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THE WILSONVILLE ADVANCED COAL LIQUEFACTION RESEARCH AND DEVELOPMENT FACILITY, WILSONVILLE, ALABAMA

Topical Report No. 5
6,000 TPD SRC-I Demonstration Plant Support

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Catalytic, Inc.
Wilsonville, Alabama



U. S. DEPARTMENT OF ENERGY

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TOPICAL REPORT NO. 5
6,000 TPD SRC-I DEMONSTRATION PLANT SUPPORT
(Published: August 1983)

Prepared by
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Wilsonville, Alabama 35186

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ABSTRACT

From 17 October 1981 to 14 October 1982, the Advanced Coal Liquefaction R & D Facility at Wilsonville, Alabama, was operated partly in support of the 6,000 TPD SRC-I demonstration plant design effort undertaken by the International Coal Refining Company (ICRC). During this period, two bituminous coals were processed, and several special tests and operations were completed. This topical report summarizes the operating data and results from the demonstration plant support tasks completed during this period.

1. INTRODUCTION

Operation of the Wilsonville Advanced Coal Liquefaction R & D Facility began in January 1974 to study the key steps in the solvent refined coal (SRC) process. Support for this program came from the Edison Electric Institute (EEI) and Southern Company Services, Inc. (SCS). Catalytic, Inc. designed, built and has operated the plant since its inception. The Electric Power Research Institute (EPRI) assumed the role of EEI in the program in April 1973. The United States Department of Energy (DOE), formerly the Energy Research and Development Agency (ERDA), became a co-sponsor of the project in 1976. SCS is the prime contractor and provides overall project management services for DOE and EPRI.

Initially, the Wilsonville facility consisted of a single stage (thermal) process, also known as the SRC-I process. Over the past few years, the original plant has been expanded to become an advanced two-stage coal liquefaction facility. A Critical Solvent Deashing (CSD) unit was installed in 1978 and a second stage catalytic hydrogenation (HTR) unit was installed in 1981. The principal product of the first stage is a low sulfur solid fuel. The reaction product is deashed by the CSD unit using a proprietary process developed by the Kerr-McGee Corporation. The hydrotreater, or the second stage, was installed primarily for further enhancement of product properties, process flexibility, and overall hydrogen utilization efficiency. In the decoupled mode of operation, the HTR unit has no direct effect on the SRC unit. This operating mode is called the non-integrated two-stage liquefaction (NTSL) process and is shown in Figure 1.

From 17 October 1981 to 14 October 1982, the Advanced Coal Liquefaction R & D Facility at Wilsonville, Alabama, was operated partly in support of the 6,000 TPD SRC-I demonstration plant design effort undertaken by ICRC. The ICRC support tests and operations performed during this period were:

- Run 235 (17 October to 21 December 1981)
with Kentucky 9 (Fies) coal
- Run 240 (31 May to 20 July 1982)
with Illinois 6 (Burning Star) coal
- CSD unit second stage variability study
- CSD unit continuous ash removal system study
- SRC solidification test
- Wastewater sampling operation
- Residual fuel oil blending operation

The operations and results of each test and operation are discussed in Section 4, and the conclusions are summarized in Section 2.

Demonstration plant support work carried out at Wilsonville prior to October 1981 is described in another topical report entitled "Operation of Wilsonville SRC Pilot Plant in Support of SRC-I Demonstration Plant" (Ref. 1).

Radian Corporation obtained various gas samples to analyze for trace contaminants on 7 to 9 May 1983. This program completed the Wilsonville demonstration plant support tasks for ICRC.

2. CONCLUSIONS

In this section, conclusions are presented for the ICRC demonstration plant studies. The test scope included the operation of the SRC, CSD and HTR units for Runs 235 and 240. Additional tests and operations performed for ICRC are summarized.

SRC Unit

Yield structures for Kentucky 9 and Illinois 6 coals were established at ICRC demonstration plant design conditions. The results obtained for Runs 235 and 240 indicated that the Kentucky 9 coal was a better feedstock for producing +450°F distillate with lower hydrogen consumption per pound of distillate. However, the total hydrogen consumptions for the NTSL process were similar for these runs on a coal feed basis.

For Run 240, Illinois 6 coal distillate yields were increased by doubling the SRC unit space velocity at constant temperature. This result was unexpected and indicated that kinetic modeling studies must be performed to optimize the SRC unit. The test at increased space velocity also provided heat of reaction data for an alternate ICRC demonstration plant design case which used two reactors in series.

CSD UNIT

Runs 235 and 240 demonstrated that stable operation of the unit is dependent on the performance of the first stage settler. A change in CSD unit feed properties (witnessed during Run 240) affected the operation of the unit and caused lower SRC recoveries.

The CSD unit variability studies at constant pressure demonstrated that LSRC production can be controlled by altering either the second stage separator temperature or the DAS parameter. Correlations developed from this data may be used to adjust operating conditions. Changes in the second stage separator pressure were also shown to affect LSRC production.

The CSD unit continuous ash removal study indicated that equipment modifications are required for continuous operation. Equipment performance was acceptable, but ash plugs limited the on-stream time.

HTR Unit

Run 240 showed a higher hydrogen utilization efficiency (lower consumption) than Run 235 in the HTR unit. This result was the reverse of what occurred in the SRC unit.

Catalyst deactivation data indicated that SRC conversion, desulfurization and denitrogenation were temperature and time dependent. Preasphaltene conversion was not temperature dependent over the temperature range studied, but was dependent on catalyst age. Deactivation studies should be performed at longer on-stream times.

SPECIAL TOPICS AND OPERATIONS

SRC SOLIDIFICATION TEST

Fumes that were generated during the solidification process could be effectively removed with the High Efficiency Air Filter (HEAF) filter. Heat and material balances and particle size distributions were obtained and this information may be used for commercial plant design.

SRC WASTE WATER SAMPLING OPERATION

Waste water generated by the SRC process can be treated to low contaminant levels with conventional treatment facilities. The data obtained from this study may be used to design biological plants for the SRC demonstration plant. In addition, conventional leaching techniques did not produce a leachate from SRC product which had detectable trace organic compounds.

RESIDUAL FUEL OIL BLENDING OPERATION

Deashed SRC and solvent produced at Wilsonville were blended on a large scale and a fuel oil of acceptable viscosity was obtained. Temperature, time and exposure to air were shown to affect the viscosity of the blend, but these effects have not been quantified.

TRACE CONTAMINANT SAMPLING OPERATION

Five gas streams were sampled by Radian Corp. for ICRC. Catalytic, Wilsonville provided coal and process solvent composite samples for analyses by Radian. The results of this operation will be reported by Radian Corp. to ICRC.

3. PROCESS DESCRIPTION

The non-integrated two-stage liquefaction (NTSL) system sequentially combines three unit processes. These are:

- The SRC process for thermal dissolution of coal,
- The CSD process for separation of ash and undissolved coal from SRC products, and
- The HTR process for product upgrading.

The NTSL system does not recycle any process streams from the HTR unit to the SRC and/or CSD units. This differentiates the NTSL from the integrated two-stage coal liquefaction (ITSL) process. Figure 1 is a conceptual flow diagram of the NTSL process. The following sections provide a brief description of the three process units.

3.1. SRC Unit

The SRC process is a direct coal liquefaction process in which coal, hydrogen, and a process solvent are reacted at elevated temperatures (750°F to 840°F) and pressures (1,400 to 2,400 psig). This process is similar to other direct coal liquefaction processes in that the properties of the coal-derived solvent are very important, but differs from them in one or more of the following ways:

- The principal product is a solid at ambient conditions.
- A solid-liquid separation process is required.
- An extraneous catalyst is not required.

Figure 2 is a schematic flow diagram of the SRC process. In this process, pulverized coal is mixed with a process-derived solvent (450°F IBP) and the resulting slurry (up to 40% coal by weight) is pressurized to 2,400 psig. A hydrogen-rich recycle gas stream (85% hydrogen purity) is added to the slurry, and the mixture is allowed to react in a thermal dissolver for 30 to 60 minutes at a desired temperature (750°F to 840°F). Several complex reactions occur in the dissolver. The net products are nonvolatile residue (SRC, unreacted coal and ash), +C₆ distillates, C₁-C₅ gases, water and acid gases. The desired products are the SRC and the C₆ and heavier distillates. The effluent from the dissolver is flashed and the gases are separated and scrubbed to remove acidic components. Most of the scrubbed gas is recycled and fresh make-up hydrogen is added to maintain the desired flow rate and concentration. The purge gas (hydrogen and net C₁-C₅) is flared, but in a commercial-size unit, the net C₁-C₅ gases could

be efficiently separated and utilized within the complex as a fuel or as a reformer feedstock. After an intermediate pressure reduction, the liquid and solid products are processed in a vacuum column (T102). SRC residue, containing some solvent to maintain viscosity, is separated as a bottoms stream and pumped to the CSD unit for solids separation.

The recovered liquids from the SRC unit are fractionated into two cuts using the T102 Vacuum and T104 Fractionating (atmospheric) Columns. The -450°F fraction is recovered as the overhead product of the T104 column, and the +450°F fraction is removed as a sidedraw product (trays 3 and 8) of the T102 column. The +450°F fraction is termed "distillate solvent". This stream is a major component of the recycle "process solvent" used as a feed to the SRC unit. The other process solvent component in the NTSL mode is light SRC (5 to 30 weight percent). Light SRC is a recycle stream from the CSD unit.

3.2. CSD Unit

The ash and unreacted coal are separated from SRC by the Critical Solvent Deashing Unit (Figure 3). This proprietary process was developed by the Kerr-McGee Corporation.

The extraction of SRC from the ash and unreacted coal occurs near the critical point of the deashing solvent (DAS). The feed to the unit is mixed with recycle and fresh DAS and is fed to the first-stage settler. DAS dissolves the soluble SRC and causes the formation of two phases. The heavy phase (containing ash, unreacted coal, DAS, and some SRC) is rejected from the bottom of the settler. This phase is processed to recover the DAS.

The light phase from the first stage settler enters the second stage settler where the process conditions are altered to reduce the solubility of the deashed SRC in the deashing solvent. This causes the heavier SRC fraction, with an equilibrium quantity of DAS, to settle as a heavy phase. This phase is then processed to remove the DAS and produce the solid SRC product. The SRC is normally fed to the HTR unit for further product upgrading.

The light phase from the second stage settler is sent to the third stage settler where the lighter fraction of the soluble SRC (LSRC) is separated from DAS solvent. By controlling the second stage settler conditions, the ratio of SRC to LSRC can be varied over a wide range. In the NTSL mode, the entire LSRC product is recycled as a component of the SRC unit process solvent.

3.3. HTR Unit

SRC recovered from the CSD unit is charged to the catalytic hydrotreater unit and is mixed with hydrotreated recycle solvent and hydrogen-rich recycle gas. The hydrotreater unit was designed by Hydrocarbon Research, Inc. and is shown in Figure 4.

The principal difference between the SRC and HTR processes is the ebullated bed catalytic reactor. The catalyst promotes SRC conversion, desulfurization and denitrogenation even at a relatively low temperature of 650°F. The reaction volume of the system consists of three phases - each with distinct hydrodynamic characteristics:

- The gas phase enters the reactor from the bottom and, after bubble formation, is assumed to move upward in a plug-flow manner.
- The liquid phase enters the reactor at the bottom and moves upward with considerable back mixing caused by the random motion of the catalyst pellets. The catalyst-free liquid phase at the top of the reactor is withdrawn and a major portion is recycled to the bottom of the reactor with an ebullating pump. This recycle flow provides the superficial liquid velocity needed to support the fluidized bed.
- The catalyst solid phase exists as a fluidized bed within the reactor and the bed height is maintained by recycling liquid. Catalyst activity can be maintained at a desired level by adding sulfided catalyst at the top and withdrawing spent catalyst from the bottom of the reactor.

The efficient heat transfer between the catalyst pellets and the bulk liquid phase is a unique feature of the hydrotreater. Temperature gradients are minimized, since the large recycle flow and the random motion of the bed effectively remove the exothermic heat of reaction and maintain a well-mixed reactor volume.

4. DISCUSSION OF RESULTS

From 17 October 1981 to 14 October 1982, two demonstration plant support runs were made in the NTSL mode. Kentucky 9 (Fies) and Illinois 6 (Burning Star) coals were processed and the runs were designated Runs 235 and 240, respectively. The Kentucky coal was a potential feedstock for the proposed 6,000 TPD SRC-I demonstration plant in Newman, Kentucky. The primary objective of these runs was the verification of the yield structures that were used by ICRC for the demonstration plant design. In addition, several special tests and operations were conducted in support of the demonstration plant design efforts. The results of these tests and operations are discussed in Sections 4.3 to 4.8.

4.1 Run 235

Kentucky 9 coal from Fies mine was processed for Run 235, which began on 17 October 1981 and ended on 21 December 1981. The detailed results of this run were reported in Reference 2. A brief summary of the two stage reaction conditions and the results for this run are presented in this section.

SRC Unit

Average operating conditions for Run 235 were:

- High temperature (835°F)
- 2,100 psig pressure
- 550 lb/hr MF coal rate
- 39% MAF coal slurry concentration
- 30 Mscf recycle gas per ton of coal at 85% hydrogen purity
- 39 lb/hr-ft³ space velocity
- Low LSRC recycle (5.3% in the process solvent)
- High temperature (740°F) and low residence time in the HP Separator
- 2.4 lb/hr addition of sodium carbonate for corrosion control

The SRC unit reaction conditions (with the exception of sodium carbonate addition) are approximately the conditions used for the demonstration plant base case design. The SRC unit yield structure for these conditions was defined.

Four material balance periods were selected from this run for detailed product workup. Actual operating conditions for these periods are summarized in Table 1 and the feed coal analyses are presented in Table 2. The yield structures obtained from these material balance periods are summarized in Table 3.

CSD Unit

For Run 235, the CSD unit was operated in the high temperature and high pressure DAS recycle mode. Deashing solvent A was used. The CSD unit feed analyses, product analyses, and performance summary are shown in Tables 4, 5, and 6.

The following CSD unit performance parameters were attained during Run 235:

SRC Recovery	79-85% of soluble feed
Energy Rejection	19-25% coal heating value
Total DAS losses	2.4-4.2% feed

DAS, the extraction solvent for SRC, is recovered within the CSD unit and is recycled. However, the CSD unit requires a net make-up of DAS to compensate for uncontrolled losses. Although DAS losses are an important parameter for CSD unit performance evaluation, the data generated at Wilsonville are not meaningful for a commercial plant design.. The product-related DAS loss is a function of the recovery system design, while the non-product loss is a function of the plant size. Large-scale commercial systems, with a more effective recovery system design, should experience smaller product-related DAS losses as a percentage of plant throughput.

HTR Unit

The primary run objectives for the HTR unit were: definition of reactor performance and estimation of catalyst deactivation rates at two temperatures (780°F and 810°F). Commercially available cobalt-molybdenum hydrodesulfurization catalyst (American Cyanamid HDS-1442B) was used. Thirteen material balances (seven at 780°F and six at 810°F) were obtained. Operating conditions and the unit performance for these material balance periods are summarized in Table 7. Feed and product SRC analyses are shown in Table 8. Fresh and spent catalyst analyses are presented in Table 9. The catalyst deactivation data and a complete description of the HTR unit performance were reported in Reference 3.

NTSL Yield Data

The combined two-stage yields for Run 235 are presented on the following page.

Run Date	235AB 15-16 Nov 1981	235C 25 Nov 1981	235D 16 Dec 1981	235E 18 Dec 1981
<u>Yields, % MAF coal</u>				
<u>Gases</u>	13.1	11.8	13.7	12.5
CO	0.2	0.1	0.2	0.2
CO ₂	1.4	1.3	1.2	1.2
NH ₃	0.6	0.5	0.6	0.6
H ₂ S	1.7	1.8	2.1	2.0
C ₁ -C ₅	9.2	8.1	9.6	8.5
<u>Liquids</u>				
H ₂ O	6.9	6.7	6.2	6.3
<u>Total recovered distillate</u>	33.2	34.4	36.2	40.3
Naphtha	6.0	4.6	7.0	8.1
Middle distillate	6.4	7.3	7.8	7.0
Distillate solvent	20.8	22.5	21.4	25.2
<u>Ash concentrate (CSD Stage I) (a)</u>	26.2	28.6	30.0	29.0
<u>Hydrotreated GRC (a)</u>	32.8	29.4	27.5	24.7
Total yield	112.2	110.9	113.6	112.8
Less net input	100.0	100.0	100.0	100.0
Hydrogen consumption, % MAF coal (b)	3.4	3.2	4.3	3.8

(a) DAS-free, includes distillate solvent.

(b) Calculated by ethylene injection.

A detailed description of these results was presented in Reference 2.

4.2 Run 240

Illinois 6 coal from Burning Star mine was continuously processed from 31 May to 20 July 1982. This test was designated as Run 240 and the results were reported in Reference 4.

The main objective of this run was determination of the yield structure for Illinois 6 coal in the SRC reactor at the demonstration plant basis of design conditions. Additional tests were performed to verify operation at other conditions and to obtain information for special tests and operations.

SRC Unit

The normal operating conditions for Run 240 were similar to those for Run 235, except the dissolver temperature was 825°F (instead of 835°F) and sodium carbonate was not added.

From 8 to 13 July, the coal feed rate to the dissolver was doubled to simulate an alternate demonstration plant design basis. This design case operates two dissolvers in series, rather than parallel as in the base case, and the pressure profile is different. Temperature control in the second dissolver is affected by the heat evolved in the first dissolver. Yield structures were determined for the alternate case.

Three overall material balance periods were selected for detailed product analyses. Operating conditions for these periods are summarized in Table 1. The feed coal analyses and yield structures obtained are summarized in Tables 2 and 3.

Liquid samples from the SRC unit low pressure Flash Tank (V110) were collected for ICRC. These samples were analyzed and characterized by ICRC to provide vapor and liquid equilibrium data for the demonstration plant design. This information will be reported in a future ICRC Quarterly Report (Ref. 5).

CSD Unit

For Run 240, the CSD unit was operated at conditions similar to those for Run 235 (high temperature and high pressure DAS recycle). Deashing solvent A was used. The performance summary, CSD feed analyses and product analyses are presented in Tables 4, 5, and 6.

The following CSD unit performance parameters were attained at normal SRC unit operating conditions for Run 240:

SRC Recovery	83% of soluble feed
Energy Rejection	20% coal heating value
Total DAS Losses	3.5-4.5% feed

At the alternate SRC unit design conditions, when the dissolvers were operated in series with a higher coal feed rate (8-13 July), SRC recovery dropped to 70% and the energy rejection was approximately 33%.

Additional tests and operations on this unit were performed during Run 240. The variation of LSRC/SRC ratio with respect to process variables in the second stage settler was investigated. This study is discussed in Section 4.3. The operation of the CSD unit ash separation equipment was tested and the results are presented in Section 4.4.

HTR Unit

The yield structures and deactivation characteristics of a commercially available cobalt-molybdenum catalyst (American Cyanamid HDS-1442A) were studied at a reactor temperature of 760°F for Run 240. Nine HTR unit material balances were developed. The operating conditions, feed and product analyses and unit performance for these material balances are listed in Tables 7 and 8.

Fresh and spent catalyst analyses are summarized in Table 9. The deactivation data are thoroughly discussed in a topical report (Ref. 3). These results may be compared to data obtained at low thermal unit severity for Run 236 with the same coal (Ref. 6).

NTSL Yield Data

The combined two-stage yields for Run 240 are presented as follows:

Run Date	240A 11 June 1982	240B 21 June 1982	240C 12 July 1982
<u>Yields, % MAF coal</u>			
<u>Gases</u>			
CO	14.3	13.5	8.8
CO ₂	0.3	0.3	0.1
NH ₃	1.4	1.5	1.3
H ₂ S	0.7	0.8	0.0
H ₂ S	2.2	2.3	1.6
C ₁ -C ₅	9.7	8.6	5.2
<u>Liquids</u>			
H ₂ O	7.8	7.9	6.6
<u>Total recovered distillate</u>	34.0	34.6	34.6
Naphtha	6.6	5.2	4.4
Middle distillate	6.0	5.4	3.4
Distillate solvent	21.4	24.0	26.8
<u>Ash concentrate (CSD Stage I) (a)</u>	32.9	31.2	42.4
<u>Hydrotreated SRC (a)</u>	27.8	30.3	23.7
Total yield	116.8	117.5	116.1
Less net input	100.0	100.0	100.0
Hydrogen consumption, % MAF coal (b)	4.0	4.2	3.1

(a) DAS-free, includes distillate solvent.

(b) Calculated by ethylene injection.

A detailed description of these results was presented in Ref. 4.

4.3 CSD Unit Second Stage Variability Study

From 15 to 19 July, a series of experiments was performed to investigate the effects of CSD second stage operating parameters on SRC and LSRC production. These tests occurred at demonstration plant base case conditions for the SRC unit during the Run 240 test period. Six tests were conducted and were arranged in the following experimental design:

Test No.	Second Stage Separator (a)		
	Pressure, psig	Temperature, °F	DAS Parameter
1	P ₁	T ₁	R ₁
2	P ₁	T ₂	R ₁
3	P ₁	T ₃	R ₁
4	P ₁	T ₂	R ₂
5	P ₁	T ₂	R ₃
6	P ₂	T ₂	R ₁

(a) Due to the proprietary nature of the Kerr-McGee data, the actual values are not included.

These tests indicated that a good relationship exists between LSRC production and second stage temperature at constant operating pressure and DAS parameter in the second stage settler. The

results are depicted in Figure 5. Also, LSRC production is related to second stage DAS parameter at constant operating pressure and temperature in the second stage settler. Figure 6 demonstrates this relationship. The effect of a change in operating pressure is also shown in Figures 5 and 6.

The results confirmed previous test work performed by Kerr-McGee for the demonstration plant design. Those tests measured DAS content in the second and third stage underflows.

Unit performance for the second stage variability study was excellent, except for transient periods which followed changes in test conditions. Stable operation and consistent first stage letdown occurred during the test period.

4.4. CSD Unit Continuous Ash Removal System Study

The technical feasibility of a continuous ash processing system was tested on numerous occasions from October 1981 to August 1982. The continuous ash removal system operating results for this period, Runs 235 through 241, were previously reported (Ref. 2, 4, 6, 7, 8). A scaled-up version of the system has been proposed for the demonstration plant by Kerr-McGee. The most recent operating results are summarized in this section.

For Runs 238 and 239, the continuous ash processing system could not be operated in a satisfactory manner due to various design and operating problems. The system was modified several times. A detailed description of the specific problems and the required modifications are proprietary information.

The ash processing system was operated for approximately 19 hours during Run 239, with 11.3 hours on 27 April 1982 as the longest continuous period. The ash discharge system plugged and terminated the test.

Total operating time for Run 240 was 25.2 hours. The continuous ash system operating summary for Run 240 is presented below. Continuous ash system performance was acceptable, but ash discharge system plugging terminated most of the tests.

<u>Date,</u> <u>1982</u>	<u>Test</u> <u>Duration,</u> <u>hr</u>	<u>Tests</u>	<u>Remarks</u>
June 23	0.3	a. b. c.	Test aborted. Test aborted. Test aborted.
	2.0	a. b.	Downstream component plugged. Upstream component problem.
	5.8	a. b.	Downstream component plugged. Upstream and downstream com- ponents plugged.
		c.	Good operation. Test aborted due to upstream component.
		a. b. c. d.	Good operation. Upstream component problems. Upstream component problems. Upstream component problems.
29	6.5	a.	Test aborted, possibly due to upstream problem.
July 4	0.2	a.	Downstream component plugged.
	0.9	a.	Instrumentation problem.
		b.	Good operation. Test aborted due to mechanical failure.

The continuous ash processing system operated for 39.7 hours during Run 241. An operating summary is presented below. Plugging of the first stage discharge system terminated the majority of the tests.

<u>Date,</u> <u>1982</u>	<u>Test</u> <u>Duration,</u> <u>hr</u>	<u>Tests</u>	<u>Remarks</u>
August 10	8.2	a.	Good operation. Upstream component plugged.
		b.	Downstream component plugged.
	7.0	a. b. c.	Upstream component plugged. Upstream component pluded, Upstream component plugged.
	8.8	a.	Good operation.
	8.0	a.	Upstream and downstream compononto plugged.
		b.	Downstream component plugged.
	1.9	a.	Good operation. Upstream component plugged.
	5.8	a.	Test aborted due to internal and downstream component plugging.

Tests of the continuous ash processing system at Wilsonville for Runs 235 to 241 indicated:

- 1) The ash removal system was susceptible to plugging when flushing was required.
- 2) Overhead system plugging was not usually a problem if target operating temperatures were maintained.
- 3) The operability of the continuous ash processing system was closely linked to the stability of the CSD first stage separator.

4.5. SRC Solidification Test

An SRC solidification test, in support of the demonstration plant design effort, was conducted from 11 to 14 October 1982, after Run 241 was completed. The objectives of this test were:

- (a) Calculation of heat and material balances for the existing indirect water-cooled Rexnord solidifier
- (b) Characterization of the fumes generated from the solidification process
- (c) Determination of efficiencies for the Anderson High Efficiency Air Filter (HEAF) while processing the fumes generated from the solidification process

The existing solidification system at Wilsonville was modified to include additional thermocouples and related instruments for rapid temperature monitoring and recording. In addition, rotameters were installed for an accurate determination of cooling water flow to the Rexnord solidifier. A scale was provided to weigh each batch of solidified SRC. The exhaust stack of the Rexnord was modified and attached to the HEAF filter unit. Fume sampling ports were installed in the exhaust stack before and after the HEAF filtration unit.

The HEAF unit is a rotary drum filtration unit which continuously presents a clean filter element to the inlet gas stream. No provisions are included for dirty filter element blowback or cleaning.

Mid Atlantic Infrared Services (MAIS) performed infrared thermography tests and quantified the convective and radiative heat losses from the solidifier. This allowed an estimate to be made of the SRC temperature profile during solidification.

A flow diagram, which shows the transfer of SRC to the Rexnord solidifier, is presented in Figure 7. The Rexnord thermocouple locations are shown in Figure 8. A schematic diagram of the HEAF unit is given in Figure 9.

Detailed test results and conclusions will be reported by ICRC under DOE Contract DE-AC05-78-ORD-3054 (Ref. 9).

A summary of the results reported by ICRC is:

- Design parameters for the Rexnord solidifier and the HEAF filter were established.
- Total fume loads during solidification were 0.022 to 0.03 grains/scf. This was 0.003 to 0.005 lb fume/hr-ft² based on the installed equipment surface area or approximately 1.3×10^{-7} lb fume/lb of SRC solidified.
- 100 percent of the fume particles were larger than 0.4 microns and 90 percent were larger than 0.7 microns.
- The HEAF removal efficiency ranged from 87 to 97 percent of the total particulate loading.

4.6. SRC Waste Water Sampling Operation

Catalytic, Inc. performed an evaluation of waste water generation and treatment for ICRC at the Wilsonville facility to provide data for the demonstration plant design. The study included: sampling on a scheduled basis from 29 April to 30 July 1982; taking grab samples during Runs 239 and 240; and correlating the previous data from the period of February 1978 to December 1981. This evaluation has been provided to ICRC by Catalytic, Inc. (Ref. 10).

Pilot plant waste water sampling, from 29 April through 30 July 1982, took advantage of material balance sampling periods at different steady state conditions. Wilsonville personnel analyzed the samples for pH and sulfide concentration. Other results were determined in outside laboratories. Process water discharge streams and biological treatment plant streams were analyzed to determine the range of contaminant concentrations encountered at Wilsonville. Analysis of the biological system provided information on nitrification, denitrification, and organic and suspended solids removal. This information was used to evaluate the performance of the biological system. Sampling of the waste water treatment system continued through two periods when the pilot plant unit was shut down, since plant upsets are sometimes accompanied by shock loads to a waste water treatment system. The general sample matrix consisted of weekly composites of the "process samples", which were composites of grab samples taken 4 or 5 days per week. "Treatment plant samples" were daily grab samples, generally 5 days per week. Chlorides were weekly composites of daily samples. Table 10 presents a list of process water and waste water streams, sample frequencies and analytical requirements.

In addition to these samples, there were two sets of grab samples taken during plant Runs 239 and 240. These were analyzed for trace organics, trace metals and other inorganics. Also, a sample of SRC product from Run 236 was leached by various methods and analyzed by GC/MS for specified trace organics. Pollutants which were of interest to this study are described in Tables 11 and 12.

Wilsonville treatment plant operating data and SRC "sour water" analyses from February 1978 to December 1981 were compared to the pilot plant operating data from the quarterly reports for these periods. The data were inspected for variability and correlated with respect to operating conditions. The biological treatment data were also evaluated to determine if any design or operating parameters could be derived for demonstration plant use. No sour water data were available for the hydrotreater unit (HTR), since it was not in operation until May of 1981.

A summary of the waste water sampling study is:

- 1) Design waste water loads and variabilities were determined in the SRC sour water (V105) for these major pollutants: Total organic carbon (TOC), chemical oxygen demand (COD), ammonia, as nitrogen (NH₃-N), sulfide and phenolics.
- 2) A log-normal statistical analysis of data from SRC sour water yielded the following waste water characteristics:

	50 percentile values	Hourly Rate(a)
TOC	13,100 mg/l	0.22 lb/hr
COD	53,000 mg/l	1.00 lb/hr
NH ₃ -N	10,400 mg/l	0.19 lb/hr
Sulfide	7,200 mg/l	0.12 lb/hr
Phenol	2,590 mg/l	0.06 lb/hr

(a) Hourly rate is based on 6 tons per day coal feed rate.

- 3) Major pollutants can be consistently removed to very low levels, indicating high treatment efficiencies, with conventional waste treatment facilities.
- 4) SRC product does not produce a leachate which contains trace organic compounds when analyzed with standard leaching test methods.
- 5) Waste water data from Wilsonville prior to December 1981 do not provide any strong correlations between the process waste water analyses and the process variables. These waste water data do not provide design data for plant scale-up, since the waste treatment plant is insufficiently loaded and kinetic correlations cannot be obtained.
- 6) Organic priority pollutants were detected in the process waste water which goes to waste treatment. However, these pollutants are effectively removed. In addition, waste water characterization analysis yielded the following efficiencies:

- a. BOD removal has averaged 99 percent.
- b. COD removal has averaged 91.8 percent.
- c. Phenol removal has averaged 99.9 percent.

7) Low levels of boron, mercury, selenium and zinc were found in the effluent from treatment. The main source of boron, mercury and selenium is apparently the SRC sour water (V105). Zinc was also found in this stream. However, effluent zinc may also be caused by the use of galvanized pipe in the waste water treatment area.

4.7. Residual Fuel Oil Blending Operation

Approximately 240,000 pounds of a synthetic fuel oil blend was prepared for ICRC from deashed SRC and coal-derived solvents. The SRC concentration was approximately 52 weight percent. The test was initiated on 19 October and was completed on 6 November 1982. Six tank trucks were sent to the Pittsburgh Energy and Technology Center (PETC) on 17 and 18 November for combustion and handling tests. This study was briefly mentioned in Run 241 Technical Progress Report (Ref. 8). The combustion test results will be reported at a later date (Ref. 11).

The synthetic fuel oil composition was established by ICRC, who prepared a 50/50 SRC-solvent blend for viscosity-temperature determination at Wilsonville. These viscosity determinations were used to check the plant fuel oil blending operations and to establish the pumping requirements at storage conditions. Interpolation of the Wilsonville viscosity data indicated that 1,050 cp is obtained at 195°F, as shown in Figure 10. The raw data are also presented in Table 13.

The deashed SRC was obtained from Wilsonville Runs 210, 211, 214, 218, 219, 225, 227, 228, 230 to 232, 235 and 236. This material, which was previously stored in sealed drums, was pulverized by Empire Coke Company and returned to Wilsonville in numbered tote bins. The material was not segregated during the grinding operation. The ash content of the SRC in each bin was determined and tote bins with ash concentrations greater than 0.17 weight percent were excluded from the blending study.

The solvent was prepared from Run 241 SRC plant vacuum tower product (tray 3) and Run 240 hydrotreated process solvent in unspecified proportions. The blended solvent properties were:

Density at 75°F, 0.98 (g/ml)
ash, wt % 0.01

G. C. Simulated Distillation		Wt % (Avg.)
Boiling Point Range, °F		
IBP-350		0.10
350-450		7.90
450-500		20.90
500-550		14.90
550-650		23.20
650-850		25.30
850-EP		7.80

Blending Procedure

The initial blending procedure consisted of slowly adding the deashed SRC to the solvent blend at 250°F in the existing coal slurry vessels. A schematic representation of the equipment is shown in Figure 11. Slow addition rate and high solvent temperature were employed to insure that complete dissolution occurred. Since the equipment capacity is limited, several batches were required to fill a tank truck. A viscosity determination was made before the batch was transferred. Solvent weights were determined by the V101 weigh scale and SRC weights were measured with the tote bin weigh scale.

This procedure was used for the first two batches, which were unsuccessful. The viscosity rose to unacceptable values after the blending operation was completed. These results are discussed later.

After the first two batches proved unsuccessful, ICRC project engineers visited the Wilsonville plant site. The following procedure was developed and agreed upon by ICRC and Wilsonville personnel:

Blend concentration: 45 percent SRC, 55 percent solvent

Mixing vessel temperature: $230 \pm 5^{\circ}\text{F}$

Solids addition rate: 20 percent maximum in 30 minutes
100 percent in 2.5 hr

1. Use a nitrogen purge on V101A.
2. Continuously agitate and circulate the vessel contents during solids addition.
3. Sample the blend at 50 percent and 80 percent solids addition and measure the viscosity.
4. Fifteen minutes after all solids are added, determine the viscosity. Maintain agitation and circulation.
 - a. If the viscosity at 195°F is less than 1,000 cp and is constant with shear rate, stop mixing and pump to the tank truck. Obtain a sample for laboratory aging tests.
 - b. If the viscosity at 195°F is less than 1,000 cp but is not constant with shear rate, the mixture is apparently not homogeneous. Continue to mix and recheck the viscosity in 30 minutes.
 - c. If the viscosity at 195°F is greater than 1,000 cp, add solvent and recheck the viscosity in 1 hr. Remove the blend from the vessel if the viscosity reaches 5,000 cp at 195°F.

Discussion of Results

Difficulties were encountered in the preparation of the first two fuel oil batches, and unaccountably high viscosities were obtained. Attempts were made to salvage the batches by adding additional solvent, but the viscosity continued to rise above the 1,050 cp target value. Operating conditions for these runs were:

Batch Number:	1	2	
Tote Bin No.	2	3	
Solids addition time, hrs.	9	6	
Mixing temp., °F	250-300	200-250	
Time after solids addition, hr	Viscosity, cp, at 195°F	Time after solids addition, hr	Viscosity, cp, at 195°F
0	NA	0	NA
2	170		
8 (a)	1,350	11	2,500
14	3,300	14 (b)	5,800
15	4,100	14	2,200
17-1/2	13,000		
19-1/2	17,500	19	3,000
		27	14,000

(a) 200 lb fresh solvent added.

(b) 600 lb fresh solvent added.

These batches were pumped into drums. The coal slurry vessel was hydroblasted after Batches 1 and 2 to insure that all material was removed.

The remaining fuel oil batches were prepared without incident, although the ICRC guidelines could not be strictly followed. Deashed SRC flow was uncontrolled and solids were added to the solvent in less than 15 minutes. Temperatures varied from 175 to 225°F, rather than the desired 225 to 230°F range. Solvent content averaged 48.6 weight percent, but laboratory distillations of several samples indicated the actual solvent content may have been higher. Table 14 presents the operating summary and analyses for the fuel oil batches. The highest viscosity sample from each tank truck was saved for an aging study. The viscosities were unexpectedly lower than the viscosity of the laboratory samples prepared by ICRC.

A laboratory investigation was initiated at Wilsonville in order to explain the high viscosities obtained for the first two fuel oil batches and the low values obtained for later mixing operations. Blends were prepared at 40, 50 and 55 weight percent SRC concentration. Both the SRC and the solvent were obtained from the same materials that were used for the full scale blending operation. A summary of the laboratory fuel oil preparation method is given in Table 15. Three 50 weight percent SRC samples were saved and used for an aging study..

Aging Study

The laboratory SRC blends and the samples taken from the plant blending operation were aged to measure viscosity changes with respect to time under storage conditions. For the laboratory SRC blends, samples were aged at different temperatures and open or closed environments. Viscosities were determined at various storage times. The results of this study are presented in Table 16.

Each plant fuel oil sample was divided into six 1-ounce samples and stored in sealed glass vials at 180°F. Aging data are summarized in Table 17. The data indicate that the viscosity increased slightly at 180°F storage temperature and sealed conditions.

Viscosity data for the unsuccessful plant Batches 1 and 2 are compared to the laboratory blends in Figure 12. The data appear to be inconclusive. The plant data have lower initial viscosities than the laboratory blends, which should not have occurred, since temperature and time effects should have been greater for these plant batches. The low initial viscosities may have been caused by incomplete dissolution or solvent concentrations greater than 50 weight percent. The steep increase in viscosity was possibly caused by combined temperature and time effects, oxygen or foreign material present in the SRC.

The laboratory open beaker viscosity data demonstrated that oxygen caused an increase in viscosity. Blend A had an initial slope similar to that for Batches 1 and 2. Blend C, at lower temperature, also showed a rapid change in viscosity. The laboratory samples which were aged in closed containers, Blend B and Blend A (after 1.5 hours), showed a slower increase in viscosity than the open samples. Laboratory blends were not aged under closed conditions at different temperatures.

In summary, the fuel oil blending study results were:

- 1) Deashed SRC and solvent were successfully blended to produce a synthetic fuel oil with a viscosity less than 1,050 cp at 195°F.
- 2) Blended fuel oil, stored at 180°F under closed conditions, did not exhibit a rapid increase in viscosity with respect to time.
- 3) Blending temperature, storage temperature, exposure to air and storage time were shown to affect the viscosity of fuel oil blends. These variables have not been quantified, but the tests indicated that short mixing times (less than 2 hours), an inert atmosphere and low mixing temperatures (below 225°F) are preferred. Aging studies indicated that 180°F and closed storage conditions are acceptable.

4.8 Trace Contaminant Sampling Operation

Radian Corp. sampled the following gas streams from 7 to 9 May 1983:

- HP Vent Separator (V104) overhead
- Water Scrubber (V106) overhead
- Combined Solvent Decanter (V105) and Solvent Column (T104) vents
- Hydrotreater Recycle Gas (T1059) inlet
- Hydrotreater Vent Gas (T1059) outlet

Catalytic-Wilsonville obtained composite samples of the coal and process solvent for analysis by Radian.

The trace components which were of interest to ICRC were:

<u>Trace Compounds</u>		<u>Trace Elements</u>
H ₂ S	NH ₃	Hg
CO ₂	HCl	Be
CS ₂	HCN	F
Mercaptans	NO _x	As
Thiophenes	SO ₂	Pb

These results will be reported to ICRC by Radian Corp. (Ref.12).

REFERENCES

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9. ICRC, Subcontract to Catalytic, Inc., to be reported in ICRC Quarterly Technical Report - DOE/OR/03054.
10. Catalytic, Inc., "Wilsonville Wastewater Sampling Program," ICRC Program 22.2, Catalytic Contract 99922, January (1983).
11. ICRC, "SRC-I Test Firing Program", to be reported, DOE/OR/03054.
12. ICRC, Subcontract to Radian Corp., "Trace Contaminants Analyses", ICRC Program 16.2, to be reported DOE/OR/03054.

APPENDIX
DEFINITIONS

Ash	Residual mineral matter obtained by muffle furnace burning at 900°C for 3 hours (adapted ASTM D-482).
Ash concentrate	A product of the Kerr-McGee CSD unit first stage separator that is rich in cresol insolubles (ash and UC) with lesser amounts of SRC and solvent.
CSD SRC	A low preasphaltene, ash, and UC product of the CSD unit second stage separator.
Deashing solvent (DAS)	A solvent used to deash the vacuum column bottoms which are fed to the CSD unit.
Distillate solvent	A coal-derived distillate fraction with a boiling range between 450°F at 760 mm Hg (GC and ASTM D-86) and 600°F at 0.1 mm Hg in the laboratory (flask).
Energy rejection	The heating value lost to the ash concentrate as a fraction of the feed coal heating value.
HTR conversion	The fraction of the SRC feed to the HTR unit that is converted to gases and liquids: $\text{Percent conversion} = \frac{\text{HTR SRC in} - \text{HTR SRC out}}{\text{HTR SRC in}} \times 100$
HTR SRC	The hydrotreated solvent refined coal product of the HTR unit.
Light solvent refined coal (LSRC)	A low preasphaltene SRC product of the CSD unit third stage separator.

APPENDIX (continued)

DEFINITIONS

Middle distillate	A coal-derived distillate fraction with a boiling range between 350 and 450°F at 760 mm Hg (GC and ASTM D-86).
Naphtha	A coal-derived distillate fraction with a boiling range from that of C ₅ up to but less than 350°F at 760 mm Hg (GC and ASTM-86).
Process solvent	Feed solvent to the SRC unit and HTR unit, which may be the distillate solvent or a blend of distillate solvent with light SRC and/or HTR SRC in variable concentrations.
Solvent refined coal (SRC)	A cresol-soluble product of the coal liquefaction process which is non-distillable at 600°F and 0.1 mm Hg in the laboratory.
T102 Vacuum Column bottoms (CSD feed)	A residual mixture of SRC, solvent, ash, and UC.
UC	Organic material that is insoluble in boiling cresol.

Table 1
SRC Unit Operating Conditions

Run no.	Coal type	Preheater inlet pressure, psig	Dissolver pressure, psig	Temperature, °F				R101 volume in use, %	Feed coal rate, lb/hr	LSRC added, lb/hr	Coal space rate, MF, 1b/hr-ft ³ (c)			Wt % LSRC in process solvent
				Preheater outlet	R101 Bottom	Dissolver Outlet	MF MAF				Slurry conc, % MF coal			
235AB	Ky 9	2,144	2,100	750	797	837	75	531	490	46	39.1	37.5	5.3	
235C	Ky 9	2,145	2,100	793	804	833	75	520	485	51	38.3	38.4	6.2	
235D	Ky 9	2,144	2,100	792	804	834	75	535	493	51	39.4	37.7	5.9	
235E	Ky 9	2,148	2,100	787	803	836	75	546	504	49	40.2	39.3	5.9	
240A	Ill 6	2,185	2,125	789	817	825	50	346	307	36.1	36.5	36.5	6.0	
240B	Ill 6	2,190	2,130	785	817	825	50	351	310	28.3	36.8	36.8	4.7	
240C	Ill 6	2,227	2,150	779	814	826	50	734	650	65.7	38.0	38.0	5.5	

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Run no.	Feed gas scfh	Mscf Ton of MF coal	Mole % H ₂ in feed gas	Hydrogen partial pressure, psia(b)				Temperature (°F)		Conversion to cresol solubles, % MAF coal	Coal reactivity, % long run(a)	Microautoclave conversion, dist. solv. act., %	
				B102 inlet	R101 outlet	% MAF coal	V103 HP separator	V110 LP separator				Kinetic	Equilibrium
235AB	8,100	30.5	85.4	1,840	1,320	2.20	730	667	93.6	78.4	74.3	61.7	
235C	8,060	31.0	84.5	1,830	1,310	2.10	725	660	94.5	77.6	70.5	64.7	
235D	7,790	29.1	84.7	1,830	1,170	2.60	724	664	93.7	78.5	72.3	60.7	
235E	8,360	30.6	87.8	1,900	1,380	2.10	706	659	94.0	80.5	76.4	60.6	
240A	5,240	30.3	84.7	1,863	1,315	2.26	685	628	93.7	78.5	74.6	64.9	
240B	5,250	29.9	84.5	1,863	1,354	2.49	687	630	92.1	78.4	70.3	66.4	
240C	7,250	19.8	83.5	1,872	1,200	1.78	700	663	88.3	78.4	72.3	63.2	

(a) Thirty minutes at 750°F. 2:1 solvent/coal ratio. Solvent - 25% tetralin and 75% 1-methylnaphthalene.

(b) Dissolver outlet hydrogen partial pressure (psia) = (P at B102 inlet (psia)) x (mole frac. H₂ in B102 feed gas) x (1 - $\frac{H_2 \text{ consumed (1b/hr)}}{H_2 \text{ in B102 feed (1b/hr)}}$)

(c) Using 75% volume = 13.58 ft³, 50% volume = 9.2 ft³.

Table 2
Feed Coal Analyses

Coal Date Run	Ky 9 15-16 Nov 81 235AB	Ky 9 25 Nov 81 235C	Ky 9 16 Dec 81 235D	Ky 9 18 Dec 81 235E	III 6 11 June 82 240A	III 6 21 June 82 240B	III 6 12 July 82 240C
<u>Ultimate analysis, wt %</u>							
Carbon	74.77	73.18	73.12	72.54	67.04	67.92	67.80
Hydrogen	5.15	4.92	4.88	5.00	4.39	4.51	4.43
Nitrogen	1.34	1.75	2.00	1.56	1.26	1.11	1.31
Ash	7.68	6.87	7.61	7.67	11.33	11.65	11.41
Sulfur	3.07	3.22	2.79	2.84	3.13	3.28	3.51
Oxygen	7.92	10.01	9.51	10.34	12.77	11.46	11.51
Chlorine	0.07	0.05	0.09	0.05	0.08	0.07	0.03
<u>Proximate analysis, wt %</u>							
Moisture	1.47	1.61	1.70	2.29	2.78	0.80	3.21
Ash	7.57	6.76	7.48	7.50	11.02	11.56	11.06
Volatile matter	32.09	37.34	33.53	35.67	35.00	39.54	36.15
Fixed carbon	58.87	54.29	57.24	54.54	51.20	48.10	49.58
Coal reactivity, %	78.4	77.6	78.5	80.5	78.5	78.4	78.4
Dry heating value, BTU/lb	13,250	13,444	13,234	13,709	12,354	12,168	12,255
<u>Sulfur forms, wt %</u>							
Pyritic	1.20	0.91	1.18	1.13	1.28	1.17	1.21
Sulfate	0.06	<0.01	0.03	<0.01	<0.01	0.10	0.11
Sulfide	0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.02
Organic	1.80	1.92	1.80	1.94	2.08	2.01	2.07
<u>Mineral analysis, wt %</u>							
Phos. pentoxide, P ₂ O ₅	0.07	0.07	0.07	0.07	0.07	0.03	0.10
Silica, SiO ₂	49.53	49.05	51.86	49.06	46.75	47.37	46.09
Ferric oxide, Fe ₂ O ₃	21.56	20.59	19.54	19.88	17.34	16.64	17.82
Alumina, Al ₂ O ₃	19.00	18.77	19.32	20.03	17.27	18.45	20.07
Titania, TiO ₂	1.30	1.05	1.05	0.97	0.77	0.79	0.96
Lime, CaO	2.14	2.07	2.17	2.23	7.29	8.34	6.53
Magnesia, MgO	0.96	0.96	0.79	0.63	1.01	0.89	0.94
Sulfur trioxide, SO ₃	1.97	2.00	2.37	2.70	4.27	3.97	4.79
Potassium oxide, K ₂ O	1.57	1.10	1.66	1.84	1.55	1.61	1.73
Sodium oxide, Na ₂ O	0.43	0.45	0.73	0.49	1.28	0.51	0.47
Vanadium oxide, V ₂ O ₅	0.12	0.07	0.06	0.07	<0.10	0.06	0.05
Undetermined	1.35	3.84	0.38	2.03	2.40	1.34	0.45
<u>Trace metal analysis, ppm</u>							
Arsenic	2	2	2	2	2	2	2

Table 3
SRC Unit Performance Summary

Coal (Mine) Run Date	Kentucky 9(Fies)				Illinois 6(Burning Star)		
	235AB 15-16 Nov 1981	235C 25 Nov 1981	235D 16 Dec 1981	235E 18 Dec 1981	240A 11 June 1982	240B 21 June 1982	240C 12 July 1982
Coal conversion at V110, % MAF coal	93.6	94.5	93.7	94.0	93.2	92.8	91.9
<u>Input Data</u>							
<u>Coal feed rate, lb/hr</u>							
As-is	537	527	543	554	352	356	746
MF	531	520	535	546	346	351	734
MAF	490	485	493	504	307	310	650
<u>Net input, % MAF coal</u>							
Hydrogen consumption	2.2	2.1	2.6	2.1	2.3	2.5	1.8
Coal ash	8.4	7.1	8.4	8.3	12.8	13.2	12.9
TOTAL NET INPUT	10.6	9.3	11.0	10.4	15.1	15.7	14.7
<u>Yield, % MAF coal</u>							
C ₁	2.5	2.3	2.0	1.8	2.5	2.2	1.2
C ₂	1.8	1.7	1.6	1.6	2.0	2.1	1.1
C ₃	1.6	1.5	1.3	1.4	1.6	1.7	0.9
C ₄	1.2	0.8	1.3	0.4	0.6	0.6	0.4
C ₅ -C ₆	0.7	0.6	0.7	0.5	0.7	0.8	0.4
CO	0.2	0.1	0.2	0.2	0.3	0.2	0.1
CO ₂	1.4	1.3	1.2	1.2	1.4	1.5	1.3
NH ₃	0.2	0.3	0.2	0.2	0.2	0.2	0.2
H ₂ S	1.3	1.5	1.7	1.7	1.9	1.9	1.4
H ₂ O	4.7	4.6	4.0	3.5	5.7	4.8	4.5
Naphtha (IBP-350°F)	3.4	3.2	3.1	3.9	3.4	2.9	1.9
Middle distillate (350-450°F)	5.2	6.1	5.8	5.0	4.6	4.2	2.0
Distillate solvent (450°F-EP)	12.7	17.2	14.5	18.9	12.9	14.3	19.3
<u>SRC</u>							
Oil	11.9	8.1	8.4	8.3	8.5	8.9	6.3
Asphaltene	31.3	28.6	29.4	25.6	32.7	30.4	28.5
Preasphaltene	15.5	18.6	19.3	22.2	17.1	18.0	22.3
Ash(a)	7.8	6.5	7.5	7.5	12.8	13.1	12.6
U%	7.3	6.5	8.9	6.7	6.3	8.0	10.5
TOTAL YIELD (less net input)	110.7 (100)	109.5 (100)	111.1 (100)	110.6 (100)	115.2 (100)	115.8 (100)	114.9 (100)

(a) Corrected for sodium carbonate addition.

Table 4
CSD Unit Feed Analyses

Run no.	Composition, wt %					Soft, point, °F(a)	Elemental analysis, wt %					
	Ast.	UC	Solv	O/I	Asph.		Carbon	Sulfur	Nitrogen	Hydrogen	Oxygen	
235A8	8.5	9.5	7.1	15.1	41.7	18.1	267	77.85	1.46	1.70	5.53	4.93
235C	8.2	7.7	10.5	11.3	40.2	22.1	281	78.46	1.51	1.77	5.16	4.90
235D	9.0	10.0	9.3	12.1	37.8	21.8	298	78.63	1.29	1.90	5.22	3.96
235E	9.2	7.8	9.9	12.7	34.6	25.8	284	77.96	1.58	1.72	5.22	4.32
240A	13.6	6.7	8.6	11.2	41.6	18.3	307	74.37	1.78	1.52	4.90	3.83
240B	14.0	8.5	8.7	12.0	37.4	19.4	368	74.44	1.84	1.56	4.93	3.23
240C	11.9	10.8	9.1	8.3	35.0	24.9	411	74.14	1.67	1.42	4.94	5.93

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CSD Unit Product Distributions

Run no.	CSD feed rate, lb/hr	DAS free	DAS free	DAS free	DAS losses			SRC recovery, %	Energy rejection, %
		ash conc., lb/hr	SRC, lb/hr	LSRC, lb/hr	Total, lb/hr	Product, lb/hr	Non-product, lb/hr		
235A3	401.8	125.7	239.0	46.1	12.8	5.7	7.2	85.0	19.4
235C	409.9	138.6	220.1	51.1	9.9	6.7	3.2	80.7	23.2
235D	421.6	142.1	228.3	51.2	17.6	9.1	8.5	83.6	23.8
235E	435.8	145.9	240.9	49.0	12.6	6.3	6.3	79.0	25.2
240A	281.2	98.5	146.6	36.1	11.0	3.3	7.7	83.0	19.7
240B	339.6	113.3	193.0	33.3	16.0	8.4	7.6	83.0	21.3
240C	634.9	275.8	293.4	65.7	23.5	20.1	3.4	70.7	32.8

(a) Run 235 values are average melting points, Run 240 values are by ring and ball test (silicone).

Table 5
CSD Unit Product Analyses

Run no.	Ash concentrate, wt %							LSRC, wt %			
	Ash	UC	Solv	Oil	Asph.	Preasph.	DAS	Ash	UC	Oil + Asph. + Preasph.	Solv(a)
235AB	31.7	46.3	1.2	1.7	5.4	18.6	1.1	0.01	0.32	60.97	38.7
235C	25.1	39.7	1.9	-	2.9	29.8	1.6	0.02	0.08	65.90	34.0
235D	27.1	42.9	2.4	-	3.5	22.4	2.7	< 0.01	0.03	65.47	34.5
235E	25.9	46.6	0.5	0.9	2.5	20.6	3.0	0.01	0.04	73.45	26.5
240A	39.7	32.3	2.7	0.6	5.8	17.7	1.2	0.03	0.47	71.10	28.4
240B	39.4	34.6	5.1	-	4.0	18.6	-	0.01	0.15	78.84	21.0
240C	27.0	36.0	7.3	1.4	5.4	17.8	5.1	0.04	0.12	83.94	15.9

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Run no.	Composition, wt %							Soft point, °F(b)	Elemental analysis, wt %				
	Ash	UC	Solv	Oil	Asph.	Preasph.	DAS		Carbon	Hydrogen	Sulfur	Nitrogen	Oxygen
235AB	0.20	0.13	8.7	9.4	62.3	17.57	1.7	326	86.00	5.86	0.79	1.52	5.70
235C	0.09	0.13	9.9	10.4	49.9	27.68	1.9	333	85.85	5.81	0.93	2.02	5.30
235D	0.04	0.45	8.3	15.5	56.8	16.71	2.2	333	85.82	5.97	0.69	1.84	5.64
235E	0.04	0.16	10.1	12.9	56.6	19.50	0.7	314	85.06	5.84	0.79	1.75	6.52
240A	0.07	0.23	7.5	14.6	60.1	16.10	1.4	328	85.34	5.90	0.98	1.77	5.94
240B	0.06	0.10	9.9	14.0	55.8	16.64	3.5	324	86.10	6.00	0.83	1.66	5.35
240C	0.07	0.06	9.0	12.5	60.4	16.17	1.8	319	84.18	6.09	1.07	1.58	7.08

(a) Includes DAS.

(b) By ring and ball test (glycerol).

Table 6
CSL Unit Performance Summary

Run Date	235AB 15-16 Nov 81	235C 25 Nov 81	235D 16 Dec 81	235E 18 Dec 81	240A 11 June 82	240B 21 June 82	240C 12 July 82
<u>Yield, % T102 bottoms</u>							
<u>Ash concentrate</u>	30.6	33.9	33.7	33.5	35.2	33.4	43.5
Distillate solvent	0.7	0.8	0.8	1.2	1.0	0.6	3.3
Oil	0.5	0.1	0.6	0.3	0.2	0.1	1.7
Asphaltenes	1.7	1.0	1.2	0.8	2.1	1.4	2.5
Preasphaltenes	5.7	10.1	7.5	6.9	6.3	6.3	7.1
UC	12.3	13.4	14.5	15.6	11.5	11.7	16.5
Ash	9.7	8.5	9.1	8.7	14.1	13.3	12.4
<u>SRC</u>	58.2	53.7	54.2	55.3	52.0	56.8	46.2
Distillate solvent	6.1	6.3	5.7	6.0	4.0	5.8	4.3
Oil	5.5	5.6	8.4	7.1	7.7	8.3	5.9
Asphaltenes	36.2	26.8	30.8	31.3	31.7	32.8	28.4
Preasphaltenes	10.2	14.9	9.1	10.8	8.5	9.8	7.6
UC	0.1	0.1	0.2	0.1	0.1	0.1	0.0
Ash	0.1	0.0	0.0	0.0	0.0	0.0	0.0
<u>LSRC</u>	11.2	12.4	12.1	11.2	12.8	9.8	10.3
Distillate solvent	4.3	4.2	4.2	3.0	3.6	2.1	1.6
Oil							
Asphaltenes	6.9	8.2	7.9	8.2	9.1	7.7	8.7
Preasphaltenes							
UC	0.0	0.0	0.0	0.0	0.1	0.0	0.0
Ash	0.0	0.0	0.0	0.0	0.0	0.0	0.0

Table 7
HTR Unit Performance Summary and Operating Conditions

Run number		SRC unit	Feed, lb/hr	SRC in feed, wt %	Reactor temp, °F	Hydrotreater yields, % SRC							Conversion, %	WHSV feed rate, (lb/hr) lb catalyst	
HTR unit						H ₂ cons.	NH ₃ , H ₂ S	H ₂ O	C ₁ -Cs	Naphtha	Middle distillate	Distillate solvent			
-	3-235-1	235AB	507	46.5	776	-2.6	1.6	4.7	2.3	6.5	3.8	16.1	67.6	32.4	2.01
-	3-235-2		542	46.0	783	-3.3	1.4	4.9	3.0	5.0	3.2	18.0	67.8	32.2	2.14
-	3-235AB	235C	507	45.4	782	-2.9	1.6	5.0	3.1	5.8	2.8	17.9	66.8	33.2	2.00
-	3-235-3		538	45.0	782	-2.9	1.2	5.4	3.2	4.4	1.7	18.9	68.2	31.8	2.13
-	3-235-4	235C	398	45.5	781	-3.5	1.6	6.9	3.7	5.9	2.9	17.4	65.1	34.9	1.57
-	3-235C		512	45.9	782	-2.8	1.5	5.3	3.1	3.5	3.0	14.6	71.8	28.2	2.02
-	3-235-5		525	45.9	784	-2.7	1.2	5.3	3.1	4.6	1.8	11.5	75.1	24.9	2.16
-	4-235-6	235D	482	48.3	809	-4.1	1.5	6.6	6.0	10.3	5.0	24.0	50.7	49.3	1.92
-	4-235-7		454	45.5	806	-3.5	1.6	5.1	5.4	8.1	3.9	25.1	54.3	45.7	1.81
-	4-235D	235E	469	46.3	809	-4.0	1.6	5.0	6.1	9.1	4.6	19.5	58.2	41.8	1.87
-	4-235E		443	48.4	810	-3.9	1.7	6.4	6.6	9.9	4.6	22.5	52.2	47.8	1.77
-	4-235-8	235E	430	47.3	810	-3.7	1.8	5.9	4.6	12.0	6.1	17.4	55.9	44.1	1.71
-	4-235-9		468	47.6	810	-3.0	1.7	4.6	5.5	11.3	5.2	16.9	57.9	42.1	1.86
-	8-240-1	240A	342	47.2	763	-3.8	1.9	6.0	3.0	4.9	1.9	30.4	55.7	44.3	1.37
-	8-240-2		289	48.2	763	-3.8	2.2	5.3	3.5	6.7	2.4	24.9	58.8	41.2	1.15
-	8-240A	240B	317	47.5	762	-3.8	1.9	4.6	5.2	7.1	3.1	23.5	58.5	41.5	1.27
-	8-240-3		295	48.2	762	-3.3	2.0	5.2	2.9	5.4	2.0	27.1	58.8	41.2	1.18
-	8-240-4	240C	288	49.5	762	-3.8	1.9	4.7	2.7	5.9	2.4	26.1	60.1	39.9	1.15
-	8-240B		308	49.8	760	-3.5	1.9	6.6	2.5	4.8	2.4	26.1	59.2	40.8	1.23
-	8-240-5	240C	299	48.2	761	-3.5	2.0	5.7	5.7	2.3	0.7	28.9	58.1	41.9	1.20
-	8-240-6		306	48.1	761	-3.8	2.2	4.9	4.0	5.8	3.4	23.2	60.4	39.6	1.22
-	8-240C		326	48.6	762	-3.4	1.8	4.6	3.2	6.3	3.6	27.0	56.3	43.7	1.30

(Table continued)

Table 7 (continued)
HTR Unit Performance Summary and Operating Conditions

Run number	SRC unit	HTR unit	Solvent activity, % (V1074)		Gas rate, scfh		Recycle gas, H ₂ mole %	Inlet hydrogen partial pressure, psia
			Kinetic	Equilibrium	Hydrogen B1200(a)	Recycle gas, B1201		
235AB	-	3-235-1	82.0	84.1	-	4,550	80.9	2,362
	-	3-235-2	75.1	82.0	-	4,340	83.1	2,478
	-	3-235AB	79.2	80.5	-	4,990	74.9	2,430
	-	3-235-3	77.6	77.4	-	5,970	-	2,360
	-	3-235-4	77.1	79.7	-	7,100	77.6	2,290
235C	-	3-235C	79.9	83.1	-	6,300	81.8	2,360
	-	3-235-5	75.0	83.2	-	5,800	84.2	2,414
235D	-	4-235-6	75.5	84.6	-	2,460	69.7	2,350
	-	4-235-7	75.6	78.8	-	2,380	71.8	2,340
	-	4-235-	76.2	84.1	-	3,620	-	2,290
	235E	4-235E	81.4	81.9	-	3,670	75.8	2,360
	-	4-235-8	73.5	80.5	-	3,570	75.3	2,340
34	-	4-235-9	70.8	82.1	-	3,490	-	2,390
	-	8-240-1	76.8	79.1	1,640	3,614	81.1	2,383
	-	8-240-2	77.1	78.8	1,470	3,413	78.8	2,304
	240A	8-240A	76.9	81.6	1,390	3,500/5,600	80.1	2,295
	-	8-240-3	71.9	-	1,240	5,680	79.3	2,277
	-	8-240-4	69.7	82.1	1,090	5,250	77.2	2,277
	240B	8-240B	77.3	81.8	1,170	5,860	79.2	2,300
	-	8-240-5	73.3	78.0	1,130	4,980	74.7	2,234
	-	8-240-6	70.9	78.1	1,070	5,313	74.5	2,230
	240C	8-240C	69.3	81.4	1,240	5,200	77.8	2,243

(a) The makeup hydrogen flow meter was faulty for Run 235.

Table 8
HTR Unit Analytical Data

SRC unit	HTR unit	CSD-SRC/HTR-SRC, wt % (a)					Elemental analysis, wt % (CSD-SRC/HTR-SRC)					
		Oil	Asph.	Preasph.	UC	Ash	Carbon	Sulfur	Nitrogen	Hydrogen	Oxygen	Ash
235AB	3-235-1	17.3/40.4	61.1/59.0	21.5/0.5	0.0/0.1	0.1/0.0	86.59/88.22	0.85/0.09	1.05/1.16	5.85/6.96	5.57/3.54	0.09/0.03
	3-235-2	12.4/39.8	66.7/59.6	20.9/0.1	0.0/0.5	0.0/0.0	85.88/88.84	0.89/0.33	1.46/1.02	5.81/6.79	5.94/3.01	0.02/0.01
	3-235AB	10.5/43.2	69.5/55.3	19.6/1.5	0.2/0.0	0.2/0.0	86.00/88.90	0.79/0.20	1.52/1.48	5.86/6.68	5.70/2.70	0.13/0.04
	3-235-3	9.2/43.3	72.6/52.4	13.1/4.2	0.1/0.1	0.1/0.0	85.70/88.57	0.51/0.13	1.57/1.34	6.06/6.82	6.10/3.05	0.06/0.09
	3-235-4	14.4/43.9	64.7/53.0	20.8/3.1	0.0/0.0	0.1/0.1	86.18/88.70	0.82/0.30	1.54/1.54	5.84/6.65	5.55/2.74	0.07/0.07
235C	3-235C	11.8/38.5	56.6/53.6	31.4/7.5	0.1/0.3	0.1/0.1	85.54/87.79	0.88/0.36	2.15/1.92	5.93/6.44	5.43/3.38	0.07/0.11
	3-235-5	18.4/40.1	64.6/51.8	15.8/7.6	0.1/0.2	0.1/0.1	85.78/87.87	0.65/0.30	1.67/1.70	6.08/6.52	5.77/3.54	0.05/0.07
235D	4-235-6	17.3/54.3	68.1/45.6	14.5/0.0	0.0/0.0	0.1/0.0	85.78/88.90	0.74/0.16	1.16/0.84	5.84/6.68	6.43/3.39	0.05/0.03
	4-235-7	17.4/50.4	66.7/49.4	15.8/0.0	0.1/0.1	0.1/0.0	86.00/88.88	0.85/0.23	1.78/1.58	5.89/6.72	5.40/2.55	0.08/0.04
	4-235D	17.3/50.1	63.5/46.1	13.7/3.8	0.5/0.0	0.0/0.1	85.76/88.48	0.77/0.13	2.10/1.40	6.03/6.54	5.27/3.38	0.07/0.07
	4-235E	14.5/34.6	63.5/59.4	21.9/5.9	0.2/0.1	0.0/0.0	85.58/88.95	0.83/0.09	1.81/1.64	5.88/6.50	5.89/2.81	0.01/0.01
	4-235-8	15.8/42.5	62.5/50.5	21.5/6.9	0.2/0.0	0.0/0.0	85.60/89.04	0.93/0.17	1.86/1.33	5.93/6.30	5.65/3.14	0.03/0.02
240A	4-235-9	15.6/33.5	57.8/59.3	26.5/7.1	0.0/0.0	0.0/0.0	85.63/88.23	1.02/0.40	1.90/1.56	5.98/6.06	5.41/3.67	0.06/0.08
	8-240-1	15.4/47.5	60.7/51.2	22.6/0.9	1.1/0.4	0.1/0.0	84.91/88.38	0.87/0.19	1.97/1.69	5.96/6.90	6.22/2.77	0.07/0.07
	8-240-2	14.4/49.4	67.8/49.0	17.7/0.8	0.1/0.8	0.0/0.1	86.03/88.64	0.91/0.17	1.88/1.40	5.90/7.06	5.27/2.65	0.01/0.08
	8-240A	15.7/53.1	70.0/46.2	14.2/0.1	0.0/0.5	0.0/0.1	85.76/88.36	0.86/0.32	1.98/1.53	5.95/6.96	5.44/2.76	0.01/0.07
	8-240-3	17.0/48.7	52.9/48.8	30.1/2.5	0.0/0.0	0.0/0.0	86.15/89.08	0.85/0.33	2.19/1.75	6.00/6.85	4.76/1.96	0.05/0.00
240B	8-240-4	19.3/49.6	64.4/48.1	16.2/2.0	0.0/0.2	0.0/0.1	85.77/88.54	0.79/0.29	1.70/1.40	6.01/6.91	5.73/2.77	0.00/0.09
	8-240B	17.6/45.9	61.4/50.6	20.9/3.2	0.1/0.2	0.0/0.1	85.71/88.04	0.92/0.23	1.66/1.79	5.92/6.55	5.79/3.34	0.00/0.05
	8-240-5	25.2/37.7	58.1/55.8	16.5/6.0	0.1/0.4	0.1/0.1	85.59/88.30	0.95/0.37	1.66/1.32	5.88/6.30	5.85/3.58	0.07/0.13
	8-240-6	17.4/33.3	64.5/59.0	18.1/7.6	0.0/0.2	0.0/0.0	85.35/88.17	0.94/0.26	1.70/1.55	5.90/6.57	6.11/3.44	0.00/0.01
	8-240C	15.4/38.0	65.9/53.9	18.5/8.1	0.1/0.0	0.1/0.1	84.73/87.33	1.00/0.64	1.76/1.70	6.39/6.62	6.06/3.64	0.06/0.07

(a) Oil, asphaltene, preasphaltene, UC and ash values are renormalized on a distillate solvent free basis.

Table 9
Hydrotreating Catalyst Analyses

SRC run no. Catalyst HTR run No. State Analytical pretreatment Laboratory (d)	235						240			
	Fresh none	Fresh none	3 Presulfided THF	3 EOR Toluene	4(b) Presulfided Toluene	4 EOR Toluene	Fresh catalyst Galbraith	Calcined 850°F HRI	8-240 16 July Toluene Extracted Galbraith	HRI
<u>Component, wt %</u>										
Carbon	-	<0.01	7.47	22.14	18.01	13.61	-	-	10.54(c)	12.59
Hydrogen	-	0.68	1.01	2.47	1.93	1.09	-	-	1.15(c)	1.27
Nitrogen	-	0.19	0.21	0.43	0.13	0.26	-	-	0.69(c)	<0.05
Sulfur	-	0.19	5.19	3.78	3.76	4.78	-	0.182	4.19(c)	5.89
Chlorine	-	0.036	0.08	0.084	0.089	-	0.04	-	0.07	-
Vanadium	-	<0.002	0.24	0.024	0.16	-	-	-	-	-
Zinc	-	0.003	0.08	0.0037	0.0063	-	-	-	-	-
Sodium	-	0.16	1.83	0.59	3.73	2.02	0.026	0.036	0.19	0.172
Cobalt	-	2.73	1.94	1.65	1.44	1.88	2.85	2.73	2.14	2.10
Molybdenum	7.85	7.39	5.28	5.57	4.86	5.81	8.62	9.93	7.16	7.63
Titanium	-	<0.01	3.08	0.32	2.60	1.88	<0.05	-	0.74	1.09
Calcium	-	0.015	0.24	0.14	0.57	0.11	0.053	-	0.35	0.189
Silicon	-	0.034	0.076	0.015	0.016	-	-	-	-	-
Iron	-	0.010	1.57	0.043	0.13	0.015	0.026	-	0.33	0.22
Boron	-	-	-	-	-	-	0.63	-	2.03	-
Extracted, %	-	-	17.8	9.5	16	76.2	-	-	26.8	-
Ash before extraction, %	-	-	49.3	69.4	-	58.6	-	-	60.4	-
Ash after extraction, %	-	-	-	-	-	-	-	-	81.7	-
Process time, hr	0	0	-	431	-	272	0	0	703	703

Note: EOR is "End of run."

(a) Sample of 3 November.

(b) Sample from Run 4-235.

(c) Wilsonville data.

(d) Run 235 analyses are from Galbraith.

Table 10
A Summary of Pilot Plant and Waste Treatment Sample Analyses and Sample Frequencies

	K-111	V105	Hydro-treater**	Raw waste	Feed	*** 1st bioreactor		*** 2nd bioreactor	
						Mixed liquor	Clarifier effluent	Mixed liquor	Clarifier effluent
pH	d	d	d	d	d	d	d	d	d
Sulfide	3	3	3	3	3	3	3	3	3
Chloride	c, m ⁵	c, m ⁵	c, m ⁵	c, m ⁵					
Ammonia*	c	c	c	c	d	d	d	d	d
Phenoolics	c	c	c	c	d	d	d	d	d
Thiocyanate	c	c	c	c	d	d	d	d	d
Total cyanide	c	c	c	c	d	d	d	d	d
TOC	c	c	c	c	d	d	d	d	d
BOD	c	c	c	c	c	c	c	c	c
TSS	-	-	-	d	d	d	d	d	d
Alkalinity	-	-	-	d	d	d	d	d	d
Metals	m ⁴	m ⁴	m ⁴	m ⁴	m ¹¹	m ¹	m ¹	m ¹	m ¹
Nitrate						d	d	d	d
Nitrite						d	d	d	d
TDS						d	d	d	d
Organics	m ²	m ²	m ²	m ²	m ¹	m ¹	m ¹	m ¹	m ¹

* - Ammonia composite done by distillation, one sample/month done in triplicate.

** - Consists of three separate streams V1070, V1080, and caustic scrubber blowdown.

*** - Notation made at each sampling as to whether powdered activated carbon was being fed to the system, or when it was last added.

c - One analysis per week on a composite of daily samples.

d - Daily analyses.

m¹ - Sampled and analyzed once per different steady state run.

m² - Capillary gas chromatograph run on 5 different steady state runs for organics listed in Table 19. Initial run analysis will include gas chromatograph/mass spectrometer identification for calibration.

m⁴ - Two sets (Table 19) to be run in conjunction with the sets run on bioreactor feed and effluents.

m⁵ - Two complete sets (Table 19) will be run in conjunction with the two complete organic samplings. A third sampling might be required.

3 - Sampling and analysis done three times per week.

Table 11
Pollutants Identified for Analysis in Wilsonville Wastewater

Organics

Benzene
Ethylbenzene
Toluene

Base/Neutral

Acenaphthene
Acenaphthalene
Anthracene
Benzo-anthracene
Benzo-pyrene
Benzo-fluoranthene
Benzo-perylene
Chrysene
Dinitrotoluene
Fluoranthene
Fluorene
Naphthalene
Phenanthrene
Phthalates
Pyrene

Acid Compounds

Dimethylphenol
Phenol
Phenolics, total

Metals and Inorganics

Antimony, total
Arsenic, total
Barium, total
Boron, total
Beryllium, total
Cadmium, total
Chromium, total
Copper, total
Lead, total
Magnesium, total
Mercury, total
Nickel, total
Potassium, total
Selenium, total
Silver, total
Sodium, total
Thallium, total
Zinc, total
Cyanide, total
Fluoride, total

Table 12
Organic Pollutants Identified for Analysis
in SRC-I Solid Product Leachate

Volatiles

Acrolein
Acrylonitrile
Benzene
Bromoform
Ethylbenzene
Methyl bromide
Methyl chloride
Methylene chloride
Toluene
Vinyl chloride

Conventional and Nonconventional
Pollutants

Oil and grease
Total organic carbon
Total organic nitrogen

Base/Neutral

Acenaphthene
Acenaphthalene
Anthracene
Benzidine
Benzo-anthracene
Benzo-pyrene
Bromophenyl phenyl ether
Benzo-fluoranthene
Benzo-perylene
Chrysene
Dibenzo-anthracene
Dinitrotoluene
Diphenylhydrazine
Fluoranthene
Fluorene
Indeno-pyrene
Isophorone
Naphthalene
Nitrobenzene
Phenanthrene
Phthalates
Pyrene

Acid Compounds

Dimethylphenol
Dinitrocresol
Dinitrophenol
Nitrophenol
Phenol

Table 13

Viscosity as a Function of Shear Rate and Temperature for the 50/50 SRC-Solvent Blend from ICRC.

<u>Temperature, °F</u>	<u>Shear rate, sec⁻¹</u>	<u>Viscosity, cp.</u>	<u>Viscosity @ 10 sec⁻¹</u>
170	1.7	5,600	
	0.8	5,620	
	0.4	5,680	5,600
	0.2	5,700	
	0.1	6,000	
200	16.8	790	
	8.4	792	
	3.4	805	
	1.7	840	
	0.8	880	790
	0.4	1,000	
	0.2	1,100	
	0.1	1,800	
300	16.8	30	
	8.4	36	
	3.4	40	34
	1.7	50	

Table 14
Composition and Analyses of the Plant SRC-Solvent Fuel Oil Blends for CRC

Date	Tote bin and blend #	Wt % ash tote bin	SRC lbs	Solvent lbs	Wt. % solvent	Blending time, hrs	Wt. % solvent by distillation	Blenc % ash	Blend viscosity 195°F, 10s ⁻¹	Tank truck #
10/20/82	2	0.10	2,580	2,780	51.9	28.5	57.8	-	17,000	Drum
10/25/82	3	0.13	2,500	2,500	50.0	33	52.5	-	13,000	Drum
10/28/82	5	0.08	2,526	3,900	60.7	2		0.04	330	1
10/28/82	6	0.11	2,665	3,257	55.0	2		-	148	1
10/29/82	12	0.14	2,868	3,505	55.0	1		0.02	140	1
10/29/82	13	0.10	2,552	2,500	49.5	1.2		0.06	358	1
10/29/82	14	0.08	2,520	2,500	49.8	1		0.04	640	1
10/29/82	15	0.09	2,545	2,400	48.5	1		0.04	828	1(1)
10/29/82	16	0.08	2,535	2,400	48.6	1.5		0.04	680	1
10/29/82	17	0.06	2,595	2,430	48.4	2		0.05	338	1
10/30/82	18	0.08	2,604	2,400	48.0	2		0.08	396	2
10/31/82	19	0.10	2,486	2,250	47.5	1.5		0.05	600	2
10/31/82	20	0.08	2,348	2,250	48.9	1		0.06	356	2
10/31/82	21	0.07	2,445	2,300	48.5	1	51.4	0.03	280	2
10/31/82	22	0.09	2,570	2,200	46.1	1.5		0.02	606	2
10/31/82	23	0.07	2,462	2,100	46.0	1.5		0.04	708	2(1)
10/31/82	24	0.06	2,494	2,100	45.7	1.5		0.05	530	2
10/31/82	25	0.06	2,949	2,300	43.8	7	50.7	0.05	462	2
11/1/82	26	0.01	3,095	2,500	44.7	5.5	48.0	0.03	676	2
11/1/82	27	0.01	2,401	2,200	47.8	2		0.06	814	3(1)
11/1/82	28	0.08	2,664	2,400	47.4	1.5		0.05	558	3
11/1/82	29	0.08	2,614	2,400	47.9	1.2		0.06	598	3
11/2/82	30	0.08	2,226	2,055	48.0	1.5		0.07	392	3
11/2/82	31	0.11	2,502	2,310	48.0	1.5		0.08	340	3
11/2/82	32	0.15	2,844	2,522	47.0	1.5	48.9	0.11	308	3
11/2/82	34	0.10	2,700	2,400	47.1	2		0.07	570	3
11/2/82	38	0.10	2,960	2,700	47.7	2		0.07	350	3
11/3/82	39	0.13	2,780	2,566	48.0	2.5		0.09	646	4
11/3/82	40	0.07	2,464	2,274	48.0	1.5		0.04	650	4
11/3/82	41	0.10	2,722	2,512	48.0	1.5		0.03	610	4
11/3/82	42	0.08	2,699	2,491	48.0	1.5		0.07	450	4
11/3/82	43	0.10	2,565	2,367	48.0	1.5			582	4
11/3/82	44	0.09	2,582	2,383	48.0	2		0.08	580	4
11/3/82	45	0.12	2,464	2,274	48.0	2		0.05	822	4(1)
11/4/82	46	0.07	2,590	2,391	48.0	1.5		0.05	378	4
11/4/82	47	0.09	2,588	2,388	48.0	1.5		0.06	500	5
11/4/82	48	0.13	2,416	2,230	48.0	1.5		0.02	610	5(1)
11/4/82	49	0.10	2,470	2,280	48.0	1.5		0.05	448	5
11/4/82	50	0.12	2,495	2,303	48.0	1.5		0.05	488	5
11/4/82	51	0.07	2,446	2,257	48.0	1.2		0.04	474	5
11/4/82	52	0.09	2,558	2,361	48.0	1		0.04	350	5
11/4/82	53	0.06	2,478	2,287	48.0	1.5		0.02	328	5
11/4/82	54	0.04	2,478	2,287	48.0	1.5		0.02	548	5
11/6/82	55	0.05	2,757	2,544	48.0	1.3		0.04	586	6
11/6/82	56	0.08	2,434	2,247	48.0	1		0.05	146	6
11/6/82	57	0.11	2,664	2,459	48.0	1.5		0.04	174	6
11/6/82	58	0.05	2,560	2,363	48.0	2		0.05	176	6
	Total		121,460	114,823	48.6					

Note 1: Sample taken for aging study.

Table 15
Wilsonville Laboratory Fuel Oil Blend Preparation.

<u>SRC, wt %</u>	<u>Preparation temp., °F</u>	<u>Viscosity @ 195°F and 10 s⁻¹ shear rate</u>	<u>Observations</u>
55	250	1,800	last 1 to 5 % SRC dissolved slowly, some crust formation
50	250	720	no problems with preparation
50	230	650	no problems with preparation
50	210	500	last 5 to 10 % SRC dissolved slowly, clump and crust form.
50	190	560	same prblems as with 210 preparation
40	230	86	no problems with preparation
40	210	75	no problems with preparation
40	190	60	last 1 to 5 % SRC dissolved slowly, crust formation

Notes 1) Deashed SRC from tote bin #3 and solvent from V123 were dissolved in a stirred open beaker.

2) Blends A, B and C were used for the aging study.

Table 16
Aging Data for the Wilsonville Laboratory 50 Weight Percent SRC Blends

<u>Sample</u>	<u>Method</u>	<u>Storage Temp., °F</u>	<u>Storage Time, hrs</u>	<u>Viscosity @ 195 °F(1)</u>
Blend A	open beaker	250	0	720
		250	1.5	940
	closed container	230	3.8	1,070
		230	7	1,050
		230	10	1,100
		230	18	1,225
		230	28	1,285
		230	37	1,510
Blend B	closed container	230	0	650
		230	7	620
		230	12	705
		230	19	690
		230	31	740
		230	38	1,050
		230	62	1,080
Blend C	open beaker	200	0	560
		200	14	850
		200	31	1,250

(1) Shear rate constant at 10 s^{-1} .

Table 17
Aging Data for the ICRC Fuel Oil Blends

Plant Batch No.	Truck (Serial no.)	Viscosity at temp., °F	Storage time, days														
			0	4	6	8	10	11	13	14	16	17	18	19	22	23	24
15	1 (216-6-994)	195°	750	830	-	1,084	-	1,232	-	1,338	-	1,575	-	-	-	2,025	-
		280°	-	44	-	48	-	50	-	60	-	64	-	-	-	74	-
23	2 (216-3-888)	195°	708	1,064	-	1,512	-	1,524	-	1,722	-	1,698	-	-	-	2,080	-
		280°	-	68	-	64	-	62	-	68	-	72	-	-	-	70	-
27	3 (216-8-761)	195°	814	1,320	-	1,432	1,220	-	1,512	-	2,000	-	-	-	180	-	-
		280°	-	90	-	70	64	-	85	-	72	-	-	-	92	-	-
45	4 (216-6-180)	195°	822	-	1,310	-	1,924	-	2,000	-	-	-	2,225	-	-	2,550	-
		280°	-	60	66	-	74	-	74	-	-	-	-	-	-	90	-
48	5 (215-3-911)	195°	610	530	646	-	960	-	740	-	-	-	-	958	-	-	1,230
		280°	-	42	48	-	58	-	50	-	-	-	-	-	-	63	-

Notes: 1) Samples were stored at 180°F in closed vials.
2) Viscosities were determined at a constant shear rate of 10 s⁻¹.

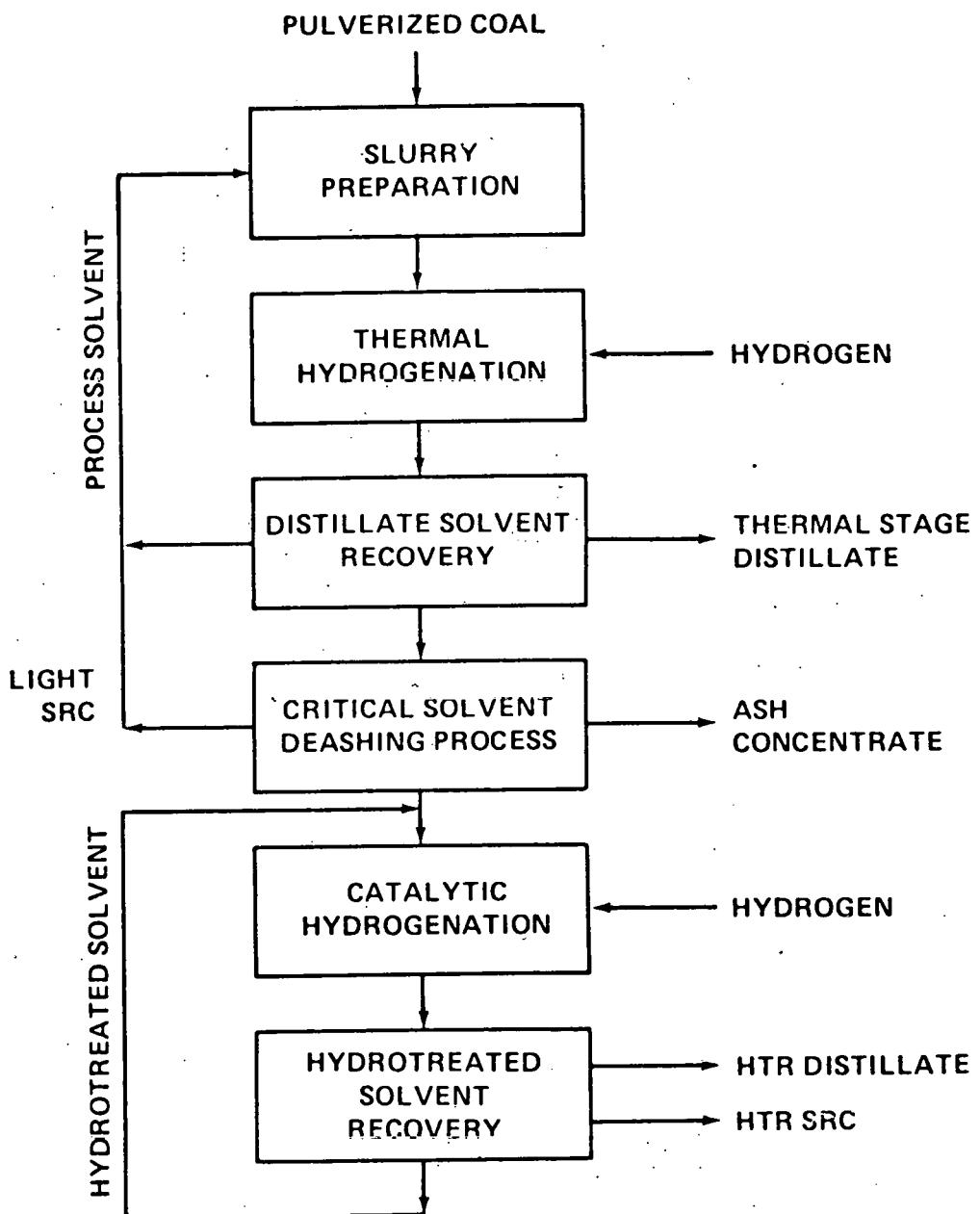


FIG 1. TWO-STAGE LIQUEFACTION
(NON-INTEGRATED MODE)

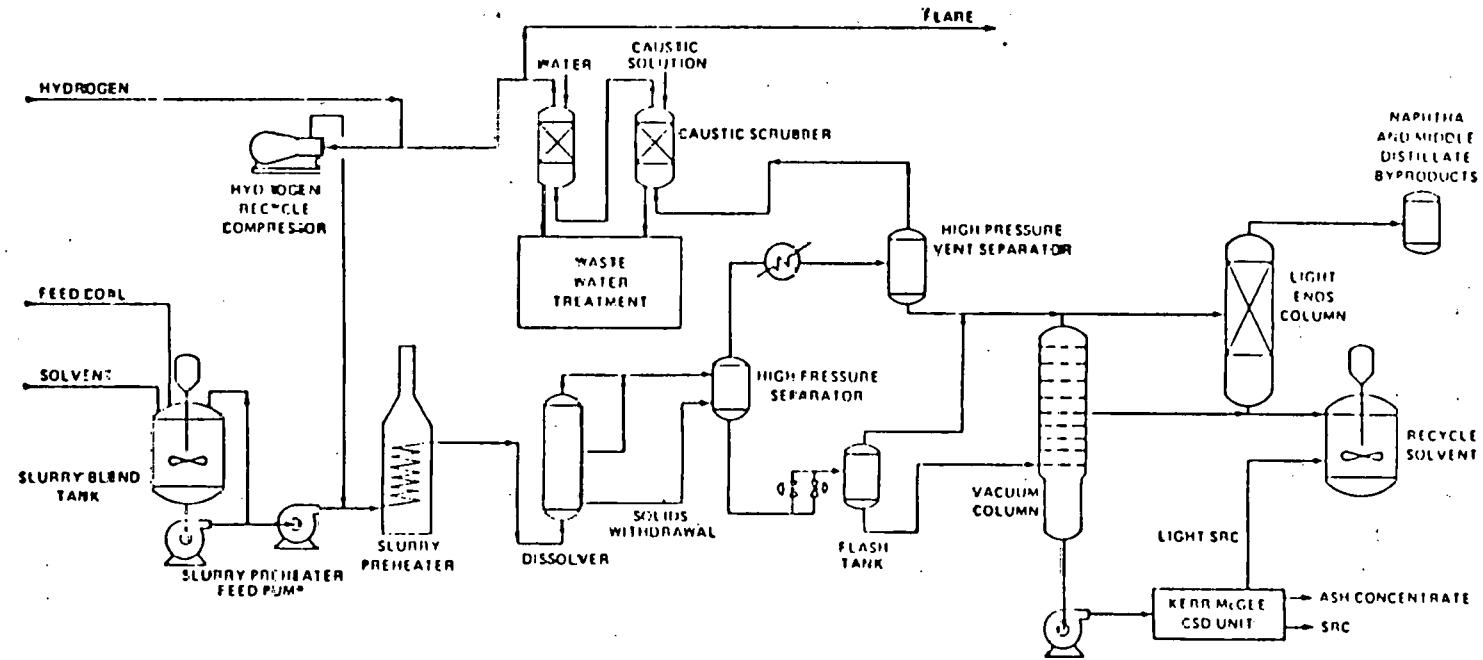


FIGURE 2. SOLVENT REFINED COAL PROCESS

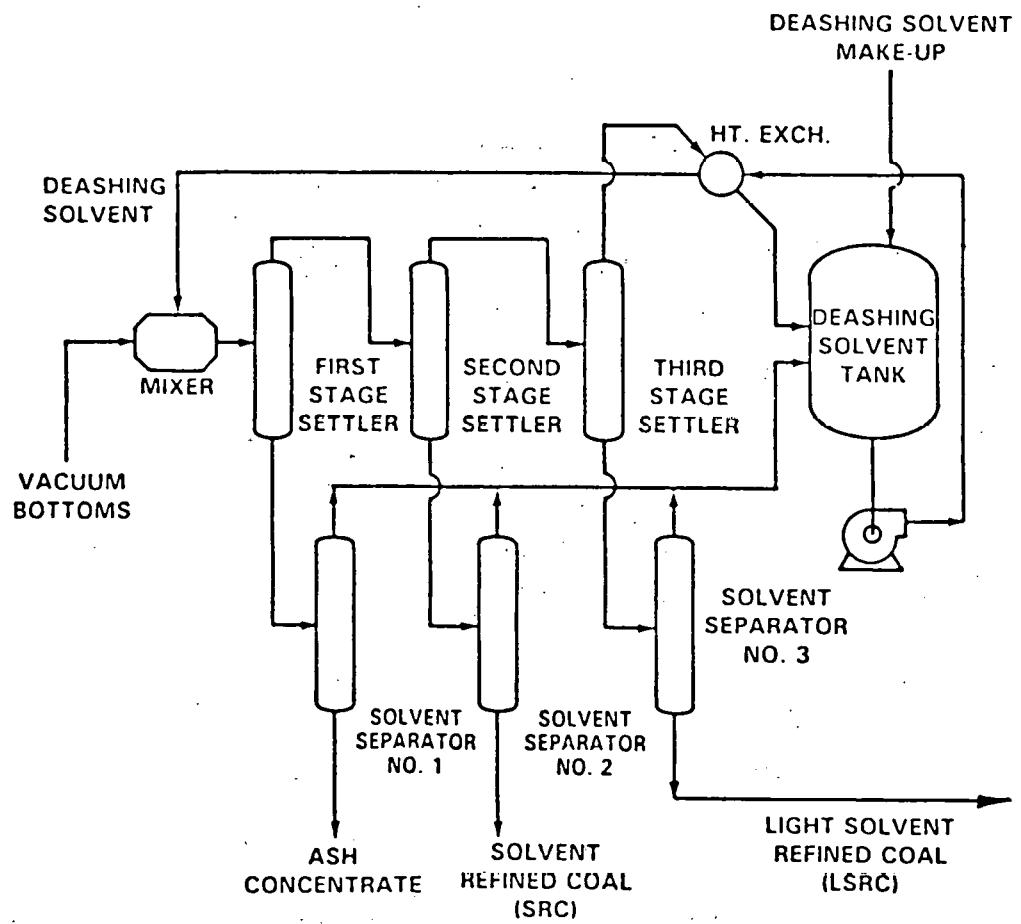


FIGURE 3. CRITICAL SOLVENT DEASHING PROCESS

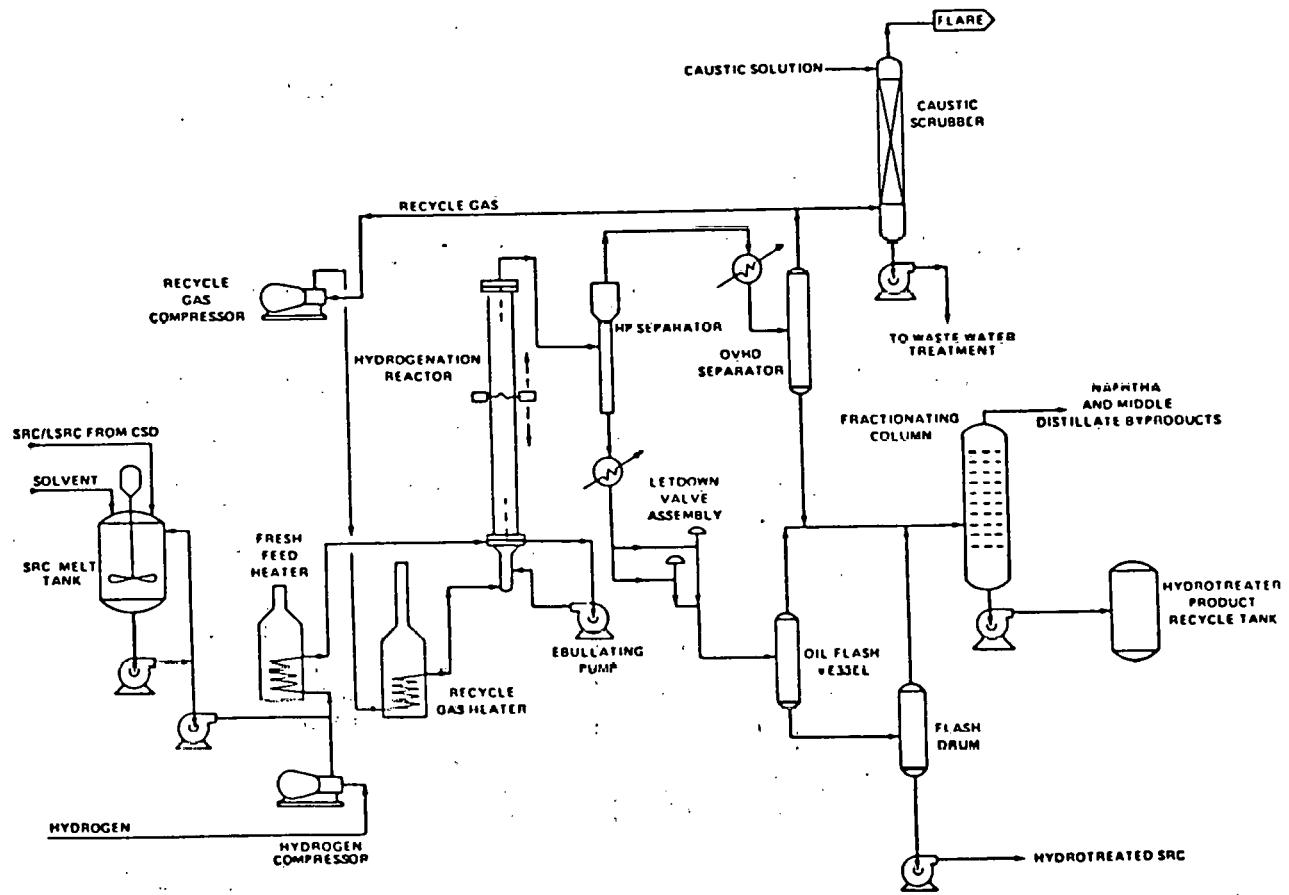


FIGURE 4. EBULLATED BED HYDROTREATER PROCESS

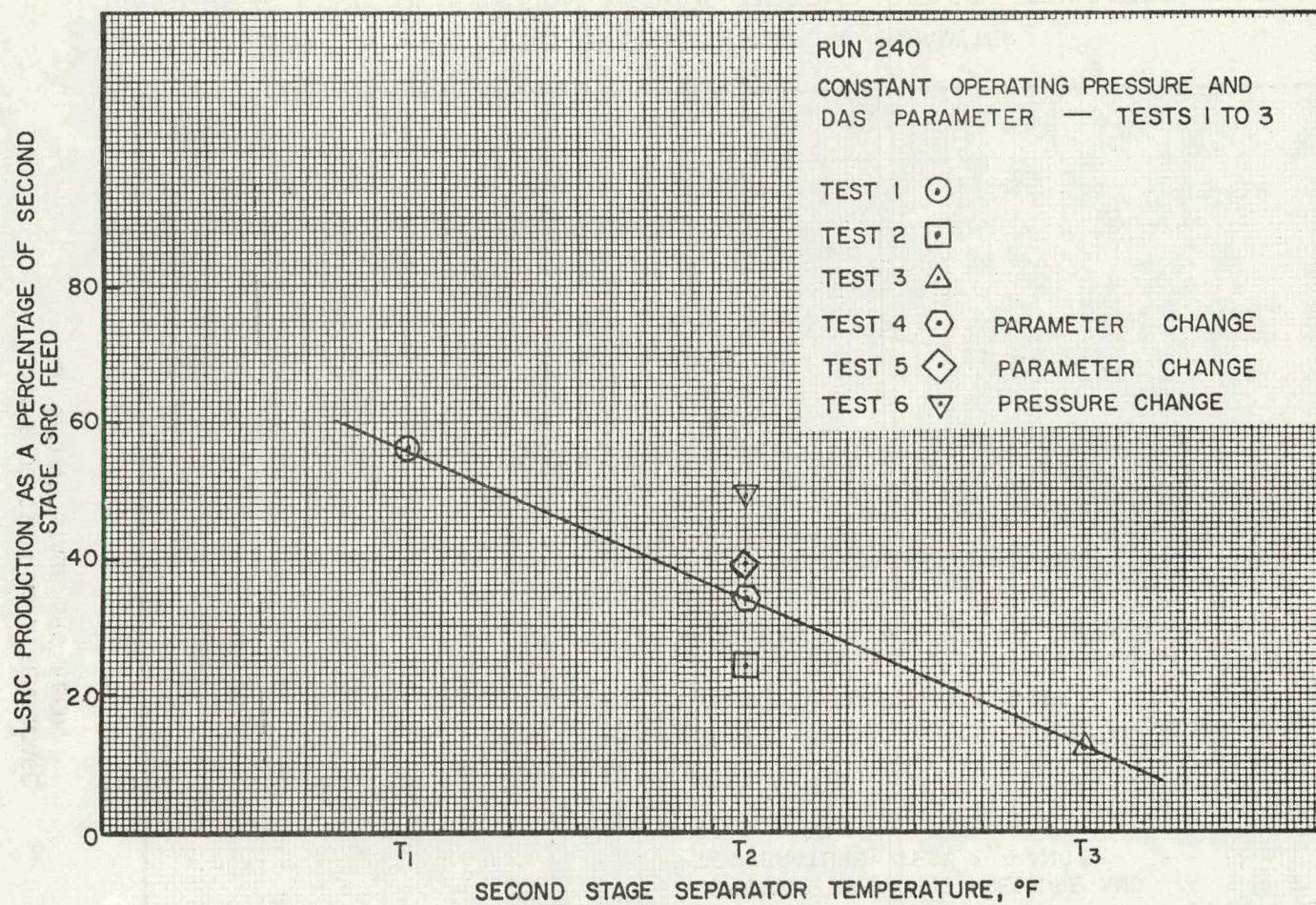


FIGURE 5. LSRC PRODUCTION VERSUS SECOND STAGE SEPARATOR TEMPERATURE

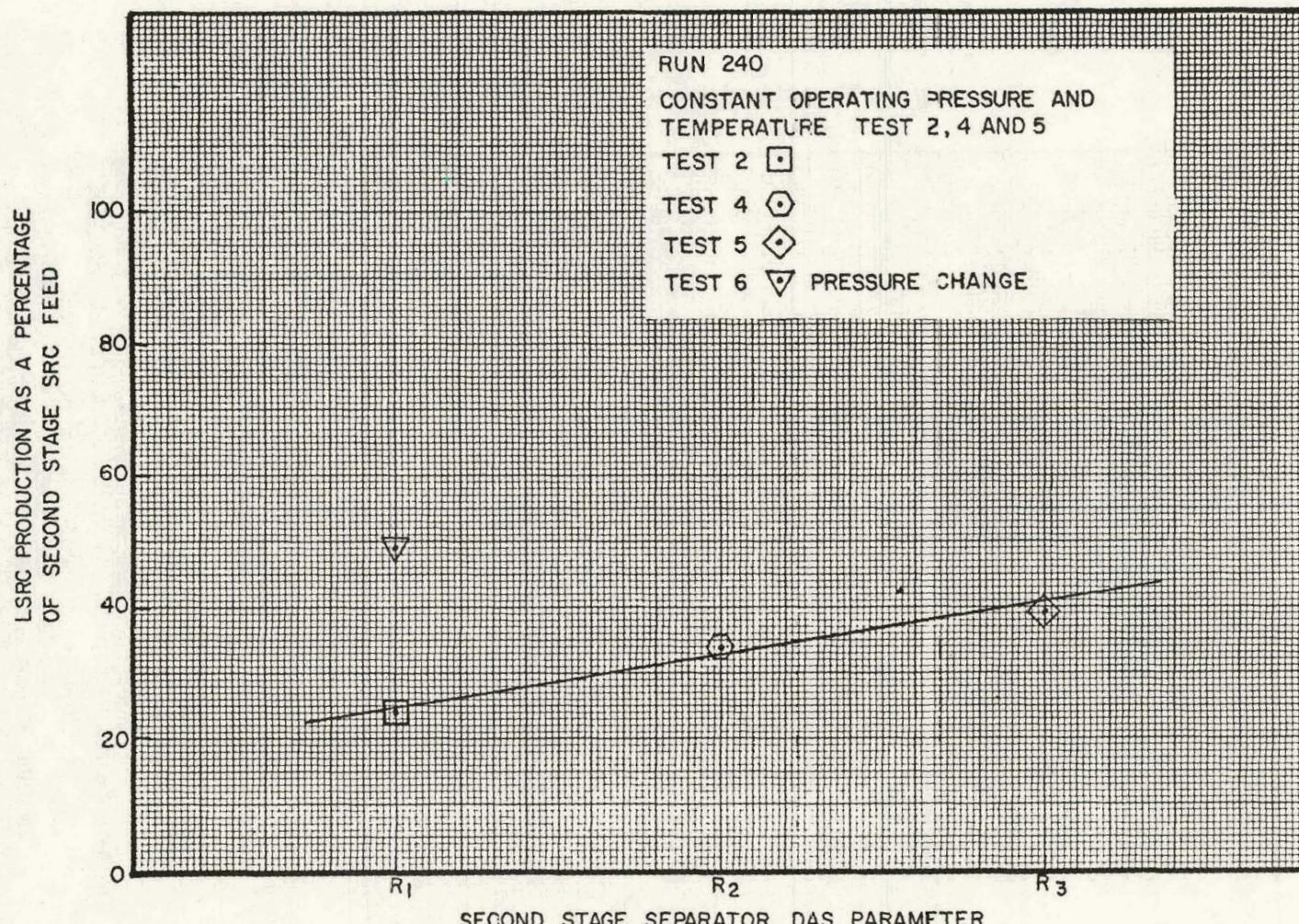


FIGURE 6. LSRC PRODUCTION VERSUS SECOND STAGE SEPARATOR DAS
PARAMETER

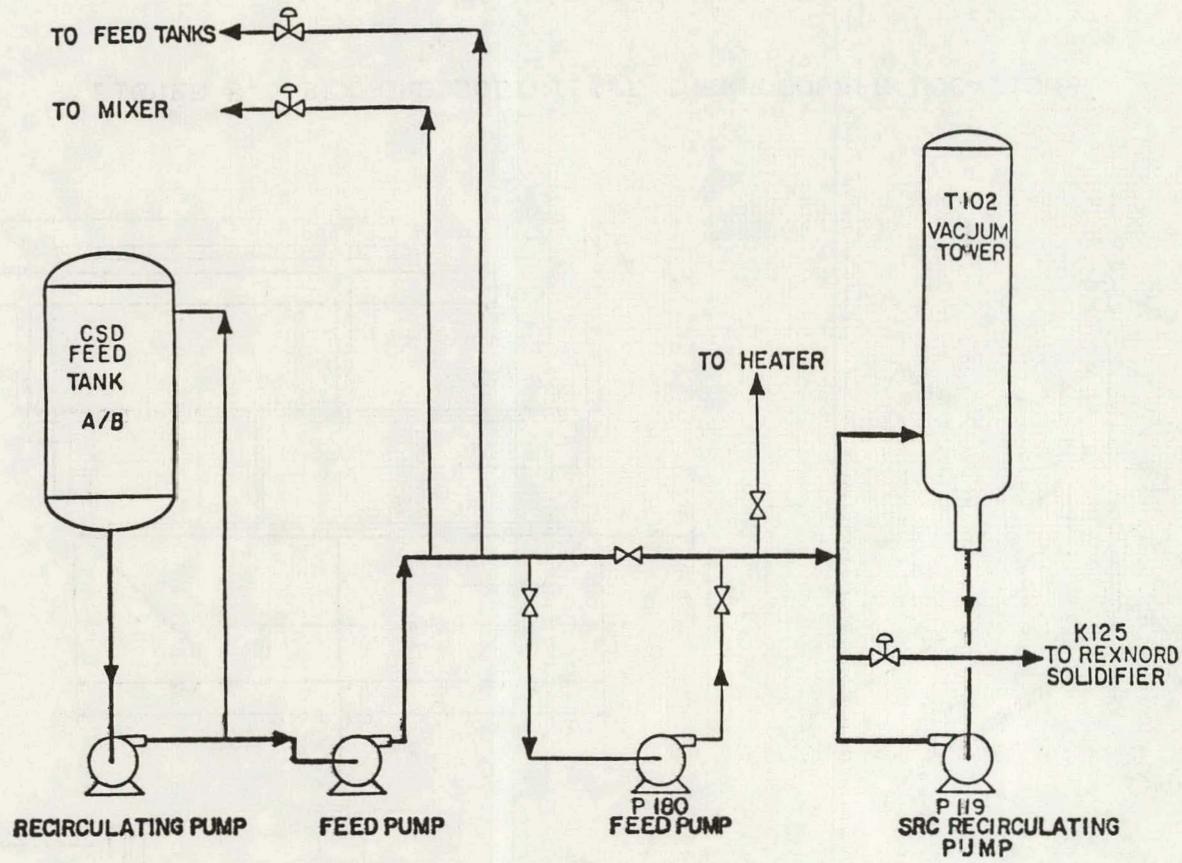


FIGURE 7. REXNORD SOLIDIFICATION TEST - SRC FLOW DIAGRAM

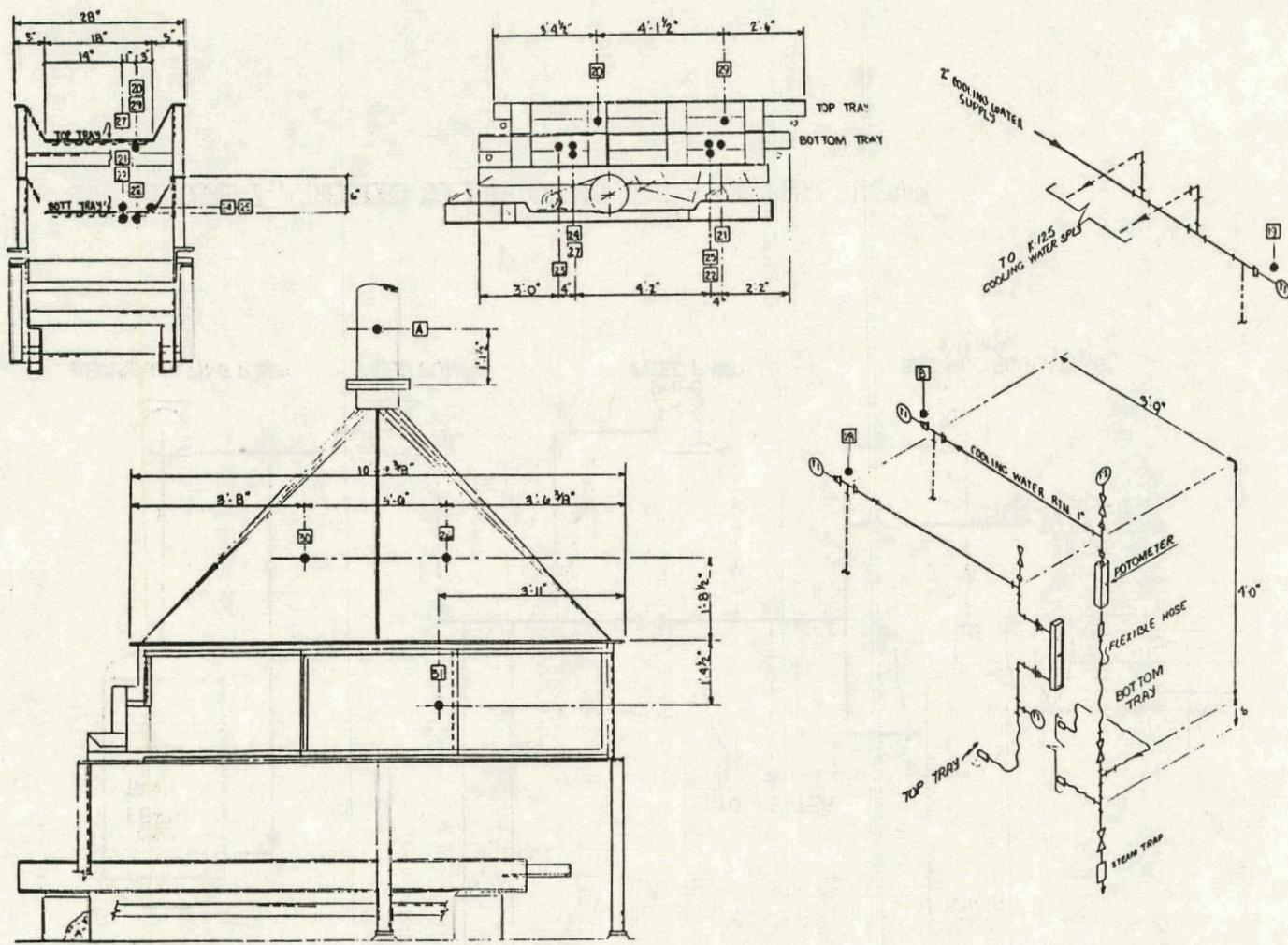


FIGURE 8. REXNORD SOLIDIFIER THERMOCOUPLE LOCATIONS

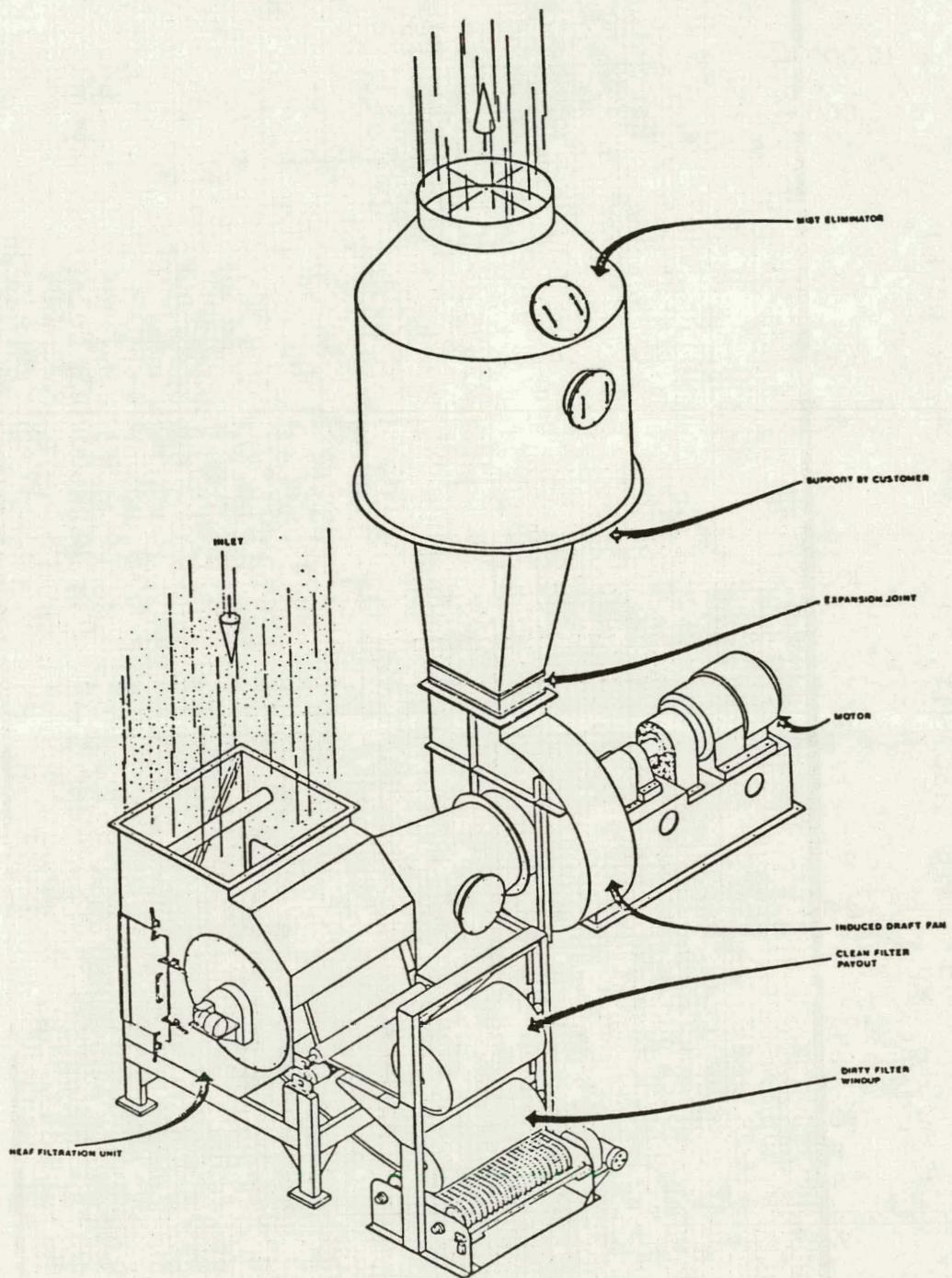


FIGURE 9. ROTARY DRUM - HEAF

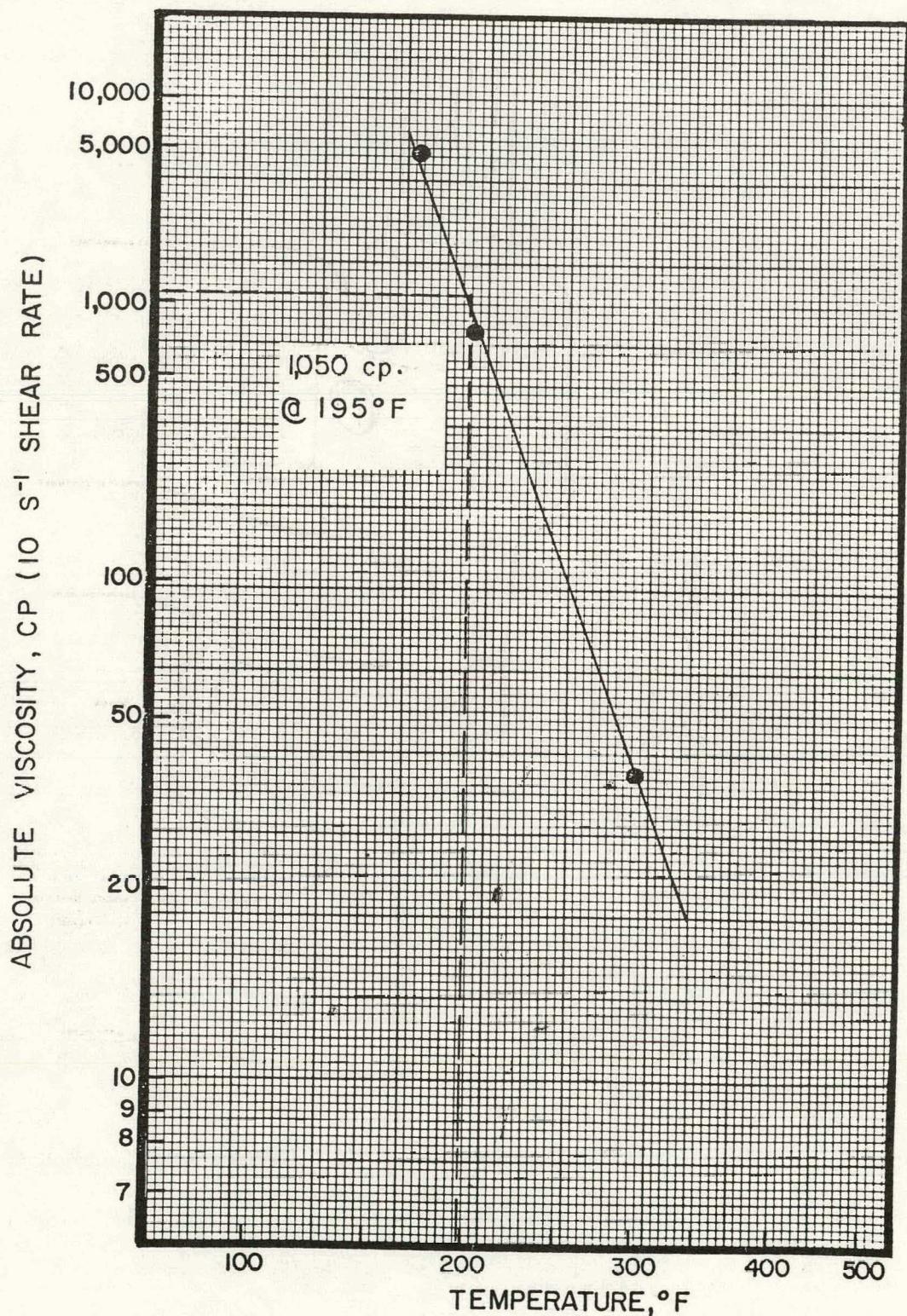


FIGURE 10. VISCOSITY VERSUS TEMPERATURE FOR THE 50/50 DEASHED SRC-SOLVENT BLEND FROM ICRC

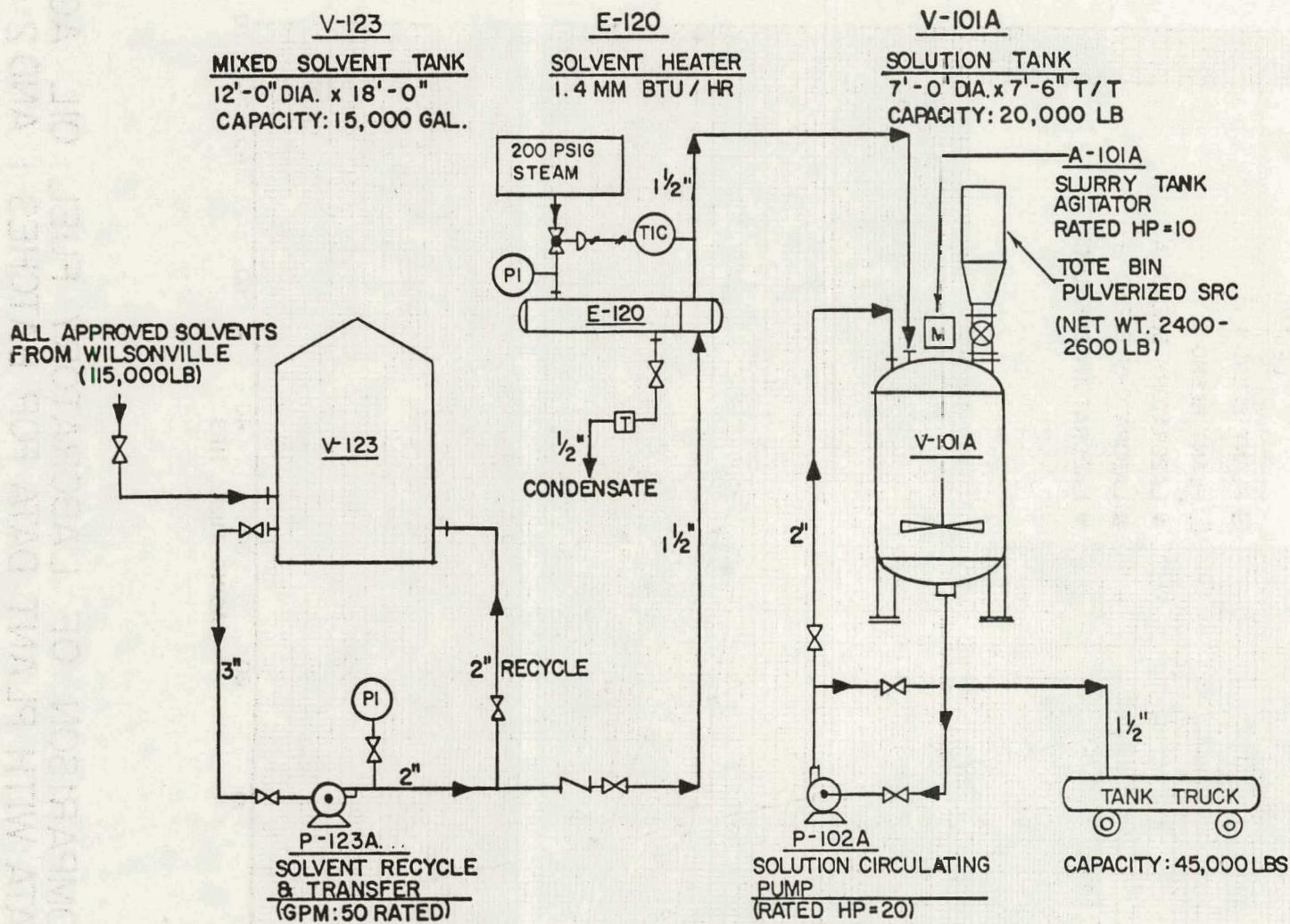


FIGURE 11. RESIDUAL OIL BLENDING SCHEMATIC FLOW DIAGRAM - WILSONVILLE
ONSITE FACILITIES

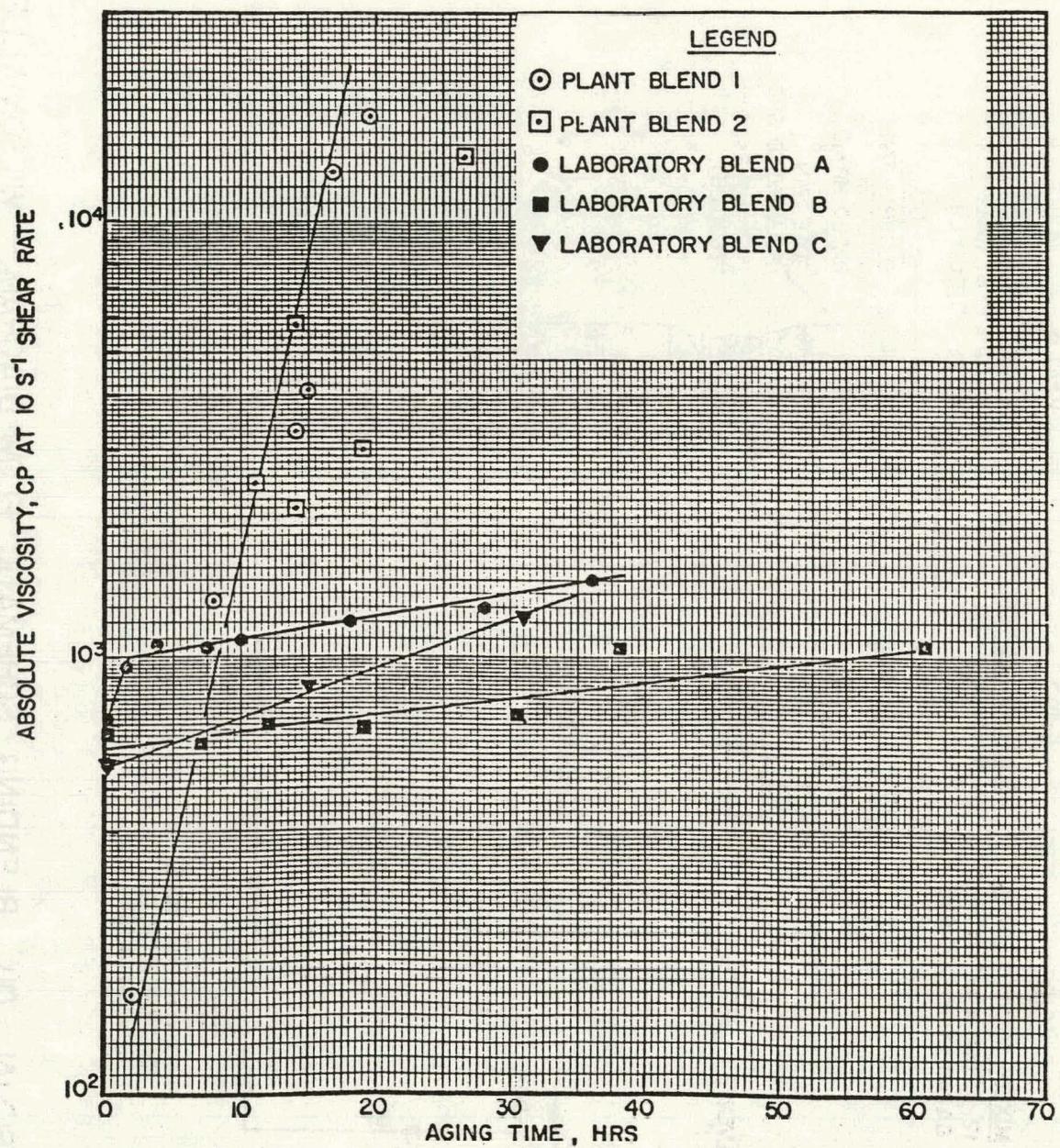


FIGURE 12. COMPARISON OF LABORATORY FUEL OIL AGING DATA WITH PLANT DATA FOR BATCHES 1 AND 2