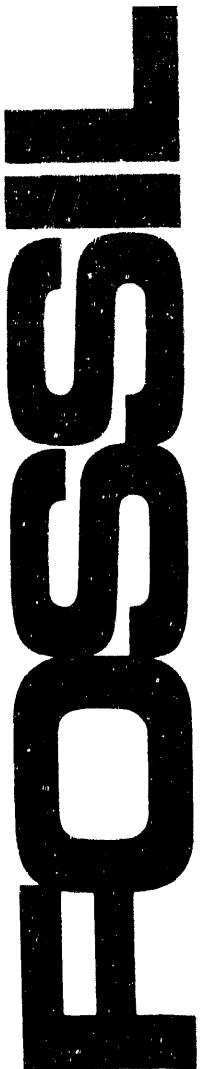


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THE EFFECT OF INTEGRATION CONFIGURATIONS ON TWO-STAGE COAL
LIQUEFACTION PROCESS YIELDS AND PERFORMANCE

Topical Report

May 1990
Date Published

Work Performed Under Contract No. AC22-82PC50041

For
U.S. Department of Energy
Pittsburgh Energy Technology Center
Pittsburgh, Pennsylvania

By
Catalytic, Inc.
Wilsonville, Alabama

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DOE/PC/50041-82
(DE91002478)
Distribution Category UC-108

DOE/PC/50041--82

DE91 002478

THE WILSONVILLE
ADVANCED COAL LIQUEFACTION
RESEARCH AND DEVELOPMENT FACILITY
Wilsonville, Alabama

TOPICAL REPORT

The Effect of Integration
Configurations on Two-Stage Coal
Liquefaction Process Yields and Performance

Published: May 1990

Prepared by
CATALYTIC, INC.
Wilsonville, Alabama 35186

Subcontractor to
SOUTHERN COMPANY SERVICES, INC.
Birmingham, Alabama 35202
Contract No. 43850

Prepared for
THE UNITED STATES DEPARTMENT OF ENERGY
Contract No. DE-AC22-82PC50041

and

ELECTRIC POWER RESEARCH INSTITUTE
Palo Alto, California 94303
Contract No. RP1234-1-2

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ABSTRACT

The investigation of various Two-Stage Liquefaction (TSL) process configurations was conducted at the Wilsonville Advanced Coal Liquefaction R&D Facility between July 1982 and September 1986. The primary objective of the TSL program has been to develop a commercially feasible method for conversion of coal to residual oils or distillate fuels.

The facility combines three process units. There are the liquefaction unit, either thermal (TLU) or catalytic, for the dissolution of coal, the Critical Solvent Deashing unit (CSD) for the separation of ash and undissolved coal, and a catalytic hydrogenation unit (HTR) for product upgrading and recycle process solvent replenishment. The various TSL process configurations were created by changing the process sequence of these three units and by recycling hydrotreated solvents between the units.

This topical report presents a description of the TSL configurations investigated and an analysis of the operating and performance data from the period of study. During this period, Illinois No. 6 Burning Star Mine coal and Wyodak Clovis Point Mine coal were processed. Cobalt-molybdenum and disposable iron-oxide catalysts were used to improve coal liquefaction reactions and nickel-molybdenum catalysts were used in the hydrotreater.

Both placing the CSD after the second stage (HTR) and ash recycle significantly improved process performance. The distillate yield improved to 70 wt % MAF coal. The organic rejection reduced to 9 wt %. Coal conversion improved to 95 wt %.

I. BACKGROUND AND INTRODUCTION

BACKGROUND

Operations at the Advanced Coal Liquefaction R & D Facility at Wilsonville, Alabama, are funded by the U. S. Department of Energy (DOE) and the Electric Power Research Institute (EPRI). Southern Company Services, Inc. (SCS) manages the Wilsonville program on behalf of DOE and EPRI. Catalytic, Inc. operates the facility under a subcontract with SCS.

The facility combines three process units: a liquefaction unit either thermal (TLU) or catalytic, a Critical Solvent Deashing (CSD) unit, and a catalytic hydrogenation (HTR) unit. The TLU was designed by Catalytic, Inc., using technology initially developed in Germany and later refined by the Pittsburg and Midway Coal Mining Co. Until November 1985, with the inception of close-coupled operations, this unit was used for coal dissolution. Since that time the TLU has been used for coal preparation, gas scrubbing and recompression and solvent distillation. The CSD unit was designed and developed by the Kerr-McGee Corporation. The HTR unit uses H-Oil® technology, developed by Hydrocarbon Research, Inc. (HRI), and was constructed by Catalytic, Inc. The HTR unit has been modified by Catalytic, Inc. to allow close-coupled operations. The modification primarily consisted of adding a new reactor in close proximity to the existing HTR reactor. These close-coupled reactors can be used for both the thermal-catalytic and the catalytic-catalytic modes of operation. The two reactors are commonly referred to as the 1st and 2nd stage reactors. The combined three-unit system is generally known as a Two-Stage Liquefaction (TSL) process.

The TSL process is an advanced coal liquefaction concept, where the severities in the 1st and 2nd stages may be independently varied, allowing for improvement in product slate flexibility as compared with single-stage liquefaction. Accordingly, a heavy fuel oil with low sulfur may be produced, or alternatively, emphasis may be placed on maximum production of low nitrogen distillate products with efficient hydrogen utilization.

INTRODUCTION

This report presents a process configuration analysis of TSL system process operating and performance data generated at Wilsonville between July 1982 (Run 241) and September 1986 (Run 251). During this period, five different process configurations were investigated by changing the process sequence of the three units (TLU, CSD and HTR) and by recycling hydrotreated process solvent between the units. Section II provides further details of each configuration listed below:

- NTSL (Non-Integrated Two-Stage Liquefaction)
- ITSL (Integrated Two-Stage Liquefaction)
- DITSL (Double-Integrated Two-Stage Liquefaction)
- RITSL (Reconfigured-Integrated Two-Stage Liquefaction)
- CC-ITSL (Close-Coupled Integrated Two-Stage Liquefaction)

Reports of several small-scale TSL process development studies can be found in the literature. Lummus and Cities Service studied the ITSL process (Ref. 1) and the RITSL process (Ref. 2). HRI and Chevron investigated the CC-ITSL process (Ref. 3).

The process performance of the TSL system and of each unit has been fully analyzed in the various Technical Progress Reports (Refs. 4-14). Recent development results have been published (Refs. 15-19).

Fifteen representative stable operation periods were selected for the analyses of process configuration comparison data. These are detailed in Section III Process Operating Details. In addition, twelve operating periods were studied for specific comparisons of certain configurations and process variables.

The following major process parameters were evaluated during these selected periods:

- Coal type
 - Illinois No. 6 Burning Star Mine
 - Wyodak Clovis Point Mine
- Operation mode
 - Thermal-catalytic
 - Catalytic-catalytic
- Solids recycle
- Reaction temperature (average)
 - 1st stage (790 to 835°F)
 - 2nd stage (620 to 780°F)
- Inlet hydrogen partial pressure
 - 1st stage (2100, 2600 psia)
 - 2nd stage (2600 psia)
- Coal space rate (low to high)

- Feed space velocity
 - 1st stage (2.7-4.2 lb feed/hr-lb cat)
 - 2nd stage (0.6-3.8 lb feed/hr-lb cat)
- Catalyst type and age
 - 1st stage
 - Amocat 1A (Co-Mo) 1/16" bimodal age, 2800 lb(resid+CI)/lb cat
 - Fe₂O₃ as a disposable catalyst
 - 2nd stage
 - ArmaK-Ketjen (Ni-Mo) 1/32" unimodal age, 390 lb (resid+CI)/lb cat
 - Shell 324 (Ni-Mo) 1/32" unimodal age, 1800 lb(resid+CI)/lb cat
 - Amocat 1C (Ni-Mo) 1/16" bimodal age, 2800 lb (resid+CI)/lb cat
- CSD deashing solvent (DAS) type
 - 2100-3100-4100

II. DESCRIPTION OF CONFIGURATIONS

Five different TSL process configurations were investigated between July 1982 and September 1986 by changing the sequence of the three process units and by recycling hydrotreated solvents between the units. This section provides details of each configuration. Figures 1-7 are block diagrams of the various configurations. Figures 8-10 are schematic flow diagrams of the TLU, HTR and CSD process units. Figure 11 is a schematic of the CC-ITSL system.

The ITSL configuration involves the recycle of the atmospheric flash bottoms stream from the HTR to the TLU, and/or recycle of hydrotreated resid and distillate solvent to the TLU. The NTSL configuration does not recycle any process streams from the HTR to the TLU. For ITSL and NTSL the process unit sequence is TLU, CSD and HTR.

The RITSL and CC-ITSL configurations employ a different process unit sequence, namely, 1st stage¹, 2nd stage and CSD. Accordingly, the hydrotreating catalyst is exposed to an ash, UC, and preasphaltene-rich environment in the RITSL and CC-ITSL configurations. RITSL was a step in the process development towards CC-ITSL. CC-ITSL has several process advantages, compared to NTSL and ITSL, such as (1) smaller CSD unit required, (2) reduction of fractionation equipment and (3) improved product quality.

In addition, CC-ITSL can employ a highly active catalyst in the 1st stage reactor, which leads to a high TSL C₄₊ distillate yield, high product quality and low catalyst requirement.

The key features of each configuration are described below:

(1) NTSL (Non-Integrated Two-Stage Liquefaction) (Figure 1)

- Process operating sequence is TLU, CSD and HTR.
- The TLU and HTR are decoupled, without recycle of HTR products back to the TLU.
- Deashed heavy thermal resid is fed to the HTR, along with hydrotreated distillate solvent. Deashed light thermal resid is recycled back to the TLU, along with thermal distillate solvent.

¹ In the CC-ITSL mode, the reactor associated with coal dissolution is referred to as the 1st stage rather than the TLU reactor. Similarly the HTR reactor is referred to as the 2nd stage.

- Process is suitable for the production of high quality residual utility fuels (No. 6 fuel oil equivalent).
- Armak-Ketjen (Ni-Mo) 1/32" unimodal catalyst was used for the batch deactivation study in the HTR.

(2) ITSL (Integrated Two-Stage Liquefaction) (Figure 2)

- Process operating sequence is TLU, CSD and HTR.
- The TLU and HTR are coupled, with recycle of the atmospheric flash bottoms from the HTR to the TLU.
- Deashed thermal resid and thermal distillate solvent are fed to the HTR.
- Process is suitable for the production of "all-distillate" product.
- Three different reactor types were tested in the TLU: Short Contact Time (SCT), Low Contact Time (LCT) and High Contact Time (HCT).
- Shell 324 (Ni-Mo) 1/32" unimodal catalyst was used for the batch deactivation study in the HTR and for the equilibrium catalyst activity run (commercial plant operation simulation by employing catalyst addition and withdrawal in the HTR).
- Iron oxide was used as a disposable catalyst in the TLU to improve coal conversion with Wyodak subbituminous coal and to promote resid conversion for several LCT runs with Illinois No. 6 bituminous coal.

(3) DITSL (Double-Integrated Two-Stage Liquefaction) (Figure 3)

- Process operating sequence is TLU, CSD and HTR.
- The TLU and HTR are coupled, with recycle of the atmospheric flash bottoms from the HTR to the TLU.
- Deashed light thermal resid and thermal distillate solvent are fed to the HTR. Deashed heavy thermal resid is recycled back to the TLU, along with the atmospheric flash bottoms from the HTR.
- Process is suitable for the production of "all-distillate" product.
- Potential advantages are that a smaller hydrotreater might be possible and a catalyst deactivation rate could be reduced because only the light thermal resid stream is hydrotreated.

- Shell 324 (Ni-Mo) 1/32" unimodal catalyst was used for the batch deactivation study in the HTR.
- Iron oxide was used as a disposable catalyst in the TLU to improve coal conversion with Wyodak subbituminous coal and to promote resid conversion with Illinois No. 6 bituminous coal (LCT runs).

(4) RITSL (Reconfigured-Integrated Two-Stage Liquefaction)
(Figure 4)

- Process operating sequence is TLU, HTR and CSD.
- The TLU and HTR are coupled, with recycle of deashed resid from the CSD and hydrotreated distillate solvent from the HTR in the operation mode without solids recycle. In the operation mode with solids recycle, the TLU and HTR are coupled, with recycle of the atmospheric flash bottoms from the HTR to the TLU.
- Products from the TLU are sent directly to the HTR without prior deashing in the CSD. Thermal distillate solvent and vacuum bottoms are fed directly to the HTR. HTR vacuum bottoms are sent to the CSD in the operation mode without solids recycle, and deashed resid is then recycled to the TLU along with hydrotreated distillate solvent. In the operation mode with solid recycle, HTR vacuum bottoms are used as an ash purge stream without CSD operation.
- This configuration is an intermediate step between the ITSL and CC-ITSL process developments. It does not completely simulate CC-ITSL because of interstage depressurizing between the TLU and the HTR.
- Process is suitable for the production of high quality "all-distillate" product.
- Program was designed to study the catalyst deactivation rate with non-deashed feed to the HTR and the CSD deashing performance with hydrotreated feed to the CSD.
- In the operation mode with solids recycle, an alternate deashing method was tested.
- Shell 324 (Ni-Mo) 1/32" unimodal catalyst was used for the batch deactivation study in the HTR.
- Iron oxide was used as a disposable catalyst in the TLU to improve coal conversion with Wyodak subbituminous coal.

(5) CC-ITSL (Close-Coupled Integrated Two-Stage Liquefaction)
(Figures 5, 6 and 11)

- Process operating sequence is 1st stage, 2nd stage and CSD.
- Two reactors (1st and 2nd stages) are directly coupled without any pressure letdown.
- 1st and 2nd stages are coupled, with recycle of deashed resid from the CSD and hydrotreated distillate solvent from the 2nd stage in the operation mode without solids recycle. In the operation mode with solids recycle, 1st and 2nd stages are coupled, with recycle of the atmospheric flash bottoms from the 2nd stage to the 1st stage, along with deashed resid from the CSD and hydrotreated distillate solvent from the 2nd stage, if necessary.
- Process is suitable for the production of high quality "all-distillate" product.
- Potential advantages are: a smaller CSD might be possible due to solids recycle and high resid conversion reactivity in the 2nd stage, requirements of fractionation equipment could be reduced increasing process thermal efficiency and product quality might be improved because of high resid conversion reactivity without cooling, depressurizing and deashing between reaction stages.
- Amocat 1A (Co-Mo) 1/16" bimodal catalyst was used for the batch deactivation study in the 1st stage in the catalytic-catalytic mode of operation.
- Shell 324 (Ni-Mo) 1/32" unimodal catalyst and Amocat 1C (Ni-Mo) 1/16" bimodal catalyst were used for the batch deactivation study in the 2nd stage.
- Iron oxide was used as a 1st stage disposable catalyst to improve coal conversion with Wyodak subbituminous coal.

III. PROCESS OPERATING DETAILS

Run objectives, operating details and the selection of representative stable operation periods for each configuration are discussed in this section.

Detailed run operating procedures and conditions were reported in Technical Progress Reports Run 241 to 251 (Refs. 4-14). Figure 7 presents TSL configurations tested at Wilsonville during Run 241 to Run 249, which are NTSL (Run 241), ITSL (Runs 242, 243, 244, 245, 246 and 248), DITSL (Runs 246 and 248), and RITSL (Runs 247 and 249).

The CC-ITSL configuration was tested at Wilsonville during Runs 250 and 251 with two different operation modes namely thermal (1st stage) - catalytic (2nd stage) and catalytic-catalytic.

(1) NTSL (Non-Integrated Two-Stage Liquefaction)

Run 241 (25 July - 11 November 1982) was a NTSL run processing Illinois No. 6 Burning Star Mine coal. The objective of this run was the operation of TLU, CSD and HTR to optimize fuel oil production with maximum thermal resid recovery at the CSD. Four overall special product workup periods were selected (241A-D) and process performance data were analyzed in detail. A good quality No. 6 fuel oil blend product was produced with 0.12 wt % sulfur and 0.84 wt % nitrogen content (Refs. 4, 20). Runs 241C and D performance data are selected for representative yield structures in the NTSL configuration and are compared to other configurations in Section IV. Process Performance Data Comparisons.

(2) ITSL (Integrated Two-Stage Liquefaction)

ITSL has been the most studied configuration at Wilsonville. Six runs from Run 242 to Run 248 (21 November 1982 - 5 May 1985) excluding RITSL Run 247 were studied to optimize process variables in the ITSL configuration. Main emphases were on reaction temperature, hydrogen partial pressure, reaction residence time and HTR catalyst age effects on process performance, to determine optimum process variables for a commercial plant design. Three different reactor types in the TLU were tested as shown below:

Reactor type	SCT	LCT	HCT
Run	<u>242</u>	<u>248</u>	<u>243, 244, 245</u>
Dimension	1 1/4" (Sch 160) x 606' L preheater coil tube	5.2" ID x 23' H new dissolver	12" ID x 23' H original dissolver

The objective of these runs was to optimize "all-distillate" product production. Illinois No. 6 Burning Star Mine coal was processed during Runs 242, 243, 244, 245 and 248, and Wyodak

Clovis Point Mine coal was processed during Run 246. C₄₊ distillate yields were high, 54-59 wt % MAF coal with Illinois No. 6 bituminous coal (Refs. 5, 6, 7, 8, 11), and 53 wt % MAF coal with Wyodak subbituminous coal (Ref. 9).

Run 245 was a proof-of-concept run to simulate a commercial plant operation by employing catalyst addition and withdrawal in the HTR. Overall performance was maintained during three months of operation with aged HTR catalyst by replacing it at a rate of 1 lb of fresh catalyst per ton of coal feed. Run 245 confirmed process performance with what was effectively an equilibrium catalyst. The run provided a good estimate of steady-state performance from typical Illinois No. 6 coal. C₄₊ distillate yields were generally greater than 58 wt % MAF coal. The majority of this product boils above naphtha, the range of interest for utility applications, and has a low-heteroatom and high hydrogen content compared to that of single stage catalytic processes. It appears that a major portion of the ITSL liquid would be particularly well suited for direct use by utilities without further upgrading (Ref. 8).

Iron oxide and DMDS were added in the coal slurry during Run 248 to improve coal conversion and coal hydrogenation reactions. Very high C₄₊ distillate was produced during Run 248D, that is, 64 wt % MAF coal. Iron oxide addition significantly improved TSL process performance with Illinois No. 6 bituminous coal (Ref. 11), as similarly observed during Run 246 with Wyodak subbituminous coal (Ref. 9).

Representative stable operation periods for the ITSL configuration are selected as follows, based on TSL process performance and are compared to other configurations in Section IV Process Performance Data Comparisons.

<u>Run</u>	<u>Operating Period</u>	<u>Period Selected for Configuration Comparison</u>	<u>Remarks</u>
<u>(Illinois No. 6 Burning Star)</u>			
242	A-D(4)	242BC	SCT(a)
243	A-M(13)	243JK/244B	HCT(a)
244	A-G(7)		
245	A-H(8)	245BCDF	HCT Cat A/W(b)
248	C,D,F,G(4)	248D	LCT(a) Fe ₂ O ₃ in slurry

(Wyodak Clovis Point)

246	F,G,H(3)	246G	HCT Fe ₂ O ₃ in slurry
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- (a) TLU reactor types: SCT for short contact time,
LCT for low contact time, and
HCT for high contact time.
- (b) Catalyst addition and withdrawal in the 2nd stage.

(3) DITSL (Double-Integrated Two-Stage Liquefaction)

DITSL was studied at Wilsonville during Run 248 (8 Feb - 23 March 1985) with Illinois No. 6 Burning Star Mine coal and during Run 246 (31 March - 9 June 1984) with Wyodak Clovis Point Mine coal. The LCT reactor was used in the TLU for Run 248 and the HCT reactor was used for Run 246. Iron oxide was added to the coal slurry to improve coal conversion and coal hydrogenation reactions. The objective of those runs was to produce "all-distillate" product in the DITSL configuration. C₄₊ distillate yield was high, 49-56 wt % MF coal, with Wyodak subbituminous coal during Run 246 (Ref. 9). With Illinois bituminous coal during Run 248, the C₄₊ distillate yield was low and the preasphaltene content was high in resid streams (Reference 11). Raising the hydrotreater reaction temperature could increase the C₄₊ distillate yield by converting the excess resid produced in the hydrotreater. However, the temperature increase in the hydrotreater would not lower the preasphaltene content in the CSD TR and prevent the preasphaltene buildup in the TLU. For DITSL to be a viable TSL process, the preasphaltenes must be converted in the TLU to produce distillate products.

Representative stable operation periods for the DITSL configuration are selected as follows based on TSL process performance and are compared to other configurations in Section IV Process Performance Data Comparisons.

<u>Run</u>	<u>Coal</u>	<u>Operating Period</u>	<u>Period Selected for Configuration Comparison</u>	<u>Remark</u>
248	Illinois No. 6	A(1)	248A	LCT Fe ₂ O ₃ in slurry
246	Wyodak	A-E(5)	246DE	HCT Fe ₂ O ₃ in slurry

(4) RITSL (Reconfigured-Integrated Two-Stage Liquefaction)

RITSL was a step in the process development leading to CC-ITSL. RITSL does not completely simulate CC-ITSL because of interstage depressurizing between the TLU and the HTR.

Run 247 with Illinois No. 6 Burning Star Mine coal (2 Sept 1984 - 6 Jan 1985) and Run 249 with Wyodak Clovis Point Mine coal (1 June - 18 Sept 1985) were tested at Wilsonville to study catalyst deactivation with non-deashed feed to the HTR and to study the CSD deashing performance with hydrotreated feed to the CSD. The objective of these runs was to simulate TSL process performance in the CC-ITSL configuration. The HCT reactor was used in the TLU for both runs and a forced-backmixing pump was installed in the TLU during Run 249 to improve dissolver mixing with Wyodak subbituminous coal. Iron oxide was added to the coal slurry during Run 249 to improve coal conversion and coal hydrogenation reactions. During part of Run 249 an alternate deashing method was tested. Solids were recycled and ash removed in the vacuum bottoms.

C₄₊ distillate yield was high, 55-62 wt % MAF coal, with Illinois bituminous coal (Ref. 10) and 53-57 wt % MAF coal with Wyodak subbituminous coal (Ref. 12).

Representative stable operation periods for the RITSL configuration are selected as follows based on TSL process performance and are compared to other configurations in Section IV Process Performance Data Comparisons.

<u>Run</u>	<u>Coal</u>	<u>Operating Period</u>	<u>Period Selected for Configuration Comparisons</u>	<u>Remark</u>
247	Illinois No. 6	C-II,D,G-I(3)	247D	HCT
249	Wyodak	D,E,F,H(4)	249E	HCT
			249H	Fe ₂ O ₃ in slurry Solids recycle

(5) CC-ITSL (Close-Coupled Integrated Two-Stage Liquefaction)

CC-ITSL is the most advanced TSL configuration in the currently available direct coal liquefaction technology. CC-ITSL has several process advantages compared to NTSL and ITSL, such as (1) smaller CSD unit, (2) reduction of fractionation equipment, (3) high process thermal efficiency, (4) improved product quality, and (5) synergistic process performance improvements in the catalytic-catalytic mode of operation.

Run 250 (13 Nov 1985 - 19 Mar 1986) in the thermal-catalytic mode of operation with and without solids recycle and Run 251 (27 April - 30 Sept 1986) in the thermal-catalytic and catalytic-catalytic modes of operation with solids recycle were studied to optimize process variables in the CC-ITSL configuration. Illinois No. 6 coal was processed during Runs 250 and 251-I and Wyodak coal was processed during Run 251-II. Process variable studies concentrated on the effects of reaction temperature, reaction residence time, catalyst type, catalyst age and solids recycle on TSL process performance, to determine optimum process variables for commercial plant design. A new close-coupled reactor unit was installed prior to Run 250. This new 1st stage reactor is identical to the existing 2nd stage reactor.

The objective of these runs was to maximize the production of high quality "all-distillate" product. Iron oxide was added in the coal slurry during Run 251-II to improve coal conversion and coal hydrogenation reactions. C₄₊ distillate was high, 54-65 wt % MAF coal with Illinois bituminous coal in the thermal-catalytic mode of operation (Run 250) (Ref. 13) and very high, 58-70 wt % MAF coal in the catalytic-catalytic mode of operation (Run 251-I) (Ref. 14). With Wyodak subbituminous coal C₄₊ distillate was high, 61 wt % MAF coal in the thermal-catalytic mode of operation (Run 251-II).

Representative stable operation periods for the CC-ITSL configuration are selected as listed below based on TSL process performance and are compared to other configurations in Section IV Process Performance Data Comparisons.

<u>Run</u>	<u>Coal</u>	<u>Operating Period</u>	<u>Period Selected for Configuration Comparisons</u>	<u>Remark</u>
250	Illinois No. 6	A-E(4)	250D	CC reactor thermal-catalytic
		F-H(3)	250G	CC reactor thermal-catalytic solids recycle
251-I	Illinois No. 6	A-G(7)	251-IE	CC reactor catalytic-catalytic solids recycle
251-II	Wyodak	A-D(4)	251-IIB	CC reactor thermal-catalytic solids recycle Fe_2O_3 in slurry

STABLE OPERATING PERIOD SUMMARY

A complete list of selected representative stable operating periods for each configuration is as follows. These periods will be compared and discussed in the following Section IV, Parts 1 thru 5.

<u>Configuration</u>	<u>Coal</u>	<u>Period</u>	<u>Remark</u>
NTSL	Illinois No. 6	241CD	HCT reactor
ITSL	Illinois No. 6 Wyodak	242BC	SCT reactor
		243JK/244B	HCT reactor
		245BCDF	HCT with Cat A/W
		248D 246G	LCT with Fe_2O_3 in slurry HCT with Fe_2O_3 in slurry
DITSL	Illinois No. 6 Wyodak	248A	LCT with Fe_2O_3 in slurry
		246DE	HCT with Fe_2O_3 in slurry
RITSL	Illinois No. 6 Wyodak	247D	HCT reactor
		249E	HCT with Fe_2O_3 in slurry
		249H	HCT with Fe_2O_3 in slurry
CC-ITSL	Illinois No. 6 Wyodak	250D	CC reactor/thermal-catalytic
		250G	CC/thermal-catalytic/solids recycle
		251-IE	CC/catalytic-catalytic/solids recycle
CC-ITSL	Wyodak	251-IIB	CC with Fe_2O_3 in slurry/thermal- catalytic/solids recycle

Process operating conditions for the above selected periods for configuration comparisons are summarized in the following tables. Note that the fifteen periods have been grouped by coal type. Ten of the fifteen periods are with Illinois No. 6 coal, and the others are with Wyodak coal. Detailed operating conditions for these periods can be found in Tables 3 and 4 (Appendix C).

Process Operating Conditions
(Coal: Illinois #6 Burning Star)

Configuration	NTSL 241CD	DITSL 248A	ITSL 242BC	ITSL 2431K/244B	ITSL 245BCDF	ITSL 248D	ITSL 247D	ITSL 250D	CC-ITSL 2505 (a)	CC-ITSL 251-1E(a)
Run no.										
Operation mode										
1st stage	thermal	thermal	thermal	thermal	thermal	thermal	thermal	thermal	catalytic	catalytic
2nd stage	catalytic	catalytic	catalytic	catalytic	catalytic	catalytic	catalytic	catalytic	catalytic	catalytic
Catalyst type										
1st stage	none(b)	none(b)	none	none	none	none	none	none	none	none
2nd stage	Armak-Ketjen	Shell 324	Shell 324	Shell 324	Shell 324	Shell 324	Shell 324	Shell 324	Amocat 1C	Amocat 1A
Coal feed rate, MF lb/hr	396	178	191	195	177	177	277	274	301	
Coal conc. in slurry, wt % MF	38.2	35.5	35.9	35.8	35.1	35.7	35.6	33.3	34.0	
Resid content, wt %										
1st stage process solvent	24 (g)	45-51	46	47	46	45	44	50	40	40
2nd stage feed	48	39	55	55	54	57	51	53	46	39
Reaction temperature, °F										
1st stage	805	832	860 (d)	810	834	810	824	829	808	
2nd stage	780	648	720	720	720	705	711	742	750	758
Inlet H ₂ part. press., psia										
1st stage	2150	2180	2150	2060	2130	2150	2500	2500	2470	
2nd stage	2240	2270	2600	2660	2590	2730	2690	2580	2540	2470
Coal space rate, lb/hr/ft ³ (>700°F)										
20	32	43 (e)	28	26	31	27	high	high	high	
Space velocity, lb feed/hr-lb cat										
1st stage	-	-	-	-	-	-	-	-	-	2.67
2nd stage	1.17	0.57	.99	1.0	0.75	0.69	0.90	2.08	2.23	(36.3) (h)
(lb MF coal/hr-cu ft cat.)	(60.2)	(21.2)	(25.1)	(25.7)	(21.5)	(20.9)	(19.5)	(34.2)	(33.8)	2.28
Catalyst age, lb (res+Cl)/lb cat										
1st stage	-	-	-	-	-	-	-	-	-	(37.2)
2nd stage	260-387	37-70	278-441	350-850	1300-1400 (f)	260-287	446-671	697-786	346-439	2159-2245
CSD										
DAS type	2100	2803-2853	2853	2803-2903	2803-2903	2653	2453	2303	2604	

(a) Ash recycle.
(b) Fe₂O₃ added in the coal slurry as a disposable catalyst.

(c) Reactor outlet temperature.
(d) Preheater outlet temperature.

(e) Reaction volume was 4.4 ft³ (preheater section = 2.1 ft³, transfer line = 2.3 ft³).
(f) Range of average catalyst age (catalyst addition rate = 1 lb cat/ton MF coal).

(g) Light thermal resid (LTR) from CSD (solvent content = 20 wt %).
(h) 1B MF coal/hr-cu ft catalyst.

Process Operating Conditions
(Coal: Wyodak Clovis Point)

Configuration Run no.	DITSL 246DE	ITSL 246G	RITSL 249E	RITSL 249H(a)	CC-ITSL 251-IIIB(a)
<u>Operation mode</u>					
1st stage	thermal	thermal	thermal	thermal	thermal
2nd stage	catalytic	catalytic	catalytic	catalytic	catalytic
<u>Catalyst type</u>					
1st stage	none(b) Shell 324	none(b) Shell 324	none(b) Shell 324	none(b) Shell 324	none(b) Amocat 1C
2nd stage					
<u>Coal feed rate, MF lb/hr</u>					
Coal conc. in slurry, wt % MF	249 35.1	244 34.4	251 40.9	251 33.2	353 33.0
<u>Resid content, wt %</u>					
1st stage process solvent	24	30	29	22	25
2nd stage feed	22	34	34	27	31
<u>Reaction temperature, °F</u>					
1st stage	825 624	813 623	796 700	802 700	819 743
2nd stage					
<u>Inlet H₂ part. press., psia</u>					
1st stage	2160 2810	2040 2780	2160 2580	2170 2610	2510 2550
2nd stage					
<u>Coal space velocity</u>					
Space velocity, lb feed/hr-lb cat					
1st stage	—	—	—	—	—
2nd stage	0.9 (1lb MF coal/hr-cu ft cat)	1.0 (27.4)	1.0 (26.9)	1.2 (31.5)	1.6 (43.6)
<u>Catalyst age, lb (res+Cl)/lb cat</u>					
1st stage	—	—	—	—	—
2nd stage	252-279	496	1208-1246	1683-1703	915-968
<u>CSD</u>					
DAS type	2453-2503	2353	3504-3704	2104-2154	2204

(a) Ash recycle.
(b) Fe₂O₃ added in the coal slurry as a disposable catalyst.

IV. PROCESS PERFORMANCE DATA COMPARISONS

This section presents a process configuration analysis of TSL system process operating and performance data generated between July 1982 (Run 241) and September 1986 (Run 251). The fifteen representative stable operating periods detailed in Section III are used for analysis in parts 1 thru 5 of this section. Twelve additional periods were studied for further detailed data analyses of specific comparisons for different configurations and process variables in parts 6 and 7 of this section.

TSL system process performance data for these periods are summarized in the following tables. Detailed performance data for these periods can be found in Tables 5 and 6 (Appendix C).

Process Performance Data
(Coal: Illinois #6 Burning Star)

Configuration Run no.	NTSL 241CD	DTSL 248A	NTSL 242BC	NTSL 244B 245BCDF	NTSL 248D	NTSL 247D	CC-NTSL 250D	CC-NTSL 250G (e)	CC-NTSL 251-1E (e)
<u>Operation mode</u>									
1st stage	thermal catalytic	thermal catalytic	thermal (SCT) catalytic	thermal catalytic	catalytic (cat A/W)	thermal (LCT) catalytic	thermal catalytic	thermal catalytic	catalytic
2nd stage	none	none (d)	none	none	none	none	none	none	catalytic
<u>Catalyst type</u>	Armak-Ketjen	Shell 324	Shell 324	Shell 324	Shell 324	Shell 324	Shell 324	Shell 324	Amocat 1A Amocat 1C
<u>Yield, wt % MAF coal</u>									
H ₂ consumption	4.2	4.7	4.9	5.2	5.6	5.6	6.1	6.4	6.8
C ₁ -C ₃ gas	7	7	4	6	8	6	7	8	7
C ₄ + distillate	40	45	54	59	61	64	62	64	63
C ₄ + resid	63	63	62	65	64	66	65	62	70
Organics rejected	20	22	25	20	21	18	21	22	15
<u>H₂ efficiency</u>									
1b C ₄ + dist./1b H ₂ cons.	9.5	9.7	11.0	11.3	10.9	11.3	10.2	10.5	9.8
<u>C₁-C₃ selectivity (X100)</u>									
to C ₄ + distillate	18	15	7	10	10	12	10	11	12
<u>Energy rejection, %</u>									
<u>Coal conversion, wt % MAF coal (a)</u>	20	24	23-27	20-23	20-24	19	22	23	16
1st stage	89	92	92	92	92	91	93	92	93
1st and 2nd stages	-	-	-	-	-	-	92	94	94
Two stage	88	88	87	90	89	90	90	91	93
<u>Resid:UC conversion, wt % feed(b)</u>									
1st stage (c)	27(36)	22(45)	21(40)	24(47)	24(51)	27(51)	21(40)	21(42)	34(67)
2nd stage	44(18)	30(19)	23(30)	22(28)	22(27)	22(26)	25(37)	24(38)	24(37)
									15(19)

Note: Runs 241 to 248 were with the CSD unit between reactors.

(a) Creosol solubles.

(b) Data in parentheses are based on wt % MAF coal.

(c) MAF coal at 100 wt % UC.

(d) Fe₂O₃ added in the coal slurry as a disposable catalyst.

(e) Ash recycle.

Process Performance Data
(Coal: Wyodak Clovis Point)

Configuration	RITSL 246DE	RITSL 246G	RITSL 249E	RITSL 249H(e)f	RITSL 251-11B(e)g
<u>Operation mode</u>					
1st stage	thermal	thermal	thermal	thermal	thermal
2nd stage	catalytic	catalytic	catalytic	catalytic	catalytic
<u>Catalyst type</u>					
1st stage	none(d)	none(d)	none(d)	none(d)	none(d)
2nd stage	Shell 324	Shell 324	Shell 324	Shell 324	Shell 324
<u>Yield, wt % MAF coal</u>					
H ₂ consumption	5.4	5.4	5.8	6.3	6.3
C ₁ -C ₃ gas	9	9	6	7	8
C ₄ + distillate	55	53	57	57	61
C ₄ + resid	54	54	53	62	65
Organics rejected	21	21	24	14	9
<u>H₂ efficiency</u>					
lb C ₄ + dist./lb H ₂ cons.	10.2	9.8	9.9	8.9	9.7
<u>C₁-C₃ selectivity (x100)</u>					
to C ₄ + distillate	15	18	10	12	13
<u>Energy rejection, %</u>					
Coal conversion, wt % MAF coal(a)	21-24	22-24	30	18	13
1st stage	89	90	89	94	94
1st & 2nd stage	-	-	90	95	95
Two stage	97	88	89	92	95
<u>Resid+UC conversion, wt % feed(b)</u>					
1st stage(c)	44(70)	39(64)	39(57)	38(64)	35(61)
2nd stage	24(9)	16(15)	25(23)	17(19)	23(27)

(a) Cresol solubles.
 (b) Data in parentheses are based on wt % MAF coal.
 (c) MAF coal as 100 wt % UC.
 (d) Fe₂O₃ added in the coal slurry as a disposable catalyst.
 (e) Ash recycle.
 (f) Projected yields including CSD performance data from a separate operation.
 (g) Performance data may not be representative for the set process conditions due to a high solid buildup in the reactor.

(1) TSL System Process Performance Comparisons

Theoretically, the optimum integration configuration for a commercial plant would have the following process performance characteristics.

- High C₄₊ distillate
- High hydrogen efficiency
- Low hydrogen consumption
- Low C₁-C₃ gas selectivity
- Low organic rejection
- Low catalyst requirements
- Good product quality

The interactions of these performance characteristics are highly complex. Advanced mathematical and statistical analyses are required to better understand them in TSL system performance. This type of study is not in the scope of this report. Mathematical process modeling will provide better insights in understanding process variable interactions (Ref. 22).

Illinois No. 6 Coal Comparisons

As stated earlier, ten of the fifteen stable operating periods under consideration were with Illinois No. 6 Burning Star Mine coal. These periods are compared in the tables of TSL system operating conditions and process performance data on pages 16 and 19. Figure 12 is a plot of TSL system process performance characteristics versus TSL C₄₊ distillate yields.

The following are main observations for configuration comparisons with Illinois No. 6 bituminous coal.

- CC-ITSL catalytic-catalytic mode of operation with solids recycle (251-IE) produced the highest C₄₊ distillate, 70 wt % MAF coal.
- ITSL (243JK/244B, 248D, 242BC and 245BCDF) had higher hydrogen efficiency (11-12 lb C₄₊ dist/lb H₂ consumed) than CC-ITSL, RITSL and NTSL. ITSL-LCT with iron oxide addition in the coal slurry (248D) produced the highest C₄₊ distillate, 64 wt % MAF coal, in the ITSL configuration.
- ITSL-SCT (242BC) had the lowest C₁-C₃ gas selectivity, 7% of C₄₊ dist, but had the highest organic rejection, 25 wt % MAF coal.

- Solids recycle reduced the organic rejection to 15 wt % MAF coal (250G and 251-IE).
- NTSL (241CD) showed the lowest hydrogen consumption, 4.2 wt % MAF coal, but produced the lowest C₄₊ distillate, 40 wt % MAF coal.

CC-ITSL with solids recycle (250G and 251-IE) had the highest two-stage coal conversion, 93 wt % MAF coal, while ITSL-SCT (242BC) and NTSL (241CD) showed the lowest two-stage coal conversion, 87-88 wt % MAF coal. This low two-stage coal conversion in ITSL-SCT and NTSL is probably due to a low severity operation in the TLU and consequently unstable CSD feed being repolymerized in CSD. In contrast, CC-ITSL with solids recycle had the least repolymerization in the CSD and high TSL hydrogen consumption. CSD feed is hydrotreated before deashing in the CC-ITSL configuration. Repolymerization in the CSD was less severe in the other TSL configurations (ITSL-LCT, ITSL-HCT, RITSL and CC-ITSL without solids recycle) than ITSL-SCT and NTSL. In Figure 13, as the TSL hydrogen consumption increases, the UC increase in the CSD (repolymerization) decreases.

With the CSD after the second stage, RITSL (247D) and CC-ITSL (250D) reduced the UC increase in the CSD (repolymerization) by approximately 1 wt % MAF coal, compared to ITSL (243JK/244B and 245BCDF) with the CSD before the second stage. The organic rejection was not significantly affected due to this improvement.

A maximum hydrogen efficiency (11.3 lb C₄₊ dist/lb H₂ cons.) was observed at around 60 wt % MAF coal C₄₊ distillate yield in the thermal-catalytic mode of operation and after that point, the hydrogen efficiency rapidly declines as the C₄₊ distillate yield increases (Figure 12). The addition of iron oxide in the coal slurry significantly reduces the decline of the hydrogen efficiency with an increase of 5 wt % MAF coal C₄₊ distillate yield. This indicates that iron oxide catalyst is selectively involved in the production of C₄₊ distillate with relatively small additional hydrogen consumption. Therefore, the hydrogen efficiency remains high with a higher C₄₊ distillate yield. In the catalytic-catalytic mode of operation the hydrogen efficiency declined a relatively small degree compared to the thermal-catalytic mode of operation with a significant increase of 11 wt % MAF coal C₄₊ distillate yield. This indicates that a synergism in process performance improvements can be achieved by employing catalysts in both stages. However, the hydrogen efficiency is lower than that in the thermal-catalytic mode of operation with iron oxide addition in the slurry. Further process studies are required to improve the hydrogen efficiency in the catalytic-catalytic mode of operation by elimination of over-hydrogenation.

A minimum C₁-C₃ gas selectivity to C₄₊ distillate (7% of C₄₊ dist) was observed at around 54 wt % MAF coal C₄₊ distillate yield in the thermal-catalytic mode of operation and after that point, the C₁-C₃ gas selectivity moderately increases as the C₄₊ distillate yield increases (Figure 12). The addition of iron oxide in the coal slurry does not lower the gas selectivity. In

the catalytic-catalytic mode of operation the C₁-C₃ gas selectivity increase relative to the C₄₊ distillate increase is much less than in the thermal-catalytic mode. This indicates that employing catalysts in both stages selectively reduces C₁-C₃ gas make with more C₄₊ distillate production.

A maximum organic rejection (25 wt % MAF coal) was observed at around 54 wt % MAF coal C₄₊ distillate in the thermal-catalytic mode of operation and after that point, the organic rejection significantly declines as the C₄₊ distillate yield increases (Figure 12) and as the TSL hydrogen consumption increases (page 19). Solids recycle also significantly reduces the organic rejection by 5-7 wt % MAF coal and at the same time increases the TSL hydrogen consumption. Higher TSL hydrogen consumption with solids recycle may be partly involved in lowering the organic rejection, along with less polymerization in the CSD with solids recycle (higher two-stage coal conversion) (Figure 13).

In summary, for TSL system process performance comparisons with Illinois No. 6 bituminous coal,

- CC-ITSL does not significantly improve TSL system process performance characteristics such as hydrogen efficiency, C₁-C₃ gas selectivity and organic rejection, compared to ITSL and RITSL.
- Solids recycle in the CC-ITSL thermal-catalytic mode of operation significantly affects TSL system process performance characteristics; reducing the hydrogen efficiency by 0.7 lb C₄₊ dist/lb H₂ and decreasing the organic rejection by 7 wt % MAF coal. The C₁-C₃ gas selectivity is not affected significantly.
- The catalytic-catalytic mode in CC-ITSL significantly affects TSL system process performance characteristics, compared to the thermal-catalytic mode; increasing the hydrogen efficiency and reducing the C₁-C₃ gas selectivity. The organic rejection is not affected significantly.

Wyodak Coal Comparisons

The five representative stable operation periods with Wyodak Clovis Point Mine coal are compared in the tables of TSL system operating conditions and process performance data on pages 17 and 20. Figure 14 is a plot of TSL system process performance characteristics versus TSL C₄₊ distillate yields.

The following are main observations for configuration comparisons with Wyodak subbituminous coal.

- CC-ITSL thermal-catalytic mode of operation with solids recycle (251-IIB) produced the highest C₄₊ distillate, 61 wt % MAF coal.

- All configurations except for RITSL with solids recycle had a similar hydrogen efficiency (10 lb C₄₊ dist./1b H₂ cons). Solids recycle in RITSL appears to lower the hydrogen efficiency significantly (249E vs 249H).
- RITSL without solids recycle (249E) and at a lower first stage reaction temperature had the lowest C₁-C₃ gas selectivity, 10% of C₄₊ dist. However, it had the highest organic rejection, 24 wt % MAF coal.
- Solids recycle reduced the organic rejection by 10 wt % MAF coal (249E vs 249H). CC-ITSL with solids recycle (251-IIB) had the lowest organic rejection, 9 wt % MAF coal.
- DITSL (246DE) and ITSL (246G) showed the lowest hydrogen consumption, 5.4 wt % MAF coal. However, they had the lowest C₄₊ distillate yield, 53-55 wt % MAF coal.

CC-ITSL with solids recycle (251-IIB) had the highest two-stage coal conversion, 95 wt % MAF coal without having a significant repolymerization in CSD (no increase of UC in CSD), while DITSL (246DE), ITSL (246G) and RITSL without solids recycle (249E) had low two-stage coal conversion, 87-89 wt % MAF coal. Although RITSL with solids recycle (249H) increases coal conversion by 3-5 wt % MAF coal, repolymerization in CSD is significant, reducing coal conversion by 3 wt % MAF coal (a 3 wt % UC increase in CSD).

Trends of TSL system process performance characteristics with Wyodak subbituminous coal (Figure 14) do not show clear maximum or minimum points. This may be due to insufficient data points obtained with this coal. However, maximum or minimum points appear to occur at around 55-60 wt % MAF coal C₄₊ distillate yield, as similarly observed with Illinois No. 6 bituminous coal (Figure 12).

In summary, for TSL system process performance comparisons with Wyodak subbituminous coal,

- Solids recycle in RITSL significantly affects TSL system process performance characteristics; reducing the hydrogen efficiency by 1.0 lb C₄₊ dist./1b H₂ cons. and decreasing the organic rejection by 12 wt % MAF coal. The C₁-C₃ gas selectivity is not affected significantly. A similar observation was made with Illinois No. 6 bituminous coal.
- CC-ITSL with solids recycle significantly affects TSL system process performance characteristics, compared to RITSL with solids recycle; increasing the hydrogen efficiency by 0.8 lb C₄₊ dist./1b H₂ cons. and decreasing the organic rejection by 5 wt % MAF coal. The C₁-C₃ gas selectivity is not affected significantly. With Illinois No. 6 coal, CC-ITSL configuration effects on TSL process performance characteristics

were not significant. It appears that the impact of the CC-ITSL configuration on performance differs with different ranks of coal, subbituminous and bituminous, probably due to different reactivities in coal and resid conversions and different selectivity in the distillate yield.

(2) TSL System Distillate Product Distribution

TSL system distillate product distribution data for the fifteen selected periods for configuration comparisons are summarized in the following tables:

Distillate Product Data
(Coal: Illinois #6 Burning Star)

Configuration Run no.	NTSL 241CD	DTDSL 248A	ITSL 242BC	ITSL 243JK/244B	ITSL 245BCDF	ITSL 248D	ITSL 247D	ITSL 250D	CC-ITSL 250G (a)	CC-ITSL 251-IE (a)
<u>Operation mode</u>										
1st stage	thermal catalytic	thermal catalytic	thermal (SCT) catalytic	thermal catalytic	thermal catalytic (cat A/W)	thermal (LCT) catalytic	thermal catalytic	thermal catalytic	thermal catalytic	catalytic
2nd stage	none	none(b)	none	none	none	none(b)	none	none	none	catalytic
<u>Catalyst type</u>										
1st stage	Armak-Ketjen	Shell 324	Shell 324	Shell 324	Shell 324	Shell 324	Shell 324	Shell 324	Amocat 1C	Amocat 1A
2nd stage									Amocat 1C	Amocat 1C
<u>Yield, wt % MAF coal</u>										
NTSL										
C ₄ + naphtha	10	10	12	11	14	11	13	15	19	
Middle distillate	4	7	7	8	9	8	7	8	8	
Distillate solvent	26	28	40	42	41	43	44	40	43	
Sum	40	45	54	61	64	62	64	63	70	
<u>1st stage</u>										
C ₄ + naphtha	6	8	5	6	9	6	5	6	12	
Middle distillate	2	6	5	5	7	5	5	4	6	
Distillate solvent	14	23	18	24	21	16	21	23	39	
Sum	23	37	28	35	37	27	31	33	57	
<u>2nd stage + CSD</u>										
C ₄ + naphtha	4	2	5	5	5	5	8	9	7	
Middle distillate	1	1	2	3	2	3	2	4	2	
Distillate solvent	12	5	19	16	27	20	23	17	4	
Sum	17	8	26	24	35	27	33	30	13(c)	
<u>C₄+ distillate reaction</u>										
Yield contribution, %										
1st stage	58	82	52	59	43	58	44	48	52	
2nd stage + CSD	42	18	48	41	57	42	56	52	48	
										81
										19(c)

(a) Ash recycle.
(b) Fe₂O₃ added in the coal slurry as a disposable catalyst.
(c) High catalyst ages employed in Run 251-IE reduced the second stage contribution. Refer to the table on page 43.

Distillate Product Data
(Coal: Wyodak Clovis Point)

Configuration	DITSL <u>246UE</u>	ITS _L <u>246G</u>	RTITS _L <u>249E</u>	RTITS _L <u>249H(a)</u>	CC-ITS _L <u>251-LIB(a)</u>
<u>Operation mode</u>					
1st stage	thermal	thermal	thermal	thermal	thermal
2nd stage	catalytic	catalytic	catalytic	catalytic	catalytic
<u>Catalyst type</u>					
1st stage	none(b)	none(b)	none(b)	none(b)	none(b)
2nd stage	Shell 324	Shell 324	Shell 324	Shell 324	Amocat 1C
<u>Yield, wt % MAF coal</u>					
<u>TSL</u>					
C ₄ + naphtha	15	16	16	15	20
Middle distillate	13	12	9	9	10
Distillate solvent	27	25	32	33	31
Sum	55	53	57	57	61
<u>1st stage</u>					
C ₄ + naphtha	12	12	10	10	9
Middle distillate	10	11	7	7	9
Distillate solvent	24	15	19	25	23
Sum	46	38	36	42	41
<u>2nd stage + CSD</u>					
C ₄ + naphtha	3	4	6	5	11
Middle distillate	3	1	2	2	1
Distillate solvent	3	10	13	8	8
Sum	9	15	21	15	20(c)
<u>C₄ distillate reaction</u>					
<u>Yield contribution,</u>					
1st stage	84	72	63	74	67
2nd stage + CSD	16	28	37	26	33(c)

(a) Ash recycle.

(b) Fe₂O₃ and DMDS added in the coal slurry as a disposable catalyst.

(c) A high coal space velocity and a high second stage reaction temperature were employed in Run 251-LIB.
Refer to the table on page 44.

Illinois No. 6 Coal Comparison

TSL system distillate product distribution data with Illinois No. 6 coal are plotted in Figure 15.

ITSL (242BC, 243JK/244B, 245BCDF), RITSL (247D) and CC-ITSL (250D) in the thermal-catalytic mode of operation all showed a similar distillate product distribution. It appears that an increase of TSL hydrogen consumption in the range of 4.9 to 6.1 wt % MAF coal does not affect the product distribution. However the total C₄₊ distillate yield increased significantly (see the table on page 27). The distillate product is composed of 19 wt % C₄₊ naphtha, 13 wt % middle distillate and 68 wt % distillate solvent. The iron oxide addition in the coal slurry (248D) increases C₄₊ naphtha by 3 wt % and middle distillate by 1 wt %, and decreases distillate solvent by 4 wt %. This indicates that iron oxide enhances hydrocracking of distillate products by its catalytic action. The iron oxide addition also increases the C₄₊ distillate yield. Solids recycle in CC-ITSL (250G) significantly affects the product distribution; increasing C₄₊ naphtha by 4 wt % and middle distillate by 2 wt %, and decreasing distillate solvent by 6 wt %. Solids recycle improves catalytic hydrocracking of distillate products, but does not increase the C₄₊ distillate yield.

CC-ITSL catalytic-catalytic mode of operation with solids recycle (251-IE) increases C₄₊ naphtha by 3 wt %, and decreases middle distillate by 2 wt % and distillate solvent by 1 wt %, compared to the thermal-catalytic mode of operation.

In summary, for TSL system distillate product distribution with Illinois No. 6 bituminous coal,

- Process configurations do not affect the product distribution, although they significantly affect the TSL C₄₊ distillate yield and TSL hydrogen consumption. This is true for ITSL, RITSL and CC-ITSL. NTSL and DITSL showed a 3-6 wt % higher C₄₊ naphtha composition in distillate products than other configurations. Since NTSL and DITSL produced much smaller C₄₊ distillate yields, the actual C₄₊ naphtha yield in NTSL and DITSL was slightly lower.
- Iron oxide addition and solids recycle in the thermal-catalytic mode and the use of an active catalyst in the 1st stage in the catalytic-catalytic mode significantly affect the TSL system distillate product distribution. They increase the C₄₊ naphtha composition by 3-4 wt % in distillate products by enhancing catalytic hydrocracking of distillate products. The increase of the actual C₄₊ naphtha yield was 2-4 wt % MAF coal.

Wyodak Coal Comparison

TSL system distillate product distribution data with Wyodak Clovis Point Mine coal are plotted in Figure 16.

DITSL (246DE) and ITSL (246G) produced more middle distillate by 3-4 wt % MAF coal, compared to RITSL (249E, 249H). The middle distillate composition is higher by 7-8 wt % and the distillate solvent is lower by 9 wt %. The C₄₊ naphtha composition is similar. Solids recycle in RITSL (249H) does not significantly affect the distillate product distribution, compared to RITSL without solids recycle (249E). CC-ITSL with solids recycle (251-IIB) in the thermal-catalytic mode of operation increases C₄₊ naphtha by 7 wt % and decreases distillate solvent by 7 wt % compared to RITSL with solids recycle (249H). The actual increase of the C₄₊ naphtha yield is 5 wt % MAF coal.

In summary, for TSL system distillate product distribution with Wyodak subbituminous coal,

- Process configurations are very important. DITSL and ITSL are in favor of the middle distillate production and CC-ITSL is in favor of the C₄₊ naphtha production. These process configuration effects were not seen with Illinois No. 6 bituminous coal.
- Solids recycle does not affect the distillate product distribution. This is a different observation than that for Illinois No. 6 bituminous coal, where solids recycle increased the C₄₊ naphtha composition in distillate products.

(3) Process Solvent Quality and Hydrogen Transfer Data

Process solvent quality and hydrogen transfer data for the fifteen selected periods are summarized in the following tables:

Solvent Quality Data
(Coal: Illinois #6 Burning Star)

Configuration	Run no.	DITSL 241CD	ITSL 248A	ITSL 242BC	ITSL 243JK/244B	ITSL 245BCDF	ITSL 248D	ITSL 247D	RITSL 250D	CC-ITSL 250G (a)	CC-ITSL 251-LE(a)
<u>Operation mode</u>											
1st stage		thermal	thermal	thermal (SCR)	thermal	thermal	thermal (LCT)	thermal	thermal	thermal	catalytic
2nd stage		catalytic	catalytic	catalytic	catalytic	catalytic	catalytic	catalytic	catalytic	catalytic	catalytic
<u>Catalyst type</u>											
1st stage	none	none(b)	none	none	none	none	none(b)	none	none	none	none
2nd stage	Arnak-Ketjen	Shell 324	Shell 324	Shell 324	Shell 324	Shell 324	Shell 324	Shell 324	Amocat 1C	Amocat 1C	Amocat 1A
<u>Hydrogen content, wt %</u>											
Process solvent (as-is)	7.90	8.46	8.81	9.02	8.84	9.00	9.16	8.61	8.22	7.74	
Resid	6.29(f)	6.63	7.41	7.44	7.14	7.49	7.49	7.38	7.24	7.08	
Distillate	8.24	10.50	10.11	10.66	10.23	10.18	10.54	9.99	10.11	9.93	
<u>Solvent quality, % (c)</u>											
	58	83	87	90	88	89	87	87	87	87	84(e)
<u>Hydrogen transfer</u>											
wt % hydrogen											
(feed-product) 1st stage											
Resid	-(g)	0.4	0.7	1.2	0.8	1.1	1.1	1.0	0.5	0.2	
Distillate	-(g)	0.8	0.5	0.8	0.7	0.8	0.9	0.9	0.7	0.3	
Total	-(g)	1.2	1.2	2.0	1.5	1.9	2.0	1.9	1.2	0.5	
<u>H₂ consumption (gaseous)</u>											
wt % MAP coal											
TSL	4.2	4.7	4.9	5.2	5.6	5.6	6.1	6.1	6.4	6.8	
1st stage	2.6	1.6	0.7	1.3	1.7	1.8	1.7	1.7	1.8	4.5	
2nd stage	1.6	3.1	4.2	3.9	3.9	3.8	4.4	4.4	4.6	2.3	
<u>Exotherm, °F</u>											
1st stage	72	11	-	5	17	0	-5	15	40	1.03(d)	
2nd stage (d)	-	0.27	-	0.44	-	0.50	0.76	1.12	1.00	0.44	

(a) Ash recycle.
(b) Fe₂O₃ added in the coal slurry as a disposable catalyst.
(c) By the equilibrium test.
(d) Relative to 250G 2nd stage exotherm.
(e) Tests were done with Cl-free samples.
(f) Light thermal resid (LTR) from CSD (solvent content = 20 wt %).
(g) Probably insignificant compared to other configurations.

Solvent Quality Data
(Coal: Wyodak Clovis Point)

Configuration Run no.	DITSL 246DE	ITSL 246G	RITSL 249E	RITSL 249H(a)	CC-ITS _L 251-1B(a)
<u>Operation mode</u>					
1st stage	thermal	thermal	thermal	thermal	thermal
2nd stage	catalytic	catalytic	catalytic	catalytic	catalytic
<u>Catalyst type</u>					
1st stage	none(b)	none(b)	none(b)	none(b)	none(b)
2nd stage	Shell 324	Shell 324	Shell 324	Shell 324	Amocat 1C
<u>Hydrogen content, wt %</u>					
<u>Process solvent (as-is)</u>					
Resid	9.18	9.02	9.54	7.98	7.55
Distillate	6.78	6.94	7.66	7.59	7.29
Solvent quality, % (c)	10.23	9.62	10.31	10.84	10.51
	87	88	88	79	81
<u>Hydrogen transfer</u>					
wt % hydrogen					
(Feed-Product) 1st stage					
Resid	0.5	0.8	1.2	1.0	0.8
Distillate	0.8	0.6	0.7	0.7	0.9
Total	1.3	1.4	1.9	1.7	1.7
<u>H₂ consumption</u>					
wt % MAP coal					
ITSL	5.4	5.4	5.8	6.3	6.3
1st stage	2.7	2.7	2.2	3.3	2.8
2nd stage	2.7	2.7	3.6	3.0	3.5
<u>Knotholes, %</u>					
1st stage	99	101	45	109	83
2nd stage(d)	0.52	0.40	0.78	0.47	1.00

(a) Ash recycle.

(b) Fe₂O₃ added in the coal slurry as a disposable catalyst.

(c) by the equilibrium test.

(d) Relative to 251-1B 2nd stage exotherm.

Illinois No. 6 Coal Comparison

Figure 17 is a plot of process solvent quality versus hydrogen content with Illinois No. 6 coal.

Process solvent quality increases as hydrogen contents of process solvent, resid and distillate portions of process solvent increase. Solvent quality gives a relative indication of the concentration of donatable hydrogen in the process solvent (Appendix B). NTSL (241CD) solvent quality was the lowest, 58%, while the other ITSL configurations had high solvent quality, 83-90%. The amount of donatable hydrogen in the NTSL process solvent is very small because the NTSL solvent is not hydro-treated. Because of this, NTSL required high hydrogen consumption in the 1st stage to accomplish sufficient coal liquefaction reactions and consequently, NTSL had a very high reactor exotherm, 72°F, in the 1st stage.

DITSL (248A) solvent quality was relatively low, 83%, compared to other ITSL configurations in the thermal-catalytic mode of operation (87-90%). This low solvent quality in DITSL is attributed to the low hydrogen content of the resid portion of process solvent due to non-hydrotreated heavy thermal resid being recycled from the CSD to the 1st stage.

ITSL-SCT (242BC) hydrogen transfer value was relatively low, 1.2, although solvent quality was high, 87%. This low hydrogen transfer value in ITSL-SCT appears due to the short contact time using the preheater in the 1st stage.

High solvent quality, 87-90%, was observed in the ITSL (243JK/244B, 248D), RITSL (247D) and CC-ITSL (250D) thermal-catalytic mode of operation without solids recycle. Hydrogen transfer values for these configurations were high, 2.0, and 1st stage reactor exotherms were low in the range of -5 to 15°F. (The negative exotherm means that the heat loss around the reactor is higher than the heat generated by reactions.)

Solids recycle (250G) in the CC-ITSL configuration significantly reduced the hydrogen transfer value from 1.9 to 1.2. Since the hydrogen consumption in the 1st stage was not changed due to the solids recycle, the reactor exotherm in the 1st stage increased from 15 to 40°F, because of the relative reduction of endothermic reactions with solids recycle. The significant decrease of hydrogen efficiency with solids recycle (from 10.5 to 9.8 lb C₄⁺ dist./lb H₂ cons.) appears due to this low hydrogen transfer in the 1st stage.

CC-ITSL in the catalytic-catalytic mode of operation (251-IE) showed a relatively low solvent quality, 84%, because of decreases in hydrogen contents of resid and distillate portions of process solvent, as illustrated in Figure 17. High hydrogenation reactions in the 1st stage resulted in a very high reactor exotherm in the 1st stage. A low hydrogen transfer value

(0.5) is misleading, for the hydrogen transfer in the catalytic-catalytic mode of operation indicates net dehydrogenation of recycle process solvent in the 1st stage as a result of combination of thermal dehydrogenation (hydrogen transfer from recycle process solvent to coal) and catalytic rehydrogenation with gaseous hydrogen. Detailed discussion was reported in Technical Progress Report Run 251 (Ref. 14).

Figure 18 is a plot for process solvent hydrogen transfer data. High hydrogen transfer improves TSL hydrogen efficiency. High hydrogen transfer decreases the reactor exotherm in the 1st stage. It appears that hydrogen transfer is not strongly dependent on process configurations (DITSL, ITSL-HCT, ITSL-LCT, CC-ITSL), but more dependent on process variables such as residence time, solids recycle and hydrotreating of recycle process solvent.

Wyodak Coal Comparison

Process solvent quality and hydrogen transfer data with Wyodak Clovis Point Mine coal are plotted in Figures 19 and 20. The available data points are not sufficient to make meaningful observations for generalization.

Solids recycle (249H) in the RITSL configuration significantly reduced solvent quality from 88 to 79%. This decrease in solvent quality is probably due to overhydrogenation of the distillate portion of recycle process solvent. The hydrogen content of distillate increased from 10.31 to 10.84 wt %. The hydrogen transfer value slightly decreased from 1.9 to 1.7 and the 1st stage hydrogen consumption significantly increased from 2.2 to 3.3 wt % MAF coal, resulting in a higher reactor exotherm (109°F) than without solids recycle (45°F). A significant decrease in hydrogen efficiency (from 9.9 to 8.9 lb C₄₊ dist./lb H₂ cons.) was observed with Wyodak coal, as with Illinois No. 6 coal, in the CC-ITSL configuration.

As a result of analyses of process solvent quality and hydrogen transfer data, the following observations can be made:

- Solvent quality is a function of hydrogen contents of resid and distillate portions of recycle process solvent.
- Optimum hydrogen content of process solvent for high solvent quality may be different with different coals.
- High hydrogen transfer of process solvent to coal in the 1st stage improves TSL hydrogen efficiency.
- Solids recycle decreases hydrogen transfer and hydrogen efficiency, but reduces organic rejection.

- Hydrogen transfer is not strongly dependent on process configurations, but more dependent on process variables such as residence time, solids recycle and hydro-treating of recycle process solvent.
- There is a good correlation between hydrogen transfer, 1st stage reactor exotherm and TSL hydrogen efficiency.

(4) Unit Contribution Effect on TSL Hydrogen Efficiency

Interstage dependency on overall TSL process performance is illustrated in Figures 21 and 22, by comparing the C₄₊ distillate unit contribution effect on TSL hydrogen efficiency in ITSL configurations. NTSL (241CD) was not included for comparisons because NTSL is a non-integrated configuration.

With Illinois No. 6 Burning Star Mine coal (Figure 21) the maximum hydrogen efficiency can be achieved at around 60% 1st stage contribution in TSL C₄₊ distillate products. Experimental data points are not sufficient to locate the optimum 1st stage contribution for the maximum hydrogen efficiency. The CC-ITSL catalytic-catalytic mode of operation (2¹l-IE) had a low hydrogen efficiency with a high 1st stage contribution (81%), as similarly observed in DITSL (248A).

With Wyodak Clovis Point Mine coal (Figure 22) it appears that the 1st stage unit contribution does not significantly affect TSL hydrogen efficiency.

(5) Product Quality

TSL system distillate products produced during Runs 241 to 251 were analyzed to study process configuration effects on distillate product quality. The distillate products from the TSL process were characterized in two ways:

- (1) Each distillate product stream was analyzed individually.
- (2) A blend of distillate products was prepared, fractionated and analyzed by boiling point range.

Distillate products were analyzed by gas chromatography to determine boiling range fractions and by an elemental analyzer to determine elemental composition such as carbon, hydrogen, nitrogen, sulfur and oxygen (by difference). GC boiling range fractions are defined as: naphtha (IBP-350°F), middle distillate (350-450°F), and distillate solvent (450°F-EP).

Distillate product analytical data and product quality data for each stream for the periods selected for analyses of configuration comparisons are tabulated in Tables 9 to 12. Distillable product blend data for periods, 244CD, 242BC, 245BCDF, and 249E are not available. Detailed product quality data for each configuration were reported in Technical Progress Report Runs 241 to 251 (Refs. 4-14).

The primary product of the NTSL configuration (241CD) with Illinois No. 6 Burning Star Mine coal was a low-sulfur coal-derived substitute for No. 6 fuel oil. This substitute was prepared by blending hydrotreated resid with distillate solvents from the 1st and 2nd stages in varying proportions to meet a given viscosity specification. Improvements in the properties of the residue achieved by hydrotreating, combined with the fact that more distillates can be produced in a two-stage system, make the blending of distillates and residue technically feasible. A good quality No. 6 fuel oil blend product was produced with 0.12 wt % sulfur and 0.84 wt % nitrogen contents (Refs. 4 and 20). Properties of No. 6 fuel oil are compared below:

Coal Derived
Fuel Oil No. 6
 (56% solv + 44% HTR resid)

Petroleum Derived
Fuel Oil No. 6

Elemental analysis (wt %)

Carbon	87.3	86.5-90.2
Hydrogen	8.7	9.5-12.0
Nitrogen	0.8	-
Sulfur	0.1	0.7-3.5
Ash	0.1	0.01-0.5
Oxygen (by diff.)	3.0	-

Viscosity (CS)

At 122°F (storage)	360	92-638
At 230°F (Atomizing)	20	-

Viscosity increase on aging

At 122°F for 6 weeks	NEGL	NEGL
At 230°F for 1 hr	NEGL	NEGL

Gravity (°API at 60°F)	1	7-22
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Results of aging tests indicate that the substitute No. 6 fuel oil produced by the NTSL configuration is relatively stable and can be stored and handled in a manner similar to petroleum derived residual oils.

Light distillate from the hydrotreater obtained during Run 244 in the ITSL configuration with Illinois No. 6 coal was submitted to Amoco Oil Company and compared as-is with aviation gasoline specifications. The distillation test results and elemental analysis are shown below. The light distillate was found to pass the freeze point test, Reid vapor pressure test, and copper strip corrosion test but it narrowly failed the octane number and potential gum specifications.

Aviation Gasoline Specifications

Sample Source: Wilsonville Run 244
(Cat. Age 1600 lbs Resid/lb Cat, HTR Temp 730°F)

Test	Result		Spec.	Comment	Test	Comment
	(vol % - °F)					
D-86	10% - 198		Max 167	Fail	Reid vapor pressure	Pass
	40% - 208		Min 167	Pass	Copper strip corrosion	Pass
	50% - 212		Max 221	Pass	Freezing point	Pass
	90% - 242		Max 275	Pass	Octane no.	Fail
	FBP-287		Max 338	Pass	Potential gum	Fail
	Dist. residue-0.8		Max 1.5			
Elemental analysis	%C = 86.85					
	%H = 13.15					
	%S = 27 ppm		Max 0.05% Pass			
	%N = 58 ppm					

Distillable product blend analyses for ITSL Run 244 and RITSL Run 247D with Illinois No. 6 coal are compared in the following table:

Distillation cut	Wt % of sample	Elemental (wt %)					API gravity (60/60)
		C	H	N	S	O(diff)	

Run 244 ITSL configuration (a)

Naphtha (IBP-360°F)	18.4	85.21	12.86	846 ppm	0.36	1.50	43.0
Distillate (360°F-650°F)	45.7	86.44	10.74	0.24	0.22	2.35	18.4
Gas Oil (650°F+)	35.9	89.07	9.69	0.31	0.16	0.76	7.4

Run 247D RITSL configuration (b)

Naphtha (IBP-350°F)	15.9	85.15	13.90	800 ppm	0.29	0.58	46.6
Distillate (350°F-650°F)	52.3	87.09	11.52	0.21	0.13	1.05	20.1
Gas oil (650°F+)	31.8	89.37	10.24	0.25	0.10	0.49	9.3

(a) Catalyst age 1600 lbs resid/lb cat, HR temp 730°F.

(b) Catalyst age 446-671 (1bs resid+UC+ash)/lb cat, HTR temp 710°F.

The hydrogen contents of comparable fractions is higher, by 0.8-1.2 wt %, for RITSL. The H/C atomic ratios are higher by 0.10-0.16. In general, the RITSL product had lower levels of nitrogen and sulfur. These results appear to be a natural consequence of having an increased content of hydrotreated product. Younger catalyst ages, a lower HTR reaction temperature, an increased feed reactivity by placing the CSD after the HTR unit and further hydrotreating of a portion of the distillate product in the first stage in 247D could also have affected the product quality in 247D (refer to the operating conditions in the

table on page 16). The hydrogen efficiency decreased as the hydrogen consumption increased, as shown in the table on page 19. Therefore, the higher hydrogen consumption seems to be a trade off with improved product quality.

Properties of a fractionated distillable product blend from period 248D processing Illinois No. 6 coal (ITSL-LCT with iron oxide) are shown below. As can be seen, the product boiling below 650°F is 67% of the total blend, which is comparable to that obtained in Run 244 without iron oxide. The naphtha from 248D had higher hydrogen content (13.76 vs 12.86 wt %) but the nitrogen and sulfur were higher. The analyses for the distillate and gas oil fractions were similar, with 248D showing slightly higher nitrogen and sulfur contents. The API gravities showed that the naphtha and distillate for 248D were heavier than those from Run 244. Younger catalyst ages, a lower HTR reaction temperature, a lower space velocity in 248D could have affected the product quality (refer to the operating conditions in the table on page 16). The hydrogen efficiency did not change as the hydrogen consumption increased, as shown in the table on page 19. This indicates that the product quality of the total distillate blend is similar for both runs, although the distillate product selectivity is different in the table on page 27 and in the figure on page 143.

Distillation cut	Wt % of sample	elemental (wt %)					API gravity (60/60)
		C	H	N	S	O(diff)	
<u>Run 248D ITSL configuration (a)</u>							
Naphtha (IBP-350°F)	18.6	84.63	13.76	0.06	0.65	0.90	42.4
Distillate (350°F-650°F)	48.1	86.07	10.95	0.31	0.38	2.29	13.0
Gas oil (650°F+)	33.3	89.11	9.41	0.46	0.18	0.84	0.3

(a) Catalyst age 300 lbs resid/lb cat, HTR temp 700°F.

Distillable product blend analyses for the CC-ITSL configuration processing Illinois No. 6 coal are listed below, to compare with results from ITSL Run 244.

Distillation cut	Wt % of sample	elemental (wt %)					API gravity (60/60)
		C	H	N	S	O(diff)	
<u>Run 250B (Shell 324)</u>							
Naphtha (IBP-350°F)	19.0	85.40	14.53	0.02	0.01	0.04	52.5
Distillate (350-650°F)	46.8	87.42	12.13	0.10	0.06	0.29	22.9
Gas oil (650°F+)	34.2	88.41	10.88	0.19	0.04	0.48	10.4
<u>Run 250C (SV* = Low)</u>							
Naphtha (IBP-350°F)	17.4	86.00	13.62	0.05	0.27	0.06	48.8
Distillate (350-650°F)	42.5	86.91	11.03	0.26	0.10	1.70	20.2
Gas oil (650°F+)	40.1	88.99	9.97	0.44	0.09	0.51	7.5
<u>Run 250D (SV* = High)</u>							
Naphtha (IBP-350°F)	18.6	85.41	14.26	0.04	0.18	0.11	49.9
Distillate (350-650°F)	45.0	87.45	11.73	0.22	0.09	0.51	21.8
Gas oil (650°F+)	36.4	88.77	10.27	0.36	0.07	0.53	9.7
<u>Run 250E (SV* = High)</u>							
Naphtha (IBP-350°F)	21.7	85.13	13.74	0.04	0.18	0.91	52.3
Distillate (350-650°F)	49.6	87.10	11.13	0.19	0.08	1.50	21.6
Gas oil (650°F+)	28.7	89.09	9.83	0.27	0.07	0.74	8.7
<u>Run 250H</u>							
Naphtha (IBP-350°F)	26.8	84.54	14.04	0.03	0.11	1.28	50.4
Distillate (350-650°F)	51.7	87.54	11.59	0.17	0.07	0.63	24.4
Gas oil (650°F+)	21.5	88.88	10.21	0.28	0.08	0.55	14.1

*SV refers to coal space velocity.

In the comparison of ITSL and CC-ITSL product quality, the catalyst type and catalyst age were the same in Run 244 and Run 250B. The products from Run 250B were better than those from Run 244 in every respect. For comparable fractions the hydrogen contents are 1.2-1.6 wt % higher and the H/C atomic ratios are 0.17-0.23 higher for CC-ITSL. The distillate fraction is lighter for CC-ITSL, 22.9 vs 18.4 API gravity. Also, Run 250B products had lower levels of nitrogen, sulfur, and oxygen. As discussed in Section IV. (1) TSL System Process Performance Comparisons, the product quality improvements were accompanied by an increase in hydrogen consumption. In Run 250B, all the TSL product was derived from the hydrotreater, whereas in the ITSL Run 244, approximately equal amounts were derived from the thermal stage and catalytic stage. This was the reason for the higher hydrogen consumption and higher quality products in Run 250B.

The product quality did not change significantly at the higher coal space velocities. The H/C atomic ratios for the product blends were in the range of 1.54-1.58 for Run 250C, D, and E periods. A comparison of Run 244 and Run 250D data shows that products from higher space velocity CC-ITSL were superior to those from the lower space velocity ITSL.

Product quality data for the CC-ITSL catalytic-catalytic mode of operation with Illinois No. 6 coal are shown below, to compare with results from the CC-ITSL thermal-catalytic mode of operation with solids recycle (250H).

Distillation cut	Wt % of sample	elemental (wt %)					API gravity (60/60)		
		C	H	N	S	O(diff)			
<u>Run 251-IC</u>									
Higher space velocity									
Naphtha (IBP-350°F)	21.1	84.29	13.87	0.03	0.04	1.77	53.2		
Distillate (350-650°F)	49.2	86.75	11.24	0.22	0.02	1.77	20.4		
Gas oil (650°F+)	29.8	88.46	10.27	0.35	0.03	0.89	10.6		
<u>Run 251-IE</u>									
Naphtha (IBP-350°F)	20.9	85.90	13.98	0.02	0.05	0.05	50.9		
Distillate (350-650°F)	42.9	87.74	11.81	0.14	0.02	0.29	24.2		
Gas oil (650+)	36.2	89.64	10.04	0.22	0.03	0.07	9.4		

The coal space velocities were roughly similar in Run 250H and Run 251-IE but the second stage catalyst age was higher in Run 251-IE by a factor of four. For comparable fractions the hydrogen contents were similar, but there was more naphtha and distillate in the Run 250H blend. The higher first stage temperature in Run 250H might have promoted the cracking reactions to form more light cuts. The nitrogen and oxygen were slightly lower in the products from the low space velocity catalytic-catalytic mode of operation (251-IE), compared to the high space velocity Run 251-IC and the thermal-catalytic mode of operation Run 250H.

Processing Wyodak Clovis Point mine coal, ITSL (Run 246), RITSL (Run 249F) and RITSL with solids recycle (Run 249H) product quality data are compared below to see the effects of configurations.

Distillation cut	Wt % of sample	elemental (wt %)					API gravity (60/60)
		C	H	N	S	O(diff)	
<u>ITSL (Run 246)</u>							
Naphtha (IBP-350°F)	19.4	83.06	13.23	0.16	0.51	3.02	47.6
Distillate (350-650°F)	68.9	86.02	10.80	0.25	0.07	2.85	20.1
Gas oil (650°F+)	11.7	88.85	9.23	0.42	0.05	1.45	-
<u>RITSL (Run 249F)</u>							
Naphtha (IBP-350°F)	31.2	84.47	13.71	0.06	0.09	1.67	45.6
Distillate (350-650°F)	58.5	86.77	11.73	0.23	0.03	1.29	23.4
Gas Oil (650°F+)	10.3	88.78	10.68	0.38	0.08	0.09	23.1
<u>RITSL with solids recycle (Run 249H)</u>							
Naphtha (IBP-350°F)	27.9	84.85	13.88	0.09	0.08	1.10	47.2
Distillate (350-650°F)	55.8	85.77	11.81	0.19	0.02	2.21	22.9
Gas oil (650°F+)	16.3	88.49	11.21	0.32	0.01	0.01	16.2

The RITSL configuration resulted in a higher hydrogen content for each fraction than for those obtained in the ITSL configuration of Run 246. This same effect was seen in earlier comparisons of products from the RITSL and ITSL configurations using Illinois No. 6 bituminous coal. The improved hydrogen content appears to be a natural consequence of having an increased content of hydrotreated product in the total product blend with the RITSL configuration. The percent of the total product that was from the hydrotreater was 44 and 53% (249F) for the ITSL and RITSL samples, respectively. The overall product with the RITSL configuration is lighter because of an increase in the fraction of naphtha product from 19 to 31 wt % (249F) of the total. The heteroatom content also shows the quality improvement in the RITSL configuration.

(6) Specific Comparisons for Different Configurations and Process Variables

Seven specific comparisons are discussed as follows, to better understand configuration effects on TSL system process performance. These are:

- (A) Effect of Solids Recycle.
- (B) CC-ITSL Comparison with ITSL.
- (C) CC-ITSL Comparison with RITSL.
- (D) High Coal Space Rate Comparisons.
- (E) CC-ITSL Thermal-Catalytic and Catalytic-Catalytic Mode Comparisons.
- (F) CC-ITSL Catalyst Type, Reaction Temperature and Space Velocity Studies.
- (G) Heteroatom Removal Comparisons.

(A) Effect of Solids Recycle

In the CC-ITSL thermal-catalytic mode of operation with Illinois No. 6 Burning Star coal, 250D and 250G are used for solids recycle comparisons because of their similar 1st and 2nd stage space velocities. Amocat 1C 1/16" catalyst was used in the 2nd stage reactor. For solids recycle, the 1st stage resid concentration in the recycle process solvent was decreased from 50 to 43 wt % (CI-free basis), the 1st stage reaction temperature was increased from 824 to 829°F, and the HTR reaction temperature was increased from 742 to 750°F. Overall TSL process performance was not significantly enhanced by the solids recycle. The TSL C4+ distillate yield was similar, 63-64 wt % MAF coal. The CSD unit performance was considerably improved. The organic rejection to the CSD ash concentrate was reduced from 22 to 15 wt % MAF coal, the lowest observed at Wilsonville. This improvement led to an increase in the TSL resid production, from -1.3 (in 250D) to +5.4 (in 250G) wt % MAF. The following table shows a comparison of TSL process operating conditions and performance data. (Refer to the detailed data in Tables 3 and 5.)

Run no.	250D	250G
Solids recycle	no	yes
<u>Thermal stage</u>		
Average reactor temperature (°F)	824	829
Inlet hydrogen partial pressure (psi)	2500	2500
Coal space velocity	High	High
Solvent-to-coal ratio	1.8	2.0
Solvent resid content (wt %)	50	40
<u>Catalytic stage</u>		
Reactor temperature (°F)	742	750
Space velocity (lb feed/hr-lb cat)	2.1	2.2
(lb MF coal/hr-cu ft cat)	34.2	33.8
Feed resid content (wt %)	53	46
Catalyst age (lb resid+CI)/lb cat	697-786	346-439
Coal conversion (wt % MAF cresol solubles)	92	94
<u>Yield (wt % MAF coal)</u>		
C ₁ -C ₃ gas (total gas)	7(12)	8(13)
Water	10	10
C ₄₊ distillate	64	63
Resid	-1.3	5.4
Hydrogen consumption	-6.1	-6.4
<u>Hydrogen efficiency</u>		
(lb C ₄₊ dist/lb H ₂ consumed)	10.5	9.8
<u>Distillate selectivity</u>		
(lb C ₁ -C ₃ /lb C ₄₊ dist)	0.11	0.12
Energy rejection (% of coal heat value)	23	16
Organics rejected to ash conc. (% MAF coal)	22	15

Major effects of solids recycle in the CC-ITSL thermal-catalytic mode of operation with Illinois No. 6 bituminous coal are summarized below:

- TSL hydrogen consumption increased by 0.3 wt % MAF coal (0.1 in 1st, 0.2 in 2nd).
- C₁-C₃ gases increased by 0.6 wt % MAF coal (0.1 in 1st, 0.5 in 2nd).
- No significant changes in C₄₊ distillate.
 - C₄₊ naphtha increased by 2 wt % MAF coal (1 in 1st, 1 in 2nd).
 - No significant changes in middle distillate.

- Distillate solvent decreased by 4 wt % MAF coal (increased by 2 in 1st, decreased by 6 in 2nd).
- Organic rejection decreased by 7 wt % MAF coal. C₄₊ resid (potential liquid fuel) yield increased from 62 to 68 wt % MAF coal.
- No significant changes in resid + organics rejected.
- Coal conversion increased by 2 wt % MAF coal.
- Resid + UC conversion increased by 3 wt % MAF coal in 1st stage and decreased by 1 wt % MAF coal in the 2nd stage.
- Hydrogen efficiency decreased by 7% (relative).
- C₁-C₃ gas selectivity increased by 9% (relative).
- 1st stage total reactor exotherm increased by 25°F, while 2nd stage exotherm decreased by 11%.

Solids recycle during period 250G (compared to 250D), did not significantly improve TLU process performance, probably due to the low resid and solids content in the recycle process solvent. A slight improvement is indicated by a 3 wt % MAF coal increase in the TLU C₄₊ distillate yield. A 25°F increase of the total dissolver exotherm was seen which indicates more reaction in the thermal stage. TLU hydrogen consumption did not increase significantly.

In the RITSL configuration with Wyodak Clovis Point coal, 249F and 249H are used for solids recycle comparisons. Shell 324 1/32" catalyst was used in the 2nd stage reactor. Overall TSL process performance was slightly enhanced by solids recycle. The TSL C₄₊ distillate yield was 56 wt % MAF coal, an increase of 1 wt % MAF coal. An additional 2 wt % MAF coal of distillate solvent was lost in HTR vacuum bottoms product. This compares to a normal solvent content in the CSD ash concentrate of less than 1 wt % MAF coal. Thus, overall solvent yield had actually increased by 3 wt % MAF coal during solids recycle. The TSL resid yield was negative (-4.4 wt % MAF coal); more resid was lost in 2nd stage vacuum bottoms than was made. The following table shows a comparison of TSL process operating conditions and performance data. (Refer to the detailed data in Tables 4 and 6.)

Run no.	249F	249H
Solids recycle	no	yes
<u>Thermal stage</u>		
Average reactor temperature (°F)	805	803
Inlet hydrogen partial pressure (psi)	2180	2170
Coal space velocity	Low	Low
Solvent-to-coal ratio	1.4	2.0
Solvent resid content (wt %)	28	21
Iron oxide addition	Yes	Yes
<u>Catalytic stage</u>		
Reactor temperature (°F)	700	700
Space velocity (lb feed/hr-1b cat) (lb MF coal/hr-cu ft cat)	1.2 31.5	1.6 31.5
Feed resid content (wt %)	24	27
Catalyst age ((lb resid+UC+ash)/lb cat)	1317-1362	1683-1825
Coal conversion (wt % MAF cresol solubles)	91	95
<u>Yield (wt % MAF coal)</u>		
C ₁ -C ₃ gas (total gas)	8(17)	7(16)
Water	15	14
C ₄ + distillate	55	56
Resid	-4	-7
Hydrogen consumption	-6.1	-6.3
Hydrogen efficiency (lb C ₄ + dist/lb H ₂ consumed)	9.0	8.9
Distillate selectivity (lb C ₁ -C ₃ /lb C ₄ + dist)	0.14	0.12
Energy rejection (% of coal heating value)	27	- (18)*
Organics rejected to ash conc. (% MAF coal)	24	26** (14)*

*Projected yields with CSD operation.

**Vacuum concentrate organic rejection, including 2.2 wt % solvent.

Major effects of solids recycle in the RITSL configuration with Wyodak subbituminous coal are summarized below:

- TSL hydrogen consumption increased by 0.2 wt % MAF coal (0.6 wt % MAF increase in 1st and 0.4 wt % MAF decrease in 2nd).
- C₁-C₃ gases decreased by 1 wt % MAF coal (mainly in 1st).

- C₄₊ distillate increased by 3 wt % MAF coal including distillate in rejection (6 wt % MAF increase in 1st).
 - C₄₊ naphtha decreased by 1 wt % MAF coal.
 - Middle distillate decreased by 2 wt % (mainly in 1st).
 - Distillate solvent increased by 6 wt % MAF coal (8 wt % MAF coal increase in 1st).
- Coal conversion increased by 4 wt % MAF coal.
- No significant changes in resid + organics rejected.
- Resid + UC conversion increased by 4 wt % MAF coal in the 1st stage and decreased by 6 wt % feed (was similar as wt % MAF coal) in the 2nd stage.
- No significant changes in hydrogen efficiency.
- C₁-C₃ gas selectivity decreased by 14%.
- 1st stage total reactor exotherm increased by 56°F, while 2nd stage exotherm decreased by 42%.

In comparing data from 249F and 249H, the solids recycle significantly improved the 1st stage performance as indicated by 6 wt % MAF coal increase in the C₄₊ distillate yield. The dissolver exotherm was very high (109°F) due to a significant enhancement in coal hydrogenation and dissolution reactions by the solids recycle. This 1st stage process improvement appears to be aided by the recycle of iron compounds in the recycle process solvent (5.9 wt % MAF coal).

Major effects of solids recycle in the 1st stage are summarized below:

- 1st stage hydrogen consumption increased by 0.6 wt % MAF coal.
- C₁-C₃ gases decreased by 1 wt % MAF coal.
- C₄₊ distillate increased by 6 wt % MAF coal.
 - No significant changes in C₄₊ naphtha.
 - Middle distillate decreased by 3 wt % MAF coal.
 - Distillate solvent increased by 8 wt % MAF coal.
- No significant changes in resid.
- Coal conversion increased by 4 wt % MAF coal.
- Dissolver exotherm increased by 56°F.
- Solvent quality decreased from 89 to 79% due to the unconverted coal and ash in the process solvent.

Smaller 1st stage performance improvements for solids recycle were observed with bituminous coal in Run 250 compared to improvements seen with Wyodak subbituminous coal in Run 249. The 1st stage C₄₊ distillate increase was 3 wt % MAF coal in Run 250 (6 wt % increase in Run 249). This may be due to the low resid and solids content in the recycle process solvent in Run 250.

TSL process operating condition and performance data changes during the solids recycle tests (249F vs 249H and 250D vs 250G) with the two different coals are compared below:

Configuration Runs compared	RITSL <u>249F vs 249H</u>	CC-ITSL <u>250D vs 250G</u>
Coal processed	Wyodak	Illinois #6
Iron oxide addition	Yes	No
<u>Process condition changes</u>		
Solids in process solvent, wt %	+25	+9
Resid in process solvent, wt % (CI-free basis)	0	-7
<u>Exotherm, °F</u>		
1st stage	+56	+25
2nd stage (%)	-42	-11
<u>Performance changes, wt % MAF coal</u>		
<u>1st stage</u>		
Hydrogen consumption	+0.6	+0.1
Coal conversion (a)	+4	-1
C ₄₊ distillate	+6	+3
C ₁ -C ₃ gas	-1	0
<u>TSL</u>		
Hydrogen consumption	+0.2	+0.3
Coal conversion (a)	+4	+2
C ₄₊ distillate	+3	-1
C ₁ -C ₃ gas	-1	+1
Organics rejected	0(b) (-10)(c)	-7

(a) Coal conversion to cresol solubles.

(b) Solvent-free basis for 249H. HTR vacuum bottoms purged.
CSD was not operated.

(c) Projected rejection with CSD operation.

Smaller increases in 1st stage total dissolver exotherm, hydrogen consumption and coal conversion for Run 250 compared to Run 249 confirmed that the solids recycle improvements in the 1st stage were smaller for bituminous coal in Run 250. TSL improvements in C₄₊ distillate, coal conversion, and organic rejection were also smaller for bituminous coal in Run 250.

TSL preasphaltene yield changes during the solids recycle tests (249F vs 249H and 250D vs 250G) with two different coals are compared below:

Configuration	RITS		CC-ITS	
Run	<u>249F</u>	<u>249H</u>	<u>250D</u>	<u>250G</u>
Ash recycle	No	Yes	No	Yes
<u>Preasphaltene yield (wt % MAF coal)</u>				
1st Feed	0.0	7.1	3.7	5.9
Product	8.1	12.0	22.9	21.2
Net Yield	8.1	4.9	19.2	15.3
2nd Feed	8.1	12.0	22.9	21.2
Product	2.6	6.6	8.7	11.0
Net Conversion	5.5	5.4	14.2	10.2
Overall Yield	2.6	-0.5	5.0	5.1
CSD rejection	2.6	-	5.0	5.1
2nd stage rejection	-	1.7	-	-
TSL accumulation	0.0	-2.2	0.0	0.0
<u>Preasphaltene conversion (wt % feed)</u>				
1st stage (a)	92	89	78	80
2nd stage	68	45	62	48

(a) Assume MAF coal is 100 wt % preasphaltenes.

Run 249H and 250G with solids recycle produced 3-4 wt % MAF coal less preasphaltenes in the 1st stage and decreased 2nd stage conversion by 0-4 wt % MAF coal. The 2nd stage conversion was significantly lower by 14 wt % feed with solids recycle. These results confirmed that solids recycle improves the 1st stage performance, but decreases the 2nd stage performance.

(B) CC-ITS Comparison with ITS

Run 250B (CC-ITS) results are compared with Run 244CD (ITS). Both runs used a Shell 324 unimodal Ni-Mo catalyst (1/32" diameter) in the 2nd stage. Illinois No. 6 Burning Star mine coal was processed without solids recycle. Catalyst age was 940-1470 lb (res+UC+ash)/1b cat. Catalyst age data as a unit of 1b MF coal/lb catalyst were not calculated for these runs. Therefore, they are not available. Catalyst histories in these runs were very complex, preventing accurate calculations. Estimates based on aging rates measured in Runs 244CD and 250B are 1107 and 982 lb MF coal/lb catalyst, respectively. These

ages correspond to 1.8 and 1.7 lb/ton MF coal catalyst replacements at steady state operation. Process operating conditions were similar for these runs, except for higher 1st stage and lower 2nd stage hydrogen partial pressures for 250B.

Major observations in performance are:

- CC-ITSL hydrogen consumption was higher by 1.2 wt % MAF coal.
- CC-ITSL C₄₊ distillate was higher by 4 wt % MAF coal.
- CC-ITSL C₄₊ naphtha was higher by 4 wt % MAF coal.
- Organic rejection was similar for both configurations.
- CC-ITSL hydrogen efficiency was lower by 13% (relative).
- CC-ITSL C₁-C₃ gas selectivity was higher by 9% (relative).

High hydrogen consumption with CC-ITSL is attributed to high resid reactivity in the catalytic stage and to production of high quality distillate products. CC-ITSL distillate products had higher hydrogen contents by 1.2-1.6 wt % and higher H/C atomic ratios by 0.15-0.23. Catalyst activity was higher due to the high resid reactivity (Reference 13). A higher hydrogen partial pressure (680 psia) in the first stage could have increased the hydrogen consumption.

Operating period	244CD	250B
Mode	ITSL	CC-ITSL
Dates	<u>7/28-8/15/83</u>	<u>12/4-7/85</u>
Catalyst type	Shell 324	Shell 324
<u>1st stage</u>		
Reaction temp., °F (average)	810(a)	809
Coal space rate	Low	Low
Inlet hydrogen partial pressure (psi)	2120	2800
<u>2nd stage</u>		
Reaction temp., °F (average)	730	725
Space rate, WHSV, hr ⁻¹	1.1	1.0
1b MF coal/lb-cu ft cat	26.1	19.6
Inlet hydrogen partial pressure (psi)	2580	2470
Catalyst age		
1b (rest+UC+ash)/lb cat	943-1139(b)	1432-1470(b)
1b MF coal/lb cat	1107(c)	982(c)
<u>TSL yield, wt % MAF coal</u>		
Hydrogen consumption	5.2	6.4
C ₁ -C ₃ gas (total gas)	6(11)	6(11)
Water	10	10
C ₄ + distillate	57	61
C ₄ + naphtha	11	15
Resid	8	6
Ash concentrate	19	18
<u>Hydrogen efficiency</u>		
1b C ₄ + dist/lb H ₂ cons	11.0	9.6
<u>C₁-C₃ selectivity (X100)</u>		
to C ₄ + distillate	11	10
<u>Energy rejection, %</u>	20-22	20
(a) Reactor outlet temperature.		
(b) Lb resid/lb cat.		
(c) May not be accurate data because of the complexity of catalyst histories. Estimates based on aging rates measured in each period.		
(c) <u>CC-ITSL Comparison with RITSL</u>		
Run 250C (CC-ITSL with Amocat 1C 2nd stage catalyst) results are compared with Run 247C and D (RITSL with Shell 324 2nd stage catalyst). Amocat 1C is a bimodal catalyst with a 1/16" diameter. Illinois No. 6 Burning Star coal was processed without solids recycle. Coal space rates for these runs were similar. Process operating conditions were similar for these runs except for (1) higher 1st stage hydrogen partial pressure for 250C (300-330 psia), (2) slightly higher resid content in the process solvent for 250C, (3) less catalyst charge (higher 2nd stage space velocity) for 250C, and (4) lower 2nd stage hydrogen partial pressure for 250C (170-180 psia).		

Major observations in TSL performance are:

- CC-ITSL with Amocat 1C (250C) showed lower hydrogen consumption (by 0.5 wt % MAF coal), compared to RITSL with Shell 324 (247C). C₄₊ distillate yield was similar (60-61 wt % MAF coal).
- CC-ITSL with Amocat 1C (250C) showed 11% (relative) higher hydrogen efficiency compared to RITSL with Shell 324 (247C).
- CC-ITSL with Amocat 1C (250C) showed 18% (relative) better C₁-C₃ gas selectivity compared to RITSL with Shell 324 (247C).

These overall process performance improvements, high hydrogen efficiency and low C₁-C₃ gas selectivity, are due to the CC-ITSL configuration without fractionation between stages and the bimodal Amocat 1C catalyst in the 2nd stage. The higher 1st stage hydrogen partial pressure (300-330 psia) and the higher resid recycle could have affected the performance by improving the performance in the 1st stage. The low hydrogen consumption with the high hydrogen efficiency appears to decrease the hydrogen content of the distillate product, as shown in tables on pages 40 and 42. The hydrogen content of recycle resid was higher in 250C, although the second stage operated at a lower reaction temperature, lower inlet hydrogen partial pressure and a higher coal space rate (Reference 13). Amocat 1C catalyst has a good hydrogenation activity of the resid. However, the distillate yield in the 2nd stage was 7 wt % MAF coal lower. This could be due to the lower 2nd stage hydrogen partial pressure, the higher coal space rate and the higher resid recycle (Table 3 on pages 102 and 103). Amocat 1C catalyst might also have a selectivity in hydrogenation and hydrocracking of the resid different from that for Shell 324 catalyst. A detailed performance comparison is given as follows:

Operating period	247C	247D	250C
Mode	RITSL	RITSL	CC-ITSL
Dates	<u>10/29-26/84</u>	<u>11/1-19/84</u>	<u>1/11-16/86</u>
Catalyst type	Shell 324	Shell 324	Amocat 1C
<u>1st stage</u>			
Reaction temp., °F (average)	812	810	809
Coal space rate	Low	Low	Low
Inlet hydrogen partial pressure (psi)	2180	2150	2480
<u>2nd stage</u>			
Reaction temp., °F (average)	698	711	703
Space rate, WHSV, hr ⁻¹	0.94	0.90	1.34
lb MF coal/hr-cu ft cat	20.1	19.5	22.7
Inlet hydrogen partial pressure (psi)	2700	2690	2520
Catalyst age			
lb (rest+UC+ash)/lb cat	283-372(a)	446-671(a)	302-396
Resid in solvent, wt %	43	44	48
<u>TSL yield, wt % MAF coal</u>			
Hydrogen consumption	6.1	6.1	5.6
C ₁ -C ₃ gas (total gas)	7(12)	6(12)	5(11)
Water	10	9	10
C ₄ + distillate	60	62	61
C ₄ + naphtha	12	11	11
Resid	5	3	2
Ash concentrate	20	21	22
<u>Hydrogen efficiency</u>			
lb C ₄ + dist/lb H ₂ cons	9.8	10.2	10.9
<u>C₁-C₃ selectivity (X100)</u>			
to C ₄ + distillate	11	10	9
<u>Energy rejection, %</u>			
	22	22	24

(a) Lb resid/lb cat.

CC-ITSL Run 250C also showed better 1st stage process performance with higher C₄+ distillate by 8 wt % MAF coal, less C₁-C₃ gas by 1-2 wt % MAF coal, and lower resid by 5 wt % MAF coal, compared to RITSL Runs 247C and 247D. The higher hydrogen partial pressure (300-330 psia) and the higher resid recycle could have improved the 1st stage performance.

TSL preasphaltene yields for CC-ITSL Run 250C and RITSL Run 247D are compared as follows:

Configuration	RITS	CC-ITSL
Run	247D	250C
Catalyst type	Shell 324	Amocat 1C
<u>Preasphaltene yield (wt % MAF coal)</u>		
1st Feed	5.4	1.7
Product	27.4	19.6
Net Yield	22.0	17.9
2nd Feed	27.4	19.6
Product	12.2	8.0
Net Conversion	15.2	11.6
Overall yield	6.8	6.3
CSD rejection	6.8	6.3
<u>Preasphaltene conversion wt % of feed</u>		
1st (a)	74	81
2nd	55	59

(a) Assume MAF coal is 100 wt % preasphaltenes.

CC-ITSL Run 250C produced 4 wt % of MAF coal less preasphaltenes in the 1st stage and decreased 2nd stage conversion by 4 wt % MAF coal. The 1st and 2nd stage preasphaltene conversion were increased by 7 and 4 wt % of feed, respectively, for CC-ITSL Run 250C. This indicates that CC-ITSL with Amocat 1C catalyst improved preasphaltene conversion reactivity compared to RITS with Shell 324 catalyst. The higher hydrogen partial pressure (300-330 psia) and the higher resid recycle could have improved the 1st stage conversion, while the lower hydrogen partial pressure, the higher coal space rate and the higher resid recycle could have decreased the 2nd stage conversion.

For Wyodak Clovis Point coal with solids recycle, Run 251-IIB (CC-ITSL with Amocat 1C 2nd stage catalyst) results are compared with Run 249H (RITS with Shell 324 2nd stage catalyst). The following table shows a comparison of TSL process operating conditions and performance data.

Run no.	249H	251-IIB
Configuration	RITSL	CC-ITSL
<u>Thermal stage</u>		
Average reactor temperature (°F)	803	819
Inlet hydrogen partial pressure (psi)	2170	2510
Coal space velocity	Low	Low
Solvent-to-coal ratio	2.0	2.0
Solvent resid content (wt %)	21	25
Iron oxide (wt %)	1.5	0.8
<u>Catalytic stage</u>		
Catalyst	Shell 324	Amocat 1C
Reactor temperature (°F)	700	743
Space velocity (lb feed/hr-lb cat) (lb MF coal/hr-cu ft cat)	1.6 31.5	2.8 43.6
Feed resid content (wt %)	27	31
Catalyst age (lb (resid + CI)/lb cat)	1680-1830	910-970
CSD unit	off-line	on-line
<u>Yield (wt % MAF coal)</u>		
C ₁ -C ₃ gas (total gas)	7(16)	8(18)
Water	14	14
C ₄ ⁺ distillate	56	(57)
Resid	-7	(4)
Hydrogen consumption	-6.3	-6.3
<u>Hydrogen efficiency</u>		
(lb C ₄ ⁺ dist/lb H ₂ consumed)	8.9	9.7
<u>Distillate selectivity</u>		
(lb C ₁ -C ₃ /lb C ₄ ⁺ dist)	0.12	0.13
Energy rejection (% of coal heating value)	-	(18)
Organics rejected to ash conc. (% MAF coal)	(26)**	(14)

*Projected yields with CSD operation.

**Vacuum concentrate organic rejection.

RITSL Run 249H yields for operation with the CSD unit were projected from 249H yields by using CSD data obtained at the end of Run 249. Although many differences in process operating conditions such as reaction temperature, catalyst type and age, space velocity and hydrogen partial pressure make the direct comparison difficult, the following observations can be made:

- CC-ITSL operation with subbituminous coal in the thermal-catalytic mode achieved an "all-distillate" yield slate while operating with a space velocity 40% higher than in the RITSL configuration.

- CC-ITSL with Amocat 1C (251-IIB) showed higher C₄₊ distillate by 4 wt % MAF coal, compared to RITSL with Shell 324 (249H). C₄₊ distillate yield was high, 61 wt % MAF coal and potential liquid product yield (C₄₊ resid) was 65 wt % MAF coal.
- CC-ITSL showed lower organic rejection by 5 wt % MAF coal. Organic rejection was low, 9 wt % MAF coal.
- CC-ITSL showed 9% (relative) higher hydrogen efficiency. Hydrogen consumption was similar (6.3 wt % MAF coal).
- CC-ITSL showed higher 2nd stage resid + UC conversion by 6 wt % of feed (8 wt % MAF coal), but lower 1st stage resid + UC conversion by 3 wt % of feed (3 wt % MAF coal).

The 1st stage performance declined with CC-ITSL Run 251-IIB compared to RITSL Run 249H, that is, lower hydrogen consumption by 0.5 wt % MAF coal, lower resid + UC conversion by 3 wt % of feed (3 wt % MAF coal), and higher resid by 4 wt % MAF coal. The decrease of 1st stage reactor exotherm by 26°F also confirmed the decline of 1st stage performance. The 2nd stage reactor exotherm increased by 110% and 2nd stage hydrogen consumption increased by 0.5 wt % MAF coal, indicating higher hydrogenation in the 2nd stage with CC-ITSL Run 251-IIB. The higher reaction temperature and younger catalyst age in the second stage could affect the process improvements, although 251-IIB operated at 40% higher space velocity than 249H.

(D) High Coal Space Rate Comparisons

Three different 1st stage reactor types were studied to investigate effects of coal space rates and process configurations on process performance. Reactor dimensions are given below:

Reactor type	SCT (Short Contact Time)	LCT (Low Contact Time)	CC (Close Coupled)
Configuration	ITSL	ITSL	CC-ITSL
Run	242	248	250
Dimension	1 1/4" (Sch 160) x 606'L preheater coil tube	5.2" ID x 23'H no backmixing	11 7/8" OD x 40'H forced backmixing

CC-ITSL 250E is compared with 242BC (ITSL-SCT) and 248F (ITSL-LCT) as follows. Run 250E used an Amocat 1C bimodal (1/16" diameter) and Run 242BC and 248F used a Shell 324 unimodal Ni-Mo catalyst (1/32" diameter) in the 2nd stage. Illinois #6 Burning Star coal was processed without solids recycle. Coal space rates for these runs were high. There are many differences in process operating conditions, such as (1) catalyst type and age, (2) reaction temperature, (3) 1st stage hydrogen partial pressure, and (4) HTR space rate.

Major observations in TSL performance are:

- CC-ITSL (250E) with Amocat 1C catalyst produced more C₄₊ distillate by 4 wt % MAF coal compared to ITSL-LCT (248F) and ITSL-SCT (242BC) with Shell 324 catalyst.
- CC-ITSL (250E) had higher coal conversion by 1-3 wt % MAF coal.
- CC-ITSL (250E) had lower organic rejection by 3-5 wt % MAF coal.
- CC-ITSL (250E) had higher hydrogen consumption by 0.4-0.8 wt % MAF coal.
- ITSL-SCT (242BC) showed high hydrogen efficiency with low C₁-C₃ gas selectivity.

Better TSL process performance with CC-ITSL, high C₄₊ distillate, high coal conversion and low organic rejection, are due to the CC-ITSL configuration and the bimodal Amocat 1C catalyst in the 2nd stage.

A more detailed comparison is given in the following table:

Operating period	242BC	248F	250E
Mode	ITSL	ITSL	CC-ITSL
Dates	<u>12/20/82-1/2/83</u>	<u>4/21-25/85</u>	<u>2/12-16/86</u>
Catalyst type	Shell 324	Shell 324	Amocat 1C
<u>1st stage</u>			
Reactor	SCT(preheater)	LCT(50%)	CC(50%)
Reaction temp., °F (average)	860(a)	835	829
Coal space rate	High(b)	High(b)	High(b)
Inlet hydrogen partial pressure, psia	2180	2170	2520
<u>2nd stage</u>			
Reaction temp., °F (average)	720	728	750
Space rate, WHSV, hr ⁻¹	1.0	0.71	2.59
1b MF coal/hr-cu ft cat	25.1	21.5	41.9
Catalyst age,			
lb (rest+UC+ash)/lb cat	278-441	359-396	1,040-1,191
lb MF coal/lb cat	NA	NA	605-701
<u>TSL yields, wt % MAF</u>			
Hydrogen	-4.9	-5.3	-5.7
C ₁ -C ₃ gas (total gas)	5(9)	7(12)	6(12)
Water	10	9	9
C ₄ + distillate	54	54	58
C ₄ + naphtha	10	13	14
Middle distillate	7	8	7
Distillate solvent	37	33	36
Resid	8	8	7
Organic rejection	24	22	19
Coal conversion, wt % MAF	92	90	93
<u>Hydrogen efficiency</u>			
lb C ₄ + dist/lb H ₂ cons.	11.0	10.2	10.2
<u>C₁-C₃ selectivity (x100)</u>			
to C ₄ + distillate	8.0	12.0	11.0
<u>Energy rejection, %</u>			
	23-27	23	22

(a) Preheater outlet temperature.

(b) Space velocity representation restricted by confidentiality agreements with Hydrocarbon Research, Inc.

1st stage performance was similar for 242BC, 248F and 250E, which all had a high coal space rate. C₄+ distillate was 25-28 wt % MAF coal, and C₁-C₃ gas was 3-5 wt % MAF coal. CC-ITSL Run 250E showed higher coal conversion by 1-3 wt % MAF coal and higher resid by 4-6 wt % MAF coal.

TSL preasphaltene yields were calculated for 242BC (ITSL-SCT), 248F (ITSL-LCT) and 250E (CC-ITSL) and are compared below:

Run	242BC	248F	250E
Dates	(12/20/82-1/2/83)	(4/21-25/85)	(2/12-16/86)
Configuration	ITSL-SCT	ITSL-LCT	CC-ITSL
Catalyst	Shell 324	Shell 324	Amocat 1C

Preasphaltene yield
(wt % MAF coal)

1st Feed	6.3	5.4	6.8
Product	27.2	28.7	27.8
Net Yield	20.9	23.3	21.0
2nd Feed	20.9	22.0	27.8
Product	6.3	5.4	13.2
Net Conversion	14.6	16.6	14.6
Overall yield	6.3	6.7	6.4
CSD rejection	6.3	6.7	6.4

Preasphaltene conversion
wt % of feed

1st (a)	74	73	74
2nd	70	73	53

(a) Assume MAF coal is 100 wt % preasphaltenes.

Similar net preasphaltene yield (1st stage), net preasphaltene conversion (2nd stage) and 1st stage conversion were observed for these runs. The decrease of 2nd stage preasphaltene conversion appears to be mainly due to a higher preasphaltene rate in the 2nd stage feed stream.

(E) CC-ITSL Thermal-Catalytic and Catalytic-Catalytic Mode Comparisons

TSL process performance is compared for CC-ITSL thermal-catalytic mode Run 250 and catalytic-catalytic mode Run 251-IE. Illinois #6 Burning Star coal was processed with solids recycle. Run 251-IE with an Amocat 1A 1st stage catalyst and an Amocat 1C 2nd stage catalyst, and Run 250H with no 1st stage catalyst and Amocat 1C 2nd stage catalyst are selected for comparisons because of similar coal feed rates (280-300 MF lb/hr). Several process operating conditions were different for these runs such as (1) lower 1st stage reaction temperature for 251-IE, (2) lower coal space rate for 251-IE, and (3) high 1st and 2nd stage catalyst ages for 251-IE.

- Catalytic-catalytic mode (251-IE) showed slightly higher hydrogen consumption by 0.3 wt % MAF coal and higher C₄₊ distillate by 8 wt % MAF coal.
- Catalytic-catalytic mode (251-IE) showed 8% higher hydrogen efficiency.
- Catalytic-catalytic mode (251-IE) showed 8% better C₁-C₃ gas selectivity.

These overall TSL process performance improvements, high hydrogen efficiency and low C₁-C₃ gas selectivity, are due to the use of a highly active Amocat 1A catalyst in the 1st stage. Amocat 1A catalyst for 251-IE increased the 1st stage C₄₊ distillate by 24 wt % MAF coal. The 1st stage hydrogen consumption increased by 2.4 wt % MAF coal. The 1st stage resid + UC conversion increased by 11 wt % feed (22 wt % MAF coal), while the 2nd stage resid + UC conversion decreased by 8 wt % feed (15 wt % MAF coal). The C₄₊ resid (potential liquid fuel) yield are the same for both modes, 69 wt % MAF coal, with the same organic rejection, 15 wt % MAF coal. A detailed performance comparison is given in the following table:

Run no.	<u>250H</u>	<u>251-IE</u>
<u>First stage</u>		
Catalyst	none	Amocat 1A
Average reactor temperature (°F)	834	808
Inlet hydrogen partial pressure (psi)	2410	2470
Coal space velocity	High(a)	High(a)
Space velocity (lb feed/hr-lb cat)	-	2.7
(lb MF coal/hr-cu ft cat)	-	36.3
Solvent-to-coal ratio	2.0	2.0
Solvent resid content (wt %)	39	40
Catalyst age (lb (resid+CI)/lb cat)	-	2160-2250
<u>Second stage</u>		
Catalyst	-----Amocat 1C-----	
Reactor temperature (°F)	759	758
Space velocity (lb feed/hr-lb cat)	2.3	2.3
(lb MF coal/hr-cu ft cat)	34.7	37.2
Feed resid content (wt %)	46	39
Catalyst age (lb (resid + CI)/lb cat)	500-630	2310-2370
<u>Yield (wt % MAF coal)</u>		
C ₁ -C ₃ gas (total gas)	8(13)	7(12)
Water	9	10
C ₄ + distillate	62	70
Resid	7	-1
Hydrogen consumption	-6.5	-6.8
<u>Hydrogen efficiency</u>		
(lb C ₄ + dist/lb H ₂ consumed)	9.5	10.3
<u>Distillate selectivity</u>		
(lb C ₁ -C ₃ /lb C ₄ + dist)	0.12	0.11
Energy content of feed coal rejected to ash conc. (%)	18	17
Organics rejected to ash conc. (% MAF coal)	15	15
(a) Space velocity representation restricted by confidential agreements with Hydrocarbon Research, Inc.		
TSI preasphaltene yields were calculated for Run 251-1B (cata- lytic-catalytic mode) and are compared with 250H (thermal- catalytic mode).		
<ul style="list-style-type: none"> ● Catalytic-catalytic mode produced less 1st stage net preasphaltenes by 8 wt % MAF coal. ● Catalytic-catalytic mode produced similar overall TSI net preasphaltene. 		

- Catalytic-catalytic mode increased 1st stage preasphaltene conversion by 8 wt % feed, but decreased 2nd stage conversion by 41 wt% feed. The decrease of preasphaltene conversion in the 2nd stage is probably due to the operation at a higher space rate and higher catalyst ages.

Run	250H (3/13-3/16/86)	251-IB (5/11-5/14/86)
Operation Mode	Thermal-Catalytic	Catalytic-Catalytic
Catalyst type		
1st stage	None	Amocat 1A
2nd stage	Amocat 1C	Amocat 1C
Preasphaltene yield (wt % MAF coal)		
1st Stage Feed	5.5	4.8
Product	19.1	10.2
Net Yield	13.6	5.4
2nd Stage Feed	19.1	10.2
Product	9.0	9.0
Net Conversion	10.1	1.2
Overall Yield	4.5	4.2
CSD Rejection	4.5	4.2
Preasphaltene conversion wt % of feed		
1st stage (a)	82	90
2nd stage	53	12(b)

(a) Assume MAF coal is 100 wt % preasphaltenes.

(b) Operated at a higher coal space velocity and higher catalyst ages.

(F) CC-ITSL Catalyst Type, Reaction Temperature and Space Velocity Studies

During Runs 250 and 251 many process conditions were varied to study TSL system process performance responses in the CC-ITSL configuration. Major process variables evaluated during these runs are:

- Coal type: Illinois No. 6 Burning Star Mine
Wyodak Clovis Point Mine
- Operation mode: thermal-catalytic
catalytic-catalytic
- Solids recycle
- Reaction temperature
1st stage: 775-835°F
2nd stage: 700-775°F
- Coal space rate: Low to High
- Space velocity
1st stage: 2.7-4.2 lb feed/hr-lb cat
(35-60 lb MF coal/hr-cu ft cat)
2nd stage: 2.3-3.9 lb feed/hr-lb cat
(23-60 lb MF coal/hr-cu ft cat)
- Catalyst type
1st stage: Amocat 1A 1/16" (Co-Mo)
2nd stage: Shell 324 1/32" (Ni-Mo)
Amocat 1C 1/16" (Ni-Mo)
- CSD DAS type: 3504-3100, 2203-2653, 2504-2754

Specifically, reaction temperature, space velocity and catalyst activity effects on TSL system process performance were emphasized to investigate their selectivity on C₄⁺ distillate, C₄⁺ naphtha and C₁-C₃ gases.

Results from the CC-ITSL configuration study were reported in Technical Progress Report Run 250 (Ref. 13) and Technical Progress Report Run 251 (Ref. 14).

(G) Heteroatom Removal Comparisons

TSL system H_2O , H_2S and NH_3 yields for 250D and G (thermal-catalytic mode) and 251-IE (catalytic-catalytic mode) in the CC-ITSL configuration are compared below, to study effects of Amocat 1A (Co-Mo) 1/16" catalyst in the 1st stage on TSL system heteroatom (O, S, N) removal. Illinois No. 6 Burning Star Mine coal was processed during these runs.

Run	250D (2/2-2/5/86)	250G (3/8-3/11/86)	251-IE (6/3-6/5/86)
Operation Mode	thermal-catalytic	thermal-catalytic	catalytic-catalytic
Solids recycle	No	Yes	Yes
<u>Catalyst type</u>			
1st stage	none	none	Amocat 1A
2nd stage	Amocat 1C	Amocat 1C	Amocat 1C
<u>Catalyst age, lb(res+Cl)/lb cat</u>			
1st stage	-	-	2159-2245
2nd stage	697-706	346-439	2313-2374
<u>Reaction temp., °F</u>			
1st stage	824	829	808
2nd stage	742	750	758
<u>Space velocity, lb feed/hr-lb cat</u>			
1st stage	-High (a)	-(High) (a)	2.67(High) (a) (36.3) (b)
2nd stage	2.08 (lb MF coal/hr-cu ft cat)	2.23 (33.8)	2.28 (37.2)
<u>H_2O yield, wt % MAP coal</u>			
TSL	9.5	10.2	10.3
1st stage	5.6	5.9	5.9
2nd stage	3.9	4.3	4.4
<u>H_2S yield, wt % MAP coal</u>			
TSL	2.7	2.7	2.9
1st stage	1.6	1.5	2.6
2nd stage	1.1	1.2	0.3
<u>NH_3 yield, wt % MAP coal</u>			
TSL	1.2	1.3	1.4
1st stage	0.3	0.1	0.7
2nd stage	1.0	1.2	0.7
<u>H_2 consump., wt % MAP coal</u>			
TSL	6.1	6.4	6.8
1st stage	1.7	1.8	4.5
2nd stage	4.4	4.6	2.3

(a) Coal space rate.

(b) Lb MF coal/hr-cu ft cat.

The catalytic-catalytic mode with Amocat 1A catalyst in the 1st stage, produced more H_2S and NH_3 in the 1st stage with higher hydrogen consumption, while producing less H_2S and NH_3 in the 2nd stage with lower hydrogen consumption. Overall TSL system heteroatom removal was similar for both modes or slightly better

in the catalytic-catalytic mode. Sour water analyses (Table 7) confirmed the above observed high catalytic activity of Amocat 1A (1st stage) in the catalytic-catalytic mode in heteroatom removal. The 1st stage sour water produced in the catalytic-catalytic mode contained much higher sulfur and nitrogen than that in the thermal-catalytic mode. However, the 2nd stage sour water had similar sulfur and nitrogen contents for both modes, which is inconsistent with the decreases of 2nd stage H_2S and NH_3 yields in the catalytic-catalytic mode, compared to the thermal-catalytic mode. Sour water analyses data for other configurations in the thermal-catalytic mode of operation (Tables 7 and 8) were not different enough to see configuration effects on heteroatom removal.

(7) Catalyst Activity and Requirements Comparisons

Catalyst activity and deactivation in coal liquefaction and resid + UC hydrotreating reactions are very complex, involving numerous chemical compounds and reaction routes. Generally, deactivation is understood a catalyst activity decline due to the loss of active sites caused by coke formation on the catalyst, metal poisoning, sintering, etc. Catalyst pore volume and surface area become reduced by the deactivation. In order to understand the catalytic resid + UC conversion reaction for the distillate production, a simplified first-order kinetics model has been extensively tested and developed for an ideal continuous stirred-tank reactor (CSTR). The model bases and assumptions were reported in Topical Report No. 4, "Catalytic Hydrogenation Unit Studies" (Ref. 23) and are also attached in Appendix A. This model has been continuously applied and practiced in various configuration studies (NTSL, ITSL, DITSL, RITSL and CC-ITSL) to evaluate the catalyst activity and deactivation in the TSL process. Catalyst activity and deactivation study results have been published (Ref. 4-14, 22-28).

Basically catalyst activity is defined as a function of space velocity, resid + UC conversion, reaction temperature and catalyst age. These process variables are experimentally measured for activity calculations. All runs except for Run 245 operated in the batch mode. Run 245 was a proof-of-concept run to simulate a commercial plant operation by employing 1 lb/ton MF coal catalyst replacement in the second stage (Ref. 8 and 27).

Catalyst activities were calculated assuming that the resid + UC conversion reaction follows first-order kinetics for a continuous stirred tank reactor. The conversion rate constant (K) is expressed in terms of two experimentally determined quantities: feed weight-hourly space velocity (WHSV) and resid + UC conversion (ϵ).

$$K = \text{WHSV} \cdot \frac{\epsilon}{1-\epsilon}$$

The dependence of the conversion rate constant on temperature (T) is described by the Arrhenius equation and the deactivation of the rate constant with catalyst age (t), [expressed as lb (resid + CI/lb cat)] is described by a deactivation model.

$$K = A \cdot e^{-E/RT} \cdot e^{-\alpha t}$$

In this equation, A is the frequency factor, E is the apparent activation energy, and α is the deactivation coefficient. The equilibrium catalyst activity (K_{eq}) with catalyst replacement for commercial plant simulation is determined by using the residence time distribution function, $RTD(t)$.

$$K_{eq} = \int_0^{\infty} RTD(t) \cdot K(t) \cdot dt$$

The linear regression analysis equation for the conversion rate constant (K) becomes:

$$\ln K = \ln A - E/R (1/T) - \alpha t$$

K: rate constant, [1/hr]
 A: frequency factor
 E: activation energy, [Btu/lb-mole]
 R: ideal gas constant, [Btu/lb-mole-°F]
 T: reaction temperature, [°R]
 α : deactivation coefficient, [1/t]
 t: catalyst age, [lb (resid+CI)/lb cat]

Catalyst requirements for the commercial plant simulation are projected by using a continuous catalyst replacement at infinite time (Ref. 25-28). The equilibrium catalyst activity (K_{eq}) with an equilibrium catalyst age distribution at infinite time can be calculated as shown below. Three different deactivation coefficients (α_1 , α_2 and α_3) are assumed for this calculation.

$$K_{eq} = \int_0^\infty RTD(t) K(t) dt$$

$$RTD(t) = 1/t e^{-t/\tau}$$

τ = catalyst hold-up/catalyst addition rate

$$K(t) = k_0 e^{-\alpha_1 t} \quad 0 < t \leq t_{sc1}$$

$$K(t) = k_0' e^{-\alpha_2 t} \quad t_{sc1} \leq t \leq t_{sc2}$$

$$K(t) = k_0'' e^{-\alpha_3 t} \quad t \geq t_{sc2}$$

$$K_{eq} = \left(\frac{k_0}{1 + \alpha_1 \tau} \right) \left[1 - e^{-t_{sc1}(\alpha_1 + 1/\tau)} \right] +$$

$$\left(\frac{k_0'}{1 + \alpha_2 \tau} \right) \left[e^{-t_{sc1}(\alpha_2 + 1/\tau)} - e^{-t_{sc2}(\alpha_2 + 1/\tau)} \right] +$$

$$\left(\frac{k_0''}{1 + \alpha_3 \tau} \right) e^{-t_{sc2}(\alpha_3 + 1/\tau)}$$

The above equation applies only for the continuous catalyst replacement. For the discrete replacement mode of operation, the average catalyst bed activity can be calculated from the history of the discrete replacement, as practiced in Run 245 (Ref. 8 and 27). It is a weighted average activity by using the age and activity distribution in the catalyst bed at a particular time.

It is evident from the above discussion that the catalyst requirement projection involves a complex theoretical calculation accommodating activity level, deactivation path and age distribution. Specifically, the projection calculation requires the data of catalyst age (t), activity (K), deactivation coefficient (α) and age residence time distribution (RTD).

CC-ITSL catalyst batch deactivation trends without solids recycle (Run 250CDE) are compared to ITSL (Runs 242, 243 and 244) and RITSL (Run 247) operations in Figure 23. Illinois No. 6 Burning Star Mine coal was processed for these configurations. CC-ITSL catalyst deactivation rates were lower than for ITSL or RITSL. In general Amocat 1C catalyst with CC-ITSL showed higher catalyst activity at catalyst ages above 400 lb (res+UC+ash)/lb cat than observed with Shell 324 catalyst for ITSL or RITSL.

In order to compare catalyst activity and deactivation impacts on the overall TSL process performance with different configurations and catalysts, equilibrium activity (K_{eq}) and resid conversion (ϵ_{eq}) are calculated and compared below.

- equilibrium activity (K_{eq}) at set process conditions

- catalyst addition = 1 lb/ton coal = 2.5 lb/day
- WHSV = 1.0 lb/hr feed/lb cat
- catalyst inventory = 410 lb
- catalyst aging rate = 13 lb/lb cat/day
- temperature = 720°F
- coal = Illinois #6 Burning Star

Configuration	Run	Catalyst	$K_{eq} (hr^{-1})$	$\epsilon_{eq} (\%)$	$A (hr^{-1})$	$E (\text{Btu/lb-mole})$
ITSL	242, 243, 244	Shell 324	0.197	16.5	6.94×10^6	50,000
RITSL	247	Shell 324	0.293	22.6	1.27×10^7	39,500
CC-ITSL	250B	Shell 324	0.330	24.8	-	-
CC-ITSL	250CDE	Amocat 1C	0.457	31.4	1.97×10^8	44,600
CC-ITSL	250FCM	Amocat 1C	0.357	26.3	3.90×10^7	42,500

CC-ITSL equilibrium conversion (ϵ_{eq}) was higher than RITSL by 10-40%, based on a catalyst weight basis. It indicated that CC-ITSL may require less catalyst inventory and less reactor size for fixed TSL performance with the same catalyst addition/withdrawal rate. The following equation is used to convert from a catalyst weight basis to a catalyst volume basis with the different catalyst densities and catalyst charge volumes:

$$\left(\frac{K_{\text{Amocat}}}{K_{\text{Shell}}} \right) \text{ volume} = 0.82 \left(\frac{K_{\text{Amocat}}}{K_{\text{Shell}}} \right) \text{ weight}$$

CC-ITSL equilibrium conversion (ϵ_{eq}) was again higher than RITSL by 10-20 %, based on a catalyst volume basis.

Amocat 1C catalyst showed higher equilibrium conversion (ϵ_{eq}) compared to Shell 324 catalyst, by 20% based on a catalyst weight basis, and by 10% based on a catalyst volume basis. CC-ITSL with the Amocat 1C catalyst may require less catalyst inventory and

less reactor size for fixed TSL performance with the same catalyst replacement rate, compared to CC-ITSL with the Shell 324 catalyst.

The projected catalyst replacement rate of Amocat 1C in CC-ITSL (no solids recycle) for an "all-distillate" product slate was less than 1 lb cat/ton MF coal at a 750°F HTR reaction temperature and a WHSV of 2.1 hr⁻¹. Results are summarized below:

Set operating conditions:

- WHSV = 2.1
- catalyst inventory = 340 lb
- catalyst aging rate = 30 lb/lb cat/day

	<u>Catalyst</u> (lb/ton coal)	<u>Temperature, (°F)</u>	<u>K_{eq} (hr⁻¹)</u>	<u>ε_{eq} (%)</u>
Case I	1.0	720	0.416	16.5
Case II	1.0	740	0.579	21.6
Case III	1.0	750	0.681	24.5
Case IV	0.75	750	0.622	22.9
Case V	0.5	750	0.527	20.1

1st stage catalyst (Amocat 1A) deactivation trends (overall, catalytic and thermal rate constants) in the CC-ITSL catalytic-catalytic mode of operation (Run 251-I) with Illinois No. 6 Burning Star Mine coal are plotted in Figure 24. The first stage reaction temperature selected is 805°F. The thermal rate constant (K_T) was calculated with limited data available during Run 250CDE, in order to compare projected catalyst replacement rates with both overall (K) and catalytic (K_C) rate constants. Results were not significantly different, as shown below:

Assuming the thermal reaction rate constant at 805°F (0.58 hr⁻¹ from Run 250CDE) is not dependent on the catalyst age, then the catalytic reaction rate constants at 805°F can be assumed as follows:

$$\text{Catalyst age} \quad (0-100) \quad K_C = K - \frac{\text{WHSV}}{\text{SV}} K_T$$

$$K_C = 4.24 (e^{-0.00493t}) - 0.58$$

$$(100-2800) \quad K_C = 2.68 (e^{-0.00034t}) - 0.58$$

Although $\ln K_C$ equations are non-linear, approximate linear equations are used for projected catalyst replacement calculations.

$$\text{approximate equation} \quad (0-100) \quad K_C = 3.66 e^{-0.006t}$$

$$(100-2800) \quad K_C = 2.10 e^{-0.0005t}$$

Projected catalyst replacement rates in the 1st stage were similar for both with overall (K) and catalytic (K_C) rate constants. Run 251-IAF at two different WHSV (2.7 and 4.2 hr^{-1}) was selected for calculations as tabulated as follows:

<u>Run</u>	<u>WHSV</u> <u>hr⁻¹</u>	<u>Rate constant</u> <u>used</u>	<u>Target Resid + UC</u> <u>conversion, wt % feed</u>	<u>Projected catalyst</u> <u>replacement</u> <u>lb/ton coal</u>
251-IAF	2.7	overall(K)	33.8	1.5
		catalytic(K_C)	22.9	1.3
	4.2	overall(K)	31.5	3.2
		catalytic(K_C)	24.3	3.3

(A) WHSV = 2.7

<u>Projected</u> <u>catalyst</u> <u>replacement</u> <u>(lb/ton coal)</u>	<u>Temperature (°F)</u>	<u>K_C eq (hr⁻¹)</u>	<u>ϵ_C eq (wt %)</u>
Case I	1.0	0.689	20.3
Case II	1.3	0.821	23.3
Case III	1.5	0.894	24.9

(B) WHSV = 4.2

<u>Projected</u> <u>catalyst</u> <u>replacement</u> <u>(lb/ton coal)</u>	<u>Temperature (°F)</u>	<u>K_C eq (hr⁻¹)</u>	<u>ϵ_C eq (wt %)</u>
Case I	3.0	1.294	23.6
Case II	3.5	1.379	24.7

Catalytic-catalytic mode (251-IAF) catalyst batch deactivation trends are compared to the previous thermal-catalytic mode with solids recycle (250FGH) in Figure 25, using overall rate constants. Catalyst deactivation of Amocat 1C in the 2nd stage in the catalytic-catalytic mode was insignificant and much smaller than in the thermal-catalytic mode. This low deactivation in the 2nd stage is probably due to removal of catalyst deactivation and poison material in the coal slurry feed by employing a catalyst in the 1st stage.

Activation energy (E), frequency factor (A), deactivation coefficient (a), and overall rate constant (K) were calculated and are summarized as follows:

Run	Solids recycle	Stage	Catalyst	Freq. factor A(hr ⁻¹)	Act. energy E(Btu/lb-mole)	Deact. coeff. α
250FGH	Yes	1st	None	-	-	-
		2nd	Amocat 1C	3.98×10^7	42,500	2.4×10^{-4}
251-IAF	Yes	1st	Amocat 1A	1.32×10^{10}	56,100	3.4×10^{-4}
		2nd	Amocat 1C	3.01×10^7	42,300	5.0×10^{-5}

Rate constant (K) equation

Run	Temperature, °F	Catalyst age 1b(res+CI)/1b cat	Equation for K(hr ⁻¹)
250FGH 760 (2nd stage)		0-100	$K = 3.58 e^{-0.0134t}$
		100-1700	$K = 0.96 e^{-0.00024t}$
251-IAF 805 (1st stage)		0-100	$K = 4.24 e^{-0.00493t}$ (assumed)
		100-2800	$K = 2.68 e^{-0.00034t}$
251-IAF 760 (2nd stage)		0-100	$K = 1.29 e^{-0.00493t}$ (assumed)
		100-2800	$K = 0.80 e^{-0.00005t}$

Projected catalyst replacement rates for "all-distillate" product slates were calculated with both catalytic-catalytic mode and thermal-catalytic mode, and are summarized below:

Catalyst addition rate for "all-distillate" slate

Run	Base Data	Stage	Catalyst	WHSV, hr ⁻¹	TSL C ₄ + dist wt % MAF	Projected catalyst replacement lb/ton coal
250FGH	250G	1st(thermal)	None	-	68	-
		2nd(catalytic)	Amocat 1C	2.3		2.9
251-IAF	251-IE	1st(catalytic)	Amocat 1A	2.7	70	1.5
		2nd(catalytic)	Amocat 1C	2.3		0.15
251-IAF	251-IC	1st(catalytic)	Amocat 1A	4.2	67	3.2
		2nd(catalytic)	Amocat 1C	3.8		0.7

(A) Run 250FGH (2nd stage - Amocat 1C)

- WHSV = 2.3
- Catalyst inventory = 340 lbs
- Catalyst aging rate = 30 lb (resid + CI)/lb cat/day
- Base data = 250G: 2nd stage conversion = 27.3 wt % feed
TSL C₄₊ dist = 68 wt % MAF
Organic rejection = 15 wt % MAF

Projected
catalyst
replacement

	<u>(lb/ton coal)</u>	<u>Temperature (°F)</u>	<u>K_{eq} (hr⁻¹)</u>	<u>ε_{eq} (wt %)</u>
Case I	1.0	760	0.59	20.3
Case II	2.0	760	0.77	25.1
Case III	2.9	760	0.86	27.3
Case IV	3.0	760	0.87	27.5
Case V	4.0	760	0.94	29.1

(B) Run 251-IAF (1st stage - Amocat 1A)

- WHSV = 2.7
- Catalyst inventory = 340 lb
- Catalyst aging rate = 45 lb (resid + CI)/lb cat/day
- Base data = 251-IE: 1st stage conversion = 33.8 wt % feed
TSL C₄₊ dist = 70 wt % MAF
Organic rejection = 15 wt % MAF

Projected
catalyst
replacement

	<u>(lb/ton coal)</u>	<u>Temperature (°F)</u>	<u>K_{eq} (hr⁻¹)</u>	<u>ε_{eq} (wt %)</u>
Case I	1.0	805	1.113	29.2
Case II	1.5	805	1.390	34.0
Case III	2.0	805	1.589	37.1

Run 251-IAF (2nd stage - Amocat 1C)

- WHSV = 2.3
- Catalyst inventory = 340 lb
- Catalyst aging rate = 30 lb (resid + CI)/lb cat/day
- Base data = 251-IE: 2nd stage conversion = 14.6 wt % feed
TSL C₄₊ dist = 70 wt % MAF
Organic rejection = 15 wt % MAF

Projected catalyst replacement (lb/ton coal)	Temperature (°F)	K _{eq} (hr ⁻¹)	ϵ_{eq} (wt %)
Case I	0.15	760	0.413
Case II	0.25	760	0.513
Case III	0.5	760	0.62
Case IV	1.0	760	0.708

(C) Run 251-IAF (1st stage - Amocat 1A)

- WHSV = 4.2
- Catalyst inventory = 340 lb
- Catalyst aging rate = 70 lb (resid + CI)/lb cat/day
- Base data = 251-IC: 1st stage conversion = 31.5 wt % feed
TSL C₄₊ dist = 67 wt % MAF
Organic rejection = 18 wt % MAF

Projected catalyst replacement (lb/ton coal)	Temperature (°F)	K _{eq} (hr ⁻¹)	ϵ_{eq} (wt %)
Case I	805	1.132	21.2
Case II	805	1.608	27.7
Case III	805	1.758	29.5
Case IV	805	1.876	30.9
Case V	805	1.971	31.9

Run 251-IAF (2nd stage - Amocat 1C)

- WHSV = 3.8
- Catalyst inventory = 340 lb
- Catalyst aging rate = 50 lb (resid + CI)/lb cat/day
- Base data = 251-IC: 2nd stage conversion = 14.9 wt % feed
TSL C₄+ dist = 67 wt % MAF
Organic rejection = 18 wt % MAF

Projected catalyst replacement (lb/ton coal)	Temperature (°F)	K _{eq} (hr ⁻¹)	ϵ_{eq} (wt %)
Case I	0.5	760	0.621
Case II	0.75	760	0.674
Case III	1.0	760	0.705
Case IV	2.0	760	0.760

At a coal feed rate of 280-300 MF lb/hr, the catalytic-catalytic mode (251-IAF) required less catalyst addition/withdrawal rates than the thermal-catalytic mode, that is, 1.7 versus 4.5 lb/ton coal. This lower catalyst requirement in the catalytic-catalytic mode is mainly due to high catalyst activity of Amocat 1A in the 1st stage, low catalyst deactivation for both 1st and 2nd stage catalysts, and enhanced hydrogen transfer in the 1st stage because of high catalytic rehydrogenation of recycle process solvent.

Projected catalyst requirement at a higher coal feed rate of 480 MF lb/hr in the catalytic-catalytic mode was 2.3 times higher (3.9 vs 1.7 lb/ton coal) than at a coal feed rate of 300 MF lb/hr.

Detailed data for catalyst characterization and analytical data were reported in Technical Progress Report Run 250 (Reference 13) and Technical Progress Report Run 251 (Ref. 14).

The CSTR 1st order kinetics model was developed from data generated in the ITSL configuration (thermal-catalytic mode). This model was also successfully applied in the CC-ITSL catalytic-catalytic configuration in Run 251-I (Ref. 14). To further verify the model in the CC-ITSL configuration, a parity plot of estimated coal feed rates against actual experimental coal feed rates was developed for periods 251-IB, IC, ID and IE, and is illustrated in Figure 26. Comparison results are summarized in Table 13. This comparison study shows that the CSTR 1st order kinetics model represents very well the experimental data with less than 13% error (relative) in the coal feed rate. Periods 251-ID and IE have high errors, 11-13% (relative), probably due to the transitional period for 251-ID and the lower experimental activity in the second stage than the

model predicted for 251-IE. Zero-time responses due to the coal space velocity changes experimentally measured in periods 251-IC, ID and IE follow the CSTR 1st order kinetics model (Figure 27).

Figures 28-31 show calculated equilibrium catalyst age distributions (RTD) for Runs 253JK/244B, 250G and 251-IC. The age distribution is dependent on the catalyst replacement rate. The equilibrium catalyst activity (K_{eq}) with catalyst replacement also varies due to the age distribution. Projected catalyst requirements discussed in this report are calculated by using K_{eq} for the commercial plant operation simulation. This projection is different from the replacement to achieve the steady-state operation, as compared to the actual plant operation.

Run	Coal feed MF lb/hr	Stage	Catalyst age lb MF coal/lb cat	Steady-state catalyst addition lb/ton MF coal	Projected catalyst replacement at equilibrium lb/ton MF coal
250G	280	2nd	236	7.3	2.9 ± 0.3
251-IE	300	1st	1098	1.8	1.5 ± 0.1
		2nd	1504	<u>1.3</u>	<u>0.2 ± 0.1</u>
		total		<u>3.1</u>	<u>1.7 ± 0.2</u>
251-IC	480	1st	763	2.4	3.2 ± 0.2
		2nd	1168	<u>1.7</u>	<u>0.7 ± 0.2</u>
		total		<u>4.1</u>	<u>3.9 ± 0.4</u>

The catalyst addition rate based on the every other day addition practiced at the plant was calculated to achieve the steady-state operation at a constant catalyst age measured for each period. It is quite obvious that this addition is different from the projected replacement at equilibrium. This addition does not account for the age distribution at equilibrium (Figures 28-30) the average activity at equilibrium (K_{eq}) and the deactivation slope (α). One might easily visualize that when operated at this addition rate the process performance is different from the measured for each period. The projected catalyst replacement at equilibrium was calculated to achieve the same performance measured for each period. The projection better represents the catalyst requirement for the commercial plant operation in each period tested at the plant.

The projected catalyst replacement at equilibrium for Run 251-IE is 0.2 ± 0.1 lb/ton MF coal in the second stage. This value seems unrealistically low, probably due to the rate constant equation developed based on limited number of data points measured in Run 251. Future runs are recommended to test at steady-state operation with catalyst replacement to verify the model used in calculation of the projected catalyst replacement at equilibrium. This test requires lengthy operation to investigate the impact of the catalyst replacement on the process performance, i.e., yield structure, etc. Sensitivity studies are also suggested to better understand the effects of catalyst activity level and deactivation coefficient on the catalyst replacement.

V. CONCLUSIONS AND RECOMMENDATIONS

CONCLUSIONS

The following conclusions were drawn from the TSL data configuration analysis presented in this report.

1. Processing Illinois No. 6 Burning Star Mine coal, the CC-ITSL catalytic-catalytic mode of operation with solids recycle produced the highest C₄₊ distillate, 70 wt % MAF coal. Hydrogen efficiency declined slightly due to the high hydrogen consumption required to produce high C₄₊ distillate, compared to the ITSL configuration. The organic rejection was the lowest, 15 wt % MAF coal, probably due to the solids recycle. The C₄₊ naphtha yield was the highest, 19 wt % MAF coal. The C₄₊ distillate product was composed of 27 wt % C₄₊ naphtha, 11 wt % middle distillate and 62 wt % distillate solvent. Distillable product quality was very good, having a high hydrogen content and low levels of nitrogen and sulfur. Good product quality was accompanied by high hydrogen consumption and a slight decline of hydrogen efficiency. Therefore, the high hydrogen consumption appears to be a trade off with improved product quality. "All-distillate" product slate was produced at high coal space rates, without lowering distillable product quality. Catalyst requirements were low because of high catalyst activity, low catalyst deactivation rates and performance improvements by employing catalyst in both stages.
2. Processing Wyodak Clovis Point Mine coal, the CC-ITSL thermal-catalytic mode of operation with solids recycle produced the highest C₄₊ distillate, 61 wt % MAF coal. Hydrogen efficiency was similar to that of other configurations. The organic rejection was the lowest, 9 wt % MAF coal, due to the solids recycle and the CC-ITSL configuration. The C₄₊ naphtha yield was the highest, 20 wt % MAF coal. The C₄₊ distillate product was composed of 33 wt % C₄₊ naphtha, 16 wt % middle distillate and 51 wt % distillate solvent. "All distillate" product slate was achieved while operating with space velocity 40% higher than in the RITSL configuration. The higher reaction temperature and the younger catalyst age could affect the process improvements in the CC-ITSL configuration, although operated at 40% higher space velocity. Good TSL process performance, high C₄₊ distillate, high hydrogen efficiency and low organic rejection are due to the CC-ITSL configuration and high catalyst activity of the bimodal Amocat 1C catalyst in the 2nd stage.

3. Solids recycle significantly affects TSL system process performance characteristics with both Illinois No. 6 and Wyodak coal; reducing the hydrogen efficiency and decreasing the organic rejection. The C₁-C₃ gas selectivity is not affected significantly.
4. A high hydrogen efficiency was observed in the ITSL configuration with Illinois No. 6 coal (11.3 lb C₄₊ dist/lb H₂) and in the DITSL configuration with Wyodak coal (10.2 lb C₄₊ dist/lb H₂). However, both ITSL and DITSL configurations produced less C₄₊ distillate than the CC-ITSL catalytic-catalytic mode with Illinois No. 6 coal and the CC-ITSL thermal-catalytic mode with Wyodak coal. The ITSL-LCT configuration with the iron oxide addition processing Illinois No. 6 coal produced the highest C₄₊ distillate in ITSL, 64 wt % MAF coal, with a high hydrogen efficiency.
5. The process configuration affects the TSL distillate product distribution more with Wyodak coal than with Illinois No. 6 coal. With Illinois No. 6 coal, the catalyst addition or charge in the 1st stage such as the iron oxide addition in the coal slurry, solids recycle and the use of an active hydrotreating catalyst in the CC-ITSL catalytic-catalytic mode are more important factors than the process configuration. Processing Wyodak coal, DITSL and ITSL configurations produce more middle distillate and the CC-ITSL configuration produces more C₄₊ naphtha, compared to other configurations. Processing Illinois No. 6 coal, the catalyst addition or charge in the 1st stage increases the C₄₊ naphtha yield by enhancing catalytic hydro-cracking of distillate products.
6. High hydrogen transfer from process solvent to coal in the 1st stage improves TSL hydrogen efficiency.
7. Solvent quality of process solvent is a function of hydrogen contents of resid and distillate portions of recycle process solvent. Optimum hydrogen content of process solvent for high solvent quality may be different with different coals.
8. The maximum hydrogen efficiency can be achieved at around 60% 1st stage contribution in TSL C₄₊ distillate products, processing Illinois No. 6 coal. The CC-ITSL catalytic-catalytic mode of operation showed a relatively low hydrogen efficiency with a high 1st stage contribution (81%).
9. CC-ITSL and RITSL configurations improve distillable product quality by consuming more hydrogen, compared to the ITSL configuration. CC-ITSL and RITSL configurations produce a high quality "all-distillate" product slate with a high hydrogen content and low levels of nitrogen and sulfur.

RECOMMENDATIONS

Future study areas for potential improvements in the process performance and economics and to expand the coal liquefaction technology data-base include:

1. Improve the hydrogen efficiency in the CC-ITSL catalytic-catalytic mode of operation processing Illinois No. 6 bituminous coal, by determining optimum process operating conditions. This would include the 1st stage reaction temperature, coal space rate, feed space velocity, hydrogen partial pressure, 2nd stage reaction temperature, catalyst activity and deactivation, process solvent activity and hydrogen transfer of recycle process solvent in the 1st stage.
2. Expand the data base of both the CC-ITSL thermal-catalytic and catalytic-catalytic modes of operation with Wyodak subbituminous coal, in order to determine optimum operating conditions for high hydrogen efficiency.
3. Study the CSD performance with solids recycle to reduce the organic rejection below 10 wt % MAF coal with Illinois No. 6 coal.
4. Test alternate coals such as bituminous, subbituminous, lignite and cleaned coal in both CC-ITSL thermal-catalytic and catalytic-catalytic mode of operation in order to generalize the TSL system process performance.
5. Test inexpensive and better catalysts with high catalyst activity and selectivity to reduce plant design and operation costs for commercialization.
6. Develop TSL system process performance models to allow accurate predictions of the system response to changes in process variables and the concurrent effect on the process economics.
7. Improve reaction system configurations.
8. Develop alternate deashing processes.
9. Determine optimum catalyst requirements employing catalyst cascading at two-stage equilibrium activity levels.
10. Eliminate the interstage separation and test low reaction system pressures for the capital cost reduction.
11. Reduce the solvent-to-coal ratio and eliminate ash recycle.

12. Increase reactive resid recycle for distillate production improvement.
13. Increase gas-oil recycle for the improved distillate product quality.
14. Experiment with Wyodak subbituminous coal at high coal throughputs and at high catalyst ages with a high slurry drying capacity to expand the process database.
15. Increase second stage reaction temperature to improve catalyst activity.

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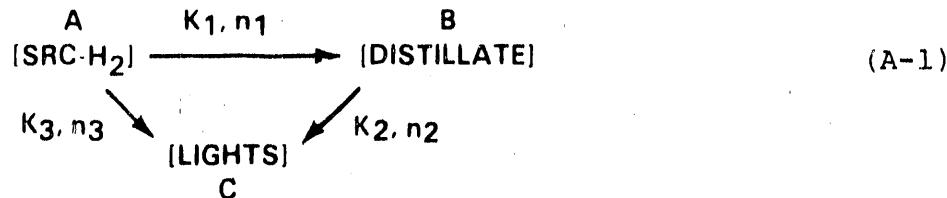
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APPENDIX A
Catalyst Activity and Deactivation
Theoretical Background (Ref. 23)

A. LUMPED KINETICS (Ref. A-1)

Normally, when complex feedstocks are involved in any reaction, describing the kinetics of each individual species can be a monumental task. One effective method of simplifying this problem is to group (or "lump") many of the species and then develop reaction models on this basis. It must be recognized that this so called simplified basis can, in principle, be developed to any desired level of sophistication and complexity. The degree of refinement of a lumped model is dictated by the final objectives of the research, the quality and quantity of available data, and available human and computer resources.

For the purposes of this study, a simple three-lump model is proposed. SRC (+850°F), distillates (350°F-EP), and light-ends were defined as the three basic pseudo species, and the reaction mechanism was assumed to be as follows:



In the above series/parallel reaction, k_1 , k_2 , and k_3 are the reaction velocity constants, and n_1 , n_2 , and n_3 are the orders of reaction. These constants are defined in general terms by the following differential equation:

$$-r = \frac{dC}{dt} = kC^n, \quad (\text{A-2})$$

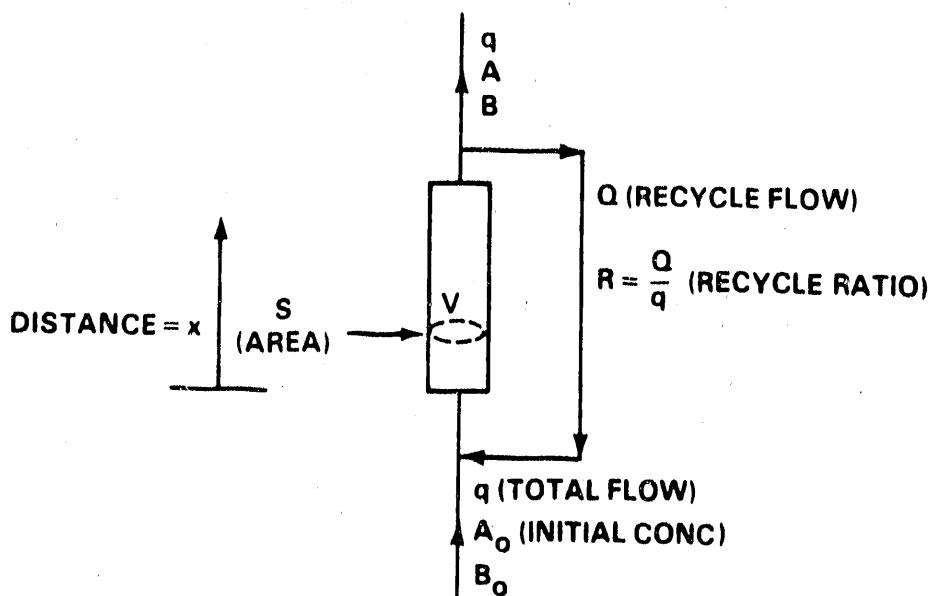
where r is the reaction rate or the differential change in concentration, C , with respect to time, t .

The above mentioned equations describe the reaction mechanism and kinetics. However, this in itself does not provide the full picture. Mass and energy balances around the reactor provide the

basis for relating the production rate and chemical composition of the products to the chemical reaction rate. We do this by postulating an ideal reactor that we believe closely approximates the real system.

B. EQUATIONS OF CONTINUITY (Ref. A-2)

The simplest of the ideal reactor models that describes an ebullated bed system is an isothermal recycle reactor shown below.



The basic equations for a recycle reactor are:

$$\frac{Q + q}{S} \frac{dC_A}{dx} = - k_1 C_A^{n_1} - k_3 C_A^{n_3} \quad (A-3)$$

$$\frac{Q + q}{S} \frac{dC_B}{dx} = + k_1 C_A^{n_1} - k_2 C_B^{n_2} \quad (A-4)$$

The above equations describe the differential change in concentration of species A and B along the length, x , of the reactor in terms of the kinetic constants defined earlier. In an integrated form, these equations define the production (or yield) of species B and disappearance (or conversion) of species A.

In order to integrate the above equations explicitly, the following assumptions were made:

<u>Assumptions</u>	<u>Implications</u>
(a) $n_1 = n_2 = n_3 = 1$	First order kinetics
(b) $k_3 = 0$	Parallel reaction neglected
(c) $k_1 \neq k_2 \neq 0$	Series reaction
(d) Space rate replaces residence time, V_R/q	Constant density, single phase and uniform catalyst distribution.
(e) Recycle ratio = $R = Q/q$	

Based on these assumptions, the following forms of integrated conversion models were derived:

IF $R \neq 0 \& < \infty$ (RECYCLE REACTOR)

$$\epsilon = \text{CONVERSION} = \left(1 - \frac{A}{A_0}\right) = 1 - \frac{\exp\left(-\frac{k_1}{WHSV(R+1)}\right)}{(R+1) - R \exp\left(-\frac{k_1}{WHSV(R+1)}\right)} \quad (A-5)$$

IF $R = 0$ (PLUG FLOW)

$$\epsilon = 1 - \exp\left(-\frac{k_1}{WHSV}\right) \quad (A-6)$$

IF $R = \infty$ (STIRRED TANK)

$$\epsilon = \frac{k_1}{k_1 + WHSV} \quad (A-7)$$

Equation (A-5) is the model for a recycle reactor. In the limiting form, when R becomes too small, the recycle reactor approaches a plug flow system [Equation (A-6)]. If R is too large, the recycle reactor approaches a stirred tank [Equation (A-7)].

Figure A-1 is a parametric plot of conversion as a function of space velocity for various values of R and assuming k is 1.12. This plot shows that an isothermal plug-flow reactor is inherently more efficient in terms of conversion than an isothermal back mix reactor. However, with an increasing recycle ratio, the performance of a plug-flow reactor rapidly approaches that of the back mix reactor. At the recycle ratios of the Wilsonville reactor, the assumption of complete back mixing would not introduce any significant error. This assumption was also experimentally confirmed for the ebullated H-Coal catalyst bed by Bickel and co-workers at Sandia National Laboratories (Ref. A-3). Hence, use of Equation (A-7) in the correlation of conversion data is justified. Equation (A-7) can be rewritten as:

$$k = WHSV \frac{\epsilon}{1-\epsilon} \quad (A-8)$$

Equation A-8 can be used to calculate the reaction velocity constants from the observed data on space velocity versus SRC conversion.

C. TEMPERATURE DEPENDENCE (Ref. A-4)

For many reactions, the rate expression [Equation (A-2)] can be written as a product of a temperature-dependent term, k , and a composition dependent term, C^n . For such reactions, k has been found to be well represented by Arrhenius law:

$$k = A \exp (-E/RT), \quad (A-16)$$

where E is the activation energy and A is the frequency factor. This expression has been experimentally validated for many reactions and over a wide range of temperature.

Equation (A-16) forms the basis of interpolation of the data along the temperature coordinate. This equation implies that an increase in temperature always increases the reaction velocity constant.

Equation (A-16) can be modified to describe the temperature dependency of the selectivity parameter as follows:

$$k_2/k_1 = (A_2/A_1) \exp (-(E_2-E_1)/RT) \quad (A-17)$$

Unlike the reaction velocity constants, the selectivity parameter can decrease or increase with increases in temperature depending upon whether $E_1 > E_2$, or $E_2 > E_1$.

D. CATALYST DEACTIVATION (Ref. A-4)

In most catalytic reactions, the activity of the catalyst decreases as it is being used. A generalized model for catalyst deactivation was proposed by Levenspiel as follows:

$$\phi = \frac{-r_A}{r_{A0}} = \frac{\text{rate at which catalyst converts A}}{\text{rate of reaction of A with fresh pellet}} \quad (A-18)$$

In terms of nth-order kinetics and Arrhenius temperature dependency, and isothermal catalyst pellet, reaction rate for a given pellet can be written as:

$$-r_A = k_C_A^n = k_0 \exp (-E/RT) \cdot \phi \cdot C_A^n \quad (A-19)$$

and for deactivation which in general is dependent on concentration of a species i :

$$-\frac{d\phi}{dt} = \alpha \cdot C_i^{n_1} \cdot \phi^D = \alpha_0 \exp(-E_D/RT) \cdot C_i^{n_1} \cdot \phi^D \quad (A-20)$$

where α_0 is the decay coefficient, D is the order of deactivation, n_1 measures the concentration dependence and E_D is the activation energy or temperature dependency of deactivation.

Assuming concentration independent, first-order deactivation, Equation (A-20) can be written as:

$$\frac{-d\phi}{dt} = \alpha \cdot \phi, \quad (A-21)$$

Integrating Equation (A-21) for unit initial decay function, or ϕ_0 equal to 1, this becomes:

$$\phi = \exp(-\alpha t), \quad (A-22)$$

Hence, from Equation (A-19),

$$k = k_0 \exp(-E/RT) \cdot \exp(-\alpha t) \quad (A-23)$$

The above equation relates the reaction velocity constant to time and temperature.

APPENDIX A REFERENCES

- A-1 Weekman, V. W., Jr., "Lumps, Models and Kinetics in Practice," AIChE Monograph Series, Vol. 75, No. 11 (1979).
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- A-3 Bickel, T. C., and Thomas, M. G., "Catalyst Deactivation in H-Coal Coal Liquefaction Process - Catalyst Residence Time Distribution," Ind. Eng. Chem. Process Des. Dev., Vol. 21, p. 377 (1982).
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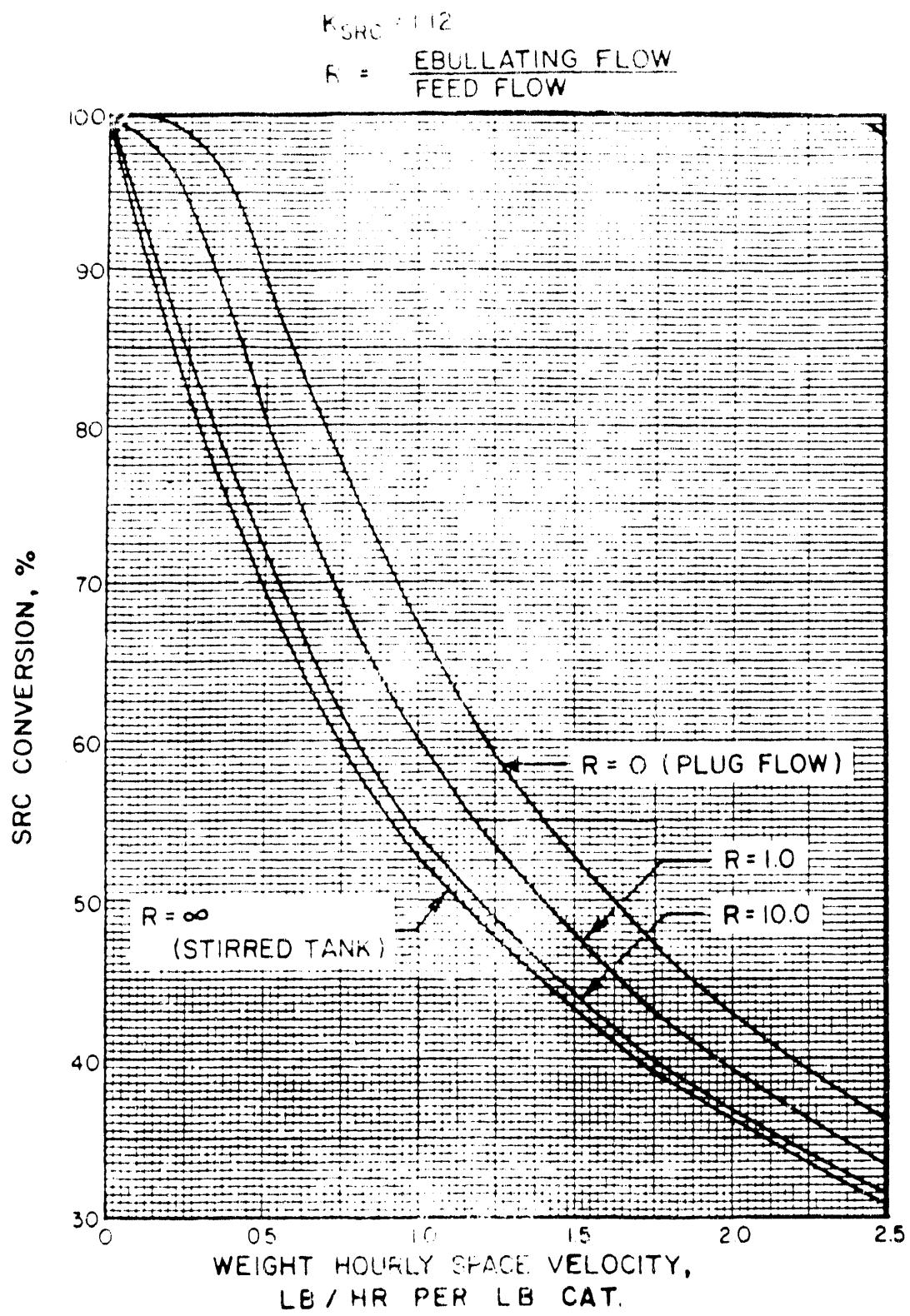


FIGURE A-1. EFFECT OF EBULLATION ON REACTOR HYDRODYNAMICS

APPENDIX B

Microautoclave Activity Test Descriptions

Solvent Quality

Solvent quality is determined in the Wilsonville laboratory as follows:

Standard coal (Indiana V) and solvent are charged to a 30 cc microautoclave reactor unit to which a mixing ball is added. During ash recycle, the solvent contains ash and unconverted coal. These are removed by cresol extraction before the solvent and coal are finally mixed. The slurry is mixed and heated to a specific temperature ($\pm 5^{\circ}\text{F}$) within a two minute period and is maintained for a specific period of time. The microautoclave is quenched in water and the reaction products are washed and extracted with tetrahydrofuran (THF).

The ratio of the amount of reacted coal to the original sample weight is expressed as a percentage conversion which is referred to as "solvent quality".

The kinetic test gives a relative indication of the hydrogen transfer rate and hydrogen shuttling ability of the solvent. the equilibrium test gives a relative indication of the concentration of donatable hydrogen in the solvent.

The conditions used for both microautoclave tests are listed below:

<u>Test Type</u>	<u>Temp, °F</u>	<u>Solvent-to- coal ratio</u>	<u>Reaction time, min.</u>
Kinetic	750		10
Equilibrium	750	2:1	30

Catalyst Activity

Catalyst activity is determined in the Wilsonville laboratory as follows:

The catalyst sample is washed thoroughly with toluene, then tetrahydrofuran (THF). The washed sample is then sonicated in THF for two hours. Fresh THF is added to the sample after sonication. A constant catalyst volume (equivalent to 7.0 inches when stacked in a capillary tube) is used for the test.

Two grams of 10% naphthalene in hexadecane is catalytically hydrogenated in a microautoclave reactor at the following conditions:

Temperature, °F	720
Hydrogen pressure, psig	1,000 (cold)
Agitation, strokes/min	800 (no ball or rod added)
Reaction time, min	15

The reactor is quenched with cold water and the contents are filtered. The concentrations of tetralin and decalin, which are the products of naphthalene hydrogenation, and naphthalene are determined by gas chromatography. The hydrogen consumption is then determined by stoichiometric calculations.

The test determines hydrogen consumption during the hydrogenation of a model compound, naphthalene, in the presence of the catalyst. This gives an indication of relative catalyst activity, independent of HTR reactor and/or TSL system performances.

The test also may be used for measuring the completeness of catalyst sulfiding.

APPENDIX C

Nomenclature and Definitions

All-distillate slate	The product yields with resid extinction.
Ash	Non-organic material obtained by muffle furnace burning at 800°C for 4 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 residue and solvent.
Asphaltenes	A benzene-soluble and pentane-insoluble product of the coal liquefaction process which is non-distillable at 600°F and 0.1 mm Hg in the laboratory.
CC-ITSL	Close Coupled Integrated Two-Stage Liquefaction
CSD feed solvency index	Laboratory analysis for fraction of CSD feed soluble in actual deashing solvent compared to solubility in a solvent standard.
Deashing solvent (DAS)	A solvent used to deash the vacuum column bottoms which is fed to the CSD unit.
Distillate solvent	A coal-derived distillate fraction boiling above 450°F that is distillable at 600°F at 0.1 mm Hg in a laboratory batch distillation apparatus.
DITSL	Double Integrated Two-Stage Liquefaction
DMDS	Dimethyl disulfide
Energy rejection	The heating value lost to the ash concentrate as a fraction of the feed coal heating value.
ITSL	Integrated Two-Stage Liquefaction
LCT	Low Contact Time

APPENDIX C (conitnued)

Names, Nature and Definitions

LTR	Light Thermal Resid
MAF	Moisture and Ash Free
MB Period	Material Balance Period
MF	Moisture Free
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 an IBP-350°F boiling range at 760 mm HG (GC and ASTM D-86).
Oils	A pentane-soluble product of the coal liquefaction process which is non-distillable at 600°F and 0.1 mm Hg in the laboratory.
Practical resid extinction	Plant resid extinction operations are achieved with + or - 2 wt % MAF coal resid yield.
Preasphaltenes	A cresol-soluble and benzene-insoluble product of the coal liquefaction process which is non-distillable at 600°F and 0.1 mm Hg in the laboratory.
Process solvent	Feed solvent to the TL unit which is normally a blend of distillate solvent and resid in variable concentrations.
Reactor exotherm	The reactor temperature increase due to exothermic reactions is measured by the difference between inlet and outlet reactor temperatures.
Resid	A cresol-soluble product of the coal liquefaction process which is non-distillable at 600°F and 0.1 Hg in the laboratory.

APPENDIX C (continued)

Nomenclature and Definitions

Resid conversion	The fraction of the resid feed to the HTR unit that is converted to gases and liquids:
	$\text{Percent conversion} = \frac{\text{Resid in} - \text{resid out}}{\text{resid in}} \times 100$
Resid recovery	The percent of CSD feed resid that is recovered in the deashed resid and is not lost to the ash concentrate.
RITSL	Reconfigured Integrated Two-Stage Liquefaction
TR	Thermal Resid
T102 Vacuum Column bottoms	A nonvolatile mixture of resid, distillate solvent, ash, and UC.
Unconverted Coal (UC)	Organic material that is insoluble in hot cresol.
WHSV	Weight Hourly Space Velocity (HTR), lb/hr feed per lb catalyst.

APPENDIX D

Table 1

FEED COAL ANALYSES (ILLINOIS NO. 6)

Run no.	Date	241CD	242BC		243JK/244B		244CD		245BCDF		247D			
			12/2/82-1/2/83	ITSL SCF	5/29-7/21/83	ITSL	7/28-8/15/83	ITSL	12/2/83-1/8/84	ITSL CAT A/W	11/1-9/84	ITSL		
<u>Proximate analysis, wt. %</u>														
<u>Volatile matter</u>														
Fixed carbon	37.24	36.16	35.20		35.79		36.48		35.63		49.23			
Ash	49.47	50.49	50.29		50.00		49.91		49.38		10.98			
Moisture	10.94	10.38	10.78		10.10		10.11		4.16		4.16			
	2.36	2.98	3.73		4.00		3.49							
<u>Ultimate analysis, wt. %</u>														
Carbon	68.25	68.27	68.97		70.46		70.00		70.34					
Hydrogen	4.47	4.49	4.62		4.68		4.64		4.85					
Nitrogen	1.30	1.39	1.32		1.32		1.37		1.19					
Sulfur	3.50	3.33	3.30		3.35		3.37		3.55					
Chlorine	0.06	0.11	0.03		0.05		0.07		0.07					
Ash	11.20	10.69	10.98		10.61		10.46		11.46					
Oxygen (by difference)	11.24	11.78	11.62		11.85		10.12		8.54					
N/C atomic ratio	0.78	0.79	0.79		0.79		0.79		0.79		0.83			
Dry heating value, Btu/lb	12,525	12,660	12,384		12,634		12,646		12,605					
Sulfur forms, wt. %														
Pyrite	1.14	1.09	1.24		1.01		1.16		1.10					
Sulfate	0.11	0.12	0.05		0.14		0.17		0.15					
Sulfide	<0.01	0.01	<0.01		0.01		<0.01		<0.01					
Organic	2.15	2.22	2.09		2.19		2.07		2.30					
<u>Mineral analysis, wt. % (ignited basis)</u>														
Phos. pentoxide, P_2O_5	0.05	0.07	0.07		0.06		0.05		0.05		0.05			
Silica, SiO_2	48.21	49.74	48.23		48.40		47.39		48.00					
Ferric oxide, Fe_2O_3	16.52	17.30	17.79		18.58		18.41		17.73					
Alumina, Al_2O_3	17.50	17.74	18.67		19.03		18.28		17.49					
Titania, TiO_3	0.96	0.81	0.96		0.93		0.90		0.86					
Lime, CaO	7.26	6.13	6.34		5.25		5.89		5.66					
Magnesia, MgO	0.97	0.74	0.87		0.79		0.84		0.92					
Sulfur trioxide, SO_3	4.41	5.09	3.71		4.35		5.10		7.54					
Potassium oxide, K_2O	1.92	1.87	1.77		2.05		1.84		1.93					
Sodium oxide, Na_2O	0.76	0.54	0.48		0.54		0.43		0.68					
Vanadium, V_2O_5	0.05	0.05	0.04		0.04		0.03		0.03					
Undetermined	1.46	0.17	1.08		0.01		1.77		—					

Table 1 (continued)

Run no.	248A	248D	250B	250C
Date	3/10-17/85	4/8-11/85	1/11-16/86	2/2-5/86
Configuration	DITSL	ITSL	CC-ITSL	CC-ITSL
<u>Proximate analysis, wt. %</u>				
Volatile matter	36.46	35.47	36.24	35.75
Fixed carbon	50.06	48.68	50.19	50.47
Ash	10.71	10.45	10.62	11.18
Moisture	2.77	5.40	2.95	2.60
<u>Ultimate analysis, wt. %</u>				
Carbon	70.53	70.18	70.12	70.22
Hydrogen	4.93	4.58	4.61	4.70
Nitrogen	1.38	1.09	1.13	1.35
Sulfur	3.68	3.79	3.74	3.75
Chlorine	0.05	0.08	0.07	0.05
Ash	11.02	11.05	10.94	11.91
Oxygen (by difference)	8.41	9.23	9.39	8.02
N/C atomic ratio	0.84	0.78	0.79	0.80
Dry heating value, Btu/lb	12,670	12,653	12,668	12,529
<u>Sulfur forms, wt. %</u>				
Pyrite	1.58	1.41	1.42	1.40
Sulfate	0.05	0.11	0.07	0.01
Sulfide	<0.01	<0.01	<0.01	0.02
Organic	1.92	2.27	2.25	2.32
<u>Mineral analysis, wt. % (ignited basis)</u>				
Phos. pentoxide, P_2O_5	0.05	0.07	0.09	0.06
Silica, SiO_2	50.01	43.33	42.81	44.85
Ferric oxide, Fe_2O_3	16.45	21.51	21.22	46.25
Alumina, Al_2O_3	16.95	16.70	17.61	19.07
Titania, TiO_3	0.98	0.85	0.94	16.97
Lime, CaO	5.69	5.27	5.62	0.98
Magnesia, MgO	0.88	1.03	0.97	5.87
Sulfur trioxide, SO_3	6.89	6.61	6.69	6.09
Potassium oxide, K_2O	2.71	3.06	3.02	1.97
Sodium oxide, Na_2O	0.43	0.47	0.48	0.58
Undetermined			0.55	0.39

Table 1 (continued)

FEED COAL ANALYSES (ILLINOIS NO. 6)

Run no.	Date	2/12-16/86 CC-ITSL	250G		251-1C 5/11-14/86 CC-ITSL		251-1E 5/16-25/86(a) CC-ITSL		251-1G 6/3-5/86(a) CC-ITSL	
			3/8-11/86(a) CC-ITSL	Ash Recycle	5/11-14/86 CC-ITSL	Ash Recycle	5/16-25/86(a) CC-ITSL	Ash Recycle	6/3-5/86(a) CC-ITSL	Ash Recycle
<u>Proximate analysis, wt. %</u>										
Volatile matter	35.86	-	-	-	35.78	-	-	-	-	-
Fixed carbon	50.86	-	-	-	49.34	-	-	-	-	-
Ash	10.77	-	-	-	10.59	-	-	-	-	-
Moisture	2.51	-	-	-	4.29	-	-	-	-	-
<u>Ultimate analysis, wt. %</u>										
Carbon	70.24	-	-	-	70.15	-	-	-	-	-
Hydrogen	4.95	-	-	-	4.74	-	-	-	-	-
Nitrogen	1.27	-	-	-	1.22	-	-	-	-	-
Sulfur	3.76	-	-	-	3.53	-	-	-	-	-
Chlorine	0.04	-	-	-	0.11	-	-	-	-	-
Ash	11.05	-	-	-	11.14	-	-	-	-	-
Oxygen (by difference)	8.69	-	-	-	9.11	-	-	-	-	-
H/C atomic ratio	0.85	-	-	-	0.81	-	-	-	-	-
Dry heating value, Btu/lb	-	-	-	-	-	-	-	-	-	-
<u>Sulfur forms, wt. %</u>										
Pyrite	1.20	-	-	-	1.19	-	-	-	-	-
Sulfate	0.09	-	-	-	0.08	-	-	-	-	-
Sulfide	<0.01	-	-	-	<0.01	-	-	-	-	-
Organic	2.47	-	-	-	2.26	-	-	-	-	-
<u>Mineral analysis, wt. % (ignited basis)</u>										
Phos. pentoxide, P_2O_5	0.19	-	-	-	-	-	-	-	-	-
Silica, SiO_2	46.41	-	-	-	-	-	-	-	-	-
Ferric oxide, Fe_2O_3	17.70	-	-	-	-	-	-	-	-	-
Alumina, Al_2O_3	17.93	-	-	-	-	-	-	-	-	-
Titania, TiO_2	0.84	-	-	-	-	-	-	-	-	-
Lime, CaO	6.22	-	-	-	-	-	-	-	-	-
Magnesia, MgO	0.82	-	-	-	-	-	-	-	-	-
Sulfur trioxide, SO_3	6.57	-	-	-	-	-	-	-	-	-
Potassium oxide, K_2O	2.22	-	-	-	-	-	-	-	-	-
Sodium oxide, Na_2O	0.71	-	-	-	-	-	-	-	-	-
Undetermined	0.39	-	-	-	-	-	-	-	-	-

(a) Data not available.

Table 2
FEED COAL ANALYSES (WYODAK)

Run no.	246DE	246CC	246CC	246CC	246CC	246CC	246CC
Date	6/1-7/84	7/19/84	7/28/85	8/4-7/85	9/7-15/85	9/7-15/85	9/7-15/85
Configuration	DTDSL	TRSL	TRSL	TRSL	TRSL	TRSL	TRSL
<u>Proximate analysis, wt %</u>							
Volatile matter	35.97	35.65	33.45	34.34	34.35	36.44	36.44
Fixed carbon	34.09	32.87	32.85	32.72	33.53	33.12	33.12
Ash	5.96	6.27	6.87	6.94	6.46	7.53	7.53
Moisture	23.62	24.76	24.63	24.00	25.67	22.91	22.91
<u>Ultimate analysis, wt %</u>							
Carbon	67.20	67.26	63.83	63.89	66.96	65.77	65.77
Hydrogen	4.91	4.87	4.31	4.23	4.71	4.77	4.77
Nitrogen	0.73	0.90	0.77	0.76	0.78	0.90	0.90
Sulfur	1.02	1.02	1.08	1.10	1.08	1.14	1.14
Chlorine	0.03	0.03	<0.01	<0.01	<0.01	0.03	0.03
Ash	7.75(a)	8.38(a)	11.80	11.76	8.69	9.77	9.77
Oxygen (by difference)	18.38	17.54	18.16	18.10	17.78	17.62	17.62
H/C atomic ratio	0.87	0.86	0.82	0.82	0.84	0.87	0.87
Dry heating value, Btu/lb	11,505	11,498	10,797	10,783	11,357	11,359	11,359
<u>Sulfur forms, wt %</u>							
Pyrite	0.06	0.13	0.12	0.12	0.16	0.18	0.18
Sulfate	0.67	0.01	0.08	0.08	0.91	0.13	0.13
Bulfide	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Organic	0.80	0.86	0.88	0.90	1.31	0.83	0.83
<u>Mineral analysis, wt % (ignited basis)</u>							
Phos. pentoxide, P_2O_5	0.47	0.48	0.46	0.46	1.14	0.53	0.53
Silica, SiO_2	27.82	28.79	40.86	41.21	34.10	38.09	38.09
Ferric oxide, Fe_2O_3	5.93	6.36	5.16	5.29	6.78	6.55	6.55
Alumina, Al_2O_3	12.71	15.16	19.38	19.62	14.20	16.12	16.12
Titania, TiO_3	0.74	0.84	0.86	0.96	0.87	0.70	0.70
Lime, CaO	24.98	26.37	15.45	15.53	22.53	16.74	16.74
Magnesia, MgO	4.21	3.92	3.32	3.31	4.42	3.36	3.36
Sulfur trioxide, SO_3	18.28	15.31	11.19	11.51	13.91	15.63	15.63
Potassium oxide, K_2O	0.28	0.53	0.61	0.70	0.37	0.26	0.26
Sodium oxide Na_2O	2.08	1.36	1.49	1.72	1.35	1.37	1.37
Vanadium, V_2O_5	0.03	-	-	-	0.33	-	-
Undetermined	1.13	0.18	1.22	0.18	0.65	-	-

(a) SO_3 free ash.

Table 3

TSI PROCESS OPERATING CONDITIONS (ILLINOIS NO. 6)

Run no.	241CD	2429C	243JK/244B	244CD	245BCDF	247C	247D
Date	9/15-24/82	12/20/82-1/2/83	5/29-7/31/03	7/28-8/15/83	12/2/83-1/18/84	10/19-26/84	11/1-9/84
Configuration	NTSL	NTSL	NTSL	NTSL	NTSL	NTSL	NTSL
Operation mode							
1st stage	thermal	thermal	thermal	thermal	thermal	thermal	thermal
2nd stage	catalytic	catalytic	catalytic	catalytic	catalytic	catalytic	catalytic
Catalyst type							
1st stage	none	none	none	none	none	none	none
2nd stage	Arak-Ketjen	Shell 324	Shell 324	Shell 324	Shell 324	Shell 324	Shell 324
Coal feed rate, MF lb/hr	396	191	195	198	177	182	177
Coal conc. in slurry, wt % MF	38.2	35.9	35.8	35.6	35.1	35.9	35.7
Process solvent, wt %							
Resid, wt %	24 (d)	46	47	47	46	43	44
CI, wt %	-	-	-	-	-	-	-
Hydrogen, wt %	7.90	8.81	9.02	8.74	8.84	9.15	9.18
H/C atomic ratio	-	1.22	1.21	1.17	1.18	1.22	1.22
Fe ₂ O ₃ addition, wt % MF coal	-	-	-	-	-	-	-
LiDOS addition, wt % MF coal	-	-	-	-	-	-	-
1st stage							
Reaction temp., °F (average)	860 (a)	860 (a)	810 (b)	811 (b)	812	810	
Inlet H ₂ part. press., psia	2170	2180	2150	2120	2060	2180	2150
Space velocity, lb feed/hr-lb cat	-	-	-	-	-	-	-
Coal space rate	low	high	low	low	low	low	low
Catalyst age, lb (res+CI)/lb cat	-	-	-	-	-	-	-
Temperature profile, °F							
Preheater outlet	752	859	818	821	815	836	839
Dissolver inlet	733 (..19)	-	805 (-13)	799 (-22)	794 (-21)	817 (-19)	815 (-18)
10% (Δ)	777 (+44)	-	-	-	-	811 (-6)	810 (-5)
25% (Δ)	-	-	810 (+5)	810 (+11)	811 (+17)	-	810 (+0)
50% (Δ)	-	-	-	-	-	-	-
75% (Δ)	-	-	-	-	-	-	-
100% (Δ)	805 (+28)	-	-	-	-	-	-
Exotherm							
(Outlet - 10%)	+28	-	-	-	-	+1	+0
(Outlet - Inlet)	+72	-	+5	+11	+17	-5	-5
Backmixing pump	No	No	No	No	No	No	No
2nd stage							
Reaction temp., °F (average)	780	720	730	720	698	711	
Inlet H ₂ part. press., psia	2240	2600	2660	2580	2590	2700	2690
Space velocity, lb feed/hr-lb cat	1.17	0.99	1.00	1.09	0.74	0.94	0.90
1 lb MF coal/hr-cu ft ³ cat	60.2	25.1	25.7	26.1	21.5	20.1	19.5
Feed resid content, wt %	48	55	55	55	49	51	
Catalyst age, 1b (res+CI)/lb cat	260-387	278-441	350-850	943-1139	1300-1400 (c)	283-372	446-671
CSD							
DAS type	2100	2853	2803-2903	2903	2803-2903	2353-2453	2453-2503

(a) Preheater outlet temperature.
 (b) Reactor outlet temperature.
 (c) Range of average catalyst age.
 (d) Light thermal resid (LTR) from CSD (solvent content = 20 wt %).

Table 3 (continued)

TSL PROCESS OPERATING CONDITIONS (ILLINOIS NO. 6)

Run no.	248A	248D	248F	250B	250D
Date	3/10-17/85	4/8-11/85	4/21-25/85	12/4-7/85	1/11-16/86
Configuration	DIRSL LCT	ITSL LCT	ITSL LCT	CC-ITSL No Int VS	CC-ITSL
Operation mode					
1st stage	thermal catalytic	thermal catalytic	thermal catalytic	thermal catalytic	thermal catalytic
2nd stage	none Shell 324	none Shell 324	none Shell 324	none Shell 324	none Amocat 1C
Catalyst type					
1st stage					
2nd stage					
Coal feed rate, MP lb/hr	178	176	181	178	184
Coal conc. in slurry, wt %	35.5	35.7	36.0	36.0	35.6
Process solvent, wt %					
Resid, wt %	45-51	45	45	49	48
CI, wt %	-	-	-	-	50
Hydrogen, wt %	8.46	9.0	8.76	9.05	8.89
H/C atoalc ratio	1.14	1.20	1.17	1.22	1.19
Fe ₂ O ₃ addition, wt % MF coal	2.1	2.0	0	-	-
DMS addition, wt % MF coal	1.3	1.4	0	-	-
1st stage					
Reaction temp., °F (average)	632	834	835	809	824
Inlet H ₂ Part. press., psia	2150	2130	2170	2800	2480
Space velocity, lb feed/hr-lb cat	-	-	-	-	-
Coal space rate	low	low	high	low	low
Catalyst age, lb (res+CI)/lb cat	-	-	-	-	high
Temperature profile, °F					
Preheater outlet	854	868	868	804	814
Dissolver inlet	829(-25)	841(-27)	844(-24)	792(-8)	811(-3)
10% (A)	826(-3)	829(-3)	836(-8)	804(+12)	807(-4)
25% (A)	829(+3)	831(+3)	836(0)	-	822(+12)
50% (A)	832(+3)	834(+1)	837(+1)	811(+7)	-
75% (A)	839(+7)	840(+6)	-	810(+3)	825(+8)
100% (A)	840(+1)	841(+1)	-	-	-
Exotherm					
(Outlet - 10%)	+14	+12	+1	+7	+3
(Outlet - inlet)	+11	0	-7	+19	-1
Backmixing pump	No	No	No	Yes	Yes
2nd stage					
Reaction temp., °F (average)	648	705	728	725	703
Inlet H ₂ Part. press., psia	2770	2730	2590	2470	2520
Space velocity, lb feed/hr-lb cat	0.57	0.69	0.71	0.95	1.34
lb MP coal/hr-cu ft cat	21.2	20.9	21.5	19.6	22.7
Feed resid content, wt %	39	57	50	52	53
Catalyst age, lb (res+CI)/lb cat	37-70	26.0-287	359-396	1432-1470	302-396
CSD	2803-2853	2753-2853	2653	2303	2453
DAS type					

Table 3 (continued)

TSL PROCESS OPERATING CONDITIONS (ILLINOIS NO. 6)

Run no.	2507	2506	250H	251-1B	251-1C	251-1E	251-1G
Date	2/12-16/86	3/8-11/86	3/13-16/86	5/11-14/86	5/16-25/86	6/3-12/86	6/10-13/86
Configuration	CC-TTSI	CC-TTSI	Ash Recycle	CC-TTSI	CC-TTSI	CC-TTSI	CC-TTSI
Operation mode			Ash Recycle				
Catalyst type							
1st stage	none	none	none	catalytic	catalytic	catalytic	catalytic
2nd stage	Amocat 1C	Amocat 1C	Amocat 1C	thermal	thermal	catalytic	catalytic
Coal feed rate, MP lb/hr	339	274	281	472	482	301	481
Coal conc. in slurry, wt % MF	35.6	33.3	33.8	33.8	34.2	33.9	34.0
Process solvent, wt %							
Resid., wt %	49	40	39	40	40	40	39
CI, wt %	-	9	9	9	12	12	12
Hydrogen, wt %	8.3	8.22	8.10	8.57	7.86	7.74	7.10
H/C atomic ratio	1.11	1.17	1.15	1.21	1.14	1.13	1.02
Fe ₂ O ₃ addition, wt % MP coal	-	-	-	-	-	-	-
DMS addition, wt % MP coal	-	-	-	-	-	-	-
1st stage							
Reaction temp., °F (average)	829	829	834	792	806	808	834
Inlet H ₂ part. press., psia	2520	2500	2410	2600	250,0	2470	2620
Space velocity, lb feed/hr-lb catalyst	-	-	-	-	4.18	4.21	4.21
lb MF coal/hr-cu ft cat	-	-	-	-	56.9	58.1	58.0
Coal space rate	high	high	high	high	high	low	high
Catalyst age, lb (res+CI)/lb cat	-	-	-	851-1048	1181-1722	2159-2245	2573-2777
Temperature profile, °F							
Preheater outlet	804	785	791	-	-	-	-
Dissolver inlet	809 (+5)	791 (+6)	797 (+6)	-	-	-	-
10% (Δ)	827 (+18)	826 (+35)	831 (+34)	-	-	-	-
25% (Δ)	-	-	-	-	-	-	-
50% (Δ)	830 (+3)	831 (+5)	835 (+5)	-	-	-	-
75% (Δ)	-	-	-	-	-	-	-
100% (Δ)	-	-	-	-	-	-	-
Exotherm							
(Outlet - 10%)	+4	+5	+5	-	-	-	-
(Outlet - inlet)	+21	+40	+39	-	-	-	-
Backmixing pump	Yes	Yes	Yes	Yes	Yes	Yes	Yes
2nd stage							
Reaction temp., °F (average)	750	750	759	760	759	758	775
Inlet H ₂ part. press., psia	2580	2540	2570	2550	2520	2460	2510
Space velocity, lb feed/hr-lb catalyst	2.59	2.23	2.25	3.82	3.78	2.28	3.64
lb MF coal/hr-cu ft cat	41.9	33.8	34.7	58.3	59.5	37.2	59.4
Feed resid content, wt %	56	46	40	40	39	43	43
Catalyst age, lb (res+CI)/lb cat	1,040-1,191	346-439	502-594	1,377-1,470	1,570-1,992	2,313-2,374	2,625-2,753
CSD	2503-2603	2303	2353	3604-3100	3100-2504	2504	2604
DAS type							

Table 4

TSL PROCESS OPERATING CONDITIONS (WYODAK)

Run no.	246DE	246G	249D	249E	249F	249H	251-11B
Date	6/1-7/84	7/19/84	7/28-8/2/85	8/4-7/85	8/14-18/85	9/7-15/85	8/2-5/86
Configuration	D-TSL	T-TSL	R-TSL	R-TSL	R-TSL	R-TSL	CC-TTS
Operation mode	thermal catalytic	Ash Recycle					
1st stage	none	none	none	none	none	none	none
2nd stage	Shell 324	Shell 324	Shell 324	Shell 324	Amocat 1C	Shell 324	Amocat 1C
Catalyst type	Shell 324	Shell 324	Shell 324	Shell 324	Amocat 1C	Shell 324	Amocat 1C
Coal feed rate, MF lb/hr	249	244	244	25	250	251	353
Coal conc., in slurry, wt. % MF	35.1	34.4	34.4	40.9	40.9	33.2	33.0
Process solvent, wt. %	-	-	-	-	-	-	-
Resid, wt. %	24	31	30	29	28	21	25
CI, wt. %	-	-	-	-	-	25	24
Hydrogen, wt. %	9.18	9.02	9.45	9.54	9.69	7.98	7.55
H/C atomic ratio	1.24	1.21	1.26	1.28	1.30	1.27	1.22
Fe2O3 addition, wt. % MF coal	2.0	2.0	1.6	1.5	1.4	1.5	0.8
DMS addition, wt. % MF coal	1.2	1.2	0	0	0	0	0
1st stage							
Reaction temp., °F (average)	826 (a)	796	796	805	803	819	819
Inlet H ₂ part. press., psia	2160	2140	2140	2180	2170	2510	2510
Space velocity, lb feed/hr-lb cat	-	-	-	-	-	-	-
Coal space rate	Low	Low	Low	Low	Low	Low	Low
Catalyst age, lb (res+CI)/lb cat	-	-	-	-	-	-	-
Temperature profile, °F							
Preheater outlet	754	745	795	781	777	730	744
Disolver inlet	748 (-6)	740 (-5)	772 (-23)	755 (-26)	757 (-20)	702 (-28)	746 (+2)
10% (Δ)	804 (+58)	780 (+40)	793 (+2)	792 (+37)	801 (-44)	795 (+93)	811 (+65)
25% (Δ)	806 (+2)	804 (+24)	795 (+2)	794 (+2)	803 (+2)	799 (+4)	-
50% (Δ)	847 (+41)	836 (+32)	796 (+1)	796 (+2)	805 (+2)	802 (+3)	-
75% (Δ)	-	841 (+5)	798 (+2)	798 (+2)	808 (+3)	806 (+4)	-
100% (Δ)	-	-	800 (+2)	800 (+2)	810 (+2)	811 (+5)	829 (+16)
Exotherm							
(Outlet - 19°)	+43	+61	+7	+8	+9	+16	+18
(Outlet - Inlet)	+99	+101	+28	+45	+53	+109	+83
Backmixing pump	No	No	Yes	Yes	Yes	Yes	Yes
2nd stage							
Reaction temp., °F (average)	624	623	699	701	700	743	743
Inlet H ₂ part. press., psia	2810	2780	2580	2650	2530	2550	2550
Space velocity, lb feed/hr-lb cat	0.86	1.02	1.25	1.24	1.19	1.63	2.79
1b MF coal/hr-cu ft cat	27.4	26.9	30.6	31.5	31.5	31.5	43.6
Feed resid content, wt. %	22	34	22	34	34	27	31
Catalyst age, lb (res+CI)/lb cat	252-279	496	1119-1183	1208-1246	1317-1362	1693-1825	915-968
CSD	2403-2503	2253-2403	2453-2503	3703-3504	2104-2304	no CSD	2204

(a) Average temperature of bottom and outlet of dissolver.

Table 5

TSL PROCESS YIELD DATA (ILLI'VIS NO. 6)

Run no.	241CD		242BC		243JK/244		244CD		245BCDF		247C		247D	
	9/15-24/B2	12/20/H2-1/2/B3	ITSL	ITSL	ITSL	ITSL	ITSL	ITSL	CAT A/W	ITSL	CAT A/W	ITSL	ITSL	11/1-19/84
Configuration														
1st stage average temp., °F	805	860	810	810	811	811	812±1	810±1						
2nd stage average temp., °F	780	720	720	720	720	720	698±1	711±1						
Coal space rate	Low	High	Low	Low	Low	Low	High	High						
WHSV (lb feed/hr/lb cat)														
1st stage	—	—	—	—	—	—	—	—						
2nd stage	1.17	0.99	1.00	1.09	0.74	0.74	0.94±.78	39.88±0.58						
(lb MP coal/hr-cu ft cat)	(60.2)	(25.1)	(25.7)	(26.1)	(21.5)	(20.1)	(19.5)							
Cat age (lb (res+ash+UC)/lb cat)	—	—	—	—	—	—	—	—						
1st stage	26.0-387	278-441	350-850	943-850	1300-1400	1300-1400	283-372	446±671						
2nd stage	—	—	—	—	—	—	—	—						
C4+ dist/H ₂ conc.	9.5	11.0	11.3	10.9	10.9	10.9	9.79±0.42	10.15±0.51						
C ₁ -C ₃ /C ₄ + dist.	0.18	0.07	0.10	0.10	0.10	0.10	0.11±0.01	0.10±0.01						
1st stage yield, wt % MAF coal														
H ₂	-0.63±0.42	-0.68±0.37	-1.28±0.24	-1.44±0.11	-1.44±0.11	-1.44±0.11	-1.52±0.24	-1.67±0.25						
CO, CO ₂	1.65±0.19	1.35±0.01	1.12±0.18	1.34±0.18	1.34±0.18	1.34±0.18	1.10±0.27	1.49±0.28						
H ₂ O	8.00±2.09	4.99±1.51	4.13±1.07	6.08±0.83	7.20±1.44	6.80±0.98	6.16±0.99	6.16±0.99						
NH ₃	0.31±0.14	0.21±0.14	0.40±0.23	0.33±0.17	0.18±0.10	0.31±0.28	0.21±0.17	0.21±0.17						
H ₂ S	2.59±0.11	1.42±0.11	2.59±0.34	1.92±0.13	1.92±0.26	1.92±0.16	1.85±0.26	1.85±0.26						
C ₁ -C ₃	5.42±0.10	3.33±0.49	5.29±1.05	4.77±0.93	4.94±0.44	5.6±0.33	4.99±0.57	4.99±0.57						
C ₄ + dist	23.24±0.95	25.11±1.17	35.3±4.96	31.19±1.25	26.49±1.89	27.40±1.89	27.89±2.59	27.89±2.59						
C ₄ -C ₆	1.26±0.18	0.82±0.38	2.02±0.35	1.60±0.35	1.32±0.14	1.5±0.18	1.97±0.43	1.97±0.43						
IBP-350	4.47±0.96	4.19±0.95	3.33±0.57	8.13±0.74	4.93±0.30	4.27±0.78	4.27±0.59	4.27±0.59						
350-450	3.26±1.14	4.65±0.32	4.74±0.09	4.40±0.46	5.1±0.62	5.76±0.70	5.61±0.41	5.61±0.41						
450-EP	14.26±2.77	16.42±4.19	24.69±4.08	22.07±2.21	15.23±3.29	15.40±1.78	16.47±2.54	16.47±2.54						
Resid	53.65±0.71	52.87±3.37	45.34±3.40	47.11±3.85	51.97±3.27	49.86±2.27	49.86±2.27	49.86±2.27						
Ash	12.61±0.19	11.97±0.20	12.55±0.18	11.8±0.17	11.68±0.26	12.65±0.13	12.65±0.31	12.65±0.31						
UC	7.79±0.18	7.92±0.84	7.60±1.39	8.47±0.64	8.42±0.44	8.66±0.56	8.66±0.56	8.66±0.56						
2nd stage yield, wt % soln. & ash-free feed														
H ₂	-3.79±0.50	-3.26±0.05	-3.19±0.38	-2.80±0.18	-3.11±0.15	-3.17±0.09	-2.95±0.17							
CO, CO ₂	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00						
H ₂ O	1.13±0.52	3.93±0.54	4.24±1.33	3.02±0.76	2.40±0.75	2.40±0.75	2.40±0.75	2.40±0.75						
NH ₃	1.58±0.58	0.42±0.17	0.56±0.15	0.55±0.19	0.74±0.14	0.81±0.09	0.75±0.14	0.75±0.14						
H ₂ S	0.79±0.13	0.53±0.06	0.51±0.20	0.50±0.08	0.53±0.08	0.58±0.03	0.59±0.03	0.59±0.03						
C ₁ -C ₃	4.14±0.42	1.01±0.22	0.92±0.29	0.83±0.06	0.6±0.13	0.59±0.17	0.64±0.13	0.64±0.13						
C ₄ + dist	40.32±0.26	20.49±4.43	19.35±0.94	20.76±2.54	23.47±1.92	23.68±3.25	23.47±1.92	23.47±1.92						
C ₄ -C ₆	1.53±0.42	0.35±0.13	0.45±0.30	0.31±0.99	0.34±0.16	0.38±0.21	0.34±0.16	0.34±0.16						
IBP-350	8.06±0.25	3.16±1.78	3.34±0.52	4.35±1.32	3.76±0.18	3.56±0.64	3.14±0.57	3.14±0.57						
350-450	3.11±0.44	1.89±0.28	2.15±0.14	1.84±0.55	2.00±0.35	1.58±0.23	1.64±0.16	1.64±0.16						
450-EP	27.61±5.25	15.09±4.96	12.42±1.36	14.27±2.29	13.52±0.79	18.16±3.55	18.35±0.79	18.35±0.79						
UC	55.83±0.22	76.83±4.05	77.60±0.43	77.52±3.08	78.43±0.84	69.97±3.32	70.41±1.85	70.41±1.85						
CSD unit yield, wt % feed														
Ash conc.	27.4±0.14	19.75±0.35	18.77±0.32	17.40±0.85	19.50±0.63	25.57±1.22	26.0±1.07							
CSD resid	36.13±0.31	80.25±0.35	81.23±0.32	82.60±0.85	80.50±0.63	74.43±1.22	73.99±1.07							
CSD light resid	36.65±0.35	—	—	—	—	—	—	—						

Table 5 (continued)
TSL PROCESS YIELD DATA (ILLINOIS NO. 6)

Run no.	Date	Configuration	241CD		242BC		243JK/244B		244CD		245BCDF		247C		247D		
			9/15-24/82	12/20-82-1/2/83	TSL	TSL	5/29-7/2/83	TSL	7/28-8/15/83	TSL	CAT A/W	TSL	CAT A/W	10/19-26/84	RITSL	11/1-19/84	RITSL
<u>TSL Yield, wt % MAP</u>																	
H ₂	-4.21±0.20	-4.88±0.15	-5.23±0.37	-5.21±0.99	-5.57±0.13	-6.14±0.20	-6.07±0.21	-6.07±0.20	-6.07±0.20	-6.07±0.20	-6.07±0.20	-6.07±0.20	-6.07±0.20	-6.07±0.20	-6.07±0.20	-6.07±0.20	
CO, CO ₂	1.65±0.10	1.35±0.01	1.12±0.18	1.34±0.18	0.58±0.05	1.10±0.17	1.49±0.28	1.49±0.28	1.49±0.28	1.49±0.28	1.49±0.28	1.49±0.28	1.49±0.28	1.49±0.28	1.49±0.28	1.49±0.28	
H ₂ O	8.47±1.87	10.16±0.63	9.39±1.44	9.64±0.37	10.95±0.21	1.10±0.22	1.30±0.75	1.30±0.75	1.30±0.75	1.30±0.75	1.30±0.75	1.30±0.75	1.30±0.75	1.30±0.75	1.30±0.75	1.30±0.75	
NH ₃	0.77±0.05	2.60±0.14	1.22±0.07	1.08±0.44	1.10±0.22	2.57±0.26	2.79±0.16	2.79±0.16	2.79±0.16	2.79±0.16	2.79±0.16	2.79±0.16	2.79±0.16	2.79±0.16	2.79±0.16	2.79±0.16	
H ₂ S	2.91±0.06	4.00±0.25	2.90±0.20	2.83±0.25	5.89±1.01	5.93±0.34	6.47±0.20	5.95±0.60	5.95±0.60	5.95±0.60	5.95±0.60	5.95±0.60	5.95±0.60	5.95±0.60	5.95±0.60	5.95±0.60	
C ₁ -C ₃	7.14±0.25	4.00±0.25	6.06±0.90	59.08±2.56	56.97±0.27	60.75±1.21	60.11±3.77	61.53±1.06	61.53±1.06	61.53±1.06	61.53±1.06	61.53±1.06	61.53±1.06	61.53±1.06	61.53±1.06	61.53±1.06	
C ₄ + dist	40.22±0.29	54.16±2.82	2.57±0.56	2.01±0.21	1.61±0.13	2.52±0.37	2.52±0.37	2.52±0.37	2.52±0.37	2.52±0.37	2.52±0.37	2.52±0.37	2.52±0.37	2.52±0.37	2.52±0.37	2.52±0.37	
C ₄ -C ₆	1.89±0.36	1.28±0.56	9.30±1.25	9.98±1.06	9.81±0.72	9.45±1.31	9.45±1.31	9.45±1.31	9.45±1.31	9.45±1.31	9.45±1.31	9.45±1.31	9.45±1.31	9.45±1.31	9.45±1.31	9.45±1.31	
IBP-350	7.82±0.92	8.26±1.30	7.41±0.17	6.89±0.30	7.63±0.28	8.05±0.65	8.05±0.65	8.05±0.65	8.05±0.65	8.05±0.65	8.05±0.65	8.05±0.65	8.05±0.65	8.05±0.65	8.05±0.65	8.05±0.65	
350-450	4.55±1.30	7.13±0.13	39.8±2.97	39.09±1.31	41.70±1.50	40.08±3.48	42.47±3.26	42.47±3.26	42.47±3.26	42.47±3.26	42.47±3.26	42.47±3.26	42.47±3.26	42.47±3.26	42.47±3.26	42.47±3.26	
450-EP	25.95±2.15	37.50±4.77	5.55±3.49	8.22±0.13	2.88±1.76	4.48±2.85	3.01±3.02	3.01±3.02	3.01±3.02	3.01±3.02	3.01±3.02	3.01±3.02	3.01±3.02	3.01±3.02	3.01±3.02	3.01±3.02	
Resid	23.19±0.42	7.55±3.12	-	-	-	0.00±0.01	0.00±0.01	0.00±0.01	0.00±0.01	0.00±0.01	0.00±0.01	0.00±0.01	0.00±0.01	0.00±0.01	0.00±0.01	0.00±0.01	
Int accum-DC	-	23.72±0.12	19.32±0.35	19.24±1.27	20.24±1.64	19.50±1.36	-	-	-	-	-	-	-	-	-	-	-
Ash conc. (ash-free)	19.67±1.08	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
<u>1st stage contribution, wt % MAP</u>																	
H ₂	-1.52±0.24	-1.67±0.25	-1.52±0.24	-1.52±0.24	-1.52±0.24	-1.52±0.24	-1.52±0.24	-1.52±0.24	-1.52±0.24	-1.52±0.24	-1.52±0.24	-1.52±0.24	-1.52±0.24	-1.52±0.24	-1.52±0.24	-1.52±0.24	
CO, CO ₂	1.10±0.27	1.49±0.28	1.10±0.27	1.10±0.27	1.10±0.27	1.10±0.27	1.10±0.27	1.10±0.27	1.10±0.27	1.10±0.27	1.10±0.27	1.10±0.27	1.10±0.27	1.10±0.27	1.10±0.27	1.10±0.27	
H ₂ O	6.80±0.98	6.16±0.99	6.80±0.98	6.80±0.98	6.80±0.98	6.80±0.98	6.80±0.98	6.80±0.98	6.80±0.98	6.80±0.98	6.80±0.98	6.80±0.98	6.80±0.98	6.80±0.98	6.80±0.98	6.80±0.98	
NH ₃	0.21±0.17	0.31±0.28	0.21±0.17	0.21±0.17	0.21±0.17	0.21±0.17	0.21±0.17	0.21±0.17	0.21±0.17	0.21±0.17	0.21±0.17	0.21±0.17	0.21±0.17	0.21±0.17	0.21±0.17	0.21±0.17	
H ₂ S	1.55±0.16	1.85±0.26	1.55±0.16	1.55±0.16	1.55±0.16	1.55±0.16	1.55±0.16	1.55±0.16	1.55±0.16	1.55±0.16	1.55±0.16	1.55±0.16	1.55±0.16	1.55±0.16	1.55±0.16	1.55±0.16	
C ₁ -C ₃	5.61±0.33	4.99±0.57	5.61±0.33	5.61±0.33	5.61±0.33	5.61±0.33	5.61±0.33	5.61±0.33	5.61±0.33	5.61±0.33	5.61±0.33	5.61±0.33	5.61±0.33	5.61±0.33	5.61±0.33	5.61±0.33	
C ₄ + dist.	23.70±2.06	24.85±1.17	23.70±2.06	23.70±2.06	23.70±2.06	23.70±2.06	23.70±2.06	23.70±2.06	23.70±2.06	23.70±2.06	23.70±2.06	23.70±2.06	23.70±2.06	23.70±2.06	23.70±2.06	23.70±2.06	
C ₄ -C ₆	1.97±0.43	1.54±0.18	1.97±0.43	1.97±0.43	1.97±0.43	1.97±0.43	1.97±0.43	1.97±0.43	1.97±0.43	1.97±0.43	1.97±0.43	1.97±0.43	1.97±0.43	1.97±0.43	1.97±0.43	1.97±0.43	
IBP-350	4.30±0.78	4.34±0.65	4.30±0.78	4.30±0.78	4.30±0.78	4.30±0.78	4.30±0.78	4.30±0.78	4.30±0.78	4.30±0.78	4.30±0.78	4.30±0.78	4.30±0.78	4.30±0.78	4.30±0.78	4.30±0.78	
350-450	6.20±0.63	6.07±0.44	6.20±0.63	6.20±0.63	6.20±0.63	6.20±0.63	6.20±0.63	6.20±0.63	6.20±0.63	6.20±0.63	6.20±0.63	6.20±0.63	6.20±0.63	6.20±0.63	6.20±0.63	6.20±0.63	
450-EP	11.23±2.51	12.90±4.88	11.23±2.51	11.23±2.51	11.23±2.51	11.23±2.51	11.23±2.51	11.23±2.51	11.23±2.51	11.23±2.51	11.23±2.51	11.23±2.51	11.23±2.51	11.23±2.51	11.23±2.51	11.23±2.51	
<u>2nd stage contribution, wt % MAP</u>																	
H ₂	-4.61±0.17	-4.40±0.19	-4.61±0.17	-4.61±0.17	-4.61±0.17	-4.61±0.17	-4.61±0.17	-4.61±0.17	-4.61±0.17	-4.61±0.17	-4.61±0.17	-4.61±0.17	-4.61±0.17	-4.61±0.17	-4.61±0.17	-4.61±0.17	
CO, CO ₂	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	
H ₂ O	3.51±1.10	2.80±0.22	3.51±1.10	3.51±1.10	3.51±1.10	3.51±1.10	3.51±1.10	3.51±1.10	3.51±1.10	3.51±1.10	3.51±1.10	3.51±1.10	3.51±1.10	3.51±1.10	3.51±1.10	3.51±1.10	
NH ₃	1.17±0.13	1.11±0.20	1.17±0.13	1.17±0.13	1.17±0.13	1.17±0.13	1.17±0.13	1.17±0.13	1.17±0.13	1.17±0.13	1.17±0.13	1.17±0.13	1.17±0.13	1.17±0.13	1.17±0.13	1.17±0.13	
H ₂ S	0.84±0.05	0.88±0.10	0.84±0.05	0.84±0.05	0.84±0.05	0.84±0.05	0.84±0.05	0.84±0.05	0.84±0.05	0.84±0.05	0.84±0.05	0.84±0.05	0.84±0.05	0.84±0.05	0.84±0.05	0.84±0.05	
C ₁ -C ₃	0.86±0.24	0.95±0.21	0.86±0.24	0.86±0.24	0.86±0.24	0.86±0.24	0.86±0.24	0.86±0.24	0.86±0.24	0.86±0.24	0.86±0.24	0.86±0.24	0.86±0.24	0.86±0.24	0.86±0.24	0.86±0.24	
C ₄ + dist.	36.30±2.54	36.59±4.59	36.30±2.54	36.30±2.54	36.30±2.54	36.30±2.54	36.30±2.54	36.30±2.54	36.30±2.54	36.30±2.54	36.30±2.54	36.30±2.54	36.30±2.54	36.30±2.54	36.30±2.54	36.30±2.54	
C ₄ -C ₆	0.56±0.31	0.51±0.25	0.56±0.31	0.56±0.31	0.56±0.31	0.56±0.31	0.56±0.31	0.56±0.31	0.56±0.31	0.56±0.31	0.56±0.31	0.56±0.31	0.56±0.31	0.56±0.31	0.56±0.31	0.56±0.31	
IBP-350	5.15±0.92	4.62±0.80	5.15±0.92	5.15±0.92	5.15±0.92	5.15±0.92	5.15±0.92	5.15±0.92	5.15±0.92	5.15±0.92	5.15±0.92	5.15±0.92	5.15±0.92	5.15±0.92	5.15±0.92	5.15±0.92	
350-450	1.83±0.29	1.98±0.29	1.83±0.29	1.83±0.29	1.83±0.29	1.83±0.29	1.83±0.29	1.83±0.29	1.83±0.29	1.83±0.29	1.83±0.29	1.83±0.29	1.83±0.29	1.83±0.29	1.83±0.29	1.83±0.29	
450-EP	28.73±2.54	29.48±4.62	28.73±2.54	28.73±2.54	28.73±2.54	28.73±2.54	28.73±2.54	28.73±2.54	28.73±2.54	28.73±2.54	28.73±2.54	28.73±2.54	28.73±2.54	28.73±2.54	28.73±2.54	28.73±2.54	
Int accum-resid	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	
Int accum-DC	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
CSD contribution, wt % MAP	0.11±0.07	0.09±0.14	0.11±0.07	0.11±0.07	0.11±0.07	0.11±0.07	0.11±0.07	0.11±0.07	0.11±0.07	0.11±0.07	0.11±0.07	0.11±0.07	0.11±0.07	0.11±0.07	0.11±0.07	0.11±0.07	
450-EP	4.48±2.85	3.01±3.02	4.48±2.85	4.48±2.85	4.48±2.85	4.48±2.85	4.48±2.85	4.48±2.85	4.48±2.85	4.48±2.85	4.48±2.85	4.48±2.85	4.48±2.85	4.48±2.85	4.48±2.85	4.48±2.85	
Resid	19.50±1.36	20.95±1.55	19.50±1.36	19.50±1.36	19.50±1.36	19.50±1.36	19.50±1.36	19.50±1.36	19.50±1.36	19.50±1.36	19.50±1.36	19.50±1.36	19.50±1.36	19.50±1.36	19.50±1.36	19.50±1.36	
Ash conc. (ash-free)	-	-	-</td														

Table 5 (continued)

TSL PROCESS YIELD DATA (ILLINOIS NO. 6)

Run no.	248A	248D	248F	250B	250C	250D
Date	3/10-17/85	4/8-11/85	4/21-25/85	12/4-7/85	1/11-16/86	2/2-5/86
Configuration	DTSL	ITSL	ITSL	CC-ITSL	CC-ITSL	CC-ITSL
LCT	LCT	LCT	LCT	No Int vs	No Int vs	No Int vs
1st stage average temp., °F	832±1	834±1	835±1	809±1	809±1	824±0
2nd stage average temp., °F	648±2	705±1	728±1	724±1	703±1	724±1
Coal space rate	High	High	High	Low	Low	High
WHSV (lb feed/lb cat)						
1st stage						
2nd stage						
Cat age (lb(MP coal)/hr-cu ft cat)						
(lb(MP coal+ash+UC)/lb cat)						
1st stage						
2nd stage						
C ₄ + dist/H ₂ conc						
C ₁ -C ₃ /C ₄ dist.						
<u>1st stage yield, wt % MAP coal</u>						
H ₂	-1.58±0.43	-1.75±0.28	-1.07±0.18	-6.46±0.23	-1.57±0.16	-1.65±0.10
CO, CO ₂	1.76±0.10	1.71±0.08	1.71±0.28	0.38±0.01	1.41±0.13	1.26±0.06
H ₂ O	5.68±0.61	6.13±0.82	5.22±0.75	9.81±0.67	5.45±0.32	5.61±0.70
NH ₃	0.16±0.15	0.17±0.10	0.25±0.14	1.55±0.09	0.05±0.03	0.21±0.07
H ₂ S	2.49±0.26	2.03±0.38	2.41±0.28	3.31±0.09	1.96±0.14	1.58±0.18
C ₁ -C ₃	6.49±0.24	6.55±0.27	5.25±0.38	6.15±0.17	3.94±0.67	4.35±0.26
C ₄ + dist	26.19±1.55	36.45±2.09	24.73±2.16	64.25±1.77	35.2±2.43	30.64±2.72
C ₄ -C ₆	1.58±0.05	1.85±0.05	1.74±0.11	1.67±0.17	1.22±0.11	1.22±0.07
IBP-350	6.09±0.34	7.33±0.22	5.09±0.45	13.44±0.80	3.35±0.28	3.47±0.29
350-450	6.63±0.82	6.43±0.31	5.18±0.46	6.34±0.24	4.68±0.49	4.90±0.12
450-EP	1.84±2.14	20.83±2.39	12.72±2.23	42.80±2.04	25.99±2.44	21.04±2.44
Resid	48.26±2.61	41.14±1.98	51.03±0.77	14.63±1.90	45.65±2.12	50.67±3.19
Ash	11.96±0.09	12.44±0.19	12.24±0.05	13.90±0.09	12.81±0.22	13.00±0.08
UC	10.57±1.51	7.57±0.65	10.40±0.84	6.58±0.47	7.90±0.45	7.24±0.26
<u>2nd stage yield, wt % solv & ash-free feed</u>						
H ₂	-4.76±0.92	-3.23±0.19	-3.30±0.11	(a)	-2.71±0.20	-2.79±0.07
CO, CO ₂	0.00±0.00	0.00±0.00	0.00±0.00	-	0.00±0.00	0.00±0.00
H ₂ O	2.84±0.72	1.48±0.15	2.57±0.31	-	2.80±0.72	2.46±0.75
NH ₃	1.31±0.30	0.79±0.13	0.78±0.06	-	0.77±0.02	0.61±0.08
H ₂ S	0.66±0.08	0.50±0.07	0.38±0.14	-	0.67±0.04	0.71±0.08
C ₁ -C ₃	0.49±0.06	0.77±0.25	1.04±0.08	-	0.86±0.06	1.65±0.20
C ₄ + Dist	29.12±6.69	21.38±0.80	20.93±2.19	-	18.51±1.59	21.04±1.45
C ₄ -C ₆	0.87±0.55	0.88±0.06	0.63±0.22	-	0.18±0.02	0.40±0.10
IBP-350	2.06±0.68	3.19±0.28	4.39±0.33	-	4.04±0.27	5.02±0.60
350-450	1.17±0.75	1.62±0.23	1.84±0.18	-	1.35±0.24	1.37±0.09
450-EP	25.02±1.59	15.69±0.91	14.07±2.45	-	12.94±1.57	14.26±1.90
Resid	0.00±0.00	0.00±0.00	0.00±0.00	-	4.86±0.15	4.38±0.15
UC	70.34±7.06	78.32±0.55	77.74±2.09	-	74.24±1.56	71.43±0.99
<u>CSD unit yield, wt % feed</u>						
Ash conc.	18.57±0.51	18.84±0.28	19.62±0.29	22.56±0.57	26.11±0.65	25.94±0.14
CSD resid	36.68±3.57	81.16±0.28	80.38±0.29	77.44±0.57	73.89±0.65	74.06±0.14
CSD light resid	44.75±3.80	-	-	-	-	-

(a) CCR-1st & 2nd stage combinations.

Table 5 (continued)

TSL PROCESS YIELD DATA (ILLINOIS NO. 6)

Run no.	248A		248D		248F		250B		250C	
	3/10-17/85	DITSL	4/8-11/85	ITSL	LCT	4/21-25/85	ITSL	LCT	CC-ITSL	1/11-16/86
<u>TSL Yield, wt % MAP</u>										
H2	-4.71±0.41		-5.64±0.17		-5.28±0.13		-6.46±0.23		-5.57±0.14	-6.08±0.13
CO, CO ₂	1.76±0.10		1.71±0.08		1.71±0.28		0.38±0.01		1.41±0.13	1.26±0.06
H2O	7.54±0.86		7.92±0.79		8.51±0.49		9.81±0.67		9.58±0.53	9.53±1.48
NH ₃	1.01±0.15		1.12±0.11		1.24±0.10		1.35±0.09		1.19±0.03	1.24±0.10
H ₂ S	2.93±0.23		2.63±0.39		2.89±0.42		3.31±0.09		2.95±0.16	2.72±0.14
C ₁ -C ₃	6.82±0.26		7.48±0.56		6.58±0.33		6.15±0.17		5.24±0.64	6.99±0.18
C ₄ + dist	45.20±1.40		63.62±1.38		53.83±3.08		61.03±2.29		60.46±3.15	63.55±1.60
C ₄ -C ₆	2.13±0.36		2.91±0.12		2.55±0.19		1.67±0.17		1.47±0.10	1.63±0.20
IBP-350	7.42±0.60		11.18±0.32		10.69±0.68		13.44±0.80		9.33±0.65	11.47±1.06
350-450	7.43±0.41		8.39±0.34		7.52±0.47		6.34±0.24		6.68±0.48	7.08±0.14
450-EP	28.22±1.90		41.13±1.99		33.08±4.19		39.59±2.74		42.99±2.94	43.45±2.69
Resid	17.50±1.51		2.81±0.95		8.36±2.10		6.14±1.73		2.34±2.68	-1.70±1.92
Int accm-UC	0.06±0.04		0.00±0.00		0.00±0.00		-		-	-
Ash conc (ash-free)	21.92±0.66		18.35±0.33		22.17±1.09		18.30±1.39		22.42±0.80	22.18±0.40
<u>Int stage contribution, wt % MAP</u>										
H2	-1.58±0.43		-1.75±0.28		-1.07±0.18		-6.46±0.23		-1.57±0.16	-1.65±0.10
CO, CO ₂	1.76±0.10		1.71±0.08		1.71±0.28		0.38±0.01		1.41±0.13	1.26±0.06
H2O	5.68±0.61		6.13±0.82		5.22±0.75		9.81±0.67		5.05±1.32	5.61±0.70
NH ₃	0.16±0.15		0.17±0.10		0.25±0.14		1.35±0.09		0.24±0.03	0.24±0.07
H ₂ S	2.49±0.26		2.03±0.38		2.41±0.28		3.31±0.09		1.96±0.14	1.58±0.18
C ₁ -C ₃	6.49±0.24		6.55±0.27		5.23±0.38		6.15±0.17		3.94±0.67	4.35±0.26
C ₄ + dist	34.83±1.91		46.64±5.16		31.82±3.98		60.86±2.30		18.45±1.19	16.74±1.06
C ₄ -C ₆	1.58±0.05		1.85±0.05		1.74±0.11		1.67±0.17		1.20±0.11	1.22±0.07
IBP-350	5.98±0.35		7.44±0.31		5.16±0.40		13.44±0.80		3.45±0.33	3.46±0.29
350-450	6.66±0.47		6.87±0.61		5.41±0.37		6.34±0.24		5.03±0.41	5.07±0.12
450-EP	20.61±3.81		30.48±5.02		19.51±4.54		39.42±2.76		8.80±1.02	6.99±0.76
<u>2nd stage contribution, wt % MAP</u>										
H2	-3.13±0.35		-3.90±0.17		-4.22±0.09		(a)		-4.44±0.09	-4.44±0.09
CO, CO ₂	0.00±0.00		0.00±0.00		0.00±0.00		-		0.00±0.00	0.00±0.00
H2O	1.86±0.31		1.79±0.16		3.28±0.40		-		4.13±1.02	3.91±1.15
NH ₃	0.86±0.13		0.95±0.16		0.99±0.09		-		1.14±0.05	0.97±0.12
H ₂ S	0.44±0.06		0.61±0.07		0.48±0.19		-		1.44±0.14	2.64±0.32
C ₁ -C ₃	0.33±0.03		0.93±0.30		1.32±0.08		-		1.32±0.10	4.19±2.41
C ₄ + dist	10.03±4.10		16.97±5.51		22.03±1.10		-		41.92±4.10	47.11±1.70
C ₄ -C ₆	0.55±0.32		1.06±0.07		0.81±0.27		-		0.27±0.03	0.63±0.16
IBP-350	1.44±0.28		3.75±0.42		5.53±0.41		-		5.92±0.42	8.01±0.92
350-450	0.77±0.16		1.52±0.12		2.12±0.22		-		2.01±0.14	2.01±0.20
450-EP	7.33±4.18		10.65±5.50		13.57±1.12		-		34.09±3.92	36.46±2.64
Int accm-resid	7.73±8.70		2.81±0.95		8.34±2.10		-		-	-
Int accm-UC	-		-		-		-		-	-
<u>CSD contribution, wt % MAP</u>										
450-EP	0.28±0.24		0.00±0.00		0.00±0.00		0.17±0.03		0.09±0.08	0.00±0.01
Resid	9.78±8.53		0.00±0.00		0.00±0.00		6.14±1.73		2.34±2.68	-1.70±1.92
Ash conc (ash-free)	21.92±0.66		18.35±0.33		22.17±1.09		18.30±1.39		22.42±0.80	22.18±0.40

(a) CCR-1st and 2nd stages combination.

Table 5 (continued)

TSL PROCESS YIELD DATA (ILLINOIS NO. 6)

Run no.	250E	250G	250H	251-IB	251-IC	251-IG
Date	2/12-16/86	3/8-11/86	3/13-16/86	5/11-14/86	5/16-25/86	6/10-13/86
Configuration	CC-ITSL	CC-ITSL	CC-ITSL	CC-ITSL	CC-ITSL	CC-ITSL
1st stage average temp., °F	829±1	829±0	834±1	792±1	806±2	834±0
2nd stage average temp., °F	750±0	750±0	759±1	760±0	759±1	775±1
Coal space rate	High	High	High	—	—	—
WHSV (lb feed/hr/lb cat.)	—	—	—	—	—	—
1st stage (lb MP coal/hr-cu ft cat)	(—)	(—)	(—)	4.18±0.08	4.21±0.05	4.21±0.01
2nd stage (lb MP coal/hr-cu ft cat)	2.59±0.04	2.23±0.01	2.23±0.03	(56.9)	(58.1)	(58.0)
Cat age (lb (res+ash+HC)/lb cat)	(41.9)	(33.8)	(34.7)	3.82±0.09	3.78±0.05	3.64±0.02
1st stage	—	—	—	949±85	1532±210	2202±43
2nd stage	1106±65	392±40	548±40	1394±66	1845±165	2344±31
C4+ dist/H ₂ cond	10.24±0.18	9.81±0.18	9.51±0.18	9.91±0.23	10.08±0.18	10.29±0.13
C ₁ -C ₃ /C ₄ dist	0.11±0.00	0.12±0.00	0.12±0.00	0.08±0.00	0.01±0.01	0.11±0.01
1st stage yield, wt % MAF coal	H ₂	—	—	—	—	—
CO, CO ₂	-1.23±0.18	-1.84±0.20	-2.11±0.20	-3.28±0.37	-3.25±0.44	-4.47±0.34
H ₂ O	1.44±0.31	1.42±0.10	1.34±0.06	0.20±0.02	0.21±0.10	0.54±0.19
NH ₃	5.63±0.63	5.88±0.77	5.92±0.35	7.60±1.00	6.08±0.76	5.24±0.37
H ₂ S	0.23±0.18	0.13±0.02	0.39±0.36	0.59±0.08	0.73±0.12	0.68±0.21
C ₁ -C ₃	1.86±0.38	1.46±0.21	2.01±0.15	2.48±0.21	2.61±0.28	2.61±0.13
C ₄ dist	3.42±0.05	4.93±0.04	4.13±0.13	2.33±0.12	3.67±0.53	4.74±0.06
C ₄ -C ₆	25.23±1.85	33.32±3.10	33.37±1.09	42.97±3.15	57.17±0.34	38.65±1.12
IBP-250	1.00±0.01	1.54±0.01	1.36±0.13	1.19±0.76	1.55±0.21	2.21±0.03
350-450	4.14±0.53	4.10±0.42	4.00±0.75	5.28±0.42	5.76±0.66	9.89±0.92
450-EP	4.53±0.39	4.53±0.39	5.46±0.38	2.76±1.02	5.06±0.26	6.40±0.98
Resid	15.38±2.33	23.15±3.23	22.55±1.52	33.75±4.08	32.94±3.48	38.67±0.50
Ash	56.62±1.05	47.45±2.64	48.66±1.44	37.69±3.35	35.24±1.85	21.84±1.96
UC	12.51±0.36	11.58±0.17	12.56±0.22	12.57±0.10	12.78±0.13	41.71±2.20
	6.81±0.25	7.69±1.25	6.41±1.07	9.42±1.56	9.51±1.17	13.04±0.38
2nd stage yield, wt % solv & ash-free feed	H ₂	-2.69±0.14	-3.03±0.23	-2.94±0.21	-2.01±0.24	-1.96±0.20
CO, CO ₂	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	-2.29±0.05
H ₂ O	2.23±0.08	2.87±0.36	2.26±0.28	2.60±0.73	2.66±0.55	0.00±0.00
NH ₃	0.65±0.06	0.78±0.05	0.72±0.27	0.50±0.06	0.38±0.09	3.38±0.06
H ₂ S	0.45±0.18	0.82±0.12	0.73±0.13	0.31±0.12	0.36±0.13	0.57±0.17
C ₁ -C ₃	1.78±0.06	2.08±0.06	2.24±0.06	1.69±0.15	1.80±0.23	0.24±0.11
C ₄ dist	20.09±0.87	20.28±0.77	19.80±0.48	12.97±0.47	11.46±1.03	0.67±0.12
C ₄ -C ₆	0.38±0.08	0.47±0.04	0.46±0.08	0.52±0.14	0.57±0.06	2.20±0.13
IBP-350	5.15±0.23	5.92±0.40	7.46±0.38	5.66±0.34	5.08±0.55	10.18±0.72
350-450	1.62±0.33	2.13±0.36	2.48±0.23	3.04±0.78	1.45±0.37	13.14±1.76
450-EP	12.86±0.66	11.75±0.11	9.40±1.05	3.62±1.13	4.36±1.67	0.52±0.04
UC	4.46±0.35	8.31±0.54	8.66±0.58	10.70±0.65	11.25±0.28	5.63±0.04
Resid	7.3.10±0.29	6.7.28±0.84	6.8.45±1.51	7.3.25±0.52	7.3.25±1.39	5.50±0.27
CSD unit yield, wt % feed	H ₂	—	—	—	—	—
Ash conc.	22.42±0.57	37.70±0.91	37.23±0.98	43.19±1.58	43.56±1.54	44.05±1.18
CSD resid	77.58±0.67	62.30±0.91	62.77±0.98	56.81±1.58	56.34±1.54	55.96±1.18

Table 5 (continued)
TSL PROCESS YIELD DATA (ILLINOIS NO. 6)

Run no.	250E	250G	250H	251-IB	251-IC	251-IE
Date	2/12-16/86	3/8-11/86	3/13-16/86	5/11-14/86	5/16-25/86	6/3-5/86
Configuration	CC-ITSL	CC-ITSL	CC-ITSL	CC-ITSL	CC-ITSL	CC-ITSL
TSL Yield, wt % MAP						
H ₂	-5.66±0.12	-6.41±0.19	-6.49±0.14	-6.15±0.15	-6.02±0.38	-6.09±0.03
CO, CO ₂	1.44±0.31	1.42±0.10	1.34±0.06	0.20±0.02	0.26±0.10	0.21±0.02
H ₂ O	9.30±0.55	10.22±0.58	9.28±0.53	11.31±0.11	9.84±0.18	10.31±0.08
NH ₃	1.31±0.13	1.31±0.05	1.46±0.08	1.30±0.07	1.26±0.16	1.42±0.05
H ₂ S	2.61±0.10	2.70±0.04	3.10±0.06	2.92±0.06	2.33±0.12	2.93±0.06
C ₁ -C ₃	6.34±0.12	7.63±0.04	7.46±0.11	4.75±0.25	2.49±0.54	2.96±0.18
C ₄ + dist	57.91±1.83	62.90±2.67	61.71±1.35	61.09±2.68	60.60±4.02	70.22±0.04
C ₄ -C ₆	1.62±0.12	2.25±0.06	2.04±0.12	2.07±0.24	2.35±0.25	56.64±2.45
IBP-350	12.62±0.80	13.06±0.48	15.11±0.54	13.38±0.74	12.92±0.88	3.25±0.05
350-450	7.37±0.22	7.76±0.17	9.16±0.11	7.09±0.36	7.09±0.67	16.18±0.48
450-EP	36.30±1.59	39.83±2.58	35.40±1.46	36.45±2.30	38.25±2.46	8.00±0.18
Resid	7.35±1.84	5.42±2.31	7.17±1.43	4.79±3.29	4.28±1.45	29.22±2.70
Int accum-UC	-	0.00±0.30	-0.29±0.65	-0.03±0.22	-0.63±0.38	-0.72±0.65
Ash conc (ash-free)	19.43±1.47	14.80±0.43	15.25±0.76	19.91±0.64	18.21±1.04	8.55±1.91
					0.00±0.22	-0.16±0.28
					15.05±0.28	17.20±0.16
1st stage contribution, wt % MAP						
H ₂	-1.23±0.18	-1.84±0.20	-2.11±0.20	-3.28±0.37	-3.25±0.44	-4.47±0.34
CO, CO ₂	1.44±0.31	1.42±0.10	1.34±0.06	0.20±0.02	0.26±0.10	0.21±0.02
H ₂ O	5.63±0.63	5.88±0.77	5.92±0.35	7.60±1.00	6.08±0.76	5.88±0.10
NH ₃	0.23±0.19	0.13±0.02	0.39±0.36	0.59±0.08	0.73±0.12	0.68±0.21
H ₂ S	1.86±0.38	1.46±0.21	2.01±0.15	2.48±0.21	2.60±0.28	2.61±0.13
C ₁ -C ₃	3.42±0.05	4.49±0.04	4.13±0.13	2.33±0.12	3.67±0.53	1.99±0.06
C ₄ + dist	14.79±0.96	18.36±1.16	19.56±2.24	20.17±1.35	21.96±0.83	4.74±0.53
C ₄ -C ₆	1.00±0.01	1.54±0.01	1.36±0.13	1.19±0.06	1.55±0.21	29.81±0.17
IBP-350	4.16±0.48	4.15±0.38	4.32±0.37	5.29±0.42	5.78±0.64	2.21±0.03
350-450	5.01±0.14	4.67±0.31	5.50±0.47	3.32±0.86	5.22±0.25	9.88±0.91
450-EP	4.62±0.58	8.00±1.33	8.29±1.96	10.37±2.08	9.41±0.55	6.36±0.94
					11.17±0.34	12.78±2.76
2nd stage contribution, wt % MAP						
H ₂	-4.43±0.24	-4.58±0.28	-4.38±0.29	-2.88±0.32	-2.76±0.28	-2.35±0.37
CO, CO ₂	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00
H ₂ O	3.66±0.13	4.35±0.50	3.36±0.41	3.71±0.00	3.76±0.82	4.43±0.06
NH ₃	1.08±0.10	1.19±0.06	1.07±0.40	0.71±0.07	0.53±0.12	0.74±0.23
H ₂ S	0.75±0.30	1.24±0.20	1.09±0.19	0.44±0.18	0.51±0.19	0.32±0.15
C ₁ -C ₃	2.92±0.10	3.14±0.08	3.33±0.09	2.42±0.19	2.53±0.27	0.96±0.17
C ₄ + dist	43.00±1.92	44.29±1.57	41.64±2.10	40.54±2.13	38.00±3.52	3.17±0.15
C ₄ -C ₆	0.62±0.14	0.72±0.07	0.69±0.12	0.89±0.19	0.80±0.07	4.45±0.47
IBP-350	8.46±0.44	6.91±0.54	10.80±0.45	8.09±0.38	7.14±0.72	0.68±0.05
350-450	2.36±0.31	3.08±0.45	3.56±0.45	3.77±0.99	1.87±0.45	6.78±0.36
450-EP	31.57±1.80	31.58±1.65	26.59±1.78	27.79±2.19	28.19±2.79	7.95±0.41
Int accum-resid	-	0.22±2.25	-0.86±1.72	-0.29±1.59	-4.27±2.57	1.30±0.62
Int accum-UC	-	0.00±0.30	-0.29±0.64	-0.03±0.22	-0.63±0.38	1.64±0.15
					0.00±0.22	0.00±0.28
CSD contribution, wt % MAP						
450-EP	0.11±0.11	0.25±0.07	0.52±0.29	0.29±0.26	0.65±0.39	-0.04±0.05
Resid	7.35±1.84	5.19±0.02	9.03±3.96	5.08±3.61	11.28±1.97	-0.72±0.85
Ash conc (ash-free)	19.43±1.47	14.80±0.43	15.25±0.76	19.91±0.64	18.21±1.04	15.05±0.28
						17.20±0.16

Table 6

TSL PROCESS YIELD DATA (WYODAK)

Run no.	246DE 6/1-7/84	246G 7/19/84	249D 7/28-8/2/85	249E 8/4-7/85	249P 8/14-18/85	249H 9/7-15/85	251-LB 8/2-5/86
Date	ITSL	ITSL	Water Add	ITSL	ITSL	ITSL	CC-ITSL
Configuration							Ash Recycle
1st stage average temp., °F	826	810	796±1	796±1	805±0	803±0	819±0
2nd stage average temp., °F	624	623	699±1	701±1	700±2	701±1	743±1
Coal space rate	Low	Low	Low	Low	Low	Low	Low
WHSV (lb feed/hr/lb cat)							
1st stage	0.86	1.02	1.25±0.01	1.24±0.06	1.19±0.02	1.63±0.05	2.79±0.02
2nd stage	(27.4)	(27.9)	(30.6)	(31.5)	(31.5)	(31.5)	(43.6)
Cat. age (lb (rest+ash+UC)/lb cat)							
1st stage	—	—	—	—	—	—	—
2nd stage	252-279	496	1152±24	1227±16	1343±23	1755±72	971±49
C ₄₊ dist./H ₂ cons.	10.2	9.8	9.30±0.25	9.86±0.16	9.02±0.35	8.94±0.38	9.65±0.14
C _{1-C₃} /C ₄₊ dist.	0.15	0.18	0.14±0.01	0.10±0.01	0.14±0.01	0.12±0.00	0.13±0.01
1st stage Yield, wt % MAF coal							
H ₂	-2.74±0.04	-2.69	-1.82±0.20	-2.15±0.23	-2.74±0.27	-3.34±0.06	-2.62±0.10
CO, CO ₂	7.72±0.20	8.31	9.22±0.43	7.30±0.26	8.04±0.13	8.72±0.15	8.87±0.36
H ₂ O	10.21±0.33	10.25	9.65±0.82	9.37±1.50	10.58±0.65	10.03±0.32	7.92±0.65
NH ₃	0.10±0.01	0.13	0.21±0.15	0.09±0.03	0.09±0.01	0.20±0.11	0.25±0.17
H ₂ S	0.73±0.23	0.92	0.15±0.08	0.11±0.03	0.23±0.09	0.12±0.08	0.05±0.07
C _{1-C₃}	8.18±0.93	9.11	6.73±0.17	5.69±0.30	7.21±0.74	6.38±0.37	4.78±0.60
C ₄₊ dist.	46.24±1.03	37.82	33.32±0.63	36.09±0.82	35.98±0.69	41.72±2.9	41.07±0.92
C _{4-C₆}	2.32±0.07	3.16	2.16±0.10	2.00±0.08	2.50±0.18	2.13±0.30	1.68±0.17
IBP-350	9.63±0.34	9.15	8.82±1.03	7.93±0.99	7.03±0.90	7.66±0.43	7.28±0.59
350-450	10.42±1.13	11.16	8.26±1.25	7.47±0.63	9.96±1.60	10.06±0.42	9.17±0.68
450-EP	23.87±0.30	14.35	14.09±3.30	18.70±2.69	16.49±2.37	24.86±3.05	22.95±1.86
Resid.	18.79±1.83	25.30	33.57±1.85	32.01±1.38	30.37±1.46	29.52±3.33	33.67±1.76
Ash	6.77±0.26 (a)	7.64 (a)	13.20±0.19	13.16±0.39	9.20±0.03	9.69±0.11	9.67±0.81
UC	10.86±0.58	10.35	8.25±0.83	10.81±0.84	9.57±0.30	6.54±0.73	5.84±0.77
Coal sulfur to FeS	—	—	0.71±0.02	0.68±0.01	0.66±0.01	0.67±0.01	0.36±0.01
2nd stage Yield, wt % MAF solv & ash-free feed							
H ₂	7.00±0.77	3.52	-4.15±0.15	-4.02±0.10	-3.98±0.19	-2.75±0.09	-3.08±0.16
CO, CO ₂	0.00±0.00	0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.05±0.00
H ₂ O	5.91±1.14	1.40	4.50±0.47	5.08±1.53	4.80±0.54	3.50±1.36	4.95±0.47
NH ₃	0.74±0.18	0.60	0.67±0.13	0.75±0.04	0.81±0.03	0.63±0.08	0.45±0.17
H ₂ S	0.34±0.11	0.11	0.09±0.07	0.17±0.07	0.09±0.06	0.28±0.10	0.28±0.09
C _{1-C₃}	0.86±0.75	0.46	0.50±0.10	0.47±0.04	0.45±0.14	0.34±0.11	2.89±0.26
C ₄₊ dist.	22.71±0.93	16.78	20.49±1.62	22.47±1.64	21.18±1.45	15.36±4.89	17.89±1.35
C _{4-C₆}	1.37±0.42	1.16	0.15±0.01	0.17±0.02	0.16±0.01	0.13±0.03	0.61±0.06
IBP-350	8.14±0.01	2.96	6.07±0.55	6.07±0.50	6.86±0.69	4.24±0.55	8.76±1.70
350-450	6.13±0.12	1.06	2.05±1.47	1.96±0.97	1.69±0.56	1.88±0.55	0.98±0.38
450-EP	7.08±1.48	11.60	12.23±3.75	13.27±2.16	12.47±1.57	9.11±4.70	7.54±1.69
UC	0.00±0.00	0.00	8.78±0.19	10.60±0.58	10.95±0.82	25.14±2.40	19.23±1.97
Resid.	76.49±0.23	84.17	69.13±2.48	64.49±2.33	65.69±0.79	57.43±1.83	57.35±1.33
CSD unit Yield, wt % feed							
Ash conc.	26.15±1.20	25.40	37.80±1.32	43.83±4.58	43.56±0.71	-(b)	57.80±1.12
CSD resid	30.50±1.41	74.60	62.20±1.32	56.18±4.58	56.44±0.71	-(b)	42.20±1.12
CSD light resid	43.25±0.21	—	—	—	—	—	—

(a) SO₃ free-ash
 (b) CSD unit was not in operation.

Table 6 (continued)
TSL PROCESS YIELD DATA (WYODAK)

Run no.	246DE 6/1-7/84	246G 7/19/84	249D 7/28-8/2/85 RTSL	249E 8/4-7/85 RTSL	249F 8/14-18/85 RTSL	249H 9/7-15/85 RTSL	251-11B 8/2-9/86 CC-RTSL
Configuration			Water Add			Ash Recycle	Ash Recycle
TSL Yield, wt % MAP							
H ₂	-5.42±0.15	-5.36	-5.66±0.15	-5.79±0.17	-6.08±0.20	-6.26±0.17	-6.33±0.10
CO, CO ₂	7.64±0.20	8.31	9.22±0.43	7.30±0.26	8.04±0.13	8.17±0.15	8.93±0.36
H ₂ O	12.46±0.01	11.31	13.81±0.68	13.97±1.29	14.61±0.35	13.78±1.44	13.57±1.11
NH ₃	0.38±0.08	0.58	0.83±0.05	0.77±0.03	0.77±0.02	0.87±0.05	0.76±0.15
H ₂ S	0.86±0.27	0.90	0.24±0.05	0.27±0.04	0.31±0.10	0.41±0.14	0.36±0.15
C ₁ -C ₃	8.51±1.22	9.46	7.19±0.08	6.12±0.32	7.59±0.68	6.75±0.38	6.08±0.29
C ₄ + dist	55.11±0.69	52.64	52.59±1.66	57.02±1.10	54.73±0.33	55.94±3.19	61.03±1.34
C ₄ -C ₆	2.84±0.07	4.04	2.30±0.10	2.15±0.06	2.63±0.18	2.28±0.29	2.38±0.12
IBP-350	12.75±0.46	11.39	14.42±1.13	13.42±1.29	12.80±0.13	12.14±0.84	17.29±1.07
350-450	12.77±1.08	11.96	10.14±1.05	9.24±0.85	11.37±1.23	9.07±0.63	10.29±0.60
450-EP	26.76±5.17	25.25	25.73±1.50	32.21±0.77	27.93±1.54	32.46±3.55	31.08±1.92
Rauid	-0.58±2.28	0.70	0.48±2.69	-3.91±3.71	-4.36±1.01	-4.41±1.98	3.79±2.18
Int accm-UC	-	-	-	-	-	-2.04±0.98	0.07±0.40
Ash conc (ash-free)	21.04±0.89	21.45	20.59±1.52	21.60±4.30	23.72±1.20	26.12±2.20	9.38±0.82
Coal sulfur to P ₂ O ₅	-	-	0.71±0.02	0.68±0.01	0.66±0.01	0.67±0.01	0.36±0.01
1st stage contribution, wt % MAP							
H ₂	-2.74±0.04	-2.69	-1.82±0.20	-2.15±0.23	-2.74±0.27	-3.34±0.06	-2.82±0.10
CO, CO ₂	7.64±0.20	8.31	9.22±0.43	7.30±0.26	8.04±0.13	8.17±0.15	8.93±0.36
H ₂ O	10.21±0.33	10.25	9.65±0.82	9.37±1.50	10.58±0.65	10.03±0.32	7.92±0.65
NH ₃	0.10±0.01	0.13	0.21±0.15	0.09±0.03	0.09±0.01	0.20±0.11	0.25±0.17
H ₂ S	0.73±0.23	0.82	0.15±0.08	0.11±0.03	0.23±0.09	0.12±0.08	0.05±0.07
C ₁ -C ₃	8.19±0.94	9.11	6.73±0.17	5.69±0.30	7.21±0.74	6.38±0.37	4.78±0.60
C ₄ + dist	43.91±9.27	35.69	21.86±1.95	21.17±1.15	32.86±2.32	20.86±5.39	17.87±5.42
C ₄ -C ₆	2.32±0.07	3.16	2.16±0.10	2.00±0.08	2.50±0.18	2.13±0.30	1.68±0.17
IBP-350	10.21±0.06	9.83	8.97±0.65	7.90±0.06	7.11±0.96	7.62±0.47	6.39±0.40
350-450	11.84±1.84	11.79	6.61±0.54	5.46±0.92	7.46±0.71	5.07±0.70	5.58±0.72
450-EP	19.54±7.42	10.91	4.12±2.16	5.81±4.24	15.91±2.55	6.04±5.65	4.22±4.34
2nd stage contribution, wt % MAP							
H ₂	-2.68±0.19	-2.67	-3.84±0.20	-3.64±0.06	-3.34±0.07	-2.92±0.16	-3.51±0.17
CO, CO ₂	0.00±0.00	0.00	9.00±0.00	9.00±0.00	0.00±0.00	0.00±0.00	0.06±0.00
H ₂ O	2.26±0.35	1.06	4.16±0.48	4.60±1.38	4.03±0.42	3.76±1.34	5.65±0.48
NH ₃	0.28±0.06	0.45	0.62±0.11	0.68±0.04	0.65±0.02	0.67±0.10	0.52±0.21
H ₂ S	0.13±0.04	0.08	0.08±0.06	0.16±0.06	0.07±0.04	0.29±0.11	0.31±0.10
C ₁ -C ₃	0.33±0.28	0.35	0.45±0.10	0.43±0.03	0.39±0.08	0.37±0.12	3.30±0.32
C ₄ + dist	8.58±8.98	16.97	30.72±2.68	36.17±4.30	22.98±2.50	35.08±5.07	41.65±6.23
C ₄ -C ₆	0.52±0.14	0.88	0.14±0.01	0.16±0.02	0.13±0.00	0.14±0.02	0.70±0.08
IBP-350	2.54±0.54	1.56	5.45±0.55	5.52±0.43	5.69±0.83	4.51±0.49	10.89±1.01
350-450	0.93±0.76	0.17	3.53±0.52	3.78±0.38	3.91±0.54	3.00±0.23	4.71±0.17
450-EP	7.21±7.42	14.36	21.60±2.75	26.72±3.89	12.35±3.91	26.42±4.62	25.35±6.34
Int accm-resid	-0.48±1.83(a)	0.70(a)	-	-	-	-4.41±1.98	0.28±1.21
Int accm-UC	-	-	-	-	-	-2.04±0.98	0.07±0.40
CSD Contribution, wt % MAP							
450-EP	-	-	0.01±0.23	-0.32±0.29	-0.33±0.08	- (D)	1.50±1.19
Rauid	-0.11±0.45	-	0.48±2.69	-3.9±3.71	-4.36±1.01	- (b)	3.51±2.70
Ash conc (ash-free)	21.04±0.89	21.45	20.59±1.52	23.60±4.36	23.72±1.20	26.12±2.20	9.38±0.82
Coal sulfur to P ₂ O ₅	-	-	0.71±0.02	0.68±0.01	0.65±0.01	0.67±0.01	0.36±0.01

(a) Hydrotreated resid product.
(b) CSD unit was not in operation.

Table 7

SOUR WATER ANALYSES (ILLINOIS NO. 6)(a)

Configuration	NTSL 24 ICD	DISTL 248A	ITSL 242BC	ITSL 241JK/244B	ITSL 245BCDF	ITSL 248D	ITSL 247D	ITSL 250D	CC-ITS _L 2506(b)	CC-ITS _L 251-1E(b)
<u>Operation mode</u>										
1st stage	thermal	thermal	thermal(SCF)	thermal	thermal	thermal(LCT)	thermal	thermal	thermal	catalytic
2nd stage	catalytic	catalytic	catalytic	catalytic	catalytic	catalytic(cat A/W)	catalytic	catalytic	catalytic	catalytic
<u>Catalytic type</u>										
1st stage	none	none(c)	none	none	none	none(c)	none	none	none	none
2nd stage	Aralk-Etjen	Shell 324	Shell 324	Shell 324	Shell 324	Shell 324	Shell 324	Shell 324	Shell 324	Shell 324
<u>1st stage sour water</u> (V105)										
Total organic carbon	16,380	23,883	16,050	20,525	24,490	20,917	22,387	26,500	—	3,600-4,075
Kjeldahl nitrogen	—	17,080	—	11,540	13,151	16,380	12,285	15,960	—	11,620-33,180
Ammonia nitrogen	11,760	—	6,475	—	—	—	—	—	—	—
Sulfide sulfur	9,859	9,009	5,266	5,443	5,382	11,561	5,939	8,816	—	14,200-51,504
Chlorine	21	1,232	—	475	459	1,336	780	874	—	155
Oil & grease	343	148	540	5,153	619	768	2,363	375	—	521
Phenols	4,660	—	6,840	6,938	7,240	6,900	7,952	3,200	—	2,012
Inorganic carbon	105	—	—	1,681	3,141	3,820	2,635	5,450	—	1,573-2,935
<u>2nd stage sour water</u> (V1080)										
Total organic carbon	1,470	1,220	1,840	2,787	2,180	1,540	1,515	2,140	—	1,860-2,400
Kjeldahl nitrogen	15,960	7,840	12,775	11,270	10,166	13,230	10,465	16,380	—	12,180-17,080
Ammonia nitrogen	16,450	—	11,270	—	—	—	—	—	—	—
Sulfide sulfur	10,151	2,517	10,123	8,528	6,573	5,915	3,062	16,800	—	19,052-19,800
Chlorine	97	36	35	49	19	60	19	31	—	273
Oil & grease	170	237	117	101	46	129	233	8	—	95
Phenols	801	—	1,630	1,093	1,030	585	696	670	—	937
Inorganic carbon	145	—	57	67	53	107	64	240	—	251-322

(a) All results are reported as mg/liter.

(b) Ash recycle.

(c) Fe_2O_3 added in the coal slurry as a disposable catalyst.(d) Data for 8 May, 12 May and 12 June, 1986 in the catalytic-catalytic mode of operation.
High water injection at 80 lb/hr after H.P. Went Separator in the 1st stage dilutes component concentrations by one-fourth to one-fifth.

Table 8

SOUR WATER ANALYSES (WYODAK) (a)

Configuration	DITSL <u>246DE</u>	ITSL <u>246G</u>	RITSL <u>249E</u>	RITSL <u>249H(b)</u>	CC-ITS _L <u>251-IIIB(b)</u>
<u>Operation mode</u>					
1st stage	thermal	thermal	thermal	thermal	thermal
2nd stage	catalytic	catalytic	catalytic	catalytic	catalytic
<u>Catalyst type</u>					
1st stage	none(c)	none(c)	none(c)	none(c)	none(c)
2nd stage	Shell 324	Shell 324	Shell 324	Shell 324	Amocat 1C
<u>1st stage sour water</u>					
(W105)					
Total organic carbon	26,785	27,700	33,417	31,333	29,600
Kjeldahl nitrogen	10,475	11,200	8,260	10,780	8,960
Sulfide sulfur	9	31	0.1	0.2	0.1
Chlorine	95	43	33	177	16
Oil & grease	4,170	4,710	658	162	3,643
Phenols	7,625	6,875	7,150	7,400	5,200
Inorganic carbon	4,269	3,307	500	2,085	3,750
<u>2nd stage sour water</u>					
(W1080)					
Total organic carbon	813	1,170	1,470	1,820	1,900
Kjeldahl nitrogen	3,980	7,280	7,980	8,260	10,465
Sulfide sulfur	607	882	7,504	5,915	7,420
Chlorine	99	14	5	12	9
Oil & grease	145	448	25	104	128
Phenols	332	462	165	210	376
Inorganic carbon	71	88	187	258	1,032

(a) All results are reported as mg/liter.

(b) Ash recycle.

Table 9

 ILLINOIS NO. 6 COAL
 DISTILLATE PRODUCT ANALYTICAL DATA

Distillate Product Stream	Run 241C				Run 241D			
	T104 Overhead	V178 Distillate	V1078 Solvent	V1072 Overhead	T104 Overhead	V178 Distillate	V1078 Solvent	V1072 Solvent
<u>Boiling point fraction, wt %</u>								
IBP-350°F	61.40	0.2	58.65	0.05	53.34	0.2	60.74	0.04
350-450°F	32.92	13.6	35.93	2.78	44.65	14.1	30.96	2.90
450-500°F	0.68	56.5	5.42	14.11	1.93	50.8	8.01	15.20
500-550°F	0.00	24.9	0.00	19.82	0.08	24.8	0.26	20.70
550-650°F	0.00	4.8	0.00	63.24	0.00	10.1	0.03	61.16
650-850°F	-	-	-	-	-	-	-	-
850°F-EP	70	368	70	381	83	365	83	385
Residue	460	740	478	1008	466	773	531	949
<u>IBP, °F</u>								
EP, °F								
<u>The blend consisted of, wt %</u>								
H/C atomic ratio	1.65	1.31	1.81	1.44	1.58	1.29	1.77	1.43
<u>Elem. comp., wt %</u>								
Carbon	82.0	83.43	85.70	87.21	82.60	85.72	86.70	87.90
Hydrogen	11.38	8.36	13.02	10.56	10.92	8.12	12.91	10.56
Nitrogen	0.39	0.72	0.26	0.26	0.39	1.11	0.06	0.46
Sulfur	0.42	0.38	0.03	0.01	0.85	0.41	0.02	0.04
Oxygen (diff.)	5.81	4.68	1.19	1.96	5.24	4.64	0.31	1.04

Table 9 (continued)

ILLINOIS NO. 6 COAL
DISTILLATE PRODUCT ANALYTICAL DATA

Distillate Product Stream	Run 242B			Run 242C		
	T104 Overhead	V178	V1078 Distillate	T104 Overhead	V178	V1078 Distillate
<u>Boiling point fraction, wt %</u>						
IBP-350°F	38.2	-	16.8	47.0	-	16.1
350-450°F	54.5	-	9.0	46.4	0.1	9.4
450-500°F	7.3	9.0	23.6	6.6	7.6	24.5
500-550°F	-	16.9	27.0	-	17.2	27.9
550-650°F	-	74.1	23.6	-	75.1	22.1
650-850°F	-	-	-	-	-	-
850°F-EP	-	-	-	-	-	-
Residue	-	-	-	-	-	-
IBP, °F	68	464	97	69	463	97
EP, °F	468	1001	864	466	1003	822
<u>The blend consisted of, wt %</u>						
H/C atomic ratio	1.62	1.32	1.66	1.74	1.31	1.62
<u>Elem. comp., wt %</u>						
Carbon	83.42	87.19	87.69	82.88	87.82	87.51
Hydrogen	11.36	9.64	12.18	12.13	9.63	11.90
Nitrogen	0.13	0.23	0.13	0.34	0.07	0.10
Sulfur	0.42(0.79)	0.19(0.26)	-(0.06)	0.53(0.88)	0.18(0.30)	0.02(0.03)
Oxygen (diff.)	4.67	2.75	-	4.12	2.30	0.47

Note: Values in parenthesis are from Galbraith Lab.

Table 9 (continued)

 ILLINOIS NO. 6 COAL
 DISTILLATE PRODUCT ANALYTICAL DATA

Distillate Product Stream	Run 243J			Run 243K			Run 244B			V1067
	T104 Overhead	V178 Distillate	V1078 Distillate	T104 Overhead	V178 Distillate	V1078 Distillate	T104 Overhead	T102 Overhead	T102 Tray 6 Distillate	
Boiling Point fraction, wt. %										
IBP-350°F	43.2	0.0	18.8	45.4	0.0	16.5	77.5	1.4	0.0	16.7
350-450°F	49.6	0.2	8.0	50.3	0.2	9.0	22.5	25.7	0.2	10.2
450-500°F	7.2	11.2	31.1	4.3	9.5	27.5	-	55.7	5.0	25.8
500-550°F	0.0	18.2	26.8	0.0	16.9	25.2	-	16.3	17.8	25.8
550-650°F	0.0	70.4	15.3	0.0	73.4	21.8	-	0.9	77.0	21.5
650-850°F	-	-	-	-	-	-	-	-	-	69.9
Residue	-	-	-	-	-	-	-	-	-	-
IBP, °F	67	460	97	119	463	132	80	291	463	105
EP, °F	466	1018	779	466	1017	836	364	685	1059	838
The blend consisted of, wt. %	-	-	-	-	-	-	-	-	-	-
H/C atomic ratio	1.65	1.32	1.59	1.75	1.36	1.62	1.84	1.49	1.31	1.66
Elem. comp., wt. %										
Carbon	63.88	88.04	88.13	84.44	88.39	87.9	85.37	86.98	88.16	87.69
Hydrogen	11.58	9.78	11.74	12.39	10.07	11.98	13.18	10.88	9.69	12.19
Nitrogen	0.46	0.29	0.08	0.32	0.35	0.09	0.29	0.18	0.32	0.06
Sulfur	0.37	0.19	0.05	0.56	0.20	0.03	0.20	0.37	0.20	0.24
Oxygen (diff.)	3.74	1.7	-	2.29	0.99	-	0.96	1.59	1.63	0.01

Table 9 (continued)

 ILLINOIS NO. 6 COAL
 DISTILLATE PRODUCT ANALYTICAL DATA

Distillate Product Stream	Boiling point fraction, wt %	Run 245B				Run 245C				
		T104 Overhead	T102 Overhead	T102 Tray 8	V1078 Distillate	V1072 Solvent	T104 Overhead	T102 Overhead	T102 Tray 8	V1078 Distillate
IBP-350°F	60.4	1.9	0.0	15.3	0.3	22.1	1.3	0.0	18.2	0.25
350-450°F	39.1	16.9	0.0	8.5	2.5	16.3	17.2	0.0	8.9	2.25
450-500°F	0.5	48.3	2.6	15.5	18.1	19.6	48.9	3.1	16.8	17.02
500-550°F	0.0	26.2	9.7	27.1	35.9	19.8	26.6	12.6	23.9	28.29
550-650°F	0.0	6.7	87.7	33.6	43.2	22.2	6.0	84.2	32.2	52.18
650-850°F	-	-	-	-	-	-	-	-	-	-
850°F-EP	68.9	299.9	492.0	97.0	357.7	68.9	298.2	474.9	133.4	356.8
EP, °F	851.2	1033.1	838.0	777.4	925.8	838.0	1050.8	-	774.5	937.4
The blend consisted of wt %	-	-	-	-	-	-	-	-	-	-
H/C atomic ratio	1.78	1.47	1.28	1.62	1.53	1.57	1.47	1.27	1.59	1.53
ELEM. COMP., wt %										
Carbon	84.17	85.83	88.57	87.86	87.14	85.85	85.88	88.91	88.21	86.54
Hydrogen	12.55	10.58	9.48	11.94	11.21	11.28	10.6	9.44	11.78	11.09
Nitrogen	0.28	0.40	0.06	0.17	0.20	0.37	0.44	0.44	0.00	0.19
Sulfur	0.79	0.03	0.24	0.03	0.01	0.36	0.34	0.19	0.01	0.03
Oxygen (diff.)	2.21	2.89	1.06	0.10	1.44	2.14	2.74	1.02	0.00	2.15

Table 9 (continued)

 ILLINOIS NO. 6 COAL
 DISTILLATE PRODUCT ANALYTICAL DATA

Distillate Product Stream	Run 245D						Run 245F					
	T104 Overhead	T102 Overhead	T102 Tray 8	V1078 Distillate	V1072 Solvent	T104 Overhead	T102 Overhead	T102 Tray 8	V1078 Distillate	V1072 Solvent		
<u>Boiling point fraction, wt %</u>												
IBP-350°F	18.1	1.4	0.0	18.6	0.89	58.5	0.6	0.2	22.1	0.11		
350-450°F	15.6	19.4	0.0	8.1	1.65	41.3	16.9	0.2	9.40	1.27		
450-500°F	18.4	47.1	3.0	15.0	19.63	0.2	49.3	1.5	17.7	12.03		
500-550°F	19.8	25.8	12.8	23.3	37.05	0.0	24.8	12.8	23.8	22.39		
550-650°F	28.1	6.4	84.2	35.0	40.78	0.0	8.4	85.2	27.0	64.20		
650-850°F	-	-	-	-	-	-	-	-	-	-		
Residue	-	-	-	-	-	-	-	-	-	-		
IBP, °F	68.9	298.2	477.0	97.0	240.8	60.5	336.8	492.0	68.1	398.6		
EP, °F	859.6	797.1	1044.4	865.8	838.0	435.6	819.8	973.0	338.0	991.8		
<u>The blend consisted of, wt %</u>												
H/C atomic ratio	1.55	1.47	1.29	1.61	1.53	1.76	1.44	1.26	1.64	1.52		
<u>Elem. comp., wt %</u>												
Carbon	86.23	85.76	88.82	87.84	87.99	84.16	85.62	88.50	86.90	88.24		
Hydrogen	11.20	10.55	9.58	11.87	11.27	12.46	10.32	9.36	11.96	11.24		
Nitrogen	0.59	0.66	0.40	0.11	0.17	0.75	0.55	0.40	0.13	0.11		
Sulfur	0.49	0.35	0.21	0.18	0.29	1.10	0.44	0.25	0.01	0.02		
Oxygen (diff.)	1.49	2.68	0.99	0.00	0.28	1.53	3.07	1.42	0.00	6.39		

Table 9 (continued)

 ILLINOIS NO. 6 COAL
 DISTILLATE PRODUCT ANALYTICAL DATA

Distillate Product Stream	Run 247D						Run 248A					
	T104 Overhead	T102 Overhead	V178 Solvent	V182 Distillate	V1078 Solvent	V1072 Overhead	T104 Overhead	T102 Overhead	V178 Solvent	V182 Distillate	V1067 Distillate	V1067 Distillate
Boiling point fraction, wt %												
IBP-350°F	60.58	1.02	0.00	16.9	16.65	0.08	58.95	0.35	0.00	9.96	28.9	0.01
350-450°F	38.80	2.135	0.02	21.97	8.08	0.26	40.63	16.33	0.13	13.31	13.3	0.21
450-500°F	0.59	35.22	1.17	18.48	7.22	1.17	0.40	26.67	1.20	17.8	12.49	0.93
500-550°F	0.00	18.36	2.73	14.57	8.50	1.76	0.02	20.06	2.42	16.98	12.12	2.00
550-650°F	0.00	20.40	17.29	19.68	22.57	9.69	0.00	28.95	13.60	29.92	25.58	9.49
650-850°F	3.65	59.26	8.40	35.68	52.17	0.00	7.64	53.19	12.03	7.60	44.22	-
850°F-EP	0.04	0.00	16.54	0.00	1.09	34.87	0.00	26.46	0.00	0.00	19.44	-
Residue	0.00	0.00	3.00	0.00	0.00	0.00	0.00	3.00	0.00	0.00	23.70	-
IBP, °F	89.4	327.3	479.6	176.0	97.0	463.6	69.6	350.0	465.8	231.1	133.4	465.8
EP, °F	466.0	740.0	937.4	819.2	868.0	1065.4	435.6	756.3	1060.9	798.5	728.6	1019.0
The blend consisted of, wt %	12.9	17.0	6.6	0.8	34.8	27.9	22.9	34.5	15.3	5.0	13.5	8.8
H/C atomic ratio	1.85	1.50	1.35	1.64	1.70	1.40	1.81	1.43	1.30	1.64	1.72	1.41
Elem. comp., wt %												
Carbon	82.3	84.8	87.2	87.4	87.2	89.2	82.79	86.22	88.42	87.65	86.97	88.71
Hydrogen	12.8	10.7	9.9	12.0	12.5	10.5	12.56	10.36	9.64	12.04	12.55	10.50
Nitrogen	0.3	0.4	0.3	0.5	0.2	0.1	0.45	0.51	1.51	0.21	0.34	0.35
Sulfur	0.7	0.4	0.2	0.0	0.0	0.0	0.72	0.34	0.27	0.05	0.12	-
Oxygen (diff.)	3.9	3.7	2.4	0.1	0.1	0.2	3.49	2.58	1.16	0.06	0.02	-

Table 9 (continued)

 ILLINOIS NO. 6 COAL
 DISTILLATE PRODUCT ANALYTICAL DATA

Distillate Product Stream	Run 248D						Run 250D					
	T104 Overhead	T102 Overhead	V178 Solvent	V182 Distillate	V1078 Distillate	V1067 Distillate	T104 Overhead	T104 Bottles	V1078 Distillate	V1072 Solvent		
<u>Boiling Point fraction, wt. %</u>												
IBP-350°F	61.45	0.41	0.00	4.13	24.71	0.07	56.49	0.03	21.23	0.01		
350-450°F	38.24	15.73	0.11	16.40	10.07	0.52	39.36	1.70	10.83	0.27		
450-500°F	0.04	27.65	1.15	21.00	8.99	1.44	3.95	10.01	9.52	0.85		
500-550°F	0.00	19.10	2.65	17.11	8.17	2.20	0.00	10.18	8.45	1.47		
550-650°F	0.00	28.39	10.98	30.42	22.87	11.71	0.00	25.92	20.45	7.98		
650-850°F	0.00	8.70	53.27	10.93	25.18	32.44	0.20	43.74	27.76	42.38		
850°F-EP	0.27	0.00	28.85	0.20	0.00	5.71	0.00	8.42	1.37	47.04		
Residue	0.00	0.00	3.00	0.00	0.00	45.90	0.00	0.00	0.00	0.00		
IBP, °F	97.00	350.00	465.80	231.10	97.00	424.00	87.6	405.0	97.0	464.0		
EP, °F	436.00	745.00	1074.10	728.10	828.40	1019.30	477.6	937.4	877.4	1214.7		
<u>The blend consisted of, wt. %</u>												
H/C atomic ratio	1.85	1.46	1.26	1.60	1.65	1.36	1.80	1.41	1.63	1.30		
<u>Elem. comp., wt. %</u>												
Carbon	82.67	85.35	85.56	87.54	86.99	89.09	83.50	87.60	87.50	89.90		
Hydrogen	12.86	10.47	9.37	11.73	12.07	10.17	12.60	10.40	12.00	9.80		
Nitrogen	0.36	0.59	0.47	0.16	0.46	0.33	0.30	0.50	0.30	0.30		
Sulfur	1.10	0.37	0.23	0.15	0.04	—	0.50	0.20	0.03	0.01		
Oxygen (diff.)	3.02	3.23	0.97	0.43	0.45	—	3.10	1.40	0.20	0.00		

Table 9 (continued)

ILLINOIS NO. 6 COAL
DISTILLATE PRODUCT ANALYTICAL DATA

Distillate Product Stream	Run 250G			
	T104 Overhead	T104 Bottoms	V1078 Distillate	V1072 Solvent
<u>Boiling point fraction, wt %</u>				
IBP-350°F	57.52	0.02	21.73	0.10
350-450°F	36.98	2.21	10.25	0.42
450-500°F	4.72	11.02	10.42	1.46
500-550°F	0.00	8.15	8.31	1.39
550-650°F	0.03	22.53	16.78	8.49
650-850°F	0.58	45.93	30.19	47.01
850°F-EP	0.18	10.34	2.32	41.12
Residue	0.00	0.00	0.00	0.00
IBP, °F	68.90	405.00	67.40	442.70
EP, °F	808.70	985.00	882.40	1143.30
The blend consisted of, wt %	11.70	16.40	70.40	1.50
H/C atomic ratio	1.79	1.42	1.63	1.34
<u>Elem. comp., wt %</u>				
Carbon	83.40	87.30	87.60	89.40
Hydrogen	12.60	10.40	12.00	10.10
Nitrogen	0.24	0.40	0.12	0.29
Sulfur	0.53	0.22	0.01	0.01
Oxygen (diff.)	3.20	1.70	0.30	0.20

Table 9 (continued)

ILLINOIS NO. 6 COAL
 DISTILLATE PRODUCT ANALYTICAL DATA
 Run 251-IE

<u>Distillate Product Stream</u>	<u>T104 Overhead</u>	<u>T104 Bottoms</u>	<u>V1078 Distillate</u>	<u>V1072 Solvent</u>
<u>Boiling point fraction, wt %</u>				
IBP-350°F	71.0	0.5	23.9	0.01
350-450°F	24.6	2.8	12.0	0.5
450-500°F	4.4	21.8	20.7	4.3
500-550°F	0.0	24.5	19.4	11.1
550-650°F	0.0	50.4	24.0	84.1
650-850°F	0.0	0.0	0.0	0.0
850°F-EP	0.0	0.0	0.0	0.0
Residue	0.0	0.0	0.0	0.0
IBP, °F	81.5	350.3	71.9	443.1
EP, °F	467.9	1003.5	863.5	1053.9
The blend consisted of, wt %	17.81	22.89	51.64	7.66
H/C atomic ratio	1.89	1.42	1.62	1.31
<u>Elem. comp., wt %</u>				
Carbon	85.41	87.88	86.68	89.82
Hydrogen	13.51	10.48	11.81	9.85
Nitrogen	0.27	0.45	0.28	0.32
Sulfur	0.06	0.03	0.01	0.01
Oxygen (diff.)	0.75	1.16	1.04	0.00

Table 10

WYODAK COAL
DISTILLATE PRODUCT ANALYTICAL DATA

Distillate Product Stream	Run 246D				Run 246E			
	T104 Overhead	T102 Overhead	V178	V182	V1078 Distillate	T104 Overhead	V178	V182
<u>Boiling point fraction, wt %</u>								
IBP-350°F	52.5	1.7	-	3.8	19.1	57.2	0.9	0.1
350-450°F	43.9	27.3	0.4	11.6	13.2	40.8	23.4	0.1
450-500°F	3.6	54.0	6.8	36.5	26.7	2.0	57.4	9.5
500-550°F	-	14.4	14.4	34.3	29.1	-	14.7	5.7
550-650°F	-	2.6	78.4	13.8	11.9	-	3.6	31.6
650-850°F	-	-	-	-	-	-	78.2	27.2
850°F-EP	-	-	-	-	-	-	-	38.6
Residue	-	-	-	-	-	-	-	30.2
IBP, °F	138	291	450	231	143	70	332	462
EP, °F	465	722	1070	780	761	466	828	1063
<u>The blend consisted of, wt %</u>								
N/C atomic ratio	1.70	1.46	1.28	1.58	1.66	1.79	1.45	1.26
<u>Elem. comp., wt %</u>								
Carbon	81.67	84.37	87.66	87.59	86.18	81.07	84.83	88.42
Hydrogen	11.64	10.31	9.43	11.60	12.03	12.21	10.35	9.37
Nitrogen	0.56	0.32	0.21	0.52	0.18	0.72	1.37	0.54
Sulfur	0.21	0.19	0.04	0.02	0.01	0.02	0.11	0.28
Oxygen (diff.)	5.62	4.81	2.66	0.27	1.60	5.98	3.34	1.56

Table 10 (continued)

WYODAK COAL
DISTILLATE PRODUCT ANALYTICAL DATA

Distillate Product Stream	Run 246G				Run 249E				Run 247E			
	T104 Overhead	T102 Overhead	V178 Solvent	V1078 Distillate	T104 Overhead	T102 Overhead	V178 Solvent	V182	V1078 Distillate	V1072 Distillate	V1072 Solvent	
Boiling Point fraction, wt. %												
IBP-350°F	53.6	0.9	-	12.8	61.49	1.29	0.02	1.71	16.52	0.00		
350-450°F	44.6	25.7	1.4	10.9	37.62	30.47	1.64	9.88	11.06	0.70		
450-500°F	2.4	53.5	7.8	24.9	0.88	26.12	3.61	18.01	12.41	2.43		
500-550°F	-	10.6	14.9	26.8	0.00	13.09	3.90	17.62	10.90	3.38		
550-650°F	-	9.3	75.9	24.6	0.00	18.61	15.49	32.99	25.91	12.87		
650-850°F	-	-	-	-	0.00	10.42	48.44	18.94	22.38	55.86		
850°F-EP	-	-	-	-	0.00	0.60	23.90	0.85	0.81	24.77		
Residue					0.00	0.00	3.00	0.00	0.00	0.00	0.00	
IBP, °F	91.0	336.0	350.0	165.0	87.9	336.9	405.0	307.7	156.2	405.0		
EP, °F	466.0	869.0	1114.0	838.0	461.1	809.9	1071.1	869.9	857.2	1019.6		
The blend consisted of, wt. %												
H/C atomic ratio	1.76	1.41	1.22	1.56	1.82	1.43	1.33	1.59	1.65	1.39		
Elem. comp., wt. %												
Carbon	82.94	85.10	88.51	87.84	82.69	84.57	87.13	88.13	87.43	88.57		
Hydrogen	12.24	10.07	9.04	11.53	12.62	10.17	9.73	11.72	12.07	10.32		
Nitrogen	0.69	0.94	0.42	0.50	0.22	0.32	0.36	0.67	0.17	0.23		
Sulfur	0.19	0.06	0.02	-	0.21	0.10	0.07	0.01	0.01	0.01		
Oxygen (diff.)	3.94	3.83	1.99	-	4.27	4.84	2.71	3.08	0.03	0.88		

Table 10 (continued)

WYODAK COAL
DISTILLATE PRODUCT ANALYTIC DATA
PERIOD RUN 249H

<u>Distillate Product Stream</u>	<u>T104 Overhead</u>	<u>T102 Overhead</u>	<u>V178 Solvent</u>	<u>V182</u>	<u>V1078 Distillate</u>	<u>V1072 Solvent</u>
<u>Boiling point fraction, wt %</u>						
IBP-350°F	65.23	2.23	0.08	1.14	12.65	1.76
350-450°F	34.61	40.09	2.22	8.11	9.73	0.58
450-500°F	0.07	28.97	4.63	17.88	11.43	2.66
500-550°F	0.03	12.98	5.35	19.56	11.43	5.04
550-650°F	0.06	13.17	19.81	36.04	27.50	17.58
650-850°F	0.00	2.53	53.56	16.99	26.00	51.68
850°F-EP	0.00	0.04	11.36	0.28	1.20	20.70
Residue	0.00	0.00	3.00	0.00	0.00	0.00
IBP, °F	69.8	291.0	359.6	332.9	156.2	281.1
EP, °F	424.0	740.0	974.3	838.0	875.7	1059.7
The blend consisted of, wt %	22.3	3.70	1.30	9.00	65.3	-1.60
H/C atomic ratio	1.82	1.48	1.43	1.62	1.68	1.47
<u>Elem. comp., wt %</u>						
Carbon	82.29	84.53	87.14	87.89	87.27	88.69
Hydrogen	12.56	10.49	10.48	11.97	12.30	10.93
Nitrogen	0.44	0.37	0.40	0.09	0.23	0.37
Sulfur	0.16	0.10	0.05	0.01	0.01	0.01
Oxygen (diff.)	4.56	4.52	1.93	0.04	0.19	0.00

Table 10 (continued)

WYODAK COAL
DISTILLATE PRODUCT ANALYTICAL DATA
RUN 251-IIB

<u>Distillate Product Stream</u>	<u>T104 Overhead</u>	<u>T104 Bottoms</u>	<u>V1078 Distillate</u>	<u>V172 Solvent</u>	<u>V178</u>
<u>Boiling point fraction, wt %</u>					
IBP-350°F	55.5	0.5	28.0	0.03	2.6
350-450°F	40.5	5.8	12.0	0.5	15.7
450-500°F	3.4	22.6	19.0	4.0	43.6
500-550°F					
550-650°F	0.2	25.5	20.0	12.6	25.9
650-850°F	0.4	44.6	21.0	82.9	12.2
850°F-EP					
Residue	-	-	-	-	-
IBP, °F	88.6	355.7	67.9	442.6	297.1
EP, °F	507.2	974.9	899.6	1069.0	825.3
The blend consisted of, wt %	19.1	3.3	69.0	4.7	13.3
H/C atomic ratio	1.81	1.45	1.71	1.44	1.60
<u>Elem. comp., wt %</u>					
Carbon	81.45	86.39	86.54	88.78	87.05
Hydrogen	12.37	10.54	12.42	10.74	11.69
Nitrogen	0.45	0.75	0.46	0.43	0.40
Sulfur	0.23	0.07	0.01	0.05	0.04
Oxygen (diff.)	5.96	2.25	0.57	0.00	0.87

Table 11

 ILLINOIS NO.6 DISTILLATE
 PRODUCT QUALITY DATA
 PERIOD 244 (9/21/83)

Fraction	<u>IBP-350°F</u>	<u>350-450°F</u>	<u>450-650°F</u>	<u>650-EP</u>	<u>Calc Blend</u>	<u>Actual Blend</u>
Wt % of blend	18.4	11.0	34.7	35.9	100.0	100.0
GC dist., wt %						
IBP-350°F	-	-	-	-	-	-
350-450°F	-	-	-	-	-	-
450-650°F	-	-	-	-	-	-
650-EP	-	-	-	-	-	-
Specific gravity	0.811	0.917	0.951	1.019	0.940	0.937
<u>Elem. Comp., wt %</u>						
Carbon	85.21	84.43	86.95	89.07	87.11	86.86
Hydrogen	12.86	10.86	10.69	9.69	10.75	10.73
Nitrogen	(846 ppm)	0.22	0.24	0.31	0.23	0.25
Sulfur	0.36	0.23	0.21	0.16	0.22	0.23
Oxygen-by diff.	1.50	4.62	1.41	0.76	1.69	1.93
Oxygen-direct	-	4.07	1.48	1.16	-	1.92
H/C atomic ratio	1.80	1.53	1.47	1.30	1.47	1.47

Table 11 (continued)

 ILLINOIS NO.6 DISTILLATE
 PRODUCT QUALITY DATA
 PERIOD 247D

Fraction	IBP-350°F	350-450°F	450-650°F	650-EP	Calc Blend	Actual Blend
Wt % of blend	15.88	13.64	38.66	31.82	100.00	100.00
GC dist., wt %						
IBP-350°F	85.04	0.86	0.00	0.00	13.62	20.93
350-450°F	14.96	64.67	0.53	0.00	11.40	15.16
450-650°F	0.00	34.47	76.27	0.00	34.19	35.13
650°F-EP	0.00	0.00	23.20	100.00	40.79	28.78
Specific gravity	0.7945	0.9042	0.9427	1.0050	0.928	0.9309
<u>Elem. comp., wt %</u>						
Carbon	85.15	85.13	87.65	89.37	87.45	86.99
Hydrogen	13.90	11.44	11.53	10.24	11.49	11.46
Nitrogen	0.08	0.16	0.23	0.25	0.20	0.20
Sulfur	0.29	0.20	0.10	0.05	0.13	0.16
Oxygen-diff.	0.58	3.07	0.49	0.09	0.73	1.19
H/C atomic ratio	1.95	1.60	1.57	1.37	1.57	1.57

TSL products for 247D were blended according to elementally balanced product rates for (5) days: 01 Nov 85, 11 Nov 85, 13 Nov 85, 15 Nov 85, 19 Nov 85.

Nitrogen by Kjeldahl.

Table 11 (continued)

 ILLINOIS NO.6 DISTILLATE
 PRODUCT QUALITY DATA
 PERIOD 248A

Fraction	<u>IBP-350°F</u>	<u>350-450°F</u>	<u>450-650°F</u>	<u>650-EP</u>	<u>Calc Blend</u>	<u>Actual Blend</u>
Wt % blend	19.83	12.87	40.97	26.33	100.00	100.00
GC Dist., wt %						
IBP-350°F	86.77	1.69	0.06	0.00	17.45	18.08
350-450°F	13.23	83.57	5.49	0.00	15.63	17.36
450-650°F	0.00	14.74	91.08	0.11	39.24	41.18
650°F-EP	0.00	0.00	3.37	99.89	27.68	23.37
Specific gravity						
<u>Elem. comp., wt %</u>						
Carbon	83.93	84.18	86.34	88.18	86.07	85.47
Hydrogen	13.46	11.28	10.99	9.70	11.18	11.00
Nitrogen (1)	0.10	0.14	0.28	0.45	0.27	0.25
Sulfur	0.74	0.35	0.20	0.16	0.32	0.40
Oxygen-by diff.	1.77	4.05	2.19	1.51	2.17	2.88
Oxygen-direct	2.76	5.17	3.15	1.84	2.99	3.04
H/C atomic ratio	1.91	1.60	1.52	1.31	1.55	1.53

TSL products for 248A were blended according to elementally balanced product rates for (4) days: 10 Mar 85, 11 Mar 85, 14 Mar 85, 17 Mar 85.

(1) Nitrogen by Kjeldahl.

Table 11 (continued)

 ILLINOIS NO.6 DISTILLATE
 PRODUCT QUALITY DATA
 PERIOD 248D

Fraction	IBP-350°F	350-450°F	450-650°F	650-EP	Calc Blend	Actual Blend
Wt of blend	18.57	11.04	37.03	3336	100.00	100.00
GC dist., wt %						
IBP-350°F	91.59	5.37	0.21	0.00	17.68	21.58
350-450°F	8.41	79.51	5.54	0.00	12.39	14.57
450-650°F	0.00	15.12	89.46	0.00	34.80	37.61
650°F-EP	0.00	0.00	4.79	100.00	35.13	26.24
Specific gravity	0.8135	0.9362	0.9911	1.0738	0.9704	0.9718
<u>Elem. comp., wt %</u>						
Carbon	84.63	84.38	86.47	89.11	86.78	85.72
Hydrogen	13.76	11.42	10.80	9.41	10.95	10.79
Nitrogen (1)	0.06	0.12	0.37	0.46	0.31	0.28
Sulfur	0.65	0.50	0.34	0.18	0.36	0.43
Oxygen-by diff.	0.90	3.58	2.02	0.84	1.59	2.78
Oxygen-direct	2.37	4.09	2.67	1.65	2.43	2.47
H/C atomic ratio	1.94	1.61	1.49	1.26	1.50	1.50

TSL products for 248D were blended according to elementally balanced product rates for (4r) days: 8 Apr 85, 9 Apr 85, 10 Apr 85, 11 Apr 85.

(1) Nitrogen by Kjeldahl.

Table 11 (continued)

 ILLINOIS NO.6 DISTILLATE
 PRODUCT QUALITY DATA
 PERIOD 250D

Fraction	<u>IBP-350°F</u>	<u>350-450°F</u>	<u>450-650°F</u>	<u>650-EP</u>	<u>Calc Blend</u>	<u>Actual Blend</u>
Wt % of blend	18.60	10.00	35.00	36.40	100.00	100.00
GC dist., wt %						
IBP-350°F	93.17	4.89	0.00	0.00	17.82	20.93
350-450°F	6.83	86.12	1.16	0.00	10.29	11.45
450-650°F	0.00	8.99	85.95	0.08	31.01	35.65
650°F-EP	0.00	0.00	12.89	99.92	40.88	31.97
Specific gravity	0.780	0.884	0.934	1.002	0.918	0.925
<u>Elem. comp., wt %</u>						
Carbon	85.41	86.19	87.81	88.77	87.55	87.18
Hydrogen	14.26	12.44	11.53	10.27	11.67	11.40
Nitrogen (1)	0.04	0.13	0.25	0.36	0.24	0.24
Sulfur	0.18	0.13	0.08	0.07	0.10	0.12
Oxygen-by diff.	0.11	1.11	0.33	0.53	0.44	1.06
Oxygen-direct	0.00	0.00	0.00	0.00	0.00	0.00
H/C atomic ratio	1.99	1.72	1.56	1.38	1.59	1.56

TSL products for 250D were blended according to elementally balanced product rates for (4) days: 2 Feb 86, 3 Feb 86, 4 Feb 86, 5 Feb 86.

(1) Nitrogen by Kjeldahl.

Table 11 (continued)

ILLINOIS NO.6 DISTILLATE
 PRODUCT QUALITY DATA
 PERIOD 250G

Fraction	IBP-350°F	350-450°F	450-650°F	650-EP	Calc Blend	Actual Blend
Wt % of blend	23.00	13.90	37.40	25.70	100.00	100.00
GC dist., wt %						
IBP-350°F	93.98	5.14	0.00	0.00	22.33	23.38
350-450°F	6.02	79.92	1.03	0.00	12.88	13.16
450-650°F	0.00	14.94	78.26	0.04	31.36	35.49
650°F-EP	0.00	0.00	20.71	99.96	33.44	27.97
Specific gravity	0.766	0.874	0.939	1.007	0.898	0.894
<u>Elem. comp., wt %</u>						
Carbon	84.81	85.72	87.45	89.18	87.05	86.95
Hydrogen	13.83	11.93	11.32	10.19	11.69	11.60
Nitrogen (1)	0.05	0.14	0.19	0.28	0.17	0.16
Sulfur	0.15	0.10	0.06	0.05	0.08	0.11
Oxygen-by diff.	1.16	2.11	0.98	0.30	1.00	1.18
Oxygen-direct	0.00	0.00	0.00	0.00	0.00	0.00
H/C atomic ratio	1.94	1.66	1.54	1.36	1.60	1.59

TSL products for 250G were blended according to elementally balanced product rates for (4) days: 8 Mar 86, 9 mar 86, 10 Mar 86, 11 Mar 86.

(1) Nitrogen by Kjeldahl.

Table 11 (continued)
 ILLINOIS NO. 6 DISTILLATE
 PRODUCT QUALITY DATA
 PERIOD 251-IE

Fraction	IBP-350°F	350-450°F	450-650°F	650°F-EP	Calc Blend	Actual Blend
Wt % of blend	20.9	8.6	34.3	36.2	100.0	100.0
<u>GC dist., wt %</u>						
IBP-350°F	94.19	8.43	0.00	0.00	20.41	25.97
350-450°F	5.81	88.33	4.18	0.00	10.24	11.65
450-650°F	0.00	3.24	95.82	0.24	33.23	32.26
650°F-EP	0.00	0.00	0.00	99.76	36.12	30.13
Specific gravity	0.776	0.862	0.921	1.004	0.907	0.900
<u>Elem. comp., wt %</u>						
Carbon	85.90	87.23	87.90	89.64	88.05	87.18
Hydrogen	13.98	12.35	11.68	10.04	11.62	11.62
Nitrogen (a)	0.02	0.08	0.15	0.22	0.14	0.14
Sulfur	0.05	0.04	0.02	0.03	0.03	0.03
Oxygen-by diff.	0.05	0.30	0.25	0.07	0.16	1.03
Oxygen-direct	0.00	0.00	0.00	0.00	0.00	0.00
H/C atomic ratio	1.94	1.69	1.58	1.33	1.57	1.60

TSL products for 251-IE were blended according to Elementally Balanced product rates for (3) days: 03 June 86, 04 June 86, 05 June 86.

(a) Nitrogen by Kjeldahl.

Table 12

WYODAK DISTILLATE
PRODUCT QUALITY DATA
PERIOD 246 (8/7/84) (a)

Fraction	IBP-360°F	360-460°F	460-650°F	650-EP	Calc Blend	Actual Blend
Wt % of blend	19.4	20.4	48.5	11.7	100.0	100.0
<u>GC dist., wt %</u>						
IBP-350°F	-	-	-	-	-	-
350-450°F	-	-	-	-	-	-
450-650°F	-	-	-	-	-	-
650-EP	-	-	-	-	-	-
Specific gravity	0.790	0.924	0.938	-	-	0.912
<u>Elem. comp., wt %</u>						
Carbon	83.06	83.11	87.34	88.85	85.82	85.46
Hydrogen	13.23	10.55	10.91	9.23	11.09	10.86
Nitrogen	0.18	0.34	0.21	0.42	0.26	0.26
Sulfur	0.51	0.17	0.03	0.05	0.15	0.21
Oxygen-by diff.	3.02	5.83	1.51	1.45	2.68	3.21
Oxygen-direct	3.71	6.20	2.03	1.81	-	3.52
H/C atomic ratio	1.90	1.51	1.49	1.24	1.54	1.52

(a) Samples were collected at end of Run 246, which results may be considered to be similar for periods 246G & H.

Table 12 (continued)

WYODAK DISTILLATE
PRODUCT QUALITY DATA
PERIOD 249F

Fraction	IBP-360°F	360-460°F	460-650°F	650-EP	Calc Blend	Actual Blend
Wt % of blend	26.90	13.40	40.00	19.70	100.00	100.00
Adj. blend	31.18	15.53	46.36	22.83	115.90	115.90
GC dist., wt %						
IBP-350°F	86.31	3.78	0.00	0.00	23.72	24.37
350-450°F	13.69	74.83	1.98	0.00	14.50	20.02
450-650°F	0.00	21.39	92.63	0.00	39.92	42.43
650°F-EP	0.00	0.00	5.39	100.00	21.86	13.18
Specific gravity	0.799	0.897	0.921	0.981	0.892	0.890
Elem. comp., wt %						
Carbon	84.47	85.23	87.32	89.07	86.62	86.20
Hydrogen	13.71	11.83	11.68	10.53	12.02	12.01
Nitrogen	0.13	0.21	0.62	0.33	0.38	0.22
Sulfur	0.09	0.06	0.02	0.04	0.05	0.05
Oxygen-by diff.	1.60	2.67	0.36	0.03	0.94	1.52
Oxygen-direct	2.62	4.03	1.75	1.60	2.26	2.52
H/C atomic ratio	1.93	1.65	1.59	1.41	1.65	1.66

TSL products for 249F were blended according to elementally balanced product rates for (3) days: 14 Aug 85, 17 Aug 85, 18 Aug 85.

Note: There was a negative contribution for V1072 HTR solvent for this period. Adjusted blend and fraction analyses are shown in separate tables.

Table 12 (continued)

 WYODAK DISTILLATE
 PRODUCT QUALITY DATA
 PERIOD 249H

Fraction	<u>IBP-360°F</u>	<u>360-460°F</u>	<u>460-650°F</u>	<u>650-EP</u>	<u>Calc Blend</u>	<u>Actual Blend</u>
Wt % of blend						
Adj. blend	27.94	11.18	44.64	16.24	100.00	100.00
GC dist., wt %						
IBP-350°F	89.17	2.57	0.00	0.00	25.20	25.84
350-450°F	10.83	91.30	1.21	0.00	13.77	16.05
450-650°F	0.00	6.12	92.08	-0.01	41.79	44.81
650°F-EP	0.00	0.00	6.68	100.01	19.22	13.30
Specific gravity	0.792	0.888	0.923	0.958	0.884	0.886
Elem. comp., wt %						
Carbon	84.85	84.96	85.94	88.45	85.93	85.72
Hydrogen	13.88	11.99	11.75	11.21	12.28	12.23
Nitrogen ⁽²⁾	0.09 ⁽¹⁾	0.16 ⁽¹⁾	0.20	0.32	0.19	0.19
Sulfur	0.08	0.06	0.01	0.01	0.04	0.05
Oxygen-by diff.	1.10	2.83	2.10	0.01	1.56	1.82
Oxygen-direct	1.74	3.01	1.38	1.08	1.62	2.40
H/C atomic ratio	1.95	1.68	1.61	1.51	1.70	1.70

TSL products for 249H were blended according to elementally balanced product rates for (4) days: 7 Sep 85, 8 Sep 85, 14 Sep 85, 15 Sep 85.

Note: There was a negative contribution for V1072 HTR solvent for this period. Adjusted blend and fraction analyses are shown in this table.

(1) Nitrogen by Kjeldahl.

(2) Nitrogen by CHN analyzer.

Table 13

Parity Plot Data for Model Verification
in CC-ITS-L Catalytic-Catalytic Configuration

Rate Constant Equations for Run 251-I(a)

Run	Stage	Temp. (T), °F	Catalyst age (t) lb(R+CI)/lb cat	Number of data	Rate constant (K), 1/hr $\ln K = \ln A - (E/R)(1/T) - \alpha t$	Activation energy (E), Btu/lb mole	Deactivation coefficient (α)	Correlation coefficient (r)
251-I	1st	775-820 (775, 790) (805, 820)	400-2500	21	23.3-28200(1/T)-0.00034t	56 100±3100	0.00034±0.00004 (r ² = 0.83)	0.91
	2nd	760-775 (760-775)	1000-2800	19	17.2-21300(1/T)-0.00005t	42300±5600	0.00005±0.00005 (r ² = 0.52)	0.72

Experimental and Model Predicted Coal Feed Rate Comparisons

Run	Stage	Temp., °F	Catalyst age (t) lb(R+CI)/lb cat	Model predicted (b)			Experimental		
				Rate constant (K), 1/hr	Conversion (ε), wt % feed	C ₄ + dist yield wt % MAF coal	Rate constant (K), 1/hr	Conversion (ε), wt % feed	C ₄ + dist yield wt % MAF coal
251-IB	1st	792	949	1.574	27.4	61.4	1.546	27.0	61.0
	2nd	760	1394	0.720	15.9	0.733	0.733	16.1	476
251-IC	1st	806	1532	1.592	27.4	60.8	1.654	28.2	60.6
	2nd	759	1845	0.730	16.2	0.662	0.662	14.9	484
251-ID (trans- itional)	1st	807	1922	1.476	30.4	68.0	1.401	29.3	482
	2nd	759	2143	0.684	18.6	0.528	0.528	15.0	385
251-IE	1st	808	2202	1.366	33.8	78.7	1.363	33.8	346
	2nd	758	2344	0.667(f)	22.6	0.390	0.390	14.6	301

(a) CSTR first order resid + UC conversion rate constant determined by the multiple linear regression analysis technique.

(b) Experimental overall distillate selectivity was used for prediction of the C₄+ distillate yield.

(c) Coal feed rate predicted for the C₄+ distillate yield as the experimental.

(d) % error in the coal feed rate = [i - experimental/predicted] × 100.

(e) High error due to the transitional period.

(f) High error due to the lower experimental activity in the second stage than the model predicted.

two-stage liquefaction non-integrated mode (NTSL)

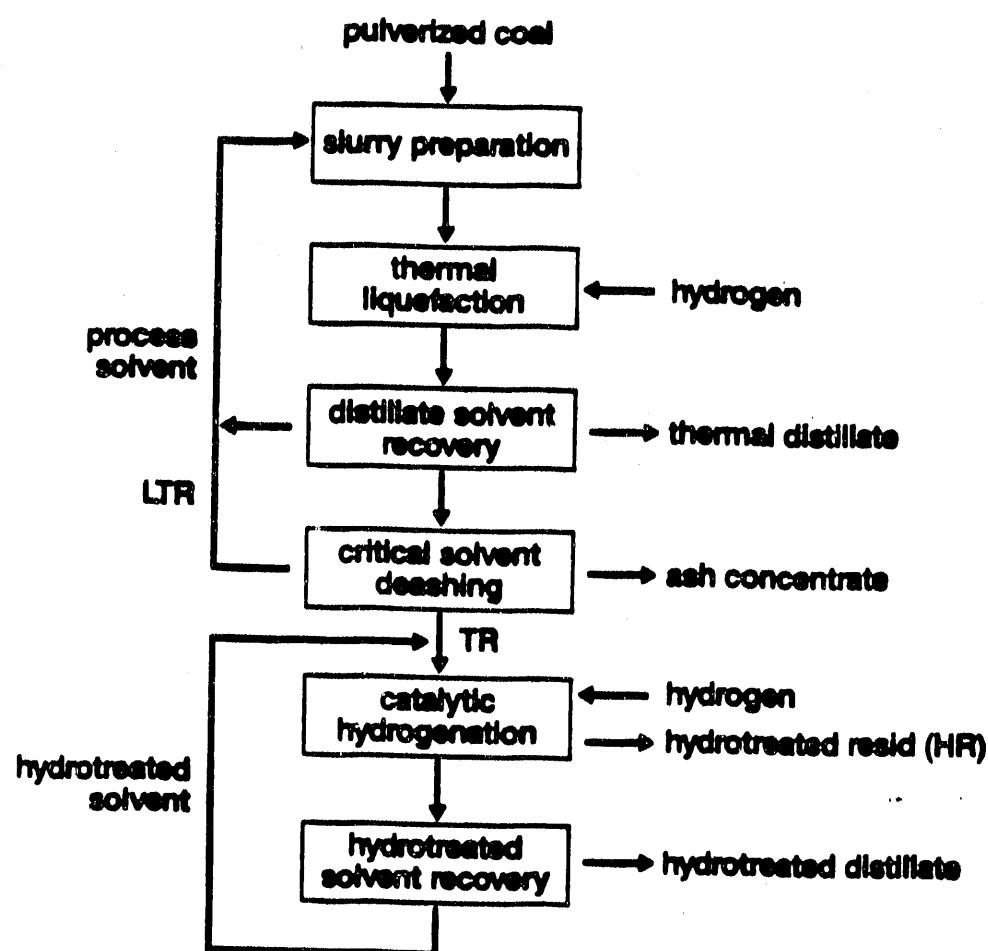


FIGURE 1. BLOCK FLOW DIAGRAM OF NTSL OPERATION

two-stage liquefaction Integrated mode (ITSL)

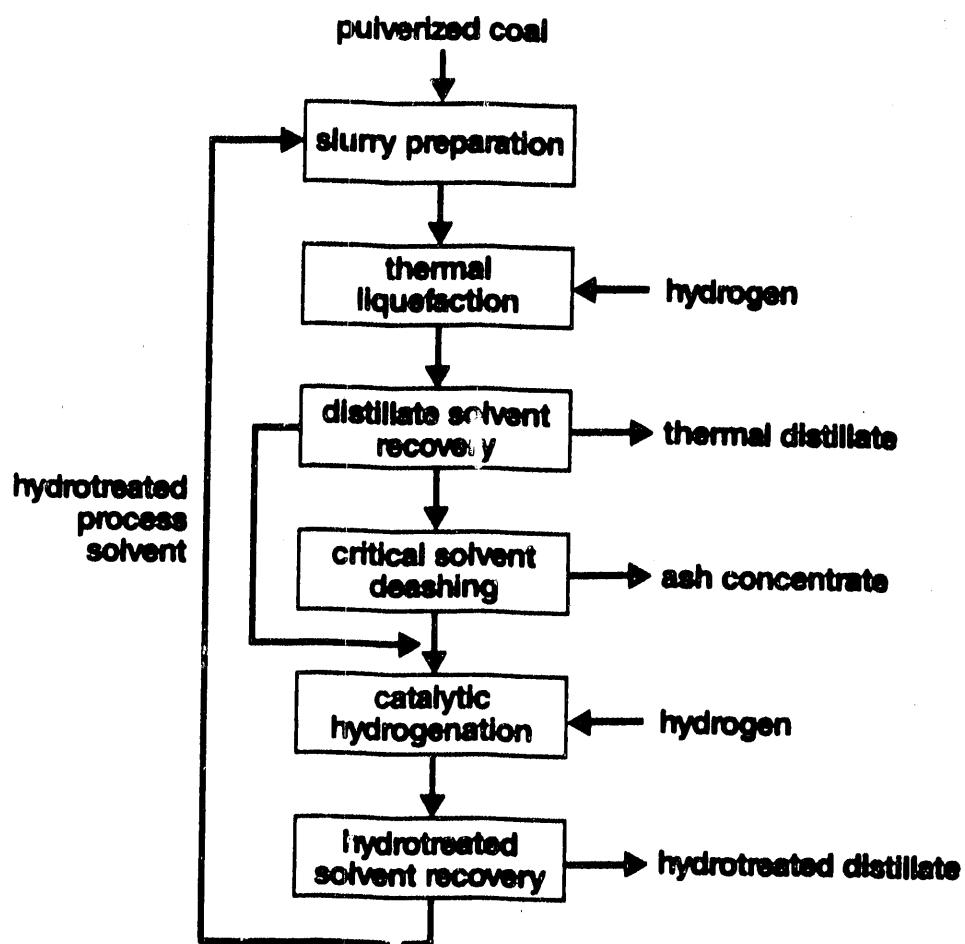


FIGURE 2. BLOCK FLOW DIAGRAM OF ITSL OPERATION

two-stage liquefaction double-integrated mode (DITSL)

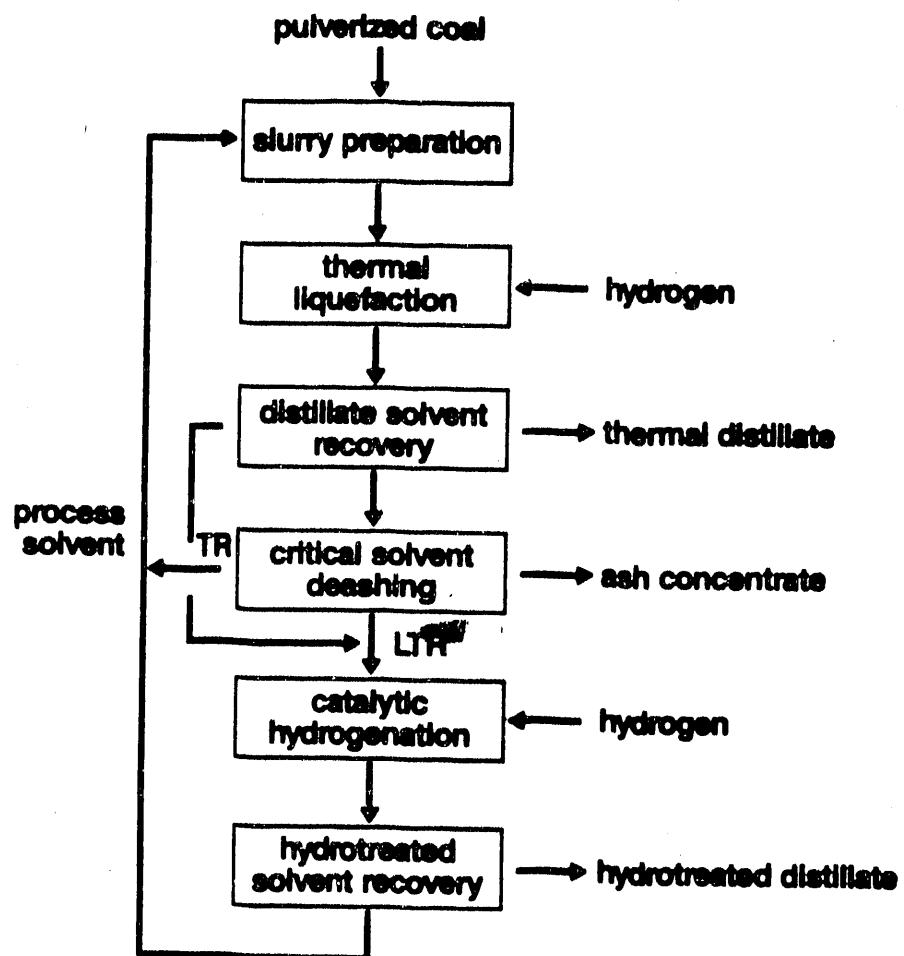


FIGURE 3. BLOCK FLOW DIAGRAM OF DITSL OPERATION

two-stage liquefaction reconfigured mode (RITSL)

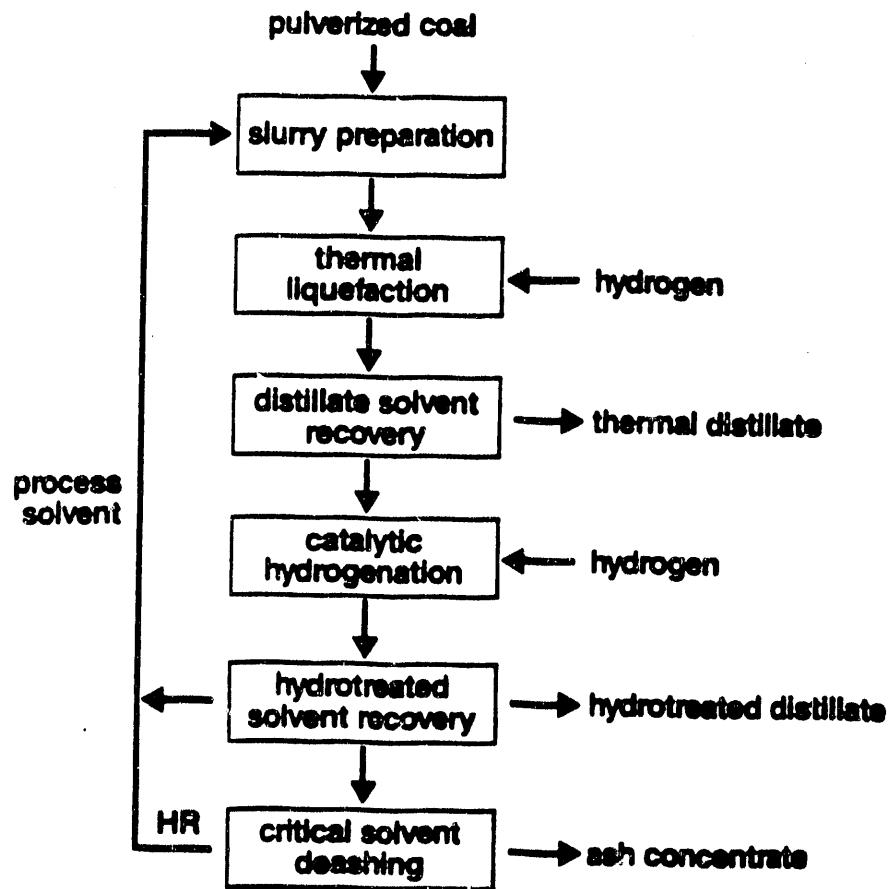


FIGURE 4. BLOCK FLOW DIAGRAM OF RITSL OPERATION

CC-ITSL
two-stage liquefaction
close-coupled Integrated mode

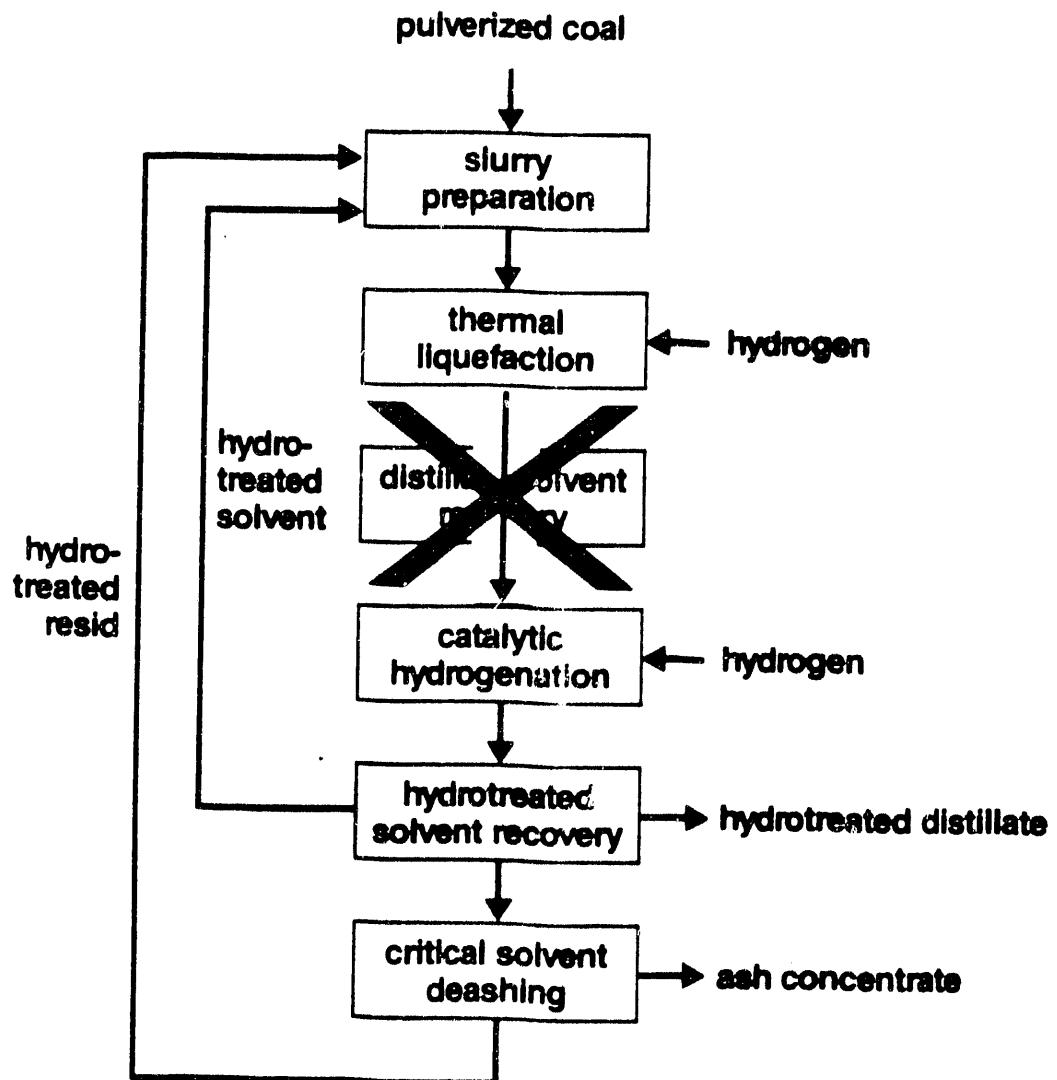


FIGURE 5. BLOCK FLOW DIAGRAM OF CC-ITSL OPERATION

**CC-ITSL with ash recycle
two-stage liquefaction
close-coupled mode**

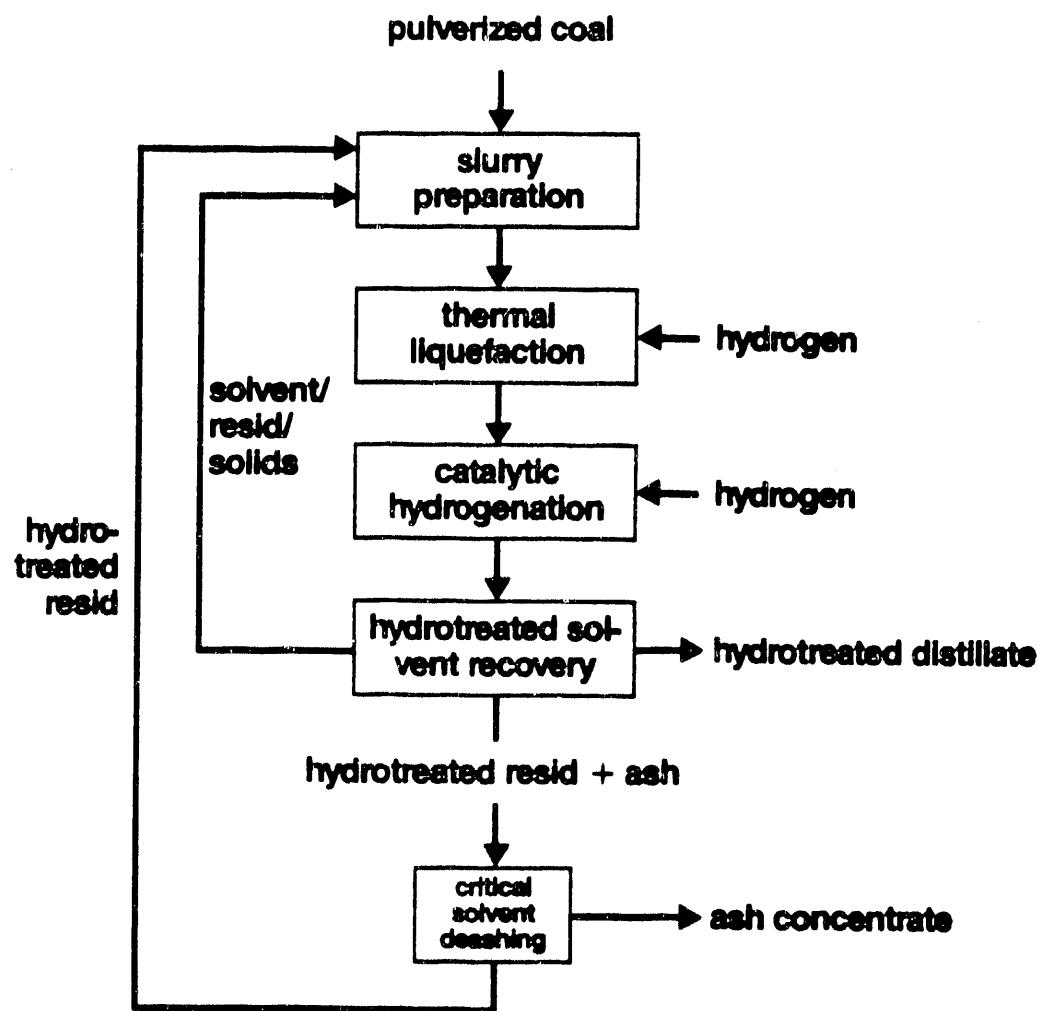
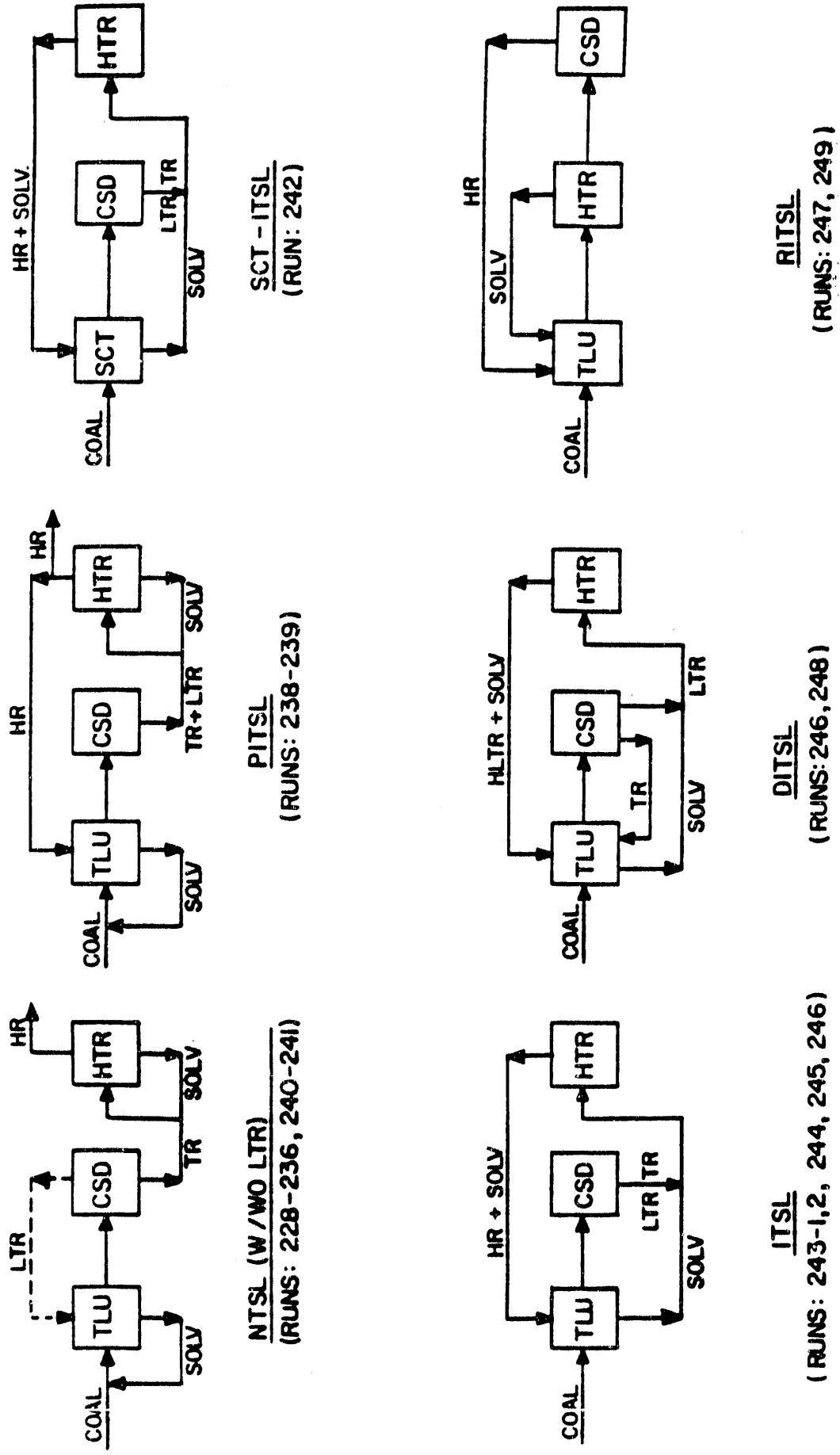


FIGURE 6. BLOCK FLOW DIAGRAM OF CC-ITSL
OPERATION WITH SOLIDS RECYCLE

FIGURE 7
TWO STAGE LIQUEFACTION MODES TESTED



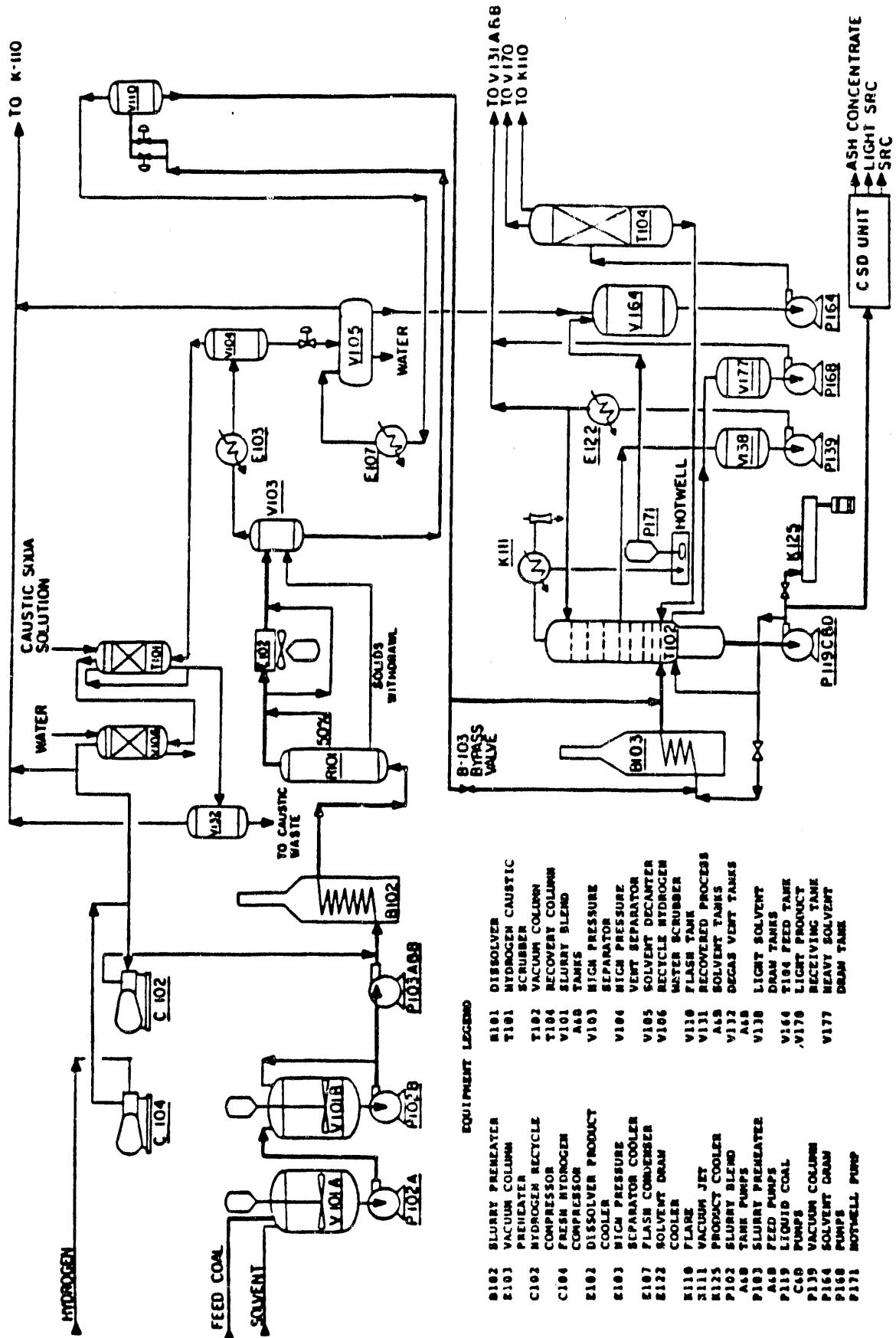


FIGURE 8. SCHEMATIC FLOW DIAGRAM OF TLU

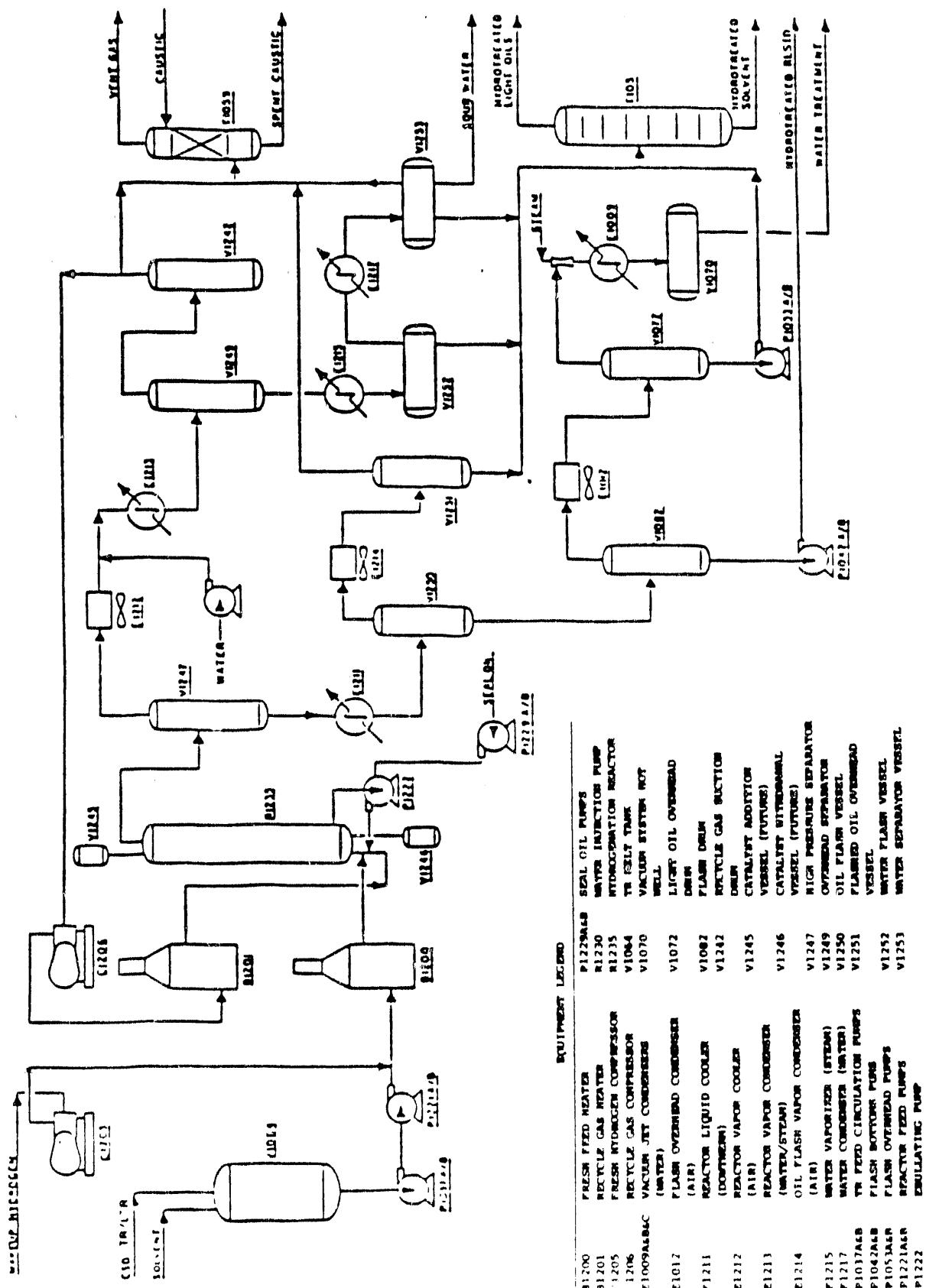


FIGURE 2. SCHEMATIC FLOW DIAGRAM OF HTR UNIT

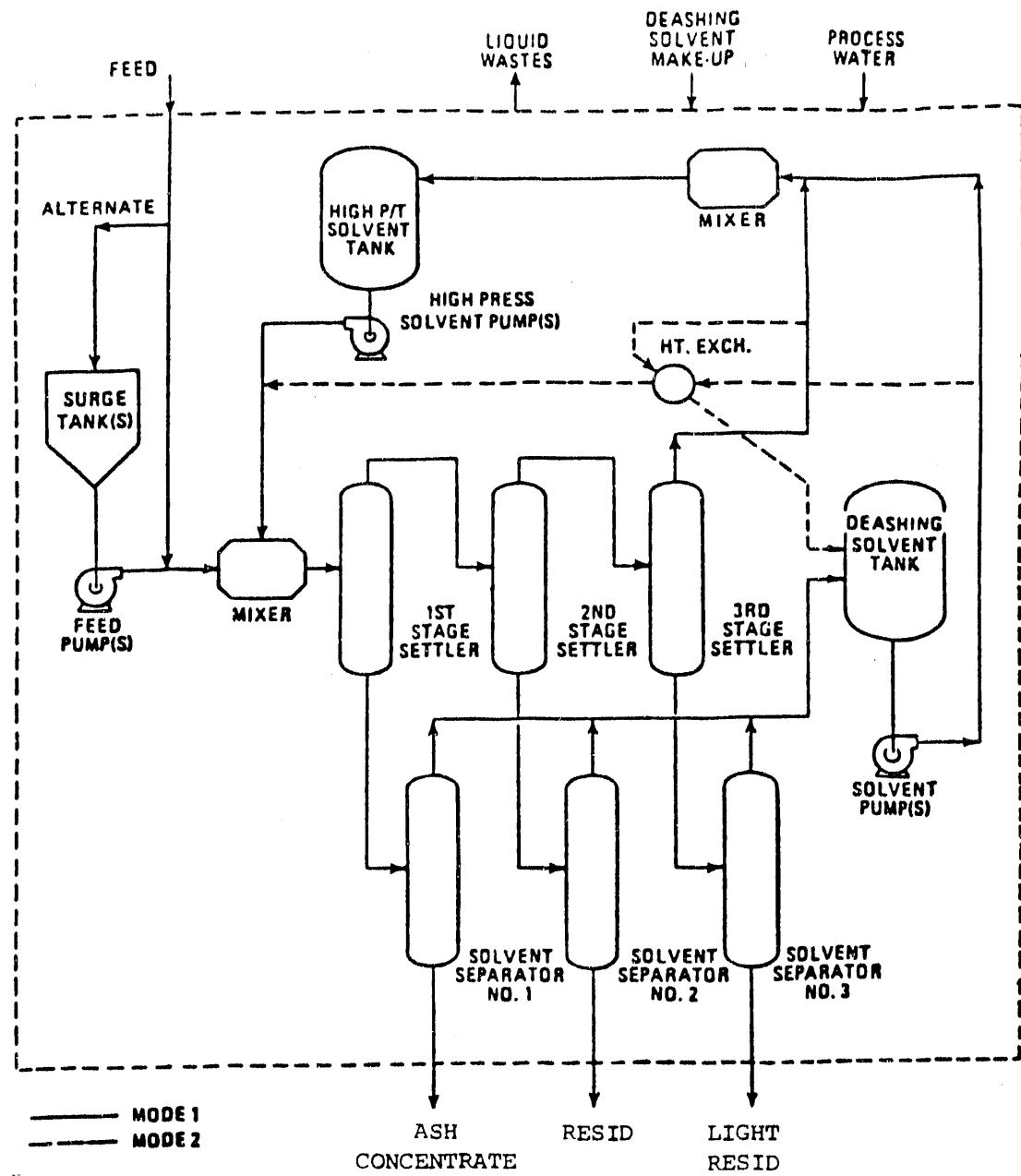


FIGURE 10. SCHEMATIC FLOW DIAGRAM OF CSD UNIT

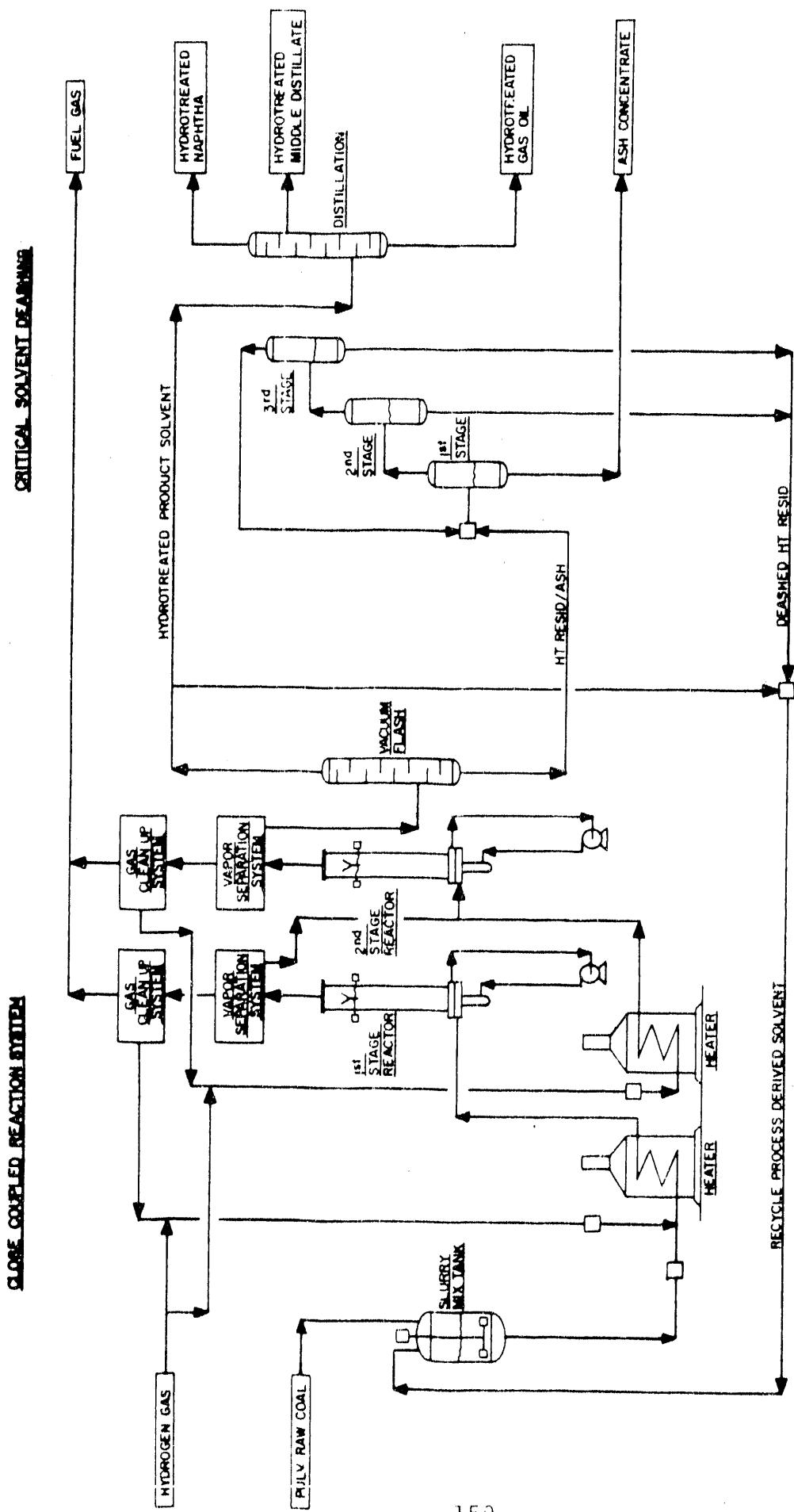


FIGURE 11. CLOSE COUPLED INTEGRATED TWO STAGE LIQUEFACTION SYSTEM

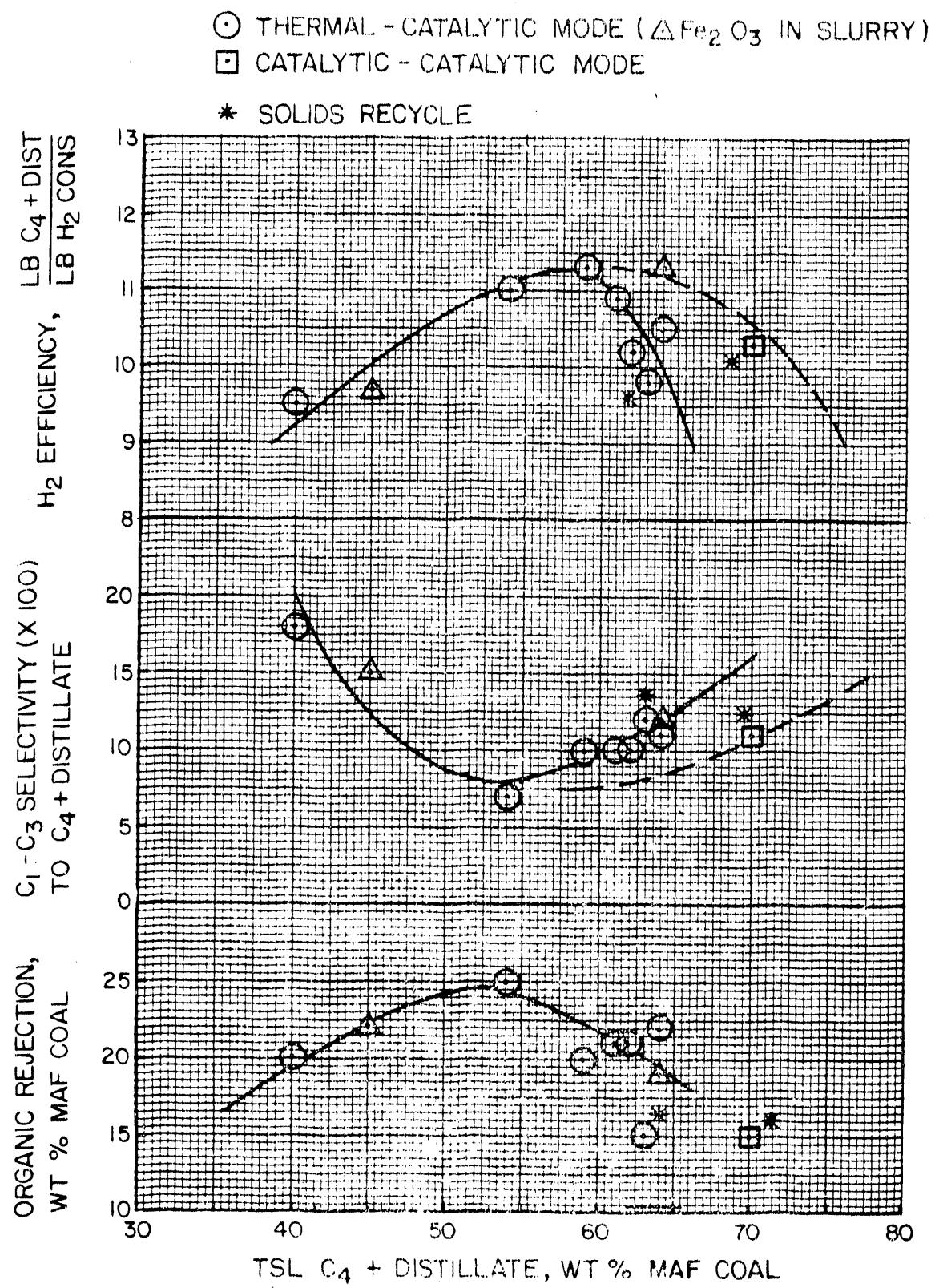


FIGURE 12. TSL SYSTEM PROCESS PERFORMANCE CHARACTERISTICS
(ILLINOIS NO. 6)

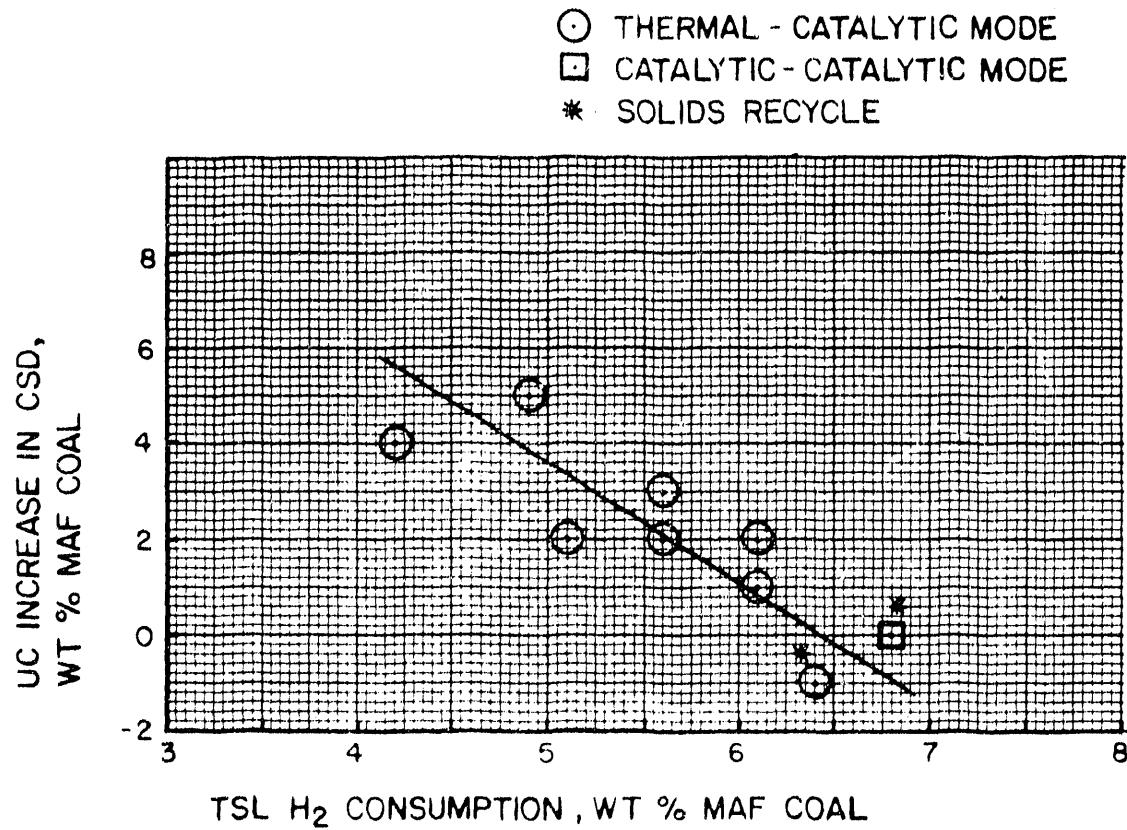


FIGURE 13. UC INCREASE IN CSD VS TSL H₂ CONSUMPTION
(ILLINOIS NO. 6)

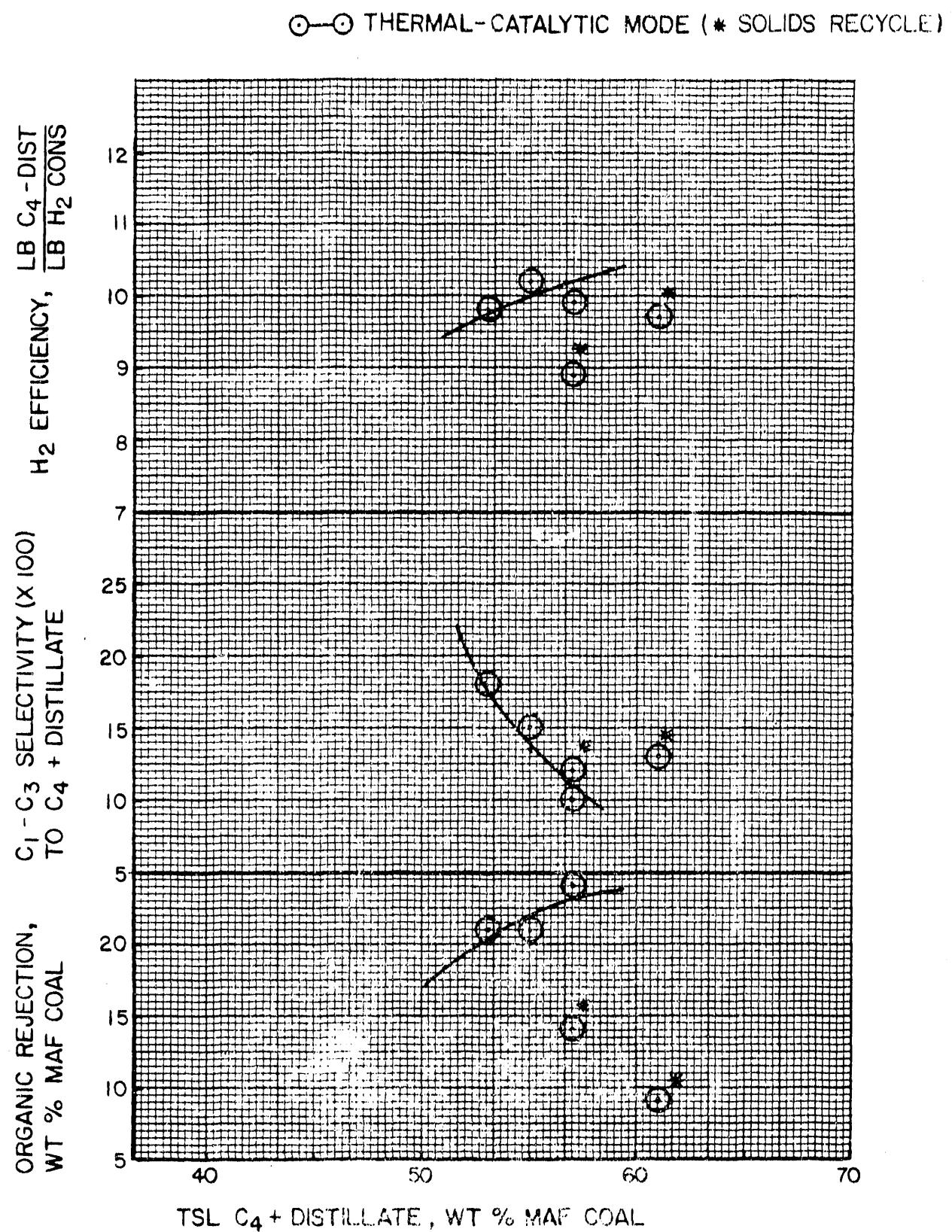


FIGURE 14. TSL SYSTEM PROCESS PERFORMANCE CHARACTERISTICS (WYODAK)

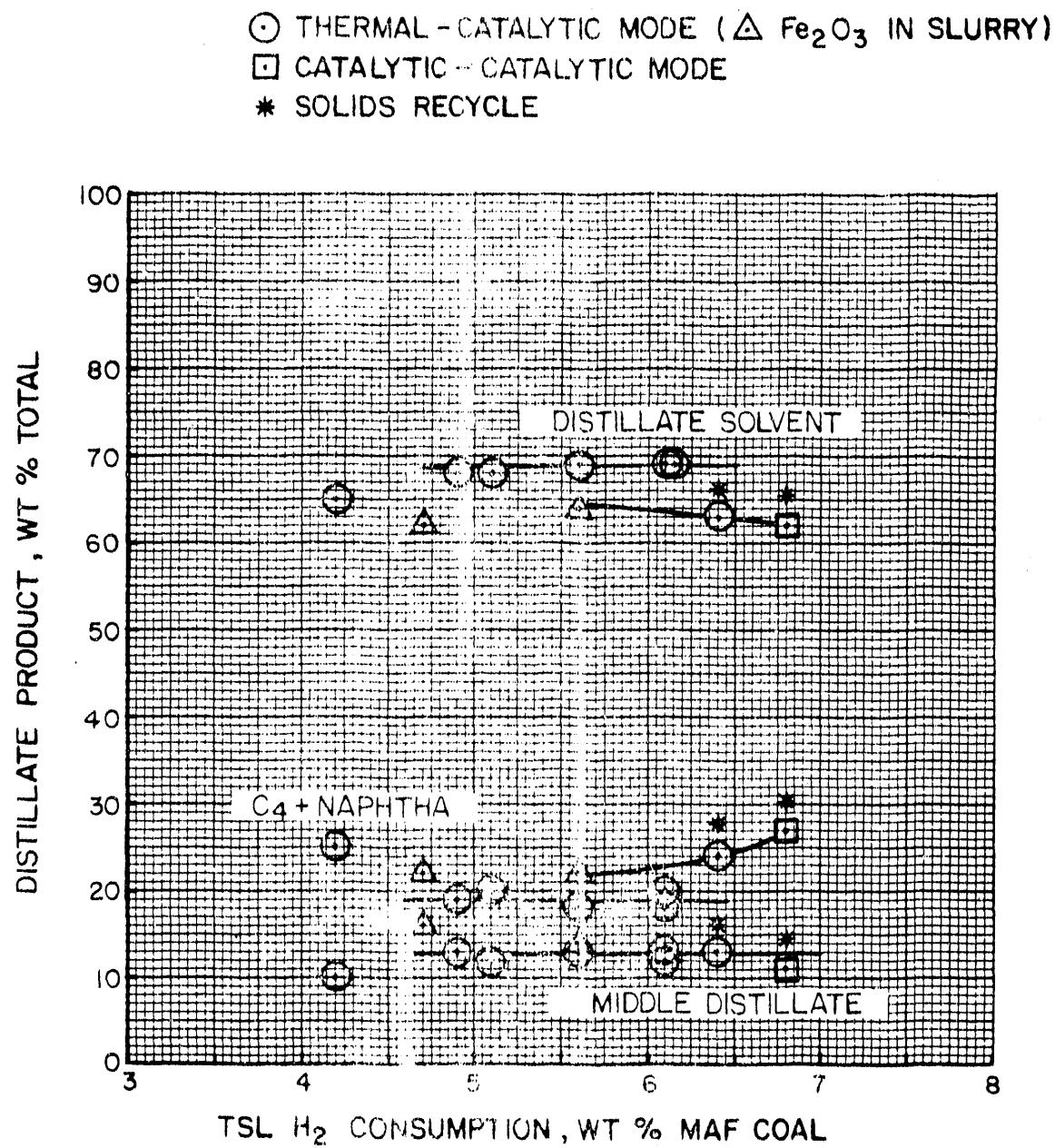


FIGURE 15. TSL SYSTEM DISTILLATE PRODUCT DISTRIBUTION
(ILLINOIS NO. 6)

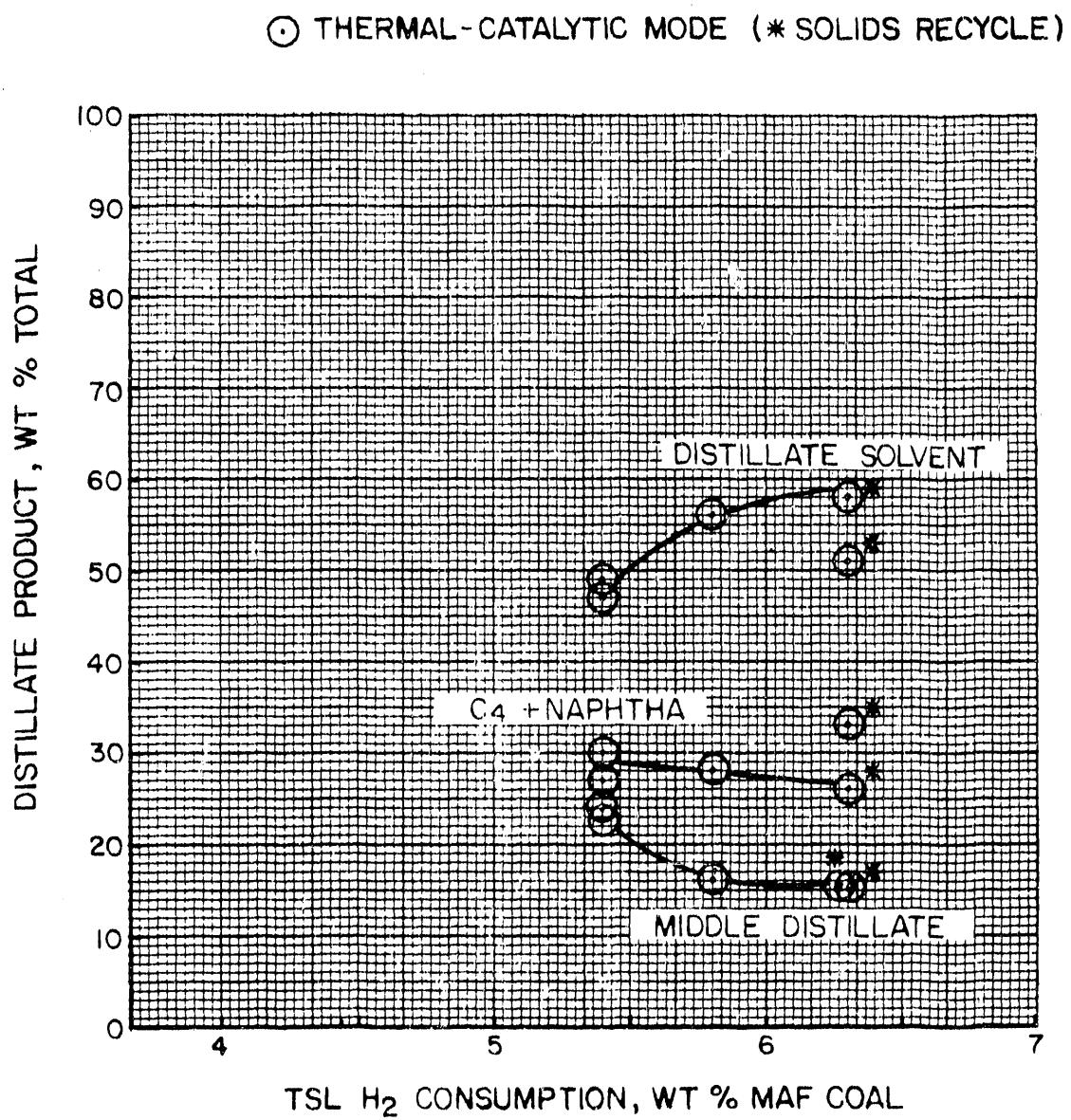


FIGURE 16. TSL SYSTEM DISTILLATE PRODUCT DISTRIBUTION (WYODAK)

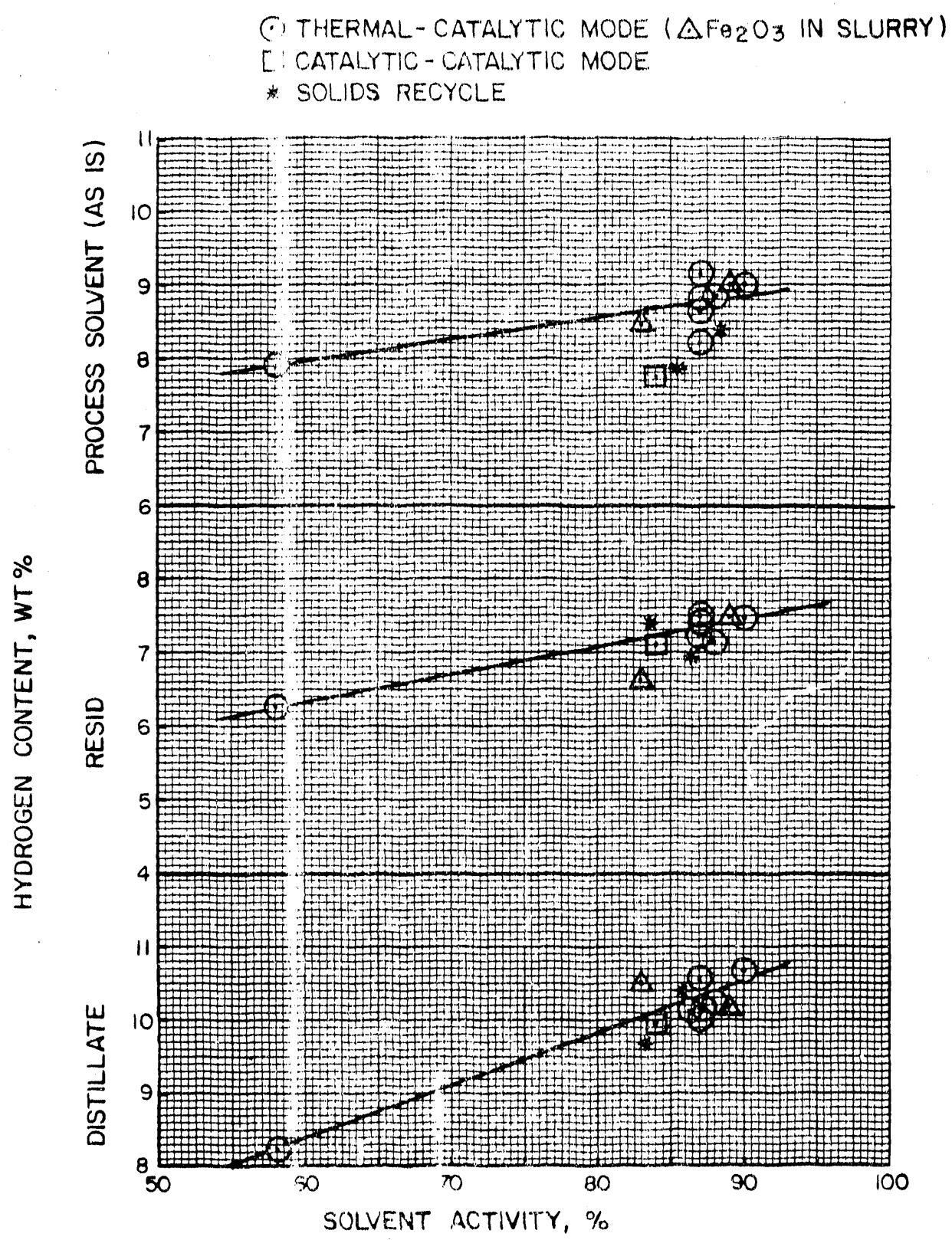


FIGURE 17. PROCESS SOLVENT ACTIVITY VS HYDROGEN CONTENT
(ILLINOIS NO. 6)

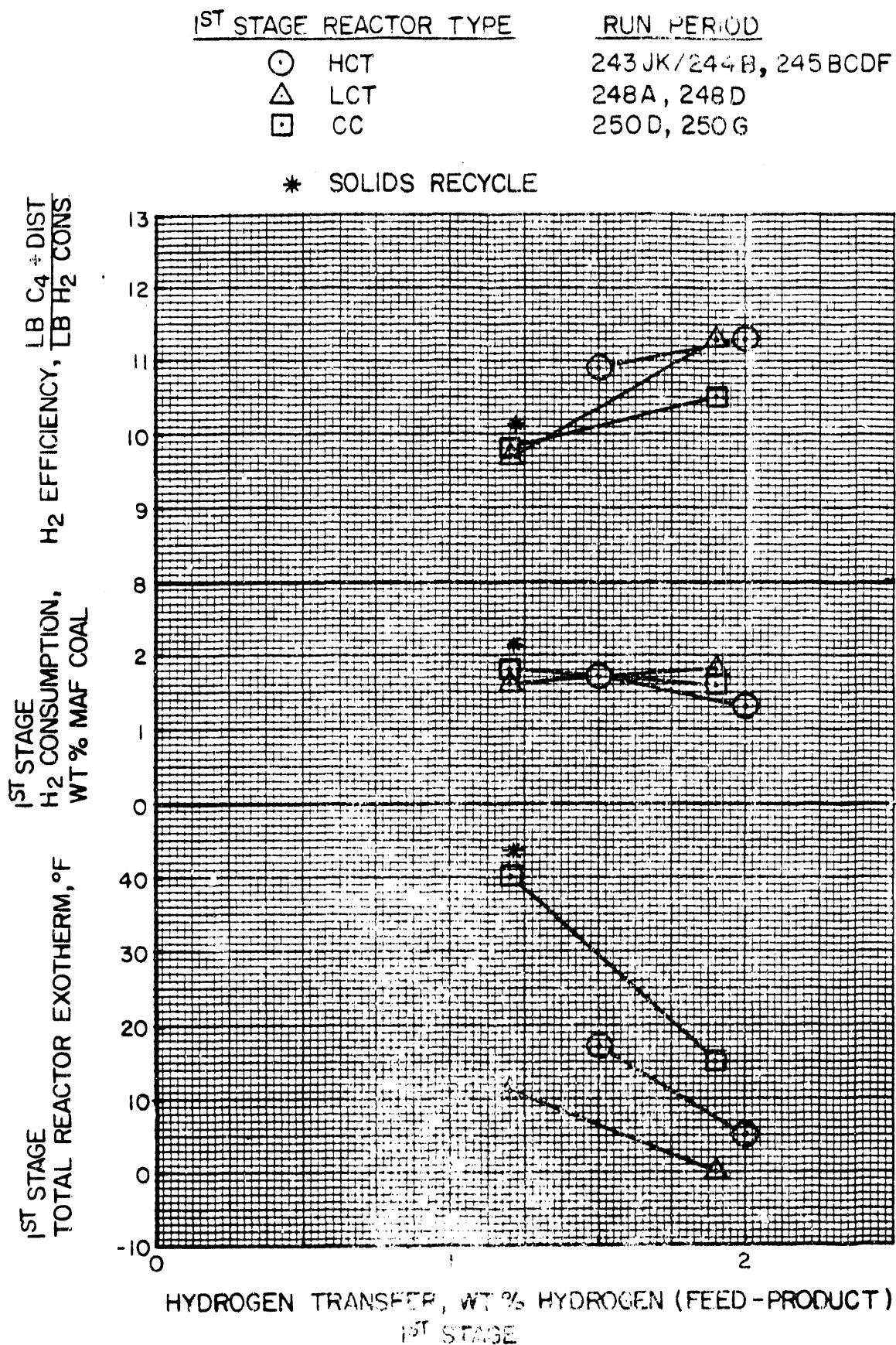


FIGURE 18. PROCESS SOLVENT HYDROGEN TRANSFER DATA
(ILLINOIS NO. 6)

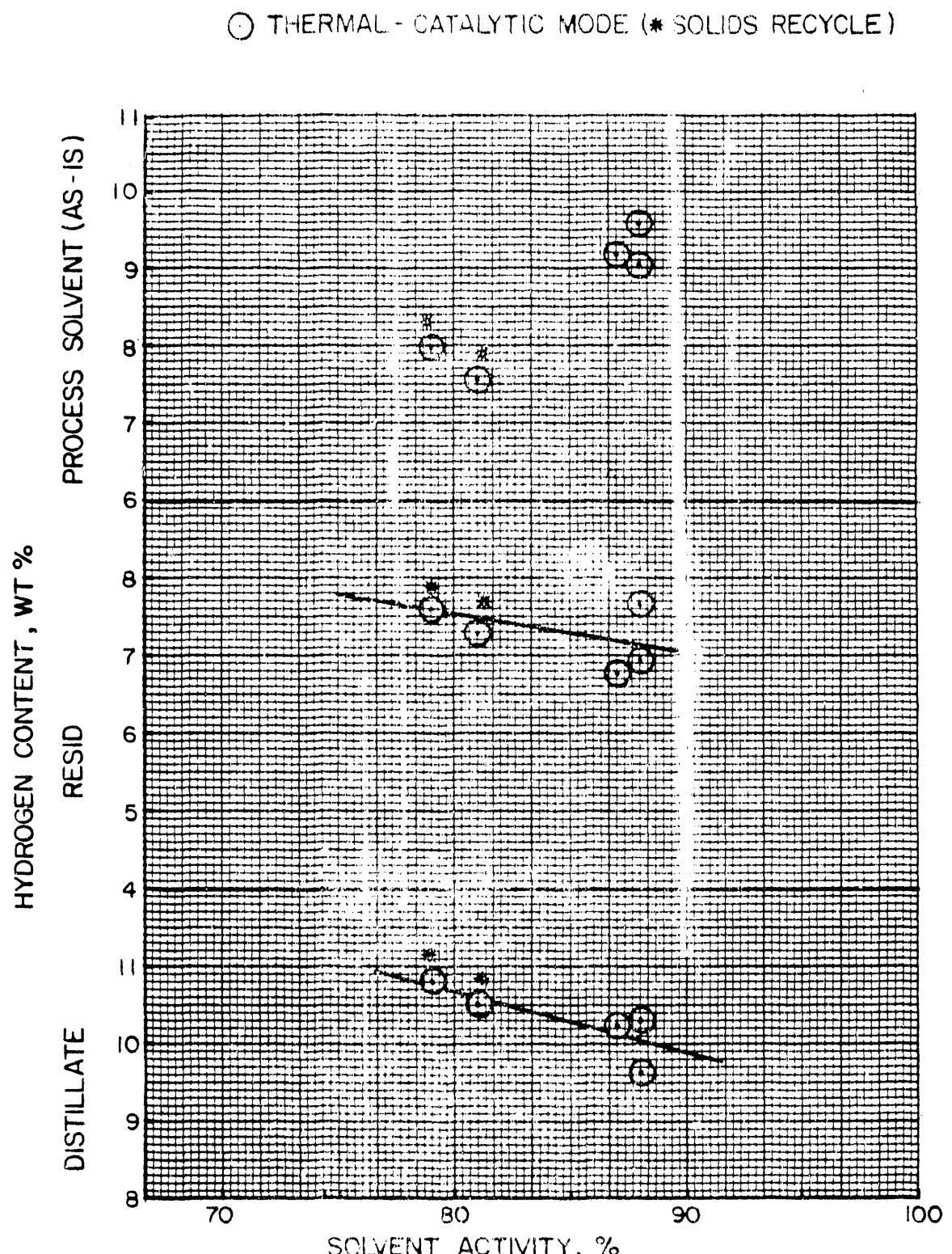


FIGURE 19. PROCESS SOLVENT ACTIVITY VS HYDROGEN CONTENT (WYODAK)

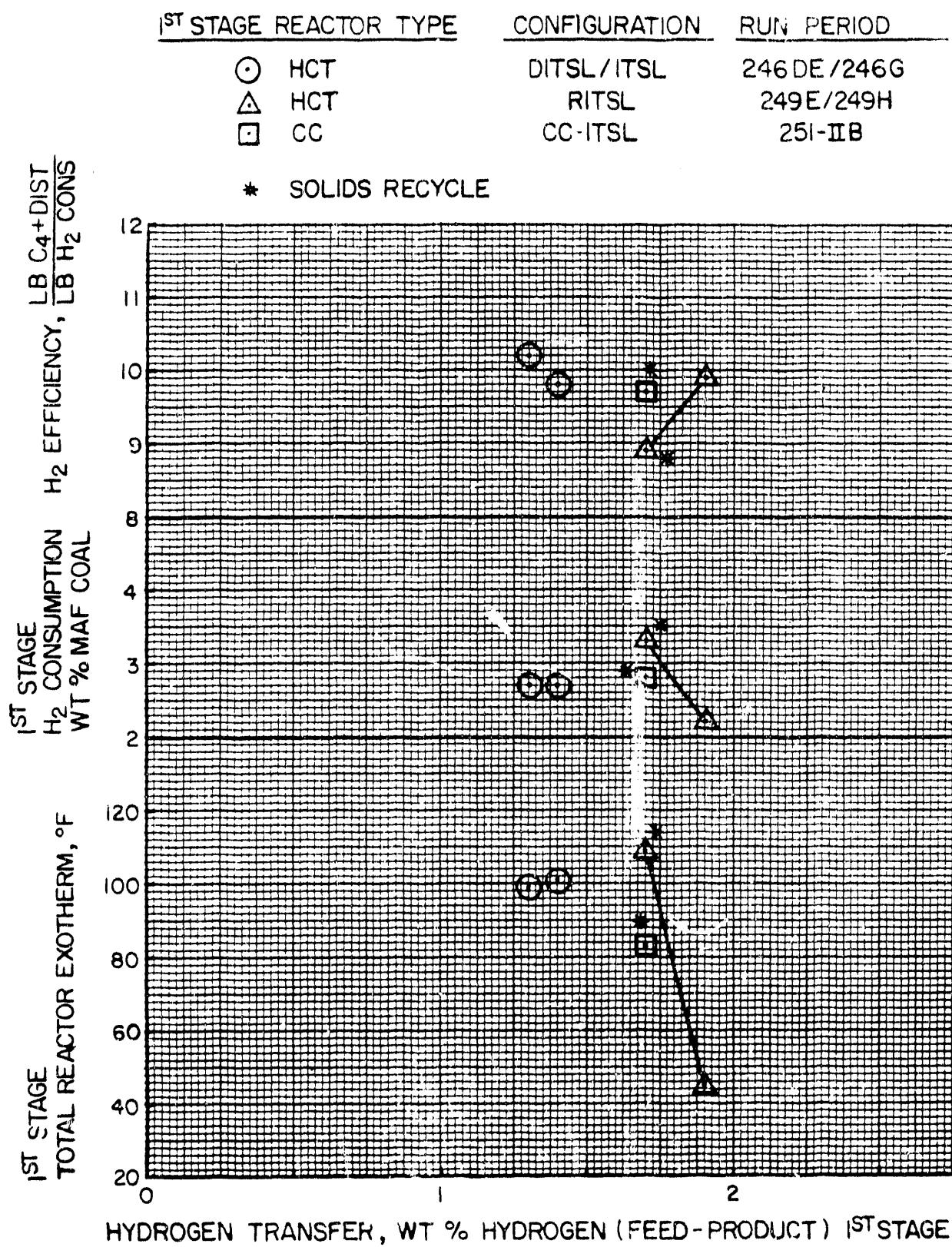


FIGURE 20. PROCESS SOLVENT HYDROGEN TRANSFER DATA (WYODAK)

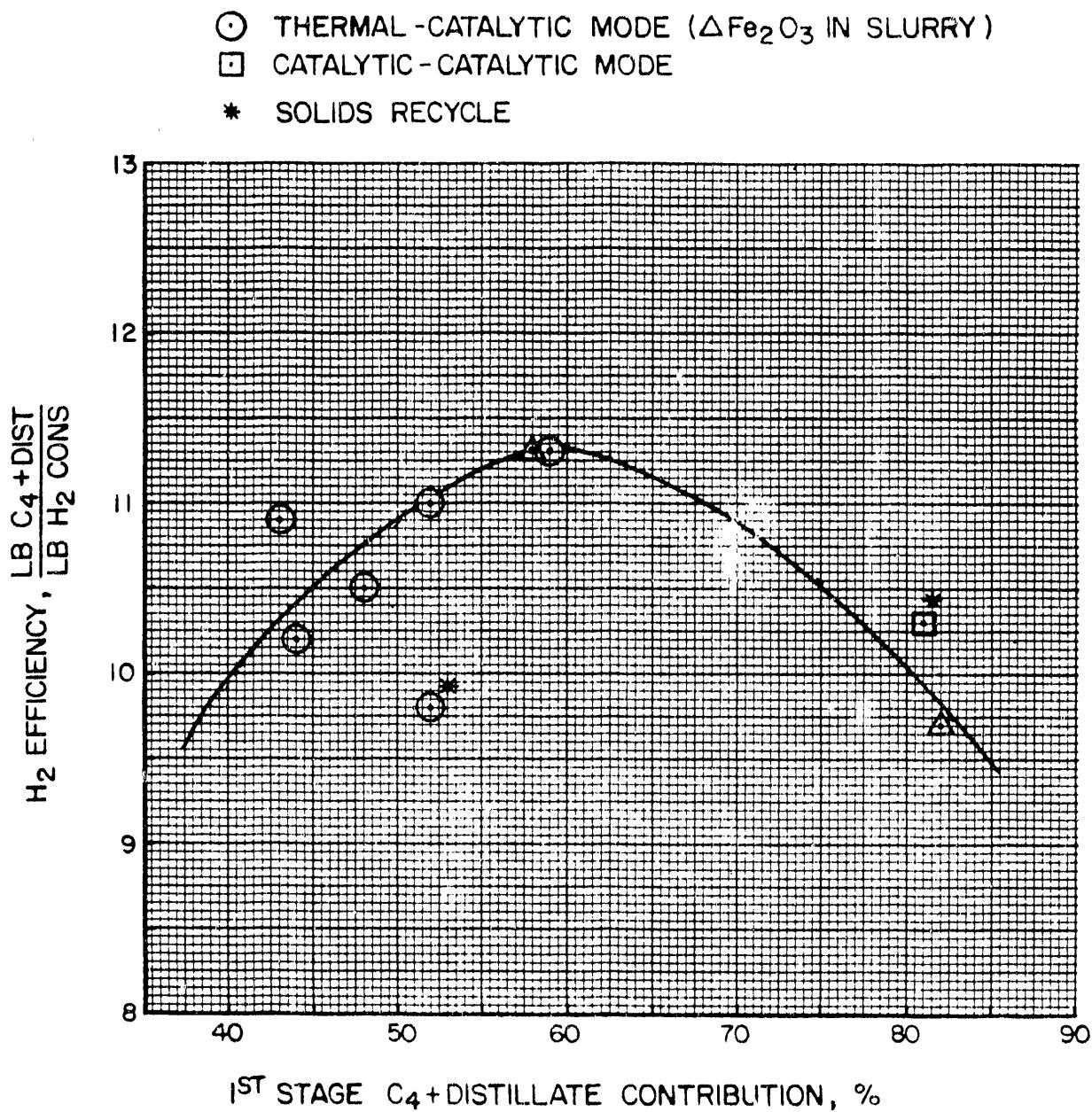


FIGURE 21. UNIT CONTRIBUTION EFFECT ON TSL HYDROGEN EFFICIENCY
(ILLINOIS NO. 6)

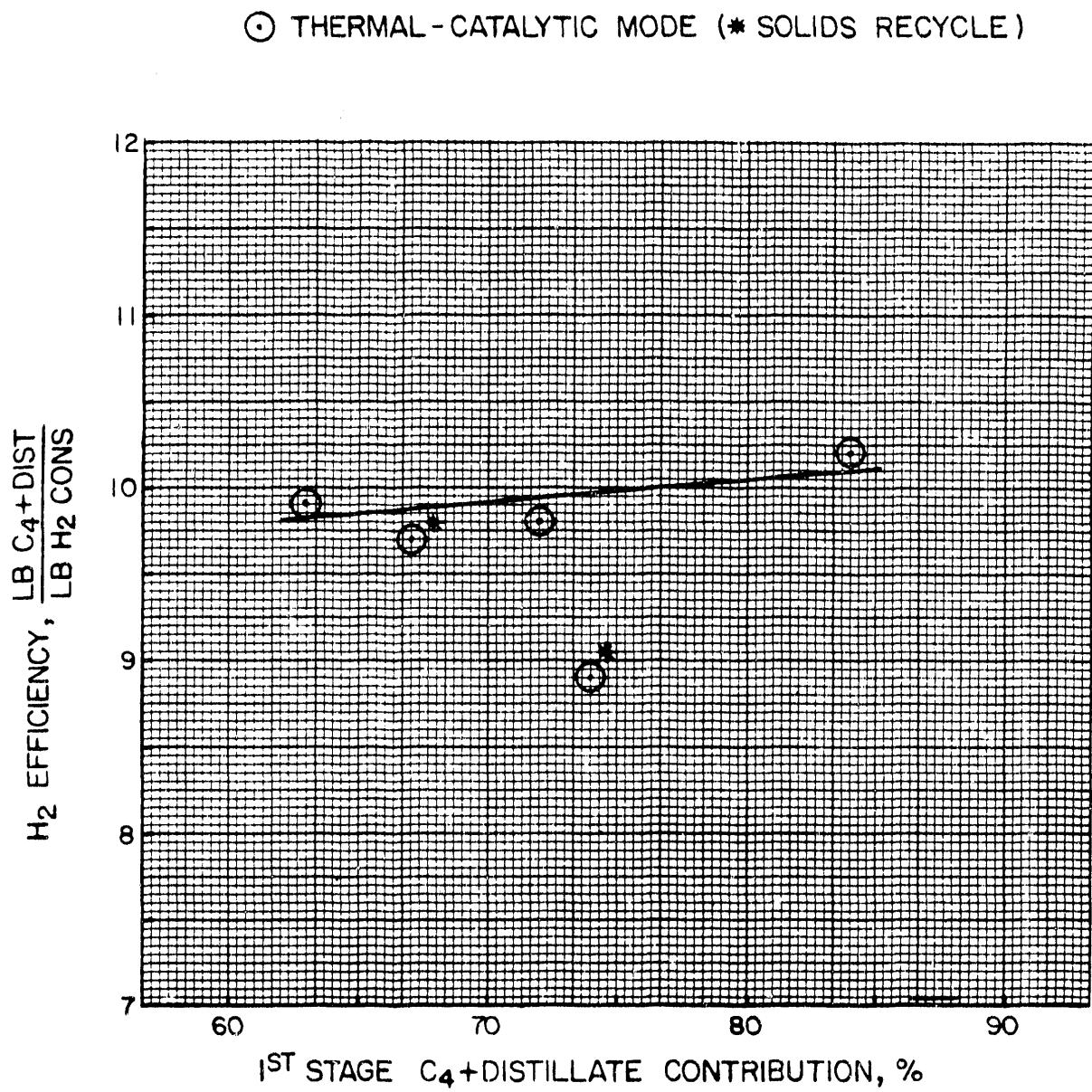


FIGURE 22. UNIT CONTRIBUTION EFFECT ON TSL HYDROGEN EFFICIENCY (WYODAK)

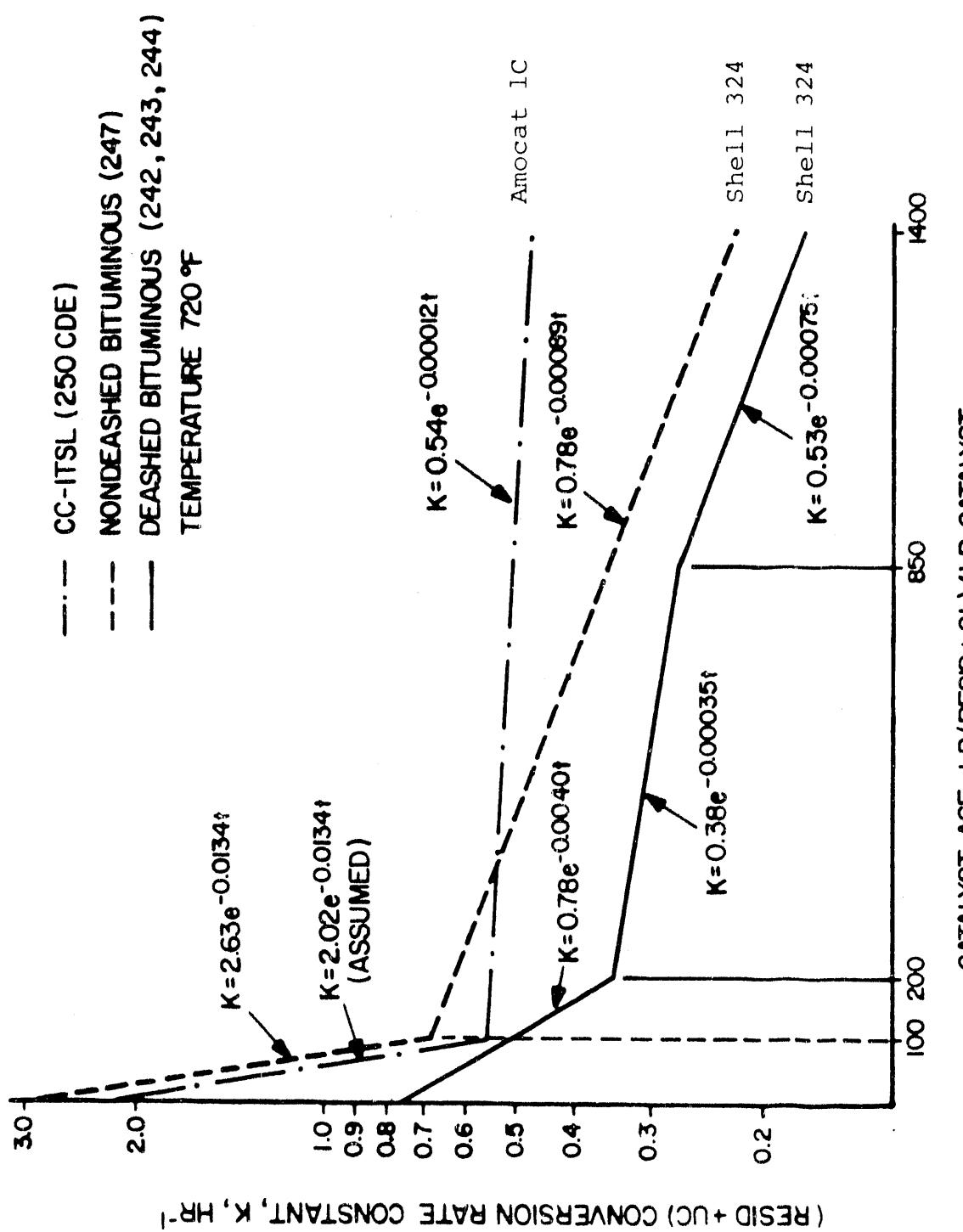


FIGURE 23. CATALYST ACTIVITY AND BATCP DEACTIVATION TRENDS

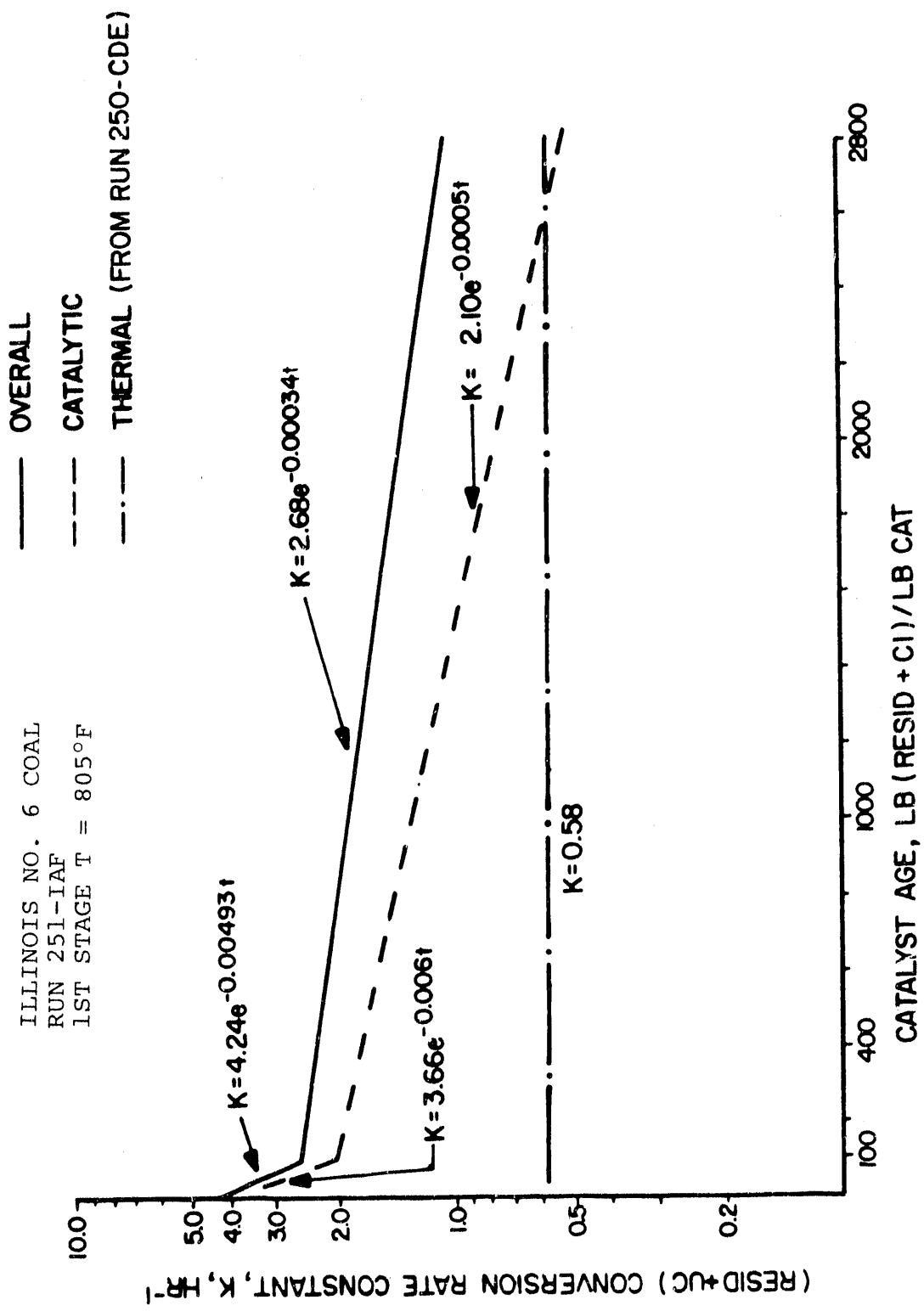


FIGURE 24. CATALYST ACTIVITY AND BATCH DEACTIVATION TRENDS

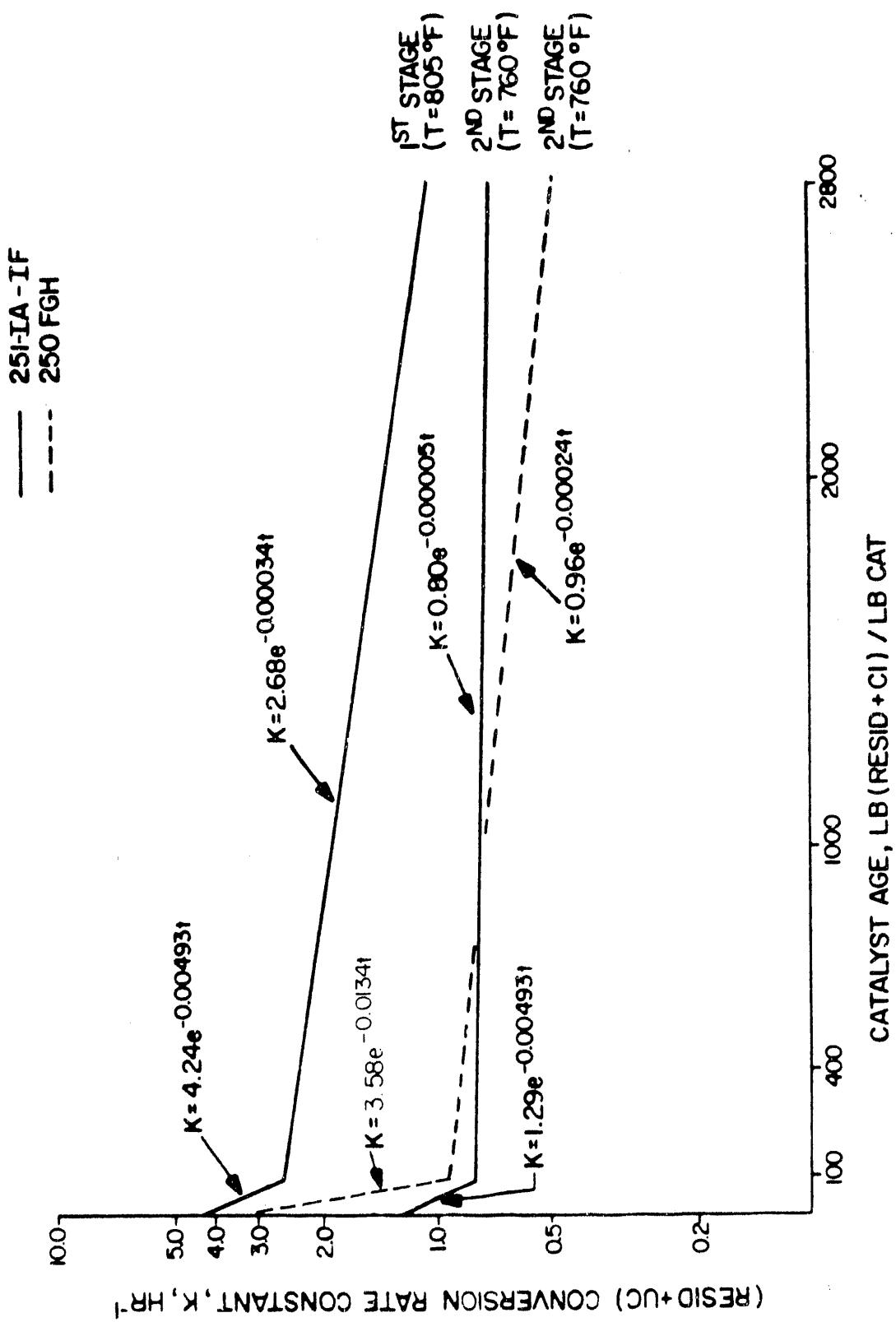


FIGURE 25. CATALYST ACTIVITY AND BATCH DEACTIVATION TRENDS

PARITY PLOT FOR MODEL VERIFICATION
COAL FEED RATE COMPARISON

Run 251-1 CC-TSL Catalytic
CSTR 1st order kinetics model verification

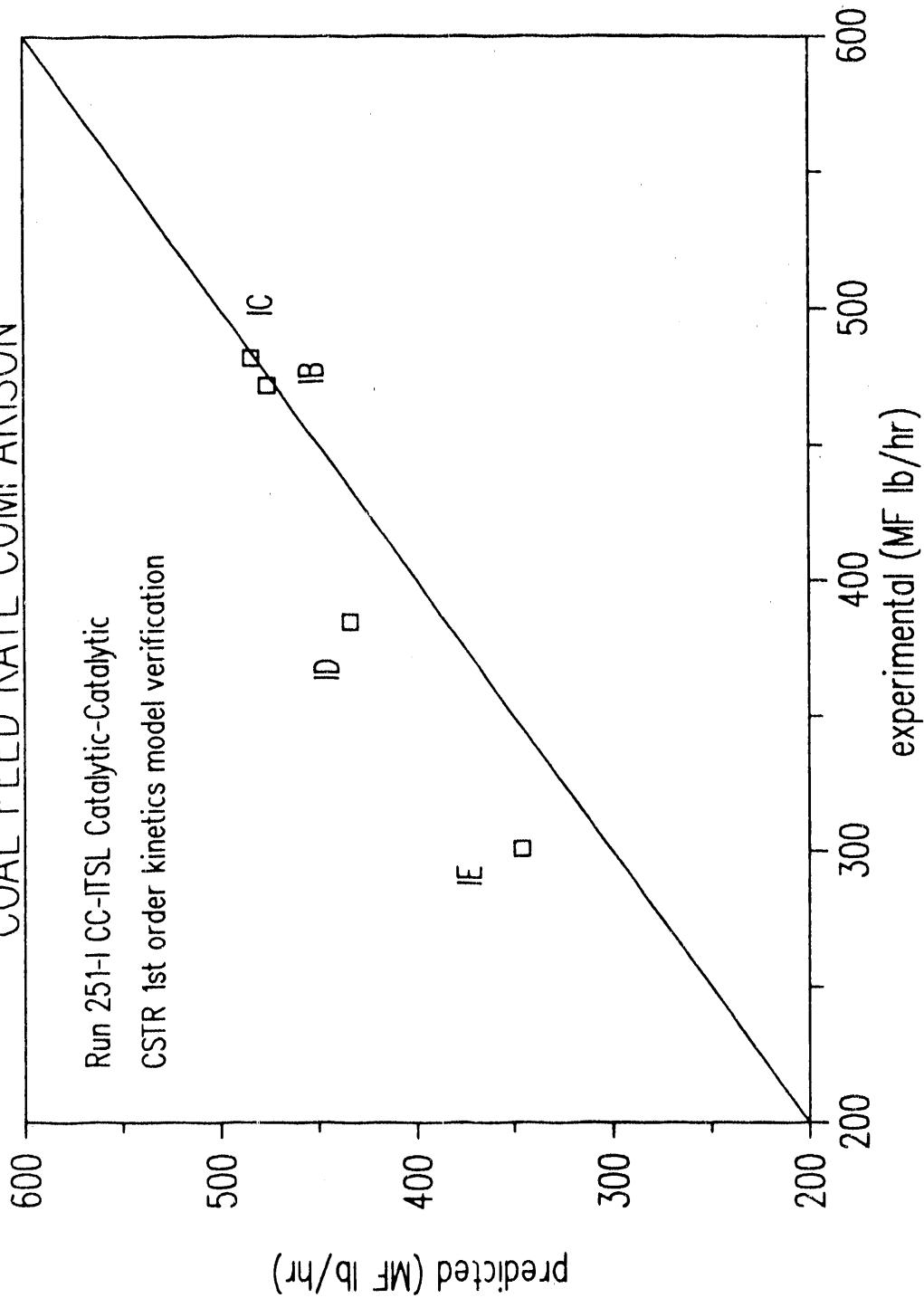


FIGURE 26. PARITY PLOT FOR MODEL VERIFICATION
- COAL FEED RATE COMPARISON

OVERALL TWO-STAGE CONVERSION ACTIVITY
RESID+UC CONVERSION PROJECTION

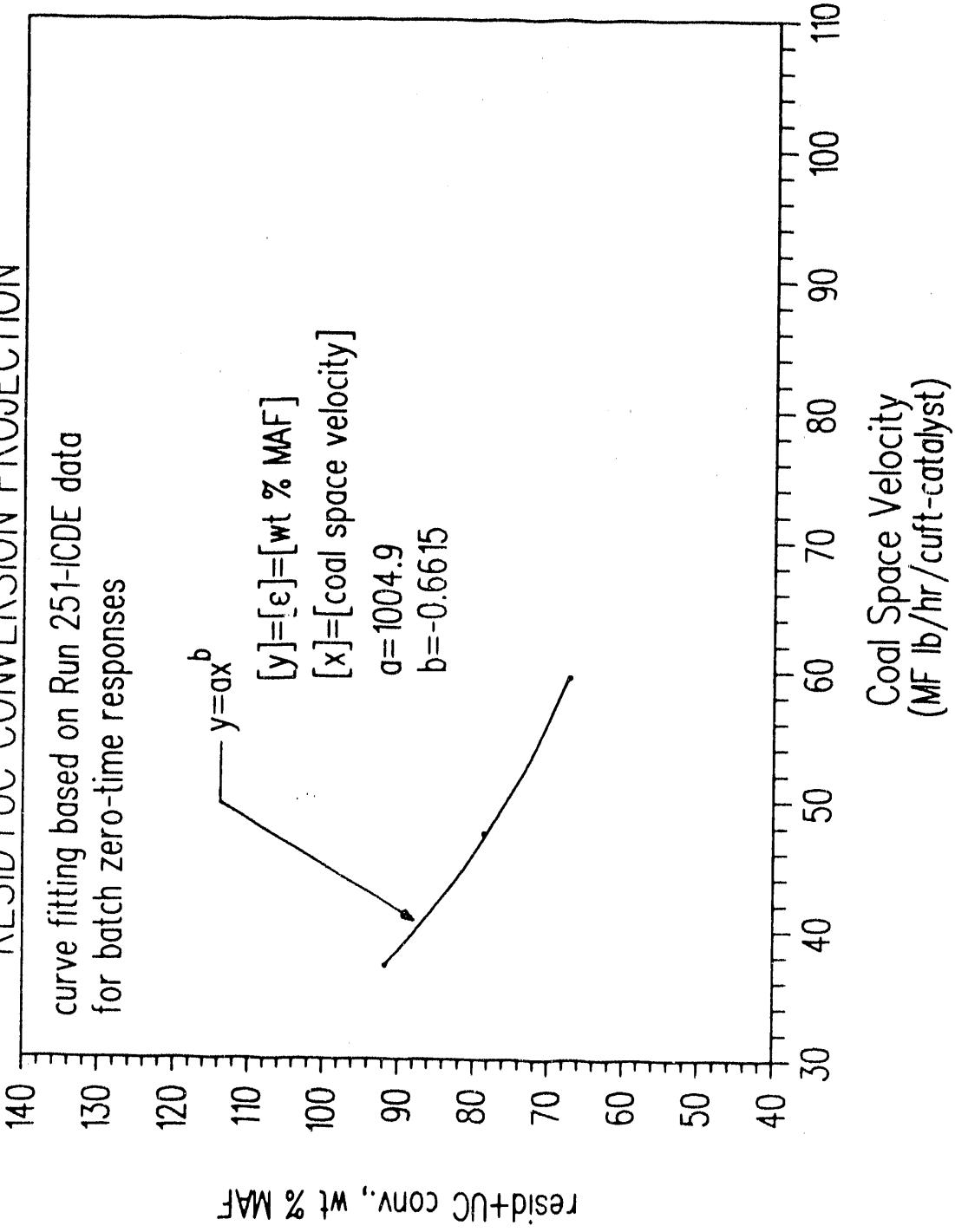


FIGURE 27. EFFECT OF COAL SPACE VELOCITY ON OVERALL TWO-STAGE RESID + UC CONVERSION (UNIT: WT % MAF) (RUN 251-ICDE)

CATALYST RESIDENCE TIME DISTRIBUTION
AT EQUILIBRIUM (250G)

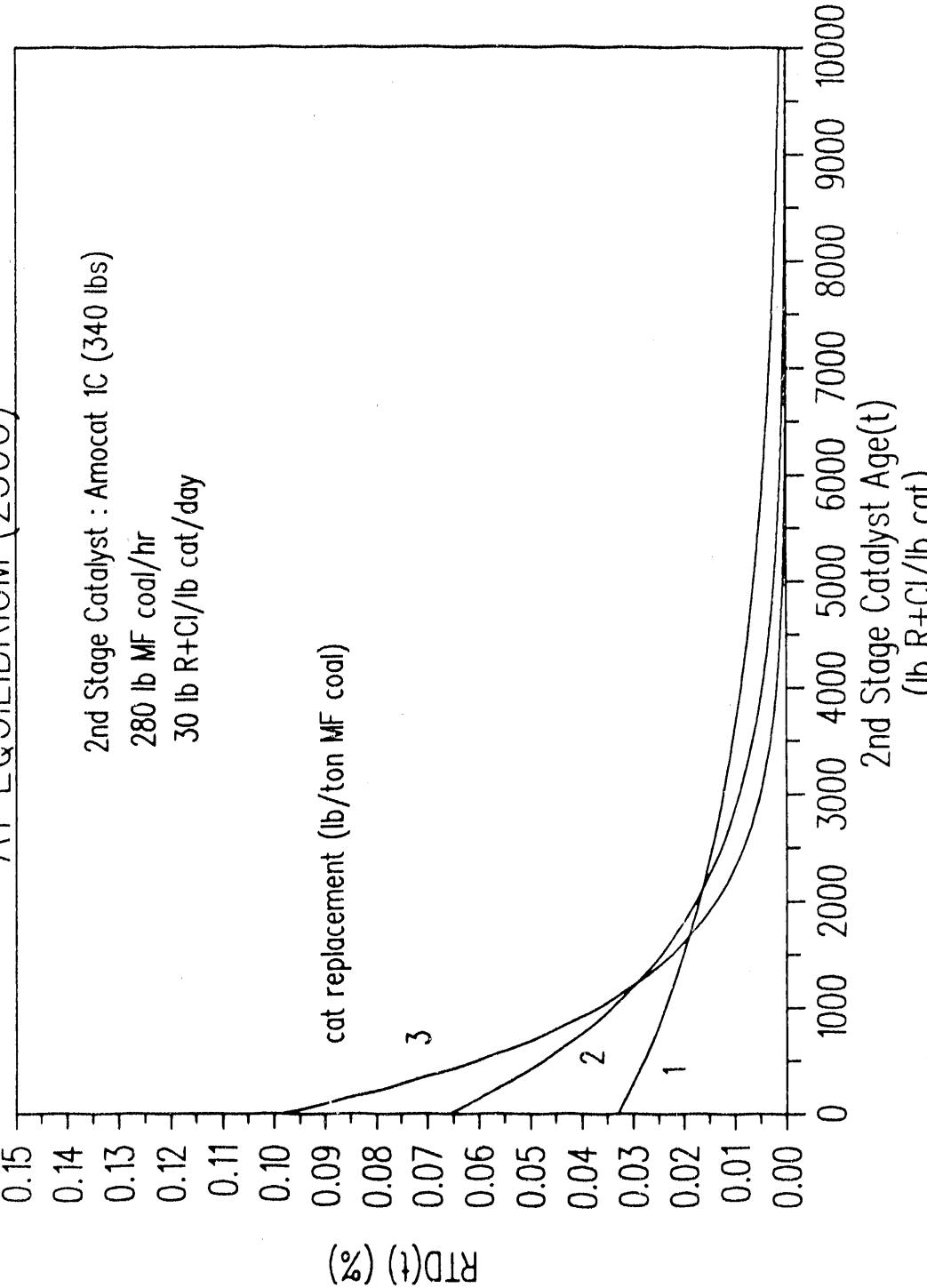


FIGURE 28. CALCULATED EQUILIBRIUM CATALYST AGE DISTRIBUTION
- RUN 250G SECOND STAGE

CATALYST RESIDENCE TIME DISTRIBUTION
AT EQUILIBRIUM (251-IC)

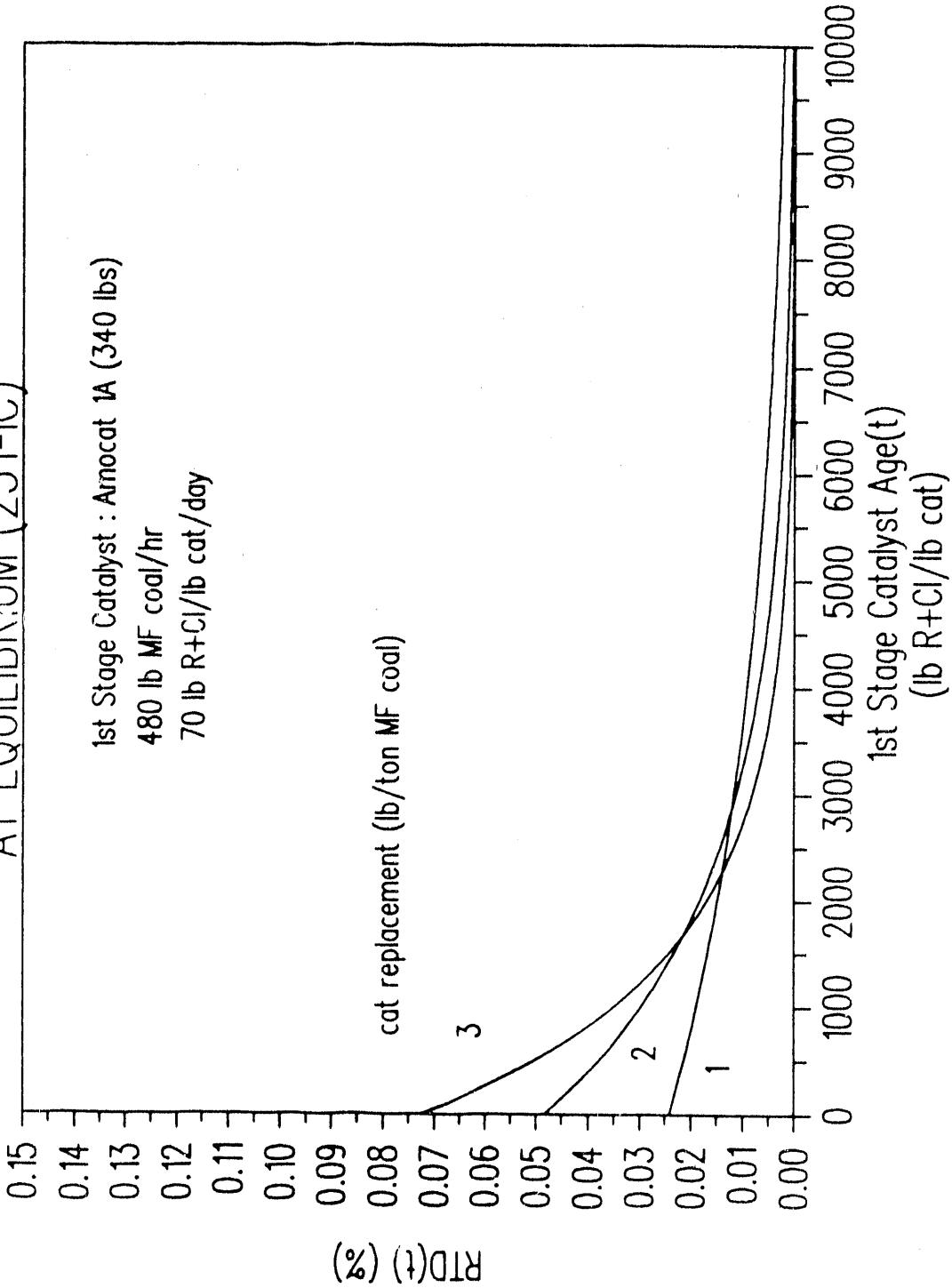


FIGURE 29. CALCULATED EQUILIBRIUM CATALYST AGE DISTRIBUTION
- RUN 251-IC FIRST STAGE

CATALYST RESIDENCE TIME DISTRIBUTION
AT EQUILIBRIUM (251-IC)

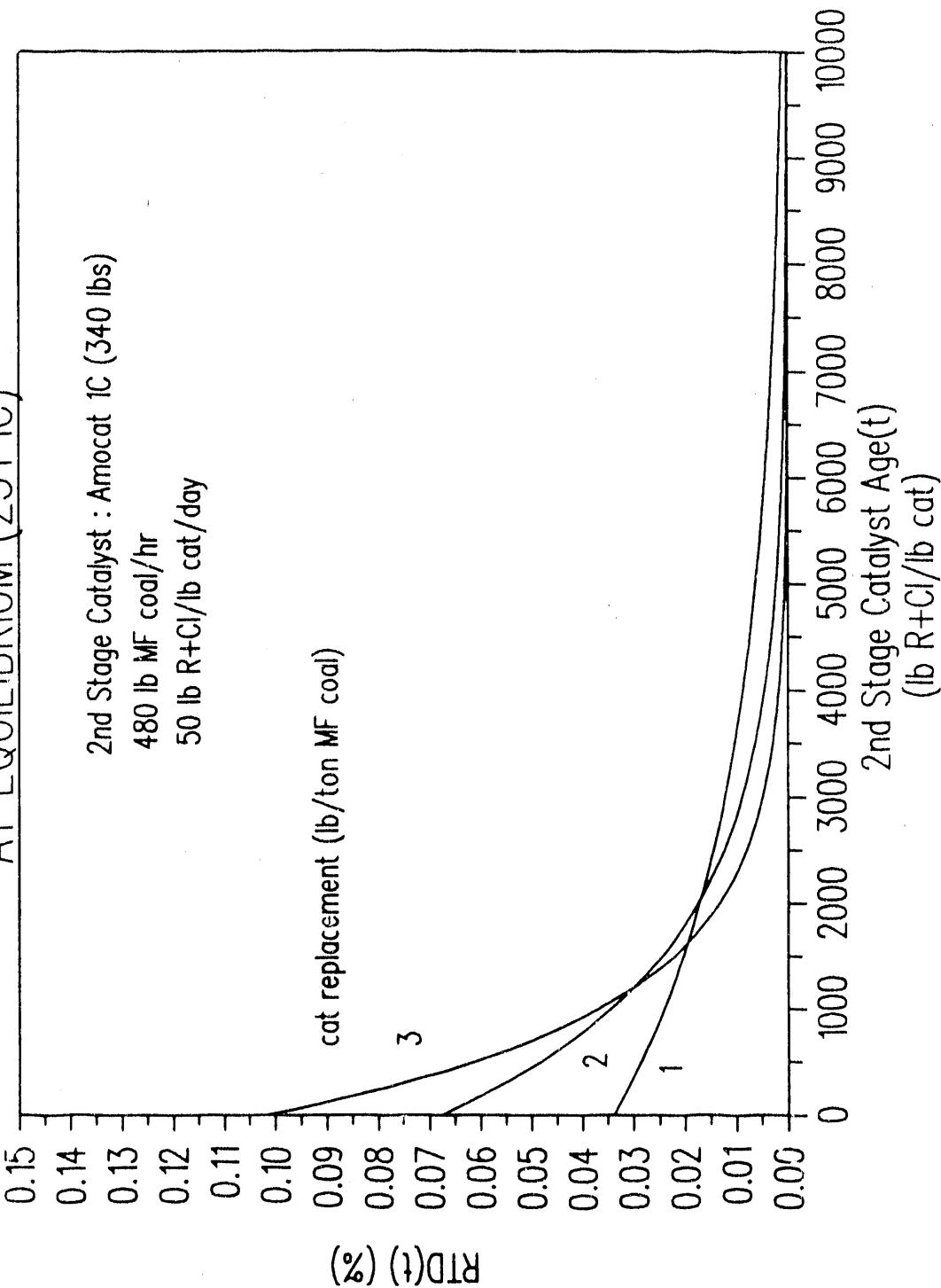


FIGURE 30. CALCULATED EQUILIBRIUM CATALYST AGE DISTRIBUTION
- RUN 251-IC SECOND STAGE

CATALYST RESIDENCE TIME DISTRIBUTION
AT EQUILIBRIUM (243JK/244B)

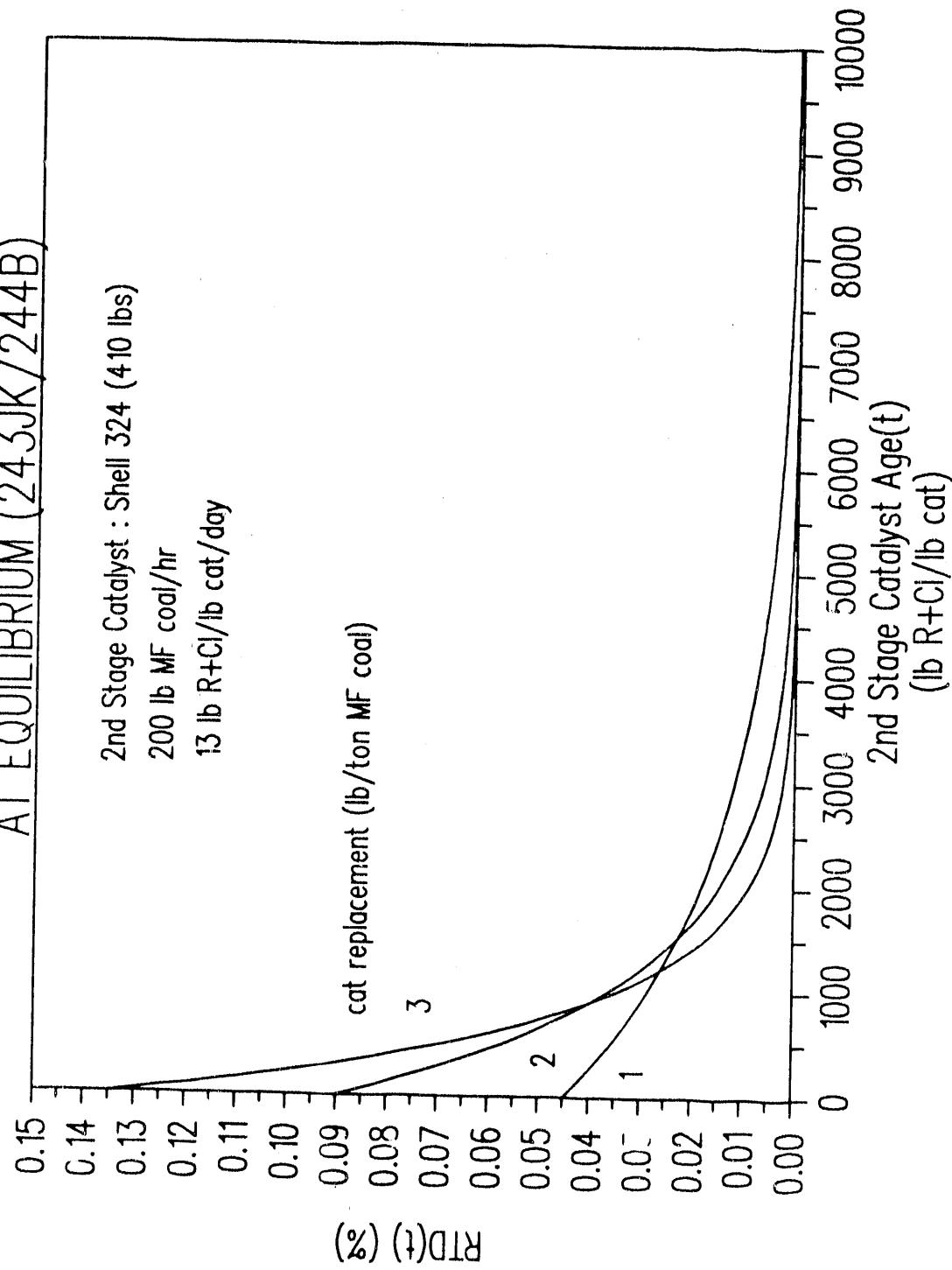


FIGURE 31. CALCULATED EQUILIBRIUM CATALYST AGE DISTRIBUTION
- RUN 243JK/244B

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101/07/91

