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SOLVENT REFINED COAL (SRC) PROCESS
OPERATION OF SOLVENT REFINED COAL PILOT PLANT
Wilsonville, Alabama

QUARTERLY TECHNICAL PROGRESS REPORT
for the period
JULY – SEPTEMBER 1978

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SYMBOLS AND ABBREVIATIONS

α (alpha)	average specific cake resistance
approx	approximate
ASTM	American Society for Testing and Materials
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)
CCDC	Conoco Coal Development Company
CF	capacity factor
CI	cresol insoluble(s)
cm/sec^2	centimeters per second per second
coeff	coefficient
col	column
comp	composite
conc	concentrate, concentrated, concentration
cons	consumed
conv	conversion
corr	corrected
cp	centipoise(s)
CSD	Critical Solvent Deashing
cum	cumulative
cfm	cubic feet per minute
cyc	cycle(s)
dia	diameter
DOE	United States Department of Energy
EEI	Edison Electric Institute
EP	end point
EPRI	Electric Power Research Institute
ERDA	Energy Research and Development Administration
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	foot (feet) per pound mass
ft^2	square foot (feet)
ft^3	cubic foot (feet)
ft-lb	foot-pound(s)
g	acceleration due to gravity
gal	gallon(s)
gm	gram(s)
gm/cc	gram(s) per cubic centimeter
gph	gallons per hour
gpm	gallons per minute

H/C	hydrogen to carbon atomic ratio
HP	high pressure; horsepower
hr	hour(s)
hr^{-1}	reciprocal hour(s)
HRI	Hydrocarbon Research Institute
HVB	High Volatile Bituminous
HVSB	High Volatile Subbituminous
IBP	initial boiling point
ID	inside diameter
in.	inch(es)
K	vapor-liquid equilibrium constant
KM	Kerr-McGee
lab	laboratory
L/D	length to diameter ratio
lb	pound(s)
lb/ft^2	pounds per square foot
lb/ft^3	pounds per cubic foot
lb/hr	pounds per hour
$lb/hr\text{-}ft^2$	pounds per hour per square foot
$lb/hr\text{-}ft^3$	pounds per hour per cubic foot
lgt org	light organic
LP	low pressure
lt	light
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/gm	milligram per gram
mid	middle
min	minimum; minutes
ml	milliliter(s)
mm	millimeter(s)
MM	million(s)
moist	moisture
mol %	mole percent
mol wt	molecular weight
MP	melting point
Mscf	thousands of standard cubic feet
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

P & M	Pittsburg and Midway Coal Mining Company
$p(H_2)$	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	degrees Rankine
R & D	research and development
ref.	reference
R_m	filter medium resistance
rpm	revolutions per minute
scf	standard cubic foot or feet at 525°F and one atmosphere
scfh	standard cubic feet per hour
scfm	standard cubic feet per minute
SCS	Southern Company Services, Incorporated
sec	second
sec^{-1}	reciprocal seconds
SG	specific gravity
SLS	solid-liquid separation
SN	sample number
solv	solvent
SOR	start of run
sp gr	specific gravity
spec	specification
sq ft	square foot(feet)
SRC	solvent refined coal
SRT	short residence time
SS	stainless steel
std	standard
sulf	sulfur
TBP	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	U. S. Filter Corporation
vac	vacuum
visc	viscosity
vol	volume
vs	versus
w/	with
wt	weight
wgtd	weighted
wt %	weight per cent
@	at
ΔHR	heat of reaction
Δt	temperature differential
ΔP	pressure differential
$<$	less than
$>$	more than
$\approx$	approximately
Σ	sigma (sum)

COAL IDENTITY

Coal processed during the quarter is listed below, together with the nomenclature used in this report for identification:

Company:	Old Ben Coal Company
State:	Indiana
Mine:	Old Ben No. 1
Seam:	Indiana V (Five)
Abbreviated identity:	Ind V

Company:	Pyro Mining Company
State:	Kentucky
Mine:	Pyro
Seam:	6 and 11
Abbreviated identity:	Ky 6 and 11

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

ABSTRACT

Operating conditions and test results obtained at the Solvent Refined Coal (SRC) pilot plant in Wilsonville, Alabama during the third quarter of 1978 are summarized in this report.

Indiana V coal processing (begun in June 1977) was concluded during this quarter. The feedstock was changed in September to Kentucky 6 and 11 from Pyro Mining Company's Pyro Mine.

A run was completed on Indiana V coal wherein the inventory of dissolver solids was controlled by use of a bottom withdrawal system. The solids concentration was varied from 5 to 37 lb/ft^3 to study its effects on SRC yield.

Operation of the Kerr-McGee Critical Solvent Deashing (CSD) unit was continued during the quarter. SRC containing 0.06-0.75% ash was produced at feed rates as high as 300 lb/hr. SRC recovery ranged from 73 to 84%.

I. INTRODUCTION

In March 1972, the Southern Company System and the Edison Electric Institute (EEI) jointly began a pilot-scale study of key steps in the solvent refining process for making low-sulfur and low-ash solid fuel from coal. Southern Company Services, Inc., representing the Southern Company System, provides project management.

In April 1973, the Electric Power Research Institute (EPRI) assumed the functions of utility industry project supervision formerly performed by EEI. The United States Energy Research and Development Administration (ERDA), now Department of Energy (DOE), became co-sponsor for the calendar years 1976, 1977, and 1978.

The six-ton-per-day solvent refined coal (SRC) pilot plant at Wilsonville, Alabama, was designed, constructed, and is being operated by Catalytic, Inc. Operation with coal has continued since January 1974. This report presents a summary of activities during the third quarter of 1978. Earlier work has been documented in prior monthly, quarterly, and annual Technical Progress Reports.

Current objectives of the Wilsonville SRC program are:

- To evaluate solid-liquid separation (SLS) processes, including
  - Antisolvent deashing with the Sharples centrifuge
  - Kerr-McGee Critical Solvent Deashing (KM-CSD)
  - Alternative deashing processes (such as a U. S. Filter Corporation vertical-leaf filter)
- To evaluate SRC process improvements, including
  - Short residence time (SRT) coal conversion
  - Feed gas bypass around the preheater
  - Three-stage Critical Solvent Deashing
- To improve process equipment and operations, including
  - Measurement and control of dissolver solids inventory
  - Reduction of in-plant process solvent inventory

- Demonstration of steady-state operation and process solvent balance when processing certain bituminous and subbituminous coals

The following progress toward fulfillment of these objectives was made during the first quarter of 1978:

- SRC containing less than 0.2% ash was produced using the Sharples solid-bowl centrifuge with the addition of antisolvent in the ratio of 0.4 lb per lb of reaction product.
- Tests confirming the high rate of Indiana V coal conversion at short residence times were completed. Coal conversion of 93% was attained when most of the hydrogen-rich gas was bypassed around the preheater.
- Nuclear density gauges were installed to measure the solids concentration in the dissolver.

Indiana V coal processing (begun in June 1977) was continued in the second quarter of 1978. The following experimental programs were emphasized during that period:

- Investigating hydrogen partial pressure and its effects on SRC yield
- Measuring the dissolver solids concentration
- Deashing (with centrifuges) using an anti-solvent
- Evaluating the performance of filter precoats
- Evaluating an experimental drum-type SRC solidifier

Operation of the Kerr-McGee Critical Solvent Deashing unit began on 25 June 1978. The following progress was made during the third quarter of 1978:

- A demonstration run with the KM-CSD unit in operation produced SRC containing less than 0.15% ash at feed rates up to 300 lb/hr. Continuous production of specification SRC was maintained for a period of 89 hours.
- Installation of a USF pressure-leaf filter unit to replace the Funda filter was begun.
- Two dissolver bypass runs were made: one at 300 lb/hr coal feed and a second at 600 lb/hr. Approximately 3,600 lb of SRC were obtained for SRT studies.

- Additional preheater gas bypass runs were completed at coal rates of 350 to 800 lb/hr and a dissolver pressure of 2,400 psig.

- The Kerr-McGee Third Stage Recycle System installation was completed.

- The solids withdrawal system was operated to maintain a low inventory of solids in the dissolver over a 10-day run. A system to measure the withdrawal rate has been designed and will be installed later.

- The nuclear density gauges were calibrated over a temperature range of 300 to 800°F on solvent and hydrogen flow and with coal slurry at various solids loadings.

- Correlations of specific gravity with solids concentration in the dissolver were developed.

- Methods were developed and modifications were completed to operate with a solvent inventory which is 50% less than that used with the filter system in service. This reduced inventory can be achieved with the Kerr-McGee CSD unit in service.

II. SUMMARY

In the SRC process, pulverized coal is slurried with a recycled, internally generated solvent. Hydrogen is added to the mixture, which is heated and fed to a high-pressure dissolver. Gas leaving the dissolver is scrubbed to remove hydrogen sulfide and carbon dioxide. Makeup hydrogen is added and the gas is recycled. Undissolved coal solids are separated from the SRC solution, and the solids-free liquid is distilled to separate the solvent from the SRC. The SRC is subsequently solidified by cooling, and the solvent is divided into a low-boiling fraction (350 - 450°F) for filter cake washing and a high-boiling fraction (450 - 900°F) for recycle to the system.

During most of the third quarter of 1978, Indiana V, a high-vitrinite bituminous coal containing 4% sulfur and 10% ash, was processed. Run 141 was completed with the full dissolver in service at 2,400 psig and 825°F in order to hydrogenate process solvent. Under these operating conditions, a series of preheater gas bypass tests was also completed. During these tests, pressure drop across the coil decreased as the gas was bypassed and coal conversion tended to increase with preheater outlet temperature. At preheater outlet temperatures above 800°F, however, conversion in Run 141 was lower than noted in the previous run because the hydrogen-donor capability of the solvent was also lower in Run 141.

Run 142 was made to supply high-ash SRC to the Kerr-McGee CSD unit for a demonstration run. In Run 143, the dissolver pressure was decreased from 2,400 to 1,700 psig to improve deashing of SRC in the Kerr-McGee CSD unit.

Runs 143 and 147 were made to study the effects of dissolver solids concentration on yields. The solids concentration was decreased from 37 lb/ft^3 during Run 143 to 5 lb/ft^3 during Run 147 by using the dissolver solids withdrawal system. The full-dissolver volume was used (coal space rate equal to 25 $lb/hr\text{-}ft^3$) at 825°F, 1,700 psig. The gas rate was 40 Mscf/ton of coal (85% hydrogen purity). There were no major differences in yields for these runs. The SRC yield from the reaction section was 53% of the MAF coal. Sulfur in SRC was 0.7 to 0.8%. Solvent make was 14 to 18% with hydrogen consumption at 3 to 3.2% of the MAF coal. Although there were no significant yield differences, the coal conversion decreased from 94 to 92% and solvent quality (as indicated by microautoclave tests) decreased during Run 147.

A short run (144) was made with the dissolver pressure at 2,400 psig in order to test the Kerr-McGee CSD unit while feeding SRC produced at the higher pressure. Runs 145 and 146 were short runs with coal feed rates of 300 and 600 lb/hr, respectively, and with the dissolver bypassed to obtain reaction product for SRT (short residence time) studies. Conversions of 92 to 94% were obtained with the preheater outlet at 860°F and feed gas rates of 3-10 Mscf/ton coal.

Material balances were completed with the Kerr-McGee CSD unit on-stream during Runs 143 and 147. During Run 143, upsets in the KM-CSD system resulted in intermittent increases in the SRC ash content. This problem was not evident during Run 147. Ash in SRC was 0.75% for Run 143 and 0.06% for Run 147. SRC recovery was 73% during Run 147 compared to 84% for Run 143.

The deashing solvent loss to the products was 0.4% of the feed. As a result of poor operation of the ash concentrate processing and discharge systems, the total deashing solvent loss was 5 to 6% of the feed.

Enough process solvent was produced during each run to increase the process solvent inventory while the KM-CSD unit was on-stream.

III. PROCESS DESCRIPTION

A. Coal Slurry Preparation

Figure 1 is a simplified flowsheet of the Wilsonville SRC pilot plant. Coal, pulverized so that 95% is smaller than 200 mesh (74 x 74 microns), is mixed with process-generated solvent in V101A Slurry Blend Tank. The resulting slurry is then transferred to V101B Slurry Feed Tank where it is agitated and recirculated to maintain a uniform suspension. P102B Slurry Circulating Pump is used both for recirculation and to feed P103A or B Slurry Preheater Feed Pump. The boiling range of solvent used to prepare the coal slurry is 450 to 900°F.

B. Slurry Preheating

Hydrogen-rich feed gas is normally added to the coal slurry upstream of the preheater inlet. The feed gas stream consists of scrubbed recycle gas plus sufficient makeup hydrogen to bring the gas to the desired composition (usually 85% hydrogen by volume).

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

C. Coal Dissolving

The coal slurry and gas mixture leaving the preheater flows upward through R101 Dissolver. The dissolver is 23 ft high and one foot in 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. Effluent from the dissolver is cooled to 600 to 650°F by E102 Dissolver Product Cooler. Outlets are located at the 25, 50, 75, and 100% volume levels to permit experimental variations in residence time.

Improvements made to the dissolver include 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 using normal pressure drop through the flow system from R101 outlet through E102 to V103.

D. High Pressure Gas and Slurry Separation

The vapor and slurry phases are separated in V103 High Pressure Separator. Vapor from the separator is cooled to about 150°F by E103 High Pressure 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.

E. 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.

F. Filtration

A schematic flow diagram of the filtration system is shown in Figure 2. Undissolved solids are removed from the coal solution by F103 Funda Pressure-Leaf Filter. This unit will accommodate 37 horizontal, circular filter leaves with metal wire screens having a total effective filtration area of 100 ft^2. The filter is operated at 480 to 580°F and 150 to 200 psig, with a maximum pressure drop of 80 psi between the slurry inlet and filtrate outlet.

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

(1) precoating

(2) filtration

(3) washing

(4) cake blowing

(5) depressurization

(6) vacuum flash drying

(7) cake discharging

(8) repressurization

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

Installation of a USF filter unit was begun in September. Evaluation will be started in October.

G. Mineral Residue Processing

After depressurization, the pressure in the filter is reduced to one or two 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.

H. 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 up to 900°F. Liquid SRC is drawn off at the bottom, solvent fractions from trays 3 and 8, and vapors in in the overhead. The column overhead passes through a vacuum jet precondenser and condensed light organic vapors are pumped to V164 Feed Tank and then to T104.

T102 is three feet in diameter and contains valve trays, tunnel cap trapout trays, and Koch-Sulzer packing. Overhead pressure is maintained at 0.3 to 1.5 psia by an overhead 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.

I. 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 ft^2. The SRC solidifies into brittle sheets which shatter into small fragments. The fragmented SRC is conveyed to storage.

J. Critical Solvent Deashing (CSD)

A schematic flow diagram of the CSD unit is shown in Figure 3. 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 SRC containing solvent, which can be added to the recycle solvent (third stage). The separations are made using a proprietary deashing solvent operating in the region of the critical temperature and pressure of the solvent. The various products are discharged into drums. The deashing solvent is recycled.

K. Gas Recovery and Recompression

Vapor from V104 High Pressure Vent Separator contains 60 to 80 mol % hydrogen, plus hydrocarbon gases, hydrogen sulfide, and carbon dioxide. The hydrogen sulfide and carbon dioxide are removed in T101 Hydrogen Scrubber by a dilute solution of caustic soda. The exit gas from T101 is scrubbed with water in V106 Recycle Hydrogen Water Scrubber to remove entrained caustic.

Scrubbed recycle gas is blended with pure hydrogen to provide a feed gas containing usually 85 mol % hydrogen. 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.

L. Light Solvent Recovery

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

T104 is six inches in diameter and contains two 5-ft sections: 1/4-in. "Pro-Pak" packing in the top, and 5/8-in. SS Pall rings in the bottom section. 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.

M. Solvent Fractionation

The material from V160 is fed to T105 Fractionation Column. It is heated to 220°F by the T105 bottom product stream, then enters at either trays 10, 12, 14, or 16, depending upon 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. However, at the design reflux ratio (reflux to overhead product rate) of 5, the required duties are about 55 to 60% of design.

N. Solvent Storage

The following solvent storage is provided:

(1) V123 Process Solvent Storage, startup and makeup;

(2) V131 Recovered Solvent Tank, recycle process solvent (boiling range 450 to 900°F);

(3) V124B Wash Solvent Storage Tank (350 to 450°F boiling range solvent); and

(4) V124A Light Oil Product Tank, light oil and excess wash solvent as total organic liquid by-product (boiling range normally below 350°F).

IV. OPERATIONS AND RESULTS

A. Run Summary

1. SRC Process

During the third quarter of 1978, coal was fed to the SRC pilot plant for 1,520 hours or 68.8% of the available time. A total of 332 tons of Indiana V coal and 17 tons of Kentucky 6 and 11 was processed. Run 140, in progress at the beginning of the quarter, and eight other runs were completed. A ninth run was in progress when the quarter ended.

Tables 1 through 13 present the following operating data:

Table 1	-	Operating Hours and Filter Cycles
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Table 12	-	Solvent Refined Coal Analyses
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A summary log of operations is given in Appendix A.

Three material balances were completed: Run 140B MB, Run 143 MB, and Run 147 MB. Results for Run 140B MB were presented in the report for the second quarter of 1978. (Ref. 2)

Operating conditions for Runs 143 and 147 were similar except that solids were withdrawn from the dissolver during Run 147.

Dissolver solids were allowed to accumulate during Run 143.

Ranges of operating conditions for the material balances were:

	Run 143 MB	Run 147 MB
Coal rate, MF lb/hr	435 - 489	411 - 481
Concentration, % MF coal	37.3 - 39.6	37.1 - 38.7
Gas rate, Mscf/ton MF coal		
To B102 inlet	41 - 45	43 - 46
To B102 outlet	0	0
Hydrogen in feed gas, mol %	84 - 88	83 - 86
Temperature, R101 outlet, °F	821 - 832	815 - 834
Pressure, psig	1700	1700 - 1720
Process solvent, % <450 °F	5 - 7	4 - 5
Dissolver volume, %	100	100

Ranges of results for the material balance periods were:

	Run 143 MB	Run 147 MB
Conversion, % MAF coal	94.0 - 94.8	90.2 - 93.2
Hydrogen consumption, % MAF coal	2.8 - 3.0	2.5 - 3.2
SRC yield, % MAF coal	53.5	54.0
Sulfur in SRC, %	0.7 - 0.8	0.8 - 0.9
Process solvent inventory change	Gain	Gain
Solvent microautoclave conversion, %	77.0	74.6

Run 141

Operating conditions for Run 141 were:

Coal	Indiana V
Dissolver Volume, ft^3	18.1
Coal feed rate, MF lb/hr	450
Slurry concentration, % MF	38.5
Gas feed rate, scfh	10,000
Dissolver pressure, psig	2,400
Dissolver temperature, °F	845

Coal feed was started at 1700 hr on 6 July.

The four purposes of Run 141 were:

- to test the mechanical aspects of the solids withdrawal system;
- to hydrogenate the process solvent;
- to complete a series of slurry preheater feed gas bypass tests; and
- to provide a high-ash feed from T102 bottoms to the Kerr-McGee Critical Solvent Deashing (KM-CSD) unit.

The withdrawal system designed to remove solids from the bottom of the dissolver was put into operation at the beginning of Run 141. A control valve designed to pass 50 lb/hr of the bottoms slurry was installed. This solids withdrawal valve was left in the "open" position for about four hours, during which time the solids level in the dissolver (as indicated by the nuclear density instruments) decreased faster than was expected. Over the next 24 hours, the valve was operated in the "off" position 95 to 98% of the time. It was subsequently removed from service and inspected. The stem and plug, made of Stellite No. 6, were severely worn.

During Run 141, 17 preheater gas bypass runs were also completed. The pressure in the R101 Dissolver was 2,400 psig while the gas rate through the B102 Slurry Preheater was varied from 0 to 10,000 scfh. The coal feed rate was varied from 350 to 800 lb/hr, and the slurry preheater outlet temperature ranged from 750 to 854°F. All of the B102 Slurry Preheater differential pressure transducers were in service. A high-pressure sample was taken at the outlet of the slurry preheater at each condition.

A total of 434 lb of blowdown solids was collected after Run 141 was completed on 16 July. The hydrogen-donor quality of the process solvent (as shown by microautoclave tests) did not increase during the 236 hours of Run 141.

Run 142

Run 142 operating parameters were the same as for Run 141 except the dissolver outlet temperature, which was lower for Run 142:

Coal	Indiana V
Dissolver volume, ft^3	18.1
Coal feed rate, MF lb/hr	450
Slurry concentration, % MF	38.5
Gas feed rate, scfh	10,000
Dissolver pressure, psig	2,400
Dissolver temperature, °F	825

Coal feed was begun at 0530 hr on 20 July. Blowdown solids from Run 140 (411 lb) and Run 141 (434 lb) were added to the slurry mix tank at the start of the run. A power outage occurred during a rainstorm on 22 July. After minor problems were corrected, feeding of coal was resumed. A plugged SRC discharge line (31 hours into the run) necessitated a Kerr-McGee CSD unit shutdown. During the maintenance downtime, the Funda filter was brought on-line to test Solka-Floc and Super-Cel precoat materials. The CSD run was resumed on 27 July. Because of the high ash content of the SRC product from the CSD unit, the run was stopped after 62 hours.

Run 143

A reduction in dissolver pressure from 2,400 to 1,700 psig was made at 0015 hr on 30 July. This was done to determine the effect of dissolver conditions on the performance of the CSD unit.

The run conditions were:

Coal	Indiana V
Dissolver volume, ft^3	18.1
Coal feed rate, MF lb/hr	450
Slurry concentration, MF lb/hr	38.5
Gas feed rate, scfh	10,000
Dissolver pressure, psig	1,700
Dissolver temperature, $^{\circ}F$	825

The Kerr-McGee CSD unit was shut down 3 hours after startup because the ash concentrate and SRC discharge lines became plugged. The unit was cleaned and the deashing run was resumed on 5 August.

Coal feed was stopped for approximately 10 hours on 12 and 13 August due to power outages. A 48-hr material balance with the CSD unit in operation was completed at 2400 hr on 21 August.

Run 144

The purpose of Run 144 was to determine the operability of the CSD unit with SRC produced under the following conditions:

Coal	Indiana V
Dissolver volume, ft^3	18.1
Coal feed rate, MF lb/hr	450
Slurry concentration, % MF	38.5
Gas feed rate, scfh	10,000
Dissolver pressure, psig	2,400
Dissolver temperature, $^{\circ}F$	825

The Kerr-McGee CSD unit produced specification SRC, but on 26 August, due to failure of the B102 burner, the dissolver was blown down and 675 lb of solids were removed. Run 144 was continued on 27 August. The dissolver was again blown down at 0600 on 28 August and 172 lb of solids were recovered.

Density gauge readings indicated that 100 lb of this material had remained in the dissolver from the previous blowdown, bringing the total to 775 lb for Run 144 (compared to 845 lb which had been added at the start of Run 143). The dissolver was not blown down at the end of Run 143.

Runs 145 and 146

Two dissolver bypass runs were completed on 29 August. These runs were made at the following conditions:

Coal	Indiana V	
Run	145	146
Dissolver volume, ft^3	0	0
Coal feed rate, MF lb/hr	300	600
Slurry concentration, % MF	38.5	38.5
Gas feed rate, scfh	1,500	1,000
B102 outlet pressure, psig	2,400	2,400
B102 outlet temperature, $^\circ F$	860	860

The combined duration of the two runs was 20.6 hours. During the lined-out period of each run, the dissolved coal slurry was withdrawn into drums from V110. Approximately 1,760 and 1,900 lb of SRC were obtained during Runs 145 and 146, respectively. The SRC was sent to C-E Lummus Company for short residence time studies under a separate DOE contract.

Run 147

Run 147 was a duplicate of Run 143 except that no solids were added to the reaction system and the solids withdrawal system was operated to maintain a low dissolver solids inventory. Calibration of the nuclear density gauges was in progress at the beginning of September.

Operating conditions were:

Coal	Indiana V
Dissolver volume, ft^3	18.1
Coal feed rate, MF lb/hr	450
Slurry concentration, % MF	38.5
Gas feed rate, scfh	10,000
Dissolver pressure, psig	1,700
Dissolver temperature, $^\circ F$	825

Coal feed for Run 147 started at 0600 hr on 2 September, but leaking dissolver relief and bypass valves forced a shutdown until 1300 hr on 4 September.

The solids withdrawal valve was put in service but the flow rate from the bottom of the dissolver was excessive. The withdrawal valve cycle time had to be shortened (less time with valve open) and the valve opening reduced. By the end of the run, the valve was in the fully-closed position, but it was still passing enough solids to maintain a low solids inventory in the dissolver. At the completion of Run 147 the valve trim was found to be severely eroded.

A 48-hr material balance was completed at 0001 hr on 9 September and the plant was shut down to complete tie-ins for the addition of the Kerr-McGee third stage system and for installation of the USF vertical-leaf filter unit. The dissolver was blown down and 97 lb of solids were collected. A total of 775 lb of solids was collected when the dissolver was operated without the withdrawal system.

Run 148

Run 148 was started at 1020 hr on 23 September using Indiana V coal. Prior to the introduction of coal, the nuclear density gauges on R101 were calibrated on solvent flow at 300 and 800°F. From 23 September to 1200 hr on 27 September, coal was fed at 400 lb/hr with specified amounts of dissolver blowdown solids added to the feed slurry in order to check the response of the nuclear density gauges. A total of 800 lb of solids was added. A changeover to Kentucky 6 and 11 coal was made during Run 148 on 27 September. Run conditions were as follows:

Coal	Kentucky 6 and 11
Dissolver volume, ft^3	18.1
Coal feed rate, MF lb/hr	450
Slurry concentration, % MF	38.5
Gas feed rate, scfh	10,000
Dissolver pressure, psig	1,700
Dissolver temperature, °F	825

Run 148 was terminated at 0930 hr on 29 September when a fuel oil leak developed in the slurry preheater. Some difficulty was experienced with deashing of SRC in the Kerr-McGee CSD unit during the run.

Run 149

Coal feed was resumed at 1500 hr on 29 September at the following conditions:

Coal	Kentucky 6 and 11
Dissolver volume, ft^3	18.1
Coal feed rate, Mf lb/hr	450
Slurry concentration, % MF	38.5
Gas feed rate, scfh	10,000
Dissolver pressure, psig	1,700
Dissolver temperature, °F	810

The dissolver temperature was lowered from the previous run to determine the effect on the performance of the Kerr-McGee CSD unit. Three hundred fifty pounds of recovered dissolver solids were added at the start of the run. The quarter ended with this run in progress.

2. Kerr-McGee Critical Solvent Deashing Process

The Kerr-McGee CSD unit was operated on feed from T102 bottoms for 1,032 hours (46.7% of the time) during the third quarter of 1978. A total of 76 tons of T102 bottoms was processed at an average rate of 1.8 tons per day. Feed rates during steady operating periods from each run ranged from 181 to 278 lb/hr. The on-stream factor (based upon feed availability from the SRC unit) during Runs 143 and 147 was 65 and 88%, respectively. Results of these runs are summarized below:

Run	143	147
Average feed rate, lb/hr	231	278
Total feed, tons	30	11
Run period	5-21 Aug	5-8 Sept
Run duration, days	16.5	3.7
On-stream factor, %[a]	65	88
Reasons for downtime (as a per cent of downtime)		
A. Heat exchanger fouling	53	-
B. Temporary plugging-letdown lines	11	14
C. Temporary plugging-vent lines	10	73
D. Feed pump problems	13	6
E. Miscellaneous	13	7
Total	100	100

(a) Based upon total feed availability from SRC pilot plant

Operating data for the Kerr-McGee CSD unit are summarized in Tables 2, 3, and 4. Results from the material balances around the CSD unit are presented in Table 7. Table 11 shows the amount of T102 bottoms processed by the Kerr-McGee CSD unit during the quarter. Table 12 shows the analyses of the KM-feed and KM-SRC product.

Run 141

Run 141 was made using the Kerr-McGee CSD unit to evaluate equipment performance and process parameters. Liquid feed was transferred directly to the CSD unit. There were three attempts to make a process demonstration and material balance run. These attempts were terminated prior to completion due to high ash (>0.5%) in the SRC product and to the plugging of the ash concentrate discharge line.

During Run 141, approximately 17 tons of feed were processed in the CSD unit at an average rate of 181 lb/hr. During a lined-out period from 8 to 14 July, the product SRC contained 0.04 to 0.25% ash. The recovery of SRC from the feed was approximately 82%. The ash concentrate contained approximately 43% ash, 27% undissolved carbon, and 30% SRC.

Run 143

A process demonstration and material balance run (143) was completed on 21 August. Approximately 30 tons of high-ash SRC feed were processed at an average rate of 231 lb/hr. During the two-day material balance period, 6.5 tons were processed at an average rate of 290 lb/hr. Three objectives of the demonstration run were achieved in this material balance:

- easily handled, powdery ash concentrate was produced,
- deashing solvent loss to the products was acceptable, and
- continuous operation was maintained.

The fourth objective (producing SRC containing less than 0.16% ash) was not achieved over the 48-hr period as a result of upsets during the first 24 hours of the MB period. The average ash content was 0.75% for the total MB period. During the last 24 hours of the MB period, the ash in the SRC averaged 0.15%. Some SRC samples taken during the MB contained as low as 0.01% ash.

Run 144

The high-ash SRC obtained during Run 142 could not be deashed in the Kerr-McGee CSD unit until the process conditions were changed during Run 144. At that time, however, the ash in this SRC was reduced to 0.05%. About 10 tons of feed were processed at an average rate of 212 lb/hr. Reaction products obtained both with and without a high solids inventory in the dissolver were successfully deashed. Ash in the SRC ranged from 0.05 to 2.5% during Run 144.

Run 147

A second demonstration run was started on 5 September. A 48-hr material balance period was completed on 9 September, during which approximately 6 tons of SRC feed containing ash were processed at an average rate of 256 lb/hr. The product SRC contained an average of 0.06% ash for the 48-hr period.

B. Slurry Preparation

All coal slurries were prepared at a nominal 38.5% MF concentration. Repairs to slurry pumps P101A and B were made on 6 August. No other problems were experienced with slurry makeup or handling.

Unpulverized Indiana V coal contained about 8% moisture, and unpulverized Kentucky 6 and 11 coal contained 4.5% moisture. Pulverized Indiana V coal averaged 2.0% moisture content for the quarter with a range of 0.77 to 5.2%. Moisture content of the pulverized Kentucky 6 and 11 coal averaged 0.76% with a range of 0.60 to 0.93%.

Indiana V coal contained about 11% ash and 4% sulfur compared to 9% ash and 3% sulfur for Kentucky 6 and 11 coal. Detailed coal analyses for material balance periods of Runs 143 and 147 are given in Table 13.

Hydrogen-to-carbon atomic ratios of 0.76 and 0.80 were obtained for Indiana V coal for the two material balance periods. A H/C ratio of 0.87 was obtained for a sample of Kentucky 6 and 11 coal.

Variations in coal composition may occur due to variations in the seam, mining methods, coal handling, and pulverizing. A 30-cc laboratory microautoclave is used to determine changes in reaction performance which may be related to these variations in coal composition. The THF coal conversion is determined by using the coal composites from material balance periods. A standard solution of 25% tetralin in 1-methylnaphthalene is used as the solvent. Microautoclave conditions were:

Reaction time, minutes	30[a]
Solvent to coal ratio	2
Temperature, °F	750

(a) Includes 2-minute heatup period.

Results obtained during the third quarter were:

Run	THF Conversion, %
143 MB	76.4
147 MB	78.4

Specific gravity data for coal slurries (obtained by labatory analyses) were:

Coal	Indiana V		Kentucky 6 and 11	
Conc, wt %	38.5		38.5	
Temp, °F	60	150	60	150
Sp gr	1.116	1.100	1.115	1.098

Slurry viscosity data for Kentucky 6 and 11 coal at 38.5% slurry concentration were:

Temperature, °F	Shear Rate, sec^{-1}	Viscosity, cp
80	20.4	308
	10.2	315
150	20.4	95
	10.2	105
200	20.4	65
	10.2	75
250	20.4	38
	10.2	45

Viscosity data for Indiana V coal slurries have been presented in previous quarterly reports. (Ref. 1, 2)

C. Preheating and Dissolving

1. Preheater Gas Bypass Runs

A series of 17 short runs was made during Run 141 using Indiana V coal at different operating conditions with part or all of the feed gas bypassed around the preheater. Normally, all of the hydrogen gas is introduced at the preheater inlet and the preheater outlet temperature is adjusted to maintain a constant dissolver outlet temperature. If part of the cold hydrogen gas is bypassed around the preheater and used to quench the feed to the dissolver, higher preheater outlet temperatures can be maintained.

Table 14 summarizes slurry preheater operating data. Thermocouple locations are shown in Figure 4.

The percentage coal conversion, SRC, and sulfur in SRC were determined from preheater effluent samples. These results are summarized in Table 8. Results from dissolver effluent samples are also included for comparison. Analyses of the SRC and cresol insoluble fractions of the preheater effluent samples from material balance periods (Runs 143 and 147) are included in Table 5.

a. Pressure Drop

The preheater pressure drop and skin/fluid temperature data indicate that there was no significant coke formation in the preheater during the series of 17 short runs:

Test	Coal rate, MF lb/hr	Gas rate, scfh	Preheater outlet temp, °F	Δp across preheater, psi
I	785	3,070	787	50
II	835	0	829	71
L	747	0	837	36
LL	765	0	854	57

The following data show the effects of gas rate on the coil pressure drop for a coal feed rate of 500 lb/hr at 37.5% slurry concentration:

Test	Gas rate, scfh	Preheater outlet temp, °F	Δp across preheater, psi
E	8,200	800	56
H	3,130	815	41

The following data show the general trend of increased coil pressure drop with increased slurry flow rate (at 3,000 scfh gas flow and 36% slurry concentration):

Test	Coal rate Mf lb/hr	Preheater outlet temp, °F	Δp across preheater, psi
G	288	832	26
H	464	815	41
I	785	787	50

The effects of changing the preheater outlet temperature on coil pressure drop are not known, but are believed to be small due to the small differences in temperature.

The following data show the effects of changes in slurry concentration on the coil pressure drop with all gas by-passed:

Test	Coal rate, MF lb/hr	Conc, MF coal, %	Preheater outlet temp, °F	Δp across preheater, psi
K	492	36.8	848	33.0
N	444	26.7	848	26.0

b. Coal Conversion and Solvent Quality

Coal conversion at the preheater outlet varied from about 57 to 93% of the MAF coal for the following ranges of operating conditions (2,400 psig operating pressure):

Coal feed rate, MF lb/hr	290-860
Slurry conc, % MF coal	26-38
Gas feed rate, scfh	0-10,000
Preheater outlet temp, °F	750-855

A hydrogen purge gas flow of about 200 scfh was maintained at all times.

In the ranges of operating conditions used, coal conversion at the preheater outlet was primarily a function of preheater outlet temperature. This relationship is shown in Figure 5 which also presents the data obtained in January and July 1978. Preheater conversions in July were lower than in January due to differences in the donor capabilities of the process solvent.

Test	Coal feed rate, MF lb/hr	Slurry conc, % MF coal	Gas rate scfh	Preheater outlet temp, °F	Conversion % MAF coal
P (Jan '78)	832	37.8	0	848	89.2
L-L (July '78)	765	36.4	0	854	78.2
G (Jan '78)	434	37.5	3,010	823	88.9
H (July '78)	464	35.3	3,130	815	72.6

The following data show the solvent donor capabilities during January and July 1978:

Test Period	Process solvent from	Microautoclave[a] conversions, %
1-6-78	V131	80.2
7-2-78	V131	71.2
7-12-78	T105 btms	73.2
7-13-78	T105 btms	73.2

(a) Microautoclave long run method (using standard Indiana V coal).

During January, the effect of residence time on coal conversion was significant. For example, at similar conditions, the effects of coal feed rate (hence residence time) on coal conversion were:

Test	Coal feed rate, MF lb/hr	Slurry conc, % MF coal	Gas rate scfh	Preheater outlet temp, °F	Conversion, % MAF coal
J	382	36.4	0	850	88.9
L-L	765	36.4	0	854	78.2

Appendix B of the report for the first quarter of 1978 (Ref. 1) presented the results of the reaction kinetics for coal dissolution in the preheater at various gas bypass conditions. The rate data followed a first order kinetics model. Residence time was based upon effective reaction volume occupied by the slurry. Liquid holdups were estimated from the Lockhart-Martinelli correlations (Ref. 3) for two-phase flow. The effect of temperature on the coal dissolution rate coefficient, K_p, followed the Arrhenius law:

$$K_p = K_o e^{-E/(RT)} \qquad (1)$$

where

K_o = Frequency factor, hr^{-1}

E = Activation energy, Btu/lb-mole of coal

R = Gas law constant, 1.987 Btu/(lb-mole)(°R)

T = Preheater outlet temperature, °R

The July preheater conversion data were fitted to the coal conversion model. These data are contained in Appendix B. Estimated activation energies and frequency factors at 2,400 psig operating pressure for each period were:

	Jan 1978	July 1978
Solvent quality		
THF conversion,[a] %	80.2	72.6
Activation energy, E, Btu/lb mole	37,100	19,100
Frequency factor, $K_O \times 10^{-6}$, hr^{-1}	174	0.13

(a) Long run conditions.

As observed during the short residence time coal conversion tests in January 1978, the solvent quality for short residence time operations cannot be maintained without additional rehydrogenation of the solvent. The following data indicate that the solvent hydrogen-donor capability decreases in the preheater. Solvent was separated from four of the preheater samples by laboratory distillation and subjected to a standard microautoclave test to determine hydrogen-donor capability. Results were:

Test Period	Preheater conversion, % MAF coal	Microautoclave conversions for distillate, % short run	long run
Process solvent(2 July 78)	-	77.9	71.2
Process solvent(12 July 78)[a]	-	-	73.2
Test			
B	57.7	-	67.8
E	64.0	-	69.7
H	72.6	62.9	71.5
J	88.9	68.2	67.5

(a) T105 bottoms

The following data show coal conversions at the preheater outlet and overall conversions attained at the dissolver outlet during Runs 143 and 147:

Run	Date, 1978	Outlet	Temp, °F	Feed gas to preheater scfh	Conv. % MAF coal	Sulfur in SRC, %[a]
143	19 Aug	Preheater	765	10,000	60.3	1.47
	19 Aug	Dissolver	820	-	94.4	0.81
147	6 Sept	Preheater	766	10,000	54.7	1.45
	6 Sept	Dissolver	820	-	93.1	0.84

(a) Laboratory filtered and distilled @ 600°F, 0.1 mm Hg

c. <u>SRC Yield and Composition</u>

Earlier studies (Ref. 1) have shown that at short reaction times, yields of SRC are usually greater than 75% of the MAF coal at high coal conversions (80% or above). Estimated SRC yield data for the preheater gas bypass tests, based upon ash balances, are given in Table 8.

SRC products from Tests B, H, and K were fractionated according to the solubility of their constituents in pentane, benzene, and cresol. The SRC components which are soluble in pentane are classified as oil and the SRC components which are soluble in benzene but insoluble in pentane are classified as asphaltenes. The benzene insolubles (cresol solubles) are termed preasphaltenes. According to a published reaction mechanism for coal dissolution, (Ref. 4) coal is first converted to preasphaltenes which then are converted to asphaltenes and oils. The following data on SRC solubility confirm this mechanism:

Test	Coal Conversion % MAF coal	SRC[a] Components		
		Oil	Asphaltenes	Preasphaltenes
B	58	6.1	3.7	90.2
H	73	7.9	7.8	84.3
K	91	14.7	18.2	67.1

(a) From laboratory workup of B102 Preheater samples.

2. <u>Dissolver Bypass Runs</u>

Two dissolver bypass runs (145 and 146) were made at coal feed rates of 300 and 600 lb/hr. About 3,600 lb of short residence time SRC were produced. Coal conversions at the V110 Flash Tank outlet varied from 92-94% of the MAF coal for the following ranges of operating conditions (2,400 psig operating pressure):

Coal feed rate, MF lb/hr	300-600
Slurry conc, % MF coal	35-36
Gas rate, Mscf/ton MF coal to preheater inlet	3-11
Preheater to outlet temp, °F	355-860

a. <u>SRC Yield and SRC Solubility Analyses</u>

The following are SRC yields from Runs 145 and 146:

Run	Coal feed rate, MF lb/hr	SRC Yield	
		Ash balance[a]	V110 drumout[b]
145	287	NA	77
146	580	75	79

(a) Based upon % SRC from V110 laboratory workup and using forced ash balance.

(b) Based upon % SRC from V110 laboratory workup and using V110 drumout weights.

The following data show the solvent fractionation analyses of the SRC products from Runs 145 and 146:

Run	Coal Conversion,%	SRC Components[a]		
		Oil	Asphaltenes	Preasphaltenes
145	94.2	21.2	50.1	28.7
146	92.8	17.6	53.7	28.7

(a) SRC from laboratory distillation of filtered V110 samples at 600°F, 0.1 mm Hg.

D. Reaction

1. Stability

Reaction stability in the dissolver depends upon several operating parameters including temperature, solids accumulation, solvent quality, solvent composition, and coal composition. Nuclear density gauges are used to monitor the solids concentration at three locations in the dissolver. A solids withdrawal line is sometimes used to remove solids continuously from a point 10 inches above the distributor plate. These systems are shown in Figure 6. A 16-point temperature probe is used to monitor the temperature profile at various dissolver heights. Gas chromatography and elemental analyses are used to determine solvent composition. Solvent hydrogen donor capability and changes in coal reactivity are evaluated with a laboratory microautoclave.

a. Dissolver Solids Inventory and Temperature Control

Run 147 was made under conditions similar to Run 143, except that a reduced inventory of dissolver solids was maintained. The solids withdrawal rate was controlled by a valve (0.025 C_V) in the withdrawal line from the dissolver bottom to V103 High Pressure Separator. Table 15 presents the variations in slurry preheater outlet temperature and the temperatures over the height of the dissolver for the material balance periods. Control of the dissolver temperature was unsteady when withdrawing the solids from the dissolver bottom. The temperature variations at the preheater outlet and at the dissolver outlet were:

Run	143 MB		147 MB	
Date, 1978	20 August	21 August	7 Sept	8 Sept
B102 Preheater				
Outlet temperature, °F	760-769	667-837	761-773	752-778
R101 temperature, °F				
Outlet	821-829	793-832	819-829	815-834
Bottom	802-810	783-813	793-803	786-813

The wide ranges of temperatures noted on 21 August were due to several slurry preheater flame outages. After each outage, the flame was quickly restored.

The settings of the solids withdrawal valve cycle timer were varied to maintain temperature and to maintain a low level of solids in the dissolver. The average reading on the bottom nuclear density gauge during Run 143 MB was 62% compared to about 8% during Run 147 MB. The settings on the valve cycle timer control were varied to open the valve for periods of 3 to 15 seconds and to close it for periods of 15 to 50 seconds. On certain occasions, the valve was kept closed because it was believed to be leaking. The average dissolver solids concentration at the end of Run 147 was 5.4 lb/ft^3 compared to 37.3 lb/ft^3 at the end of Run 143. The amounts and compositions of the accumulated solids are given in Table 16. Daily average nuclear density gauge readings are given in Tables 2, 3, and 4. The average density readings at the beginning and end of the runs were:

		Average Density Reading, % Liquid Volume		
Run	Period	Top	Middle	Bottom
143	Start[(a)]	-	50	65
	End	18	64	62
147	Start	-	10	12
	End	6	6	8

(a) Eight hundred forty-five lb blowdown solids added to the system during Run 142.

Problems associated with the present withdrawal system are:

- o Rapid wear of small valve trim, and
- o Excessive flow of material through withdrawal line when valve is open, resulting in temperature and density gauge fluctuations.

Proposed solutions are:

- o To install tungsten carbide trim in the withdrawal valve, and
- o To install a flow measurement device in the reactor withdrawal line. Flow measurement is necessary to separate the effects due to dissolver solids and those attributable to bypass material around the dissolver.

b. Nuclear Density Gauge Calibration

On 20-22 September, the dissolver nuclear density gauges were calibrated as follows:

(1) With about 1,900 lb/hr solvent flow and 10,000 scfh gas (95 mole percent hydrogen) flow through the dissolver at 800°F, the three gauges were adjusted to read zero transmittance, the span controls were adjusted based on previous data, and the suppression controls were adjusted to "zero" the meters;

(2) With R101 Dissolver full of solvent at 250°F and no hydrogen flow through the dissolver, the span controls were adjusted so that all three gauges read 40% of full-scale; and

(3) With the conditions listed under point (1) repeated, minor adjustments in transmittance settings were made to "zero" the meters.

Following calibration, 160 lb of solids (from previous blowdowns) were added to 2,000 lb of coal in a 38.5% coal slurry batch and fed to the reaction section. The coal feed rate was 400 lb/hr and the gas flow was 10,000 scfh at 85% hydrogen purity. The dissolver outlet pressure and temperature were 1,700 psig and 825°F, respectively.

Following the addition of a 160 lb batch of solids, a total of 6,000 lb of coal slurry containing no solids was added. These two steps were repeated until about 800 lb of dissolver solids had been introduced. At the end of the series, the nuclear density readings were 50% at the bottom gauge, 39% at the middle gauge, and 18% at the top gauge. The dissolver was blown down and about 600 lb of solids were recovered. At the end of the solids addition tests on 25 September, coal feed (Indiana V and Kentucky 6 and 11 coals) was continued to the front end until 2 October under the following conditions:

Coal feed rate, MF lb/hr	150-450
Coal concentration, MF wt %	30-38
Gas feed rate, scfh	10,000
Dissolver pressure, psig	1,700
Dissolver outlet temperature, °F	825

Four drums of V144 blowdown material from previous blowdowns were also added between 25 September and 2 October.

When the dissolver was blown down on 2 October, about 600 lb of solids were recovered. At the resumption of coal feed, the bottom and middle nuclear density gauges indicated that all of the solids in the dissolver had been removed during blowdown.

Figure 7 shows the relationship between nuclear density gauge readings and dissolver solids content for full-dissolver operation at 10,000 scfh gas (85% hydrogen purity) and 400 lb/hr coal feed rates (38% slurry concentration), 1,700 psig and 825°F R101 Dissolver outlet temperature. The following differential pressure (DP) cell and nuclear density gauge readings were used to develop these correlations:

Period	Readings, % of Full Scale			
	DP Cell[a]	Nuclear Density Gauge		
		Top	Middle	Bottom
At the end of solids addition, lb				
0	25	-	-	-
160	30	24	16	22
320	35	12	20	37
480	40	17	24	43
640	46	17	36	46
800	78	38	46	56
Prior to dissolver blowdown on 2 October (600 lb recovered)				

(a) 300 inches of water range.

The DP cell readings of 25% (with no solids in R101 Dissolver) and 78% (with 600 lb of solids in R101) were used to construct a curve (DP cell reading vs solids in R101) similar to curves obtained previously (Ref. 5). This curve and the above data were then used to construct Figure 7.

c. Solvent Quality

The hydrogen-donor quality of the process solvent decreased during all runs except 143 and 144:

Coal	Indiana V						Kentucky 6 and 11
Date, 1978	2 July	26 July	3 August	20 August	1 Sept	7 Sept	29 Sept
Run	140B	142	143	143 MB	147	147 MB	148
Microautoclave method	S / L	S / L	S / L	S / L	S / L	S / L	S / L
THF conversion, %	78 / 71	77 / 71	73 / 69	77 / 74	76 / 73	75 / 75	73 / 73
Hydrogen, %	8.81	8.45	9.18	9.23	8.73	9.10	9.07
Carbon	86.67	87.63	86.90	86.54	86.76	86.83	86.60
H/C atomic ratio	1.21	1.15	1.26	1.27	1.20	1.25	1.25
Specific gravity	1.017	1.016	1.010	1.006	1.020	1.007	1.008

Specific solvent quality data for Run 144 are not available. Figure 8 shows microautoclave conversions for short and long run conditions as a function of the percentage of tetralin in 1-methylnaphthalene. The solvent hydrogen-donor quality was higher during Run 140 than for Runs 137 through 139 (Ref. 2). This was probably due to a larger dissolver solids inventory.

Run 141 was an attempt to hydrogenate the process solvent, but no improvement was noted using the full-dissolver with a large solids inventory at 845°F and 2,400 psig. Reducing the temperature to 825°F in Run 142 resulted in a decreased hydrogen-donor quality, which indicates that unknown factors are present. Operating the full-dissolver at 1,700 psig and 825°F with a large solids inventory (Run 143) did improve the solvent hydrogen-donor quality. In Run 143, the dissolver solids inventory was 37 lb/ft^3 and no material was bypassed around the dissolver. In Run 147, the dissolver solids inventory was reduced to 5 lb/ft^3 and some material was bypassed. The hydrogen-donor quality of the solvent was lower in Run 147 than in Run 143. This trend of decreasing the hydrogen-donor quality with decreasing solids inventory was also noted in Runs 133 (Ref. 1) and 134. (Run 134 was the first run made using the bottom solids withdrawal system). This effect was noted (Ref. 2) when low hydrogen partial pressures prevented solids accumulation in the dissolver.

The true effect of total pressure on hydrogen-donor quality is difficult to evaluate. The hydrogen content of the process solvent increased from 8 to 9% after the pressure was increased to 2,400 psig in Run 133 (Ref. 1). However, the hydrogen content remained at about 9% during the runs subsequent to Run 133 when the dissolver pressure was less than 2,400 psig. NMR data provided by Air Products' Laboratory (Ref. 6) revealed a shift in hydrogen distribution from aromatic to nonaromatic species as a result of the high-pressure operations.

Run No	Pressure total, psig	H_2, psi	Solvent: Atomic Ratio, H/C	Solvent: SG 60°F	Solvent: Aromatic Hydrogen / Nonaromatic Hydrogen	Microautoclave THF Conversion, % (Short)
125 MB	1,700	700	1.09	1.05	0.64	78
131 MB	1,700	500	1.13	1.05	0.58	78
133 MB	2,400	1,200	1.23	1.01	0.42	81

Nonaromatic hydrogen includes donatable hydrogen as well as aliphatic hydrogen. The increase in the nonaromatic hydrogen at 2,400 psig could be due to:

- o Saturation of aromatic rings to form naphthenes,
- o Hydrogenation of aromatic rings to form hydroaromatic donor molecules,
- o Opening of aromatic rings to form aliphatic chains on the aromatic compounds,
- o Cracking of highly-saturated SRC molecules.

All runs made with Indiana V coal since July 1977 produced enough solvent to eliminate the need for startup solvent (unhydrogenated anthracene oil). The solvent produced from the coal is only 1 to 2% of the daily in-plant process inventory. The solvent hydrogen content has remained high, even at low hydrogen partial pressures:

Run No	Pressure total, psig	H_2, psi	Solvent: Atomic Ratio, H/C	Solvent: SG 60°F	Solvent: Aromatic / Nonaromatic	Microautoclave THF Conversion,% (Short)
136 MB	2200	1400	1.30	1.01	0.34	77
138 MB	1700	700	1.26	1.01	0.40	74
140B	2400	1400	1.23	1.02	0.39	75
143 MB	1700	1100	1.21	1.01	0.34	77
147 MB	1700	1100	1.27	1.01	0.35	75

Microautoclave studies were made to determine the hydrogen-donor activity of various boiling range fractions of the process solvent. The results for various solvent fractions from a high quality hydrogen-donor solvent (Run 133) and a low quality hydrogen-donor solvent (Run 143) were:

Date, 1978	8 February		3 August	
Run	133		143	
	wt % of total	THF[a] conversion, %	wt % of total	THF[a] conversion, %
Process solvent boiling fraction				
IBP-550°F	56.1	65	44.6	57
550-650°F	21.2	80	30.6	73
650°F-EP	22.7	79	24.8	76
Whole process solvent	100.	78	100.	69

(a) Microautoclave long run conditions.

The hydrogen-donor quality of the solvent decreases as the boiling range decreases. Even in the high quality hydrogen-donor solvent (Run 133), the IBP-550°F fraction had a relatively low activity. Therefore, the lower boiling fractions may contain more saturated compounds (naphthenes).

Another indication of increased hydrogen-donor activity with increased solvent boiling range was obtained by blending the process solvent with the light SRC fraction from the third stage of the Kerr-McGee CSD unit. The light SRC apparently has a greater hydrogen transfer rate than the process solvent. Even though the hydrogen-donor concentration may not be as high in this material, the hydrogen transfer rate may be so much greater that a significant increase in the conversion rate results:

Material	Microautoclave THF conversion, % (a) short	Microautoclave THF conversion, % (a) long
Process solvent (1 Sept 1978)	71	71
25% blend of KM-CSD Stage III[b] light SRC and process solvent	75	68
50% blend of KM-CSD Stage III[b] light SRC and process solvent	78	61
Plus 550°F cut from process solvent (1 Sept 1978)	77	72
50% blend of KM-CSD Stage III[b] light SRC and plus 550°F cut from process solvent	81	68
50% blend of oil[c] from KM-CSD Stage III[b] light SRC and plus 550°F cut from process solvent	81	68

(a) Using standard coal which gave 74% conversion with 25% tetralin in 1-methylnaphthalene.
(b) Kerr-McGee Technical Center sample containing 20% distillate @ 600°F, 0.1 mm Hg absolute.
(c) Pentane soluble.

It should also be considered that the higher boiling fractions may have a greater ability to dissolve the coal fragments since they are more similar to them. A stable complex between the coal fragment and the heavy solvent molecules may be formed. Minimal hydrogen transfer may take place in the transition state.

d. Coal Conversion and SRC Yield

Coal conversion during the material balance periods was 94.2% of the MAF coal during Run 143 and 92.2% during Run 147 (low solids inventory). The solids apparently catalyze conversion, since the same trend was noted for Run 134 (low solids inventory) compared to Run 133 (Ref. 1).

Variations in apparent SRC yields, based on analyses of V110 Flash Tank samples, may indicate changes in dissolver performance. The following data compare daily SRC yields (based upon V110 samples) during Runs 143 and 147:

Run 143		Run 147	
Date, 1978	SRC yield,[a] % MAF coal	Date, 1978	SRC yield,[a] % MAF coal
31 July - 1 August	59.4	4-5 September	60.2
3-5 August	58.1	6 September	60.4
6-9 August	62.9		
10-13 August	55.4	7 September	56.6
15-19 August	54.1	8 September	62.7
20 August	53.2		
21 August	55.1		

(a) $\text{SRC yield, \% MAF coal} = 100 \times \frac{(\text{\% ash in MF coal}) \times (\text{\% SRC in V110})}{(\text{\% ash in V110}) \times (100 - \text{\% ash in MF coal})}$

2. Yields

Table 17 shows reaction section operating data for material balance periods. Table 6 shows the calculated yields for Runs 143 and 147. Two methods were used to calculate yields: process material balance, and V110 short method (Ref. 5). The following data show the apparent errors in the elemental balances based upon each method:

Element	% Error[a] by Element			
Run	143		147	
Method	process	V110	process	V110
Carbon	- 0.65	- 0.27	- 0.75	- 0.62
Hydrogen	- 1.39	- 0.77	- 1.33	- 0.83
Sulfur	2.54	0.90	-11.76	-12.76
Nitrogen	36.82	36.69	5.08	3.64
Ash	5.56	- 1.85	6.55	3.47
Oxygen[b]	- 2.16	- 2.27	8.18	8.33

(a) Based upon input. Positive error means the element was not completely accounted for in the output streams.

(b) By difference.

Table 6 also shows the yields calculated from adjusted process flow rates which produced elementally balanced results (Ref. 5).

a. Yield of SRC

The yield of SRC was not significantly higher in Run 147 (with low dissolver solids concentration) than in Run 143:

Run	143		147	
	Process MB[a]	V110[a] Short method[a]	Process MB[a]	V110[a] Short method[a]
Yield, % MF coal				
Oil	12.2	14.0	11.8	12.1
Asphaltene	22.1	21.7	17.8	25.5
Benzene insoluble	12.6	12.2	17.7	11.8
Total SRC	46.9	47.9	47.3	49.4

(a) Elementally balanced

b. Yields of Organic Distillates, Water and Gases

The process solvent yield was higher in Run 143 (with higher dissolver solids concentration) than in Run 147. The yields of light and wash solvents were similar, as shown below:

Run	143		147	
	Process MB[a]	V110 Short method	Process MB[a]	V110 Short Method
Yield, % MF coal				
Light organic liquids	3.2	3.1	3.9	3.9
Wash solvent	4.0	4.0	4.1	4.0
Process solvent	17.5	16.6	12.6	10.5

(a) Elementally balanced.

There was a net increase in process solvent during Runs 143 and 147.

The yield of water was higher in Run 147 than in Run 143:

Run	143	147
Water yield, % MAF coal	6.4	9.8

Hydrocarbon gas yields and the hydrogen consumptions are shown in Table 6. Variations in dissolver solids concentration did not noticeably affect the hydrocarbon gas yields. Based on elemental balances, the hydrogen consumption was 3.0% of the MF coal in Run 147 and 2.7% in Run 143. Ethylene tracer gas tests during Runs 143 and 147 indicated that leakage rates (through valves tied into the flare header) were insignificant.

Typical gas chromatographic analyses of the various product vent gases are given in Tables 18, 19, and 20. Analyses of the total flare gas are given in Table 21.

Table 22 shows analyses of reaction products from V110 Flash Tank composites for material balance periods. These, along with data from Tables 17 through 21, were used to calculate the short reaction section material balances.

3. Heteroatom Removal

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

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

where:

A = heteroatom to carbon ratio in feed coal

B = heteroatom to carbon ratio in SRC

Another equation used to calculate heteroatom removal is:

$$\text{Removal, } \% = (100\,A - BC)/A \qquad (2)$$

where:

A = heteroatom % in coal

B = heteroatom % in SRC

C = yield of SRC, % MF coal

The values calculated are:

Run	Heteroatom	Weight Ratio of Heteroatom to Carbon: Feed coal	V110 SRC	KM-SRC	Heteroatom Removal, %: Eq. (1) V110 SRC	Eq. (1) KM SRC	Eq. (2) V110 SRC	Eq. (2) KM SRC
143	Oxygen	0.157	0.058	.031	63	80	78	89
	Nitrogen	0.017	0.015	.02	12	-18	49	41
	Sulfur	0.052	0.008	.009	83	83	90	91
147	Oxygen	0.192	0.049	.036	74	81	84	91
	Nitrogen	0.017	0.020	.021	-18	-24	26	41
	Sulfur	0.055	0.009	.009	84	84	89	92

In the Kerr-McGee CSD process, the components of the feed SRC are distributed between ash concentrate, deashed SRC and light SRC, depending upon their molecular weights. Due to this separation, the oxygen contents of V110 SRC differ from those of KM-SRC. The sulfur and nitrogen contents, however, do not differ significantly. The sulfur in SRC did show a slight decrease from 0.81 to 0.75% for Run 147 with the low recovery of SRC (73%). The removal of oxygen is higher for KM-SRC than for V110 SRC because of the loss of the polyfunctional SRC fractions to the ash concentrate in the KM unit.

Organic sulfur removal (OSR) can be calculated by the following equation based on the calorific value of coal and SRC:

$$\text{OSR, \%} = (A/C - B/D) \div A/C \qquad (3)$$

where:

A = Organic sulfur in feed coal, wt %

B = Organic sulfur in SRC, wt %

C = Calorific value of coal, Btu/lb

D = Calorific value of SRC, Btu/lb

Organic sulfur removals calculated by this equation were:

Run	Hydrogen Partial Pressure, psia	Temp, °F	Hydrogen Consumption, % MF coal	Organic Sulfur: in coal, %	in V110 SRC, %	in KM SRC, %	Organic Sulfur Removal, %: V110 SRC	KM SRC
143	1130	823	2.7	1.73	0.74	0.74	65	63
147	1080	825	3.0	2.41	0.81	0.75	70	72

4. Heat of Reaction

The temperature rise from the preheater to the dissolver outlet is a measure of the heat evolved as a result of such exothermic reactions as:

- hydrogenation of the solvent and dissolved coal,
- hydrocracking of SRC and solvent into lower molecular weight compounds, and
- formation of water and gases.

The heats of reaction in the transfer line and in the dissolver can be estimated from an energy balance around these sections. Estimated heats of reaction were:

Run	Coal feed, MF lb/hr	Slurry conc, % MF coal	Btu/lb H_2 consumed	Btu/lb MF coal
143	468	38.2	8,420	205
147	447	38.0	9,990	266

Heat losses from the dissolver and the transfer line were estimated from operating data using solvent and hydrogen gas flow. It was assumed that any reactions between solvent and hydrogen are adiabatic.

Representative heat loss rate data during this quarter were as follows:

Date, 1978	2 September
Dissolver volume, %	100
Rates	
Solvent, lb/hr	1,250
Hydrogen, lb/hr	60.4
Pressure, psig	1,700
Outlet temperature, °F	
Preheater	851
Dissolver	799
Estimated heat loss, Btu/hr	60,000

E. Mineral Residue Separation

1. Filtration

Tests to determine whether Solka-Floc (a cellulose fiber) could replace asbestos were not conclusive due to erratic filter operation. During the 24 July test, using 37 screens of 60 x 60 mesh, filtrate clarities were poor (0.1 to 2.4% CI). Asbestos fibers were added, but filtrate clarities improved only to 0.7 from 1.1% solids. Between 1 and 3 August the tests were repeated using 21 screens of 60 x 60 mesh. Starting with 7.5% Solka-Floc in Hyflo Super-Cel, poor clarities (2.5 to 3.8% CI) were obtained on the first four cycles. Asbestos (10% of precoat) was added. Better filtrate clarities were obtained on the next 19 cycles. Several of these cycles, however, had high filtration rates and poor clarities, indicating leakage into the filtrate. Table 23 shows the operating data for these test periods.

Figures 9 and 10 show viscosity and specific gravity curves for reaction products for each material balance period. The reaction product was bypassed around the filtration section to the vacuum distillation section during these runs to test the KM-CSD unit.

Sonic probe tests were conducted to evaluate the value of the probe for measurement of solids in the filtrate. To be useful, the instrument must be sensitive enough to respond to 0.04% solids. The instrument's sensitivity was not satisfactory. The tests will be repeated using a modified probe.

Work was done in preparation for testing the USF vertical-leaf filter unit to be carried out during the fourth quarter of 1978. A summary of the planned USF filter unit cycle is presented in Appendix C. Appendix Figure C-1 gives the valve identification list for the USF filter system.

Two modes of operating the USF filter are planned. In the "precoat" mode, the operating procedure will be:

1. precoat with filter aid
2. filter
3. wash with wash solvent
4. blow nitrogen through cake to remove as much liquid as possible
5. depressurize
6. dry by vacuum flashing

7. discharge cake

8. repressurize

9. sluice filter leaves with filtrate

In the "no precoat" mode of operation, the procedure will be:

1. build initial cake by filter feed recirculation

2. filter

3. wash with wash solvent

4. blow nitrogen through cake to remove as much liquid as possible

5. depressurize

6. dry by vacuum flashing

7. discharge cake

8. repressurize

9. sluice leaves with filter feed

F. Solvent Recovery

1. T102 Vacuum Column

The vacuum column operated without major problems throughout the third quarter. Performance data during material balance Runs 143 and 147 are shown in Table 24. During the quarter the operating pressure of the column was varied over a range of 0.4 to 1.4 psia. Separation of solvent from SRC depends on the pressure in the column which is limited to a minimum of 0.3 psia.

The column overhead temperature was adjusted to keep the amount of light organics fed to the solvent fractionation column (T105) less than 3%, while maintaining a maximum allowable flow to the light ends separation column (T104).

Summary operating and performance data from the material balance periods were:

Run	143	147
T102 pressure, psia	1.3	1.2
T102 bottom temperature, °F	598	596
Overhead temperature, °F	192	193
Distillate in SRC, @ 600°F, 0.1 mm Hg, wt %	7.3	5.5
Melting point, °F	356	390
IBP-350°F in T105 feed, wt %	1.8	1.9

2. T105 Solvent Fractionation Column

The solvent fractionating column operated whenever required throughout the third quarter. Performance data obtained during the material balance periods of Runs 143 and 147 are shown in Table 25. Tables 26 and 27 show product analyses and compositions.

Figure 11 shows ASTM D-86, D1160, and GC simulated true boiling point (TBP) curves for the process solvent. The D1160 data were corrected to atmospheric pressure using the method shown in Appendix D. Figure 12 shows ASTM D-86 and GC simulated TBP data for the wash solvent.

Operation of the fractionating column during periods when the Kerr-McGee CSD unit was on-stream was adjusted to compensate for the reduction in wash solvent inventory (350 to 450°F cut). A 4-hour test of the column was completed during this quarter while the CSD unit was operating.

V105 Decanter and T102 overhead oil, which is normally fed to the Light Solvent Recovery Column, was blended into the fractionator feed stream. The column was operated for about 4 hours at the following conditions:

Feed rate	1.4 gpm
Reflux rate	3.4 gpm
Reboiler vapor temp	555°F
Bottoms temp	547°F
Top temp	425°F
Reflux temp	298°F

The column operated as expected and yielded process solvent with the following analyses:

	IBP °F	<350 °F (%)	350-450 °F (%)	>450 °F (%)
Feed	310	0.4	9.3	90.3
Process solvent	446	0.0	0.2	99.8

The overhead product from the fractionator was accumulated to feed the Light Solvent Recovery Column, T104. In a separate distillation operation, light organic liquid (IBP-350°F) and wash solvent (350-450°F) were separated.

The fractionating column was inspected in September. Corrosion of the 304 SS liner in the lower section was severe. Stress cracking was observed. Many of the valves in trays 11 through 20 (lower section of column) were missing. The top tray was unaffected, however.

3. T104 Light Solvent Recovery Column

The light solvent recovery column operated whenever required during the third quarter. Tables 28 and 29 show the material balance compositions of the V105 Decanter and T102 overhead oil which make up the feed to the T104 column. Performance data which were obtained during material balance periods are shown in Table 30. The column was operated without reflux at feed rates ranging from 115 to 403 lb/hr. Tables 31 and 32 show light organic product analyses and compositions. ASTM D-86 and GC simulated distillation curves for the light organic liquid product are shown in Figure 13. Figure 14 shows a vapor pressure curve for the light organic liquid. A special distillation run was carried out in an effort to generate a process-derived organic liquid fraction which could be used as an additive or blend for the Kerr-McGee deashing solvent. An evaluation of the organic liquid fraction by the Kerr-McGee Technical Center was underway at the end of the third quarter.

4. Solvent Inventory

The solvent inventories increased during the third quarter, as shown below:

Inventory, M lb	1 July	30 Sept	Change	Composition,[a] wt % IBP-350 °F	350-450 °F	450 °F-EP
Light organic liquid						
in tanks	9.2	27.5				
shipped during qtr	-	14.0				
total	9.2	41.5	32.3	39	56	5
Wash solvent	30.3	46.2	15.9	2	96	2
Process solvent						
in tanks	80.8	62.1				
in drums	-	24.4				
shipped during qtr	-	11.8				
total	80.8	98.3	17.5	-	6	94

(a) ASTM D-86

The light organic liquid product in V124A tank was inadvertently mixed with wash solvent from V124B tank during 7 and 8 August when a valve was left open. Prior to this upset, the light organic liquid product contained about 1% of material boiling above 350°F.

The wash solvent inventory increased significantly because the filter system was not in operation during most of the period.

Figure 15 shows the trends in solvent inventory during the operating period.

The wash and process solvent inventories during each run were as follows:

Run	Date	Process Solvent Inventory, lb	Wash Solvent in process solvent		Wash Solvent Inventory, lb	Process Solvent in wash solvent	
			ASTM D-86 vol, %	GC wt, %		ASTM D-86 vol, %	GC wt, %
141	6 July to 16 July	87,900 81,400	0.0	-	24,900 25,800	1.0	-
142	20 July to 30 July	78,500 86,700	2.5	6.1	36,800 35,900	-	4.0
143	30 July to 22 August	86,700 102,800	-	4.4	35,900 32,200	-	8.1
144	22 August to 28 August	102,800 106,200	-	9.1	32,200 36,600	0.0	-
145-146	29 August to 30 August	106,200 94,300	1.0	7.5	36,600 36,800	2.0	-
147	4 September to 9 September	89,300 93,500	-	4.3	38,900 39,200	-	8.8
148-149	29 September to 30 September	87,600 86,500	-	7.2	44,900 46,200	1.4	1.8

5. Process Solvent Composition

Typical process solvent compositions during the period were as follows:

Coal	Indiana V					Kentucky 6 and 11
Run	140B	142	143	143 MB	147 MB	148
Date, 1978	2 July	26 July	3 August	20 August	7 Sept	29 Sept
Boiling Fractions, wt %						
IBP-450°F	1	10	9	7	5	7
450-550°F	53	40	51	55	51	48
550-650°F	31	27	23	25	26	26
650°F-EP	15	23	17	13	18	19
Carbon %	86.67	87.63	86.90	86.54	86.83	86.60
Hydrogen %	8.81	8.45	9.18	9.23	9.10	9.07
H/C atomic ratio	1.21	1.15	1.26	1.27	1.25	1.25
Specific gravity	1.017	1.016	1.010	1.006	1.007	1.008

The process solvent showed a decrease in high boiling components (650°F-EP) during Run 143 compared to Run 142. The hydrogen content increased and the specific gravity decreased. The process solvent had a ratio of 0.35 to 0.4 aromatic hydrogen to aliphatic hydrogen based on NMR data supplied by Air Products and Chemicals, Inc. (Ref. 6). The process solvent from earlier processing of Indiana V coal prior to using a 2,400 psig operating pressure had an aromatic to aliphatic ratio of 0.60 to 0.65. The specific gravity was higher (1.03-1.06) and the hydrogen content lower (8.0-8.2%) than those of the solvent obtained after using a 2,400 psig operating pressure. Operating at low hydrogen partial pressure may result in greater production of aromatic solvent compounds from the coal.

G. SRC Critical Solvent Deashing

Ash-containing vacuum still bottoms were pumped to the Kerr-McGee CSD feed tanks. Feed processing rates ranged from 150 to 300 lb/hr. After equipment performance tests were completed, two process demonstration runs (143 and 147), both including 48-hr material balance periods, were made. The primary objectives of the demonstration runs were achieved during the 48-hr material balance periods, namely:

- SRC containing less than 0.16% ash was produced,
- Ash concentrate was produced in handle-able, powdery form,
- Acceptably low deashing solvent loss to the products was obtained, and
- Continuous operation was maintained.

SRC recovery ranged from 73 to 84% of the cresol-soluble feed. SRC contained 0.06 to 0.75% ash and 0.79 to 0.80% sulfur.

1. Process Stability

During Runs 143 and 144, upsets in the first stage caused the ash in SRC to increase from less than 0.2% to as high as 8%. A typical upset, monitored during the Run 143 MB, is shown in Figure 16. The problem was ash carryover from the ash concentrate processing system which caused contamination of the deashing solvent.

The Run 147 material was easier to deash, but gave a lower yield of SRC than the Run 143 material. It was possible to deash all the product generated during Run 147. This capability is attributed to eliminating contamination in the deashing solvent. During initial runs, an unreliable feed pumping system limited the operability of the unit. This problem has been corrected. Another problem which limited the length of continuous stable operation in the CSD unit was plugging of the vent lines and letdown system associated with processing the ash concentrate.

An area of concern is repolymerization of SRC in the feed because of residence time in the KM feed tanks. A proposal is under consideration to transfer T102 bottoms directly into the CSD unit, bypassing the feed tanks.

During most of this period, the feed rate to the unit was lower than the design rate due to heat exchanger fouling. New heat exchangers were designed and will be installed to:

- increase the fluid velocity through the tubes, and
- increase the total exchanger surface area.

2. Feed Composition

The composition and properties of the feed during material balance periods (143 and 147) were:

Run	143 MB	147 MB
Feed composition, wt %		
Oil	19.9	19.0
Asphaltene	36.0	28.5
Preasphaltene	20.6	28.3
Undissolved coal	6.8	8.5
Ash	16.7	15.7
Feed properties		
Melting point, °F	356	390
Distillate, wt %		
600 °F, 0.1 mm Hg	7.3	5.5
Sulfur, wt %	2.3	2.5

The SRC recovery in the Kerr-McGee CSD process is related to the preasphaltene content of the feed which, in turn, is related to the SRC reaction section conditions and the solvent hydrogen donor quality. The preasphaltene content in the KM-feed may also be related to repolymerization reactions which can occur downstream of the reaction section.

The preasphaltene content and melting point of the feed were higher during Run 147 than during Run 143. The higher preasphaltene content of the feed for Run 147 appears to be due to repolymerization of asphaltenes in the vacuum distillation system and/or the KM-feed tanks:

Run	143		147	
	V110 SRC	KM[(b)] Feed SRC	V110 SRC	KM[(b)] Feed SRC
Composition, wt %				
Oil	29.1	26.0	24.4	25.1
Asphaltene	45.3	47.1	51.7	37.6
Preasphaltene[(a)]	25.6	26.9	23.9	37.3

(a) Cresol soluble
(b) From Kerr-McGee feed tank

Additional study is required to better define and characterize the change in preasphaltene content.

The terphenyl solubility of the KM-feed is another measure of the quality of the feed. The terphenyl solubility is thought to simulate the solubility of the feed in the Kerr-McGee deashing stage. Terphenyl solubility data on the KM-feed for each run were:

Run	143 MB	147 MB
Terphenyl insoluble (TI), % of feed	35.0	42.8
Ash in TI, %	48.9	38.3

The Run 147 feed was less soluble in terphenyl and, therefore, the potential for SRC recovery from the Kerr-McGee CSD unit was reduced.

3. Yields

Normalized yield data for the Kerr-McGee CSD unit were:

Run	143				147			
Feed Rate, lb/hr	273				256			
Yields	% MF Coal		% of Feed		% MF Coal		% of Feed	
SRC	40.8		64.8		35.9		55.2	
oil		10.8		17.1		10.0		15.3
asphaltene		23.2		36.9		22.3		34.2
preasphaltene		6.3		9.9		3.5		5.5
undissolved coal		0.1		0.2		0.0		0.1
ash		0.3		0.5		0.0		0.0
deashing solvent		0.1		0.2		0.1		0.1
Ash Concentrate	22.5		35.6		29.3		45.1	
oil		1.4		2.2		1.4		2.2
asphaltene		1.3		2.0		1.0		1.6
preasphaltene		4.4		7.1		9.8		15.1
undissolved coal		5.0		7.9		6.8		10.5
ash		10.3		16.2		10.1		15.5
deashing solvent		0.1		0.2		0.2		0.2

The material balance closures were <1% and 6%, respectively, for Runs 143 and 147.

SRC component balances for each run were:

Run	143					147				
	KM Feed	KM - Products Ash conc.	SRC	Total	% of feed Comp.	KM Feed	KM - Products Ash conc.	SRC	Total	% of feed Comp.
Component, lb/hr										
Deashing solvent		0.52	0.62	1.14			0.56	0.43	1.01	
Oil	54.26	6.13	46.87	53.00	97.7	48.74	5.68	39.14	44.82	92.0
Asphaltene	98.29	5.42	100.33	105.75	107.6	72.95	4.08	87.57	91.65	125.6
Prepashaltene	56.14	19.20	27.15	46.35	82.6	72.46	38.70	13.98	52.68	72.7
Ash	45.56	44.29	1.29	45.58	100.0	40.29	39.67	0.17	39.84	97.6
Undissolved coal	18.55	21.56	0.53	22.09	119.1	21.70	26.95	0.21	27.16	125.2
Total	272.80	97.12	176.79	273.91	100.4	256.14	115.66	141.50	257.16	100.4

The output streams were normalized to 100-plus the deashing solvent in the products. The ash balance closures reflect the accuracy of the overall balances. The individual component balances show some maldistribution of asphaltenes, preasphaltenes, and undissolved coal. It is not known how much the differences are due to actual compositional changes attributable to the Kerr-McGee CSD process. Some of the error is imprecision in the analytical procedures. The interaction of components affects the solubility of a component in a specific solvent such as pentane, benzene, or cresol. Therefore, SRC compositional changes due to separation of the feed into ash concentrate and SRC products may affect the laboratory extraction results.

Elemental balance errors for each normalized material balance were:

Run	143 MB	147 MB
Error, % of element		
Carbon	- 0.27	0.07
Hydrogen	- 0.32	0.27
Nitrogen	8.12	4.10
Sulfur	- 2.02	- 0.71
Ash	- 0.06	1.12
Oxygen	7.52	- 6.63

The program for elementally balancing the reaction yields is being modified for the Kerr-McGee CSD unit.

4. Ash Separation

a. Ash Concentrate Processing

Ash concentrate from the underflow of the first stage of the Kerr-McGee CSD unit is discharged continuously from the ash processing systems. The percentages of the feed reporting to the ash concentrate discharge for each material balance run follows:

Run	143	147
Ash concentrate, % of feed	35.6	45.1
Ash in ash concentrate, %	45.5	34.3

The lower ash content of the ash concentrate in Run 147 was primarily due to the increased quantity of preasphaltenes in the ash concentrate. This was due partly to the increase in the preasphaltene content of the feed, and also to the higher molecular weight distribution in the preasphaltenes:

Run	143	147
Preasphaltenes, lb/hr		
in feed	56.14	72.46
in ash concentrate	19.20	38.70
difference	36.94	33.76
Preasphaltene, % of ash concentrate	19.77	33.46

The ash concentrate was discharged as a light powdery material with an approximate bulk density of 18.5 lb/ft^3. Analyses of this material are shown in Table 33.

b. SRC Recovery

The SRC recovery decreased for Run 147 compared to 143 due primarily to the difference in composition (higher preasphalten content) of the feed:

Run	143 MB	147 MB
SRC recovery (a)		
by normalized material balance	83.5	72.5
by forced ash balance	82.8	71.4

(a) Percent of cresol soluble feed recovered in SRC and light SRC products.

The recoveries for individual SRC components were:

Run	143			147		
	KM-feed	KM-Ash conc	SRC Recovery, %	KM-feed	KM-Ash conc	SRC Recovery,
Component, lb/hr						
Deashing solvent		0.52			0.58	
Oil/asphaltene	152.55	11.55	92.4	121.69	9.76	92.0
Preasphaltene	56.14	19.20	65.8	72.46	38.70	46.6
Undissolved coal	18.55	21.56		21.70	26.95	
Ash	45.56	44.29		40.29	39.67	
Total	272.80	97.12	83.8	256.14	115.66	72.3

The overall SRC recovery is calculated by subtracting oil, asphaltene, and incremental undissolved coal rates for the ash concentrate stream from the component feed rates. This corrects for any loss of SRC due to formation of undissolved coal by repolymerization in the Kerr-McGee CSD unit.

Recoveries of oil and asphaltene fractions of the SRC were about 92%. However, as the asphaltene/preasphaltene ratio in the feed decreased from 1.75 to 1.01, the preasphaltene recovery decreased significantly. Methods available for increasing the recovery of SRC from the Kerr-McGee CSD unit are:

- Change operating conditions in the SRC process
- Decrease KM-feed residence time (prior to processing)
- Change operating conditions in the CSD process.

Analyses of the ash concentrate from each material balance showed that 9 to 12% of the material is potentially soluble in the deashing solvent:

Run	143 MB	147 MB
Composition of ash concentrate, wt %		
Oil	6.8	5.4
Asphaltene	5.6	3.5
Preasphaltene	19.8	33.5
Undissolved [(a)] coal	22.2	23.3
Ash	45.6	34.3
Distillate, [(b)] wt %	5.5	5.2

(a) Cresol insoluble.
(b) 600°F, 0.1 mm Hg.

A system for recovery of soluble material in the ash concentrate may be added to the Kerr-McGee CSD unit later.

5. Light SRC Separation

Light SRC is a mixture of distillable process solvent and SRC fractions that can be recovered as a separate product from the third stage of the CSD unit. During the quarter, the CSD process was operated as a two-stage system. However, some material was sampled in the third stage to determine the future potential for recovering process solvent for the SRC process. Analyses of this material were:

Run	143 MB	147 MB
Light SRC recovery, % of SRC	3.5	13.2
Light SRC distillate,[a] wt %	9.3	20.0
SRC[b]		
distillate, wt %	10.0	6.1
melting point, °F	282	317

(a) 600°F, 0.1 mm Hg absolute.
(b) From second stage.

The increase in solvent content of the light SRC and the increase in the melting point of the SRC in Run 147, as compared to Run 143, are related to adjustment of the pressure and temperature in the CSD second stage. Further adjustment of the second stage conditions after the Run 147 material balance produced a light SRC containing 42% distillable solvent.

Elemental analyses of the light SRC from each run were:

Run	143 MB	147 MB
Element, wt %		
Carbon	88.68	86.51
Hydrogen	6.11	6.68
Nitrogen	1.70	1.57
Sulfur	0.70	0.91
Ash	0.21	0.51
Oxygen	2.60	3.82

6. Deashing Solvent Recovery

The sources of most of the deashing solvent losses were determined. Approximately 0.4% deashing solvent (based on feed input) was lost to the product streams: ash concentrate, SRC, and water. This loss is controlled primarily by the present equipment limitations. The major portion of total loss is due to deashing solvent discharge through the ventilation systems.

Run	143 MB	147 MB
Deashing solvent loss, % of feed		
Total	5.9	4.7
To products	0.4	0.4

Deashing solvent is lost primarily to the atmosphere during discharge of the products. This loss can be prevented by venting and condensing the deashing solvent in a sealed system. Prior to Run 147, the Kerr-McGee CSD unit was operated on total recycle with no discharge of products. During this period, deashing solvent losses ranged from 0.6 to 0.9% of the feed. About 50% of these losses were due to venting.

The deashing solvent contained small amounts of ash and other contaminants. The concentration of contaminants will vary depending upon the deashing solvent makeup rate. The makeup was 12 to 16 lb/hr during the demonstration runs. With total recovery of non-product losses, this can be reduced to 1 to 2 lb/hr. Therefore, the deashing solvent may not be at "equilibrium" concentration since the deashing solvent losses can be greatly reduced by a sealed ash concentrate processing system.

Further modifications in the CSD unit are required in order to operate with a recycle deashing solvent composition comparable to that which would be used in a commercial operation. The extent to which this can affect the performance of the CSD process must be determined.

7. Product Analyses and Properties

SRC from V110 and the deashed SRC were extracted with benzene. Benzene was removed by distillation and the residue was extracted with pentane. The data indicate that KM deashed SRC had a lower melting point and preasphaltene content than SRC from the reaction section:

Run	143		147	
Sample location	V110	KM	V110	KM
Material	SRC	Deashed SRC product	SRC	Deashed SRC product
Composition, wt %				
Oil	29.0	26.9	24.3	25.5
Asphaltene	45.1	56.7	51.4	62.9
Preasphaltene	25.6	15.3	23.8	11.4
Undissolved[a] coal	0.1	0.3	0.2	0.1
Ash	0.2	0.8	0.3	0.1
Sulfur, wt %	0.75	0.80	0.85	0.79
Distillate, wt %				
500 °F, 0.1 mm Hg	-	1.5	-	2.2
600 °F, 0.1 mm Hg	-	10.0	-	6.1
Melting point, °F	327	282	345	317

(a) Cresol soluble

Figure 17 shows viscosity curves at various shear rates for the KM deashed SRC products for each run.

The composition of the KM-SRC appears to be relatively constant.

The pentane-soluble oil from the KM-SRC was analyzed on the Perkin-Elmer Sigma 3/10 Gas Chromatograph System. About 25 to 30% of the oil is distillable at 600°F, 0.1 mm Hg. Only about 20% of the oil was eluted. The eluted portion of the oil had the following composition:

IBP, °F	548
Boiling component, wt %	
548-739°F	11
Pyrene	2
741-837°F	17
Chrysene	9
839-936°F	29
Perylene	5
938-952°F	28
EP, °F	952

H. Product Solidification

A portion of the high-ash SRC (T102 bottoms) was deashed and solidified in the Kerr-McGee CSD unit. The remainder was processed in the K125 Rexnord Product Solidifier. Based on the pilot plant production, the percent of SRC processed in the Kerr-McGee unit increased from 28% in July to 54% in September. There were no major problems associated with the product solidification. The Aercology fume abatement system has not performed satisfactorily, in that only a portion of the fumes are captured and returned to the process solvent (in the order of 1 lb/hr). Also, the fume exhaust system appears to be undersized, which results in lack of fume containment during SRC solidification.

V. MECHANICAL PERFORMANCE

A. Overview

Maintenance activities were routine in nature during the third quarter of 1978. The Kerr-McGee CSD unit operated without major maintenance work. Minor problems occurred with feed pumps, first and second stage letdown lines, pump packings, and plugging of the vent system.

B. Agitators

All plant agitators operated with only minor packing adjustments.

C. Centrifuges

The Sharples centrifuge was not operated during the period.

D. Compressors

During the September shutdown, all compressors were inspected and found to be in excellent condition. The hydrogen compressors have operated for over a year without packing and rod maintenance. The packing continues to be in excellent condition.

E. Dryers

The D201 Instrument Air Dryer is undersized (150 scfm) for its present service. A larger 250 scfm dryer has been ordered and delivery is expected in mid-November.

F. Fired Heaters

Because of voltage fluctuations in July, the heaters tripped out on several occasions. To eliminate these fluctuations, constant voltage transformers were installed. It was found that most shutdowns followed fuel deliveries when the storage tank was agitated during refilling. The tank was emptied and water and sludge were removed.

During the September shutdown, all of the heater burners were cleaned and inspected. A smaller burner was installed in B102 Slurry Preheater, which improved control of the heater temperature.

G. Heat Exchangers

Heat exchanger plugging problems occurred in the Kerr-McGee CSD unit.

H. Filter

The F103 Funda filter was removed from the pilot plant on 24 August. The area is being prepared for installation of a U. S. Filter Corporation vertical-leaf filter unit.

I. Pumps

The P111A and B Filter Feed and P143 Precoat pumps, coated with the Eutectic 6715 hard coatings, operated at 1,750 rpm from the January 1978 startup until August. At 1,750 rpm, these pumps showed little wear and the seal life was improved.

Pumps in the Kerr-McGee unit have had some packing problems. Changing the style of packing and method of packing pumps has greatly improved the operation.

The P102C Slurry Feed Pump seal failed after approximately 8,000 hours of maintenance-free operation.

The causes of major pump failures during the period were:

SRC Unit

Seals	28
Packing	2
Bearings	10
Gasket	1
Motor Bearings	1
Ball check	2
Total	44

Kerr-McGee Unit

Packing	8
Seals	4
Bearings	2
Ball check	3
Total	17

J. Valves

LV415A and B High Pressure Letdown Valves continued to operate without a trim change. The trim sets currently in use were installed on 22 February 1978.

Plugging occurred in the first and second stage letdown lines of the Kerr-McGee unit. This was due to the very small size of the system.

K. Reactors, Towers, and Columns

During the September shutdown, corrosion coupons were removed from plant reactors, towers, and vacuum columns. Inspections were made and samples were taken from the corrosion coupons. Vessel wall thicknesses were also checked by ultrasonics. T105 Solvent Fractionation Column showed evidence of corrosion in the unclad sections. All other process vessels were in excellent condition.

VI. PROJECTS

A. Project 4117 - Waste Water Treatment

Construction of the waste water treatment facility began on 23 September. All major equipment orders have been placed. The biological unit is scheduled for shipment during the week of 30 October. Target completion date is early December.

B. Project 4120 - V144 Blowdown Tank Mixer

This project was completed in September.

C. Project 4122 - New Ash Cooler

Delivery of the new ash cooler has been delayed until late November. Target completion date is mid-December.

D. Project 4123 - Kerr-McGee Stage III

Construction work is in progress. The process and utility tie-ins were completed during the September shutdown. Scheduled completion date is 6 October.

E. Project 4125 - U. S. Filter Corporation Pressure-Leaf Filter

Construction work is in progress. The filter was installed on 14 September. Scheduled completion date is 13 October.

F. Project 4126 - New On-Line Gas Chromatographs

This project was completed in July.

G. Project 4127 - Lawrence Slurry Pump

This project is approved. It provides for the installation of a fully-lined, horizontal, high-temperature, high-pressure slurry pump manufactured by Lawrence Pump, Inc. This pump will be installed in filter feed service. The pump is scheduled for shipment in mid-December.

H. Project 4128 - Weigh Cells for V110 Flash Tank

This project is approved. It provides for the installation of three weigh cells under the V110 Flash Tank and support electronic equipment. This system is designed to improve the accuracy of measuring the flow rate of material leaving the reaction system of the pilot plant. The delivery of the weigh cells and associated equipment is scheduled for mid-October.

I. Project 4129 - New V141 Precoat Tank

This project is approved. It provides for the installation of a new precoat tank with sufficient volume to precoat the USF unit. Delivery of the new precoat tank is scheduled for 6 October.

J. Project 4130 - New Instrument Air Dryer

This project is approved. It provides for the installation of a 250 scfm air dryer to meet increasing plant demand. Completion is scheduled for early December.

VII. CONCLUSIONS

While operating with the full dissolver at 1,700 psig and 825°F, it was found that reducing the inventory of dissolver solids from 37 to 5 lb/ft^3 had little effect on yield structure or on SRC desulfurization. Solvent production was slightly higher and water production was lower at the higher dissolver solids concentration. The solids do appear to catalyze coal conversion and solvent hydrogenation reactions. Solvent hydrogen-donor quality was low at the lower dissolver solids inventory amd the SRC produced under these conditions was unstable. Polymerization of asphaltenes occurred downstream of the reaction section and reduced SRC recovery in the Kerr-McGee CSD unit. A reduction in the feed SRC asphaltene content from 50 to 37% (with a resultant increase in preasphaltene content) reduced the SRC recovery from 84 to 73%.

The Kerr-McGee CSD unit produced specification-grade SRC at feed rates up to 300 lb/hr. With a hydrogen partial pressure of 1,100 psig at the dissolver outlet and a coal space rate of 25 $lb/hr\text{-}ft^3$, the highest SRC recovery (84% of feed) was achieved with the Kerr-McGee CSD unit. Data indicate that the SRC recovery may be related to solvent hydrogen-donor quality. Lower hydrogen-donor quality solvent apparently produces unstable asphaltenes which repolymerize during the vacuum distillation or in the Kerr-McGee feed tanks. The recovery of preasphaltenes is a function of the concentration of preasphaltenes in the feed. As the asphaltene/preasphaltene ratio in the SRC decreased from 1.75 to 1.01, the preasphaltene recovery decreased from 66 to 47%. SRC product from the Kerr-McGee CSD unit contained 10-15% preasphaltenes over the range of the SRC feed conditions.

Future goals for improving the operation of the Kerr-McGee CSD unit include the following:

- Reduce deashing solvent losses to less than 1% of the feed,
- Minimize contaminants in the deashing solvent,
- Increase the capacity of the process, and
- Maximize recovery of preasphaltenes from the ash concentrate.

VIII. FUTURE PLANS

An extended run of 20 to 30 days with the Kerr-McGee CSD unit is planned to investigate the long-term operability of the unit and to improve SRC recovery. Process solvent inventory will be minimized during the run. Light SRC from the CSD unit may be blended with recycle solvent if necessary to maintain the solvent hydrogen donor concentration. Operating conditions of the CSD unit will be adjusted during the run to maximize SRC recovery.

After completion of the extended Kerr-McGee CSD run, testing of the U. S. Filter pressure-leaf unit will begin. During the testing of this filter, the Kerr-McGee CSD unit will be operated using remelted ash-containing SRC in order to study the recovery of solvent from the light SRC fraction.

REFERENCES

1. Quarterly Technical Progress Report (FE-2270-34) for the period January - March 1978, Southern Company Services, Inc., Project No. 43080 (prepared by Catalytic, Inc.).

2. Quarterly Technical Progress Report (FE-2270-36) for the period April - June 1978, Southern Company Services, Inc., Project No. 43080 (prepared by Catalytic, Inc.).

3. Lockhart, R. W., and Martinelli, R. C., Chemical Engineering Progress, Vol. 45, 139 (1949).

4. Technical Report (RP-713-1)-Task 1: Literature Survey "Investigation of Mechanisms of Reactions Involving Oxygen-Containing Compounds in Coal Hydrogenation", June 1976, prepared by Gulf Research and Development Company for Electric Power Research Institute.

5. Annual Technical Progress Report (FE-2270-31- for the period January - December 1977, Southern Company Services, Inc., Project No. 43080 (prepared by Catalytic, Inc.).

6. R. Skinner, Air Products and Chemicals, Inc., to W. H. Weber, Catalytic, Inc., personal communication, 26 October 1978.

APPENDIX A
OPERATING LOG

July

1. The first of three 24-hr material balances to conclude Run 140 was started at 0001 hr. Seventeen filter cycles were completed. Ash in the product SRC averaged 0.19% and sulfur averaged 0.75%. Operations were smooth.

2. Plugging of the filter limited the number of cycles to 13 for the day. Average ash in SRC was 0.13% and sulfur averaged 0.81%.

4. The material balance ended at 0001 hr. Coal feed was continued, however, to run out the slurry in V101B. R101 was blown down to V144. Four hundred eleven pounds of solids were recovered. The reaction section of the plant was taken out of service at 0500 hr to permit a change from the half- to the full-dissolver point on the dissolver outlet and to clean T101, V106, and R101. The filter and the distillation columns were kept operating.

5. The filter was shut down after completing four cycles.

6. Coal feed was reintroduced at 1702 hr to begin Run 141, intended to be a solvent hydrogenation run. The solids withdrawal system was put into operation. The control valve C_v was 0.025 - - designed to pass 50 lb/hr. A greater withdrawal rate was experienced over the first four hours, so an on-off mode (in the off position 95 to 98% of the time) was used.

7. Excessive hydrogen demand to maintain 85% hydrogen in the feed gas was caused by the on-line GC being out of calibration. Deteriorating control of the withdrawal valve XV4078 was experienced. Transfer of T102 bottoms to the Kerr-McGee CSD unit was started at 1405. The stem and plug of the solids withdrawal valve were found to be severely worn after only 30 hours of operation.

8. XV4078 was taken out of service at 1215 hr.

9. XV4078 was operated (4 seconds open and 25 seconds closed) from 0745 to 0845 hr. Column operations were trouble-free.

10. A series of 17 preheater bypass runs was started.

11. Test D of the series at 11.5% feed gas bypass was ended at 2130 hr. Sonic probe tests were made between 1230 and 2000 hr. The probe is a device for monitoring the solids in the filter discharge. A plugged line from V113 to V141 caused suspension of tests.

12. The high pressure sample for series test G at 40.5% bypass was obtained at 1740 hr.

13. The sonic probe tests were resumed.

14. The filter was shut down as scheduled following completion of the probe tests. Transfer to the Kerr-McGee CSD unit was intermittent to conform to demand. Series test M bypass high pressure sample (100% bypass) was obtained at 1904 hr.

15. Series tests II and LL at approximately 800 lb/hr coal feed and 100% gas bypass were completed. The final test "O" was begun.

16. Coal feed was stopped at 1330 hr to end Run 141. Blowdown solids were 434 lb.

17-19. Inspection and maintenance work was completed. Analysis of the process solvent showed that the desired level of hydrogenation was not achieved in Run 141.

20. Run 142 started at 0530 hr with the introduction of coal feed. Blowdown solids from the previous two runs were added to the coal slurry. The purpose of the run was to provide a high-ash feed to the Kerr-McGee CSD unit.

21. Problems with the B204 Dowtherm Heater caused operational difficulties with T105 Fractionation Column. The first high-ash SRC transfer to the Kerr-McGee CSD unit was made at 0515 hr.

22. A momentary power outage occurred at 1625 hr.

23. Plugging developed in the Kerr-McGee CSD system.

24. Filter operation was resumed. Filtrate clarity was good at first but deteriorated with successive cycles. Addition of asbestos fibers to the precoat was tried unsuccessfully.

25. The filter was shut down. The screens were operable, but 238 lb of solids were removed from the filter body. The screens were cleaned and reinstalled.

26. The distillation columns continued to operate smoothly. Unplugging the Kerr-McGee CSD system was completed and transfer of SRC was started at 2210 hr.

27. Transfers to the Kerr-McGee CSD unit were quite steady. Kerr-McGee CSD plugging problems, however, developed.

28. T102 bottoms were diverted to K125.

29. Transfer to the Kerr-McGee CSD unit was resumed at 1130 hr. A decision was made to reduce R101 pressure from 2,400 to 1,700 psig in an effort to make the SRC more amenable to deashing in the CSD system.

30. The lower pressure was obtained at 0015 hr. Run 143 was begun with no interruption in coal feed. Plugging continued to be a problem in the Kerr-McGee CSD system.

31. T102 bottoms were diverted to K125 pending correction of the Kerr-McGee CSD system problems. The Funda filter was readied for service.

<u>August</u>

1. Filtration was resumed with Solka-Floc and Hyflo Super-Cel as precoat. The first cycles had high cresol-insolubles (CI's) in the filtrate. Asbestos fibers were added to the precoat.

2. Filtrate clarities were improved - down from 3.6% to 0.06% CI's. K125 SRC ash dropped to 0.64%. Distillation column operations were good.

3. Filtrate CI contents became erratic.

4. Substitution of Fibra-Flo 7-C as precoat did not improve filtrate clarity, so the unit was shut down for inspection.

5. Transfer of T102 bottoms to the Kerr-McGee CSD unit was resumed at 0224 hr. There was no transfer between 0700 and 1950 hr, when all material went to K125.

6. The screens were removed from the filter. No holes or tears were found. Twenty-one 150 x 150 mesh screens will be installed for test evaluation.

7. Several power blips occurred during a rainstorm. A number of units tripped out at 2145 hr, but were restarted immediately.

8. The filter reassembly was completed.

9. Tests with the sonic probe were resumed. After completing these, the filter was placed on standby.

10. Additional sonic probe data were obtained.

11. The series of probe tests was completed and the filter was shut down. The distillation columns have been operating trouble-free.

12. An electrical outage occurred at 2115 hr, and shut down the plant. C102 developed a gasket leak.

13. SRC transfer to the Kerr-McGee CSD unit was begun at 0140 hr. Coal feed was reintroduced at 0800 hr. Another power outage occurred at 1415 hr, but caused only momentary equipment downtime.

14. The filter was still on standby. All systems ran well. A pump problem caused some delay in the Kerr-McGee CSD area.

15. There were no problems.

16. There were no problems.

17. A damaged transfer line from the plant to the Kerr-McGee CSD unit was repaired.

18. The Kerr-McGee CSD unit was operated 75% of the day.

19. Plant operations were good.

20. The 48-hr material balance was started as scheduled. Plant and Kerr-McGee CSD operations were steady.

21. Again, plant and Kerr-McGee CSD operations were steady. The material balance ended at midnight.

22. Coal feed was not stopped at the end of the material balance, but was continued at increased R101 pressure as Run 144 began.

23. Emptying and cleaning the Funda filter system was begun to replace it with a USF unit.

24. The Kerr-McGee CSD unit was operated only 50% of the time due to pump and plugging problems. The distillation column operated trouble-free.

25. Approximately two hours of coal feed time were lost because of B102 flame-outs.

26. Continuing B102 flame-outs and an R101 bottom gasket leak required a coal feed outage of 13 hours. Six hundred seventy-five pounds of solids were blown down from R101.

27. The Kerr-McGee CSD unit processed material for 38% of the day.

28. Coal feed was stopped at 0600 hr to end Run 144. One hundred seventy-two pounds of solids were blown down from R101.

29. Two dissolver bypass runs (145 and 146) were made: the first was from 0200 to 1315 hr and the second from 1315 to 2230 hr. The design rate for the first was 300 lb/hr coal feed and the second 600 lb/hr. Lined-out periods were 0600 to 1300 for Run 145 and from 1700 to 2230 hr for Run 146. The reaction product from V110 was placed in drums.

30-31. No coal was fed. The Funda filter was removed from the structure.

<u>September</u>

1. The nuclear density gauges were calibrated and a heat loss test was made.

2. Coal feed to start Run 147 was begun at 0600 hr to duplicate Run 142, but without the addition of dissolver blowdown solids. Accumulated solids will be withdrawn from R101 bottom. Coal feed was suspended at 0925 hr to repair PSV408 on R101 outlet.

3. All PSV's and RO's were checked.

4. Coal feed was resumed at 1300 hr.

5. XV4078 solids withdrawal valve was in service. Transfer of T102 bottoms to the Kerr-McGee CSD unit was not steady because of ash concentrate system plugging.

6. A test program was begun to establish process conditions for wash solvent recovery and improved column stability during long Kerr-McGee CSD runs. This involves improving control through T105. A high pressure sample was obtained at B102 outlet at 2010 hr in anticipation of beginning a material balance data period.

7. Run 147 A MB was started at 0001 hr. Solids withdrawal from R101 was fairly steady. Transfer to the Kerr-McGee CSD unit was also steady despite some plugging in ash concentrate system.

8. XV4078 would not control due to apparent wear.

9. R101 was blown down at the end of the material balance. The recovered solids weighed 97 lb.

10-18. The plant was down to set the USF filter in place and to make tie-ins for the third stage of the Kerr-McGee CSD unit.

19-22. The R101 nuclear density gauges were calibrated with solvent feed in the 300 to 800°F range.

23. At 1020 hr calibration work on the nuclear density gauges was resumed with coal and dissolver solids feed. This was the start of Run 148.

25. Calibration tests were completed. The coal slurry was reduced to 25% concentration to conserve coal pending receipt of a new supply delayed by a strike.

26. Four hours of coal feed were lost due to B102 shutdown caused by a large clinker falling into the burner.

27. A switch to Kentucky 6 and 11 coal was made at 1200 hr. T102 bottoms feed to the Kerr-McGee CSD unit was for only one-half hour - from 1310 to 1340 hr.

28. Some difficulties were experienced in the operation of the new automatic transfer valve controlling T102 bottoms to the Kerr-McGee CSD unit during the first two shifts. Control was good during the third shift.

29. B102 problems (a fuel oil leak and clinker in the fire box) required an interruption of coal feed between 0930 and 1500 hr. Transfer of T102 bottoms to the Kerr-McGee CSD unit was intermittent on each shift due to difficulties with deashing and because the level in V111 was being lowered by diverting the SRC to K125. Bringing the level in V111 to zero provides a surge mechanism to handle transfer variations to the Kerr-McGee CSD unit. Coal feed was resumed at 1500 hr, and Run 149 began.

30. Transfer to the Kerr-McGee CSD unit was such that the level in V111 rose from 2 to 40% in 24 hours. Operations were otherwise relatively smooth.

APPENDIX B
PREHEATER CONVERSION MODEL

A first-order reaction model for coal conversion in B102 Slurry Preheater was developed previously (Ref. B1) based on the preheater gas bypass tests in January, 1978:

$$X_{AI} = X_M \left(1-e^{-K_p \tau_p}\right) \tag{B1}$$

where

X_{AI} = MAF coal conversion at the preheater outlet, fractional, based on cresol-solubles

X_M = Equilibrium conversion at the preheater outlet, fractional, based on cresol-solubles

K_p = Rate constant, hr^{-1}

τ_p = Volumetric residence time, hr

$$= V_e/F = 1.6\ V_L/F \tag{B2}$$

where

V_e = Effective reaction volume occupied by slurry, ft^3 = preheater volume (1.6 ft^3) x V_L

V_L = Fraction of voids occupied by coal slurry,

F = Volumetric flow rate of slurry, ft^3/hr

Since the rate of coal conversion below 600 °F is expected to be low, the reaction volume for the preheater (1.6 ft^3) was taken as that portion of coil which is at a temperature higher than 600 °F.

The liquid hold-ups (V_L) were estimated from the Lockhart-Martinelli correlations (Ref. B2). Table B-1 summarizes experimental and estimated data for the July, 1978, preheater gas bypass tests. Liquid and gas densitites were estimated using the average temperature between 600 °F and the preheater outlet temperature and using the heater outlet pressure. The molecular weight of feed gas was estimated to be 5.0. Specific gravity of coal was estimated to be 1.3. Figure B-1 shows the relationship between coal conversion, X_{AI}, and residence time, τ_p, according to equation (B1). The equilibrium MAF coal conversion, X_M, at the preheater outlet was assumed to be 96 wt % for Indiana V coal. The slopes of these lines correspond to the values of K_p at various preheater outlet temperatures.

The effect of temperature on the coal dissolution rate coefficient, K_p, at 2,400 psig is shown in an Arrehenius plot in Figure B-2. K_p can be expressed as:

$$K_p = K_o e^{-E/RT}$$

where

K_o = Frequency factor, hr^{-1}

E = Activation energy, Btu/lb-mole of coal

R = 1.987 Btu/(lb-mole)(°R)

T = Preheater outlet temperature, °R

Figure B-2 shows that the relationship between K_p and T follows the Arrhenius law. The slope of this line (slope = -E/R) provides the value of E. The estimated activation energy (E) and frequency factor (K_o) at 2,400 psig operating pressure are:

E = 19,100 Btu/lb-mole

K_o = 0.13×10^6 hr^{-1}

The above activation energy and frequency factors correspond to the Run 141 solvent and coal compositions and a solvent donor quality of about 72.6 (microautoclave conversion, long method). The corresponding conversion model parameters for the January, 1978 data were 37,100 Btu/lb-mole and 174 x 10^6 hr^{-1}, (Ref. B1). The January, 1978, solvent donor quality was 80.2 (microautoclave conversion, long method). Comparisons of calculated and experimental values for coal conversion, X_{AI}, are shown in Figure B-3.

REFERENCES

B1. Quarterly Technical Progress Report (FE-2270-34) for the the period January-March 1978, Southern Company Services, Inc., Project No. 43080 (prepared by Catalytic, Inc.).

B2. Lockhart, R. W., and Martinelli, R. C., Chemical Engineering Progress, Vol. 45, 139 (1949).

Table B-1
Comparison of Observed and Calculated Coal Conversion in Slurry Preheater
Indiana V Coal

Operating Pressure: 2,400 psig

Test	Temperature, °F	Coal feed rate, MF lb/hr	Gas feed rate, scfh	V_L estimated liquid hold-up, ft^3/ft^3 total volume	T_p Slurry residence time, hr	X_{AI}, coal conversion, % MAF Experimental	Calculated	Error
A	748	310	10,430	0.20	0.0230	64.5	63.9	+ 0.9
B	760	422	10,340	0.25	0.0191	57.7	57.3	+ 0.7
C	756	773	10,160	0.30	0.0156	56.7	50.3	+11.3
D	803	312	9,430	0.22	0.0258	74.6	77.1	- 3.4
E	800	500	8,200	0.27	0.0199	64.0	68.7	- 7.3
F	800	812	7,620	0.31	0.0142	60.5	57.0	+ 5.8
G	832	288	3,030	0.33	0.0410	84.3	91.8	- 8.9
H	815	464	3,130	0.38	0.0280	72.6	82.2	-13.2
I	787	785	3,070	0.40	0.0186	74.1	63.6	+14.2
J	850	382	0[a]	0.50	0.0463	88.9	94.0	- 5.7
K	848	492	0[a]	0.51	0.0371	90.6	91.8	- 1.3
L	837	747	0[a]	0.56	0.0268	72.6	84.3	-16.1
M	846	334	0[a]	0.51	0.0381	87.3	92.2	- 5.6
N	848	444	0[a]	0.55	0.0310	93.3	89.0	+ 4.6
I-I	829	835	0[a]	0.58	0.0251	73.9	81.4	-10.1
L-L	854	765	0[a]	0.57	0.0365	78.2	91.7	-17.3
0	847	857	0[a]	0.70	0.0201	79.0	78.4	+ 0.8

(a) A purge of 200 scfh was maintained at the preheater inlet pressure tap.

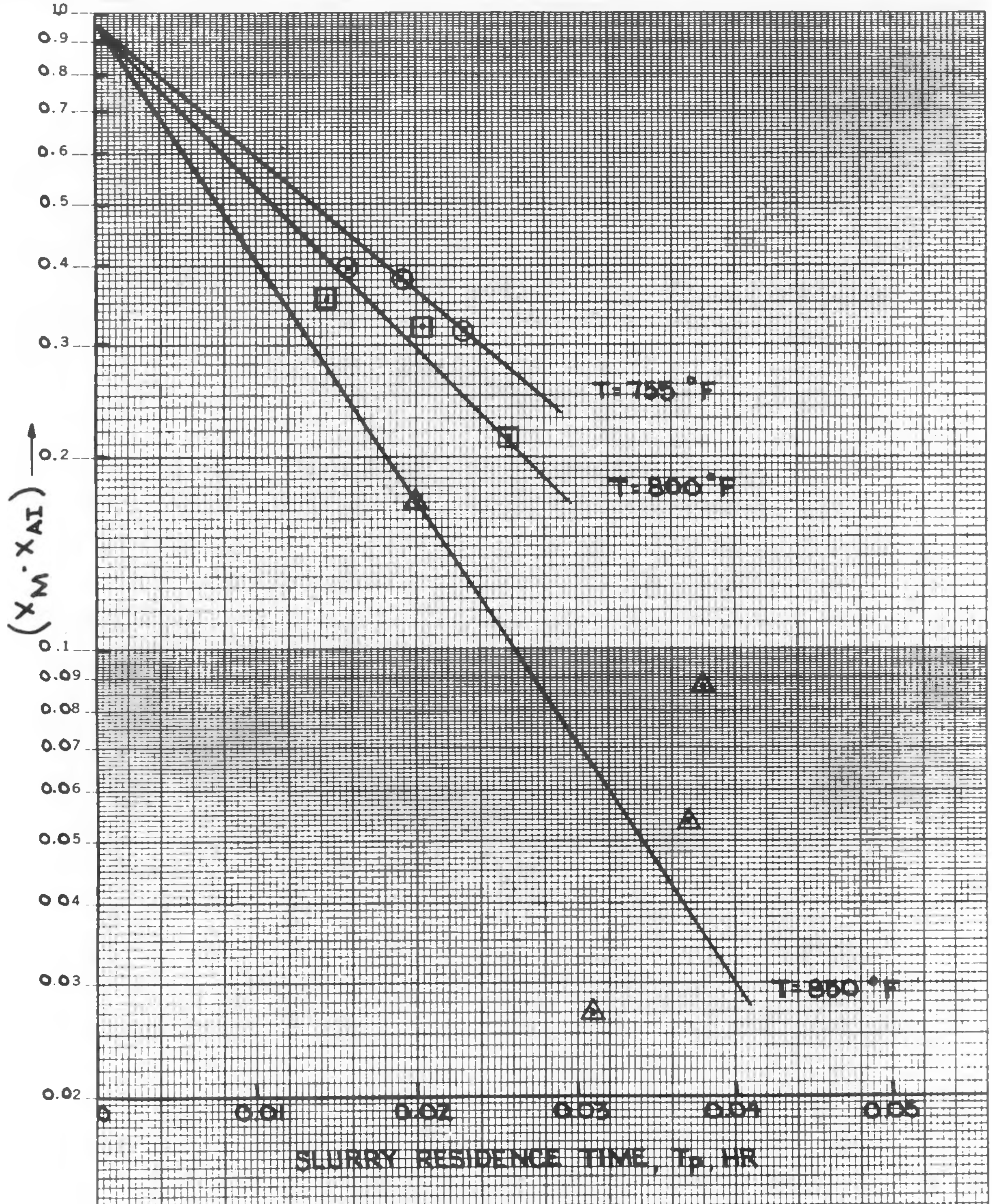

FIGURE B-1: PREHEATER FIRST ORDER CONVERSION MODEL. PRESSURE = 2400 PSIG

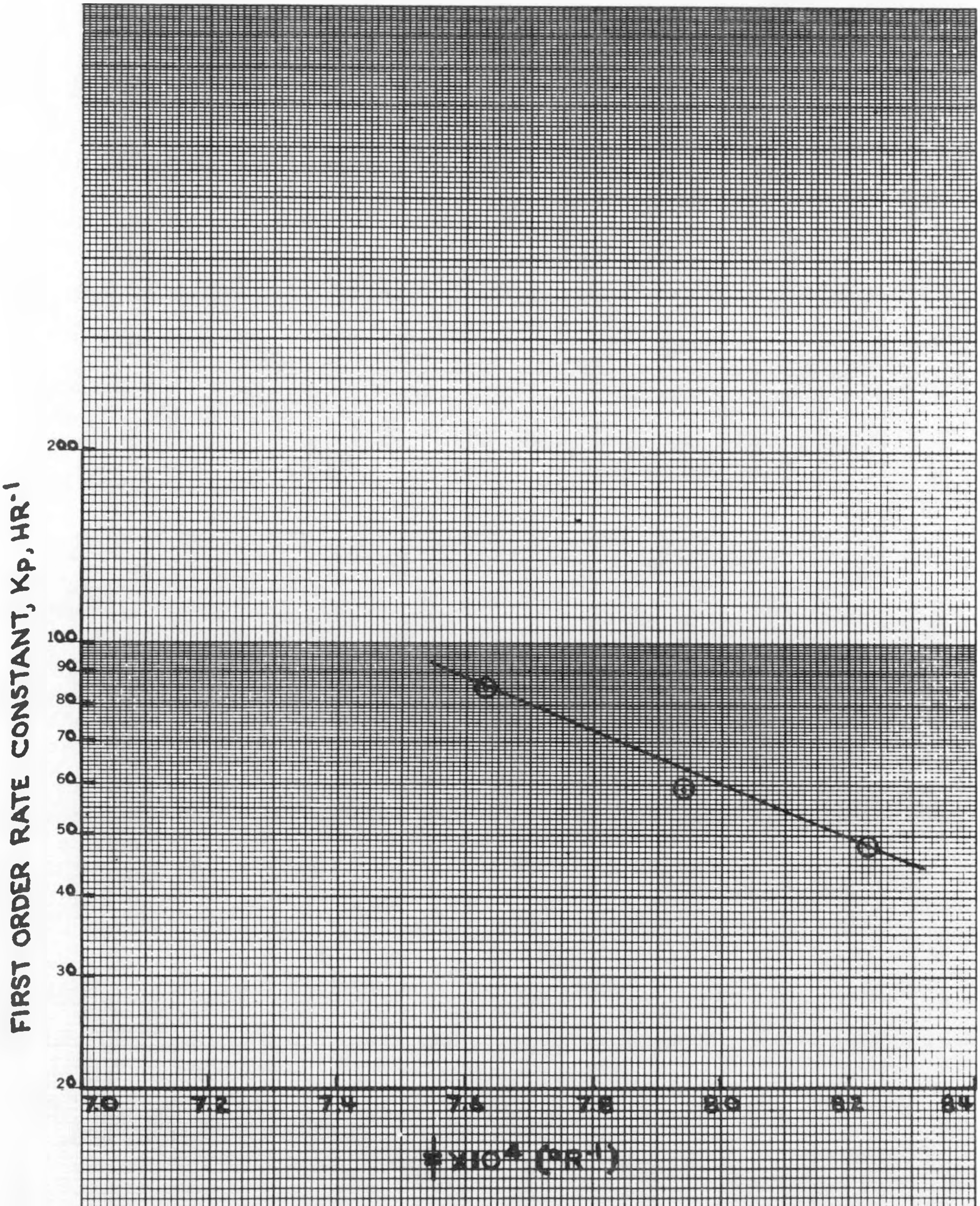

FIGURE B-2: EFFECT OF TEMPERATURE ON COAL CONVERSION RATE CONSTANT. PRESSURE = 2400 PSIG

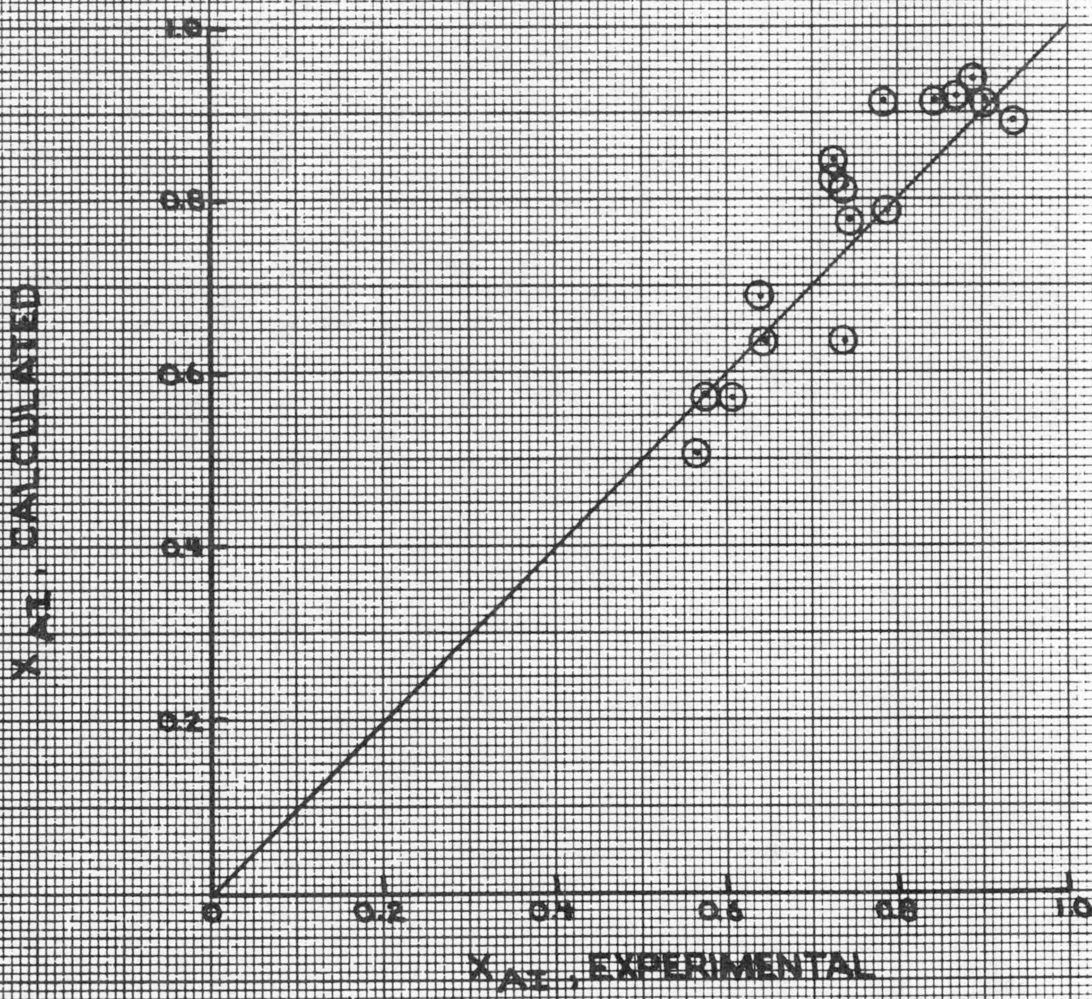

FIGURE B-3: CALCULATED VERSUS EXPERIMENTAL COAL CONVERSION

APPENDIX C: SUMMARY OF USF FILTER CYCLE

"A" PROGRAM - NO PRECOAT, SLUICE WITH FEED

STEP	DESCRIPTION	EXECUTION, SECONDS	OCCURRENCE OR MANUAL ACTION
A-1	Check "B" program.		"A" steps only if "B" is at B1.
A-2	Pressure filter.	120	Open: PV673. Step: when PSH672B trips.
A-4	Start sluicing from V112. Rotate leaves.	180	Divert: XV655 to F125. Open: DPCV6036, XV674, FV684 on PIC-6051, XV6028, XV6038, FV6026 on FIC-6026. Close: PV673, leaf rotation switch. Step: when timer T-5 is out.
A-5	Continue sluice and wait for leaves to position.	60 (max)	Close: XV674, FV684. Step: when leaf position switch indicates bottom position.
A-6	Continue sluice. Check valving.	2	Step: when XV674 limit switch indicates valve is closed.
A-7	Continue sluicing. Check valving.	2	Step: when XV654 limit switch indicates valve is diverted to V112.
A-11	Stop sluicing. Continue filter fill through bottom. Check valving.	2	Close: XV6028. Open: XV6029, XV669, XV6035, DPCV6036 on DPIC6036. Step: when XV6028 limit switch indicates valve is closed.

APPENDIX C: SUMMARY OF USF FILTER CYCLE

"A" PROGRAM - NO PRECOAT, SLUICE WITH FEED

STEP	DESCRIPTION	EXECUTION, SECONDS	OCCURRENCE OR MANUAL ACTION
A-12	Continue filter fill through bottom.	220	Step: when LSH661 (or LSH656) trips.
A-13	Prepare for recirculation. Check valving.	2	Divert: XV654 to V112. Open: XV686B. Close: XV669, DPCV6036. Step: when XV669 limit switch indicates valve is closed.
A-14	Recirculate to V112 for cake buildup.	180	Step: when DPSH650 reaches 10-40 psi (to be determined).
A-15	Switch to forward feed filtration.	Variable	Close: XV686B. Open: DPCV687. Step: when LSL640A trips or when timer K-4 is out.

Other steps same as in Precoat Program starting with Step A-27.

APPENDIX C: SUMMARY OF USF FILTER CYCLE

"A" PROGRAM - PRECOAT, SLUICE WITH PRECOAT

STEP	DESCRIPTION	EXECUTION SECONDS	OCCURRENCE OR MANUAL ACTION
A-1	Check "B" Program.	1	"A" steps only if "B" is at B1.
A-2	Pressure filter.	120	Open: PV673. Step: when PSH672B trips.
A-4	Start sluicing from V141. Rotate leaves.	180	Divert: XV658 to F125. Open: DPCV6036, XV674, FV684 on PIC-6051, XV6028,FV6026 on FIC-6026. Close: PV673, filter leaf rotation switch. Step: when timer T-5 is out.
A-5	Continue sluicing and wait for leaves to position.	60 (max)	Close: XV674, FV684. Step: when filter leaf position switch indicates bottom position.
A-6	Check valving.	2	Step: when XV674 limit switch indicates valve is closed.
A-7	Check valving.	2	Divert: XV654 to V141. Step: when XV654 limit switch indicates valve is diverted to V141.
A-11	Stop sluicing. Continue filter fill through bottom. Check valving.	2	Close: XV6028. Open: XV6029, XV669, XV6035, DPCV6036 on DPIC6036. Step: when XV6028 limit switch indicates valve is closed.

APPENDIX C: SUMMARY OF USF FILTER CYCLE

"A" PROGRAM - PRECOAT, SLUICE WITH PRECOAT

STEP	DESCRIPTION	EXECUTION SECONDS	OCCURRENCE OR MANUAL ACTION
A-12	Continue filter fill through bottom.	220	Step: when LSH661 (or LSH656) trips.
A-13	Set valves for precoating. Switch to side feed.	2	Open: XV686B, XV671. Close: XV6029. Step: when XV6029 and XV6027 limit switches indicate valves are closed.
A-14	Check valving.	2	Close: FV6026, XV669, DPCV6036. Step: when XV669 limit switch indicates valve is closed.
A-15	Precoat.	1,620	Step: when timer K-3 is out.
A-19	Switch valves to establish ΔP control.	10	Divert: XV655 to F125. Open: DPCV687 on ΔP control. Step: when timer T-1 is out.
A-20	Check valving.	2	Divert: XV658 to V141. Close: XV686B. Open: XV6038. Step: when XV658 limit switch indicates valve is to V141.
A-21	Filter to V120.	600	Step: when LSL640A trips.

APPENDIX C: SUMMARY OF USF FILTER CYCLE

"A" PROGRAM - PRECOAT, SLUICE WITH PRECOAT

STEP	DESCRIPTION	EXECUTION SECONDS	OCCURRENCE OR MANUAL ACTION
A-22	Switch filtrate to V141.	10	Open: DPCV686A on DPIC687 control. Step: when timer T-1 is out.
A-26	Fill V141.	Variable	Close: DPCV687. Step: when LSH620 (90%) or when LSL640B trips.
A-27	Establish ΔP = 10 psi using N_2 flow.	10	Open: XV6049, PV673 on PIC-6051. Step: when PSH672C trips.
A-30	Stop forward feed. Check valving.	2	Divert: XV655 to V112, XV654 to V112. Close: DPCV686A, XV6038 (E112 bypass). Step: when XV654 limit switch indicates valve is diverted to V112.
A-31	Check valving.	2	Step: when XV655 limit switch indicates valve is diverted to V112.
A-32	Drain filter feed to V112.	430	Open: FV600 on flow control. Step: when LSL656 trips.

APPENDIX C: SUMMARY OF USF FILTER CYCLE

"A" PROGRAM - PRECOAT, SLUICE WITH PRECOAT

STEP	DESCRIPTION	EXECUTION SECONDS	OCCURRENCE OR MANUAL ACTION
A-35	Position valves for wash fill. Check valving.	2	Divert: XV681 to V178. Close: FV600, XV671, XV6035. Open: XV6029, XV669, DPCV6036 on DPIC-6036. FV6026 on FIC-6026. Step: when XV681 limit switch indicates valve is diverted to V178.
A-36	Fill with wash solvent.	430	Open: XV6027. Step: when LSH661 (or LSH656) trips.
A-37	Cake wash. Check valving.	2	Close: XV669, DPCV6036, PV673, XV6049. Open: XV6035, DPCV687 on DPIC-687. Step: when XV669 limit switch indicates valve is closed.
A-38	Cake wash.	360	Step: when specified amount of wash fed as set on differential level switch LSL9044.
A-39	Establish N_2 flow.	10	Open: XV6049, PV673 on PIC-6051. Close: DPCV687. Step: when timer T-1 is out.

APPENDIX C: SUMMARY OF USF FILTER CYCLE

"A" PROGRAM - PRECOAT, SLUICE WITH PRECOAT

STEP	DESCRIPTION	EXECUTION SECONDS	OCCURRENCE OR MANUAL ACTION
A-40	Close feed valves.	2	Close: XV6027, FV6026, XV6029, XV6035. Step: when XV6027 and XV6029 are closed.
A-43	Drain.	430	Open: FV600 on FIC600. Step: when LSL656 trips.
A-44	Check valving.	2	Divert: XV681 to XV654. Close: FV600. Open: DPCV687. Step: when XV681 limit switch indicates valve is diverted to XV654.
A-45	Cake blow.	60	Step: when timer T-2 is out.
A-46	Open filtrate line to vent and open vent valve.	2	Open: XV6037 and DPCV6036. Close: DPCV687. Step: when XV6037 limit switch indicates valve is open.
A-47	Depressure.	180	Open: XV674, FV684 on DPIC687, turn conveyor motor on. Close: PV673, XV6049. Step: when PSL672 trips.

APPENDIX C: SUMMARY OF USF FILTER CYCLE

"A" PROGRAM - PRECOAT, SLUICE WITH PRECOAT

STEP	DESCRIPTION	EXECUTION SECONDS	OCCURRENCE OR MANUAL ACTION
A-49	Dry cake in "C" program.	364	Open: XV690. Close: XV674, FV684, XV6053. Step: when "C" program is on step C-8.
A-51	Prepare for discharge.	10	Open: XV665 A and B. Close: XV690, XV6036, XV6037, XV6053. Step: when XV665 limit switch indicates valve is open.
A-52	Discharge by rotating leaves and conveyor.	180	Turn on leaf rotator. Advance "C" program to ash cool steps. Step: when timer T-5 is out.
A-53	Close bottom valve.	10	Close: XV665 A and B. Turn off leaf motor. Advance "B" program to step B1. Step: when XV665 limit switch indicates valve is closed.
A-54	Check that leaves are in position.	0-60	Step: when leaf position switch indicates bottom position.
	END OF CYCLE		Return to step A-1 to start another cycle or to A-0 if program hold is used.

APPENDIX C: SUMMARY OF USF FILTER CYCLE

"A" PROGRAM - PRECOAT, SLUICE WITH PRECOAT

STEP	DESCRIPTION	EXECUTION SECONDS	OCCURRENCE OR MANUAL ACTION
	Programmer off.		XV658 to V141, XV654 to V112, XV655 to V112, XV681 to XV654, XV604 to V140, XV542 to V111, XV801G open, XV6053 open. Other valves are normally closed.
	Downtime:		
	No precoat, sluice w/ feed	3,196	
	Precoat, sluice w/ precoat	5,261	

APPENDIX C: SUMMARY OF USF FILTER CYCLE

"B" PROGRAM - PRECOAT, SLUICE WITH PRECOAT

STEP	DESCRIPTION	EXECUTION SECONDS	OCCURRENCE OR MANUAL ACTION
B-1	Hold until started by "A" program.	2	"A" program steps "B" program on step A-16.
B-2	Refill V112 from V111.	700	Divert: XV542 to V112. Step: when V112 reaches level set on LSH640.
	TOTAL	702	Return to B-1.
	END OF CYCLE.		For addition of precoat slurry from V140, the operator must manually transfer the specified amount of precoat to V141 by diverting XV604 to V141.

APPENDIX C: SUMMARY OF USF FILTER CYCLE

"C" PROGRAM - PRECOAT, SLUICE WITH PRECOAT

STEP	DESCRIPTION	EXECUTION SECONDS	OCCURRENCE OR MANUAL ACTION
C-1	Check vent valve position.	1	Step: if 801G is open.
C-2	Hold for "A" program.	1	Step: when "A" program is on step A-49.
C-3	Continue to depressure to K115 and start vacuum.	2	Open: XV801H. Close: XV801G. Step: when limit switches indicate XV801G is closed and XV801H is open.
C-4	Dry.	300	Step: when timer K-5 is out.
C-5	Break vacuum.	60	Open: XV801C. Close: XV801H. Step: when PSH 835 indicates 0 psig.
C-6	Reset valves.	2	Open: XV801G. Close: XV801C. Step: when XV801G limit switch indicates valve is open.

APPENDIX C: SUMMARY OF USF FILTER CYCLE

"C" PROGRAM - PRECOAT, SLUICE WITH PRECOAT

STEP	DESCRIPTION	EXECUTION SECONDS	OCCURRENCE OR MANUAL ACTION
C-8	Return to "A" program. Hold until end of cake discharge.	2	"C" program steps "A". Step: when "A" program is on step A-52.
C-9	Cool ash.	600	Step: when timer K-6 times out (or manually).
C-10	Discharge ash.	300	Open: Ash cooler discharge valve (XV6046). Step: when timer K-7 times out. (Do manually when horn sounds).
	Total	1,268	
	END OF CYCLE		Return to C-1.

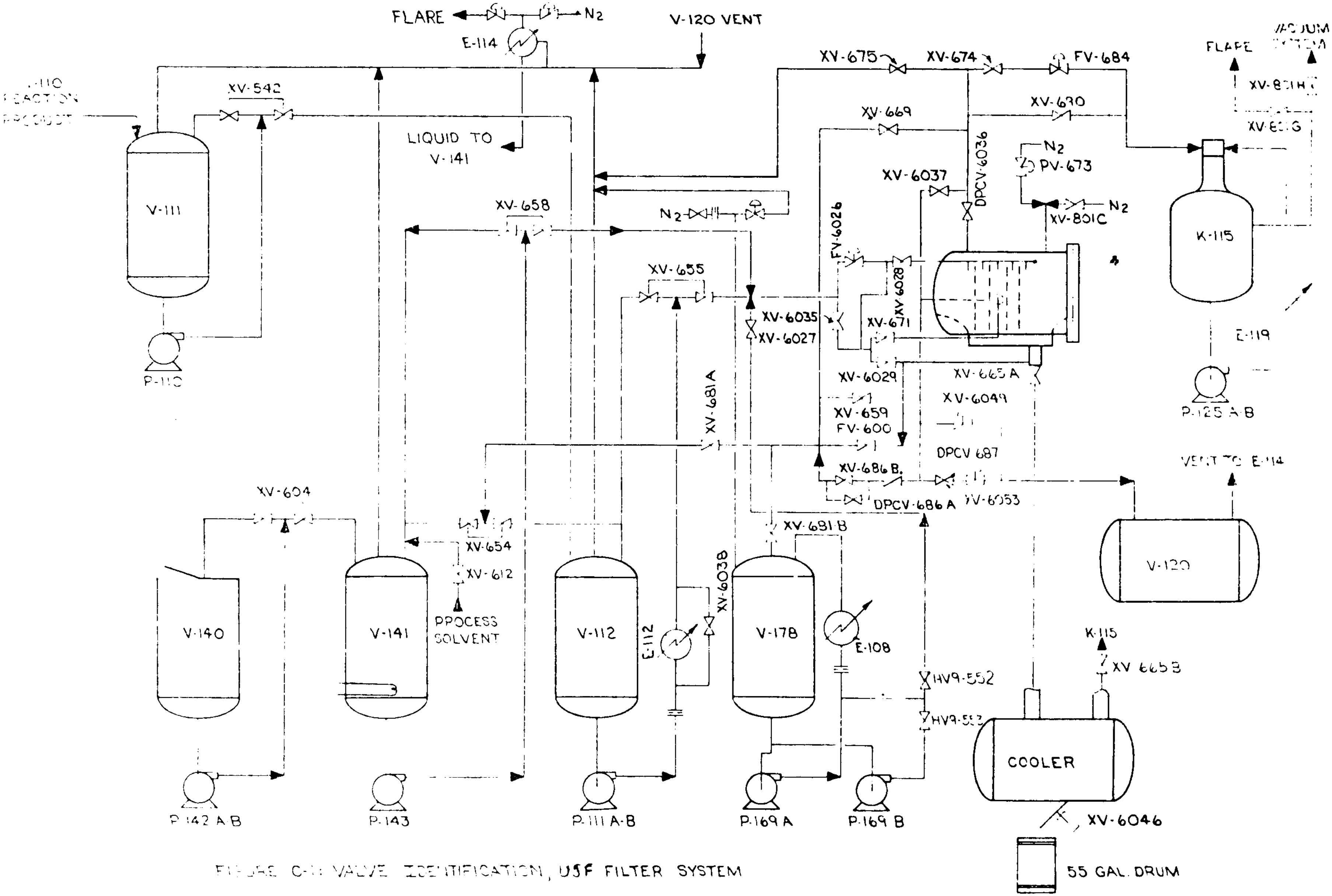

FIGURE C-[illegible] VALVE IDENTIFICATION, USF FILTER SYSTEM

APPENDIX D
Boiling Point Correction for ASTM D-1160 Method

An inconsistency is usually noted when process solvent (D-1160 and D-86) distillation results are compared (Figure D-1). The D-1160 results show higher boiling ranges than do the D-86 results.

The D-86 method is an atmospheric boiling point separation with a low reflux volume and low separation. The sample (100 ml) is evaporated, condensed, and collected. Due to the nature of the solvent and the distillation, the heavy materials in the flask may reach temperatures as high as 900°F before distilling. This may cause decomposition.

The D-1160 method, due to the high vacuum used, does not require temperatures as high as the D-86 method. There would be less decomposition at the heavy end. Decomposition products would lower the boiling temperatures of the heavy ends in D-86 distillations.

Some differences in these two methods are summarized in Table D-1. The most important difference, which is not listed, is the method of boiling point determination. The D-86 boiling point is determined by direct readout of the thermometer, while the D-1160 boiling points must be determined from both temperature and vacuum readings. This has been done in the past using a published nomograph prepared by using data from the distillation of petroleum products.

The data produced with the use of the nomograph deviate considerably from those determined by D-86 and GC distillations. The following correction for boiling point was developed:

$$\text{Temp } ^\circ\text{F} = K\,(760 - \text{Vac mm Hg})\,(460 + T_O) \qquad \text{(I)}$$

where T_O = temperature observed.

The value of K for hydrocarbons is 1.25×10^{-4}*. This factor gives temperatures which are much too low for polycyclic aromatic compounds. A standard composition for distillations (Table D-2) was prepared. This standard was analyzed by both D-86 and D-1160 methods and the data were compared (Table D-3). From these data, a K factor was determined for each point. These K factors were plotted against the D-86 boiling points (Figure D-2).

The knee in the graph is a result of the standard used. Some of the components tend to solidify in the condenser. The K factor was determined by averaging the K values of all points below the knee of the graph in Figure D-2.

*_Handbook of Chemistry_, N. A. Lange, 1949.

Table D-1
Comparison of Distillation Methods

	ASTM D-86	ASTM D-1160
Temperature of flask @ IBP, °F	400-450	60-80
Pressure, mm Hg	750-760	< 1.0
Reflux volume/100 ml, m	≈100	≈250
Temperature of flask @ EP, °F	>900	< 450
Volume of sample, ml	100	200
Temperature of condenser, °F	60-212	60-212
Atmospheric boiling point of lowest condensable component, °F	≈80	≈400 [a]

(a) A compound with a BP of 410°F will evaporate at 60°F and 0.1 mm Hg.

Table D-2
Components in Distillation Standard

Component	wt %	TBP, °F	wt % cum
phenol	7.4	360	7.4
tetralin	7.0	405	14.4
naphthalene	9.7	424	24.1
quinoline	4.1	460	28.2
2-methylnaphthalene	12.7	466	40.9
1-methylnaphthalene	5.8	473	46.7
biphenyl	10.1	491	56.8
dimethylnaphthalenes	4.5	501-9	61.3
acenaphthene	6.1	531	67.4
dibenzofuran	7.2	549	74.6
phenanthrene	11.0	643	85.6
anthracene	3.6	645	89.2
carbazole	3.6	667	92.8
fluoranthene	4.8	722	97.6
chrysene	2.4	838	100.0

Table D-

Comparison of D-86, D-1160, and True Boiling Point Distillations

%	TBP	D-86	Calc ⊡ (a) D-1160	Nomogr. △ (b) D-1160	D-1160 Data and Calculations: Temp °F	Vac(c)	T diff(d)	K x 10^4 (e)
	Temp °F							
IBP	360	380	428	419	150.8	3.20	2.29	4.96
5	360	401	439	481	158.0	0.70	2.43	5.18
10	405	416	440	491	158.0	0.45	2.58	5.50
15	424	428	440	512	158.0	0.28	2.70	5.75
20	424	440	437	524	156.2	0.19	2.83	6.06
25	460	451	437	531	156.2	0.16	2.95	6.30
30	466	458	437	534	156.2	0.15	3.02	6.45
35	466	465	440	539	158.0	0.16	3.07	6.54
40	466	476	442	541	159.8	0.16	3.16	6.71
45	473	481	449	562	164.3	0.12	3.17	6.68
50	491	489	455	563	168.8	0.12	3.20	6.70
55	491	497	466	579	176.0	0.12	3.21	6.64
60	≈505	516	476	589	183.2	0.12	3.33	6.81
65	531	545	492	600	194.0	0.13	3.51	7.06
70	549	590	518	630	212.0	0.12	3.78	7.40
(f)								

(a) ⊡ BP = (6.01×10^{-4}) (760- P) (460 + T_o).

(b) △ Determined from J. Inst. Pet. Tech. 23-311 (1937).

(c) Vacuum in mm Hg.

(d) T_{diff} = Temp (D-86)-Temp (D-1160).

(e) K = Avg K IBP-45% = 6.01×10^{-4}

K = T_{diff}/(760-P)(460 + To).

(f) Distillation ended due to solidification of components in condenser.

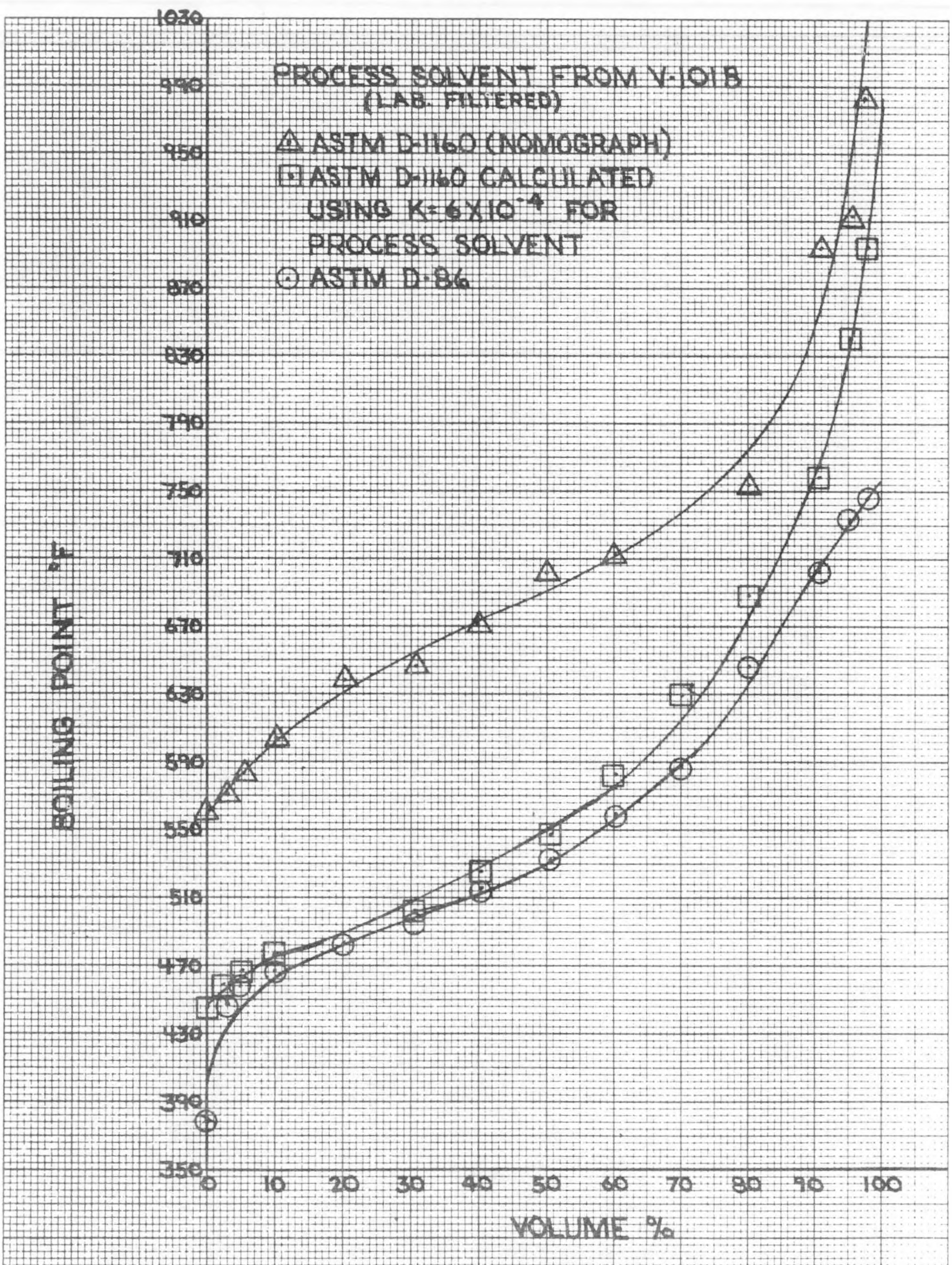

FIGURE D1 : COMPARISON OF VARIOUS DISTILLATION CURVES FOR PROCESS SOLVENT

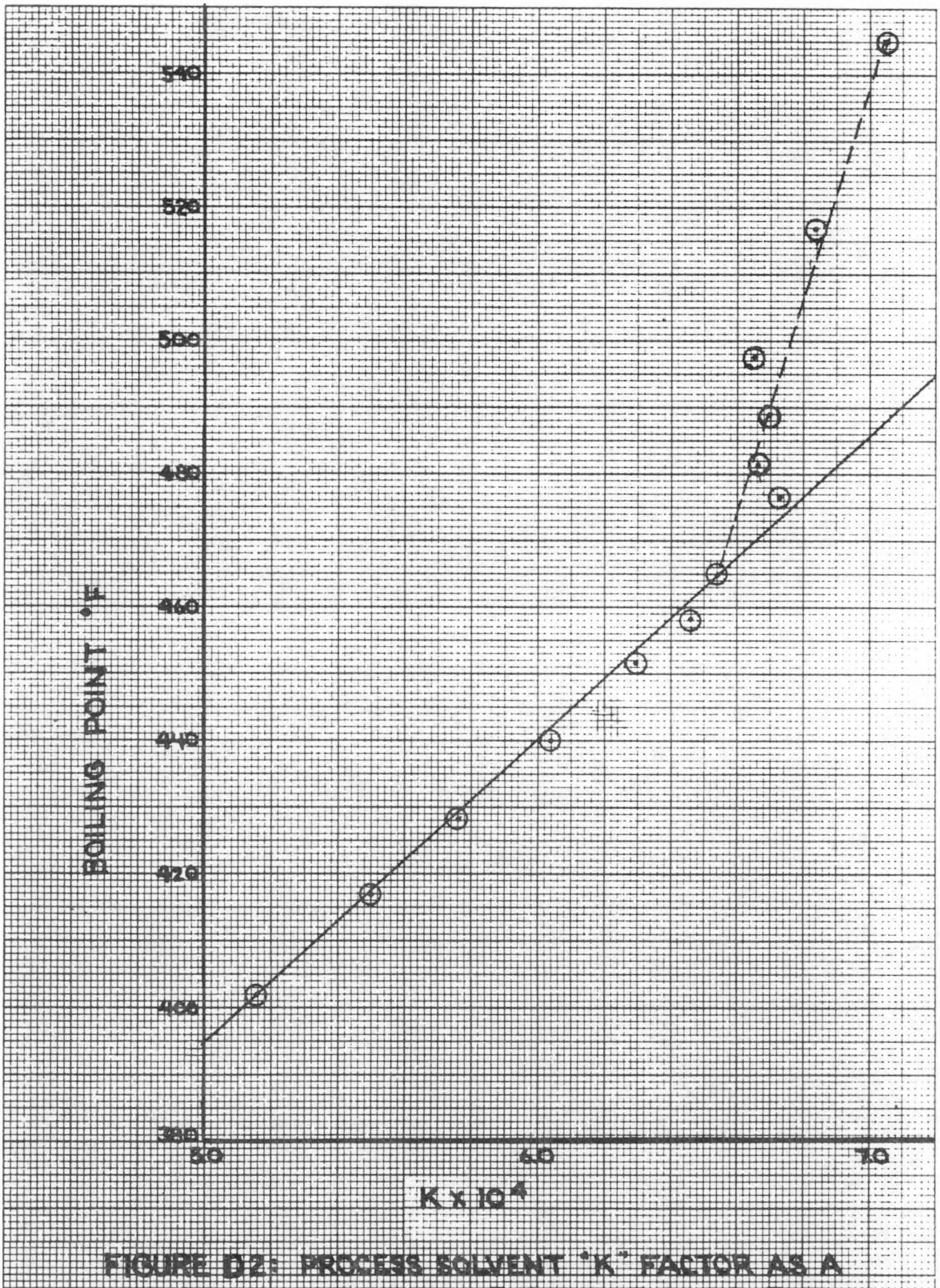

FIGURE D2: PROCESS SOLVENT "K" FACTOR AS A FUNCTION OF TEMPERATURE FOR ASTM D-1160

Table 1
SRC Pilot Plant Operating Hours and Filter Cycles

Run	w/solv & H_2 hr [a]	Reaction Section with coal slurry and hydrogen, hr [c] 5% [b]	10% [b]	20% [b]	25% [b]	27% [b]	33% [b]	38.5 [b]	40% [b]	Filtration section, cycles	Vacuum Flash section, hr
140 [e]	80							77		55	96
141	244							237			271
142	268							235		13	243
143	546							541		37	546
144	148							135			162
145	17							11			12
146	16							9			0
147	124							108			117
148	193							135			123
149 [f]	32							32			32
Report period	1,668							1,520		105	1,602
Prior 1978	3,580							3,321		779	3,524
Total 1977	5,559		39	40	324	713	992	2,574		2,319	5,625
Total 1976	6,329		111	184	1,367		69	4,098		2,893	6,610
Total 1975	4,883		48	637	2,013		1,041		300	1,575	4,892
Total 1974	4,675	23	73	69	3,193 [d]		407			1,208	4,874
Overall total to date	26,694	23	271	930	6,897	713	2,509	11,513	300	8,879	27,127

(a) On-stream hours with solvent and hydrogen through B102 Slurry Preheater and R101 Dissolver.
(b) Coal slurry concentration MF basis.
(c) On-stream hours with coal slurry and hydrogen through B102 Slurry Preheater and R101 Dissolver.
(d) Includes 165 hours on 5-20% concentration during early runs.
(e) July portion of run.
(f) September portion of run.

Table 2
Operating Data Summary for July, 1978

Date July 1978	Coal feed MF lb/hr	Coal feed hr	Slurry conc MF coal, %	Feed Gas: H_2 %	Feed Gas: total scfh	Feed Gas: to B102 scfh	B102 Preheater: inlet press psig	B102 Preheater: ΔP mid psi	B102 Preheater: ΔP top psi	B102 Preheater: ΔP total psi	B102 Preheater: outlet temp °F	R101 Dissolver: Temperature btm °F	R101 Dissolver: Temperature outlet °F	R101 Dissolver: Density btm %	R101 Dissolver: Density mid %	R101 Dissolver: Density top %
- Run 141 Indiana V -																
6	522	6.0	36.2	85			2,450	9	10	51	761	791	834	34	28	-
7	462	24.0	36.6	86	10,298	10,298	2,434	11	14	56	746	790	845	43	40	-
8	482	24.0	36.1	85	10,051	10,051	2,427	9	15	53	727	799	846	36	28	-
9	449	24.0	36.1	85	10,074	10,074	2,429	9	14	50	727	803	846	40	30	-
10	460	24.0	32.4	86	10,201	10,201	2,422	9	10	50	748	806	843	43	35	-
11	550	24.0	36.4	85	9,673	1,644	2,432	10	14	58	757	805	843	48	41	-
12	545	24.0	37.2	85	5,614	2,453	2,437	8	8	46	810	808	847	54	45	-
13	625	24.0	36.6	88	2,822	3,155	2,458	7	5	37	801	799	836	59	54	-
14	566	24.0	33.7	88	402	4,759	2,447	7	4	26	841	799	833	61	53	-
15	570	24.0	28.0	87	1,485	4,194	2,464	6	18	47	818	804	842	59	51	-
16	508	14.5	31.6	91	8,939	673	2,446	4	15	34	779	796	822	61	43	-
- Run 142 Indiana V -																
20	431	17.5	36.3	86	9,452	9,452	2,450	6	16	24	760	780	823	49	22	-
21	458	24.0	37.2	84	9,963	9,963	2,165	11	20	40	754	796	826	62	48	-
22	474	24.0	37.2	85	10,176	10,176	2,459	12	21	45	756	797	825	64	55	-
23	455	24.0	37.1	85	10,134	10,134	2,460	13	21	46	749	799	827	64	57	-
24	468	24.0	36.7	85	10,073	10,073	2,456	12	21	41	749	798	825	65	55	-
25	456	24.0	37.5	86	10,386	10,386	2,425	12	20	44	749	793	824	65	55	-
26	448	24.0	38.3	85	10,335	10,335	2,456	12	19	44	747	800	825	65	51	-
27	453	24.0	37.8	85	10,146	10,146	2,453	12	18	44	749	799	826	65	49	-
28	435	24.0	37.6	86	10,379	10,379	2,455	11	19	43	752	800	822	66	52	-
29	449	24.0	37.4	84	9,960	9,960	2,451	11	19	41	752	798	825	67	53	-
30	478	1.3	37.3	87	10,192	10,192	2,450	12	20	42	752	798	825	66	53	-
- Run 143 Indiana V -																
30	478	22.7	37.3	87	10,192	10,192	1,800	19	21	48	758	803	824	65	52	-
31	457	24.0	37.1	85	9,983	9,983	1,792	22	10	52	758	800	809	66	51	-

Table 2 (continued)
Operating Data Summary for July, 1978

Date July 1978	Performance: Coal conv % MAF	Performance: SRC yield(a) % MAF coal	Performance: SRC sulfur(b) %	H_2 consumed, % MAF	T102 Vacuum Column: Temperature btm °F	T102 Vacuum Column: Temperature top °F	T102 Vacuum Column: press top psia	T104(c) lgt org +350 °F wt %	T105 Frac Column(c): btms -450 °F wt %	T105 Frac Column(c): wash solvent -350 °F wt %	T105 Frac Column(c): wash solvent +450 °F wt %
- Run 141 Indiana V -											
6	93.8				598	192	1.1		2.4	0	0.5
7				3.4	598	191	0.6		0	3.0	0
8	96.0	47.5	0.67	3.3	597	192	0.8	0	1.5	0.3	1.5
9	94.3		0.68	3.9	597	192	1.0	0	1.6	1.7	0
10	93.3		0.50	3.5	598	191	1.0	0	2.5	2.5	5.5
11	94.3	61.9	0.61	3.6	602	192	0.7	2.0	0	3.9	0
12	94.0			3.4	596	191	0.5	0	0	7.3	0
13	94.1		0.68	2.7	596	190	0.5	0	0	-	-
14	93.9	62.6	0.76	2.6	596	190	0.5	0	0	5.5	0
15	93.8		0.74	2.7	596	192	0.5	0	0	5.0	0
16	93.5		0.82	3.0	596	191	0.5	0	0	6.0	0
- Run 142 Indiana V -											
20			0.72	3.1	582	188	1.1	-	-	-	-
21	94.6			3.2	591	191	0.9	0	1.1	0	14.0
22	94.0	51.2	0.75	3.1	592	191	1.4	0	3.0	0	0
23	94.1		0.75	3.2	592	191	1.1	0	3.0	3.0	10.0
24	93.9		0.68	3.0	589	191	0.9	0	3.5	-	-
25	94.1	43.8	0.71	3.0	594	191	1.1	1.5	0	-	-
26	94.4		0.72	3.4	599	191	1.3	0	2.5	2.5	0
27	93.9	47.8	0.75	3.2	600	192	1.0	0	1.5	2.0	0
28	93.6		0.68	3.0	586	191	1.0	9.0(d)	8.4(d)	-	-
29	94.0		0.65	3.1	587	191	1.2	5.0(d)	7.1(d)	-	-
30	-		0.72		585	193	1.4				
- Run 143 Indiana V -											
30	94.4		0.72	2.9	591	190	1.1	0(d)	2.4(d)	0.6(d)	16.7(d)
31	94.5	60.1	0.77	2.7	591	192	1.2	-	0.1(d)	1.0(d)	31.0(d)

Table 2 (continued)
Operating Data Summary for July, 1978
Critical Solvent Deashing Unit

Date July 1978	Feed Rate, T102 Btms, lb/hr	Feed Time, hr	Feed From T102 Bottoms					Ash Concentrate				SRC				
			Ash, %	UC, %	Solvent,(e) %	Sulfur, %	Melting Point, F	Ash, %	UC, %	SRC, %	Solvent,(e) %	Ash, %	Sulfur, %	Solvent(e)	Melting Point, °F	Recovery (f)
								- Run 141 Indiana V -								
8	177	17.1	16.7	8.7	5.0	-	335	42.7	27.2	25.0	-	0.06	0.65	-	275	81.6
9	151	18.8	16.2	8.0	6.6	-	317	41.9	26.6	28.6	-	0.25	0.66	-	267	80.9
10	237	17.7	16.1	7.6	6.2	1.90	340	42.2	24.2	31.6	-	0.08	0.57	-	265	81.1
11	191	24.0	15.2	8.1	6.0	1.87	330	42.1	28.0	27.0	-	N.D.	0.49	-	260	83.3
12	179	16.0	15.6	8.2	-	-	-	43.4	29.2	27.2	-	0.07	0.62	-	-	84.1
13	160	22.2	15.9	8.1	8.1	-	332	42.4	27.8	-	-	0.10	-	-	282	82.2
14	181	21.2	14.3	7.7	5.9	-	357	41.5	25.0	33.3	-	0.11	-	-	287	84.0
15	185	17.3	14.2	7.5	-	2.0	342	38.6	28.9	30.0	-	0.07	0.83	-	287	80.7
16	118	20.8	13.9	-	-	1.9	347	-	-	-	-	0.14	0.88	-	297	-
17	256	16.0	-	-	-	-	-	35.2	29.3	33.5	-	0.04	0.85	-	302	-
								- Run 142 Indiana V -								
21	-	0.9	-	-	-	-	-									
22	-	0.3	13.0	6.3	12.3	1.78	298	- Unit Plugged -		-	-	-	-	-	-	-
27	-	10.6	15.2	7.7	4.6	1.81	344	41.3	28.5	29.3	3.1	5.0	1.04	6.3	298	82.2
28	-	6.4	14.1	7.4	5.8	1.81	333	41.7	26.7	33.4	3.5	5.0	1.02	9.5	386	84.3
29	-	7.3	-	-	-	-	-	-	-			0.8-15				
30	-	1.3	-	-	-	-	-	- Unit Plugged -		-	-	-	-	-	-	-

Table 3
Operating Data Summary for August, 1978

Date Aug 1978	Coal feed MF lb/hr	Coal feed hr	Slurry conc MF coal, %	Feed Gas H_2 %	Feed Gas total scfh	Feed Gas to B102 scfh	B102 Preheater inlet press psig	B102 Preheater ΔP mid psi	B102 Preheater ΔP top psi	B102 Preheater ΔP total psi	B102 Preheater outlet temp °F	R101 Dissolver Temperature btm °F	R101 Dissolver Temperature outlet °F	R101 Dissolver Density btm %	R101 Dissolver Density mid %	R101 Dissolver Density top %
							- Run 143 Indiana V - continued -									
1	451	24	37.1	84	9,574	9,574	1,785	18	21	46	758	797	823	66	-	
2	461	24	37.2	84	9,644	9,644	1,795	19	25	56	771	802	826	66	-	-
3	414	24	37.2	85	9,862	9,862	1,796	16	23	54	770	801	825	66	-	-
4	508	24	36.4	85	9,079	9,979	1,798	16	23	66	769	800	825	66	-	-
5	475	24	37.1	85	9,934	9,934	1,801	13	22	63	770	799	816	67	-	-
6	480	24	37.3	85	10,012	10,012	1,800	12	23	61	766	801	825	67	-	-
7	480	24	36.8	84	9,878	9,878	1,800	18	22	55	767	800	824	67	-	-
8	458	24	37.4	85	10,164	10,164	1,802	15	24	61	768	800	824	68	-	-
9	439	24	37.0	86	10,234	10,234	1,800	14	21	56	769	801	824	67	-	-
10	442	24	37.3	85	10,125	10,125	1,798	11	22	55	768	801	825	67	-	-
11	445	24	37.5	85	9,991	9,991	1,794	13	23	54	767	801	818	56	-	-
12	447	22.3	37.2	85	10,061	10,061	1,798	9	23	55	766	803	820	64	-	-
13	468	15	37.6	88	9,587	9,587	1,786	12	17	40	795	788	789	65	-	-
14	456	24	37.3	85	10,013	10,013	1,795	9	22	55	770	803	815	64	-	-
15	470	24	37.1	85	9,967	9,967	1,796	19	22	51	766	803	826	62	-	-
16	474	24	36.8	84	9,857	9,857	1,797	16	23	52	765	805	825	62	-	-
17	479	24	37.1	85	9,825	9,825	1,794	16	23	55	766	804	825	62	-	-
18	471	24	37.6	85	9,937	9,937	1,795	11	22	54	763	806	825	63	65	23
19	478	24	37.3	85	10,041	10,041	1,800	17	18	55	762	804	824	62	64	23
20	474	24	27.9	85	10,025	10,025	1,796	5	23	47	765	806	825	62	64	21
21	461	24	38.4	86	10,142	10,142	1,811	11	24	51	765	802	821	62	64	15
22	445	1	38.4	85	9,494	9,494	2,449	15	15	35	756	796	820	62	63	18
							- Run 144 Indiana V -									
22	445	23	38.4	85	9,494	9,494	2,449	15	15	35	756	799	825	60	64	34
23	445	24	37.5	85	10,034	10,034	2,444	14	15	41	754	800	825	62	65	50
24	455	24	37.3	85	10,052	10,052	2,447	15	15	41	754	804	825	63	66	51
25	425	22.1	35.5	86	10,443	10,443	2,464	15	15	44	760	802	823	63	66	41
26	430	11	36.8	86	10,151	10,151	2,475	15	16	49	750	802	823	62	66	40
27	419	24	36.0	85	9,922	9,922	2,469	13	14	38	748	790	825	44	48	31
28	409	7	33.9	85	10,104	10,104	2,472	15	15	47	754	794	825	47	50	33
							- Run 145 Indiana V -									
29	374	11.3	35.8	88	2,209	2,209	2,400	4	2	11	855					
							- Run 146 Indiana V -									
29	462	9.3	36.2	88	1,031	1,031	2,400	7	3	18	858					

Table 3 (continued)
Operating Data Summary for August, 1978

Date Aug 1978	Performance: Coal conv % MAF	Performance: SRC yield(a) % MAF coal	Performance: SRC sulfur(b) %	H_2 consumed, % MAF	T102 Vacuum Column: Temperature btm °F	T102 Vacuum Column: Temperature top °F	T102 Vacuum Column: press top psia	T104(h) lgt org +350 °F wt %	T105 Fractionation Column(h): btms -450 °F wt %	T105 Fractionation Column(h): wash solvent -350 °F wt %	T105 Fractionation Column(h): wash solvent +450 °F wt %
				- Run 143 Indiana V - continued -							
1	93.3		0.81	3.1	575	191	1.4	-	0.7	1.3	
2	94.1	43.2		2.9	589	192	0.5	-	2.2	0.2	
3	94.8		0.70	3.2	596	192	0.4	-	7.6	1.2	0.8
4	94.8		0.71	2.7	594	192	0.8	[illegible].0	8.6	1.1	5.3
5	94.8			3.0	597	190	1.1	8	7.9	1.3	7.3
6	94.7		0.77	3.1	598	192	1.2	.6	5.1	1.5	12.1
7	93.8	60.1	0.67	3.4	597	191	1.2	.9	4.3	1.8	-
8	94.2		0.70	3.5	596	192	1.2	.5	6.8	3.6	3.0
9	94.2		0.67	2.9	601	192	1.1	.7	7.4	4.0	7.9
10	94.2		0.64	2.9	598	190	0.9	-	6.6	6.6	2.0
11	93.9	64.0	0.74	2.9	600	192	1.0	-	4.7	2.8	-
12	94.6		0.70	3.0	599	192	1.0	2.0	1.8	1.4	-
13	95.2		0.70	2.3	597	191	1.0	7.7	5.4	0.7	-
14	95.1	62.2	0.67	2.9	598	191	1.0	0.0(c)	3.0(c)	13.0(c)	0.0(c)
15	94.4		0.68	2.8	597	192	1.1	-	-	-	0.0(c)
16	94.7		0.68	2.8	600	192	1.1		2.0(c)	-	0.0(c)
17	94.8	65.4	0.73	2.8	597	191	1.1	0.0(c)	-	-	-
18	94.5		0.74	2.9	596	192	1.1	-	-	-	-
19	94.8		0.83	2.8	595	192	1.3	5.0(c)	-	-	-
20	94.3	52.7	0.81	2.8	595	192	1.3	6.9	-	9.46	13.0
21	94.5		0.71	3.0	599	191	1.3	0.0(c)	-	-	-
22	94.0			3.2	598	192	1.2	-	-	-	-
				- Run 144 Indiana V -							
22	94.0	47.8		3.2	598	192	1.2	-	-	-	-
23	94.7			3.3	595	192	1.1	-	-	-	-
24	93.6			3.4	597	191	0.9	-	-	-	-
25	94.2			3.6	597	191	0.7	-	-	-	-
26	95.1			3.4	599	193	0.7	-	-	3.9	2.6
27	94.9	35.3	0.77	3.7	600	191	1.0	7.9	-	2.3	1.9
28	95.1			4.1	596	191	1.0	-	4.9	2.4	9.8
				- Run 145 Indiana V -							
29	94.4		1.29	1.7					5.8	2.7	-
				- Run 146 Indiana V -							
29	93.1		1.31	1.2				-	5.8	2.7	-

Table 3 (continued)
Operating Data Summary for August, 1978
Critical Solvent Deashing Unit

Date Aug 1978	Feed Rate, T102 Btms, lb/hr	Feed Time, hr	Feed From T102 Bottoms: Ash %	UC, %	Solvent,(e) %	Sulfur, %	Melting Point, °F	Ash Concentrate: Ash, %	UC, %	SRC, %	Solvent,(e) %	SRC: Ash, %	Sulfur, %	Solvent,(e) %	Melting Point, °F	Recovery, (f)	(g)
								- Run 143 Indiana V -									
4	-	-	15.3	7.6	-	2.67	335	40.1	49.0	12.0	-	0.09	0.47	-	292	80	-
5	-	-	-	-	-	-	-	39.6	30.9	27.5	-	0.04	0.45	-	292	-	-
6	147	15.2	16.5	2.3	7.7	2.12	340	43.5	18.1	35.1	4.6	0.99	0.76	7.9	295	76	82
7	179	20.1	16.4	6.7	7.4	2.05	340	40.5	19.5	41.3	8.3	0.23	0.71	10.4	280	73	75
8	231	9.7	16.3	9.4	5.9	2.01	336	45.7	25.9	27.9	4.3	0.15	0.76	9.8	302	87	89
9	184	19.3	16.8	6.9	-	-	342	43.9	24.2	33.0	4.1	0.14	-	9.6	282	-	80
11	143	7.1	17.3	6.9	7.7	2.08	352	43.6	28.0	31.5	3.1	0.24	0.69	-	292	80	85
12	223	16.4	16.3	6.6	5.8	2.02	347	43.7	23.5	32.8	3.7	0.13	0.65	10.9	292	81	81
13	214	14.1	16.9	7.5	5.5	2.24	347	43.3	25.1	32.2	4.3	0.05	0.63	11.4	292	81	80
14	289	15.4	16.4	7.7	6.4	2.09	347	45.8	30.3	23.2	4.2	0.38	0.66	10.9	292	85	86
15	248	17.8	17.1	7.4	5.2	-	377	46.1	25.7	28.7	4.5	2.87	0.90	-	286	83	80
16	228	3.3	17.1	6.5	9.8	2.15	-	45.5	24.3	29.6	3.7	0.76	0.70	6.9	-	82	70
17	261	20.5	17.2	6.1	9.2	2.22	340	45.6	26.6	27.8	5.1	0.03	0.68	6.6	311	81	84
18	282	18.2	17.5	6.6	9.6	-	325	46.9	21.0	40.0	4.7	1.9	0.95	9.0	290	83	81
19	258	22.3	17.4	7.2	9.4	2.26	335	46.2	26.7	28.0	3.8	0.2	0.68	6.9	309	83	-
20	287	23.6	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
21	294	21.5	16.7	6.8	7.3	2.31		45.6	22.2	28.5	5.5	0.75	0.8	6.4/13.5	283	84	82
								- Run 144 Indiana V -									
22	136	16.1	17.0	4.6	6.4	2.24	340	47.6	24.3	28.4	2.9	0.23	0.68	9.0	290	-	75
23	172	16.9	17.0	7.7	5.7	2.22	350	45.5	21.3	28.6	4.5	2.5	0.88	7.4	311	83	-
24	126	11.8	-	-	-	-	-	47.2	21.3	30.6	3.1	0.27	0.70	6.3	318	-	-
25	277	11.9	17.7	7.9	7.8	2.56	352	46.4	22.0	29.1	3.8	0.05	0.70	10.9	310	83	87
26	280	12.7	16.5	8.3	7.2	2.56	354	48.5	20.8	29.1	2.8	1.62	0.70	7.1	311	88	80
27	308	9.1	17.3	6.9	4.6	2.39	353	47.3	20.2	32.5	3.4	0.13	0.75	7.2	304	84	91
28	254	12.2	15.9	6.4	5.3	2.21	348	47.3	21.6	33.5	-	1.55	0.91	7.2	314	85	-

Table 4
Operating Data Summary for September, 1978

Date Sept 1978	Performance: Coal conv % MAF	Performance: SRC yield(a) % MAF coal	Performance: SRC sulfur(b) %	H_2 consumed, % MAF	T102 Vacuum Column: Temperature btm °F	T102 Vacuum Column: Temperature top °F	T102 Vacuum Column: press top psia	T104(h) lgt org +350 °F wt %	T105 Fractionation Column(h): btms -450 °F wt %	T105 Fractionation Column(h): wash solvent -350 °F wt %	T105 Fractionation Column(h): wash solvent +450 °F wt %
				- Run 147 Indiana V Coal -							
4	93.7		1.05	-	-	-	-	-	3.7	8.0	18.0
5	94.7	54.8	0.67	3.3	593	191	1.3	0	10.4	8.6	8.1
6	93.2		0.88	2.6	595	192	1.3	2.3	5.1	11.7	9.6
7	91.5	54.0	0.88	2.5	595	192	1.2	-	6.8	7.6	8.9
8	92.8		0.85	3.2	597	194	1.2	0	10.2	5.3	6.4
9											
				- Run 148 Indiana V Coal -							
25			0.49	-	601	192	1.3	-	5.3	-	-
25	97.2				603	192	1.0	-	5.3	-	-
26	97.5				600	191	1.1	-	4.7	10.8	5.9
27					600	187	1.5	6.0	4.7	-	-
				- Run 148 Kentucky 6 and 11 Coal -							
27	94.4		1.13	3.0	594	199	0.9	6.0	4.7	-	-
28	93.3		0.68	3.0	603	192	0.8	-	5.2	6.5	5.7
29	94.0		0.66	3.5	601	192	1.0	13.0	8.8	9.4	1.8
				- Run 149 Kentucky 6 and 11 Coal -							
29	94.0		0.66	2.5	597	193	1.0	13.0	8.8	9.4	1.8
30	93.9	63.0	0.74	2.9	601	193	0.9	0	8.7	6.9	2.2

Table 4 (continued)
Operating Data Summary for September, 1978

Date Sept 1978	Coal feed MF lb/hr	Coal feed hr	Slurry conc MF coal, %	Feed Gas H_2 %	Feed Gas to B102 scfh	Feed Gas total scfh	B102 Preheater inlet press psig	B102 Preheater ΔP btm psi	B102 Preheater ΔP mid psi	B102 Preheater ΔP top psi	B102 Preheater outlet temp °F	R101 Dissolver Temperature btm °F	R101 Dissolver Temperature outlet °F	R101 Dissolver Density btm %	R101 Dissolver Density mid %	R101 Dissolver Density top %
- Run 147 Indiana V Coal -																
2		3.4														
3		0														
4	473	10.0	38.3				1,755	-	10	9	850	798	805	9	12	19
5	406	24.0	38.4	85	10,054	10,054	1,775	-	13	14	818	807	812	8	8	9
6	445	24.0	37.6	85	9,970	9,970	1,785	-	17	18	778	803	825	8	6	9
7	454	24.0	37.7	85	10,132	10,132	1,789	-	17	17	767	798	825	8	6	6
8	439	24.0	38.3	85	9,982	9,982	1,786	-	17	18	766	801	826	8	6	10
9	453	2.4	38.2	85	9,982	9,982	1,780	-	11	16	748	801	826	-	-	-
- Run 148 Indiana V Coal -																
23		12.0														
24	308	21.3	38.6	95	-	-	-	-	-	-	-	-	-	-	-	-
25		12.2			-	-	1,759	4	13	16	768	805	826	47	36	17
25		11.8			-	-	1,750	2	8	7	805	811	814	52	42	19
26	157	19.2	30.0	95	-	-	1,749	2	6	5	812	814	824	51	42	32
27		13.0			-	-	1,780	2	6	6	820	809	818	54	43	12
- Run 148 Kentucky 6 and 11 Coal -																
27	443	11.0	39.5	85	9,340	9,340	1,790	4	12	16	781	802	817	52	42	13
28	429	24.0	37.4	85	9,320	9,320	1,800	6	13	17	784	809	825	52	43	28
29	396	10.5	35.6	85	9,450	9,450	1,800	8	12	22	767	807	824	53	43	29
- Run 149 Kentucky 6 and 11 Coal -																
29	457	8.0	36.1	94	-	-	1,800	5	14	23	772	794	806	54	42	8
30	436	24.0	36.7	86	9,380	9,380	1,800	7	13	20	767	796	811	54	44	31

Table 4 (continued)
Operating Data Summary for September, 1978
Critical Solvent Deashing Unit

Date Sept 1978	Feed Rate T102 Btms, lb/hr	Feed Time, hr	Feed From T102 Bottoms: Ash, %	UC, %	Solvent,[e] %	Sulfur, %	Melting Point, °F	Ash Concentrate: Ash, %	UC, %	SRC, %	Solvent,[e] %	SRC: Ash, %	Sulfur, %	Solvent,[e] %	Melting Point, °F	Recovery (f)	Recovery (g)
								- Run 147 Indiana V -									
5	297	14.5	14.1	7.4	8.8	2.16	350	32.9	19.1	35.3	8.3	0.02	0.83	12.8	290	73	81
6	304	16.0	15.2	7.9	9.3	-	358	34.7	24.1	40.2	6.1	0.05	0.8	9.3	303	73	74
7	290	23.8	16.6	7.8	9.2	2.53	388	34.6	24.3	38.3	8.3	0.09	0.8	12.1	287	69	-
8	230	23.0	15.7	8.3	6.7	2.64	393	34.3	23.3	34.2	5.2	0.05	0.8	6.3	318	71	76
								- Run 148 Kentucky 6 and 11 -									
28	161	12.4	15.4	7.1	5.4	1.71	350	-	-	-	-	-	-	-	-	-	-
29	200	16.3	-	-	-	-	-	34.9	21.1	43.2		0.13	0.68	6.1	290	-	-
30	172	14.4	15.1	6.9	-	1.8	360	36.6	24.4	39.1	1.1	0.07	0.64	5.0	288	75	78

(a) Cresol-soluble material.
(b) From Laboratory workup of filtrate from V110 Flash Tank sample, distilled at 600°F, 0.1 mm Hg.
(c) ASTM D-86.
(d) GC.
(e) 600°F, 0.2 mm Hg
(f) % of cresol-soluble feed from T102 bottoms by forced ash balance.
(g) % of cresol-soluble feed from T102 bottoms by normalized material balance.
(h) GC as of 1 August '78.

Table 5
Conditions and Results Summary for Material Balance Periods
Indiana V Coal

Date, 1978	20-21 August	7-8 September
Run	143	147
Coal		
Volatile matter, %	36.0	34.7
H/C atomic ratio	0.80	0.76
Microautoclave conversion,%	78.2	77.5
Solvent		
IBP, °F	376	399
EP, °F	789	814
% minus 450°F/% plus 650°F	7/14	5/22
Specific gravity	1.006	1.007
H/C atomic ratio	1.27	1.26
Microautoclave conversion, %		
short run	77.0	74.6
long run	74.1	74.7
Operating conditions		
Feed		
Coal feed rate, MF lb/hr	468	447
Concentration, % MF coal	38.2	38.0
Feed gas, Mscf/ton MF coal to B102	46.3	46.7
Hydrogen purity, Mol %	87.5	85.4
Reaction		
Coal space rate, MF lb/hr-ft^3		
Cumulative (a)	22.1	21.1
Dissolver (b)	26.0	24.8
Temperature, °F		
Preheater outlet	763	766
Dissolver		
Bottom	804	800
Middle	815	818
Outlet	823	825
Pressure, psig	1750	1750
Hydrogen pressure, psig		
Preheater inlet	1580	1540
Preheater outlet	1370	1360
Dissolver outlet	1130	1080
Results		
Conversion, % MAF coal		
Preheater	60.3	54.7
Dissolver	94.4	92.2
Hydrogen consumption		
% MF coal	2.4	2.7
Sulfur, % of SRC product	0.75	0.85
Yields, % MF coal (c)		
SRC	47.4	48.0
Organic liquid	25.9	21.9

Table 5 - (continued)
Conditions and Results Summary for Material Balance Periods
Indiana V Coal

Date, 1978 Run	20-21 August 143	7-8 September 147
<u>Results (continued)</u>		
Gases		
C_1 - C_5	5.0	4.9
CO - CO_2	0.8	0.7
H_2S	2.3	2.7
NH_3	0.3	0.3
Water	5.3	7.7
Unreacted coal	4.8	6.3
Ash	10.5	10.1
Dissolver solids conc, lb/ft^3	37.3(d)	5.4

Table 5 (continued)
Conditions and Results Summary for Material Balance Periods
Indiana V Coal

Date, 1978 Run	19 August 143	6 September 147
Slurry Preheater Results(g)		
Preheater outlet temperature, °F	765	766
Feed gas to preheater, scfh	46.3	46.7
Conversion	60.3	54.7
Cresol insolubles (CI), %	6.4	5.4
Ash, %	1.6	1.2
SRC, %	16.4	16.3
Cresol insolubles, Ultimate analysis, wt %		
Carbon	57.30	60.19
Hydrogen	3.77	3.79
Nitrogen	1.03	1.18
Sulfur	4.14	NA
Ash	24.90	21.80
Oxygen (by difference)	8.86	NA
SRC Ultimate analysis, wt %		
Carbon	82.12	85.98
Hydrogen	5.30	4.47
Nitrogen	1.49	1.54
Sulfur	1.47	1.45
Ash	2.40	1.01
Oxygen (by difference)	7.22	5.55

Table 6
Conditions and Results Summary, Adjusted Yields
Indiana V Coal

Date, 1978

Run	143				147			
Material Balance Method	Process Method		V110 Short Method		Process Method		V110 Short Method	
Basis	Unadjusted	Elementally balanced	Unadjusted	Elementally balanced	Unadjusted	Elementally balanced	Unadjusted	Elementally balanced
Yields, % MF coal								
Gases								
H_2S	2.3	2.4	2.3	2.4	2.7	2.1	2.7	2.1
CO_2	0.7	0.7	0.7	0.7	0.6	0.5	0.6	0.5
CO	0.2	0.2	0.2	0.2	0.1	0.1	0.1	0.1
C_1	1.6	1.6	1.6	1.6	1.6	1.6	1.6	1.6
C_2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2
C_3	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2
C_4- C_5	1.0	1.0	1.0	1.0	0.9	0.8	0.9	0.8
NH_3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3
Water	5.3	6.4	5.3	6.4	7.7	9.8	7.7	9.8
Distillates								
C_5-350 °F	3.2	3.2	3.2	3.1	3.9	3.9	3.9	3.9
350-450 °F	4.2	4.0	4.0	4.0	4.2	4.1	4.1	4.0
450-950 °F	18.6	17.5	15.9	16.6	13.9	12.6	11.0	10.5
SRC								
Oil	12.3	12.2	14.3	14.0	12.1	11.8	12.3	12.1
Asphaltene	22.4	22.1	22.2	21.7	18.0	17.8	26.1	25.5
Benzene insoluble	12.7	12.6	12.6	12.2	17.9	17.7	12.0	11.8
Ash	10.5	11.1	11.6	11.4	10.1	10.9	10.7	11.1
Unreacted coal	4.8	5.1	4.9	4.8	6.3	6.8	6.3	6.6
Hydrogen consumption, % MF coal	2.4	2.7	2.4	2.7	2.7	3.0	2.7	2.9
Organic liquid yield[e]								
Distribution, % of total liquid product[f]								
IBP-350 °F	12.4	12.8	13.9	13.2	17.7	18.8	20.5	21.2
350-450 °F	16.0	16.2	17.3	16.7	19.1	20.0	21.4	21.9
450-550 °F	- 22.9	- 26.8	- 30.8	- 28.0	33.3	32.2	31.7	31.2
550-650 °F	26.0	26.2	25.8	26.0	14.9	14.3	13.0	12.6
650°F-EP	68.5	71.6	73.8	72.1	15.0	14.7	13.4	13.1

Table 7
Kerr-McGee CSD Unit Yield Data
Indiana V Coal

Run	143		147	
Feed Rate, lb/hr	273		256	
Yields	% MF Coal[h]	% of Feed	% MF Coal[h]	% of Feed
SRC	40.8	64.8	35.9	55.2
oil	10.8	17.1	10.0	15.3
asphaltene	23.2	36.9	22.3	34.2
preasphaltene	6.3	9.9	3.5	5.5
undissolved coal	0.1	0.2	0.0	0.1
ash	0.3	0.5	0.0	0.0
deashing solvent	0.1	0.2	0.1	0.1
Ash Concentrate	22.5	35.6	29.3	45.1
oil	1.4	2.2	1.4	2.2
asphaltene	1.3	2.0	1.0	1.6
preasphaltene	4.4	7.1	9.8	15.1
undissolved coal	5.0	7.9	6.8	10.5
ash	10.3	16.2	10.1	15.5
deashing solvent	0.1	0.2	0.2	0.2
SRC Recovery[i]		83.5		72.5
Distillate[j]				
in SRC, wt %	10.0			6.1
in light SRC, wt %	9.3			20.0
Deashing Solvent Loss, %				
total		5.9		4.7
to products		0.4		0.4

Table 8
Preheater Gas Bypass Runs Data
9-16 July 1978

							B102 Preheater						Results, B102 Preheater				R101 Dissolver				
Test	Feed Gas, scfh	Gas Bypassed, scfh	Date, July	Time	Slurry % MF	Coal MF lb/hr	Inlet press psig	ΔP Mid psi	ΔP Top psi	ΔP Total psi	Outlet temp, °F	Mix °F	Conv, %	SRC, wt %	SRC[k] yield %	Sulfur in SRC, %	inlet °F	outlet °F	Conv % MAF	SRC[k] Yield	Sulfur in SRC, %
A	10,430	0	10	0225	36.6	310	2,420	14.1	9.0	61.5	748	734	64.5	30.7	67.8	0.96	744	848	93.3	42.5	0.50
B	10,340	0	11	0245	33.5	422	2,450	9.0	14.4	63.0	760	743	57.7	17.4	48.0	1.40	745	845	93.9	61.3	0.61
C	10,160	0	11	1530	37.2	773	2,450	9.6	18.3	55.5	756	749	56.7	15.9	39.8	1.34	750	846	93.3	78.8	0.74
D	9,430	1,230	11	2130	37.4	312	2,390	8.4	6.3	51.0	803	752	74.6	20.5	131.7	1.25	754	831	94.6	42.3	0.55
E	8,200	2,140	12	0210	37.7	500	2,450	10.8	9.3	55.5	800	741	64.0	28.5	98.5	1.21	748	844	93.4	51.6	0.69
F	7,620	3,250	12	0918	38.1	812	2,450	9.9	13.5	57.0	800	750	60.5	28.6	211.9	-	758	856	93.9	56.0	0.72
G	3,030	2,070	12	1740	36.8	288	2,450	5.1	3.0	25.5	832	743	84.3	34.8	92.2	1.24	748	840	94.7	62.8	2.24
H	3,130	2,340	13	0200	35.3	464	2,460	6.0	4.8	40.5	815	742	72.6	21.4	51.0	1.19	755	848	93.7	61.8	0.57
I	3,070	2,530	13	1125	37.4	785	2,460	8.4	9.0	49.5	787	726	74.1	22.6	69.8	1.29	753	849	94.2	65.6	0.68
J	0	5,450	13	2030	36.4	382	2,460	4.5	2.1	21.0	850	678	88.9	23.5	69.4	1.28	692	829	94.3	66.1	0.87
K	0	4,800	14	0335	36.8	492	2,445	8.1	3.3	33.0	848	720	90.6	31.9	45.8	1.36	733	824	93.9	54.6	0.76
L	0	5,120	14	1330	36.7	747	2,440	9.6	13.2	36.0	837	751	72.6	23.3	38.7	1.39	763	848	93.5	74.2	0.80
M	0	6,320	14	1905	26.7	334	2,450	3.0	2.7	22.5	846	760	87.3	21.7	63.1	0.95	762	827	94.4	80.2	0.73
N	0	3,540	15	0001	26.7	444	2,450	3.3	5.1	25.5	848	788	93.3	21.8	53.3	1.31	786	846	94.1	49.4	0.77
I-I	0	4,940	15	1620	37.1	835	2,430	8.4	>30.0	70.5	829	755	73.9	26.6	136.9	1.36	764	845	93.6	77.7	0.81
L-L	0	5,250	15	1025	36.4	765	2,490	8.1	25.2	57.0	854	761	78.2	26.0	128.4	1.34	763	851	93.7	62.9	0.84
O	0	3,670	16	0100	26.4	857	2,480	3.6	8.7	33.0	847	788	79.0	23.4	146.0	1.17	777	856	03.5	71.1	0.88

Table 9
Dissolver Bypass Runs Data

Run	145		146
Date, 1978	29 Aug		29 Aug
Time Period	0430-1300		1720-2205
Solvent			
IBP, °F		374	
EP, °F		838	
% minus 450°F/% plus 650°F		6.7/20.1	
Specific gravity		1.005	
H/C atomic ratio		1.26	
Microautoclave conversion, %			
short run (kinetic)		78.1	
long run (equilibrium)		72.7	
Feed			
Coal rate, MF lb/hr	287		580
Concentration, % MF	35.8		36.2
Gas rate, Mscf/ton MF coal			
to preheater inlet	10.5		3.5
Hydrogen purity, mol %	87		87
Reaction Conditions			
Coal space rate,(l) MF lb/hr-ft^3	73		147
Temperature, °F			
Turn 15 skin/fluid	629/587		628/598
Turn 23 skin/fluid	713/616		717/599
Turn 31 skin/fluid	797/782		795/778
Turn 37 skin/fluid	853/817		869/822
Outlet fluid	856		860
Pressure, psig			
Outlet	2,370		2,380
Pressure Drop, psi			
Inlet to turn 23	5		8
Turn 23 to 31	4		7
Turn 31 to outlet	2		3
Results			
Conversion, % MAF coal	94.2		92.8
Hydrogen consumption,(m) % MAF coal	1.6		0.8
SRC yield, (n) % MAF coal	77		79
Sulfur in SRC, %	1.3		1.3

(a) Using 1.6 ft^3 as preheater reaction volume, plus 1.5 ft^3 for full-dissolver transfer line (2.3 ft^3 for dissolver bypass line) plus dissolver volume.
(b) Using 18.11 ft^3 as full-dissolver volume.
(c) Based upon unadjusted yields, process method.
(d) Estimated from density gauge correlation.
(e) Liquid fractions by GC determination.
(f) A negative value indicates that the amount of that fraction decreased because part of that fraction was consumed in the process.
(g) From high pressure sample taken at outlet of slurry preheater.
(h) Based upon adjusted coal feed and KM feed (from T102 bottom) rates which produced elementally balanced material balances.
(i) Percent of cresol soluble feed recovered in SRC products.
(j) @ 600°F and 0.1 mm Hg.
(k) Wt % MAF coal, based on ash balance.
(l) Using 1.6 ft^3 as preheater section volume plus 2.3 ft^3 for transfer line.
(m) Based upon on-line hydrogen analyzers and gas flow rates.
(n) Based upon laboratory distillation of filtered V110 sample at 600°F, 0.1 mm Hg absolute; and using V110 drumout weights.

Table 10
Coal Feed Summary
Indiana V and Kentucky 6 and 11 Coals

Run	Feed Slurry Conc.		Coal Feed, Pounds			Weighted Average, wt %		
	MF lb/hr	% MF Coal	As-is	MF	MAF	Moist.	Ash	Sulf.
140[a]	436	37.6	34,506	33,421	29,943	3.1	10.4	3.5
141	520	35.6	125,919	122,974	110,066	2.3	10.5	3.1
142	453	37.3	109,232	106,345	95,477	2.6	10.2	3.3
143	463	37.3	255,217	250,614	221,653	1.8	11.6	3.5
144	439	36.7	60,204	59,293	52,700	1.5	11.1	3.9
145	371	35.9	4,253	4,187	3,746	1.6	10.5	3.6
146	489	36.7	4,607	4,546	4,050	1.3	10.9	3.6
147	449	37.9	49,594	48,627	43,199	2.0	11.2	4.0
148[b]	297	36.8	40,968	40,119	35,297	2.1	12.0	3.1
149[c]	439	36.6	14,138	14,036	12,533	0.7	10.7	2.9

(a) July portion of run.

(b) Started run on Indiana V; changed to Kentucky 6 and 11 coal on 27 September at 1200 hr.

(c) Kentucky 6 and 11 coal; September portion of run.

Table 11

SRC Production Summary

Run	Coal	MCIF, lb/hr K125	MCIF, lb/hr KM	MCIF yield, % MAF coal	Analysis, wt % Solv	Analysis, wt % CI	Analysis, wt % Ash	Analysis, wt % S	MP °F	SRC Pounds[a] MF K125	SRC Pounds[a] MF KM	SRC Pounds[a] MAF K125	SRC Pounds[a] MAF KM	SRC Pounds[a] MCIF K125	SRC Pounds[a] MCIF KM
140[b]	Ind. V	200	-	51.3	3.3	0.2	0.2	0.8	362	15,385	0	15,356	0	15,355	0
141	Ind. V	157	109	57.1	6.6	23.8	15.0	1.9	326	48,777	33,719	41,459	28,661	37,168	25,694
142	Ind. V	198	45	59.7	7.0	19.8	11.8	1.7	313	58,009	13,095	51,153	11,547	46,508	10,500
143	Ind. V	151	90	58.8	7.1	21.3	15.4	2.0	341	101,685	64,069	87,177	53,068	81.603	48,787
144	Ind. V	88	128	55.3	7.3	24.3	17.3	2.4	353	15,771	22,708	12,982	18,825	11,881	17,265
145[c]	Ind. V	216	-	65.0	-	24.0	17.0	-	-	3,205	0	2,660	0	2,436	0
146[c]	Ind. V	355	-	81.5	-	24.0	17.0	-	-	4,340	0	3,603	0	3,299	0
147	Ind. V	66	208	68.9	8.3	23.3	15.3	2.4	372	9,408	29,380	7,983	24,884	7,202	22,566
148[d]	Ind. V/Ky 6/11	99	38	52.3	-	22.5	16.0	-	-	17,246	6,578	14,495	5,529	13,366	5,098
149[e]	Ky 6 and 11	123	83	68.5	5.2	22.1	14.6	1.8	342	7,621	3,404	6,504	2,907	5,933	2,651
Averages		148	87	58.7	7.2	21.4	14.4	1.9	344						
Totals										281,447	172,953	243,372	145,421	224,751	132,561

(a) T102 bottoms.
(b) July portion of run.
(c) Calculated SRC (drums plus transition).
(d) Equipment calibration run.
(e) September portion of run.

Table 12
Solvent Refined Coal Analyses

Date, 1978	20-22 August			7-9 September		
Run	143			147		
Sample	KM-Feed	KM-SRC	V110(a)	KM-Feed	KM-SRC	V110(a)
Proximate Analysis, wt %						
Volatile Matter	-	48.76	47.66	-	47.00	44.97
Fixed Carbon	-	50.27	52.29	-	52.88	54.65
Ash	-	0.97	0.05	-	0.12	0.37
Ultimate Analysis, wt %						
Carbon	71.53	87.57	86.51	72.95	87.85	86.76
Hydrogen	4.93	6.34	6.17	4.90	6.27	5.98
Nitrogen	1.52	1.76	1.32	1.69	1.88	1.80
Sulfur	2.31	0.80	0.75	2.50	0.79	0.85
Ash	16.70	0.75	0.21	15.7	0.06	0.33
Oxygen (by difference)	3.01	2.78	5.04	2.26	3.15	4.28
Heating Value, Btu/lb	13,435	15,502	16,341	13,633	15,928	15,740
Sulfur Forms, wt %						
Pyritic	-	0.02	<0.01	-	<0.01	<0.01
Sulfate	-	<0.01	<0.01	-	<0.01	<0.01
Sulfide	-	0.04	0.01	-	0.04	0.04
Organic	-	0.74	0.74	-	0.75	0.81
Melting Point, °F	356	282	327	390	317	345
Distillate						
at 500°F, wt %	-	1.5	-	-	2.2	-
Vacuum, mm Hg	-	1.0	-	-	1.0	-
at 600°F, wt %	7.3	10.0	-	5.5	6.1	-
Vacuum, mm Hg	0.10	0.15	-	0.10	0.20	-
Solvent Fractionation Analysis, wt %						
Oil(b)	19.9	26.9	29.0	19.0	25.6	24.3
Asphaltenes (c)	36.0	56.7	45.1	28.5	62.9	51.4
Benzene insoluble (d)	20.6	15.3	25.6	28.3	11.3	23.8
Cresol insoluble coal	6.8	0.3	0.1	8.5	0.1	0.2
Ash	16.7	0.8	0.2	15.7	0.1	0.3

(a) Laboratory filtered and vacuum distilled.
(b) Pentane soluble.
(c) Benzene soluble, pentane insoluble.
(d) Cresol-soluble.

Table 13
Feed Coal Analyses
Indiana V Coal

Date, 1978	20-22 August	7-9 September
Run	143	147
Proximate Analysis, wt %		
Moisture	2.34	2.59
Ash	11.65	10.86
Volatile Matter	36.03	34.71
Fixed Carbon	49.98	51.84
Ultimate Analysis, wt %		
Carbon	68.42	67.29
Hydrogen	4.60	4.28
Nitrogen	1.22	1.15
Sulfur	3.58	3.73
Ash	11.40	10.60
Oxygen (by difference)	10.78	12.95
Dry Heating Value, Btu/lb	12,735	12,486
Sulfur Forms, wt %		
Pyritic	1.75	1.39
Sulfate	0.19	0.20
Sulfide	0.01	<0.01
Organic	1.63	2.14
Mineral Analysis, wt %		
Phos. pentoxide, P_2O_5	0.11	0.18
Silica, SiO_2	45.64	51.26
Ferric oxide, Fe_2O_3	25.30	19.08
Alumina, Al_2O_3	20.27	19.19
Titania, TiO_2	1.15	1.17
Lime, CaO	3.24	2.69
Magnesia, MgO	0.86	0.83
Sulfur Trioxide, SO_3	0.25	3.35
Potassium oxide, K_2O	1.36	1.61
Sodium oxide, Na_2O	0.63	0.43
Undetermined	1.19	0.21

Table 14
Slurry Preheater Operating Data
Preheater Gas Bypass Tests
Indiana V Coal

Date, July 1978	10	11	11	11	12	12	12	13
Time	0100	0100	1500	2100	0100	0900	1700	0100
Test/Run	A	B	C	D	E	F	G	H
Fluid	$Slurry/H_2$	$Slurry/H_2$	$Slurry/H_2$	$Slurry/H_2$	$Slurry/H_2$	$Slurry/H_2$	$Slurry/H_2$	$Slurry/H_2$
Temperature[(a)] °F								
Feed inlet								
TR 344-7	135	135	150	130	135	150	150	152
Turn 7 skin/fluid								
TR 344-6/5	350/315	345/308	375/330	385/340	375/338	385/340	420/370	395/358
Turn 7 skin/flud								
Δt	35	37	45	45	37	45	50	37
Turn 15 skin/fluid								
TR 401-1/2	537/501	515/476	522/476	575/537	553/515	545/500	595/558	572/533
Turn 15 skin/fluid								
Δt	36	39	46	38	38	45	37	39
Turn 19 skin/fluid								
TR 401-3/4	605/542	577/518	580/515	662/574	617/556	604/539	676/595	636/573
Turn 19 skin/fluid								
Δt	63	59	65	88	61	65	81	63
Turn 23 skin/fluid								
TR 401-5/6	627/527	614/546	618/550	659/531	651/532	647/533	682/561	664/548
Turn 23 skin/fluid								
Δt	100	68	68	128	119	114	121	116
Turn 27 skin/fluid								
TR401-7/8	687/566	678/575	680/581	716/602	713/599	714/603	743/647	725/617
Turn 27 skin/fluid								
Δt	121	103	99	114	114	111	96	108
Turn 31 skin/fluid								
TR 401-9/10	695/679	692/675	715/693	741/725	729/712	746/722	759/745	747/733
Turn 31 skin/fluid								
Δt	16	17	22	16	17	24	14	14
Turn 33 skin/fluid								
TR 401-11/12	712/669	704/661	723/670	757/718	748/708	755/701	774/727	763/721
Turn 33 skin/fluid								
Δt	43	43	53	39	40	54	47	42
Turn 35 skin/fluid								
TR 401-13/14	740/695	747/684	773/699	785/737	783/731	798/734	800/754	796/739
Turn 35 skin/fluid								
Δt	45	63	74	48	52	64	46	57
Turn 37 skin/fluid								
TR 401-15/16	766/723	764/715	785/722	803/760	802/753	816/761	815/781	818/746
Turn 37 skin/fluid								
Δt	43	49	63	43	49	55	34	72

Table 14 (continued)
Slurry Preheater Operating Data
Preheater Gas Bypass Tests
Indiana V Coal

Date, July 1978	13	13	14	14	14	14	15	15	16
Time	1100	1900	0300	1100	1900	2300	1500	0900	0100
Test/Run	I	J	K	L	M	N	I-I	L-L	0
Fluid	Slurry/H_2	Slurry/H_2	Slurry/H_2	Slurry/H_2	Slurry/H_2	Slurry/H_2	Slurry/H_2	Slurry/H_2	Slurry/H_2
Temperature[a] °F									
Feed inlet									
TR-344-7	155	150	150	160	140	155	150	160	150
Turn 7 skin/fluid									
TR 344-6/5	390/350	420/380	413/372	435/385	406/370	415/370	430/380	440/385	405/360
Turn 7 skin/fluid									
Δt	40	40	40	50	36	45	50	55	45
Turn 15 skin/fluid									
TR 401-1/2	550/509	616/573	611/567	598/556	599/562	586/549	595/551	605/562	568/514
Turn 15 skin/fluid									
Δt	41	43	44	42	37	37	44	43	54
Turn 19 skin/fluid									
TR 401-3/4	605/540	691/604	681/597	667/577	665/612	647/590	652/583	682/586	624/555
Turn 19 skin/fluid									
Δt	65	87	84	90	53	57	69	96	69
Turn 23 skin/fluid									
TR 401-5/6	645/549	701/573	704/584	694/570	701/613	692/618	691/569	704/578	687/607
Turn 23 skin/fluid									
Δt	96	128	120	124	88	74	122	126	80
Turn 27 skin/fluid									
TR 401-7/8	708/596	760/669	753/668	746/644	762/677	754/661	748/639	757/659	755/642
Turn 27 skin/fluid									
Δt	112	91	85	102	85	93	109	98	113
Turn 31 skin/fluid									
TR 401-9/10	736/716	780/765	779/767	772/730	784/776	784/771	778/763	782/770	801/783
Turn 31 skin/fluid									
Δt	20	15	12	42	8	13	15	12	18
Turn									
TR401-11/12	750/699	797/759	791/753	794/745	799/769	804/768	798/753	808/762	817/764
Turn 33 skin/fluid									
Δt	51	38	38	49	30	36	45	46	53
Turn 35 skin/fluid									
TR 401-13/14	790/739	823/778	827/779	842/773	829/784	844/786	842/777	853/783	870/794
Turn 35 skin/fluid									
Δt	60	45	48	69	45	58	65	70	76
Turn 37 skin/fluid									
TR 401-15/16	807/754	842/806	845/808	869/792	844/809	865/812	883/801	880/805	909/818
Turn 37 skin/fluid									
Δt	53	36	37	77	35	53	82	75	91

Table 14 (continued)
Slurry Preheater Operating Data
Preheater Gas Bypass Tests
Indiana V Coal

Date, July 1978	10	11	11	11	12	12	12	13
Time	0100	0100	1500	2100	0100	0900	1700	0100
Test/Run	A	B	C	D	E	F	G	H
Fluid	Slurry/H_2	Slurry/H_2	Slurry/H_2	Slurry/H_2	Slurry/H_2	Slurry/H_2	Slurry/H_2	Slurry/H_2
B102 outlet								
TR401-17[b] °F	748	726	756	801	798	800	821	808
(TE 344-12)								
TR 401-18	765	756	761	803	799	800	822	815
(TE 329)								
TR 401-19	760	758	762	806	802	800	821	817
(TE 368)								
Stack TR 308	870	920	1025	880	940	1050	885	930
Pressure, psig								
Inlet	2420	2450	2450	2390	2450	2450	2450	2460
Outlet (by difference)	2358	2387	2394	2339	2394	2393	2424	2420
Pressure drop, ΔP, psi								
Inlet to turn 23 [c]	38.4	39.6	27.6	36.3	35.4	33.6	17.4	29.7
Turn 23 to turn 31, DPT 370	14.1	9.0	9.6	8.4	10.8	9.9	5.1	6.0
Turn 31 to coil outlet, DPT 371	9.0	14.4	18.3	6.3	9.3	13.5	3.0	4.8
Total, DPT 369	61.5	63.0	55.5	51.0	55.5	57.0	25.5	40.5
Fuel oil, gph								
FR 307	7.5	9.3	12.6	6.9	9.3	12.9	6.6	8.6

Table 14 (continued)
Slurry Preheater Operating Data
Preheater Gas Bypass Tests
Indiana V Coal

Date, July 1978	13	13	14	14	14	14	15	15	16
Time	1100	1900	0300	1100	1900	2300	1500	0900	0100
Test/Run	I	J	K	L	M	N	I-I	L-L	O
Fluid	Slurry/H_2	Slurry/H_2	Slurry/H_2	Slurry/H_2	Slurry/H_2	Slurry/H_2	Slurry/H_2	Slurry/H_2	Slurry/H_2
B102 outlet									
TR 401-17[b] °F (TE 344-12)	780	845	847	836	847	851	768	841	850
TR 401-18 (TE 329)	793	850	850	840	849	853	828	851	850
TR 401-19 (TE 368)	790	842	827	813	830	839	799	821	829
Stack TR 308	1010	900	950	1040	920	1010	1045	1050	1180
Pressure, psig									
Inlet	2460	2460	2445	2440	2450	2450	2430	2490	2480
Outlet (by difference)	2410	2439	2412	2404	2427	2425	2360	2433	2447
Pressure drop, ΔP, psi									
Inlet to turn 23[c]	32.1	14.4	21.6	13.2	16.8	17.1	< 32.1	23.7	20.7
Turn 23 to turn 31, DPT 370	8.4	4.5	8.1	9.6	3.0	3.3	8.4	8.1	3.6
Turn 31 to coil outlet, DPT 371	9.0	2.1	3.3	13.2	2.7	5.1	> 30.0	25.2	8.7
Total, DPT 369	49.5	21.0	33.0	36.0	22.5	25.5	70.5	57.0	33.0
Fuel oil, gph									
FR 307	12.0	7.5	8.1	12.0	7.8	11.4	12.3	12.0	18.3

Table 14 (continued)
Slurry Preheater Operating Data
Indiana V Coal

Date, 1978	20 August	7 September
Time	1900	1700
Test/Run	143	147
Fluid	Slurry/H_2	Slurry/H_2
Temperature[a] °F		
Feed inlet		
TR 344-7	140	140
Turn 7 skin/fluid		
TR 344-6/5	390/340	390/350
Turn 7 skin/fluid		
Δt	50	40
Turn 15 skin/fluid		
TR 401-1/2	543/505	542/513
Turn 15 skin/fluid		
Δt	38	29
Turn 19 skin/fluid		
TR 401-3/4	598/541	598/540
Turn 19 skin/fluid		
Δt	57	58
Turn 23 skin/fluid		
TR 401-5/6	630/515	626/514
Turn 23 skin/fluid		
Δt	115	112
Turn 27 skin/fluid		
TR 401-7/8	692/588	688/586
Turn 27 skin/fluid		
Δt	104	102
Turn 31 skin/fluid		
TR 401-9/10	713/696	711/693
Turn 31 skin/fluid		
Δt	17	18
Turn 33 skin/fluid		
TR 401-11/12	730/691	729/683
Turn 33 skin/fluid		
Δt	39	46
Turn 35 skin/fluid		
TR 401-13/14	769/710	767/702
Turn 35 skin/fluid		
Δt	59	65
Turn 37 skin/fluid		
TR 401-15/16	796/735	791/736
Turn 37 skin/fluid		
Δt	61	55

Table 14 (continued)
Slurry Preheater Operating Data
Indiana V Coal

Date, 1978	20 August	7 September
Time	1900	1700
Test/Run	143	147
Fluid	Slurry/H_2	Slurry/H_2
B102 outlet		
TR 401-17[b] °F	762	766
(TE 344-12)		
TR 401-18	766	768
(TE 329)		
TR 401-19	758	767
(TE 368)		
Stack TR 308	950	940
Pressure, psig		
Inlet	1800	1790
Outlet (by difference)	1752	1748
Pressure drop, ΔP, psi		
Inlet to turn 23[c]	7.5	7.5[d]
Turn 23 to turn 31, DPT 370	18.0	16.5
Turn 31 to coil outlet, DPT 371	22.5	18.0
Total, DPT 369	48.0	42.0[e]
Fuel oil, gph		
FR 307	9.3	7.8

(a) See Figure 3 for location of thermocouples.
(b) Used as outlet temperature.
(c) By difference: total - turn 31 to coil outlet - turn 23 to 31.
(d) DPT 368
(e) Σ (DPT 368, 370 and 371); DPT 369 indicated 39.0 psi (average).

Table 15
Dissolver Temperature Profile
Indiana V Coal

Date, 1978	20-22 Aug			7-9 Sept		
Run	143			147		
Dissolver Volume in use, %	100			100		
Temperature, °F	Min	Avg	Max	Min	Avg	Max
TR-401-17 (Preheater Outlet)	667	758	837	752	767	778
TR-401-21 (Feed Inlet)	738	749	811	735	748	759
TR-401-22 (Dissolver Bottom)	783	786	813	786	799	813
TR-401-23	798	805	812	790	803	813
TR-401-24	795	806	814	794	805	814
TR-401-25	796	807	815	797	808	818
TR-401-26	793	807	814	802	808	819
TR-401-27	796	809	817	804	811	822
TR-401-28	788	812	819	807	814	823
TR-401-29	798	814	820	809	816	827
TR-401-30	788	814	821	811	818	828
TR-401-31	798	815	822	814	820	831
TR-401-32	799	816	823	814	822	832
TR-401-33	790	816	826	815	823	833
TR-401-34	797	817	828	816	823	833
TR-401-35	800	821	831	819	826	836
TR-401-36	801	822	832	820	826	836
TR-401-37 (Dissolver Top)	793	823	832	815	825	834
TR-401-39 (Dissolver Outlet)	785	814	824	800	810	818

Table 16
Reaction Solids Analyses
(V144 Emergency Blowdown Tank)

Coal	Indiana V		Indiana V
Date, 1978	26 August		9 September
Blowdown Solids	V144/AWS	V101A/AWS	V101A/AWS
Quantity, lb	675		97
Proximate Analysis, wt %			
Moisture	0.04	0.07	0.75
Ash	85.57	65.49	42.54
Volatile Matter	7.06	13.38	23.40
Fixed carbon, by difference	7.33	21.06	33.31
Ultimate Analysis, wt %			
Carbon	14.52	29.96	48.22
Hydrogen	0.38	1.59	3.02
Nitrogen	0.11	0.37	1.00
Sulfur	6.35	11.54	4.86
Ash	78.64	56.42	42.00
Oxygen (by difference)	-	0.12	0.90
Sulfur Forms, wt %			
Pyritic	0.03	4.00	0.56
Sulfate	0.39	0.42	0.57
Sulfide	5.65	6.94	1.24
Organic	0.28	0.18	2.49
Mineral Analysis of Ash, wt %			
Phos. pentoxide, P_2O_5	0.12	0.11	0.18
Silica, SiO_2	44.85	30.60	49.94
Ferric oxide, Fe_2O_3	22.01	50.74	21.06
Alumina, Al_2O_3	5.35	8.66	12.21
Titania, TiO_2	0.85	0.67	1.83
Lime, CaO	12.10	3.72	7.73
Magnesia, MgO	0.44	0.50	0.74
Sulfur Trioxide, SO_3	9.11	3.50	3.25
Potassium oxide, K_2O	0.29	0.85	0.78
Sodium oxide, Na_2O	0.61	0.65	1.24
Undetermined	4.27	-	1.04
Carbon dioxide, wt %	13.31	3.63	2.51
Wet Screen Analysis, wt %			
retained on mesh			
50	2.2	0.7	0.06
100	7.0	1.4	0.70
200	39.5	3.9	2.62
270	23.9	4.7	2.88
325	5.6	7.1	6.30
-325	21.8	82.2	87.44
Solids density, gm/cc	-	0.8	-

Table 17
Reaction Section Operating Data
Indiana V Coal

Date	20-21 August	7-8 September
Run	143	147
Flow rates, lb/hr		
MF coal to B102	468	447
Solvent to B102	749	721
Light solvent from V105	193	146
Water from V105	34	36
Gas flow rates, scfh		
Feed gas to B102	10,830	10,440
Makeup hydrogen	3,325	3,165
Recycle gas from V104	12,345	10,730
HP purge gas	1,053	850
LP vent gas from V105	389	315
Degas vent[(a)]	122	87
Gas removed by caustic scrubber	122	140
Temperature, °F		
B102 outlet	763	766
R101 bottom	804	800
R101 middle[(b)]	815	818
R101 top[(b)]	823	825
V103 HP flash	620	600
V104 HP vent	88	82
V110 LP flash	560	555
Pressure, psig		
B102 inlet	1,800	1,790
B102 outlet	1,750	1,750
V110 outlet gas	125	128
V105 outlet gas	7	7
Hydrogen purity, vol %		
Feed gas to B102	87.5	85.4
Recycle gas from V104	83.7	80.6
Recycle gas from V106	84.6	81.5
LP vent gas from V105	37.1	29.7
Degas vent	65.4	68.0

(a) Includes compressor vents, plus on-line GC sample streams.
(b) Of liquid volume.

Table 18
High Pressure Vent Separator Gas Analyses (V104)
Indiana V Coal

Date, 1978		20-22 Aug	7-9 Sept
Run		143	147
Component	mol wt	mole %	mole %
H_2	2.02	83.7	80.6
N_2	28.02	0.1	0.1
CO	28.00	0.6	0.7
CO_2	44.00	0.2	0.3
H_2S	34.08	0.8	1.1
CH_4	16.03	10.6	12.5
C_2H_2	26.02	ND	ND
C_2H_4	28.03	ND	ND
C_2H_6	30.05	2.7	3.2
C_3H_6	42.05	ND	ND
C_3H_8	44.06	1.1	1.3
iC_4H_{10}	58.08	ND	ND
nC_4H_{10}	58.08	0.2	0.2
iC_5H_{12}	72.15	ND	ND
nC_5H_{12}	72.15	ND	ND
C_6H_{14}	86.18	ND	ND

Table 19
Decanter Vent Gas Analyses (V105)
Indiana V Coal

Date, 1978		20-22 Aug	7-9 Sept
Run		143	147
Component	mol wt	mole %	mole %
H_2	2.02	37.1	29.7
N_2	28.02	29.8	39.4
CO	28.00	0.9	0.1
CO_2	44.00	0.3	0.3
H_2S	34.08	4.1	3.5
CH_4	16.03	9.2	9.0
C_2H_2	26.02	ND	ND
C_2H_4	28.03	ND	ND
C_2H_6	30.05	7.1	6.7
C_3H_6	42.05	ND	ND
C_3H_8	44.06	6.6	6.8
iC_4H_{10}	58.08	0.4	0.5
nC_4H_{10}	58.08	2.1	2.8
iC_5H_{12}	72.15	0.3	0.3
nC_5H_{12}	72.15	2.1	0.9
C_6H_{14}	86.18	ND	ND

Table 20
Degas Vent Gas Analyses (V132)
Indiana V Coal

Date, 1978		20-22 Aug	7-9 Sept
Run		143	147
Component	mol wt	mole %	mole %
H_2	2.02	65.4	68.0
N_2	28.02	19.9	13.4
CO	28.00	0.4	0.5
CO_2	44.00	0.2	ND
H_2S	34.08	1.0	ND
CH_4	16.03	9.2	12.9
C_2H_2	26.02	ND	ND
C_2H_4	28.03	ND	ND
C_2H_6	30.05	2.8	3.8
C_3H_6	42.05	ND	ND
C_3H_8	44.06	1.0	1.3
iC_4H_{10}	58.08	ND	ND
nC_4H_{10}	58.08	0.1	0.1
ic_5H_{12}	72.15	ND	ND
nC_5H_{12}	72.15	ND	ND
C_6H_{14}	86.18	ND	ND

Table 21
Flare Gas Analyses (K110)
Indiana V Coal

Date, 1978		20-22 Aug	7-9 Sept
Run		143	147
Component	mol wt	mole %	mole %
H_2	2.02	45.5	35.2
N_2	28.02	47.1	54.1
CO	28.00	0.1	0.1
CO_2	44.00	ND	0.1
H_2S	34.08	0.2	ND
CH_4	16.03	4.1	5.8
C_2H_2	26.02	ND	ND
C_2H_4	28.03	ND	ND
C_2H_6	30.05	1.6	2.5
C_3H_6	42.05	ND	ND
C_3H_8	44.06	1.1	1.7
iC_4H_{10}	58.08	ND	ND
nC_4H_{10}	58.08	0.3	0.5
iC_5H_{12}	72.15	ND	ND
nC_5H_{12}	72.15	ND	ND
C_6H_{14}	86.18	ND	ND

Table 22
Low Pressure Flash (V110) Product Analyses
Indiana V Coal

Date, 1978	20-22 August		7-9 September	
Run	143		147	
Composition, wt %				
Cresol Insoluble (CI)	8.0		7.9	
Ash	5.6		4.9	
SRC[a]	23.6		23.3	
Distillate[b]	68.4		68.8	
Distillate Composition, wt %	GC[c]	ASTM[d]	GC[c]	ASTM[d]
IBP-350 °F	0	0	0	0
350-450 °F	5.1	3.5	4.4	2.5
450-550 °F	41.1	45.5	43.9	54.0
550-650 °F	27.3	23.0	27.1	19.5
650 °F -EP	26.5	28.0	24.6	24.0
Distillate, lab				
Ultimate Analysis, wt %				
Carbon	86.74		86.44	
Hydrogen	9.15		9.06	
Nitrogen	0.61		0.64	
Sulfur	0.29		0.33	
Ash	0.00		0.00	
Oxygen (by difference)	3.21		3.53	
SRC, lab				
Solvent Fractionation and Ultimate Analysis[e]				
Cresol Insoluble, lab				
Ultimate Analysis, wt %				
Carbon	21.34		28.09	
Hydrogen	1.10		1.48	
Nitrogen	0.00		0.23	
Sulfur	5.38		6.74	
Ash	69.60		62.50	
Oxygen (by difference)	2.58		0.96	

(a) Distillation conditions: 316 °C @ 0.1 mm Hg.
(b) Distillate + 100-(CI + SRC).
(c) Fisher 4800 gas chromatographic simulated TBP distillation, weight percent.
(d) ASTM D-86 method volume percent.
(e) See Table 12.

Table 23
Filter Performance Summary
Indiana V Coal

Date, 1978	Run No.	No of Cyc	Filt time, min	Cycle time, min	Wash solv, lb/cyc	V111 Vol Filt, gal	Filtration Rate, gpm	Filtration Rate, gph/ft²	Precoat, lb/cyc	ΔP, psig Precoat range	ΔP, psig Filtration range	ΔP, psig Filtration avg	Filter Cake % solv	Filter Cake % ash	Filter Cake % UCC	Filter Cake % SRC	Filter Cake wt, lb	Solids Filtrate, wt %	Solids Filt Feed, wt %	Filt Temp, °F	Filtrate, Visc, cp	Filtrate, Temp, °F	Filt Feed Temp, °F	Pre-coat
24 July[a]	142	13	11	70	680	273	25	15	45	3-7	17-50	36	-	-	-	-	191	0.04-2.4	-	518	-	521	541	(b)
1 Aug[c]	143	7	10	77	758	218	22	23	27	3-19	8-60	29	-	-	-	-	129	0.06-3.8	-	528	-	523	542	(b)
2		15	20	87	741	232	12	12	27	15-21	44-62	50	6.5	61.8	32.5	8.3	173	0.02-1.9	7.0	541	1.0	527	547	(d)
3		15	18	85	783	238	13	14	27	15-22	49-54	50	3.3	72.7	26.5	3.2	236	0.01-1.21	-	541	1.0	541	549	(d)

(a) Thirty-seven (37) screens, 60 x 60 mesh, 0.0075-in. wire, plain square weave.
(b) 7.5% Solka-Floc with Hyflo Super-Cel.
(c) 2.4% CI in filtrate to 21 screens, 60 x 60 mesh, 0.0075-in. wire, plain square weave.
(d) Fibra-Flo 7-C.

Table 24
Vacuum Column Operating Data
T102
Indiana V Coal

Date, 1978	20-21 Aug	7-8 Sept
Run	143	147
Operating Conditions		
Pressure, psia	1.3	1.2
Flow rates, lb/hr		
Feed		
from V120	972	967
Overhead Light Solvent		
to T104 feed	34	30
Tray 1 reflux	1,600	1,570
Bottoms (SRC)		
to K125	293	288
to B103	14,600	10,600
Temperature, °F		
Tray 1 (ovhd)	192	193
Tray 8	365	365
Packing reflux	188	174
B103 outlet	-	612
Bottom	598	596
Product Pump Power, amps	34	36

Table 25
Fractionating Column Operating Data
T105
Indiana V Coal

Date, 1978	20-21 Aug	7-8 Sept
Run	143	147
Operating Conditions		
Pressure, psig		4.0
Rate, lb/hr		
Feed	845	802
Overhead product	65	68
Reflux	2,700	2,200
Vent	0	0
Reboiler	78,400	77,700
Temperature, °F		
Top	394	411
Reflux	342	331
Middle	420	440
Feed	292	322
Bottom	537	551
Reboiler	545	557
Composition,[a] wt %		
Overhead		
Light organic liquid	9.5	7.6
Wash solvent	87.1	83.8
Bottom		
Light organic liquid	0.2	0.0
Wash solvent	3.0	5.6
Feed		
Light organic liquid	1.8	1.9
Wash solvent	21.1	22.0

(a) By Fisher 4800 Gas Chromatograph.

Table 26
Recovered Solvent Analyses
Indiana V Coal

Date, 1978	20-22 Aug		7-9 Sept	
Run	143		147	
Recycle solvent from T105[a]				
Boiling fractions, wt %				
(IBP, °F)	(406)[e]		(395)[c]	
IBP-350°F	0		0	
350-450°F	3		6	
450-550°F	35		51	
550-650°F	27		26	
650°F - EP	35		17	
(EP, °F)	(989)		(814)	
Specific gravity	1.004		1.005	
Ultimate analysis, wt %				
Carbon	86.67		87.48	
Hydrogen	9.31		9.07	
Nitrogen	1.24		0.74	
Sulfur	0.25		0.43	
Ash	0		0	
Oxygen (by difference)	2.53		2.28	
Wash solvent from T105[b]				
Boiling fractions, wt %	GC[c]	ASTM[d]	GC[c]	ASTM[d]
(IBP, °F)	(197)	-	(216)	-
IBP-350°F	10	30	7	14
350-450°F	87	70	84	86
450-550°F	3	-	9	-
550-650°F	0	-	0	-
650°F - EP	0	-	0	-
(EP, °F)	(463)	-	(473)	-
Specific gravity	0.927		0.937	
Ultimate analysis, wt %				
Carbon	82.67		83.41	
Hydrogen	10.20		10.09	
Nitrogen	1.42		0.58	
Sulfur	0.10		0.23	
Ash	0		0	
Oxygen (by difference)	5.61		5.69	

(a) Bottom Product.
(b) Overhead Product.
(c) Fisher-4800 gas chromatographic simulated TBP distillation, weight percent.
(d) ASTM D-86 method, volume percent.
(e) Perkin-Elmer gas chromatographic simulated TBP distillation, weight percent.

Table 27
Recovered Solvent Compositions (wt %)
Indiana V Coal

Date, 1978	20-22 Aug		7-9 Sept	
Run	143		147	
	T105 Ovhd	T105 Btms	T105 Ovhd	T105 Btms
Component or boiling range, °F				
100-155				
hexane				
157-175				
cyclohexane				
177-230			1	
toluene	1		1	
232-280	1		1	
xylene (p-,m-)				
o-xylene				
292-349	7		5	
indane	17		5	
351-359				
phenol	26		14	
360-383	23		10	
c-decalin	3		23	
385-395	3		3	
cresol				
396-404	4		4	1
tetralin	8		9	2
406-423	2	1	11	1
naphthalene	2		4	1
425-459		4	2	1
quinoline	3	1	3	5
461-465			1	2
2-methylnaphthalene			2	7
1-methylnaphthalene			1	6
473-491		8		
biphenyl		5		8
diphenyl-ether				
497-531		12		13
acenaphthene		2		4
532-548		3		2
dibenzofuran		1		4
549-568		7		3
fluorene		1		3
569-629		14		12
dibenzothiophene		1		2
phenanthrene		1		4

Table 27 (continued)
Recovered Solvent Compositions (wt %)
Indiana V Coal

Date, 1978	20-22 Aug		7-9 Sept	
Run	143		147	
	T105 Ovhd	T105 Btms	T105 Ovhd	T105 Btms
Component or boiling range, °F				
644-675		15		
1-methylphenanthrene		1		1
677-684		2		2
9-methylanthracene		1		3
686-721				5
fluoranthene		3		1
722-739		1		
pyrene		1		2
741-837		10		2
chrysene		1		
unknowns > chrysene		4		

Table 28
Organic Liquid Analyses
Indiana V Coal

Date, 1978	20-22 Aug		7-9 Sept	
Run	143		147	
Organic liquid from V105	GC[a]	ASTM[b]	GC[a]	ASTM[b]
Boiling fractions, wt %				
(IBP, °F)	(148)	-	(146)	-
IBP-200°F	5	-	9	-
200-350°F	3	6	3	12
350-450°F	19	29	16	26
450°F - EP	73	65	72	62
(EP, °F)	(699)	-	(696)	-
Specific Gravity	0.940		0.937	
Ultimate Analysis, wt %				
Carbon	85.96		87.01	
Hydrogen	10.00		10.14	
Nitrogen	1.05		0.56	
Sulfur	0.26		0.26	
Ash			0.03	
Oxygen (by difference)	2.73		2.00	
Organic liquid from T102 ovhd				
Boiling fractions, wt %				
(IBP, °F)	(231)		(231)	
IBP-200°F	0		0	
200-350°F	6		3	
350-450°F	80		79	
450°F - EP	14		18	
(EP, °F)	(473)		(492)	
Specific Gravity	0.921		0.920	

(a) Fisher-4800 gas chromatographic simulated TBP distillation, weight percent.
(b) ASTM D-86 method, volume percent.

Table 29
Organic Liquid Compositions (wt %)
Indiana V Coal

Date, 1978	20-22 Aug		7-9 Sept	
Run	143		147	
	V105 Decanter	T102 Ovhd	V105 Decanter	T102 Ovhd
Component or boiling range, °F				
100-155	1	-	3	-
hexane	1	-	2	-
157-175	-	-	-	-
cyclohexane	2	-	2	-
177-230	1	-	2	-
toluene	1	-	1	1
232-280	1	1	1	1
xylene (p-,m-)	-	-	-	-
o-xylene	-	-	-	-
292-349	1	4	-	1
indane	1	10	-	3
351-359	-	-	-	-
phenol	1	12	1	13
360-383	3	-	1	7
c-decalin	-	16	3	15
385-395	-	6	-	3
cresol	-	-	-	-
396-404	2	7	1	4
tetralin	5	13	4	11
406-423	2	1	-	8
naphthalene	2	7	2	6
425-459	2	8	4	8
quinoline	9	8	9	3
461-465	2	2	2	8
2-methylnaphthalene	9	4	10	5
1-methylnaphthalene	6	1	4	1
473-491	-	-	3	-
biphenyl	9	-	9	1
diphenyl-ether	-	-	-	-
497-531	13	-	16	-
acenaphthene	4	-	2	-
532-548	-	-	-	-
dibenzofuran	2	-	3	-
549-568	5	-	2	-
fluorene	2	-	4	-
569-629	7	-	5	-
dibenzothiophene	1	-	1	-
phenanthrene	2	-	1	-
644-675	-	-	-	-
1-methylphenanthrene	1	-	1	-
9-methylanthracene	1	-	1	-
686-721	1	-	-	-
fluoranthene	-	-	-	-
722-739	-	-	-	-
pyrene	-	-	-	-
741-837	-	-	-	-
chrysene	-	-	-	-

Table 30
T104 Light Solvent Recovery Column Operating Data
Indiana V Coal

Date, 1978	20-21 Aug	7-8 Sept
Run	143	147
Operating Conditions		
Pressure, psig	5.6	7.8
Flow rates, lb/hr		
Feed		
from V105	193	146
from T102	34	30
Overhead		
Product	26	23
Reflux[a]	-	-
Vent	-	-
Temperature, °F		
Top	311	306
Middle	313	300
Bottom	506	500
Feed	70	70
Composition,[b] wt %		
Feed		
IBP-350°F	9.0	9.5
350-450°F	22.0	21.5
450°F - EP	69.0	69.0
Water in feed		
from V105	0.76	4.41
from T102	0.75	0.8
Overhead oil		
IBP-350°F	75.3	96.49
350-450°F	24.7	3.51
450°F - EP	0.0	0.0
Bottom		
IBP-350°F	1.6	1.16
350-450°F	22.8	24.53
450°F - EP	75.6	74.31
Specific gravity		
Overhead	0.790	0.780
Bottom	0.996	0.960

(a) T104 Reflux shut down.
(b) Fisher-4800 gas chromatograph simulated distillation.

Table 31
Organic Liquid Product Analyses

Date, 1978	20-22 August		7-9 September	
Run	143		147	
Recycle solvent (V131)				
Boiling fractions, wt %	GC [(a)]	ASTM [(b)]	GC [(a)]	ASTM [(b)]
(IBP, °F)	(376)		(400)	-
IBP-350 °F	0	0	0	0
350-450 °F	7	2	5	7
450-550 °F	55	50	48	43
550-650 °F	25	26	26	33
650°F - EP	13	22	21	17
(EP, °F)	(789)	-	(814)	-
Specific gravity	1.001			
Ultimate analysis, wt %				
Carbon	87.40		86.88	
Hydrogen	9.31		9.20	
Nitrogen	1.36		0.77	
Sulfur	0.33		0.35	
Ash	0.00		0.00	
Oxygen (by difference)	1.60		2.80	
Light Organic Liquid from T104 overhead				
Boiling fractions, wt %	GC [(a)]	ASTM [(b)]	GC [(a)]	ASTM [(b)]
(IBP, °F)	(147)	-	(148)	-
IBP-200 °F	43	0	50	-
200-350 °F	32	95	46	100
350-450 °F	25	5	4	-
450°F - EP	0	-	0	-
(EP, °F)	(374)	-	(360)	-
Specific gravity	0.791		0.778	
Ultimate analysis, wt				
Carbon	83.89		85.44	
Hydrogen	12.86		13.37	
Nitrogen	0.86		0.53	
Sulfur	0.18		0.23	
Ash	0.00		0.00	
Oxygen (by difference)	2.21		0.43	

(a) Fisher - 4800 gas chromatographic simulated TBP distillation, weight percent.
(b) ASTM D-86 method, volume percent.

Table 32
Organic Liquid Product Compositions (wt %)
Indiana V Coal

Date, 1978	20-22 Aug		7-9 Sept	
Run	143		147	
	V131	T104 Ovhd	V131	T104 Ovhd
Component or boiling range, °F				
100-155	-	6	-	7
hexane	-	11	-	13
157-175	-	-	-	-
cyclohexane	-	-	-	17
177-230	-	27	-	14
toluene	-	11	-	12
232-280	-	7	-	9
xylene (p-,m-)	-	2	-	3
292-349	-	11	-	22
indane	-	3	-	-
351-359	-	-	-	-
phenol	-	15	-	3
360-383	1	7	-	-
c-decalin	-	-	-	-
385-395	-	-	-	-
cresol	-	-	-	-
396-404	1	-	1	-
tetralin	1	-	1	-
406-423	2	-	1	-
naphthalene	1	-	1	-
425-459	1	-	2	-
quinoline	5	-	4	-
461-465	2	-	2	-
2-methylnaphthalene	8	-	7	-
1-methylnaphthalene	-	-	5	-
473-491	6	-	-	-
biphenyl	10	-	8	-
diphenyl-ether	-	-	-	-
497-531	14	-	16	-
acenaphthanene	4	-	2	-
532-548	2	-	-	-
dibenzofuran	4	-	4	-
549-568	6	-	3	-
fluorene	4	-	3	-
569-629	11	-	12	-
dibenzothiophene	1	-	2	-
phenanthrene	1	-	5	-
644-675	-	-	3	-
1-methylphenanthrene	3	-	1	-
9-methylanthracene	2	-	2	-
686-721	2	-	5	-
fluoranthene	-	-	1	-
722-739	-	-	-	-
pyrene	1	-	2	-
741-837	1	-	2	-
chrysene	-	-	-	-
unk > chrysene	6	-	5	-

Table 33
Ash Concentrate Analyses (KM-CSD Unit)
Indiana V Coal

Date, 1978	20-21 Aug	7-8 Sept
Run	143	147
<u>Composition, wt %</u>		
Ash	44.8	35.4
Undissolved coal	21.8	24.0
Solvent refined coal	28.0	35.3
Solvent	4.9	4.8
Deashing solvent	0.5	0.5
<u>Solvent Extraction Analysis, wt %</u>		
Oil	6.8	5.4
Asphaltenes	5.6	3.5
Benzene insoluble (cresol soluble)	19.8	33.5
Cresol insoluble	22.2	23.3
Ash	45.6	34.3
<u>Ultimate Analysis, wt %</u>		
Carbon	44.76	54.5
Hydrogen	2.58	3.03
Nitrogen	0.56	1.34
Ash	45.6	34.3
Sulfur	5.19	4.63
Oxygen (by difference)	1.31	2.2

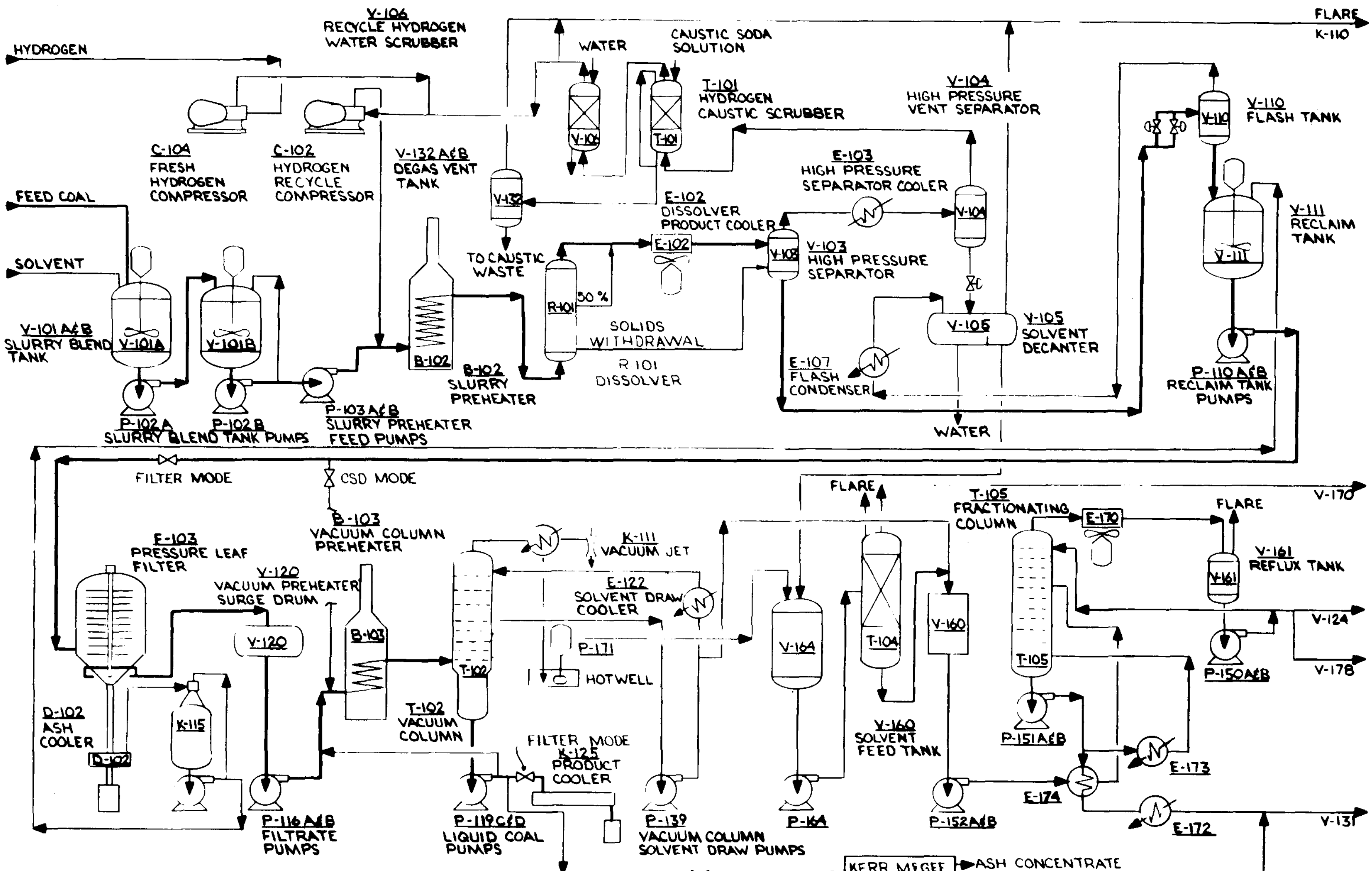

FIGURE 1: SRC FLOWSHEET

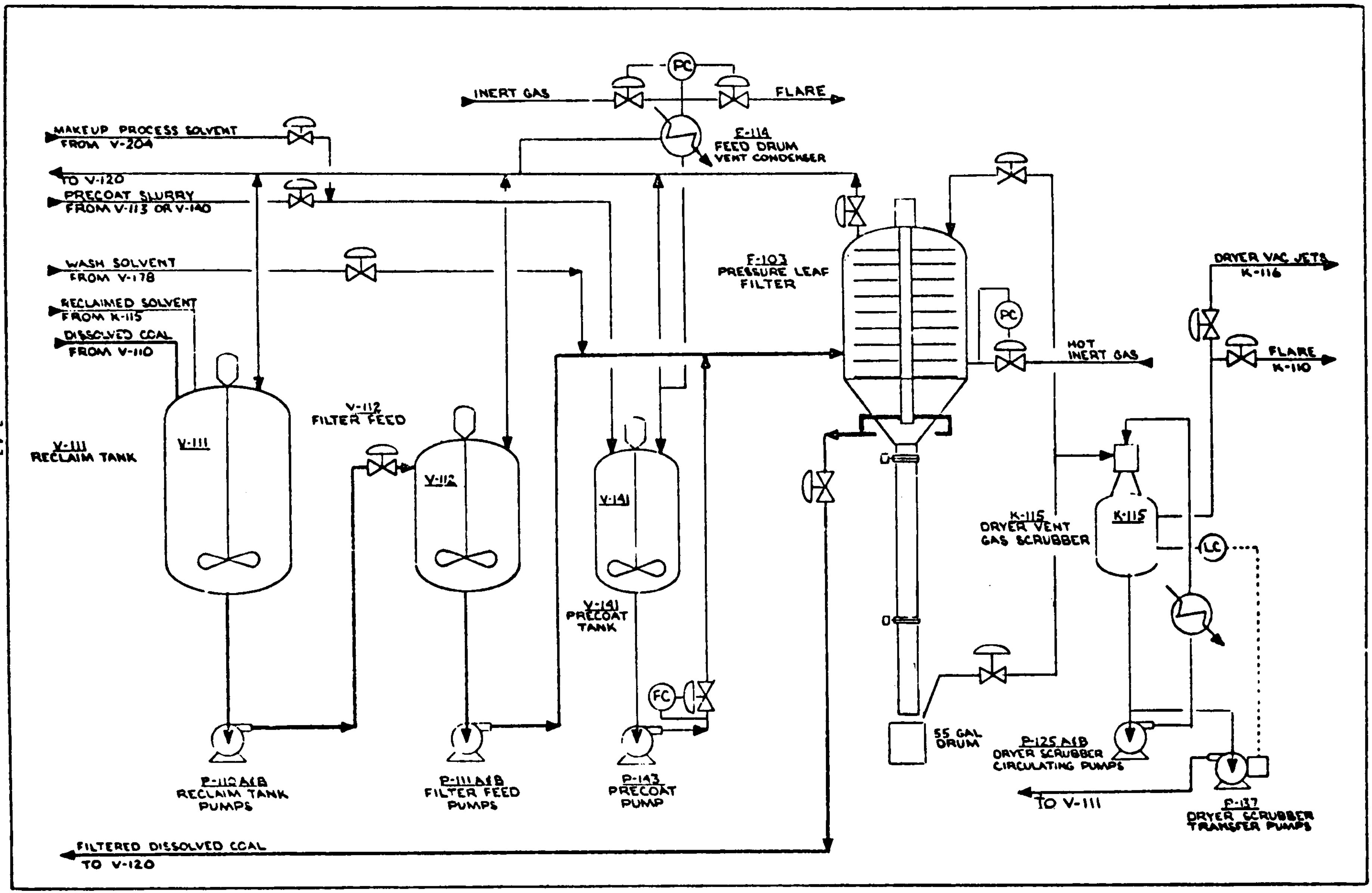

FIGURE 2: FILTRATION FLOWSHEET (FUNDA FILTER)

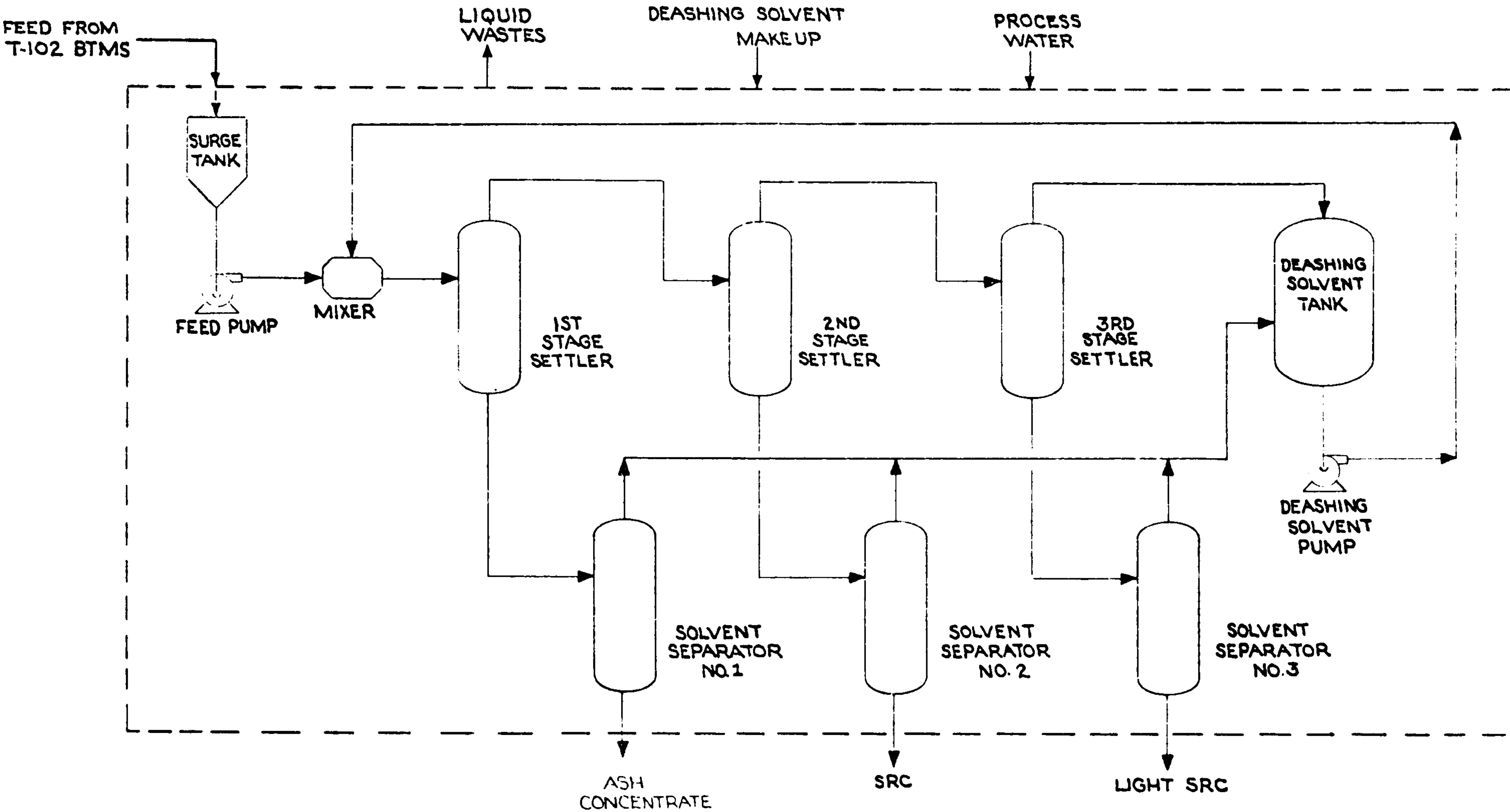

FIGURE 3: CRITICAL SOLVENT DEASHING FLOWSHEET

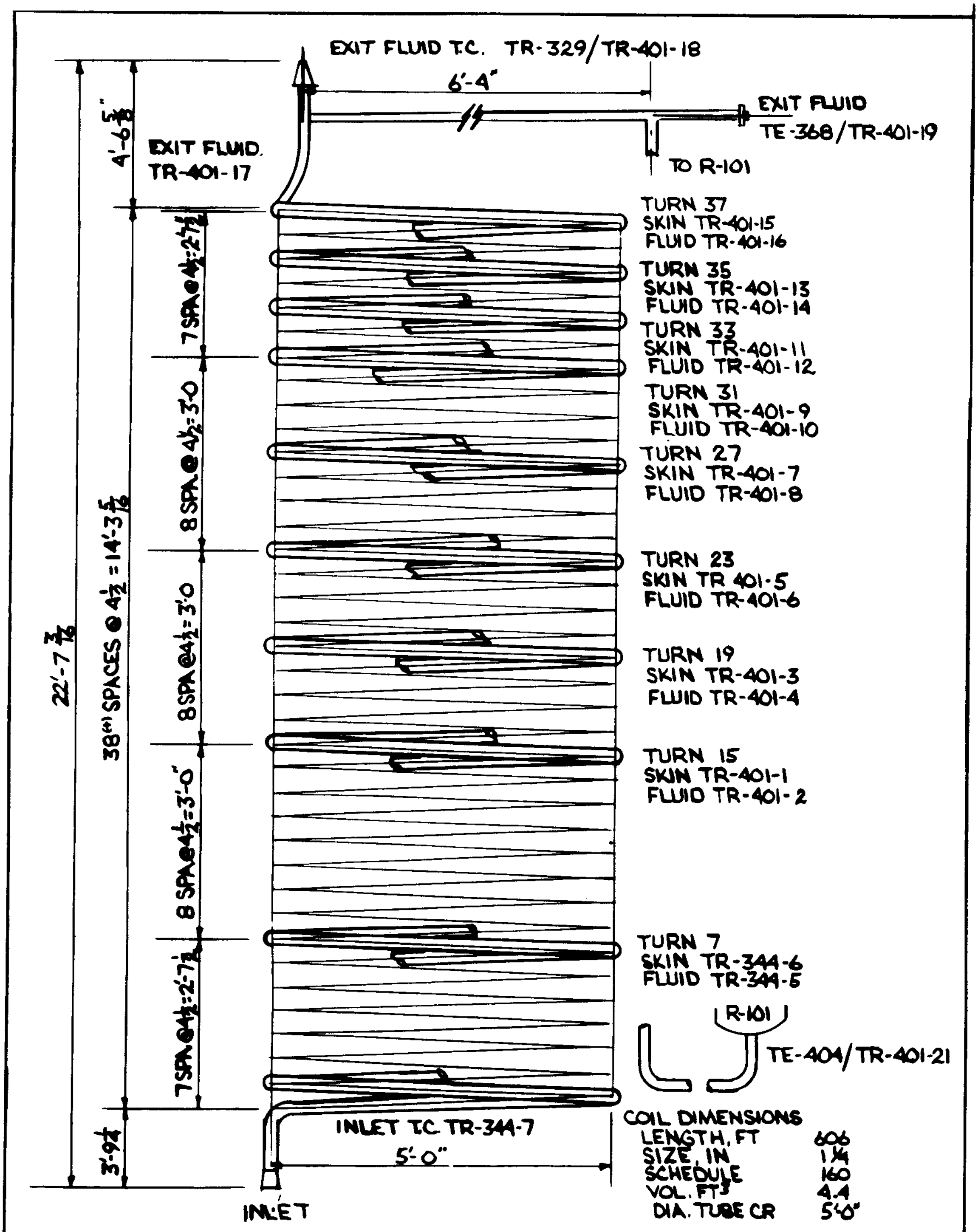

FIGURE 4: B-102 SLURRY PREHEATER THERMOCOUPLE LOCATIONS

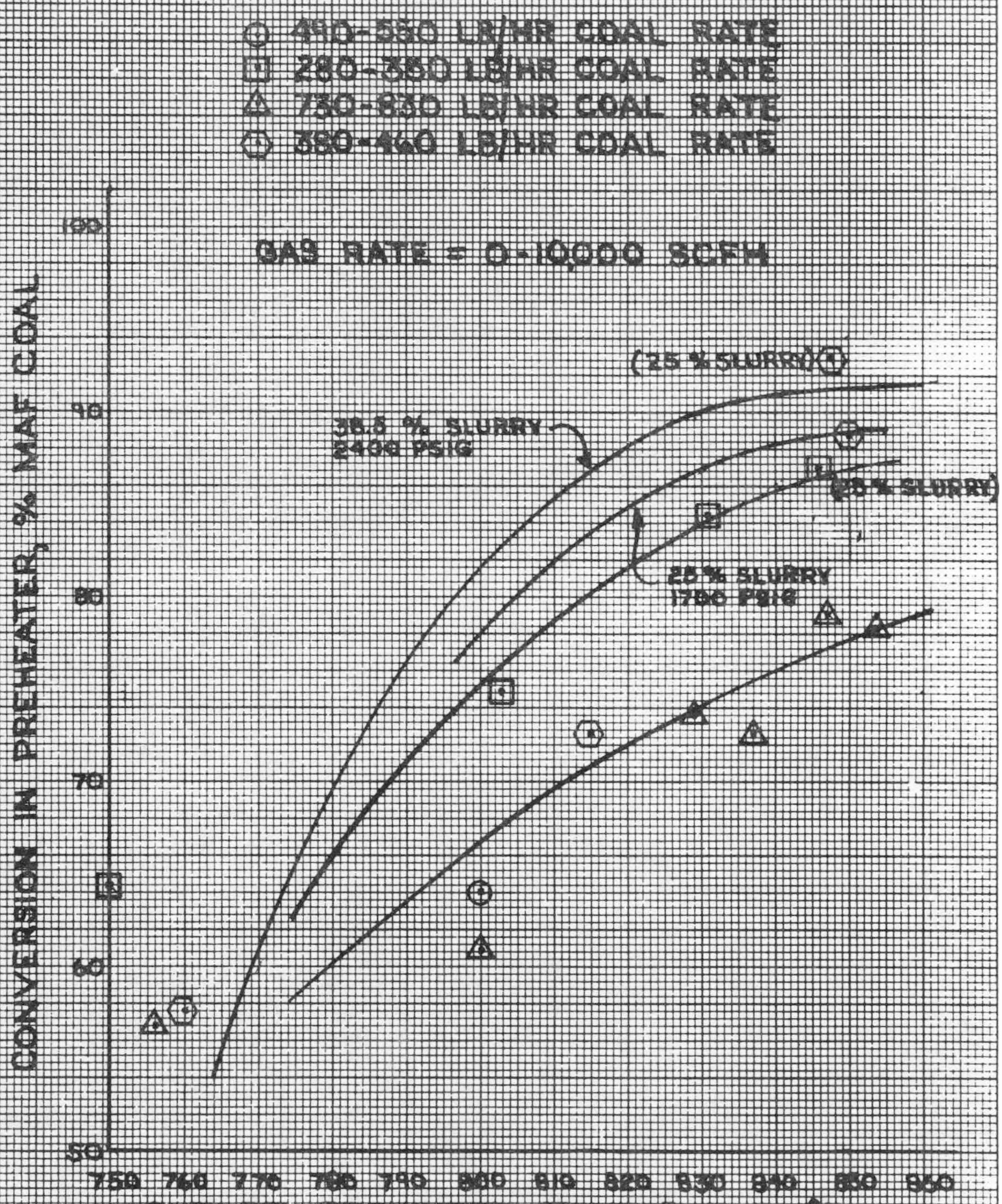

FIGURE 5: EFFECT OF TEMPERATURE ON INDIANA V COAL CONVERSION IN PREHEATER

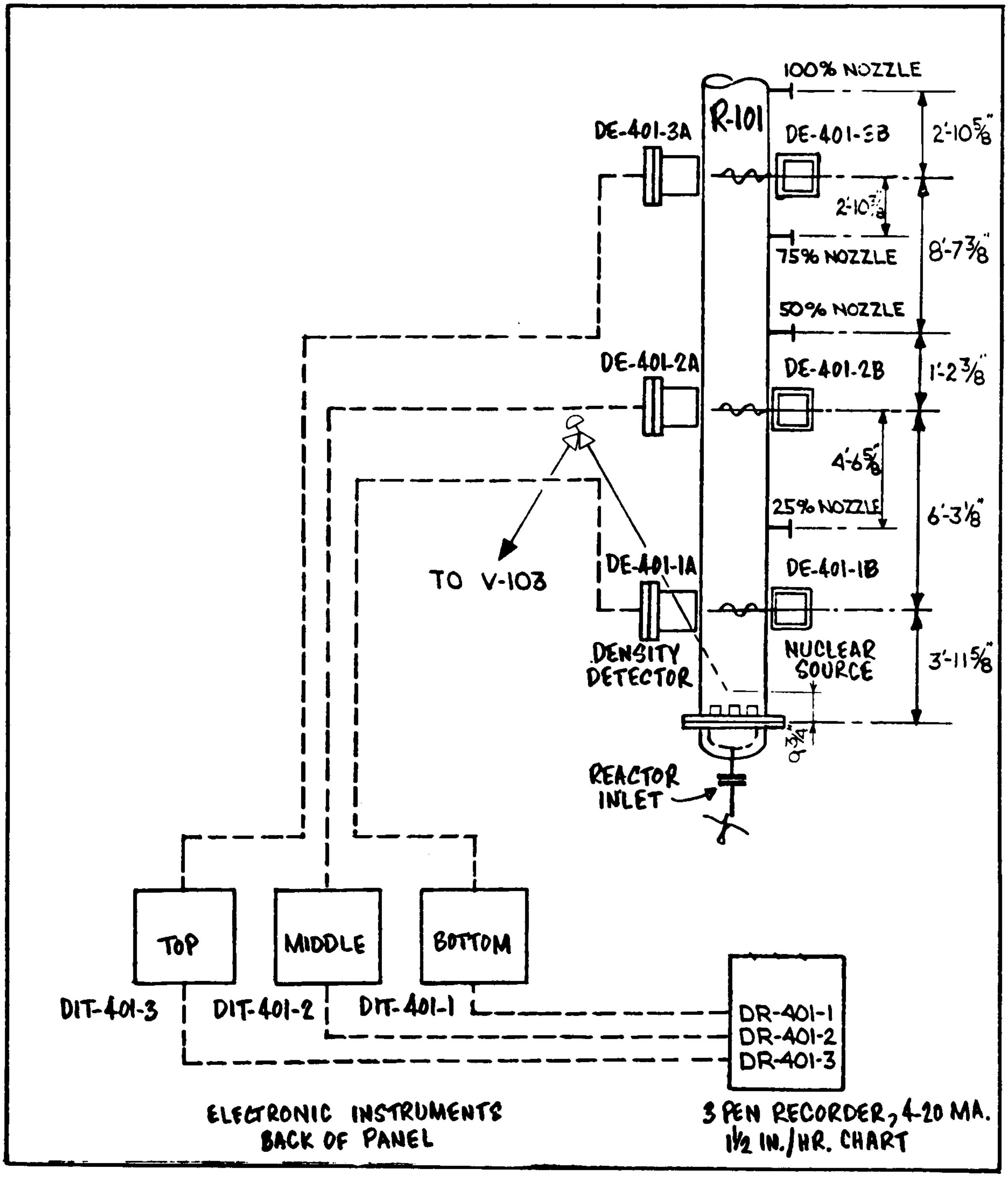

FIGURE 6: DISSOLVER SOLIDS MEASUREMENT AND CONTROL SYSTEM

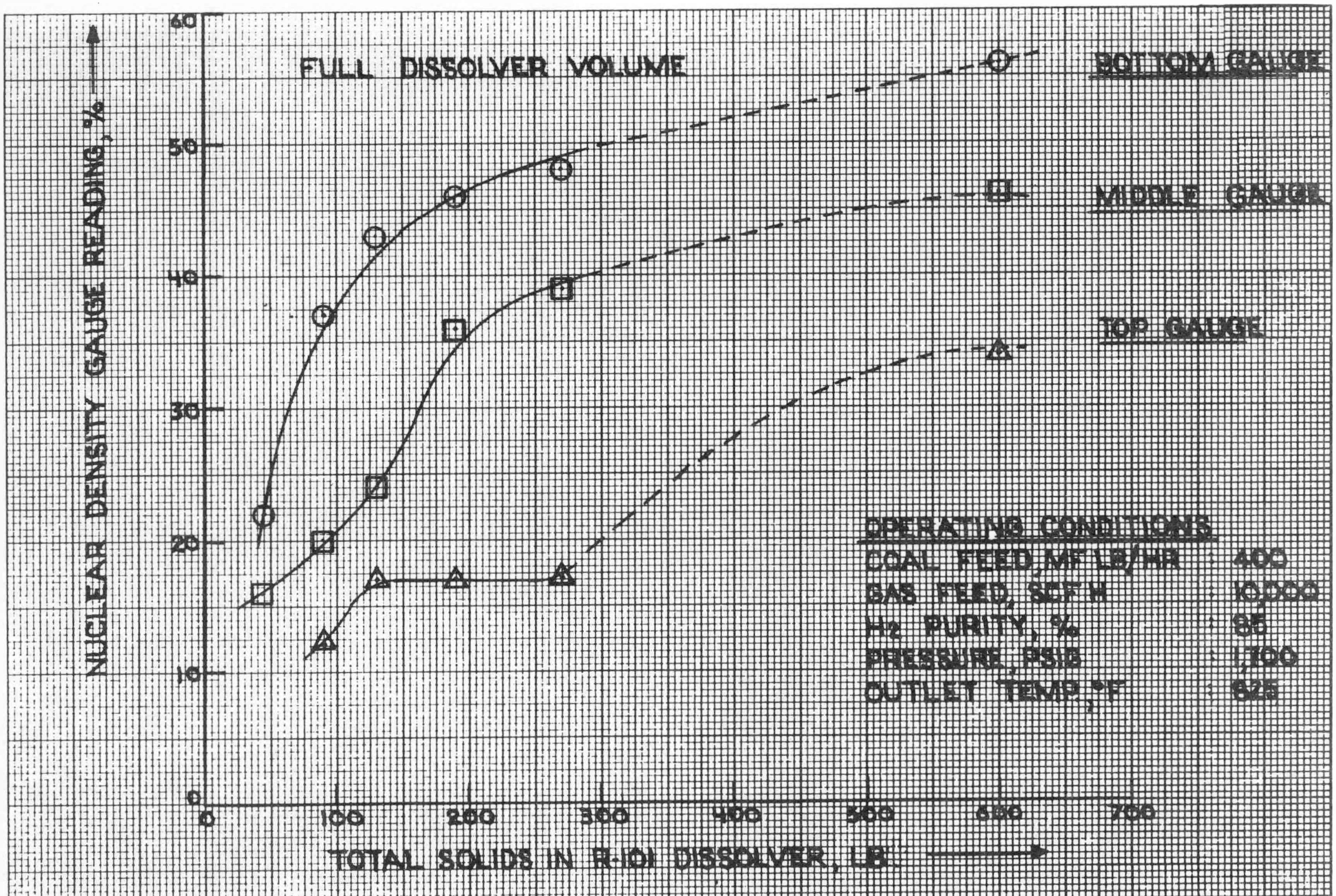

FIGURE 7: EFFECT OF DISSOLVER SOLIDS INVENTORY ON R-101 DENSITY GAUGE READING

RUN	TEMP °F	SOLVENT TO COAL RATIO	REACTION TIME,(A) MIN
SHORT	750	8:1	10
LONG	750	2:1	30

(A) INCLUDES HEAT-UP PERIOD

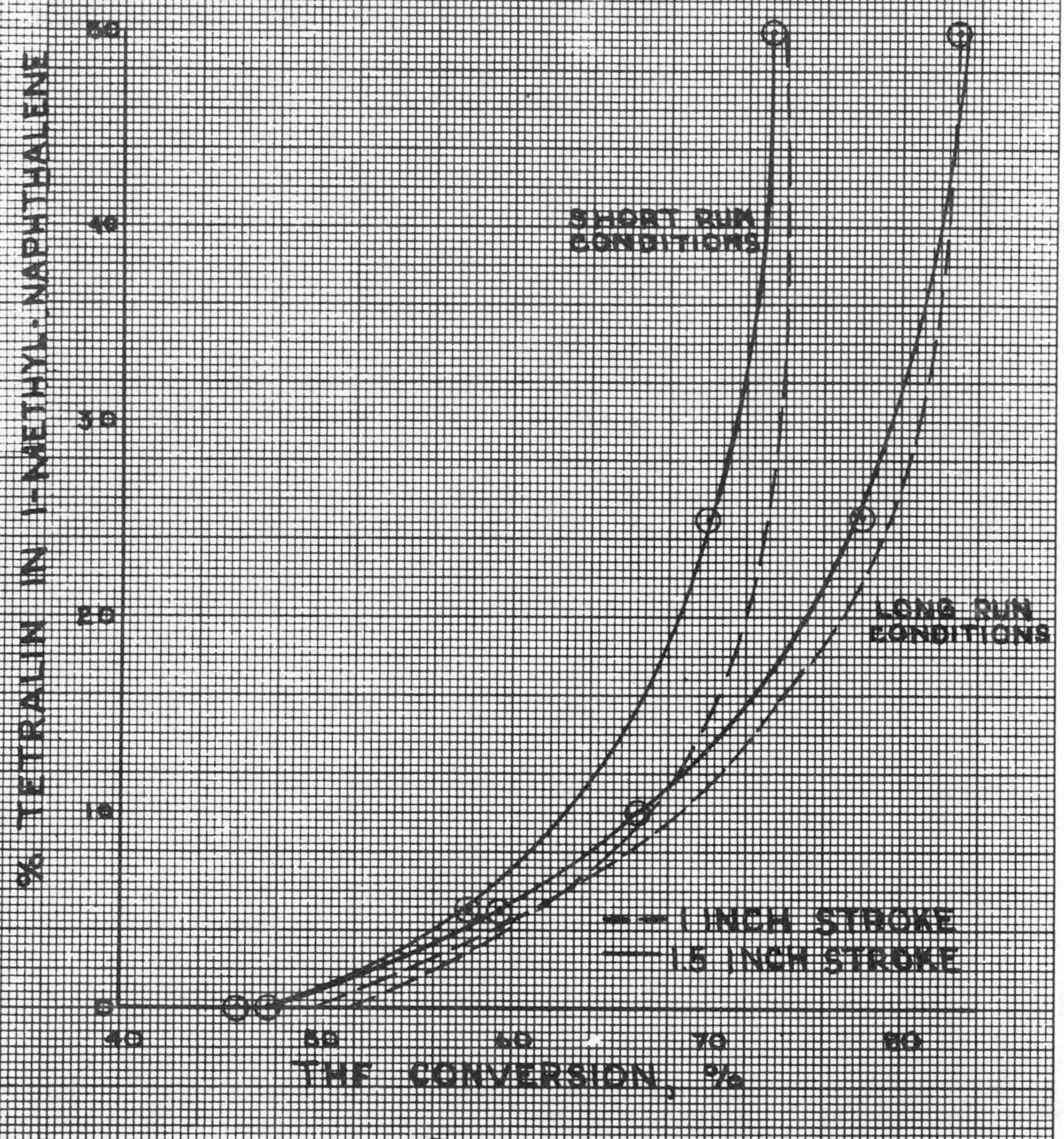

FIGURE 8: MICROAUTOCLAVE CALIBRATION CURVES

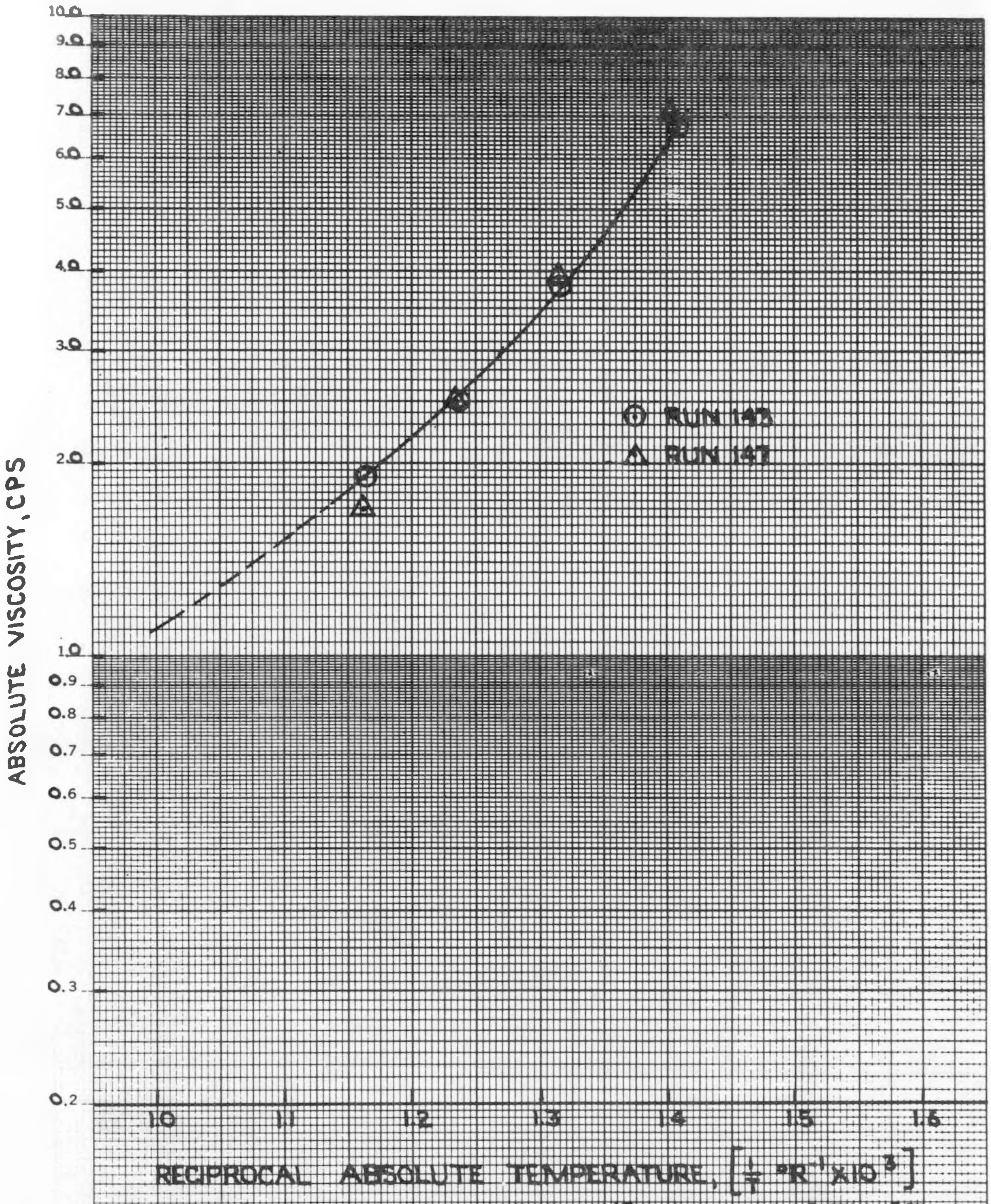

FIGURE 9: EFFECT OF TEMPERATURE ON VISCOSITY OF REACTION PRODUCTS FOR INDIANA V COAL

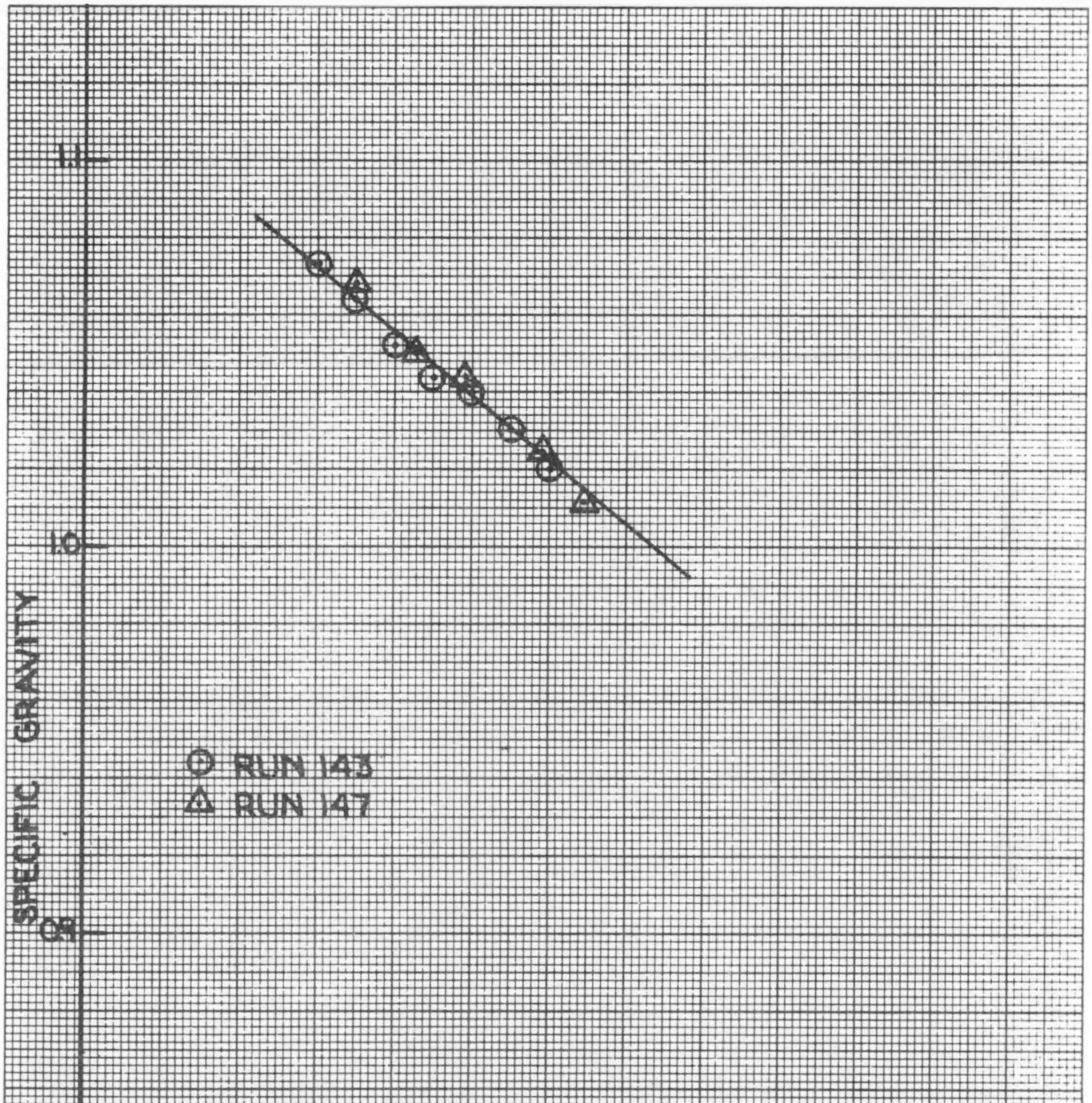

FIGURE 10: SPECIFIC GRAVITY VERSUS TEMPERATURE FOR REACTION PRODUCT FROM INDIANA V COAL

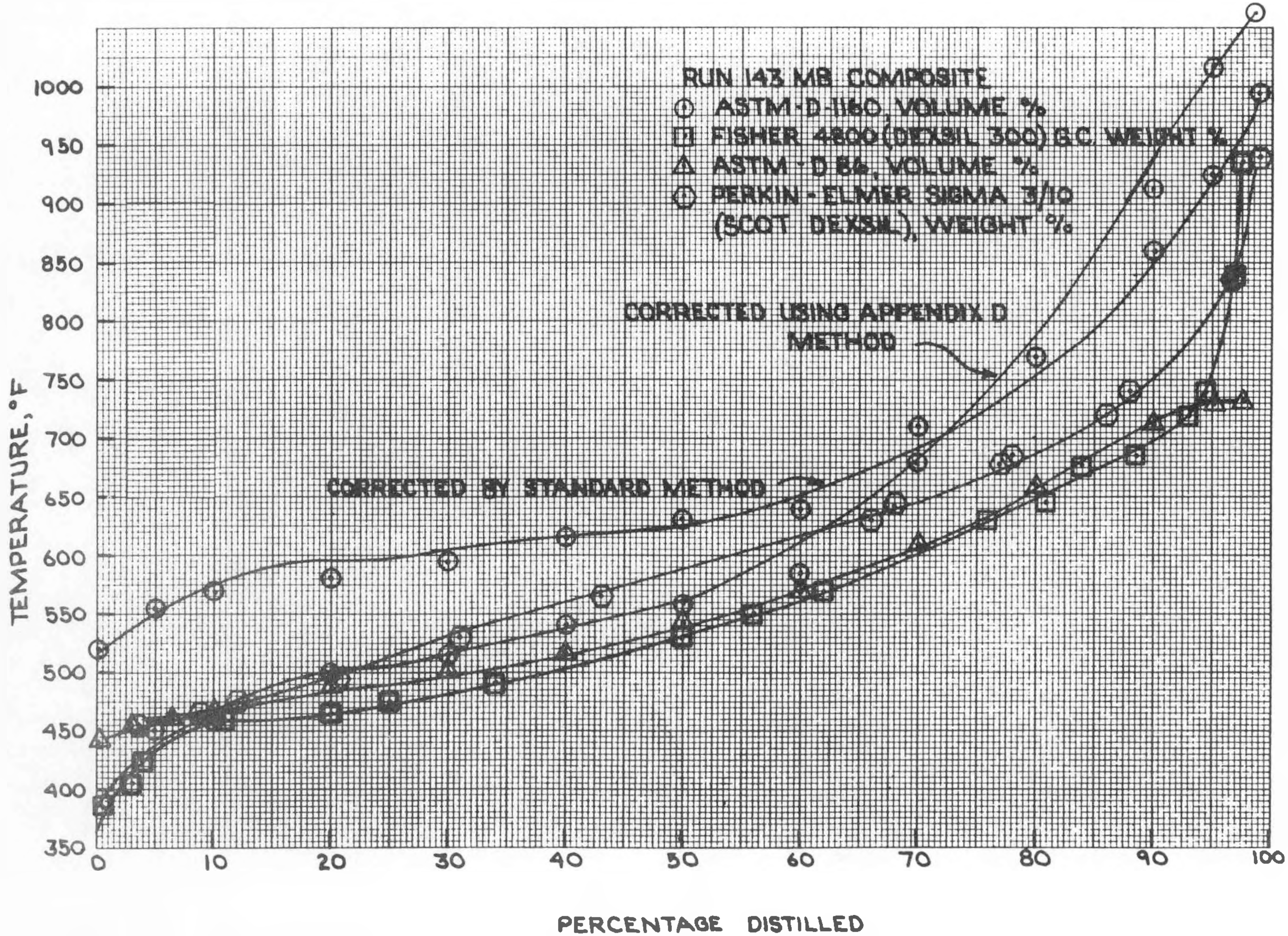

FIGURE 11 : PROCESS SOLVENT DISTILLATION CURVES

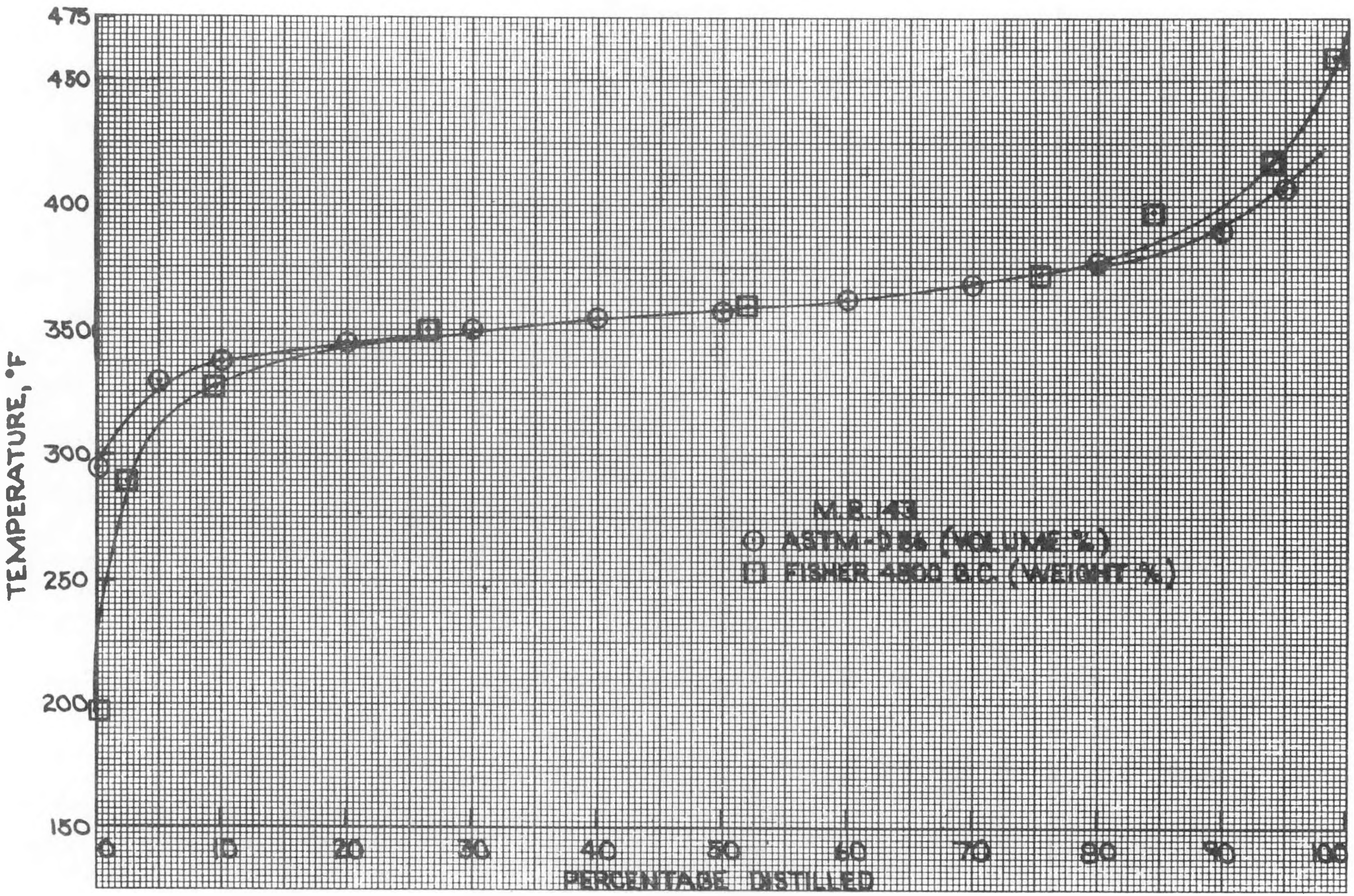

FIGURE 12: WASH SOLVENT DISTILLATION CURVES

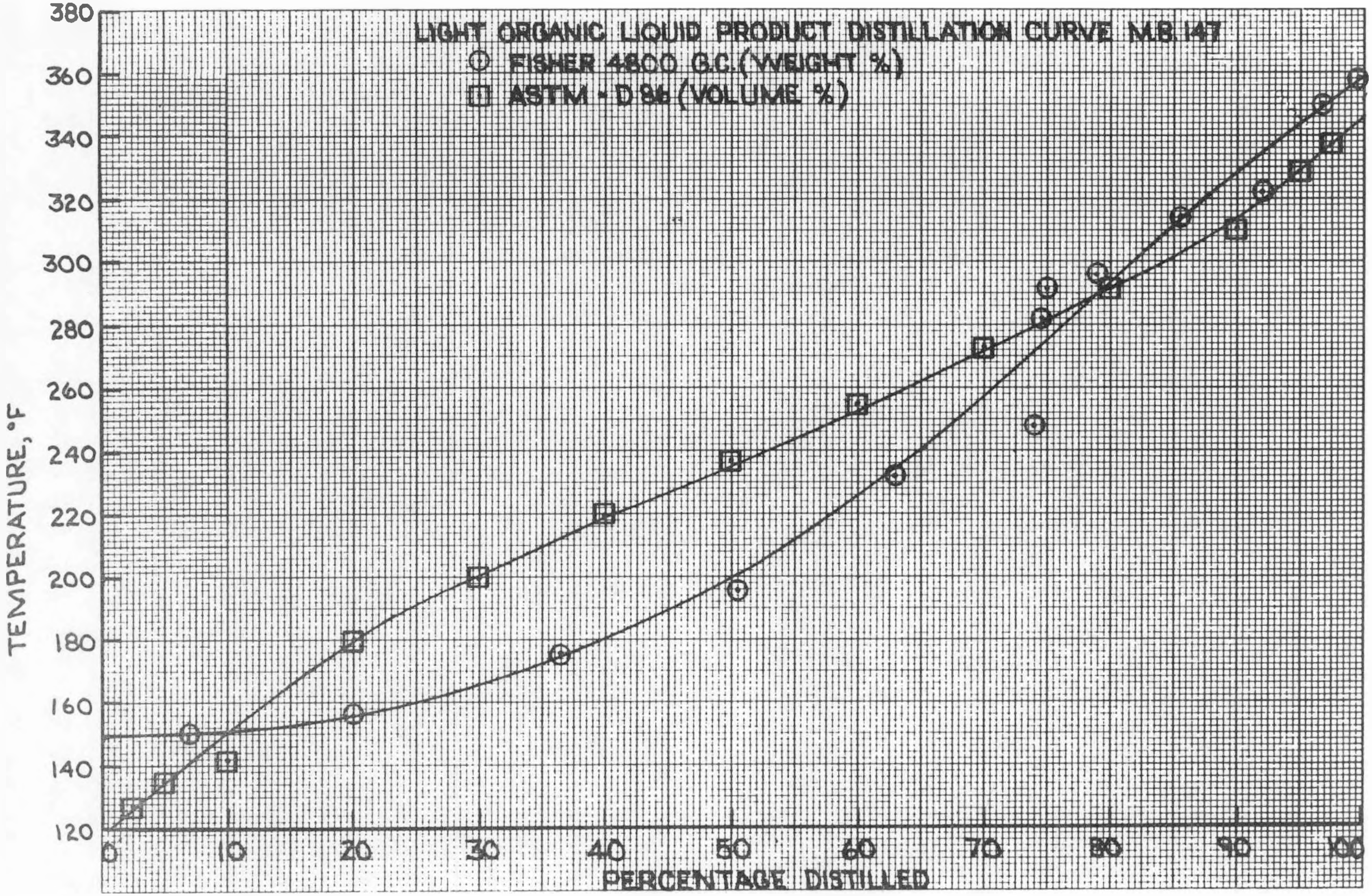

FIGURE 13: LIGHT ORGANIC LIQUID PRODUCT DISTILLATION CURVES

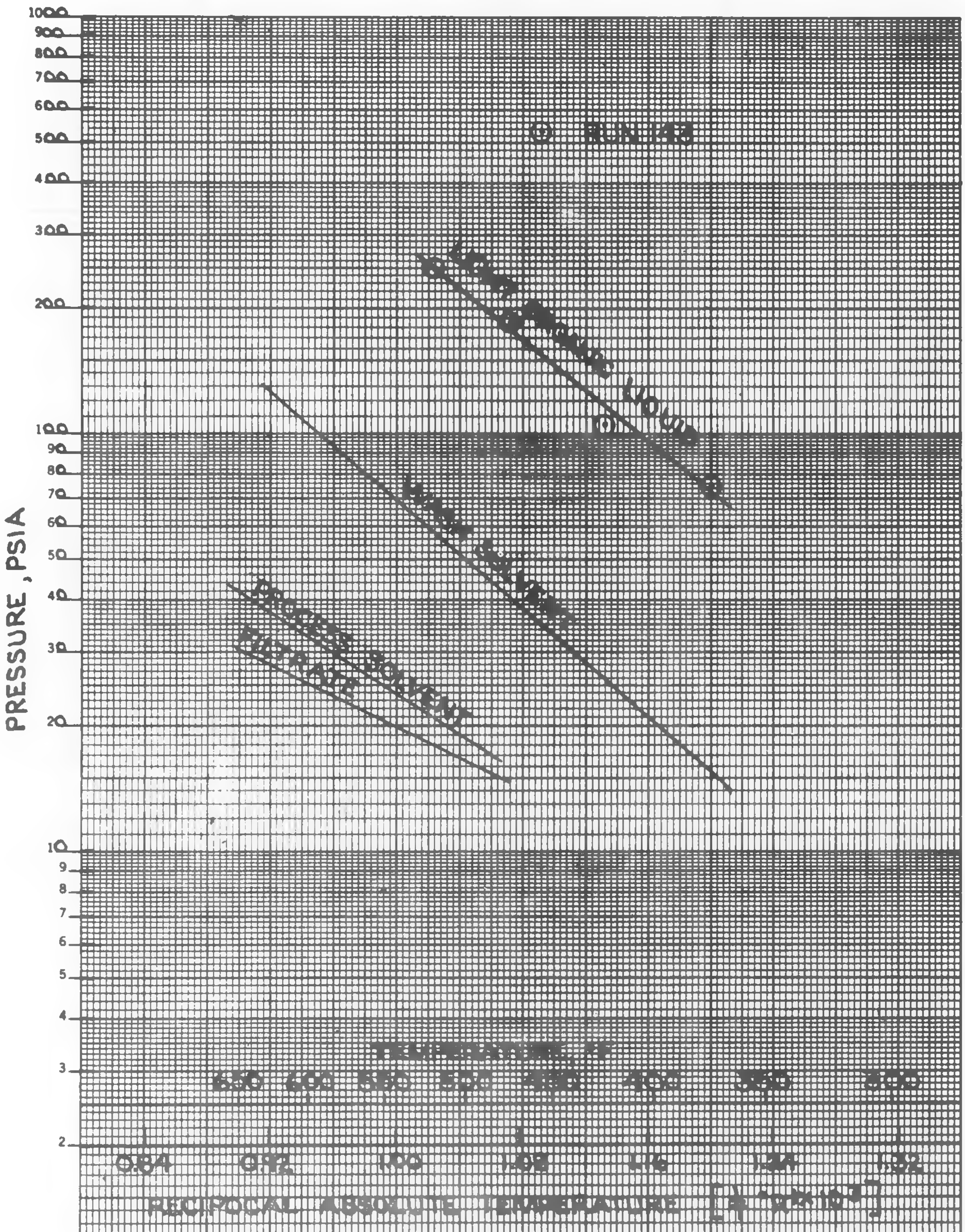

FIGURE 14: ORGANIC LIQUID FRACTION VAPOR PRESSURE CURVE

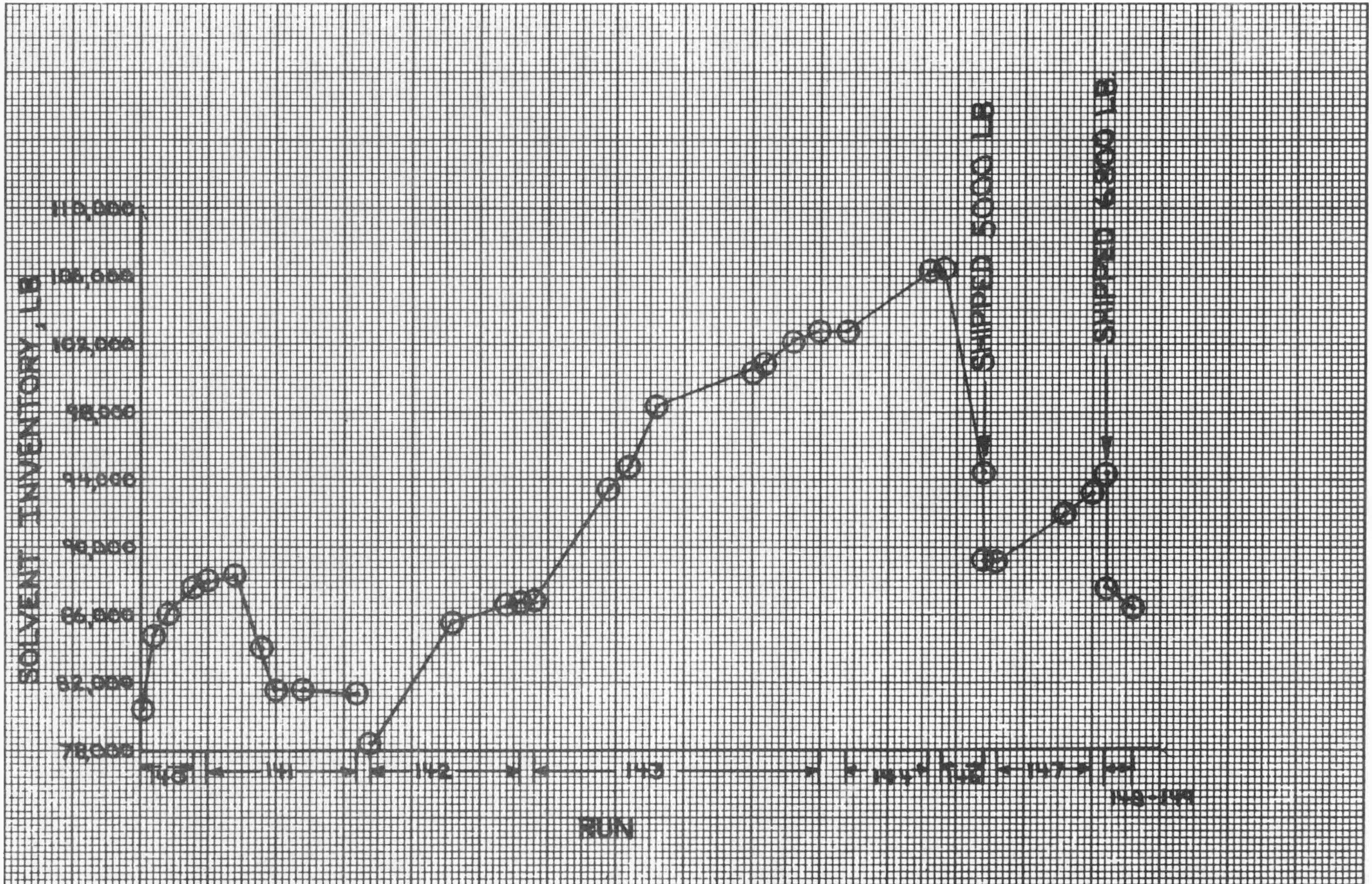

FIGURE 15: ADJUSTED SOLVENT INVENTORY

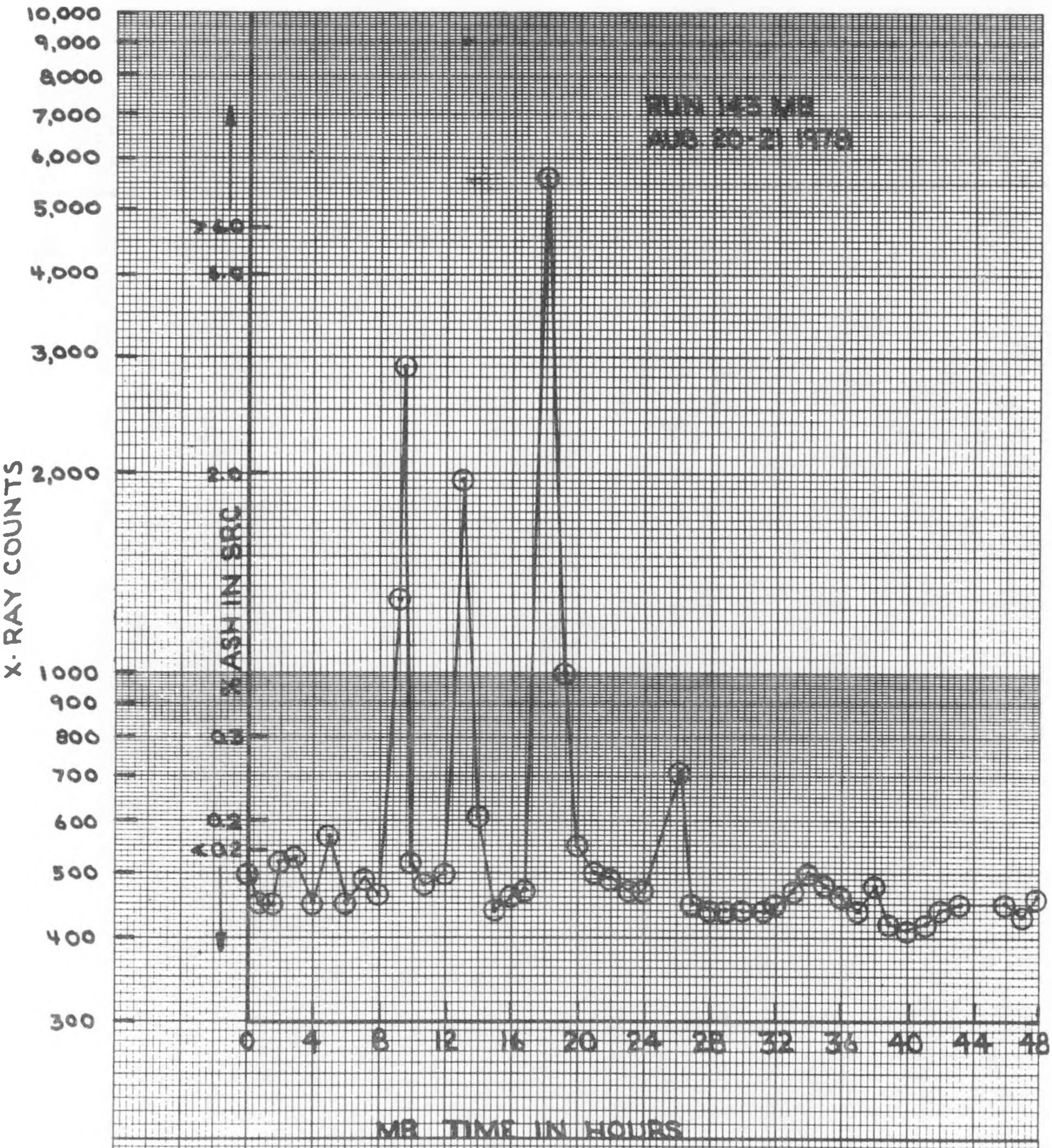

FIGURE 16: ASH CONTENT IN SRC DURING A MATERIAL BALANCE PERIOD

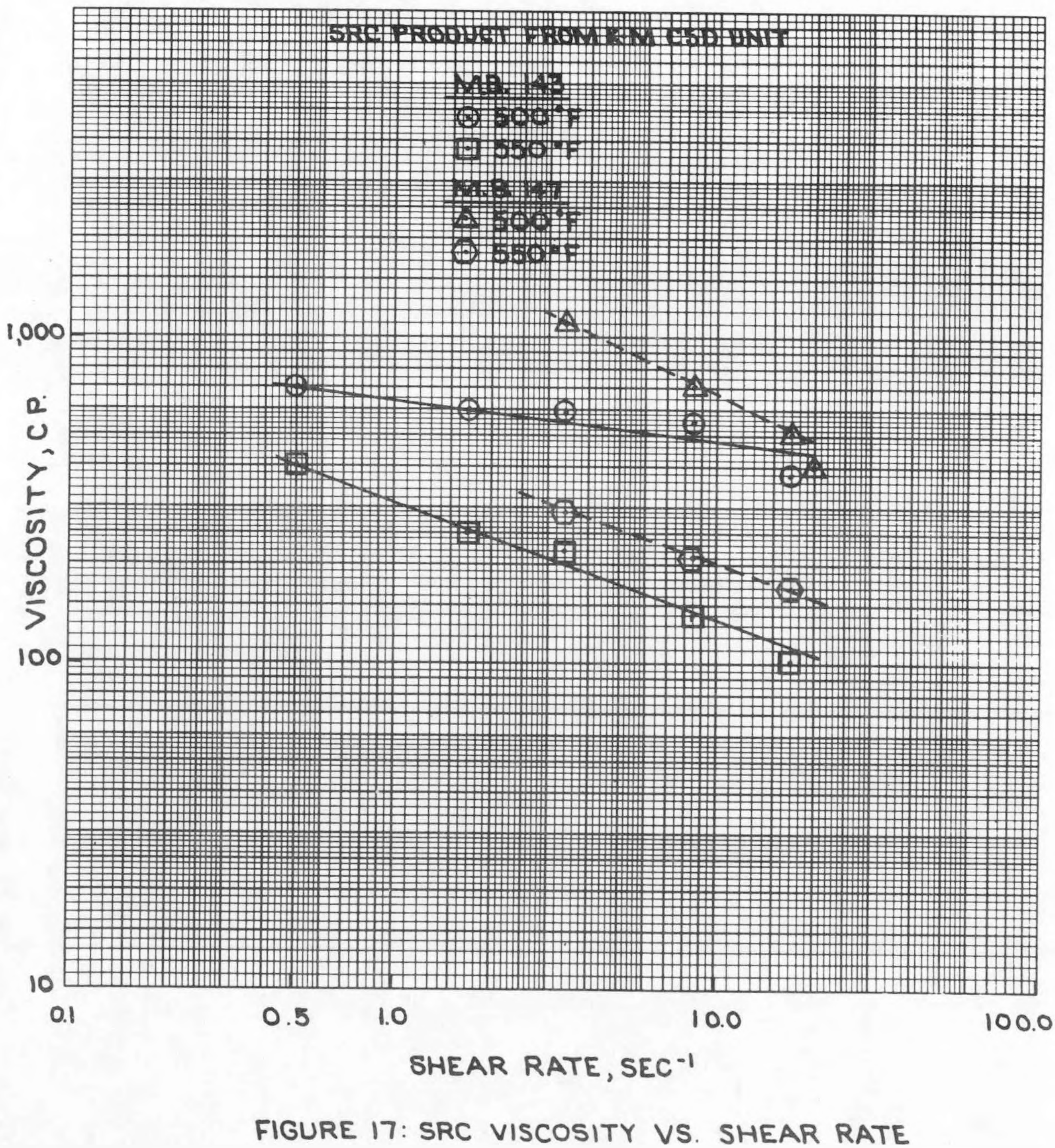

FIGURE 17: SRC VISCOSITY VS. SHEAR RATE

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