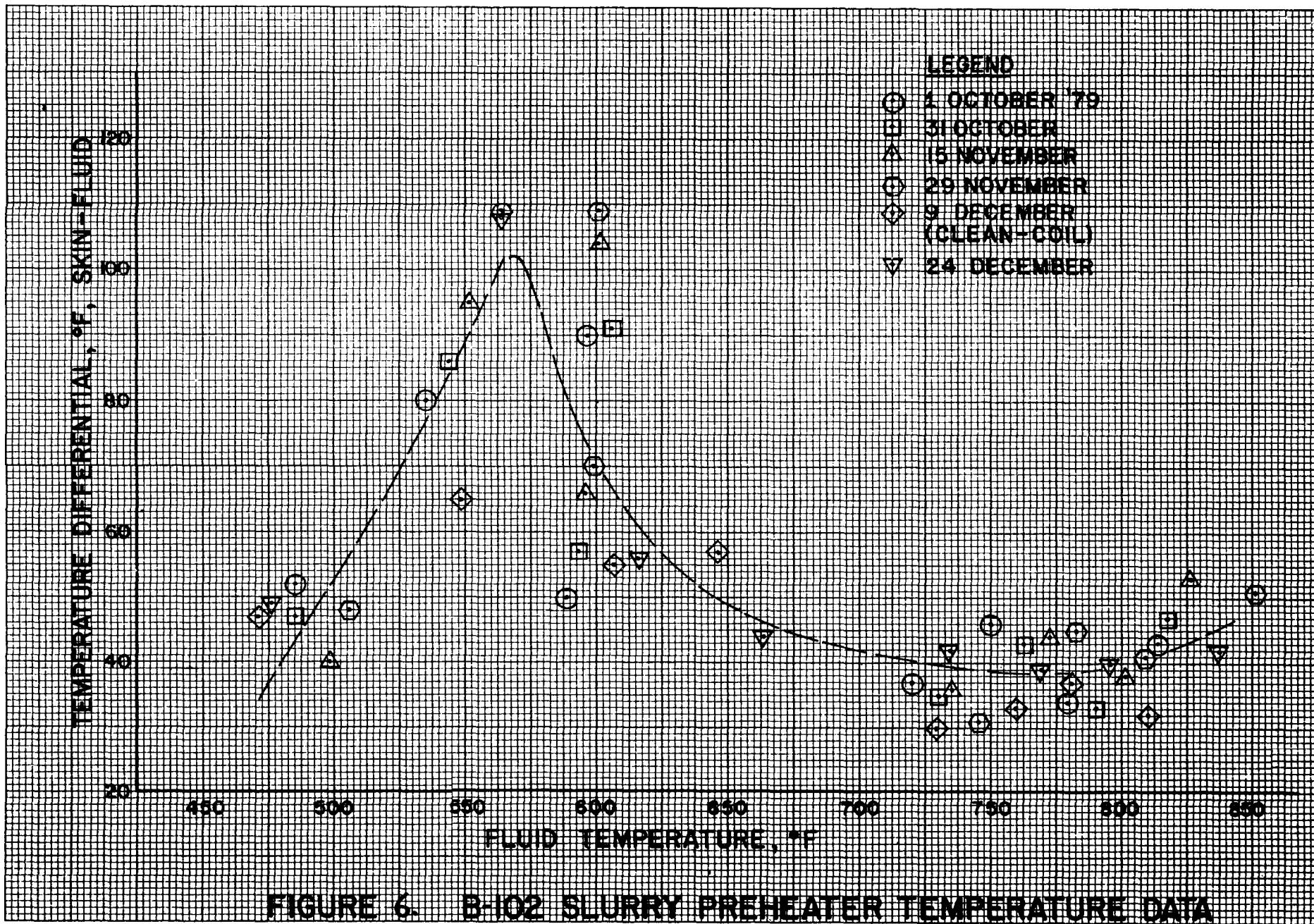


FIGURE 6. B-102 SLURRY PREHEATER TEMPERATURE DATA

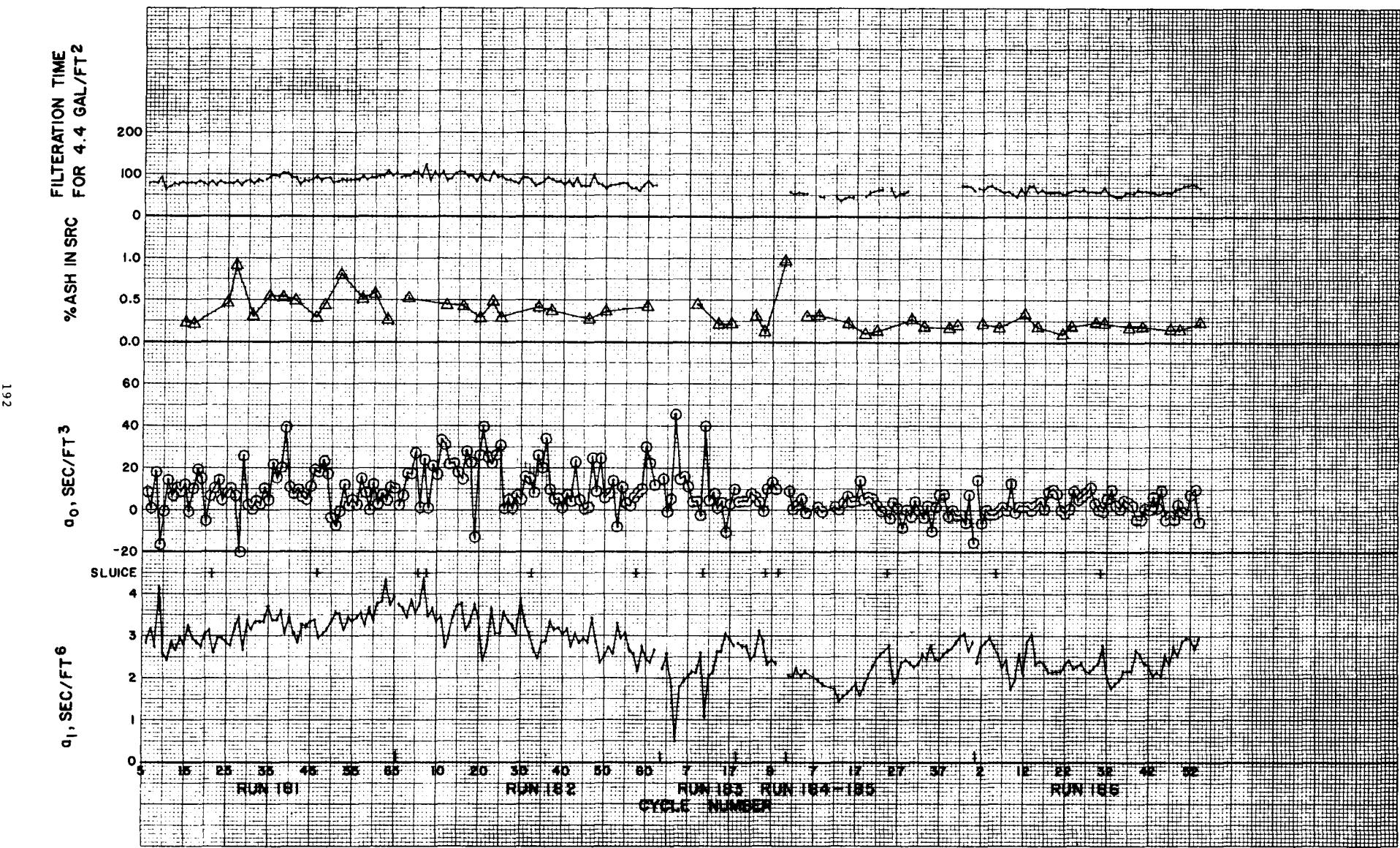


FIGURE 7. FILTER CYCLE DATA FOR NOVEMBER 1979

E6T

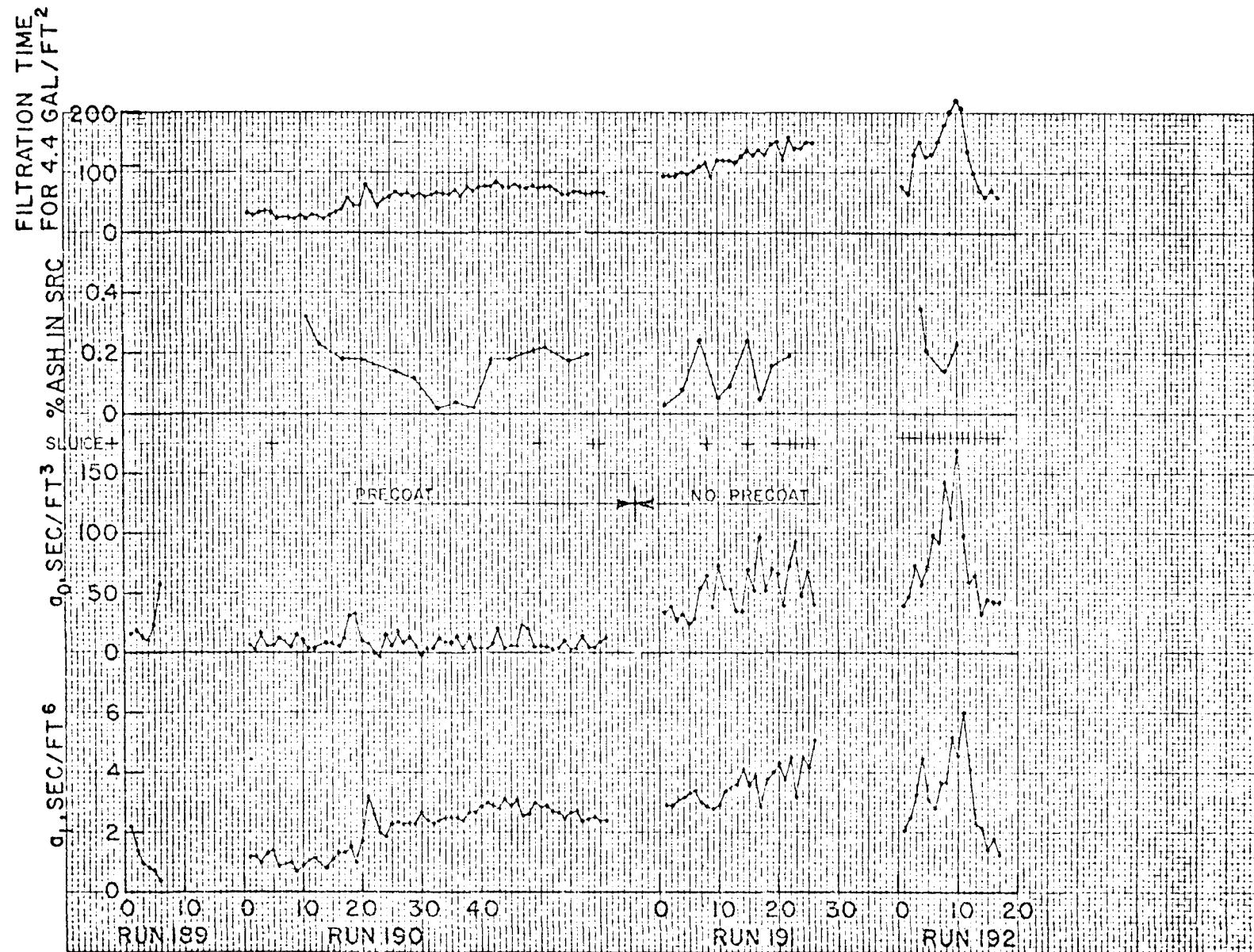


FIGURE 8. FILTER CYCLE DATA FOR DECEMBER 1979

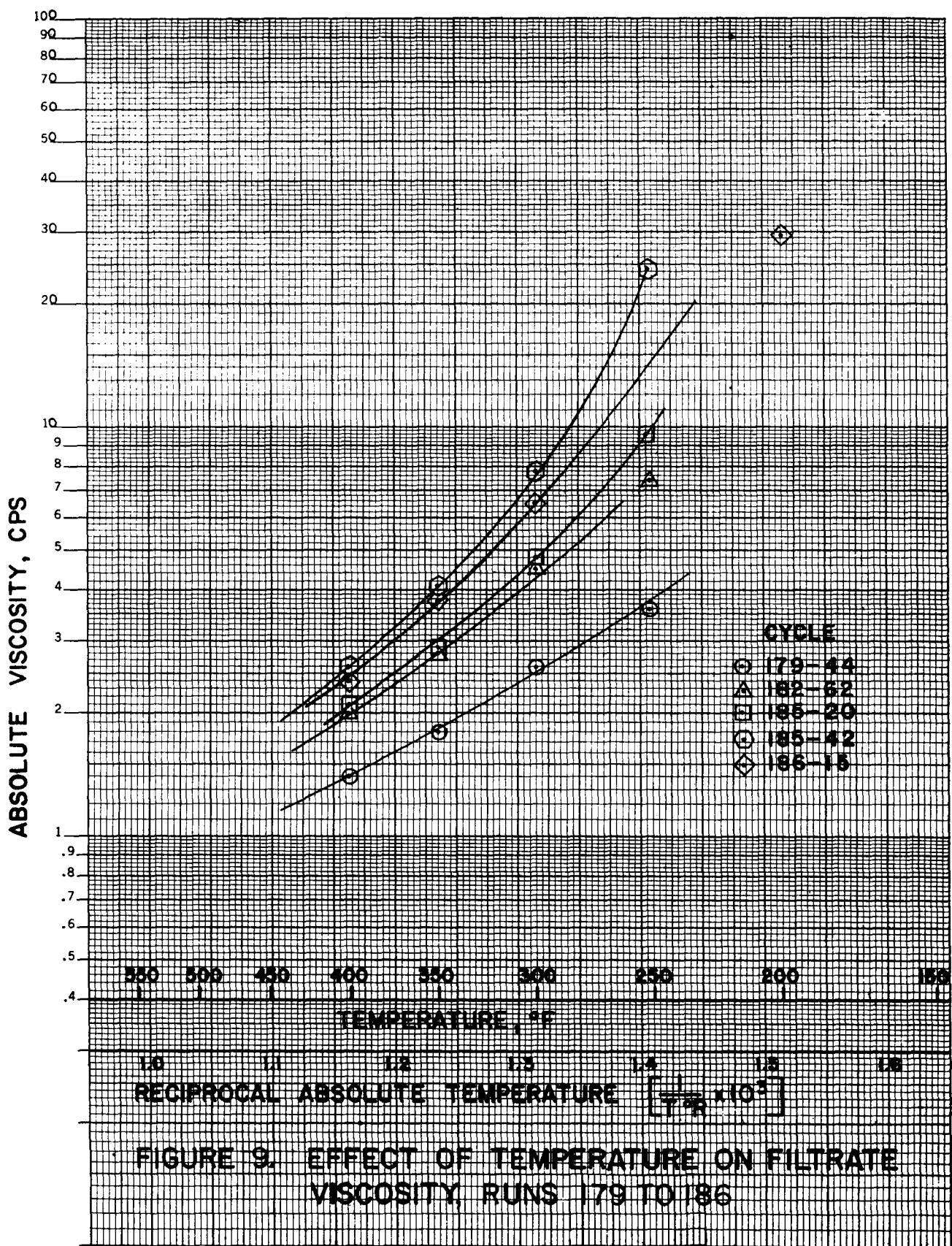
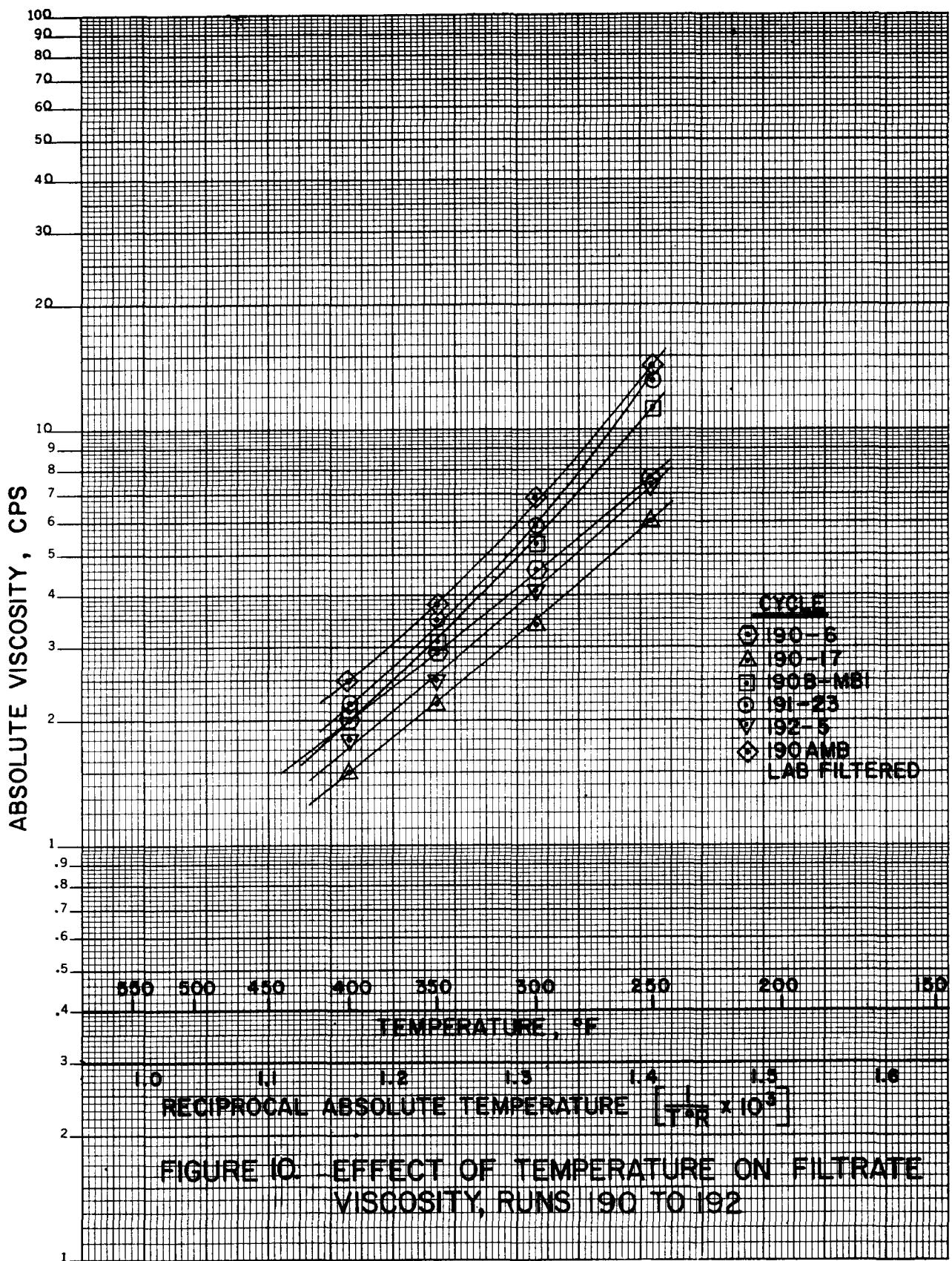


FIGURE 9. EFFECT OF TEMPERATURE ON FILTRATE VISCOSITY, RUNS 79 TO 86



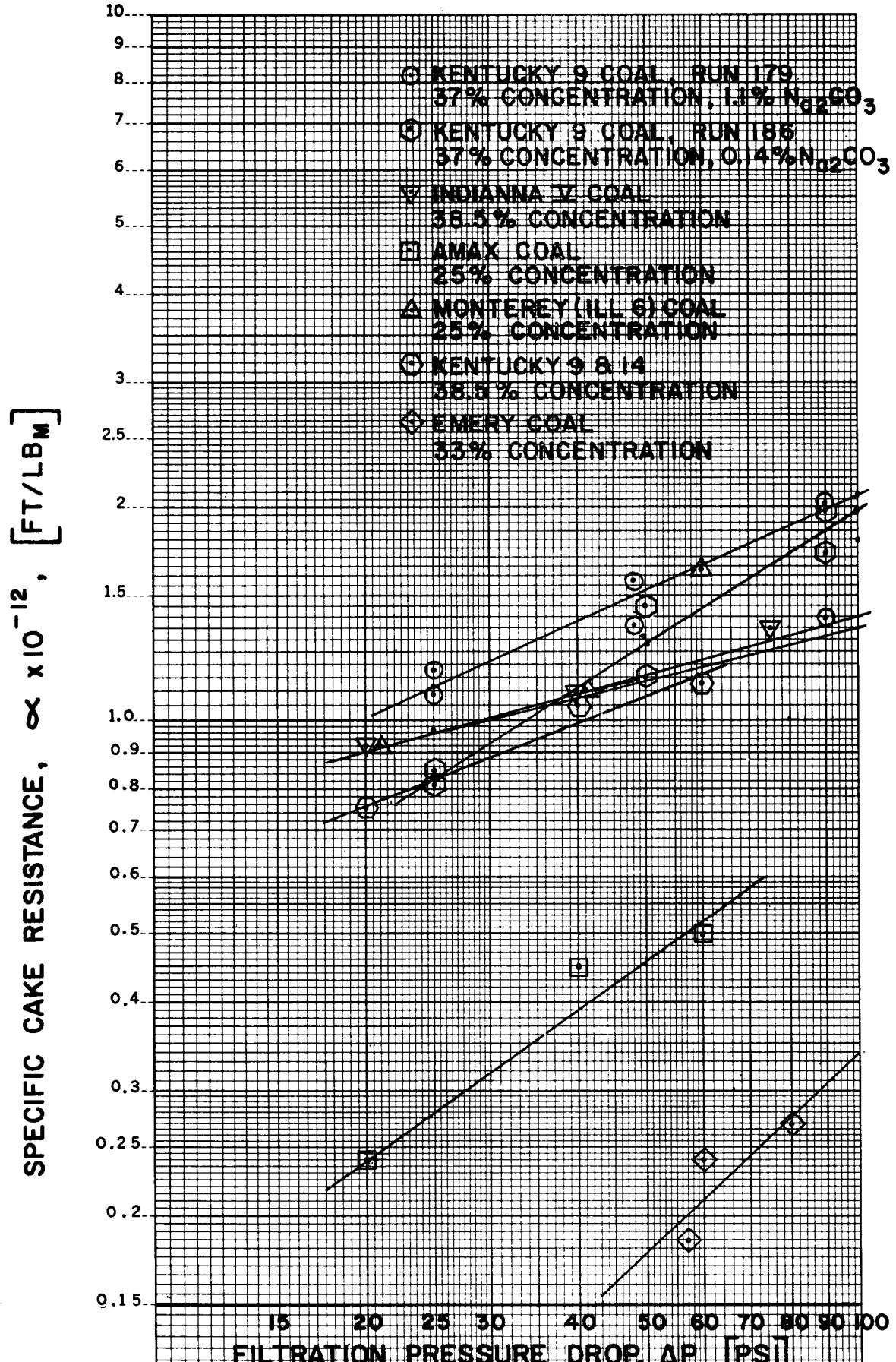


FIGURE II. SPECIFIC CAKE RESISTANCE VS FILTRATION PRESSURE DROP

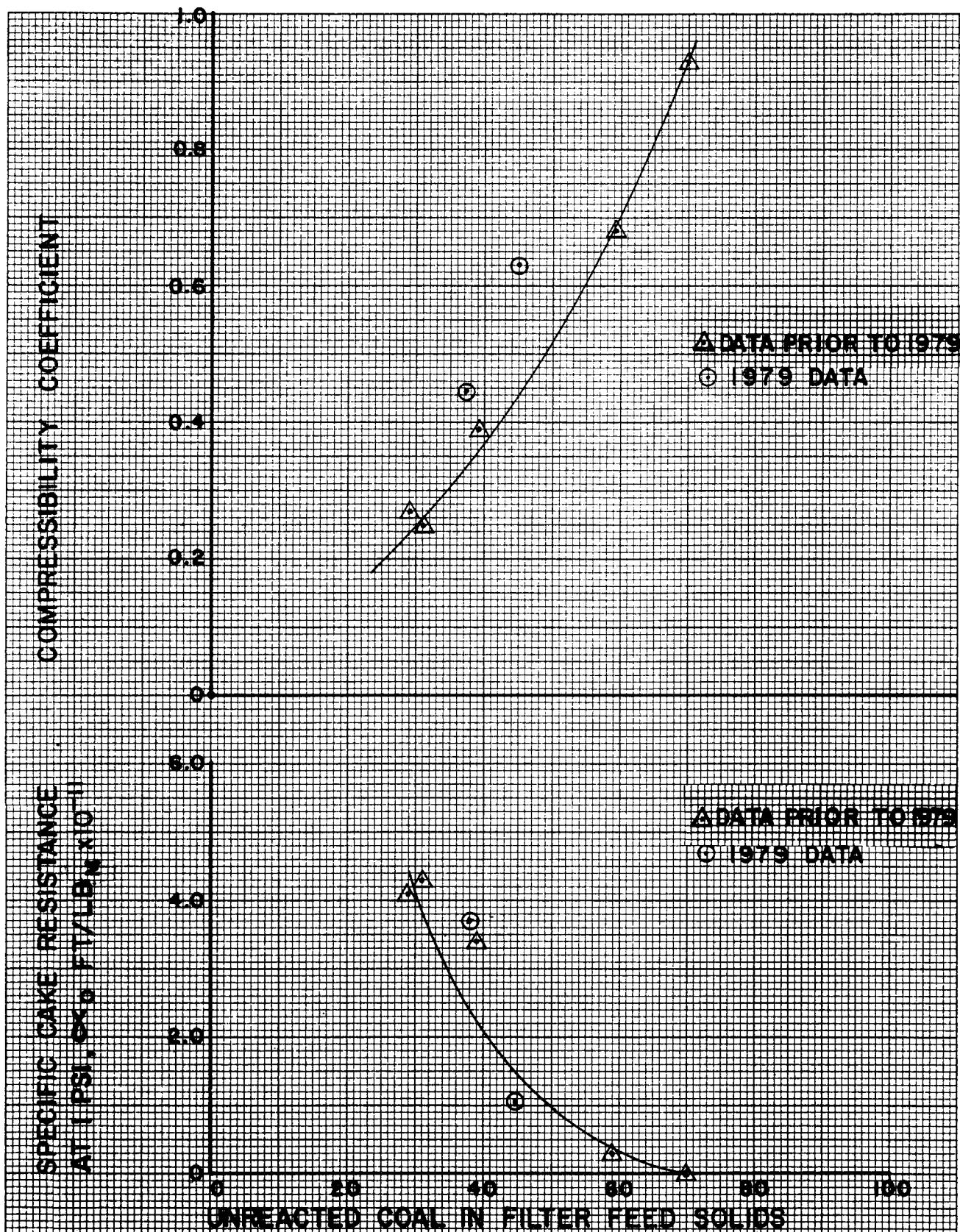


FIGURE 12. EFFECT OF FILTER FEED COMPOSITION ON FILTRATION MODEL PARAMETERS

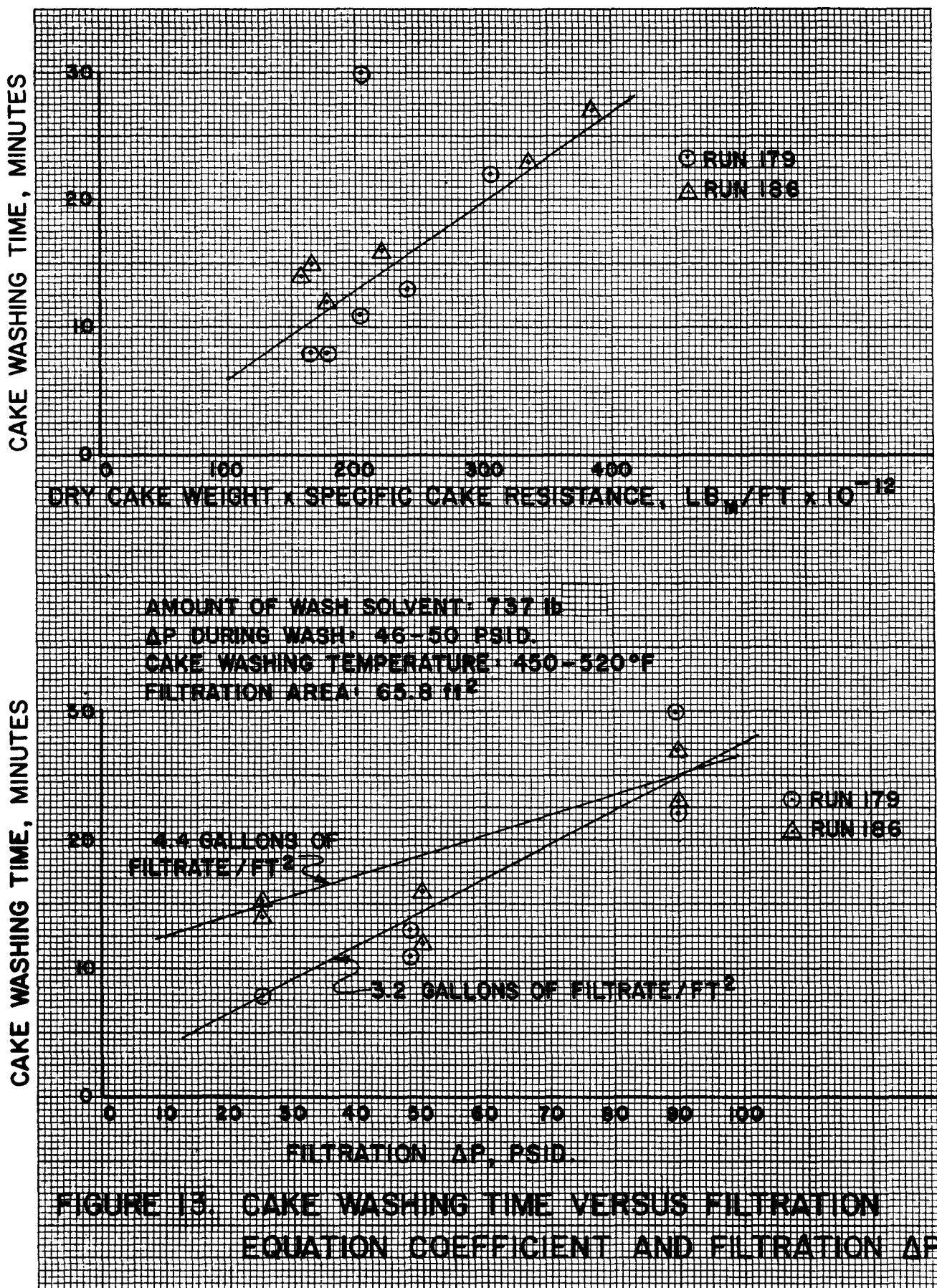


FIGURE 13. CAKE WASHING TIME VERSUS FILTRATION EQUATION COEFFICIENT AND FILTRATION AP

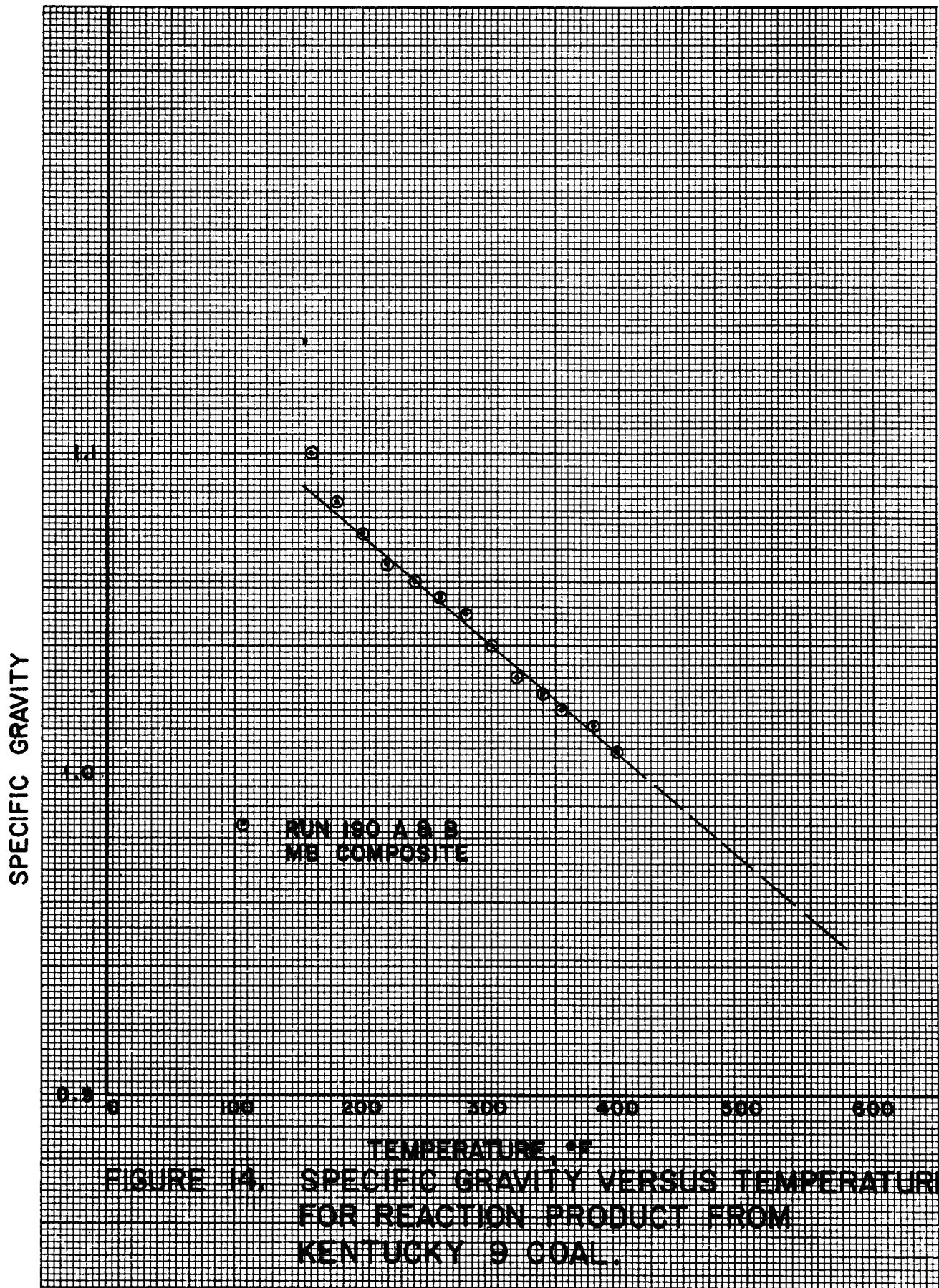


FIGURE 14. SPECIFIC GRAVITY VERSUS TEMPERATURE FOR REACTION PRODUCT FROM KENTUCKY #9 COAL.

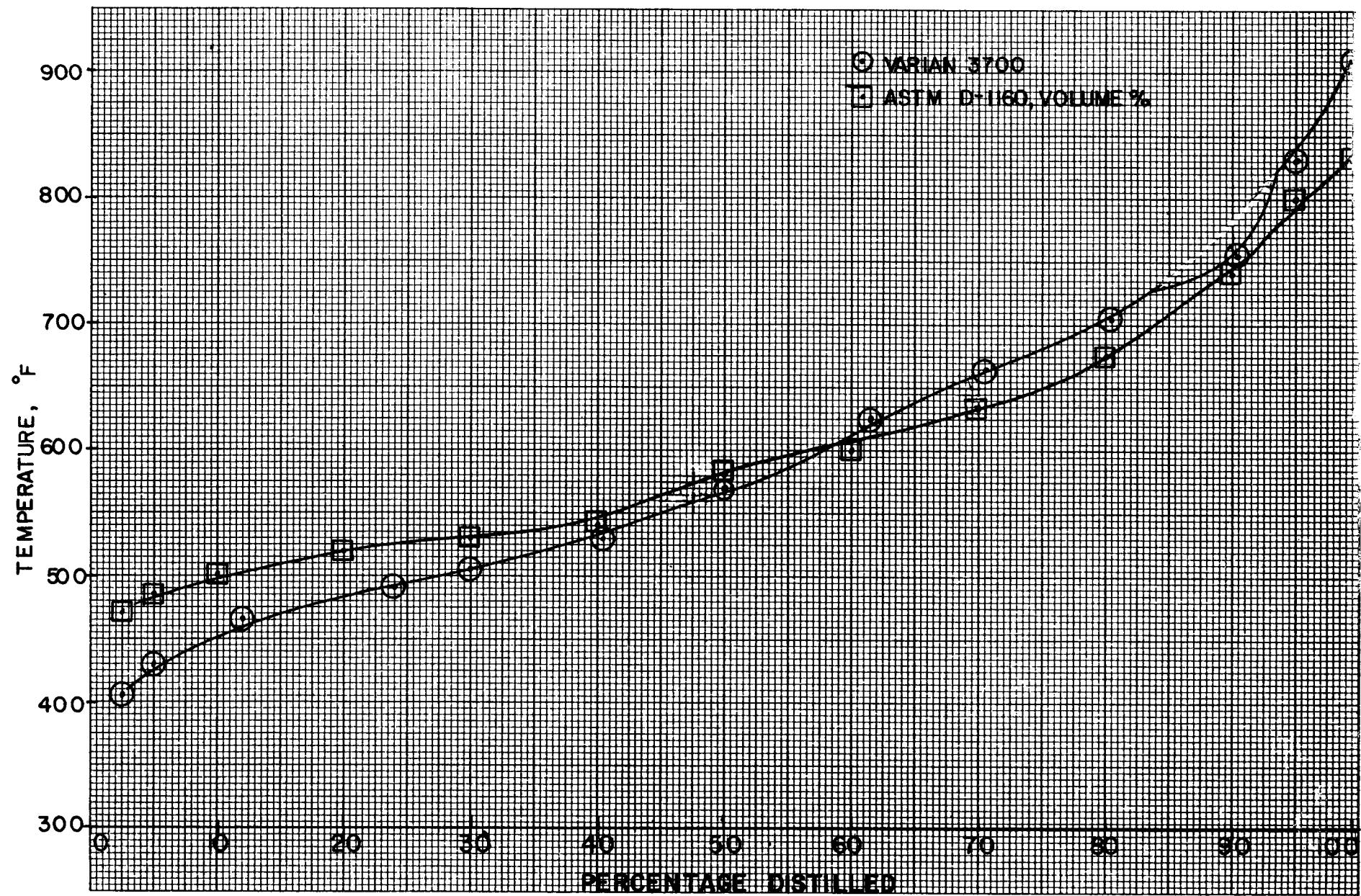


FIGURE 15. PROCESS SOLVENT DISTILLATION CURVES, RUN 171 AB-MB

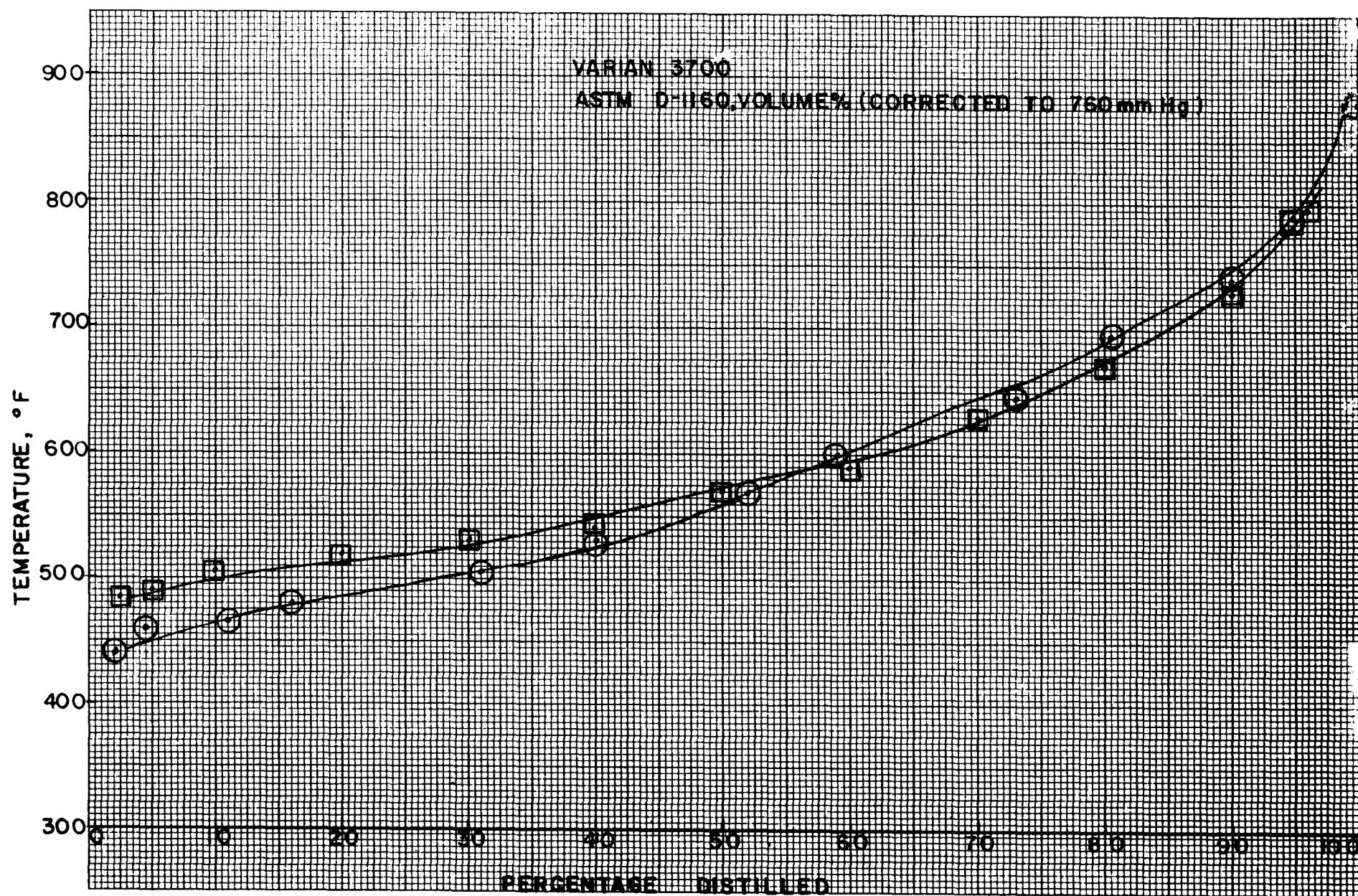


FIGURE 16. PROCESS SOLVENT DISTILLATION CURVES, RUN 172 A-MB

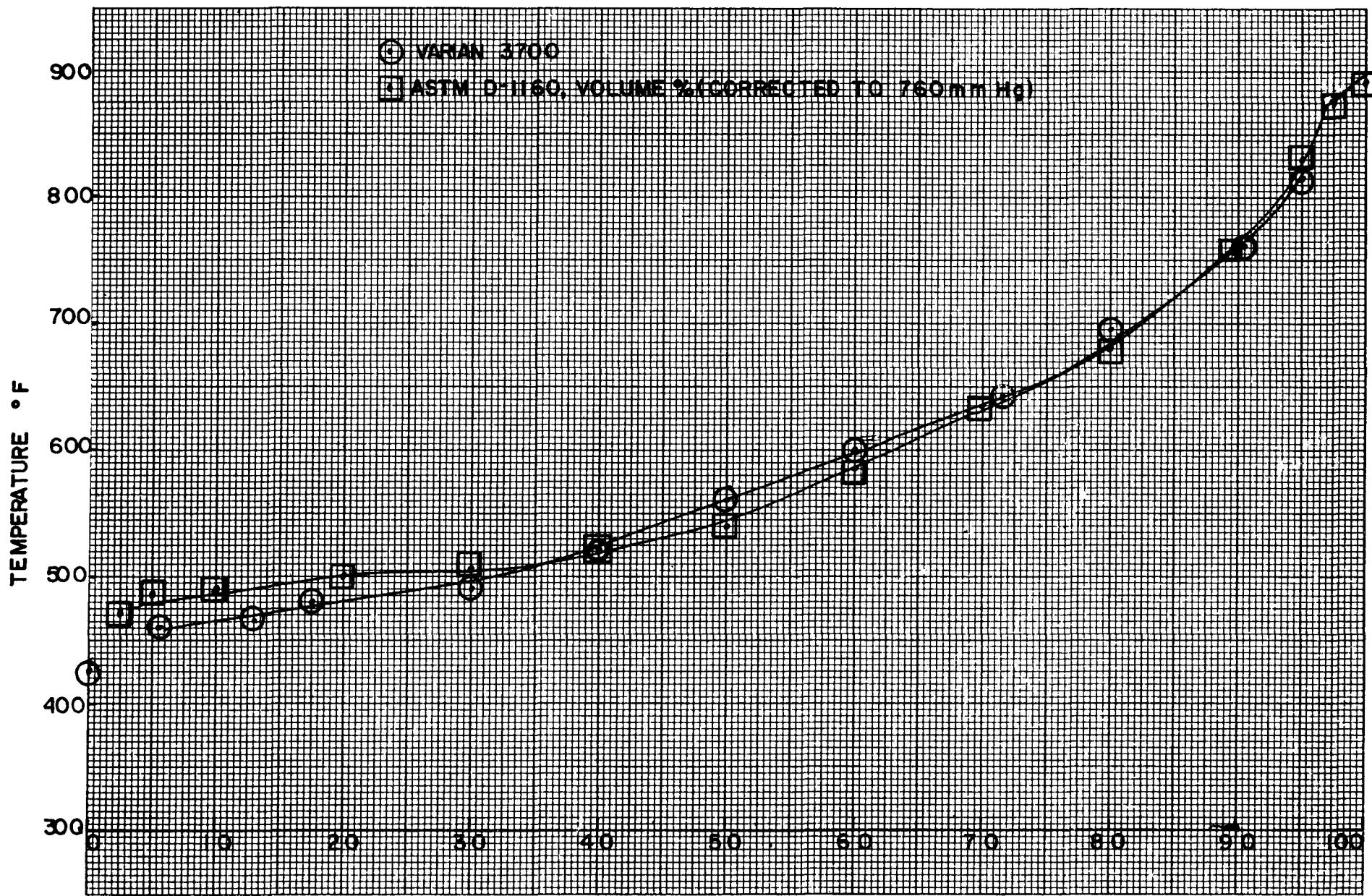


FIGURE 17. PROCESS SOLVENT DISTILLATION CURVES, RUN 190 AB-MB
BY ASTM D-1160 METHOD

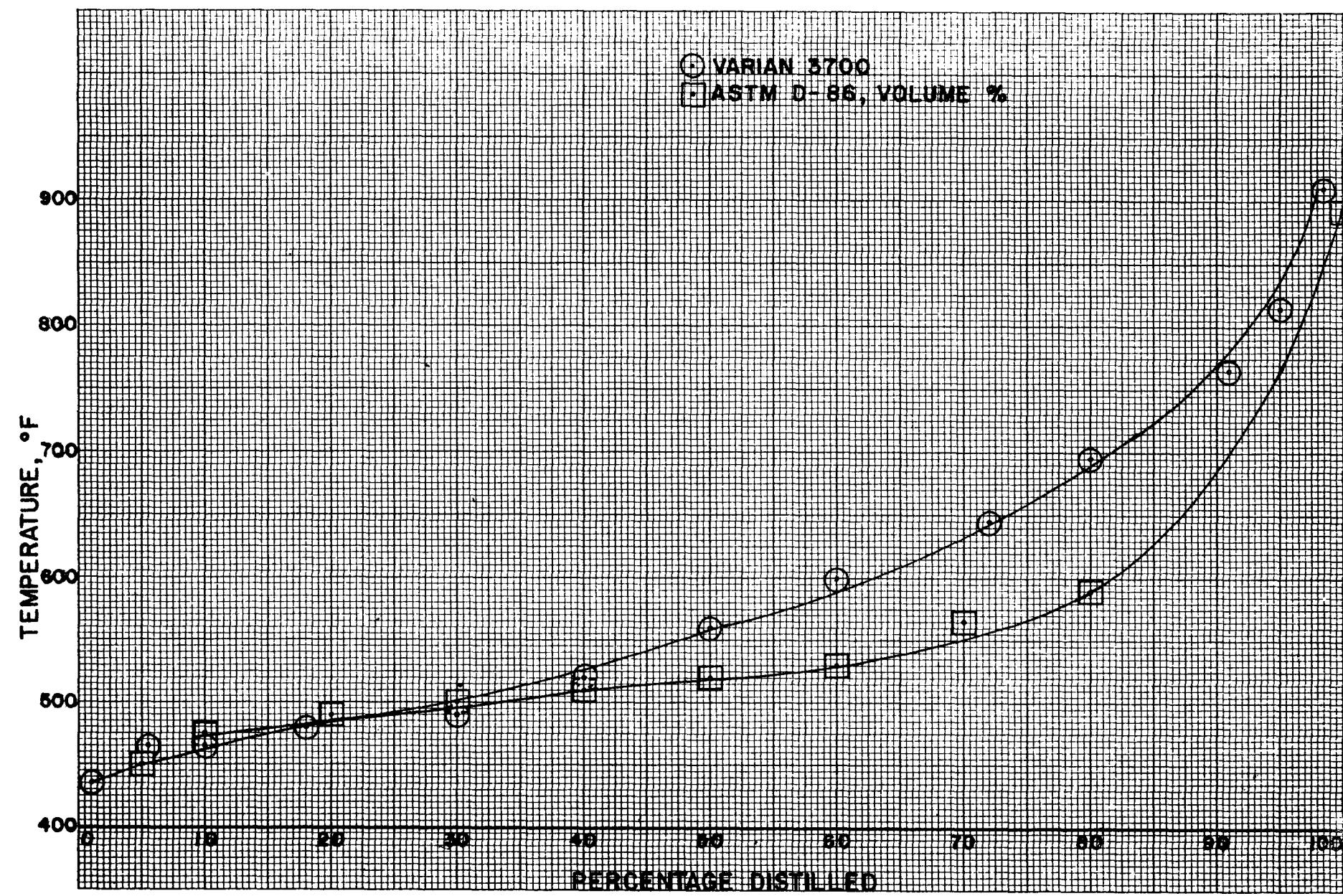


FIGURE 18. PROCESS SOLVENT DISTILLATION CURVES, RUN 190 AB-MB
BY ASTM D-86 METHOD

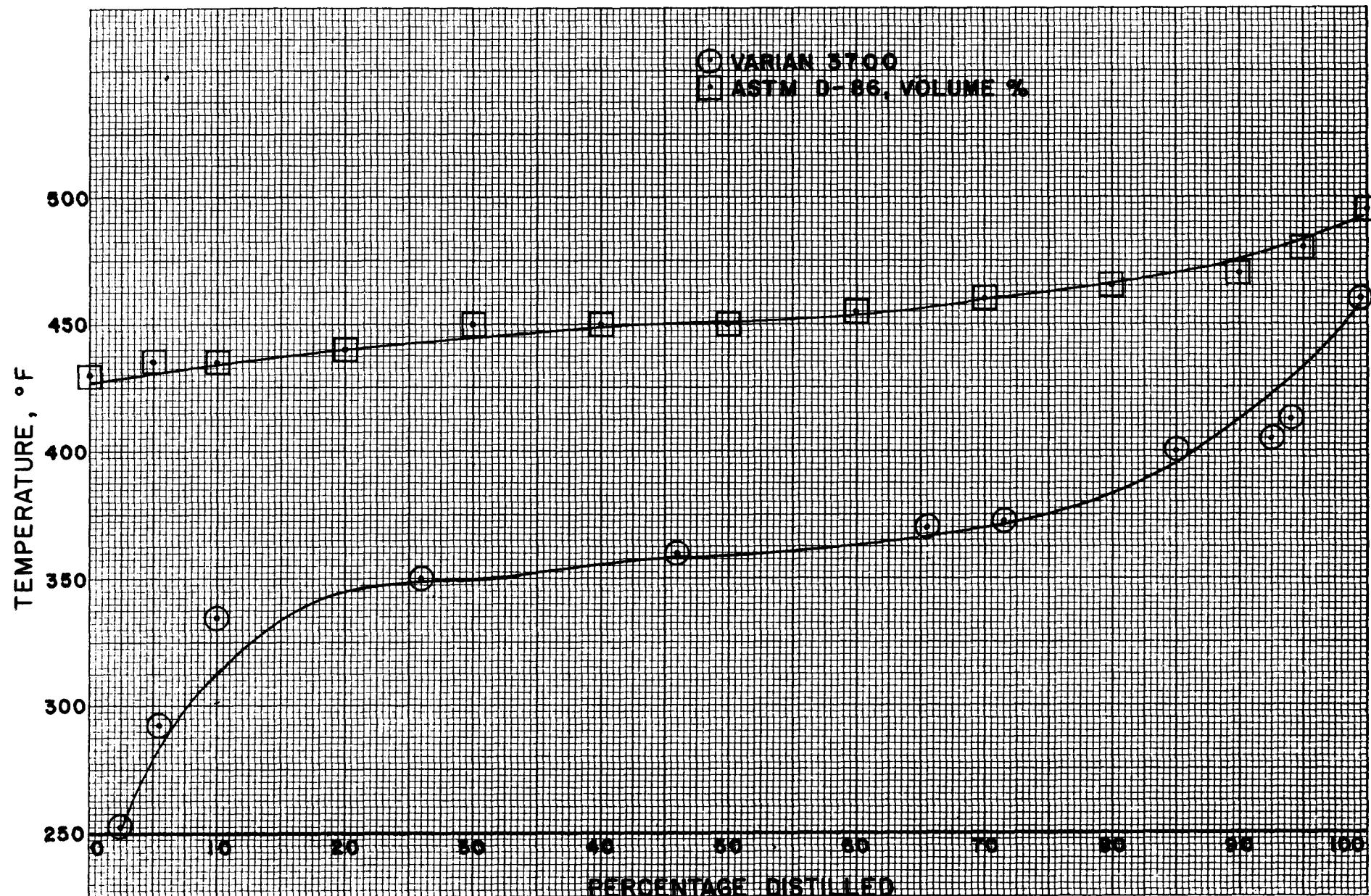


FIGURE 19. WASH SOLVENT DISTILLATION CURVES, RUN 171 A-MB

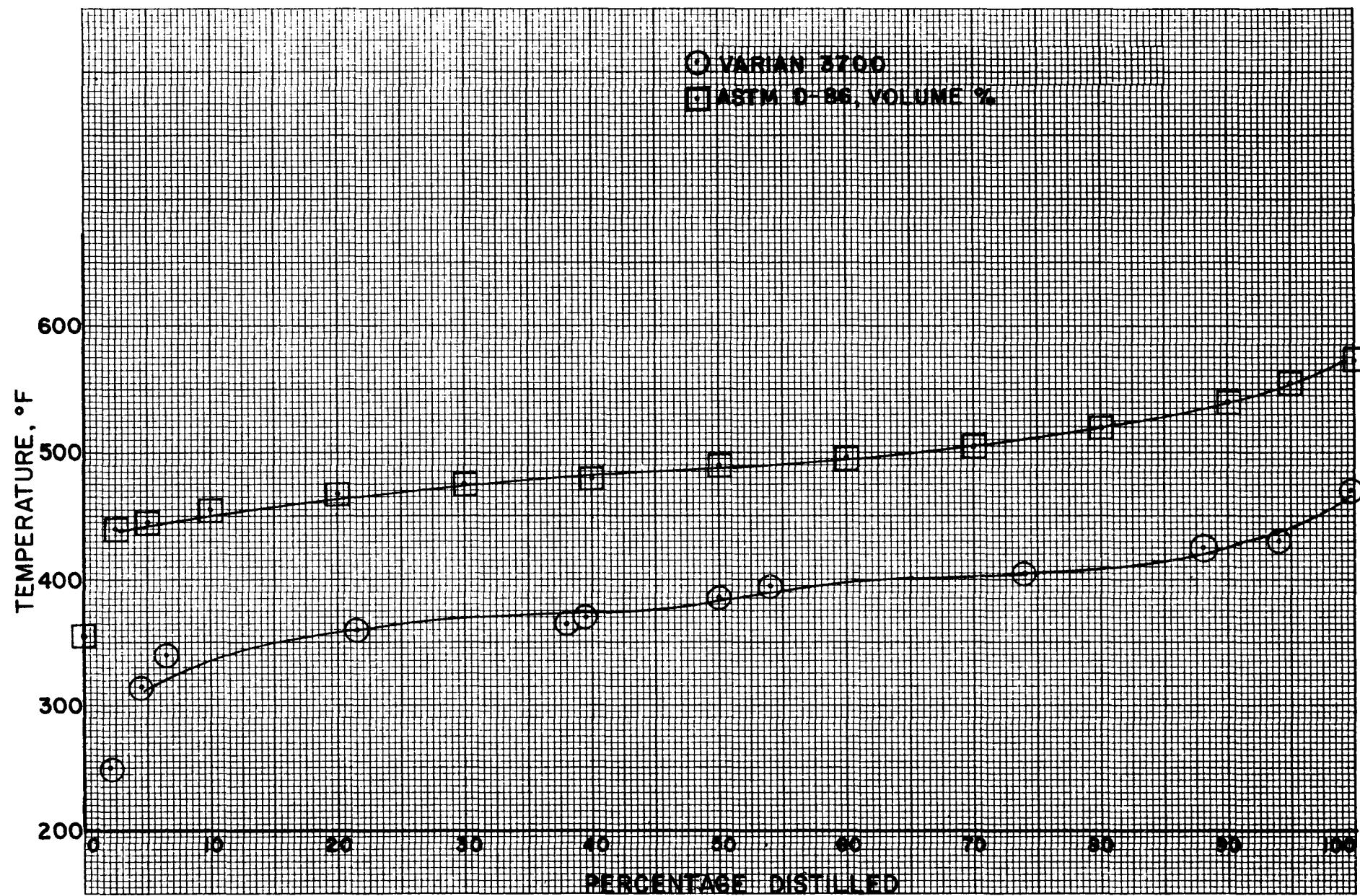


FIGURE 20. WASH SOLVENT DISTILLATION CURVES, RUN 172 A-MB

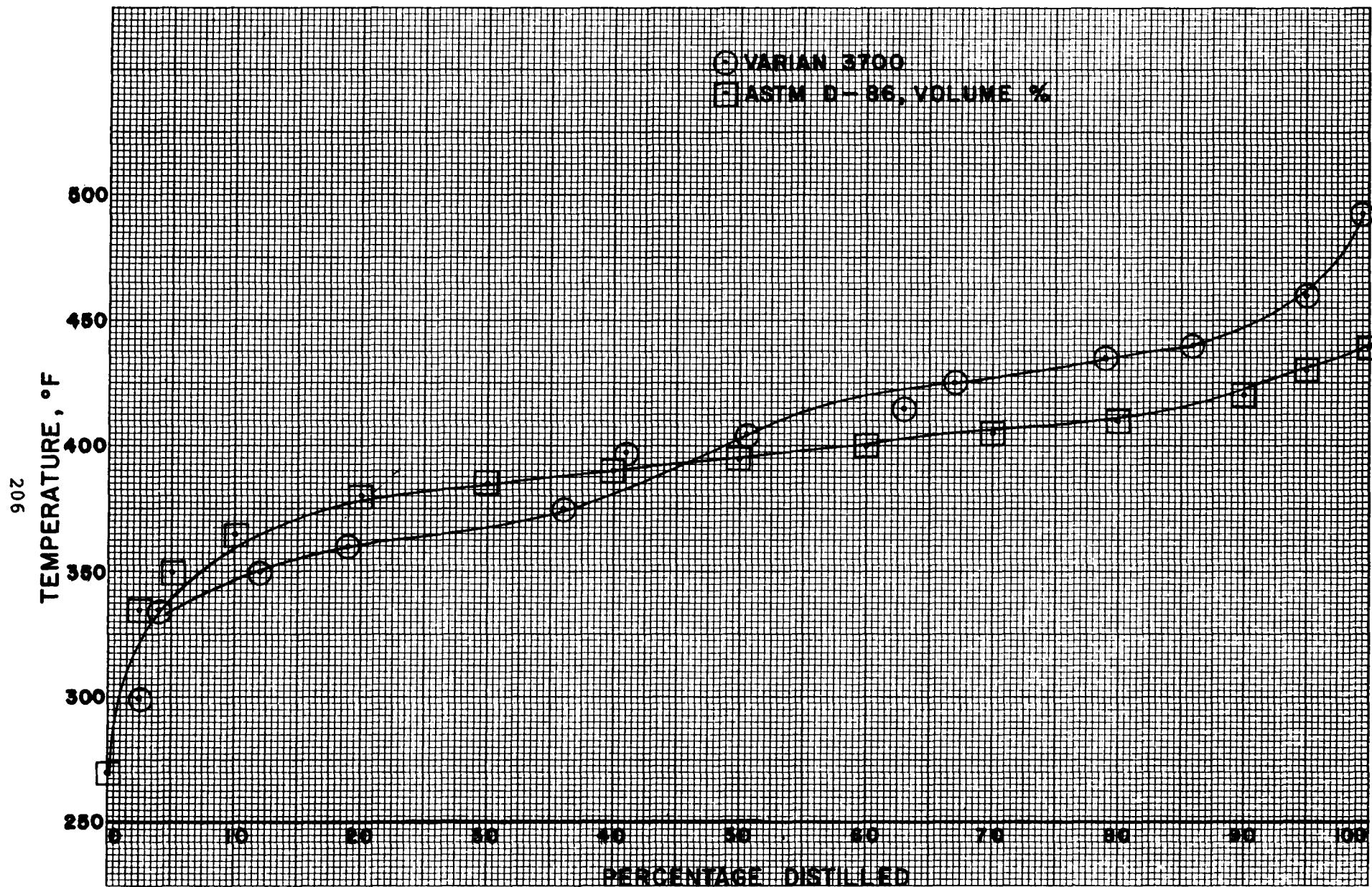


FIGURE 21. WASH SOLVENT DISTILLATION CURVES, RUN 190 AB-MB

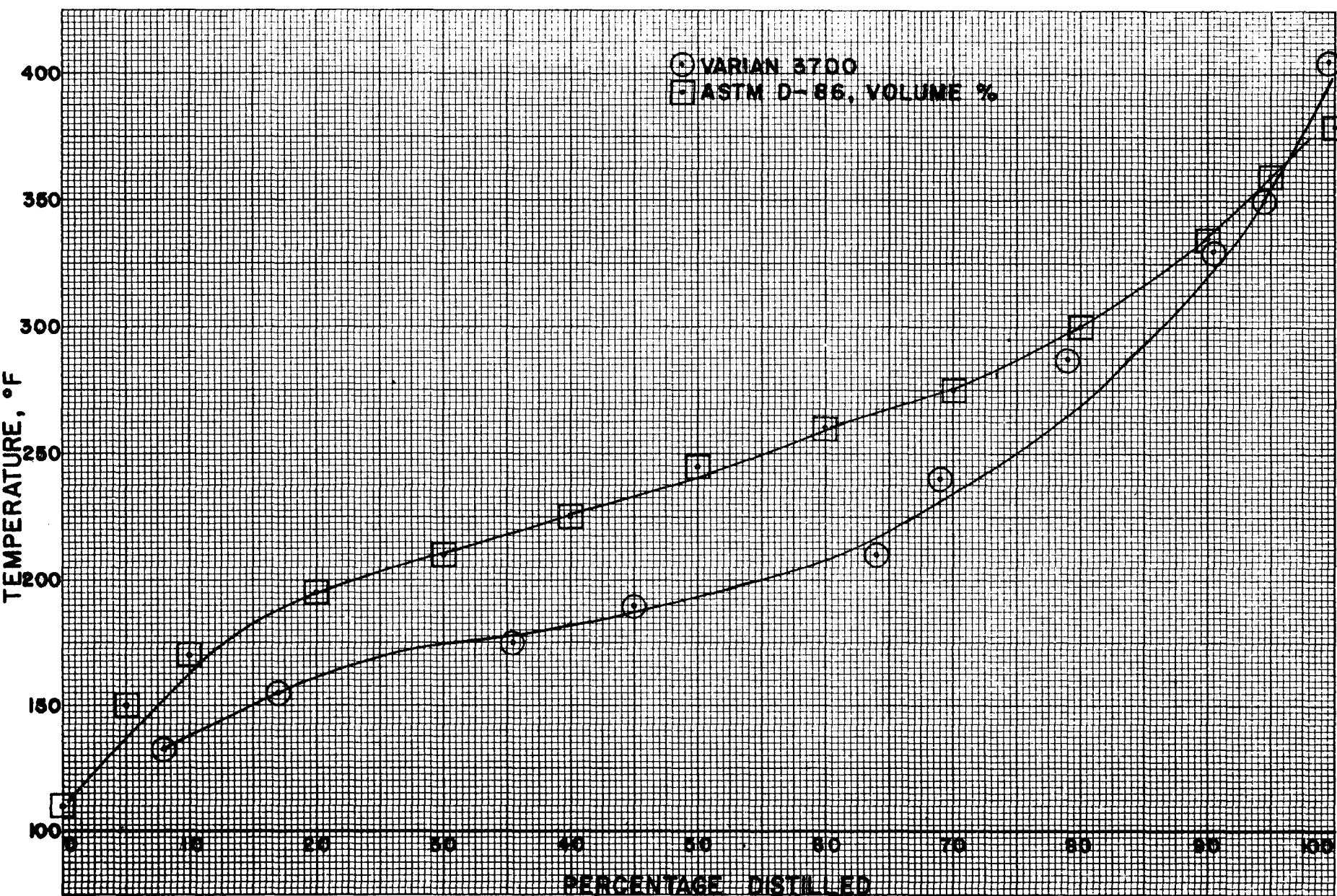


FIGURE 22. LIGHT ORGANIC LIQUID PRODUCT DISTILLATION CURVES, RUN 171A-MB

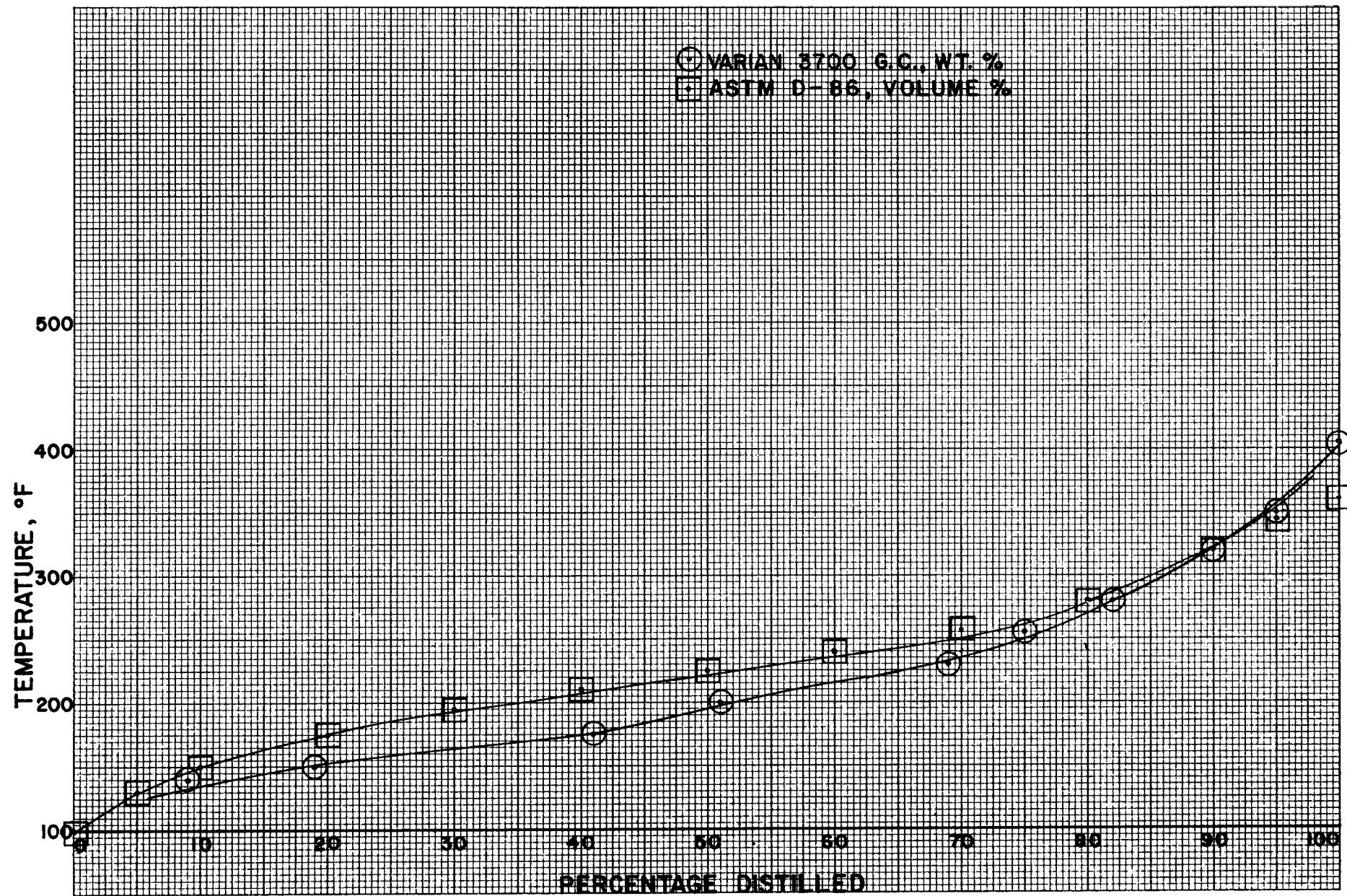


FIGURE 23. LIGHT ORGANIC LIQUID PRODUCT DISTILLATION CURVES, RUN 172 A-MB

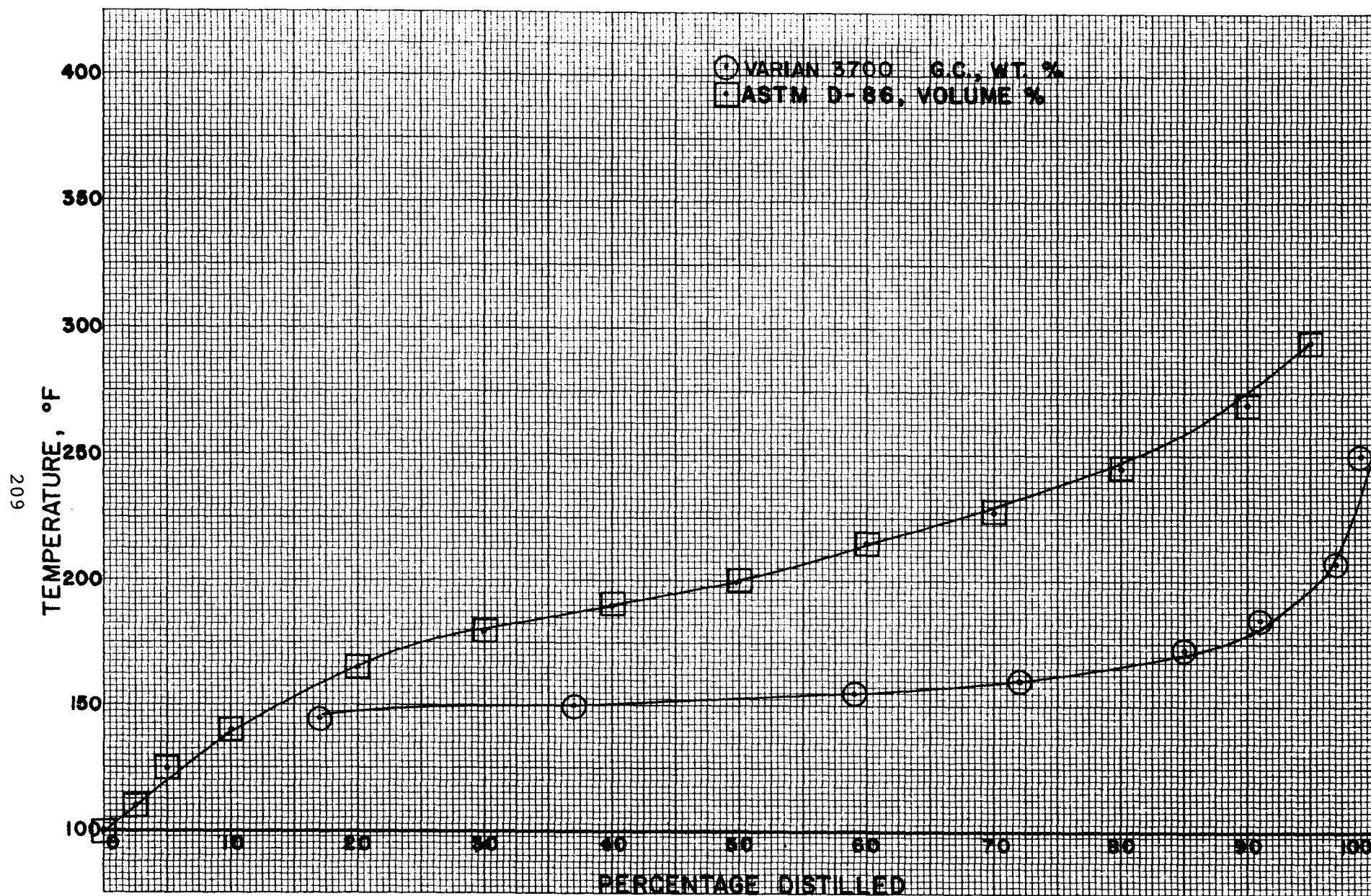


FIGURE 24. LIGHT ORGANIC LIQUID PRODUCT DISTILLATION CURVES, RUN 190 AB-MB

% ASH IN ASH CONCENTRATE (y)

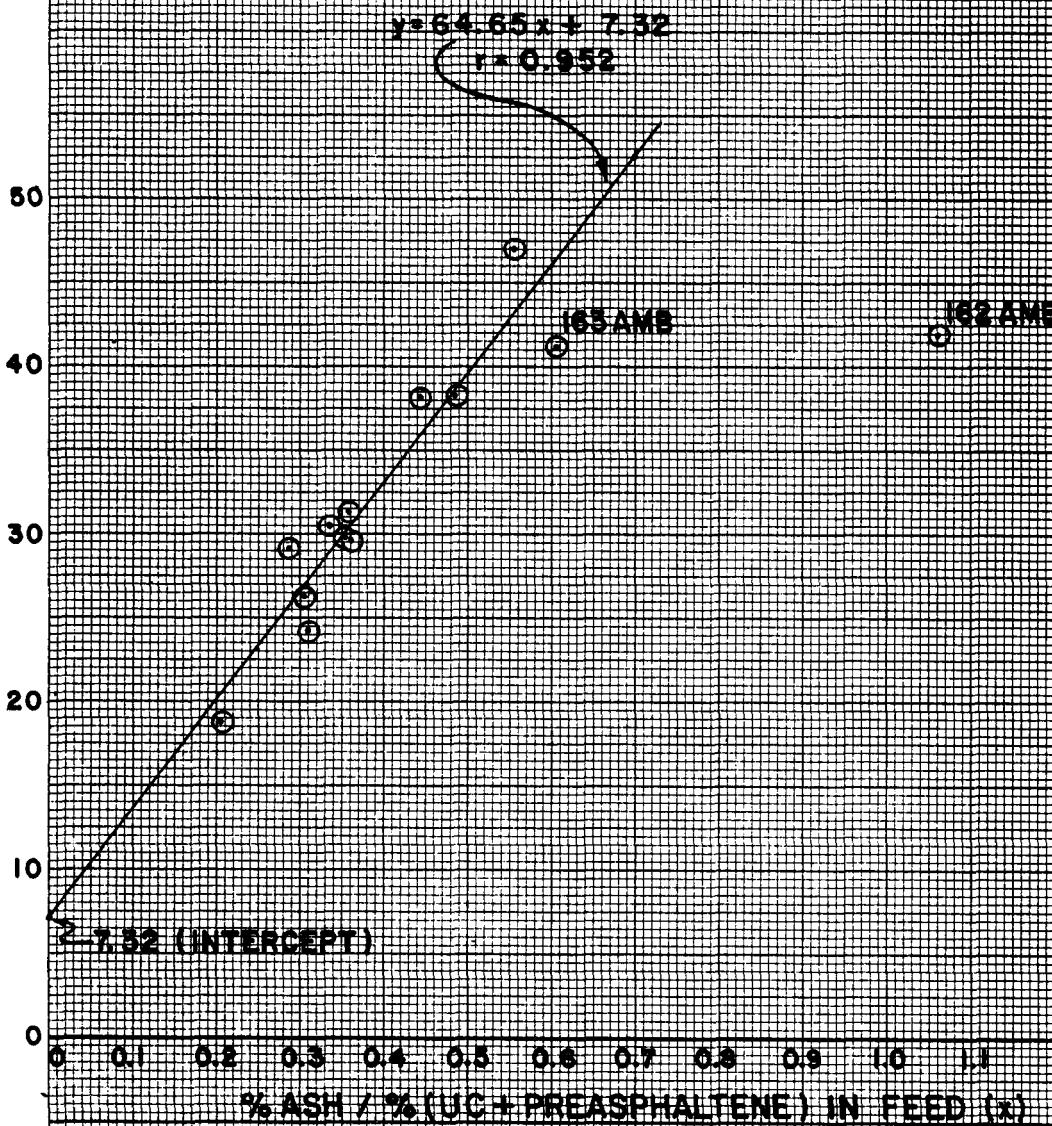
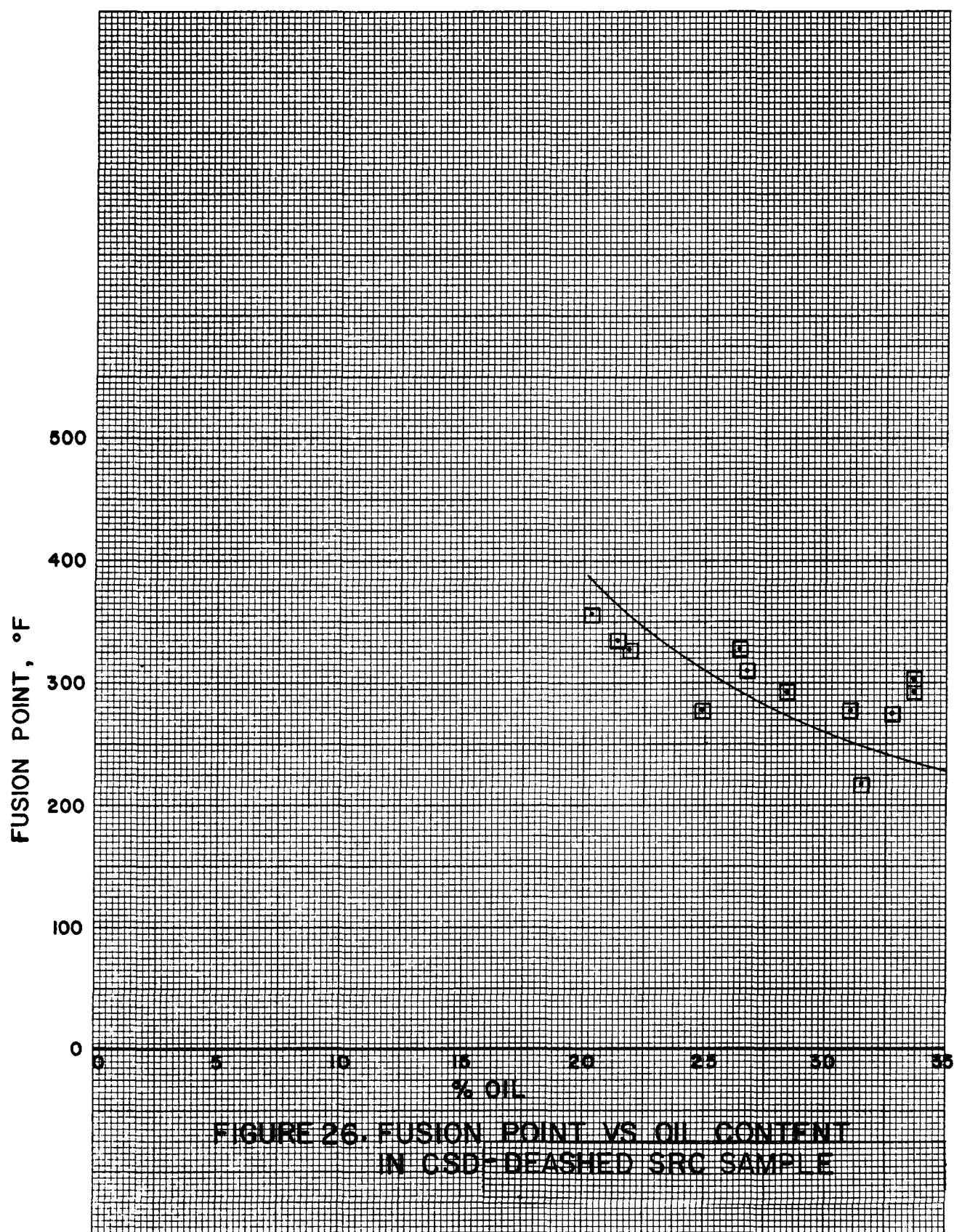


FIGURE 25. % ASH IN ASH CONCENTRATE VS
% ASH / % (UC + PREASPHALTENE)
IN FEED



FUSION POINT, °F

500
400
300
200
100
0

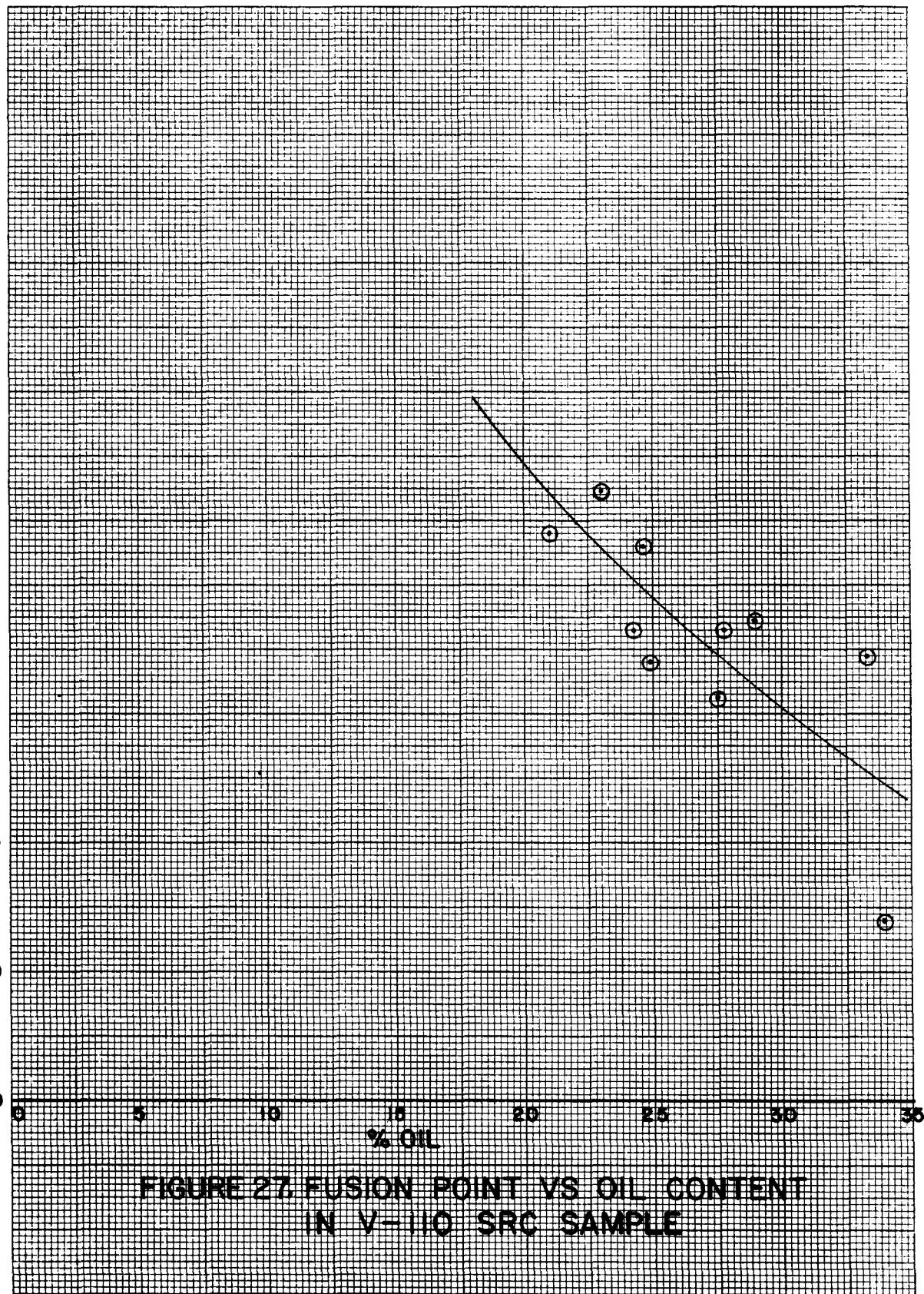
0

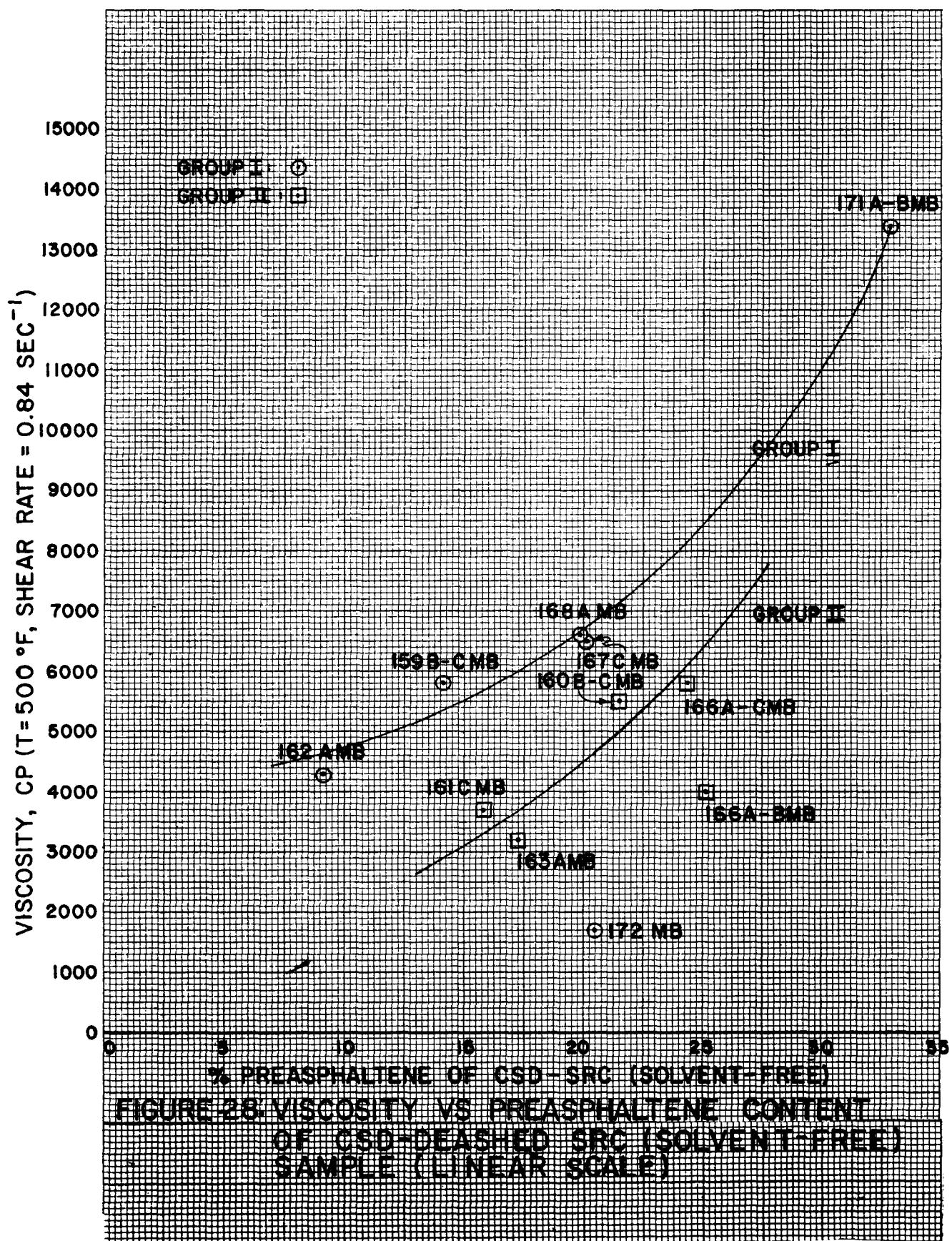
5 10 15 20 25 30 35

% OIL

212

FIGURE 27. FUSION POINT VS OIL CONTENT
IN V-110 SRC SAMPLE





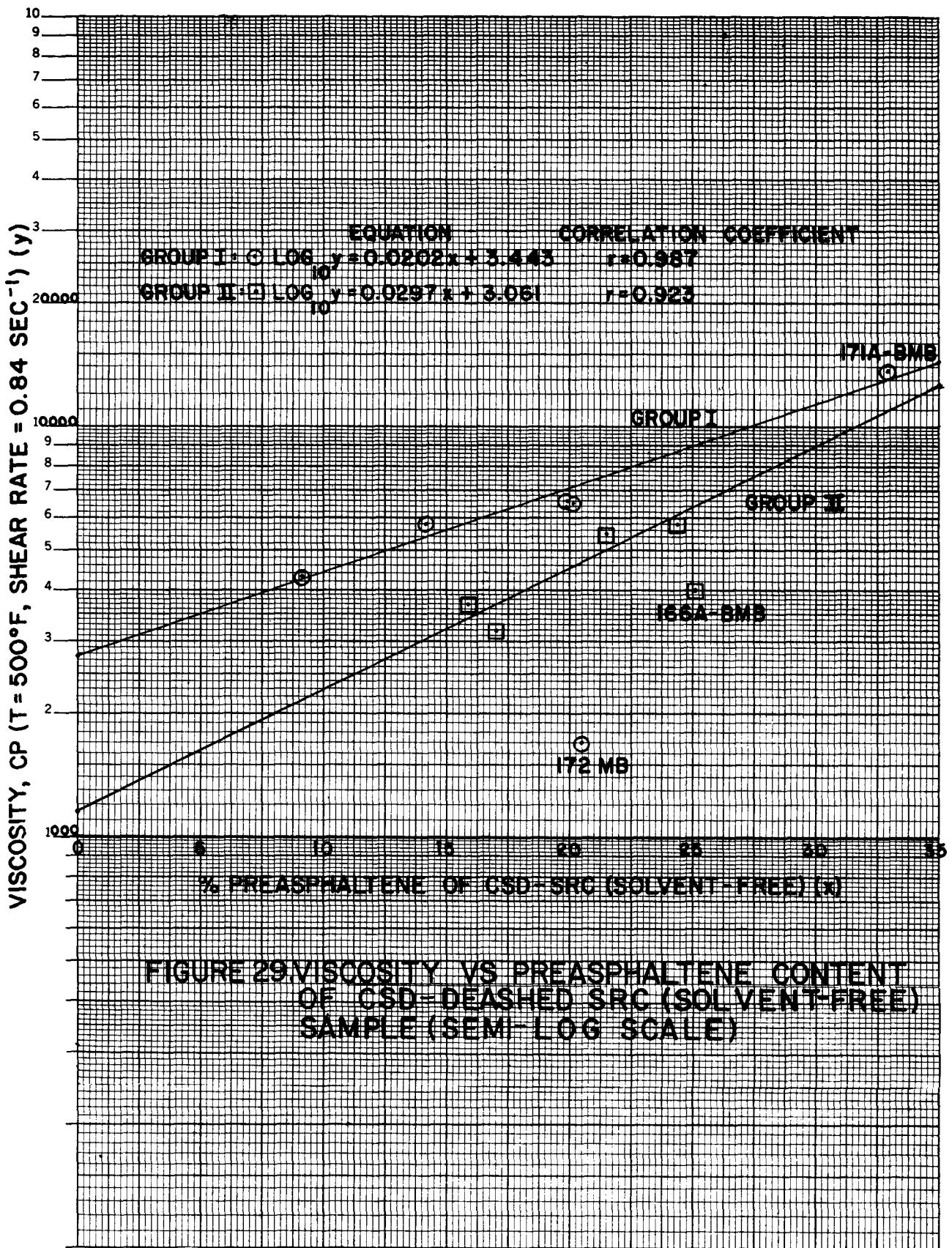


FIGURE 29 VISCOSITY VS PREASPHALTENE CONTENT
OF CSD-DEASHED SRC (SOLVENT-FREE)
SAMPLE (SEMI-LOG SCALE)

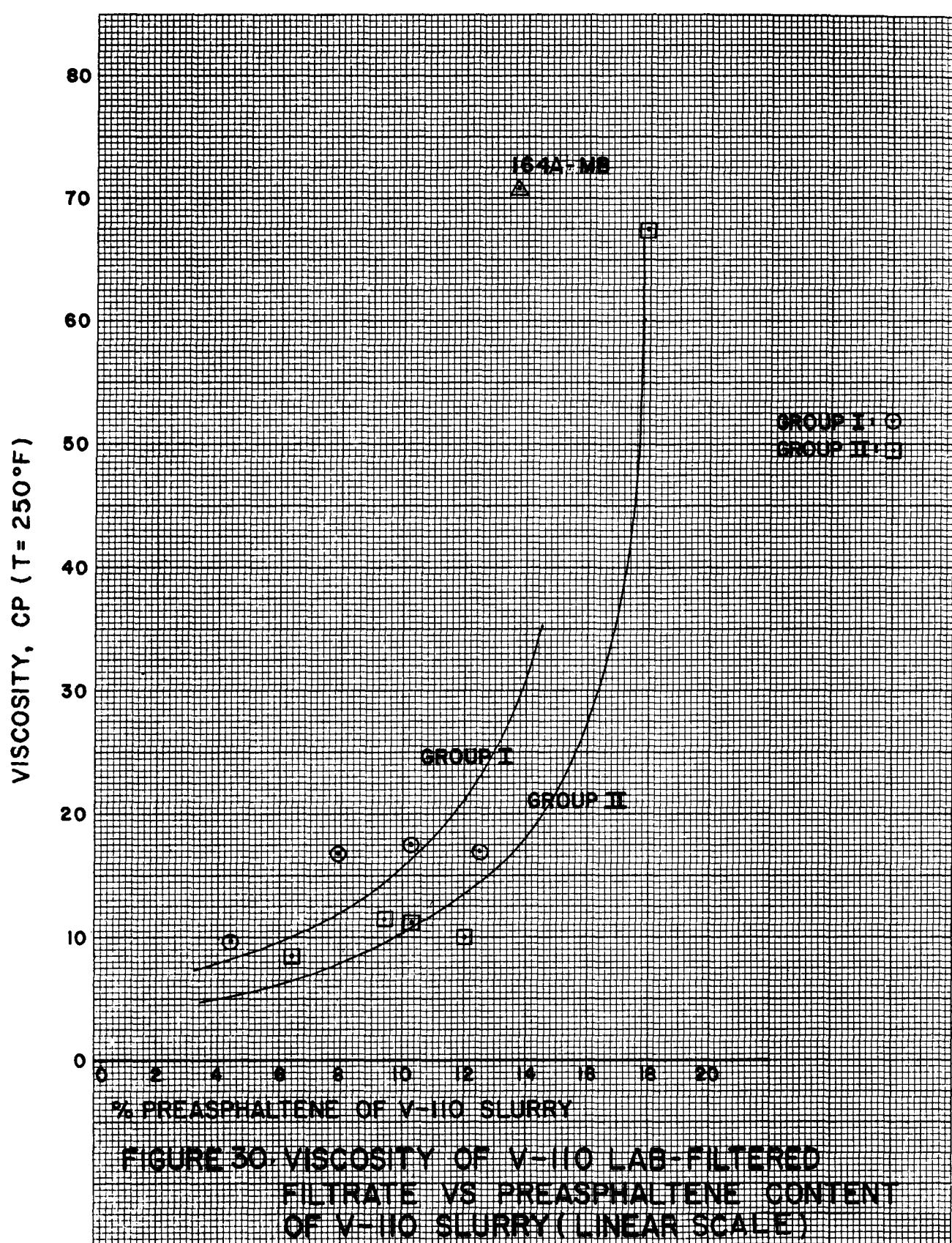


FIGURE 30. VISCOSITY OF V-110 LAB-FILTERED FILTRATE VS PREASPHALTENE CONTENT OF V-110 SLURRY (LINEAR SCALE)

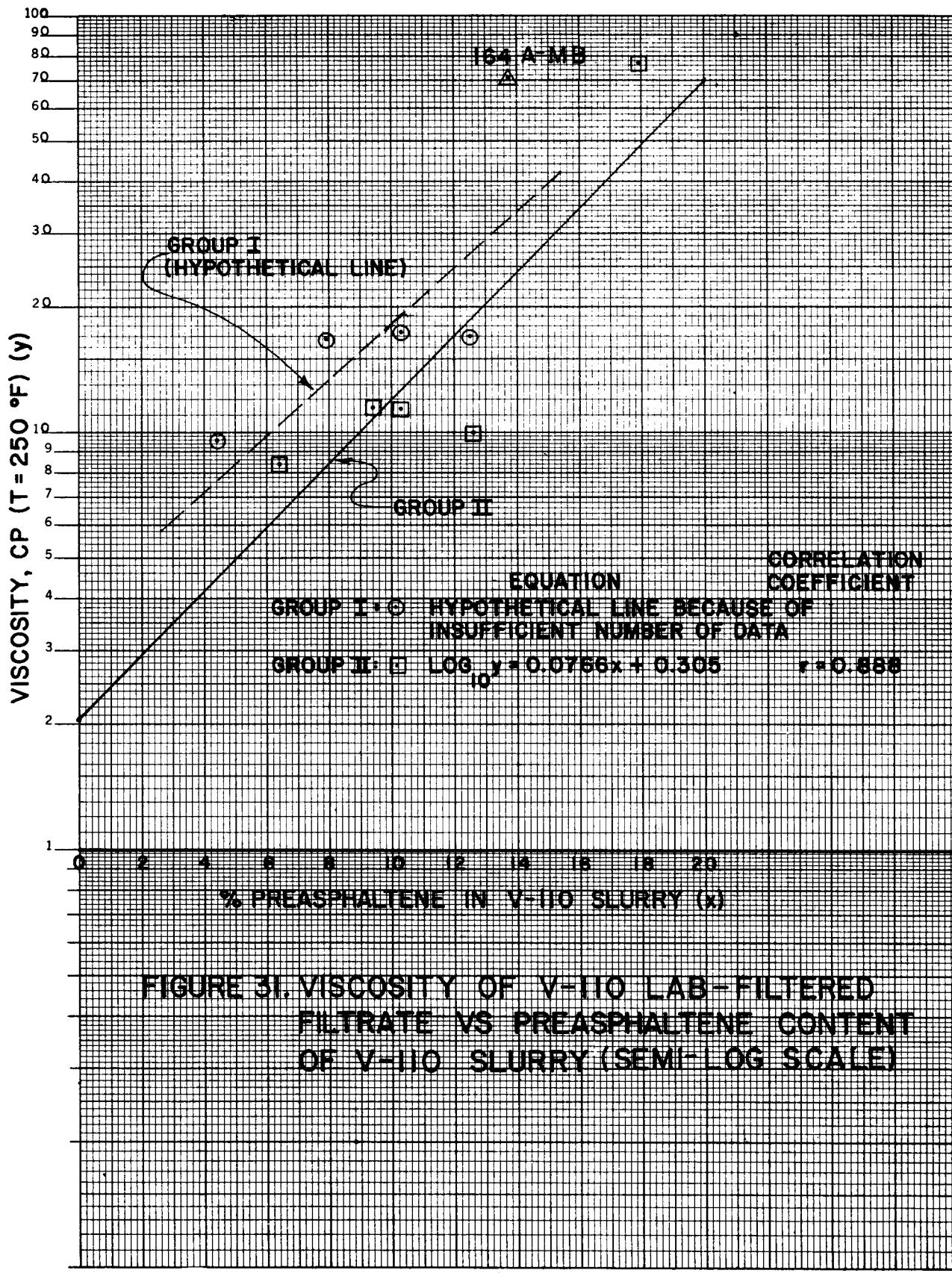


FIGURE 31. VISCOSITY OF V-IIO LAB-FILTERED
FILTRATE VS PREASPHALTENE CONTENT
OF V-IIO SLURRY (SEMI-LOG SCALE)