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ADVANCED COAL LIQUEFACTION

RESEARCH AND DEVELOPMENT FACILITY

Wilsonville, Alabama

TECHNICAL PROGRESS REPORT

Run 261 with Illinois No. 6 Burning Star Mine Coal

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PREFACE

Operation of the Advanced Coal Liquefaction R & D Facility at the Clean Coal Research Center in Wilsonville, Alabama, is funded by the U. S. Department of Energy (DOE), the Electric Power Research Institute (EPRI), and Amoco Corporation. Southern Company Services, Inc. (SCS) manages the Wilsonville program on behalf of DOE, EPRI and Amoco.

The Wilsonville R & D Facility combines two process units: a Close-Coupled Reactors (CCR) unit and a Residuum Oil Supercritical Extraction - Solids Rejection (ROSE-SR℠) unit. The CCR unit uses H-Oil® technology, developed by Hydrocarbon Research, Inc. (HRI) and was constructed and modified by Catalytic, Inc. to allow close-coupled operation. The modification primarily consisted of adding a second reactor in close proximity to a pre-existing reactor. These close-coupled reactors can be used for various modes of operation --- thermal/catalytic, catalytic/catalytic. The ROSE-SR℠ unit uses a proprietary solid-liquid separation process developed by the Kerr-McGee Corporation. The process separates ash and unconverted coal (UC) from resid as a heavy fluid phase, termed bottoms product, using a deashing solvent near its critical point. The combined two-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 first and second stage can be independently varied, allowing for improvement in product slate flexibility. During Run 261, emphasis was placed on achieving high distillate yields at high coal throughputs in a bituminous coal liquefaction operation.

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ABSTRACT

This report presents the results of Run 261 performed at the Advanced Coal Liquefaction R & D Facility in Wilsonville, Alabama. The run started on January 12, 1991 and continued until May 31, 1991, operating in the Close-Coupled Integrated Two-Stage Liquefaction mode processing Illinois No. 6 seam bituminous coal (from Burning Star #2 mine).

In the first part of Run 261, a new bimodal catalyst, EXP-AO-60, was tested for its performance and attrition characteristics in the catalytic/catalytic mode of the CC-ITSL process. The main objective of this part of the run was to obtain good process performance in the low/high temperature mode of operation along with well-defined distillation product end boiling points. Higher thermal severity operation with higher reaction temperatures allowed higher coal throughput, resulting in higher distillate production. "All-distillate" product slate was achieved in two different test periods during steady state operation with catalyst replacement rates of 3 and 1.5 lb/ton MF coal. The data at the two different catalyst replacement rates provided information for tradeoff calculations between catalyst cost and reactor cost. Compared to Runs 257 and 259, a 27-53% increase in distillate production rate was obtained with EXP-AO-60 catalyst operation in Run 261. The EXP-AO-60 catalyst attrition was low and was comparable to unimodal catalysts.

In the second part of Run 261, Criterion (Shell) 324 catalyst was tested. The objective of this test was to evaluate the operational stability and catalyst and process performance while processing the high ash Illinois No. 6 coal. Increasing viscosity and preasphaltenes made it difficult to operate at conditions similar to EXP-AO-60 catalyst operation, especially at lower catalyst replacement rates. At conditions operated, the distillate production rates were 10-24% lower with the Criterion catalyst compared to EXP-AO-60 catalyst. The interpretation of results based on calculated rate constants and deactivation rates might be biased negatively because of uncertainty in catalyst charge in the second reactor (only 77% of catalyst charged was recovered at the end of the run). The average end boiling point of distillate product was 764°F and 786°F during EXP-AO-60 and Criterion catalyst operations. The lower hydrogen content observed in the process streams and lower nitrogen removal found in the products suggested that hydrogenation was being constrained by thermodynamic limitations at the higher temperatures employed with the Criterion catalyst.

A new continuous resid injection system was tested near the end of Run 261 in order to reduce equilibration time for the data periods. In this system, coal was batch slurried with heavy gas oil 2(V1074) or V1074+deashed resid, and the stream containing

resid and solids (vacuum flash bottoms) and deashed resid were continuously injected into the reactor feed line. The best operable conditions were determined. For smooth operations, the maximum coal concentration was 45 wt % in V1074+deashed resid which would give 30 wt % coal concentration in slurry to reactor feed.

The close-coupled reactor unit was on-stream for 2333 hours for an on-stream factor of 85.4% and the ROSE-SR℠ unit was on-feed for 2240 hours for an on-stream factor of 99.0% for the entire run.

1. INTRODUCTION

The Close-Coupled Integrated Two-Stage Liquefaction (CC-ITSL) mode was used in Run 261 in the catalytic/catalytic, low/high temperature mode of operation. Prior runs in the CC-ITSL configuration include Runs 250-260 (Ref. 1-11).

In the CC-ITSL mode, the product from the first stage reactor is sent directly to the second stage reactor, without depressuring or deashing. In Run 261, a high pressure gas-liquid flash separator was used between the two stages to remove the recycle and vent gases, water, and light hydrocarbons that were produced in the first stage. The remainder of the first stage product was sent to the second stage.

The effluent from the second stage is separated using three flashes: a high pressure flash, an atmospheric flash, and a vacuum flash. Part of the bottoms from the vacuum flash, which contains resid, unconverted coal, and ash, is fed to the Residuum Oil Supercritical Extraction - Solids Rejection (ROSE-SR^{SM}) unit. The remaining portion is recycled. The solids-free resid recovered in the ROSE-SR^{SM} unit is recycled with second stage distillate and the vacuum flashed bottoms for coal slurry preparation. A simplified flow diagram of the CC-ITSL process is shown in Figure 1.

In the first two test periods, atmospheric flashed bottoms were used for preparing the process solvent. In other test periods, the vacuum flashed bottoms were used to obtain 12 wt % solids concentration in the process solvent. This allowed operation with all the distillate routed to the distillation system.

Two different supported catalysts were tested in this run. In the first part of the run, a new bimodal catalyst, EXP-AO-60 (developed by Amoco Oil Company), was tested for its performance and attrition characteristics. The unimodal Criterion (Shell) 324 catalyst was tested in the second part of the run to evaluate its performance in the liquefaction of the high ash Illinois coal.

Near the end of the run, a new continuous resid injection system was tested. In this system, coal was slurried with either the heavy gas oil or with a mixture of heavy gas oil and deashed resid. The vacuum flash underflow stream containing the solids were injected directly into the preheater/reactor feed line. If the deashed resid was not used for slurrying coal, then it was also injected continuously into the preheater/reactor feed line.

In the following, a brief summary of Integrated Two-Stage Liquefaction (ITSL) runs conducted at Wilsonville is given:

Table A. Summary of Runs Since 242

Run	Coal	Configuration	Comments/Process Mode
242	Illinois No. 6	ITSL[1]	SCT[2]
243	Illinois No. 6	ITSL	
244	Illinois No. 6	ITSL	
245	Illinois No. 6	ITSL	On-line cat. replacement in second stage
246	Wyoming	DITSL/ITSL	Iron oxide addition in first stage[3]
247	Illinois No. 6	RITSL[4]	First stage dissolver tracer study
248	Illinois No. 6	DITSL/ITSL	Low Contact Time liquefaction[5]
249	Wyoming	RITSL[4]	Forced back-mixed dissolver
250	Illinois No. 6	CC-ITSL	Thermal/Catalytic
251	Ill./Wyo.	CC-ITSL	Thermal/Catalytic and Catalytic/Catalytic[6]
252	Illinois No. 6	CC-ITSL	Catalytic/Catalytic
253	Illinois No. 6	CC-ITSL	Catalytic/Catalytic
254	Ohio No. 6	CC-ITSL	Catalytic/Catalytic
255	Texas Lignite	CC-ITSL	Thermal/Catalytic and Catalytic/Catalytic
256	Ohio No. 6	CC-ITSL	Catalytic/Catalytic[7]
257	Illinois No. 6	CC-ITSL	Catalytic/Catalytic[8]
258	Bl. Thunder/Sp. Creek	CC-ITSL	Thermal/Catalytic[9,10]
259	Pittsburgh No. 8	CC-ITSL	Catalytic/Catalytic
260	Black Thunder	CC-ITSL	Thermal/Catalytic and Catalytic/Thermal[11]
261	Illinois No. 6	CC-ITSL	Catalytic/Catalytic[12]

(1) In Runs 242-250, the first stage was a thermal liquefaction stage and the second stage was a catalytic stage.
(2) SCT denotes Short Contact Time liquefaction. A dissolver was not used.
(3) Iron oxide and dimethyl disulfide (to form sulfided iron catalyst in first stage) are usually added in low rank coal runs
(4) RITSL was a precursor to CC-ITSL. RITSL differs from CC-ITSL in that the reactor interstage stream is cooled and depressured.
(5) A 5.2" ID dissolver was used.
(6) Catalytic/catalytic mode denotes that a supported catalyst was present in both reactors.
(7) On-line catalyst replacement capability added to second reactor.
(8) Also tested half-volume reactors and low/high temperature severity operation.
(9) Spring Creek/Black Thunder Mine coals from Powder River Basin.
(10) Half-volume reactors were used throughout the run.
(11) Full-volume thermal stage and three-quarters-volume catalytic stage.
(12) EXP-AO-60 and Criterion catalysts used.

Figure 2 shows, in a block diagram form, all of the operating modes tested at Wilsonville since Run 242. Figures 3, 4, and 5 are flow diagrams of the coal slurry preparation system, ebullated bed reactor, and the ROSE-SR^{SM} unit, respectively. The nomenclature and definitions are given in Appendix A.

Objectives

The primary objectives of Run 261 were:

- To obtain high performance in the CC-ITSL process using catalytic/catalytic and low/high temperature modes of operation with Illinois No. 6 coal and a new bimodal catalyst, EXP-AO-60.
- To test a new bimodal catalyst (EXP-AO-60 1/16" cylindrical extrudate) and evaluate catalyst performance.
- To obtain a well defined end point for the distillate product processing Illinois No. 6 coal.
- To obtain data points at steady-state operation with catalyst replacement using Criterion (Shell) 324 unimodal catalyst while processing Illinois No. 6 coal.

The main objective of this part of the run was to obtain good process performance in the low/high temperature mode of operation along with well-defined distillation product end boiling points. In the low/high severity operation, the temperature constraint on the EXP-AO-60 catalyst will not be exceeded and at the same time certain thermal efficiencies projected can be implemented in the tests. Higher thermal severity operation with higher reaction temperatures will allow higher coal throughput, resulting in higher distillate production. Run 261 was the first run in which fresh catalyst was batch deactivated in the low/high severity operation mode.

The EXP-AO-60 catalyst used in this run is similar to the 1/12" Amocat 1C catalyst tested in Run 257. However, the Amocat 1C catalyst had poor integrity with excessive breakage. The EXP-AO-60 catalyst was developed to improve the attrition characteristics of bimodal catalysts. The main objective of this run was to test the new catalyst for its catalytic activity and strength while processing Illinois No. 6 coal at conditions for highest possible process performance.

The end points of distillate products of Illinois No. 6 coal from Run 257 were uncertain due to problems with the T102 vacuum distillation column operation. Also, the atmospheric flash which has about 5 wt % 750°F- material was recycled for process solvent makeup. Since Run 257, the vacuum distillation column problems

were corrected, and vacuum flash bottoms recycle was implemented. For better evaluation of coal liquefaction economics using Run 257 data, one of the objectives of this run was to obtain a better-defined end point for product distillates of Illinois No. 6 coal.

Run 259 was the first run in which a unimodal catalyst was tested over an extended period of time in the CC-ITSL process with a bituminous coal. Both low- and high-ash Pittsburgh No. 8 seam coals had been tested in Run 259, and the operational problems in using the unimodal catalyst were overcome by suitable process condition changes. The high ash coal was tested in just one period in Run 259, and the data indicated that the combined activity of coal and catalyst with the high ash coal was higher, and the operations were smoother. It was more likely that the feedstock reactivity changed, when switched to the high ash coal. Therefore, one of the objectives of this run was to determine the possible beneficial effects of the unimodal Criterion (Shell) 324 catalyst (aged in Run 259) in processing the high ash Illinois No. 6 coal.

2. CONCLUSIONS AND RECOMMENDATIONS

2.1 Conclusions

The conclusions for Run 261 are:

- The "all-distillate" product slate was achieved in 261B processing Illinois No. 6 coal with EXP-AO-60 catalyst. The experimentally observed C4+ distillate was 64.5 wt % MAF coal with 4.7 wt % resid and 17.9 wt % organic rejection. This was obtained at a high coal space velocity, 66.9 MF lb/hr/cuft-catalyst per stage, corresponding to 548 lb MF coal/hr. The projected C4+ distillate yield with resid extinction is 68.3 wt % with 18.0 wt % organic rejection at 61.4 MF lb/hr/cuft-catalyst per stage coal space velocity. The projected coal feed rate is 503 MF lb/hr.

- The distillate production was significantly improved in period 261B processing Illinois No. 6 coal with EXP-AO-60 catalyst which resulted in a 27-53% increase compared to periods 257DE, 257H, 259DE and 259H.

- The interpretation of Criterion catalyst results based on calculated rate constants and deactivation rates might be biased negatively because of uncertainty in catalyst charge in the second reactor (due to a low only 77% catalyst recovery at the end of the run).

- The "all-distillate" product slate was achieved in period 261D at steady-state operation with catalyst replacement processing Illinois No. 6 coal with EXP-AO-60 catalyst. The experimentally observed C4+ distillate was 65.6 wt % MAF coal with 3.7 wt % resid and 15.3 wt % organic rejection. This was obtained at a low catalyst replacement of 1.5 lb/ton MF coal per stage. The projected C4+ distillate yield with resid extinction is 68.9 wt % with 15.0 wt % organic rejection at 52.7 MF lb/hr/cuft-catalyst per stage coal space velocity, based on experimentally measured Phase 2 data. The projected coal feed rate for resid extinction is 431 MF lb/hr. (See Table E and Figure 71.) If the projection is based on the elementally-balanced adjusted Phase 3 yield data (□ symbol in Figure 71), the projected coal feed rate for resid extinction becomes 6% lower at 405 MF lb/hr.

- Operation in period 261D with EXP-AO-60 catalyst at a lower catalyst replacement and a higher resid recycle resulted in a reduction in coal throughput by 15% and distillate production by 13%, compared to period 261B. This indicates that a higher catalyst replacement (6 vs 3 lb/ton MF coal) significantly improves the coal throughput even at a lower

resid recycle.

- The "all-distillate" product slate was not achieved in periods 261E-G at steady-state operation with catalyst replacement processing Illinois No. 6 coal with Criterion 324 catalyst. The experimentally observed C4+ distillate yield was 58.4-64.3 wt % MAF coal with 5.1-10.3 wt % resid yield and 15.6-16.7 wt % organic rejection. The projected C4+ distillate yield with resid extinction is 66.1-68.9 wt % with 15.0 wt % organic rejection. The projected coal space velocities for resid extinction are 56.5, 41.6, and 39.7 MF lb/hr-cuft-catalyst per stage; and the projected coal feed rates are 460, 339, and 323 MF lb/hr, respectively, for periods 261E-G.

- Period 261E at a higher catalyst replacement rate (6 vs 4.5 lb/ton MF coal) and 25°F higher second stage reaction temperature improved distillate production by 20-21%, compared to periods 261F and G.

- Operation in periods 261E-G with Criterion 324 catalyst lowered the distillate production rate by 10-24%, compared to periods 261B and 261D with EXP-AO-60 catalyst. If the effect of recycle resid concentration is considered in the comparison, then approximately 35% higher distillate production is possible with EXP-AO-60 catalyst.

- Operation in periods 261E-G at higher first stage reaction temperatures (and at higher second stage temperature in period 261E) lowered the distillate selectivities to conversion to 78-81%, compared to 81-83% in periods 261B and 261D. This was primarily due to higher C_1-C_3 gas selectivities to distillate in periods 261E-G, 12-13%, compared to 8-9% in periods 261B and 261D. Hydrogen efficiencies were lower at 9.7-11.2 lb C_4+ distillate/lb H_2 consumed due to higher gas selectivities.

- Overall, good product quality characteristics were observed in periods 261A-D with EXP-AO-60 catalyst where properties were similar except for an increase in nitrogen for the oil in period 261D. Nitrogen in the product distillate increased by 44% to 3196 ppm in period 261D primarily due to higher resid recycle and lower replacement rate giving decreased catalytic conversion activity. Ranges of values for some distillate product properties with EXP-AO-60 catalyst in periods 261A-D were as follows:

    - Hydrogen content 11.27-11.78 wt %
    - Nitrogen 2,214-3,196 ppm
    - Sulfur 0.020-0.035 wt %
    - Oxygen 0.62-0.87 wt % by direct analysis
    - End Point 752-780°F.

- Ranges of values for product characteristics with Criterion catalyst in periods 261E and 261F were as follows:

    . Hydrogen content 10.65-10.86 wt %
    . Nitrogen 4,277-4,337 ppm
    . Sulfur 0.058-0.075 wt %
    . Oxygen, by difference, 1.65-2.38 wt %
    . End Point 786-925°F.

  Operation conditions such as reaction temperatures, space velocities and catalyst age as well as catalyst characteristics (bimodal vs unimodal) may have had an effect on the observed decrease in product quality with Criterion catalyst. Decreasing nitrogen removal at sufficiently high temperatures could have been due to thermodynamic limitations. However, both EXP-AO-60 and Criterion catalyst yielded product oils with the following properties:

    . Nitrogen increases in each boiling cut.
    . The total oil product is predominantly middle distillate.
    . Sulfur was generally highest in the naphtha fraction.

Major Observations in Run 261 were:

- High catalyst activities with EXP-AO-60 in both stages improved coal throughput and distillate production. Higher severity operating conditions employed might have improved conversion activities.

- Catalyst deactivation rate is relatively low, comparable to that in Run 257 processing Illinois coal with Amocat 1C catalyst and lower than that in Run 259 processing Pittsburgh coal with Shell 324 catalyst.

- If the effect of increased resid recycle from 40 to 50 wt % observed in periods 257D-E with Illinois coal and Amocat 1C catalyst is considered (a 27% increase in coal throughput and a 29% increase in distillate production), then just the effect of increased catalyst replacement rate becomes more significant, approximately 50% increase in coal throughput when periods 261B and 261D are compared on a 40 wt % resid recycle basis. The effect of increased resid recycle from 40 to 50 wt % was lower in periods 259D-F with Pittsburgh coal and Shell 324 catalyst (19% increase in coal throughput and 23% increase in distillate production). Note that Run 259 was conducted at higher thermal severity than Runs 257 and 261B-D, and thus the recycling of additional resid might have been less effective. Also, the resid in Run 259 was more likely to contain refractory components, since more of the initial coal-derived resid had already been converted to products (higher distillate yields, etc.). Furthermore, the

reactivity of the organic matrix as well as contributions of the mineral matter might have been different also for the two coals studied.

- In periods 261E-G with Criterion 324 catalyst, calculated rate constant values for both stages were very similar, even though operating reaction temperatures were different - 810°F in the first stage and 800-825°F in the second stage. It seemed that a decrease of 25°F in the second stage had no effect on the reaction rate constant, which appeared to be due to changes in the reactivity of the feedstock as it proceeded through the two reaction stages.

- In Run 261 processing Illinois coal, operation with Criterion 324 catalyst at 810/825°F in period 261E gave similar calculated rate constant values in both stages when compared to those with EXP-AO-60 catalyst operated at 790/810°F, at the same volume-basis catalyst age; however, when compared at the same weight-basis catalyst age, period 261E with Criterion 324 had significantly lower rate constant values than those in period 261B with EXP-AO-60 at 790/810°F. Operation with Criterion 324 at 810/800°F in period 261FG gave lower rate constant values than those in 261D with EXP-AO-60 at 790/810°F, primarily due to higher deactivation rates experienced with Criterion 324. The interpretation of Criterion catalyst results based on calculated rate constants and deactivation rates might be biased negatively because of a decrease in catalyst charge in the second reactor during operations (i.e., only 77% catalyst recovery at the end of the run).

- Operation with Criterion 324 in period 261E processing high-ash Illinois coal at 810°F resulted in a first stage calculated rate constant value similar to that at 825°F with Shell 324 in periods 259H processing high-ash Pittsburgh coal, when compared at the same catalyst age (ln K = 3.8 vs. 3.7 hr^{-1}); gave higher second stage values at 825°F than that at 790°F with Pittsburgh coal (ln K = 3.9 vs. 3.4 hr^{-1}). Deactivation rates appeared to be similar for both Criterion and Shell catalysts, although they were used at different reaction temperatures and with different coals. It seemed that the Illinois coal was more reactive than the Pittsburgh coal. This observation would confirm the preliminary assessment based on results from Runs 259A and 257J using Amocat 1C catalyst. The interpretation of Criterion catalyst results in period 261E based on calculated rate constants and deactivation rates might be biased negatively because of uncertainty in catalyst charge in the second reactor (i.e., only 77% catalyst recovery at the end of the run).

- At a coal feed rate of 494 lb/hr and a Criterion 324

261E, the C_4+ distillate yield was about 61 wt % MAF coal with 5.4 wt % resid yield. However, the average hydrogen partial pressure was about 100 psi lower than the periods with EXP-AO-60 catalyst in each stage. The lower hydrogen partial pressure might have been a contributing factor.

- Lowering the Criterion catalyst replacement rate from 3 to 2.25 lb/ton (each stage) in period 261F necessitated a lowering of the coal feed rate to 360 lb/hr and the coal concentration to 30 wt %. Because of the reduced exotherms at lower coal feed rates, the second stage temperature could be maintained only at 800°F instead of the target 825°F. The distillate yield was low at 58 wt % MAF coal with 10 wt % resid yield at lower space velocity due to both catalyst deactivation and lower second stage temperature.

- Compared to Run 257, the resid hydrogen content in both the process solvent and the interstage stream for Run 261 with EXP-AO-60 catalyst was lower. The hydrogen content of both the process solvent and the interstage stream in Run 261 decreased significantly with Criterion 324 catalyst operation compared to operation with EXP-AO-60 catalyst. Process conditions (such as reaction temperature, hydrogen partial pressure and space velocity) and the inherent nature of the catalyst might have had an effect on hydrogen content. The trend in hydrogen content in these runs seemed to follow the expected with thermal severity, related to thermodynamic equilibrium and hydrogenation. Carbon deposition on the catalyst was also related to the thermal severity affecting hydrogenation. The lower second stage hydrogen partial pressure in Run 257 due to no interstage separation and again in Run 261 the lower partial pressure with Criterion catalyst compared to that with EXP-AO-60 catalyst might have also affected hydrogenation.

- The solvent quality of the process solvent in Run 261 processing high-ash Illinois coal was much greater with operation using EXP-AO-60 catalyst than with operation using Criterion catalyst at the conditions tested (81-86% vs. 76-77%). The solvent quality in Run 259H processing high-ash Pittsburgh coal was similar using Shell 324 catalyst compared to Run 261 with Illinois coal and EXP-AO-60 catalyst (85% vs. 81-86%).

- Unlike bimodal catalysts previously tested at Wilsonville, the attrition characteristics of the new EXP-AO-60 catalyst were much better and comparable to unimodal catalysts. The EXP-AO-60 catalyst recovered from the reactors at the end-of-test had only 4 to 5 wt % catalyst passing a 14 mesh screen. In comparison, Runs 257 and 259A had 14 to 16 wt % Amocat 1C catalyst passing a 14 mesh screen.

- The carbon deposition on the THF extracted EXP-AO-60 catalyst used in this run was much greater than the carbon deposition on the Amocat 1C 1/12" catalyst used in Run 257. The wt % carbon on the EXP-AO-60 catalyst in the first stage was 12 to 13 wt %, and in the second stage, the deposition was 14 to 15 wt %. In Run 257, the carbon deposits ranged from 10-12 wt % in both the first and second stages. The higher reaction temperatures in Run 261 might have had an effect on the higher carbon depositions on EXP-AO-60 catalyst. The carbon laydown on the Criterion catalyst in this run was 16-20 wt % in both stages, even higher when operated at higher temperatures.

- The preasphaltenes in the ROSE-SR^{SM} feed for both Run 261 with EXP-AO-60 catalyst and Run 257 with Amocat catalyst were in the 4 to 7 wt % range. The preasphaltenes increased significantly to about 14 wt % once operation with Criterion 324 catalyst was started. However, process condition changes helped reduce the preasphaltenes to about 8 wt %.

- The distillation cutpoint by equal wt % overlap method was 780 to 825°F with 6 to 9 wt % overlap in this run with EXP-AO-60 catalyst. In comparison, for Run 257 with Amocat 1C catalyst, the cutpoint was 795 to 860°F with 12 to 15 wt % overlap. The end points of distillate products for Run 257 had uncertainty due to the problem with the fractionation unit without finely separating distillate products from the recylce gas oil.

- The decrease in resid recycle by 10 wt % with a corresponding increase in heavy gas oil recycle resulted in a decrease in end boiling point of the distillate product from 780 to 753°F (period 261D vs 261C). This decrease was less than that observed in Run 259 (54°F decrease in end boiling point for a similar decrease in resid recycle) due to lower catalyst ages in period 261C compared to period 261D.

- During the continuous resid injection test, the centrifugal pumps were not operable at the desirable coal + heavy gas oil (V1074) compositions to give 30-33 wt % coal concentration in the slurry to reactor feed. This first necessitated increasing the distillate portion of the slurry, then later required the addition of deashed resid directly to the process solvent tank. Best operable conditions were obtained with 45 wt % coal in V1074+deashed resid, giving a 30 wt % coal concentration in slurry to reactor feed. The transitional period obtained with resid injection indicated reasonable yields, which in fact were better than those for the previous period 261F without using the continuous resid injection.

2.2 Recommendations

The recommendations for future runs are:

- Continue testing with the new bimodal catalyst, EXP-AO-60, to increase the distillate production at resid recycle levels higher than 50 wt % in the process solvent by varying the slurry composition.

- Determine the effect of thermal/catalytic volume ratio on mixing characteristics and process performance and if desired, test at high coal space velocities with half-volume reactors and recycle resid levels higher than 50 wt % to improve distillate yields and production rates.

- Using EXP-AO-60 catalyst, test deeply cleaned bituminous coals with <5 wt % ash content, i.e., low-ash reactive Illinois coals, to improve the distillate production, selectivity, and hydrogen efficiency.

- Further test the low/high thermal severity configuration in catalytic/catalytic mode with higher space velocity at higher thermal severity, i.e., half-volume reactor operation at 805/825°F reaction temperatures, to improve the thermal efficiency, catalyst utilization, distillate selectivity, and hydrogen efficiency.

- Determine optimum catalyst requirement with cascading in the low/high mode, i.e., from the first stage to second stage with 10-50% replacement of the second stage fresh catalyst addition. Cascading would most benefit in reducing catalyst requirement, if the first stage compared to the second stage operates at a lower temperature with a higher catalyst replacement, resulting in lower carbon deposition and higher catalyst activity at a younger catalyst age, if used in the second stage. However, it is possible that depending on the actual combination of thermal severity and space velocity, the staging effect on carbon and metal depositions might be greater than the thermal severity effect.

- Test upgrading the distillate product for better quality. Also, test integrated upgrading schemes for an overall improvement in process performance and coal liquefaction economics.

- Test distillate product for use as turbine fuel.

- Test continuous operation of catalyst replacement to improve operation stability. Evaluate the economics of beneficial effects of high catalyst replacement rates. Test high catalyst replacement rates with rejuvenated catalysts.

- Test alternate catalysts with high activity and selectivity.
- Develop alternate deashing processes
- Evaluate the performance of pretreated/upgraded coals.
- Further test the continuous resid injection to evaluate the performance improvement, i.e., yields, and to improve the injection system operability with better slurry composition. Methods to attain coal concentrations above 45 wt % in V1074 in the slurry tanks with the resid injection system should be explored. Methods to achieve better incipient wetting of coal and alternatives to centrifugal pumps for slurry recirculation should be explored.
- Based on Run 261, the distillate yield can be improved by lowering organic rejection below 18 wt % MAF coal, which could be achieved by increasing coal conversion above 92 wt %. Higher solids (CI) recycle would increase coal conversion. The coal throughput can be increased, if operated at higher coal concentrations in the slurry. Such a mode should be tested if economics are attractive.
- Expand the product quality characterization testing to include metals, aromatics, paraffins and toxicological effects.

3. OPERATING DATA AND PROCESS PERFORMANCE

The main objectives of this run included testing the EXP-AO-60 and Criterion 324 catalysts (both 1/16" cylindrical extrudates) using Illinois No. 6 coal from Burning Star #2 mine. In addition, the new continuous resid circulation system was also tested near the end of the run. Full-volume reactors with interstage separation were used in this run.

3.1 TSL System Stability

TSL system stability is judged by evaluating the material balance closure errors, plant operation stability, and plant performance stability. Criteria for selection of stable days include:

- Mass balance closure errors for CCR unit (first and second stages), and ROSE-SR^{SM} must be less than 10 wt % MAF coal.
- The sum of absolute values of inventory changes including drumouts, must be less than 15 wt % MAF coal for the following locations.
    a) Between the second stage and ROSE-SR^{SM} unit,
    b) Between the ROSE-SR^{SM} unit and the first stage.

A description of the elemental balancing procedure and a more detailed description of the selection criteria are given in Appendix B (Material Balance Methodology).

There were several mechanical problems resulting in eight outages. Most of the outages summarized below occurred during the first two months.

January 13	Steam leak in main stream line, 13.6 hours.
January 21, 22	Suction bolts loose on slurry pumps, 18.3 hours.
February 3-7	Block valves on high pressure letdown valves leaking through, 109 hours.
February 7-8	High slurry viscosities, 10.3 hours.
February 16	Leak on V1082 vacuum line, 11.2 hours.
February 17-19	Leak on line from V1079 to V1067, 79 hours.
Feb 26-Mar 2	P1236 ebullation pump failed, 92.5 hours.
May 12-14	Leak at bottom of V1250, 63.4 hours.

In addition to these outages, there was a scheduled outage between March 19 and April 12 to change the catalyst in the reactors and presulfide 1690 lbs of Criterion 324 catalyst.

The three process leaks occurred in lines that were worn or corroded to where the pipe was thin and all were in elbows. These lines handled high solids and resid streams. An analysis of the ash from the build up on the P1236 pump shaft showed about equal amounts of zinc oxide and phosphorous pentoxide - components of oil additives.

Operating conditions during Run 261 with both EXP-AO-60 and Criterion catalysts are given in Table B. Process performance and yield data are summarized in Table C. Two run periods were declared transitional (periods 261A and 261G). Early operation up to January 28 was unstable due to over conversion of the subbituminous resid (start up material) and due to ash carryover in the ROSE-SR^{SM} unit. Changing the first stage temperature to 790°F also delayed reaching satisfactory operating conditions during the first month of operation. During the Run 261A period, there was still some drift in the amount of resid in the process solvent and the resid content was low. Period 261G, during the resid injection test, is also considered transitional due to difficulty in reaching the target coal and process solvent compositions. The period was also during a test to determine the limitations of the resid injection equipment. Period 261F was considered unstable due to material balance errors. Two suspect meters were recalibrated, but no change in the calibration factors were found.

Periods 261A and 261B were conducted with V1067 recycle to V131B rather than V1082 recycle. The feed rate to V1082 was thought to be too high to obtain low distillate content in the bottoms. Over conversion of subbituminous resid and high solvent content at the start of the run caused most of the problem. Periods 261C through 261G were conducted with V1082 recycle.

Periods 261E, 261F and 261G used Criterion 324 catalyst while periods 261A through 261D used EXP-AO-60 catalyst. When the Criterion catalyst test was started on April 12 at more severe operating conditions, the resid inventories were reduced requiring a higher feed rate. The feed rate in combination with higher preasphaltenes led to higher viscosities (Figures 6 and 7) and high pressure drop in the slurry feed line and in the preheater. When the slurry pump discharge pressure reached near relief settings, the reactor pressures were reduced during April 19-30 which includes period 261E. On April 30, the coal concentration was reduced from 33 to 30 wt % to reduce the coal slurry viscosity. The coal feed rate was lowered and the second stage temperature (interstage heater temperature was too high) was reduced to 800°F. This combination of changes resulted in a reasonable viscosity for period 261F.

Table B. Summary of Operating Conditions

Operating Period	261A(transitional)	261B	261C	261D
Date, 1991	Jan 29-Feb 2	Feb 11-15	March 7-11	March 14-18
Operational Days	16-20	24-28	40-44	47-51
Coal				
Feed rate, MF lb/hr	543.0	547.9	420.0	423.0
Ash, wt % MF	12.2	12.1	11.2	11.8
Conc. in slurry, wt % MF	33.0	32.9	32.8	31.9
Process solvent, wt %				
Resid	32.7	38.3	38.3	48.4
CI	12.0	12.1	11.8	11.9
1st stage				
Reactor temp.,°F (average)	790	789	790	791
Inlet H_2 part. press., psia	2611±20	2610±23	2643±14	2656±22
Total gas flow, scfh	5390	5760	5080	5285
Recycle gas, scfh	800	1040	710	880
Space velocity,				
lb feed/hr-lb cat	6.3	6.4	5.0	5.0
lb MF coal/hr-ft^3 cat	66.4	66.9	51.3	51.7
Catalyst type	EXP-A0-60	EXP-A0-60	EXP-A0-60	EXP-A0-60
Catalyst age, lb(R+CI)/lb cat	1328±74	1324±55	2537±120	2812±45
lb MF coal/lb cat	680±35	671±28	1256±53	1375±20
Catalyst replacement rate, lb cat/ton MF coal	3.0(a)	3.0	0(b)	1.5
2nd stage				
Reactor temp.,°F (average)	810	808	809	809
Inlet H_2 part. press., psia	2479±13	2515±27	2478±16	2472±7
Total gas flow, scfh	4860	4650	4400	4140
Recycle gas, scfh	1610	1640	2210	1930
Space velocity,				
lb feed/hr-lb cat	5.9	6.0	4.6	4.6
lb MF coal/hr-ft^3 cat	66.4	66.9	51.3	51.7
Catalyst type	EXP-A0-60	EXP-A0-60	EXP-A0-60	EXP-A0-60
Catalyst age, lb(R+CI)/lb cat	1128±117	1090±46	2067±97	2238±35
lb MF coal/lb cat	723±78	689±30	1269±53	1360±20
Catalyst replacement rate, lb cat/ton MF coal	3.0(a)	3.0	0	1.5
ROSE-SR℠ Unit				
DAS type	2414	2394	2514	2544

(a) Started on February 1 in First Stage and February 2 in Second Stage.
(b) Catalyst batch deactivation.

Table B. Summary of Operating Conditions (Continued)

Operating Period	261E	261F(unstable)	261G(transitional)
Date, 1991	April 20,23,24,25,26	May 17-21	May 24-27
Operational Days	59,62-65	84-88	91-94 (Resid Injection)
Coal			
Feed rate, MF lb/hr	494.3	359.1	357.0
Ash, wt % MF	11.6	11.8	11.5
Conc. in slurry, wt % MF	33.0	29.7	28.8
Process solvent, wt %			
Resid	48.9	48.9	47.4
CI	12.0	11.8	11.4
1st stage			
Reactor temp.,°F (average)	809	809	809
Inlet H_2 part. press., psia	2538±64	2624±8	2636±10
Total gas flow, scfh	5370	5518	5688
Recycle gas, scfh	830	1400	1353
Space velocity,			
lb feed/hr-lb cat	3.5	2.9	3.0
lb MF coal/hr-ft^3 cat	60.7	44.1	43.8
Catalyst type	Criterion 324	Criterion 324	Criterion 324
Catalyst age, lb(R+CI)/lb cat	1345±31	1936±25	1966±24
lb MF coal/lb cat	660±14	885±10	884±10
Catalyst replacement rate, lb cat/ton MF coal	3.0	2.25	2.25
2nd stage			
Reactor temp.,°F (average)	824	800	800
Inlet H_2 part. press., psia	2354±58	2444±15	2308±14
Total gas flow, scfh	5020	4455	4759
Recycle gas, scfh	1975	2476	2775
Space velocity,			
lb feed/hr-lb cat	3.3	2.6	2.7
lb MF coal/hr-ft^3 cat	60.7	44.1	43.8
Catalyst type	Criterion 324	Criterion 324	Criterion 324
Catalyst age, lb(R+CI)/lb cat	1450±38	1800±22	1790±23
lb MF coal/lb cat	669±13	861±9	860±10
Catalyst replacement rate, lb cat/ton MF coal	3.0	2.25	2.25
ROSE-SR℠ Unit			
DAS type	2654	2684	2654

Table C. Overall TSL Yields with Illinois No. 6 Coal

Operating Period Date, 1991 Operational Days	261A (transitional) Jan 29-Feb 2 16-20	261B Feb 11-15 24-28	261C March 7-11 40-44	261D March 14-18 47-51
Yield, wt % MAF coal				
H_2	-5.4±0.4	-5.7±0.2	-6.1±0.3	-6.0±0.2
Water	7.7±0.7	8.3±1.5	9.1±0.5	9.7±0.5
H_2S	3.5±0.5	3.6±0.5	3.4±0.1	3.3±0.1
CO, CO_2	0.4±0.1	0.3±0.1	0.6±0.4	0.4±0.1
NH_3	1.3±0.1	1.4±0.1	1.3±0.1	1.5±0.4
C_1-C_3 gas	5.4±0.5	5.1±0.6	6.0±0.4	6.5±0.7
C_4+ distillate	59.0±0.5	64.4±2.6	64.8±3.8	65.6±1.6
C_4-C_6	2.7±0.2	2.4±0.5	2.6±0.3	2.4±0.2
IBP-350°F	13.4±0.2	13.2±0.5	14.9±0.6	18.9±5.5
350-450°F	7.4±0.4	7.4±0.5	8.2±0.1	9.2±1.2
450-EP°F	35.5±2.1	41.4±2.7	39.1±3.4	35.1±6.9
Resid (a)	7.4±2.8	4.7±3.3	5.1±1.6	3.7±2.6
Bottoms Product (ash-free)	20.9±1.0	17.9±1.4	15.8±1.6	15.3±0.7
H_2 efficiency lb C_4+ dist/lb H_2 cons	10.9±0.7	11.3±0.4	10.6±0.3	10.9±0.3
C_1-C_3 selectivity to C_4+ distillate	9.2±0.8	7.9±0.7	9.2±0.9	9.8±0.9
Coal Conversion, wt % MAF				
1st stage	84.5±1.7	87.0±1.0	89.7±0.5	90.5±0.8
1st and 2nd stage	91.1±0.9	92.4±0.2	93.8±0.2	93.9±0.3
Overall TSL	91.1±0.5	91.7±0.7	93.6±0.4	93.0±0.2
Resid+UC conversion, wt % feed				
1st stage	24.3±1.0	21.9±1.0	23.7±1.3	22.9±0.8
2nd stage	17.2±2.6	20.7±1.3	20.0±0.9	17.3±1.2
Resid+UC conversion, wt % MAF coal				
1st stage	45.8±2.8	43.8±2.1	46.8±3.2	50.4±1.7
2nd stage	25.8±2.1	33.7±2.0	32.3±1.5	30.8±2.4
Overall	71.7±2.3	77.5±2.2	79.1±3.2	81.2±2.6

(a) Includes UC accumulation

Table C. Overall TSL Yields with Illinois No. 6 Coal (Continued)

Operating Period	261E	261F(unstable)	261G(transitional)
Date, 1991	April 20,23,24,25,26	May 17-21	May 24-27
Operational Days	59,62-65	84-88	91-94
Yield, wt % MAF coal			
H_2	-5.4±0.2	-6.0±0.2	-6.2±0.1
Water	9.7±0.5	9.8±1.1	9.0±1.1
H_2S	3.4±0.0	2.7±0.0	2.8±0.1
CO, CO_2	0.6±0.1	0.5±0.1	0.5±0.0
NH_3	1.3±0.2	1.0±0.1	1.3±0.2
C_1-C_3 gas	7.7±0.5	7.7±0.2	7.6±0.3
C_4+ distillate	60.6±2.8	58.4±2.0	64.3±2.6
C_4-C_6	2.9±0.3	3.3±0.1	3.2±0.1
IBP-350°F	14.0±1.8	13.1±0.7	14.3±0.3
350-450°F	8.9±1.2	8.1±0.3	8.2±0.2
450-EP	34.8±3.0	33.8±2.0	38.5±2.0
Resid (a)	5.4±3.6	10.3±2.9	5.1±2.2
Bottoms Product (ash-free)	16.7±1.5	15.6±0.8	15.6±0.7
H_2 efficiency, (%)			
lb C_4+ dist/lb H_2 cons	11.2±0.4	9.7±0.4	10.3±0.2
C_1-C_3 selectivity, (%)			
to C_4+ distillate	12.7±0.6	13.2±0.5	11.8±0.4
Coal Conversion, wt % MAF			
1st stage	92.4±0.6	92.2±0.7	92.0±0.7
1st and 2nd stage	93.9±0.3	93.7±0.1	93.7±0.5
Overall TSL	92.7±0.6	92.5±0.4	92.2±0.2
Resid+UC conversion, wt % feed			
1st stage	19.4±1.6	17.6±2.7	19.4±1.1
2nd stage	20.0±1.8	16.4±1.8	15.8±0.9
Resid+UC conversion, wt % MAF coal			
1st stage	42.5±3.9	41.2±6.8	47.2±2.2
2nd stage	35.4±3.2	32.9±4.5	32.1±1.7
Overall	77.9±3.4	74.1±2.6	79.2±2.9

(a) Includes UC accumulation.

The operational problems and steps taken to rectify the problem, and the process condition changes made to achieve the objectives of Run 261 are summarized below:

DIFFICULTY OR CHANGE	CONSEQUENCE
Sulfided 2100 pounds of EXP-AO-60 catalyst, Jan 1-9.	Some fines present in batches.
Water line to caustic scrubber developed crack, Jan 9.	Down to repair line.
Timer went bad on water softener, Jan 10.	Water to boiler not soft.
Start of Run 261, Jan 12.	
Low steam pressure due to underground leak in steam line, Jan 13.	Outage of 13.6 hours.
High solvent content in ROSE-SR^{SM} feed, thought to be due to high feed rate, Jan 15.	ROSE-SR^{SM} unit not deashing. Coal rate lowered from 450 to 400 lb/hr.
Withdrawal tube flush rate to second stage erratic, Jan 15.	Intermittent WTF flow rate affecting DT.
Change valves and rings on second stage hydrogen compressor, Jan 17.	Low hydrogen flow to second stage.
Boiler shutdown due to low water level, Jan 18.	Loss of vacuum.
Resid inventory low, Jan 18.	Increased coal rate to 425 lb/hr.
Resid inventory low, Jan 21.	Increased coal rate to 450 lb/hr.
Boiler pressure low, Jan 21.	Lost vacuum 1250-1320 hrs.
Unable to recirculate V101A slurry tank. Foaming in line, Jan 21.	Outage of 18.3 hours. Recirculation pump suction pipe bolts loose.
Repaired several problems.	On feed at 1600 hrs, Jan 22.
Solvent content of ROSE-SR^{SM} feed still high, Jan 22.	Changed to ash recycle from V106?.

DIFFICULTY OR CHANGE	CONSEQUENCE
Increased first stage temperature to 790°F and brought catalyst charge to 270 pounds, Jan 23.	
Low steam pressure, Jan 24.	Loss of vacuum; blocked in unnecessary users.
Interstage separator valve leaking through, Jan 24.	Caused gas blow through to second stage and loss of ebullating pump for 10 minutes. Coal rate lowered temporarily.
Low resid inventory, Jan 24.	Coal feed rate to 500 lb/hr.
Power outage and interstage separator valve leaking through, Jan 26.	System upset.
Low resid in process solvent, Jan 27.	Adding extra solvent to process solvent.
Interstage separator valve not controlling, Jan 27.	Valve repaired again.
Low resid inventory, Jan 28.	Coal feed rate to 550 lb/hr. Process solvent adjusted to 30 wt % resid on Jan. 28 and then to 35 wt % Jan. 29.
Water injection rate too high to second stage, Jan 29.	Large amounts of water present in distillate.
One high pressure letdown valve not controlling and other valve not enough capacity, Jan 30.	Coal rate lowered temporarily.
Condensate from interstage separator overheads routed to wrong tank, till Feb 1.	Low indicated rate for separator overheads.
Block valves downstream of high pressure letdown valves not holding when attempting to change valve trim, Feb 1.	Decide to shutdown to repair block valves.
Catalyst addition started on Feb 1 to first stage and on Feb 2 to second stage at 3 lbs/ton MF.	

DIFFICULTY OR CHANGE	CONSEQUENCE
Repair of block valves for high pressure letdown valves and of catalyst addition valves on second stage, Feb 3-7.	Outage of 108 hours.
During outage, temperature of V101A slurry tank with process solvent increased to 440°F. Viscosity of V101B tank high, Feb 4-6.	Possible oxidation due to method of operating new vent system. Slurries were diluted.
On coal feed, 1200 hrs on Feb 7.	
Unable to circulate V101A tank.	Off coal feed: 22:45 hrs on Feb 7 - 10:00 hrs on Feb 8.
Change to 40 wt % resid in process solvent, Feb 8.	
Catalyst addition valves on first stage not opening all the way, Feb 12.	Catalyst addition problems. Valves repaired.
Both water softeners accidently tripped off line, Feb 14.	Loss of vacuum for 3 hours.
Dowtherm heater running a lower temperature during cooler weather, Feb 14.	Temperature in second stage dropped.
Both water softeners tripped off line - reason unknown, Feb 15.	Loss of vacuum for 2 hours.
Bottoms pump on T102 stopped. Shutdown on high stack temperature, Feb 15.	B102 preheater tubes may be coked.
Both water softeners tripped off line, Feb 16.	Loss of vacuum. Automatic regeneration disabled.
Unable to maintain vacuum in V1082 vacuum flash vessel, Feb 16.	Off coal feed at 1110 - 2230 hrs to repair hole in elbow.
Pin hole leaks in line from V1079 to V1067, Feb 17.	Off coal feed at 0230. Line thinned to 1/16" from 0.154.

DIFFICULTY OR CHANGE	CONSEQUENCE
P1222 ebullating pump inoperable, Feb 17.	Repair.
On coal feed at 0930, Feb 20.	
Steam trap not functioning, Feb 20-22. Repaired.	Much water present in second stage flashed overheads to distillation columns.
B1200 shutdown three times, Feb 22-24.	Small temperature reductions. Cause unknown at this time.
Computer not running programs correctly, Feb 25.	Programs were rerun after rebooting computer.
P1236 ebullating pump failed, Feb 26. Off coal feed at 09:30.	Maximum temperature of 828°F reached in R1236 catalyst bed.
Poor circulation in B102 since Feb 15.	B102 was decoked, Feb 27.
On coal feed at 06:10, Mar 2 at 425 lb MF/hour with V1082 Recycle.	
Overhead condenser on T105 developed leak.	T105 down March 4 to bypass condenser.
Diaphragm broke on water pump to vent separator overhead line, March 5.	Pump off line to repair.
Ball valve at top of coal slurry tank restricted, March 8.	Coal addition slow.
DP developing across seal in ebullating pumps, almost daily.	Flow rate of seal oil raised for short period of time each day to drop DP.
Impulse line on R1235 DP plugged, March 11.	R1235 DP readings not valid.
To 50 wt % resid in the process solvent, March 12.	
Off coal feed at 02:35, March 19.	Scheduled Shutdown.
Sulfided 1690 pounds of Criterion 324 catalyst, April 6-10.	

DIFFICULTY OR CHANGE	CONSEQUENCE
Added Run 259 catalyst to reactors, April 11.	
Started coal feed at 19:55, Apr 12	
Water present in ROSE-SR^{SM} DAS, April 13-15.	Causing variation in process solvent composition.
Fill valves on ROSE-SR^{SM} feed measurement not operating,	Unmelted solids in valve. Repaired. April 16.
Several power blips, April 17.	
DP developing across seal on ebullating pumps, daily.	Flow rate of seal oil increased for short period of time each day to lower DP.
Increased coal feed rate to 500 lb MF/hour, April 18.	
Preheater, B1200, DP very high. Slurry feed pump discharge pressure high. April 19 - 30.	Lowering reactor pressures due to high DP in feed in feed line and heater.
Decreased coal feed rate to 425 lb MF/hour, April 27.	
Decreased coal feed rate to 360 lb MF/hour, April 29.	
Coal concentration decreased from 33% to 30%, April 30.	
High temperature at second stage outlet.	Reduced second stage preheater temperature to 800°F.
At lower feed rates and coal concentration, pressure drop in lines are less, April 30.	Increasing reactor pressures back to normal levels.
Hydrogen compressor for second down for valve change, May 1.	Low purity gas from first stage stage used for 1 hour.
Leak on K.O. pot of second stage recycle compressor, May 1.	Compressor was overpressured when valve was closed. Upset.
Computer off line, May 2.	Loss of data for 1.2 hours.

DIFFICULTY OR CHANGE	CONSEQUENCE
During heavy rain, electronic controller on interstage separator quit, May 5.	Pressure upset.
R1235 catalyst tube plugged, May 7-10.	Could not withdraw catalyst.
Line from ROSE resid tank to V131B plugged. Found heat media to be throttled too much, May 7-9.	Process solvent makeup was very slow and difficult to calculate.
Leak in line at bottom of V1250, May 12.	Unit on solvent to repair leak. Carbon steel pipe at elbow was thin.
Plug formed in B1200 or R1235 on startup, May 14.	Cleared R1235 feed line.
On coal feed at 2250 on May, 14.	
Changed valves on second stage hydrogen compressor, May 16.	Using first stage gas. Upset conditions.
Computer card for resid injection project was bad, May 16.	Computer was taken off line 2 hours to repair.
Interstage overhead water separator plugged, May 21.	V1258 overhead flow uncertain.
Withdrawal tube flush pump quit on R1236, May 22.	Change in reactor overall DT.
Material balances off about 3%.	V1072 and V1258 Overheads meters tested okay.
Start resid injection project test, May 23.	
Difficulty pumping 53% coal, 47% V1074 slurry, May 23.	Backflushed V101B to V101A to get centrifugal pumps to pump.
Could not pump 57% coal, 43% V1074 slurry, May 24.	Diluted slurry. Reasonable results at 45% coal in slurry.
End of Run 261, May 31.	

3.2 Feed Coal

Illinois No. 6 coal from Consolidation Coal Company's Burning Star No. 2 mine was the feed coal in Run 261. This coal has been used as the feed coal at Wilsonville in several experimental runs over the past five years. It was last used in Run 257 with Amocat 1C catalyst. It is desirable to have the reactivity of coal remain the same throughout the run. Thus, independent of process performance, the analytical laboratory conducts a variety of tests and the information is used to determine if the coal reactivity remains the same.

Handling of the feed coal before liquefaction is as described below. It is washed at the mine and then trucked to the grinding facility at Empire Coke in Holt, Alabama. Usually, three to four weeks before an experiment starts, the three to four hundred ton lot shipment arrives at Empire. When the plant is nearly ready for coal feed, the grinder is notified and a small portion of the lot is ground such that approximately 95 wt % of it will pass through a 200 mesh (75 microns) screen. Ground coal is stored in a large metal bin blanketed with nitrogen and then shipped to the pilot plant. Oxidation effects on coal reactivity are a major concern as some of the coal can weather for several weeks before an experimental run is finished.

The data in Table 1 is a summary of coal analysis conducted throughout Run 261. It is typical analysis, but is not necessarily the data used for material balance calculations. In general, the various analyses indicate that coal properties remained about the same for the duration of the experimental run. No single element shows a significant deviation from the analysis gathered on the original pile sampled fresh from the mine. Substantial changes could indicate that the coal became contaminated with another source such as coke or another coal of different rank. The mineral analysis is similar to that observed in the Illinois feed coal from Run 257. Silica, iron, and aluminum are the primary constituents of the ash. The pyritic sulfur content is not unusual. As past experience has shown, approximately 33 wt % of the total sulfur is associated with pyritic iron. The sulfate sulfur content can be used as an indication of coal oxidation. A sulfate sulfur content less than 0.10 wt % is common for freshly mined coal. A value greater than 0.30 wt % could indicate oxidation. Through period 261E, there was no appreciable increase in sulfate sulfur.

Feed coal samples are randomly selected for reactivity tests throughout the run. The test measures the difficulty of breaking the coal down into tetrahydrofuran (THF) soluble components under standard reaction conditions using a standard solvent. The test data show about a ten percent decrease in conversion values toward the end of the run. The lower values are consistent with coal that underwent the most weathering and thus indicate that

perhaps slight changes in coal reactivity did occur towards the end of the run.

After the coal arrives at the pilot plant, its storage time is for only a few days before it is slurried with the solvent. The hot slurry is pumped into the pre-heater and then into the first stage reactor.

3.2.1 Slurry Preparation

The process solvent used for coal slurry blend was a mixture of V1074 heavy gas oil (T102 vacuum column bottoms), full range ROSE-SR^{SM} resid, and the V1067 atmospheric flashed bottoms for periods 261A and 261B. For periods 261C through 261F, the V1082 vacuum flashed bottoms was used for solids recycle. For period 261G, V1074 solvent was used to prepare coal slurry in combination with the resid injection scheme.

Recycle process solvent analytical data is in Table 2. In Run 261, the target process solvent composition was 40 wt % resid, 48 wt % distillate and 12 wt % CI for periods 261A through 261C. The target process solvent composition was 50 wt % resid, 38 wt % distillate and 12 wt % CI for periods 261D through 261F. Coal concentration was 33 wt % up to April 30 (periods 261A-E). Period 261F was at 30 wt % coal. In period 261G, coal concentrations varied between 26 to 35 wt % during the resid injection test period.

3.3 Close-Coupled Reactor (CCR) Unit

The Closed-coupled reactor (CCR) unit had the following objectives for Run 261:

- Attain the highest conversions and distillate yields possible with high throughput when processing Illinois No.6 coal in the CC-ITSL configuration,
- Maintain steady-state operation with catalyst replacement in both reactors and watch for any signs of catalyst breakage when testing the new, bimodal EXP-AO-60 catalyst,
- Evaluate process performance and operational stability while processing the Illinois No. 6 coal with Criterion 324, 1/16" catalyst,
- Maintain steady-state operation with catalyst replacement in both reactors with Criterion 324 catalyst, and
- Test the continuous resid injection system and determine the process limitations.

In the first part of Run 261, Illinois No. 6 coal was processed with the new EXP-AO-60 catalyst. The fresh catalyst was batch deactivated to an age equivalent to a catalyst replacement rate of 3 lb cat/ton coal. The aged catalyst was further batch deactivated to 1.5 lb cat/ton coal replacement rate. Both batch and steady state (catalyst activity) data were obtained. In the second part of the run, Criterion 324, a unimodal catalyst, was tested with the Illinois coal to evaluate the process performance and operational stability. Aged catalyst from Run 259 was used, and steady state data at 3 and 2.25 lb cat/ton replacement rates and batch deactivation data were taken. Near the end of Run 261, a new resid injection system was tested. With this system, coal was slurried in V1074 heavy gas oil, and the deashed resid and the stream containing the solids were directly injected into the feed line to the preheater.

Feed slurry composition, coal feed rate, and reactor temperatures were changed to maintain smooth operation and obtain high distillate yields at high coal throughputs. Coal conversion, hydrogen consumption, and resid and distillate yields were monitored daily as performance indicators. Catalyst samples were routinely analyzed to evaluate the catalyst for activity and integrity.

3.3.1 CCR Unit Operations and Process Performance

Before the start of Run 261, the plant was off feed for routine maintenance and for implementing a new vent system for the coal slurry mix tanks. The interstage separator, V1258, was included in the process. Full volume reactors were used for Run 261 with R1235 and R1236 being the first and second stage reactors, respectively.

On January 1, sulfiding of fresh EXP-AO-60 catalyst was started. Four batches of two full volume reactors were sulfided to give 2220 pounds for use in the catalyst replacement program during the run. Part of the last batch was left in the reactors for startup. The initial catalyst charge for startup was 241 lbs (dry) in R1235 and 242 lbs dry in R1236. The plant went on coal feed on January 12. On January 14, the catalyst charge in R1236 was increased to the run conditions (270 lbs). The R1235 catalyst charge was not increased at this time because of overconversion of resid and poor deashing. On January 23 (operation day, OD=10), the R1235 catalyst charge was increased to 270 lbs.

The plant was put on coal feed on January 12 at the following conditions:

Coal Feed:	
MF Coal Feed Rate	450 lb/hr
Wt % Coal in Slurry	33 wt %

Process Solvent	40 (target)/48/12 wt % :: Resid/Heavy Gas Oil/CI
R1235 First Stage Reactor:	
Reaction Temperature	760°F
Catalyst	EXP-AO-60
Initial Catalyst Charge	242 lbs
Catalyst Replacement Rate	Batch
R1236 Second Stage Reactor:	
Reaction Temperature	810°F
Catalyst	EXP-AO-60
Initial Catalyst Charge	241 lbs
Catalyst Replacement Rate	Batch

The following process changes were made since startup during tests with EXP-AO-60 catalyst:

Jan 15	Coal feed decreased to 400 MF lb/hr due to operational problems at the ROSE-SR℠ unit with high solvent content in the ROSE-SR℠ feed.
Jan 18	Coal feed increased to 425 MF lb/hr.
Jan 21	Coal feed increased to 450 MF lb/hr; V1067 Recycle to reduce V1082 feed.
Jan 23	R1235 temperature increased to 790°F; R1235 catalyst charge to 270 lbs.
Jan 24	Coal feed increased to 500 MF lb/hr.
Jan 28	Coal feed increased to 550 MF lb/hr; resid in process solvent set at 35 wt % due to overconversion.
Feb 1	Started catalyst replacement in R1235 (3 lb/T MF coal).
Feb 2	Started catalyst replacement in R1236 (3 lb/T MF coal).
Feb 8	Resid in process solvent set at 40 wt %.
Feb 20	Started batch deactivation in both stages.
Mar 2	Coal feed decreased from 550 to 425 MF lb/hr; V1082 Recycle established.
Mar 12	Resid in process solvent set at 50 wt %.
Mar 13	Cat. replacement started in R1236 at 1.5 lb/ton MF coal.
Mar 14	Cat. replacement started in R1235 at 1.5 lb/ton MF coal.
Mar 18	End-of-tests with EXP-AO-60 catalyst.

In Run 261, the process solvent was prepared by mixing the deashed resid from the ROSE-SR℠ unit, recycled resid containing solids (Cresol Insolubles, CI = ash + unconverted coal) from V1082 vacuum flash bottoms, and heavy gas oil (V1074) from the T102 vacuum column bottoms (98 wt % boiling above 750°F). The process solvent was blended in V131B vessel and then sent to the coal slurry preparation tank (V101A). The coal slurry in V101A

was transferred to the feed tank V101B. The slurry was fed using the P103 pump into the B1200 preheater and first stage reactor (Figure 3). The process solvent used at the start of the run contained heavy gas oil from Run 257 (Illinois coal) and solids and resid from Run 260 (Black Thunder mine subbituminous coal).

With the interstage separator (V1258) in service, cleaned gas from the flashed overheads was recycled to the first stage. The gas from the high flash separator (V1247) was cooled to knock out organics and compressed and recycled back to the second stage. The first and second stage recycle gases were mixed with fresh hydrogen in order to maintain the desirable hydrogen partial pressures in reactor feed. To keep the catalyst withdrawal tube in both reactors free from plugging, a withdrawal tube flush solvent is continuously pumped through the tube. This flush solvent was the T102 vacuum column overheads (V182) material during Run 261.

On January 15, the coal feed was decreased from 450 to 400 lb/hr to reduce the amount of solvent in the ROSE-SR^{SM} feed. With high amounts of solvent in the ROSE feed, the unit could not deash the resid. The deashing solvent strength in ROSE-SR^{SM} unit was as low as it could be. The use of subbituminous resid at the start of the run could have compounded the problem at the ROSE-SR^{SM} unit. The coal feed was increased on January 18 to 425 lb/hr because of a low solvent inventory.

Up until January 21, the solids were recycled using the V1082 vacuum flash bottoms. After this date, the solids were recycled by V1067 atmospheric flash bottoms because with higher feed rates to V1082, there were high solvent concentrations in the ROSE-SR^{SM} feed. The coal feed rate was also increased on January 21 to increase the process solvent inventory and amount of resid in the process solvent. Only 30 to 33 wt % resid concentration could be maintained in the process solvent.

On January 23 (OD=10), the catalyst charge in R1235 was brought up to 270 lbs. Also, the R1235 temperature was raised from 760 to 790°F to obtain higher distillate yields. Since this higher first stage temperature and added catalyst caused higher resid conversion, the coal feed rate was increased to 500 lb/hr on January 24 to increase the resid inventory.

On January 28 (OD=15), the coal feed rate was further increased to 550 lb/hr due to low inventory and lower than target resid concentration in the process solvent. There had been variations in the resid content of the process solvent between different batches of slurry. As a result, the resid concentration in the process solvent was targeted at 35 wt %.

Catalyst replacement began on February 1 (OD=19) at 3 lb/ton MF coal rate in the first stage. Catalyst replacement in both

reactors kept the equilibrium age constant during period 261B. Replacement was scheduled for every other day in each reactor. On February 2, due to the problems with the catalyst addition valves on the second stage (R1236), fresh catalyst could not be added and the plant went off coal feed on February 3. Also, the low pressure side block valves (located after R1236 and V1247) were leaking in the high pressure letdown system.

The plant was put on coal feed on February 7, but the V101A circulation line plugged bringing the plant off coal feed to clean the line out. The plant went on feed on February 8 (OD=21), and the resid concentration in the process solvent was changed to the target condition of 40 wt %. The resid content in the process solvent ranged from 34-40 wt % between February 1-24. After obtaining a data point, batch deactivation began on February 20 (OD=29).

There was down time starting February 16 to replace pipe that had holes in the lines from V1067 to V1082 and from V1079 to V1067. Also, during this time, problems with the P1222 ebullating pump were corrected. The plant was put on coal feed on February 20. However, on February 26, the plant was off line because the P1236 ebullating pump locked up. Both ebullating pumps were pulled, inspected, and repaired.

When the plant went on coal feed on March 2 (OD=35), the coal feed rate was reduced to 425 from 550 MF lb/hr. Also, solids recycle was established from the vacuum flash bottoms (V1082) instead of the atmospheric flash bottoms (V1067) where it was recycled from previously. A data point at 50 wt % resid in the process solvent was desired, therefore, batches of process solvent with the new composition were started on March 11. March 12 (OD=45) was the first full day of 50 wt % resid in the process solvent fed to the CCR unit.

Catalyst replacement in the second stage (R1236) began on March 13 (OD=46), and in the first stage (R1235), replacement began on March 14 (OD=47). Replacement at a rate of 1.5 lb catalyst/ton MF coal was continued by alternating addition/withdrawal in each reactor.

The plant went off feed for a planned shutdown on March 19 to remove the EXP-AO-60 catalyst from the reactors for an evaluation of its integrity. The consecutive withdrawals from one reactor approximately represent the successive layers of the catalyst bed in the reactor starting from the bottom of the reactor. Details about catalyst analyses are given in Section 5. The first and second stage catalyst recoveries were 109.0 and 89.7%, respectively.

In the second part of the Run 261, Criterion 324, a unimodal catalyst, was tested with the Illinois No. 6 coal to determine

operational stability and process performance. Run 259 was the first run in which the Shell 324 unimodal catalyst (recently Criterion Catalyst Co. changed the name of the Shell 324 catalyst to Criterion 324) was tested in CC-ITSL process over an extended period of time with a bituminous coal (Pittsburgh No. 8 seam coal from Ireland mine). Both low- and high-ash (about 4.7 and 14.7 wt % MF coal) Pittsburgh coal was tested, but the high ash coal with Shell catalyst was tested in just one period.

During the shutdown, 1690 lbs of Criterion 324 catalyst were presulfided from April 6 to 11. Two batches of catalyst, each batch with two full volume reactors, were sulfided for use in the catalyst replacement program. Part of the last batch was left in the reactor. Shell 324 1/16" catalyst from Run 259 was added to the reactors to make the 440 lbs catalyst charge in each reactor. The aged catalyst from Run 259 that was added to the first stage was 286.3 lbs (dry) and second stage was 302.6 lbs (dry). The aged catalyst from Run 259 plus the fresh catalyst gave starting ages of 527 lb MF coal/lb catalyst and 1024 lb (R+CI)/lb catalyst in the first stage (R1235) and 565 lb MF coal/lb catalyst and 1356 lb (R+CI)/lb catalyst in the second stage (R1236).

On April 12 (OD=52), the plant was put on coal feed for the second part of Run 261 at the conditions shown below. The initial startup of the CCR unit for this part of Run 261 was very smooth compared to Run 259. The targeted operating conditions were:

Coal Feed:	
MF Coal Feed Rate	425 lb/hr
Wt % Coal in Slurry	33 wt %
Process Solvent	50 wt % Resid/12 wt % CI/ 38 wt % Heavy Gas Oil
R1235 First Stage:	
Reaction Temperature	810°F
Reactor Volume	Full
Catalyst	Criterion 324 1/16", 440 lbs
Catalyst Replacement Rate	Batch
R1236 Second Stage:	
Reaction Temperature	825°F
Reactor Volume	Full
Catalyst	Criterion 324 1/16", 440 lbs
Catalyst Replacement Rate	Batch

The process changes made during tests with Criterion catalyst are listed below.

Apr 18	Coal feed was increased from 440 to 500 MF lb/hr.
	Cat. replacement in R1236 at 3.0 lb/ton MF coal.
Apr 19	Cat. replacement in R1235 at 3.0 lb/ton MF coal.

Apr 27 Batch deactivation.
Coal feed was decreased from 500 to 425 MF lb/hr.
Apr 29 Coal feed was decreased from 425 to 360 MF lb/hr.
Apr 30 Coal conc. in slurry decreased from 33 to 30 wt %.
2nd stage temp. decreased from 825 to 800°F.

May 8 Cat. replacement in R1236 at 2.25 lb/ton MF coal.
May 10 Cat. replacement in R1235 at 2.25 lb/ton MF coal.

May 23 Continuous resid injection system in service.

May 28 Adding ROSE-SR℠ deashed resid by V131B instead of V1090.

Since the V1082 bottoms was recycled during the tests with Criterion catalyst, all the distillate was sent to the T105/T102 distillation system. All the material boiling primarily above 850°F was recycled since the T102 bottoms was used as the heavy gas oil fraction in the pasting solvent makeup. The line from V1082 vacuum flash vessel to V131B (process solvent makeup vessel) was flushed after every transfer with heavy gas oil (V1074) after April 21. This was to help prevent plugging in that line. The material balances accounted for the flush material.

On April 18 (OD=57), the coal feed rate was increased from 425 to 500 lb MF coal/hr due to decreasing solvent inventories. Catalyst replacement was started in the second stage (R1236) at a rate of 3.0 lb catalyst/ton MF coal. Catalyst replacement was scheduled for every other day for each reactor, alternating reactors each day. Catalyst replacement began in the first stage (R1235) on April 19 (OD=58) at the same rate. At these conditions, process performance with Criterion catalyst could be compared with EXP-AO-60 catalyst (261B) at operating temperatures which possibly gives good process performance with the respective catalysts.

Batch deactivation commenced on April 27 (OD=66) to age the catalyst to a target age equivalent to 1.5 lb catalyst/ton MF coal. The coal feed rate was decreased from 500 to 425 lb MF coal/hr so that period 261D, with EXP-AO-60 catalyst, could be compared to operations using Criterion catalyst at similar coal feed rates and catalyst replacement rates.

As deactivation of the Criterion catalyst proceeded, the preasphaltenes in the process stream began to increase. Due to high viscosities and high discharge pressures on the slurry feed pump, the coal feed rate and the coal concentration in the slurry were lowered from 425 to 360 lb MF coal/hr and from 33 to 30 wt %, respectively, on April 29 and 30. Operations could only achieve temperatures below 810°F in the second stage reactor at the lower coal feed rate (without exceeding the maximum

interstage heater outlet temperature for safe operation). The second stage temperature was set at 800°F (a decrease from the target temperature of 825°F) on April 30 (OD=69) in anticipation that further deactivation would result in a decrease in the second stage reactor temperature.

Even at a lower coal feed rate and coal concentration in the slurry, continued batch deactivation resulted in a gradual increase in discharge pressure on the feed pump. The discharge pressure was once again approaching the relief settings. The operating constraints at these conditions made it difficult to batch deactivate to an age equivalent to 1.5 lb catalyst/ton MF coal as previously planned. Therefore, it was decided to perform catalyst replacement at 2.25 lb catalyst/ton MF coal.

On May 7, the first stage withdrawal tube plugged and catalyst replacement could not be accomplished. Catalyst replacement in the second stage was executed on May 8 (OD=77). The plug in the first stage was cleared on May 10 and double the required amount of catalyst was withdrawn and added. A double addition/-withdrawal was also completed in the second stage on May 11. These additional catalyst replacements ensured that the catalyst age in both reactors was equivalent to 2.25 lb catalyst/ton MF coal replaement rate.

The plant went off-feed on May 12 due to a leak below the atmospheric flash vessel (V1250). The leak was caused by a worn elbow in the line. The line was approximately ten years old. After repairs, the plant went on-feed on May 14. Catalyst replacement at 2.25 lb catalyst/ton MF coal continued to be scheduled every other day for each reactor. The test with Criterion catalyst at steady state conditions with catalyst replacement ended on May 22 (OD=89).

The new continuous resid injection system was put in service on May 23 (OD=90). This new system injects the ROSE-SR^{SM} deashed resid and the V1082 bottoms (solids recycle) into the feed line directly. The coal is slurried with V1074 solvent (heavy gas oil from T102 vacuum column bottoms) in the coal slurry tank (V101A). The ROSE-SR^{SM} resid rate is determined from a calibration/holding pot, V1090. The feed pump of the ROSE-SR^{SM} resid is P1090. The V1082 bottoms (solids recycle stream) was calibrated in V1091 and pumped by P1091.

Continuous resid injection has the potential to decrease the resid inventory by about one-third, and this may reduce resid degradation. Also, the time taken to reach steady state, once an operational condition change is made, can be minimized. A detailed discussion of the new continuous resid injection system and the tests performed to determine the operational limitations are given in Section 7.1.

In the first test with the new resid injection system, the targeted coal concentration in the V1074 solvent was 53 wt %. This would give 30 wt % coal concentration in the slurry to the reactor feed. In order to keep circulation in V101A, the slurry was backflushed many times from V101B to V101A. For almost all batches, additional V1074 solvent had to be added to V101A to alleviate pumping problems. The additional solvent decreased the coal concentration from 53 to 50 wt %. Keeping circulation on both V101A and V101B at high coal concentration was a major battle throughout the test.

On May 28 (OD=95), the ROSE-SR^{SM} resid (deashed resid) injection by P1090 was discontinued. Instead, ROSE-SR^{SM} resid was added to V131B at 29 wt % along with V1074 solvent at 71 wt %. The coal was slurried in this mixture at a concentration of 47 wt %. This would give 33 wt % coal concentration in the slurry to the reactor feed. The V1082 bottoms material continued to be injected into the feed stream by P1091. On the 29th of May, the coal concentration was set at 45 wt % coal in the pasting solvent (to give 30 wt % coal concentration in the reactor feed). Smooth operation with slurry preparation was realized at 45 wt % coal concentration.

Run 261 ended on May 31, 1991 at 07:40 hrs. During the entire run, the CCR unit was on stream for 2333 hours for an on-stream efficiency of 85.4%. The Criterion catalyst was withdrawn and sampled for activity and integrity. The catalyst recovery was 96.2% in the first stage and 77.0% in the second stage.

The TSL yields before elemental balancing (Phase 2 data) and the close-coupled reactors operating data are summarized in Tables 3 and 4. The resid+UC conversion data from Phase 2 material balance is shown in Figure 8. The R+UC conversion decreased sharply in the first stage more so than the second stage during initial batch deactivation of the fresh EXP-AO-60 catalyst. When the first stage temperature was increased to 790°F and the catalyst charge was brought up to the target level (270 lbs) on January 23 (OD=10), the first stage R+UC conversion was higher than the second stage R+UC conversion.

During catalyst replacement at 3 lb catalyst/ton MF coal (OD=19-28), the R+UC conversions remained constant in both stages. Higher first stage conversions compared to second stage are seen at 50 wt % recycle resid operation (OD=45-51) at a catalyst replacement rate of 1.5 lb catalyst/ton MF coal.

With Criterion catalyst operation, at a catalyst replacement rate of 3 lb/ton MF coal, the first stage conversions appear to be lower than the second stage (OD=58-65). Batch deactivation of Criterion catalyst in both stages show first stage R+UC conversions declining more rapidly compared to second stage conversions. A significant gradual decline in overall conversion was observed during batch deactivation. The overall R+UC

conversion shows a significant decrease at 2.25 lb catalyst/ton coal replacement with Criterion catalyst (OD=78-89) when compared to operations with EXP-AO-60 catalyst at a replacement rate of 1.5 lb catalyst/ton coal (OD=29-44).

A qualitative representation of the second stage ebullation flow rates for the entire run is shown in Figure 9. The first stage ebullation flow rates are not plotted due to measurement errors. Ebullation flow rates are adjusted as necessary to maintain the catalyst bed ebullated to a fixed height in the reactor. Operations with Criterion catalyst (from OD=52) show higher ebullation rates than with EXP-AO-60 catalyst. Lowering of second stage temperature and and coal feed rates (OD=66) results in an increase in ebullation rates.

Higher ebullation rates promote mixing in the reactor which results in lower temperature rises within the reactor. One would expect much higher ebullation rates with unimodal catalyst due to their higher compacted bulk density (54 lb/cu ft for Criterion versus 33 lb/cu ft for EXP-AO-60 catalyst). But, in this run, the ebullation rates with Criterion catalyst was not much different with EXP-AO-60 catalyst, persumably due to the higher viscosity of the process material during Criterion catalyst operation. The gas rates, which also has an effect on bed expansion, did not vary much during the entire run.

The temperature rise in the reactor bed and across the reactor are shown in Figure 10 for both reactors. Deactivation of fresh EXP-AO-60 catalyst from the start of the run to an age equivalent to 1.5 lb catalyst/ton replacement rate at the end of period 261C decreased the average bed exotherm from 43 to 31°F in the first stage. The increase in recycle resid concentration from 40 to 50 wt % in the process solvent in period 261D increased the first stage average bed exotherm from about 31 to 36°F.

The average in-bed temperature rise was lower with the Criterion 324 catalyst operation (25 to 28°F) than with EXP-AO-60 catalyst operation (31 to 43°F) in the first stage. The average in-bed difference in temperature for Run 257 was about 40°F in the first stage with Amocat 1C catalyst and at a coal feed rate of 420 to 530 lb MF coal/hr. At a coal feed rate of 345 lb MF coal/hr in Run 257, the average in-bed temperature rise in the first stage was about 33°F.

During batch deactivation of Criterion catalyst from an age equivalent to 3 to 2.25 lb catalyst/ton MF coal, the average temperature rise in the bed steadily decreased in the first stage from 28 to 25°F. In the second stage with Criterion catalyst, the temperature rise in the bed decreased from 12.6 to about 7.5°F when the coal feed rate was lowered to 360 lb MF coal/hr and the average second stage reactor temperature was decreased from 825 to 800°F.

3.4 Residuum Oil Supercritical Extraction - Solids Rejection (ROSE-SR℠) Unit

The objectives of Run 261 for the Residuum Oil Supercritical Extraction - Solids Rejection (ROSE-SR℠) unit were to

- Evaluate unit performance while using both EXP-AO-60 and Criterion 324 catalysts with Illinois No. 6 coal.
- Recover full range resid used in coal slurry preparation while achieving acceptable deashing.
- Obtain optimum resid recovery with minimum energy rejected into the bottoms product.

3.4.1 Unit Operation and Process Performance

The unit operated well during Run 261. Performance is summarized in Tables 5 and 6, and Figures 11 and 12. The temperature and pressure of the separating vessels and the strength of the deashing solvent were varied to prevent letdown header pluggage and to improve process performance and operability. The ROSE-SR℠ unit was on feed for 2240.1 hours to give an on-stream efficiency of 99.0 % Run 261.

The unit went on feed January 13. The feed to the ROSE-SR℠ unit came from V1082 (vacuum flash) bottoms. At the beginning of Run 261, the amount of solvent in the feed was high causing the unit to be unable to extract the resid from the slurry. Ash carryover occurred from January 13 to January 20 due to very low preasphaltenes as well as a higher amounts of solvent in the ROSE-SR℠ feed. The beginning of the run started by processing subbituminous resid (Black Thunder coal) from Run 260. The DAS strength had to be the lowest possible in order to acheive reasonable deashing. Solvent in the ROSE-SR℠ feed acts as a DAS strengthening agent. Therefore, when needing the lowest DAS strength to deash the slurry, the high solvent in the feed strengthened the DAS causing unacceptable deashing. On January 21, the solids recycle was changed from the V1082 vacuum flash bottoms to the V1067 atmospheric flash bottoms to reduce the rate through V1082. Ash carryover occurred on February 14 due to problems with the second stage pressure transmitter diaphragm causing the first stage pressure to float on the second stage pressure.

On April 13, the ROSE-SR℠ unit was put on feed for the second part of Run 261 with Criterion 324 catalyst and higher reactor temperatures. Ash carryover occurred April 13 through 15 due to water in the deashing solvent. On April 16, the fill valves on the feed measuring pot had solids in them causing the unit to be down 4.5 hours. The resid content in the process solvent varied

during this time due to operational problems at the ROSE-SR^{SM} unit. Once these startup problems were solved, the unit ran well after April 17. The deashing solvent strength gradually increased when operating at these new conditions.

The unit operated very well through 261E, 261F, and 261G. There was no down time during these periods and the only ash carry over occurred on the last day of 261G and was minor. The performance was excellent during resid injection (261G) with resid recovery at 88.2 wt %, energy rejection at 17.8%, and organic rejection at 15.0 wt %.

On May 22, the resid injection system was put into service which upset the material balance at the ROSE-SR^{SM} unit since both feed and resid were going to drums. However, the unit performed very well during the resid injection test except for ash carryover on May 27 and May 29 which was caused by operational problems not related to resid injection.

Preasphaltenes were low for most of Run 261 using EXP-AO-60 catalyst. After switching to Criterion 324, day 53 on Figure 13, the preasphaltene level increased dramatically but then decreased somewhat due to a reduction in reactor temperature and feed rates.

Figures 14 and 15 show that the nitrogen and sulfur levels in the bottoms product did not significantly change when switching from EXP-AO-60 catalyst to Criterion 324.

The ROSE-SR^{SM} resid as a percentage of total resid in process solvent ranged from 32% to 37% in periods 261B-E. These periods were chosen to give examples of both EXP-AO-60 and Criterian 324 catalysts and operations at both 40 and 50 wt % recycle resid in the process solvent. The ROSE-SR^{SM} resid as a percentage of total resid ranged from 6 to 24% in Run 260 for subbituminous coal.

3.5 Distillation System

The objectives of the V1082/T105/T102 distillation system for Run 261 were:

- To remove low boiling point products in T105 from the interstage separator overheads (when in service) and from the liquid flashed overhead in the second stage reactor.

- To split in T102, the T105 bottoms and heavy vacuum flashed overheads (from V1082) into a product stream and a recycle distillate stream that is in balance with the recycle distillate needs for blending with the coal.

Run 261 was the third consecutive run to use T102 for adjusting the entire split between recycled distillates and product distillates except for periods 261A and 261B as stated in Section 3.1.1. In periods 261A and 261B, the solvent present in the V1067 recycle stream contained some distillates boiling below 650°F. Essentially all of the cut points (obtained from GC simulated distillation and by equal weight percent overlap method) were above 770°F (Table 7 and Figure 16).

The lowest cut point was during period 261G, however, this is primarily the result of a significant shift in inventories due to the resid injection test. In the resid injection scheme, most of the inventory of resid and CI is replaced with distillate. The average overlap for the run averaged from 6 to 9 wt % (Table 7, Figure 17).

The light distillates (V161 in Figure 3) was the overheads from the second stage. The T105 overheads had a boiling point below 600°F. About 50% of the T105 overheads boiled below 350°F. Prior to period 261E, the boiling point distribution changed when, mistakenly, the heavier V1072 distillate was also fed to the column resulting in more light products in T105 bottoms and hence in V182 (T102 overheads, Figure 19). For most of the run, T102 overheads (V182, Figure 19) had little material boiling above 850°F by GC simulated distillation. The recycle distillate (V1074 in Figure 20) was very heavy with 70-80 wt % boiling above 850°F by GC simulated distillation. This stream also contained very little boiling below 750°F.

4. OVERALL TWO-STAGE LIQUEFACTION YIELDS

The overall TSL yields are the result of averaged elemental balances around the unit. The analytical data used for these balances are presented in Tables 8 - 10. Operating conditions, averaged elementally balanced overall yields and unit contributions are summarized in Tables B & C for all representative periods. The yield contributions are also shown schematically in material balance flow diagrams (Figures 21-27).

4.1 TSL Performance

The initial part of Run 261 consisted of testing the EXP-AO-60, 1/16" (Ni-Mo, bimodal) catalyst, using full-volume reactors at two different catalyst replacement rates. The Illinois No. 6 coal from Burning Star #2 mine was used throughout the run. Although it was intended to recycle the vacuum flash bottoms, due to operational problems, the first two periods were obtained with atmospheric flash recycle. Vacuum flash recycle was used for the remainder of the run. These tests were made in low/high temperature mode.

In period 261A, the coal feed rate was 543 lb/hr at a coal concentration of 33 wt %. Although the target resid concentration in the process solvent was 40 wt %, because of the high activity of the fresh catalyst and low process solvent inventory, only about 33 wt % resid concentration could be obtained. However, attempts were made during this period to increase resid concentration in the process solvent. Also, in this period, catalyst replacement was started at a rate of 3 lb/ton of MF coal. Thus, this period was termed transitional. In period 261A, the distillate yield and net resid yield were 59.0 and 7.4 wt %, respectively. The first stage and overall TSL conversions were 84.5 and 91.1 wt % MAF coal, respectively.

A high coal feed rate of 550 lb/hr was again maintained in period 261B. However, the resid concentration could be increased to 38.3 wt %. The distillate and net resid yields were 64.4 and 4.7 wt % MAF coal, respectively. The first stage and TSL coal conversions were 87.0 and 91.7 wt % MAF coal, respectively. Lower first stage temperature and high space velocity have probably contributed to the low first stage coal conversion. The C_1-C_3 yield appeared to be low in this period.

In period 261C, vacuum flash recycle was started. In this period, the catalyst was being deactivated to an age equivalent to 1.5 lb/ton replacement rate. The coal feed rate was about 420 lb/hr. No catalyst replacement was performed during this period. The coal conversion was about 94 wt % and the C_4+distillate yield was about 64.8 wt %.

In period 261D, catalyst replacement was performed in each stage at a rate of 1.5 lb/ton. The C_4+ distillate yield and net resid yield were 65.6 and 3.7 wt %, respectively. The organics in the ROSE-SR℠ bottoms product was low in this period, only 15.3 wt % MAF coal.

Period 261E was the first period with Criterion 324 catalyst. The aged catalyst from Run 259 was used in both stages; however, some fresh catalyst was added to obtain appropriate catalyst age, which required a short period of batch deactivation. The coal feed rate was about 494 lb/hr. The catalyst replacement was 3 lb/ton in each stage. The C_4+ distillate yield was about 61 wt % and the net resid make was about 5 wt %. Periods 261B and 261E were obtained at similar catalyst replacement rates. However, there were some differences: in period 261B, coal feed rate was about 550 lb/hr, solids recycle was through atmospheric flash bottoms, and resid concentration in the solvent was 38.3 wt %. Whereas, in period 261E, resid concentration in process solvent was 48.9 wt %, and solids were recycled using vacuum flash bottoms.

The catalyst was deactivated and a data point was obtained at a replacement rate of 2.25 lb/ton of coal. Because of the reduced catalyst replacement rate, the coal feed rate was reduced to 359 lb/hr. At this lower coal feed rate, the exotherms were low and heat losses could be relatively higher; thus, the second stage temperature could not be maintained at the target 825°F. It was set at about 800°F. The distillate yield decreased to 58.4 wt %, and resid yield increased to 10.3 wt %. This period was termed unstable because of high closure errors in Phase 2 data. It may be noted that because of high operation temperatures, from period 261E onwards the C_1-C_3 gas make increased to about 7.7 wt % MAF coal.

After obtaining period 261F, the new continuous resid injection test was started. In this test, coal and the heavy gas oil portion of the process solvent were mixed in the slurry tank. The vacuum flash bottoms and ROSE-SR℠ resid were added to the high pressure feed line to the B1200 preheater. However, the coal + heavy gas oil slurry prepared to give target process solvent and slurry compositions gave a high viscosity which caused recirculation pump problems in the slurry tank. The operable coal + heavy gas oil composition was further explored. Since the slurry could not be maintained at target composition, period 261G was termed transitional. In this period, the distillate yield was higher than in period 261F (64.3 vs 58.4 wt %). The net resid yield was lower in period 261G compared to 261F (5.1 vs 10.3 wt %).

4.2 Process Solvent Quality and Hydrogenation

The process solvent qualities for different periods in Run 261 are given in Table 11. Operation with EXP-AO-60 catalyst gave much higher process solvent quality than operation with Criterion 324 catalyst. The solvent qualities in Run 261 ranged from 81 to 85% with EXP-AO-60 catalyst operation and from 76 to 77% with Criterion 324 catalyst operation. Besides differences in catalyst during these two test periods, the reaction temperatures were higher in Criterion catalyst operation compared to EXP-AO-60 catalyst operation. The higher reaction temperatures may have had a negative affect on the solvent quality.

The solvent quality decreased from 83.5 to 81% with higher EXP-AO-60 catalyst ages and higher resid concentration (40 vs. 50 wt %) when comparing the quality in 261B and 261D. With higher Criterion 324 catalyst ages the solvent quality decreased from 77.2 to 75.9% in 261E and 261F, respectively.

When comparing the solvent quality during Run 261 with EXP-AO-60 catalyst to the quality during Run 257 with Amocat 1C catalyst, the solvent qualities are similar. Table 12 shows the process solvent quality and preasphaltene content of the ROSE-SR^{SM} feed for Runs 261 and 257. The preasphaltene content in both runs were similar also.

The hydrogen content of the process solvent and interstage streams are shown in Figure 28. At the beginning of the run, the hydrogen in the process solvent was very high at 8.5 wt % causing problems with deashing in the ROSE-SR^{SM} unit. Thoughout this run, the amount of hydrogen in both the process solvent and the interstage stream has been very similar. There was a decrease in the hydrogen content in both streams when the first stage temperature was increased from 760 to 790°F and the feed rate was increased to 550 lb MF coal/hr. The hydrogen content remained constant during catalyst replacement and decreased during batch deactivation.

During batch deactivation from an age equivalent to 3.0 to 1.5 lb catalyst/ton MF coal, the process solvent resid hydrogen declined faster than the interstage resid hydrogen.

When the amount of distillate in the coal feed slurry was reduced by targeting a higher amount of resid (40 vs. 50 wt %), there was a reduction in hydrogen content. When the Criterion catalyst test began, the hydrogen content in the process solvent and the interstage stream decreased from 7.1 with EXP-AO-60 catalyst to 6.2 wt % with Criterion catalyst. Besides differences in catalyst, higher reaction temperatures with Criterion catalyst operation contributed to the decrease in hydrogen content.

Both the distillate and resid portions of the process solvent and

of the interstage stream had less hydrogen content with Criterion catalyst operation than with EXP-AO-60 catalyst operation (Figures 29 and 30). Evidently at higher reaction temperatures with Criterion catalyst, hydrogenation activity seemed to be controlled by thermodynamic limitations rather than by kinetics. In the interstage stream, however, there was a greater change in the resid portion (a change from 7 to 6 wt % hydrogen). Even with the higher reaction temperatures with Criterion catalyst which should give higher catalyst activity, there was less resid hydrogen, indicating that the Criterion catalyst was cracking more than hydrogenating. And, since there was less hydrogenation, the solvent quality was lower during the Criterion catalyst periods.

Table 13 compares the hydrogen content in the process solvent and interstage streams during Run 257 and Run 261. The resid hydrogen in both the process solvent and the interstage stream was lower in Run 261 than in Run 257. Again, thermodynamics seemed to favor hydrogenation at lower reaction temepratures in Run 257. The resid hydrogen in the process solvent averaged 7.4 wt % for Run 261, compared to a range of 7.4 wt % to 8.2 wt % in Run 257. The interstage resid hydrogen averaged 7.1 wt % in Run 261 and in Run 257 it ranged from 7.2 to 8.0 wt %.

5. Catalyst

The 1/16" EXP-AO-60 catalyst tested in this run has a bimodal pore structure support as the 1/12" Amocat 1C catalyst tested in Run 257. A comparison of properties of various bimodal catalysts tested at Wilsonville is given in Table 14. The EXP-AO-60 catalyst is a Ni-Mo (2.5/10.7 wt %) catalyst. Table 14 shows that the Ni-Mo content is similar to other bimodal catalysts. The EXP-AO-60 catalyst has more surface area compared to Amocat 1C 1/16" catalyst (241 vs 190 sqm/g) and lower pore volume (0.78 vs 0.85 cc/g) and lower bulk density (33 vs 42 lb/cuft). The EXP-AO-60 catalyst is an improvement over Amocat 1C catalyst in that it is expected to have a better physical strength, i.e., less breakage with a smaller size and a less pore volume and to improve catalyst activity with a higher surface area. Also, a problem during Amocat 1C manufacturing process which might have contributed to its decreased strength was carefully eliminated by catalyst manufacturer during EXP-AO-60 catalyst production.

On January 1, sulfiding of fresh EXP-AO-60 catalyst was started. Four batches of two, full-volume reactors were sulfided to give 2220 pounds for use in the catalyst replacement program during the run. Part of the last batch was left in the reactors for startup. The initial catalyst charge for startup was 241 lbs dry in R1235 (first stage reactor) and 242 lbs dry in R1236 (second stage reactor). On January 14, the catalyst charge in R1236 was increased to the run conditions (270 lbs). The R1235 catalyst charge was not increased at this time because of overconversion of resid and poor deashing at the ROSE-SR℠ unit. The catalyst charge in the first stage was raised to 270 lbs on January 23.

Catalyst replacement began on February 1 at a 3 lb catalyst/ton MF coal rate in the first stage. On February 2, due to problems with the catalyst addition valves on the second stage, fresh catalyst could not be added and the plant went off coal feed from February 3 to 7. Catalyst replacement in both reactors kept the equilibrium age constant during period 261B. Replacement was scheduled for every other day in each reactor. After obtaining a data point at 3 lb catalyst/ton MF coal, batch deactivation began on February 20.

Catalyst replacement in the second stage (R1236) at a rate of 1.5 lb catalyst/ton MF coal began on March 13. On March 14, replacement began in the first stage (R1235). Replacement was continued by alternating addition/withdrawal in each reactor.

The plant went off feed for a planned shutdown on March 19 to remove the EXP-AO-60 catalyst from the reactors for an evaluation of its integrity.

During the shutdown, 1690 lbs of Criterion 324 catalyst were

presulfided from April 6 to 11. Two batches of catalyst, each batch encompassing two, full-volume reactor amounts, were sulfided for use in the catalyst replacement program. Part of the last batch was left in the reactor. Shell 324 1/16" catalyst from Run 259 was added to the reactors to make the 440 lbs catalyst charge in each reactor. The catalyst from Run 259 that was added to the first stage was 286.3 lbs and had an age of 810 lb MF coal/lb catalyst or 1573 lb (R+CI)/lb catalyst. The catalyst added to the second stage from Run 259 was 302.6 lbs with an age of 821 lb MF coal/lb catalyst or 1971 lb (R+CI)/lb catalyst. The aged catalyst from Run 259 plus the presulfided catalyst gave a starting age of

R1235	527	lb MF coal/lb catalyst
	1024	lb (R+CI)/lb catalyst
R1236	565	lb MF coal/lb catalyst
	1356	lb (R+CI)/lb catalyst.

On April 18, catalyst replacement was started in R1236 at a rate of 3.0 lb catalyst/ton MF coal. At this time, operation with EXP-AO-60 catalyst was compared to operation with Criterion catalyst at conditions for maximum process performance. Catalyst replacement began in R1235 on April 19 at the same rate. Batch deactivation commenced on April 27.

Originally, it was planned to batch deactivate until the catalyst age was equivalent to 1.5 lb catalyst/ton MF coal, however, operating constraints made it impossible to do so. The viscosity and coal slurry discharge pressure increased during batch deactivation to such a high extent that the coal feed rate was lowered from 425 to 360 lb MF coal/hr and the coal concentration was decreased from 33 to 30 wt % to have acceptable viscosity. It was decided to perform catalyst replacement at a rate of 2.25 lb catalyst/ton MF coal since the coal slurry discharge pressure and viscosity continued to increase.

On May 7, the first stage withdrawal tube plugged and catalyst replacement could not be accomplished. Catalyst replacement in the second stage was performed on May 8. The plug in the first stage was cleared on May 10 and a double amount of catalyst was withdrawn and added. A double addition/withdrawal was also completed in the second stage on May 11.

During the test with the resid injection system, the catalyst ages in both stages were maintained with a replacement rate of 2.25 lb catalyst/ton MF coal in each reactor.

5.1 Catalyst Sulfiding Procedure

The EXP-AO-60 and Criterion 324 catalysts used in Run 261 were

sulfided using the following procedure:

The fresh catalyst was sulfided with dimethyl disulfide (DMDS) at 1.5 wt % initial concentration in recirculating No. 2 diesel (fuel) oil. The feed gas rate was maintained at 3,000 SCFH, and catalyst bed ebullation began when the reactor temperature reached 250°F. The reactor was heated stepwise by 50°F from 250 to 400°F. The temperature was held at 400°F until the hydrogen sulfide "breakthrough" occurred, indicating the end point of the sulfiding at this temperature. During the sulfiding, the hydrogen-rich vent gas was recycled and DMDS was added to the diesel oil at a rate of 12 lbs/hr. The reactor temperature was again increased stepwise by 50°F and held at 500°F, 600°F, and 700°F until the breakthrough occurred. At the maximum temperature (700°F), the reactor was held until the analysis of the catalyst samples indicated a sulfur content (wt % S = 100 x "as-is" wt % S/wt % ash) of at least 8 wt % for the EXP-AO-60 catalyst and 9 wt % for the Criterion 324 catalyst. The reactor was cooled at a maximum rate of 100°F/hr until it was less than 300°F. The catalyst was withdrawn and stored in drums at ambient conditions and covered with fuel oil.

The analytical results for the fresh sulfided EXP-AO-60 catalyst, fresh Criterion 324 catalyst, and withdrawn catalyst along with the daily analytical results, are shown in Tables 15 and 16. Fresh sulfided EXP-AO-60 catalyst had an average naphthalene activity of 186 mmoles H_2 consumed. In the first stage and 192 mmoles H_2 consumed in the second stage. The average carbon buildup during sulfiding was 1.0 wt % in the first stage and 1.1 wt % in the second stage. Some breakage occurred to the EXP-AO-60 catalyst during the sulfiding process:

EXP-AO-60	U.S. Standard Screen Size Distribution			
	# 14	# 18	# 25	- 25
R1235	91.4	4.6	1.7	2.3
R1236	91.6	4.9	1.7	1.8

The fresh, sulfided Criterion 324 catalyst in the first stage had an average naphthalene activity of 180 mmoles of H_2 consumed and in the second stage the catalyst had an activity of 195 mmoles H_2 consumed. The carbon deposited on the fresh, sulfided Criterion 324 catalyst in the first stage was 1.4 wt % and on the second stage catalyst the carbon deposition was 1.0 wt %. Very little breakage of the Criterion 324 catalyst occurred during the presulfiding process:

Criterion 324 Catalyst	U.S. Standard Screen Size Distribution			
	#14	#18	#25	-25
R1235	98.4	1.3	0.2	0.1
R1236	98.8	0.8	0.2	0.2

5.2 Analytical Results and Recovery

Analytical results of each catalyst sample include an elemental analysis, a screen analysis, an ash evaluation, and a naphthalene activity test. The carbon content was evaluated to determine any buildup that would inhibit catalyst activity. The naphthalene activity test determines the hydrogen consumption during the hydrogenation of a fixed volume of catalyst in a laboratory microautoclave reactor, measured in units of millimoles of hydrogen consumed per 100 g of liquid sample. Naphthalene is the model compound used to measure relative catalyst activity in a laboratory microautoclave reactor and is independent of pilot plant reactor or TSL system performance.

Recovery of catalyst was calculated when the entire catalyst bed was removed from the reactor. Since the reported weight was always a dry weight, the ash analysis was used to determine the difference in the actual wet weight and the theoretical dry weight. Due to the additional process ash remaining in the reactor after shutdown, the optimum recovery was usually 107 wt%.

Several catalyst samples were sent to outside laboratories (Alabama Power Company General Laboratory and Galbraith Laboratories) for metals deposition analysis. These results are shown in Table 22.

5.2.1 Catalyst Size and Recovery

In Run 257 with Amocat 1C 1/12" catalyst, there was a considerable amount of breakage and a significant amount of catalyst fines were suspected to be found in the top of the bed. Therefore, with the new bimodal catalyst, the integrity was monitored closely. After testing the EXP-AO-60 catalyst, it was removed from the reactors for an evaluation of its integrity. Each withdrawal of catalyst was placed in a separate, labelled, 5 gallon bucket. The buckets were combined to make seven composite samples for each reactor with Composite #1 representing the bottom of the bed and Composite #7 the top of the bed. Table 17 and 18 show the "End-of-Test" composite mesh analysis. The first and second stage EXP-AO-60 catalyst recoveries were 109.0 and 89.7%, respectively. Good integrity for the EXP-AO-60 catalyst was shown by the screen analysis for both stages. The EXP-AO-60 catalyst had the same integrity as Shell 324 from Run 259 and higher integrity than Amocat 1C in Runs 257 and 259.

The Criterion catalyst was withdrawn and sampled for activity and integrity at the end of the run. The mesh analysis at the end of the run is shown in Tables 19 and 20. The catalyst recovery was 96.2% in the first stage and 77.0% in the second stage. A number of circumstances may explain the low recovery in the second stage. New plenum chambers were installed for this run. At the

end of the entire run, the top surface of the plenum chamber in the second stage had grooves worn where the process stream hits the surface and distributes into the reactor. In the process of shutdown, the interstage separator was depressured without the overheads blocked. When ebullation flow could not be reestablished, an abrupt shutdown was made. No catalyst was found in the interstage separator or in the B1201 interstage heater. With both the EXP-AO-60 and the Criterion 324 catalysts, the initial catalyst charge after presulfiding was determined by subtraction. The amount of catalyst withdrawn after presulfiding was subtracted from the amount of catalyst charged to determine the amount of catalyst left in the reactor for startup. Samples of ROSE-SR^{SM} bottoms product were sent for metal analysis to check for nickel and molybdenum to verify if the catalyst was ground and carried over during the run. Metal analysis results did not show any indication of catalyst carryover, since all bottoms product samples from January 30, April 17, April 26, May 4, May 20 and May 25 had nickel oxide and molybdenum oxide contents (0.1 wt % in ash) similar to those in Illinois feed coal ash. Theoretically, if catalyst was continuously carried over at the same loss rate for the period with Criterion catalyst (45 days), the molybdenum oxide content in the bottoms product should have been increasing gradually above 0.2 wt % in ash.

5.2.2 Catalyst Elemental Analysis and Naphthalene Activity

The carbon deposition in the first stage ranged from 12 to 13 wt % in Run 261 with EXP-AO-60 catalyst. In Run 257, the carbon deposition on the bimodal Amocat 1C 1/12" catalyst was slightly lower at 11 to 12 wt%. The reaction temperature of the first stage was 790°F for both Run 261 with EXP-AO-60 catalyst and for Run 257 with Amocat 1C catalyst.

The carbon deposition in the second stage increased gradually reaching a high of 15 wt % in Run 261 with EXP-AO-60 catalyst. In Run 257, the carbon deposition on the Amocat 1C ranged from 9 to 11 wt % which is quite a bit lower than in Run 261. The second stage temperature in Run 257 was 807°F in 257H, 789°F in 257G, and 760°F in all other periods. In Run 261 with EXP-AO-60 catalyst operation, the second stage temperature was 810°F. See Table 21 for carbon deposition on the EXP-AO-60 catalyst in Run 261 and on the Amocat 1C catalyst in Run 257.

Once Criterion 324 catalyst was used with higher first stage temperatures (810°F), the carbon deposits on the catalyst increased to 18 wt%. In Run 259 when the second stage was operated at 790°F, the carbon deposition on the Shell 324 catalyst was 15 wt%, and when the temperature was 810°F in the first stage, the carbon deposit was 14.5 wt%. See Figure 31 for the carbon deposition on the EXP-AO-60 catalyst and the Criterion catalyst in the first stage during Run 261.

With the second stage temperature at 825°F for Criterion 324 catalyst operation, the carbon deposits increased to 18 wt%. Once the second stage temperature was decreased to 800°F, the carbon deposition on the Criterion catalyst also decreased to 16.5 wt %. In Run 259 with first stage reactor temperatures of 810 and 825°F and using Shell 324 catalyst, the carbon deposition was similar to that found for the Criterion 324 catalyst in Run 261. See Figure 32 for the carbon deposition on the catalysts in Run 261 in the second stage.

In the first stage, the carbon to hydrogen atomic ratio of the Criterion catalyst (1.5) was higher than the ratio of the EXP-AO-60 catalyst (1.2) in the first stage. The ratio for the Criterion catalyst was similar to the ratio for the Shell 324 catalyst in Run 259. In the second stage, the carbon to hydrogen ratio was similar for both catalysts (1.5) possibly due to operation at similar reactor temperatures.

The sulfur on the catalysts in Run 261 are shown in Figures 31 and 32. Sulfur deposits were slightly greater on the Criterion catalyst than on the EXP-AO-60 catalyst (7.0 wt % vs. 6.4 wt %).

The nitrogen retained on the Criterion 324 catalyst was higher than on the EXP-AO-60 catalyst (0.4 vs. 0.3 wt %) in both reactors.

During the EXP-AO-60 catalyst test, the naphthalene activity remained constant at approximately 50 mmoles of hydrogen consumed in the first stage. The catalyst withdrawn at the end of the EXP-AO-60 test from the first stage also had this activity. The Criterion catalyst had a lower naphthalene activity of 30 mmoles of hydrogen due to higher carbon retained on the catalyst. This activity was similar to what was found in Run 259.

The naphthalene activity of the EXP-AO-60 catalyst in the second stage was within the range of 50 to 60 mmoles of hydrogen. The EXP-AO-60 second stage catalyst withdrawn at the end of the test had a lower naphthalene activity of 30 mmoles of hydrogen. The Criterion catalyst in the second stage had naphthalene activity similar to that in Run 259 of around 30 mmoles of hydrogen.

Figure 33 shows naphthalene activity vs. carbon deposition for Run 261. The second stage had higher naphthalene activity at similar carbon deposition. Due to higher second stage temperatures during the EXP-AO-60 catalyst operation, there was more carbon deposition in the second stage than in the first stage.

Metal analyses of the EXP-AO-60 and Criterion 324 catalysts during the run are given in Table 22. Catalyst samples were THF extracted and then calcined before performing metal analysis. The analyses reported in Table 22 are for metals in the most

probable form of their oxides. The calcium and iron increased in both stages throughout EXP-AO-60 catalyst operation but to a greater extent in the first stage. The magnesium increased in the first stage but not in the second stage. There was no loss of nickel in either stage. Importantly, the titanium increased in both reactors but to a greater extent in the first reactor.

5.2.3 Catalyst Characterization - Comparisons to Previous Runs

Figure 34 compares bimodal catalyst naphthalene activity versus carbon deposition from Runs 253, 254, 257 and 261. The naphthalene activity at similar carbon deposition for EXP-AO-60 catalyst used in Run 261 was greater than both Amocat 1C and Shell 317 catalysts used in Runs 257 and 254, respectively, and was similar to that for Shell 317 catalyst used in Run 253. During normal operation, both the EXP-AO-60 and the Amocat 1C catalysts had similar naphthalene activity which was higher than the naphthalene activity of the Criterion (Shell) 324 catalyst (Figures 33 and 34).

5.3 Run 261 Catalyst Activity Analysis

Catalyst activity data were evaluated and analyzed based on Phase 2 and 3 trend data. Normally washed, "standard" level high-ash Illinois No. 6 coal with 11-12 wt % ash was processed with EXP-AO-60 1/16" catalyst during January 12 - March 18 (Operation Days, OD=1-51), and with Criterion 324 1/16" catalyst during April 13 - May 30 (OD=52-97). The interstage separator was in use for the entire run. A portion of V1082 vacuum bottoms stream was used for solids recycle in most of the run. During January 20 - March 2, the V1067 atmospheric bottoms were used for solids recycle, since V1082 Vacuum Vessel did not properly operate at coal feed rates higher than 425 MF lb/hr.

EXP-AO-60 1/16" catalyst activities during both batch and steady-state operating conditions with catalyst replacement were analyzed based on Phase 2 TSL resid yield by adjusting to a common organic rejection (COR) of 15 or 20 wt % MAF coal. Run 261 started on January 12 in the catalytic/catalytic mode of operation. Batch deactivation operation continued until February 1, when catalyst ages in both stages became equivalent to the equlibrium ages with catalyst replacement at 3 lb/ton MF coal. Catalyst replacement was 3 lb/ton MF coal in each stage during February 1-15 (OD=19-28); 1.5 lb/ton MF coal in each stage during March 13-18 (OD=46-51). Batch operation periods without catalyst replacement were January 12-31 (OD=1-18) and February 16 - March 12 (OD=29-45). Reaction temperatures studied for this portion of Run 261 with EXP-AO-60 catalyst were 760-790°F in the first stage and 810°F in the second stage. Coal feed rates were 400-550 MF lb/hr, and recycle resid and CI concentrations in the recycle

process solvent were 32-50 and 10-14 wt %, respectively. Initial catalyst charge was 240 lbs in each reactor. The catalyst charge was increased to 270 lbs in the second stage on January 14 and in the first stage on January 23.

Process performance results are compared in Figures 35 to 39. Cracking activity (resid conversion) is not the only function of the catalyst. Hydrogenation activity of the catalyst was not considered in these catalyst activity analyses. Trend data for the catalyst hydrogenation were discussed based on hydrogen contents of the recycle resid and distillate in Section 4.2.

During the initial startup and batch deactivation periods, TSL resid yield gradually increased from -1 to 5 wt % MAF coal with 20 wt % common organic rejection (Figure 35). The recycle resid concentration in the process solvent was below the target 40 wt %, continuously declining from 38 to 32 wt %. This decline indicated that the process operation severity studied for the initial startup appeared to be higher than that for resid balance operation. The recycle resid concentration started back to increase after January 31 and gradually increased to the target 40 wt % on February 24. The 1st & 2nd stage coal conversion did not vary significantly and was in the range of 92-93 wt % MAF coal, although the first stage coal conversion increased from 78 to 84-87 wt %, as the first stage reaction temperature was increased from 760 to 790°F on January 22 (Figure 37). Organic rejection in the ROSE-SR bottoms solids product was in the range of 20-21 wt % (Figure 38).

During March 4-8 (OD=37-41) period (batch deactivation operation without catalyst replacement) resid + UC conversion increased by 6-9 wt % due to the lower space velocity (Figures 35 and 36). Lowering the coal feed rate from 550 to 425 MF lb/hr improved coal conversion to 94 wt % MAF coal (Figure 37). As a result, organic rejection decreased to 16 wt % (Figure 38). The TSL excess resid yield gradually increased from 0 to 6 wt % during March 7-11 (OD=40-44) (Figure 35). Common organic rejection used for comparison was 15 wt %. The recycle resid concentration in the process solvent was increased from 40 to 50 wt % on March 12 (Figure 39). Catalyst replacement at 1.5 lb/ton MF coal started on March 13. Coal conversion remained high at 94 wt %; organic rejection stayed low at 15 wt %. The extrapolated trend analysis in Figure 35 showed a significant improvement in the resid + UC conversion, approximately 9 wt % due to the combined effect of resid recycle increase and catalyst replacement.

Criterion 1/16" catalyst activities at both steady-state operation with catalyst replacement and batch operation were analyzed based on Phase 2 TSL resid yield by adjusting to a common organic rejection (COR) of 15 or 20 wt % MAF coal. Batch deactivation operation continued during April 13-17 (OD=52-56), when catalyst ages in both stages became equivalent to the

equlibrium ages with catalyst replacement at 3 lb/ton MF coal. Catalyst replacement was 3 lb/ton MF coal in each stage during April 18-26 (OD=57-65); 2.25 lb/ton MF coal in each stage during May 8-30 (OD=77-97). Batch operation periods without catalyst replacement were April 13-17 (OD=52-56) and April 27 to May 7 (OD=66-76).

During Criterion catalyst operation, the reaction temperatures studied were 810°F in the first stage and 800-825°F in the second stage. Coal feed rate was 360-500 MF lb/hr, and recycle resid and CI concentrations in the recycle process solvent varied in the range of 35-53 and 11-17 wt %, respectively. Catalyst charge was 440 lbs in each reactor. The recycle resid concentration in the process solvent decreased to 36-43 wt % on April 16-17, below the target 50 wt %. This decline indicated that the process operation severity studied for this initial startup period appeared to be higher than that for the resid balance operation. During April 20-28 (OD=59-67), the inlet hydrogen partial pressure declined by 200-300 psia due to the differential pressure buildup in the preheater area. The coal concentration in the feed slurry was decreased to 30 wt % and the coal feed rate was decreased to 360 MF lb/hr to recover the system pressure back to normal operating conditions. At this lower coal feed rate, with 2.25 lb/ton MF coal catalyst replacement rate, the second stage reaction temperature could not be maintained at the target 825°F due to the lower process exothermic heat generated. Therefore, the second stage was operated at a lower reaction temperature, 800°F for the remainder of the run. The new resid injection system was tested during May 23-30 (OD=90-97). The test included a significant variation in feed slurry composition; 26-35 wt % coal concentration in feed slurry and 35-53 wt % resid and 8-17 wt % CI in the recycle process solvent, respectively.

Results are illustrated in Figures 40 to 44. During April 16-17 (OD=55-56), batch deactivation operation without catalyst replacement, resid + UC conversion was high with 2 wt % MAF coal TSL excess resid yield (Figures 40 and 41). Coal conversion was high at 94 wt % MAF coal (Figure 42); organic rejection was low at 15 wt % (Figure 43). Recycle resid and CI concentrations were in the range 36-43 and 12-13 wt % in the process solvent, respectively (Figure 44).

5.3.1 Overall Two-Stage Catalyst Activity

Overall two-stage catalyst activities were calculated for periods with EXP-AO-60 catalyst: January 18-21 (initial startup); periods 261A (transitional due to combined batch deactivation and catalyst replacement), 261B (steady-state), 261C (batch) and 261D (steady-state); and for periods with Criterion 324 catalyst, 261E (steady-sate), 261F (steady-state) and 261G (steady-state). TSL resid yields were compared for these periods along with Phase 3

data. Standard deviation of the resid yield for the steady-state operation periods was 2-3 wt % MAF coal, as shown below.

Period		Resid with OR = 20/15 wt % wt % MAF coal Phase 2	Phase 3
(w/ EXP-AO-60 catalyst)			
Jan. 18-20	(COR=20%)	-1.3 ± 0.6	-
261A (transitional)			
	(COR=20%)	5.4 ± 1.7	8.3 ± 2.3
	(COR=15%)	10.4 ± 1.7	13.3 ± 2.3
261B (steady)	(COR=20%)	1.3 ± 2.0	2.5 ± 2.2
	(COR=15%)	6.3 ± 2.0	7.5 ± 2.2
Feb. 23-25 (batch)			
	(COR=20%)	3.6 ± 1.4	-
	(COR=15%)	8.6 ± 1.4	-
261C (batch)	(COR=20%)	-1.5 ± 2.2	0.9 ± 3.2
	(COR=15%)	3.5 ± 2.2	5.9 ± 3.2
261D (steady)	(COR=20%)	-4.8 ± 1.3	-1.0 ± 2.5
	(COR=15%)	0.2 ± 1.3	4.0 ± 2.5
(w/ Criterion catalyst)			
261E (steady)	(COR=20%)	-3.1 ± 3.0	2.2 ± 3.3
	(COR=15%)	1.9 ± 3.0	7.2 ± 3.3
261F (steady)	(COR=20%)	-1.6 ± 1.8	5.9 ± 2.6
	(COR=15%)	3.4 ± 1.8	10.9 ± 2.6
261G (steady)	(COR=20%)	-2.4 ± 2.6	0.8 ± 2.9
	(COR=15%)	2.6 ± 2.6	5.8 ± 2.9

Overall catalyst activities can not be compared directly for these periods based on the resid yields, because several process conditions were changed affecting the performance. Key process variable changes are summarized below.

Period	Process condition change
(w/ EXP-AO-60 catalyst)	
Jan. 18-21	startup w/ subbituminous resid; batch deactivation; $T_{1st}/_{2nd}$=760/810°F; 410-420 lb MF coal/hr; 36 and 13 wt % resid and CI recycles; 240/270 lbs catalyst charge (1st/2nd).
261A	catalyst replacement at 3 lb/ton MF started in mid period (1st stage); T_{1st}=790°F; 545 lb MF coal/hr; 33 and 12 wt % resid and CI recycles; 270 lbs catalyst charge (1st stage).
261B	catalyst replacement in both stages; 550 lb MF coal/hr; 38 wt% resid recycle.
Feb. 23-25	batch deactivation; 38-40 wt % resid recycle.

261C	425 lb MF coal/hr.
261D	50 wt % resid recycle; catalyst replacement at 1.5 lb/ton MF.

(w/ Criterion catalyst)

261E	$T_{1st}/_{2nd}$=810/825°F; 500 lb MF coal/hr; catalyst replacement at 3 lb/ton MF.
261F	unstable w/ high MB closure error; $T_{1st}/_{2nd}$=810/800°F; 360 lb MF coal/hr; catalyst replacement at 2.25 lb/ton MF.
261G	transitional testing w/ resid injection; 26-31 wt % coal concentration; 41-50 wt % resid recycle; 9-12 wt % CI recycle.

Resid + UC conversion data for these periods are compared in Table D. Standard deviation of the overall two-stage conversion for the steady-state operation periods was 2-3 wt % MAF coal. Standard deviation for each stage conversion was higher at 1-7 wt % in periods 261E and F (Table D).

During the initial startup and batch deactivation period, January 14 - February 2 (OD=2-20, including 261A with catalyst replacement on February 1 in the first stage), the resid + UC conversion declined from 81 to 74 wt % MAF coal (Figure 36). As a result, the TSL excess resid yield increased from -1 to 5 wt % with 20 wt % common organic rejection (Figure 35). This decline was primarily due to catalyst deactivation (refer to Sections 5.3.1 and 5.3.2), coal feed rate increases from 400 to 550 MF lb/hr in several steps, and resid recycle decreaseS from 36 to 32 wt %. Increasing the reaction temperature from 760 to 790°F as well as the catalyst charge from 240 to 270 lbs in the first stage could have affected the performance.

In period 261B, at steady-state operation with catalyst replacement, the "all-distillate" product slate was achieved with 1-3 wt % MAF coal TSL resid yield, estimated at 20 wt % common organic rejection (Figure 35 and Table 23). The experimentally observed C4+ distillate yield was 64.5 wt % with 4.7 wt % resid yield and 17.9 wt % organic rejection. This was obtained at a high coal space velocity, 66.9 MF lb/hr/cuft-catalyst per stage, corresponding to 548 lb MF coal/hr. The projected C4+ distillate yield with resid extinction is 68.3 wt % with 18.0 wt % organic rejection at 61.4 MF lb/hr/cuft-catalyst per stage coal space velocity (refer to Section 5.4). The projected coal feed rate is 503 MF lb/hr. The projection line used for estimating the resid extinction conditions was developed primarily using Phase 2 data.

During batch deactivation period February 23-25, the TSL resid yield increased by 2-3 wt % MAF coal due to the decrease of resid + UC conversion.

Table D
Phase 2 and 3 Resid + UC Conversions

Period	Resid + UC conversion wt % MAF coal Phase 2	Phase 3
(w/ EXP-AO-60 catalyst)		
Jan. 18-20 (startup)		
1st stage	34.6 ± 2.4	-
2nd stage	46.6 ± 2.6	-
Overall	81.3 ± 0.6	-
261A (transitional)		
1st stage	45.3 ± 1.6	45.8 ± 2.8
2nd stage	29.4 ± 1.5	25.8 ± 2.1
Overall	74.6 ± 1.7	71.7 ± 2.3
261B (steady)		
1st stage	43.1 ± 1.4	43.8 ± 2.1
2nd stage	35.6 ± 2.5	33.7 ± 2.0
Overall	78.7 ± 2.0	77.5 ± 2.2
Feb. 23-25 (batch)		
1st stage	40.6 ± 4.8	-
2nd stage	35.9 ± 4.9	-
Overall	76.4 ± 1.4	-
261C (batch)		
1st stage	45.8 ± 2.2	46.8 ± 3.2
2nd stage	35.8 ± 1.3	32.3 ± 1.5
Overall	81.7 ± 2.2	79.1 ± 3.2
261D (steady)		
1st stage	50.7 ± 2.1	50.4 ± 1.7
2nd stage	34.1 ± 2.2	30.8 ± 2.4
Overall	84.8 ± 1.3	81.2 ± 2.6
(w/ Criterion catalyst)		
261E (steady)		
1st stage	44.1 ± 4.2	42.5 ± 3.9
2nd stage	39.0 ± 1.3	35.5 ± 3.2
Overall	83.1 ± 3.0	77.9 ± 3.4
261F (steady w/ catalyst replacement) (unstable)		
1st stage	41.9 ± 6.2	41.2 ± 6.9
2nd stage	39.7 ± 4.7	32.9 ± 4.5
Overall	81.7 ± 1.8	74.1 ± 2.6
261G (steady w/ catalyst replacement) (transitional)		
1st stage	48.0 ± 2.2	47.2 ± 2.2
2nd stage	34.4 ± 1.5	32.1 ± 1.7
Overall	82.4 ± 2.6	79.3 ± 2.9

In period 261C, with catalyst batch deactivation operation, lowering the coal feed rate from 550 to 425 MF lb/hr significantly improved the resid + UC conversion by 6-9 wt % MAF coal during March 4-8 due to the lower space velocity (Figures 35 and 36). The TSL excess resid yield gradually increased from 0 to 6 wt % during March 7-11 due to batch deactivation. Common organic rejection used for comparison was 15 wt %.

In period 261D, at steady-state operation with catalyst replacement, the "all-distillate" product slate was achieved with 3.7 wt % MAF coal TSL resid yield. The experimentally observed C4+ distillate yield was 65.6 wt % with 15.3 wt % organic rejection. This was obtained at a low catalyst replacement rate of 1.5 lb/ton MF coal per stage. The projected C4+ distillate yield with resid extinction is 68.9 wt % with 15 wt % organic rejection at 52.7 MF lb/hr/cuft-catalyst per stage coal space velocity (refer to Section 5.4). The projected coal feed rate is 431 MF lb/hr. The projection line used for estimating resid extinction conditions was developed primarily using Phase 2 data. A significant variation of 4 wt % in resid + UC conversion was observed between Phase 2 and 3 data (84.8 vs 81.2 wt %) in period 261D.

In period 261E, at steady-state operation with 3 lb/ton MF coal catalyst replacement in each stage, the "all-distillate" product slate was not achieved with 5.4 wt % MAF coal TSL resid yield (Figure 40 and Table 26). The experimentally observed C4+ distillate yield was 60.6 wt % with 16.7 wt % organic rejection. A significant variation of 5 wt % in resid + UC conversion was observed between Phase 2 and 3 data (83.1 vs 77.9 wt %). Phase 2 data showed lower resid yield and organic rejection, 1.2 and 15.7 wt %, respectively, indicating the "all-distillate" product slate in contrast with Phase 3 data.

In period 261F, at steady-state operation with 2.25 lb/ton MF coal catalyst replacement rate in each stage, the "all-distillate" product slate was not achieved with 10.3 wt % MAF coal TSL resid yield. The experimentally observed C4+ distillate yield was 58.4 wt % with 15.6 wt % organic rejection. Again, there was a significant variation of about 8 wt % observed between Phase 2 and 3 data in resid + UC conversion (81.7 vs 74.1 wt %). Phase 2 data showed lower resid yield and organic rejection, 3.5 and 14.9 wt %, respectively, indicating the "all-distillate" product slate in contrast with Phase 3 data. This high variation might be partly due to the high MB closure errors experienced in 261F.

In period 261G, testing the new resid injection system at steady-state operation with 2.25 lb/ton MF coal catalyst replacement rate in each stage, the "all-distillate" product slate was not achieved with 5.1 wt % MAF coal TSL resid yield. The experimentally observed C4+ distillate yield was 64.3 wt % with

15.6 wt % organic rejection. A variation of 3 wt % was observed between Phase 2 and 3 data in resid + UC conversion (82.4 vs 79.3 wt %). Phase 2 data showed lower resid yield and organic rejection, 2.6 and 15.0 wt %, respectively.

Trend analyses are summarized in Tables 23 to 28. Resid + UC conversion, TSL excess resid yield, coal conversion, and organic rejection data are compared.

5.3.2 First Stage Catalyst Activity

Catalyst activities were calculated assuming that the resid + UC conversion reaction follows a 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).

$$K = WHSV\ \epsilon/(1-\epsilon)$$

The dependence of the conversion rate constant on temperature (T) is described by the Arrhenius equation and the decrease in catalyst activity due to aging (t) is described by the following deactivation model:

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

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 is projected by using the residence time distribution function, RTD(t).

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

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

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

K: first stage resid + UC conversion activity, [WHSV]
A: frequency factor
E: apparent activation energy, Btu/lb-mole
R: ideal gas constant, Btu/lb-mole °R
T: reaction temperature, °R
α: deactivation coefficient, [1/t]
t: first stage catalyst age

EXP-AO-60 1/16" and Criterion 324 1/16" catalyst activities at both batch and steady-state operation with catalyst replacement in the first stage were analyzed based on Phase 2 resid + UC

conversion data. Results are illustrated in Figures 45 to 51.

The average ln K values for all the periods in Run 261 including January 18-20 and February 23-25 were calculated and are compared below along with Phase 3 data. Catalyst volume was used in calculating the conversion rate constant (K).

	Average ln K (1/hr)	
Period	Phase 2	Phase 3
(w/ EXP-AO-60 catalyst)		
Jan. 18-20 (startup)	3.65 ± 0.11	-
261A (transitional)	4.19 ± 0.06	4.19 ± 0.05
261B (steady)	4.05 ± 0.04	4.07 ± 0.06
Feb. 23-25	3.96 ± 0.14	-
261C (batch)	3.89 ± 0.07	3.91 ± 0.08
261D (steady)	3.88 ± 0.05	3.88 ± 0.04
(w/ Criterion catalyst)		
261E (steady)	3.83 ± 0.12	3.86 ± 0.10
261F (steady)	3.47 ± 0.17	3.40 ± 0.13
261G (steady)	3.65 ± 0.02	3.38 ± 0.07

Catalyst activity trend data in Figure 45 show that the activity in the first stage significantly increased during period 261A, compared to the initial startup period January 18-20. The average calculated rate constant value (K) increased by 72%. This increase was primarily due to the increase of reaction temperature from 760 to 790°F. The increase of catalyst charge from 240 to 270 lbs on January 23 might have affected the activity by increasing the ratio of catalytic to thermal volume in the reactor.

In period 261B, at steady-state operation with catalyst replacement, the average calculated rate constant value lowered by 13%, compared to 261A (transitional). This decrease was probably due to data scattering experienced with catalyst replacement and/or limited data points available for data analysis.

During the batch deactivation period February 23-25, the average calculated rate constant value slightly decreased, compared to 261B, due to catalyst deactivation; however, the value was significantly lower than that in 261A. This indicates that the deactivation might have been be higher than that observed during the initial batch deactivation period January 17-31.

In period 261C, at batch deactivation operation without catalyst replacement, the average calculated rate constant value slightly decreased, compared to the period Feb. 23-25, due to catalyst deactivation; however, the value was significantly lower than

those in periods 261A and 261B. This again indicates that the deactivation might be higher than that observed during the initial batch deactivation period January 17-31.

In period 261D, at steady-state operation with catalyst replacement, the average calculated rate constant value was similar to that in 261C with batch operation, indicating that the deactivation apparently might have stopped.

The activity trend data (Figure 45) for the batch operation period January 14-31 showed that the deactiavtion rate in the first stage was relatively low. However, as the run progressed, additional data obtained in periods 261C and 261D suggested that the deactivation rate could be higher, if all data were included in the analysis. Linear regression results (Figure 46) using all data at 790°F are summarized below.

(periods Jan. 26-31, Feb. 23 - Mar. 11 at T = 790°F)

$$\ln K = 4.57 - 0.000018\ t$$

($r^2 = 0.82$, standard deviation of slope, $\sigma(\alpha) = \pm 0.000002$)

Multiple linear regression results using data at 760 and 790°F are shown in Figure 47 and are summarized below. These results might have been affected by the lower catalyst charge (240 lbs) during operation at 760°F. Transitional data for January 14, 15, 16 and 23 were not included in the linear regression calculations.

(periods Jan. 17-31, Feb. 23 - Mar. 11 at T = 760 and 790°F)

$$\ln K = \ln A - (E/R)(1/T) - \alpha\ t$$
$$\ln K = 34.55 - 37500\ (1/T) - 0.000018\ t$$

(r^2=0.84; ln A=34.55; $E/R = 37500 \pm 3800$; α=0.000018 $\pm$ 0.000002)

Multiple linear regression results show a high apparent activation energy, 75,000 Btu/lb mole. This activation enregy value might have been affected by the lower catalyst charge during operation at 760°F.

Figure 48 compares Run 261 catalyst activity data. Period 261B has a lower calculated rate constant value below the batch trend line, while period 261D has a higher value. Both periods operated at steady-state with catalyst replacement. This discrepancy seems probably due to data scattering experienced with catalyst replacement and/or limited data points available for analysis.

In period 261E, with Criterion 324 catalyst at steady-state operation with 3 lb/ton MF coal catalyst replacement (Figure 49),

the average calculated rate constant value was lower, compared to 261B with EXP-AO-60, although 261E operated at a higher reaction temperature, 810 vs 790°F. 261E had a higher catalyst age based on catalyst volume, $3.55x\ 10^4$ vs $2.22x10^4$ lb MF coal/cuft-cat; a similar catalyst age based on catalyst weight, 657 vs 671 lb MF coal/lb-cat. When compared at the same catalyst age based on catalyst volume by extrapolation or interpolation of linear regression trend lines of batch aging in Figures 51 and 48, the first stage calculated rate constant values are similar (4.1-4.2 hr^{-1}) at a young catalyst age of $2.22x10^4$; however, 261E has a lower value (3.8 vs 4.0 hr^{-1}) at a higher catalyst age, $3.55x10^4$. Catalyst charge volume was the same for both catalysts, 8.1-8.2 cuft of slump catalyst in each reactor. However, catalyst charge weight was different due to different compacted bulk densities, 54 and 33 lb/cuft-slump catalyst with Criterion and EXP-AO-60 catalysts, respectively.

In periods 261F and G, with Criterion 324 at steady-state operation with 2.25 lb/ton MF coal catalyst replacement, the average calculated rate constant values were significantly lower than those in 261E due to operations with high catalyst ages (Figure 51); lower than that in 261D with EXP-AO-60 catalyst despite operation at a higher reaction temperature, 810 vs 790°F, and at a higher catalyst replacement rate, 2.25 vs 1.5 lb/ton (Figure 48). The deactivation rate was higher in 261E-G with Criterion 324 than in 261A-D with EXP-AO-60 catalyst (Figures 46 and 50), and therefore, the differences in the rate constant value became more significant at a higher catalyst age ($4.5x10^4$ lb MF coal/cu ft cat), 3.4 vs 3.9 hr^{-1}. Linear regression results using all batch aging data generated at 810°F with Criterion catalyst in 261E-G and at 790°F with EXP-AO-60 catalyst in 261A-D are summarized below.

(w/ Criterion 324 in 261E-G, as illustrated in Figure 50)

$$\ln K = 5.17 - 0.000037\ t, \quad r^2 = 0.74$$

(w/ EXP-AO-60 in 261A-D, as illustrated in Figure 46)

$$\ln K = 4.57 - 0.000018\ t, \quad r^2 = 0.82$$

Figure 52 shows that in 261A-D with EXP-AO-60 catalyst, both stages show very similar calculated rate constant values, even though operating reaction temperatures were different, 790°F in the first stage and 810°F in the second stage.

Figure 53 shows that in 261E-G with Criterion 324 catalyst, both stages again show very similar calculated rate constant values, even though operating reaction temperatures were different, 810°F in the first stage and 800-825°F in the second stage.

As illustrated in Figures 52 and 53, Run 261 with Illinois coal and Criterion 324 catalyst at 810/825°F (261E period) gave similar calculated rate constant values in both stages which are

also similar to those with EXP-AO-60 catalyst operated at 790/810°F, when compared at the same volume-basis catalyst age, 3.55×10^{-4} lb MF coal/cuft-cat; however, when compared at the same weight-basis catalyst age, 660-670 lb MF coal/lb-cat, 261E with Criterion at 810/825°F had significantly lower rate constant values than those in 261B with EXP-AO-60 catalyst at 790/810°F. In both periods, catalyst replacement rate was maintained at 3 lb/ton MF coal. Operation with Criterion 324 catalyst at 810/800°F in 261FG gave lower rate constant values than those in 261D with EXP-AO-60 catalyst at 790/810°F, primarily due to higher deactivation rates experienced with Criterion 324 catalyst.

In Figures 53 and 54, operation at 810°F with Criterion catalyst in 261E processing Illinois coal gave a first stage calculated rate constant value similar to that at 825°F with Shell 324 catalyst in 259H-J processing Pittsburgh coal, when compared at the same catalyst age; and gave higher second stage rate constant values at 800-825°F than that obtained at 790°F with Pittsburgh coal. Deactivation rates appeared to be similar with both catalysts, although operated at different reaction temperatures and with different coals.

Catalyst activity data in Runs 261, 259, 257 and 254 with three different coals (Illinois No. 6, Pittsburgh No. 8, and Ohio No. 6) and four different catalysts (EXP-AO-60, Shell 324, Amocat 1C and Shell 317) are compared in Figures 48, 51, 55-59.

Figure 57 compares Runs 261 and 257 processing Illinois coal with two different catalysts, EXP-AO-60 and Amocat 1C. The calculated rate constant value at 790°F for Run 261 with EXP-AO-60 is much higher than that for Amocat 1C in Run 257.

Figure 58 compares Runs 261, 257 and 259. The calculated rate constant value at 810 and 825°F for Run 259 processing Pittsburgh coal with Shell 324 catalyst is much lower than those at 790°F for Runs 261 and 257 processing Illinois coal with EXP-AO-60 and Amocat 1C catalysts, respectively.

Figure 59 compares Runs 261, 257, 259 and 254. The calculated rate constant value at 810°F for Run 254 processing Ohio coal with Shell 317 catalyst is higher than those at 810 and 825°F for Run 259 processing Pittsburgh coal with Shell 324 catalyst; lower than those at 790°F for Runs 261 and 257 processing Illinois coal with EXP-AO-60 and Amocat 1C catalysts, respectively.

Trend analyses are summarized in Tables 29 and 30.

5.3.3 Second Stage Catalyst Activity

EXP-AO-60 1/16" and Criterion 324 1/16" catalyst activities at

both batch and steady-state operation with catalyst replacement in the second stage were analyzed based on Phase 2 resid + UC conversion data. Results are illustrated in Figures 60-63. Second stage catalyst activity trend data are compared in a manner similar to that used for the first stage catalyst activity analysis. As discussed in Section 5.2.1, the low recovery of Criterion catalyst in the second stage at the end of run suggested a possible lower charge than assumed for the catalyst activity calculations, which might impart a significant negative bias on the calculated rate constants and deactivation rates. Catalyst breakage could have occurred due to several operational problems experienced such as plenum chamber wear and abrupt shutdown.

The average ln K values for periods 261A-G including January 18-20 and February 23-25 were calculated and are compared below along with Phase 3 data. Catalyst volume was used in calculation of the conversion rate constant (K).

	Average ln K (1/hr)	
Period	Phase 2	Phase 3
(w/ EXP-AO-60 catalyst)		
Jan.18-20 (startup)	4.18 ± 0.07	-
261A (transitional)	3.89 ± 0.10	3.75 ± 0.18
261B (steady)	4.07 ± 0.10	4.01 ± 0.08
Feb. 23-25 (batch)	4.03 ± 0.13	-
261C (batch)	3.84 ± 0.04	3.69 ± 0.06
261D (steady)	3.66 ± 0.10	3.52 ± 0.09
(w/ Criterion catalyst)		
261E (steady)	3.93 ± 0.05	3.86 ± 0.10
261F (steady)	3.62 ± 0.14	3.40 ± 0.13
261G (steady)	3.43 ± 0.07	3.38 ± 0.07

Catalyst activity trend data in Figure 45 show that the activity in the second stage continuously declined during the initial startup and batch deactivation periods. In period 261B, at steady-state operation with catalyst replacement, the average calculated rate constant value increased by 19-28%, compared to 261A (transitional). This increase might be due to data scattering experienced with catalyst replacement and/or limited data points available for data analysis. Note that in contrast the value decreased by 13% in the first stage (refer to Section 5.3.2).

During the batch deactivation period February 23-25, the average calculated rate constant value slightly decreased, compared to 251B, due to the catalyst deactivation. However, the value was significantly higher than that in 261A. This indicated that, in contrast with the first stage deactivation increase as discussed

in Section 5.3.2, the second stage deactivation might be lower than that observed during the initial batch deactivation period January 17-31.

In period 261C, with batch deactivation operation, the average calculated rate constant value significantly decreased, compared to the period February 23-25, due to catalyst deactivation; the value was similar to that in 261A. This indicates that the deactivation might be less than that observed during the initial batch deactivation period January 17-31.

In period 261D, at steady-state operation with catalyst replacement, the average calculated rate constant value was significantly lower than that in 261C at batch operation. This lower value seems probably due to data scattering experienced with catalyst replacement and/or limited data points available for analysis.

The activity trend data (Figure 45) for the batch operation period January 14 - February 2 showed that the deactiavtion rate in the second stage was relatively low and similar to that in the first stage. However, as the run progressed, additional data obtained in periods 261C and 261D suggested that the deactivation rate could be lower, if all the data were included in analysis. Linear regression results (Figure 60) using all data at 810°F are summarized below:

(periods Jan. 14 - Feb. 2, Feb. 23 - Mar. 11 at T = 790°F)

$$\ln K = 4.17 - 0.000008\ t$$
$$r^2 = 0.56$$

Figure 61 compares Run 261 catalyst activity data. In contrast with the first stage data (Figure 48), period 261B has a higher calculated rate constant value, located above the batch trend line, while period 261D has a lower value. Both periods operated at steady-state with catalyst replacement. This discrepancy seems probably due to data scattering experienced with catalyst replacement and/or limited data points available for analysis.

In period 261E, with Criterion 324 catalyst at steady-state operation with 3 lb/ton MF coal catalyst replacement (Figure 62), the average calculated rate constant value was lower compared to 261B with EXP-AO-60 catalyst, although period 261E was operated at a higher reaction temperature, 825 vs 810°F. Period 261E had a higher catalyst age based on catalyst volume, 3.62×10^4 vs 2.28×10^4 lb MF coal/cuft-cat; a similar catalyst age based on catalyst weight, 671 vs 690 lb MF coal/lb-cat. When 261E and B are compared at the same catalyst age based on catalyst volume by extrapolation or interpolation of linear regression trend lines of batch aging (Figures 63 and 61), the second stage calculated rate constant values in 261E are higher (4.3-4.1 hr^{-1}) at a young

catalyst age of 2.28×10^4 and a similar value (3.9- 4.0 hr^{-1}) at a higher catalyst age of 3.62×10^4 lb MF coal/cuft-cat. Catalyst charge volume was the same for both catalysts, 8.1-8.2 cuft of slump catalyst in each reactor. However, catalyst charge weight was different due to different compacted bulk densities, 54 and 33 lb/cuft-slump catalyst with Criterion 324 and EXP-AO-60 catalysts, respectively.

In periods 261F and G, with Criterion 324 at steady-state operation with 2.25 lb/ton MF coal catalyst replacement rate, the average calculated rate constant values were significantly lower than that in 261E due to operations with high catalyst ages (Figure 63); lower than that in 261D with EXP-AO-60 catalyst. Periods 261F and G operated at a lower reaction temperature, 800 vs 810°F, and at a higher catalyst replacement rate, 2.25 vs 1.5 lb/ton, compared to 261D (Figure 61). The deactivation rate was higher in 261E-G with Criterion 324 catalyst than in 261A-D with EXP-AO-60 catalyst, and therefore, the difference in the rate constant values become more significant at a higher catalyst age (4.5×10^4 lb MF coal/cuft cat), 3.5 vs 3.8 hr^{-1}. Linear regression results using all batch aging data generated at 825 and 800°F with Criterion 324 catalyst in 261E-G and at 810°F with EXP-AO-60 catalyst in 261A-D are summarized below:

(w/ Criterion 324 in 261E-G, as illustrated in Figure 62)

at 825°F, $\ln K = 5.09 - 0.000033\ t$, $r^2 = 0.62$
at 800°F, $\ln K = 5.24 - 0.000037\ t$, $r^2 = 0.58$

(w/ EXP-AO-60 in 261A-D, as illustrated in Figure 60)

at 810°F, $\ln K = 4.17 - 0.000008\ t$, $r^2 = 0.56$

The temperature dependence on the calculated rate constant value appeared to be not significant due to a change in reaction temperature from 825 to 800°F, as shown in Figure 62. This lack of temperature dependency might be due to the change made from the high to the low thermal severity and/or limited data points obtained in these periods. As shown in Figure 51, the first stage rate constant values for April 30 and May 1-5 are lower than the linear regression trend line. The interstage sampling problems could slightly shift the rate constant values in each stage in opposite directions. If the first stage values are lower than the trend line, then the second stage values will be higher than the trend line. The observed shift might also be partly contributed to the lack of temperature dependency in the second stage. Multiple linear regression analysis showed an unusually low apparent activation energy, 5200 Btu/lb-mole, and results are summarized below:

(w/ Criterion 324 in 261E-G at 825 and 800°F)

$$\ln K = \ln A - (E/R)(1/T) - \alpha t$$
$$\ln K = 7.14 - 2600\ (1/T) - 0.000033\ t$$

(r^2=0.87; ln A = 7.14; E/R = 2600 $\pm$ 5900; α=0.000033 $\pm$ 0.000009)

The above low calculated activation energy might be an indication of reaction control by hydrogenation equilibrium and/or significant regressive reactions at these operating temperatures.

As already discussed in Section 5.3.3, Figures 52 and 53 show that in periods 261A-D with EXP-AO-60 catalyst and in periods 261E-G with Criterion 324 catalyst, the calculated rate constant values for both stages were very similar, even though the operating reaction temperatures were different.

Catalyst activity data in Runs 261, 259, 257 and 254 with three different coals (Illinois No. 6, Pittsburgh No. 8, and Ohio No. 6) and four different catalysts (EXP-AO-60, Shell 324, Amocat 1C and Shell 317) are compared in Figures 61, 63, 64-68.

Figure 66 compares Runs 261 and 257 processing Illinois coal with two differnt catalysts, EXP-AO-60 and Amocat 1C. The calculated rate constant value at 810°F for Run 261 with EXP-AO-60 catalyst is higher than those at 760 and 790°F for Run 257 with Amocat 1C catalyst; is similar to that at 810°F for Run 257H with Amocat 1C catalyst. However, the deactivation rate at 810°F is higher than those at 760 and 790°F. The calculated rate constant values become similar for these temperatures at high catalyst ages, above 50,000 lb MF coal/cuft-cat.

Figure 67 compares Runs 261, 257 and 259. The calculated rate constant values at 760 and 790°F for Run 259 processing Pittsburgh coal with Shell 324 catalyst are much lower than those at 810°F for Runs 261 and 257H processing Illinois coal with EXP-AO-60 and Amocat 1C catalysts; are similar to those at 760 and 790°F for Run 257 processing Illinois coal with Amocat 1C catalyst.

Figure 68 compares Runs 261, 257, 259 and 254. The calculated rate constant values at 760 and 790°F for Run 254 processing Ohio coal with Shell 317 catalyst are lower than those at 760 and 790°F for Run 259 processing Pittsburgh coal with Shell 324 catalyst; are lower than that at 760°F for Run 257 processing Illinois coal with Amocat 1C catalyst. The value at 810°F for Run 254 processing Ohio coal with Shell 317 catalyst appears to be similar to that at 810°F for Run 261 processing Illinois coal with EXP-AO-60 catalyst, when the deactivation curves are extrapolated for comparison at similar catalyst ages.

Trend analyses are summarized in Tables 31 and 32.

5.4 Catalyst Requirement Comparisons

Phase 3 overall resid + UC conversion data for periods 261B, and D-G are plotted in Figures 69-74. Projected lines for these periods generated by assuming CSTR first order kinetics are also plotted. The variations in the resid content of the process solvent and the inlet hydorgen partial pressure (Table B) were not considered in the projections. The resid content was 38.3 wt % in 261B, while it was in the range 47.4-48.9 wt % during periods 261D-G. If adjusted for target resid levels, the projection lines could be slightly affected. Coal space velocity was selected as the x-coordinate variable to evaluate process responses in resid + UC conversion and catalyst requirement.

To better evaluate the catalyst requirement and coal throughput for Run 261 with EXP-AO-60 1/16" (bimodal) and Criterion 324 1/16" (unimodal) catalysts, coal feed rates were projected for resid extinction by using process performance responses estimated by assuming CSTR first order kinetics (Figures 70-74). Catalyst replacement rates for steady-state operation were calculated for comparison with actual replacement rates used at the plant. Results are listed in Table E. TSL process performance data measured at the plant are shown in Table F for comparison. The projected coal feed rate for resid extinction could be slightly affected due to the variations in the resid recycle level (Table B) and in the inlet hydrogen partial pressure (Table F).

In period 261B, at steady-state operation with catalyst replacement, the "all-distillate" product slate was achieved with 1-3 wt % MAF coal TSL resid yield, estimated at 20 wt % common organic rejection (Figure 35 and Table 23). The experimentally observed C4+ distillate yield was 64.5 wt % with 4.7 wt % resid yield and 17.9 wt % organic rejection (Table F). This was obtained at a high coal space velocity, 66.9 MF lb/hr/cuft-catalyst per stage, corresponding to 548 lb MF coal/hr. The projected C4+ distillate yield with resid extinction is 68.3 wt % with 18.0 wt % organic rejection at 61.4 MF lb/cuft-catalyst coal space velocity (Table E). The projected coal feed rate is 503 MF lb/hr.

In period 261D, at steady-state operation with catalyst replacement, the "all-distillate" product slate was achieved with 3.7 wt % MAF coal resid yield. The experimentally observed C4+ distillate yield was 65.6 wt % with 15.3 wt % organic rejection. This was obtained at a low catalyst replacement rate of 1.5 lb/ton MF coal per stage. The projected C4+ distillate yield with resid extinction is 68.9 wt % with 15.0 wt % organic rejection at 52.7 MF lb/hr/cuft-catalyst per stage coal space velocity. The projected coal feed rate is 431 MF lb/hr. The projection line used for estimating resid extinction was developed primarily using Phase 2 data. Daily plant operation was monitored by the resid balance through the TSL system. The

Table E

Projection of Coal Feed Rate for Resid Extinction and
Calculation of Steady-State Catalyst Replacement

Period	Projected (a) coal feed rate (MF lb/hr)	Projected (a) space velocity (MF lb/hr/cuft-cat)	Adjusted yield (wt % MAF coal) C4+ dist	Adjusted yield (wt % MAF coal) Common(b) organic rej.	Calculated steady-state catalyst replacement (lb/ton MF) 1st	2nd	Total
261A	-	-	-	-	2.6	2.4	5.0
261B	505	51.4	68.3	18.0	2.6	2.5	5.1
261C	400	40.8	69.6	15.0	1.5	1.5	3.0
261D	431	52.7	68.9	15.0	1.4	1.4	2.8
261E	460	56.5	66.1	15.0	2.7	2.6	5.3
261F	339	41.6	67.0	15.0	2.1	2.2	4.3
261G	323	39.7	68.9	15.0	2.1	2.2	4.3

Note: (a) Projected by using CSTR 1st order kinetics.
(b) Estimated.

Table F

TSL Process Performance Summary

Period	Reactor volume %	Recycle Resid (wt %)	Reaction temp.(F) 1st	2nd	Coal feed (MF lb/hr)	Yield (wt % MAF coal) C4+ dist	Organic rejection	Resid	Catalyst replacement (lb/ton MF) 1st	2nd	Total	Catalyst age (lb MF coal /lb cat) 1st	2nd	Coal space velocity (lb MF coal/ hr-cuft cat) 1st	2nd	Interstage separator	Inlet H2 pp (psia) 1st	2nd
261A(a)	100	33	790	810	543	59	21	7	3.0	0.0	3.0	680	722	66.4	66.4	Yes	2610	2480
261B	100	38	789	808	548	64	18	5	3.0	3.0	6.0	671	690	66.9	66.9	Yes	2610	2520
261C	100	38	790	809	420	65	16	5	0.0	0.0	0.0	1256	1269	51.3	51.3	Yes	2640	2480
261D	100	48	791	809	423	66	15	4	1.5	1.5	3.0	1375	1360	51.7	51.7	Yes	2660	2470
261E(b)	100	49	809	824	494	61	17	5	3.0	3.0	6.0	660	669	60.7	60.7	Yes	2540	2350
261F(c)	100	49	809	800	359	58	16	10	2.25	2.25	4.5	885	861	44.1	44.1	Yes	2620	2440
261G(d)	100	47	809	800	357	64	16	5	2.25	2.25	4.5	884	860	43.8	43.8	Yes	2640	2310

Note: (a) Transitional due to catalyst replacement started in the mid of 261A.
(b) Changed to Criterion 324 catalyst from EXP-AO-60.
(c) Unstable due to high MB closure errors.
(d) Transitional testing a new resid injection system.

resid yield of Phase 2 data is an experimentally measured value at the plant through the resid balance. There was a significant variation of 4 wt % observed between Phase 2 and 3 data in resid + UC conversion (84.8 vs 81.2 wt %) in period 261D.

Data analysis was performed by using the daily measured resid + UC conversion, and the rate constant values were based on the measured conversions. Phase 3 data are the adjusted yields by the elemental balance, and are good for estimating the distillate yield and selectivity.

In period 261E, at steady-state operation with 3 lb/ton MF coal catalyst replacement rate in each stage, the "all-distillate" product slate was not achieved with 5.4 wt % MAF coal resid yield. The experimentally observed C4+ distillate yield was 60.6 wt % with 16.7 wt % organic rejection. There was a significant variation of 5 wt % observed between Phase 2 and 3 data in resid + UC conversion (83.1 vs 77.9 wt %). Phase 2 data showed lower resid yield and organic rejection, 1.2 and 15.7 wt %, respectively, indicating a "all-distillate" product slate in contrast to Phase 3 data. The projected C4+ distillate yield with resid extinction is 66.1 wt % with 15.0 wt % organic rejection at 52.8 MF lb//hr/cuft-catalyst per stage coal space velocity. The projected coal feed rate is 432 MF lb/hr.

In period 261F, at steady-state operation with 2.25 lb/ton MF coal catalyst replacement rate in each stage, the "all-distillate" product slate was not achieved with 10.3 wt % MAF coal resid yield. The experimentally observed C4+ distillate yield was 58.4 wt % with 15.6 wt % organic rejection. Again, there was a significant variation of 8 wt % observed between Phase 2 and 3 data in resid + UC conversion (81.7 vs 74.1 wt %). Phase 2 data showed lower resid yield and organic rejection, 3.5 and 14.9 wt %, respectively, indicating a "all-distillate" product slate in contrast to Phase 3 data. This high variation might be partly due to the high MB closure errors experienced in 261F. The projected C4+ distillate yield with resid extinction is 67.0 wt % with 15.0 wt % organic rejection at 41.6 MF lb//hr/cuft-catalyst per stage coal space velocity. The projected coal feed rate is 339 MF lb/hr.

In period 261G, testing the new resid injection system at steady-state operation with 2.25 lb/ton MF coal catalyst replacement rate in each stage, the "all-distillate" product slate was nearly achieved with 5.1 wt % MAF coal resid yield. The experimentally observed C4+ distillate yield was 64.3 wt % with 15.6 wt % organic rejection. A variation of 3 wt % was observed between Phase 2 and 3 data in resid + UC conversion (82.4 vs 79.3 wt %). Phase 2 data showed lower resid yield and organic rejection, 2.6 and 15.0 wt %, respectively. The projected C4+ distillate yield with resid extinction is 68.9 wt % with 15.0 wt % organic rejection at 39.7 MF lb/hr/cuft-catalyst per stage coal space

velocity. The projected coal feed rate is 325 MF lb/hr.

5.4.1 Distillate Production Comparison

Figure 75 compares resid + UC conversion data for Runs 261BD, 259DEH, 257DEJ, 254BG and 251-IE with three different coals (Illinois No. 6, Pittsburgh No. 8, and Ohio No. 6) and five different catalysts (EXP-AO-60, Shell 324, Amocat 1C, Amocat 1A and Shell 317). Distillate selectivity for 259H was higher than the trend established for other runs processing high-ash coal (10-15 wt %). The distillate selectivity for 259H was 0.88, compared to 0.81-0.84 for 261BD and 257DE. The distillate selectivity for 259DE and 254G processing low-ash coal (5-7 wt %) was 0.85. Three projection lines generated for periods 257F, I and J by assuming CSTR first order kinetics were included for trend analysis in resid + UC conversion.

Several process operation variables were different for these runs: reactor severity sequence, half- or full-volume reactor operation, reaction temperatures, coal space velocity, recycle resid concentration, additional cleaning, etc. Reactor and catalyst volumes for these runs are summarized below:

Run	254	257D-H	257I-K	259B-J	261	251-I
reactor	full	full	half	full	full	full
volume (xfactor)	1.00	1.00	0.47	1.00	1.00	1.00
catalyst volume						
(slump) (ft^3)	8.11	8.29	3.14	8.15	8.18	8.20
(ratio)	0.98	1.00	0.38	0.98	0.99	0.99

Runs 254 and 257 was operated without interstage separation, which reduced the second stage inlet hydrogen partial pressure approximately by 200-300 psia. This lower hydrogen partial pressure without the interstage separator could lower resid + UC conversion in the second stage. The treatment of half-volume reactor results did not include the potential effects of the higher thermal-to-catalytic volume in the reactors and of the higher thermal severity in the preheater and the interstage heater. A commercial plant design for scaleup using half-volume data might be more complicated than with full-volume data.

Distillate production rates projected for the "all-distillate" product slate in Runs 257DE, 257H, 257J, 251-IE, 261B, 261D, 254B, 259H, 254G and 259DE are compared in Tables G-J, and illustrated in Figures 76-81. Common organic rejection was assumed to determine the resid + UC conversion for each period. Catalyst replacement rates for Runs 251-IE, 254B and 254G were estimated at steady-state operation from batch aging data. Results for Runs 254 and 257 could have been affected by the

Table G

Distillate Production Comparison (Illinois Coal - High Ash)

run	257DE	257H	257J(b)	251-1E	261B	261D	261E	261F	261G
coal	Illinois								
ash content (wt %)	11.3	11.3	11.2	11.3	12.1	11.8	11.6	11.8	11.5
catalyst	Amo 1C	Amo 1C	Amo 1C	Amo 1A/1C	EXP-AO-60	EXP-AO-60	---- Criterion 324 ----		
recycle resid (wt %)	50	40	50	40	38	48	49	49	47
inlet hydrogen partial pressure (psia)									
1st stage	2660	2710	2590	2470	2610	2660	2540	2620	2640
2nd stage	-	-	-	2460	2520	2470	2350	2440	2310
"all-distillate" yield (wt % MAF coal)									
C4+ distillate	69	70	67	70	68	69	66	67	69
organic rejection	18	18	18	15	18	15	15	15	15
distillate selectivity to conversion	0.84	0.85	0.82	0.82	0.83	0.81	0.78	0.79	0.81
projected coal space velocity (MF lb/hr/cuft-cat-each)	47	40	71	38	62	53	57	42	40
(relative)	(100)	(85)	(151)	(81)	(132)	(113)	(121)	(89)	(85)
catalyst replacement (lb/ton MF coal)									
1st stage	3	1.5	3	2(a)	3	1.5	3	2.25	2.25
2nd stage	1.5	3	1.5	1(a)	3	1.5	3	2.25	2.25
total	4.5	4.5	4.5	3	6	3	6	4.5	4.5
(relative)	(100)	(100)	(100)	(67)	(133)	(67)	(133)	(100)	(100)
reaction temperature (deg F)									
1st stage	790	760	810	810	790	790	810	810	810
2nd stage	760	810	760	760	810	810	825	800	800
(delta T total)	(base)	(+20)	(+20)	(+20)	(+50)	(+50)	(+85)	(+60)	(+60)
(delta T average)	(base)	(+10)	(+10)	(+10)	(+25)	(+25)	(+43)	(+30)	(+30)
projected distillate production (lb/hr/cuft-cat-each)	28.8	24.8	42.2	23.6	37.1	32.3	33.3	24.8	24.4
(relative)	(100)	(86)	(147)	(82)	(129)	(112)	(116)	(86)	(85)

(a) Estimated at steady-state operation from batch aging data.
(b) With half-volume reactors.
(c) Run 257 operated without interstage separation.

Table II

Distillate Production Comparison

(Ohio and Pittsburgh Coals)

run	254B	259H	254G	259DE
coal	Ohio	Pitt.	Ohio	Pitt.
ash content (wt %)	9.5	14.8	6.3	4.5
catalyst	S 317	S 324	S 317	S 324
recycle resid (wt %)	40	50	50	50
inlet hydrogen partial pressure (psia)				
1st stage	2620	2710	2730	2630
2nd stage	-	2500	-	2500
"all-distillate" yield (wt % MAF coal)				
C4+ distillate	70	70	78	78
organic rejection	16	20	8	9
distillate selectivity to conversion	0.81	0.88	0.85	0.85
projected coal space velocity (MF lb/hr/cuft-cat-each)	40	48	46	32
(relative)	(85)	(102)	(98)	(68)
catalyst replacement (lb/ton MF coal)				
1st stage	4(a)	3.6	3	4
2nd stage	4(a)	3.6	3(a)	4
total	8	7.2	6	8
(relative)	(178)	(160)	(133)	(178)
reaction temperature (deg F)				
1st stage	810	825	810	825
2nd stage	760	790	790	790
(delta T total)	(+20)	(+65)	(+50)	(+65)
(delta T average)	(+10)	(+33)	(+25)	(+33)
projected distillate production (lb/hr/cuft-cat-each)	25.3	28.6	33.3	23.8
(relative)	(88)	(99)	(116)	(83)

Table I

Effect of Resid Recycle on Distillate Production

run	257DE	257F	259DE	259F
coal	Illi.		Pitt.	
ash content (wt %)	11.3		4.5	
catalyst	Amo 1C		S 324	
recycle resid (wt %)	50	40	50	40
inlet hydrogen partial pressure (psia)				
1st stage	2660	2660	2630	2690
2nd stage	-	-	2500	2490
"all-distillate" yield (wt % MAF coal)				
C4+ distillate	69	68	78	75
organic rejection	18	18	9	9
distillate selectivity to conversion	0.84	0.83	0.85	0.81
projected coal space velocity (MF lb/hr/cuft-cat-each)	47	37	32	27
(relative)	(100)	(79)	(68)	(57)
catalyst replacement (lb/ton MF coal)				
1st stage	3	3	4	4
2nd stage	1.5	1.5	4	4
total	4.5	4.5	8	8
(relative)	(100)	(100)	(178)	(178)
reaction temperature (deg F)				
1st stage	790		825	
2nd stage	760		790	
(delta T total)	(base)		(+65)	
(delta T average)	(base)		(+33)	
projected distillate production (lb/hr/cuft-cat-each)	28.8	22.3	23.8	19.3
(relative)	(100)	(77)	(83)	(67)

(a) Estimated at steady-state operation from batch aging data.
(b) Comparison values relative to Run 257DE in the previous table with Illinois coal.
(c) Runs 254 and 257 operated without interstage separation.

Table J

Effect of Catalyst Replacement Rate on Distillate Production

run	261B	261D	254G	254C	254I	254J	259DE	259I	259J
coal	Illinois		Ohio				Pittsburgh		
ash content (wt %)	high (12.1)	(11.8)	low (6.3)	(6.5)	(5.9)	(5.6)	low (4.5)	(4.5)	(4.5)
catalyst	EXP-AO-60		Shell 317				Shell 324		
recycle rate (wt %)	38	48	50	50	50	50	50	50	50
inlet hydrogen partial pressure (psia)									
1st stage	2610	2660	2730	2710	2700	2740	2630	2680	2680
2nd stage	2520	2470	-	-	-	-	2500	2510	2510
"all-distillate" yield (wt % MAF coal)	68	69	78	75	77	75	78	78	75
C4+ distillate	18	15	8	8	8	8	9	9	9
organic rejection									
distillate selectivity to conversion	0.83	0.81	0.85	0.83	0.83	0.81	0.85	0.85	0.82
projected coal space velocity (MF lb/hr/cuft-cat-each)	62	53	46	41	37	43	32	26	22
(relative)	(132)	(113)	(98)	(87)	(79)	(91)	(68)	(55)	(47)
catalyst replacement (lb/ton MF coal)									
1st stage	3	1.5	3	3	3	3	4	3(a)	2.5(a)
2nd stage	3	1.5	3(a)	2.1(a)	1.5(a)	1.3(a)	4	3(a)	2.5(a)
total	6	3	6	5.1	4.5	4.3	8	6	5
(relative)	(133)	(67)	(133)	(113)	(100)	(96)	(178)	(133)	(111)
reaction temperature (deg F)									
1st stage	790		810				825		
2nd stage	810		790				790		
(delta T total)	(+50)		(+50)				(+65)		
(delta T average)	(+25)		(+25)				(+33)		
projected distillate production (lb/hr/cuft-cat-each)	37.1	32.3	33.3	28.8	26.8	30.4	23.8	19.4	15.8
(relative)	(129)	(112)	(116)	(100)	(93)	(106)	(83)	(67)	(55)

(a) Estimated at steady-state operation from batch aging data.
(b) Comparison values relative to Run 257DE in the previous table with Illinois coal.
(c) Runs 254 and 257 operated without interstage separation.

lower hydrogen partial pressure to the second stage without the interstage separation.

5.4.1.1 Illinois Coal - Low/High vs High/Low Severity Mode

The distillate production was significantly improved in period 261B processing Illinois coal with EXP-AO-60 catalyst (37.1 lb/hr cuft-catalyst per stage); a 29-57% increases compared to 257DE, 257H and 251-IE, as shown in Table G. However, the distillate production was 12% lower than in 257J. The improvements obtained in period 261B are described in detail in the following.

Period 261B with EXP-AO-60 catalyst in the low/high thermal severity mode improved distillate production by 29% compared to 257DE with Amocat 1C catalyst in the high/low severity. This was achieved at a lower resid recycle (40 vs 50 wt %), a higher second stage reaction temperature (810 vs 760°F) and a higher catalyst replacement rate in the second stage (3 vs 1.5 lb/ton MF coal).

Period 261B with EXP-AO-60 catalyst in the low/high severity improved distillate production by 57% compared to 251-IE with Amocat 1A/1C catalyst in the high/low severity. This was achieved at higher catalyst replacement rates in both stages (3 vs 2 in the first stage, 3 vs 1 lb/ton MF coal in the second stage), a lower first stage reaction temperature (790 vs 810°F) and a higher second stage reaction temperature (810 vs 760°F).

Period 261B with EXP-AO-60 catalyst improved distillate production by 50% compared to 257H with Amocat 1C catalyst. This was achieved at a higher first stage reaction temperature (790 vs 760°F) and a higher catalyst replacement rate in the first stage (3 vs 1.5 lb/ton MF coal). Both periods were operated in the low/high temperature severity mode.

Period 261B with EXP-AO-60 catalyst in the low/high severity had distillate production which was 12% lower compared to 257J with Amocat 1C catalyst in the high/low severity. This was observed with full-volume reactors operation in 261B compared to half-volume reactors operation in 257J. The half-volume reactor operation provided more isothermal reactor temperature profiles. In 261B, compared to 257J, resid recycle was lower (40 vs 50 wt %); first stage reaction temperature was lower (790 vs 810°F); second stage reaction temperature was higher (810 vs 760°F); catalyst replacement in the second stage was higher (3 vs 1.5 lb/ton MF coal). Period 257J with half-volume reactors had a 24% less catalytic-thermal volume ratio, and operated at higher thermal severity in the preheater and the interstage heater due to lower process heat generation. The treatment of half-volume reactor results did not include the potential effects of the higher thermal severities. The effect of reaction temperature

and recycle resid level on coal throughput appeared to be very significant and similar to those with full-volume reactors, as seen in periods 257I and K.

Period 261B improved distillate production by 12%, compared to period 261D. Both periods were operated with EXP-AO-60 catalyst in the low/high temperature severity mode. The improvement was achieved at a higher catalyst replacement rate (6 vs 3 lb/ton MF coal total) and a lower resid recycle (40 vs 50 wt %).

5.4.1.2 Ohio and Pittsburgh Coals - Effect of Coal Cleaning

With additional coal cleaning with heavy media, the distillate production significantly improved while processing Ohio with Shell 317 catalyst, a 16% improvement in period 254G when compared with period 257DE (Tables G and H). In contrast the distillate production was lower by 17% while processing Pittsburgh coal with Shell 324 catalyst in period 259DE when compared with period 257DE.

As shown in Table H, period 254G, processing additionally cleaned Ohio coal with Shell 317 catalyst, showed a 32% improvement in distillate production, compared to 254B with normally washed high-ash Ohio coal. Period 254G operated at a higher resid recycle (50 vs 40 wt %), a higher second stage reaction temperature (790 vs 760°F) and a lower catalyst replacement (6 vs 8 lb/ton MF coal total). It seemed that the additional coal cleaning had no detrimental effect on the distillate production. The additionally cleaned coal had a similar pyrite content compared to the normally washed coal (1.5 vs 1.9 wt %). The resid + UC conversion activity was similar for both coals, if the aging effect was considered in comparison (Ref. 5).

Processing both low- and high-ash Pittsburgh coals with Shell 324 catalyst in Run 259 showed that period 259DE with additional cleaned coal (with heavy media) lowered distillate production by 17%, compared to period 259H with normally washed high-ash coal. Operation conditions were similar for both periods except for the slightly higher catalyst replacement in 259DE to compensate for the lower ash content (8 vs 7.2 lb/ton MF coal total). It seemed that, in contrast to the combination of Ohio coal and Shell 317 catalyst, the additional coal cleaning had a significant effect on the distillate production. The additional coal cleaning reduced the pyrite content from 1.6 to 0.6 wt % and lowered the resid + UC conversion activity in Run 259 with Pittsburgh coal and Shell 324 catalyst.

With high-ash coals (Tables G and H), the combination of Illinois coal and EXP-AO-60 catalyst in period 261B showed a 30-47% higher distillate production than with the Ohio coal and Shell 317 catalyst (254B) and Pittsburgh coal and Shell 324 catalyst (259H)

combinations. Full-volume reactors were used for all these periods. Period 261B was in the low/high severity, while 254B and 259H were in the high/low severity. Compared to 254B and 259H, period 261B was operated at a lower catalyst replacement rate (6 vs 7-8 lb/ton MF coal total), a lower first stage reaction temperature (790 vs 810-825°F) and a higher second stage reaction temperature (810 vs 760-790°F). Recycle resid concentration was similar for 261B and 254B at 40 wt % in the process solvent; and different for 259H at 50 wt %.

With low-ash coals (Tables G and H), which were obtained by additional cleaning with heavy media, the combination of Ohio coal and Shell 317 catalyst (254G) showed a 40% higher distillate production compared to that with Pittsburgh coal and Shell 324 catalyst (259DE). However, period 254G had a 10% lower distillate production rate compared to period 261B with Illinois coal and EXP-AO-60 catalyst combination.

5.4.1.3 Effect of Recycle Resid Concentration

The effect of recycle resid concentration on distillate production was projected for both Runs 257D-F and 259D-F. Results are summarized in Table I and illustrated in Figure 80. The recycle resid level increase from 40 to 50 wt % improved the distillate production by 29% in period 257D-F and by 23% in 259D-F. Periods 257D-F was with Illinois coal and Amocat 1C catalyst and 259D-F was with Pittsburgh coal and Shell 324 catalyst. Both runs were operated in the high/low temperature mode.

5.4.1.4 Effect of Catalyst Replacement Rate

The effect of catalyst replacement rate on distillate production was projected for Runs 261BD, 254G-J, and 259DEIJ. Results are summarized in Table J and illustrated in Figure 81. If the effect of resid recycle increase from 40 to 50 wt % observed in 257D-F with Illinois coal and Amocat 1C catalyst is considered (a 29% increase in the distillate production), then just the effect of catalyst replacement rates in 261B and D becomes more significant, approximately a 50% increase in coal throughput by a 3 lb/ton MF coal total replacement rate increase in 261B compared to 261D with Illinois coal and EXP-AO-60 catalyst. The distillate production in 261B at higher resid recycle rate (50 wt %) is estimated at 48 lb/hr/cuft-cat per stage, higher than that in 257J (42.2).

The effect of catalyst replacement rate increase on coal throughput and distillate production was also significant in 254G-J with Ohio coal and Shell 317 catalyst, and in 259DEIJ with Pittsburgh coal and Shell 324 catalyst. Both coals were cleaned with heavy media to lower the ash content and operated at 50 wt %

resid recycle level. Periods 254G-J with a 1-1.5 lb/ton MF coal total catalyst replacement rate increase showed a 12-24% increase in the coal throughput and a 16-24% increase in the distillate production; 259DEIJ with a 2-3 lb/ton MF coal total catalyst replacement rate increase showed a 23-45% increase in the coal throughput and a 23-51% increase in the distillate production.

Data from periods 251-IE and 257DEJ in the high/low mode were also included for comparison in Figure 81. Recycle resid concentration was 40 wt % in period 251-IE and 50 wt % in periods 257DEJ. Operating conditions for these runs are listed in Table G. Period 257J had the highest distillate production, primarily due to half-volume reactors operation resulting in more isothermal temperature distribution and better mixing and a higher resid recycle level at 50 wt %. Periods 251-IE and 257DE with full-volume reactors operation had lower distillate production rates primarily due to lower resid recycle (40 wt %) operation in 251-IE and a lower first stage reaction temperature (790°F) operation in 257DE.

Period 259H with high-ash Pittsburgh coal and Shell 324 catalyst had a similar distillate production as in 257DE with high-ash Illinois coal and Amocat 1C catalyst (28-29 lb/hr/cuft-cat per stage); a 17% higher production compared to periods 259DE with low-ash Pittsburgh coal and Shell 324 catalyst (Figure 81). Period 259H operated at a 33°F higher reaction temperature (average two-stage) and a 60% higher total catalyst replacement rate compared to periods 257DE in the high/low mode. Recycle resid concentration for these periods were same at 50 wt %.

5.4.1.5 EXP-AO-60 Bimodal vs Criterion 324 Unimodal Catalyst

Distillate production rates, projected for the "all-distillate" product slate for periods 261B, D, E, F and G processing Illinois coal, are compared in Table G and illustrated in Figures 82 and 83. Run 257DE data, which were used as a baseline case, could have been affected by the lower hydrogen partial pressure in the second stage due to operation without the interstage separator. Periods 261BD was operated using EXP-AO-60 bimodal catalyst, while periods 261E-G were with Criterion 324 unimodal catalyst. The effect of catalyst replacement rate on the distillate production is illustrated in Figure 84 for periods 261B and D-G.

Several observations can be made from this comparison:

- Period 261E with Criterion 324 catalyst had a 10% lower distillate production rate compared to 261B with EXP-AO-60 catalyst. Period 261E was operated at a similar catalyst replacement rate (3 lb/ton MF coal in each stage), 18°F higher reaction temperature, 10 wt % higher recycle resid concentration (50 vs 40 wt %), and 100-200 psia lower inlet

hydrogen partial pressure (2540 vs 2610 psia in the first stage, 2350 vs 2520 psia in the second stage). Period 261E operated unstably due to the decline in the system pressure caused by slurry pumping problems. However, reaction temperature profiles were stable in 261E. If the effect of increased resid recycle from 40 to 50 wt % observed in 257DE with Illinois coal and Amocat 1C catalyst is considered in comparison (a 29% increase in the distillate production), then the distillate production in 261E with Criterion catalyst would be lower by 31%, compared to 261B with EXP-AO-60 catalyst.

- Period 261E with Criterion 324 catalyst had a 4% higher distillate production compared to period 261D with EXP-AO-60 catalyst. Period 261E was operated at a higher catalyst replacement rate (3 vs 1.5 lb/ton MF coal in each stage), a 18°F higher reaction temperature, and a similar recycle resid concentration (50 wt %).

- Periods 261F and G which were operated at a lower catalyst replacement rate (2.25 vs 3 lb/ton MF coal in each stage) and a 7°F lower reaction temperature, compared to 261E, lowered distillate production by 25-27%. These periods were operated using Criterion 324 catalyst at the same recycle resid concentration (50 wt %). The second stage reaction temperature could not be raised above 800°F in 261F and G due to less process heat generated. During period 261G, the new continuous resid injection system was tested.

- Periods 261F and G with Criterion 324 catalyst had a 24% lower distillate production, compared to 261D with EXP-AO-60 catalyst. Periods 261F and G were operated at a higher catalyst replacement rate (2.25 vs 1.5 lb/ton MF coal in each stage), a 5°F higher reaction temperature, and a similar recycle resid concentration (50 wt %).

- Periods 261E, F and G which were operated at higher reaction temperatures in the first stage (and higher in the second stage in 261E) had lower distillate selectivities to conversion (0.78-0.81) than periods 261B and D (0.81-0.83), due to higher C_1-C_3 gas selectivities to distillate, 12-13%, compared to 8-9%. Hydrogen efficiencies were lower, 9.7-11.2 lb C4+ distillate/lb H2 consumed.

5.5 Thermal History in TSL System

Thermal histories in Run 261 are illustrated in Figures 85-89 by using temperature profiles and approximate relative residence times through the TSL process system. Run 261 was operated with the full-volume reactors and interstage separator. Process times for slurry blend and feed tanks, and process solvent storage tank

were significantly reduced for illustration. In the following table, major vessels/reactors/tanks in the CC-ITSL process are associated with the location number for illustration in Figures 85-89.

Key Equipment	Location Number	Description
V101A	6-10	Slurry blend tank
V101B	10-14	Slurry feed tank
B1200	16-18	Preheater
R1235	19-34	1st stage reactor
V1258	36	Interstage separator
B1201	38-40	Interstage heater
R1236	42-57	2nd stage reactor
V1247	59	High press. separator
V1067	61	Atm. flash vessel
V1082	63	Vac. flash vessel
V131B	65-69	Process sol. storage

The outlet temperature of the B1201 interstage heater was significantly higher than the secod stage reaction temperature in Run 261 except for period 261B (Figure 85). The higher thermal severity operation of the interstage heater might have affected the performance in periods 261D-G, complicating the scale-up of data.

6. DISTILLATE PRODUCT QUALITY AND UNIT SOLVENTS

6.1 Product Quality

Physical and chemical properties of coal liquids were measured in order to determine their quality. The coal liquids resulted from converting Illinois No 6 coal to oil products in the CC-ITSL process. Six evaluations were performed when operating conditions were different. General product quality characteristics are discussed with possible effects of operating conditions on quality.

A substantial liquefaction data base has been compiled with Illinois No. 6 coal from the Burning Star No. 2 mine. In the past five years, it has served as the feed coal in about seven runs at Wilsonville. It was last used in Run 257 in the CC-ITSL mode with Amocat 1C catalyst. Usually 60-70 wt % MAF of the Illinois coal is converted to a distillate product in the CC-ITSL configuration. The results from Run 261 were thus typical with about 62 wt % of the coal being converted to oils.

The market place destination for liquefaction oils is not yet decided. They could end up as commercial transportation products such as jet fuel, gasoline, diesel fuel, or perhaps as additives to a commercial fuel. However, their present characteristics suggests that their quality must be improved in order for them to meet specifications for commercial fuels. Presently, they are highly aromatic which is consistent with a lower hydrogen content and their heteroatom content is too high for them to compete as a market ready fuel. Thus from the process level the next step is likely to be to a commercial hydroprocessing facility for upgrading.

The feed characteristics to a hydroprocessing facility are very important. Heavy ends and nitrogen can adversely effect catalyst activity. Lower hydrogen content is also an indication the feed will be more difficult to upgrade. Thus there is a continuous need to evaluate the liquefaction oils on the process level. The evaluations are indicative of what will be required for upgrading and will also help determine if process improvements or changes are affecting the overall quality of the products.

6.1.1 Methodology and Blending of Process Streams

Some typical laboratory test indicative of product quality were carried out on the oils from the respective evaluation periods. These tests included carbon, hydrogen, nitrogen, oxygen and sulfur. Simulated distillation by gas chromatography gave a breakdown of the product by weight percent and boiling point. A separate sample was used to distill the product oils into fractions of naphtha, middle distillate, and gas oil. This gave

the percentage of each fraction present and also yielded enough of each cut that they too could be characterized. By the ASTM D1160 procedure, the sample was carefully distilled to end point.

Presently, there is not a single process stream sample available that represents the net product at the liquefaction process level. Hence, representative stream samples have to be combined in order to come up with the correct product. At Wilsonville, there are two streams that can be combined to comprise the desired product and both are overhead products from distillation columns. One is the T105 atmospheric column, designated as sample V161, and the other is the T-02 vacuum column overhead sample designated as V182 oil. The V161 material is naphtha-like and contains about 13.0 wt % hydrogen. Naturally, it imparts a great deal of hydrogen to the total product. The vacuum column sample is heavier and boils in the 400 to 850°F range. It contains about 11.0 wt % hydrogen and influences heavily the end point and heteroatom content of the final product.

Figure 90 shows a partial schematic of the CC-ITSL process with distillate product separation. The sample locations are indicated and Table 33 gives the respective amounts of each stream that were combined to make the final product blends. The percentages relate to production rates calculated from material balances.

6.1.2 Product Quality Data and Results

Total Oil

Table 34 contains some typical product quality characteristics of the total oil product. For evaluation periods 261A-D, hydrogen content was above 11.0 wt % and averaged 11.51 wt %. In periods 261E and 261F, hydrogen dropped significantly and averaged 10.75 wt %. The heteroatom content of the products was lowest in the first four evaluation periods. Nitrogen averaged 2,481 ppm and showed a slight increase for period 261D. Sulfur did not change much in these four periods and averaged 0.026 wt %. Oxygen also changed little in these periods and direct analyses results averaged 0.74 wt %. In the last two evaluation periods, 261E and 261F, heteroatom content of the product oils increased. Nitrogen, for example, increased to over 4,000 ppm and averaged 4,307 ppm. Sulfur increased to 0.068 wt % and oxygen to 2.03 wt % by difference.

Distillations on the product oils gave end points and also the percentages of naphtha, middle distillate, and vacuum gas oil. This data is summarized in Table 34 also and shows the product to contain predominantly middle distillate, 350-650°F, boiling components. Naphtha constitutes about 18 wt % of the product and the gas oil fraction around 28 wt %. For periods 261A-D,the

product end point varied about twenty degrees between the periods and averaged 764°F. The end point increased significantly in periods 261E and 261F. For period 261E, it was 786°F and for 261F it reached 925°F. The 925°F end point was most unusual but was confirmed in a repeat of the distillation test.

Naphtha Cut (IBP-350°F)

This light boiling fraction showed some of the same trends as the total oil product. For example, in the first four periods, the hydrogen content of the naphtha cut was above 14.0 wt % and averaged 14.20 wt %. A significant decrease in hydrogen is observed in this cut for periods 261E and 261F wherein the hydrogen averaged 13.61 wt %. Again, the lowest heteroatom content is observed in the first four periods. Nitrogen averaged 259 ppm and sulfur was found to be 0.037 wt %. For periods 261E and 261F, nitrogen increased to 687 ppm and sulfur went up to 0.081 wt %. Oxygen, by difference, averaged 1.39 wt % which was a significant increase compared to 0.14 wt % by difference in the first four periods. The naphtha data are summarized in Table 35.

Middle Distillate Cut (350-450°F)

The properties of the lighter middle distillate fraction are given in Table 36 and the data show some trends are beginning to develop. Nitrogen has increased in all of these cuts compared to nitrogen in the naphtha cuts. But again, the nitrogen and other properties are clearly different by evaluation periods. The lowest nitrogen are seen in periods 261A-D where the average value was 1,350 ppm. In periods 261E and 261F, the nitrogen increased to 2,460 ppm. However, either value is much higher than the nitrogen in the naphtha which overall averaged 473 ppm. Hydrogen is also different for the respective periods with 12.34 wt % being a typical value in the first four periods. It decreases to 11.79 wt % in periods 261E and 261F. Sulfur was below 0.10 wt % for all of the cuts but again it was higher in the last two evaluation periods where it averaged 0.067 wt %. This was almost double the 0.037 wt % value typical in periods 261A-D.

Middle Distillate Cut (450-650°F)

This fraction has the greatest influence on the overall properties of the total oil product because it typically makes up thirty to forty weight percent of the distillate. Its characteristics are given in Table 37 and again the properties can be discussed in two segments. Overall, the best properties of this cut are clearly in the first four evaluation periods. Hydrogen, for example, averages 11.29 wt % but drops to 10.69 wt % for the last two periods. Nitrogen is typically 2,652 ppm in periods 261A-D but increases significantly to 4,652 ppm in periods 261E and 261F. The trend continues of increasing

nitrogen content by boiling fraction. Sulfur also shows a large difference with 0.025 wt % the average for periods 261A-D and 0.069 wt % the value for periods 261E and 261F.

Vacuum Gas Oil Cut (650°F-End Point)

The properties of the gas oil are shown in Table 38. This cut concludes some noticeable trends such as having the lowest hydrogen content but the highest nitrogen content. Approximately fifty percent of the nitrogen in the total oil product is due to this fraction. Although not as pronounced as in other fractions, the general qualities of the oils in the first four evaluation periods are better than the qualities of the oils in the last two periods. Nitrogen averages 3,796 ppm for the first four periods but increases to 5,261 ppm in periods 261E and 261F. Hydrogen did not change that much except for period 261E where it was 9.38 wt %. Hydrogen remained about the same in the other periods and averaged 10.06 wt %. Sulfur was also highest in the last two periods and averaged 0.075 wt % which was significantly higher than the average value of 0.018 wt % in periods 261A-D.

6.1.3 Summary

This section summarizes the physical and chemical properties of product oils obtained by liquefying Illinois No. 6 coal in the CC-ITSL process. The best product quality was achieved in the first four evaluations when the EXP-A0-60 catalyst was being used. This conclusion is based on the product oils having overall higher hydrogen content and lower heteroatoms sulfur, nitrogen and oxygen. Hydrogen averaged 11.51 wt % in the first four evaluations. Sulfur was the lowest heteroatom and averaged 0.028 wt %, oxygen was the highest heteroatom and averaged 0.74 wt % based on direct analysis. Nitrogen was intermediate and averaged 2,481 ppm. End points measured by distillation varied about twenty degrees and averaged 764°F.

Product quality declined in the second phase of Run 261 when the Criterion catalyst was being used. Hydrogen decreased to 10.75 wt % in the product and all heteroatoms increased. Nitrogen, for example, increased to 4,307 ppm, sulfur to 0.066 wt %, and oxygen, by difference, to 2.02 wt %. The product end points also increased. It reached 786°F in period 261E and then increased substantially to 925°F for the last period.

Product evaluations are performed independent of the process and therefore properties offer no clue as to how they became what they are. However, for this Run, it is clear that the best product quality is associated with the EXP-A0-60 catalyst. But in fairness to the Criterion catalyst there are no periods where direct comparisons of catalyst only can be made. In periods 261E and 261D, reactor temperatures and space velocity were also

changed. Both of these influence product quality. The closest comparison is perhaps in periods 261D and 261F with most of the conditions favoring better product quality in period 261F. However, clearly the 261D product had better qualities.

6.2 Unit Solvents

6.2.1 CCR Unit Recycle Solvent

The recycle process solvent was composed of solids-free resid recovered from the ROSE-SR^{SM} unit, solids recycle material either from the atmospheric flash bottoms (V1067) or the vacuum flash bottoms (V1082), and heavy gas oil (V1074) from the vacuuum distillation column (T102 bottoms stream). In the last few runs, the V1082 vacuum flash bottoms is used for the solids recycle material. However, at the start of the run on January 21, the solids recycle stream was changed to the V1067 atmospheric flash bottoms. This change was made because there was too much solvent in the ROSE-SR^{SM} feed and it was thought that the high flow rates through the V1082 may have caused the high solvent content. The solids recycle was changed back to V1082 material on March 2 during batch deactivation with EXP-AO-60 catalyst.

The three streams that make up the process solvent were combined to give a process solvent with the following composition from January 12 to March 12:

40 wt % Resid
12 wt % CI
48 wt % Heavy Gas Oil.

This was the target composition of the process solvent. At the start of the run, the target resid composition could not be achieved. There had been 30 to 33 wt % resid in the process solvent due to overconversion of resid. The coal feed rate was increased to raise the resid inventory and the amount of resid in the process solvent. The target resid concentration was achieved on February 21.

After March 12, the composition was changed to the following to obtain data at 50 wt % resid and to have more distillate production.

50 wt % Resid
12 wt % CI
38 wt % Heavy Gas Oil.

The coal concentration in the process solvent was 33 wt % until April 30. Due to increasing viscosity and slurry discharge pressures during operations with Criterion 324 catalyst, the coal concentration was lowered to 30 wt % in the CCR feed slurry. The

T102 vacuum distillation column overhead product was used for maintaining a constant flush on the catalyst withdrawal tubes throughout the run. This helped avoid plugging of the withdrawal tubes.

During the resid injection test, the target coal concentration was 30-33 wt % in the CCR feed slurry, however, due to circulation pump difficulties, the coal concentration could not be kept constant. To help the circulation in the coal slurry tank, more heavy gas oil material was added when needed, lowering the coal concentration in the feed to the CCR unit. See Section 7.1 for details on the resid injection test.

6.2.2 ROSE-SR℠ Unit Deashing Solvent

The ROSE-SR℠ unit uses a proprietary deashing solvent to process material containing ash. These deashing solvents are designated by numerical designations in order to prevent their disclosure. The deashing solvent was strengthened whenever necessary to optimize resid recovery while minimizing energy rejection to the ROSE-SR℠ bottoms product and maintaining efficient, stable deashing in the first stage.

7. Additional Test During Run 261

7.1 Resid Injection System

The objectives for the continuous resid injection system test were to test equipment operation and to determine operating ranges for slurry composition and evaluate process performance. Continuous resid injection has the potential to decrease the resid inventory by about one-third, and this may reduce resid degradation. Also, the time taken to reach steady state, once an operational condition change is made, can be minimized.

A schematic of the continuous resid injection system is shown in Figure 91. The P1090 pump was used to pump the deashed resid into the first stage reactor feed line and the P1091 pump was used to pump vacuum flashed bottoms, V1082, into the feed line. The coal slurry preparation system and reactor feed pump (P103) are shown in Figure 3. The test was conducted by adding coal to V1074 heavy gas oil as well as to a mixture of V1074 heavy gas oil and resid in V101A slurry tank. Centrifugal pumps circulate both slurries in V101A and V101B tanks. A positive displacement pump, P103, pumps from the V101B recirculating pump to the first stage reactor.

Prior to testing the resid injection equipment, most of the resid in the slurry tanks was displaced with V1074 heavy gas oil. The T102 cut point was lowered to provide enough solvent for making the first batch in V101A. The target composition was 53 wt % coal and 47 wt % V1074 which would be equivalent to 30% coal in the total mixture entering the reactor. The 53 wt % coal concentration caused repeatedly a loss of flow through the centrifugal circulation pumps. The feed mixture in V101B was added to V101A to establish circulation. As a result, the resulting mixture diluted to about 43 wt % coal.

Due to the problems with the first coal batch, the second V101A batch was set at 40 wt % coal in V1074 distillate. Batch compositions, pressure drop across B1200 and pertinent comments are given in Figures 92 and 93. The third batch was made with 50 wt % coal in V101A. The target for the fourth batch was 57 wt % coal since this would be the concentration necessary to give 33 wt % coal in the total slurry with a process solvent of 50 wt % resid and 12 wt % CI. However, this batch had to be diluted to 50 wt % coal. The centrifugal pumps will not pump much more than 50 wt % solids. Laboratory tests with a 57 wt % coal tended to cake up without good mixing.

Between May 24-27, the coal concentration in V101A was increased to 53 wt %. Some of these batches could be circulated while others required flushing of the centrifugal pumps and addition of V1074 to the mixture.

The ratio of hot solvent (V1074) to coal in the slurry tank was much less with the resid injection scheme. The temperature drop due to coal addition was more than when mixing all components. The E120 exchanger was put in service but operation was still not smooth. The deashed resid that was being pumped in with P1090 pump was added to the V1074 solvent in V131Bin the ratio 29 wt % resid and 71 wt % V1074. The solvent in V131B was transferred to V101A, and coal was then added to this mixture to give 48 wt % coal concentration. This would give an equivalent 33 wt % coal in the total slurry. After the first batch had started, a very high discharge pressure developed at the slurry pump and due to a high pressure drop across the B1200 preheater. The coal slurry was diluted to reduce the high discharge pressure. Subsequently, the coal slurry concentration was reduced to 45 wt %. This produced the best operability.

The P1090, P1091, and V1082 pumps performed reasonably well. The new pumps had to be reconditioned by the maintenance department prior to use.

Figures 94 and 95 give the viscosities of some of the V101A batches. These viscosities were in a reasonable range, however, the viscosities do not seem to be reliable for determining if the mixture can be pumped. The laboratory conducted time vs temperature tests to determine if the viscosity increased with time. With 50 wt % coal in V1074, only a small increase in viscosity was observed with time.

	Viscosity, cp					
	300°F shear, 1/sec			350°F shear, 1/sec		
Time , hr	16.8	8.4	3.4	16.8	8.4	3.4
2	348	390	495	239	272	345
4	322	380	525	228	266	345
6	343	400	545	230	312	425
8	346	408	535	244	294	455

The laboratory tried a similar study on a 57 wt % coal slurry; however, the stirrer only gave localized mixing and the outer perimeter did not mix. After about one-half hour the outer perimeter began to cake. After two hours, the whole slurry solidified.

In summary, under the continuous resid injection scheme, the coal concentration in slurries need to be lower unless a different type of pump can be found for pumping the coal+solvent slurry in V101A. Obtaining a 33 wt % coal slurry in a 50 wt % resid and 12 wt % CI process solvent will require a pump that can transfer thicker slurries (47-57 wt % coal in V101A). Figures 96 and 97 illustrate the composition limits for a typical bituminous coal and for a subbituminous coal operation. The subbituminous formula requires thicker V101A slurries since more resid is recycled with the higher CI content.

8. REFERENCES

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APPENDIX A

Nomenclature and Definitions

All-distillate: For experimental data, the resid yield is in the range of ±5 wt % MAF coal. Projected values are based on a zero resid yield.

Ash: Non-organic material obtained by muffle furnace burning at 800°C for 4 hours (adapted ASTM D-482).

Asphaltenes: A toluene-soluble and pentane-insoluble material which is non-distillable at 600°F and 0.1 mm Hg in the laboratory.

B102: T102 Vacuum Column Reboiler

B1200: First stage reactor preheater

B1201: Interstage heater

CC-ITSL: Close-Coupled Integrated Two-Stage Liquefaction

CCR: Close Coupled Reactor Unit

CI: Material which is insoluble in hot cresol. This material is composed of both ash and unconverted coal.

COR: Common Organic Rejection

DAS (Deashing Solvent): Deashing solvent in the ROSE-SR^{SM} unit. A solvent used to extract the resid from the feed to the ROSE-SR^{SM} unit.

Distillate solvent: A coal-derived distillate fraction which boils between 450°F and 600°F at 0.1 mm Hg in a laboratory batch distillation apparatus.

DITSL: Double Integrated Two-Stage Liquefaction

Dowtherm: Heat transfer fluid used in the process. Dowtherm is a trademark of the Dow Chemical Company.

DMDS: Dimethyl disulfide

Energy rejection: The heating value lost to the bottoms product as a percent of the feed coal heating value.

Feed Solvency Index: Laboratory analysis for fraction of ROSE-SR^{SM} feed soluble in actual deashing solvent compared to its solubility in a solvent standard.

HTR: Hydrotreater. Catalytic hydrogenation reactor unit.

IBP: Initial Boiling Point

ITSL: Integrated Two-Stage Liquefaction

K111: Vacuum System Precondenser. The vessel used as the middle distillate product condenser in T102 Vacuum Column.

LTR: Light Thermal Resid

MAF coal: Moisture- and Ash-Free coal

MB period: Material Balance period

MF coal: Moisture-Free coal

Middle distillate: A coal-derived distillate fraction which boils between 350 and 450°F at 760 mm Hg (GC and ASTM D-86).

Naphtha: A coal-derived distillate fraction which boils less than 350°F at 760 mm Hg (GC and ASTM D-86).

Naphthalene Activity: Catalyst activity test: Millimoles of hydrogen consumed per 100 g of 10% naphthalene solution.

OD: Operation Days

Oils: A pentane-soluble material which is non-distillable at 600 and 0.1 mm Hg in the laboratory.

Preasphaltenes: A cresol-soluble and toluene-insoluble material which is non-distillable at 600°F and 0.1 mm Hg in the laboratory.

Process solvent: The material mixed with coal in slurry preparation which is normally a blend of distillate solvent, resid and CI in specified concentrations.

P171: Pump used to transfer T102 overhead material to product storage. Also designates a sample point.

P1222 & P1236: Ebullation pumps on R1235 and R1236, respectively.

R1235 OTL: Interstage sample.

Resid: A cresol-soluble material which is non-distillable at 600°F and 0.1 mm Hg in the laboratory. Normally refers to deashed resid from the ROSE-SR^{SM} unit.

Resid + UC conversion: The fraction of the feed resid and UC that is converted to material that is distillable at 600°F and 0.1 mm Hg.

$$\text{Percent Conversion} = \frac{\text{Resid+UC in} - \text{Resid+UC out}}{\text{Resid+UC in}} \times 100$$

Resid recovery: The percent of ROSE-SR^{SM} feed resid that is recovered in the deashed resid and not lost to bottoms product.

RITSL: Reconfigured Integrated Two-Stage Liquefaction

ROSE-SR^{SM}: Residuum Oil Supercritical Extraction - Solids Rejection Unit. The unit used to remove ash and unconverted coal from the process.

Bottoms Product: A product of the Kerr-McGee ROSE-SR^{M} unit first stage separator that is rich in cresol insolubles (ash and UC) with lesser amounts of resid and solvent (previously known as ash concentrate).

Solvent (SOLV): A coal-derived distillate fraction which boils above 450°F and is distillable at 600°F at 0.1 mm Hg in a laboratory batch distillation apparatus.

TLU: Thermal Liquefaction Unit. Reactor operating in thermal mode.

TR: Thermal Resid

T102: Vacuum Distillation Column

T105: Atmospheric Distillation Column

Unconverted Coal (UC): Ash free organic material that is insoluble in hot cresol.

V101A: Coal Slurry Blend Tank. The vessel in which coal is added to process solvent to form a coal slurry for feed to the reactors.

V101B: Coal Slurry Feed Tank

V131B: Process Solvent Blend Tank. The vessel used to blend material from V1067 (or V1082) and V1074 with deashed resid from the ROSE-SR^{SM} unit to make process solvent for recycle to the coal slurry blend section of the plant.

V138: T102 Tray 3 Pot. The vessel used as the collection drum for the T102 Vacuum Column Tray 3 product draw.

V161: T105 Distillate Overhead Tank. The vessel used to collect

T105 overhead material for transfer to product storage.

V182: Oil/Water Separator. This vessel is used to remove water from the middle distillate product stream from the T102 Vacuum Column.

V1067: Bottoms Product Hold Tank. The vessel used to collect bottoms material from the atmospheric flash vessel.

V1072: Vacuum Overhead Drum. The vessel used to collect overhead material from the vacuum flash drum.

V1074: Distillate Bottoms Tank. The vessel used as a surge drum for the T102 Column bottoms material used in the recycle solvent.

V1078: Atmospheric Overhead Pot. The vessel used to measure overhead material from the atmospheric flash vessel.

V1079: Bottoms Measuring Pot. The vessel which measures the amount of material transferred from the atmospheric flash vessel to the V1067 Bottoms Product Hold Tank.

V1080: Sour Water Measuring Pot. The vessel ultimately used to measure the amount of water removed from the second stage reactor effluent via a series of flash drums and separators.

V1082: Vacuum Flash Vessel

V1247: The vessel used to separate the liquid and gas fractions of the second stage reactor effluent.

V1258B: Interstage Separator. The vessel used to separate the liquid and gas fractions of the first stage reactor effluent.

WHSV: Weight Hourly Space Velocity, lb/hr feed per lb catalyst. Space velocity with catalyst volume units are lb MF coal/hr-ft^3 settled catalyst.

APPENDIX B

MATERIAL BALANCE METHODOLOGY

B.1 Elemental Balancing of Yields

The total mass and elemental balances around each process unit are determined from the measured stream flow rates and laboratory analyses for the following elements in each stream.

- Carbon
- Hydrogen
- Nitrogen
- Sulfur
- Oxygen
- Ash

Elemental analyses (C, H, N, S, O) are not adjusted. Weighing factors, based on assumed flow rate errors for each stream and relative stream flow rate sizes, are applied to the process flow rates. The method minimizes the required adjustments to a stream flow rate to close the mass and elemental balance for each unit.

Since the streams are composed primarily of hydrogen and carbon, the balance is first developed based on these elements. Next, sulfur, nitrogen, an oxygen are balanced along with carbon and hydrogen, primarily by adjusting hydrogen sulfide, ammonia, and water rates. Ash is balanced by adjusting the ash analysis of output streams to equal the ash that entered with the coal. For ash recycle operations, the vacuum bottoms (V1082) rate to the ROSE-SR^{SM} unit is adjusted based on both coal ash and vacuum bottoms ash to ensure that all of the coal ash was purged in the ash concentrate (steady-state assumption). The adjusted stream flow rates between units are then corrected for inventory changes to achieve steady-state flow rate.

The CCR unit balance is developed with the above procedure. Since the ROSE-SR^{SM} has fewer streams than components, the measured stream flow rates and elemental analyses are used to calculate elemental errors. The errors are used as the basis for adjusting the compositions of the streams to close the balance.

The overall two-stage (TSL) yields are calculated by combining the balances of the CCR and the ROSE-SR^{SM} units.

B.2 Material Balance Methodology

Material balance data are routinely available for plant monitoring. In calculating the final yields, there are two intermediate stages of data. The "as-is" material balance data

(Phase 2) uses measured flow rates in calculating yields. Included with Phase 2 data are the mass balance closure errors. These data for Run 261 can be found in Table 3. When elemental analyses are complete on all input and output streams for a given day, elementally balanced yields (Phase 3) are calculated for that day. Phase 3 elementally balanced yield data are averaged for each set of stable operating conditions to obtain the final yield used throughout this report (Phase 4).

An evaluation of the material balance methodology was conducted to assess the usefulness of the different phases of material balance reports for plant monitoring, daily decision making, and technical accuracy of yields. Quantitative guidelines were desired to screen the data as it continued through the data phase system from Phase 2 to Phase 3 to Phase 4. For the evaluation, a statistical approach was used to assess the variance of the material balance data before and after elemental balancing.

In general, it was found that the Phase 2 and Phase 3 yields were in good agreement. Thus, the Phase 2 yields are considered to be adequate for plant monitoring and daily decision making. As an exception to this rule, only Phase 3 data is used to monitor plant performance with regard to production of C_4+ distillate. Daily comparison of Phase 2 and Phase 3 data proved to be useful in locating and correcting sources of balance errors.

For final yield characterization (Phase 4), Phase 3 yields are averaged for stable operating periods. These are the yields that have been used throughout this report.

B.3 Material Balance Data Selection Criteria

Statistical analysis was used to develop selection criteria for deciding which days to include in Phase 3 and Phase 4 data.

Phase 2-3 Selection Criteria are related to flow closure error, inventory changes, and plant stability. A total of ±10 wt % MAF flow closure error and ±15 wt % MAF inventory changes are allowed. Both are obtained by summing the contributions from individual units. In addition, days may be eliminated due to plant upsets or step changes in operating conditions.

Phase 3-4 Selection Criteria are related to elemental balance closure errors. Elemental closure error should be within the 95% confidence intervals for each individual unit and overall TSL system. Yields on days highlighted by excessive elemental closure errors are then compared with yields from the other elemental balance days in the Phase 4 period. If important yields on these days are outside an 80% confidence interval, the days are eliminated from the Phase 4 yields. The averages and standard deviations of the Phase 3-4 Selection Criteria are

reported in Table 39 for each representative operating period (Phase 4 Period).

In addition to the selection criteria, changes in Phase 2 or Phase 3 yields from one day to the next are used to assess TSL stability. Material balance data have defined quantitative guidelines, that indicate system stability, as the data flow through the data phase system from Phase 2 to Phase 3 to Phase 4.

APPENDIX C

Microautoclave Activity Test Descriptions

C.1 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 to which a mixing ball is added. The slurry is mixed and heated to a specific temperature (±5°F) within a three 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 percentage conversion which is referred to as "solvent quality".

Both kinetic and equilibrium tests can be performed. The equilibrium test is used to monitor solvent quality and is more commonly used. 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 these tests are listed below:

Test type	Temp., °F	Reaction Solvent-to-coal ratio	Time min.
Kinetic	750	8:1	10
Equilibrium	750	2:1	30

C.2 Catalyst Activity

Catalyst activity is determined in the Wilsonville laboratory as follows:

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 catalyst activity or naphthalene activity is the millimoles of hydrogen consumed per 100 g of 10% naphthalene solution.

Table 1

ILLINOIS NO. 6 FEED COAL ANALYSIS FOR RUN 261

Period	pre*	261A	261B	261C	261D	261E	261F	261G
Proximate (MF) %								
Volatile matter		37.1	37.4	37.5	37.4	37.2	37.5	37.1
Fixed carbon		50.8	50.7	51.2	51.1	51.1	50.9	50.8
Ash	11.8	12.1	11.9	11.3	11.5	11.7	11.6	12.1
Moisture	6.8	4.2	4.1	4.1	4.4	4.0	4.1	3.5
Ultimate (MF) %								
Carbon	71.1	70.3	71.9	71.4	71.3	70.7	71.1	70.5
Hydrogen	4.9	4.9	5.0	4.7	4.8	4.8	4.7	4.7
Nitrogen	1.4	1.4	1.4	1.4	1.4	1.4	1.3	1.4
Sulfur	3.5	3.7	3.7	3.8	3.8	3.8	3.5	3.3
Ash	10.8	12.2	11.9	11.3	11.5	11.7	11.6	12.2
Oxygen (diff.)	8.4	7.7	6.1	7.4	7.3	7.6	7.8	7.9
Dry heating value (Btu/lb)	12,490							
Sulfur forms, wt %								
Pyritic		0.99	1.38			1.46		
Sulfate		0.028	0.003			0.022		
Mineral analysis, (wt % of ash)								
Silica, SiO_2	46.9		43.2					
Ferric oxide, Fe_2O_3	19.1		16.1					
Alumina, Al_2O_3	17.7		22.4					
Lime, CaO	5.6		6.0					
Magnesia, MgO	1.1		0.9					
Potassium oxide, K_2O	0.9		1.8					
Sodium oxide, Na_2O	0.6		0.6					
Reactivity, % THF Conv.	81.0	77.0	80.5	80.9	-	76.1	70.3	70.9

*Sample from coal pile before grinding.

Table 2

RECYCLE PROCESS SOLVENT ANALYTICAL DATA

Operating Period	V131B					
	CI-Free Resid, wt %			Distillate, wt %		
	C	H	N	C	H	N
261A	88.5	7.6	1.0	87.8	9.9	0.3
261B	90.0	7.5	1.0	89.7	9.6	0.3
261C	89.7	6.8	1.3	89.9	9.4	0.4
261D	89.6	6.7	1.3	90.0	9.3	0.4
261E	89.2	6.0	1.4	89.7	8.5	0.5
261F	90.3	7.0	0.7	89.4	9.5	0.2
261G	90.7	6.8	0.9	89.8	9.4	0.3

Table 3

TSL Yields Before Elemental Balancing
(Phase 2 Data)

Yield, wt % MAF	Operating Period 261A	261B	261C	261D	261E	261F*	261G
H_2 consumed	-6.0	-6.2	-6.4	-6.3	-6.1	-6.8	-7.2
CO, CO_2	0.4	0.3	0.6	0.4	0.6	0.4	0.5
Sour Water	14.4	11.4	11.4	13.3	14.5	13.0	13.0
H_2S	1.1	1.9	1.6	1.8	1.8	1.4	1.7
C_1-C_3 gases	5.4	5.2	5.8	6.4	7.7	7.7	7.7
C_4+ distillate	59.2	63.8	63.3	64.7	61.3	56.5	66.7
C_4-C_6	2.7	2.5	2.6	2.4	2.9	3.3	3.2
IBP-350°F	13.5	13.3	14.9	19.3	14.9	13.9	14.7
350-450°F	7.3	7.3	8.1	9.2	8.9	8.0	8.3
450-750°F	33.0	36.4	35.6	32.3	35.7	40.0	35.9
750-850°F	9.7	11.5	11.4	9.1	11.3	17.3	9.3
850°F-EP	-7.1	-7.2	-9.3	-7.6	-12.3	-26.1	-4.7
Int. Accum.(Res. Free)	-0.9	-0.5	-0.2	-0.4	-1.4	0.0	-2.1
Resid	5.6	4.4	2.6	0.4	2.2	3.5	4.7
Bottoms Product(Ash-free)	20.7	17.3	16.0	15.1	15.9	18.8	15.0
Closure	0.3	2.5	5.4	4.6	3.5	9.5	-0.1
Resid+UC conv., wt % Feed							
1st stage	24.3	21.7	23.2	22.9	20.1	17.4	19.8
2nd stage	19.3	21.8	22.5	19.3	21.4	19.7	16.5
Coal conversion, wt % MAF							
1st stage	84.4	86.6	89.6	90.4	92.5	92.2	91.8
TSL	91.6	92.7	94.0	94.2	94.8	93.9	94.8

Notes: EXP-AO-60 Catalyst test: Periods 261A-D.
Criterion 324 Catalyst test: Periods 261E-F.
Resid Injection test: Period 261G.

For process conditions, see Table B.

*Unstable period.

Table 4

Close-Coupled Reactors Operating Data

Operating period	Coal feed rate MF lb/hr	First Stage		Second Stage	
		Avg temp.,(°F)	Bed exotherm (°F)	Avg temp.,(°F)	Bed exotherm (°F)
261A	543	790	43.1	810	10.3
261B	548	789	35.5	808	16.5
261C	420	790	31.0	809	11.4
261D	423	791	36.2	809	12.1
261E	494	809	28.3	824	12.6
261F	359	809	25.1	800	7.4
261G	357	809	25.3	800	7.5

Table 5

ROSE Unit Analytical Data - Run 261

Operating Period	261A	261B	261C	261D	261E	261F	261G
Feed Comp (Wt%)							
Ash	20.4	20.2	19.9	18.3	17.9	17.7	16.8
UC	13.0	11.0	9.7	8.5	8.2	8.3	8.1
Resid (a)	66.7	66.5	65.7	70.8	71.9	73.3	73.3
Solvent	3.6	2.3	4.7	2.5	2.0	0.7	1.8
Carbon	70.5	71.4	71.7	72.7	72.9	72.7	74.5
Hydrogen	5.3	5.5	5.4	5.4	4.8	4.9	5.2
Nitrogen	1.0	0.9	1.1	1.1	1.2	1.2	1.2
Sulfur	1.6	1.5	1.7	1.5	1.5	1.5	1.4
Oxygen	21.7	20.7	19.8	19.3	19.9	20.1	60.1
Preasphaltenes	7.0	4.3	6.9	5.8	14.2	8.2	8.7
Soft Point (deg F)	136.0	189.0	228.2	203.3	191.2	200.0	206.2
Fusion Pt (deg F)	151.4	221.3	201.9	247.8	227.4	259.4	239.5
Bottoms Prod Comp (Wt%)							
Ash (b)	40.1	44.6	44.1	46.0	45.4	47.1	45.6
UC (b)	25.6	25.8	22.8	24.8	23.9	25.6	27.1
Resid (a) (b)	33.9	27.9	32.0	28.8	30.3	27.0	26.7
Solvent (b)	0.4	1.7	1.1	0.7	0.3	0.2	0.5
DAS	1.4	0.8	1.1	1.0	1.2	1.3	1.2
Carbon	51.9	49.2	49.3	47.6	48.4	48.4	48.0
Hydrogen	3.2	3.1	3.1	3.0	2.7	2.7	2.8
Nitrogen	1.0	0.8	1.0	0.9	0.9	0.9	0.9
Sulfur	3.1	3.2	3.5	3.5	3.5	3.2	3.3
Oxygen (a)	40.2	43.7	43.1	44.8	44.5	44.8	45.1
Recycle Resid Comp (Wt%)							
Ash (b)	0.2	0.8	0.0	0.0	0.1	0.0	0.1
UC (b)	1.0	1.6	0.1	0.0	0.1	0.0	0.3
Resid (a) (b)	86.4	89.2	85.7	93.5	96.2	98.8	95.0
Solvent (b)	12.3	8.4	14.2	6.4	3.6	1.2	4.6
DAS	1.2	0.9	0.7	0.9	2.6	2.2	1.6
Carbon	89.1	89.8	90.3	89.9	89.5	90.5	90.3
Hydrogen	7.5	7.5	7.3	7.1	6.2	6.4	6.5
Nitrogen	1.0	0.9	1.1	1.2	1.3	1.4	1.4
Sulfur	0.4	0.2	0.3	0.2	0.4	0.4	0.4
Oxygen	2.4	1.6	1.0	1.7	2.8	1.2	1.4
Soft Point (deg F)	69.4	135.9	162.3	153.0	143.4	131.4	124.2
Fusion Pt (deg F)	109.4	165.9	191.5	191.5	182.4	218.2	177.1

Note: All periods in Catalytic/Catalytic Mode

(a) Calculated by difference
(b) "DAS-free" basis

Table 6

Summary of ROSE-SR Performance Parameters

Operating Period	DAS Type	Resid Recovery (wt %)	Energy Rejection (%)	Organic Rejection (wt % MAF)	Ash Conc Toluene Solubles (wt %)	FSI (a)	Ash Consistency
261A	2344	73.7	23.0	20.7	17.0	0.65	Extruded
261B	2394	78.0	19.8	17.3	15.7	0.72	Powder
261C	2314	77.0	18.5	16.0	13.3	0.67	Extruded
261D	2544	82.2	18.1	15.1	13.7	0.71	Extruded
261E	2654	82.5	17.7	15.9	13.5	0.69	Extruded
261F	2684	84.5	17.2	14.9	11.1	0.69	Extruded
261G	2664	88.2	17.8	15.0	10.6	0.68	Extruded

(a) Feed solvency index: Ratio of feed soluble in deashing solvent to feed soluble in cresol.

Table 7

T102 Operating Conditions Summary

	261A	261B	261C	261D	261E	261F	261G
T102 Top vapor temp, °F	504	502	505	505	502	497	496
T102 Tray 2 temp, °F	492	495	503	512	500	500	489
T102 Tray 3 temp, °F	505	507	513	526	506	524	507
T102 Top pack temp, °F	552	547	556	565	553	554	543
T102 Tray 8 temp, °F	575	575	579	588	575	580	574
T102 Bottom temp, °F	625	619	625	630	624	625	625
T102 TR3 reflux temp, °F	225	212	227	235	237	236	247
T102 Top pres, psia	1.8	1.3	1.7	1.4	1.4	1.1	1.9
T102 Reflux flow, lb/hr	641	579	637	630	634	631	635
B102 Inlet temp, °F	617	612	616	621	618	617	616
B102 Outlet temp, °F	685	679	679	683	670	667	668
B102 Process flow, GPM	20	21	22	22	25	25	25
GC Cut Point, °F	801±22	825±2	782±6	815±8	800±15	822±4	769±5
GC Equal wt % Overlap	7	9	6	8	9	9	9

Table 8

ANALYTICAL DATA FOR THE INTERSTAGE STREAM

Operating Period	Interstage Sample, wt %									
					CI-Free Residue			Distillate		
	Solv	Resid	UC	Ash	C	H	N	C	H	N
261A	47.5	34.9	8.0	9.6	86.70	7.00	1.30	88.80	9.80	0.44
261B	43.2	40.2	6.9	9.7	88.74	7.25	1.05	89.34	9.87	0.37
261C	43.7	40.6	5.6	10.1	88.73	7.07	1.32	89.40	9.76	0.40
261D	37.9	46.4	5.6	10.1	88.60	6.90	1.31	89.27	9.47	0.44
261E	35.2	49.7	5.1	10.0	87.96	6.09	1.39	88.1	8.92	0.56
261F	35.1	49.9	5.0	10.0	89.46	6.69	0.89	88.1	9.63	0.16
261G	38.4	47.3	5.1	9.2	89.99	6.35	1.17	-	-	-

Table 9

ANALYTICAL DATA OF STREAMS USED IN THE ELEMENTAL MATERIAL BALANCES

Operating Period	Coal, wt % MF					
	C	H	N	S	O(a)	Ash
261A	70.1	4.9	1.4	3.7	7.8	12.2
261B	70.7	4.9	1.4	3.7	7.4	12.1
261C	70.9	4.9	1.4	3.8	7.7	11.2
261D	70.2	4.9	1.4	3.7	8.3	11.5
261E	70.2	4.8	1.4	3.8	8.3	11.6
261F	69.8	4.7	1.3	3.2	9.4	11.6
261G	71.1	4.7	1.4	3.2	8.0	11.6

Operating Period	Recycle Material*, wt %					
	C	H	N	S	O(a)	Ash
261A(V1067)	78.9	7.3	0.7	0.8	1.0	11.3
261B(V1067)	79.2	7.4	0.7	0.8	0.2	11.7
261C(V1082)	71.8	5.4	1.1	1.7	0.6	19.5
261D(V1082)	72.8	5.4	1.1	1.5	0.8	18.4
261E(V1082)	72.6	4.8	1.2	1.5	1.6	18.1
261F(V1082)	74.2	5.0	1.2	1.5	0.7	17.5
261G(V1082)	74.5	5.2	1.2	1.5	0.9	16.7

*Similar to ROSE-SR℠ feed.

Operating Period	Flashed Distillate (V1078), wt %				
	C	H	N	S	O(a)
261A	87.2	11.7	0.2	0.03	0.9
261B	87.1	11.7	0.2	0.02	1.0
261C	87.8	11.6	0.2	0.04	0.3
261D	87.7	11.6	0.2	0.07	0.5
261E	87.6	10.9	0.5	0.05	1.0
261F	87.3	11.3	0.3	0.06	1.0
261G	87.4	11.2	0.3	0.05	1.1

Operating Period	T102 Bottoms (V1074) + Withdrawal Tube Flush, wt %				
	C	H	N	S	O(a)
261A	88.7	10.0	0.4	0.05	0.9
261B	89.3	9.8	0.4	0.05	0.5
261C	89.9	9.6	0.4	0.03	0.1
261D	90.0	9.4	0.4	0.02	0.2
261E	89.1	8.6	0.6	0.07	1.6
261F	90.2	8.7	0.5	0.08	0.5
261G	90.1	8.9	0.5	0.08	0.5

Table 9 (continued)

ANALYTICAL DATA OF STREAMS USED IN THE ELEMENTAL MATERIAL BALANCES

Operating Period	Vaccum Flash Overhead (V1072), wt %				
	C	H	N	S	O(a)
261A	89.5	9.8	0.3	0.07	0.4
261B	90.1	9.5	0.4	0.06	0.0
261C	89.8	9.7	0.4	0.03	0.1
261D	90.0	9.4	0.4	0.02	0.1
261E	90.1	8.3	0.5	0.06	1.1
261F	90.3	8.8	0.6	0.06	0.3
261G	90.0	8.9	0.5	0.05	0.4

Operating Period	ROSE-SR[SM] Resid, wt %					
	C	H	N	S	O(a)	Ash
261A	89.1	7.5	1.0	0.4	1.8	0.2
261B	89.8	7.5	0.9	0.2	0.6	1.0
261C	90.3	7.3	1.1	0.2	1.1	0.0
261D	89.9	7.1	1.2	0.2	1.5	0.1
261E	89.5	6.2	1.3	0.4	2.6	0.1
261F	90.5	6.6	1.4	0.5	1.2	0.0
261G	90.4	6.4	1.4	0.4	1.2	0.1

(a) Oxygen by difference.

Table 10

Run 261 Sour Water Analysis

	1st Stage	2nd Stage
Period	V105 Water (mg/liter)	V1080 water (mg/liter)
Kjeldahl nitrogen	9,730	5,502
Total organic carbon	2,504	1,052
Inorganic carbon	2.957	552
Phenols	1,600	845
Sulfide sulfur	14,865	11,573
Chloride	5.2	3.1

Table 11

Solvent Quality During Run 261

Date	Period	Catalyst	Solvent Quality, %
Jan 21		EXP-AO-60	77.9
Jan 28	261A		83.2
Feb 3			76.8
Feb 11	261B		85.5
Feb 25			79.5
Mar 4			83.5
Mar 11	261C		81.5
Mar 18	261D		81.0
Apr 16		Criterion 324	80.3
Apr 22	261E		77.2
Apr 29			75.8
May 6			76.3
May 20	261F		75.9

Table 12

Solvent Quality Comparison of Run 261 with Run 257

Period	Catalyst Type	Solvent Quality	Preasphaltene, wt % (ROSE-SR[SM] feed)
261A	EXP-AO-60 1/16"	83	7.0
261B	EXP-AO-60 1/16"	86	4.3
261C	EXP-AO-60 1/16"	81	8.8
261D	EXP-AO-60 1/16"	81	5.8
257A	Amocat 1C 1/12"	82-83	4.3
257B	Amocat 1C 1/12"	80-81	6.8
257C	Amocat 1C 1/12"	76-81	6.1
257D	Amocat 1C 1/12"	78-83	7.5
257E	Amocat 1C 1/12"	78-83	5.0
257F	Amocat 1C 1/12"	80-82	4.4
257G	Amocat 1C 1/12"	81-82	5.4
257H	Amocat 1C 1/12"	78-80(69-71)[(a)]	4.4
257I	Amocat 1C 1/12"	75	5.3
257J	Amocat 1C 1/12"	75-77	6.2
257K	Amocat 1C 1/12"	80-81	7.1

(a) Lower values with interstage samples collected after the first stage reaction.

Note: Solvent quality tests in Runs 257 and 261 were performed with different batches of standard Indiana V coal. A comparison between the different batches of the standard coal showed that the new batch of standard coal (used from Run 259 onwards) had about 3% higher solvent quality than the old batch (used before Run 259).

Table 13

Hydrogen Content Comparison of Run 261 with Run 257

Period	Interstage	Process Solvent	Process Solvent		Interstage Stream	
			Resid(CI-Free)	Distillate	Resid	Distillate
261A	7.67	7.88	7.35	9.69	7.02	9.78
261B	7.41	7.78	7.51	9.64	7.25	9.87
261C	7.45	7.56	6.78	9.39	7.06	9.76
261D	7.09	7.16	6.66	9.28	6.90	9.46
257A	7.65	7.93	8.10	9.95	7.67	10.01
257B	7.32	7.93	7.79	9.85	7.59	9.97
257C	7.44	7.77	7.66	9.67	7.32	9.79
257D	7.71	7.91	7.81	9.99	7.59	10.26
257E	7.65	7.96	7.80	9.91	7.61	10.05
257F	8.00	8.49	8.19	10.30	7.95	10.39
257G	7.94	8.44	8.20	10.26	7.80	10.38
257H	7.84	8.19	7.91	10.02	7.90	10.32
257I	8.00	8.45	7.89	10.54	7.29	10.25
257J	7.50	7.81	7.44	10.12	7.23	10.08
257K	7.78	8.15	7.67	10.50	7.33	10.37

Table 14

COMPARISON OF CATALYST PROPERTIES

	Criterion 324 (Shell 324)	Shell 317	Amocat 1C	Amocat 1C	EXP-AO-60
Run	256,259,261	253,254,256	257,259	251,252	261
Support	Unimodal	Bimodal	Bimodal	Bimodal	Bimodal
Size, inches Shape	1/16 Cylindrical	1/20 Trilobe	1/12 Cylindrical	1/16 Cylindrical	1/16 Cylindrical
Bulk density, lb/ft^3	54	37	35	42	33
Surface area (Sqm/g)	165	235	185	190	241
Metals, wt %					
Ni	2.70	2.65	2.35	2.33	2.5
Mo	13.20	11.58	10.60	10.42	10.7
Pore volume, cc/g	0.48	0.75	0.86	0.85	0.78
Catalyst charge, lb (Full-volume)	440	300	290*	340	270

*110 lb in half-volume tests.

Table 15

First Stage (R1235) Catalyst Age and Analytical Data

Date	Operating Day	Description	Catalyst Age R+CI	Catalyst Age MF Coal	As is S	As is Ash	As is Oil	THF Extracted C	THF Extracted H	THF Extracted N	THF Extracted S	THF Extracted Ash	THF Extracted Naph	14.0	18.0	25.0	-25.0
Run 261 Part 1 - EXP-AO-60 1/16" Catalyst						(wt %)				(wt %)				Screen Size			
JAN 1		Fresh EXP-AO-60				98.3								100.0	0.0	0.0	0.0
JAN 2		1st WD Batch #1			6.2	55.2	33.0	1.0	0.7	0.2	7.1	86.5		88.7	5.5	2.3	3.6
JAN 3		Batch #1 Comp.			5.3	56.0	36.7	1.4	0.7	0.2	7.2	86.9	198	98.1	1.2	0.2	0.5
JAN 3		Fresh Batch #2				98.8								100.0	0.0	0.0	0.0
JAN 4		1st WD Batch #2			6.1	55.1	36.5	1.1	0.6	0.0	7.1	85.4		88.9	6.3	2.4	2.4
JAN 5		Batch #2 Comp.			5.3	54.9	37.9	0.7	0.6	0.0	7.4	84.9	202	83.8	8.3	3.4	4.6
JAN 5		Fresh Batch #3				98.9								100.0	0.0	0.0	0.0
JAN 6		Fresh Batch #4				99.0								100.0	0.0	0.0	0.0
JAN 6		1st WD Batch #3			5.4	55.3	34.2	0.9	0.7	0.1	7.4	86.3		89.8	5.2	2.1	3.0
JAN 7		Batch #3 Comp.			5.5	56.2	37.7	0.7	0.6	0.1	6.9	85.2	178	90.0	5.8	1.9	2.3
JAN 8		1st WD Batch #4			7.4	55.2	36.2	1.1	0.6	0.0	7.0	83.9	164	93.7	3.3	1.2	1.8
JAN 23	10	1st WD Sample	725	359	6.3	54.1	35.2	11.6	1.0	0.3	6.6	79.4	93	86.2	1.7	1.8	10.3
JAN 29	16	2nd WD Sample	1226	626	5.1	56.4	31.3	11.9	0.9	0.3	6.4	79.9	34	94.9	1.7	1.0	2.4
FEB 1	19	3rd WD A/W	1296	665	4.9	56.3	31.2	14.4	0.9	0.3	6.5	77.0	53	99.0	0.8	0.1	0.1
FEB 6	21	4th WD A/W	1391	713	4.8	53.9	34.1	10.4	0.8	0.3	6.7	80.6	90	92.2	2.2	0.9	4.6
FEB 10	23	5th WD A/W	1269	648	4.4	52.9	30.9	12.[illegible]	1.0	0.3	6.3	80.2	59	98.2	0.5	0.3	1.0
FEB 12	25	6th WD A/W	1265	643	4.5	54.2	30.3	13.1	1.0	0.3	6.1	77.3	34	93.6	3.0	1.6	1.8
FEB 14	27	7th WD A/W	1263	639	4.4	53.4	31.4	12.2	0.9	0.3	6.2	78.0	62	96.4	1.6	0.8	1.2
FEB 21	30	8th WD Sample	1577	792	4.7	51.8	36.5	11.5	0.9	0.3	6.0	78.7	43	85.1	5.2	3.3	6.3
MAR 7	40	9th WD Sample	2384	1181	4.4	54.0	38.7	11.7	0.8	0.3	6.4	81.7	60	93.7	2.2	1.8	2.3
MAR 14	47	9th WD A/W	2772	1363	4.7	55.7	31.3	12.2	0.9	0.3	6.5	79.9	54	96.4	1.9	0.5	1.2
MAR 16	49	10th WD A/W	2780	1359	4.3	52.0	36.9	12.4	0.9	0.3	6.5	80.1		84.1	5.9	3.0	7.0
MAR 18	51	11th WD A/W	2788	1356	5.1	56.1	30.3	12.7	0.9	0.3	6.3	79.7		95.0	1.1	0.9	2.9
MAR 19	51	EOT WD #1 (btm)	2788	1356	4.5	55.8	31.0	13.4	1.0	0.3	6.4	80.7	49	98.0	1.3	0.2	0.5
		EOT WD #2			4.5	56.1	30.8	13.2	0.9	0.3	6.4	80.4		99.0	0.4	0.2	0.3
		EOT WD #3			4.5	55.9	31.1	13.5	0.9	0.3	6.4	80.2		97.9	1.2	0.2	0.7
		EOT WD #4			4.4	55.6	31.2	13.8	1.0	0.3	6.4	80.1	51	98.4	0.9	0.2	0.4
		EOT WD #5			4.4	55.8	32.9	13.3	1.0	0.3	6.4	79.4		92.1	5.9	0.7	1.3
		EOT WD #6 (top)			4.4	57.3	31.6	13.0	0.9	0.3	6.5	80.7		64.7	19.5	10.9	4.9
		Btm Head (a)			5.6	53.2	38.5	20.7	1.2	0.4	6.1	71.4		66.7	11.7	6.1	15.6
		Wt Avg Composite (b)												94.9	3.2	1.2	0.7
Run 261 Part 2 - Criterion 324 1/16" Catalyst																	
APR 7		1st WD Batch #1			6.8	69.7	19.5	2.2	0.6	0.3	5.7	84.8		98.4	1.5	0.0	0.1
APR 8		Comp. Batch #1			6.7	65.8	21.6	1.4	0.7	0.3	8.0	87.7	175	98.0	1.6	0.2	0.2
APR 10		1st WD Batch #2			5.8	70.1	22.6	1.1	0.7	0.1	6.8	88.4		98.4	1.6	0.0	0.0
APR 10		2nd WD Batch #2			7.1	70.7	21.4	1.3	0.7	0.1	8.2	87.5		98.1	1.6	0.1	0.2
APR 10		Comp. Batch #2			7.6	70.7	22.4					88.7	186	98.9	0.9	0.2	0.0
APR 19	58	1st WD A/W	1290	646	6.2	65.8	14.0	18.0	1.0	0.4	7.1	77.1	28	99.6	0.2	0.1	0.1
APR 21	60	2nd WD A/W	1300	645	5.9	63.0	17.3	16.5	1.0	0.4	6.9	75.7	34	98.7	0.7	0.1	0.5
APR 23	62	3rd WD A/W	1309	644	5.8	68.0	14.7	18.1	1.1	0.4	6.9	70.6	19	98.4	0.6	0.4	0.[illegible]
APR 25	64	4th WD A/W	1318	643	5.9	63.6	10.6	18.2	1.0	0.4	6.8	69.2	31	99.6	0.2	0.0	0.2
MAY 10	79	5th WD A/W	1876	872	6.3	59.9	12.1	19.4	1.0	0.3	7.2	64.6	21	95.6	1.5	0.5	2.4
MAY 15	82	6th WD A/W	1901	878	6.1	71.6	13.4	15.1	0.9	0.3	7.0	76.9	15	99.5	0.4	0.1	0.0
MAY 17	84	7th WD A/W	1910	877	6.1	62.4	14.4	20.0	1.0	0.4	7.0	73.4	22	99.1	0.7	0.0	0.2
MAY 19	86	8th WD A/W	1919	877	6.0	61.9	12.7	18.1	1.1	0.4	7.1	65.5	40	99.2	0.5	0.0	0.3
MAY 21	88	9th WD A/W	1928	876	5.9	61.1	17.3	17.6	1.0	0.4	7.1	76.1	27	98.3	0.6	0.1	1.0
MAY 23	90	10th WD A/W	1933	875	6.1	60.5	12.6	17.0	1.0	0.4	6.9	75.4	23	99.6	0.3	0.1	0.0
MAY 25	92	11th WD A/W	1942	874	6.2	64.3	12.5	17.1	1.0	0.4	7.0	72.3	28	99.6	0.3	0.0	0.1
MAY 27	94	12th WD A/W	1949	874	6.0	54.0	19.6	17.3	1.0	0.4	7.3	75.1	32	99.1	0.6	0.2	0.1
MAY 29	96	13th WD A/W	1951	873	6.0	60.0	16.1	17.0	0.9	0.4	7.1	71.3	38	99.4	0.4	0.0	0.2

Table 15 (Continued)

First Stage (R1235) Catalyst Age and Analytical Data

JUN	1	97	EOR WD #1	2016	898	5.9	66.4	14.4	17.8	0.9	0.4	6.8	70.4	34	95.0	2.1	0.8	2.1
			EOR WD #2			6.0	66.6	14.9	17.6	1.1	0.4	6.8	73.1		92.1	2.5	1.3	4.4
			Btm Head			6.1	58.5	21.5	20.3	1.2	0.5	6.7	73.8		88.3	1.7	1.3	8.7
			Wt Avg Composite (c)												93.2	2.2	1.1	3.6

(a) -25 mesh contained mostly mineral matter from coal. In wt avg composite, assumed -25 mesh portion zero and prorated other sizes.

(b) End-of-Test with EXP-AO-60 catalyst
EOT WD #1 = composite sample of withdrawals 1-5 w/ 62.5 lbs dry wt.
EOT WD #2 = composite sample of withdrawals 6-10 w/ 68.9 lbs dry wt.
EOT WD #3 = composite sample of withdrawals 11-15 w/ 61.0 lbs dry wt.
EOT WD #4 = composite sample of withdrawals 16-18 w/ 35.6 lbs dry wt.
EOT WD #5 = composite sample of withdrawals 19-20 w/ 28.8 lbs dry wt.
EOT WD #6 = composite sample of withdrawal 21 w/ 9.8 lbs dry wt.
Btm Head = composite sample of withdrawal 22 and the catalyst off the bottom head w/ 25.8 lbs dry wt, the fines being mineral matter.

(c) Criterion 324 catalyst withdrawal from the reactor at the End-of-Run.
EOR WD #1 = composite sample of withdrawals 1-4 w/ 201.8 lbs dry wt.
EOR WD #2 = composite sample of withdrawals 5-8 w/ 193.1 lbs dry wt.
Btm Head = sample of the catalyst off the bottom head w/ 28.4 lbs dry wt.

Table 16

Second Stage (R1236) Catalyst Age and Analytical Data

R1236 Catalyst

Date	Operating Day	Description	Catalyst Age R+CI	Catalyst Age MF Coal	As is S	As is Ash (wt %)	As is Oil	THF Extracted C	THF Extracted H	THF Extracted N (wt %)	THF Extracted S	THF Extracted Ash	THF Extracted Naph	Screen Size 14.0	Screen Size 18.0	Screen Size 25.0	Screen Size -25.0
Run 261 Part 1 - EXP-AO-60 1/16" Catalyst																	
						98.3								100.0	0.0	0.0	0.0
JAN 1		Fresh EXP-AO-60				55.1	41.6	1.9	0.8	0.2	7.1	85.2		98.7	1.0	0.0	0.3
JAN 2		1st WD Batch #1			5.7	55.3	37.5	0.8	0.7	0.2	7.3	86.0	158	96.4	2.4	0.6	0.6
JAN 3		Batch #1 Comp.			5.4									100.0	0.0	0.0	0.0
JAN 3		Fresh Batch #2				98.3								97.3	1.6	0.5	0.6
JAN 4		1st WD Batch #2			5.7	56.3	35.2	0.9	0.6	0.0	7.4	86.1		88.9	5.6	2.4	3.1
JAN 5		Batch #2 Comp.			5.7	56.3	35.3	0.6	0.7	0.1	8.0	86.2	201	100.0	0.0	0.0	0.0
JAN 5		Fresh Batch #3				98.4								100.0	0.0	0.0	0.0
JAN 6		Fresh Batch #4				99.1								94.6	3.1	0.6	1.7
JAN 6		1st WD Batch #3			5.8	56.1	40.6	0.8	0.6	0.1	7.2	85.9		94.0	3.5	1.3	1.2
JAN 7		Batch #3 Comp.			5.7	56.1	35.4	0.7	0.6	0.1	7.3	85.7	209	87.0	8.0	2.6	2.4
JAN 8		1st WD Batch #4			6.4	54.4	35.9	2.3	0.6	0.0	7.3	82.4	202	96.5	2.4	0.4	0.6
JAN 21	9	1st WD Batch Deact	519	307	4.5	49.6	38.9	8.9	1.0	0.5	6.3	79.8	126	99.0	0.0	0.1	0.9
JAN 28	15	2nd WD Batch Deact	910	577	4.2	53.1	32.7	14.0	1.0	0.3	6.4	76.5	57	99.6	0.4	0.0	0.0
FEB 2	20	3rd WD A/W	1287	825	4.4	54.4	31.3	14.2	0.8	0.2	6.4	77.4	90	99.0	0.6	0.2	0.2
FEB 9	22	4th WD A/W	1078	687	4.3	52.0	30.5	16.6	1.0	0.3	6.0	76.6	64	98.9	0.6	0.2	0.3
FEB 11	24	5th WD A/W	1063	677	4.2	53.0	29.4	16.4	1.1	0.3	5.9	74.5	60	98.9	0.7	0.3	0.2
FEB 13	26	6th WD A/W	1056	668		51.9	31.0	14.8	1.0	0.3	6.2	76.1	65	98.3	1.2	0.3	0.3
FEB 15	28	7th WD A/W	1051	660	4.3	51.7	32.4	13.8	1.0	0.3	6.0	75.7	78	96.7	1.0	0.3	0.0
FEB 22	31	8th WD A/W	1307	814	4.3	51.5	33.0	14.3	0.9	0.3	5.7	76.4	52	97.6	2.2	1.8	2.3
MAR 8	41	9th WD Batch Deact	2006	1232	4.2	51.3	34.2	15.2	0.9	0.3	6.1	77.6	56	97.8	1.6	0.2	0.4
MAR 13	46	10th WD A/W	2189	1341	4.4	53.8	28.6	16.5	1.0	0.3	6.2	77.0	39	98.2	1.0	0.4	0.4
MAR 15	48	11th WD A/W	2196	1378	4.3	55.0	29.7	15.5	0.9	0.3	6.1	77.0		98.1	1.3	0.3	0.3
MAR 17	50	12th WD A/W	2205	1337	4.3	53.2	32.0	15.2	1.0	0.3	6.5	77.1		99.1	0.5	0.2	0.2
MAR 19	51	EOT WD #1	2272	1373	4.0	52.9	33.9	17.3	1.1	0.3	6.4	75.6	37	99.2	0.5	0.1	0.3
		EOT WD #2			4.2	53.2	29.1	18.5	1.0	0.3	6.2	74.5		99.4	0.2	0.2	0.2
		EOT WD #3			4.2	52.4	28.9	19.1	1.0	0.3	6.2	74.2		97.6	1.7	0.2	0.5
		EOT WD #4			4.2	52.3	29.3	18.8	1.0	0.3	6.4	74.3	24	63.9	21.6	6.4	8.2
		EOT WD #5			4.5	53.8	30.2	17.9	1.0	0.3	6.3	75.1		11.0	3.7	27.5	57.8
		EOT WD #6			6.4	54.4	35.1	17.6	0.9	0.3	6.4	74.1		85.2	2.5	0.9	11.4
		Btm Head (a)			5.6	54.0	33.1	17.6	0.9	0.3	6.1	74.2		95.7	2.3	0.8	1.2
		Wt Avg Composite (b)															
Run 261 Part 2 - Criterion 324 1/16" Catalyst																	
APR 7		1st WD Batch #1			6.0	69.6	20.9	1.6	0.7	0.3	5.8	83.8		98.2	1.3	0.1	0.4
APR 8		2nd WD Batch #1			7.1	71.0	18.7	1.0	0.6	0.2	7.1	84.0		99.0	0.9	0.0	0.1
APR 8		Comp. Batch #1			6.9	71.1	20.3	1.1	0.7	0.2	8.1	87.3	203	98.1	1.2	0.3	0.4
APR 10		1st WD Batch #2			6.4	70.6	21.7	1.3	0.7	0.0	7.4	88.5		98.7	1.0	0.2	0.2
APR 10		Comp. Batch #2			7.4	71.0	21.3	1.0	0.7	0.0	7.8	88.7	187	99.5	0.4	0.1	0.1
APR 18	57	1st WD A/W	1496	663	6.3	64.5	10.7	16.8	1.0	0.3	7.0	75.4	28	99.7	0.3	0.0	0.0
APR 20	59	2nd WD A/W	1350	673	6.1	66.3	11.0	18.6	1.0	0.3	6.9	75.2	19	99.5	0.4	0.0	0.0
APR 22	61	3rd WD A/W	1442	660	5.9	64.2	11.9	17.4	1.0	0.4	6.9	76.0	11	99.5	0.3	0.1	0.1
APR 24	63	4th WD A/W	1421	658	6.1	62.3	11.5	17.9	1.0	0.4	6.7	74.4	31	99.6	0.0	0.2	0.2
APR 26	65	5th WD A/W	1400	655	5.9	63.2	11.4	19.2	1.0	0.4	6.9	75.6	38	99.7	0.2	0.1	0.0
MAY 8	77	6th WD A/W	1688	802	6.2	59.3	12.6	16.6	0.9	0.4	7.0	75.9	26	99.8	0.0	0.0	0.2
MAY 11	80	7th WD A/W	1762	841	6.0	59.9	11.8	17.4	0.9	0.4	7.1	73.5	30	99.7	0.2	0.1	0.0
MAY 16	83	8th WD A/W	1771	848	6.0	67.7	13.3	15.8	1.0	0.3	6.8	77.9	36	98.3	0.4	0.9	0.5
MAY 18	85	9th WD A/W	1772	849	5.9	61.6	13.6	17.5	1.0	0.4	6.7	77.5	37	99.3	0.3	0.1	0.4
MAY 20	87	10th WD A/W	1774	850	5.9	65.1	12.9	16.9	1.0	0.4	7.0	77.1	36	99.5	0.2	0.0	0.3
MAY 22	89	11th WD A/W	1775	850	5.9	63.0	13.9	16.4	1.0	0.4	6.8	76.2	33	99.2	0.4	0.2	0.2
MAY 24	91	12th WD A/W	1773	850	6.0	58.8	13.2	16.5	1.0	0.4	6.9	76.2	41	99.6	0.2	0.2	0.1

Table 16 (Continued)

Second Stage (R1236) Catalyst Age and Analytical Data

MAY 26	93	13th WD A/W	1771	850	5.8	65.2	13.5	15.1	1.0	0.4	7.1	75.4	34	99.7	0.0	0.1	0.1
MAY 28	95	14th WD A/W	1771	850	5.8	63.9	13.8	15.9	0.9	0.4	5.9	76.1	39	98.1	0.6	0.5	0.8
MAY 30	97	15th WD A/W	1767	851	5.7	61.2	11.1	15.6	0.9	0.4	6.8	74.8	35	99.4	0.3	0.1	0.2
MAY 31	98	EOR WD #1	1780	857	5.7	64.6	13.6	19.7	1.2	0.3	6.4	71.5	37	91.8	4.4	1.4	2.4
		EOR WD #2			5.7	62.3	13.1	18.6	1.1	0.4	6.8	71.9		98.3	0.8	0.3	0.5
		EOR WD #3			5.6	66.0	12.2	18.1	1.1	0.4	6.6	71.1		99.7	0.1	0.2	0.1
		Btm Head (c)			5.5	61.9	14.0	21.5	1.2	0.3	6.2	67.1		92.2	3.7	1.5	2.6
														95.3	2.4	0.9	0.3

(a) -25 mesh contained mostly mineral matter from coal. In the wt avg composite, the -25 mesh size was assumed zero and the other sizes pr

(b) End-of-Test run with EXP-AO-60 catalyst
EOT WD #1 = composite sample of withdrawals 1-4 w/ 63.0 lbs dry wt.
EOT WD #2 = composite sample of withdrawals 5-8 w/ 62.6 lbs dry wt.
EOT WD #3 = composite sample of withdrawals 9-11 w/ 47.0 lbs dry wt.
EOT WD #4 = composite sample of withdrawals 12-13 w/ 31.7 lbs dry wt.
EOT WD #5 = composite sample of withdrawal 14 w/ 17.2 lbs dry wt.
EOT WD #6 = composite sample of withdrawal 15 w/ 1.6 lbs dry wt.
Btm Head = catalyst off the bottom head w/ 15.8 lbs dry wt with most of the -25 mesh size being minerals.

(c) Criterion 324 catalyst withdrawn from the reactor at the End-of-Run
EOR WD #1 = composite sample of withdrawals 1-8 w/ 148.9 lbs dry wt.
EOR WD #2 = composite sample of withdrawal 9-10 w/ 47.6 lbs dry wt.
EOR WD #3 = composite sample of withdrawal 11-13 w/ 111.1 lbs dry wt.
Btm Head = catalyst off the bottom head w/ 31.0 lbs dry wt.

Table 17

First Stage (R1235) EXP-AO-60 Catalyst Screen Analysis (Composite from End-of-Test)

Comp. #	Weight (dry) lbs	Mesh Size (wt %) 14	18	25	-25
1 (Btm)	62.5	98.0	1.3	0.2	0.5
2	68.9	99.0	0.4	0.2	0.3
3	61.0	97.9	1.2	0.2	0.7
4	35.6	98.4	0.9	0.2	0.4
5	28.2	92.1	5.9	0.7	1.3
6	9.8	64.7	19.5	10.9	4.9
7 (Top)	25.8	78.9	13.8	7.2	0.0
	sum = 291.8				
Total Composite R1235 (Weighted Average)		94.9	3.2	1.2	0.7
257H (Amocat 1C)		85.1	7.4	3.9	3.6
259A (Amocat 1C)		84.4	15.6	(-14 mesh)	
259 (Shell 324)		93.1	4.1	2.0	0.8

Table 18

Second Stage (R1236) EXP-AO-60 Catalyst Screen Analysis (Composite from End-of-Test)

Comp. #	Weight (dry) lbs	Mesh Size (wt %) 14	18	25	-25
1 (Btm)	63.0	99.1	0.5	0.2	0.2
2	62.6	99.2	0.5	0.1	0.3
3	47.0	99.4	0.2	0.2	0.2
4	31.7	97.6	1.7	0.2	0.5
5	17.2	63.9	21.6	6.4	8.2
6	1.6	11.0	3.7	27.5	57.8
7 (Top)	15.8	96.2	2.8	1.0	0.0
	sum = 238.9				
Total Composite R1236 (Weighted Average)		95.7	2.3	0.8	1.2
257H (Amocat 1C)		85.7	7.0	4.6	2.7
259A (Amocat 1C)		86.6	13.4	(-14 mesh)	
259 (Shell 324)		94.5	3.3	0.9	1.3

Table 19

First Stage (R1235) Criterion 324 Screen Analysis (Composite From the End-of-Run)

Comp. #	Weight (dry) lbs	Mesh Size (wt %) 14	18	25	-25
EOR Comp. #1 (Btm)	201.8	95.0	2.1	0.8	2.1
EOR Comp. #2 (Top)	193.1	92.1	2.5	1.3	4.4
Btm Head	28.3	88.3	1.7	1.3	8.7
Wt. Avg Composite	423.2	93.2	2.2	1.1	3.6

Table 20

Second Stage (R1236) Criterion 324 Screen Analysis (Composite From the End-of-Run)

Comp. #	Weight (dry) lbs	Mesh Size (wt %) 14	18	25	-25
EOR Comp. #1 (Btm)	148.9	91.8	4.4	1.4	2.4
EOR Comp. #2 (Mid)	47.6	98.3	0.8	0.3	0.5
EOR Comp. #3 (Top)	111.1	99.7	0.1	0.2	0.1
Btm Head	31.0	92.2	3.7	1.5	2.6
Wt. Avg. Composite	338.5	95.3	2.4	0.9	0.3

Table 21

Catalyst Carbon Deposition and Naphthalene Activity
Comparison of Run 261 with Run 257

Run 261 (EXP-AO-60 1/16" Catalyst)

	First Stage		Second Stage	
Period	THF Extracted Carbon wt %	Naphthalene Activity	THF Extracted Carbon wt %	Naphthalene Activity
261A	13.2	44	14.2	74
261B	12.6	48	15.0	62
261C	11.7	60	15.2	56
261D	12.4	54	15.3	37
Run 257 (Amocat 1C 1/12" Catalyst)				
257A	11.6	56	11.2	57
257B	11.7	47	10.8	40
257C	11.8	42	10.6	38
257D	11.9	38	11.2	44
257E	11.6	52	10.5	69
257F	11.0	59	10.1	74
257G	10.7	48	9.5	62
257H	11.7	38	11.0	71
257I	12.3	59	10.4	58
257J	12.4	52	10.3	66
257K	12.1	44	10.9	72

Table 22

Metal Analysis of First and Second Stage EXP-AO-60 Catalyst
(Wt % Metal Oxides)

	First Stage					Second Stage				
Sample No.	16611	17079	17575	17818	18205	16610	17040	17608	17863	18257
Date, 1991	1/8	1/23	2/1	2/10	2/21	1/8	1/21	2/2	2/11	2/22
Period	Starter	Batch	261A	261B	Batch	Starter	Batch	261A	261B	Batch
Metal Oxides (Base: 100% Molybdenum Oxide)										
Aluminum	475.5	472.9	476.4	478.7	489.0	463.0	469.5	472.5	467.5	482.7
Calcium	0.7	4.9	2.8	7.4	5.9	0.6	1.3	2.7	3.3	3.6
Iron	0.7	4.2	1.4	8.1	7.4	0.6	4.5	1.3	1.3	1.4
Magnesium	0.7	2.8	0.7	2.9	2.2	0.6	0.6	0.7	0.7	0.7
Molybdenum	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
Nickel	21.2	20.8	20.7	21.3	22.0	20.8	20.8	20.8	20.5	21.6
Titanium	0.7	9.7	8.6	21.3	20.6	0.6	0.6	9.4	10.6	11.5
Silicon	13.2	13.9	12.8	14.7	15.4	13.6	13.6	13.4	13.9	13.7
Metal Oxides (Original Analysis)										
Aluminum	71.8	68.1	66.7	65.1	66.5	71.3	72.3	70.4	70.6	67.1
Calcium	0.1	0.7	0.4	1.0	0.8	0.1	0.2	0.4	0.5	0.5
Iron	0.1	0.6	0.2	1.1	1.0	0.1	0.7	0.2	0.2	0.2
Magnesium	0.1	0.4	0.1	0.4	0.3	0.1	0.1	0.1	0.1	0.1
Molybdenum	15.1	14.4	14.0	13.6	13.6	15.4	15.4	14.9	15.1	13.9
Nickel	3.2	3.0	2.9	2.9	3.0	3.2	3.2	3.1	3.1	3.0
Titanium	0.1	1.4	1.2	2.9	2.8	0.1	0.1	1.4	1.6	1.6
Silicon	2.0	2.0	1.8	2.0	2.1	2.1	2.1	2.0	2.1	1.9

Table 23

TSL System Responses in Resid + UC Conversion and Excess Resid
(Phase 2 Data)

TSL SYSTEM RESPONSES IN RESID+UC CONVERSION AND EXCESS RESID
Run 261 with Illinois No. 6 Coal, Jan. 12 - Mar. 18, 1991

unit: wt % MAF coal

		resid+UC conversion			Phase 2 Data (Measured)			
							Resid	
Date	Days	First	Second	Overall	TSL resid	Org.Rej.	w/ OR=15%	OR=20%
14-Jan-91	2	46.3	37.4	83.7	-9.6	25.9	1.3	-3.7
15-Jan-91	3	61.3	42.4	103.7	-20.4	16.7	-18.7	-23.7
16-Jan-91	4	47.7	43.2	90.9	-10.7	19.8	-5.9	-10.9
17-Jan-91	5	41.4	43.1	84.5	-0.5	16.0	0.5	-4.5
18-Jan-91	6	32.2	49.5	81.7	-1.6	19.9	3.3	-1.7
19-Jan-91	7	37.0	44.5	81.5	-1.3	19.8	3.5	-1.5
20-Jan-91	8	34.7	45.9	80.6	0.4	19.0	4.4	-0.6
23-Jan-91	10	53.2	38.0	91.2	-10.4	19.2	-6.2	-11.2
26-Jan-91	13	52.3	31.0	83.3	-3.8	20.4	1.6	-3.4
27-Jan-91	14	49.9	31.4	81.3	-7.2	19.9	-2.3	-7.3
28-Jan-91	15	46.9	31.3	78.2	0.1	21.8	6.9	1.9
29-Jan-91	16	47.1	27.2	74.3	5.5	20.2	10.7	5.7
30-Jan-91	17	46.6	30.1	76.7	1.6	21.7	8.3	3.3
31-Jan-91	18	43.5	30.9	74.4	4.4	21.2	10.6	5.6
01-Feb-91	19	45.4	30.3	75.7	3.9	20.4	9.3	4.3
02-Feb-91	20	43.7	28.4	72.1	8.1	19.8	12.9	7.9
09-Feb-91	22	48.1	37.6	85.7	-3.7	18.1	-0.6	-5.6
10-Feb-91	23	55.9	28.6	84.5	-2.6	18.1	0.5	-4.5
11-Feb-91	24	42.4	34.7	77.1	4.9	18.0	7.9	2.9
12-Feb-91	25	45.4	34.1	79.5	2.0	18.5	5.5	0.5
13-Feb-91	26	43.3	32.9	76.2	6.9	16.9	8.8	3.8
14-Feb-91	27	41.9	38.4	80.3	4.7	15.0	4.7	-0.3
15-Feb-91	28	42.5	38.1	80.6	1.4	18.0	4.4	-0.6
21-Feb-91	30	50.3	36.6	86.9	-7.2	20.3	-1.9	-6.9
22-Feb-91	31	43.6	36.8	80.4	-0.1	19.7	4.6	-0.4
23-Feb-91	32	39.7	35.1	74.8	5.8	19.4	10.2	5.2
24-Feb-91	33	45.7	31.4	77.1	3.0	19.9	7.9	2.9
25-Feb-91	34	36.3	41.1	77.4	3.0	19.6	7.6	2.6
03-Mar-91	36	52.4	38.0	90.4	-4.1	13.7	-5.4	-10.4
04-Mar-91	37	50.2	32.3	82.5	2.1	15.4	2.5	-2.5
05-Mar-91	38	43.3	39.1	82.4	2.9	14.7	2.6	-2.4
06-Mar-91	39	45.0	38.6	83.6	1.0	15.4	1.4	-3.6
07-Mar-91	40	49.3	35.2	84.5	-0.4	15.6	0.2	-4.8
08-Mar-91	41	46.7	36.6	83.3	1.8	15.9	2.7	-2.3
09-Mar-91	42	43.7	37.8	81.5	4.0	14.5	3.5	-1.5
10-Mar-91	43	44.5	34.9	79.4	3.9	16.6	5.5	0.5
11-Mar-91	44	44.9	34.7	79.6	3.0	17.4	5.4	0.4
12-Mar-91	45	53.4	28.4	81.8	-1.2	19.4	3.2	-1.8
13-Mar-91	46	52.4	33.6	86.0	-3.8	17.8	-1.0	-6.0
14-Mar-91	47	51.0	32.4	83.4	1.2	15.4	1.6	-3.4
15-Mar-91	48	49.4	36.7	86.1	-2.0	15.9	-1.1	-6.1
16-Mar-91	49	48.8	36.2	85.0	-0.3	15.3	0.0	-5.0
17-Mar-91	50	50.1	33.4	83.5	1.6	14.9	1.5	-3.5
18-Mar-91	51	54.1	31.9	86.0	-0.1	14.1	-1.0	-6.0

Table 23 (Continued)

TSL System Responses in Resid + UC Conversion and Excess Resid
(Phase 2 Data)

PERIOD : Jan. 18-20							
Average	34.6	46.6	81.3	-0.8	19.6	3.7	-1.3
Stan. Dev. (σ_n)	2.0	2.1	0.5	0.9	0.4	0.5	0.5
Stan. Dev. (σ_{n-1})	2.4	2.6	0.6	1.1	0.5	0.6	0.6
PERIOD 261A: Jan. 29 - Feb. 2							
Average	45.3	29.4	74.6	4.7	20.7	10.4	5.4
Stan. Dev. (σ_n)	1.5	1.4	1.5	2.1	0.7	1.5	1.5
Stan. Dev. (σ_{n-1})	1.6	1.5	1.7	2.4	0.8	1.7	1.7
PERIOD 261B: Feb. 11-15							
Average	43.1	35.6	78.7	4.0	17.3	6.3	1.3
Stan. Dev. (σ_n)	1.2	2.2	1.8	2.0	1.3	1.8	1.8
Stan. Dev. (σ_{n-1})	1.4	2.5	2.0	2.3	1.4	2.0	2.0
PERIOD : Feb. 23-25							
Average	40.6	35.9	76.4	3.9	19.6	8.6	3.6
Stan. Dev. (σ_n)	3.9	4.0	1.2	1.3	0.2	1.2	1.2
Stan. Dev. (σ_{n-1})	4.8	4.9	1.4	1.6	0.3	1.4	1.4
PERIOD : Mar. 4-8							
Average	46.9	36.4	83.3	1.5	15.4	1.9	-3.1
Stan. Dev. (σ_n)	2.6	2.5	0.8	1.1	0.4	1.0	1.0
Stan. Dev. (σ_{n-1})	2.9	2.8	0.9	1.3	0.4	1.1	1.1
PERIOD 261C: Mar. 7-11							
Average	45.8	35.8	81.7	2.5	16.0	3.5	-1.5
Stan. Dev. (σ_n)	2.0	1.2	2.0	1.6	1.0	2.0	2.0
Stan. Dev. (σ_{n-1})	2.2	1.3	2.2	1.8	1.1	2.2	2.2
PERIOD 261D : Mar. 14-18							
Average	50.7	34.1	84.8	0.1	15.1	0.2	-4.8
Stan. Dev. (σ_n)	1.9	2.0	1.2	1.3	0.6	1.2	1.2
Stan. Dev. (σ_{n-1})	2.1	2.2	1.3	1.4	0.7	1.3	1.3

Table 24
Coal Conversion (Phase 2 Data)

RUN 261 COAL CONVERSION
Illinois No. 6 Coal, Jan. 12 - Mar. 18, 1991

unit: wt % MAF coal

Date	Days	First	Second
14-Jan-91	2	83.0	92.7
15-Jan-91	3	77.3	93.5
16-Jan-91	4	76.6	90.9
17-Jan-91	5	77.3	91.8
18-Jan-91	6	78.4	93.3
19-Jan-91	7	79.6	93.2
20-Jan-91	8	76.3	92.2
23-Jan-91	10	83.9	93.9
26-Jan-91	13	89.6	94.0
27-Jan-91	14	86.1	93.4
28-Jan-91	15	87.0	91.6
29-Jan-91	16	82.9	89.9
30-Jan-91	17	82.8	90.5
31-Jan-91	18	83.9	92.2
01-Feb-91	19	86.7	92.5
02-Feb-91	20	85.8	92.6
09-Feb-91	22	87.3	93.3
10-Feb-91	23	87.5	93.3
11-Feb-91	24	88.6	93.0
12-Feb-91	25	87.2	92.7
13-Feb-91	26	87.1	92.5
14-Feb-91	27	85.2	92.7
15-Feb-91	28	84.9	92.4
21-Feb-91	30	87.1	94.1
22-Feb-91	31	88.3	92.7
23-Feb-91	32	88.0	93.1
24-Feb-91	33	88.7	92.4
25-Feb-91	34	88.3	93.0
03-Mar-91	36	88.5	95.6
04-Mar-91	37	90.0	93.4
05-Mar-91	38	90.3	95.1
06-Mar-91	39	90.4	94.0
07-Mar-91	40	89.0	94.6
08-Mar-91	41	89.4	94.6
09-Mar-91	42	90.2	94.0
10-Mar-91	43	89.9	93.5
11-Mar-91	44	89.7	93.2
12-Mar-91	45	90.0	94.4
13-Mar-91	46	90.2	94.6
14-Mar-91	47	90.3	93.5
15-Mar-91	48	90.5	94.4
16-Mar-91	49	90.0	93.9
17-Mar-91	50	89.7	94.7
18-Mar-91	51	91.6	94.3

Table 24 (Continued)
Coal Conversion (Phase 2 Data)

PERIOD : Jan. 18-20		
Average	78.1	92.9
Stan. Dev. (σ_n)	1.4	0.5
Stan. Dev. (σ_{n-1})	1.7	0.6
PERIOD 261A: Jan. 29 - Feb. 2		
Average	84.4	91.5
Stan. Dev. (σ_n)	1.6	1.1
Stan. Dev. (σ_{n-1})	1.8	1.3
PERIOD 261B: Feb. 11-15		
Average	86.6	92.7
Stan. Dev. (σ_n)	1.4	0.2
Stan. Dev. (σ_{n-1})	1.5	0.2
PERIOD : Feb. 23-25		
Average	88.3	92.8
Stan. Dev. (σ_n)	0.3	0.3
Stan. Dev. (σ_{n-1})	0.4	0.4
PERIOD : Mar. 4-8		
Average	89.8	94.3
Stan. Dev. (σ_n)	0.5	0.6
Stan. Dev. (σ_{n-1})	0.6	0.7
PERIOD 261C: Mar. 7-11		
Average	89.6	94.0
Stan. Dev. (σ_n)	0.4	0.6
Stan. Dev. (σ_{n-1})	0.5	0.6
PERIOD 261D : Mar. 14-18		
Average	90.4	94.2
Stan. Dev. (σ_n)	0.6	0.4
Stan. Dev. (σ_{n-1})	0.7	0.5

Table 25
TSL System Responses in Resid + UC Conversion and Excess Resid
(Phase 3 Data)

TSL SYSTEM RESPONSES IN RESID+UC CONVERSION AND EXCESS RESID
Run 261 with Illinois No. 6 Coal, Jan. 12 - Mar. 18, 1991
Phase 3 Data

unit: wt % MAF coal

Date	Days	resid+UC conversion First	Second	Overall	TSL resid	Org.Rej.	Resid w/ OR=15%	OR=20%
29-Jan-91	16	44.9	27.7	72.6	6.9	20.5	12.4	7.4
30-Jan-91	17	46.4	27.3	73.7	4.0	22.3	11.3	6.3
31-Jan-91	18	41.8	26.9	68.7	9.9	21.4	16.3	11.3
01-Feb-91	19	49.4	24.3	73.7	6.0	20.3	11.3	6.3
02-Feb-91	20	46.7	23.0	69.7	10.3	20.0	15.3	10.3
11-Feb-91	24	43.0	36.8	79.8	1.7	18.5	5.2	0.2
12-Feb-91	25	47.0	32.2	79.2	1.9	18.9	5.8	0.8
13-Feb-91	26	44.2	31.7	75.9	6.9	17.2	9.1	4.1
14-Feb-91	27	41.2	33.4	74.6	9.7	15.7	10.4	5.4
15-Feb-91	28	43.6	34.4	78.0	3.1	18.9	7.0	2.0
07-Mar-91	40	51.3	32.5	83.8	2.5	13.7	1.2	-3.8
08-Mar-91	41	49.0	31.8	80.8	3.1	16.1	4.2	-0.8
09-Mar-91	42	43.4	34.6	78.0	7.3	14.7	7.0	2.0
10-Mar-91	43	45.0	32.1	77.1	6.1	16.8	7.9	2.9
11-Mar-91	44	45.3	30.5	75.8	6.6	17.6	9.2	4.2
14-Mar-91	47	51.9	33.2	85.1	-0.6	15.5	-0.1	-5.1
15-Mar-91	48	50.1	31.1	81.2	2.5	16.3	3.8	-1.2
16-Mar-91	49	48.2	30.5	78.7	5.7	15.6	6.3	1.3
17-Mar-91	50	49.4	32.4	81.8	4.3	14.9	4.2	-0.8
18-Mar-91	51	52.2	26.9	79.1	6.6	14.3	5.9	0.9
PERIOD 261A: Jan. 29 - Feb. 2								
Average		45.8	25.8	71.7	7.4	20.9	13.3	8.3
Stan. Dev. (σ_n)		2.5	1.9	2.1	2.4	0.8	2.1	2.1
Stan. Dev. (σ_{n-1})		2.8	2.1	2.3	2.7	0.9	2.3	2.3
PERIOD 261B: Feb. 11-15								
Average		43.8	33.7	77.5	4.7	17.8	7.5	2.5
Stan. Dev. (σ_n)		1.9	1.8	2.0	3.1	1.2	2.0	2.0
Stan. Dev. (σ_{n-1})		2.1	2.0	2.2	3.5	1.4	2.2	2.2
PERIOD 261C: Mar. 7-11								
Average		46.8	32.3	79.1	5.1	15.8	5.9	0.9
Stan. Dev. (σ_n)		2.9	1.3	2.9	1.9	1.4	2.9	2.9
Stan. Dev. (σ_{n-1})		3.2	1.5	3.2	2.2	1.6	3.2	3.2
PERIOD 261D: Mar. 14-18								
Average		50.4	30.8	81.2	3.7	15.3	4.0	-1.0
Stan. Dev. (σ_n)		1.5	2.2	2.3	2.6	0.7	2.3	2.3
Stan. Dev. (σ_{n-1})		1.7	2.4	2.6	2.9	0.8	2.5	2.5

Table 25 (Continued)

TSL System Responses in Resid + UC Conversion and Excess Resid
(Phase 3 Data)

			unit:	wt % feed	hr^{-1}	hr^{-1}		wt % feed	hr^{-1}	hr^{-1}
coal conversion				First stage rate constant				Second stage rate constant		
First	Second	TSL		Conv.	K_Vol	LN K_Vol		Conv.	K_Vol	LN K_Vol
83.8	90.7	91.1		24.1	65.4	4.18		19.2	48.9	3.89
82.5	89.7	90.2		25.0	68.7	4.23		19.5	50.0	3.91
83.9	91.6	91.6		22.9	61.6	4.12		18.4	46.9	3.85
86.8	91.6	91.2		25.4	70.4	4.25		14.3	34.7	3.55
85.7	91.8	91.2		24.1	65.1	4.18		14.5	34.9	3.55
88.3	92.6	91.6		21.9	58.0	4.06		22.2	60.2	4.10
87.5	92.6	91.6		23.4	63.4	4.15		19.2	50.3	3.92
87.2	92.3	91.5		22.2	59.0	4.08		19.7	51.6	3.94
85.8	92.5	92.8		20.8	54.5	4.00		21.0	56.1	4.03
86.4	92.0	91.0		21.5	55.9	4.02		21.6	57.7	4.05
89.2	93.6	94.1		25.5	55.5	4.02		20.0	40.5	3.70
89.3	94.0	93.5		24.6	52.5	3.96		20.9	42.6	3.75
90.5	94.1	94.0		22.2	45.9	3.83		20.5	41.4	3.72
90.0	93.8	93.2		23.0	47.9	3.87		20.0	40.0	3.69
89.6	93.5	93.1		23.2	48.4	3.88		18.5	36.4	3.60
90.8	93.6	93.1		23.5	49.1	3.89		18.2	35.5	3.57
90.3	93.9	92.8		22.4	47.1	3.85		17.5	34.6	3.54
90.0	93.8	93.3		22.1	46.8	3.85		17.3	34.4	3.54
89.7	94.2	92.9		22.5	47.4	3.86		18.3	36.5	3.60
91.8	94.1	92.8		24.1	51.4	3.94		15.3	29.3	3.38
84.5	91.1	91.1		24.3	66.3	4.19		17.2	43.1	3.75
1.5	0.8	0.5		0.9	3.1	0.05		2.3	6.8	0.16
1.7	0.9	0.5		1.0	3.4	0.05		2.6	7.6	0.18
87.0	92.4	91.7		21.9	58.2	4.06		20.8	55.2	4.01
0.9	0.2	0.6		0.9	3.1	0.05		1.1	3.7	0.07
1.0	0.3	0.7		1.0	3.4	0.06		1.3	4.1	0.08
89.7	93.8	93.6		23.7	50.1	3.91		20.0	40.2	3.69
0.5	0.2	0.4		1.2	3.5	0.07		0.8	2.1	0.05
0.5	0.3	0.5		1.3	3.9	0.08		0.9	2.3	0.06
90.5	93.9	93.0		22.9	48.4	3.88		17.3	34.1	3.52
0.7	0.2	0.2		0.7	1.7	0.03		1.1	2.5	0.08
0.8	0.2	0.2		0.8	1.9	0.04		1.2	2.8	0.09

Table 26

TSL System Responses in Resid + UC Conversion and Excess Resid

(Phase 2 Data)

TSL SYSTEM RESPONSES IN RESID+UC CONVERSION AND EXCESS RESID
Run 261 with Illinois No. 6 Coal, Apr. 12-May 30, 1991

unit. wt % MAF coal

		resid+UC conversion			Phase 2 Data (Measured)			
							Resid	
Date	Days	First	Second	Overall	TSL resid	Org.Rcj.	w/ OR=15%	OR=20%
13-Apr-91	52	60.2	37.5	97.7	13.3	-11.0	-12.7	-17.7
14-Apr-91	53	56.7	27.2	83.9	6.9	9.2	1.1	-3.9
15-Apr-91	54	43.9	43.0	86.9	-1.8	14.9	-1.9	-6.9
16-Apr-91	55	45.2	29.7	74.9	7.9	17.2	10.1	5.1
17-Apr-91	56	59.3	32.0	91.3	-3.8	12.5	-6.3	-11.3
18-Apr-91	57	51.1	32.1	83.2	1.1	15.7	1.8	-3.2
19-Apr-91	58	53.5	30.4	83.9	-0.4	16.5	1.1	-3.9
20-Apr-91	59	45.5	38.6	84.1	-0.7	16.6	0.9	-4.1
21-Apr-91	60	39.9	36.8	76.7	7.9	15.4	8.3	3.3
22-Apr-91	61	39.2	38.3	77.5	7.1	15.4	7.5	2.5
23-Apr-91	62	40.5	39.8	80.3	5.8	13.9	4.7	-0.3
24-Apr-91	63	47.9	38.2	86.1	-2.5	16.4	-1.1	-6.1
25-Apr-91	64	47.6	37.7	85.3	-1.6	16.3	-0.3	-5.3
26-Apr-91	65	40.4	40.4	80.8	3.0	16.2	4.2	-0.8
27-Apr-91	66	46.7	42.2	88.9	-3.4	14.5	-3.9	-8.9
28-Apr-91	67	46.9	40.6	87.5	-1.4	13.9	-2.5	-7.5
29-Apr-91	68	42.0	42.3	84.3	2.3	13.4	0.7	-4.3
30-Apr-91	69	42.1	43.2	85.3	1.1	13.6	0.3	-5.3
01-May-91	70	38.0	43.4	81.4	4.9	13.7	3.6	-1.4
02-May-91	71	41.1	43.6	84.7	2.3	13.0	0.3	-4.7
03-May-91	72	37.4	43.8	81.2	4.6	14.2	3.8	-1.2
04-May-91	73	33.6	49.0	82.6	1.7	15.7	2.4	-2.6
05-May-91	74	40.1	35.0	75.1	8.8	16.1	9.9	4.9
06-May-91	75	40.3	33.5	73.8	8.5	17.7	11.2	6.2
07-May-91	76	40.4	40.6	81.0	0.7	18.3	4.(	-1.0
08-May-91	77	43.6	39.4	83.0	3.2	16.8	5.0	0.0
09-May-91	78	38.9	35.4	74.3	8.7	17.0	10.7	5.7
10-May-91	79	40.0	40.4	0.4	0.6	19.0	4.6	-0.4
11-May-91	80	47.0	36.2	83.2	1.5	16.3	2.8	-2.2
15-May-91	82	48.9	59.2	108.1	-24.9	16.8	-23.1	-28.1
16-May-91	83	47.4	36.2	83.6	1.0	14.6	0.6	-4.4
17-May-91	84	52.2	32.6	84.8	0.9	14.4	0.3	-4.7
18-May-91	85	38.4	42.8	81.2	3.9	14.9	3.8	-1.2
19-May-91	86	36.9	44.4	81.3	3.5	15.2	3.7	-1.3
20-May-91	87	39.1	41.0	80.1	4.1	15.8	4.9	-0.1
21-May-91	88	43.1	37.8	80.9	5.1	14.0	4.1	-0.9
22-May-91	89	44.1	41.5	85.6	-3.4	17.8	-0.6	-5.6
23-May-91	90	43.2	37.5	80.7	-8.2	27.5	4.3	-0.7
24-May-91	91	48.5	32.8	81.3	3.2	15.4	3.7	-1.3
25-May-91	92	47.2	36.3	83.5	2.4	14.1	1.5	-3.5
26-May-91	93	50.7	34.7	85.4	0.2	14.4	-0.4	-5.4
27-May-91	94	45.6	33.8	79.4	4.4	16.2	5.6	0.6
28-May-91	95	33.9	46.1	80.0	6.1	13.9	5.0	0.0
29-May-91	96	44.4	40.9	85.3	5.0	9.7	-0.3	-5.3
30-May-91	97	46.8	39.3	86.1	-2.4	16.3	-1.1	-6.1

Table 26 (Continued)

TSL System Responses in Resid + UC Conversion and Excess Resid
(Phase 2 Data)

PERIOD 261E: Apr. 23-26 not including Apr. 20							
Average	44.1	39.0	83.1	1.2	15.7	1.9	-3.1
Stan. Dev. (σ_n)	3.7	1.1	2.6	3.4	1.0	2.6	2.6
Stan. Dev. (σ_{n-1})	4.2	1.3	3.0	3.9	1.2	3.0	3.0
PERIOD 261F: May 17-21							
Average	41.9	39.7	81.7	3.5	14.9	3.4	-1.6
Stan. Dev. (σ_n)	5.5	4.2	1.6	1.4	0.6	1.6	1.6
Stan. Dev. (σ_{n-1})	6.2	4.7	1.8	1.6	0.7	1.8	1.8
PERIOD 261G: May 24-27							
Average	48.0	34.4	82.4	2.6	15.0	2.6	-2.4
Stan. Dev. (σ_n)	1.9	1.3	2.3	1.5	0.8	2.3	2.3
Stan. Dev. (σ_{n-1})	2.2	1.5	2.6	1.8	1.0	2.6	2.6

wt % MAF coal

Table 27

Coal Conversion (Phase 3 Data)

RUN 261 COAL CONVERSION
Illinois No. 6 Coal, Apr. 12-May 30, 1991

unit: wt % MAF coal

Date	Days	First	Second
13-Apr-91	52	92.2	93.2
14-Apr-91	53	93.0	93.6
15-Apr-91	54	90.9	94.9
16-Apr-91	55	92.1	94.1
17-Apr-91	56	92.3	94.4
18-Apr-91	57	90.6	94.3
19-Apr-91	58	91.6	95.4
20-Apr-91	59	93.1	94.5
21-Apr-91	60	93.4	94.4
22-Apr-91	61	92.3	94.1
23-Apr-91	62	91.8	94.1
24-Apr-91	63	93.2	95.6
25-Apr-91	64	92.8	95.3
26-Apr-91	65	91.7	94.7
27-Apr-91	66	90.6	94.7
28-Apr-91	67	91.5	94.3
29-Apr-91	68	91.7	95.2
30-Apr-91	69	92.9	95.6
01-May-91	70	93.0	94.7
02-May-91	71	92.9	93.8
03-May-91	72	91.7	93.9
04-May-91	73	90.6	94.8
05-May-91	74	89.0	94.9
06-May-91	75	89.1	93.9
07-May-91	76	89.1	93.8
08-May-91	77	91.8	94.4
09-May-91	78	90.2	95.4
10-May-91	79	89.1	92.6
11-May-91	80	90.5	95.0
15-May-91	82	93.3	95.0
16-May-91	83	91.3	94.7
17-May-91	84	91.4	94.4
18-May-91	85	92.7	94.0
19-May-91	86	93.1	93.3
20-May-91	87	92.3	94.1
21-May-91	88	91.6	93.8
22-May-91	89	91.9	95.1
23-May-91	90	90.5	93.2
24-May-91	91	92.4	94.3
25-May-91	92	91.8	94.7
26-May-91	93	92.4	95.9
27-May-91	94	90.8	94.3
28-May-91	95	90.8	93.6
29-May-91	96	90.9	94.4
30-May-91	97	91.4	93.4

Table 27 (Continued)

Coal Conversion (Phase 3 Data)

PERIOD 261E: Apr. 23-26 not including Apr. 20		
Average	92.4	94.9
Stan. Dev. (σ_n)	0.6	0.6
Stan. Dev. (σ_{n-1})	0.7	0.7
PERIOD 261F: May 17-21		
Average	92.2	93.9
Stan. Dev. (σ_n)	0.6	0.4
Stan. Dev. (σ_{n-1})	0.7	0.4
PERIOD 261G: May 24-27		
Average	91.9	94.8
Stan. Dev. (σ_n)	0.7	0.7
Stan. Dev. (σ_{n-1})	0.8	0.8

Table 28

TSL System Responses in Resid + UC Conversion and Excess Resid (Phase 3 Data)

TSL SYSTEM RESPONSES IN RESID+UC CONVERSION AND EXCESS RESID
Run 261 with Illinois No. 6 Coal, Apr. 12 - May 30, 1991
Phase 3 Data

unit: wt % MAF coal

Date	Days	resid+UC conversion First	Second	Overall	TSL resid	Resid Org.Rej.	w/ OR=15%	OR=20%
20-Apr-91	59	39.8	40.5	80.3	3.0	16.7	4.7	-0.3
23-Apr-91	62	42.8	32.6	75.4	10.4	14.2	9.6	4.6
24-Apr-91	63	46.5	33.0	79.5	3.8	17.4	6.2	1.2
25-Apr-91	64	45.9	35.2	81.1	1.5	17.4	3.9	-1.1
26-Apr-91	65	37.4	36.0	73.4	8.3	18.3	11.6	6.6
17-May-91	84	52.8	25.5	78.3	5.3	16.4	6.7	1.7
18-May-91	85	37.0	37.1	74.1	10.6	15.3	10.9	5.9
19-May-91	86	35.7	35.3	71.0	13.4	15.6	14.0	9.0
20-May-91	87	38.7	34.6	73.3	10.4	16.2	11.6	6.6
21-May-91	88	41.6	32.2	73.8	11.9	14.3	11.2	6.2
24-May-91	91	45.0	32.4	77.4	6.8	15.8	7.6	2.6
25-May-91	92	45.8	32.8	78.6	6.1	15.3	6.4	1.4
26-May-91	93	49.9	33.6	83.5	1.6	14.9	1.5	-3.5
27-May-91	94	47.9	29.6	77.5	6.0	16.5	7.5	2.5
PERIOD 261E: Apr. 20,23-26								
Average		42.5	35.5	77.9	5.4	16.8	7.2	2.2
Stan. Dev. (σ_n)		3.5	2.8	3.0	3.4	1.4	2.9	2.9
Stan. Dev. (σ_{n-1})		3.9	3.2	3.4	3.8	1.6	3.3	3.3
PERIOD 261F: May 17-21								
Average		41.2	32.9	74.1	10.3	15.6	10.9	5.9
Stan. Dev. (σ_n)		6.1	4.0	2.4	2.7	0.7	2.4	2.4
Stan. Dev. (σ_{n-1})		6.9	4.5	2.6	3.1	0.8	2.6	2.6
PERIOD 261G: May 24-27								
Average		47.2	32.1	79.3	5.1	15.6	5.8	0.8
Stan. Dev. (σ_n)		1.9	1.5	2.5	2.1	0.6	2.5	2.5
Stan. Dev. (σ_{n-1})		2.2	1.7	2.9	2.4	0.7	2.9	2.9

Table 28 (Continued)

TSL System Responses in Resid + UC Conversion and Excess Resid

(Phase 3 Data)

			unit:	wt % feed	hr^{-1}	hr^{-1}	wt % feed	hr^{-1}	hr^{-1}
coal conversion				First stage rate constant			Second stage rate constant		
First	Second	TSL		Conv.	K_Vol	LN K_Vol	Conv.	K_Vol	LN K_Vol
92.9	93.7	92.9		18.4	42.1	3.74	22.8	55.4	4.01
92.1	93.4	93.5		19.4	45.6	3.82	18.0	41.8	3.73
93.0	94.1	92.9		21.2	51.3	3.94	19.1	45.0	3.81
92.4	94.1	92.5		20.8	49.8	3.91	19.7	46.7	3.84
91.7	94.1	91.8		17.3	39.7	3.68	20.3	48.3	3.88
91.7	93.8	92.4		22.3	43.9	3.78	13.7	24.3	3.19
92.3	93.7	92.5		16.0	29.3	3.38	18.5	34.7	3.55
93.3	93.5	92.3		15.6	28.3	3.34	17.0	31.3	3.44
92.5	93.7	91.9		16.3	29.7	3.39	17.1	31.4	3.45
91.4	93.6	93.1		17.6	32.6	3.48	15.9	29.0	3.37
92.5	93.4	92.3		18.0	36.0	3.58	15.7	30.6	3.42
91.9	93.9	92.3		19.1	36.7	3.60	16.5	30.5	3.42
92.5	94.3	92.2		20.1	38.2	3.64	16.6	30.1	3.40
91.0	93.1	91.9		20.3	39.4	3.67	14.6	26.4	3.28
92.4	93.9	92.7		19.4	45.7	3.82	20.0	47.4	3.86
0.5	0.3	0.6		1.5	4.4	0.10	1.6	4.5	0.09
0.5	0.3	0.6		1.6	4.9	0.11	1.8	5.0	0.10
92.2	93.7	92.4		17.6	32.8	3.48	16.4	30.2	3.40
0.7	0.1	0.4		2.4	5.8	0.16	1.6	3.4	0.12
0.7	0.1	0.4		2.7	6.4	0.18	1.8	3.8	0.13
92.0	93.7	92.2		19.4	37.6	3.63	15.8	29.4	3.38
0.6	0.5	0.2		0.9	1.3	0.04	0.8	1.7	0.06
0.7	0.5	0.2		1.1	1.5	0.04	0.9	2.0	0.07

Table 29
First Stage Catalyst Activity in Resid + UC Conversion
EXP-AO-60 Catalyst

RUN 261 FIRST STAGE CATALYST ACTIVITY IN RESID + UC CONVERSION
EXP-AO-60 1/16", Illinois No. 6 Coal
Batch Operation: Jan. 12-31 and Feb. 16 - Mar. 11, 1991
Steady-State Operation: Feb. 1-15 at 3 lb/ton, Mar. 14-18 at 1.5 lb/ton MF Coal
Full-Volume Reactor, 270 lbs Charge (240 lbs Before Jan. 24)
Phase 2 Data

Date	Days	Temp.	Factor	MF_Age	Age_Vol	SV_Cat_Vol	Conv.	K_Vol	LN K_Vol	T_Inv.
14-Jan-91	2	761	3.09	62	0.20	60.1	23.0	55.5	4.02	0.000819
15-Jan-91	3	762	3.12	103	0.34	57.2	28.1	69.7	4.24	0.000818
16-Jan-91	4	761	3.13	143	0.47	54.7	23.8	53.5	3.98	0.000819
17-Jan-91	5	759	3.13	182	0.60	53.5	21.1	44.8	3.80	0.000820
18-Jan-91	6	760	3.11	222	0.73	55.9	16.5	34.3	3.54	0.000820
19-Jan-91	7	761	3.11	264	0.87	57.5	19.4	43.0	3.76	0.000819
20-Jan-91	8	762	3.13	306	1.01	57.1	17.6	38.2	3.64	0.000818
23-Jan-91	10	776	3.09	359	1.18	60.0	28.1	72.5	4.28	0.000809
26-Jan-91	13	792	3.02	486	1.60	61.6	29.4	77.4	4.35	0.000799
27-Jan-91	14	791	3.10	530	1.75	61.0	25.9	66.1	4.19	0.000799
28-Jan-91	15	792	3.10	578	1.91	65.2	25.4	68.9	4.23	0.000799
29-Jan-91	16	790	3.11	626	2.07	66.2	25.8	71.5	4.27	0.000800
30-Jan-91	17	790	3.10	674	2.22	66.4	25.0	68.5	4.23	0.000800
31-Jan-91	18	789	3.10	723	2.39	67.0	23.7	64.4	4.17	0.000801
01-Feb-91	19	790	3.11	665	2.19	66.6	24.0	65.3	4.18	0.000800
02-Feb-91	20	789	3.10	713	2.35	66.3	22.9	61.0	4.11	0.000801
09-Feb-91	22	789	3.07	702	2.32	68.0	24.9	69.3	4.24	0.000801
10-Feb-91	23	789	3.09	648	2.14	67.6	28.3	82.5	4.41	0.000801
11-Feb-91	24	788	3.12	697	2.30	66.5	21.6	57.1	4.05	0.000801
12-Feb-91	25	789	3.10	643	2.12	66.9	22.8	61.3	4.12	0.000801
13-Feb-91	26	790	3.11	691	2.28	66.6	21.7	57.5	4.05	0.000800
14-Feb-91	27	789	3.11	639	2.11	66.5	21.2	55.7	4.02	0.000801
15-Feb-91	28	790	3.10	687	2.27	66.0	21.2	55.0	4.01	0.000800
21-Feb-91	30	791	3.11	792	2.61	66.3	25.3	69.8	4.25	0.000799
22-Feb-91	31	791	3.12	840	2.77	66.5	21.8	57.8	4.06	0.000799
23-Feb-91	32	791	3.11	889	2.93	67.0	20.0	52.2	3.95	0.000799
24-Feb-91	33	790	3.14	937	3.09	66.3	22.4	60.0	4.09	0.000800
25-Feb-91	34	790	3.11	986	3.25	66.7	18.0	45.5	3.82	0.000800
03-Mar-91	36	791	3.14	1070	3.53	52.0	24.8	53.8	3.99	0.000799
04-Mar-91	37	792	3.13	1108	3.66	52.0	24.6	53.1	3.97	0.000799
05-Mar-91	38	791	3.13	1146	3.78	51.7	21.4	44.0	3.78	0.000799
06-Mar-91	39	791	3.14	1183	3.90	51.4	22.6	47.1	3.85	0.000799
07-Mar-91	40	791	3.15	1181	3.90	51.5	25.1	54.3	3.99	0.000799
08-Mar-91	41	791	3.12	1219	4.02	51.6	23.8	50.3	3.92	0.000799
09-Mar-91	42	790	3.16	1256	4.14	51.0	22.0	45.4	3.82	0.000800
10-Mar-91	43	791	3.13	1293	4.27	51.3	22.6	46.8	3.85	0.000799
11-Mar-91	44	790	3.13	1330	4.39	51.4	22.7	47.2	3.85	0.000800
12-Mar-91	45	791	3.12	1368	4.51	52.1	25.9	56.8	4.04	0.000799
13-Mar-91	46	790	3.12	1406	4.64	51.8	24.3	51.9	3.95	0.000800
14-Mar-91	47	790	3.14	1363	4.50	51.1	23.2	48.4	3.88	0.000800
15-Mar-91	48	791	3.19	1401	4.62	51.1	22.1	46.2	3.83	0.000799
16-Mar-91	49	791	3.16	1359	4.48	52.2	22.0	46.5	3.84	0.000799
17-Mar-91	50	792	3.12	1397	4.61	52.4	22.6	47.7	3.87	0.000799
18-Mar-91	51	792	3.12	1356	4.47	51.8	24.5	52.5	3.96	0.000799

Table 29 (Continued)

First Stage Catalyst Activity in Resid + UC Conversion
EXP-AO-60 Catalyst

PERIOD : Jan. 18-20									
Average	761	3.12	264	0.87	56.8	17.8	38.5	3.65	0.000819
Stan. Dev. (s_n)	1	0.01	34	0.11	0.7	1.2	3.6	0.09	0.000001
Stan. Dev. (s_{n-1})	1	0.02	42	0.14	0.8	1.5	4.4	0.11	0.000001
PERIOD 261A: Jan. 29 - Feb. 2									
Average	790	3.10	680	2.24	66.5	24.3	66.1	4.19	0.000800
Stan. Dev. (s_n)	0	0.00	35	0.12	0.3	1.0	3.6	0.05	0.000000
Stan. Dev. (s_{n-1})	1	0.01	39	0.13	0.3	1.1	4.0	0.06	0.000000
PERIOD 261B: Feb. 11-15									
Average	789	3.11	671	2.22	66.5	21.7	57.3	4.05	0.000801
Stan. Dev. (s_n)	1	0.01	25	0.08	0.3	0.6	2.2	0.04	0.000000
Stan. Dev. (s_{n-1})	1	0.01	28	0.09	0.3	0.7	2.4	0.04	0.000001
PERIOD : Feb. 23-25									
Average	790	3.12	937	3.09	66.7	20.1	52.6	3.96	0.000800
Stan. Dev. (s_n)	0	0.01	40	0.13	0.3	1.8	5.9	0.11	0.000000
Stan. Dev. (s_{n-1})	1	0.01	49	0.16	0.4	2.2	7.3	0.14	0.000000
PERIOD : Mar. 4-8									
Average	791	3.13	1167	3.85	51.6	23.5	49.7	3.90	0.000799
Stan. Dev. (s_n)	0	0.01	38	0.12	0.2	1.3	3.8	0.08	0.000000
Stan. Dev. (s_{n-1})	0	0.01	42	0.14	0.2	1.5	4.3	0.09	0.000000
PERIOD 261C: Mar. 7-11									
Average	791	3.14	1256	4.14	51.4	23.2	48.8	3.89	0.000800
Stan. Dev. (s_n)	0	0.01	53	0.17	0.2	1.1	3.2	0.06	0.000000
Stan. Dev. (s_{n-1})	1	0.01	59	0.19	0.2	1.2	3.5	0.07	0.000000
PERIOD 261D: Mar. 14-18									
Average	791	3.14	1375	4.54	51.7	22.9	40.3	3.88	0.000799
Stan. Dev. (s_n)	1	0.02	20	0.06	0.5	0.9	2.3	0.05	0.000000
Stan. Dev. (s_{n-1})	1	0.03	22	0.07	0.6	1.0	2.5	0.05	0.000001

Table 30

First Stage Catalyst Activity in Resid + UC Conversion
Criterion 324

RUN 261 FIRST STAGE CATALYST ACTIVITY IN RESID + UC CONVERSION
Criterion (Shell) 324 1/16", Illinois No. 6 Coal
Batch Operation: Apr. 12-17, Apr. 27-May 9,1991
Steady-State Operation: Apr. 19-26 at 3; May 10-30 at 2.25 lb/ton MF Coal
Full-Volume Reactor, 440 lbs Charge
Phase 2 Data

Date	Days	Temp.	Factor	MF_Age	Age_Vol	SV_Cat_Vol	Conv.	K_Vol	LN K_Vol	T_Inv.
13-Apr-91	52	810	3.16	555	3.00	51.8	27.3	61.4	4.12	0.000787
14-Apr-91	53	809	3.14	578	3.12	52.1	25.8	56.9	4.04	0.000788
15-Apr-91	54	810	3.13	601	3.25	52.5	21.5	45.0	3.81	0.000787
16-Apr-91	55	810	3.13	624	3.37	51.8	23.6	50.1	3.91	0.000787
17-Apr-91	56	811	3.12	647	3.49	51.8	28.6	64.6	4.17	0.000787
18-Apr-91	57	810	3.10	673	3.63	58.6	24.0	57.3	4.05	0.000787
19-Apr-91	58	810	3.08	646	3.49	61.6	24.7	62.2	4.13	0.000787
20-Apr-91	59	808	3.05	673	3.63	61.3	20.8	49.2	3.89	0.000789
21-Apr-91	60	808	3.09	645	3.48	61.6	18.4	43.0	3.76	0.000789
22-Apr-91	61	811	3.13	672	3.63	61.1	18.4	43.1	3.76	0.000787
23-Apr-91	62	809	3.12	644	3.48	60.9	18.2	42.3	3.74	0.000788
24-Apr-91	63	810	3.11	672	3.63	61.1	21.5	52.1	3.95	0.000787
25-Apr-91	64	808	3.11	643	3.47	59.4	21.5	50.5	3.92	0.000789
26-Apr-91	65	809	3.05	670	3.62	60.5	18.3	41.3	3.72	0.000788
27-Apr-91	66	810	3.12	693	3.74	52.2	21.1	43.6	3.77	0.000787
28-Apr-91	67	810	3.12	716	3.87	52.0	21.1	43.4	3.77	0.000787
29-Apr-91	68	810	3.13	738	3.99	49.6	19.0	36.5	3.60	0.000787
30-Apr-91	69	809	3.34	758	4.09	44.5	18.2	33.1	3.50	0.000788
01-May-91	70	808	3.44	778	4.20	44.3	16.0	29.0	3.37	0.000789
02-May-91	71	810	3.45	797	4.30	44.3	17.3	32.0	3.47	0.000787
03-May-91	72	809	3.44	817	4.41	44.4	15.6	28.3	3.34	0.000788
04-May-91	73	809	3.45	837	4.52	44.6	18.2	34.2	3.53	0.000788
05-May-91	74	809	3.46	857	4.63	44.2	16.8	30.9	3.43	0.000788
06-May-91	75	809	3.44	876	4.73	44.4	17.1	31.5	3.45	0.000788
07-May-91	76	810	3.44	896	4.84	44.1	16.6	30.2	3.41	0.000787
08-May-91	77	810	3.34	916	4.95	44.3	18.3	33.1	3.50	0.000787
09-May-91	78	809	3.37	935	5.05	44.5	16.6	29.8	3.39	0.000788
10-May-91	79	810	3.41	872	4.71	44.2	16.9	30.7	3.42	0.000787
11-May-91	80	811	3.44	892	4.82	44.4	19.6	37.3	3.62	0.000787
15-May-91	82	806	3.72	878	4.74	40.8	20.1	38.2	3.64	0.000790
16-May-91	83	810	3.50	897	4.84	43.7	19.7	37.5	3.62	0.000787
17-May-91	84	809	3.45	877	4.74	44.4	21.8	42.8	3.76	0.000788
18-May-91	85	809	3.47	897	4.84	44.2	16.1	29.4	3.38	0.000788
19-May-91	86	810	3.46	877	4.74	44.3	15.4	27.9	3.33	0.000787
20-May-91	87	809	3.48	897	4.84	43.7	16.1	29.2	3.37	0.000788
21-May-91	88	810	3.49	876	4.73	43.8	17.7	32.9	3.49	0.000787
22-May-91	89	809	3.48	896	4.84	43.9	18.2	34.0	3.53	0.000788
23-May-91	90	809	3.66	875	4.73	41.4	18.1	33.5	3.51	0.000788
24-May-91	91	809	3.80	894	4.83	43.3	19.1	38.8	3.66	0.000788
25-May-91	92	809	3.55	874	4.72	43.7	19.7	38.0	3.64	0.000788
26-May-91	93	810	3.53	893	4.82	43.0	20.7	39.6	3.68	0.000787
27-May-91	94	809	3.42	874	4.72	45.2	19.5	37.4	3.62	0.000788
28-May-91	95	808	3.62	892	4.82	42.4	14.1	25.2	3.23	0.000789
29-May-91	96	811	3.18	873	4.71	44.6	21.5	38.8	3.66	0.000787
30-May-91	97	811	3.50	892	4.82	43.7	20.0	38.2	3.64	0.000787

Table 30 (Continued)

First Stage Catalyst Activity in Resid + UC Conversion
Criterion 324

PERIOD 261E: Apr. 23-26 not including Apr. 20									
Average	809	3.10	657	3.55	60.5	19.9	46.5	3.83	0.000788
Stan. Dev. (σ_n)	1	0.03	14	0.07	0.7	1.6	4.8	0.10	0.000000
Stan. Dev. (σ_{n-1})	1	0.03	16	0.09	0.8	1.9	5.5	0.12	0.000001
PERIOD 261F: May 17-21									
Average	809	3.47	885	4.78	44.1	17.4	32.4	3.47	0.000788
Stan. Dev. (σ_n)	0	0.01	10	0.05	0.3	2.3	5.4	0.15	0.000000
Stan. Dev. (σ_{n-1})	1	0.02	11	0.06	0.3	2.6	6.1	0.17	0.000000
PERIOD 261G: May 24-27									
Average	809	3.57	884	4.77	43.8	19.8	38.5	3.65	0.000788
Stan. Dev. (σ_n)	0	0.14	10	0.05	0.8	0.6	0.8	0.02	0.000000
Stan. Dev. (σ_{n-1})	1	0.16	11	0.06	1.0	0.7	0.9	0.02	0.000000

NOTE:

unit	deg F	$\frac{\text{lb MFcoal}}{\text{lb-cat}}$	$\frac{(\times 10^{-4})}{\text{cuft-cat}}$	$\frac{\text{lb MFcoal}}{\text{hr-cuft-c}}$	wt % of feed r+UC	hr^{-1}	hr^{-1}	$deg\ R^{-1}$

Table 31

Second Stage Catalyst Activity in Resid + UC Conversion
EXP-AO-60 Catalyst

RUN 261 SECOND STAGE CATALYST ACTIVITY IN RESID + UC CONVERSION
EXP-AO-60 1/16", Illinois No. 6 Coal
Batch Operation: Jan. 12 - Feb. 8 and Feb. 16 - Mar. 11, 1991
Steady-State Operation: Feb. 9-15 at 3 lb/ton, Mar. 13-18 at 1.5 lb/ton MF Coal
Full-Volume Reactor, 270 lbs Charge
Phase 2 Data

Date	Days	Temp.	MF_Age	Age_Vol	SV_Cat_Vol	Conv.	K	LN K	T_Inv.
14-Jan-91	2	810	55	0.18	59.8	29.7	78.1	4.36	0.000787
15-Jan-91	3	810	92	0.30	51.0	27.0	58.8	4.07	0.000787
16-Jan-91	4	810	128	0.42	48.9	29.5	64.1	4.16	0.000787
17-Jan-91	5	808	162	0.53	47.8	29.3	62.0	4.13	0.000789
18-Jan-91	6	809	199	0.66	49.9	30.9	69.3	4.24	0.000788
19-Jan-91	7	810	236	0.78	51.4	27.4	60.3	4.10	0.000787
20-Jan-91	8	810	273	0.90	51.0	29.2	65.9	4.19	0.000787
23-Jan-91	10	807	358	1.18	53.6	24.8	54.6	4.00	0.000789
26-Jan-91	13	810	485	1.60	61.7	23.2	56.3	4.03	0.000787
27-Jan-91	14	809	530	1.75	61.2	22.4	54.8	4.00	0.000788
28-Jan-91	15	810	577	1.90	65.4	20.6	52.7	3.96	0.000787
29-Jan-91	16	811	625	2.06	66.3	18.4	46.4	3.84	0.000787
30-Jan-91	17	810	674	2.22	66.5	21.2	55.4	4.01	0.000787
31-Jan-91	18	810	723	2.39	67.1	20.5	53.6	3.98	0.000787
01-Feb-91	19	811	771	2.54	66.7	18.5	47.0	3.85	0.000787
02-Feb-91	20	809	819	2.70	66.4	17.7	44.2	3.79	0.000788
09-Feb-91	22	811	687	2.27	68.5	21.5	57.6	4.05	0.000787
10-Feb-91	23	810	737	2.43	68.2	16.0	40.2	3.69	0.000787
11-Feb-91	24	810	677	2.23	67.5	20.8	55.3	4.01	0.000787
12-Feb-91	25	810	726	2.40	68.1	20.0	52.8	3.97	0.000787
13-Feb-91	26	809	668	2.20	67.8	20.9	55.8	4.02	0.000788
14-Feb-91	27	807	717	2.37	67.7	23.4	64.4	4.17	0.000789
15-Feb-91	28	806	660	2.18	67.4	23.9	65.6	4.18	0.000790
21-Feb-91	30	811	766	2.53	66.3	22.3	59.1	4.08	0.000787
22-Feb-91	31	810	814	2.69	66.5	20.5	53.4	3.98	0.000787
23-Feb-91	32	810	863	2.85	67.0	20.5	53.8	3.99	0.000787
24-Feb-91	33	810	911	3.01	66.3	19.7	51.0	3.93	0.000787
25-Feb-91	34	809	960	3.17	66.7	24.0	65.5	4.18	0.000788
03-Mar-91	36	809	1044	3.45	52.0	22.6	47.6	3.86	0.000788
04-Mar-91	37	809	1082	3.57	52.0	21.0	43.3	3.77	0.000788
05-Mar-91	38	808	1120	3.70	51.7	22.7	47.5	3.86	0.000789
06-Mar-91	39	809	1157	3.82	51.4	21.6	44.4	3.79	0.000788
07-Mar-91	40	810	1195	3.94	51.5	22.7	47.6	3.86	0.000787
08-Mar-91	41	809	1232	4.07	51.6	22.6	47.0	3.85	0.000788
09-Mar-91	42	809	1269	4.19	51.0	23.4	49.2	3.90	0.000788
10-Mar-91	43	810	1306	4.31	51.3	22.0	45.2	3.81	0.000787
11-Mar-91	44	810	1344	4.44	51.4	21.7	44.6	3.80	0.000787
12-Mar-91	45	809	1382	4.56	52.1	17.4	34.2	3.53	0.000788
13-Mar-91	46	809	1341	4.43	51.8	19.4	38.9	3.66	0.000788
14-Mar-91	47	809	1378	4.55	51.1	17.6	34.2	3.53	0.000788
15-Mar-91	48	810	1337	4.41	51.1	20.6	42.2	3.74	0.000787
16-Mar-91	49	810	1375	4.54	52.2	20.7	43.0	3.76	0.000787
17-Mar-91	50	809	1335	4.41	52.4	19.3	39.1	3.67	0.000788
18-Mar-91	51	809	1373	4.53	51.8	18.2	36.0	3.58	0.000788

Table 31 (Continued)
Second Stage Catalyst Activity in Resid + UC Conversion
EXP-AO-60 Catalyst

PERIOD : Jan. 18-20								
Average	810	236	0.78	50.8	29.2	65.2	4.18	0.000788
Stan. Dev. (sn)	0	30	0.10	0.6	1.4	3.7	0.06	0.000000
Stan. Dev. (sn-1)	1	37	0.12	0.8	1.8	4.5	0.07	0.000000
PERIOD 261A: Jan. 29 - Feb. 2								
Average	810	722	2.38	66.6	19.3	49.3	3.89	0.000787
Stan. Dev. (sn)	1	69	0.23	0.3	1.3	4.3	0.09	0.000000
Stan. Dev. (sn-1)	1	77	0.25	0.3	1.5	4.9	0.10	0.000001
PERIOD 261B: Feb. 11-15								
Average	808	690	2.28	67.7	21.8	58.8	4.07	0.000788
Stan. Dev. (sn)	2	27	0.09	0.2	1.6	5.2	0.09	0.000001
Stan. Dev. (sn-1)	2	30	0.10	0.3	1.7	5.8	0.10	0.000001
PERIOD : Feb. 23-25								
Average	810	911	3.01	66.7	21.4	56.8	4.03	0.000788
Stan. Dev. (sn)	0	40	0.13	0.3	1.9	6.3	0.11	0.000000
Stan. Dev. (sn-1)	1	49	0.16	0.4	2.3	7.7	0.13	0.000000
PERIOD : Mar. 4-8								
Average	809	1157	3.82	51.6	22.1	45.9	3.83	0.000788
Stan. Dev. (sn)	1	53	0.18	0.2	0.7	1.8	0.04	0.000000
Stan. Dev. (sn-1)	1	59	0.20	0.2	0.8	2.0	0.04	0.000000
PERIOD 261C: Mar. 7-11								
Average	810	1269	4.19	51.4	22.5	46.7	3.84	0.000788
Stan. Dev. (sn)	0	53	0.17	0.2	0.6	1.7	0.04	0.000000
Stan. Dev. (sn-1)	1	59	0.19	0.2	0.7	1.8	0.04	0.000000
PERIOD 261D : Mar. 14-18								
Average	809	1360	4.49	51.7	19.3	38.9	3.66	0.000788
Stan. Dev. (sn)	0	19	0.06	0.5	1.2	3.4	0.09	0.000000
Stan. Dev. (sn-1)	1	22	0.07	0.6	1.4	3.8	0.10	0.000000

Table 32

Second Stage Catalyst Activity in Resid + UC Conversion
EXP-AO-60 Catalyst

RUN 261 SECOND STAGE CATALYST ACTIVITY IN RESID + UC CONVERSION
Criterion (Shell) 324 1/16", Illinois No. 6 Coal
Batch Operation: Apr. 12-17, Apr. 27-May 7,1991
Steady-State Operation: Apr. 18-26 at 3; May 8-30 at 2.25 lb/ton MF Coal
Full-Volume Reactor, 440 lbs Charge
Phase 2 Data

Date	Days	Temp.	MF_Age	Age_Vol	SV_Cat_Vol	Conv.	K	LN K	T_Inv.
13-Apr-91	52	821	592	3.20	51.8	24.8	53.9	3.99	0.000781
14-Apr-91	53	824	615	3.32	52.1	25.8	56.9	4.04	0.000779
15-Apr-91	54	825	639	3.45	52.5	26.1	58.0	4.06	0.000778
16-Apr-91	55	824	662	3.57	51.8	22.3	46.5	3.84	0.000779
17-Apr-91	56	825	685	3.70	51.8	22.3	46.3	3.84	0.000778
18-Apr-91	57	824	663	3.58	58.6	19.1	42.8	3.76	0.000779
19-Apr-91	58	824	691	3.73	61.6	17.9	41.4	3.72	0.000779
20-Apr-91	59	824	661	3.57	61.3	21.4	51.0	3.93	0.000779
21-Apr-91	60	824	689	3.72	61.6	19.1	45.0	3.81	0.000779
22-Apr-91	61	822	660	3.56	61.1	21.2	51.4	3.94	0.000780
23-Apr-91	62	824	687	3.71	60.9	21.6	52.3	3.96	0.000779
24-Apr-91	63	824	658	3.55	61.1	21.8	53.0	3.97	0.000779
25-Apr-91	64	823	684	3.69	59.4	20.4	47.3	3.86	0.000779
26-Apr-91	65	823	655	3.54	60.5	21.7	51.1	3.93	0.000779
27-Apr-91	66	824	678	3.66	52.2	23.8	50.9	3.93	0.000779
28-Apr-91	67	824	701	3.79	52.0	22.1	46.0	3.83	0.000779
29-Apr-91	68	817	723	3.90	49.6	23.0	46.4	3.84	0.000783
30-Apr-91	69	802	743	4.01	44.5	21.6	41.0	3.71	0.000792
01-May-91	70	801	763	4.12	44.3	21.1	40.8	3.71	0.000793
02-May-91	71	800	782	4.22	44.3	21.2	41.1	3.72	0.000794
03-May-91	72	801	802	4.33	44.4	21.0	40.6	3.70	0.000793
04-May-91	73	801	822	4.44	44.6	19.6	37.5	3.62	0.000793
05-May-91	74	798	842	4.55	44.2	17.6	32.6	3.49	0.000795
06-May-91	75	796	861	4.65	44.4	16.0	29.1	3.37	0.000796
07-May-91	76	795	881	4.76	44.1	19.3	36.2	3.59	0.000797
08-May-91	77	799	862	4.65	44.3	17.2	30.7	3.42	0.000794
09-May-91	78	796	881	4.76	44.5	17.1	30.9	3.43	0.000796
10-May-91	79	796	901	4.87	44.2	18.9	35.1	3.56	0.000796
11-May-91	80	800	841	4.54	44.4	17.3	32.0	3.47	0.000794
15-May-91	82	786	867	4.68	40.8	27.5	57.6	4.05	0.000803
16-May-91	83	800	848	4.58	43.7	18.1	33.8	3.52	0.000794
17-May-91	84	801	868	4.69	44.4	16.4	30.1	3.40	0.000793
18-May-91	85	801	849	4.58	44.2	21.5	42.0	3.74	0.000793
19-May-91	86	800	869	4.69	44.3	21.7	42.4	3.75	0.000794
20-May-91	87	800	850	4.59	43.7	20.3	38.8	3.66	0.000794
21-May-91	88	800	869	4.69	43.8	18.9	35.6	3.57	0.000794
22-May-91	89	801	850	4.59	43.9	20.5	39.4	3.67	0.000793
23-May-91	90	800	869	4.69	41.4	19.6	37.0	3.61	0.000794
24-May-91	91	801	850	4.59	43.3	15.1	29.2	3.38	0.000793
25-May-91	92	800	869	4.69	43.7	17.9	33.8	3.52	0.000794
26-May-91	93	800	850	4.59	43.0	16.2	29.3	3.38	0.000794
27-May-91	94	800	870	4.70	45.2	16.6	30.8	3.43	0.000794
28-May-91	95	800	850	4.59	42.4	21.0	40.8	3.71	0.000794
29-May-91	96	800	870	4.70	44.6	23.8	44.3	3.79	0.000794
30-May-91	97	799	851	4.60	43.7	20.0	38.2	3.64	0.000794

Table 32 (Continued)

Second Stage Catalyst Activity in Resid + UC Conversion
EXP-AO-60 Catalyst

PERIOD 261E: Apr. 23-26 not including Apr. 20								
Average	824	671	3.62	56.0	21.4	50.9	3.93	0.000779
Stan. Dev. ($\bar{s}_n$)	1	15	0.08	4.3	0.6	2.2	0.04	0.000000
Stan. Dev. ($\bar{s}_{n-1}$)	1	17	0.09	4.9	0.7	2.6	0.05	0.000000
PERIOD 261F: May 17-21								
Average	800	861	4.65	44.1	19.8	37.8	3.62	0.000793
Stan. Dev. ($\bar{s}_n$)	0	9	0.05	0.3	2.0	4.6	0.13	0.000000
Stan. Dev. ($\bar{s}_{n-1}$)	1	11	0.06	0.3	2.2	5.1	0.14	0.000000
PERIOD 261G: May 24-27								
Average	800	860	4.64	43.8	16.5	30.8	3.43	0.000793
Stan. Dev. ($\bar{s}_n$)	0	10	0.05	0.8	1.0	1.8	0.06	0.000000
Stan. Dev. ($\bar{s}_{n-1}$)	1	11	0.06	1.0	1.2	2.1	0.07	0.000000

deg F	$\frac{\text{lb MFcoal}}{\text{lb-cat}}$	$\frac{(x10^{-4})\ \text{lb MFcoal}}{\text{cuft-c}}$	$\frac{\text{lb MFcoal}}{\text{hr-cuft-c}}$	wt % of feed r+UC	hr^{-1}	hr^{-1}	deg R^{-1}

Table 33

Blending Proportions of Atmospheric Overhead (V161) and Vacuum Tower Overhead (V182) Used To Prepare Product Quality Blends

Period	V161, wt %	V182, wt %
261A	38.0	62.0
261B	40.5	59.5
261C	41.0	59.0
261D	36.2	63.8
261E	47.7	52.3
261F	33.1	66.9

Table 34

Characteristics of Product Oils From Run 261
With Illinois Coal

Period	261A	261B	261C	261D	261E	261F
Elemental wt%						
C	87.80	87.25	87.20	87.49	86.28	87.19
H	11.60	11.42	11.78	11.27	10.86	10.65
N (ppm)	2,262	2,251	2,214	3,196	4,277	4,337
S	0.032	0.035	0.020	0.025	0.058	0.075
O by diff. (a)	0.34(0.77)	1.07(0.69)	0.78(0.62)	0.90(0.87)	2.38	1.65
°API Gravity	23.7	23.3	24.5	22.1	23.2	18.1
GC Sim. Dist., °F						
IBP	97	133	97	97	97	156
10 wt %	268	267	241	305	241	365
30 wt %	466	481	430	482	405	562
50 wt %	568	612	561	602	531	663
70 wt %	672	707	671	689	664	752
90 wt %	767	805	749	778	764	813
95 wt %	796	831	782	811	790	838
EP	838	892	812	868	856	879
Distribution, wt %						
IBP/350°F	17.7	18.9	22.3	13.9	23.0	11.9
350/450°F	11.3	11.1	16.7	15.5	17.8	10.4
450/650°F	46.3	39.8	43.0	40.0	36.2	39.3
650°F+	24.7	30.2	18.0	30.6	23.0	38.4
EP, °F (D1160)	752	772	753	780	786	925

(a) numbers in parenthesis are by direct analysis

Table 35

Product Characteristics of Naphtha (IBP-350°F)

Period	261A	261B	261C	261D	261E	261F
Elemental, wt %						
C	85.81	85.90	85.25	84.53	84.70	84.99
H	14.05	14.09	14.30	14.35	13.56	13.66
N (ppm)	242	288	265	240	687	686
S	0.053	0.037	0.014	0.043	0.083	0.079
O by diff. (a)	0.07	-0.06 (0.57)	0.41	0.06	1.59	1.20
Gravity (°API)	50.7	50.8	51.1	50.7	49.4	48.0
GC Sim. Dist. (°F)						
IBP	70	69	70	69	88	88
10 wt %	156	165	156	162	165	165
30 wt %	231	239	231	234	231	240
50 wt %	241	241	241	241	241	247
70 wt %	281	281	281	281	291	291
90 wt %	321	334	329	326	350	350
95 wt %	335	354	337	342	360	360
EP	360	360	360	360	360	363

(a) numbers in parenthesis are by direct analysis

Table 36

Product Characterization of Middle Distillate (350-450°F)

Period	261A	261B	261C	261D	261E	261F
Elemental, wt %						
C	87.61	87.46	87.37	86.78	87.38	87.01
H	12.28	12.30	12.43	12.34	11.78	11.79
N (ppm)	1,263	959	1,234	1,943	2,497	2,422
S	0.037	0.028	0.046	0.037	0.064	0.070
O by diff. (a)	-0.06	0.12(0.80)	0.03	0.65	0.53	0.89
Gravity (°API)	30.2	30.4	30.1	28.7	27.2	26.8
GC Sim. Dist. (°F)						
IBP	325	329	323	243	326	337
10 wt %	350	350	350	350	350	360
30 wt %	379	382	375	389	383	360
50 wt %	405	405	410	415	410	424
70 wt %	429	435	445	450	447	447
90 wt %	460	460	460	460	460	460
95 wt %	460	460	466	466	466	466
EP	479	482	492	475	475	492

(a) numbers in parenthesis are by direct analysis

Table 37

Product Characterization of Middle Distillate (450-650°F)

Period	261A	261B	261C	261D	261E	261F
Elemental, wt %						
C	88.38	88.37	88.51	87.43	88.16	88.15
H	11.42	11.11	11.30	11.34	10.69	10.70
N (ppm)	2,188	2,336	2,699	3,386	4,909	4,283
S	0.033	0.014	0.017	0.035	0.077	0.061
O by diff. (a)	-0.04	0.28(0.62)	-0.10	0.85	0.58	0.66
Gravity (°API)	19.4	19.2	18.6	18.7	16.7	16.3
GC Sim. Dist. (°F)						
IBP	451	450	451	445	460	460
10 wt %	481	486	482	479	492	492
30 wt %	541	539	531	538	544	538
50 wt %	592	590	568	590	581	595
70 wt %	643	643	634	643	643	643
90 wt %	672	676	670	673	680	676
95 wt %	676	676	676	676	692	680
EP	722	685	685	685	722	685

(a) numbers in parenthesis are by direct analysis

Table 38

Product Characteristics of Vacuum Gas Oil (650°F-EP)

Period	261A	261B	261C	261D	261E	261F
Elemental, wt %						
C	89.16	89.48	89.70	88.91	89.37	89.41
H	10.16	10.12	10.06	9.97	9.38	10.02
N (ppm)	3,397	3,636	3,661	4,490	5,695	4,827
S	0.014	0.021	0.015	0.021	0.072	0.079
O by diff. (a)	0.33	0.02(0.82)	-0.05	0.65	0.62	0.18
Gravity (°API)	10.8	9.7	9.9	8.8	6.7	9.4
GC Sim. Dist. (°F)						
IBP	722	685	685	685	722	685
10 wt %	722	722	722	722	722	722
30 wt %	740	740	740	740	740	740
50 wt %	776	782	765	761	761	780
70 wt %	796	810	790	781	783	800
90 wt %	838	858	815	838	838	852
95 wt %	838	858	838	850	857	868
EP	838	906	898	906	905	950

Table 39

PHASE 3-4 CRITERIA

UNIT AND ELEMENTAL CLOSURE ERRORS

WT% MAF COAL

PERIODS ->	261A	261B	261C	261D	261E	261F	261G
CCR1 UNIT DATA							
CARBON	-0.07±3.5	0.11±1.9	1.10±0.9	0.12±0.3	-0.79±1.8	0.15±0.6	2.69±2.6
HYDROGEN	-0.68±0.3	-1.08±0.2	-0.47±0.4	-0.65±0.2	-0.85±0.2	-1.69±0.2	-1.46±0.4
NITROGEN	-0.27±0.1	-0.15±0.0	-0.38±0.0	-0.29±0.0	-0.16±0.1	-0.13±0.1	-0.24±0.1
SULFUR	-1.40±0.2	-0.49±0.4	-0.73±0.2	-0.49±0.2	-0.46±0.1	-0.17±0.4	0.60±1.1
OXYGEN	1.91±3.4	1.67±2.1	-0.16±0.7	0.30±0.7	2.08±1.6	-0.25±0.4	0.10±1.0
ASH	0.50±0.4	-0.21±0.5	0.64±1.0	1.01±0.6	0.45±0.7	2.09±0.5	0.38±0.6
CCR2 UNIT DATA							
CARBON	-2.12±3.2	-1.29±3.2	-4.55±1.7	-4.02±1.5	-2.38±2.6	-8.84±2.0	-0.34±2.0
HYDROGEN	0.25±0.5	0.53±0.5	-0.30±0.2	0.10±0.3	0.20±0.1	0.20±0.3	0.92±0.1
NITROGEN	0.01±0.1	-0.26±0.1	-0.30±0.0	-0.51±0.3	-0.51±0.1	-0.24±0.1	-0.24±0.0
SULFUR	-0.29±0.5	-0.60±0.4	-0.15±0.0	-0.20±0.1	-0.34±0.2	-0.22±0.0	-0.63±0.8
OXYGEN	2.26±3.6	-1.15±1.9	0.64±0.7	1.38±0.7	0.74±1.9	1.28±1.0	1.87±1.8
ASH	-0.33±0.4	0.03±0.8	-0.99±0.4	-1.20±0.5	-0.90±1.0	-1.68±0.5	-1.22±0.3
ROSE-SR UNIT DATA							
CARBON	-0.25±0.1	-0.34±0.4	0.19±1.0	0.05±0.3	-0.57±0.8	0.40±1.1	-0.33±0.2
HYDROGEN	-0.08±0.0	-0.07±0.0	0.05±0.0	-0.01±0.0	-0.08±0.1	-0.07±0.0	-0.17±0.0
NITROGEN	0.00±0.0	0.00±0.0	-0.01±0.0	0.16±0.3	0.01±0.0	0.03±0.0	0.07±0.0
SULFUR	0.27±0.3	0.30±0.4	0.02±0.0	0.03±0.0	0.06±0.0	0.03±0.0	-0.02±0.0
OXYGEN	0.06±0.4	-0.34±0.5	0.21±0.1	-0.17±0.3	-0.07±0.2	-0.76±0.8	0.10±0.5
ASH	0.00±0.1	0.44±0.5	-0.02±0.2	-0.07±0.1	0.11±0.3	0.04±0.3	-0.29±0.1
TOTALED DATA							
CARBON	-2.44±0.8	-1.52±2.0	-3.26±1.2	-3.84±1.7	-3.75±2.1	-8.29±1.0	2.02±3.2
HYDROGEN	-0.51±0.2	-0.63±0.4	-0.72±0.5	-0.56±0.3	-0.74±0.2	-1.56±0.2	-0.72±0.5
NITROGEN	-0.26±0.0	-0.41±0.0	-0.69±0.0	-0.64±0.0	-0.66±0.1	-0.35±0.0	-0.41±0.0
SULFUR	-1.42±0.3	-0.79±0.5	-0.87±0.2	-0.66±0.3	-0.74±0.2	-0.35±0.3	-0.05±0.3
OXYGEN	4.23±0.3	0.19±1.1	0.69±0.4	1.51±0.4	2.75±0.2	0.28±0.2	2.07±0.5
ASH	0.17±0.5	0.25±0.4	-0.38±1.4	-0.26±0.4	-0.34±0.4	0.45±0.7	-1.13±0.4
ABSCLUTE SUM DATA							
CARBON	6.37±2.4	5.01±1.9	6.36±2.6	4.64±1.5	5.36±1.6	10.37±2.0	4.41±2.5
HYDROGEN	1.19±0.7	1.69±0.8	0.84±0.4	0.87±0.3	1.14±0.1	2.08±0.3	2.55±0.4
NITROGEN	0.38±0.1	0.41±0.0	0.69±0.0	0.96±0.6	0.71±0.1	0.43±0.0	0.55±0.1
SULFUR	2.07±1.0	1.41±0.7	0.92±0.2	0.74±0.2	0.87±0.2	0.62±0.2	1.49±1.8
OXYGEN	6.46±2.4	3.72±3.6	1.61±0.5	2.31±0.8	3.93±1.7	2.50±1.8	3.14±1.9
ASH	0.99±0.8	1.60±0.7	2.29±0.7	2.38±1.1	2.35±0.5	4.05±0.9	2.05±0.8

NOTES: (a) Closure error sign convention is that losses are negative. (b) Total is the arithmetic sum of the individual unit errors.

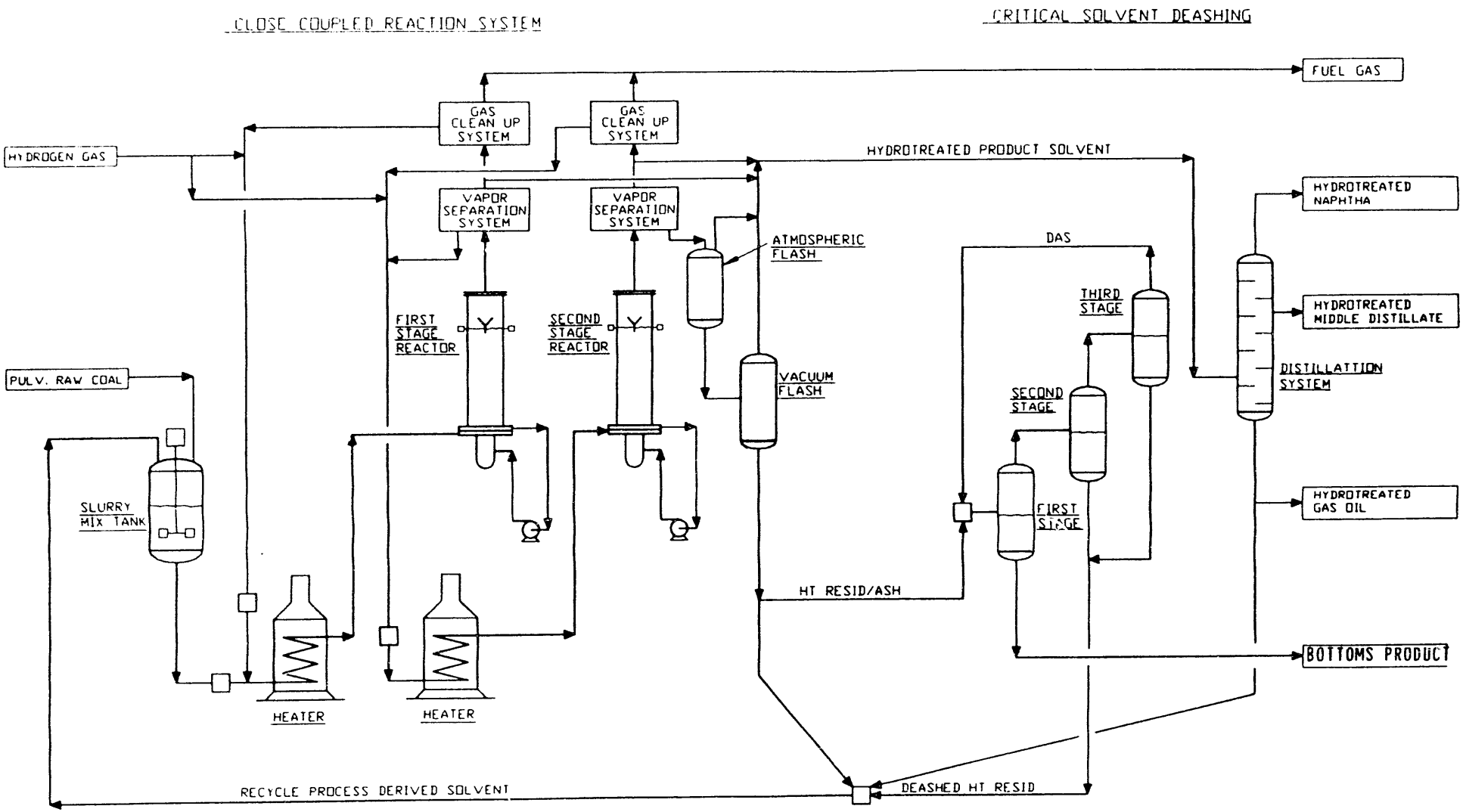
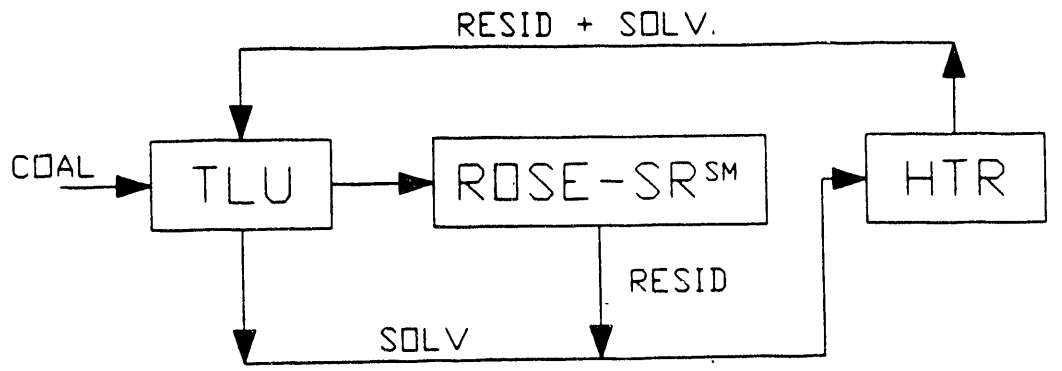
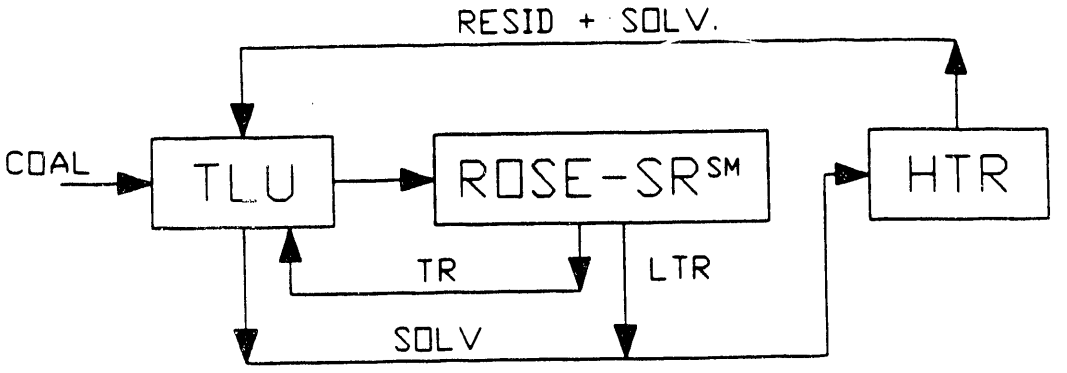


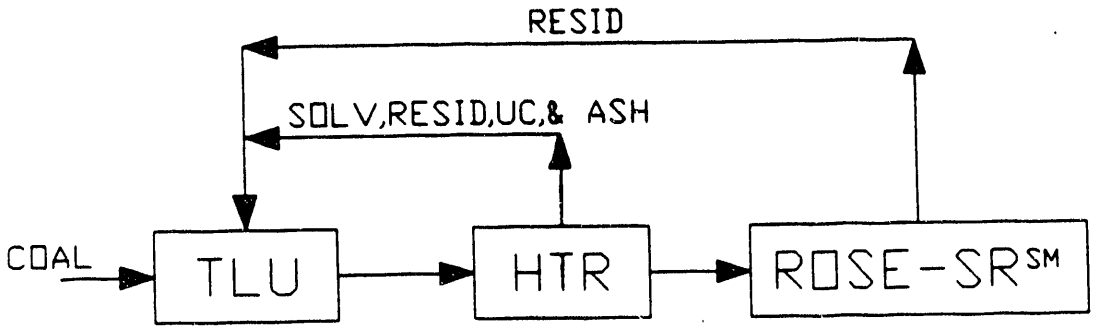
FIGURE 1. SIMPLIFIED FLOW DIAGRAM OF THE CLOSE-COUPLED ITSL COAL LIQUEFACTION SYSTEM WITH INTERSTAGE SEPARATION



INTEGRATED TWO-STAGE LIQUEFACTION

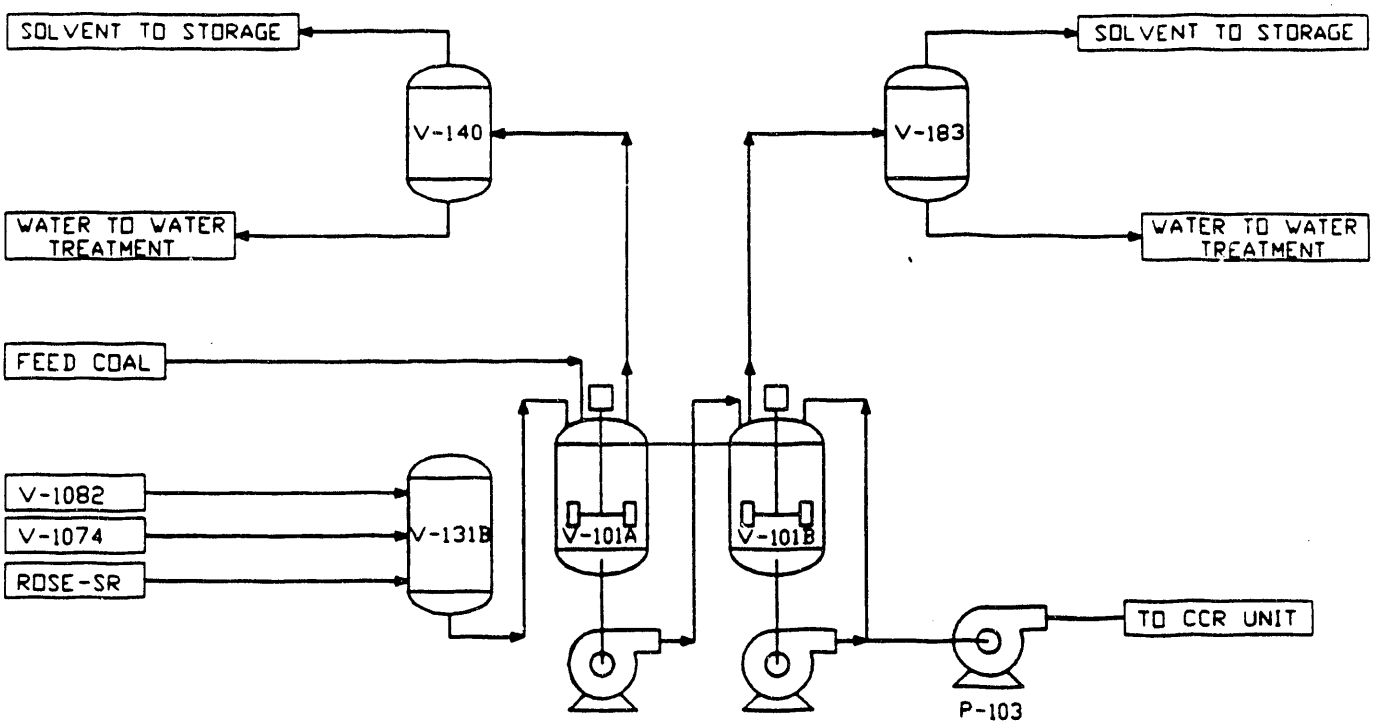


DOUBLE INTEGRATED TWO-STAGE LIQUEFACTION



CLOSE-COUPLED INTEGRATED TWO-STAGE LIQUEFACTION

FIGURE 2. LIQUEFACTION MODES TESTED SINCE RUN 242



V101A	SLURRY BLEND TANK
V101B	FEED TANK
V131B	RECYCLE PROCESS SOLVENT TANK
V140/V183	SLURRY DRYING SEPARATORS
P103	HIGH PRESSURE FEED PUMP

FIGURE 3. FLOW DIAGRAM OF COAL SLURRY PREPARATION SYSTEM

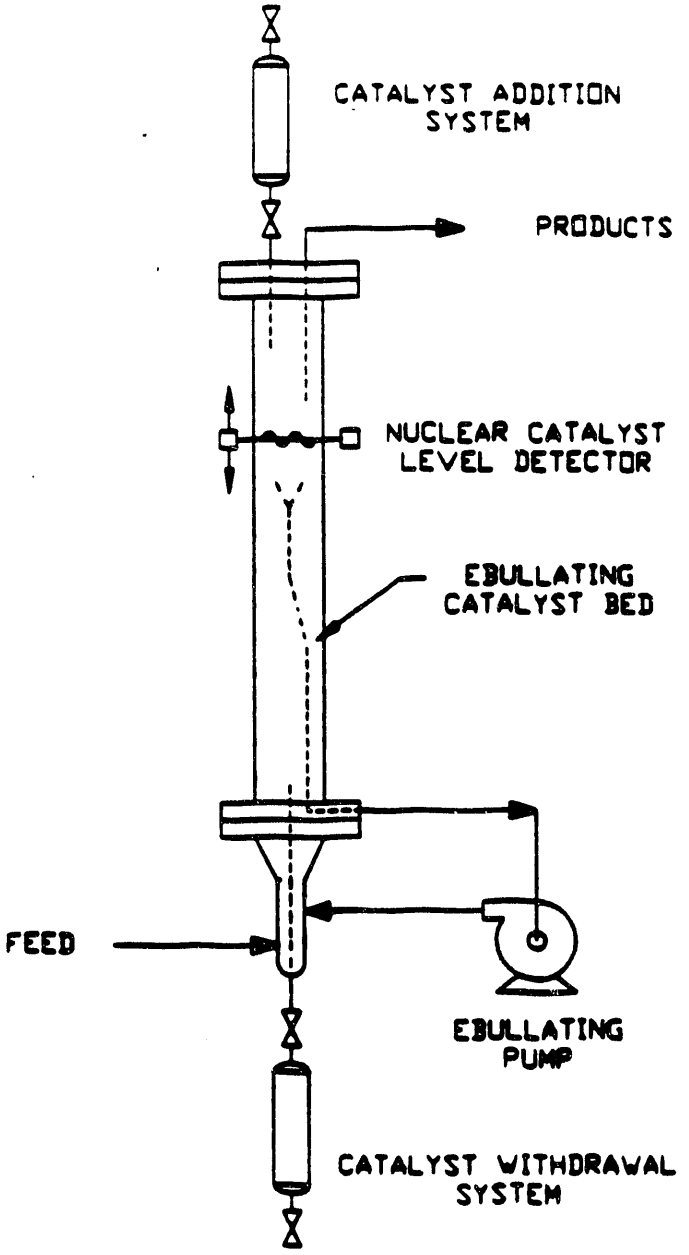


FIGURE 4. H-OIL® EBULLATED BED REACTOR

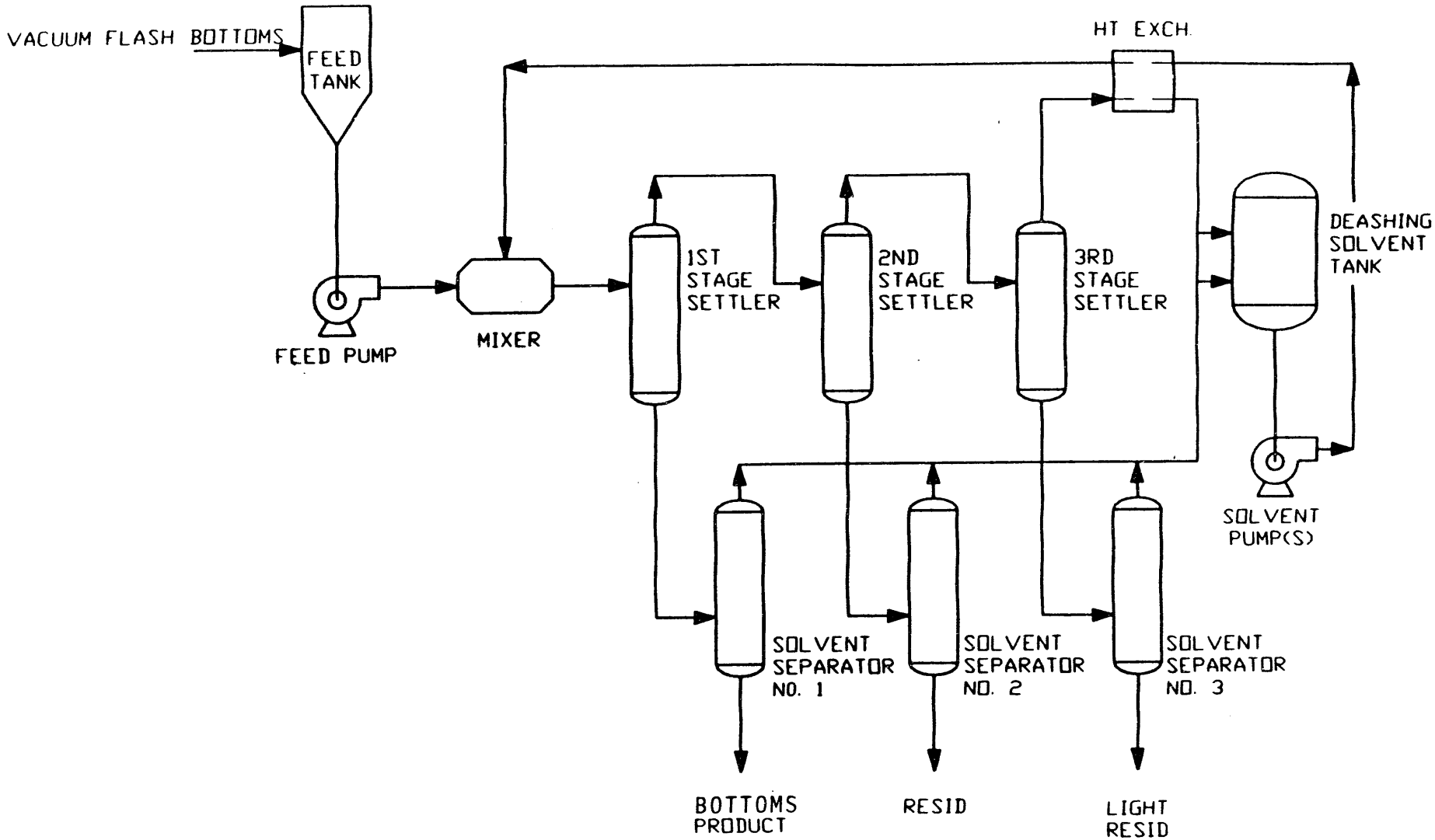


FIGURE 5. FLOW DIAGRAM OF THE RESIDUUM OIL SUPERCRITICAL EXTRACTION - SOLIDS REJECTION UNIT

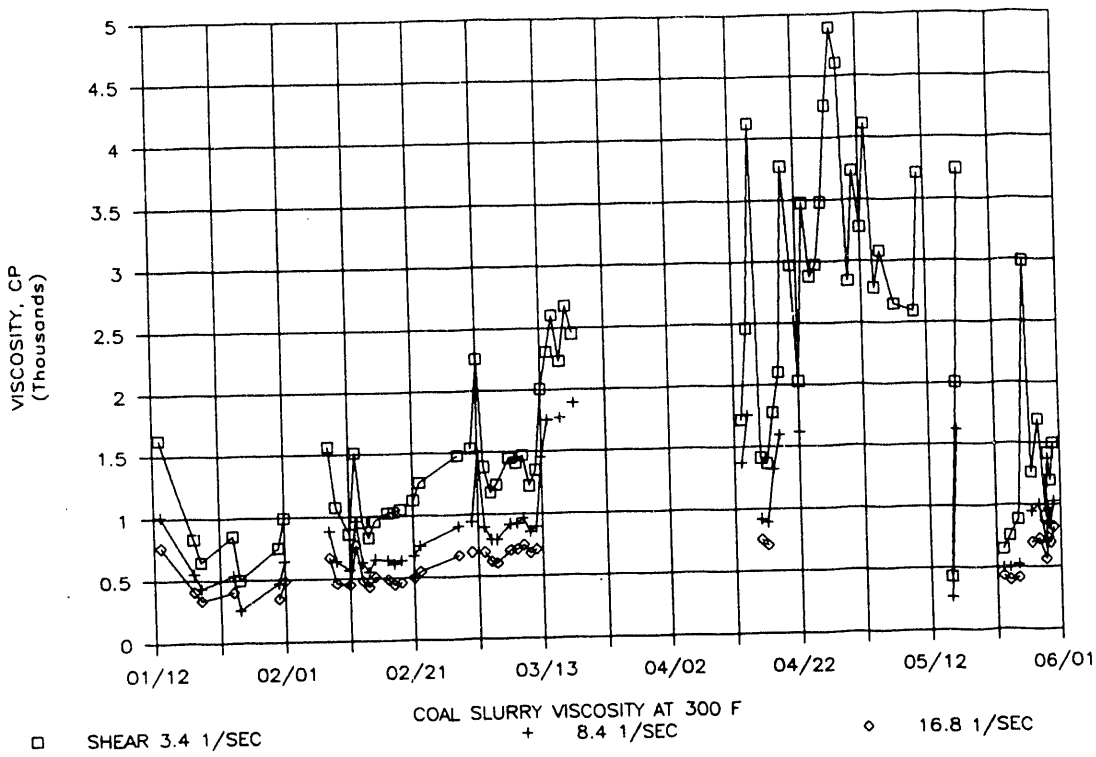


FIGURE 6. COAL SLURRY VISCOSITY AT 300°F

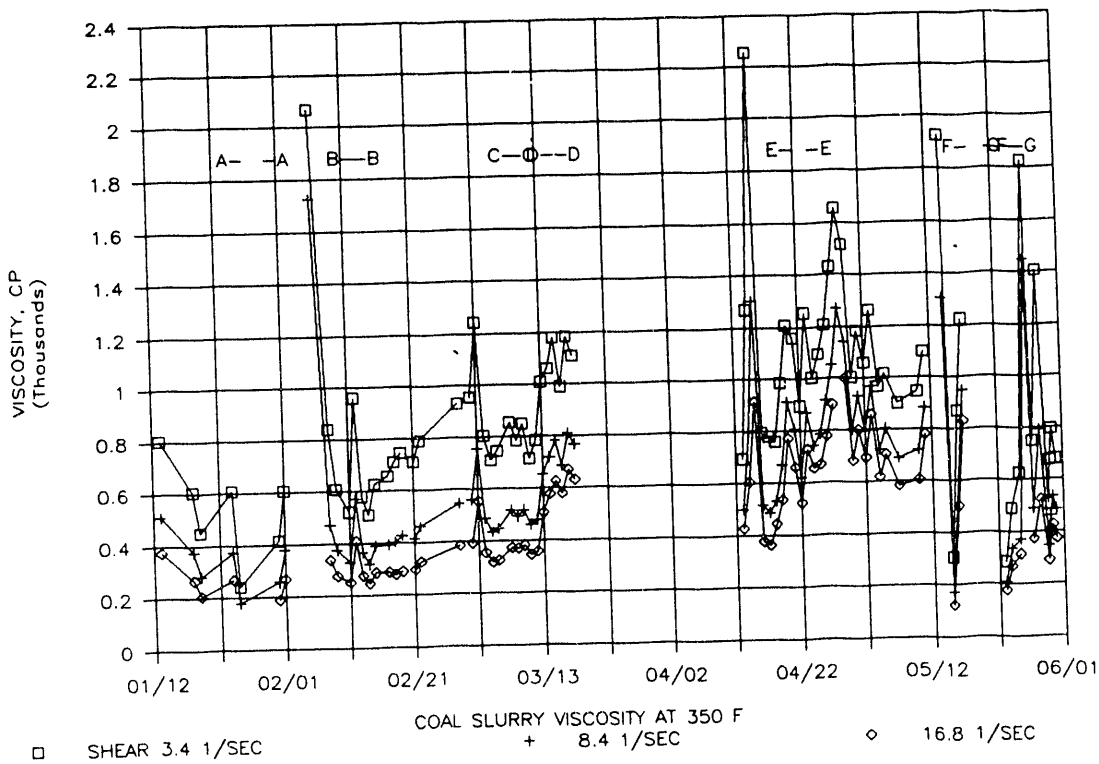
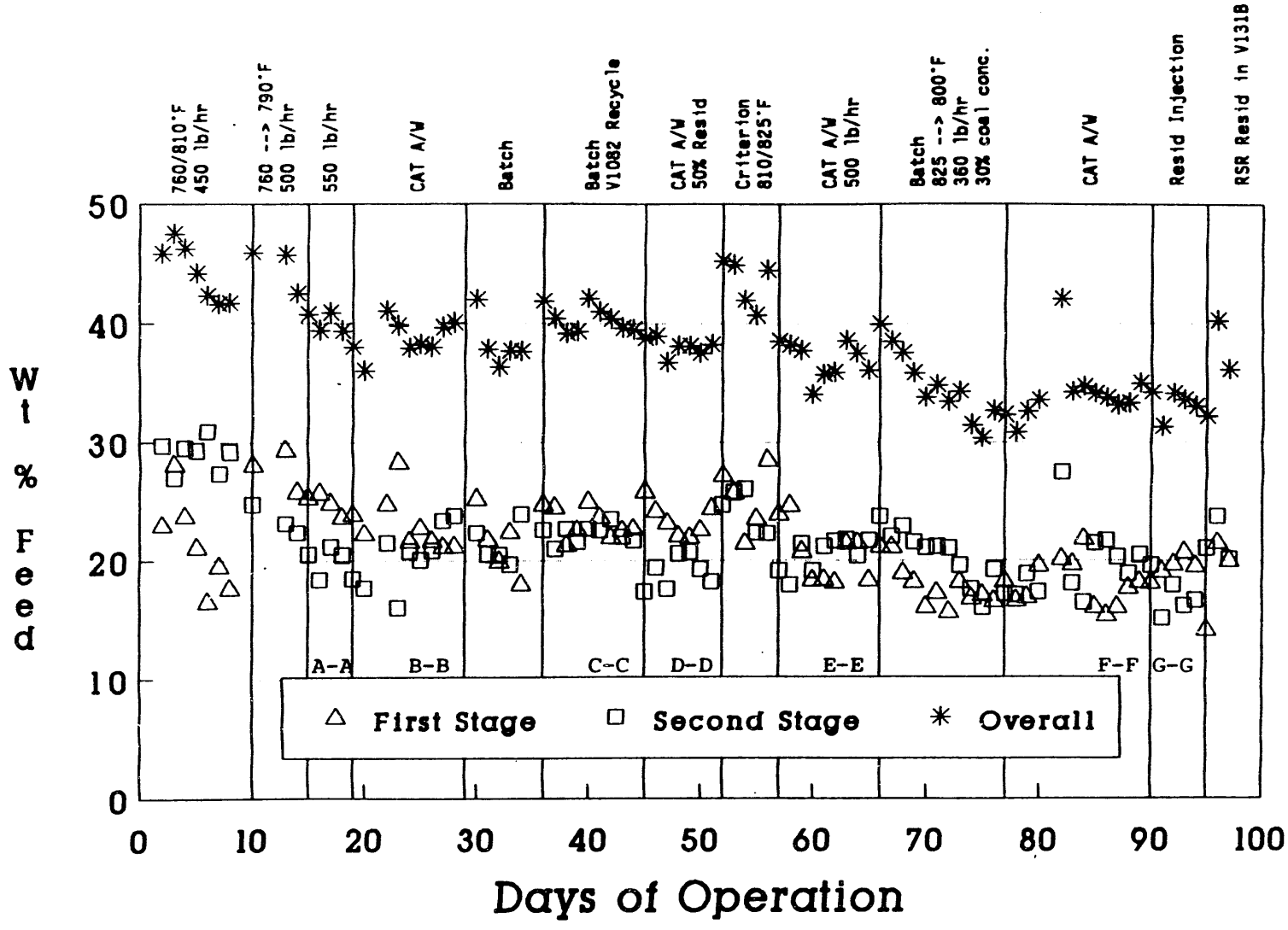
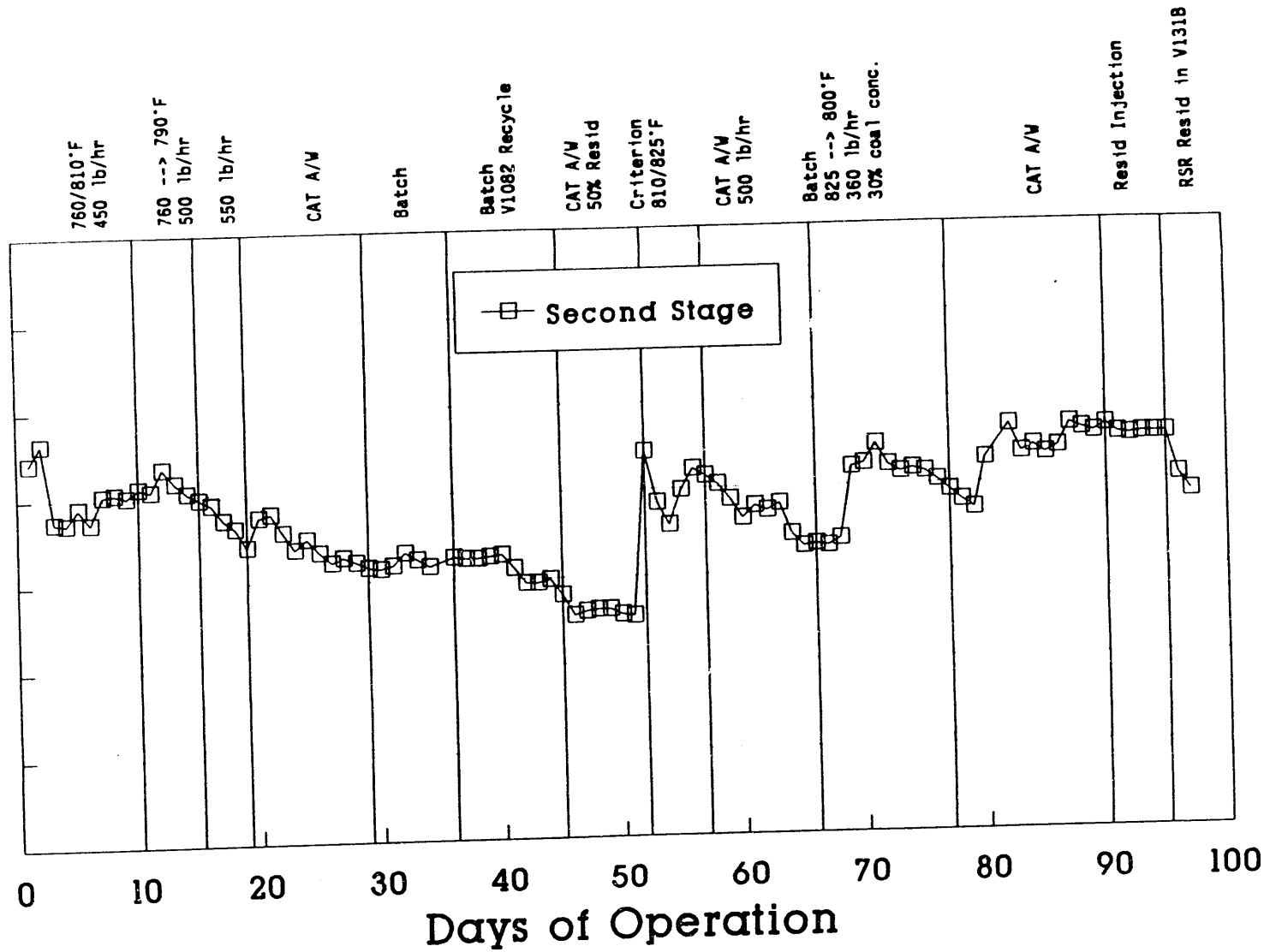


FIGURE 7. COAL SLURRY VISCOSITY AT 350°F



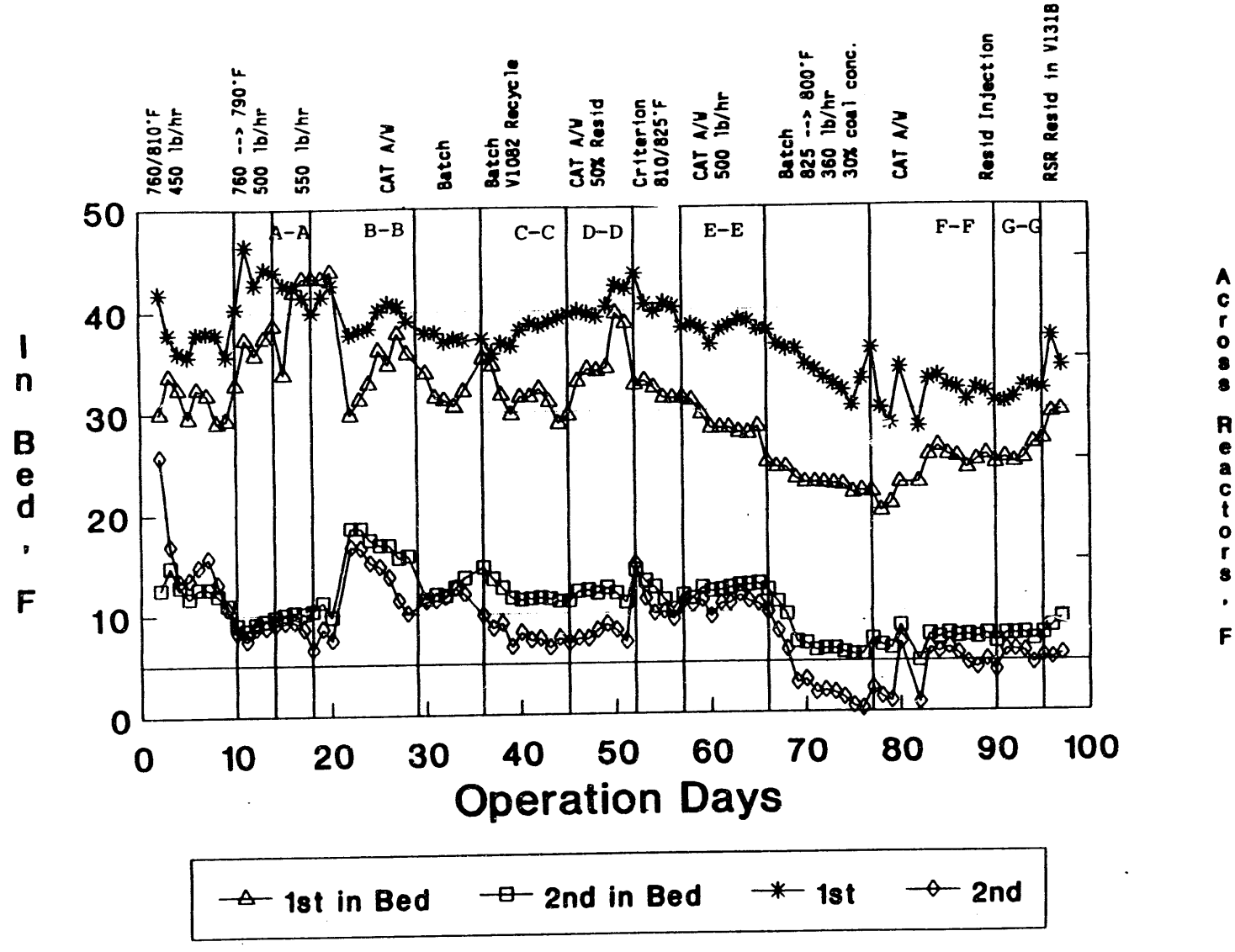
Day 10 = 1/23; 21 = 2/8; 30 = 2/21; 40 = 3/7; 50 = 3/17; 60 = 4/21; 70 = 5/1; 80 = 5/11; 90 = 5/23;

FIGURE 8. PHASE 2 RESID + UC CONVERSIONS



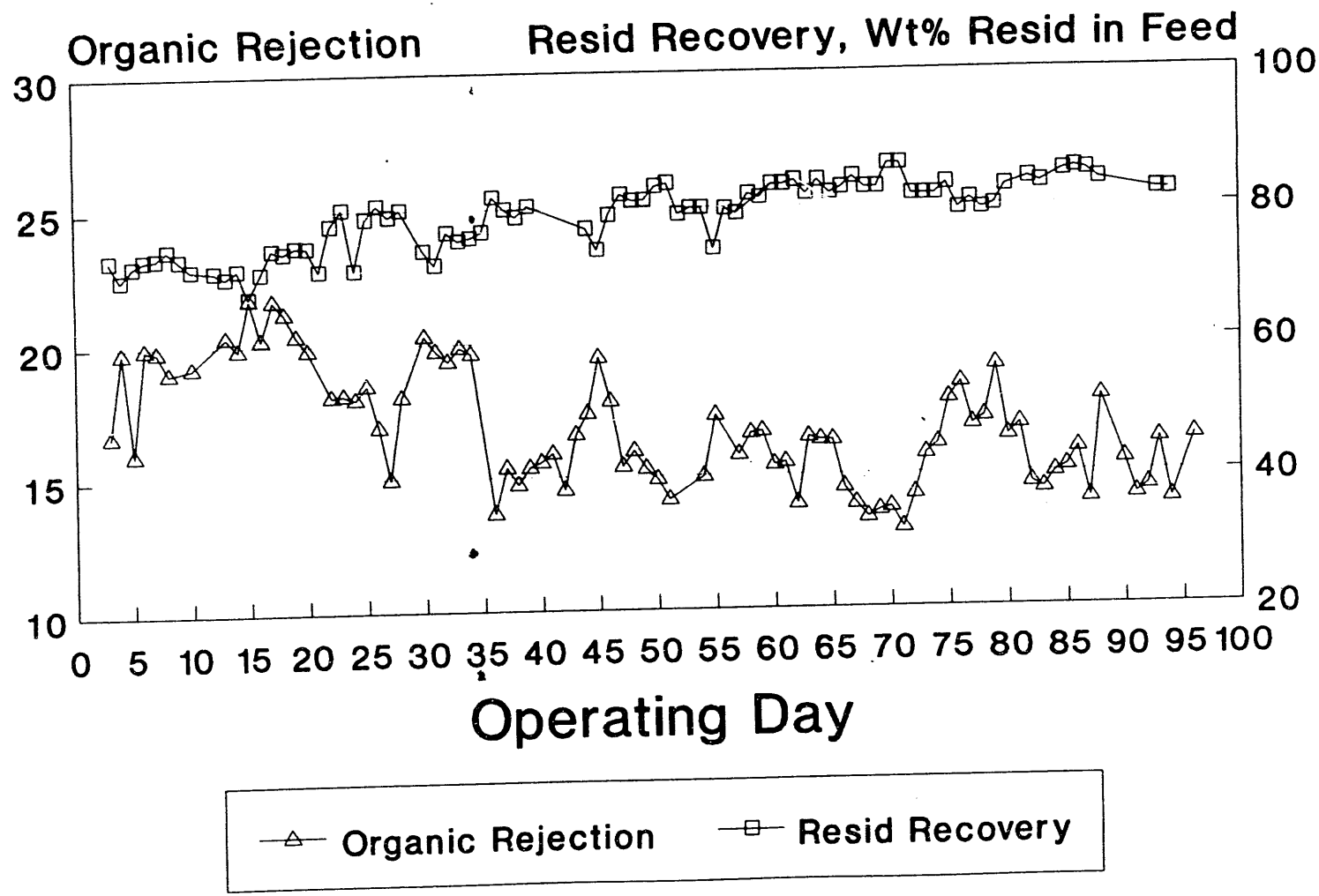
Day 10 = 1/23; 21 = 2/8; 30 = 2/21; 40 = 3/7; 50 = 3/17; 60 = 4/21; 70 = 5/1; 80 = 5/11; 90 = 5/23;

FIGURE 9. SECOND STAGE EBULLATION FLOW RATES DURING RUN 261. THE FIRST STAGE EBULLATION RATES ARE NOT REPRESENTED DUE TO MEASUREMENT PROBLEMS.



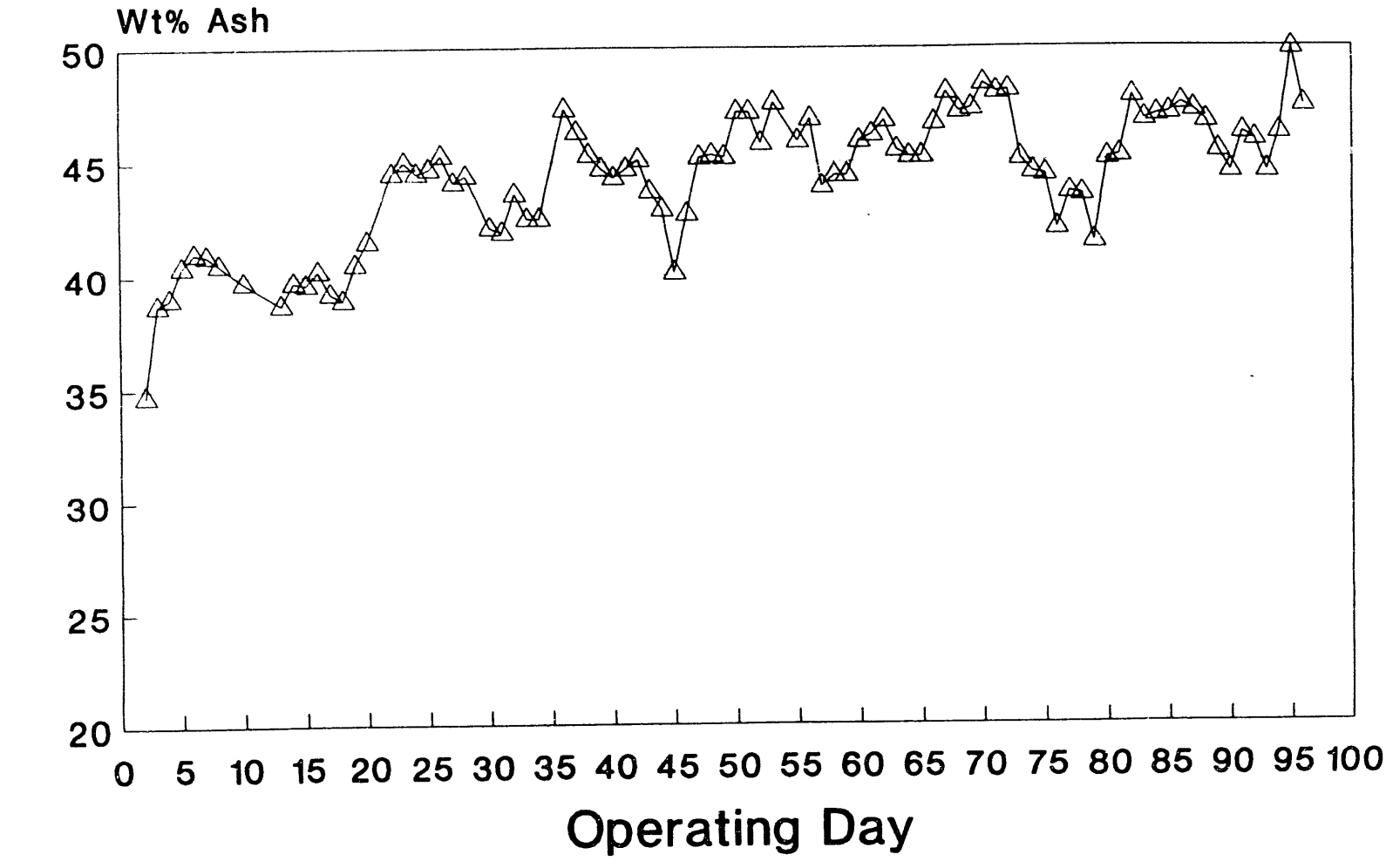
Day 10 = 1/23; 21 = 2/8; 30 = 2/21; 40 = 3/7; 50 = 3/17; 60 = 4/21; 70 = 5/1; 80 = 5/11; 90 = 5/23;

FIGURE 10. TEMPERATURE RISES IN REACTOR BEDS AND ACROSS THE REACTORS



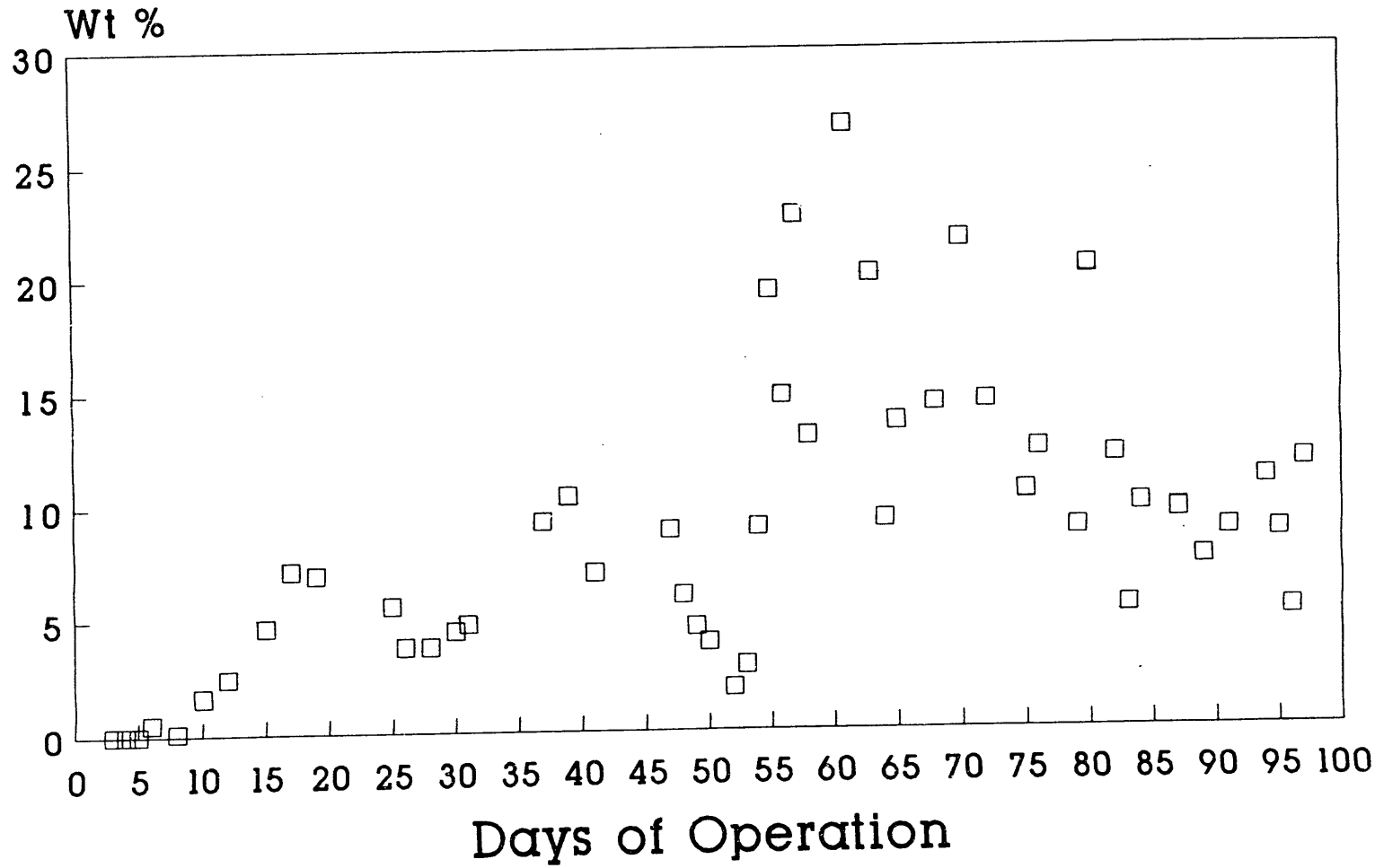
Day 10 = 1/23/91; Day 30 = 2/21/91
Day 50 = 3/17/91; Day 70 = 5/01/91
Day 90 = 5/24/91

FIGURE 11. ORGANIC REJECTION AND RESID RECOVERY IN THE ROSE-SR^{SM} UNIT



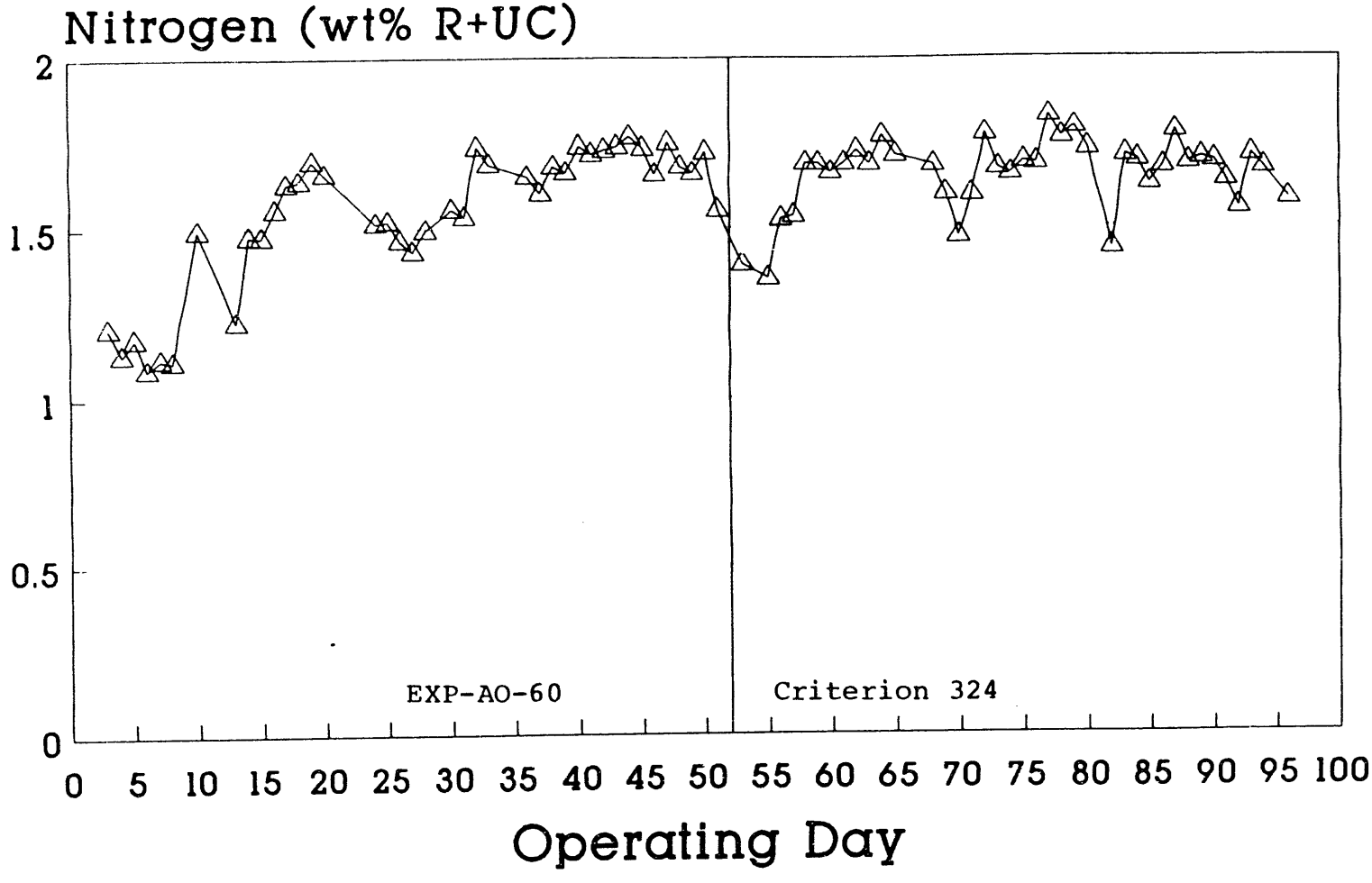
Day 10 = 1/23/91; Day 30 = 2/21/91
Day 50 = 3/17/91; Day 70 = 5/01/91
Day 90 = 5/24/91

FIGURE 12. ASH CONTENT IN BOTTOMS PRODUCT OF THE ROSE-SR^{sm} UNIT



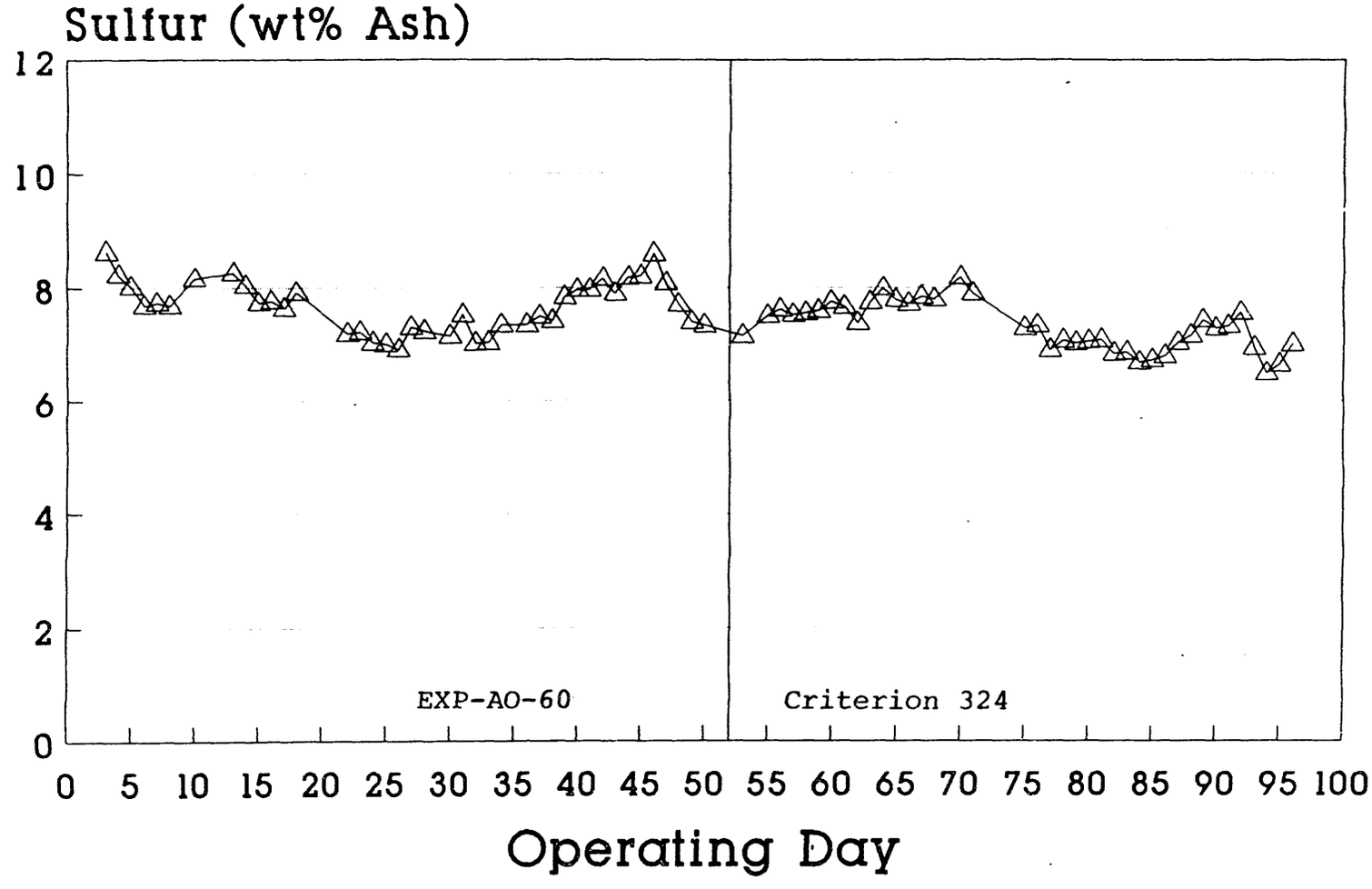
Day 10 = 1/23/91; Day 30 = 2/21/91
Day 50 = 3/17/91; Day 70 = 5/01/91
Day 90 = 5/24/91

FIGURE 13. PREASPHALTENES IN ROSE-SR^{SM} FEED



Day 10 = 1/23/91; Day 30 = 2/21/91
Day 50 = 3/17/91; Day 70 = 5/01/91
Day 90 = 5/24/91

FIGURE 14. ASH-FREE NITROGEN CONTENT IN ROSE-SR^{SM} BOTTOMS PRODUCT



Day 10 = 1/23/91; Day 30 = 2/21/91
Day 50 = 3/17/91; Day 70 = 5/01/91
Day 90 = 5/24/91

FIGURE 15. ORGANIC-FREE SULFUR IN THE ROSE-SR^{SM} BOTTOMS PRODUCT

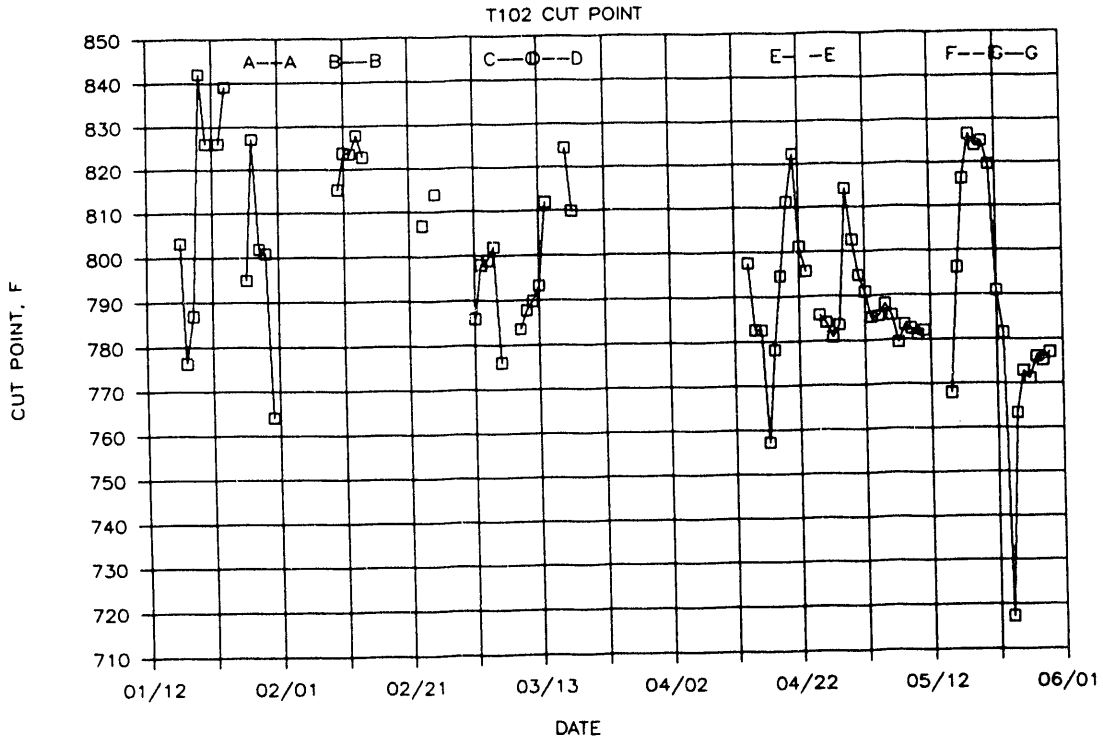


FIGURE 16. T102 VACUUM COLUMN CUT POINT BY GC

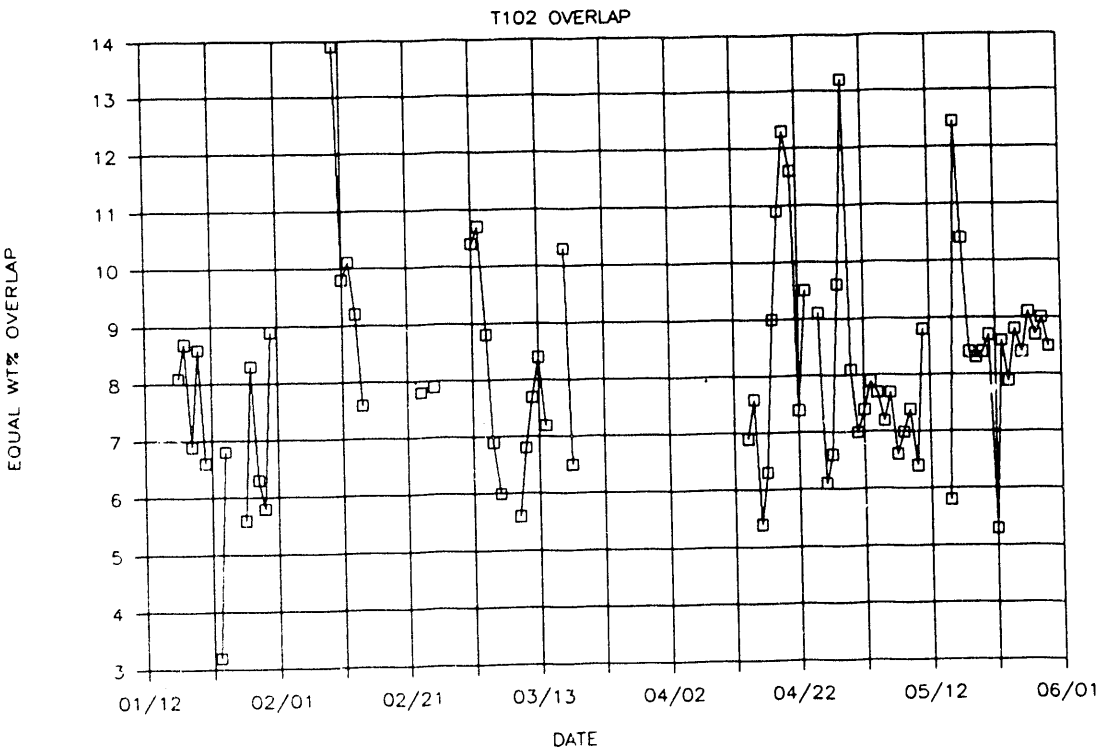


FIGURE 17. T102 VACUUM COLUMN OVERLAP BETWEEN OVERHEADS AND BOTTOMS

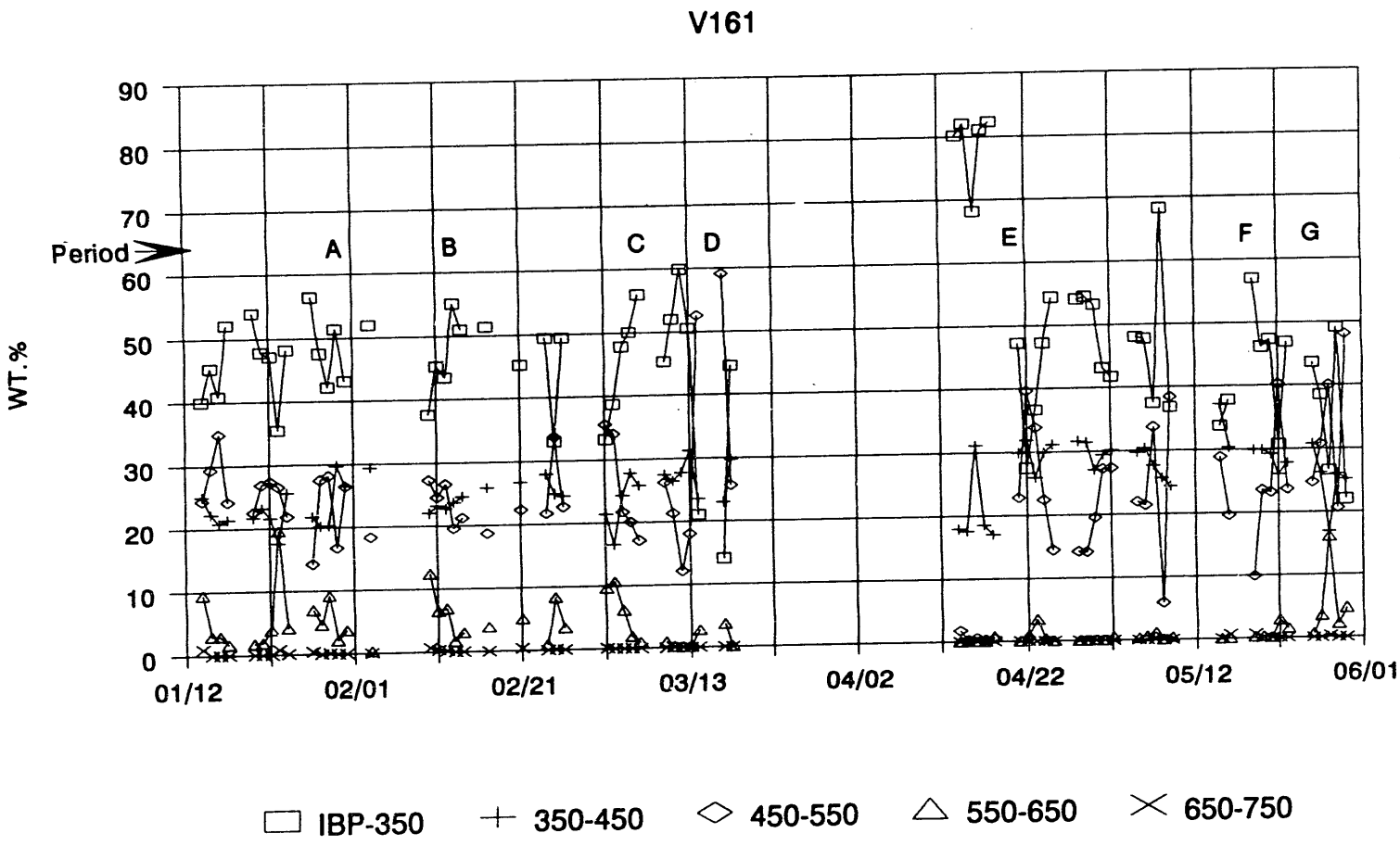


FIGURE 18. T105 OVERHEADS DISTRIBUTION

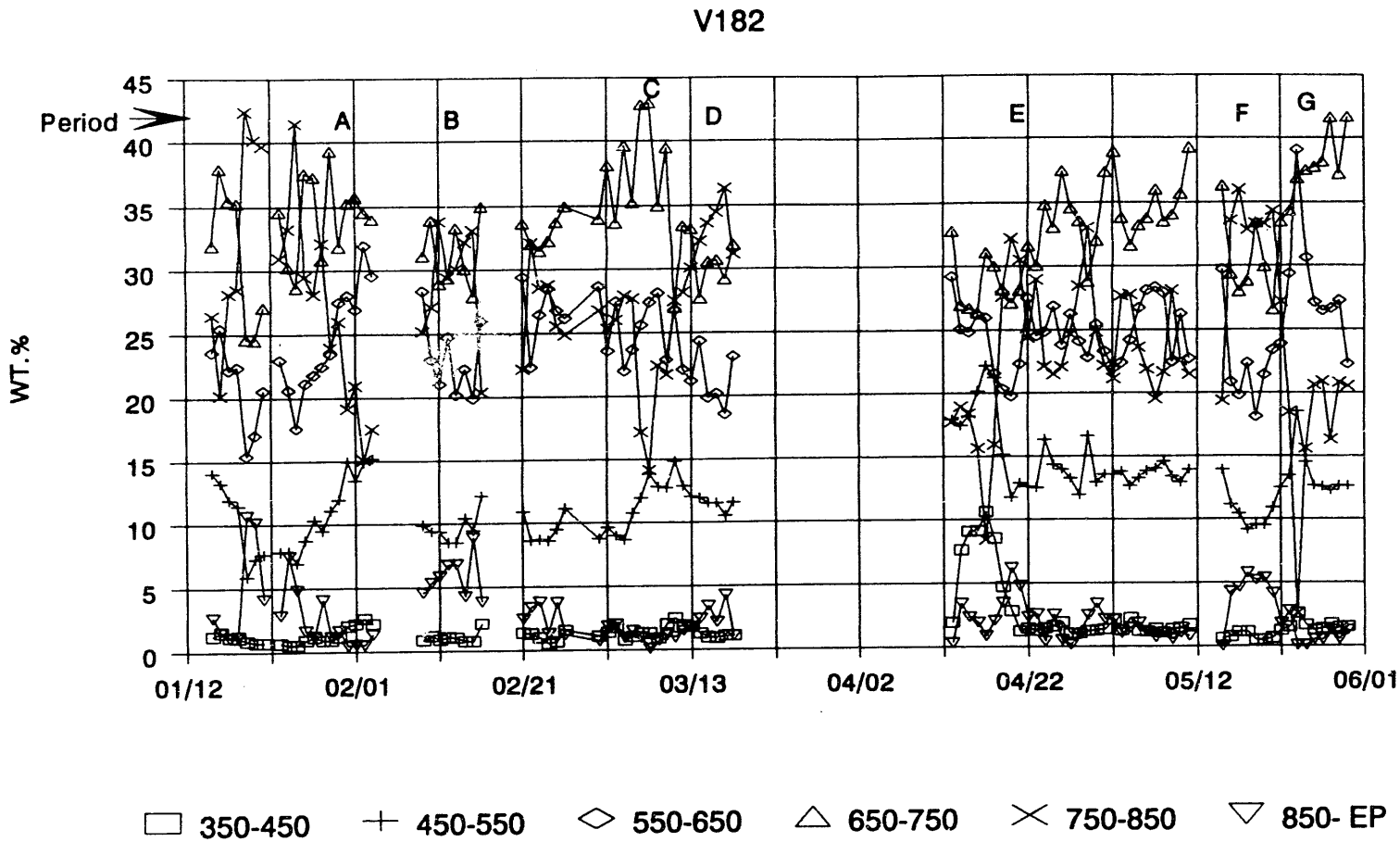


FIGURE 19. T102 OVERHEADS (V182) DISTRIBUTION

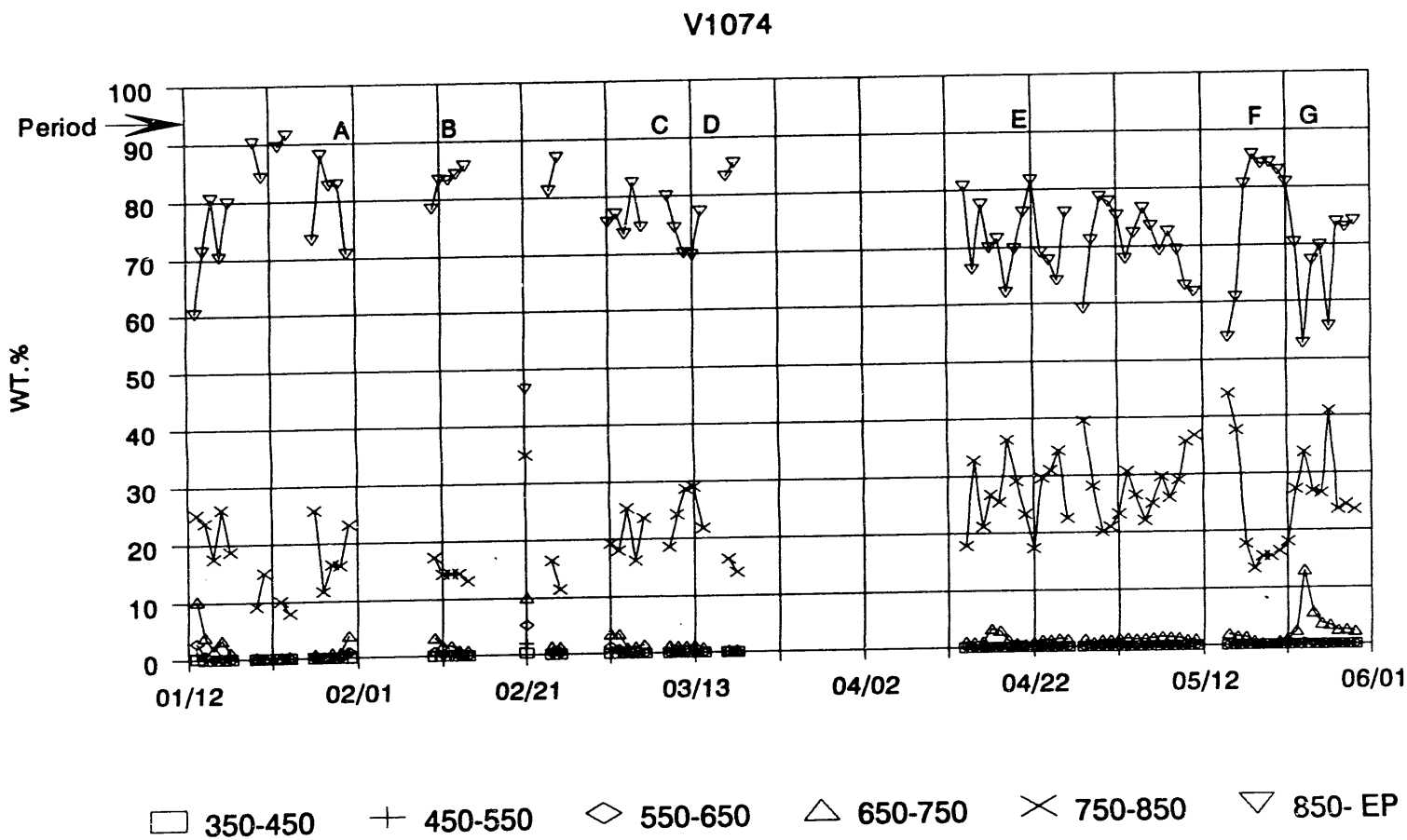


FIGURE 20. T102 BOTTOMS DISTRIBUTION

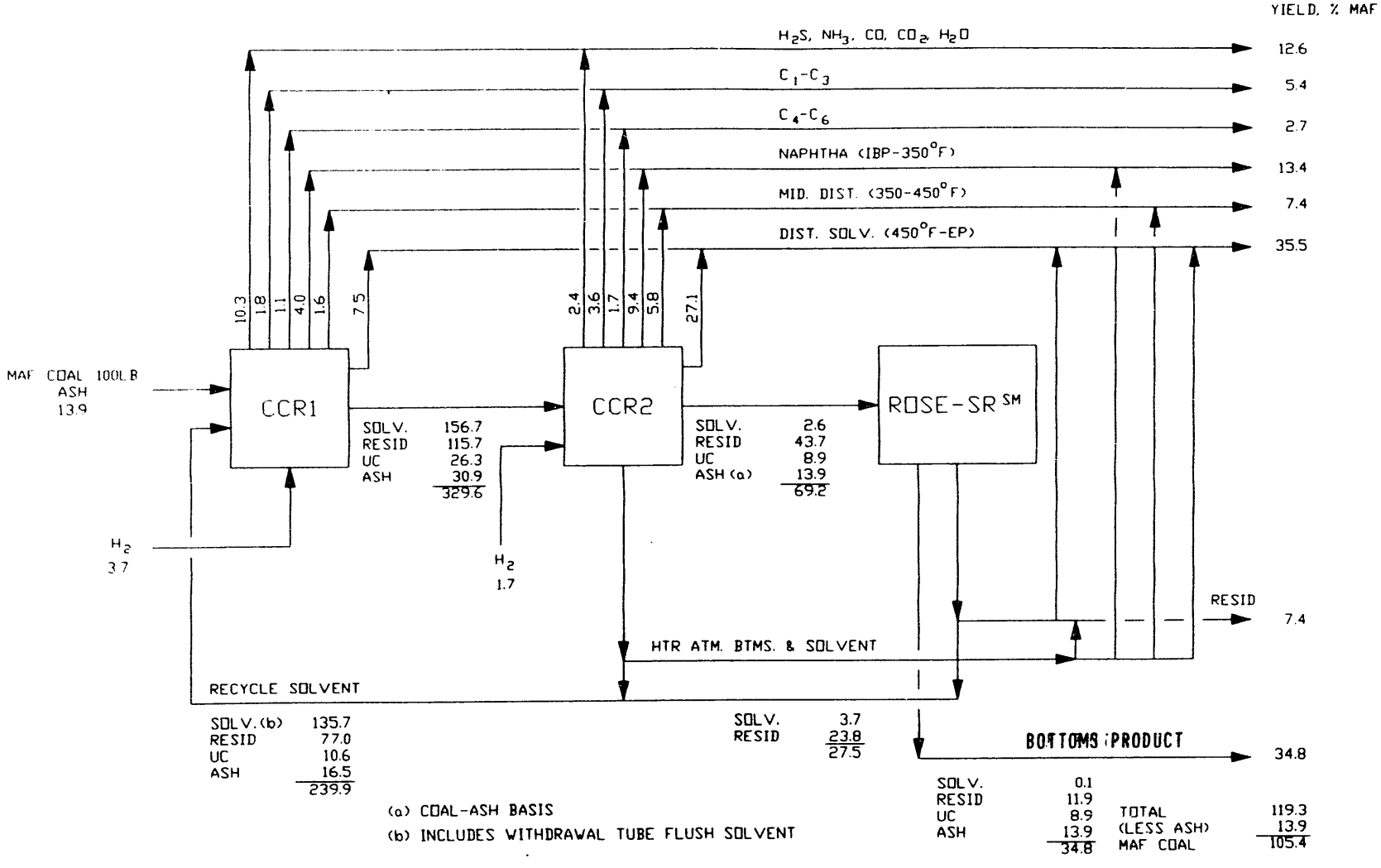


FIGURE 21. MATERIAL BALANCE FLOW DIAGRAM FOR PERIOD 261A (TRANSITIONAL)

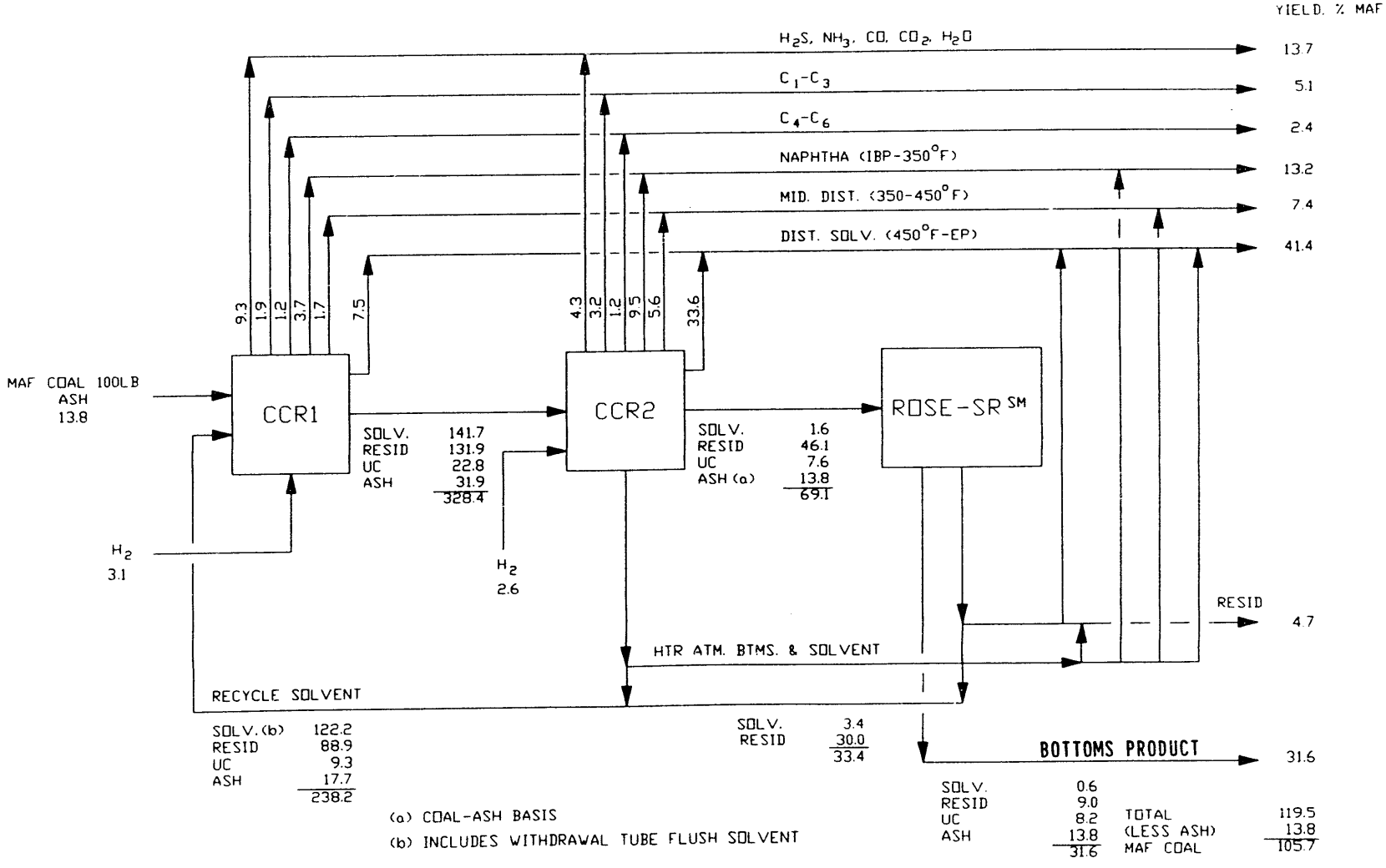


FIGURE 22. MATERIAL BALANCE FLOW DIAGRAM FOR PERIOD 261B

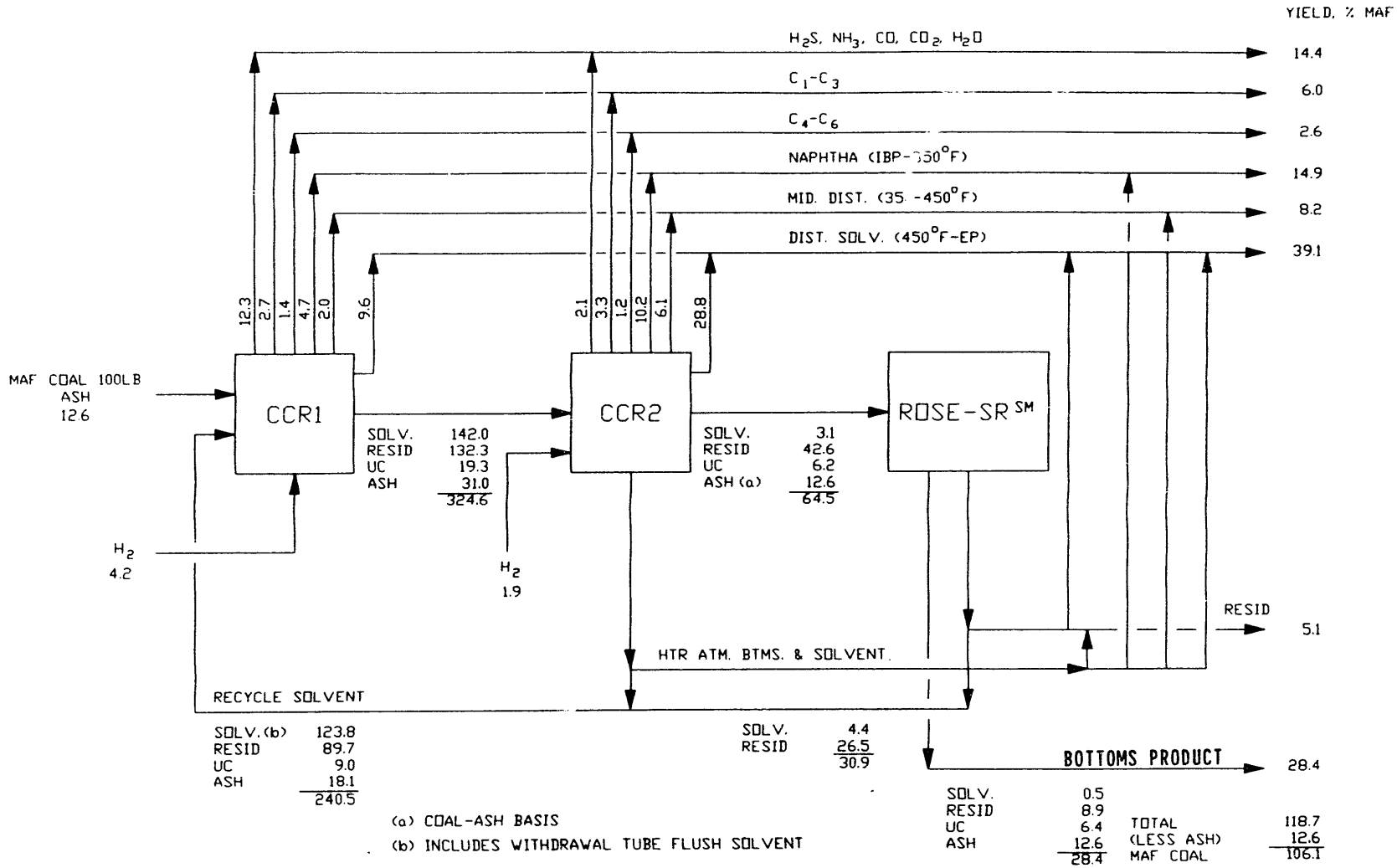


FIGURE 23. MATERIAL BALANCE FLOW DIAGRAM FOR PERIOD 261C

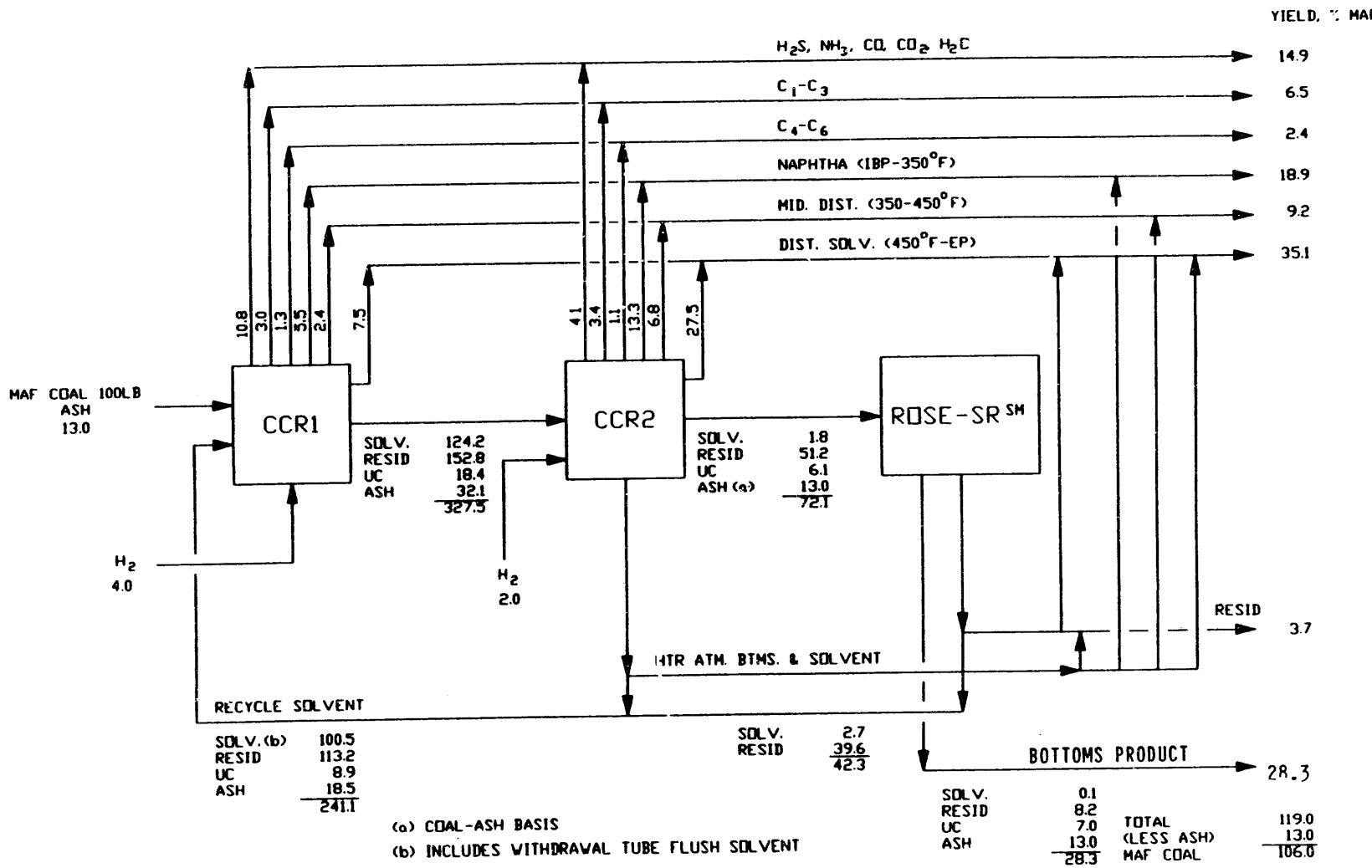


FIGURE 24. MATERIAL BALANCE FLOW DIAGRAM FOR PERIOD 261D

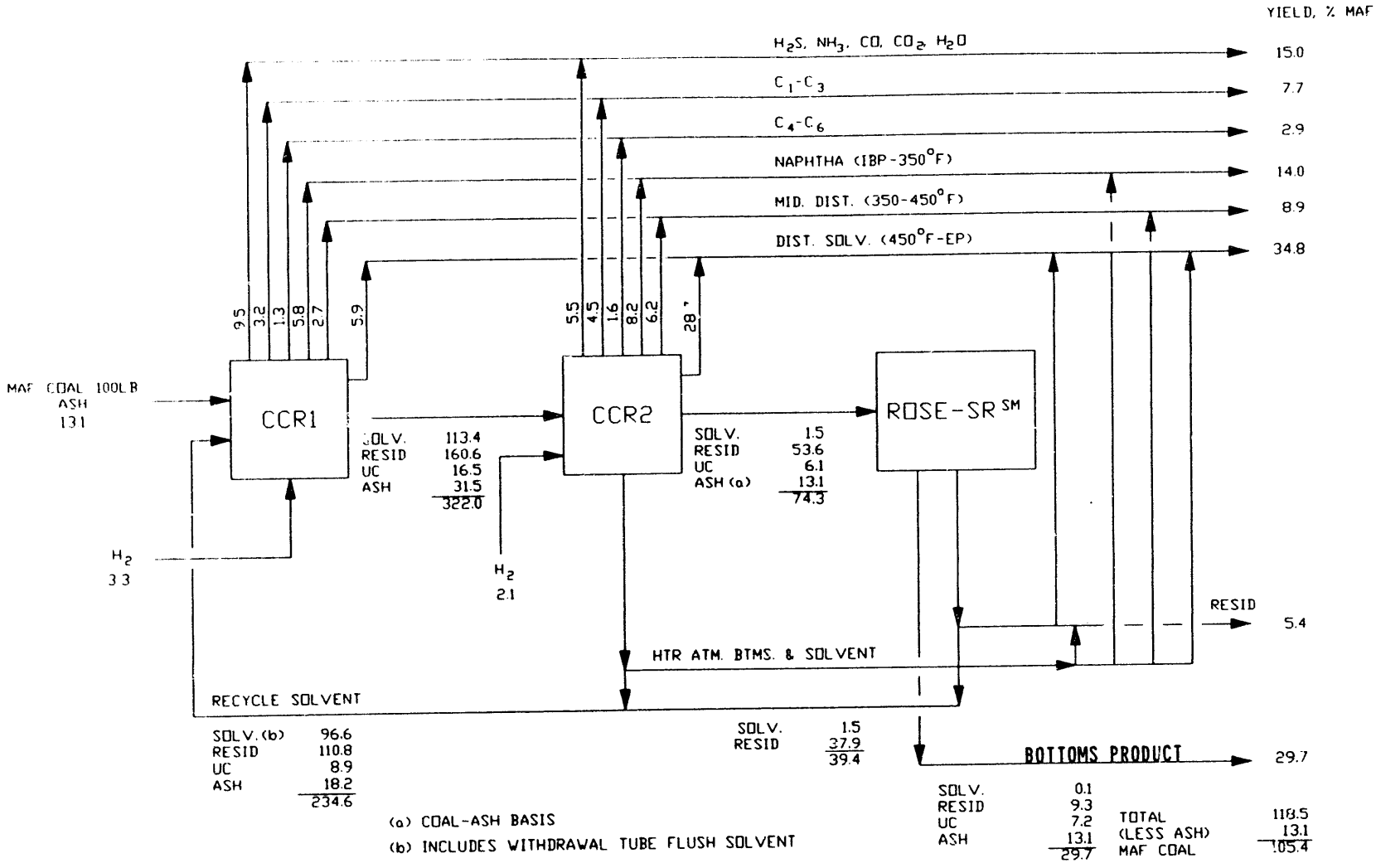


FIGURE 25. MATERIAL BALANCE FLOW DIAGRAM FOR PERIOD 261E

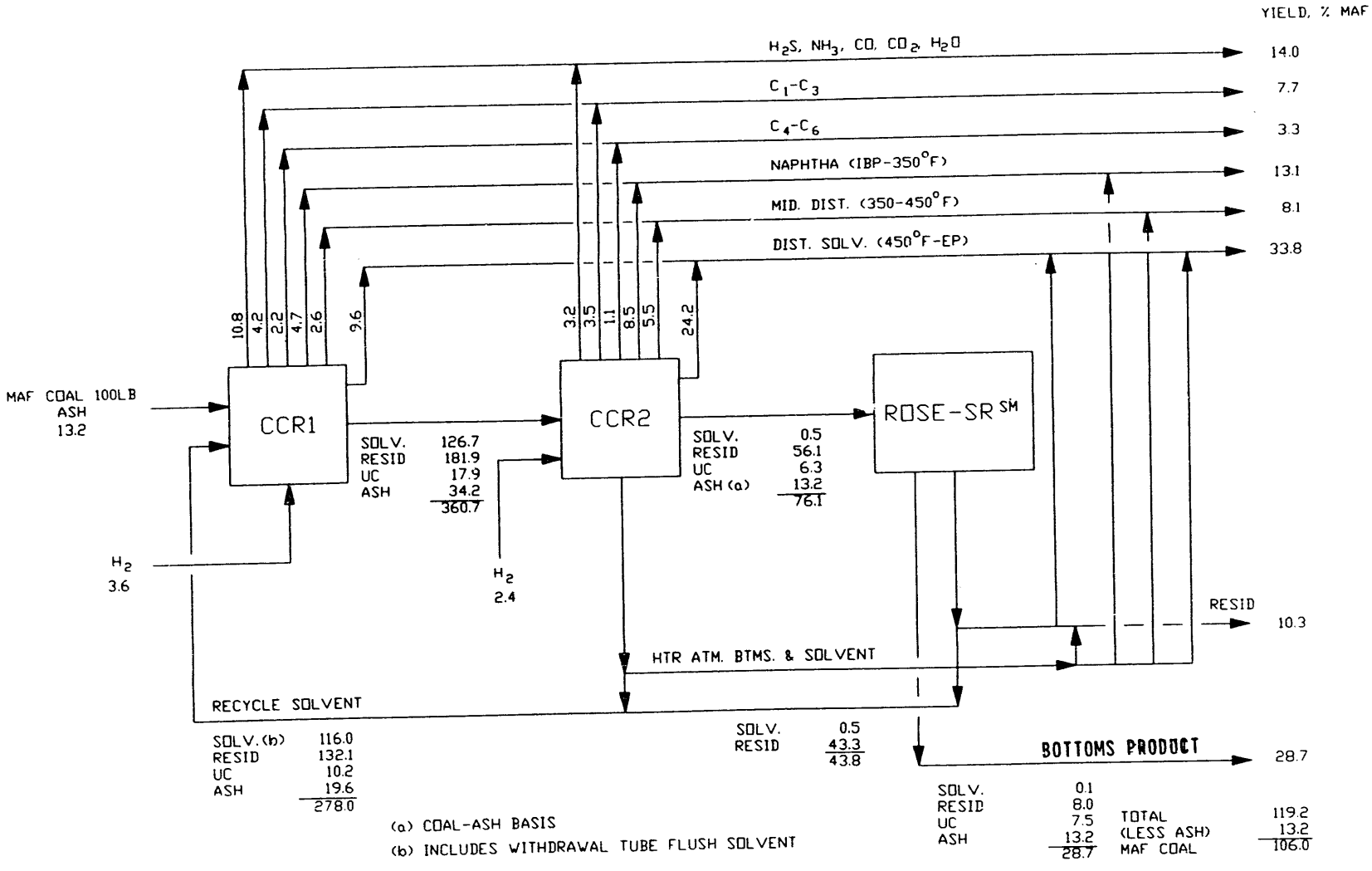


FIGURE 26. MATERIAL BALANCE FLOW DIAGRAM FOR PERIOD 261F (UNSTABLE)

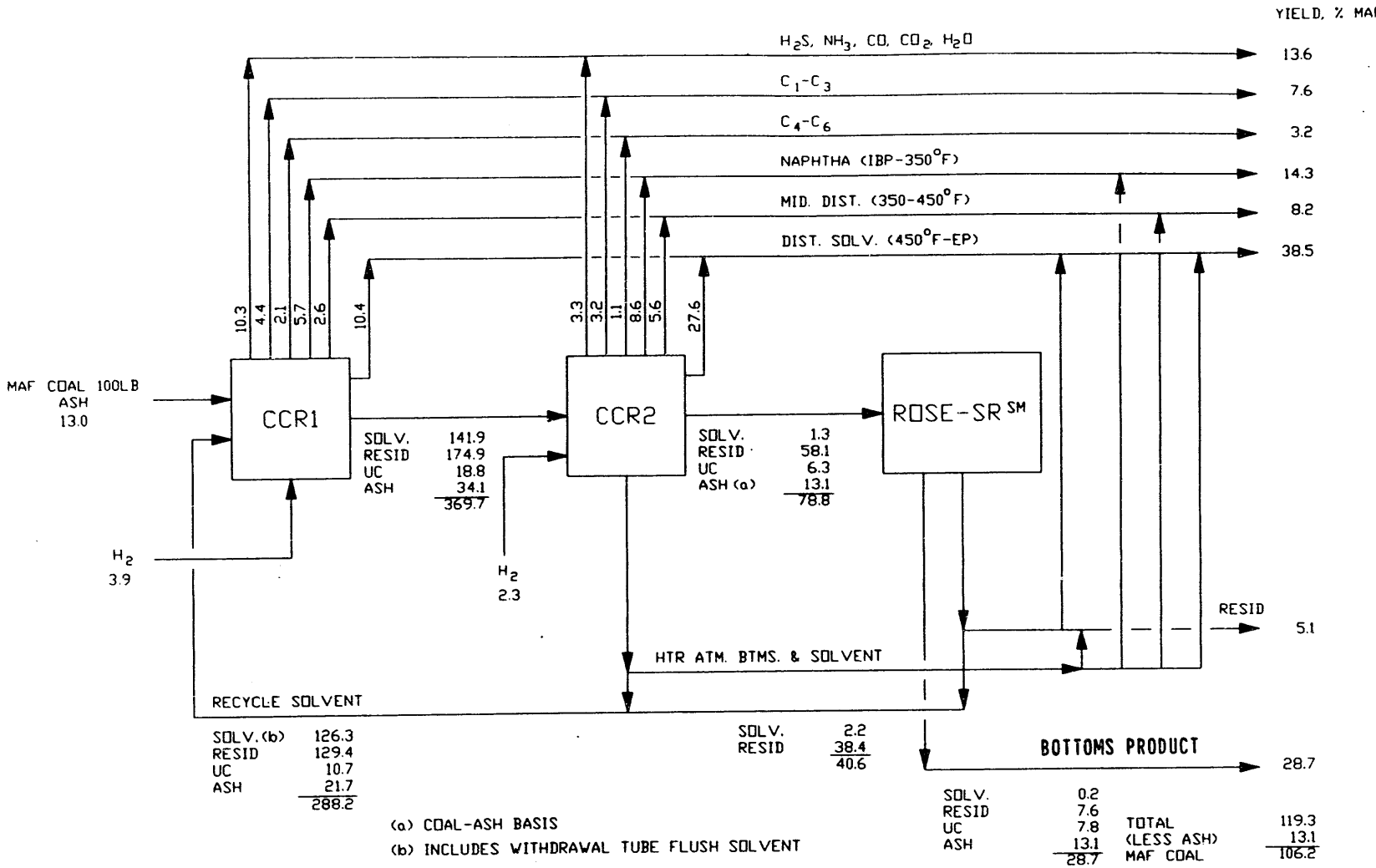
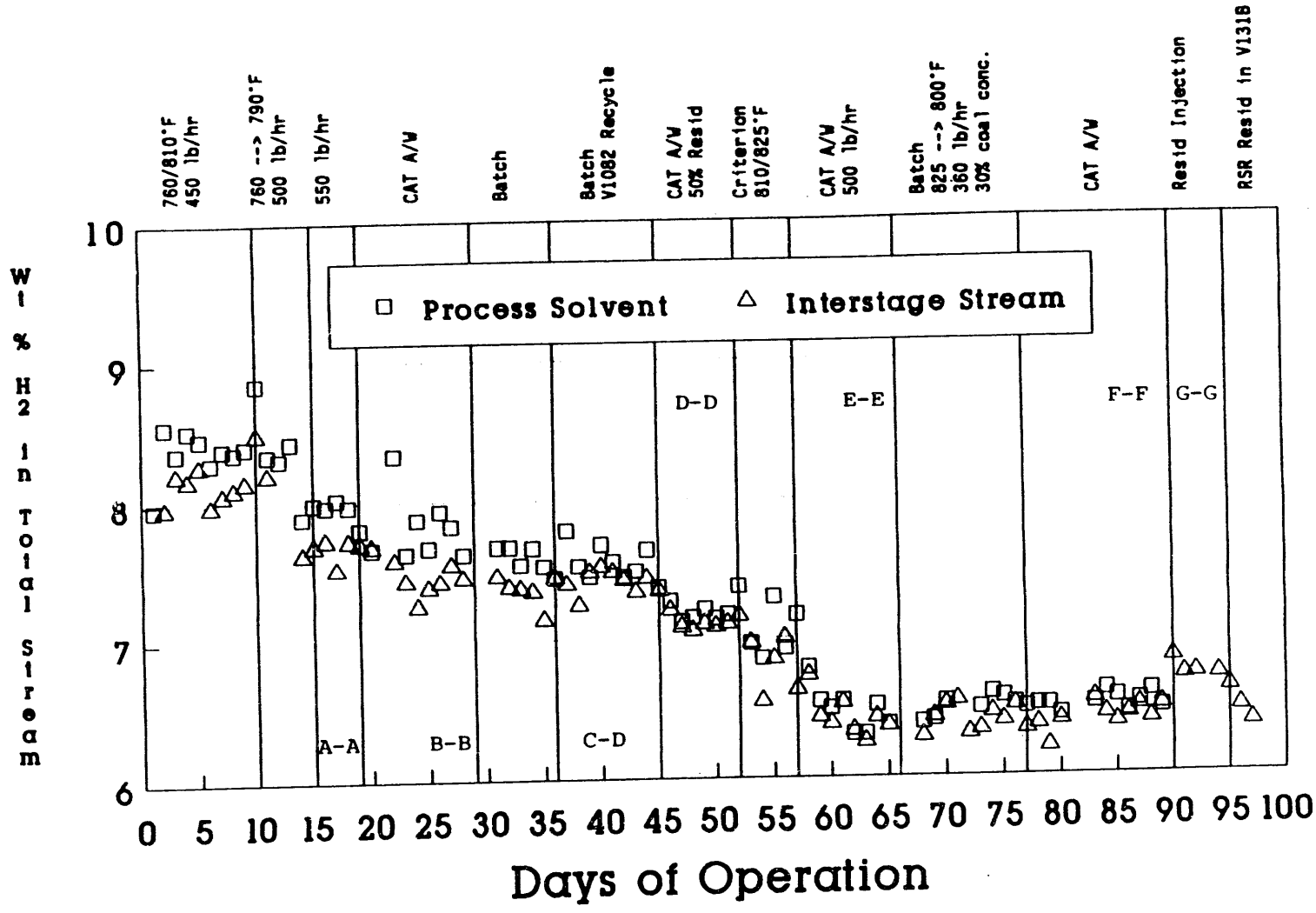
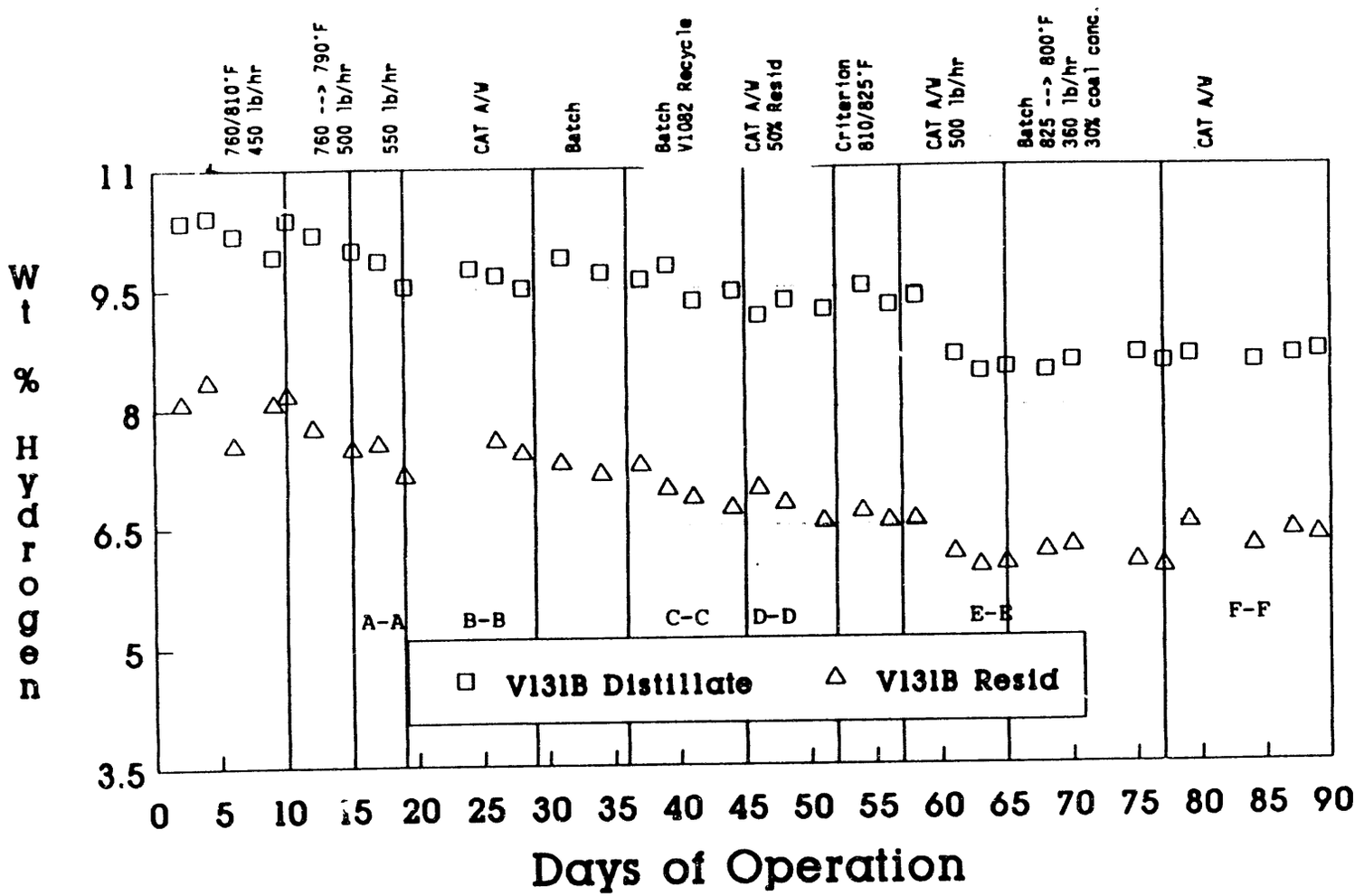


FIGURE 27. MATERIAL BALANCE FLOW DIAGRAM FOR PERIOD 261G (TRANSITIONAL)



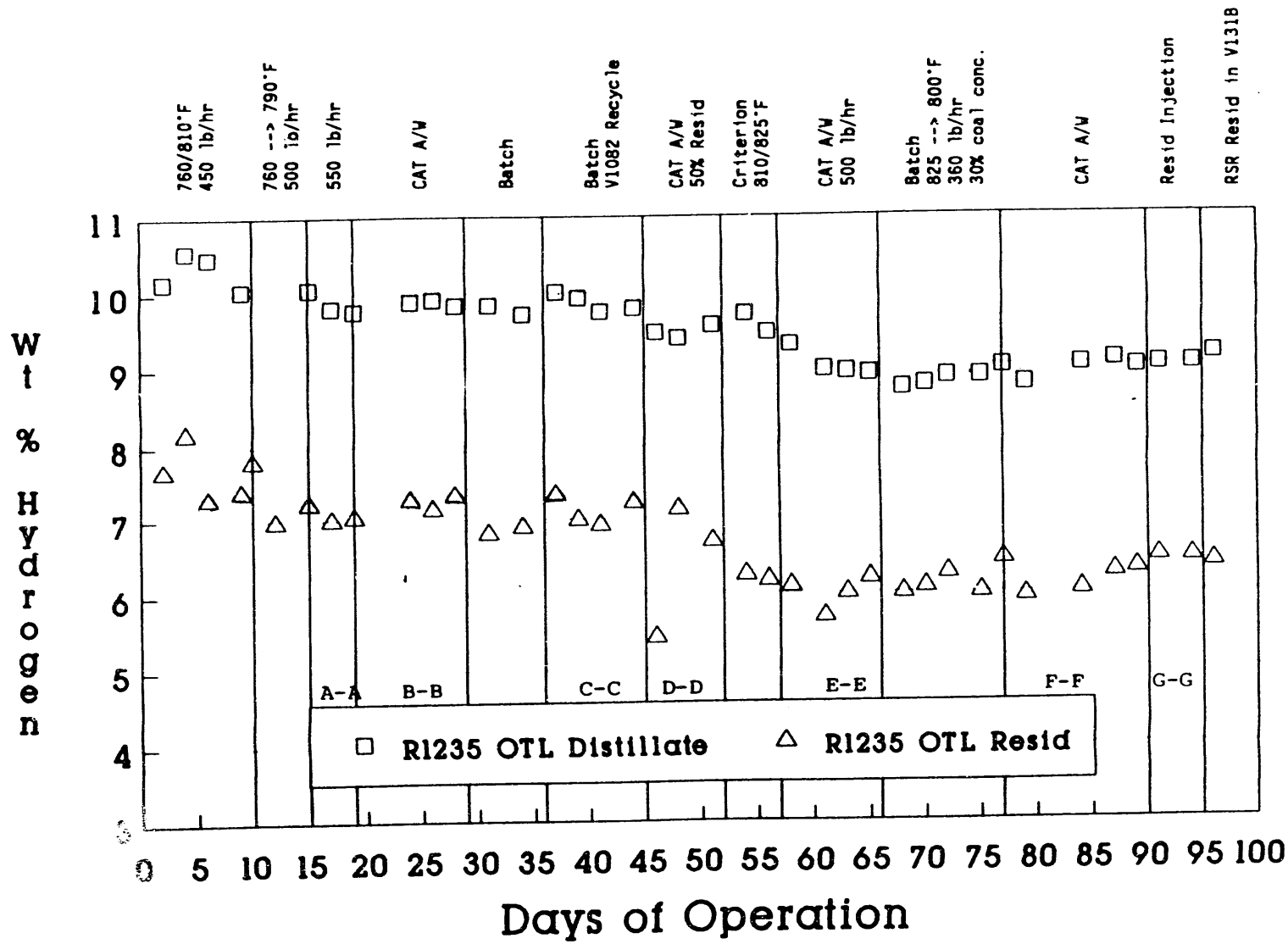
Day 10 = 1/23; 21 = 2/8; 30 = 2/21; 40 = 3/7; 50 = 3/17; 60 = 4/21; 70 = 5/1;
80 = 5/11; 90 = 5/23;

FIGURE 28. HYDROGEN CONTENT IN TOTAL PROCESS SOLVENT AND INTERSTAGE STREAMS



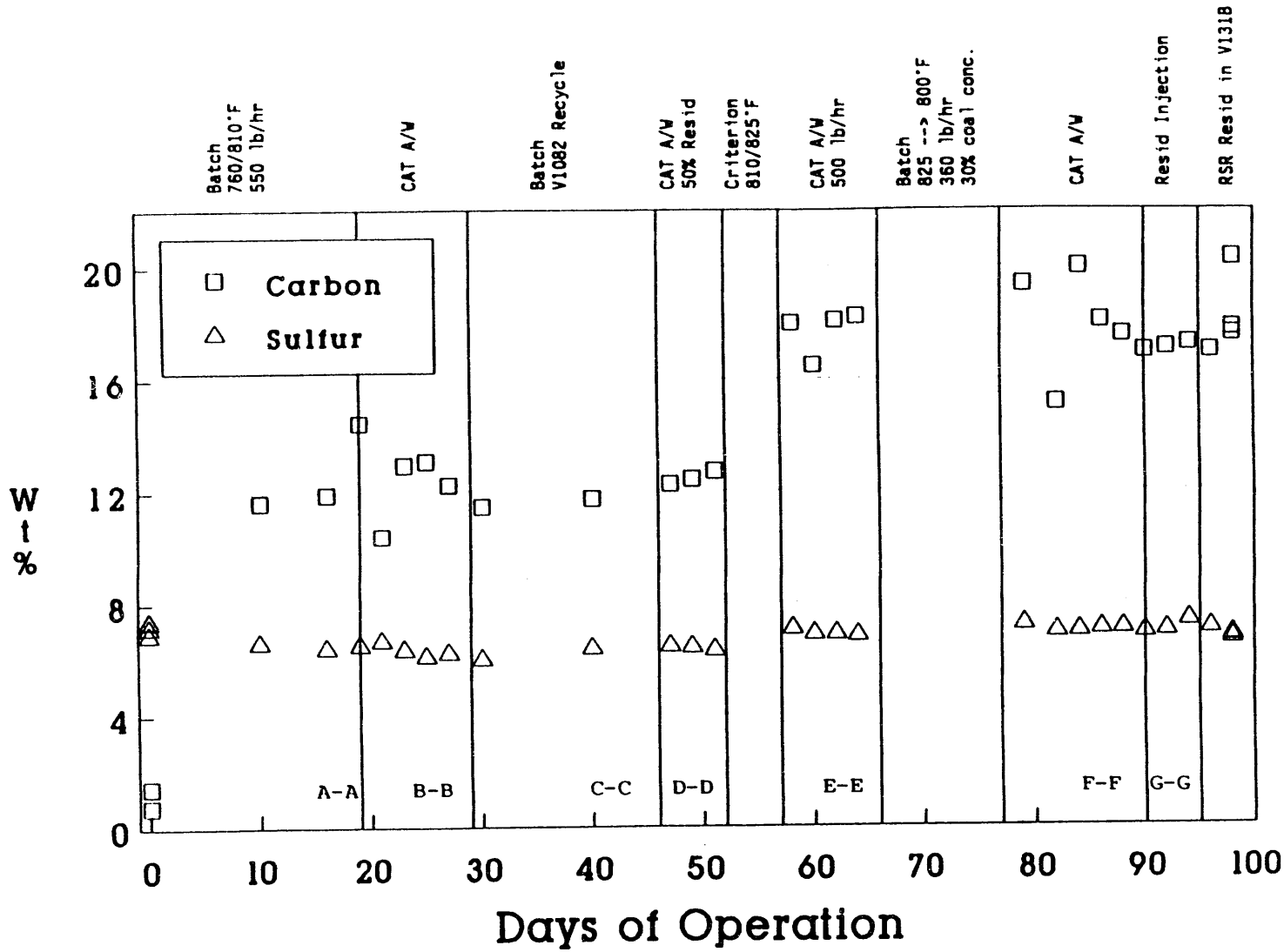
Day 10 - 1/23; 21 - 2/8; 30 - 2/21; 40 - 3/7; 50 - 3/17; 60 - 4/21; 70 - 5/1;
80 - 5/11; 90 - 5/23;

FIGURE 29. HYDROGEN CONTENT IN THE DISTILLATE AND RESID PORTION OF THE PROCESS SOLVENT



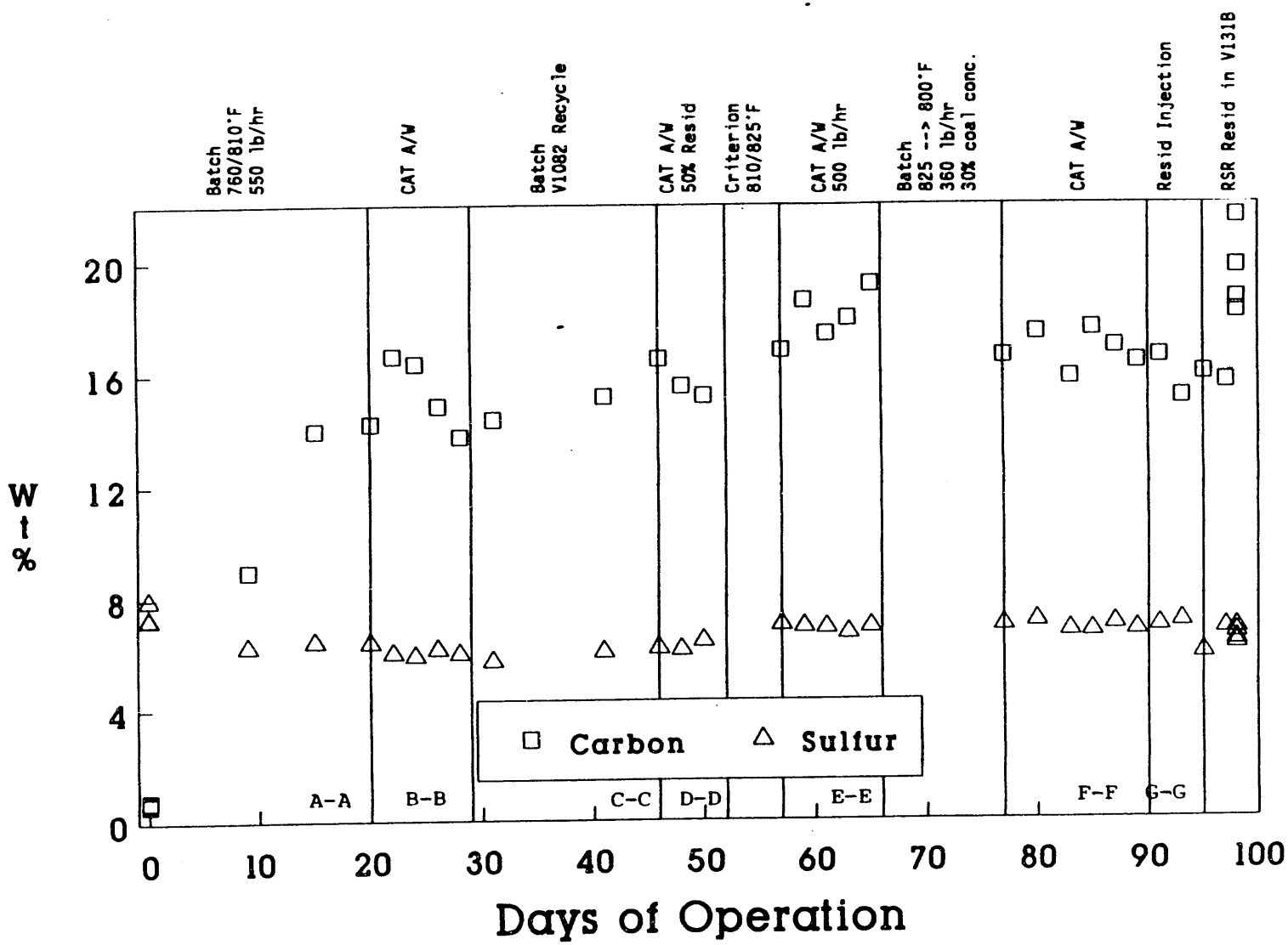
Day 10 = 1/23; 21 = 2/8; 30 = 2/21; 40 = 3/7; 50 = 3/17; 60 = 4/21; 70 = 5/1;
80 = 5/11; 90 = 5/23;

FIGURE 30. HYDROGEN CONTENT IN THE DISTILLATE AND RESID PORTIONS OF THE INTERSTAGE STREAM



Day 10 = 1/23; 21 = 2/8; 30 = 2/21; 40 = 3/7; 50 = 3/17; 60 = 4/21; 70 = 5/1; 80 = 5/11; 90 = 5/23;

FIGURE 31. FIRST STAGE THF EXTRACTION CARBON AND SULFUR DEPOSITS ON EXP-AO-60 AND CRITERION CATALYST



Day 10 = 1/23; 21 = 2/8; 30 = 2/21; 40 = 3/7; 50 = 3/17; 60 = 4/21; 70 = 5/1; 80 = 5/11; 90 = 5/23;

FIGURE 32. SECOND STAGE THF EXTRACTED CARBON AND SULFUR DEPOSITS ON EXP-AO-60 AND CRITERION CATALYSTS

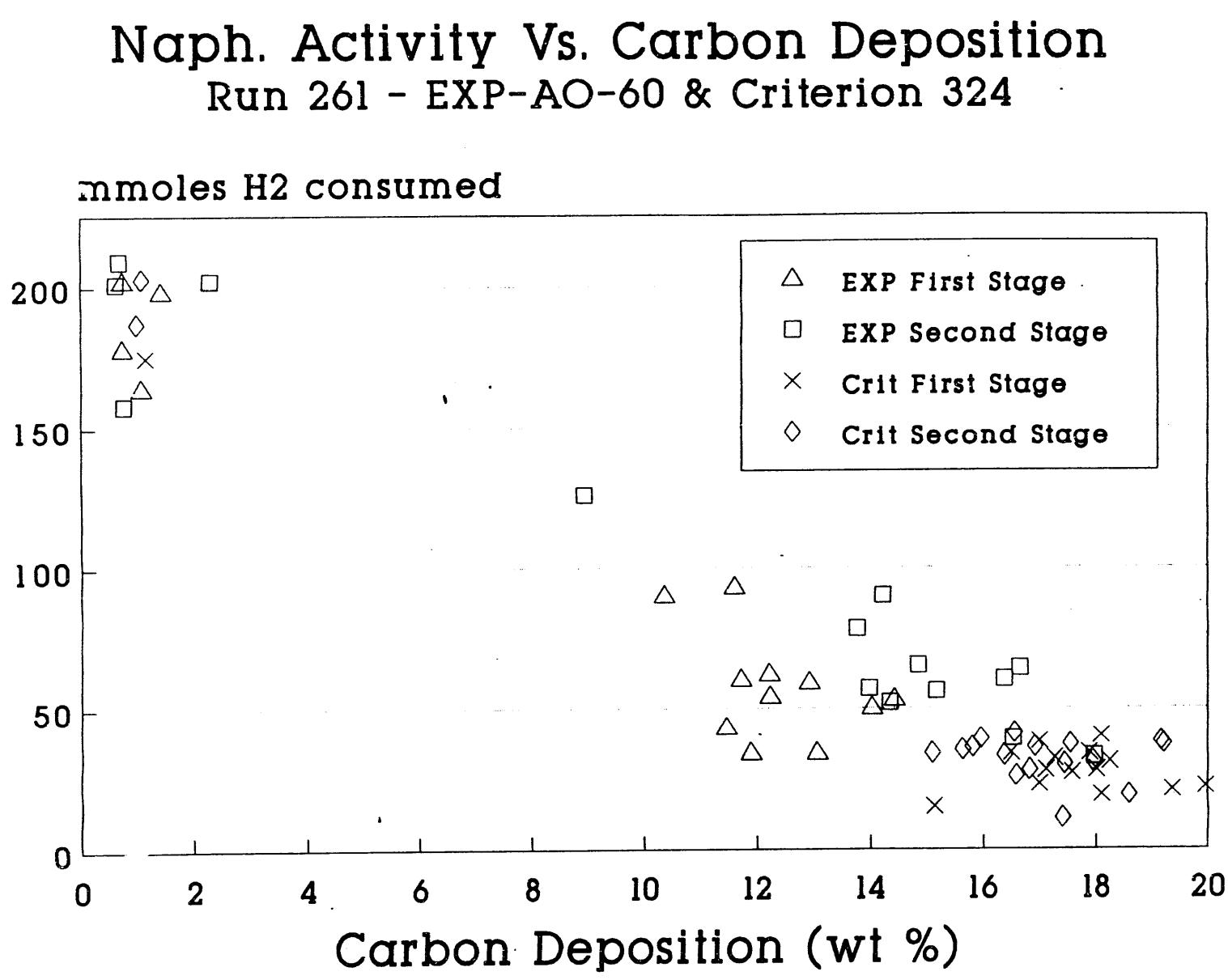


FIGURE 33. NAPHTHALENE ACTIVITY AS A FUNCTION OF CARBON DEPOSITED ON THE THF EXTRACTED CATALYST SAMPLES FROM RUN 261

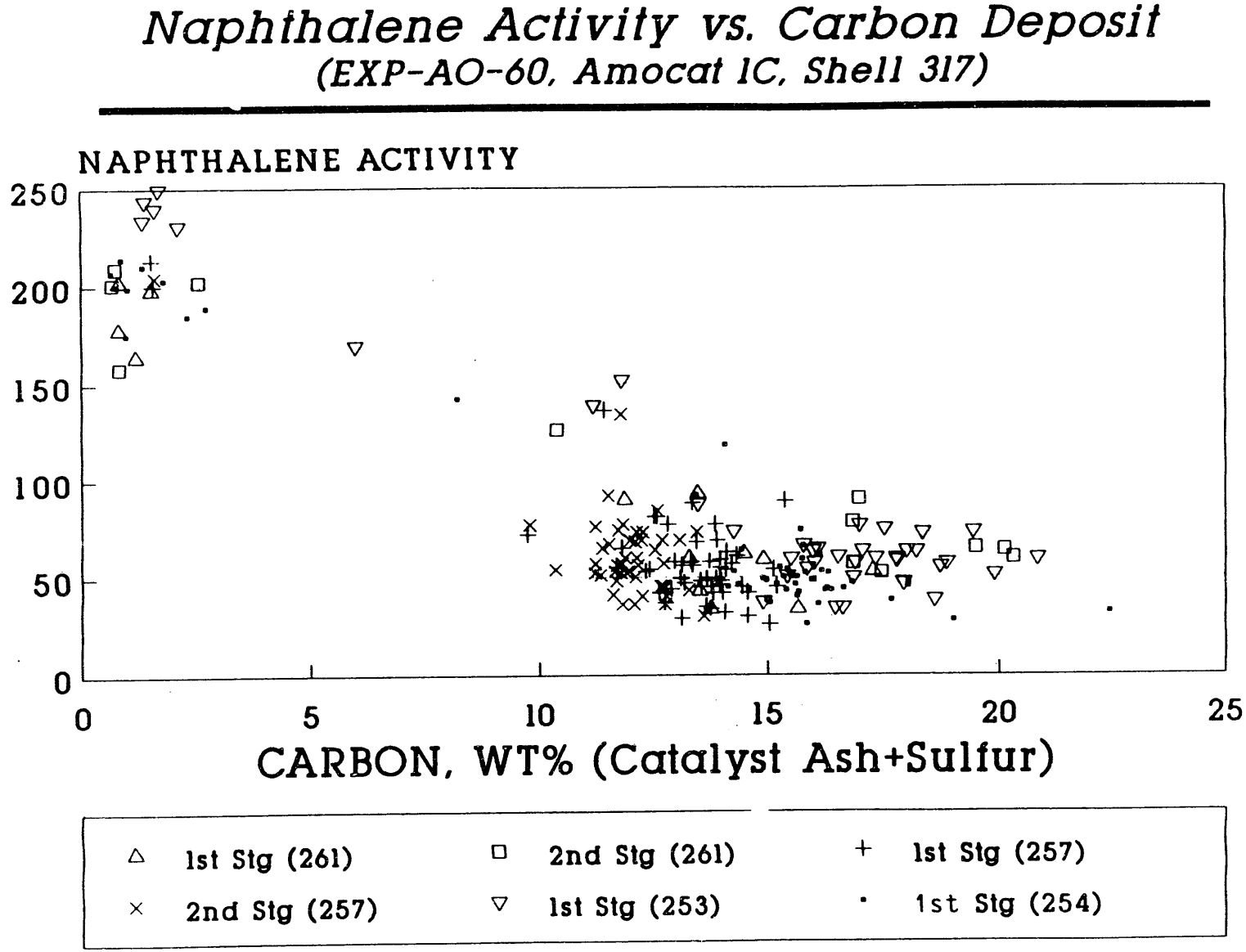
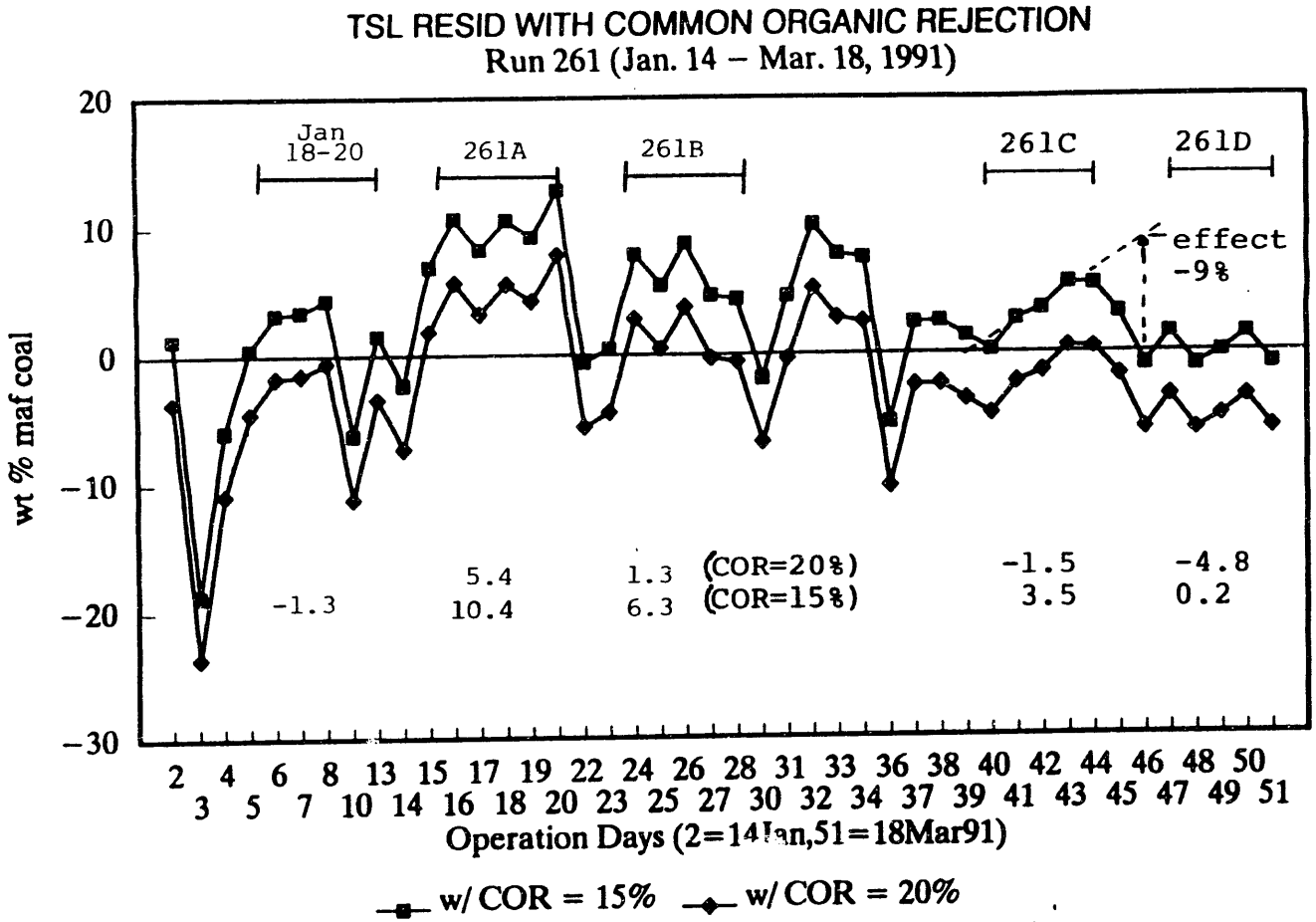


FIGURE 34. NAPHTHALENE ACTIVITY AS A FUNCTION OF CARBON DEPOSITED ON THE THF EXTRACTED CATALYST SAMPLES FROM DIFFERENT RUNS WITH BIMODAL CATALYSTS



* Illinois No. 6 with EXP−AO−60 (1/16") in catalytic/catalytic mode; each stage cat. repl. at 3 lb/ton in Feb. 1−15 (OD=19−28); 1.5 lb/ton in Mar. 13−18 (OD=46−51)

FIGURE 35. TREND DATA OF OVERALL TWO-STAGE EXCESS RESID YIELD WITH COMMON ORGANIC REJECTION (EXP-AO-60 CATALYST; 261A-D)

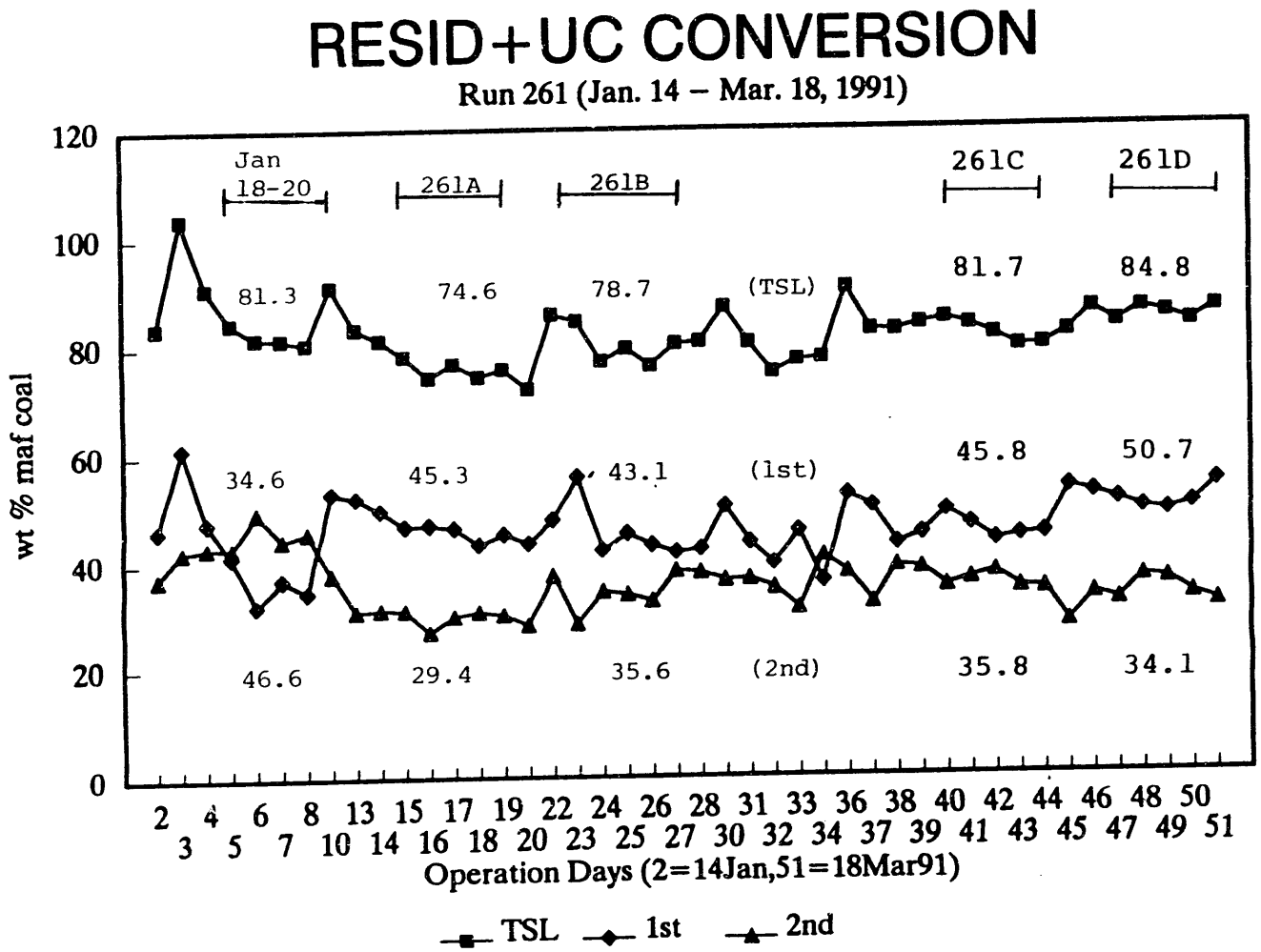


FIGURE 36. TREND DATA OF OVERALL TWO-STAGE RESID + UC CONVERSION (EXP-AO-60 CATALYST ; 261A-D)

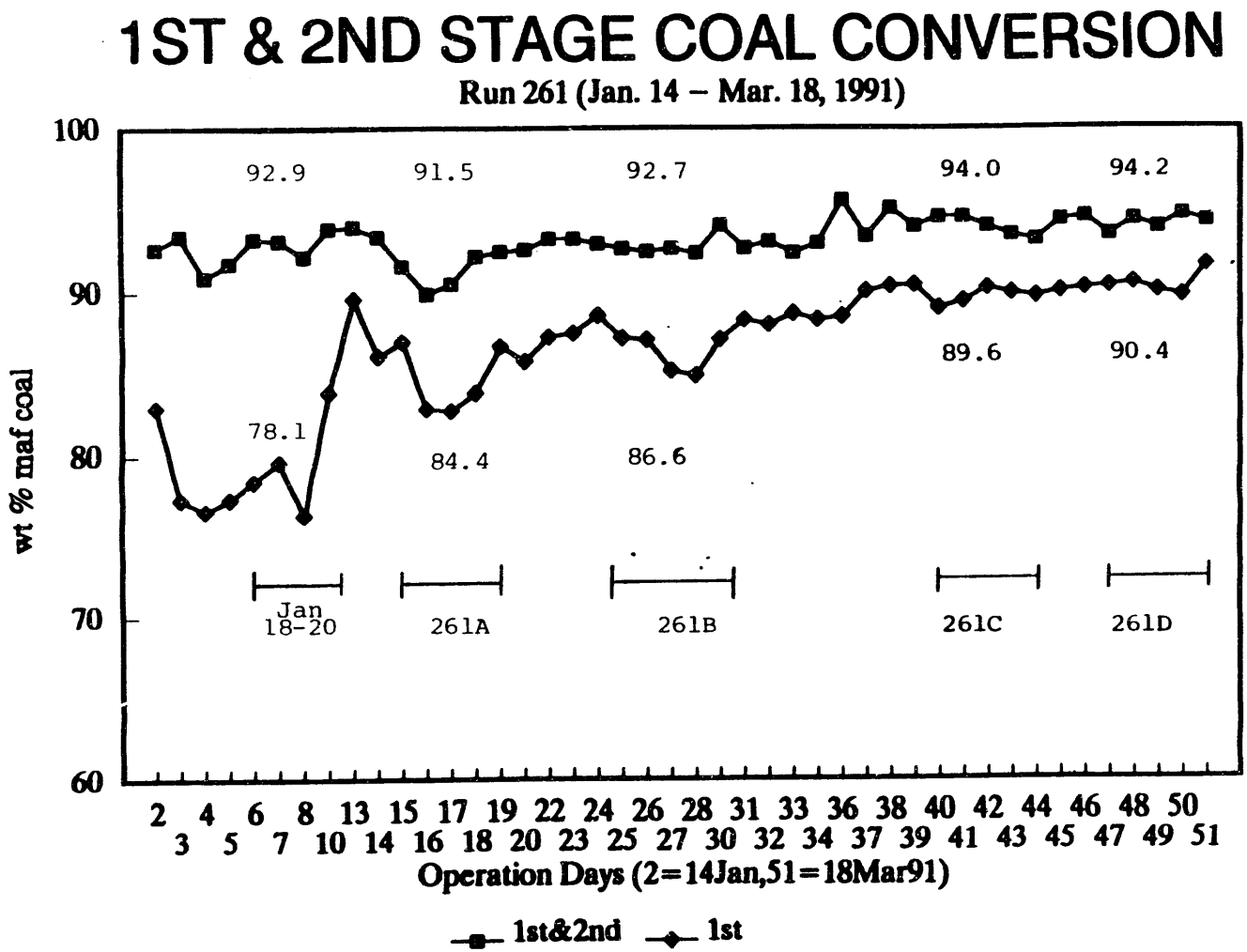


FIGURE 37. TREND DATA OF COAL CONVERSION (EXP-AO-60 CATALYST; 261A-D)

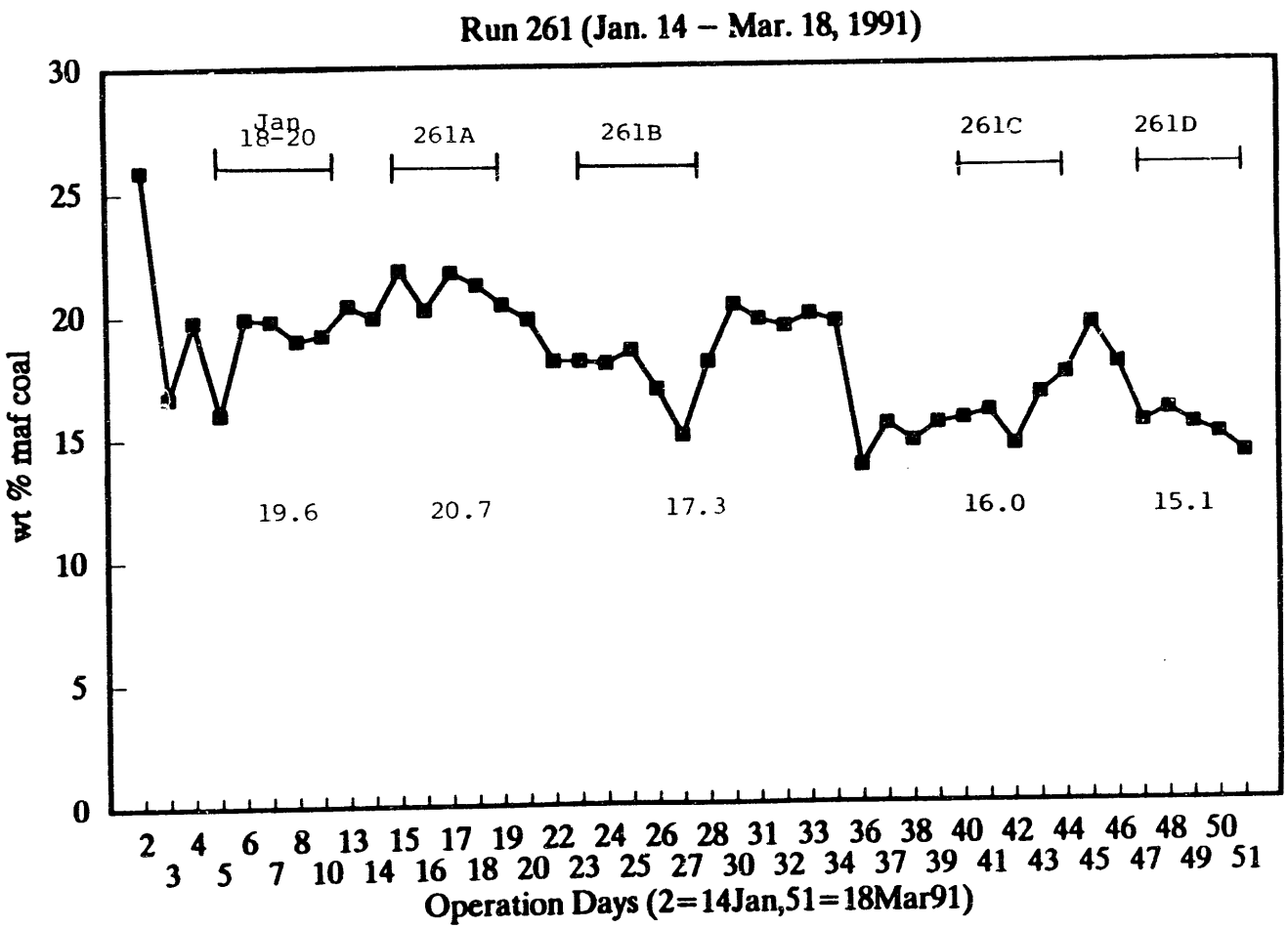


FIGURE 38. TREND DATA OF ORGANICS IN BOTTOMS PRODUCT (EXP-AO-60 CATALYST; 261A-D)

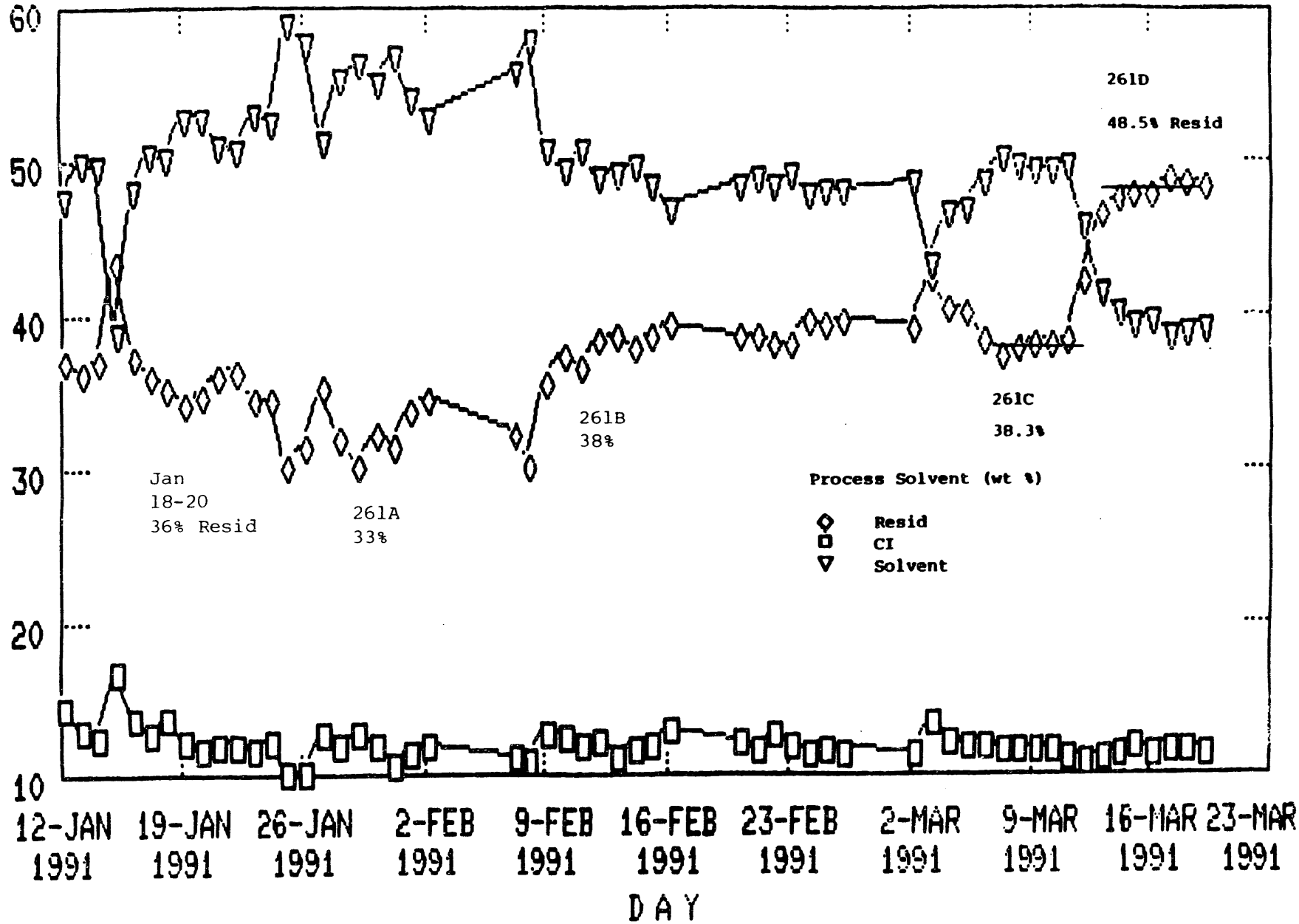
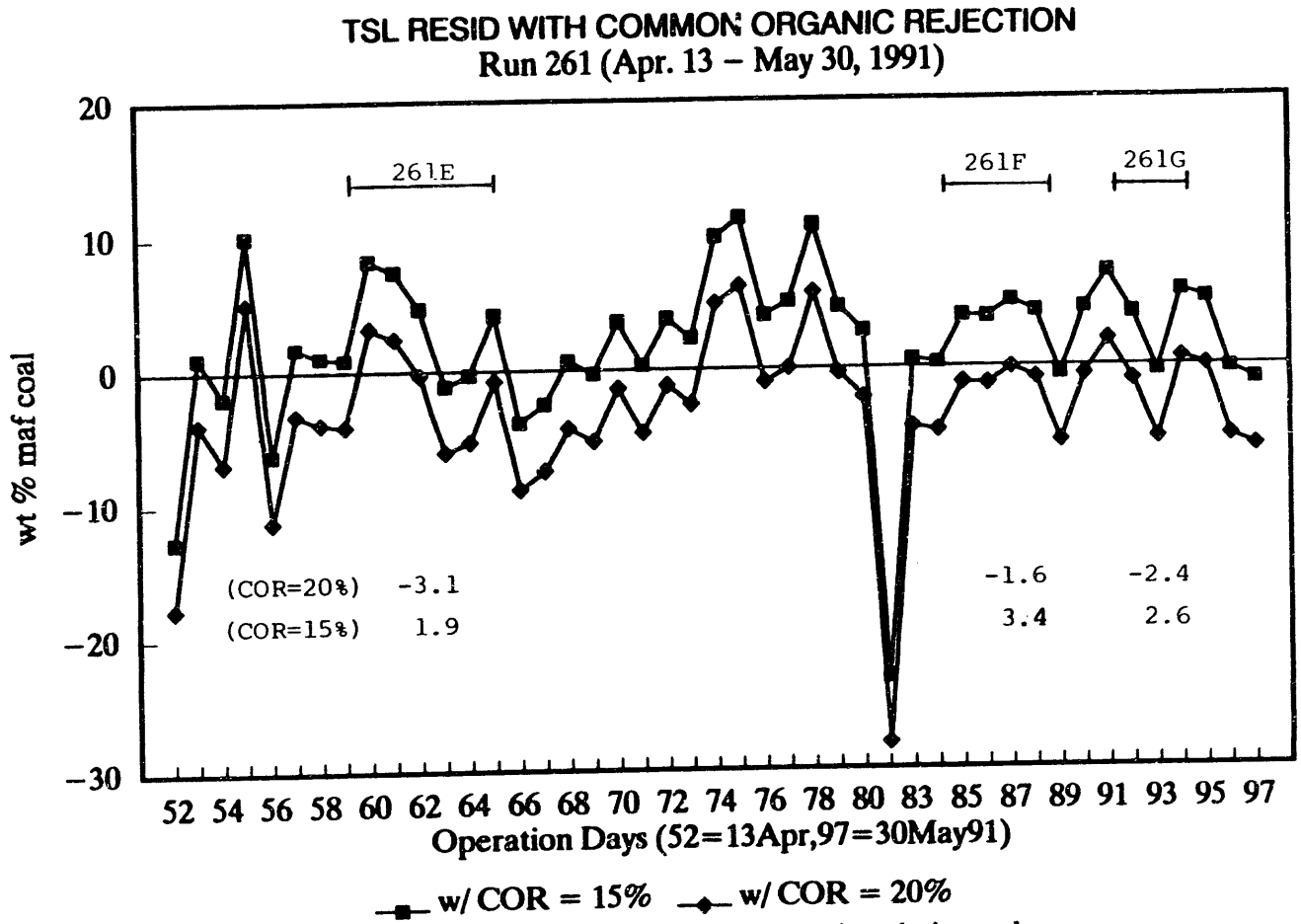


FIGURE 39. TREND DATA OF PROCESS SOLVENT COMPOSITION (EXP-AP-60 CATALYST; 261A-D)



* Illinois No. 6 with Criterion (Shell) 324 (1/16") in catalytic/catalytic mode each stage cat. repl. at 3 lb/t in Apr. 18–26 (OD=57–65); 2.25 lb/t in May 8–30 (OD=77–97)

FIGURE 40. TREND DATA OF OVERALL TWO-STAGE EXCESS RESID YIELD WITH COMMON ORGANIC REJECTION (CRITERION 324 CATALYST; 261E-G)

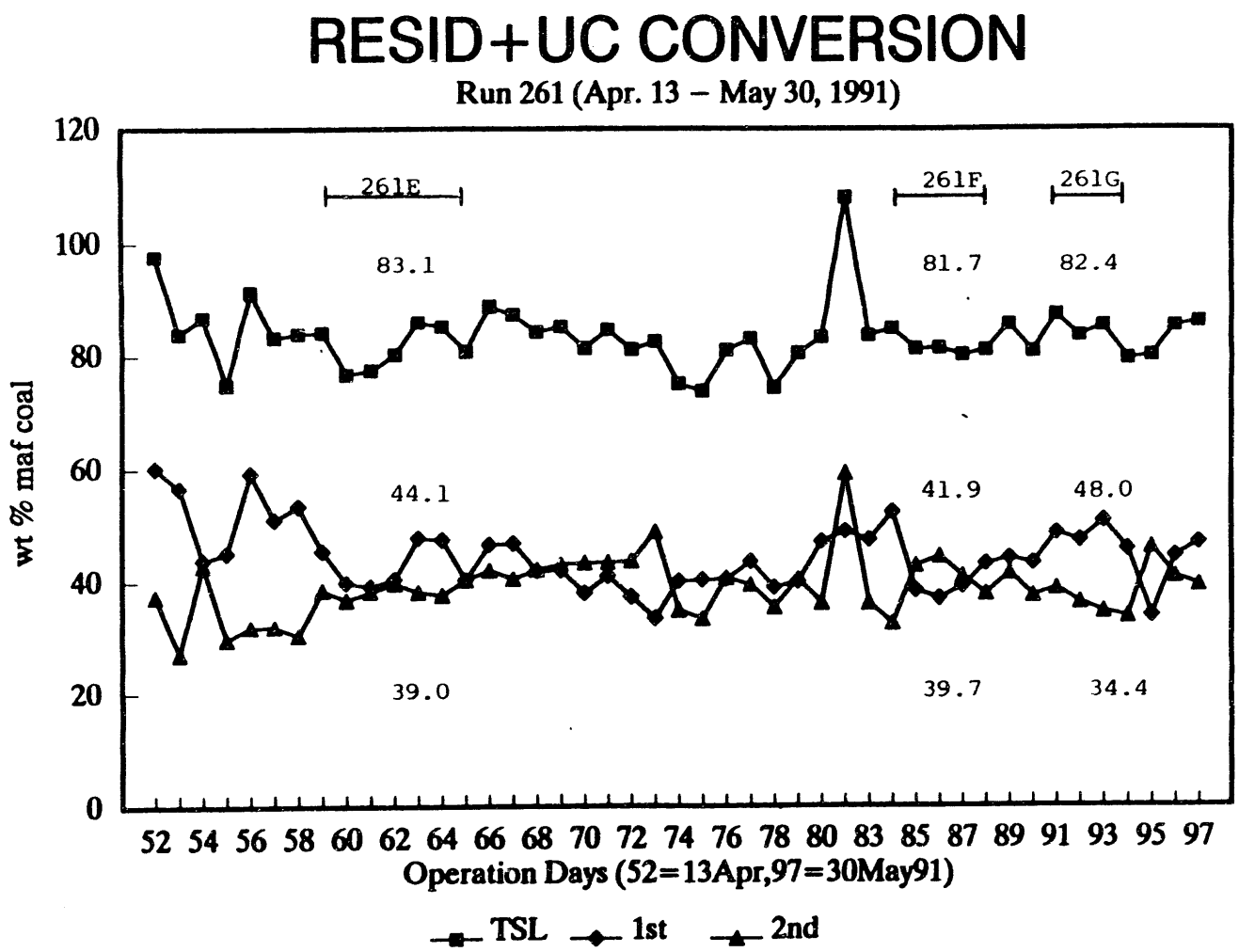


FIGURE 41. TREND DATA OF OVERALL TWO-STAGE RESID + UC CONVERSION (CRITERION 324 CATALYST; 261E-G)

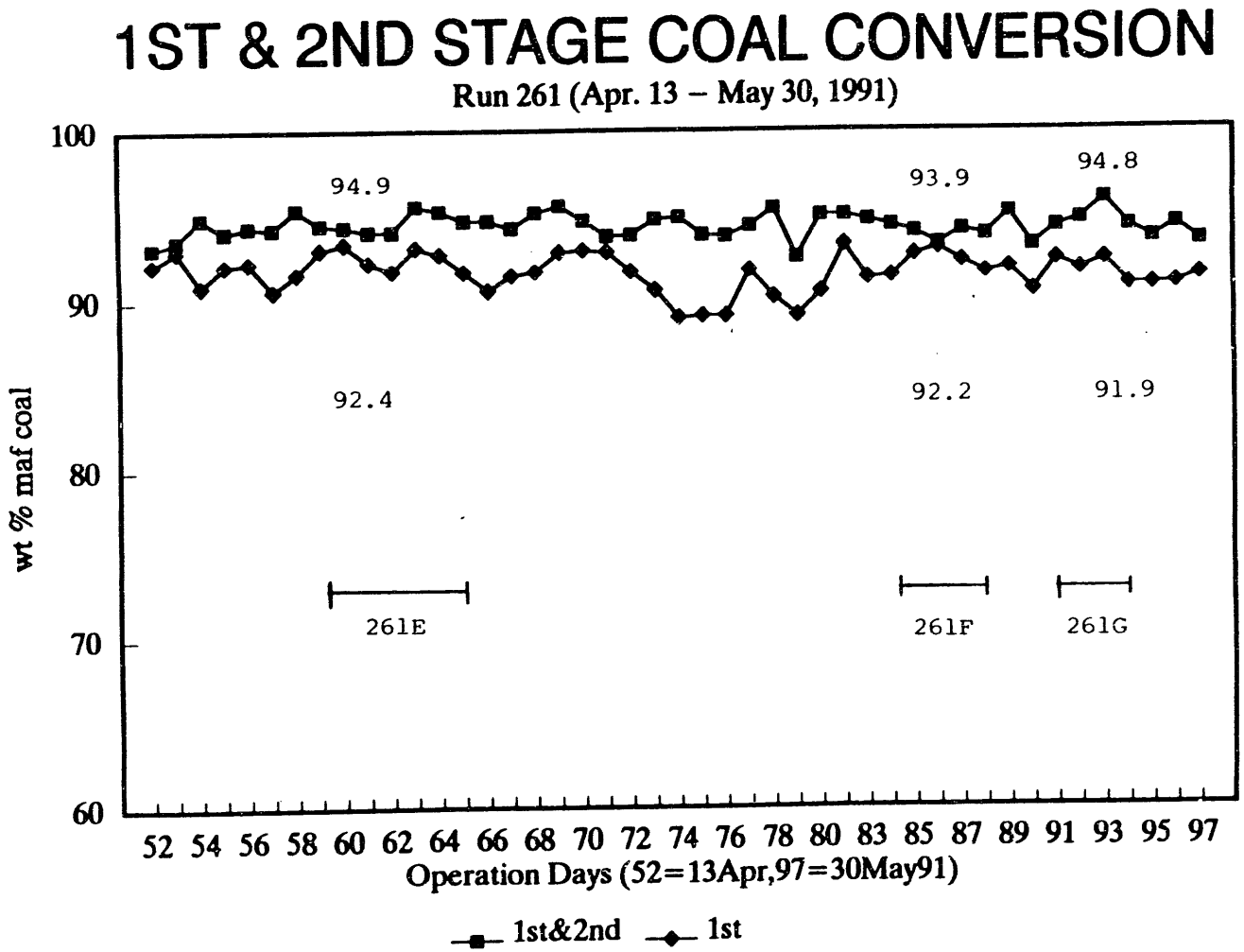


FIGURE 42. TREND DATA OF COAL CONVERSION (CRITERION 324 CATALYST; 261E-G)

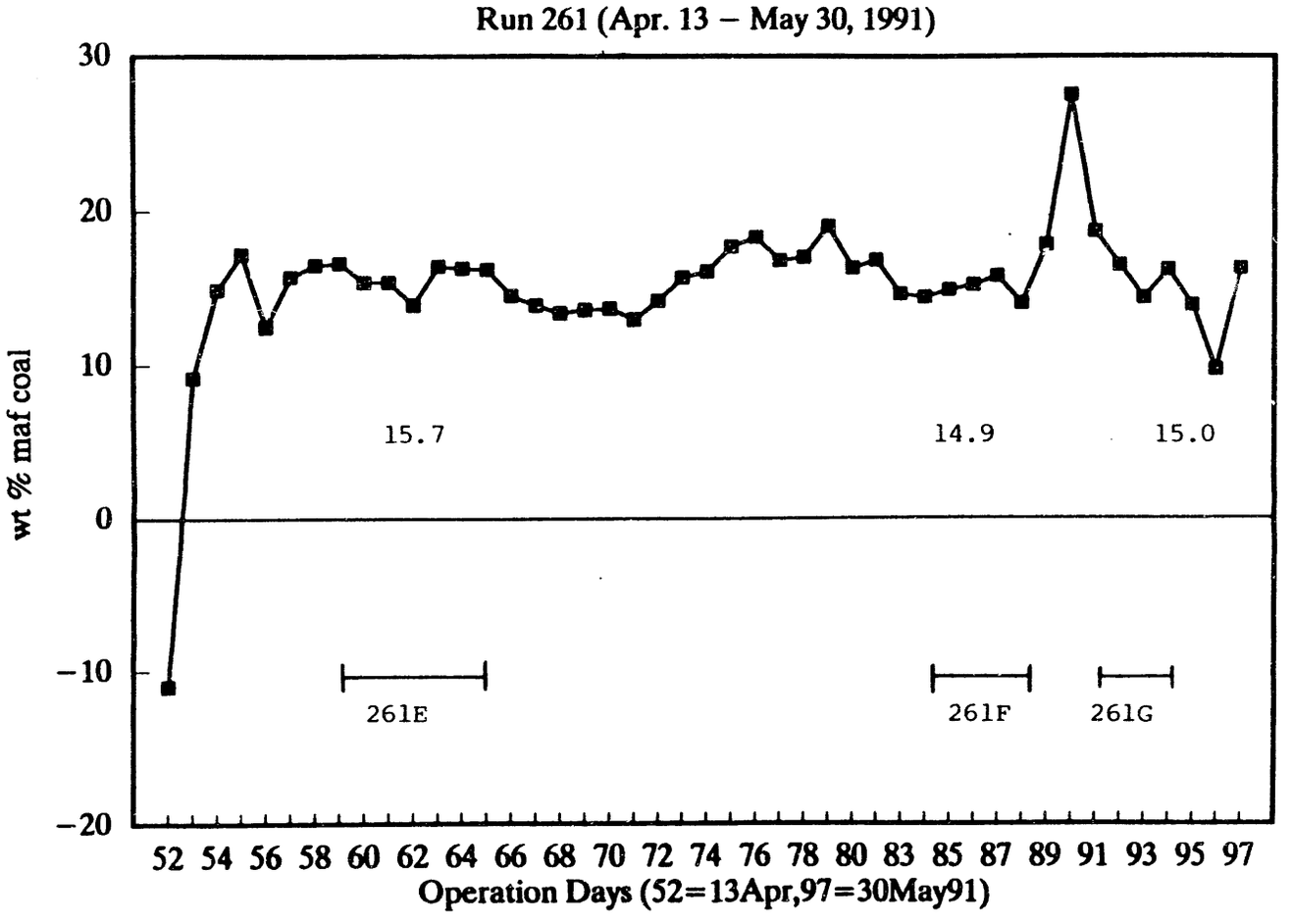


FIGURE 43. TREND DATA OF ORGANICS IN BOTTOMS PRODUCT (CRITERION 324 CATALYST; 261E-G)

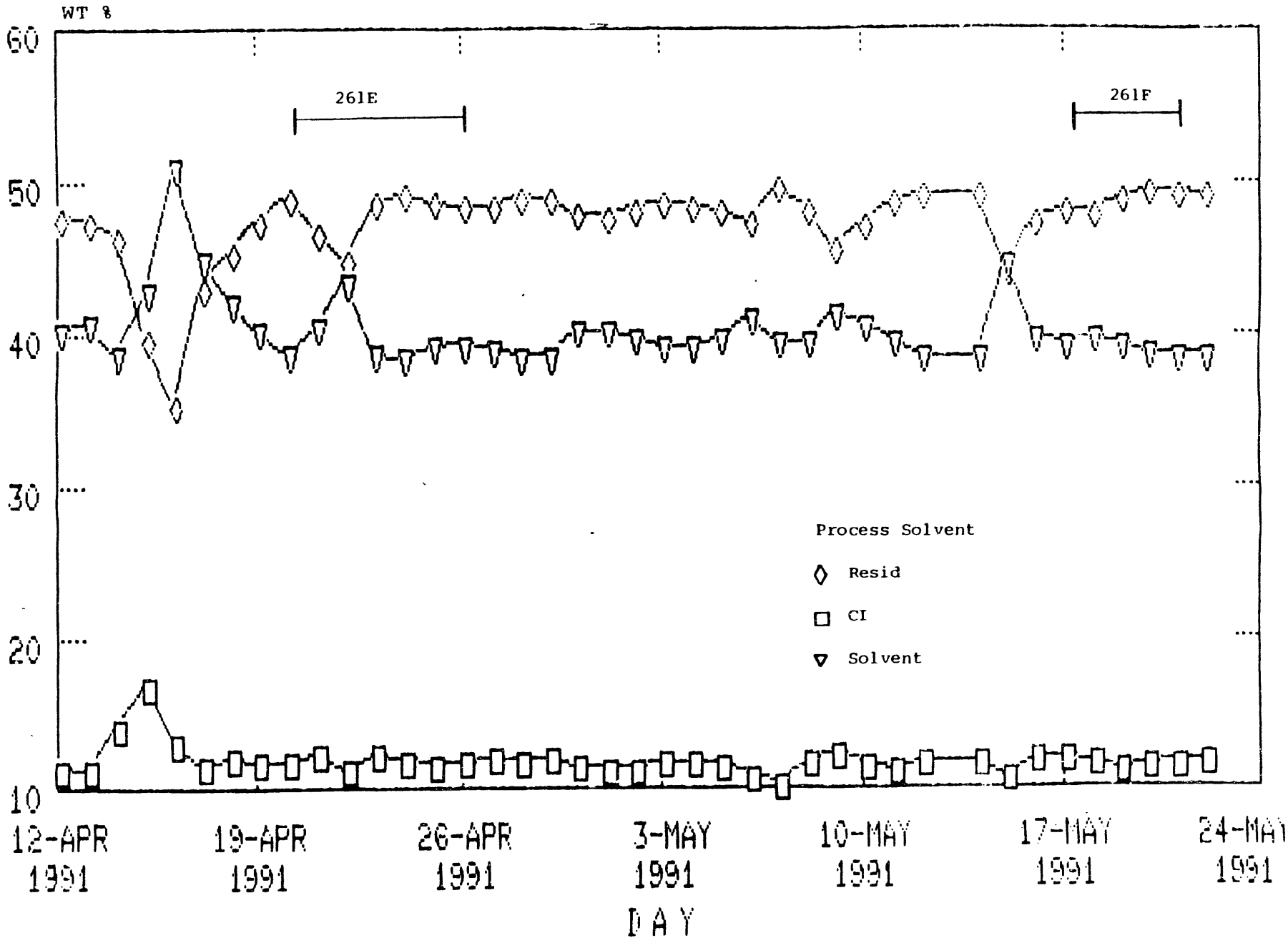


FIGURE 44. TREND DATA OF PROCESS SOLVENT COMPOSITION WITH CRITERION 324 CATALYST; 261E-F)

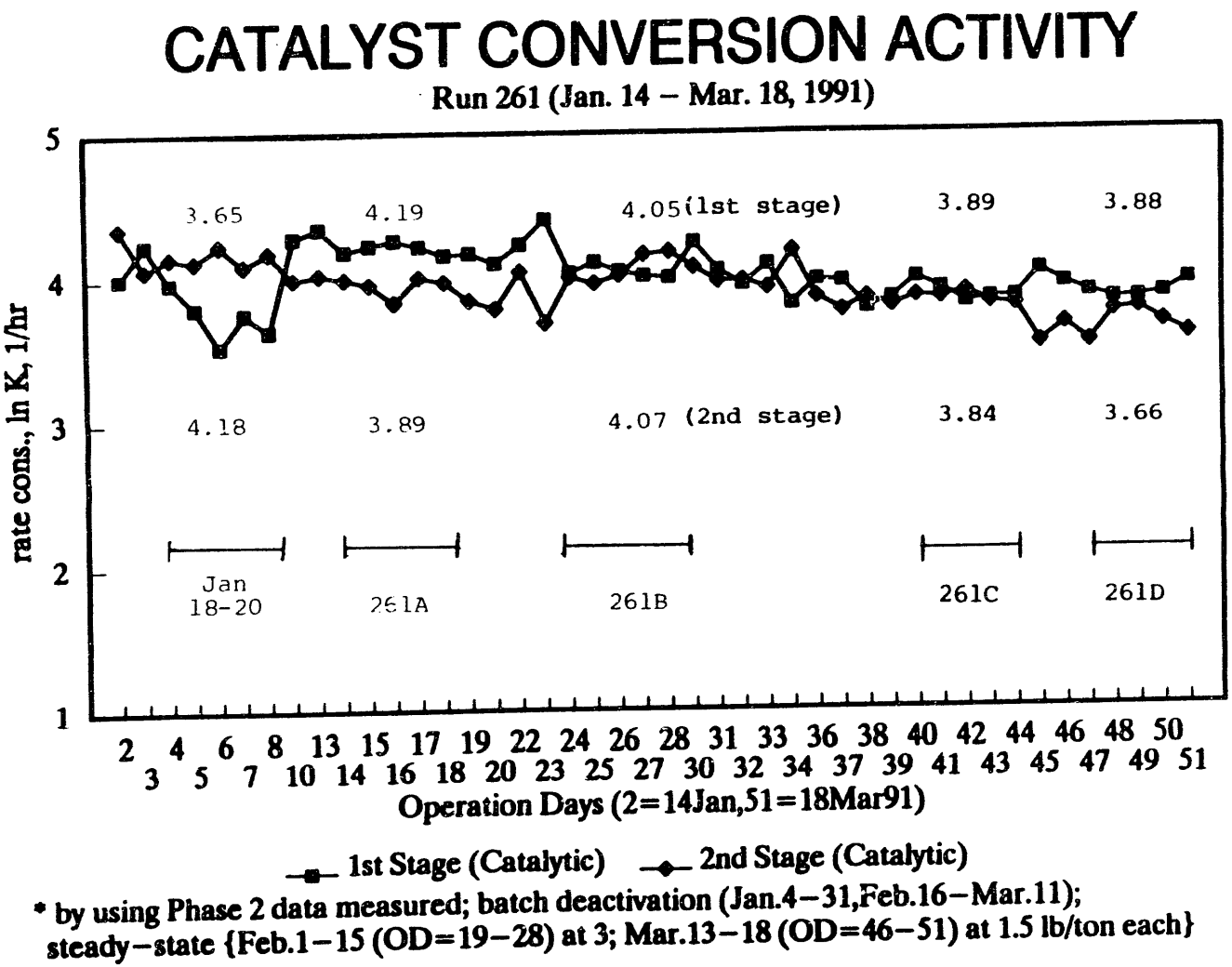


FIGURE 45. FIRST AND SECOND STAGE CATALYST ACTIVITY TRENDS (EXP-AO-60 CATALYST; 261A-D)

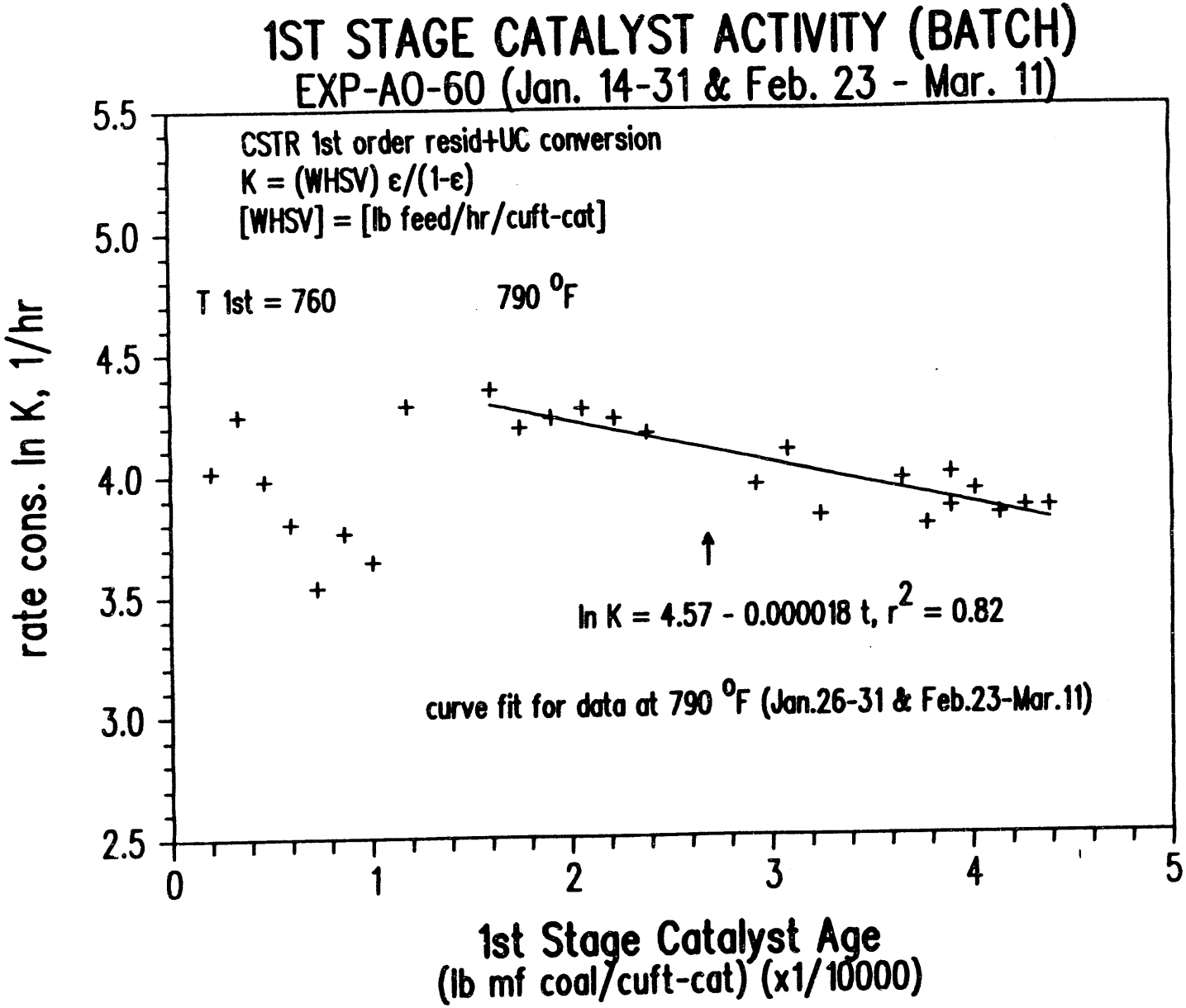


FIGURE 46. LINEAR REGRESSION ANALYSIS OF FIRST STAGE ACTIVITY TREND (BATCH DEACTIVATION OF EXP-AO-60 CATALYST)

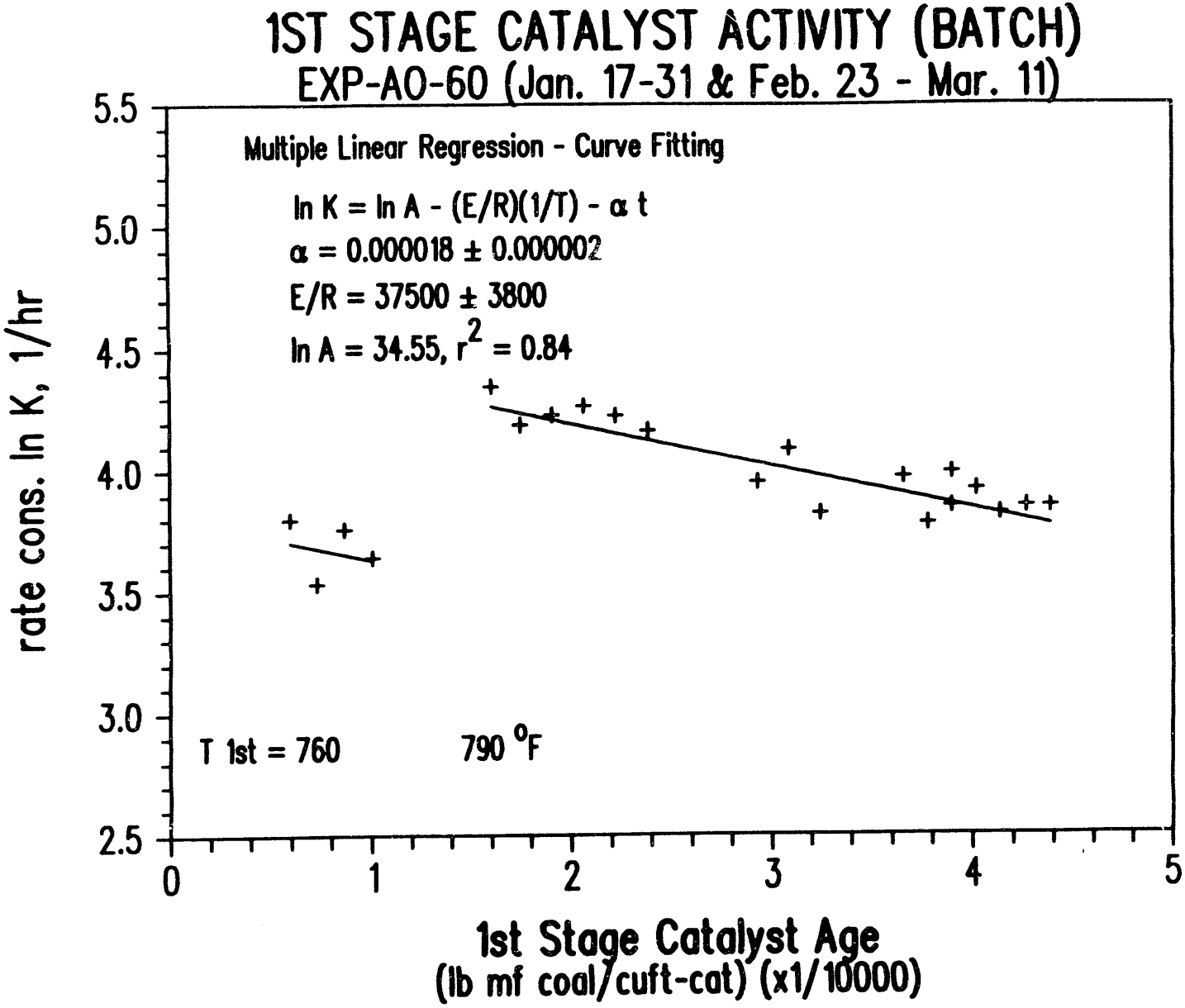


FIGURE 47. MULTIPLE LINGAR REGRESSION ANALYSIS OF FIRST STAGE ACTIVITY TREND (BATCH DEACTIVATION OF EXP-AO-60 CATALYST)

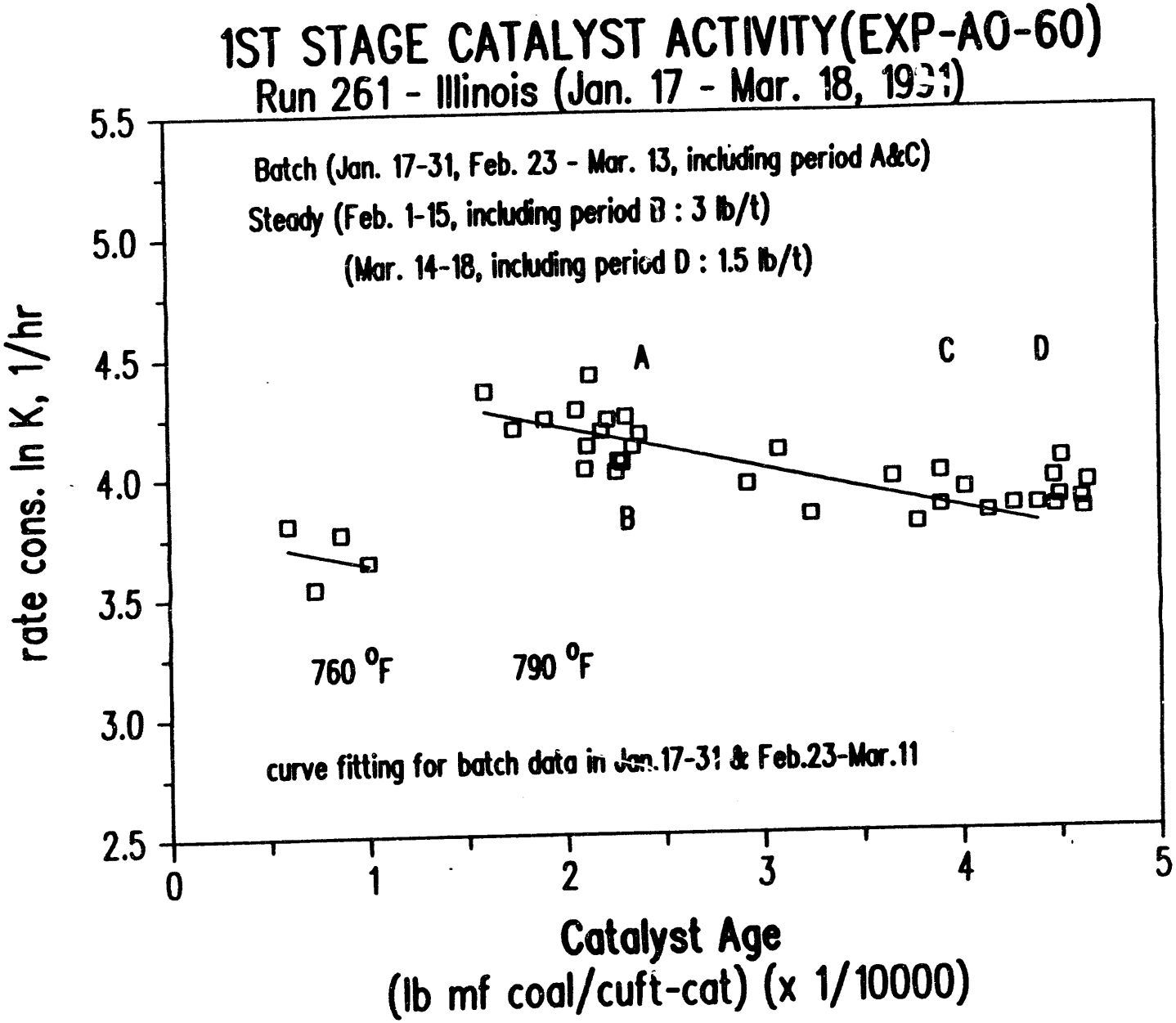


FIGURE 48. FIRST STAGE CATALYST ACTIVITY COMPARISON (EXP-AO-60 CATALYST; BATCH DEACTIVATION VS STEADY-STATE OPERATION; 261A-D)

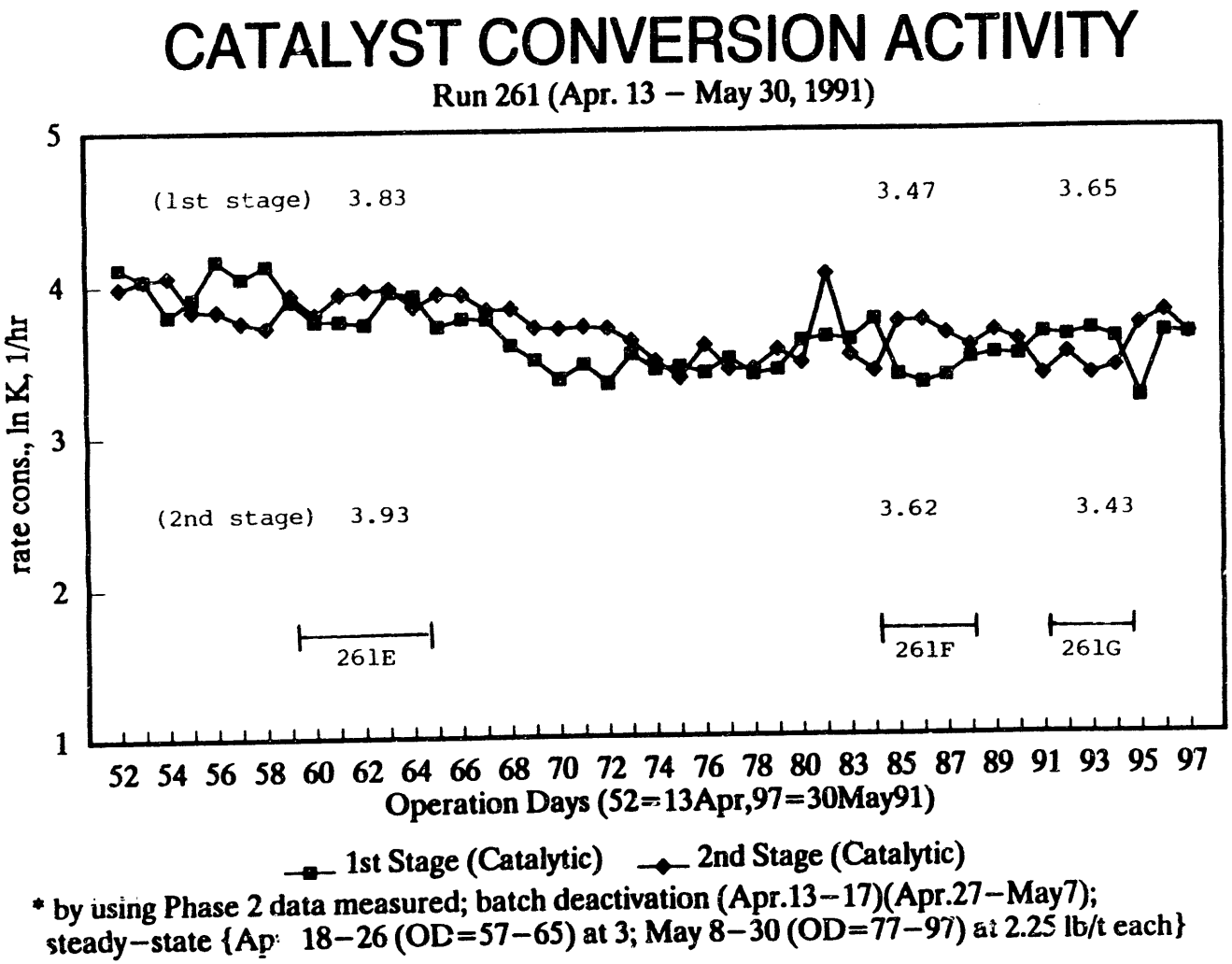


FIGURE 49. FIRST AND SECOND STAGE CATALYST ACTIVITY TRENDS (CRITERION 324 CATALYST; 261E-G)

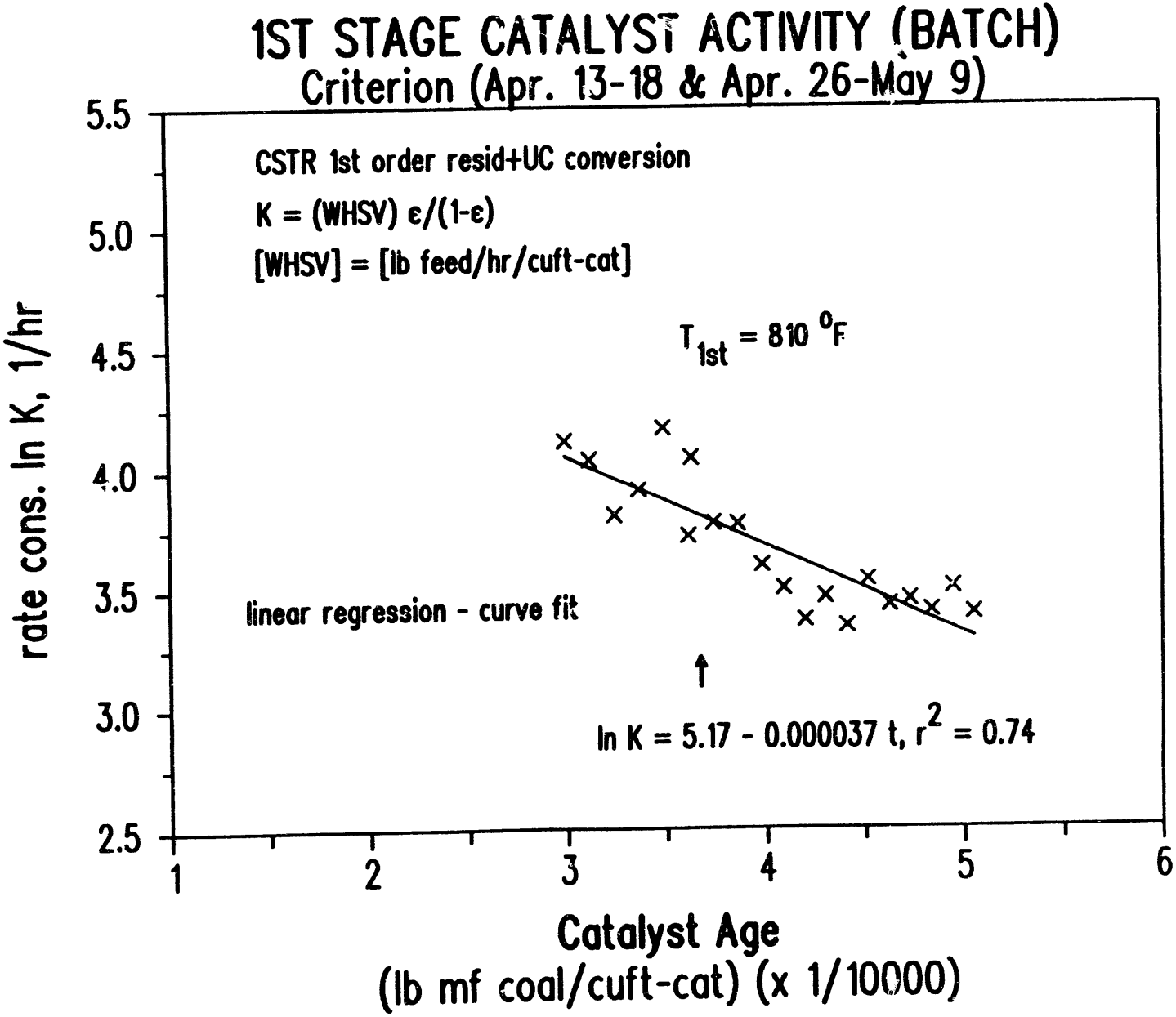


FIGURE 50. LINEAR REGRESSION ANALYSIS OF FIRST STAGE ACTIVITY TREND (CRITERION 324 CATALYST; BATCH DEACTIVATION)

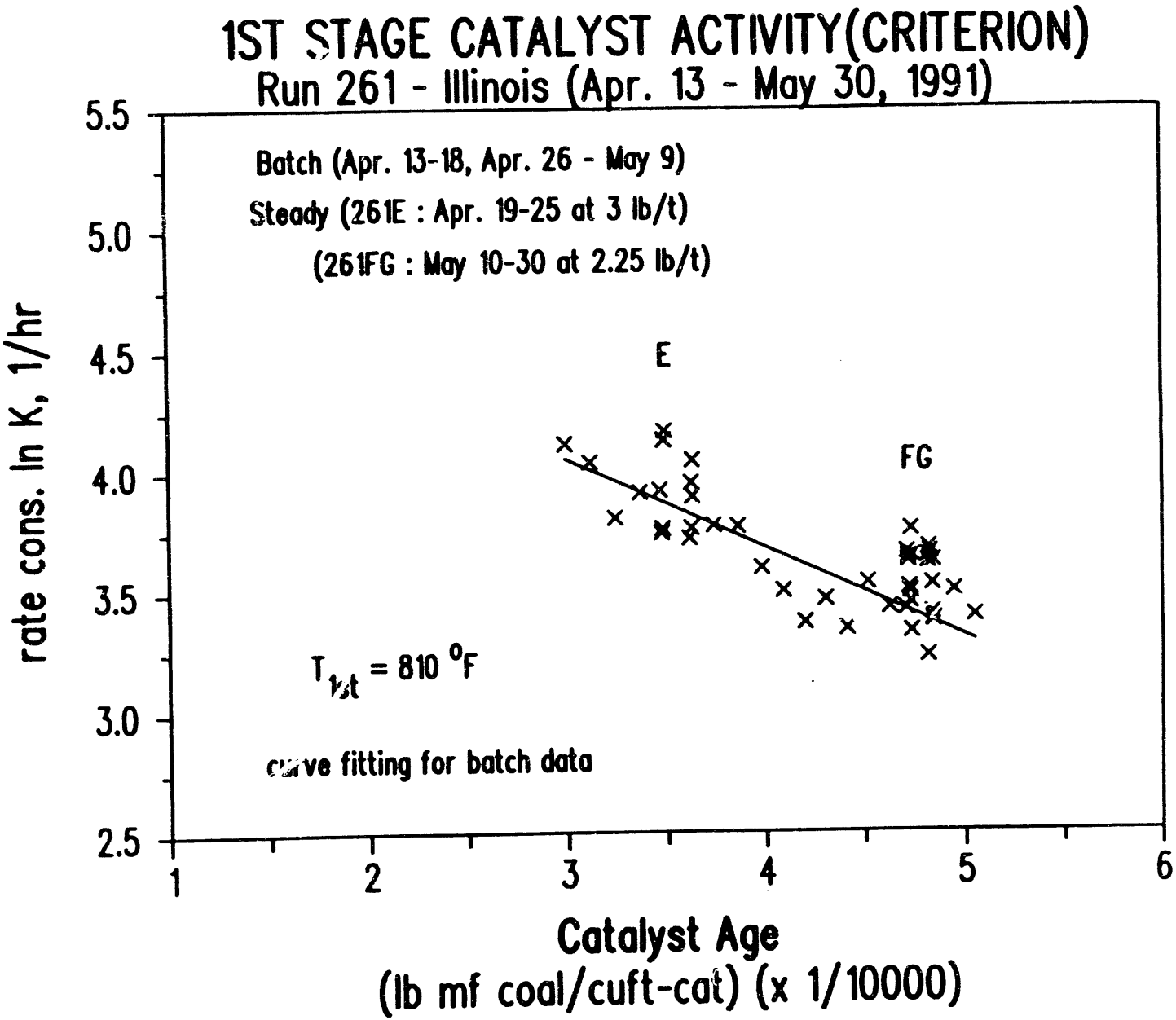


FIGURE 51. FIRST STAGE CATALYST ACTIVITY COMPARISON (CRITERION 324 CATALYST; BATCH DEACTIVATION VS STEADY-STATE OPERATION (261E-G)

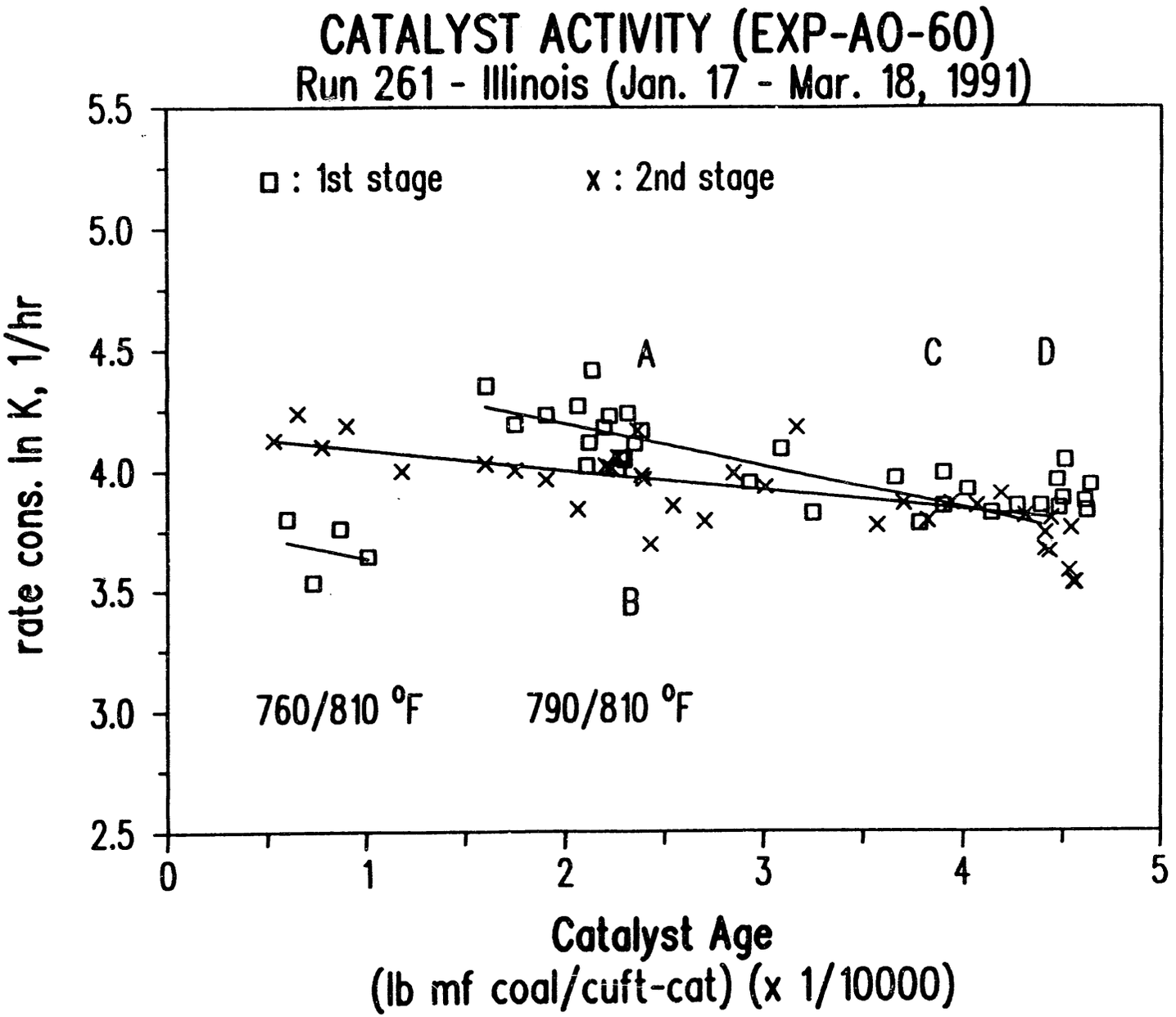


FIGURE 52. FIRST AND SECOND STAGE CATALYST ACTIVITY COMPARISON (EXP-AO-60 CATALYST; 261A-D)

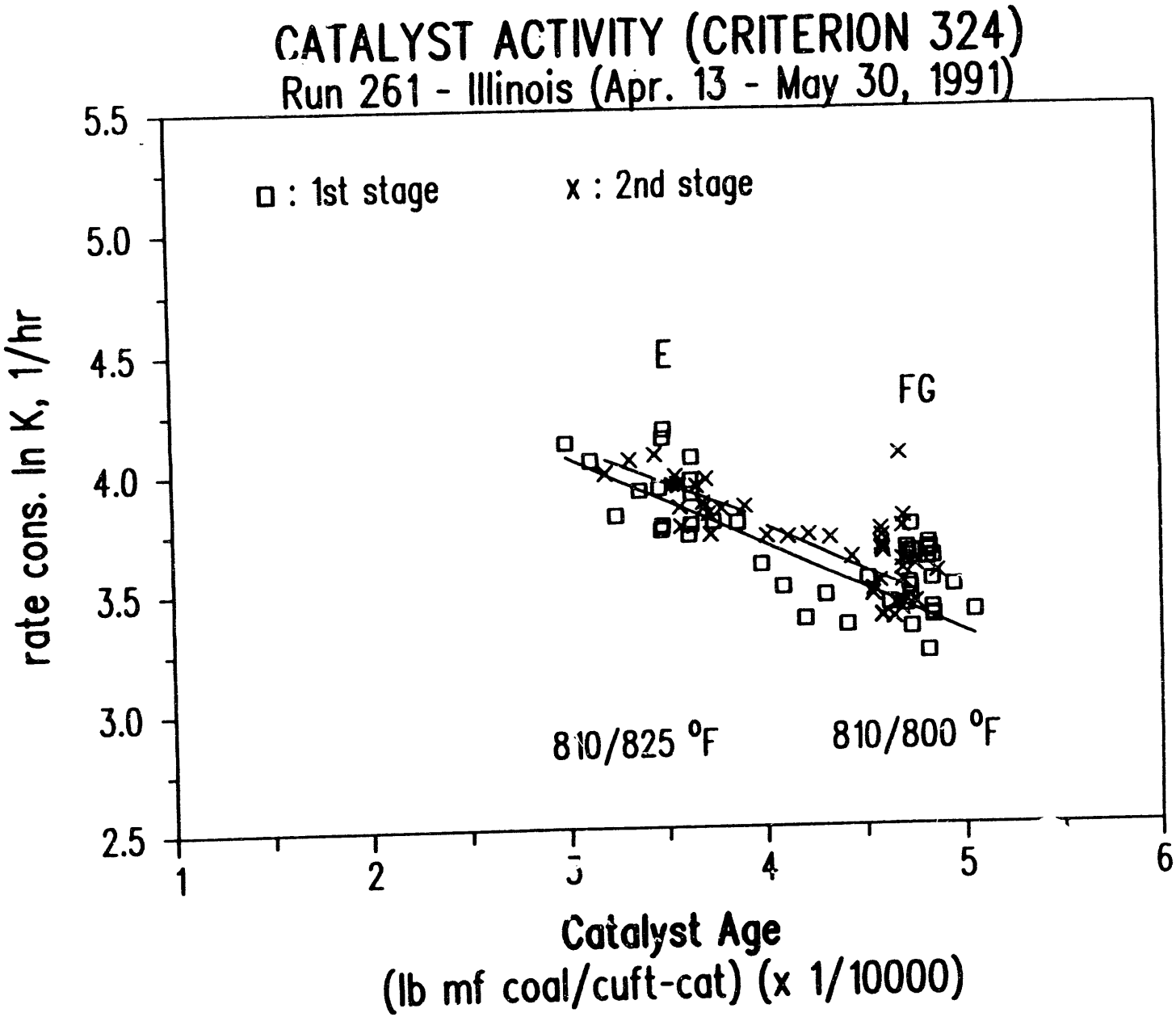


FIGURE 53. FIRST AND SECOND STAGE CATALYST ACTIVITY COMPARISON (CRITERION 324 CATALYST; 261E-G)

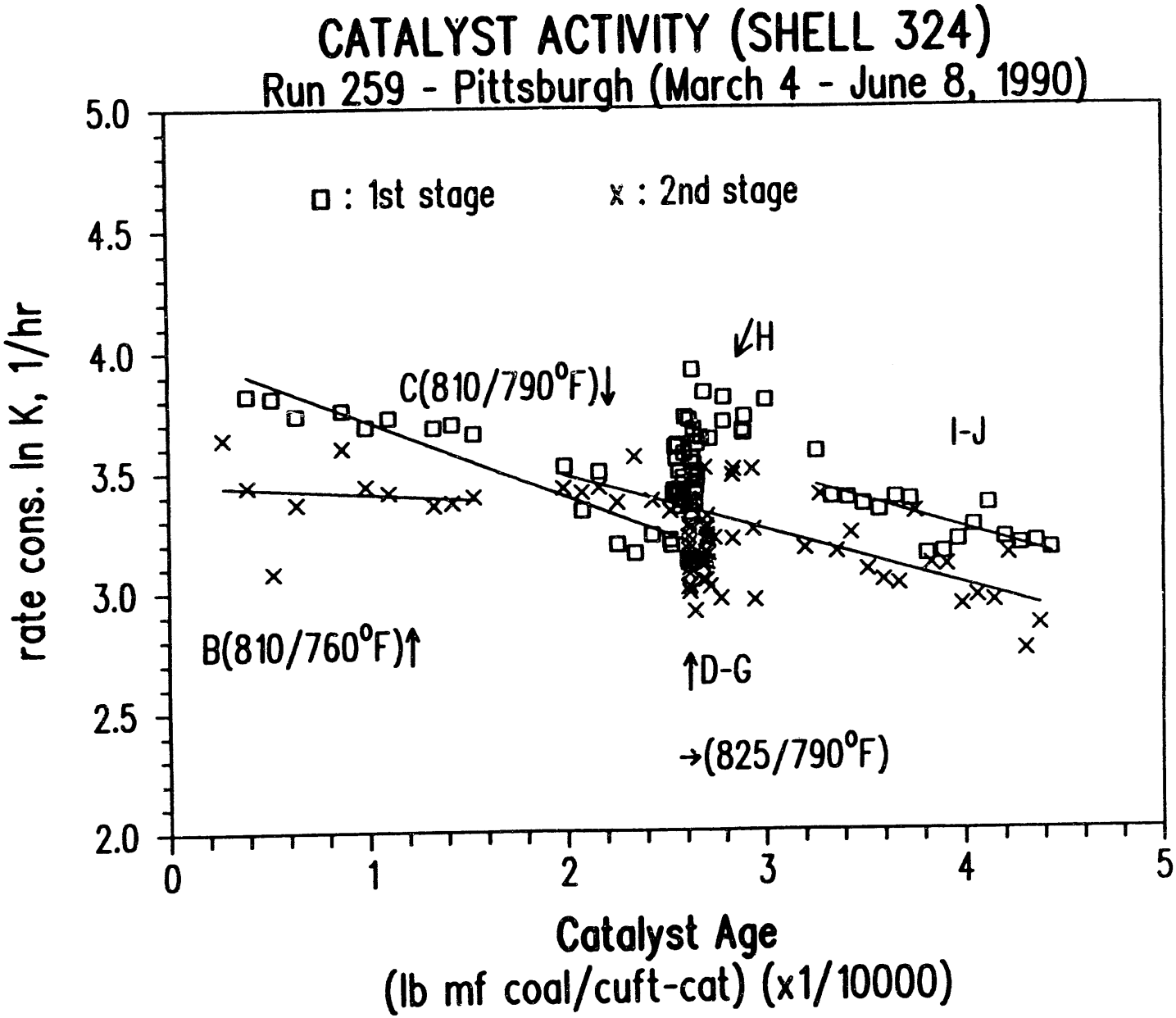


FIGURE 54. FIRST AND SECOND STAGE CATALYST ACTIVITY COMPARISON (SHELL 324 CATALYST; 259B-J)

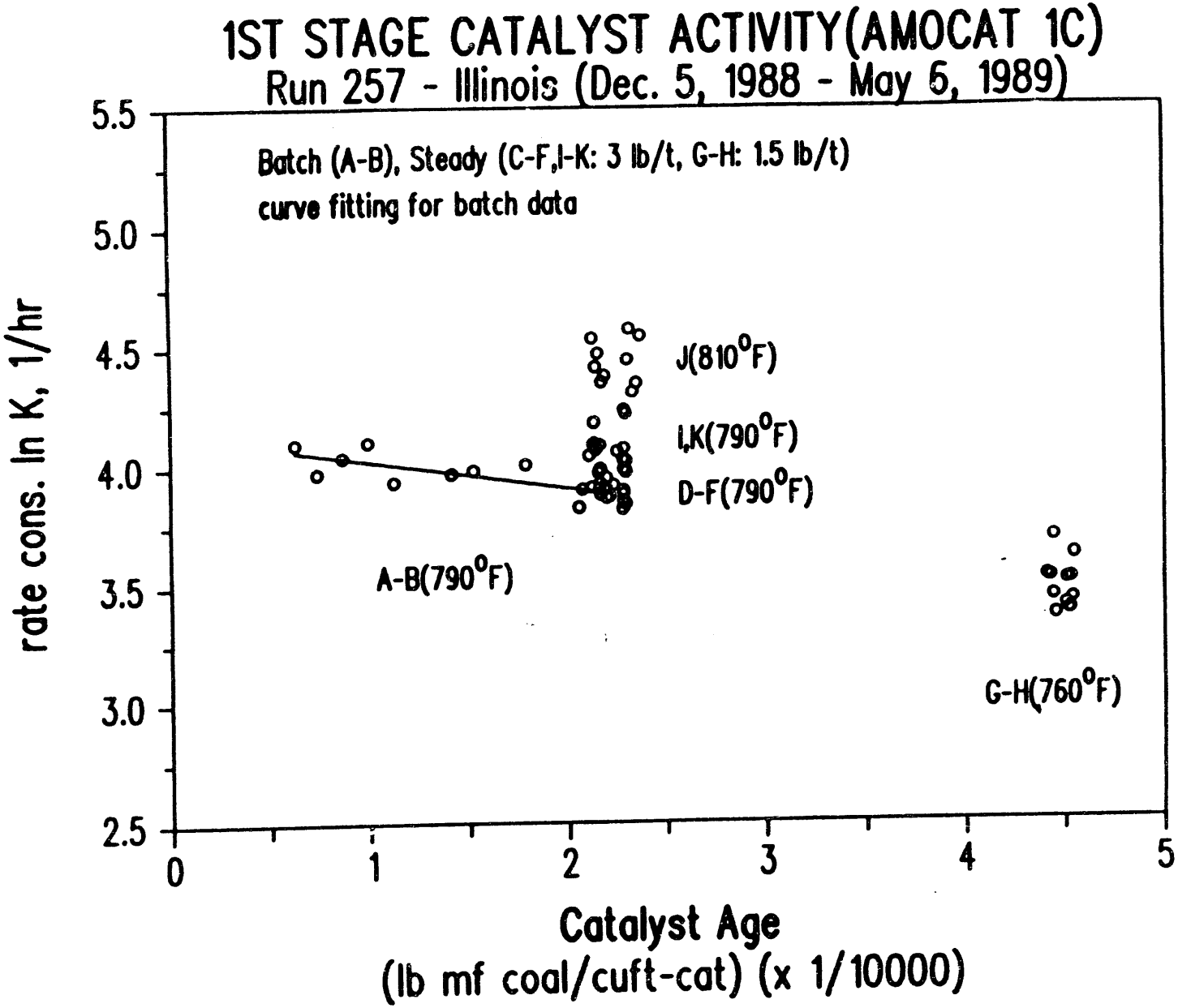


FIGURE 55. FIRST STAGE CATALYST ACTIVITY COMPARISON (AMOCAT 1C CATALYST; BATCH DEACTIVATION VS STEADY-STATE OPERATION; 257A-K)

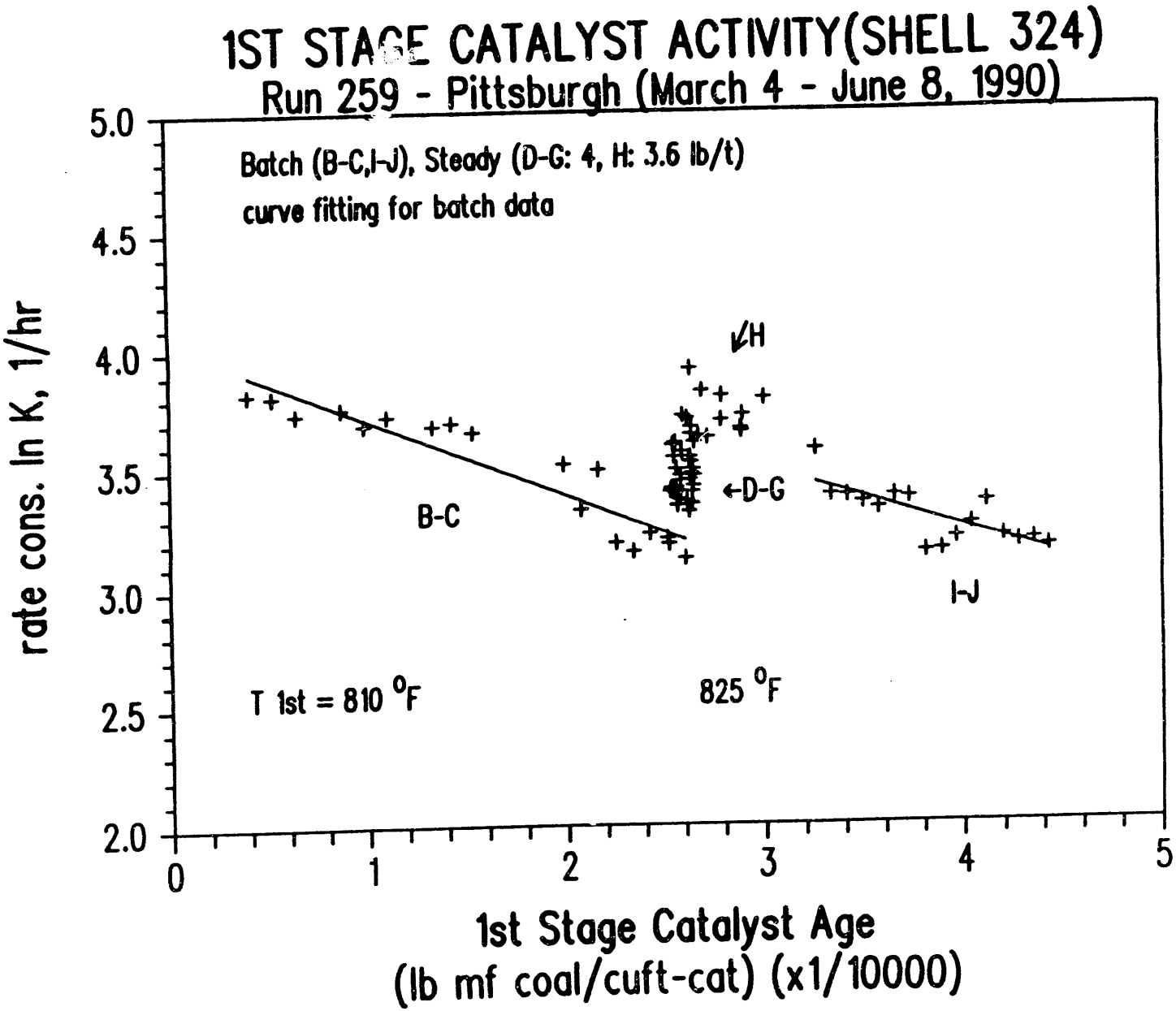


FIGURE 56. FIRST STAGE CATALYST ACTIVITY COMPARISON (SHELL 324 CATALYST; BATCH DEACTIVATION VS STEADY-STATE OPERAION; 259B-J)

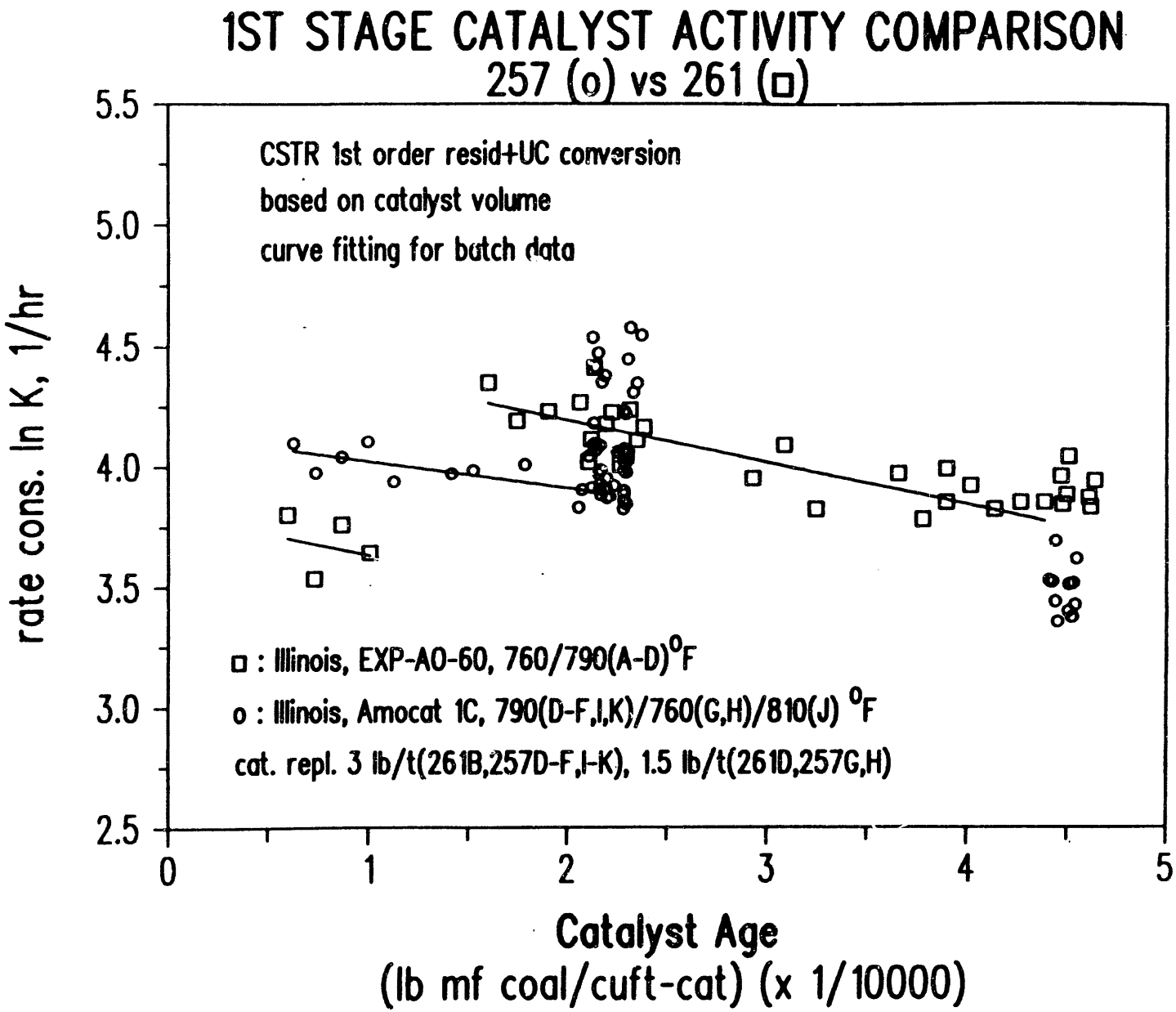


FIGURE 57. FIRST STAGE CATALYST ACTIVITY COMPARISON (RUN 261 VS 257)

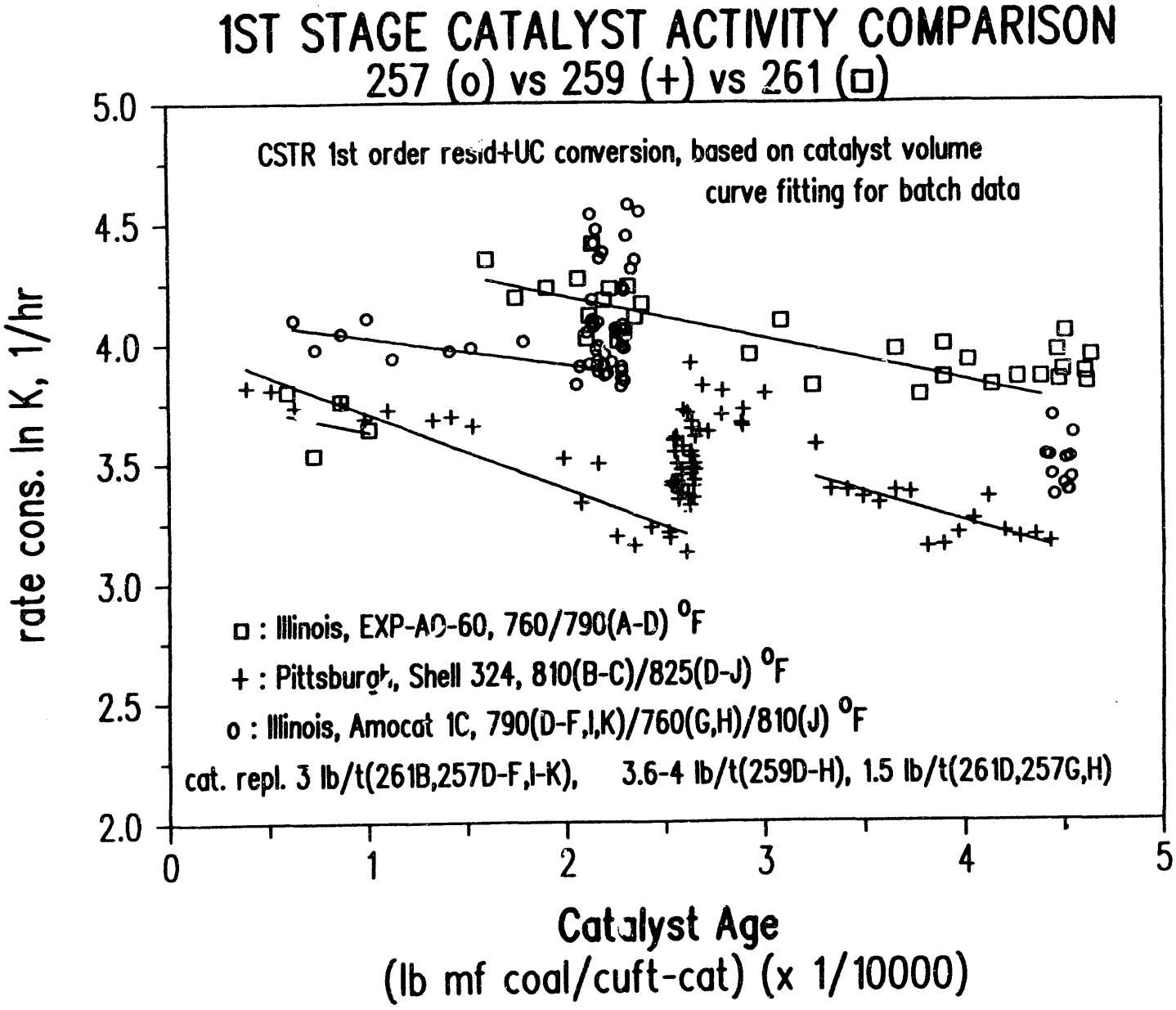


FIGURE 58. FIRST STAGE CATALYST ACTIVITY COMPARISON (RUN 261 VS 259 VS 257)

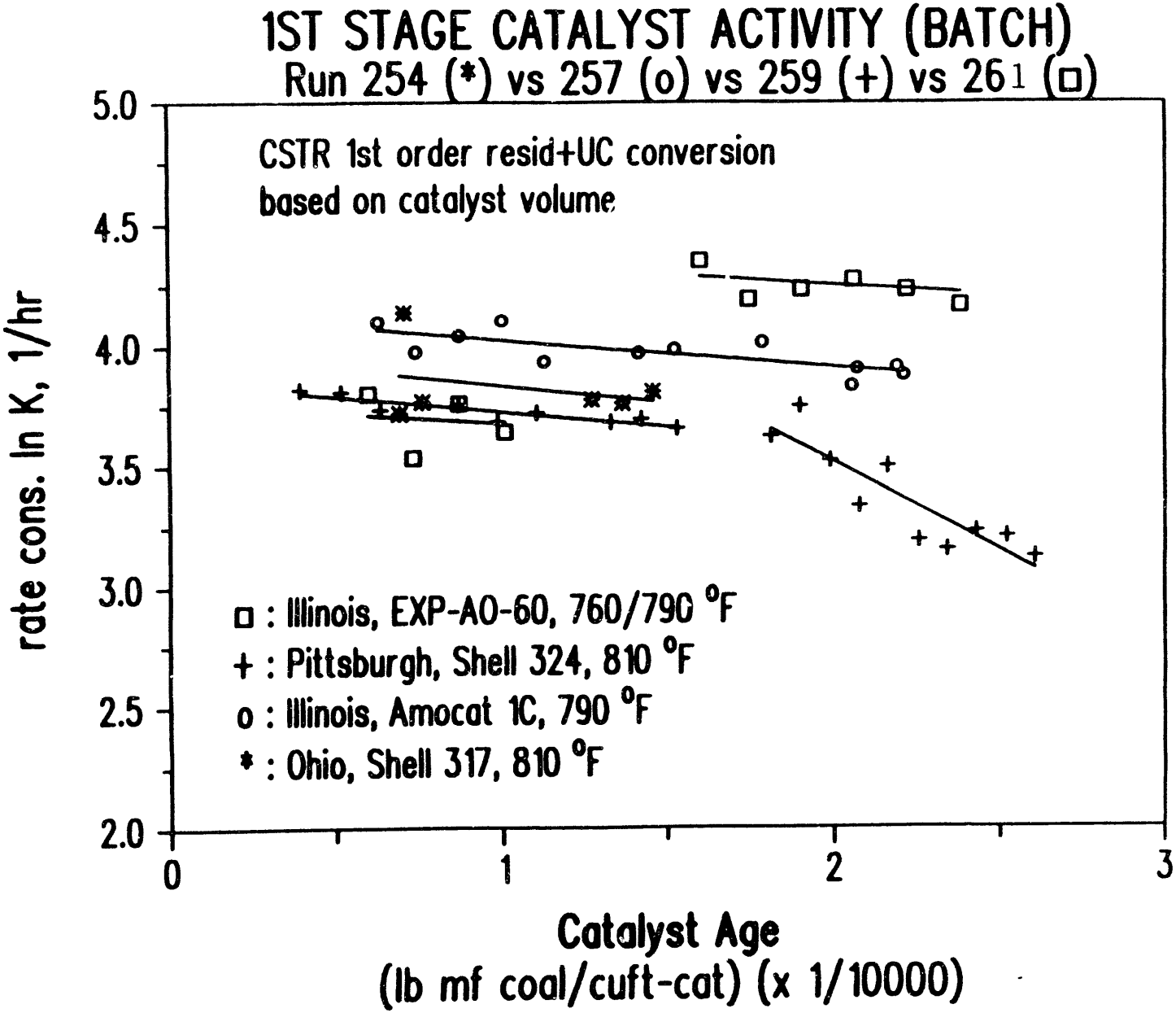


FIGURE 59. FIRST STAGE CATALYST ACTIVITY COMPARISON (BATCH DEACTIVATION) (RUN 261 VS 259 VS 257 VS 254)

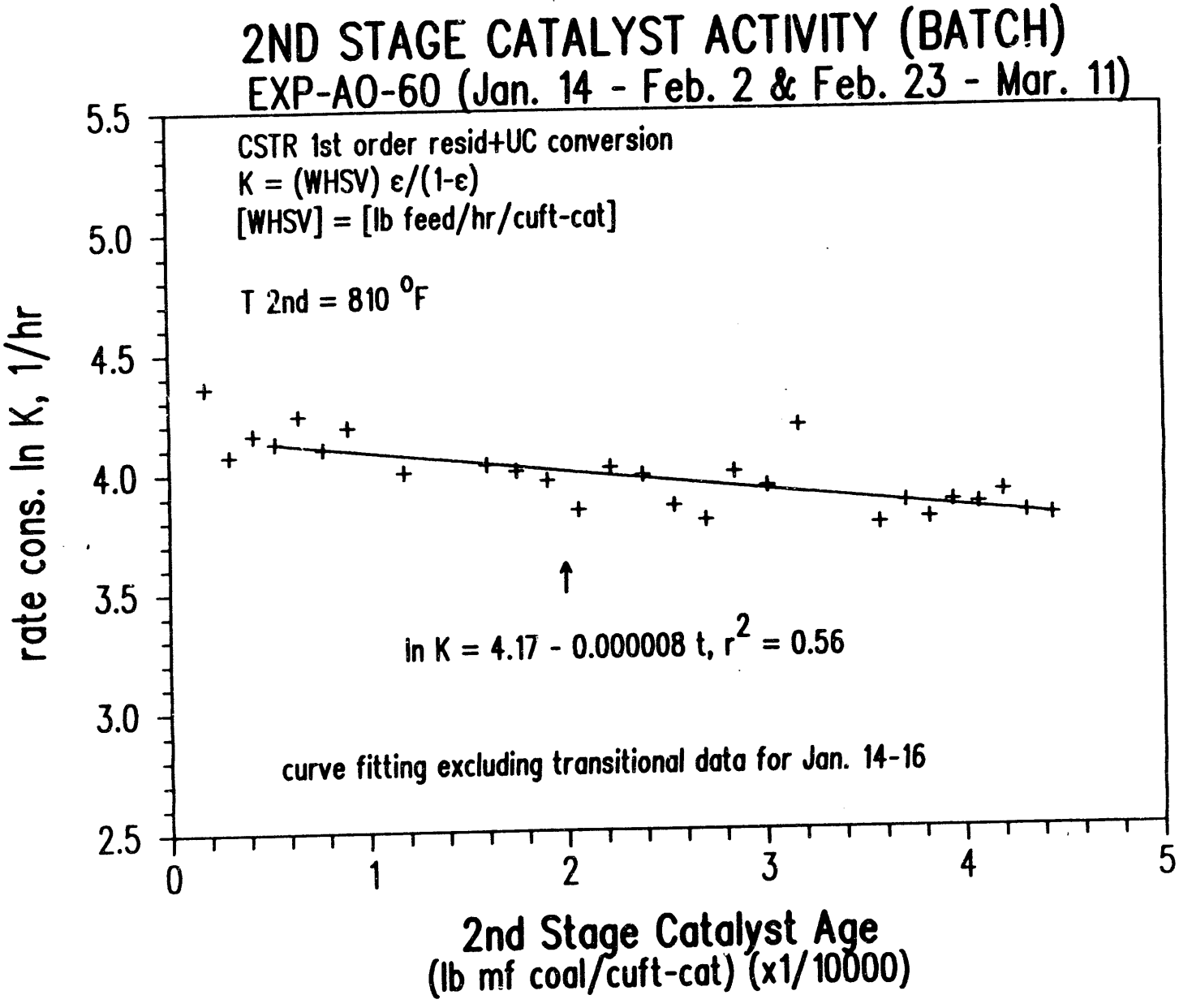


FIGURE 60. LINEAR REGRESSION ANALYSIS OF SECOND STAGE ACTIVITY TREND (EXP-AO-60 CATALYST; BATCH DEACTIVATION)

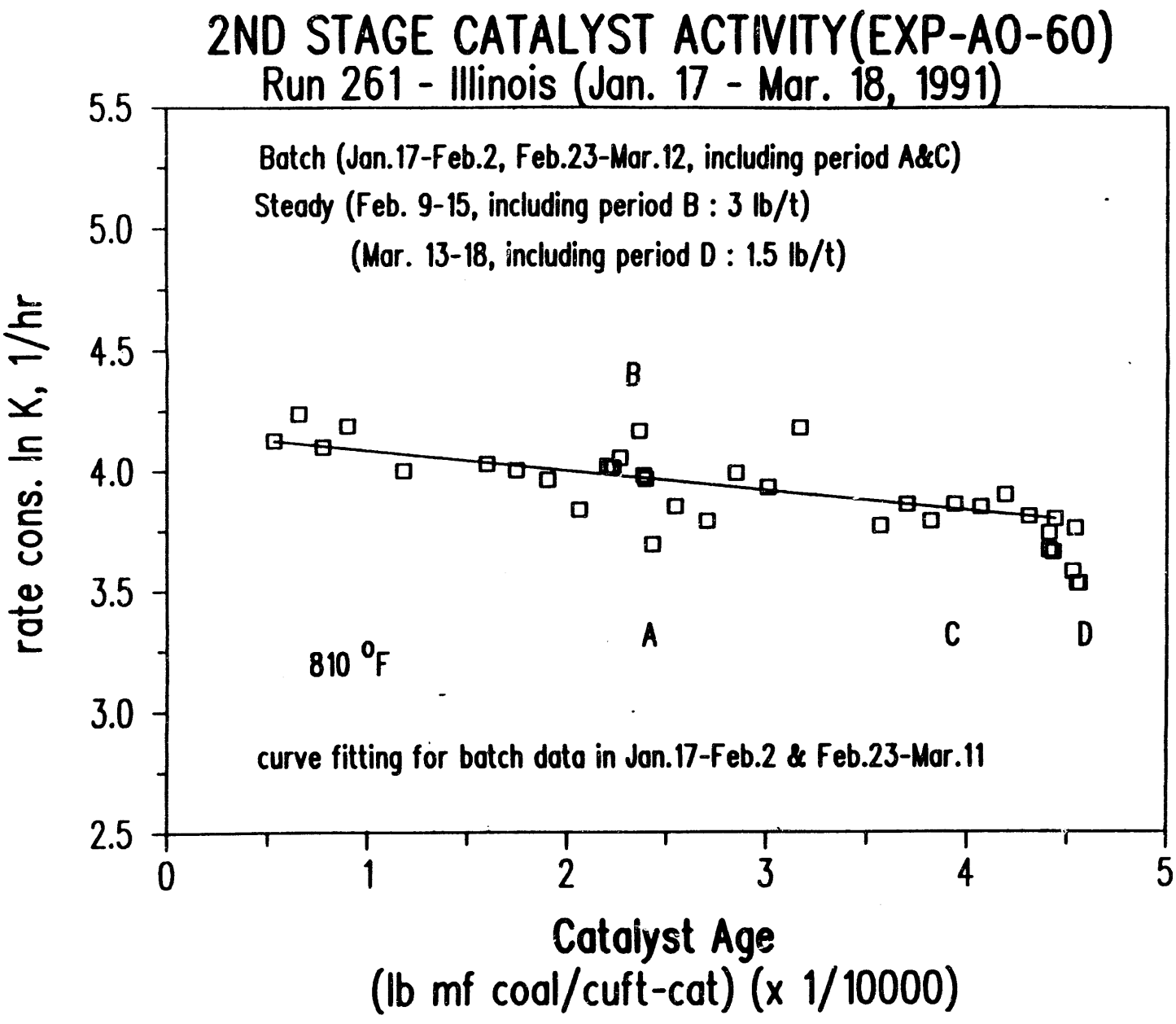


FIGURE 61. SECOND STAGE CATALYST ACTIVITY COMPARISON (EXP-AO-60 CATALYST; BATCH DEACTIVATION VS STEADY-STATE OPERATION; 261A-D)

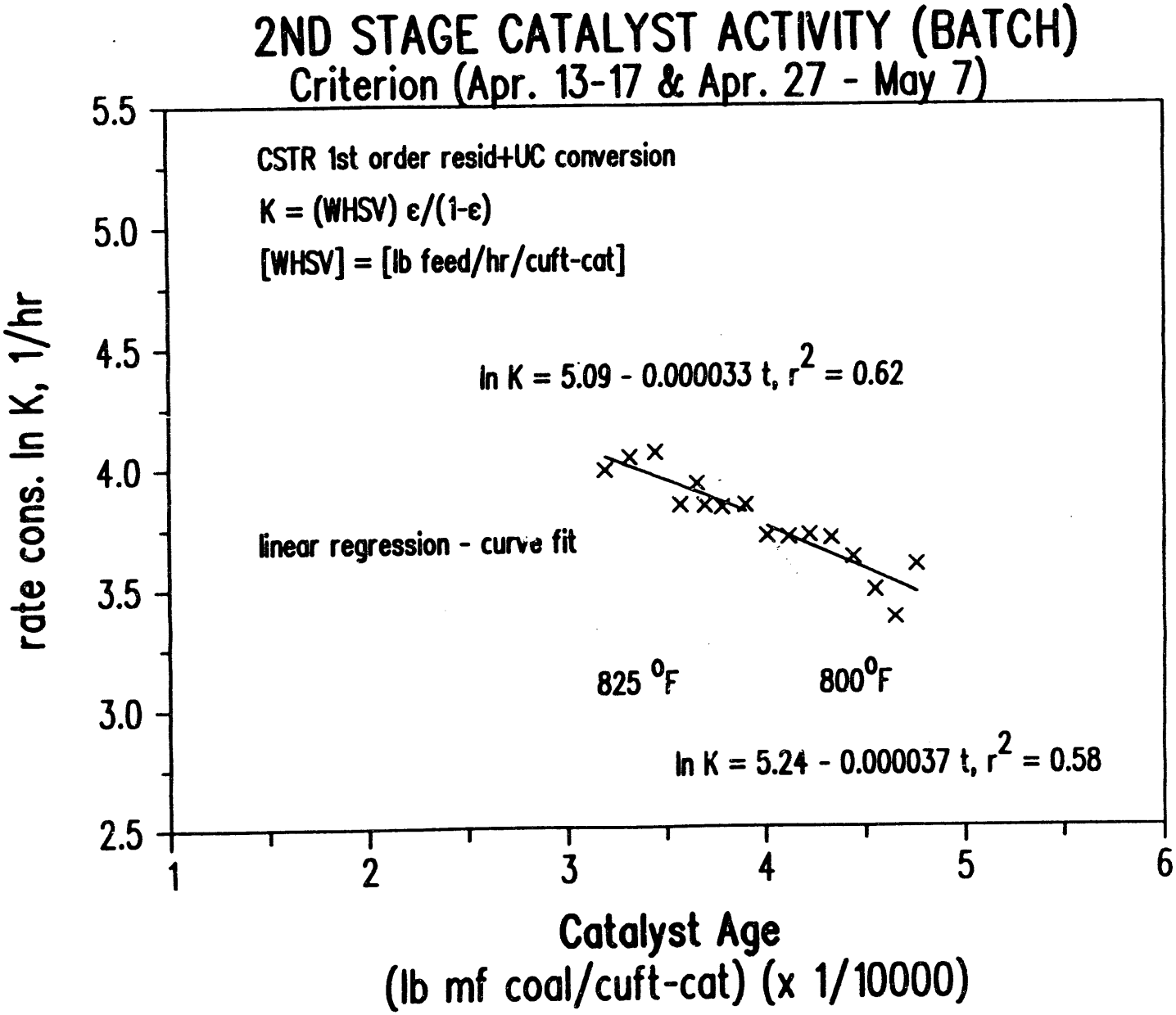


FIGURE 62. LINGAR REGRESSION ANALYSIS OF SECOND STAGE ACTIVITY TREND (CRITERION 324 CATALYST; BATCH DEACTIVATION)

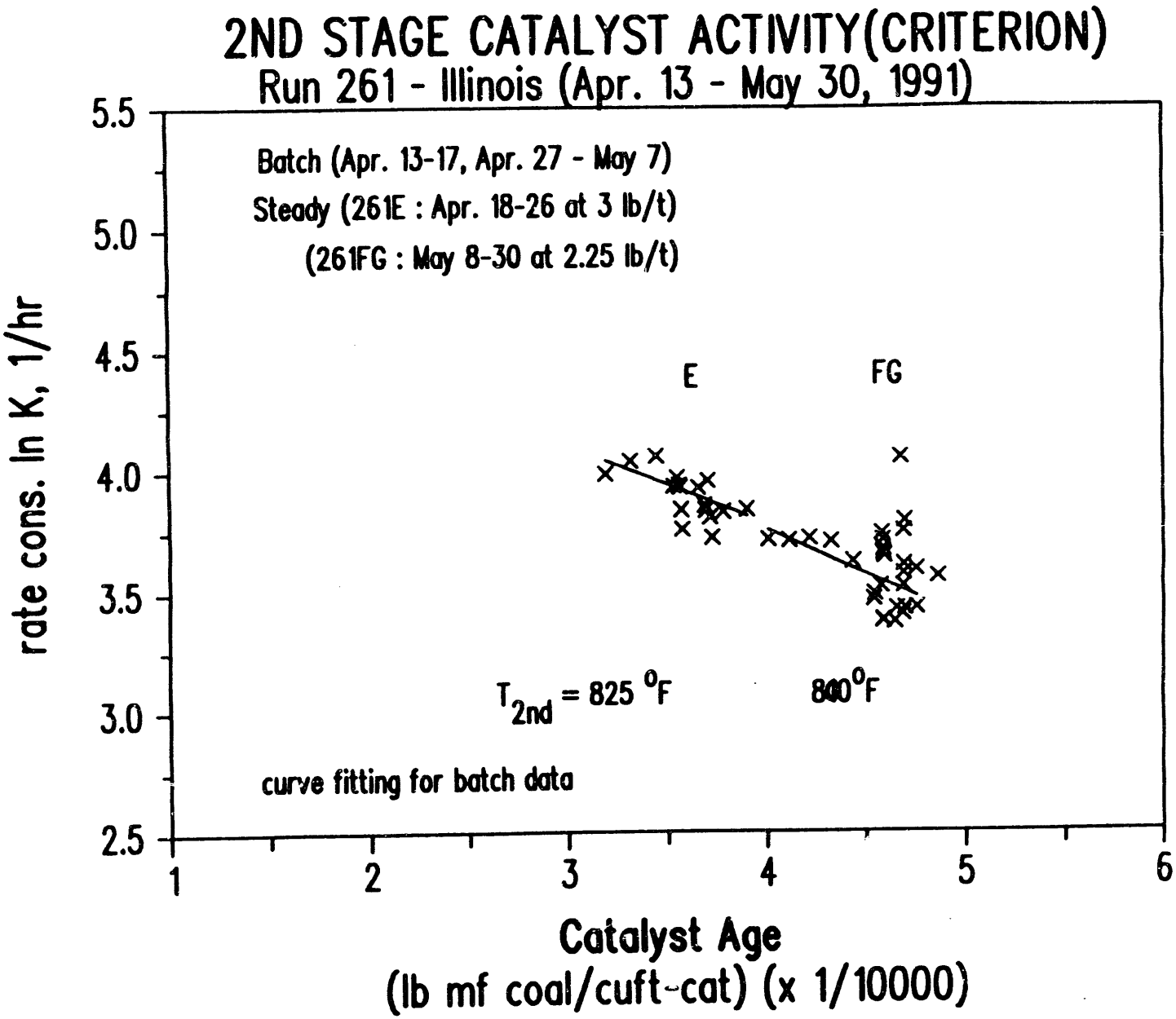


FIGURE 63. SECOND STAGE CATALYST ACTIVITY COMPARISON (CRITERION 324 CATALYST; BATCH DEACTIVATION VS STEADY-STATE OPERATION; 261E-G)

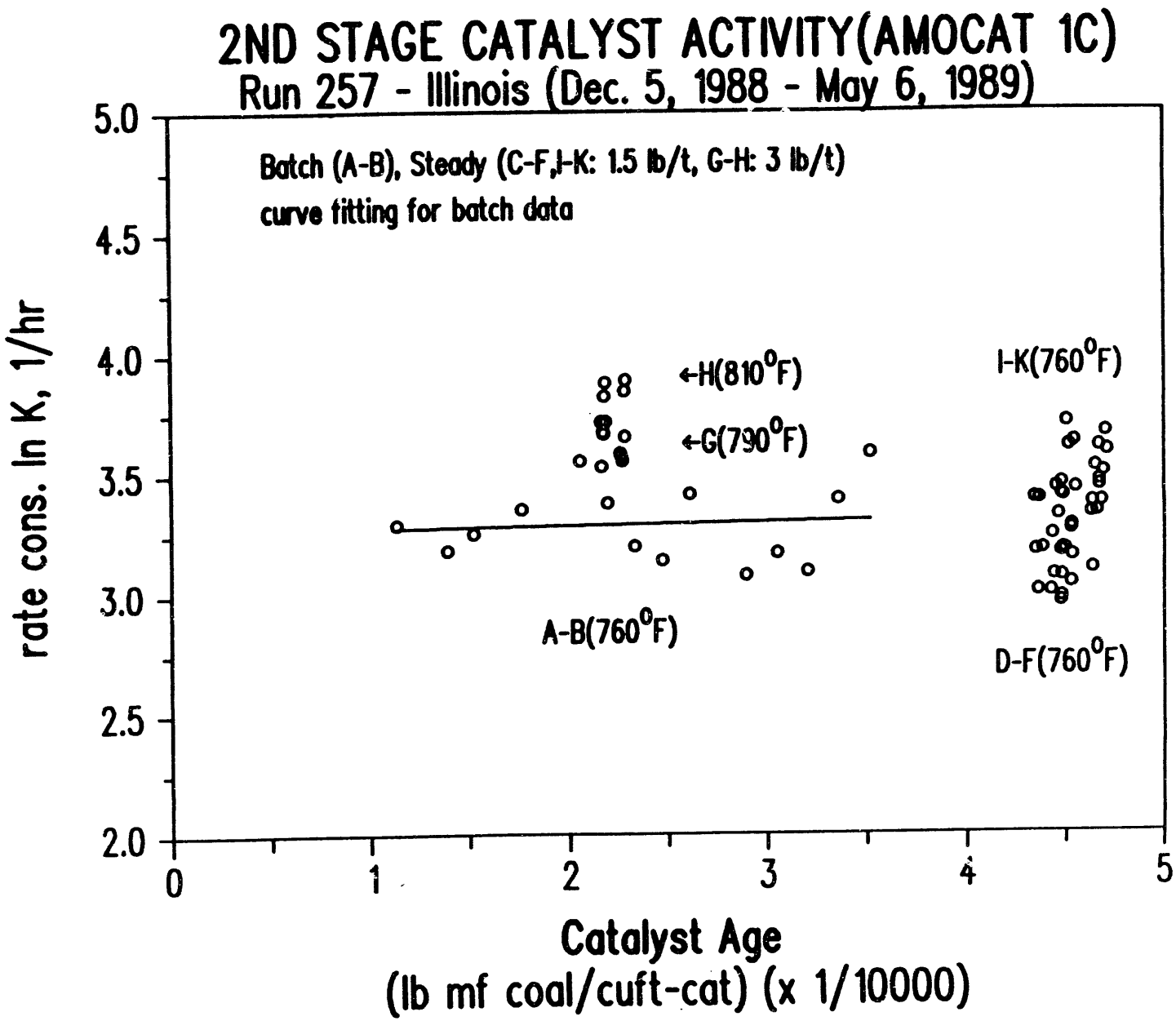


FIGURE 64. SECOND STAGE CATALYST ACTIVITY COMPARISON (AMOCAT 1C CATALYST; BATCH DEACTIVATION VS STEADY-STATE OPERATION; 257A-K)

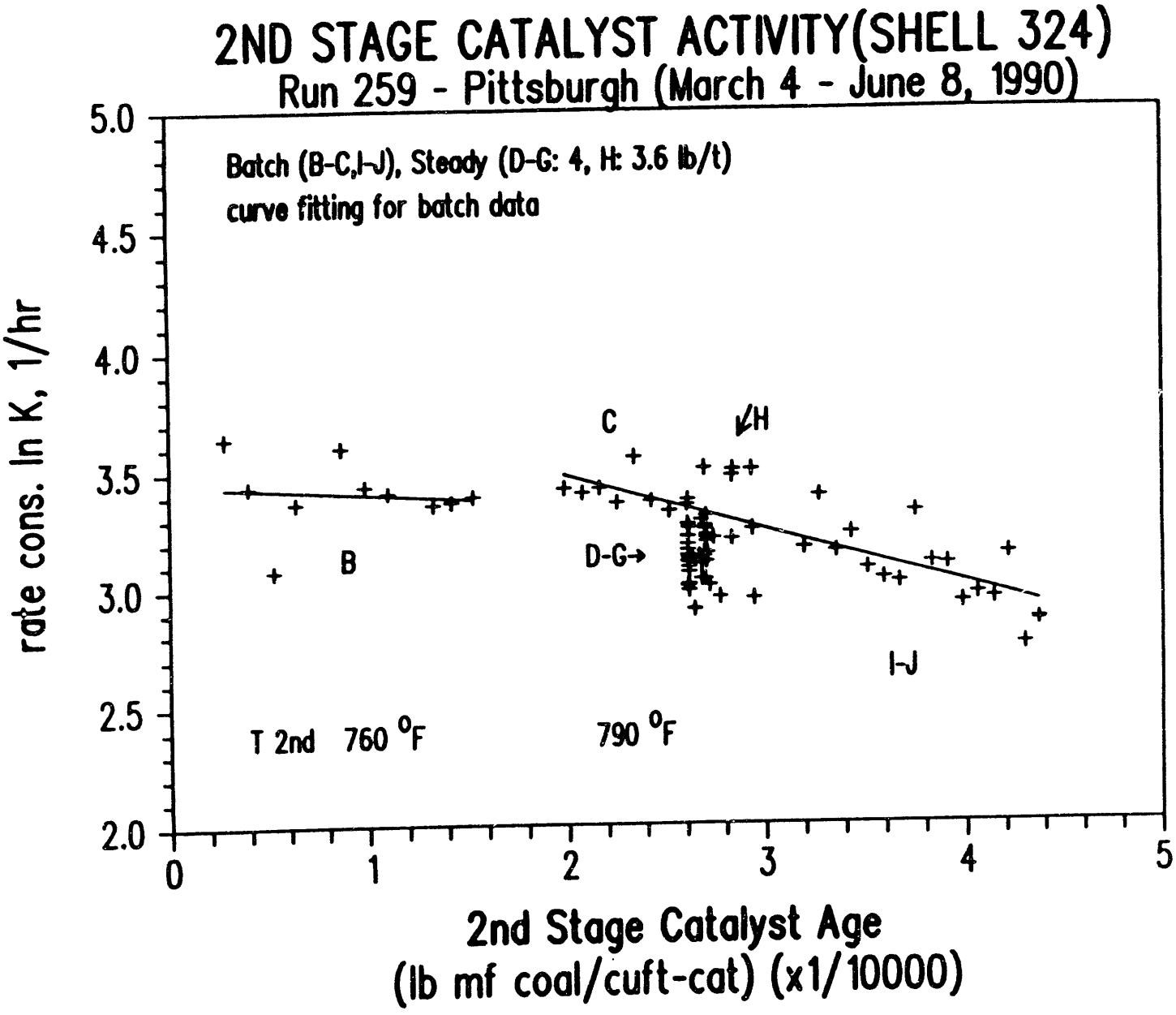


FIGURE 65. SECOND STAGE CATALYST ACTIVITY COMPARISON (SHELL 324 CATALYST; BATCH DEACTIVATION VS STEADY-STATE OPERATION; 259B-J)

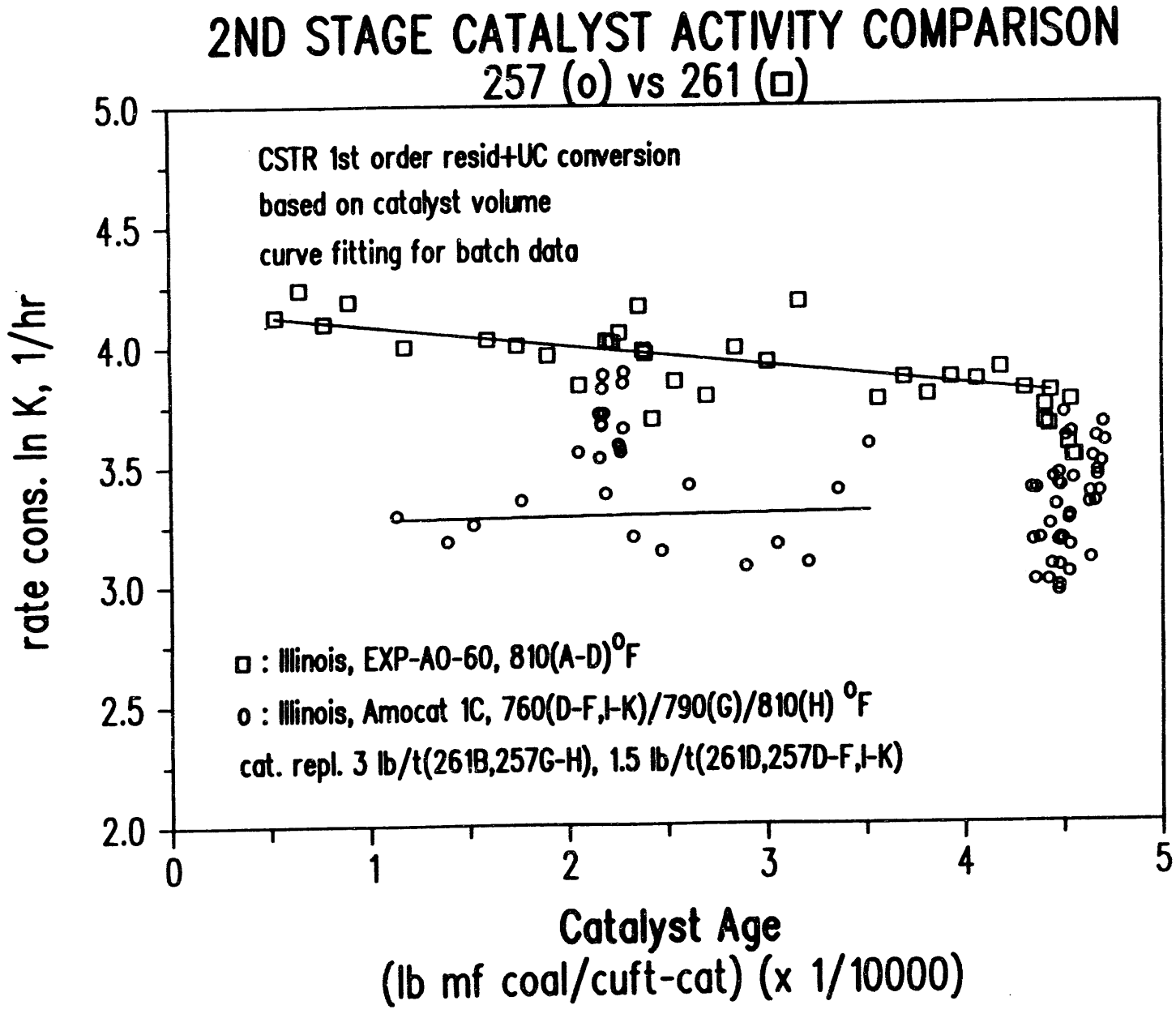


FIGURE 66. SECOND STAGE CATALYST ACTIVITY COMPARISON (RUN 261 VS 257)

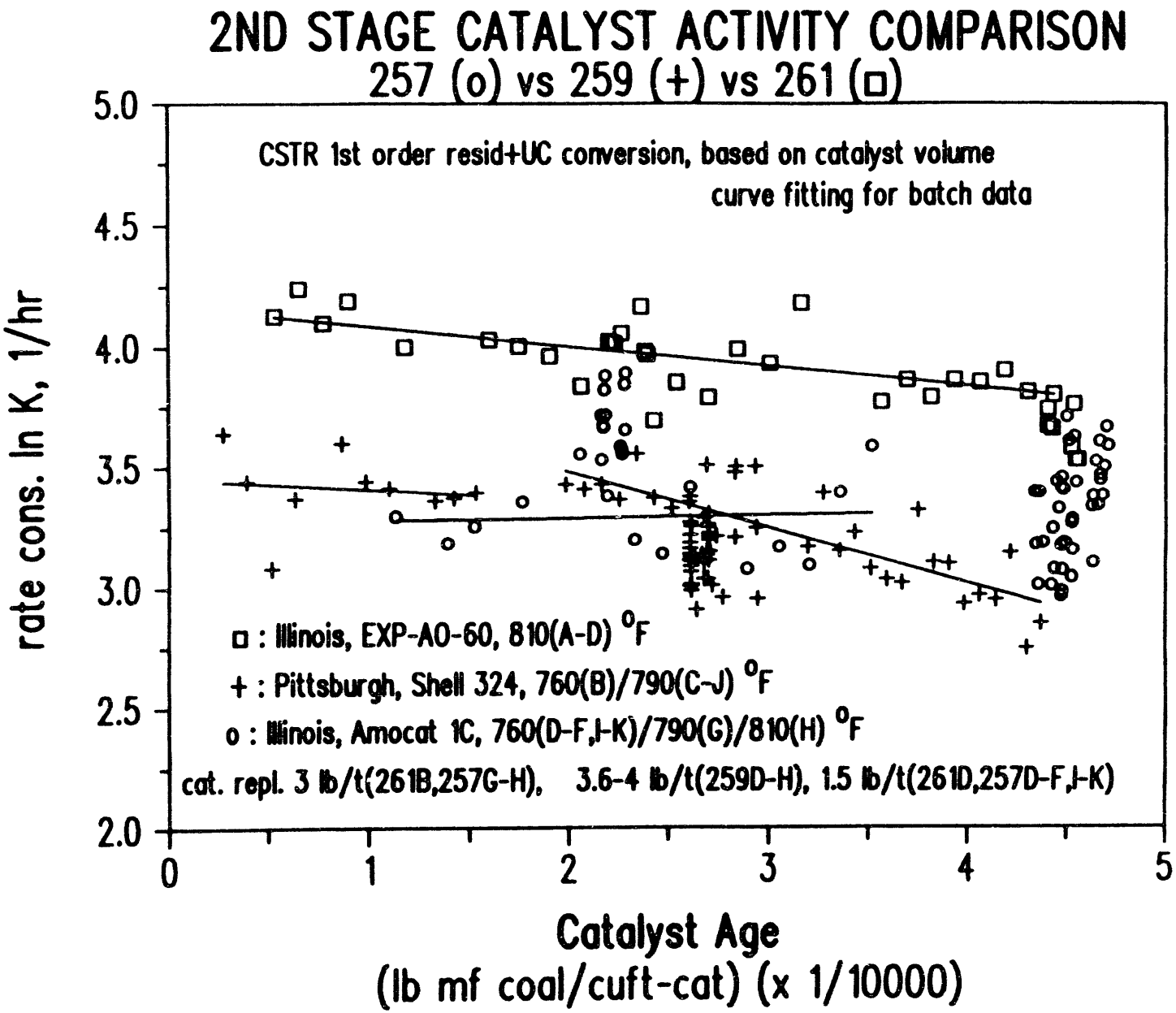


FIGURE 67. SECOND STAGE CATALYST ACTIVITY COMPARISON (RUN 261 VS 259 VS 257)

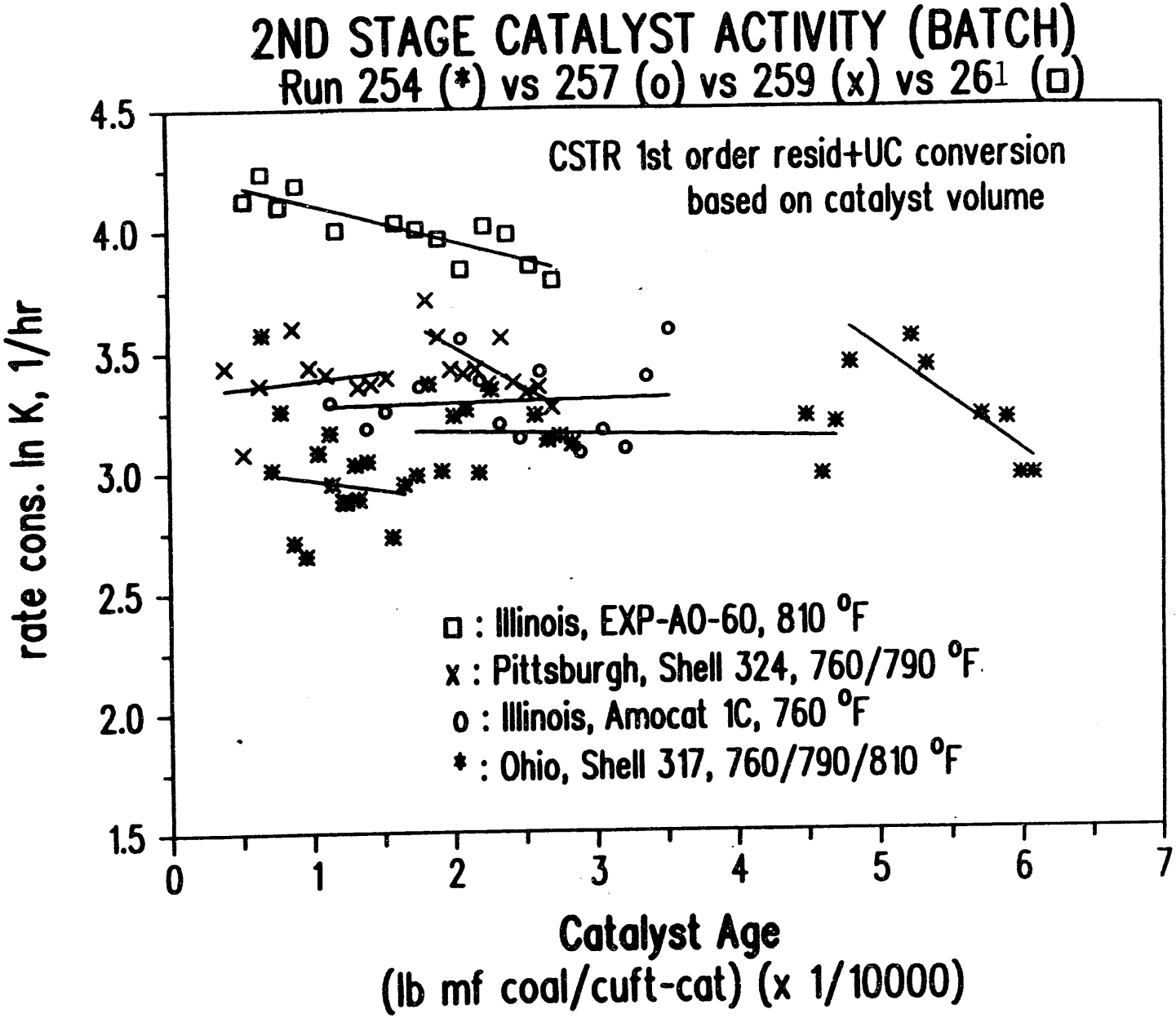


FIGURE 68. SECOND STAGE CATALYST ACTIVITY COMPARISON (BATCH DEACTIVATION) (RUN 261 VS 259 VS 257 VS 254)

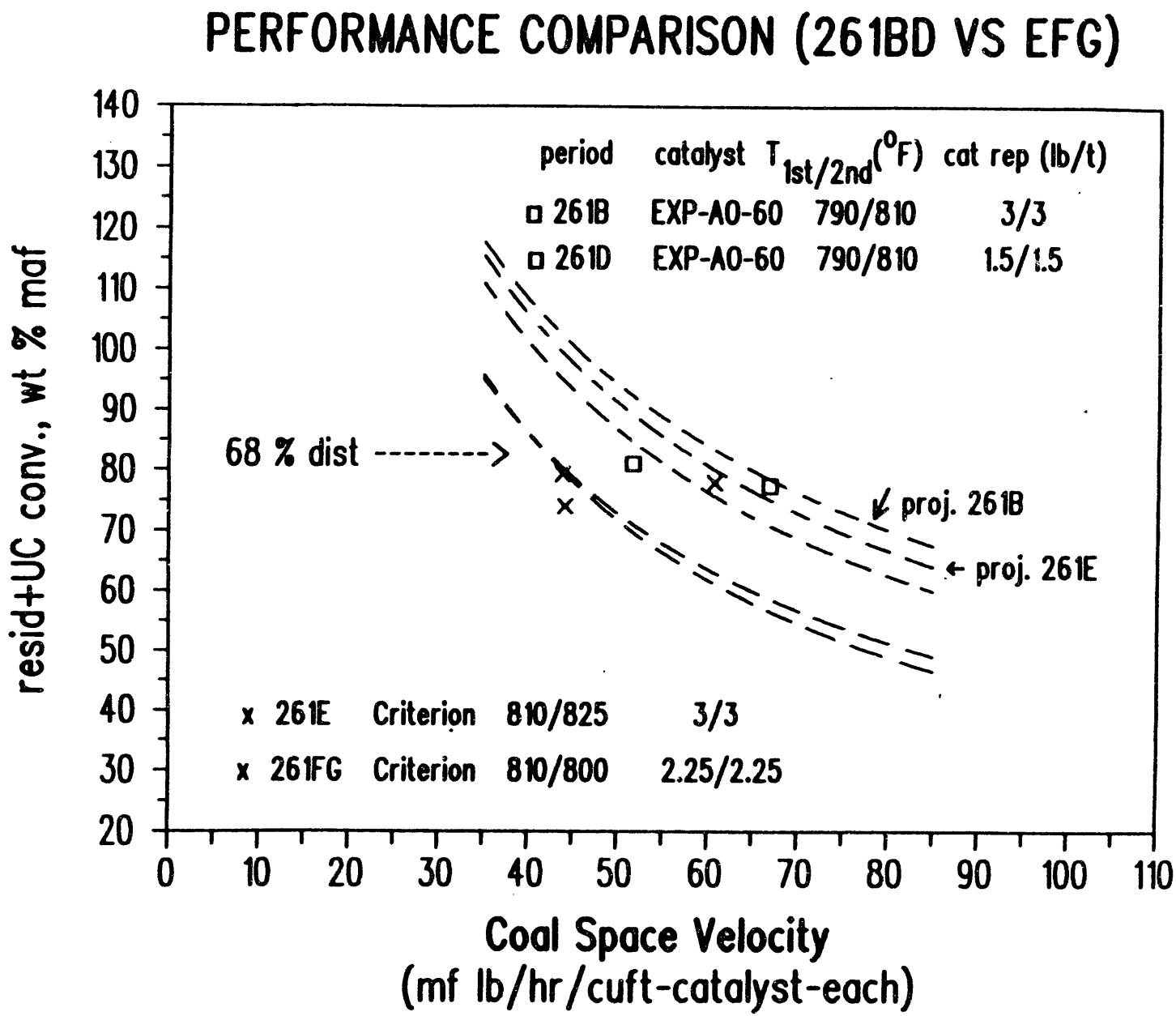


FIGURE 69. OVERALL TWO-STAGE RESID + UC CONVERSION COMPARISON AND PROJECTIONS USING CSTR FIRST ORDER RESID + UC CONVERSION MODEL (261,B,D,E-G)

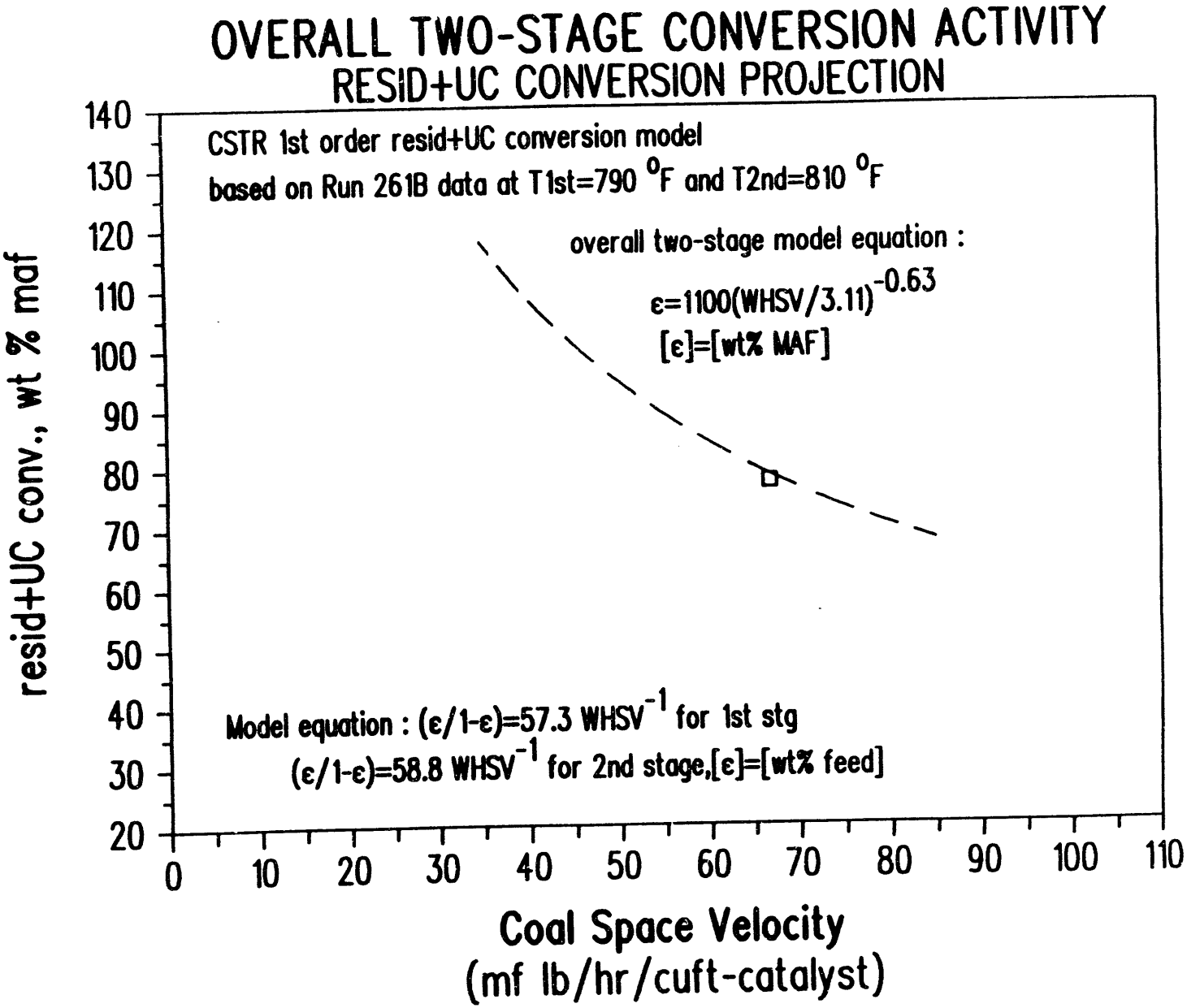


FIGURE 70. PROJECTION USING CSTR FIRST ORDER RESID + UC CONVERSION MODEL (261B)

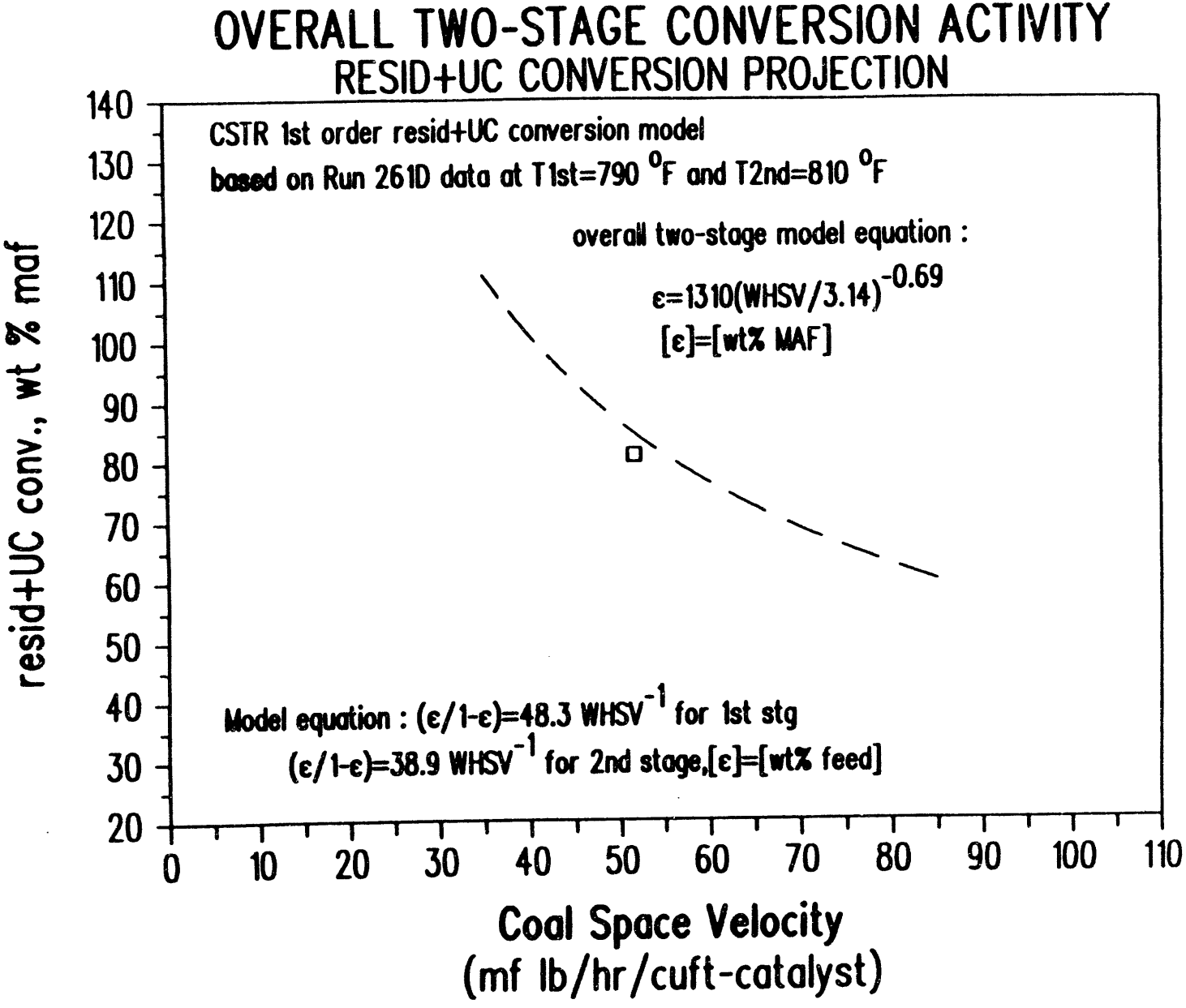


FIGURE 71. PROJECTION USING CSTR FIRST ORDER RESID + UC CONVERSION MODEL (261D)

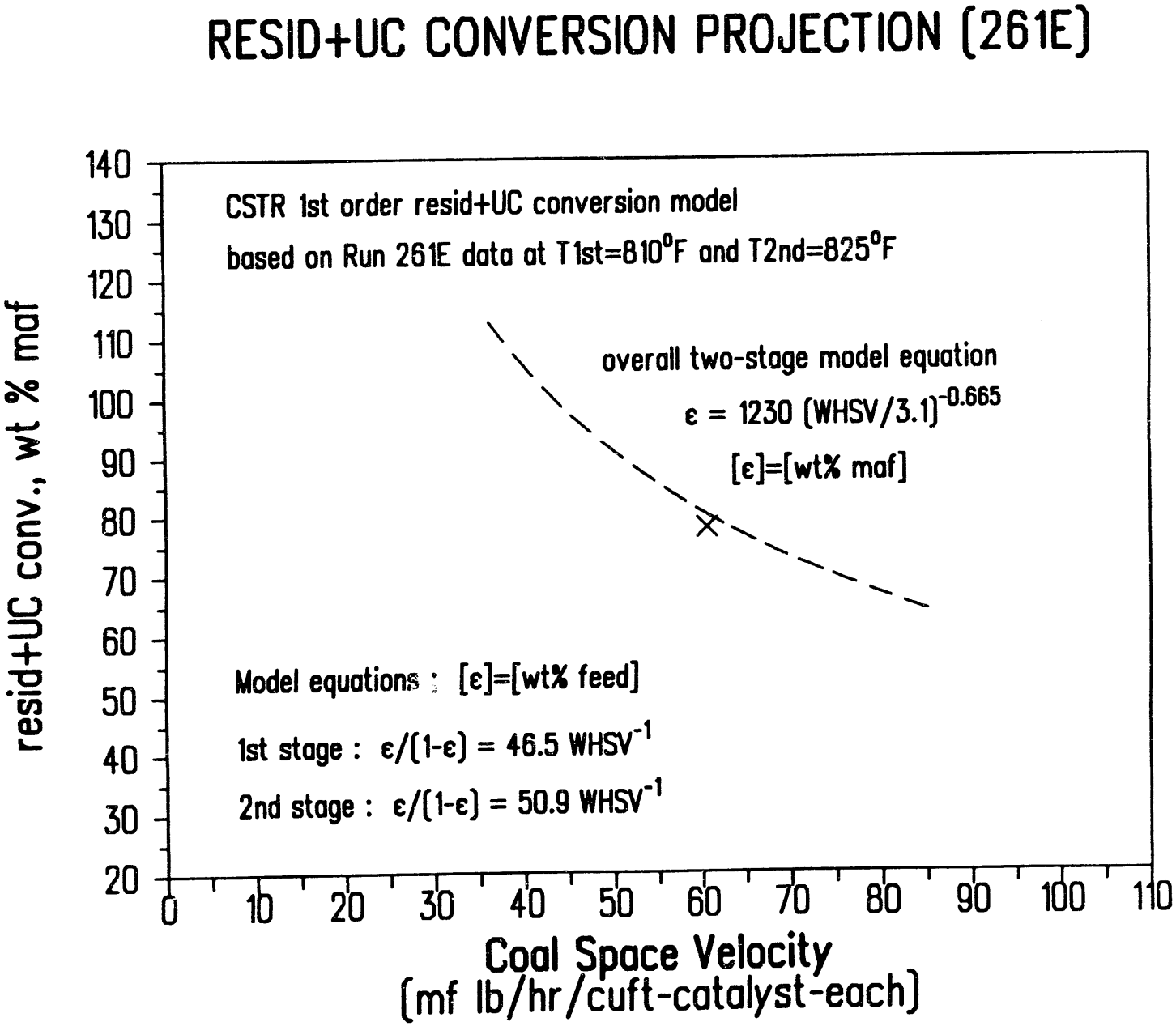


FIGURE 72. PROJECTION USING CSTR FIRST ORDER RESID + UC CONVERSION MODEL (261E)

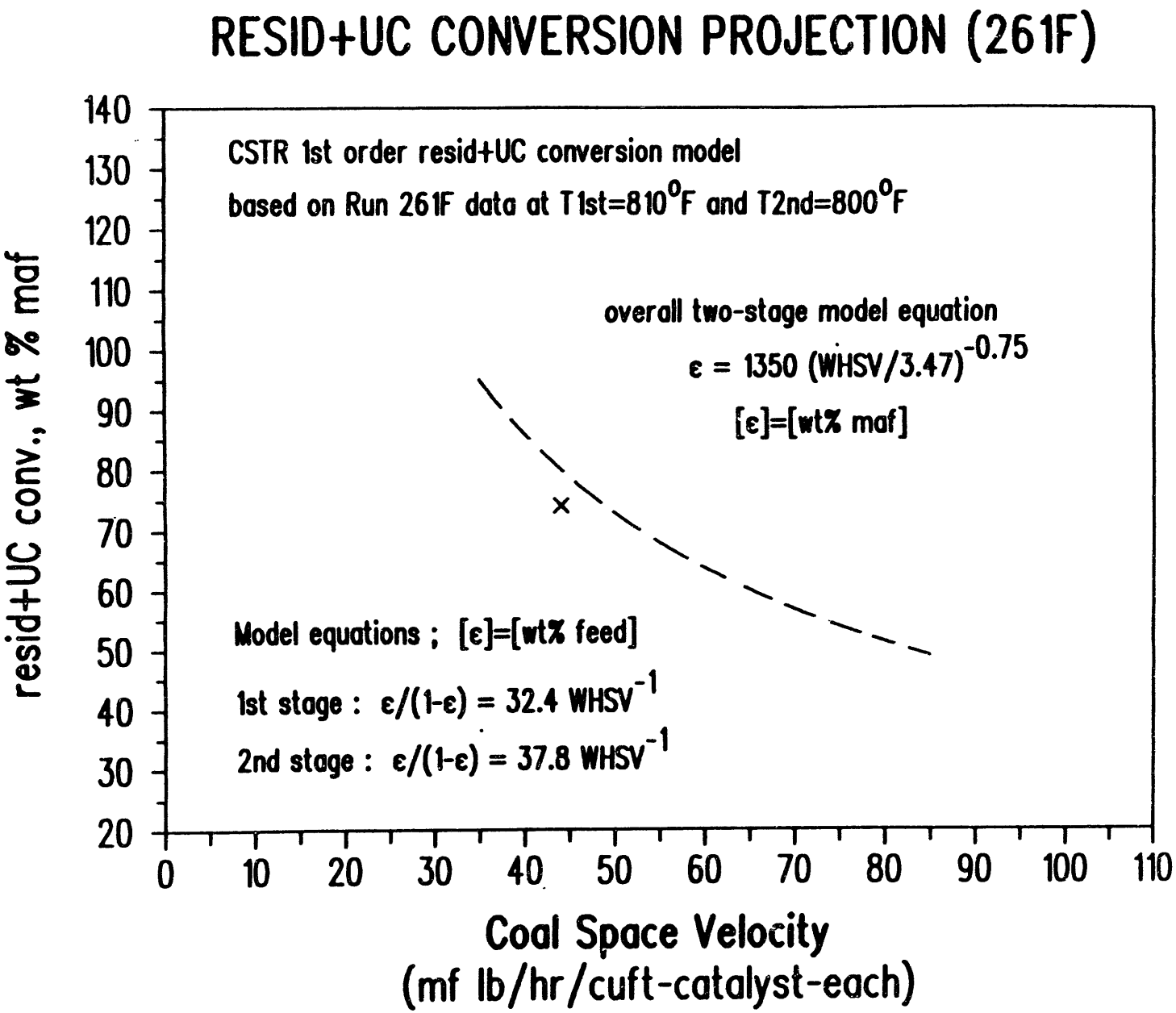


FIGURE 73. PROJECTION USING CSTR FIRST ORDER RESID + UC CONVERSION MODEL (261F)

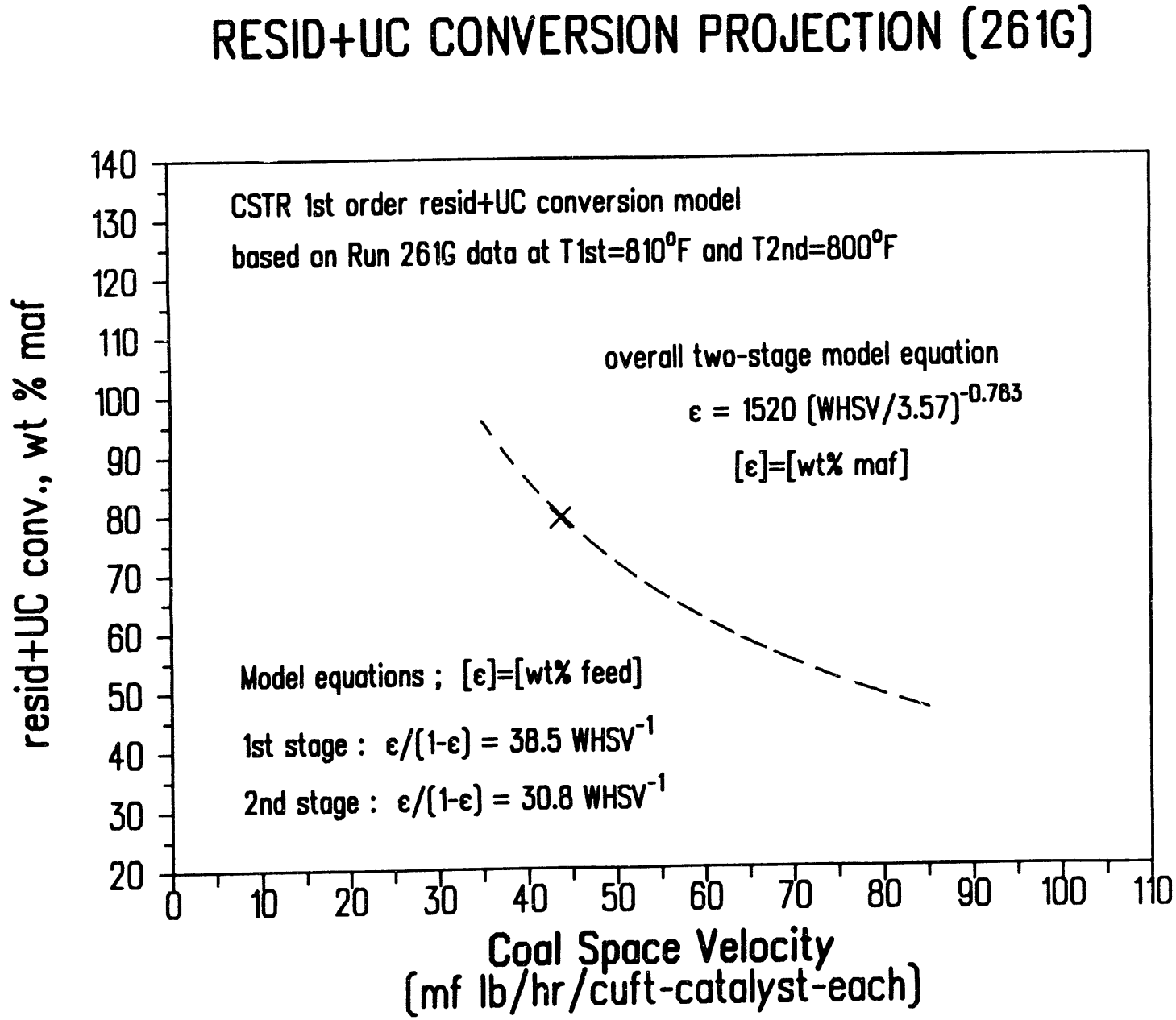


FIGURE 74. PROJECTION USING CSTR FIRST ORDER RESID + UC CONVERSION MODEL (261G)

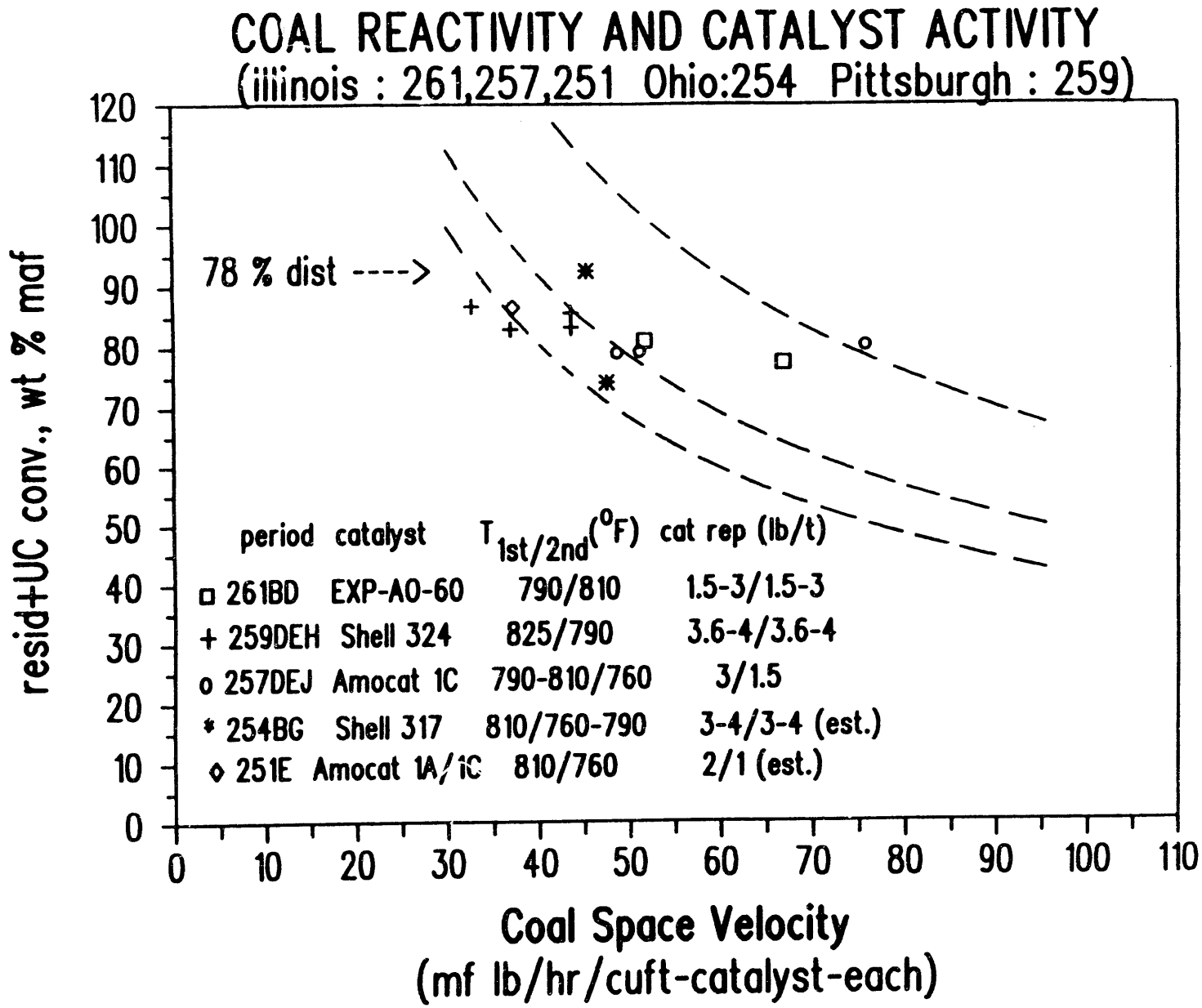
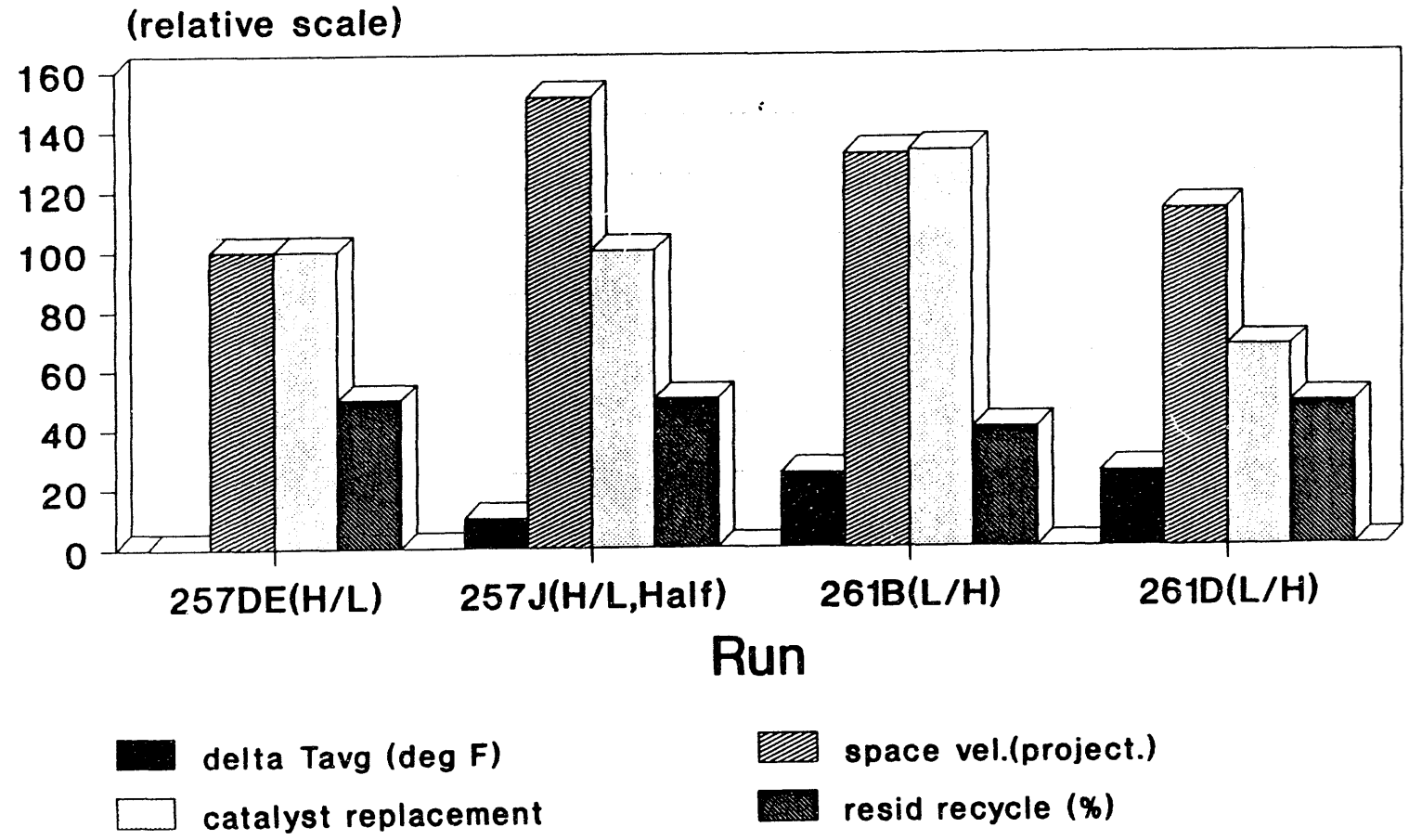


FIGURE 75. COAL REACTIVITY AND CATALYST ACTIVITY COMPARISON FOR "ALL-DISTILLATE" PRODUCT SLATE (RUN 261BD VS 259 DEH VS 257 DEJ VS 254BG VS 251-IE)

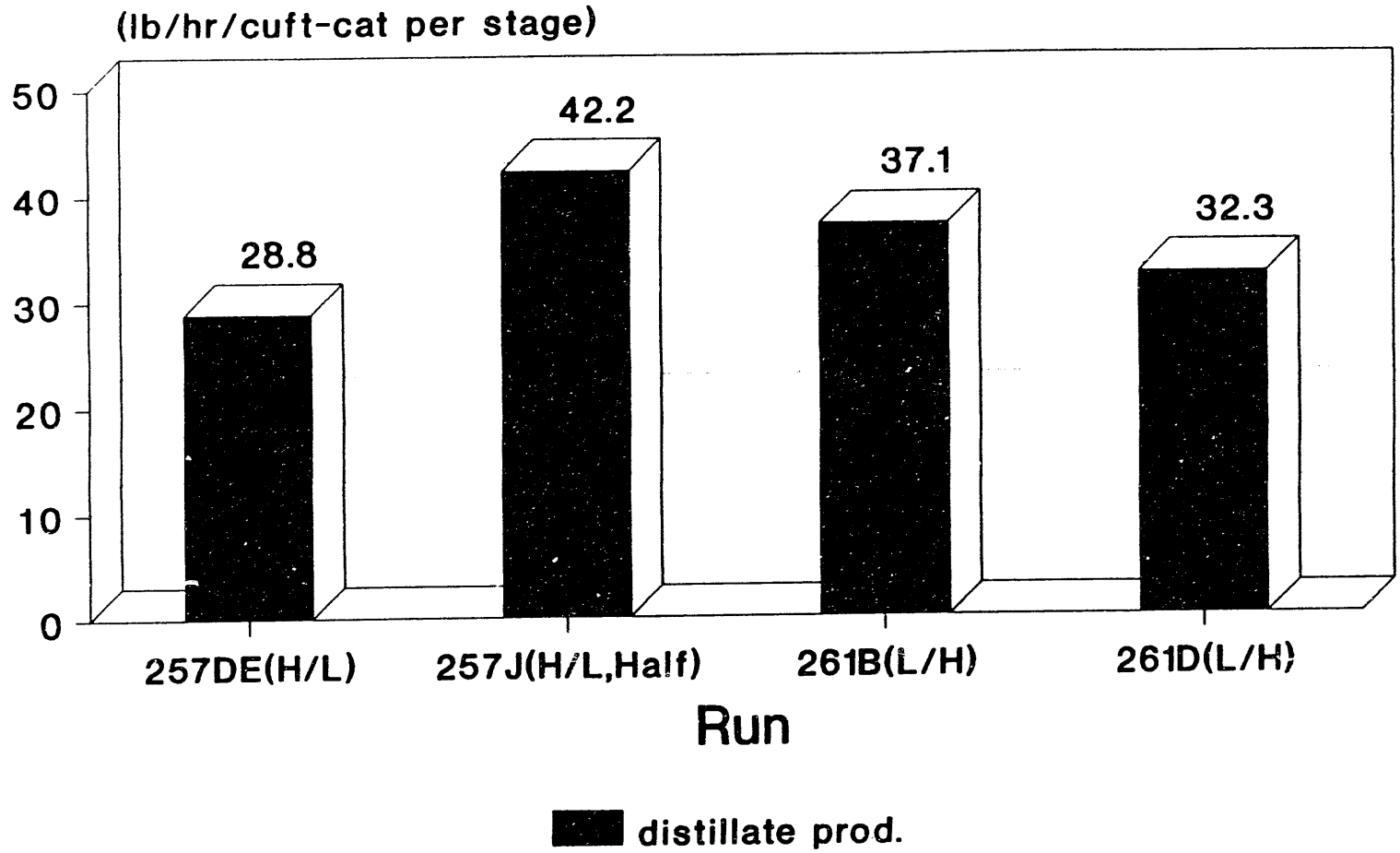
Operating Conditions with Illinois No. 6
Projection for "All-Distillate" Product



* Amocat 1C (257); EXP-AO-60 (261)

FIGURE 76. COMPARISON OF OPERATING CONDITIONS WITH ILLINOIS COAL (HIGH-ASH)

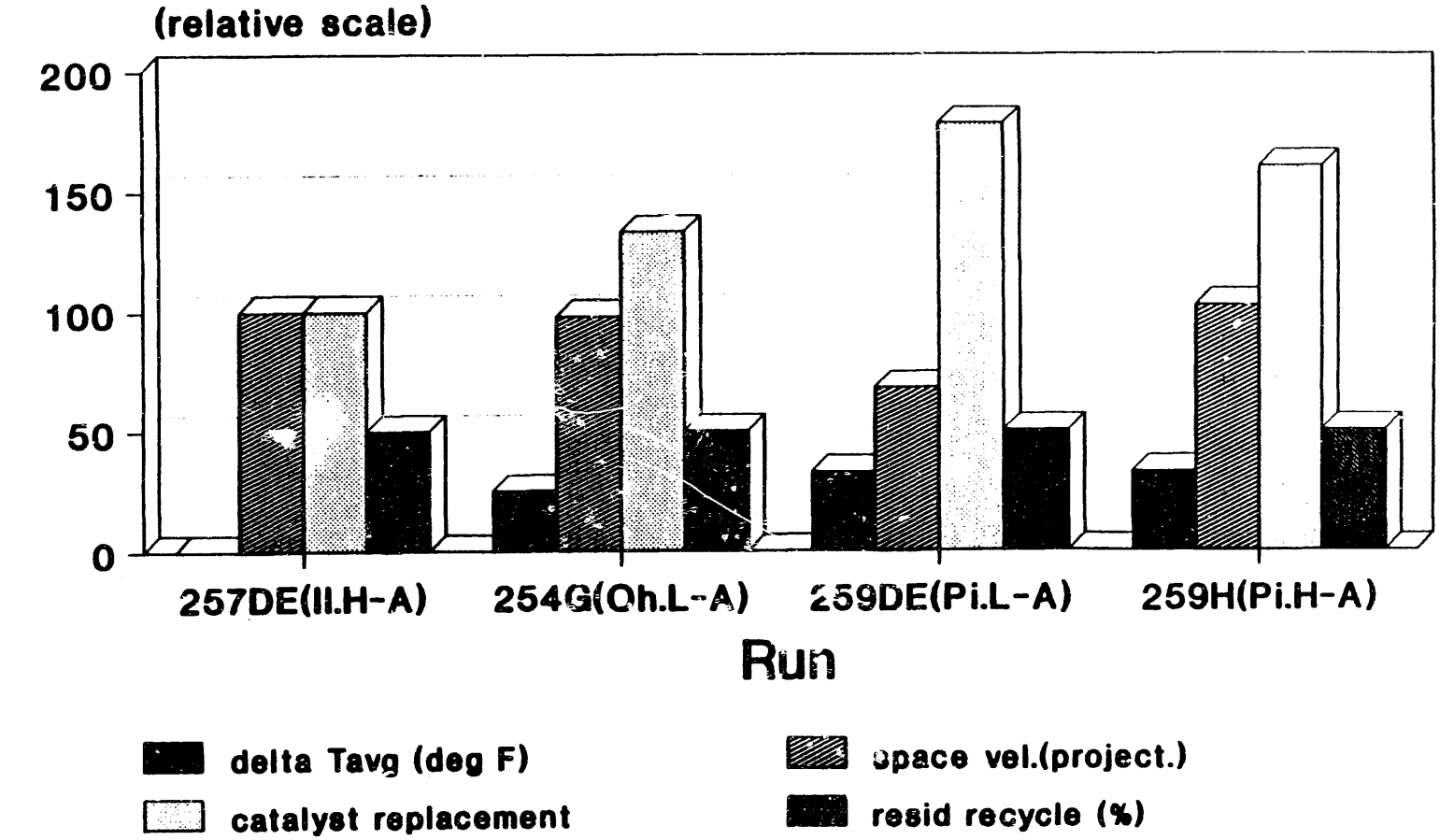
Distillate Production Comparison
Projection with Illinois No. 6 Coal



* 67-70% distillate; 15-18% OR

FIGURE 77. COMPARISON OF DISTILLATE PRODUCTIONS WITH ILLINOIS COAL (HIGH-ASH)

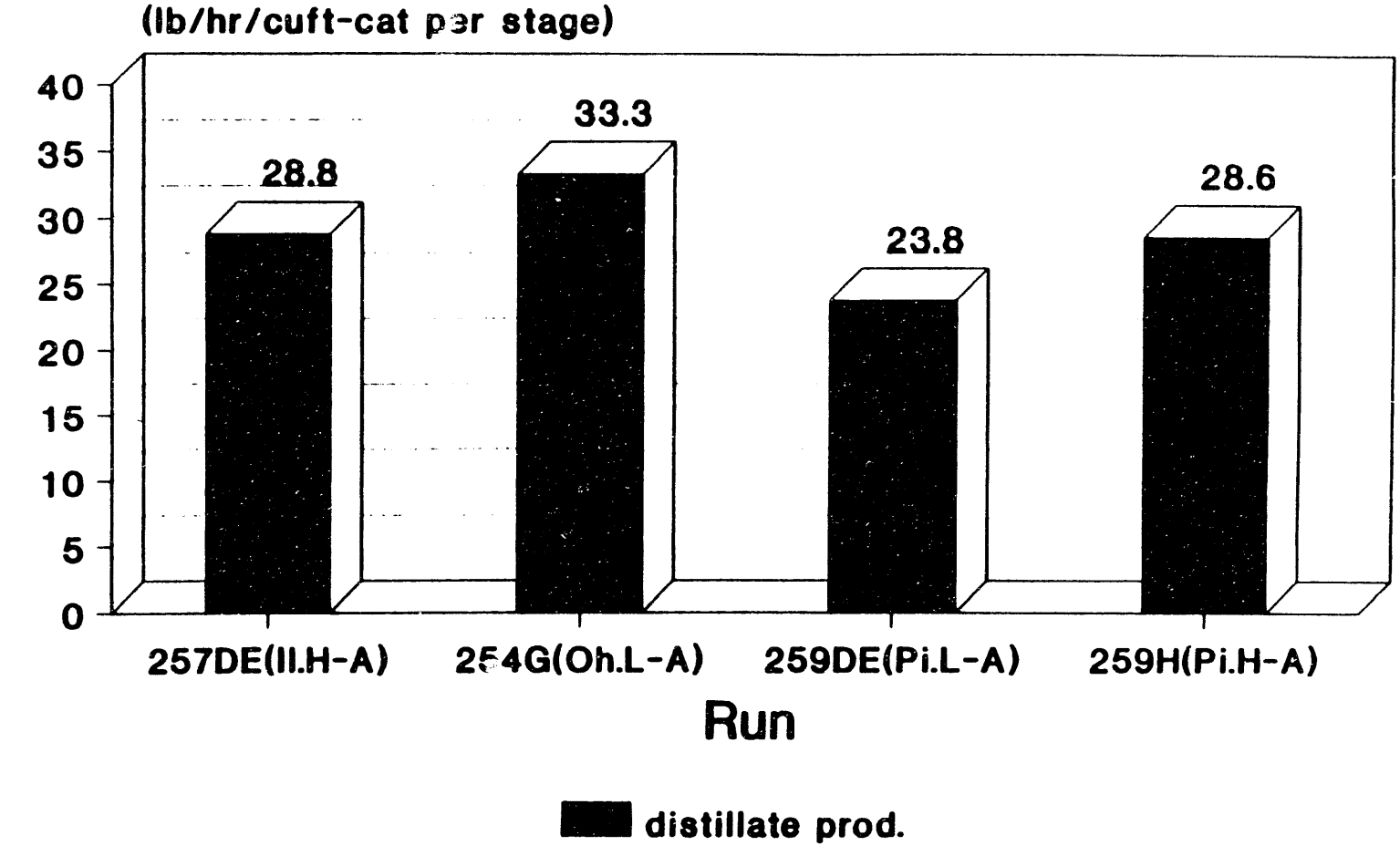
Operating Conditions
Ohio and Pittsburgh Coals



• Shell 317(254);324(259);Amocat 1C(257)

FIGURE 78. COMPARISON OF OPERATING CONDITIONS WITH OHIO AND PITTSBURGH COALS (HIGH-ASH AND LOW-ASH)

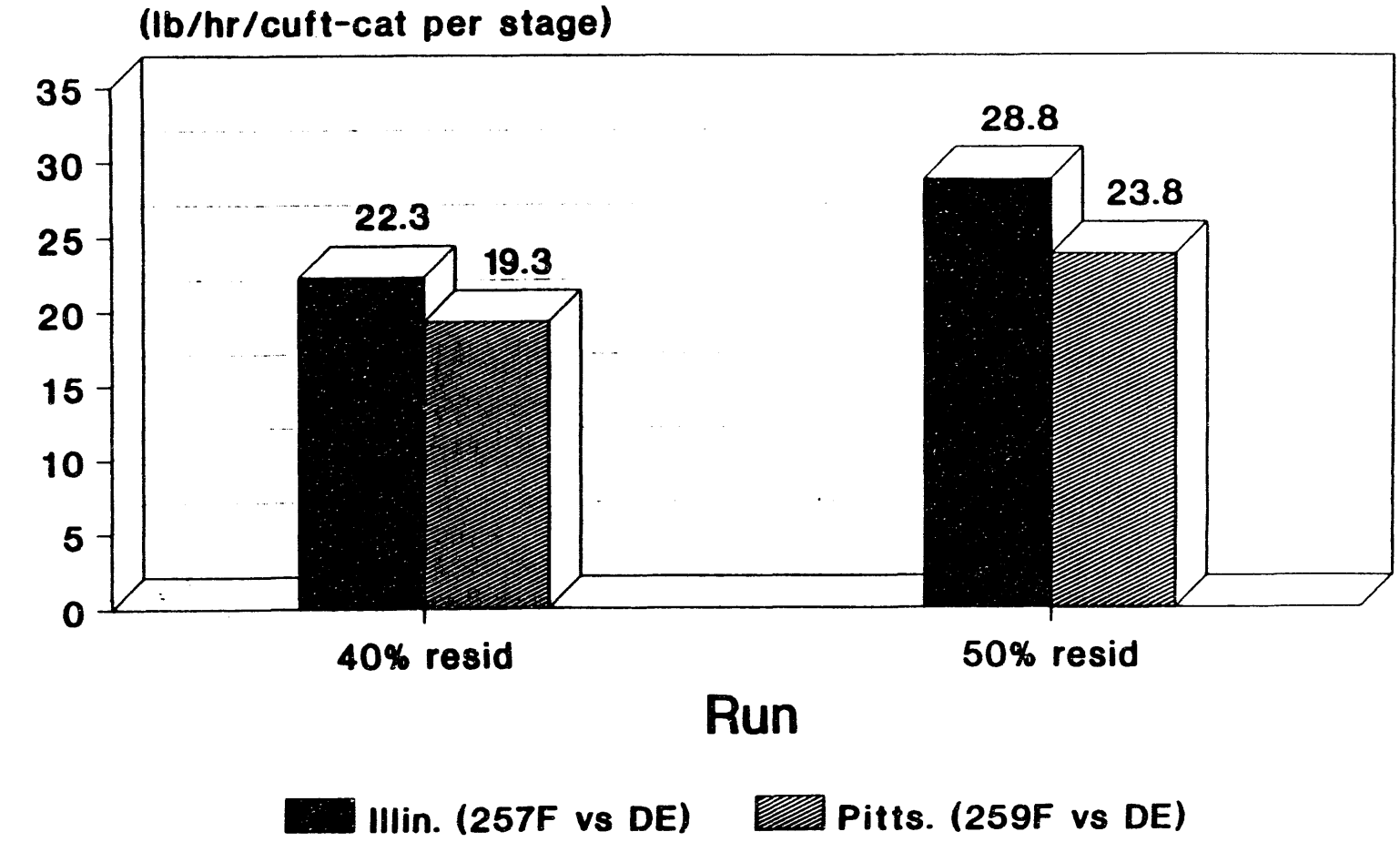
Distillate Production (Projection)
Ohio and Pittsburgh Coals



- **low-ash coals: 78% distillate, 8-9% OR**

FIGURE 79. DISTILLATE PRODUCTION COMPARISON WITH OHIO AND PITTSBURGH COALS (HIGH-ASH AND LOW-ASH)

Effect of Recycle Resid Concentration
On Distillate Production



- Amocat 1C (257); Shell 324 (259)

FIGURE 80. EFFECT OF RECYCLE RESID CONCENTRATION ON DISTILLTE PRODUCTION (RUN 257D-F AND 259D-F)

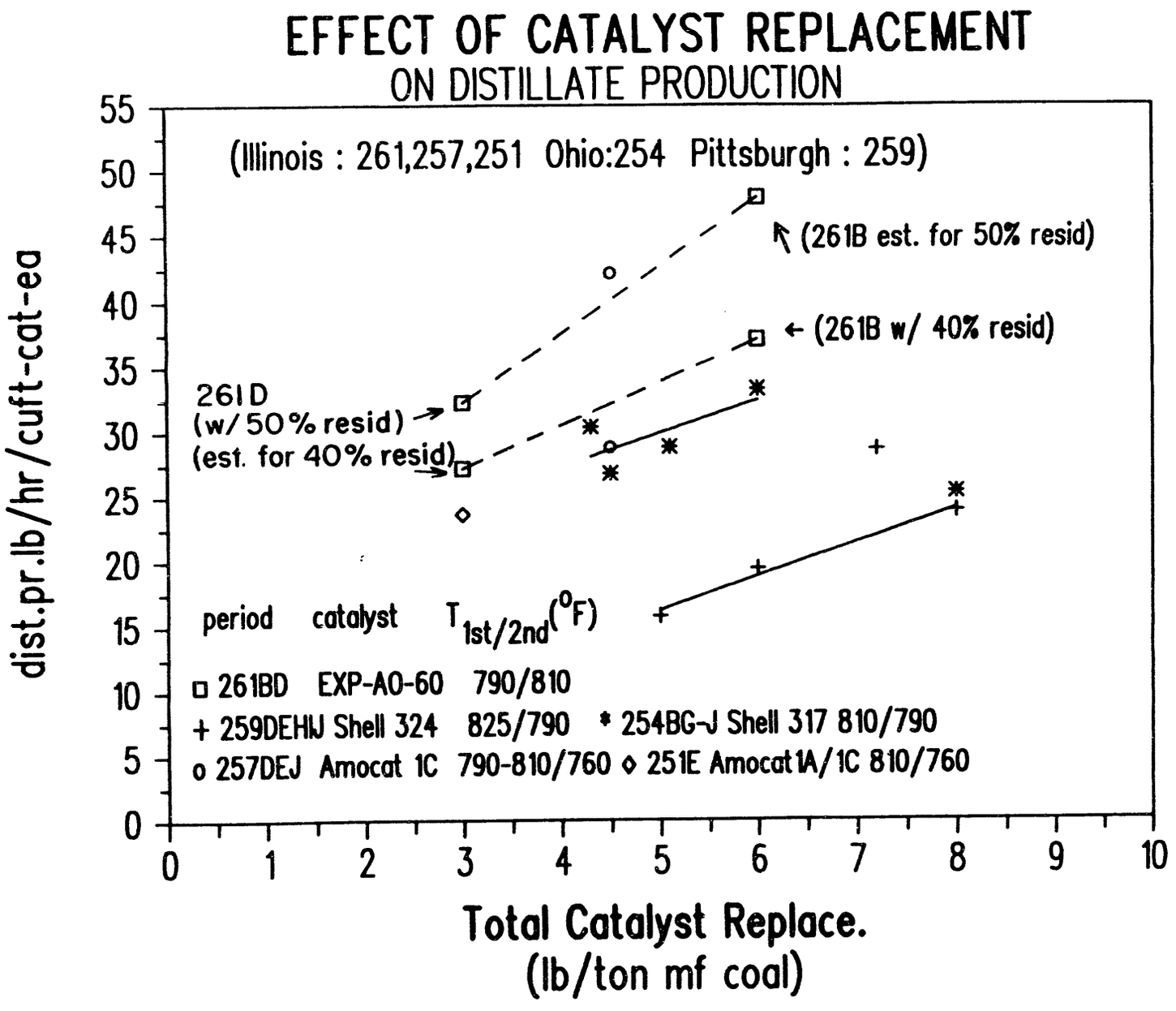
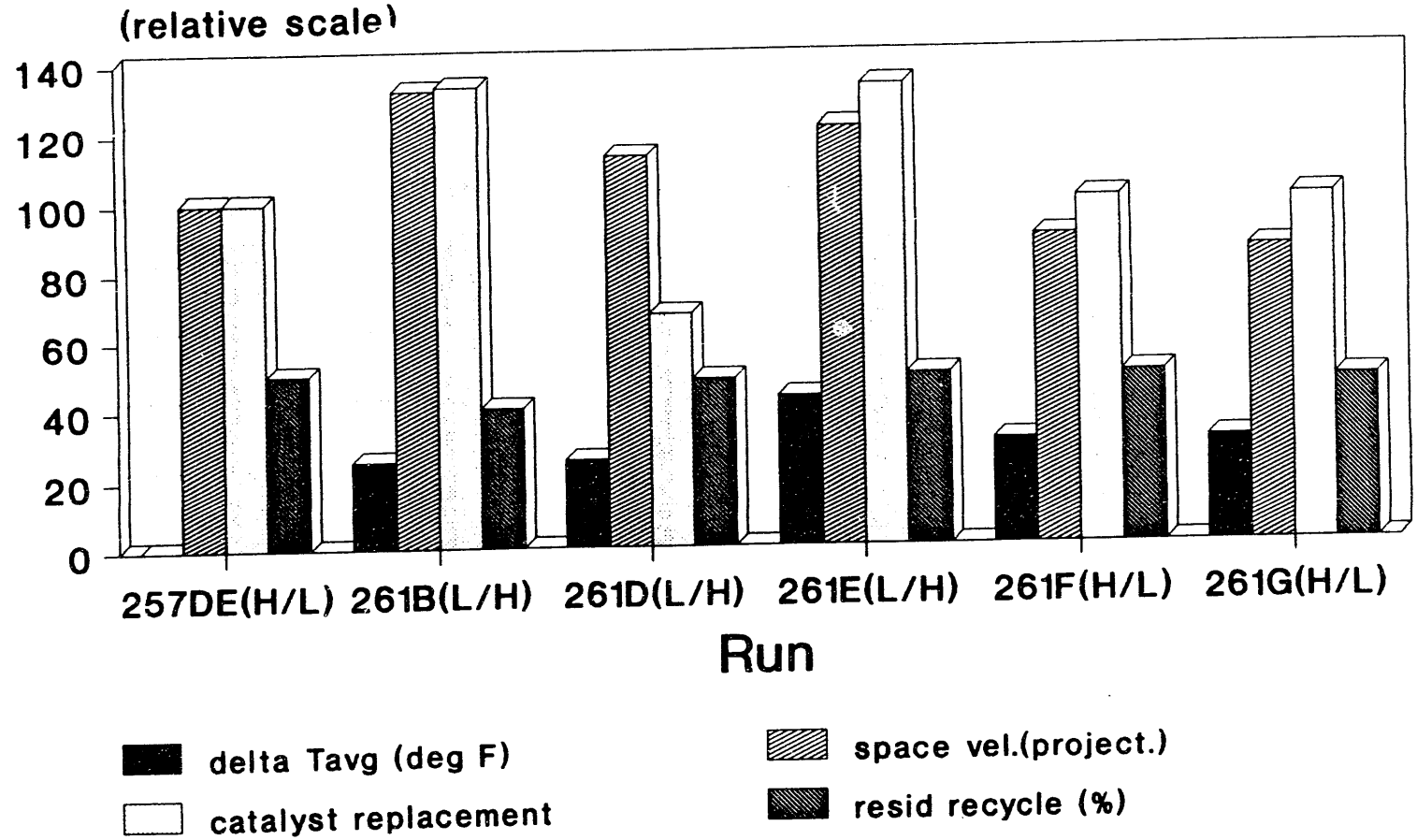


FIGURE 81. EFFECT OF CATALYST REPLACEMENT ON DISTILLATE PRODUCTION (RUN 261BD, 259DEIJ, and 254G-J)

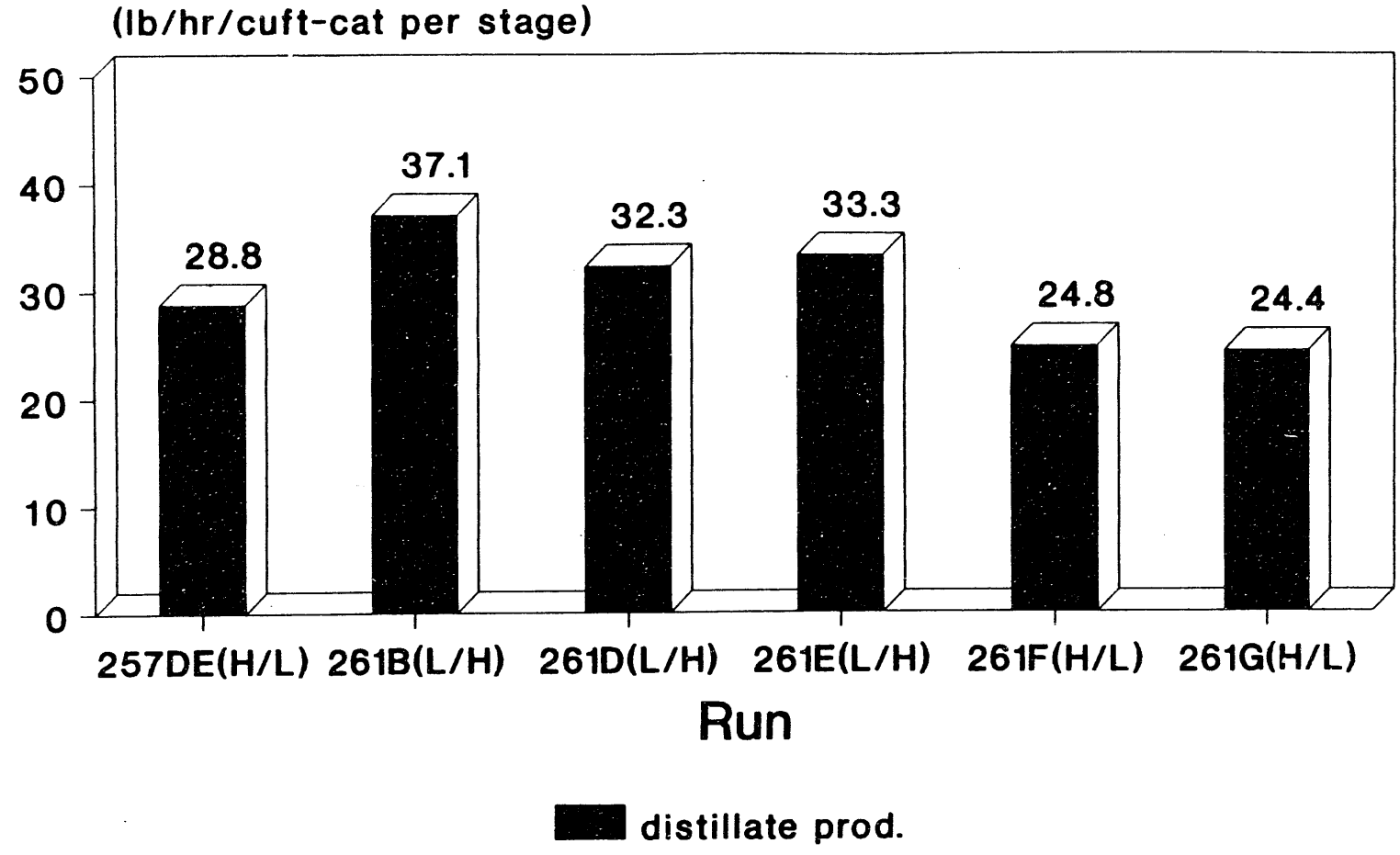
Operating Conditions with Illinois No. 6
Projection for "All-Distillate" Product



*Amo1C(257);EXPAO(261BD);Criterion(EFG)

FIGURE 82. COMPARISON OF OPERATING CONDITIONS WITH ILLINOIS COAL (EXP-AO-60 BIMODAL VS CRITERION 324 UNIMODAL CATALYST)

Distillate Production Comparison
Projection with Illinois No. 6 Coal



* 66-69% distillate; 15-18% OR

FIGURE 83. DISTILLATE PRODUCTION COMPARISON WITH ILLINOIS COAL (EXP-AO-60 BIMODAL VS CRITERION 324 UNIMODAL CATALYST)

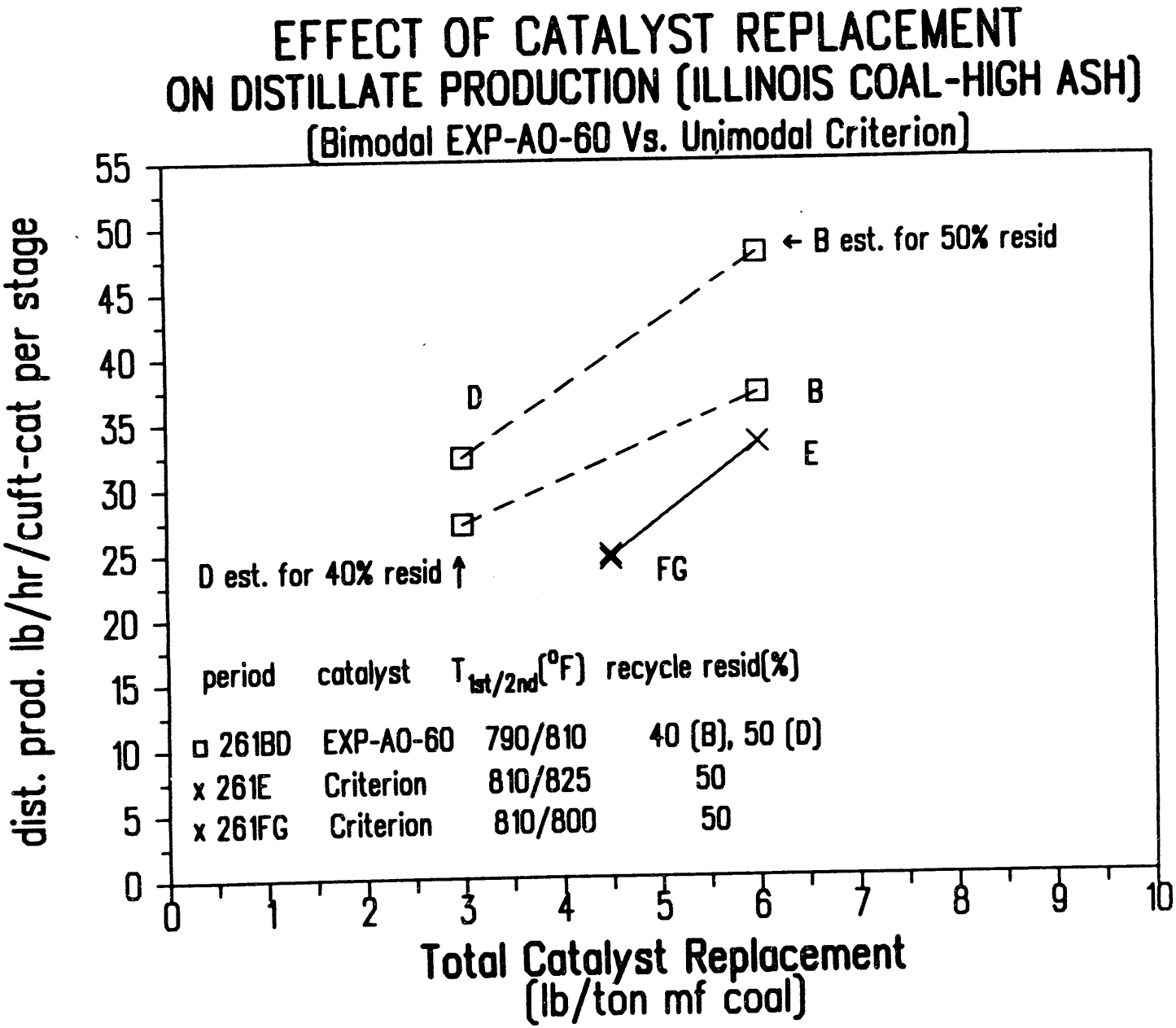


FIGURE 84. EFFECT OF CATALYST REPLACEMENT ON DISTILLATE PRODUCTION (EXP-AO-60 BIMODAL VS CRITERION 324 UNIMODAL CATALYST)

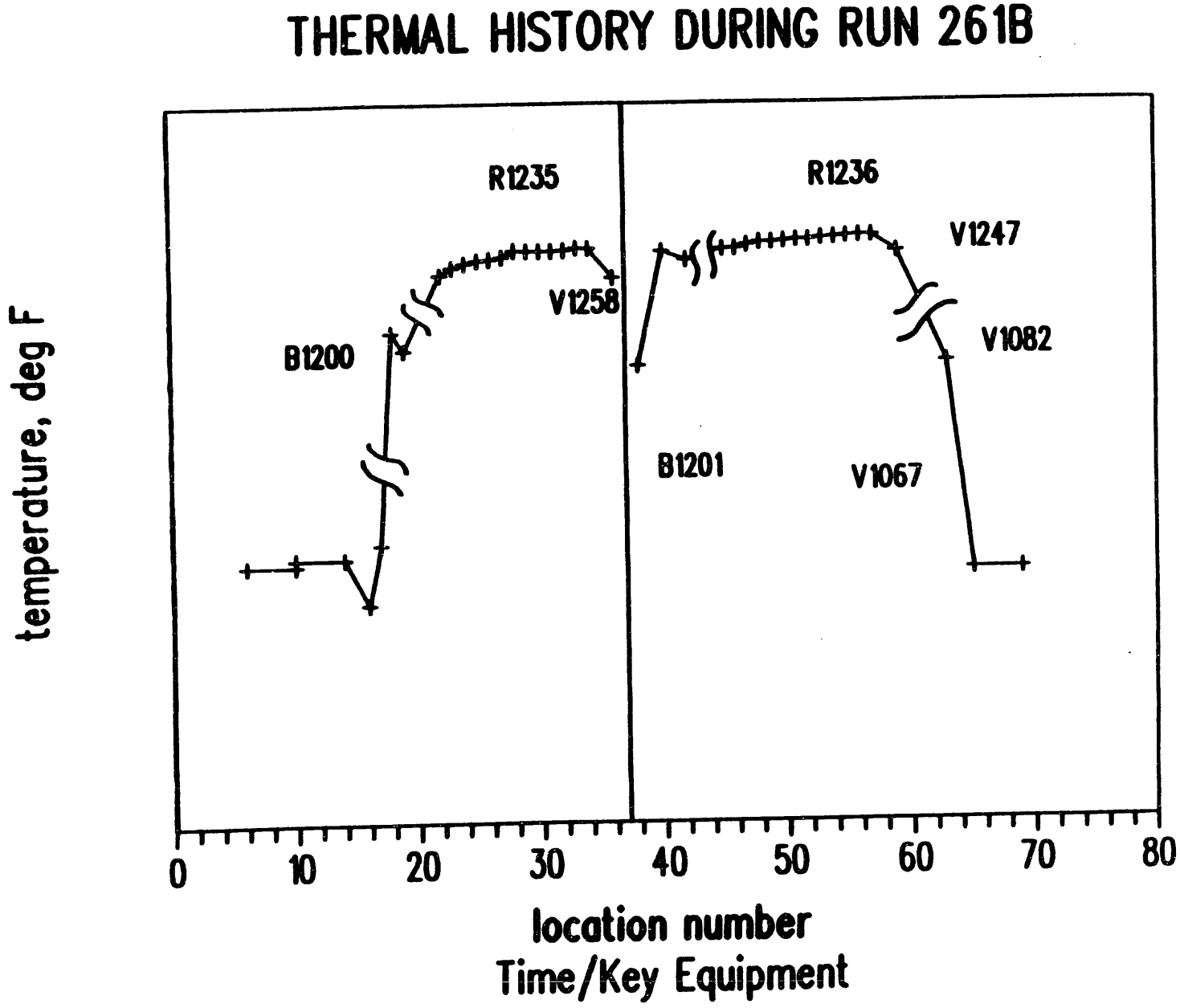


FIGURE 85. TSL THERMAL HISTORY DURING RUN 261B

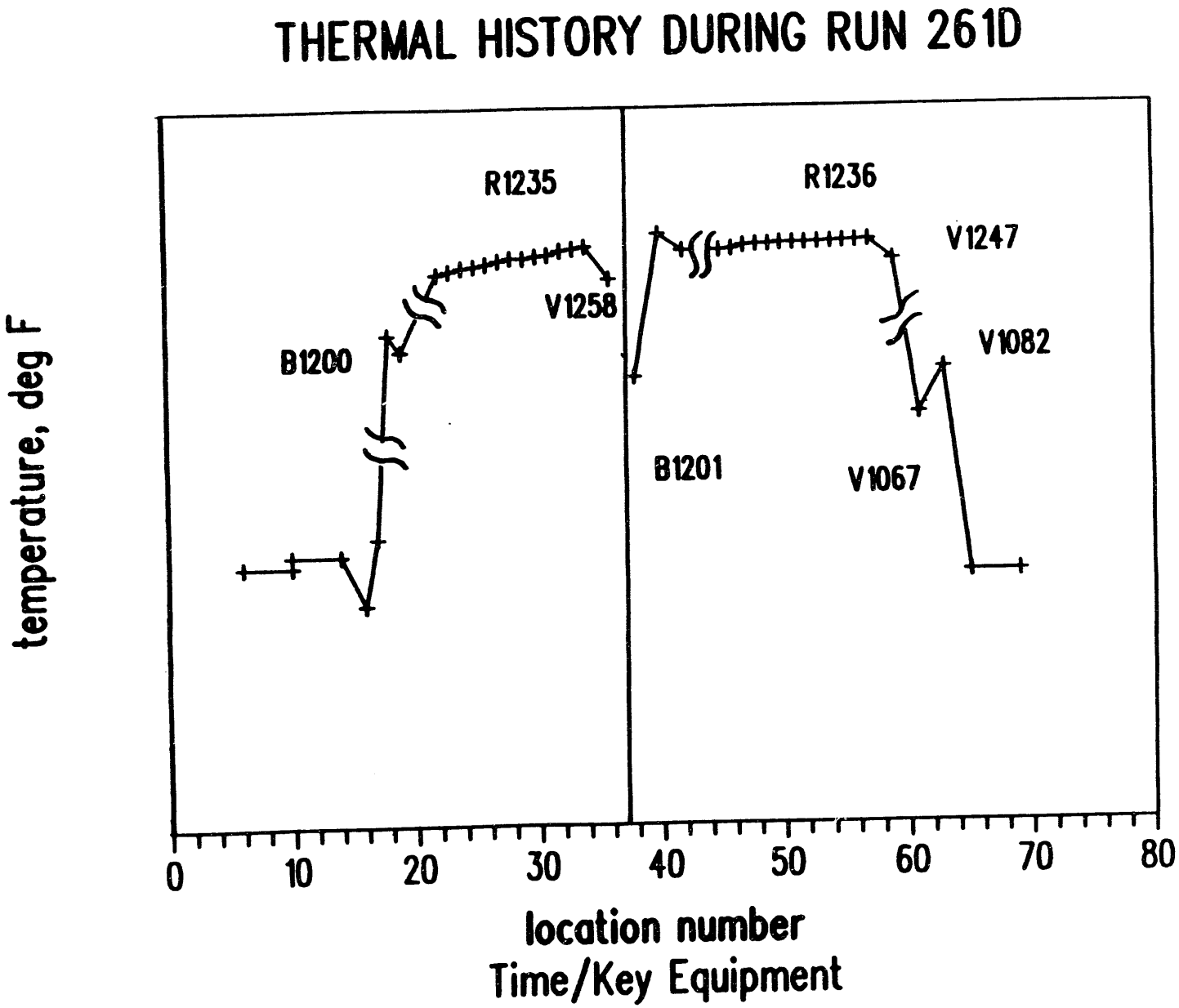


FIGURE 86. TSL THERMAL HISTORY DURING RUN 261D

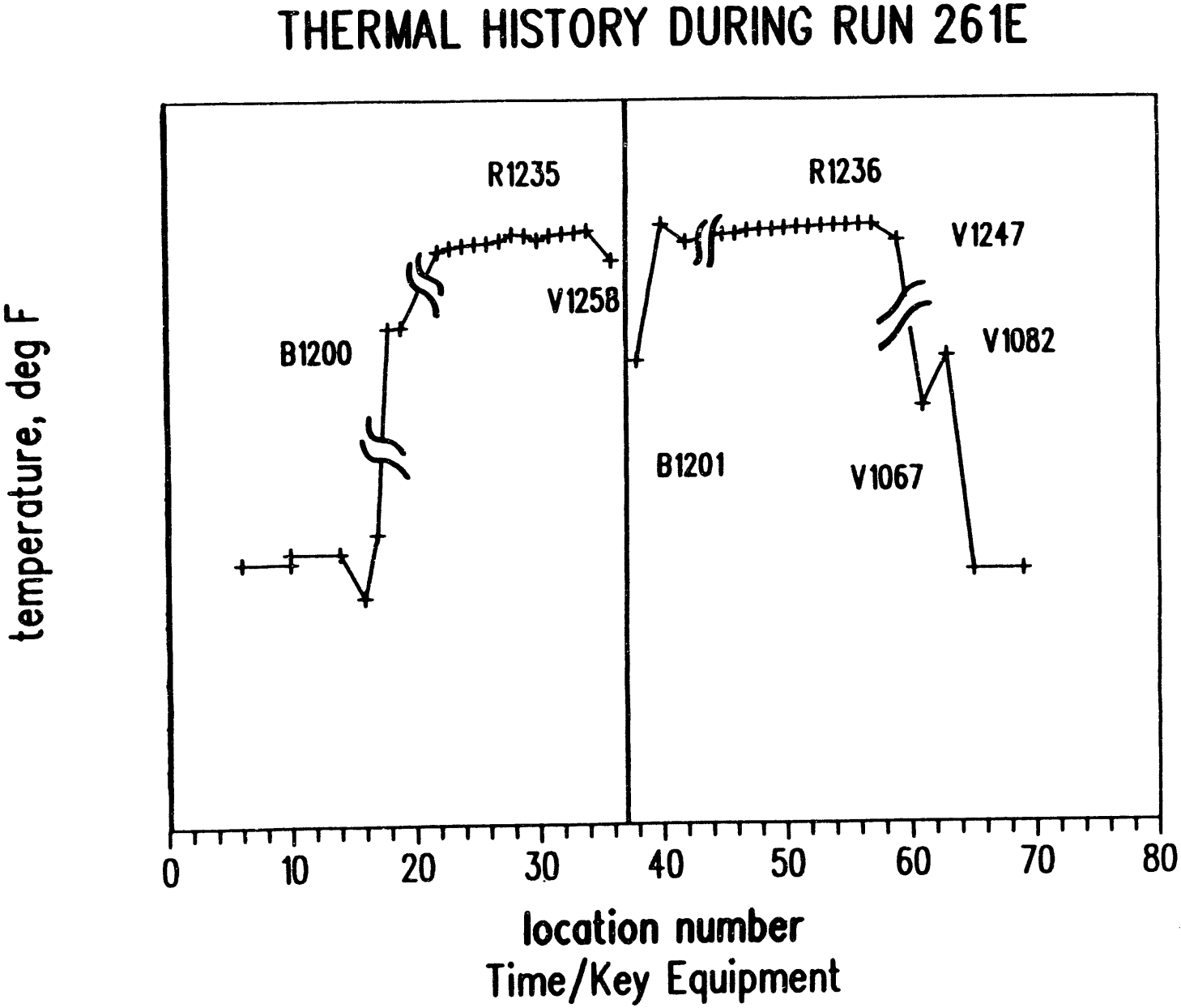


FIGURE 87. TSL THERMAL HISTORY DURING RUN 261E

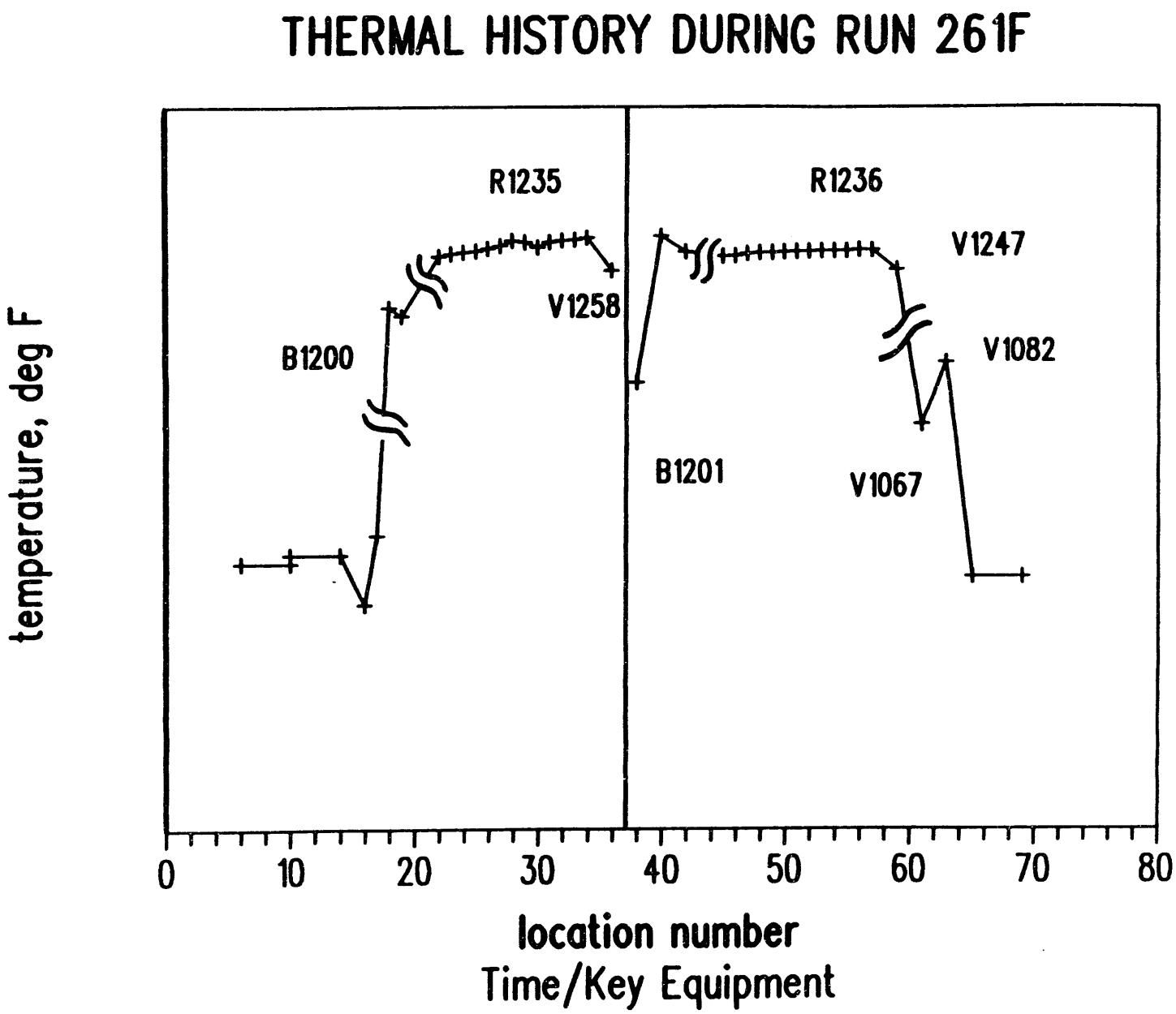


FIGURE 88. TSL THERMAL HISTORY DURING RUN 261F

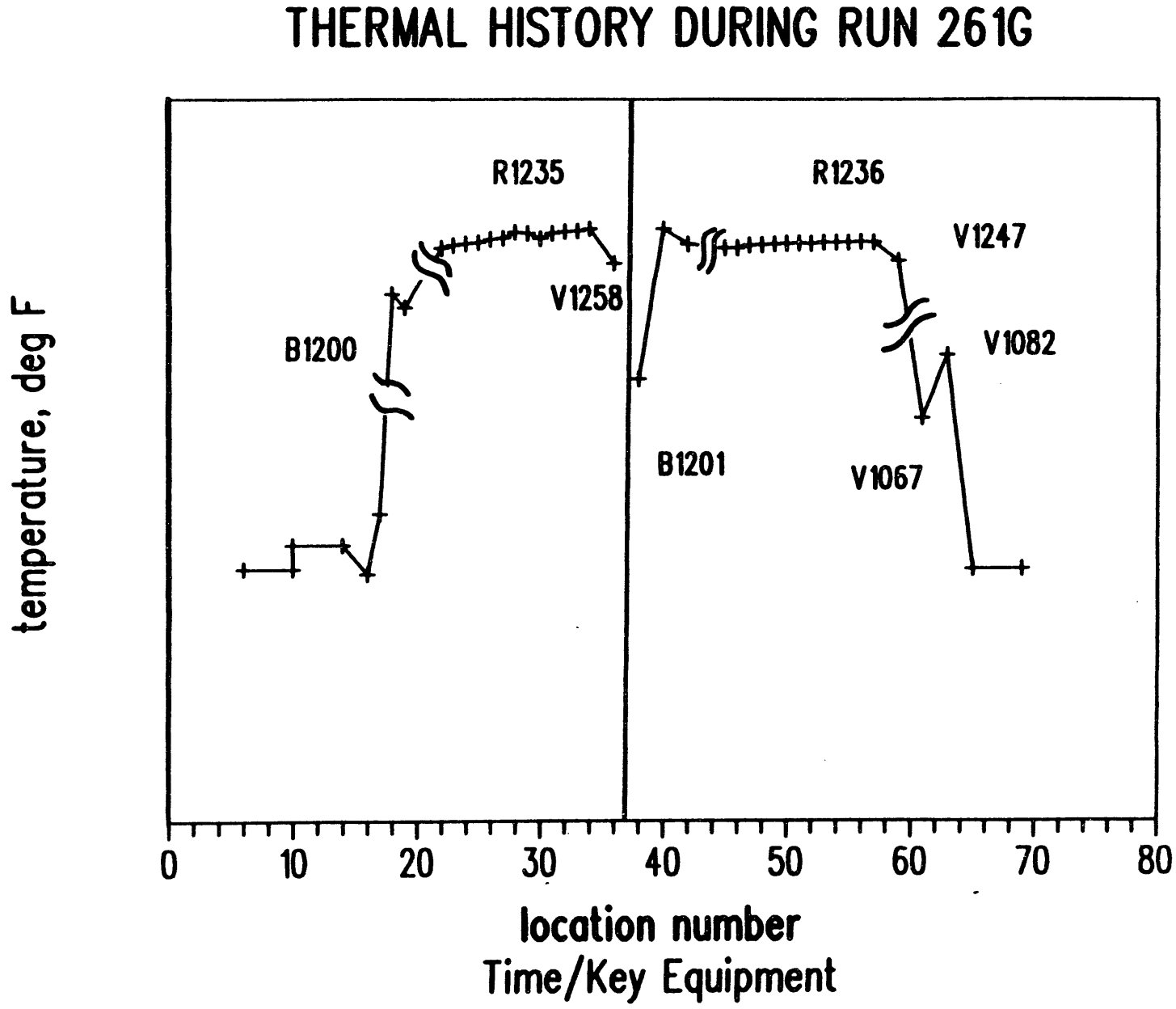


FIGURE 89. TSL THERMAL HISTORY DURING RUN 261G

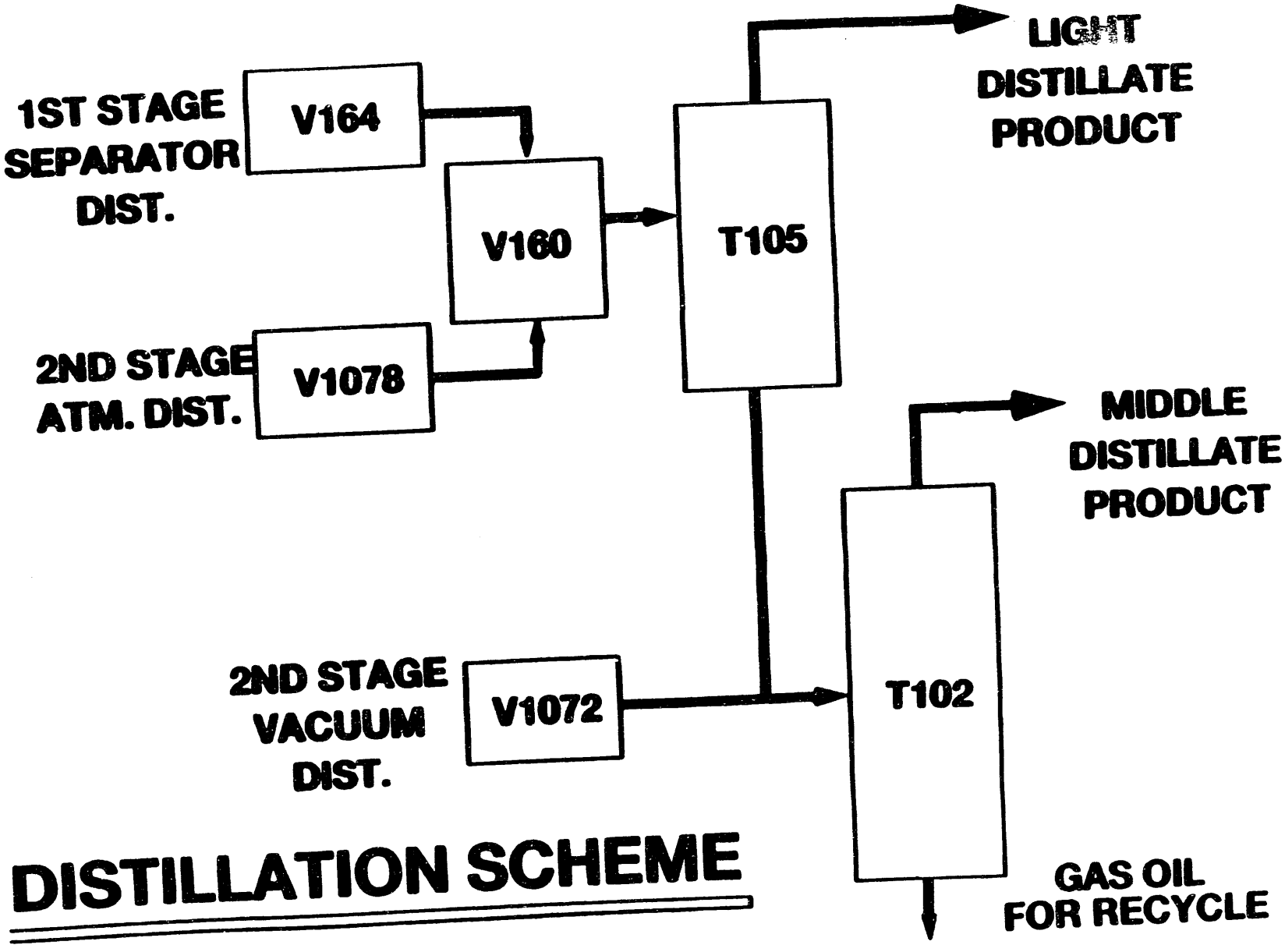


FIGURE 90. A SCHEMATIC SHOWING DISTILLATION COLUMNS FEED AND PRODUCT STREAMS

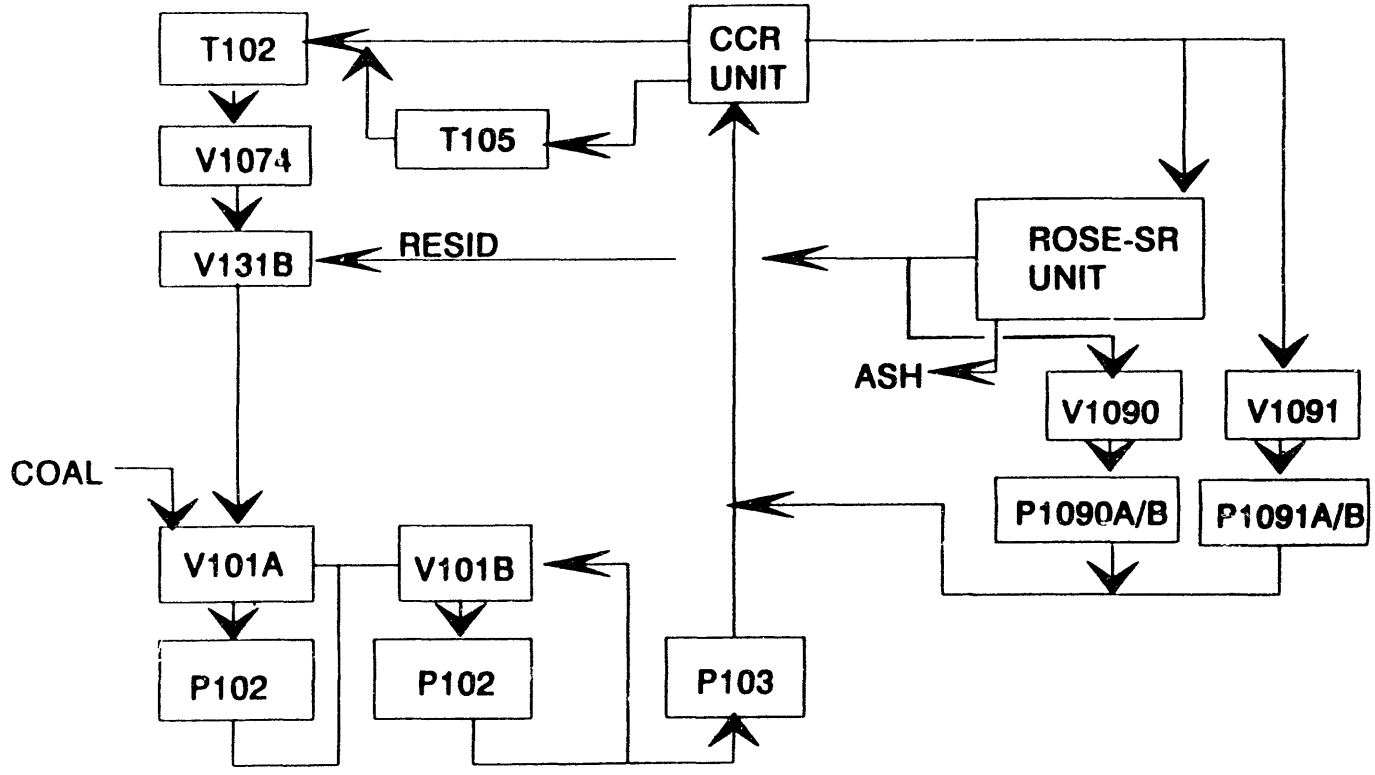


FIGURE 91. SIMPLIFIED FLOW CHART OF STREAMS DURING RESID INJECTION

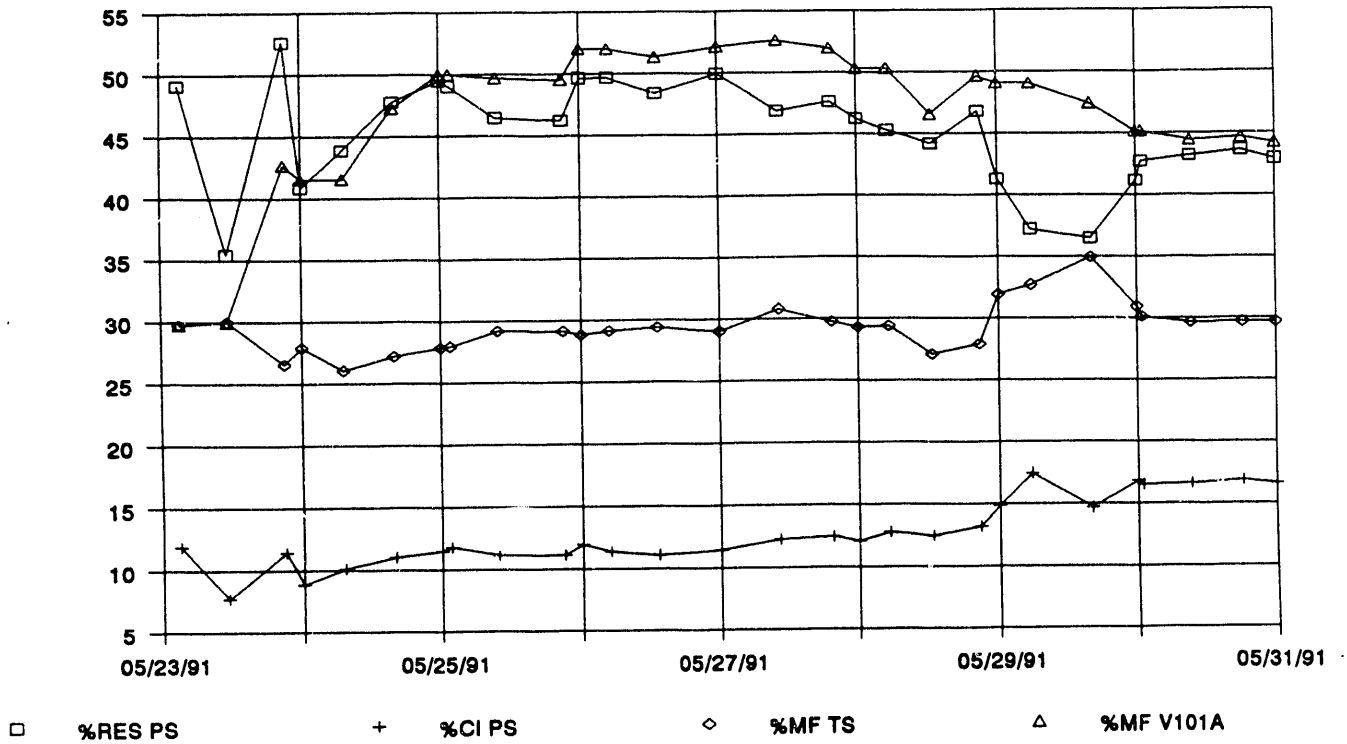


FIGURE 92. SLURRY COMPOSITION

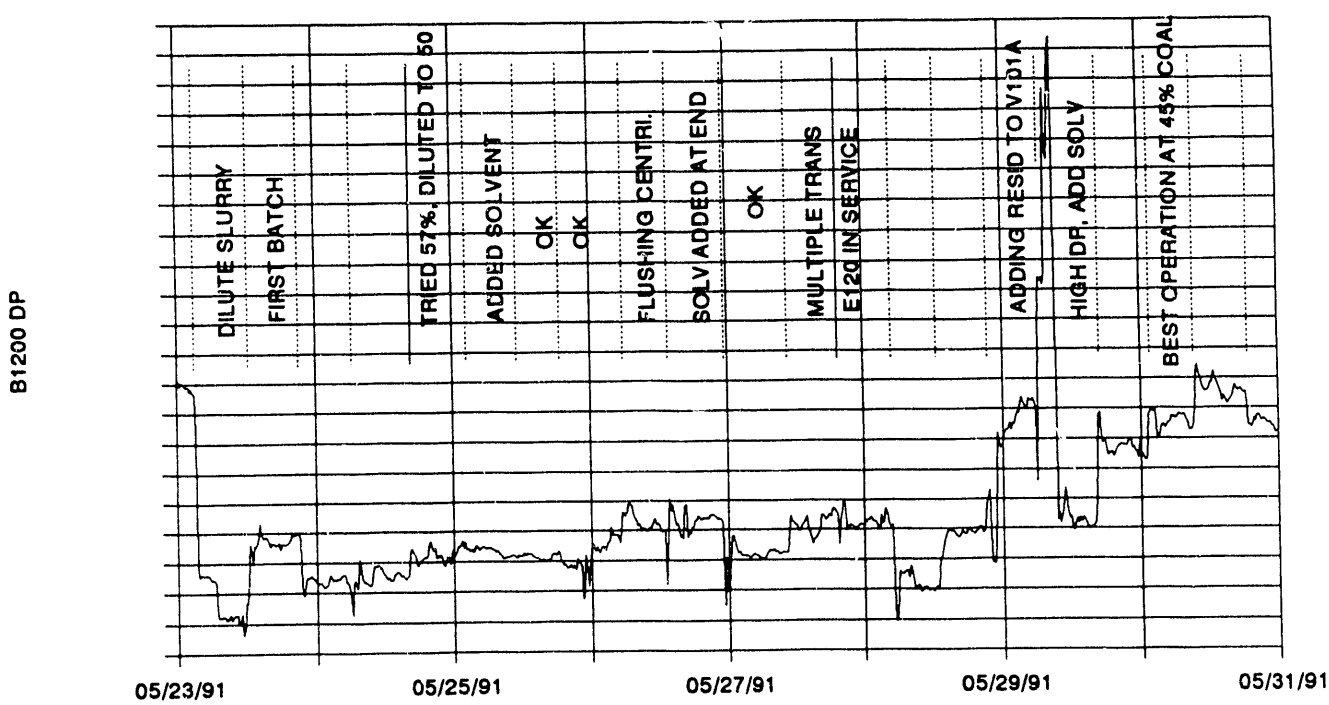


FIGURE 93. B1200 PREHEATER DIFFERENTIAL PRESSURE

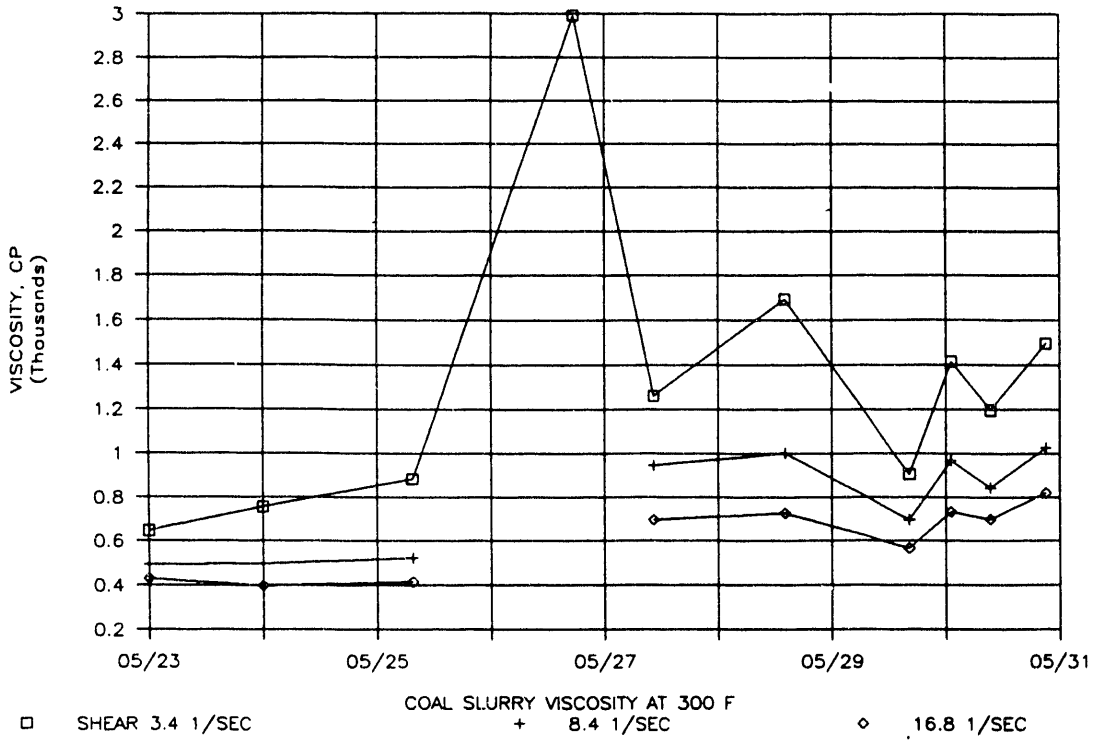


FIGURE 94. COAL SLURRY VISCOSITY AT 300°F

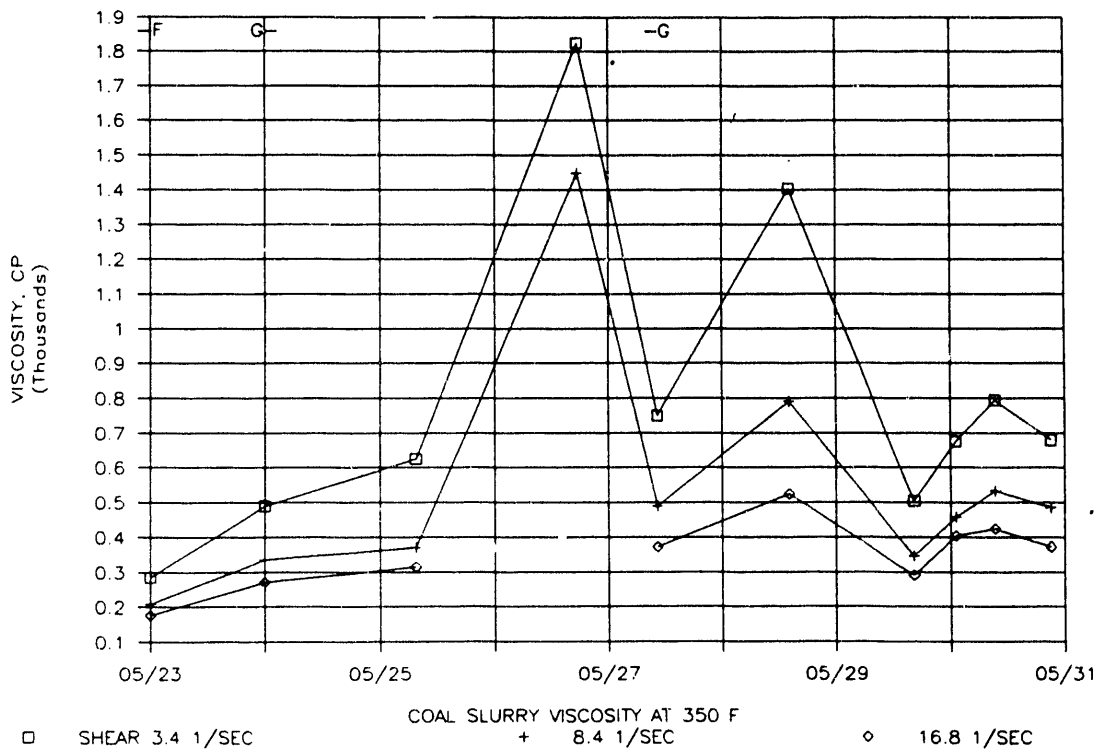


FIGURE 95. COAL SLURRY VISCOSITY AT 350°F

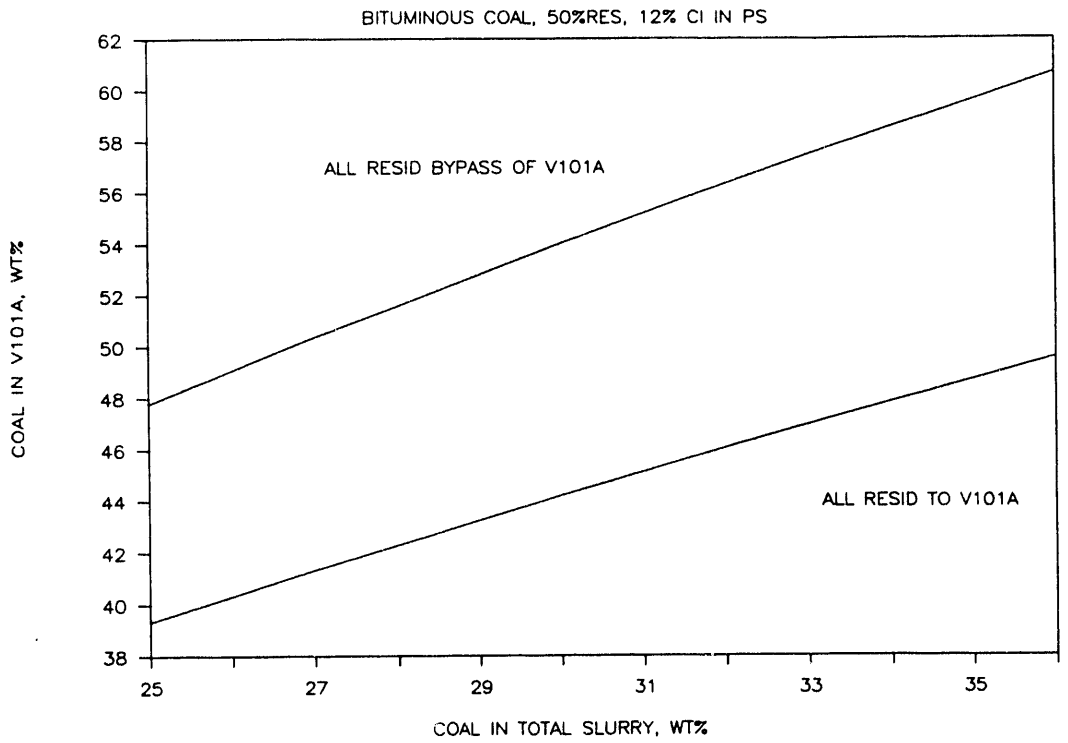


FIGURE 96. V101A SLURRY COMPOSITION VS TOTAL FOR BITUMINOUS COAL

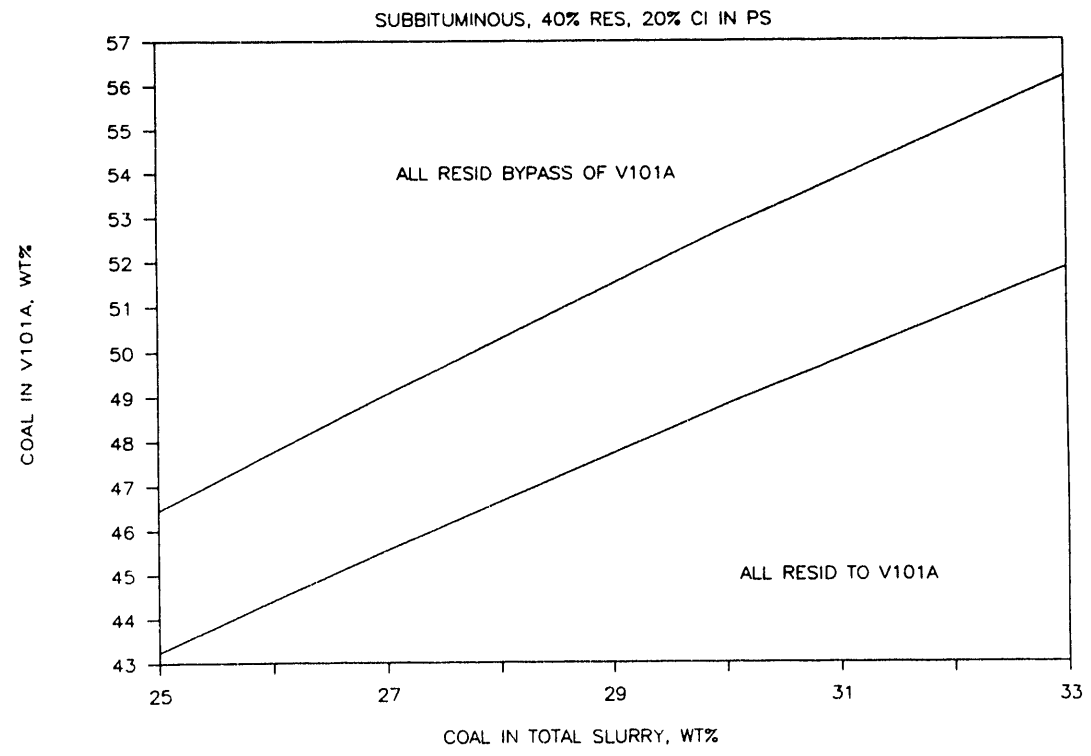


FIGURE 97. V101A SLURRY COMPOSITION VS TOTAL FOR SUBBITUMINOUS COAL

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