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RESEARCH AND DEVELOPMENT FACILITY

Wilsonville, Alabama

TECHNICAL PROGRESS REPORT

Run 263 with Black Thunder Mine Subbituminous Coal And Dispersed Molybdenum Catalysts

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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^{SM}) 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 by placing the ROSE-SR^{SM} unit after the second stage and with no significant pressure letdown between stages. A second reactor was added in close proximity to a pre-existing reactor. These close-coupled reactors can be used for various modes of operation --- thermal/catalytic, catalytic/catalytic, catalytic/thermal and thermal/thermal. The ROSE-SR^{SM} unit uses a proprietary solid-liquid separation process developed by the Kerr-McGee Corporation. The process separates ash and unconverted coal (UC) from resid 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. Black Thunder mine subbituminous coal from Powder River Basin was used in Run 263. The main emphasis was to evaluate the performance of the TSL system using dispersed molybdenum catalysts along with and without the supported Criterion 324 1/16" catalyst in the second stage.

TABLE OF CONTENTS

Section **Page**

TABLE OF CONTENTS (continued)

Section **Page**

LIST OF TABLES

No. **Tables** **Page**

LIST OF TABLES (Continued)

LIST OF FIGURES

LIST OF FIGURES (Continued)

LIST OF FIGURES (Continued)

LIST OF FIGURES (Continued)

LIST OF FIGURES (Continued)

ABSTRACT

This report presents the results of Run 263 performed at the Advanced Coal Liquefaction R & D Facility in Wilsonville, Alabama. The run started on October 31, 1991 and continued until February 23, 1992. Tests were conducted by operating the reactors in the Close-Coupled Integrated Two-Stage Liquefaction mode and by processing Black Thunder Mine subbituminous coal from Wyodak-Anderson seam in Wyoming Powder River Basin. Half volume reactors were used for the entire run.

In the first part of Run 263, a dispersed molybdenum catalyst was evaluated for its performance without a supported catalyst in the second stage. Molyvan L and Molyvan 822 (commercially available as friction reducing lubricants) were used as precursors for the dispersed molybdenum catalyst. The effect of the dispersed catalyst on eliminating the solids buildup was also evaluated.

For the second part of the run, the hybrid catalyst system was tested with supported Criterion 324 1/16" catalyst in the second stage at catalyst replacement rates of 2 and 3 lb/ton of MF coal. The molybdenum concentration was 100-200 ppm based on MF coal. Iron oxide was used as a slurry catalyst precursor at a rate of 1-2 wt % MF coal throughout the run with dimethyl disulfide (DMDS) as the sulfiding agent.

The close-coupled reactor unit was on-stream for 2482 hours for an on-stream factor of 91.2% and the ROSE-SR^{SM} unit was on-feed for 2126 hours for an on-stream factor of 96.4% for the entire run.

1. INTRODUCTION

The Close-Coupled Integrated Two-Stage Liquefaction (CC-ITSL) mode was used in Run 263 in the thermal/thermal (with no supported catalyst) or thermal/catalytic, high/low temperature mode of operation. The term "thermal reactor" implies no use of a supported transition metal catalyst, although fine iron oxide (+ DMDS) and a dispersed molybdenum catalyst were externally added in the first stage during this run. The term "slurry/slurry reactor mode of operation" seems more appropriate than the term "thermal/thermal". However, for continuity with the term "thermal reactor" is used consistent with previous run reports concerned with low-rank subbituminous coals and lignite (Runs 251-II, 251-III, 255, 258, 260 and 262). Prior runs in the CC-ITSL configuration include Runs 250-262 (Ref. 1-13).

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 263, 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 of the vacuum flash (except in period 263F in which atmospheric flash bottoms was used) which contains resid, unconverted coal and ash, is recycled to meet the targeted CI content in the process solvent. The remaining portion of the bottoms from the vacuum flash is fed to the Residuum Oil Supercritical Extraction-Solids Rejection (ROSE-SR^{SM}) unit. The solids-free resid recovered in the ROSE-SR^{SM} unit is recycled with the second stage distillate (heavy vacuum gas oil) and the vacuum flashed bottoms for coal slurry preparation. A simplified flow diagram of the CC-ITSL process is shown in Figure 1.

Dispersed molybdenum catalyst was introduced into the CC-ITSL system in concentrations ranging from 100 to 200 ppm molybdenum based on MF coal. Molyvan L and Molyvan 822, commercial friction reducing lubricants, were used as precursors for the dispersed molybdenum catalyst. For the first part of the run, the thermal/thermal (no supported catalyst) mode of operation was employed to test the dispersed molybdenum catalyst for its performance without a supported catalyst; and for the second part of the run, the thermal/catalytic (supported catalyst in the second stage) mode was employed to evaluate a hybrid slurry and supported catalyst system with Molyvan L (or Molyvan 822) and Criterion 324 1/16" supported catalyst.

The performance of the dispersed molybdenum catalyst was evaluated by processing the low-rank coal Black Thunder mine coal. Process operating conditions were optimized to increase the distillate production rate with good product quality in order to improve the process economics. High distillate yield and coal throughput increases the distillate production. The distillate production can be enhanced by improving the coal reactivity, catalyst conversion activity, and distillate selectivity.

Solids buildup in reactor transfer lines and interstage separator was investigated for possible reduction or elimination by using a dispersed molybdenum catalyst. Problems associated with the formation of deposits were discussed in the previous run report with a subbituminous coal (Ref. 13) and two different solids deposition mechanisms suggested by Davis, et al., were coking and mineral deposition (Ref. 14 and 15). The dispersed molybdenum catalyst may give acceptable conversions at moderate reaction temperatures and reduce the coking reactions, that cause solids to deposit in the transfer lines, by hydrogenating the thermally produced free radicals and possibly by preventing retrogressive reactions (Ref. 16).

Amoco Oil Company (Ref. 17) tested the dispersed molybdenum catalyst using Molyvan L and molybdenum octoate as precursors in bench-scale two-stage liquefaction with Illinois No. 6 coal and Amocat 1C bimodal supported catalyst. The UOP (Ref. 18) and Dow Chemical Company (Ref. 19) studied the dispersed catalysts with bench-scale single stage liquefaction tests. Several other investigators have also conducted liquefaction tests with the dispersed molybdenum and iron catalysts to improve conversion and their results can be found in the literature (Ref. 20-26).

In the following, a brief summary of Integrated Two-Stage Liquefaction (ITSL) runs conducted at Wilsonville along with the coals used and processing modes employed 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	Illinois/Wyoming	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	Black Thunder/ Spring 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)
262	Black Thunder	CC-ITSL	Thermal/Catalytic(10,13)
263	Black Thunder	CC-ITSL	Thermal/Thermal and Thermal/Catalytic(10,13)

(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 were used.

(13) Used dispersed molybdenum and supported Criterion catalysts.

Simplified block diagrams of all the operating modes tested at Wilsonville since Run 242 are shown in Figure 2. 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 263 in continuation of Run 262 were:

- To evaluate the performance of a dispersed molybdenum catalyst by processing a low-rank coal,
- To investigate and reduce/eliminate solids buildup that generally occurs while processing a low rank coal, and
- To evaluate the performance of a hybrid catalyst system with dispersed molybdenum and Criterion 324 supported catalysts.

During Run 263, the following tests were done to attain the objectives set forth.

- Evaluate the performance of the Molyvan L slurry catalyst at a 200 ppm molybdenum addition rate in the absence of a supported catalyst.
- Evaluate the performance of the Molyvan L slurry catalyst at a 100 ppm molybdenum addition rate.
- Increase the first stage reaction temperature to 840°F in order to improve the resid conversion.
- Decrease the coal feed rate to 265 MF lb/hr in order to improve the process performance.
- Test Molyvan 822 in place of Molyvan L as a dispersed molybdenum catalyst.
- Test atmospheric bottoms recycle in place of vacuum bottoms recycle in order to evaluate the effect on the boiling end point of the distillate product.
- Decrease the concentration of iron-oxide added in the feed slurry to 1 wt % MF coal.
- Test Criterion 324 supported catalyst in the second stage with a catalyst replacement of 3 lb/ton MF coal.
- Decrease the first stage inlet hydrogen partial pressure by 400 psia.

- Test Criterion 324 supported catalyst in the second stage with a catalyst replacement of 2 lb/ton MF coal.

In the first part of Run 263, slurry reactors with only the dispersed molybdenum and iron catalysts were used and liquefaction tests were conducted by varying the molybdenum precursor type, molybdenum and iron concentrations, first stage temperature, recycle resid concentration, solids recycle stream, and coal space rate. One of the primary objectives of the run was to reduce/eliminate the solid deposits typically experienced while liquefying low-rank coals at Wilsonville. The dispersed molybdenum catalyst may give acceptable conversions at moderate reaction temperatures, and the moderate temperatures reduce the coking reactions that may cause solids to deposit in the transfer lines.

In the second part of Run 263, a hybrid slurry and supported catalyst system was tested in verification of the previous run (Run 262) at 3 lb/ton MF coal catalyst replacement rate. The performance of the hybrid catalyst system was evaluated by varying the catalyst replacement rate, recycle resid concentration, and hydrogen partial pressure in the first stage.

2. CONCLUSIONS AND RECOMMENDATIONS

2.1 Conclusions

The conclusions for Run 263 are as follows:

- Processing Black Thunder subbituminous coal, the hybrid system with a combination of dispersed molybdenum and supported Criterion catalysts improved the distillate production by 30-60% compared to the use of either dispersed molybdenum or supported catalyst separately.

- Low molybdenum addition rate of 100 ppm MF coal using Molyvan L or 822 as the dispersed molybdenum catalyst precursor significantly improved coal and resid conversions, allowing operation at lower thermal severity in the first stage and higher severity in the second stage compared to the supported catalyst system. Also, this resulted in both higher C4+ distillate product yield and selectivity to resid+UC conversion.

- Operations in the hybrid catalyst system with a combination of Molyvan L or 822 and Criterion 324 catalysts produced better quality distillate than by just using the dispersed molybdenum catalyst. However, the boiling end point of the distillate product was in the same range for both the dispersed and hybrid catalyst systems (717-755°F).

- The supported catalyst only system had similar distillate product quality as the hybrid catalyst system. It seemed that the presence of Criterion 324 supported catalyst in the second stage improved the product quality in both hybrid and supported catalyst systems by increasing the hydrogenation and lowering the heteroatoms in the distillate product.

- The dispersed molybdenum catalyst seemed to catalyze hydrogenation of coal and/or resid in the first ("thermal") stage ("thermal reactor" refers to the absence of a supported catalyst) through stabilization by hydrogen transfer from the process solvent, resulting in higher first ("thermal") stage conversion in the hybrid catalyst system, while it seemed to have a lesser role in hydrogenation and hydrocracking of the process solvent compared to the supported catalyst.

- The addition of Molyvan L or 822 as a dispersed molybdenum catalyst precursor improved process operability by preventing process line plugging. However, solids did deposit in the interstage separator. A different interstage separator design could resolve the deposit problem.

- Black Thunder mine subbituminous coal gave a higher distillate production rate per unit volume of reactor than Illinois No. 6 bituminous coal, when process operating conditions were optimized for better coal and resid conversions, even though the subbituminous coal had lower distillate yield and selectivity.

Major Observations in Run 263 were:

- Good performance was observed at a low 100 ppm molybdenum concentration in both the slurry and hybrid catalyst systems. The lower molybdenum addition rate of 100 ppm did not reduce the conversion performance compared to the 200 ppm addition rate.

- Operation at the higher first stage temperature of 840°F, and at the lower coal space velocity with 265 MF lb/hr coal feed rate in the thermal/thermal mode (no supported catalyst) did not significantly affect the distillate selectivity to conversion. The distillate selectivity to conversion was 69-72% (71% at 825°F and 300 MF lb/hr) without excessive gas production. Coal conversion improved to 94-95 wt % MAF coal and organic rejection in the ROSE-SR^{sm} bottoms product stream decreased to 13 wt %.

- Periods 263E-H in the thermal/thermal mode using a dispersed molybdenum catalyst and without the presence of a supported catalyst in the second stage achieved the "all-distillate" product slate with 57-62 wt % MAF coal C_4+ distillate yield. The TSL resid yield was -1 to 4 wt % and organic rejection was 14-15 wt %.

- Molyvan 822 addition slightly lowered the resid+UC conversion by about 2-3 wt % MAF coal compared to Molyvan L addition, indicating that there is little difference in the conversion performance among the two molybdenum precursors.

- Lowering iron-oxide concentration from 2 to 1 wt % MF coal did not reduce the resid+UC and coal conversions.

- With Criterion 324 supported catalyst in the second stage, the hybrid catalyst system improved distillate production by facilitating operation at 26-33% higher coal space velocity. The distillate production rate increased by a similar margin (25-32%). This was observed with 3 lb/ton MF coal catalyst replacement rate. The "all-distillate" product slate was achieved in periods 263IJ with 58-61 wt % MAF coal C_4+ distillate yield. The TSL resid yield was 1 to 4 wt % and organic rejection was 14 wt %.

- At a lower Criterion 324 catalyst replacement rate of 2 lb/ton MF coal, the resid+UC conversion declined by 2-10 wt % MAF coal, decreasing the coal throughput by 2-11% for the "all-distillate" product slate with resid extinction. The distillate production rate decreased by 5-16%.

- Lowering inlet hydrogen partial pressure by 350-400 psia in the first stage decreased the resid+UC conversion by 1-5 wt % MAF coal, decreasing the coal throughput by 2-7% for the "all-distillate" product slate with resid extinction. The distillate production rate decreased by 1-8%.

- The presence of Criterion supported catalyst in the second stage improved the coal conversion more than that with Molyvan L or 822 slurry catalyst, and the coal conversion further improved by the combination of Criterion and Molyvan L or 822 catalysts.

- The use of just dispersed molybdenum catalyst (Molyvan L or 822) or Criterion supported catalyst alone gave similar resid+UC conversion, and the resid+UC conversion improved by the combination of Criterion and Molyvan L or 822 catalysts.

- The presence of a supported catalyst (Criterion, etc.) in the second stage might improve the first stage thermal conversion in the hybrid catalyst system, due to improved hydrogenation of the process solvent. The calculated average rate constant values for periods 263I, 263J, 263L and 263M operating with the hybrid catalyst system using the Criterion supported catalyst were 20-45% higher than those obtained for 263GH operating with the dispersed molybdenum catalyst only system (K/C = 91.4-112.0 vs. 74.9-80.5 hr^{-1} at 840°F).

- Results obtained at a lower first stage reaction temperature of 825°F can be compared to that in Run 262. The calculated average rate constant values for 263AB operating with the dispersed molybdenum catalyst only system were lower than those obtained for 262A-F operating with the hybrid catalyst system with Criterion catalyst (K/C = 69.3-71.2 vs. 75.9-91.9 hr^{-1} at 825°F).

- The presence of a supported catalyst (Criterion, etc.) in the second stage improved the second stage resid+UC conversion. The calculated average rate constant values for 263I-J and L-M periods operating with the hybrid catalyst system with Criterion supported catalyst were 45-115% higher than those obtained for 263B-H operating with the dispersed molybdenum catalyst only system (K = 65.9-96.4 vs. K/C = 44.1-48.6 hr^{-1} at 810°F).

- The combination of Molyvan L slurry and Criterion 324 supported catalysts showed higher first stage thermal resid+UC conversion than that obtained by using them separately.

- Apparent activation energies determined for the first stage thermal conversion with Molyvan L slurry catalyst addition were 30000-38000 Btu/lb-mole for the hybrid and dispersed catalyst systems. These values were lower than that for the supported catalyst only system (53000-64000 Btu/lb-mole), although conversion activity levels for the hybrid and dispersed catalyst systems were higher than the supported catalyst only system.

- When the first and second stage ("thermal") conversion activities for periods 263A-H were compared in Arrhenius plot to evaluate the staging effect in the dispersed molybdenum catalyst system, the staging effect in resid+UC conversion was somewhat apparent, having lower conversion in the second stage when compared by extrapolation at the same reaction temperature. Although the second stage activity data were highly scattered, the second stage average value for 810°F seemed to be present below the linear regression line extrapolated and extended for a lower reaction temperature of 810°F. The regression line was generated using the first stage activity data at 825-840°F. The hybrid catalyst system data were not included in regression.

- Lower coal feed rate does not seem to significantly affect the calculated rate constant values for both stages by assuming CSTR first-order kinetics.

- Increased first stage reaction temperature seemed not to significantly affect the calculated rate constant value of the second stage by assuming CSTR first-order kinetics.

- A good linear correlation was observed between organic rejection and coal conversion with low-rank coals and lignite.

- The atmospheric flash bottoms (V1067) recycle in 263F increased the 750°F- material in the process solvent from 2 wt% (with vacuum flash bottoms recycle in 263E) to 13 wt%. Heavy vacuum gas oil (V1074) was collected as product. The product distribution changed from 41/59 wt% V161/V182 in 263E period to 48/10/42 wt% V161/V182/V1074.

- Process performances with atmospheric flash bottoms recycle and vacuum flash bottoms recycle were similar except for product end point. In order for a net shift in boiling point distribution to be observed, a net shift in reaction rates must occur. In our tests, this was accomplished by changing the concentrations of the relevant reactants (not necessarily the rate constants themselves). That is, with vacuum bottoms recycle the heavy end crack to a greater extent. Also, lighter ends are more resistant to cracking and report to the vapor phase to a greater extent. Or, it is also possible that the effect of lighter solvent recycle appears to be a transitional trend of a physical displacement -- the lighter distillate product being replaced by heavier product by an amount equivalent to the lighter solvent being recycled.

- Reduced backmixing flow rate significantly affects the first stage reactor temperature profile, resulting in an increased reactor temperature spread from about 25-30°F to 55-65°F, and then to 110-115°F, and after February 13, to 250-315°F. The higher temperature spread are the result of unusually low temperature readings from the thermocouple in the bottom of the reactor due to solid deposition. It is also possible that the thermocouple reading was in error as indicated in period 263M with the large drop in process temperature between B1200 preheater and R1235 first reactor.

- The presence of a supported catalyst along with a dispersed molybdenum catalyst gave higher hydrogen values in the process solvent and interstage stream compared to the slurry catalyst only system, indicating additional hydrogenation by the supported catalyst.

- Decreasing the iron-oxide addition rate from 2 to 1 wt % or decreasing the inlet hydrogen partial pressure in the first stage by about 400 psia appeared not to significantly affect the hydrogen values of both the process solvent and interstage stream.

- Maintaining the first stage reactor at 840°F gave higher coal conversion and resulted in lower energy rejection.

- The GC cut point of the distillate product (T102 overheads) from the GC simulated distillation and by the equal weight percent overlap method did not vary much during the run (760-804°F, with an overlap of 6-12 wt %).

- The laboratory workup for the distillate product quality using the ASTM D1160 method gave boiling end point of the distillate product in the range 710-760°F except for period 263F with the atmospheric flash bottoms recycle. These end points are for the combined distillate product (T105 and T102 column overheads) for each period. The end boiling point increased significantly from 755°F in 263E and 708°F in 263G to 963°F in 263F. This was accomplished by changing the concentrations of the relevant reactants (not necessarily the rate constants themselves). Or it is also possible that the effect of lighter solvent recycle appears to be a transitional trend of a physical replacement.

- There appeared to be no significant difference in the nitrogen value of the total oil due to either Molyvan L or Molyvan 822 precursor (8600-8800 ppm). With the hybrid supported and slurry catalyst system the nitrogen value of the total oil significantly decreased to 6000-6600 ppm range. The oxygen content determined by difference was high with the dispersed molybdenum catalyst, e.g., up to 6.9 wt % in the 350-450°F range.

- Paraffin buildup in the process streams and in process solvent was less noticeable in this run (263) than the previous run (262). The paraffins (waxy material) appeared in the first stage distillate (V164) and second stage distillate (V1078) when the weather became cooler causing transfer problems in lines that carried these distillates. The paraffins in V1074 averaged from 8 to 12 wt % with both dispersed molybdenum catalyst system (263A-H) and hybrid catalyst system (263I-M). In Run 262, the paraffin buildup was more in the process streams. The vacuum column bottoms (V1074) had 8-28 wt % wax in highly concentrated amounts; V164 had 3-9 wt %; and V1078 had 2-9 wt %. The presence of paraffins in subbituminous coal distillates is not surprising since aliphatic groups have been postulated as bridges between aromatic clusters in this rank of coal. Thermal breakage of aliphatic bonds and consequent catalytic hydrogenation might produce paraffins. Or, paraffins with high carbon chains from C_{16} to C_{32} might be entrapped in the coal matrix and upon thermal treatment released as products.

2.2 Recommendations

- Test at molybdenum concentrations lower than 100 ppm MF coal to improve the process economics by reducing the slurry catalyst cost. Perform bench-scale tests to selectively recover molybdenum from ash concentrate and determine its activity.

- Determine the effects of solids recycle levels and iron-oxide (+DMDS) slurry catalyst concentrations on process performance in the presence of Molyvan L slurry catalyst.

- Test lignite with the slurry catalyst system to evaluate distillate production, yield and selectivity at coal conversions higher than 90 wt % MAF coal.

- Test the low/high thermal severity configuration in the catalytic/catalytic mode to improve the thermal efficiency, catalyst utilization, distillate selectivity, and hydrogen efficiency.

- Evaluate the performance of a bimodal catalyst such as EXP-

AO-60 in processing subbituminous coals and lignite.

- Evaluate the performance of a dispersed molybdenum catalyst in processing bituminous coals.
- Evaluate the effect of other molybdenum precursors such as molybdenum octoate for higher levels of hydrogenation and lower cost. Recent studies indicate that the organic part of the organometallic precursors plays an important role in *in situ* decomposition and in dispersion and activation of active species. As a result, determine the catalytic activity by varying the organic part of the Molyvan L precursor.
- Evaluate process economics with variations in catalyst systems such as the hybrid catalyst system, dispersed catalyst only system, and the supported catalyst only system.
- Dispersed molybdenum catalyst allows higher throughput, however, the nitrogen content is much higher in the distillate product. Thus, there is a need to investigate and add a fixed bed hydrotreater/hydrocracker to improve the product quality.
- Redesign the interstage separator vessel to eliminate solids buildup and achieve adequate separation of gas and liquid.
- Test SO_2 pretreated subbituminous coal to completely eliminate solid deposits by removing calcium so as not to form calcium carbonate, and consequently study the effect of pretreatment on improvements to liquefaction process performance.

3. OPERATING DATA AND PROCESS PERFORMANCE

The main objective of this run is to test a dispersed molybdenum catalyst and study its effects on solids buildup and yield performance. Molyvan L and 822, which are organic liquid additives containing molybdenum and sulfur, were used as precursors to obtain dispersed molybdenum catalyst *in situ*. In the first part of Run 263, in continuation of Run 262, slurry reactors with only dispersed molybdenum and iron catalysts were used for testing in the thermal/thermal (no supported catalyst), high/low temperature mode without the Criterion 324 catalyst in the second stage. These tests were made by varying the molybdenum precursor type, molybdenum and iron concentrations, first stage temperature, recycle resid concentration, solids recycle stream, and coal space rate.

In the second part of Run 263, a hybrid slurry and supported catalyst system was tested at 3 lb/ton MF coal catalyst replacement rate. The performance of the hybrid catalyst system was evaluated by varying the catalyst replacement rate, recycle resid concentration, and hydrogen partial pressure in the first stage.

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} unit 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).

At the end of Run 262, waxy material from the overhead flashed streams caused several plugging problems. This problem occurred in the V1258 interstage separator overhead water/oil separator and in the flashed overheads from the second stage. At the beginning of Run 263 on October 31, 1991, the lines were steam traced, however, handling the waxy solvent along with the cooler weather continued to cause loss of flow in several places including the T105 atmospheric fractionation column bottoms, T102 vacuum tower overheads, V1074 heavy vacuum gas oil vessel to withdrawal tube flush holding vessels, and T102 vacuum tower bottoms to V1074 tank.

There were six outages during Run 263. The First on November 1 was due to the seat in a block valve on V1082 vacuum flash vessel becoming loose and blocking the recirculation flow. The same problem occurred at the start of Run 262 but was corrected by welding the seat to the valve stem. The second outage on November 22 was due to the failure of the first stage ebullating pump after period 263B. The third outage on December 1, prior to period 263C, was due to an accidental depressurization of the reactors when trying to repair the C1206 recycle gas compressor in the second stage.

The fourth outage was when the ebullating pump locked up on the second stage reactor on December 19, after period 263D. Inspection of the interstage separator during this outage showed an accumulation of solids (about 100 pounds) which were removed. On startup, the bottom thermocouple in the first stage reactor began to read lower than normal which indicated an accumulation of solids. Inspection at the end of the run showed that about 45 pounds of solids was present in the bottom of the reactor. Also, on startup after the fourth outage, the first stage ebullating pump would shut down almost everyday indicating a pluggage in the pipes to the pump. In early January, nuclear density gauge showed that the deposits returned to V1258 interstage separator.

The fifth outage on January 24, 1992, during tests for period 263I, was due to an attempt to remove the solids from the V1258 separator by placing the units on solvent circulation for 12.5 hours, The solids, however, remained in the V1258 separator. This was tried since it appeared that solids were removed by solvent circulation on November 23 during the second outage.

The sixth outage occurred on February 12 during tests for period 263L. During a C102 first stage recycle gas compressor outage, a plug developed in the gas line to B1200 slurry preheater. Recycle gas was sent to B1200 preheater outlet, however, the heater temperatures were too high. The plug in the gas injection nozzle and check valve was cleared by using high pressure oil.

At the end of the run, the reactors and V1258 separator were inspected. Considerable amount of hard, dry solids was present in the V1258 interstage separator. Only about 45 pounds of solids was present in the bottom of the reactor. On inspection, the plenum chamber in the R1236 second stage reactor showed wear in several places. Prior to the start of Run 263, the gas distribution device was changed to allow more clearance from the top of the plenum chamber. Also, flow orifices and restricted orifices (RO's) were checked, but nothing was found to explain the excess flow and wear. Significant problems/process condition changes that occurred during plant operations in Run 263 are summarized below:

DIFFICULTY OR CHANGE	**CONSEQUENCE**
C1205 compressor not operating well, Oct. 26-29.	Several repairs.
Start of Run 263, Oct. 31, with iron oxide from Perox Industries.	
Block valve on V1082 not letting flow through recirculation loop.	Off coal feed, Nov. 1.
On coal feed, Nov. 1.	
Plugging in oil/water separator from V1258 overhead, Nov. 2.	Steam traced oil/water separator. Waxy nature of products.
Level not working on V1250 overhead separator, Nov. 3.	More steam tracing and insulation work done for waxy products.
Tried DMDS to second stage reactor plus lowered withdrawal tube flush rate to improve temperatures, Nov. 4.	
First stage to 825°F, second stage to 790°F, Nov. 5.	
Change target to 45 wt % resid in process solvent from 42%, Nov. 6.	
Underground soft water line leaking, Nov. 6.	Blocked line in. Started using process water for caustic scrubber.
Lost circulation on V101B. Slurry too thick.	Slurry difficult to pump. Add T102 overhead to thin out.
Second stage temperature to 810°F, Nov. 7.	
Computer quit, Nov. 8.	Loss of 50 minutes of data.
V1072 flow rate device bypassed for a while, Nov. 9.	Plugging. Flow rate estimated.

DIFFICULTY OR CHANGE	**CONSEQUENCE**
Recirculation pump on slurry tank developed an internal seal leak, Nov. 11.	Lubricating oil, 10-20 gallon, entered slurry feed.
Level/density on V1258 starting to increase, Nov. 12.	Solids accumulation in V1258?
Molybdenum (Molyvan L precursor) reduced to 100 ppm of MF coal feed, Nov. 15.	
Computer quit, Nov. 15.	Loss of 93 minutes of data.
Restriction in line to caustic scrubber, Nov. 18.	Recycle gas not being scrubbed. New water line installed. Back in service on Nov. 19.
Recycle gas compressor down to repair lubricator. Feed measuring device to ROSE-SR unit bypassed, Nov. 19.	Gas system upset. ROSE-SR feed rate estimated. Measuring device repaired, Nov. 20
C102 first stage recycle compressor, down due to bad valve, Nov. 20.	Gas system upset.
V1258 level indication erratic, November 20, 21, 22.	Blowing level transmitter legs several times per day.
P1222, ebullating pump, on first stage quit, UPS (uninterrupted power system) failed also, Nov. 22.	Off coal feed.
V1258 density/level indication dropped after 2 hours on solvent, Nov. 23.	Washout of V1258 solids?
P1222 repaired, Nov. 24.	
Unable to get flow from T102 to V1074, Nov. 24.	Could not start up.
T102 line to V1074 taken apart. 10 foot section collapsed, with 10" split in jacketed line, Nov. 26.	Line rebuilt.

DIFFICULTY OR CHANGE	CONSEQUENCE
On coal feed, first stage to 840°F, Nov. 27.	
Packing leak on C1206. While repairing C1206, a valve was left open which depressured the reactor, Nov. 30.	Off coal feed, Dec. 1.
Clearing V1258 overhead line and V1247 overhead lines, Dec. 1.	
On coal feed, Dec. 2.	
C1206 packing leak, Dec. 4.	High gas purity.
C1206 oiler tubing blown apart, Dec. 5.	Gas system upset. Packing not being oiled.
Inlet thermocouple to second stage reading wrong. Cannot repair on line, Dec. 5.	Overall DT on second stage reactor cannot be calculated.
C1206 cooling water leak to flare header. Several plugs in T102 system, Dec. 6.	Gas system upset.
V1079 bypassed. Would not let enough flow, Dec. 8.	Flow rates estimated.
Reduced coal rate to 265 lb MF/hr. Changed to 42% resid in process solvent, Dec. 10.	
Density/level increasing on V1258, Dec. 15.	Solids buildup?
Started using T102 overhead for withdrawal tube flush, Dec. 17.	V1074 solvent too thick.
Process solvent resid changed to 45%, Dec. 18.	
Computer quit, Dec. 18.	Loss of 4 2/3 hours data.
P1236, ebullating pump, on second stage locked up, Dec. 19.	Off coal feed.
V1258 inspected. 3" buildup on southwest wall. Gummy material in bottom 3 feet of the vessel, Dec. 19.	

DIFFICULTY OR CHANGE	**CONSEQUENCE**
On coal feed, Dec. 22.	
Bottom point in first stage reading lower than normal, Dec. 22.	Solids in bottom of reactor?
P1222, ebullating pump on first stage shutting down, Dec. 24, 27, 29.	Lines plugged?
Bottom thermocouple in R1235 reading low, Dec. 29.	May be solids at very bottom of R1235.
C1205 down twice to replace third stage valve, Dec. 31.	Gas recycle flow upset for 1-2 hours.
P1222, first stage ebullating pump, shutting down daily, after Jan. 1.	Recycle flow stoppage for few minutes each day.
Change to Molyvan 822 from Molyvan L at 100 ppm Mo and change to V1067 recycle instead of V1082, T102 set to 2.5 psia, Jan. 2.	
C1205 down for second and third stage valve change, Jan. 3.	Recycle gas flow upset for 1-2 hours.
Due to low flow of V1082 overheads and cool weather, the overheads condenser plugged, Jan. 4, 5, 6. T102 set back to control V1074 inventory, Jan. 7.	High solvent in ROSE-SR unit feed.
Back to V1082 recycle, Jan. 8.	
Cold wind from west, Jan. 10, 14.	R1236 temperature dropped below target.
Lost circulation of slurry on feed tank, Jan. 12.	Slight interruption of slurry feed.
Change to 1 wt % MF coal iron oxide concentration, Jan. 14.	
Instrument air outage, due to water freezing in dryer beds, Jan. 17.	Low temperature bump for both reactors.
170 lbs (dry) catalyst from Run 262 was added to R1236. Catalyst addition/withdrawal was set at 3 lb/ton MF coal, Jan. 21	

DIFFICULTY OR CHANGE	**CONSEQUENCE**
Coal concentration changed to 30 wt% and coal feed rate increased to 350 lb MF coal/hr, Jan. 22.	
New Coal pile started, Jan. 23.	
Solids apparent in V1258. Tried going on solvent to remove V1258 solids, Jan. 24.	Off coal feed for 12.5 hours. V1258 solids not affected.
Motor on slurry feed tank failed, Jan. 26.	Loss of agitation on slurry and loss of coal feed.
Out of Molyvan 822, started using Molyvan L, Jan. 27.	
Not enough totebins to keep up with feed rate, Jan. 28.	Feed rate lowered to 300 lb MF coal/hr for half day.
Change to 40 wt% resid in process solvent, Jan. 29.	
Impulse line on V1258 plugged with salts, Jan. 30.	Pressure and DP's in error.
Increase in ROSE-SR feed solids content, Jan. 31.	ROSE-SR unit feed pumps not working well for rest of run.
Oil accumulation in second stage recycle gas flow meter, Feb. 1,2,3.	Recycle gas flow rate in error.
Computer down, part of Feb. 1, 2.	Loss of 11 hours data.
Lowered hydrogen partial pressure by 400 psia on first stage reactor, Feb. 4.	
Oil accumulation in second stage recycle gas flow meter, Feb. 5.	Recycle gas flow rate in error.
Catalyst addition/withdrawal stopped to batch age catalyst to 2 lb/ton MF coal, Feb. 8.	
Changed valves on C102 two times, Feb. 9.	Recycle gas flows upset for short time.

DIFFICULTY OR CHANGE	**CONSEQUENCE**
Vacuum jets replaced on T102, Feb. 10.	Able to pull tower pressure lower than 2 psia.
Changed back to high hydrogen partial pressure in first stage, Feb. 10.	
C102 outage, Feb. 12.	On solvent to clear plugged recycle gas line.
Back on coal feed, Feb. 13.	
P1222, ebullating pump, quit pumping, Feb. 13.	P1222 was shut down and blocked in.
Catalyst addition/withdrawal restarted at 2 lb/ton MF coal, Feb. 17.	
Mid temperature in V1258 dropping, Feb. 19.	Indication of solid deposits in V1258.
Off coal feed, Feb. 23. End of Run 263.	

In Figure 6, the forced ash balance coal conversions are shown for Run 263. After the low coal conversions on startup, the major event affecting coal conversion was the increase in first stage temperature to 840°F in period 263C which increased the coal conversion. Coal conversion decreased after the increase in coal feed rate for period 263I, which also involved a change in coal concentration from 25 to 30 wt %. The coal slurry viscosity for Runs 262 and 263 are shown in Figure 7. The higher first stage temperature of 840°F decreased the coal slurry viscosity.

3.2 Feed Coal

Black Thunder mine subbituminous coal from the Thunder Basin Coal Company in Wright, Wyoming, served as the feed coal for Run 263. This Powder River Basin coal was also used in three previous runs: Runs 258, 260, and 262. Some characteristics of this coal are summarized in Table 1. This table also contains coal data from Run 262 and by comparison the feed coal properties as expected stay about the same in the two runs. The coal ash was slightly higher at 7.0 wt % compared to an average ash of 6.4 wt % for Run 262 feed coal. The carbon, hydrogen, nitrogen, and sulfur values do not change much within Run 263 or even when comparing them to Run 262 values. Carbon is typically 69 wt %, hydrogen 5 wt %, nitrogen 1 wt %, sulfur 0.5 wt %, and oxygen by difference is about 18 wt %. Microautoclave tests performed on the coal gave tetrahydrofuran (THF) soluble coal conversion varying from 57 to 65 wt %. These values did not decrease or increase all that significantly throughout the run thus indicating that by this test the coal activity remained about the same from start to finish in Run 263.

3.2.1 Slurry Preparation

The process solvent used for coal slurry blend preparation was a mixture of V1074 distillate (heavy vacuum gas oil), full range ROSE-SR^{sm} resid and V1082 vacuum flash bottoms (except for period 263F in which V1067 atmospheric flash bottoms instead of V1082 vacuum flash bottoms was used). The mixture was blended in V101A slurry blend tank with coal and was fed to the first stage reactor. Recycle process solvent analytical data is in Table 2. In Run 263, the target process solvent composition was: resid = 40-45 wt %, heavy vacuum gas oil = 35-40 wt %, and CI = 20 wt %. Coal concentration in the slurry was 25-30 wt %.

3.3 Close-Coupled Reactor (CCR) Unit

The close-coupled reactor (CCR) unit had the following objectives for Run 263:

- Evaluate the performance of the CCR unit using different dispersed molybdenum catalyst precursors,
- Evaluate process performance using the dispersed molybdenum catalyst under conditions of varied reactor temperature, coal feed rate, resid concentration in process solvent, and iron-oxide addition rate,
- Evaluate process performance with a hybrid catalyst system at two different Criterion 324 supported catalyst ages,
- Test the effect of lower hydrogen partial pressure in the first stage reactor, and
- Study the effect of dispersed molybdenum catalyst on solid deposition in the process lines and vessels when processing a low rank coal.

Feed slurry composition, coal feed rate, dispersed molybdenum catalyst precursor type and concentration, and reactor temperature were changed to maintain smooth operation and obtain performance data for the dispersed molybdenum and hybrid catalyst systems. 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 Unit Operations

After carrying out routine and preventive maintenance work, the plant was put on coal feed on October 31 for start of Run 263. The startup process solvent came from end of Run 262. Conditions for the first test period in Run 263 was nearly similar to the last test period in Run 262, and as a result, process equilibration was established within few days. Run 262 featured the same coal as in Run 263, and except for the last test period, a hybrid catalyst system was used for liquefaction tests. A dispersed molybdenum only catalyst system was used in the last test period of Run 262.

At the beginning of the Run 263, the process conditions were the following:

Coal	
Type	Black Thunder Sub.
Feed rate	300 lb MF coal/hr
Conc. in Slurry	25 wt % MF coal
Process Solvent	
Resid	42 wt %
CI	20 wt %
First Stage	
Temperature	810°F
Dispersed Moly Conc.	200 ppm MF coal
Moly Precursor	Molyvan L
Fe_2O_3 Conc.	2 wt % MF coal
Sulfiding Agent	DMDS
Reactor Volume	Half
Second Stage	
Temperature	800°F (target 825°F)
Supported Catalyst	None
Reactor Volume	Half

In contrast to previous runs (262, 260 and 258), the first eight periods (263A-H) with the dispersed molybdenum catalyst only system were obtained at a low coal concentration of 25 wt % in the feed slurry. This low coal concentration was selected based on several rationales, i.e., anticipation of low exotherms with a slurry catalyst, targeting a high second stage temperature, increased heat capacity at higher slurry flow rates, and use of a higher resid concentration in solvent.

In the first week of the run, the process changes that were made from these test conditions are listed in the following:

Nov. 5-7 First stage reactor temperature increased from 810 to 825°F and second stage reactor temperature increased from 790 to 810°F. Resid in process solvent to 45 wt%.

The target second stage temperature of 825°F in the low/high temperature mode of operation could not be achieved. As a result, the reaction temperatures were changed to 825/810°F between November 5-7, and process performance was evaluated with reactors operating in the high/low temperature mode of operation. Because of lower slurry viscosities, the resid content in the process solvent was increased to 45 wt % during this period.

At the end of Run 262, two short tests of about 4-6 hours duration were conducted on September 30 to determine the feasibility of low/high temperature mode of operation. The tests suggested that low/high operation at 810/825°F is possible at both 300 and 350 lb/hr coal feed. At the beginning of this run, the highest second stage that could be achieved was about 800°F with 810°F in the first stage. The essential difference between the September 30 test in Run 262 and the November 1-7 test in Run 263 is the presence of about 30 lbs (dry) Criterion 324 catalyst in the second stage reactor during September 30 test which apparently contributed to higher exotherm in the second stage reactor and resulting in 825°F reactor temperature. The higher donor quality of the recycle solvent in the September 30 test may also have been a contributing factor.

After the first test period, 263A, the following process condition changes were made in order to further the study of the dispersed molybdenum catalyst system.

Nov. 15 Molybdenum concentration reduced from 200 to 100 ppm.
Nov. 27 First stage reactor temperature increased from 825 to 840°F.
Dec. 10 Coal feed rate lowered from 300 to 265 MF lb/hr and resid in process solvent lowered from 45 to 41 wt %.
Dec. 18 Resid in process solvent increased from 41 to 45 wt %.

Periods 263B (from data between Nov. 15 and Nov. 22), 263C (from data between Nov. 27 and Dec. 9) and period 263D (from data between Dec. 10 and Dec. 17) were obtained. On December 19, the second stage ebullation pump failed and we went off coal feed. The interstage separator vessel (V1258) was inspected during this shutdown because of indications (from the nuclear density gauge) of severe solids buildup. About 100 lbs of mostly gummy material containing some solid chunks were obtained from V1258. The separator was cleaned and the plant was put back on coal feed on December 22. Run 263 continued by processing the Black Thunder mine subbituminous coal.

The resid content in the process solvent was increased to 45 wt% in order to obtain resid extinction and study the process response. Other operating conditions at the startup on December 22 were as follows: Coal concentration = 25 wt %; coal feed rate 260 MF lb/hr; reaction temperatures = 840/810°F; molybdenum concentration = 100 ppm (Molyvan L precursor); iron oxide = 2 wt %; and process solvent blended with 45 wt % resid; 20 wt % CI; and 38 wt % V1074 (heavy vacuum gas oil). The data at these conditions constitute period 263E.

Starting January 2, another molybdenum precursor, Molyvan 822, was tested. Based on tests with model compounds, Amoco's studies showed that Molyvan 822 (4.9 wt % molybdenum and 6 wt % sulfur) was a better hydrogenating catalyst than Molyvan L (8.1 wt % molybdenum, 12.3 wt % sulfur and 6.4 wt % phosphorus). With Molyvan L dispersed catalyst alone, the hydrogen in process solvent decreased to 6.5 wt % (263CD) from 7.1-7.6 wt % (262DE) with the hybrid catalyst system, resulting in 12% lower solvent quality and lower first stage coal conversion.

Also, starting January 2, the recycle solids requirement in the process solvent was met using the atmospheric flash (V1067) bottoms instead of the vacuum flash (V1082) bottoms.

The recycle of atmospheric flash bottoms to meet CI (solids) requirements in the process solvent resulted in an increase of 850°F- material in the process solvent from 11 wt % (with V1082 recycle) to 22 wt %. With atmospheric bottoms recycle, the 750°F- material in gas oil fraction of recycle process solvent increased from about 6 wt % to 30 wt % (an increase from 2 wt % with V1082 bottoms recycle to about 13 wt % with V1067 bottoms recycle in the process solvent).

The V1067 bottoms recycle resulted in production of V1074 (heavy vacuum gas oil with end point around 1000°F) to the tune of 17-20 wt % MAF coal as product. The V1074 (T102 bottoms) as product was obtained in spite of running the T102 vacuum column at low vacuum. In all other test periods, the column was adjusted to maintain a constant inventory of V1074.

The CI recycle from V1082 bottoms was reestablished on January 8 and a data point was taken between Jan 8-14 to compare the process performance with Molyvan 822 and Molyvan L at similar conditions. On January 15, the iron oxide concentration was lowered from 2 to 1 wt % MF coal to evaluate its effect on the process performance.

On January 21-22, the CC-ITSL operation was changed from a dispersed molybdenum catalyst only case to a hybrid catalyst system with both dispersed molybdenum and supported Criterion 324. The 170 (dry) lbs of Criterion catalyst charged to the second stage reactor was aged to 3 lb/ton MF coal replacement rate equivalent in Run 262. The catalyst used in this run came from the catalyst withdrawn in Run 262 during the catalyst addition/withdrawal procedure. The catalyst was exposed to 790°F in Run 262.

With the addition of the supported catalyst while on-stream, the following condition changes also were made: Coal feed rate was increased from 260 to 350 MF lb/hr; and the coal concentration in the slurry was increased from 25 to 30 wt %. Since excess resid was available, the resid in the process solvent was maintained at 45 wt % even though this would have resulted in a negative resid yield. Catalyst replacement at 3 lb/ton MF coal was started on January 23 and the replacement was carried out every other day.

The nuclear density gauge on interstage separator (V1258) was reading 95% indicating a solids buildup in the vessel. Since previous experience with molybdenum dispersed catalyst showed the buildup to be soft (gummy) in nature, an attempt was made on January 24 to wash the deposits away by putting the plant on V178/V1074 solvent recirculation for about 7 hours. The density gauge reading remained at 95%, and since the deposits were not causing any problems with operations, the plant was put back on coal feed on January 24.

On January 27, the molybdenum precursor was changed back to Molyvan L since no significant difference was noticed between Molyvan L and Molyvan 822 and Molyvan 822 supply was exhausted. The coal feed rate was reduced to 300 MF lb/hr for about 10 hrs on January 28 to catch up with slurry preparation. Coal addition to the process solvent was slow due to slower moisture removal because of a lower recirculation flow in the slurry tank. To circumvent the problem, the resid in the process solvent was lowered from 45 to 40 wt %. According to projections, the lower resid concentration in the process solvent should still give a low resid yield as well as allow continued operation at the same resid level for subsequent tests.

On February 3, the inlet hydrogen partial pressure in the first stage was decreased by 400 psia by increasing the amount of gas recycled back to the first stage and decreasing the make-up hydrogen. The immediate effect of this condition change was a decrease in the exotherm across the first stage reactor by about 10-15°F due to less exothermic heat generated in the reactor. The second stage reactor compensated partially by generating slightly higher exothermic heat. During this test period, the V1074 inventory continued to increase due to problems with vacuum jets on the T102 column. The vacuum jets were replaced on February 10.

The inlet hydrogen partial pressure was increased back by 400 psia to normal operating conditions on February 10. Also, the catalyst replacement was suspended and the deactivation of Criterion catalyst to higher catalyst ages was started on February 10. After a brief outage on February 12 to clear the plugged feed gas line, the ebullating pump on the first stage reactor quit pumping due to a plug in the suction line. Attempts made to clear the plug were unsuccessful. Since the temperature rise (the difference between the top and the second thermocouple from the bottom of the reactor) within the first stage reactor did not change significantly, operations were continued without the backmix flow in the first stage reactor.

Criterion catalyst replacement in the second stage at a lower rate at 2 lb catalyst/ton MF coal was started on February 17 and continued on alternate days. Sufficient data was obtained at the higher catalyst age corresponding to 2 lb/ton MF coal replacement rate. This concluded Run 263 and the plant went off coal feed on February 23 at 1223 hours.

3.3.2 Solids Buildup

In the liquefaction of low rank coals at Wilsonville, long-term operability is generally dictated by the onset and rapid growth of solid deposits in the transfer line between the first stage reactor and the interstage separator vessel (V1258) and within the V1258 vessel itself. Severe solid deposition was observed in Run 258 in which two subbituminous coals were screened for their liquefaction potential. Operations were typically restricted to 30-40 days before necessciating a shutdown to clean the deposits. Solid deposition problem is being addressed since Run 260. Process configuration and condition changes and catalysts were tested for their effects on solid deposition.

The solid deposits from Runs 258, 260, and 262 with Black Thunder coal were analyzed by Pennsylvania State University. Run 258 deposits were found to be largely caused by mesophase-derived semicoke (Ref. 14). The deposits formed during the catalytic/thermal operation of Run 260 (sample numbers 16411 and 16403) were also analyzed. These samples were from the second stage thermal reactor. In their report (Ref. 15), the minerals found by X-ray diffraction for both samples were similar. Calcite, halite, and pyrrhotite were the more predominant minerals along with spinel and quartz.

The presence of halite was unexpected because Black Thunder Mine coal typically does not contain chlorine. A recent analysis might explain the presence of chlorine in the solid deposit samples (Ref. 13). Chlorine was found to be present at 2.02 wt % in the iron oxide (purchased from Kerr-McGee Chemical Company, Mobile, Alabama) that was used as a slurry catalyst with DMDS as the sulfiding agent in Run 262. The same brand iron oxide was used in Run 260. A new brand of iron oxide (purchased from Bailey Engineers, Fairfield, Alabama) was used in Run 263 that contains only 0.28 wt % chlorine. Manufacturing methods and compositions are summarized in the following table:

source	Kerr-McGee	Bailey
process	By-product from synthetic rutile (TiO_2) process, ground by Solomon Grind-Chem Services, Inc.	Aiusruther spray roast process of spent acid from steel pickling
specifications		
screen	95% -200 mesh	95% -325 mesh
composition (wt%)		
Iron	62-65	
Fe_2O_3	89-93	98.8-99
Chloride	0.5-1.5	0.11-0.12 (Chlorine)
TiO_2	0.3-0.7	0.001
Manganese	2.5-3	0.33-0.38 (oxide)
Aluminum	0.5-0.9	0.05-0.06 (oxide)
Magnesium	0.3-0.4	0.01 (oxide)
Silica	0.3	0.03-0.08
Sodium	0.3	0.001-0.008 (oxide)
Zinc	0.1-0.3	0.005-0.014 (oxide)
Lead	0.1-0.3	0.000-0.003 (oxide)
Calcium	0.1-0.2	0.028-0.046 (oxide)
Vanadium	0.1	0.003-0.005 (oxide)
Sulfur	0.1	0.002-0.08
Nickel	0.05	0.010-0.012 (oxide)
Chromium	0.03-0.04	0.028-0.031 (oxide)
Thorium	0.02-0.03	
Phosphorus	0.02	0.019-0.021
Potassium	0.01	0.000-0.003 (oxide)
Barium	0.01	
Copper	70-80 ppm	0.007-0.008 (oxide)
Tin	<30 ppm	0.003-0.005 (oxide)
Uranium	<30 ppm	
Molybdenum		0.005-0.006 (oxide)

Run 260 was operated in the catalytic/thermal mode with the intent of running at a low first stage temperature and a high second stage temperature to prevent coke formation along with retrogressive reactions. Optical microscopy analysis of Run 260 deposits (Ref. 15) showed that the reactor bottoms sample was composed of calcium carbonate spheres, pyrrhotite aggregates with carbonate rims and coal-derived minerals cemented together with a thin layer of process-derived mesophase. The reactor wall scale was composed mainly of calcium carbonate cement. The deposits were mainly formed by the deposition or formation and then buildup of calcium carbonate with the other process and coal derived inerts. These results suggested that the process conditions of catalytic/thermal mode to prevent coking might have been effective. However, the larger problem of process-derived calcium carbonate formation has not been and may not be eliminated by changes in process configurations and conditions. This problem may have to be addressed by the removal of organically bound calcium ions before liquefaction.

In Run 262 (Ref. 13), the solid deposition problem associated with low rank coal was addressed by the use of a dispersed molybdenum catalyst to reduce the retrogressive coking reactions. The dispersed catalyst seemed to eliminate the deposits in the lines in between the reactors. However, the interstage separator had some deposition. The deposits in V1258 were semi-solid with a solid block floating in the bottom. This type of deposit (semi-solid) differed from the deposit seen in Runs 258 and 260. The transfer line deposit in Run 260 and the interstage separator deposit in Run 258 were very dense solids.

At the conclusion of Run 262, both reactors, the process lines between the reactors and the interstage separator were inspected. Neither the transfer lines nor the second stage reactor showed any appreciable deposits upon inspection. Both the first stage reactor and V1258 had little deposits, weighing 17 lbs and 37 lbs, respectively. These results were expected since the pressure drop between the reactors or between the first stage and V1258 did not increase during the run. Comparing these results with what was usually found when operating in thermal/catalytic mode, Run 262 with a dispersed molybdenum catalyst was a great improvement in significantly reducing the solid deposition.

In the first part of Run 263, the effect of slurry catalyst only system on solid deposition was studied. The second part of Run 263, which was a continuation of Run 262, featured the hybrid catalyst system and its effect on solid deposition was further studied to confirm the results obtained from Run 262.

To detect the appearance and growth of any deposition, the pressure drop between the reactors and between the first stage and the interstage separator (Figures 8-10), the gas flow rate from the overheads of the interstage separator, the interstage separator temperatures (Figure 11), and the nuclear density gauge on the interstage separator (Figure 12) were closely monitored.

The V1258 nuclear density gauge reading started to increase in the beginning of the run. This is an indication of deposits being formed in V1258. The density gauge reading was back close to zero on November 22 when the unit was shutdown due to ebullation pump problems and put on solvent. It was suspected that due to incorrect level transmitter readings, the level in the vessel was actually higher than the plane scanned by the nuclear density gauge. In normal operation, the level in the vessel is kept below the nuclear density gauge scanned section. It is also possible that gummy material may have accumulated in the vessel which might have washed off once the plant went on solvent on November 22.

The nuclear density gauge reading again started to increase once the first stage temperature was increased to 840°F on November 27. The readings continued to increase and reached 100% on December 18-19. The level in V1258 was lowered to a point much below the nuclear density gauge scanning section several times during operations; however, no change in the density gauge reading was apparent. This was a positive indication that solid deposits did occur in V1258.

The plant was put on solvent on December 19 due to ebullation pump problems, but at this time the reading on the nuclear density gauge stayed at 100% (Figure 12). On inspection, solid deposit was observed in the separator vessel. The deposits occurred 20 days after the first stage temperature was increased to 840°F from 825°F. The 100 lbs of deposits recovered on cleaning were mostly gummy material. This included about 20 lbs of hard dry chunky material which was deposited on the walls of the vessel opposite to the feed entry point. Ash and CI analyses along with metal analysis of the deposit material are given in Table 3 and a comparison with Run 262 is given in Table 4.

The interstage separator vessel was cleaned and put back in service on December 22. The run continued with the dispersed molybdenum catalyst only system. The deposits returned to V1258 in middle of January as was evidenced by the nuclear density gauge reading and ragged operation of the flash vessel. On January 24, an attempt was made to wash off the solids in V1258 by placing the unit first on V178 solvent (middle distillate) and then on V1074 (heavy vacuum gas oil. The solids in V1258, however, remained intact. The plant was put back on coal feed and operations continued. It requires a few days shutdown to cool the system and clean the vessel, and since only a few tests were remaining to end Run 263, operations were resumed with V1258 uncleaned. Towards the end of the run, the reading of the thermocouple located between the V1258 feed inlet and the bottom of the vessel started dropping (Figure 11). This is an indication of the deposits growing upwards in the vessel and covering the thermocouple.

The pressure drop across R1235-V1258 and across R1235-R1236 reactors showed small increases over a number of days (Figure 8). In absolute numbers, the R1235-V1258 pressure drop is small. Small changes in the pressure drop occurring over a number of days are probably within measurement error due to problems such as salt and process stream deposits in instrument legs. The rapid increase in pressure drop is however a good indication of solid deposits in the transfer lines. This was seen in earlier runs (Runs 258 and 260). A comparison of pressure drops in Runs 260 and 263 (Figures 9 and 10) shows that the rate of increase in pressure drop in Run 263 was much smaller than in Run 260. This is a good indication that the deposits in the transfer line in Run 263 did not occur at any time during the run in spite of processing a low rank coal for nearly 4 months.

The thermocouple in the bottom of the first stage reactor progressively started reading lower than normal values since December 29. Again, this is an indication of deposits in the bottom of the reactor and around the thermocouple.

Run 263 ended on February 23, and the end-of-run inspection showed deposit in V1258. The vessel was filled with deposit material up to a point just below the feed nozzle. About an inch diameter hole (opposite to the point where the feed enters the vessel) was left in the deposit material for the process stream to flow. The deposits were hard dry material. Small amount of deposits was also found in the bottom of the first stage reactor. Analysis of the deposit materials are given in Tables 3 and 4. The transfer lines between the first stage reactor and V1258 and between V1258 and the second stage reactor were inspected at a number of places and were found to be clean.

At the end of over six months of testing with the Black Thunder mine subbituminous coal (Runs 262 and 263), the process lines between the reactors and after the second stage reactor were clean and free of any solid deposit. This was a major improvement compared to Run 258 in which deposits in process lines precluded continuous operation for more than a month. The major difference between Run 258 and Runs 262 and 263 is the addition of the dispersed molybdenum catalyst, and it appears that it did play a role in preventing deposit formation in the process lines.

Even with molybdenum in the process, deposits were observed in the interstage separator. The deposit formation in the separator can probably be prevented with a redesign of the vessel along with a hydrogen purge. This supposition was based on the experience of the H-Coal direct liquefaction pilot plant operation at Catlettsburg, Kentucky (referred to the report published by Hydrocarbon Research, Inc., "H-Coal Pilot Plant Operations," DOE Document No. DOE/ET/10143-T37 (Volume 4), 1984. pp 4.8-38 to 4.8-48). The reactor effluent separator (Q-224) at the Catlettsburg plant was plagued with solids deposition and coking problem. Analysis of the deposits suggested a settling action followed by coking. Hydrogen was introduced through a sparge ring in Q-224 to prevent coking by increasing the concentration of dissolved hydrogen in the liquid and to provide agitation to minimize sedimentation. High hydrogen sparge gas rates, 60,000 - 130,000 SCFH, were used. In spite of this effort, large amounts of hard solid deposits, requiring air hammer to break up, resulted from bituminous coal runs using Kentucky No. 9 and Illinois No. 6 coals (Runs 7 and 8).

Further design changes to Q-224 was made at Catlettsburg to control solid deposit. The bottom geometry of the vessel was changed to reduce both sedimentation and slurry residence time. A conical bottom was fabricated by filling the annular space with refractory, and this reduced the slurry residence time from about 5 minutes to 1 minute. Inspections at the end of Runs 9 (Wyodak coal), 10 (Wyodak coal), and 11 (Illinois and Kentucky coals) showed Q-224 to be free of deposits.

Unlike the Q-224 separator in Catlettsburg plant, the V1258 separator at Wilsonville never had any deposit problems with bituminous coal runs. Recycle gas, which was mostly hydrogen, was used to purge the instrument lines on V1258. This resulted in about 500 SCFH of recycle gas (equivalent to approximately 10 volume % of the gas through the reactor) being bubbled through the liquid in V1258. The maximum hydrogen purge rate through Q-224 at Catlettsburg was 22-33 volume % of the gas flow rate through the reactor (400,000-600,000 SCFH).

The redesign of the Q-224 separator at Catlettsburg to decrease the liquid residence time in the vessel by using a conical bottom apparently prevented solid deposition problems. Similar success was noted at Wilsonville in Run 260 by a redesigned separator, V1258B. In redesigning V1258, the diameter of the separator was reduced by a factor of about four in order to increase the linear liquid velocities by a factor of 13 through the separator to scavenge away any deposits. The residence time of slurry in the separator was decreased from about 4 to 1 minute by redesign. Even though the redesigned vessel was clean when checked in the middle of the run, it did not perform adequately due to poor separation of gas and liquid. Perhaps a conical bottom for the separator, as tested at Catlettsburg, with a larger interface area for separation may be the solution to prevent deposits and at the same time achieve adequate flash vaporization.

3.3.3 Process Performance

The resid and coal conversions, hydrogen consumption, and distillate and resid yields were monitored daily as process performance indicators. Phase 2 TSL yields are summarized in Table 5 for each period of the run. Internal accumulation of UC and ash in the recycle loop, "Int. Accum. (Res. Free)", was calculated for each period and is included in the table to evaluate the TSL system performance stability. This value is affected by the imbalances of yield components (UC, ash, etc.) caused by experimental errors. The 850°F-EP distillate yield varied over a wide range, i.e., from 3.7 to -31.0 wt % MAF coal. This variation is probably due to analytical problems associated with gas chromatograph handling of heavy end distillates, resulting in a higher yield of 750-850°F distillate. The 750°F-EP distillate yield combined from the above two fractions varied less from -7.8 to +8.3 wt % except for the 263F period with +15.3

wt %. A detailed discussion of process performance and unit conversions is given in Section 4.0.

Figure 13 shows a comparison of overall resid+UC conversions from Runs 262 and 263. Except for the last test period, a hybrid catalyst system was tested in Run 262. The hybrid catalyst system tests were continued in Run 263 during periods 263I-M. A dispersed catalyst only system was tested in 263A-H periods. As seen in Figure 13, the R+UC conversions in 263A-B (upto operation day, od=22) was much lower than that obtained with the hybrid catalyst system in Run 262.

The increase in conversion for 263C is a result of increasing the first stage temperature from 825°F to 840°F on November 27 (od=23). Overall and first stage resid+UC conversion increased after the increase to 840°F, but second stage conversion remained constant. As expected, the overall and first stage resid+UC conversion also increased in 263D due to a decrease in the coal feed rate to 265 from 300 lbs/hr.

As shown in Figure 13, the resid+UC conversion for the hybrid case of dispersed catalyst through the first stage and supported catalyst in the second stage in Run 262 was higher than that for the dispersed molybdenum catalyst only system in Run 263:

	Resid + UC Conversion (wt %)		
Period	1st Stage	2nd Stage	Overall
262D	24.4	16.0	36.5
262E	20.7	17.9	34.8
263B	17.8	11.6	27.4
263C	19.5	11.4	28.7
263D	22.2	12.0	31.5

With the addition of supported catalyst to the second stage beginning period 263I (od=73), the overall resid+UC conversion increased from 31.4 wt % (based on Phase 2 feed R+UC data) in 263H to 36.9 wt % in 263I. Much of this increase was due to the increase in the second stage resid+UC conversion (12.7 wt % in 263H vs. 18.2 wt % in 263I). The first stage also showed a small increase in resid+UC conversion (from 21.4 wt % in 263H to 22.9 wt % in 263I). The overall resid+UC conversions with the hybrid catalyst system in 263IJ periods were similar to that observed in Run 262.

The performance of the second stage reactor increased as soon as the supported catalyst was added as was seen in a tremendous increase in temperature rise across the reactor. The resid+UC conversions, especially in the second stage, also increased within a day. These observations show that the process solvent characteristics and hence the conversions are reversible and can be improved, and the performance is to some extent is independent of the history. Using Phase 3 elemental balance data, the effects of changes in test parameters on the first, second and overall resid+UC conversions are further discussed in Section 4.0.

Reactor operating data for each period are listed in Table 6 and Figure 14 shows the temperature changes across the reactors and within the reactor beds. Increases in the exotherm across the first stage reactor and a simultaneous decrease in the first stage in-bed exotherm through operating day 15 might be due to the use of process solvent from the previous run. The first stage in-bed exotherm increased from 15 to 29°F beginning at period 263C (od=23). This was the result of increasing the first stage temperature to 840°F. The additional increase starting with operating day 42 was the result of thermocouple problems.

Both the first and second stage in-bed exotherms showed further increase with the addition of supported catalyst to the second stage (beginning od=73). The first stage exotherm increased from about 28°F in 263G and 32°F in 263H to 42°F in 263I and 50°F in 263J. Operating with the lower hydrogen partial pressure in the first stage in 263K decreased the first stage exotherm to about 39°F. Also, deactivation of catalyst in the second stage probably resulted in lower in-bed exotherms in the first stage in 263LM periods (38 and 37°F, respectively).

The ebullation pump on the first stage was shutdown on February 13 (od=95) for the rest of the run. Surprisingly, the first stage in-bed exotherm did not show any increase.

The second stage exotherm across the reactor increased significantly with the addition of supported catalyst (on od=73). The second stage exotherm across the reactor was less than zero (reactor and reactor outlet temperatures less than the reactor feed temperatures) for tests with the dispersed catalyst only system. This was the result of heat losses from the reactor being much higher than the amount of exothermic heat that was being generated. With the addition of supported catalyst, the hydrogen consumption and the resid+UC conversion in the second stage increased significantly resulting in positive exotherms across the reactor. The second stage in-bed exotherm also increased from about 1.6°F in periods 263E-H to 6-7°F in periods 263I-M. Catalyst deactivation did not decrease the in-bed exotherm in the second stage.

Catalyst was withdrawn from the reactor at the conclusion of the run. The recovery was only 69%. On inspection, severe wear was seen on the plenum chamber of the second stage reactor. There were holes on top of the plenum chamber and in the device for distributing the gas/slurry mixture into the reactor. The shortcircuting of the gas and slurry flow through the holes would have caused excessive catalyst attrition which would explain the low catalyst recovery.

The CCR unit on stream efficiency for the entire Run 263 was 91.2% with 2482 hours on stream. This was one of the longest runs with low rank coals without any frequent shutdown due to solid deposition. In the nearly four month long run, only once the V1258 was cleaned of deposits to improve operability. A longer run was possible without a shutdown due to solid deposits since virtually no deposit was observed in the process lines in spite of operating the first stage at 840°F (in the previous runs, temperatures above 825°F was suspected as a cause for catalyst did play a role in preventing deposit formation in the process lines, and the deposit formation in V1258 can probably be prevented with a redesign of the vessel, giving a higher linear velocity with a conical bottom vessel, and by introducing a hydrogen purge stream to the vessel as implemented in the H-Coal pilot plant (see Section 3.3.2).

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

The objectives of Run 263 for the Residuum Oil Supercritical Extraction - Solids Rejection (ROSE-SR^{SM}) unit were to:

- Evaluate unit performance while using Molyvan L as a dispersed catalyst with Black Thunder Subbituminous coal and varying the reactor temperatures, resid concentration in process solvent, and coal feed rate,
- Recover full range resid used in coal slurry preparation while achieving acceptable deashing, and
- Obtain optimum resid recovery with minimum energy rejection into the bottoms product.

3.4.1 Unit Operation and Process Performance

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 the process performance and operability.

Operations in the ROSE-SR^{SM} unit for Run 263 improved relative to Run 262 (Ref. 13). The severe plugging problems experienced in the beginning of Run 262 did not repeat. As shown in Figure 15, organic rejection and resid recovery in ROSE-SR^{SM} bottoms product also showed improvement over Run 262 (see Figure 19 in Ref. 13). This was mainly due to the higher coal conversion obtained by a higher first stage temperature starting in period 263C, however, with the hybrid system (supported catalyst in the second stage reactor), organic rejection began to increase primarily due to lower coal conversion as a result of higher space velocities in the reactors. Ash in the ash concentrate (Figure 16) showed a similar pattern of being highest when coal conversion was highest. As shown in Figure 17, the preasphaltenes of the ROSE-SR^{SM} feed were low (3-10 wt %) with the use of dispersed molybdenum catalyst at 100-200 ppm molybdenum addition rates, as was observed in Run 262 at 100-1000 ppm molybdenum addition rates (2-11 wt % preasphaltenes) (see Figure 17 in Ref. 13). The preasphaltenes were higher without the use of dispersed molybdenum catalyst, at 7-16 wt % in Run 260 (see Table 8 in Ref. 11) and 8-22 wt % in Run 258 (see Table 14 in Ref. 9).

The strength of the deashing solvent (related to solvent extraction power and recovery of resid from the ROSE-SR^{SM} feed without ash carryover) is presented in Figure 18. The feed solubility properties normally dictate the strength of the deashing solvent; a stronger solvent with a less soluble feed and vice versa. For Run 263 approximately the same strength of deashing solvent was maintained throughout the run. In period 263H, the iron-oxide was reduced from 2 wt % MF coal to 1 wt %. The initial calculated response gave a reduced organic rejection, however, as the system equilibrated there was little change in the organic or energy rejection for this test (see Table 7). The sulfur content in the bottoms product (Table 8) was less at the lower iron-oxide addition rate of 1 wt %. The oxygen content in the bottoms product showed large negative values (-1 to -5 wt %), which was similar to that observed in Run 258 with the same Black Thunder subbituminous coal. This negative oxygen content was calculated by difference, affected by the errors in estimation of other elements and probably due to the high ash content with formation of $CaSO_4$ during ashing of the bottoms product. Figures 19 to 21 illustrate the trend data for energy rejection and ash content in the ROSE-SR^{sm} bottoms product.

3.5 Distillation System

The objectives of the V1082/T105/T102 distillation system for Run 263 were:

- To remove the low boiling point products in T105 (the atmospheric distillation column) from the interstage separator overheads and from the liquids flashed overhead after the second stage reactor, and
- To split in T102, the T105 bottoms and heavy vacuum flashed overheads from V1082 into a product stream and a heavy vacuum gas oil that is in balance with the recycle distillate needs for blending with the coal.

Run 263 was the fifth consecutive run to use T102 vacuum column for adjusting the entire split between the recycled distillates and product distillates. In periods 263F and 263K, the entire split was not made in T102. Higher end boiling point product was expected in period 263F due to recycle of atmospheric flash bottoms instead of vacuum flash bottoms for process solvent makeup. Period 263F was set to take a higher end point product while maintaining the T102 vacuum at 2.5 psia, resulting in accumulation of considerable T102 bottoms (V1074 heavy vacuum gas oil) as product. Normally vacuum in the column will be adjusted to maintain a constant inventory of V1074 gas oil, and all the product will be obtained as T102 overheads. In period 263K, the vacuum jets were not functioning correctly resulting in too low a cut point and production of too much recycle V1074 gas oil.

The range of cut points for Run 263 was 760 to 804°F (see Table 9) except for period 263F (745°F) and 263K (763°F). The GC cut points are close to the end boiling points of the product stream except for 263F and K periods. The cut points for Runs 262 and 263 are plotted in Figure 22. Essentially all the cut points were above 770°F. The overlap for the cut points remained low (Figure 23 and Table 9) indicating that the vacuum column was giving similar performance.

The lightest distillate (V161 from T105 overheads in Figure 24) was primarily overheads from the second stage. The T105 overheads had a boiling point below 550°F. The data from Runs 262 and 263 are plotted on Figure 24. During periods when a supported catalyst was present, the amount of IBP-350°F naphtha fraction was higher than periods without the supported catalyst. This indicates that the supported catalyst cracked the distillates to lighter products. For most of the run, the T102 overheads (V182 in Figure 25) had little material boiling above 850°F by GC simulated distillation. The heaviest product from T102 bottoms (V1074 in Figure 26) was recycled to make the coal slurry. The T102 bottoms solvent was very high boiling with 60-80% boiling above 850° by GC simulated distillation.

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 10 to 12. Detailed operating conditions, averaged elementally balanced overall yields and unit contributions are summarized in Tables B and C for all representative periods. Internal accumulation of UC in the recycle loop, "Int. Acc. UC", was calculated for each period and is included in Table C to evaluate the TSL system performance stability. This value is affected by the imbalances of yield components (UC, ash, etc.) caused by experimental errors. The yield contributions are also shown schematically in material balance flow diagrams (Figures 27 to 39).

4.1 TSL Performance

Black Thunder mine coal was processed in Run 263 with a dispersed molybdenum catalyst (using either Molyvan L or Molyvan 822 as precursors) system as well as with a hybrid system containing both molybdenum slurry and supported Criterion 324 catalysts. Iron oxide along with dimethyl disulfide (DMDS) as sulfiding agent was also used as a slurry catalyst throughout the run.

Run 263 was started on October 31 and ended on February 23. Thirteen material balance periods were obtained in this run and test conditions and process performance in each of these periods is discussed in detail below. Periods 263A-H cover tests with the dispersed molybdenum catalyst system and periods 263I-M cover tests with the hybrid catalyst system. Molyvan 822 was used as a dispersed molybdenum catalyst precursor in periods 263F-I, and Molyvan L was used in other test periods.

Before Run 263, Black Thunder mine subbituminous coal was tested extensively in the CC-ITSL mode at Wilsonville in three different runs -- Runs 258, 260, and 262. Run 258 featured a thermal/catalytic system with Criterion (Shell) 324 unimodal catalyst and iron-oxide (+DMDS) as the slurry catalyst to promote coal conversion. Both thermal/catalytic and catalytic/thermal modes were tested in Run 260 with Criterion and iron-oxide slurry catalyst. A hybrid catalyst system was tested in Run 262 with both supported Criterion 324 catalyst and slurry iron-oxide and dispersed molybdenum catalysts. Molyvan L was used as a precursor to obtain the dispersed molybdenum catalyst *in situ*. In this run, Run 263, Black Thunder mine coal was tested with both the slurry and hybrid catalyst systems.

In Run 262, the coal concentration in the slurry (to reactor feed) was maintained at 30 wt %. However, in this run, in tests with the dispersed molybdenum catalyst only system (periods 263A-H), the coal concentration in the slurry was maintained at 25 wt

%. This low coal concentration was selected based on several rationales: anticipation of low exotherms with a slurry catalyst, targeting a high second stage temperature, increased heat capacity at higher slurry flow rates, the use of higher resid concentrations in solvent. The low coal concentration enabled operations at increased resid concentrations of up to 45 wt % in the process solvent. The coal concentration was increased back to 30 wt % for tests with the hybrid catalyst system in periods 263I-M. Preliminary process economics evaluation made by M. Peluso of LDP Associates, Piscataway, New Jersey (presented at the 115th Wilsonville Technical Review Meeting held on March 18-19, 1992) indicated potential economic tradeoffs between high coal concentration and high resid concentration. Comparing dispersed and hybrid catalyst systems (263E vs. 262E), the negative impacts of the lower coal space rate and coal concentration in the dispersed catalyst mode of operation in period 263E were more significant from an economic standpoint than the savings in the supported catalyst replacement and the improvement in coal conversion.

Large variation in ash in feed coal was observed in this run (from 6.8 wt % MF coal in 263H to 7.7 wt % in 263A). These coal ash values were higher than those observed in Run 262 (6.3-6.5 wt % MF coal). A new coal pile was used since January 23. There was less variation with this new pile, and the coal ash values ranged from 6.5 to 6.7 wt % MF coal.

Table B summarizes in detail the operating conditions for all the periods in Run 263 (263A-M). The overall TSL yields and conversions are summarized in detail in Table C. Test conditions and TSL conversions and yields from Run 263 are plotted in Figures 40-44. As shown in Figure 40, the first stage temperature was increased from 825°F in 263AB to 840°F in 263C, and the temperature was maintained around 840°F for further tests in Run 263. The second stage temperature was maintained around 810°F for all tests in Run 263. During period 263H, the second stage temperature dropped to 806°F because of higher heat losses due to cooler ambient conditions. In order to decrease the resid yield, the space velocity was decreased from about 78C lb MF coal/hr/ft^3 in 263ABC to 69C lb MF coal/hr/ft^3 in period 263D. This lower space velocity was maintained during further tests in periods 263E-H with the dispersed catalyst system. With the changeover to hybrid catalyst system in period 263I, the space velocity was increased by over 32% to 91C lb MF coal/hr/ft^3, and this space velocity was maintained for further tests during periods 263J-M.

For most of the tests with the dispersed catalyst system (263E-H), the resid in the process solvent was maintained around 43-45 wt%. The resid concentration was decreased to about 40 wt% during hybrid catalyst system tests in periods 263J-M. The decrease in resid concentration was necessitated because of insufficient resid in the system due to a limitation on the coal feed rate (350 lb MF coal/hr for subbituminous coals). Operations with lower resid content in the process solvent allowed us to increase the coal concentration in the feed slurry from 25 wt% (263A-H) to 30 wt% (263I-M). The effect of these and other operating condition changes on process performance are illustrated in Figures 41-44 and are discussed in detail below (coal conversions and organic rejection, resid+UC conversion and resid yield, C_4+ distillate yield and C1-C3 gas make, and hydrogen consumptions are plotted respectively in Figures 41-44 for each test period in Run 263).

In period 263A, at 825/810°F reactor temperatures and molybdenum at 200 ppm MF coal concentration, the resid yield was high at 9.4 wt % MAF coal with C4+ distillate yield and C1-C3 gas make being 47.9 and 6.7 wt %, respectively. The coal feed rate was around 300 MF lb/hr. The TSL coal conversion was high at 92.8 wt %; however, the organic rejection was also high at 21.3 wt %. This period could be compared to 262D with similar coal feed rate, first stage temperature and molybdenum concentration, but with a hybrid catalyst system. The TSL coal conversion in 262D was lower at 90.1 wt % (probably due to a lower second stage temperature of 790°F) with a lower organic rejection (19.0 wt %). The distillate yield in 262D was much higher (60.8 wt %) and the resid yield was much lower (-0.3 wt %) compared to 263A. The overall resid+UC conversion in 262D was much higher compared to 263A (80.8 vs. 68.7 wt % MAF).

In period 263B, a baseline data point was obtained at a lower molybdenum concentration of 100 ppm MF coal. Based on the cost of molybdenum precursor, (Molyvan L), 100 ppm molybdenum or less may be reasonable for liquefaction tests. Also, the effect of a decrease in molybdenum concentration from 200 to 100 ppm appeared to be insignificant on the process performance as evidenced from periods 262C and D with the hybrid catalyst system case. The coal conversion in both 263A and B was nearly similar (92.8 vs. 92.5 wt %); however, the organic rejection in 263B decreased considerably to 17.0 wt % compared to 21.3 wt % in 263A. The lower organic rejections observed as the run progressed was probably related to changing characteristics of the resid in the ROSE-SR^{SM} unit feed, and not due to any operation related improvement of the resid recovery process. In addition to the transitional operation and performance of the ROSE-SR^{SM} unit for periods 263A and 263B as indicated in Figures 15, 16 and 18, a slightly lower feed coal ash in 263B (7.0 wt %) might have slightly lowered the organic rejection compared to 263A in which the coal ash content was 7.7 wt %.

Higher distillate yield was obtained in 263B compared to 263A (51.8 vs. 47.9 wt % MAF coal); however, only a slight decrease in resid yield was observed (8.6 vs. 9.4 wt %). Higher distillate yield in 263B was probably due to the higher resid+UC conversion

(73.5 vs. 68.7 wt %) with lower organic rejection compared to 263A (17.0 vs. 21.3 wt %). It is interesting that in spite of lower molybdenum and resid concentrations, 263B had about 4 wt % higher distillate yield than 263A. This might again indicate a transitional trend in changing to the slurry catalyst system as a similar transition was observed in the ROSE-SR^{SM} unit performance. The overall resid+UC conversion for the dispersed molybdenum catalyst system in 263AB was less than that for the hybrid catalyst system in 262D (68.7-73.5 vs. 80.8 wt %).

The first stage temperature was increased to 840°F in period 263C. This resulted in higher first stage coal conversion at 86.1 wt % compared to 80.4-82.2 wt % in periods 263AB. However, the TSL coal conversion in 263C did not show any significant increase (92.9 vs. 92.5-92.8 wt % in 263AB). The organic rejection in 263C was similar to that in 263B (17.0-17.5 wt %). The resid yield in 263C decreased by 2.0 wt % to 6.6 wt % from 8.6 wt % in 263B. However, the C4+ distillate yield increased by only 0.3 wt % to 52.1 wt %. The C1-C3 gas make increased from 6.3 wt % in 263B to 7.8 wt % in 263C due to the increased first stage temperature operation.

The coal feed rate was decreased to about 260 MF lb/hr in 263D period in order to decrease the resid yield. This test allowed for accumulation of kinetic data at a lower space velocity and at higher distillate yields while other conditions nearly remained the same. In this way, the effect of space velocity on rate constant can be decoupled from other variables, especially the reaction temperature. In the past runs, a change in space velocity was accompanied with a change in other variables to maintain the process at steady-state operation, and this resulted in resid+UC conversion being invariant to space velocity. With the decrease in coal feed, it was anticipated that the resid in process solvent will decrease to about 38 wt % at resid extinction. However, Phase 3 data indicates that the response to a 12% decrease in space velocity was minimal and the resid yield in period 263D decreased to only about 5.5 wt %.

The resid in process solvent decreased from 43.3 wt % in 263C to 41.1 wt % in 263D. The coal conversion in 263D was similar to 263C (93.1 vs. 92.9 wt % MAF coal), but the organic rejection in 263D was less (15.8 vs. 17.5 wt %). The overall resid+UC conversion increased to 78.5 wt % in 263D compared to 75.4 wt % in 263C. The resid yield decreased only slightly from 6.6 wt % in 263C to 5.5 wt % in 263D. But, due to lower organic rejection, the distillate yield increased from 52.1 wt % in 263C to 54.6 wt % in 263D and the C1-C3 gas yield increased from 7.8 to 9.1 wt %.

The resid content in the process solvent was increased back to 44 wt % in period 263E in order to obtain resid extinction and study the process response. This resulted in a lower resid yield of

2.4 wt % MAF coal in 263E. Also, compared to period 263C (both 263C and E were at similar resid concentration in the process solvent), both the coal conversion and resid+UC conversion improved (coal conversion 92.9 vs. 94.1 wt % and resid+UC conversion 75.4 vs. 83.8 wt %), resulting in a decrease in organic rejection from 17.5 wt % in 263C to 14.1 wt % in 263E. While the distillate yield increased from 52.1 wt % in 263C to 60.7 wt % in 263E due to lower space velocity, the C1-C3 gas make also increased from 7.8 to 9.0 wt %. The higher C1-C3 gas make and distillate yield resulted in an increase in hydrogen consumption from 4.6 wt % in 263C to 5.5 wt % in 263E (Figure 44).

Comparing periods 263D and E, the effect of increase in resid in the process solvent appeared to have increased the overall TSL coal conversion (93.1 vs. 94.1 wt % MAF coal), resid+UC conversion (78.5 vs. 83.8 wt %), distillate yield (54.6 vs. 60.7 wt %), and, as shown in Figure 44, hydrogen consumption (5.2 vs. 5.5 wt %). The resid yield decreased from 5.5 wt % in 263D to 2.4 wt % in 263E and the organic rejection also decreased from 15.8 to 14.1 wt %. The increase in TSL coal conversion seemed due to an artifact of the ROSE-SR^{SM} unit operation, since the second stage coal conversions (after the second stage reactor with V1082 vacuum flash bottoms samples) were similar for both periods (94.5-94.7 wt %). A similar inconsistent result was observed for period 263D when the coal space rate was lowered from 263C (no change in the TSL conversion, but an increase in the second stage coal conversion).

In period 263F, the recycle solids came from the atmospheric flash (V1067) bottoms instead of the vacuum flash (V1082) bottoms. With atmospheric flash bottoms recycle, the 750°F- material in the process solvent increased from about 2 wt % (with V1082 recycle) to about 13 wt %. The V1067 recycle resulted in collection of V1074 (heavy vacuum gas oil with end point between 1000-1100°F) as product to the tune of 17-20 wt % MAF coal. Also, in this period, another molybdenum precursor, Molyvan 822, was used to generate the active molybdenum catalyst *in situ* in the reactors. Molyvan 822 contains 4.9 wt % molybdenum compared to 8.1 wt % in Molyvan L. Molyvan 822 was injected into the reactor along with DMDS, and the Molyvan 822 addition rate was set to give 100 ppm MF coal molybdenum concentration.

Period 263F was termed transitional since the test period was relatively short (January 2-7). It takes about 4-6 days to displace molybdenum from Molyvan L from the system. The process performance in periods 263E and F were nearly similar except for a decrease in resid yield (from 2.4 to 0.2 wt % MAF coal) and a slight increase in distillate yield (from 60.7 to 61.5 wt %) as a result of an increase in overall resid+UC conversion from 83.8 wt % in 263E to 86.0 wt % in 263F. Since statistically 263E and F process performances were equivalent, the only result of atmospheric flash bottoms recycle was in the distribution of boiling point fractions in the product towards heavier end. Process performances with atmospheric flash bottoms recycle and vacuum flash bottoms recycle were similar except for product end point. In order for a net shift in boiling point distribution to be observed, a net shift in reaction rates must occur. In our tests, this was accomplished by changing the concentrations of the relevant reactants (not necessarily the rate constants themselves). That is, with vacuum bottoms recycle the heavy end crack to a greater extent. Also, lighter ends are more resistant to cracking and report to the vapor phase to a greater extent.

The CI recycle from V1082 was reestablished on January 8 and period 263G (January 9-13) represents data comparing Molyvan 822 process performance with that of Molyvan L (period 263E) at similar conditions. Comparing period 263G with 263E, the coal conversion and organic rejection were similar. As a result of lower resid+UC conversion in 263G (81.7 vs. 83.8 wt % MAF coal), the resid yield in 263G with Molyvan 822 was higher (4.1 vs. 2.4 wt %) and the distillate yield was lower (57.0 vs. 60.7 wt %). It appeared that Molyvan 822 exhibited lower hydrogenation activity compared to Molyvan L when operated at the same 100 ppm molybdenum addition rate, although the difference was not statistically large.

In period 263H, the iron oxide concentration was lowered from 2 to 1 wt % MF coal. The first stage coal conversion decreased from 89.3 wt % MAF coal in 263G to 88.4 wt % in 263H. However, the resid+UC conversion increased from 81.7 wt % in 263G to 83.8 wt % in 263H resulting in a decrease in resid yield from 4.1 to 2.4 wt % and an increase in distillate yield from 57.0 to 60.9 wt %. The $CO+CO_2$ yield decreased from about 5.9 wt % in 263G to 4.2 wt % in 263H.

The hybrid catalyst system tests started with period 263I. Criterion 324 catalyst aged to 3 lb/ton MF coal catalyst replacement equivalent in Run 262 was used in 263I. The catalyst replacement rate was maintained at 3 lb/ton MF coal. The coal concentration was increased from 25 wt % in 263H to 30 wt % in 263I and the coal feed rate was increased from about 260 to 350 MF lb/hr. Since excess resid was available, the resid in process solvent was maintained at 45 wt %, even though this would have resulted in a negative resid yield. Period 263I was termed transitional since it was relatively a short period taken immediately after commencing operations with the hybrid catalyst system.

The process conditions for the hybrid catalyst system was chosen based on period 262E. The limited data in 262E indicated catalyst deactivation at 810°F. The initial test objective with the hybrid system was to evaluate further catalyst activity and also quantify improvements in process performance at the higher 840°F first stage temperature.

In period 263J, the resid in the process solvent was lowered from 44.8 to 40.5 wt %. This was done mainly to maintain the resid in the process solvent at the same level in subsequent tests. The process solvent contained 38.3 wt % resid in period 262E. Periods 263IJ can be compared with 262E to evaluate the benefit of higher first stage temperature of 840°F (263IJ). The coal conversion in 263IJ was higher compared to 262E (93.5 vs 91.6 wt % MAF coal) and the organic rejection was much lower at 13.6 wt % in 263IJ compared to 19.8 wt % in 262E. The distillate yields in 263J and 262E were similar, however, the resid yield in 263J was much higher (3.7 vs. -0.4 wt %) due to higher coal conversion and lower organic rejection in 263J.

The inlet hydrogen partial pressure to the first stage was reduced by about 360 psia from 2744 psia in 263J to 2386 psia in 263K. These partial pressures are based on compressor discharge pressure. Based on reactor pressure, the hydrogen partial pressure was decreased from 2566 psia in 263J to 2226 psia in 263K. The decrease in hydrogen partial pressure was achieved by increasing the recycle gas flow to first stage which translated to a commercial plant will mean a relatively smaller gas cleanup circuit with a smaller purge gas stream. The recycle gas to first stage increased from about 1187 scfh in 263IJ and 700 scfh in 263LM to 1773 in 263K. The total gas flow to first stage decreased from 4500 scfh in 263IJ and 263LM to about 4040 scfh in 263K. Other test conditions in 263K remained similar to 263J.

Comparing periods 263J and K, the overall effect of reduction in first stage hydrogen partial pressure on the process performance appeared to be marginal. However, the first stage resid+UC and coal conversions decreased (51.8 vs. 47.9 wt % resid+UC conversion; 89.6 vs. 88.2 wt % coal conversion), showing the stage interaction with the second stage conversions increased. Water yield increased in 263K compared to 263J (16.5 vs. 14.9 wt %) with simultaneous decreases in $CO+CO_2$ gas yield (6.0 wt % in 263J vs. 4.8 wt % in 263K) and C1-C3 gas yield (7.9 wt % in 263J vs. 7.4 wt % in 263K).

Criterion catalyst was batch deactivated from an age equivalent to 3 lb/ton MF coal catalyst replacement to 2 lb/ton replacement. Period 263L represents the average of data during the batch deactivation period. The first stage inlet hydrogen partial pressure was increased back from 2386 psia in 263K to 2782 psia in 263L. Period 263M represents the data taken at steady-state operations with a catalyst replacement rate of 2 lb/ton MF coal.

Comparing periods 263LM with period 263J, the TSL coal conversion decreased from 93.6 wt % MAF coal in 263J to 92.7 wt % in 263LM and the overall resid+UC conversion decreased from 82.4 to about

80.4 wt %. Even though the overall resid + UC conversion decreased in 263LM periods, the first stage resid+UC conversion in 263LM was higher than 263J (56.2 vs 51.8 wt % MAF coal), showing the effect of stage interaction. The catalyst deactivation had a greater effect in the second stage resid+UC conversion which decreased from 30.6 wt % feed in 263J to 24.1 wt % feed in 263M. The organic rejection increased from 13.4 to about 15.3 wt % MAF coal. The resid yield increased from 3.7 wt % in 263J to 5.1 wt % in 263M and the distillate yield decreased from 58.4 to 54.6 wt %. As shown in Figure 44, the hydrogen consumption decreased from 5.8 wt % in 263J to 5.5 wt % in 263L and 5.2 wt % in 263M.

Figure 41 shows that throughout Run 263, the difference between the 1st&2nd stage coal conversion and the TSL coal conversion was low and ranged from 0 to 1.4 wt% MAF coal, indicating that no significant retrogressive reactions occurred. As a result of process condition changes with the dispersed catalyst system (the increase in first stage temperature to 840°F for periods 263C-H and the decrease in space velocity to 690 lb MF coal/hr/ft^3 during periods 263D-H), the first stage and overall coal conversions improved (from 81.3 wt% MAF in 263AB to 89.1 wt% in 263E-H and from 92.7 wt% in 263AB to 94.4 wt% in 263E-H, respectively). As a result of an increase in coal space velocity from 690 (263E-H) to 910 (263I-M) lb MF coal/hr/ft^3, the overall coal conversion decreased from 94.4 to 93.2 wt%.

Due to increases in both coal conversion and resid+UC conversion (Figure 42), the organic rejection decreased from 21.3 wt% in 263A and 17.0 wt% in 263B to 14.1 wt% in 263E-H periods. Figure 42 also shows that the overall resid+UC conversion is a mirror image of resid yield--any increase in resid+UC conversion decreases the resid yield and vice versa.

The increase in resid+UC conversion and the decrease in resid yield during periods 263A-F is accompanied by an increase in C4+ distillate yield from 47.9 wt% in 263A to 61.5 wt% in 263F (Figure 43). During the dispersed catalyst system tests, the C1-C3 gas make increased from 6.5 wt% in 263AB to 9.1 wt% in 263E-H as a result of an increase in the first stage temperature to 840°F and a decrease in space velocity to 690 lb MF coal/hr/ft^3. The gas make decreased to 7.6 wt% during hybrid catalyst system tests (263I-M) primarily due to an increase in space velocity to 910 lb MF coal/hr/ft^3.

The first, second and overall two-stage hydrogen consumptions in Run 263 are shown in Figure 44. During the dispersed catalyst tests, the decrease in coal space velocity from about 780 lb MF coal/hr/ft^3 in 263A-C to 690 lb MF coal/hr/ft^3 in period 263D-H appears to have increased the hydrogen consumption from 4.6 to 5.3 wt%. The presence and the periodic replacement of supported catalyst (at 3 lb/t MF coal) in 263IJ further improved the hydrogen consumption to 5.7 wt% in spite of an increase in coal space velocity to 91C lb MF coal/hr/ft^3. Even though the overall hydrogen consumption changed from 5.3 (263D-H) to 5.7 (263IJ) wt% between the dispersed and hybrid catalyst tests, the individual stages showed a much larger change (first stage: from 4.1 wt% in 263D-H to 2.9 wt% in 263IJ; second stage: from 1.3 wt% in 263D-H to 2.8 wt% in 263IJ).

The yields and conversions from periods 263A-M are plotted and compared with data from Runs 258, 260 and 262 in Figures 45-53. Figure 45 shows a comparison of test conditions from Runs 262 and 263. Hybrid catalyst system was tested in Run 262, and a comparison of test conditions with 263I-M periods show that the resid in process solvent was 37.4 wt% in 262B-E and 39.7 wt% in 263J-M periods. The first stage temperature in Run 262 was 825°F, and in Run 263I-M, it was 840°F. The second stage temperature in Run 263I-M was maintained at 810°F, while it was increased from 760°F in 262AB to 790°F in 262CD to 810°F in 262E period. Besides those shown in Figure 45, there were other differences in operating conditions between Run 262 and Run 263I-M such as catalyst ages, first stage inlet hydrogen partial pressures and dispersed moly concentration.

Process performance from Runs 262 and 263I-M are compared in Figures 46 and 47. The effect of an increase in first stage reaction temperature from 825°F to 840°F can be deduced by comparing 262E and 263J periods. The higher first stage temperature in period 263J gave higher coal conversion (93.6 vs. 91.6 wt%) and resid+UC conversion (82.4 vs. 80.6 wt%) and lower organic rejection (13.4 vs. 19.8 wt%). The potential liquid yield (distillate+resid yields) in 263J was much higher compared to 262E (62.1 vs. 56.8 wt%). The increase in first stage temperature increased the C1-C3 gas make from 6.0 to 7.9 wt%.

In Figures 48-50, the dispersed and hybrid catalyst system tests in Run 263 are compared with the supported catalyst system tests conducted in Run 258. Only data with Black Thunder mine coal (258H-M) are plotted in these figures. Higher first stage temperatures (840-864°F vs. 825-840°F) and lower second stage temperatures (760-790°F vs. 810°F) were used in Run 258H-M compared to Run 263. The resid content in process solvent was similar (about 40 wt%) for both 258H-M and 263J-M test periods). The coal space velocities in 258H-M were much lower compared to that used with the hybrid catalyst system tests in 263I-M (65C-75C vs. 91C lb MF coal/hr/ft^3), but were in the range of dispersed catalyst system tests (69C-79C lb MF coal/hr/ft^3).

Runs 258H-M and 263 are compared in Figures 49 and 50. The hybrid catalyst system gave higher coal and resid+UC conversions and lower organic rejections when compared to the supported catalyst system in spite of operating at 21-40% higher coal space velocity in 263I-M test periods and lower first stage reaction temperature, but at higher second stage temperature. At conditions tested, Figure 50 shows that higher distillate yields and lower organic rejection and C1-C3 gas make can be obtained with both the dispersed and hybrid catalyst systems in Run 263 compared to the supported catalyst system in Run 258H-M.

Test conditions and process performance from Runs 260 and 263 are compared in Figures 51-53. A supported catalyst system was used in Run 260 and tests in thermal/catalytic mode were performed in 260A-C periods and in catalytic/thermal mode in 260D-F. Half volume reactors were used in Run 263; whereas, in Run 260, full volume thermal and three-quarters volume catalytic reactors were used. Except for period 260B (840°F first stage temperature), lower first and second stage reaction temperatures were used in Run 260 (first stage: 825°F in 260AC and 774-790°F in 260D-F vs. 840°F in 263C-M; second stage: 790°F in 260A-C and 774-804°F in 260D-F vs. 810°F in 263A-M). The resid content in the process solvent in Run 260 (40.1 wt% in 260A-C and 38.7 wt% in 260D-F) was lower than the concentrations used with the dispersed catalyst system in 263A-H and was similar to that used with the hybrid catalyst system in 263J-M periods. The coal space velocity in Run 260 was much lower compared to Run 263 (31C-43C in Run 260 vs. 69C-79C in 263A-H and 91C in 263I-M periods).

Figure 52 shows that Run 260A-C with thermal/catalytic mode of operation gave 1-2 wt% higher coal conversion and 3-4 wt% lower organic rejection compared to both the dispersed (263C-H) and hybrid (263I-M) catalyst systems. Figure 53 shows that the distillate yield in 260A-C was 2-6 wt% lower compared to 263E-H and 1-5 wt% lower compared to 263J-M. The C1-C3 gas make was approaching 14 wt% in 260B test period compared to 6-9 wt% in Run 263. In the catalytic/thermal mode of operation in 260D-F, the coal conversion was 5-7 wt% lower and the resid+UC conversions were 2-14 wt% lower and the organic rejection was 4-8 wt% higher compared to 263E-H and 263J-M periods.

4.2 Process Solvent Quality and Hydrogenation

The process solvent quality for Run 263 is given in Table D below. There appears to be not much variation in the process solvent quality between periods 263E-G with the dispersed catalyst system and periods 263I-L with the hybrid catalyst system. Compared to Run 263, the process solvent quality in Run 262 was higher at about 80% during 262A-E periods with the hybrid catalyst system.

Paraffin buildup in the process streams and in process solvent was a concern in Run 262 (Ref. 13). The paraffins (waxy material) appeared in the first stage distillate (V164) and second stage distillate (V1078) when the weather became cooler causing transfer problems in lines that carried these distillates. The vacuum column bottoms (V1074) had highly

Table B. Summary of Operating Conditions

Operating Period	263A	263B	263C	263D
Date, 1991	Nov 10-14	Nov 17,18,20-22	Dec 3,6-9	Dec 13-17
Operation Days	10-14	17,18,20-22	26,29-32	36-40
Coal				
Feed rate, MF lb/hr	299.0	298.8	293.6	259.7
Ash, wt % MF	7.7	7.0	7.6	7.2
Conc. in slurry, wt % MF	24.8	25.0	24.4	24.6
Process solvent, wt %				
Resid	45.2	44.1	43.3	41.1
CI	20.0	20.0	20.2	19.6
First Stage				
Reactor temp., °F	826	825	840	840
Inlet H_2 par. pres., psia	2756	2797	2707	2759
Total gas flow, scfh	4886	5014	4684	5044
Recycle gas, scfh	1534	1449	1196	1528
Space velocity, lb MF coal/hr/ft^3 reac.	79.0C	78.6C	77.6C	68.6C
Iron Oxide, wt % MF coal	2	2	2	2
Moly Conc., ppm MF coal	200	100	100	100
Second Stage				
Reactor temp., °F	810	809	810	810
Inlet H_2 par. pres., psia	2436	2420	2415	2477
Total gas flow, scfh	4765	4872	4757	5094
Recycle gas, scfh	2570	2775	2628	3115
Space velocity, lb MF coal/hr-ft^3 reac.	79.0C	78.6C	77.6C	68.6C
Catalyst type	--	--	--	--
Cat. age, lb(R+CI)/lb cat	--	--	--	--
lb MF coal/lb cat	--	--	--	--
Catalyst replacement rate, lb cat/ton MF coal	--	--	--	--
ROSE-SR^{SM} Unit				
DAS type	2334	2304	2394	2404

Table B. Summary of Operating Conditions (Continued)

Operating Period	263E	263F (transitional)	263G	263H
Date, 1991,1992	Dec 26-28,31,Jan 1	Jan 4-6	Jan 9-13	Jan 16-20
Operation Days	46-48,51,52	55-57	60-64	67-71
Coal				
Feed rate, MF lb/hr	260.9	254.3	263.5	263.9
Ash, wt % MF	7.1	6.9	7.1	6.8
Conc. in slurry, wt % MF	24.5	23.9	24.8	24.7
Process solvent, wt %				
Resid	44.0	43.7	44.6	44.8
CI	20.1	20.2	19.7	20.2
Recycle CI's from	Vac. Fl.	Atm. Fl.	Vac. Fl.	Vac. Fl.
First Stage				
Reactor temp.,°F	840	840	841	840
Inlet H_2 par. pres., psia	2724	2771	2771	2756
Total gas flow, scfh	4614	4769	4858	4575
Recycle gas, scfh	1234	1324	1616	1322
Space velocity, lb MF coal/hr/ft^3 reac.	68.9C	67.2C	69.6C	69.7C
Iron Oxide, wt % MF coal	2	2	2	1
Moly Conc., ppm MF coal	100	100	100	100
Moly Precursor	MV L	MV 822	MV 822	MV 822
Second Stage				
Reactor temp.,°F	809	808	809	806
Inlet H_2 par. pres., psia	2546	2533	2546	2554
Total gas flow, scfh	4850	4829	5010	4785
Recycle gas, scfh	3021	3074	3094	2917
Space velocity, lb MF coal/hr-ft^3 reactor	68.9C	67.2C	69.6C	69.7C
Catalyst type	--	--	--	--
ROSE-SR^{SM} Unit				
DAS type	2414	2364	2424	2424

Notes: MV L - Molyvan L (263A-E, I-M); MV 822 - Molyvan 822 (263 F-H);
Vac. Fl. - vacuum flash bottoms; Atm. Fl. - atmospheric flash bottoms

Table B. Summary of Operating Conditions (Continued)

Operating Period	263I(transitional)	263J	263K	263L	263M
Date, 1992	Jan 23,25,26,27	Jan 29-31,Feb 1,3	Feb 6-9	Feb 11,14-16	Feb 18-22
Operation Days	74,76-78	80-83,85	87-91	93,96-98	100-104
Coal					
Feed rate, MF lb/hr	342.6	344.9	344.1	345.1	348.0
Ash, wt % MF	6.7	6.7	6.5	6.6	6.5
Conc. in slurry, wt % MF	29.4	29.8	29.7	29.6	29.9
Process solvent, wt %					
Resid	44.8	40.5	38.9	39.8	39.6
CI	20.4	20.4	19.9	19.8	19.9
First Stage					
Reactor temp., °F	840	840	842	842	840
Inlet H_2 par. pres., psia	2773	2744	2386	2782	2785
Total gas flow, scfh	4460	4465	4042	4613	4505
Recycle gas, scfh	1162	1212	1773	755	655
Space velocity,					
lb MF coal/hr/ft^3 reac.	90.5C	91.1C	90.9C	91.2C	91.9C
Iron Oxide, wt % MF coal	1	1	1	1	1
Moly Conc., ppm MF coal	100	100	100	100	100
Second Stage					
Reactor temp., °F	810	810	809	810	810
Inlet H_2 par. pres., psia	2482	2542	2515	2537	2549
Total gas flow, scfh	4943	5721	5423	4672	4829
Recycle gas, scfh	2061	2816	2493	2141	2291
Space velocity,					
lb MF coal/hr-ft^3 reac.	90.5C	91.1C	90.9C	91.2C	91.9C
lb MF coal/hr-ft^3 cat.	108.8	109.5	109.4	109.7	110.5
Catalyst type	Crit. 324	Crit. 324	Crit. 324	Crit. 324	Cri. 324
Cat. age, lb(R+CI)/lb cat	1386	1355	1292	1758	1891
lb MF coal/lb cat	668	662	640	889	963
Catalyst replacement rate, lb cat/ton MF coal	3.0	3.0	3.0	-	2.0
ROSE-SR^{SM} Unit					
DAS type	2404	2394	2354	2374	2364

Table C. Overall TSL Yields with Black Thunder Mine Coal

Operating Period	263A	263B	263C	263D
Date, 1991	Nov 10-14	Nov 17,18,20-22	Dec 3,6-9	Dec 13-17
Operation Days	10-14	17,18,20-22	26,29-32	36-40
Yield, wt % MAF coal				
H_2	-4.3±0.3	-4.9±0.5	-4.6±0.6	-5.2±0.2
Water	11.6±1.8	13.4±1.8	13.7±0.7	13.0±0.7
H_2S	0.3±0.2	0.4±0.4	0.2±0.0	0.4±0.2
CO, CO_2	6.2±1.3	6.0±0.7	5.8±0.3	6.3±0.3
NH_3	0.4±0.1	0.3±0.0	0.3±0.2	0.5±0.1
C_1-C_3 gas	6.7±0.5	6.3±0.2	7.8±1.1	9.1±1.2
C_4+ distillate	47.9±4.8	51.8±1.8	52.1±0.6	54.6±4.3
C_4-C_6	2.4±0.3	2.7±0.6	3.0±0.3	3.0±0.3
IBP-350°F	7.9±2.0	8.0±1.2	8.9±0.9	9.1±1.2
350-450°F	8.4±1.5	9.3±1.0	11.3±0.9	10.3±1.3
450°F-EP	29.1±6.7	31.9±1.4	28.8±1.2	32.1±6.5
Resid	9.4±5.3	8.6±2.8	6.6±2.4	5.5±3.3
Int. Acc. (UC)	0.7±1.2	1.0±0.9	0.4±0.3	0.3±1.0
ROSE-SR^{SM} Bottoms Product (ash-free)	21.3±1.5	17.0±0.9	17.5±0.6	15.8±0.7
H_2 Efficiency, (%)	11.1±0.9	10.8±1.2	11.6±1.4	10.4±0.9
C_1-C_3 Selectivity, (%)	14.1±2.3	12.3±0.7	14.9±2.1	16.7±2.5
Coal Conv., wt % MAF				
First stage	80.4±5.9	82.2±3.2	86.1±3.9	87.0±1.4
Second stage	92.7±0.9	93.4±0.3	93.7±0.1	94.5±0.2
Overall TSL	92.8±1.4	92.5±1.4	92.9±0.5	93.1±0.5
Resid+UC Conv., wt % feed				
First stage	17.9±3.0	16.6±1.3	19.9±1.0	20.1±1.2
Second stage	8.4±4.2	13.7±3.4	9.6±1.5	12.7±1.5
Resid+UC Conv., wt % MAF coal				
First stage	48.7±9.7	41.8±5.2	53.4±2.7	51.9±3.3
Second stage	19.9±8.5	31.6±5.1	22.0±3.2	26.6±2.7
Overall	68.7±4.0	73.5±2.2	75.4±1.7	78.5±3.9

Table C. Overall TSL Yields with Black Thunder Mine Coal (Continued)

Operating Period	263E	263F(transitional)	263G	263H
Date, 1991,1992	Dec 26,28,31,Jan 1	Jan 4-6	Jan 9-13	Jan 16-20
Operation Days	46-48,51-52	55-57	60-64	67-71
Yield, wt % MAF coal				
H_2	-5.5±0.2	-5.2±0.2	-5.1±0.2	-5.6±0.3
Water	13.2±1.3	13.6±0.6	14.2±1.0	14.4±0.9
CO, CO_2	5.7±0.2	6.0±0.6	5.9±0.2	4.2±1.1
NH_3	0.3±0.2	0.6±0.2	0.5±0.1	0.4±0.1
H_2S	0.4±0.1	0.5±0.2	0.3±0.1	0.1±0.1
C_1-C_3 gas	9.0±0.3	9.1±0.4	8.9±0.7	9.4±1.0
C_4+ distillate	60.7±2.0	61.5±3.3	57.0±1.1	60.9±2.7
C_4-C_6	3.3±0.5	2.4±0.2	3.0±0.6	3.3±0.3
IBP-350°F	8.0±0.6	9.1±0.5	9.3±1.3	8.4±0.6
350-450°F	10.5±0.7	10.0±0.3	9.7±0.8	10.3±0.8
450°F-EP	39.0±2.4	40.0±2.6	35.0±1.8	38.9±3.1
Resid	2.4±0.9	0.2±3.1	4.1±0.6	2.4±1.8
Int. Acc. (UC)	-0.3±0.1	-0.8±0.7	0.2±0.2	0.0±0.7
ROSE-SR^{SM} Bottoms Product (ash-free)	14.1±0.4	14.5±0.9	14.0±0.7	13.8±0.6
H_2 Efficiency, (%)	11.0±0.4	11.8±0.2	11.2±0.6	10.9±0.3
C_1-C_3 Selectivity, (%)	14.9±0.7	14.8±1.0	15.6±1.2	15.4±2.2
Coal Conv., wt % MAF				
First stage	90.2±1.3	88.7±1.4	89.3±2.8	88.4±2.5
Second stage	94.7±0.4	95.2±0.4	94.9±0.3	94.9±0.4
Overall TSL	94.1±0.8	94.9±0.4	94.1±0.6	94.3±0.4
Resid+UC Conv., wt % feed				
First stage	22.7±0.6	22.8±0.7	22.7±0.8	22.0±0.7
Second stage	9.7±1.0	9.8±2.3	9.9±0.8	10.0±1.5
Resid+UC Conv., wt % MAF coal				
First stage	62.1±2.4	64.2±3.4	60.9±2.0	61.7±1.7
Second stage	21.7±1.8	21.8±3.9	20.8±2.0	22.2±3.2
Overall	83.8±0.7	86.0±2.9	81.7±1.3	83.8±1.6

Table C. Overall TSL Yields with Black Thunder Mine Coal (Continued)

Operating Period	263I(transitional)	263J	263K	263L	263M
Date, 1992	Jan 23,25,26,27	Jan 29-31,Feb 1,3	Feb 6-9	Feb 11,14-16	Feb 18-22
Operation Days	74,76-78	80-83,85	87-91	93,96-98	100-104
Yield, wt % MAF coal					
H_2	-5.6±0.1	-5.8±0.2	-5.7±0.2	-5.5±0.1	-5.2±0.1
Water	15.0±0.9	14.9±0.8	16.5±1.2	14.0±0.6	15.3±0.9
CO, CO_2	6.5±1.0	6.0±1.1	4.8±1.5	7.2±0.6	7.1±0.5
NH_3	0.7±0.1	0.8±0.2	0.7±0.0	0.6±0.0	0.6±0.1
H_2S	0.2±0.0	0.2±0.1	0.3±0.1	0.3±0.0	0.3±0.0
C_1-C_3 gas	7.5±0.2	7.9±0.8	7.4±0.5	7.9±0.3	7.4±0.3
C_4+ distillate	60.9±0.7	58.4±1.3	57.8±1.4	56.1±2.3	54.6±0.4
C_4-C_6	2.7±0.3	3.3±0.5	3.2±0.5	3.6±0.3	2.8±0.2
IBP-350°F	12.4±2.0	11.9±0.8	11.7±1.5	11.5±1.7	10.4±1.3
350-450°F	8.7±0.4	8.3±0.5	8.2±0.2	8.5±1.0	7.8±0.8
450°F-EP	37.2±3.2	35.0±1.5	34.7±2.2	32.6±1.5	33.7±2.1
Resid	1.2±1.1	3.7±1.5	4.3±1.3	3.6±1.5	5.1±0.4
Int. Acc. (UC)	0.0±0.3	0.5±0.4	0.5±0.4	0.0±0.5	0.0±0.2
ROSE-SR^{SM} Bottoms Product (ash-free)	13.7±0.6	13.4±0.3	13.5±0.1	15.9±0.7	14.8±0.6
H_2 Efficiency, (%)	10.9±0.2	10.1±0.5	10.1±0.3	10.1±0.5	10.5±0.1
C_1-C_3 Selectivity, (%)	12.4±0.4	13.6±1.3	12.8±0.9	14.1±0.9	13.5±0.6
Coal Conv., wt % MAF					
First stage	91.4±0.9	89.6±1.1	88.2±1.8	89.7±1.2	90.5±0.9
Second stage	93.7±0.2	94.1±0.3	93.8±0.1	93.5±0.2	93.5±0.1
Overall TSL	93.3±0.7	93.6±0.5	93.7±0.3	92.7±0.5	92.7±0.4
Resid+UC Conv., wt % feed					
First stage	23.0±2.0	23.2±0.3	21.9±1.4	25.1±1.0	25.4±0.6
Second stage	15.3±1.8	17.6±1.1	19.2±2.6	13.8±1.3	14.3±1.2
Resid+UC Conv., wt % MAF coal					
First stage	55.6±5.2	51.8±0.9	47.9±2.8	56.4±2.4	56.0±1.4
Second stage	29.6±4.1	30.6±2.3	33.9±4.5	24.2±3.1	24.1±2.2
Overall	85.2±1.7	82.4±1.7	81.7±1.8	80.6±1.9	80.1±0.9

Table D
Process Solvent Quality

Period	Process Solvent Quality, %
A	68.6
B	70.1
C	73.1
D	69.6
E	74.4
F	75.5
G	71.9
I	75.8
J	70.9
K	73.6
L	75.2

concentrated wax at 8-28 wt %; V164 had 3-9 wt %; and V1078 had 2-9 wt %. In this run (263), no paraffin buildup was noticed. The paraffins in V1074 averaged from 8 to 12 wt % with both dispersed molybdenum catalyst only system (263A-H) and hybrid catalyst system (263I-M). The presence of paraffins in subbituminous coal distillates is not surprising since aliphatic groups have been postulated as bridges between aromatic clusters in this rank of coal. Thermal breakage of aliphatic bonds and consequent catalytic hydrogenation might produce paraffins. It is also possible that paraffins with high carbon chains from C_{16} to C_{32} might be entrapped in the coal matrix and upon thermal treatment released as products.

Figures 54-58 illustrate hydrogen content trend data of the process solvent and interstage stream for Run 263. The data from Run 263 are also compared with that from Run 262.

The hydrogen in the process solvent and interstage stream decreased due to an increase in the first stage reaction temperature to 840°F in period 263C (Figure 54). Compared to period 263B, period 263C averages show that the process solvent hydrogen decreased from 7.1 to 6.5 wt % and interstage stream hydrogen decreased from 6.8 to 6.4 wt %. Figure 57 indicates a decrease in both the process solvent and interstage stream resid hydrogen values due to an increase in the first stage temperature to 840°F in 263C.

Figures 54 and 58 show that the presence of a supported catalyst in periods 263I-M and 262A-E resulted in higher hydrogen values in the process solvent (V131B) and interstage stream sample (R1235 OTL) when compared to the dispersed molybdenum catalyst only system values obtained in periods 263A-H. Hydrogen values from different periods are summarized below:

Period	Hydrogen (wt %) V131B	 R1235 OTL
262D	7.58	7.36
262E	7.09	6.80
263C	6.48	6.39
263D	6.49	6.50
263E	6.37	6.38
263F	6.19	6.22
263G	6.22	6.29
263H	6.39	6.43
263J	6.76	6.61 (6.47/6.75)
263K	6.79	6.50
263L	6.82	6.70
263M	6.72	6.64

The higher resid recycle in period 263E (at the expense of the V1074 heavy vacuum gas oil) decreased both the V131B process solvent and interstage stream sample (R1235 OTL) hydrogen values compared to period 263D. The V1067 atmospheric flash bottoms recycle and the addition of Molyvan 822 in period 263F further decreased the hydrogen values in V131B and R1235 OTL streams. Comparing period 263G (with Molyvan 822 precursor) with 263E (with Molyvan L precursor), it appeared that the Molyvan 822 decreased the hydrogen values in both V131B and R1235 OTL streams.

The decrease in the iron-oxide addition rate from 2 to 1 wt % MF coal in period 263H appeared to increase the V131B hydrogen value from 6.22 wt % in 263G to 6.39 wt % in 263H and the R1235 OTL hydrogen value from 6.29 to 6.43 wt %.

The hydrogen values in both V131B and R1235 OTL in the hybrid catalyst cases (263J-M) were much higher than with those from the dispersed molybdenum catalyst only test periods (263C-H). The hydrogen values gradually increased from 263H values as the hybrid catalyst system was continued to be tested. The R1235 OTL average hydrogen value in the first part of 263J was 6.47 wt % while near the end of 263J it was 6.75 wt %.

The decrease in the inlet hydrogen partial pressure in the first stage by about 400 psia in period 263K decreased the R1235 OTL hydrogen value from 6.75 wt % (in the latter part of 263J) to 6.50 wt % in 263K. The R1235 OTL hydrogen value increased back to 6.70 wt % in 263L in which the first stage inlet hydrogen partial pressure was increased back by 400 psia. The process solvent hydrogen value was not affected by the decrease in the hydrogen partial pressure in the first stage.

5. CATALYST

Criterion 324, 1/16" unimodal, supported catalyst, aged in Run 262, was charged to the second stage reactor (R1236) on January 21-22. The aged catalyst used in this run came from the catalyst accumulated through regular catalyst replacement in Run 262. With the Criterion catalyst in the second stage reactor, tests in the second part of Run 263 (periods 263I-M) featured a hybrid catalyst system with a combination of supported and dispersed catalysts.

The supported catalyst was already at an equilibrium age equivalent to 3 lb/ton MF coal catalyst replacement from operations in Run 262. The initial catalyst ages were 668 lb MF coal/lb catalyst and 1386 lb (resid+CI)/lb catalyst. No presulfiding of fresh catalyst was necessary since there was enough in inventory from previous runs (Runs 260 and 261). Table 13 compares the properties of Criterion 324 catalyst with other catalysts that were tested before at Wilsonville.

The second stage reactor (R1236) was charged with 170 lbs of Criterion catalyst on January 21-22, 1992. Catalyst replacement continued every other day until February 9 to keep the catalyst at an age equivalent to 3 lb /ton MF coal replacement. Batch deactivation was performed without any catalyst replacement between February 9-16. Catalyst replacement at 2 lb/ton MF coal rate was started on February 17 (od=98) and continued until end of the run. The equilibrium catalyst ages increased to 963 lb MF coal/lb catalyst and 1891 lb (resid+CI)/lb catalyst due to a lower replacement rate.

The second stage catalyst ages are shown in Figure 59. Since the Criterion (Shell) 324 catalyst replacement was first started on January 23, the catalyst in the initial two to three days (January 21-23) was batch deactivated resulting in slightly higher catalyst ages. With the start of catalyst replacement, the catalyst ages decreased until they reached equilibrium ages at 3 lb/ton MF coal replacement rate (periods 263I-K). The R+CI catalyst age showed a slight downward trend through the periods 263I-K. The second stage catalyst was batch deactivated during period 263L and data for period 263M was taken at steady-state operation with 2 lb/ton MF coal catalyst replacement rate. The average catalyst ages at the end of the run was 959 lb MF coal/lb catalyst and 1878 lb (resid+CI)/lb catalyst.

Throughout the run, a dispersed molybdenum catalyst was used in the form of Molyvan L or Molyvan 822. A description of Molyvan L or 822 is given in Section 5.1.4, Dispersed Molybdenum Catalyst. Also, iron-oxide slurry catalyst was added to the system at 1-2 wt % MF coal using dimethyl disulfide (DMDS) as the sulfiding agent through the entire run.

5.1 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. It is measured in units of millimoles of hydrogen consumed per 100 g of liquid sample (10% naphthalene solution). 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. Details of the test are given in Appendix C.

When the reactors were opened at the end of the run, the Criterion 324 catalyst recovery was assessed for R1236. The recovery was only 69%. On inspection, severe wear was seen on the plenum chamber of the second stage reactor. There were holes on top of the plenum chamber and in the device for distributing the gas/slurry mixture into the reactor. The catalyst is probably crushed initially by the impingement of the feed material flowing from the top surface of the plenum chamber since the top surface had eroded grooves and holes. The catalyst attrition may have aggravated due to the shortcircuiting of the gas and slurry flow through the holes.

5.1.1 Catalyst Size and Recovery

In previous runs, Runs 258 and 260, at Wilsonville with Criterion 324 1/16" catalyst, no problems were observed with breakage or fracturing of the catalyst during the run. Also, during Runs 262 and 263, no signs of breakage or fracturing occurred during the run as seen from the analysis results (Table 14) of the catalyst withdrawn during catalyst addition/withdrawal procedure. However, the end-of-run sample screen analysis showed 81.5% and 76.7% +14 mesh size, respectively, for Runs 262 and 263. Other end-of-run Criterion 324 catalyst samples have not had as much breakage. For example, the Shell 324 (same as Criterion) catalyst end-of-run sample from Run 260 withdrawn from the first stage had a 91.9% +14 mesh screen analysis. For Run 258, the second stage end-of-run sample contained 86.7% +14 mesh with Shell 324 catalyst.

The screen analysis of the end-of-run sample showed that non-supported catalytic fines (mainly agglomerated iron compounds) were not present in any significant amount as opposed to 15.4 wt % of the catalyst charge in Run 262. Table 14 summarizes the analytical results of the catalyst samples withdrawn during the run and the end-of-run samples.

5.1.2 Catalyst Elemental Analysis and Naphthalene Activity

Figure 60 gives the carbon and sulfur on the THF extracted catalyst. The carbon on the Criterion catalyst was in the range 12-14 wt % during periods 263I-M when the hybrid catalyst system was tested. Little variation in the carbon content of the catalyst was seen in periods 263J-L. The sulfur on the catalyst also did not significantly vary through the run and was present in 6.5-7.5 wt % range. These stable trends were probably due to the similar operating conditions at 840/810°F and 350 lb MF coal/hr that were used during the hybrid catalyst test periods. The carbon was slightly lower and the sulfur was slightly higher on the end-of-run samples compared to catalyst samples withdrawn during the run.

Figure 61 shows both the hydrogen and nitrogen on the THF extracted catalyst. Both the hydrogen and nitrogen on the Criterion catalyst did not show any significant variation. The hydrogen was in the range 0.8-0.9 wt % and the nitrogen was close to 0.2 wt% through periods 263I-M.

Figure 62 shows the carbon to hydrogen atomic ratio (C/H) for the THF extracted Criterion catalyst. The C/H ratio increased rapidly from 1.1 to about 1.4 in the beginning of the hybrid catalyst system tests. This increase was the result of both an increase in carbon content and a decrease in hydrogen content during period 263I. The C/H ratio decreased to about 1.3 during 263LM periods mainly due to a slight increase in hydrogen on the catalyst. The C/H ratios in periods 263J-M are higher than those observed in previous runs (1.0-1.2). The end-of-run catalyst sample, however, had a C/H ratio of 1.1 which was in the range of values observed in previous runs.

The naphthalene activity of the Criterion catalyst is shown in Figure 63. The naphthalene activity in 263JK periods was about 25 which increased to about 30-40 range in 263M period. The end-of-run catalyst sample had a naphthalene activity of about 65. The trend in naphthalene activity shows that it is inversely proportional to carbon deposit on the catalyst. The higher naphthalene activity of catalyst in 263M was somewhat unexpected since the catalyst in 263M period was at a higher catalyst age compared to 263JK periods. A possible explanation for the observed higher activity is the lower catalyst inventory in the reactor due to attrition (probably caused by holes in the plenum chamber), as indicated with the low catalyst recovery (69%) at the end of the run. The attrition probably causes the higher age catalyst to disintegrate and wash away from the reactor resulting with an inventory of lower catalyst age and higher activity.

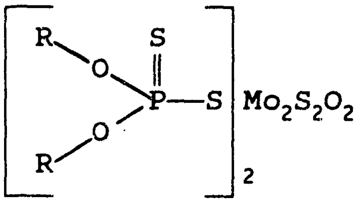
Table 15 gives the metal analysis of the withdrawn catalyst samples from the entire run. Within some variation, there was no apparent nickel loss and no buildup of any metal occurred throughout the run.

5.1.3 Comparison of Catalyst Analysis to Previous Runs

The carbon deposit on the catalyst for Run 263 (12-14 wt %) was much higher than the values obtained in Run 262 (about 9 wt %). Run 263 catalyst carbon were similar to that obtained in Run 260A-C (11-15 wt %). Reaction temperature in the catalytic reactor and space velocity influenced the deposits on the catalyst. The lower carbon deposit on the catalyst in Run 262 was likely due to a lower temperature operation at 760°F (262AB), 790°F (262CD) and 810°F (262EF) in the second stage. The second stage temperature in Run 263 was maintained at 810°F throughout the run. Even though the second stage temperature in Run 260A-C (790°F) was lower than Run 263 (810°F), the carbon on the catalyst samples were in similar range (11-15 wt %). The space velocity in Run 260A-C was much lower than in Run 263I-M (45-55 lb MF coal/hr/cuft-catalyst in 260A-C vs. 110 lb MF coal/hr/cuft-catalyst in 263I-M).

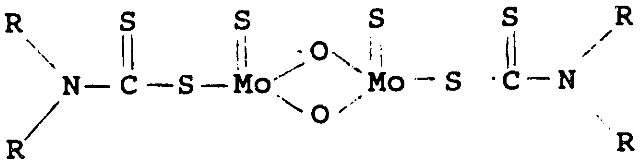
5.1.4 Dispersed Molybdenum Catalyst

To disperse molybdenum into the process stream before entering the TSL system, Molyvan L and Molyvan 822, sold as friction reducers and antiwear agents, were injected before the B1200 preheater along with DMDS. Molyvan L and Molyvan 822 are stable organic liquid additives containing molybdenum, sulfur, and phosphorous or nitrogen. They are manufactured by R.T. Vanderbilt Company, Inc. of Norwalk, Connecticut. Molyvan L contains 8.1 wt % molybdenum, 12.3 wt % sulfur, 6.4 wt % phosphorous and no nitrogen; Molyvan 822 contains 4.9 wt % molybdenum, 6.0 wt % sulfur, no phosphorous and some nitrogen. Both the molybdenum precursors are soluble in petroleum oils and greases and aliphatic and aromatic solvents. At room temperature, they are in a liquid state with a flash point of 330-350°F. The structure of Molyvan L is a sulfurized oxymolybdenum organophosphorodithioate shown below:



R is an alkyl group

Molyvan 822 structure is shown below:



R is an alkyl group

Recent studies (personal communication from R. P. Padamsey, Alberta Oil Sands Technology & Research Authority, Calgary, Alberta, Canada) suggest that the alkyl group on the organometallic catalyst plays an important role in decomposition of the precursor and in generation and dispersion of active species in solution. According to the manufacturer, the alkyl group on Molyvan L and Molyvan 822 can be changed to suit the process. It may be worthwhile to carryout bench-scale liquefaction tests to evaluate the effect of alkyl groups on the effectiveness of dispersed catalysts.

Molyvan L contains 8.1 wt % molybdenum and costs $4.00/lb, while Molyvan 822 contains 4.9 wt % molybdenum and costs $2.81/lb. In comparison, the Criterion 324 supported catalyst contains 13.2 wt % molybdenum and 2.7 wt % Ni and costs $2.75/lb. At Wilsonville, steady-state operations were typically maintained by adding the required amount of supported catalyst periodically once in 48 hours. Catalyst costs for dispersed and supported catalyst systems can be compared at 300 lb MF coal/hr feed rate and assuming 100 ppm MF dispersed catalyst addition rate and 3 lb/ton coal supported catalyst replacement rate. Over a 48 hour period, the cost of Molyvan L introduced into the system is $71.11, while the cost of supported catalyst replaced is $59.40.

5.2 Run 263 Supported Catalyst Activity and Thermal Conversion Activity with Dispersed Molybdenum Catalyst

With the addition of dispersed molybdenum catalysts (Molyvan L and 822), Criterion (Shell) 324 1/16" supported catalyst activity in the second stage and thermal conversion activities in both the first and second stages were evaluated and analyzed, based on Phase 2 and 3 resid+UC conversion trend data. The first stage conversion, the second stage conversion, and the overall two-stage conversion were compared for TSL performance evaluation. Black Thunder subbituminous coal was processed in Run 263 (between November 2, 1991 - February 22, operation days, od = 2-103). The slurry blend system involved V131B pasting solvent tank, V101A slurry blend tank and V101B slurry feed tank. The interstage separator was used in the process, and the hot, heavier V1074 was used for the withdrawal tube flush in both reactors until December 16 and switched to V178 (same as V182) solvent from T102 vacuum column overheads to reduce pumping problems. The V1067 atmospheric bottoms were recycled to meet the CI requirements in the process solvent during January 2-6 (od=53-57) in place of V1082 vacuum bottoms in order to evaluate its effect on the boiling end point of the distillate product.

Run 263 began on October 31. The thermal/thermal mode of operation with half-volume reactors in both stages without a supported catalyst (Criterion 324) was employed until January 20 (od=71), and thereafter, the plant operation was switched to the thermal/catalytic mode with Criterion 324 supported catalyst in the second stage. Catalyst replacement in the second stage was 3 lb/ton MF coal between January 23 - February 8 (od=74-90) and 2 lb/ton MF coal between February 17-22 (od=98-103). Batch deactivation mode was used between February 9-16 (od=91-97) without catalyst replacement in the second stage to obtain a higher catalyst age.

Reaction temperatures studied in this run were 825/840°F in the first stage and 810°F in the second stage. Coal feed rate was 265/300/350 MF lb/hr, coal concentration in the feed slurry was 25/30 wt %, and targeted recycle resid and CI concentrations in the recycle process solvent were 40/42/45 and 20 wt %, respectively. Molyvan L and Molyvan 822 addition rates were at 100/200 ppm molybdenum (based on MF coal). Iron-oxide addition was 1-2 wt % MF coal and DMDS was used as a sulfiding agent.

On February 4 (od=86), the hydrogen partial pressure in the first stage was reduced by 400 psia by increasing the amount of gas recycled back to the first stage and decreasing the make-up hydrogen flowrate. This lower hydrogen partial pressure seemed to decrease the total exotherm in the first stage due to less exothermic heat generated in the reactor, indicating less hydrogenation and cracking. The hydrogen partial pressure was increased back to the normal operating value on February 10 (od=92).

Since December 22 (od=42), pumping problems continued with the P1222 ebullation pump which was used as a backmixing pump in the R1235 first stage reactor. The bottom thermocouple showed much lower reading than normal, indicating a possible solids buildup inside the reactor.

Results are illustrated in Figures 64 to 69. 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, Process Solvent Quality and Hydrogenation.

5.2.1 Overall Two-Stage Conversion Analysis

Overall two-stage conversion activities were calculated for periods 263A-M, and are illustrated in Figures 64 and 65. The TSL resid yield data are listed below for these periods along with the Phase 3 data. Two different common organic rejections (COR), 15 or 20 wt % MAF coal, were selected for trend analysis. Standard deviation of the resid yield was 1-9 wt % MAF coal. Phase 2 data variation was very high (7-9 wt %) for periods C, D, F, H and I, and Phase 3 data variation was much less than Phase 2 data (2-4 wt %).

	Resid with COR = 20/15 wt % wt % MAF coal	
Period	Phase 2	Phase 3
263A (COR=15)	16.3 ± 4.7	16.3 ± 4.0
(COR=20)	11.3 ± 4.7	11.3 ± 4.0
263B (COR=15)	10.3 ± 3.0	11.2 ± 2.3
(COR=20)	5.3 ± 3.0	6.2 ± 2.3
263C (COR=15)	4.9 ± 7.1	9.6 ± 1.7
(COR=20)	-0.1 ± 7.1	4.6 ± 1.7
263D (COR=15)	1.4 ± 8.9	6.5 ± 3.9
(COR=20)	-3.6 ± 8.9	1.5 ± 3.9
263E (COR=15)	-2.3 ± 4.5	1.2 ± 0.7
(COR=20)	-7.3 ± 4.5	-3.8 ± 0.7
263F (COR=15)	-0.7 ± 8.4	-1.1 ± 2.9
(COR=20)	-5.7 ± 8.4	-6.1 ± 2.9
263G (COR=15)	0.5 ± 3.6	3.3 ± 1.3
(COR=20)	-4.5 ± 3.6	-1.7 ± 1.3
263H (COR=15)	-3.5 ± 7.5	1.2 ± 1.6
(COR=20)	-8.5 ± 7.5	-3.8 ± 1.6
263I (COR=15)	-6.0 ± 7.8	-0.2 ± 1.7
(COR=20)	-11.0 ± 7.8	-5.2 ± 1.7
263J (COR=15)	-1.7 ± 2.3	2.5 ± 1.7
(COR=20)	-6.7 ± 2.3	-2.5 ± 1.7
263K (COR=15)	3.4 ± 0.7	3.3 ± 1.8
(COR=20)	-1.7 ± 0.7	-1.8 ± 1.8
263L (COR=15)	4.1 ± 1.7	4.4 ± 1.9
(COR=20)	-0.9 ± 1.7	-0.6 ± 1.9
263M (COR=15)	7.9 ± 5.0	4.9 ± 0.9
(COR=20)	2.9 ± 5.0	-0.1 ± 0.9

Key process variable changes are summarized below.

Period	Process condition
263A	startup w/ subbituminous resid; thermal/thermal mode; V1074 hot, heavier solvent for withdrawal tube flush in reactors; Molyvan L addition = 200 ppm Molybdenum Fe_2O_3 (+ DMDS) addition = 2 wt % MF; $T_{1st}/_{2nd}$ = 825/810°F, 300 lb MF coal/hr; coal conc. in slurry = 24.8 wt %; process solvent composition (wt %) resid = 45.3 CI = 20.0
263B	Molyvan L addition = 100 ppm molybdenum
263C	T_{1st} = 840°F
263D	265 lb MF coal/hr; 42 wt % resid in process solvent
263E	45 wt % resid in process solvent; V178 solvent for withdrawal tube flush
263F	Molyvan 822 addition = 100 ppm moly; V1067 atmospheric bottoms recycle
263G	V1082 vacuum bottoms recycle
263H	Fe_2O_3 (+ DMDS) addition = 1 wt % MF
263I	Criterion 324 catalyst in 2nd stage; catalyst replacement = 3 lb/ton MF coal; 350 lb MF coal/hr; 45 wt % resid in process solvent; Molyvan L addition = 100 ppm molybdenum
263J	40 wt % resid in process solvent
263K	H_2 partial pressure reduced by 350-400 psia in 1st stage
263L	hydrogen pressure back to normal; batch deactivation in 2nd stage
263M	catalyst replacement = 2 lb/ton MF coal

Resid+UC conversion data for these periods are compared below. Standard deviation of the overall two-stage conversion was 1-9 wt % MAF coal. Individual stage conversions varied more than the overall two-stage conversion (1-14 wt %).

Period		Resid + UC conversion wt % MAF coal	
		Phase 2	Phase 3
263A	1st stage	47.6 ± 9.2	48.7 ± 9.7
	2nd stage	21.1 ± 7.9	19.9 ± 8.5
	Overall	68.7 ± 4.7	68.7 ± 4.0
263B	1st stage	47.9 ± 6.5	41.8 ± 5.2
	2nd stage	26.4 ± 6.7	31.6 ± 5.1
	Overall	74.4 ± 3.1	73.5 ± 2.2
263C	1st stage	54.6 ± 5.2	53.4 ± 2.7
	2nd stage	25.5 ± 7.6	22.0 ± 3.2
	Overall	80.1 ± 7.1	75.4 ± 1.7
263D	1st stage	55.2 ± 3.5	51.9 ± 3.3
	2nd stage	28.4 ± 8.1	26.6 ± 2.7
	Overall	83.6 ± 8.9	78.5 ± 3.9
263E	1st stage	61.6 ± 6.0	62.1 ± 2.4
	2nd stage	25.5 ± 4.2	21.7 ± 1.8
	Overall	87.1 ± 4.5	83.8 ± 0.7
263F	1st stage	54.3 ± 9.6	64.2 ± 3.4
	2nd stage	31.5 ± 11.6	21.8 ± 3.9
	Overall	85.7 ± 8.4	86.0 ± 2.9
263G	1st stage	54.5 ± 6.6	60.9 ± 2.0
	2nd stage	30.0 ± 9.0	20.8 ± 2.0
	Overall	84.5 ± 3.6	81.7 ± 1.2
263H	1st stage	58.4 ± 7.8	61.7 ± 1.7
	2nd stage	30.1 ± 13.7	22.1 ± 3.2
	Overall	88.5 ± 7.5	83.8 ± 1.6
263I	1st stage	54.7 ± 3.3	55.6 ± 5.2
	2nd stage	36.3 ± 5.1	29.6 ± 4.1
	Overall	91.0 ± 7.8	85.2 ± 1.7
263J	1st stage	50.4 ± 7.2	51.8 ± 0.9
	2nd stage	36.3 ± 9.2	30.6 ± 2.4
	Overall	86.7 ± 2.3	82.4 ± 1.7
263K	1st stage	50.2 ± 5.2	47.9 ± 2.8
	2nd stage	31.5 ± 4.8	33.9 ± 4.5
	Overall	81.7 ± 0.7	81.8 ± 1.8
263L	1st stage	58.5 ± 2.4	56.4 ± 2.4
	2nd stage	22.4 ± 3.9	24.2 ± 3.1
	Overall	80.9 ± 1.7	80.6 ± 1.9
263M	1st stage	53.3 ± 5.7	56.0 ± 1.4
	2nd stage	23.6 ± 4.3	24.1 ± 2.2
	Overall	76.9 ± 4.8	80.1 ± 0.9

In period 263A, the "all-distillate" product slate was not achieved with 10 wt % MAF coal resid yield. The performance data were similar to those obtained for Run 262F with 350 MF lb/hr coal feed rate, 30 wt % coal concentration in the feed slurry and 41 wt % resid concentration in the process solvent.

In period 263B, at a lower molybdenum addition rate (100 ppm), a higher C4+ distillate yield was observed (52 vs. 48 wt % MAF coal) compared to period 263A with a 200 ppm molybdenum addition rate. This indicates that operation at the lower molybdenum addition rate of 100 ppm did not lower the resid+UC conversion performance. Coal conversion was similar for both periods (93 wt % with Phase 3 data and 89 wt % with Phase 2 data), even though 263B had lower organic rejection than 263A (17 vs. 21 wt %). Resid+UC conversion was 5-6 wt % higher in 263B compared to 263A.

In period 263C, the increase in the first stage reaction temperature to 840°F, initially (between November 28 - December 6), significantly increased the resid conversion in the first stage, which was the primary contributor to the overall two-stage conversion increase by 11-20 wt % MAF coal. However, the later data in period 263C (between December 7-9) showed no difference compared to 263B, which was operated at a lower first stage reaction temperature of 825°F.

The high variation in conversion data in 263C made it difficult to arrive at a conclusive temperature effect. The effect was 2-5 wt % MAF coal resid+UC conversion increase based on the selected material balance days (December 3, 6-9), and was higher at 8 wt % based on the average value for the entire test period (November 28 - December 9). As already mentioned, the initial response was much higher, 11-20 wt %.

It appears that the accidental system depressuring and a plant outage caused by leaks developed in C1206 recycle gas compressor during November 30 - December 6 might have affected the performance in 263C. In addition, the variation of resid concentration in the recycle process solvent might have caused unstable operation. Daily V131B (process solvent) grab sample analytical data showed that the resid concentration varied from 41 to 50 wt %. Phase 3 data showed a similar C4+ distillate yield (52 wt %) for both 263B and C periods, and the resid+UC conversion (73.4-75.5 wt % MAF coal) and distillate selectivity to conversion (69-71%) were similar for these two periods.

In period 263D, at a lower coal feed rate of 265 MF lb/hr and a lower level of resid recycle at 42 wt %, C4+ distillate yield increased to 54.6 wt % MAF coal with 5.8 wt % excess resid yield. Phase 2 data showed a lower resid yield of 1.4 wt % due to higher resid+UC conversion, indicating a "all-distillate" product slate. Distillate selectivity to conversion was 70%, similar to those for periods 263A-C. Period 263D data suggest that higher distillate production is possible at 840°F first stage reaction temperature and 265 MF lb/hr coal feed rate without extensively producing gases. High data variation was again experienced in this period.

In periods 263E and F (transitional), the higher first stage reaction temperature, 840°F, and lower coal space velocity with 265 MF lb/hr coal feed rate did not significantly affect distillate selectivity. The distillate selectivity was 71-72% without excessive gas make (selectivity was 71% at 825°F and 300 MF lb/hr in periods 263A and B). The "all-distillate" product slate was achieved in periods 263E-F with 61-62 wt % MAF coal C_4+ distillate. In period 263F (transitional), Molyvan 822 slurry catalyst addition and atmospheric bottoms recycle did not significantly affect the performance, compared to 263E with Molyvan L slurry catalyst addition and vacuum bottoms recycle. The overall TSL resid+UC conversion was similar for both periods (84-87 wt %). On January 27 (od=78), as Molyvan 822 supply was exhausted, the molybdenum precursor was changed back to Molyvan L. Period 263E with a higher recycle resid level of 44 wt % showed a 3-5 wt % higher conversion than that for period 263D with 41 wt % recycle resid.

In period 263G with Molyvan 822 addition, the overall TSL resid+UC conversion was 2-3 wt % MAF coal lower than that for 263E with Molyvan L addition, indicating there is little difference in the conversion performance with both precursors. The "all-distillate" product slate was achieved in 263G with 57 wt % MAF coal C_4+ distillate. The excess resid yield was 4 wt %. Compared to 263F (transitional) with V1067 atmospheric bottoms used for solids recycle, period 263G, with V1082 vacuum bottoms recycle, showed 1-4 wt % lower resid+UC conversion. This difference in the conversion performance may not be significant considering the data scatter.

In period 263H, with a lower iron-oxide addition rate of 1 wt % MF coal compared to 263G with 2 wt % addition rate, the overall TSL resid+UC conversion increased by 2-4 wt %, indicating that lowering iron-oxide concentration did not reduce the conversion. The conversion for 263H was similar to that for 263E with 2 wt % iron-oxide. Coal conversion was similar for these periods (93-94 wt %). The "all-distillate" product slate was achieved in 263H with 61 wt % MAF coal C_4+ distillate. The excess resid yield was 2 wt %.

In periods 263I (transitional) and J, with Criterion 324 supported catalyst charged in the second stage, even at a higher coal feed rate of 350 MF lb/hr, the "all-distillate" product slates were achieved with 58-61 wt % MAF coal C_4+ distillate. The excess resid yields were 1-4 wt %. The conversion performance might have been affected in 263J at a lower recycle resid level of 41 wt % compared to 45 wt % in 263I (transitional). The overall TSL resid+UC conversion was 3-4 wt % lower than in 263J. Coal conversion for both periods were high at 94-95 wt %, which seemed not to have affected by the lower severity operation with Criterion catalyst in the second stage and the higher coal feed rate.

In period 263K, with a lower inlet hydrogen partial pressure (by 350-400 psia) in the first stage compared to 263J, the "all-distillate" product slate was not achieved with 58 wt % MAF coal C_4+ distillate. The excess resid yield was 5 wt %. The overall TSL resid+UC conversion was 1-5 wt % lower in 263K (lower by 1 wt % with Phase 3 data and 5 wt % with Phase 2 data). Coal conversion for 263K was lower with Phase 2 data (91 wt %) and similar with Phase 3 data (94 wt %). Distillate selectivity stayed the same at 71%. The first stage resid+UC conversion did not decline based on Phase 2 trend data; declined by 4 wt % in 263K based on Phase 3 trend data. The second stage resid+UC conversion was not consistent between Phase 2 and Phase 3 trend data.

Period 263L was transitional in a batch deactivation mode without catalyst replacement. The first stage hydrogen partial pressure was increased back by 400 psia to normal operating condition. The "all-distillate" product slate was achieved with 56 wt % MAF coal C_4+ distillate. The excess resid yield was 4 wt %.

In period 263M, with a lower catalyst replacement rate of 2 lb/ton MF coal, the overall resid+UC conversion declined by 10 wt % MAF coal using Phase 2 trend data compared to 263J with 3 lb/ton MF coal replacement, while Phase 3 data shows that the conversion decreased by only 2 wt %, a much lower response than what was observed with the Phase 2 data. The "all-distillate" product slate was not achieved with 55 wt % MAF coal C_4+ distillate yield. The excess resid yield was 5 wt %. The second stage resid+UC conversion declined in 263M by 13 wt % and the first stage slightly increased by 3 wt %, based on Phase 2 trend data. Phase 3 trend data showed 7 wt % decrease in the second stage and 4 wt % increase in the first stage.

The 1st & 2nd stage coal conversion for 263LM was lower by 1-3 wt % (Phase 2 data) than 263IJ with Criterion catalyst and 263C-H without the Criterion catalyst. This lower coal conversion might have been affected by the lower severity operation with Criterion catalyst in the second stage and higher coal feed rate (350 MF lb/hr), and lower iron-oxide concentration (1 wt %). However, the first stage coal conversion for periods 263I, 263J, 263L and 263M increased to 89-92 wt % compared to 263EG. The stage interaction effect with the supported catalyst in the second stage seemed to improve the first stage coal conversion. This probably is due to better solvent quality with recycle solvent by hydrogenating more in the second stage with an active supported catalyst.

Trend analyses are summarized in Tables 16 to 18. In these tables, the resid+UC conversion, TSL excess resid yield, coal conversion and organic rejection data are compared.

In period 262E, at a lower first stage reaction temperature of 825°F, the process performance was not so good as in period 263J at 840°F first stage reaction temperature, as shown in the following. The first stage reaction temperature increase improved the performance by increasing the resid and coal conversions at higher first stage thermal severity without lowering the distillate selectivity. The higher iron-oxide concentration in 262E might have affected the performance (2 vs 1 wt % MF). Phase 2 data showed much higher improvement than Phase 3 data.

run		262E	263J	
yield (wt % MAF)				
C4+ distillate		57.2	58.4	
resid		-0.5	4.2	
organic rejection		19.8	13.4	
conversion (wt % MAF)				
coal	(Ph 2)	92.0	95.2	(+3.2)
	(Ph 3)	92.3	94.1	(+0.8)
resid + UC	(Ph 2)	77.1	86.9	(+9.8)
	(Ph 3)	80.6	82.4	(+1.8)

5.2.2 First Stage Thermal Conversion Activity with Dispersed Molybdenum Catalyst

With the addition of dispersed molybdenum catalysts (Molyvan L and Molyvan 822), the thermal resid+UC conversion activities were calculated, assuming that the resid+UC conversion reaction follows the 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 (SV) and resid+UC conversion (ϵ).

$$K/C = (SV/C)\ \epsilon/(1-\epsilon),$$

where C: constant equating thermal reaction volume to catalytic reaction volume in the thermal/catalytic mode.

The dependence of the conversion rate constant on temperature (T) is described by the Arrhenius equation.

$$K/C = (A/C)\ e^{-E/RT},$$

where K: rate constant, [hr^{-1}]

SV: feed space velocity,
[lb feed/hr/cu ft-reactor]
ϵ: resid + UC conversion, [wt % feed]
A: frequency factor
E: apparent activation energy, [Btu/lb-mole]
R: ideal gas constant, [Btu/lb-mole-°R]
T: temperature, [°R]

The linear regression analysis equation becomes:

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

First stage thermal conversion activity with dispersed molybdenum catalysts was analyzed using on Phase 2 resid+UC conversion data. Results are illustrated in Figures 70 to 73.

The average ln K/C values for periods 263A-M were calculated and are listed below. Reactor volume was used in calculation of the conversion rate constant (K).

	Average ln K/C (1/hr)	
Period	Phase 2	Phase 3
263A	4.22 ± 0.21	4.27 ± 0.21
263B	4.26 ± 0.16	4.17 ± 0.09
263C	4.41 ± 0.11	4.39 ± 0.06
263D	4.35 ± 0.06	4.29 ± 0.06
263E	4.50 ± 0.08	4.45 ± 0.03
263F	4.28 ± 0.21	4.46 ± 0.03
263G	4.31 ± 0.16	4.46 ± 0.05
263H	4.38 ± 0.16	4.42 ± 0.04
263I	4.61 ± 0.09	4.62 ± 0.06
263J	4.50 ± 0.18	4.56 ± 0.01
263K	4.53 ± 0.14	4.49 ± 0.09
263L	4.72 ± 0.05	4.67 ± 0.05
263M	4.61 ± 0.14	4.68 ± 0.03

The average ln K/C values for periods 263A and B were lower than those obtained for 262A-F (4.22-4.26 vs. 4.32-4.49 hr^{-1}). This suggests that the presence of a supported catalyst (Criterion, etc.) in the second stage might improve the first stage thermal conversion, due to improved hydrogenation of the process solvent by the supported catalyst.

In period 263B, at a lower molybdenum addition rate (100 ppm), the average ln K/C value was similar to that for 263A (4.2-4.3 hr^{-1}). This suggests that operations with the lower molybdenum addition rate of 100 ppm did not lower the conversion performance. The average ln K/C value using all data between November 10-22 at 825°F is 4.22±0.17 hr^{-1} (K/C = 69.2±11.9 hr^{-1}, ϵ = 17.2±2.5 wt %).

The average ln K/C values during periods 263C-H at 840°F first stage reaction temperature varied in the range of 4.28-4.50 hr^{-1}. Changes in coal feed rate (265-300 MF lb/hr), Molyvan L or 822 addition, iron-oxide concentration (1-2 wt % MF coal), and resid concentration (40-45 wt %) seemed not to significantly affect the average value. The average ln K/C value using all the data for periods 263C-H at 840°F was 4.36±0.16 hr^{-1} (K/C = 79.5±12.5 hr^{-1}, ϵ = 20.8±2.5 wt %); using all the data for periods 263I-M, it was 4.55±0.16 hr^{-1} (K/C = 96.1±14.8 hr^{-1}, ϵ = 23.0±2.8 wt %); using all the data for periods 263C-M, it was 4.44±0.19 hr^{-1} (K/C = 86.0±15.6 hr^{-1}, ϵ = 21.7±2.8 wt %).

The high first stage average value for 263E (4.50 vs. 4.28-4.41 hr^{-1}) might have been due to the low second stage average value (3.43 vs. 3.70-3.83 hr^{-1}), indicating possible problems with the interstage sample.

The presence of Criterion 324 supported catalyst in the second stage seemed to improve the average rate constant values for both stages in periods 263I, 263J, 263L and 263M. The first stage average ln K/C values increased to 4.50-4.72 hr^{-1}. The average K/C values for 263I, 263J, 263L and 263M were 20-45% higher than that for 263GH without the Criterion catalyst. More hydrogenation in the second stage by the supported catalyst seemed to increase the first stage thermal conversion activity.

In period 263K, at a lower hydrogen partial pressure (by about 350-400 psia) in the first stage, the first stage average ln K/C value was similar or slightly lower than those for 263IJ at a higher hydrogen partial pressure.

Figures 71 and 72 (Arrhenius plots) show apparent activation energy calculated for the temperature change from 825 to 840°F. Apparent activation energy was determined by the linear regression analysis technique and was 30400-57200 Btu/lb-mole. Results are shown below:

(Figure 71: dispersed molybdenum catalyst system in 263A-H)
$\ln K/C = 16.2 - 15400\ (1/T),\ r^2 = 0.15$

(Figure 72: hybrid catalyst system in 263I-M and 262A-E)
$\ln K/C = 16.1 - 15000\ (1/T),\ r^2 = 0.28$

The Arrhenius plot for temperature dependence (Figure 72) compares the first stage thermal conversion activities in Runs 258, 260, 262 and 263. In these runs, the Black Thunder subbituminous coal was processed with three different catalyst systems: supported, dispersed and hybrid catalyst systems. Run

262 data generated at 825°F using a hybrid catalyst system with a combination of Molyvan L slurry and Criterion 324 supported catalysts showed the highest conversion activity compared to other runs using either slurry or supported catalysts separately (compared with Runs 263, 260 and 258 at the same first stage reaction temperature). Run 263A-H using a dispersed catalyst system with Molyvan L or Molyvan 822 precursor without the Criterion catalyst showed higher activity at 840°F than activities in Runs 260 and 258 using a supported catalyst system with Criterion (Shell) catalyst and without Molyvan L or 822 slurry catalyst. Run 263I-M using a hybrid catalyst system with Criterion and Molyvan L or 822 catalysts showed higher activity at 840°F compared to Run 263A-H using a dispersed molybdenum catalyst only system.

The hybrid catalyst system with a combination of Molyvan L or 822 and Criterion 324 catalysts showed the highest thermal conversion activity, compared to either the dispersed molybdenum or the supported catalyst system. The presence of Criterion supported catalyst in the second stage reactor improved the resid+UC conversion in the first stage, due to improved hydrogenation by the supported catalyst, producing better recycle process solvent and resulting in better thermal conversion in the first stage. It appears that the dispersed molybdenum catalyzes thermal hydrogenation of coal- and resid-derived free radicals (Ref. 16) through stabilization by hydrogen transfer from the process solvent, resulting in higher thermal conversion activity compared to just the supported catalyst system. Apparent activation energies (30000-31000 Btu/lb-mole) for the hybrid and dispersed catalyst systems were lower than that (53000-64000 Btu/lb-mole) for the supported catalyst system, although their conversion activity levels were higher than the supported catalyst system.

In Figure 73, first and second stage thermal conversion activities for periods 263A-H were plotted at the same time to evaluate the staging effect in the dispersed molybdenum catalyst system. Although the second stage activity data were highly scattered, the second stage average value for 810°F seems to be present below the linear regression line extrapolated and extended for a lower reaction temperature of 810°F. It indicates that the quality of the resid entering the second stage might be poorer, exhibiting a lower reactivity. The regression line was generated using the first stage activity data at 825-840°F. The hybrid catalyst system data are not included in regression.

Trend analyses are summarized in Table 19.

5.2.3 Second Stage Supported Catalyst Activity and Thermal Conversion Activity with Dispersed Molybdenum Catalyst

With the addition of dispersed molybdenum catalysts (Molyvan L and Molyvan 822), the thermal conversion activities in the second stage were analyzed for periods 263A-H using Phase 2 resid+UC conversion data in a manner similar to the first stage described in Section 5.2.2. The analysis result is illustrated in Figure 70.

The average ln K/C values for periods 263A-H were calculated and are listed in the following. Reactor volume was used in calculation of the conversion rate constant (K).

	Average ln K/C (1/hr)	
Period	Phase 2	Phase 3
263A	3.42 ± 0.63	3.22 ± 0.85
263B	3.71 ± 0.25	3.92 ± 0.30
263C	3.73 ± 0.41	3.53 ± 0.20
263D	3.75 ± 0.35	3.74 ± 0.13
263E	3.43 ± 0.28	3.44 ± 0.12
263F	3.83 ± 0.39	3.44 ± 0.27
263G	3.82 ± 0.31	3.47 ± 0.09
263H	3.70 ± 0.45	3.48 ± 0.17

The average ln K/C values in the second stage during periods 263B-H did not significantly vary (3.70-3.83 hr^{-1}) at the same reaction temperature of 810°F, although several other changes were made during these test periods: the first stage reaction temperature was increased from 825 to 840°F after period 263B; the coal feed rate was decreased from 300 to 265 MF lb/hr after period 263C; Molyvan 822 was used as a slurry catalyst precursor in place of Molyvan L after period 263E; recycle resid concentration varied in the range of 40-45 wt % in the recycle process solvent; and iron-oxide concentration varied in the range of 1-2 wt % MF coal.

In period 263A, the calculated rate constant value was lower than in subsequent periods, probably due to the inclusion of November 10 data with a very low conversion (3 vs. 8-14 wt % feed). Excluding this low value for November 10, the average ln K/C value increases to 3.68 hr^{-1}, similar to those for other periods. In period 263E, the calculated rate constant value was lower than those for other periods, probably due to the high first stage average value, indicating possible interstage sampling problems as discussed in Section 5.2.2. The average ln K/C value using all the data in 263B-H at 810°F was 3.74±0.38 hr^{-1} (K/C = 44.7±15.5 hr^{-1} ϵ = 12.6±3.8 wt %). The standard deviation on the second stage activity (0.12-0.85) was more than twice the deviation for the first stage activity (0.03-0.21), and as a result, trend data analysis becomes more difficult for the second stage.

In Figure 74, the ln K/C values for periods 263A-H without the presence of Criterion catalyst were lower than that obtained for 262E (average ln K/C = 3.42-3.83 vs. average ln K = 4.41 hr^{-1}). These periods operated at the same reaction temperature of 810°F. This suggests that the presence of a supported catalyst (Criterion, etc.) in the second stage additionally improved the second stage resid+UC conversion. The ln K/C values in Run 263 were similar or higher than that obtained for 262F (average ln K/C = 3.43 hr^{-1}). This observation may not be valid because of high data scatter experienced in Runs 263 and 262F.

Trend analyses are summarized in Table 20.

With the addition of dispersed molybdenum catalysts (Molyvan L and 822), Criterion (Shell) 324 1/16" catalyst activities during both batch operation and steady-state operation with catalyst replacement in the second stage were analyzed for periods 263I-M using the Phase 2 resid+UC conversion data. Results are illustrated in Figures 70 and 74.

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

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

The dependence of the conversion rate constant on temperature (T) is described by the Arrhenius equation and the change in rate constant due to deactivation with catalyst age (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) then becomes:

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

K: second stage resid+UC conversion activity
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: second stage catalyst age

The average ln K values for periods 263I-M were calculated and are listed below. Catalyst volume was used in calculating the conversion rate constant (K).

Period	Average ln K (1/hr)	
	Phase 2	Phase 3
263I	4.50 ± 0.16	4.30 ± 0.26
263J	4.54 ± 0.27	4.39 ± 0.08
263K	4.44 ± 0.16	4.50 ± 0.16
263L	3.99 ± 0.19	4.12 ± 0.11
263M	4.16 ± 0.22	4.16 ± 0.10

The presence of Criterion 324 supported catalyst in the second stage seemed to improve the average rate constant values for both stages in periods 263I, 263J, 263L and 263M. The second stage average ln K values increased to 4.16-4.54 hr^{-1}. The second stage average K values were 45-115% higher compared to the second stage average ln K/C values for previous periods 263B-H (3.70-3.83 hr^{-1}). As discussed in Section 5.2.2, the first stage average ln K/C values also increased to 4.50-4.72 hr^{-1}. The first stage average K/C values were 20-45% higher. More hydrogenation in the second stage by the supported catalyst seemed to increase the first stage thermal conversion activity.

In period 263K, at a lower hydrogen partial pressure (by 350-400 psia) in the first stage, the second stage average ln K/C value did not vary compared to 263IJ at a higher hydrogen partial pressure.

Period 263L was transitional in batch deactivation in the second stage without any catalyst replacement. The second stage average ln K value decreased to 3.99-4.12 hr^{-1}.

In period 263M, at a lower catalyst replacement rate of 2 lb/ton MF coal in the second stage, the second stage average ln K value was lower than that for 263J with 3 lb/ton MF coal catalyst replacement (4.16 vs. 4.39-4.54 hr^{-1}).

In Figure 74, the ln K values for periods 263I-K at 3 lb/ton MF coal catalyst replacement rate were similar to that obtained for 262E (4.44-4.54 vs. 4.41 hr^{-1}). These periods operated at the same reaction temperature of 810°F. The ln K value for period 263M at 2 lb/ton MF coal catalyst replacement rate was lower than those for 263I-K and 262E at 3 lb/ton replacement rate, since the catalyst age at steady-state operation was higher in 263M compared to 263I-K (960 vs. 660-670 lb MF coal/lb catalyst). Apparent activation energy for the hybrid catalyst system (263I-M and 262A-E), calculated for the temperature change in the range of 760-810°F, was 47,800 Btu/lb-mole which was higher than that with the supported catalyst (30,000-40,000 Btu/lb-mole). This high value might have been affected by molybdenum concentration variation in the range of 100-1000 ppm MF coal.

The catalytic resid+UC conversion activity of the hybrid system with a combination of Molyvan L or 822 and Criterion 324 catalysts was much higher than the thermal conversion activity of the dispersed molybdenum catalyst system using just the Molyvan L or 822 catalyst. It seems that the dispersed molybdenum may not be greatly involved in hydrogenation and hydrocracking of the process solvent compared to the supported catalyst, since operations with the dispersed molybdenum catalyst gave significantly lower hydrogen and higher heteroatoms in the distillate and other process streams. The hybrid catalyst system had much higher transition metal loadings in the reactor than the dispersed catalyst system, which appeared to enhance the conversion activity. Criterion 324 catalyst contains 13.2 wt % molybdenum and 2.7 wt % nickel. The catalyst loading with a half-volume reactor was 170 lbs, containing 22.5 lbs Mo and 4.6 lbs Ni. In contrast, the dispersed catalyst system at 100/200 ppm addition rates had very small molybdenum loadings, approximately 0.0355/0.0888 lbs (calculated using 0.2/0.5 wt % Mo_2O_5 in the recycle ash measured during Run 262 with 100/200 ppm Mo addition rates (Ref. 13)). It is possible that the nickel promoter present in the supported catalyst, which is a missing component of the dispersed molybdenum, might be playing a significant role in hydrogenation and hydrocracking. And the alumina support material might have some cracking activity enhancing conversion in the hybrid catalyst system.

Trend analyses are summarized in Table 20.

5.3 Catalyst Requirement and Performance Comparisons

Phase 2 and 3 overall resid+UC conversion data for periods 263A-H without the presence of a supported catalyst and 263I-M with the Criterion (Shell) 324 catalyst are plotted in Figures 75 and 76. These periods processed Black Thunder coal with the addition of Molyvan L or 822 slurry catalyst. The figures are plotted with respect to coal space rate using CCR reactor volume. Data for Runs 262A-F, 260A-F and 258H-M are also included for comparison in Figure 76.

Coal space velocity was selected as the x-coordinate variable to evaluate the process responses in resid+UC conversion and catalyst requirement. To better evaluate the catalyst requirement and coal throughput for Run 263, coal feed rates were projected for resid extinction by using process performance responses estimated by assuming the CSTR first-order kinetics as shown in Figures 77 to 89. Catalyst replacement rates for steady-state operation were calculated for comparison with actual replacement rates used in the plant. Results are listed in Tables 21 and 22.

Periods 263IJ with the Criterion supported catalyst in the second stage showed a similar or higher resid+UC conversion than 263E-H without the Criterion catalyst, even though 263IJ was operated at a higher coal feed rate (350 vs 265 MF lb/hr) and a higher coal concentration in the slurry (30 va 25 wt %). Catalyst replacement rate was 3 lb/ton MF coal in 263IJ, which improved the coal throughput approximately by 26-33% compared to 263H (Table 22). The distillate production rate also increased by 25-32% (See Section 5.3.1).

Period 263K, at a lower hydrogen partial pressure in the first stage by about 350-400 psia, showed a 1-5 wt % lower resid+UC conversion compared to 263J. This lower conversion affects the coal throughput by decreasing it by 1-8% for the "all-distillate" product slate with resid extinction (Table 22). The distillate production rate decreased by 1-8% (See Section 5.3.1).

Period 263M, at a lower catalyst replacement rate of 2 lb/ton MF coal, showed a 2-10 wt % lower resid+UC conversion compared to 263J with 3 lb/ton MF coal replacement rate. This lower conversion affects the coal throughput by decreasing it by 2-11% for the "all-distillate" product slate with resid extinction (Table 22). The distillate production rate decreased by 5-16% (See Section 5.3.1).

Figure 90 compares the coal conversion for Runs 263 and 262. Reactor and catalyst volumes for these runs are summarized below:

run(s)	262	263
reactor	half/half[a]	half/half[b]
reactor volume ratio to full	0.47	0.47
catalyst volume (slump) (ft^3)	- /3.1[a]	- /3.1[c]
(ratio to full)	- /0.38[a]	- /0.38[c]

[a] Thermal/catalytic mode of operation.

[b] Thermal/thermal mode of operation in 263A-H;
thermal/catalytic in 263I-M.

[c] Catalytic second stage in 263I-M

Both runs showed similar coal conversions and flat responses with respect to coal space velocity (except for Phase 2 data for

263AB, which were 4 wt % MAF coal lower than Phase 3 data), probably due to other process variable changes made at the same time (Molyvan L or 822 addition, coal space velocity, first and second stage reaction temperatures, Criterion supported catalyst replacement rates, etc.).

A thermal reaction severity factor (TRSF) is defined as a function of system pressure (P), coal space velocity above 700°F (SV) and average reaction temperature (T) as shown below in order to evaluate the process responses in coal and resid+UC conversions:

$$\begin{aligned} \mathrm{TRSF} &= f(P, SV, T) \\ &= a(P^b)(1/SV)\exp(c/T) \end{aligned}$$

where P: system pressure [psia]
SV: coal space vel. [MF lb/hr/ft^3-reaction volume]
T: volumetric average reaction temperature [°R]
a,b,c: constants

Runs 263A-M and 262A-F coal conversion data are compared in Figure 91 with the second order correlation developed for Run 260A-F. This figure does not show the data used for the correlation (Ref. 11). This comparison suggests that Molyvan L or 822 addition in 263I-M and 262A-E improved coal conversion by approximately 3-5 wt % MAF coal in both the thermal first stage and thermal/catalytic first and second stages compared to 260A-F, when Criterion 324 supported catalyst alone was present in the second stage. However, in Run 263A-H without the presence of a supported catalyst, Molyvan L or 822 addition did not improve the coal conversion, in fact 1-4 wt % MAF coal lower, compared to 260A-F which was operated with just the Criterion 324 catalyst. It seems that the Criterion supported catalyst improved coal conversion more than Molyvan L or 822 slurry catalyst, and coal conversion further improved by the combination of Criterion supported and Molyvan L (or 822) slurry catalysts in the system.

Runs 263A-M and 262A-F resid+UC conversion data are compared in Figures 92 with correlations (power function, $Y = \alpha X^\beta$) developed for Runs 260 and 258 (Ref. 11). This comparison suggests that Molyvan L or 822 addition in 263I-M and 262A-E improved the resid+UC conversion by approximately 0-10 wt % MAF coal in the thermal first stage and 5-10 wt % in overall two-stage (except in 262F where it did not improve with a small amount of catalyst left in the second stage) when Criterion 324 supported catalyst was present in the second stage. However, in Run 263A-H without the presence of a supported catalyst, Molyvan L or 822 addition did not improve resid+UC conversion, which was similar in overall two-stage and slightly lower in the first stage compared to 260A-C with Criterion 324 catalyst and without the addition of Molyvan L or 822 slurry catalyst. It seems that both Molyvan L or 822 slurry and Criterion supported catalyst had similar resid+UC conversion, and resid+UC conversion improved by the combination of Criterion supported and Molyvan L or 822 slurry catalysts, as seen in Runs 262 and 263.

A good linear correlation between organic rejection and coal conversion is shown in Figure 93. Data generated for low-rank coals and lignite runs (251-II, 251-III, 255, 258, 260, 262 and 263) are used for the linear regression calculation. In Runs 262 and 263 with Molyvan L or 822 slurry catalyst addition, organics were rejected more in the ROSE-SR^{sm} bottoms product stream, when compared at the same coal conversion. It seems that Runs 262 and 263 operated with weaker deashing solvents than normally used for optimum recovery operation of the ROSE-SR^{sm} unit in previous runs. It is also possible that the Molyvan L or 822 slurry catalyst addition might have affected the ROSE-SR^{sm} feed properties, resulting in higher organic rejection.

Regression results are summarized below:

$$Y = 173.3 - 1.69\,X;\ r^2 = 0.84$$

where Y is the organic rejection (wt % MAF coal), X is the coal conversion (wt % MAF coal), and r^2 is the determination coefficient. The X-intercept in Figure 93 indicates that a deeply cleaned coal could achieve a low organic rejection (4 wt % MAF coal), if extrapolated to 100 wt % coal conversion. A similar observation with bituminous coals was reported in the previous work (Ref. 12). Coal cleaning with heavy media improved coal conversion, and as a result reduced organic rejection, as evidenced while processing bituminous coals, Illinois No. 6, Ohio No. 6 and Pittsburgh No. 8 (Refs. 5, 10, 27, 28 and 29). However, heavy media cleaned subbituminous coals were not tested at Wilsonville and data with deeply cleaned subbituminous coals were not available in the literature.

5.3.1 Distillate Production Comparison

Experimentally measured distillate production rates are compared in Tables 23 and 24. Low-rank subbituminous coals and Texas lignite were processed in Runs 263A-M, 262B-F, 260CD, 258EHI, 255B and 251-IIB, and projected rates for the "all-distillate" product slate with resid extinction for these runs are compared in Tables 25-27. The CSTR first-order resid+UC conversion kinetics were assumed for the projection of the "all-distillate" product slate; and common organic rejection (COR) was assumed to determine the resid+UC conversion for each period. Catalyst replacement rates for Runs 251-IIB, 255B and 263L were estimated at steady-state operation from batch aging data. Figures 94-104 illustrate distillate production rates with different operating conditions, and Figures 105 and 106 compare experimental and projected theoretical distillate production rates. A good linear correlation was observed in the parity plot of projected and experimental distillate production rates (Y = X, r^2 = 0.88 in Figure 106).

In periods 263I-M, with the hybrid catalyst system, the distillate production rates were higher than those for periods 263A-H in the dispersed molybdenum catalyst system (178-195 vs. 132-150 lb/hr) (Figure 94) with higher total (supported + dispersed) molybdenum addition rates, 233-300 vs. 100-200 ppm (Figure 95) and with operation at higher coal space velocity (45C-46C vs. 34C-40C MF lb/hr/ft^3-reactors), a similar (or slightly lower) resid recycle level (40-45 wt % in process solvent) and similar (or higher in some periods) reaction temperatures (825-840/810°F) (Figure 96).

Data for the previous run periods, 262B-F in the hybrid catalyst system except for 262F in the dispersed molybdenum catalyst system, are compared in Figures 97-99, similar to Figures 94-96 for Run 263. High distillate production rates (171-185 lb/hr) were observed for periods 262C1, D and E in the hybrid catalyst system as were for 263I-M. Total molybdenum addition rates were high, 300-700 ppm; coal space velocities were 40C-46C MF lb/hr/ft^3-reactors; and resid recycle was at 36-39 wt %.

In Figures 100-102, data for Runs 258, 262 and 263 with Black Thunder subbituminous coal are compared at the same time for three different modes of operation with supported, dispersed and hybrid catalyst systems. Details concerning the distillate production for selected steady-state operation periods are discussed in Section 5.3.2. Illinois No. 6 bituminous coal result from Run 257J using Amocat 1C catalyst in the catalytic/-catalytic mode is also included for comparison. The hybrid catalyst system showed the highest distillate production, while the supported catalyst system had the lowest. The slurry catalyst system was in the middle. The distillate production of the supported catalyst system with Illinois coal in the catalytic/catalytic mode was similar to that (the lowest) for the supported catalyst system with Black Thunder coal. Total molybdenum addition rates in the hybrid system were higher than in the slurry and supported systems (233-400 vs. 100-200 ppm); and were higher or similar to that (240 ppm) for Run 257J with Illinois coal. Black Thunder coal operations employed higher reaction temperatures, higher coal space velocity, and lower recycle resid concentration compared to operation with Illinois coal.

The effect of supported catalyst replacement on distillate production is shown in Figures 103 and 104 for low-rank subbituminous coals and lignite processing. Direct comparison is not possible, since several operating conditions were different for these runs such as coal type, catalyst type, reaction temperatures, recycle resid concentration, etc. In periods 263JM, the supported catalyst replacement was varied by 1 lb/ton

MF coal rate, resulting in 6-19% increase in the distillate production (6% increase in Phase 3 data and 19% increase in Phase 2 data). The effect of supported catalyst replacement with bituminous coals was 15-20% increase in the distillate production per unit (lb/ton) catalyst replacement rate increase (Ref. 12). Further studies are necessary in order to develop meaningful correlations to quantify catalyst replacement effect with low-rank coals, especially with the hybrid catalyst system.

High distillate yields, 57-63 wt % MAF coal, were observed with the liquefaction of Black Thunder Mine subbituminous coal, as shown in the following.

	distillate yield, wt % MAF coal	
run	experimental	theoretical
263E-J	57-62	60-63 (COR=13-14)
262B-E	57-65	57-63 (COR=16-19)
258HI	55-58	60 (COR=14)

Listed below are the key process variables studied in Runs 258, 260, 262 and 263. These variables are used to maximize the distillate production by increasing the distillate yield and coal throughput while processing the Black Thunder mine subbituminous coal.

(1) Catalyst activity
  - Shell (or Criterion) 324 unimodal supported
  - dispersed molybdenum (Molyvan L/822 precursors)
  - iron-oxide + DMDS (dimethyl disulfide)

(2) Steady-state operation with catalyst replacement
  - 1.5 to 3 lb/ton MF coal in catalytic stage

(3) Molybdenum concentration (100-1000 ppm MF coal)
(4) Iron-oxide addition concentration (1-2 wt % MF coal)
(5) Recycle resid concentration
  - 35-45 wt % in the process solvent

(6) High/low thermal severity mode
(7) Heavy vacuum gas oil recycle
(8) Coal space velocity
  - 45-110 lb MF coal/hr/ft^3-catalyst in catalytic
  - 30C-90C lb MF coal/hr/ft^3-reactor in thermal
    (C: constant equating thermal to catalytic reaction volume in the thermal/catalytic mode)

(9) Reaction temperature
  - 760-860°F/760-810°F in the first/second stage

(10) Inlet hydrogen partial pressure
  - 2450-2850 psia in the first stage
  - 2400-2550 psia in the second stage

(11) Slurry composition (25-30 wt % coal, 20 wt % CI)
(12) Interstage separation
(13) Reactor operation parameters
  - temperature profiles, exotherms

- ebullation rate, gas flow, slurry flow
- slurry viscosity, etc.

Several steady-state operation periods in Runs 258, 262 and 263 with half-volume reactors in operation were selected for distillate production rate comparison in three different catalyst system: the supported, dispersed and hybrid catalyst systems. Experimental (exp.) results with Black Thunder subbituminous coal are summarized in the following table along with the theoretical (the.) distillate production projected for the "all-distillate" product slate with resid extinction using a CSTR first-order kinetic model. Illinois No. 6 bituminous coal result from Run 257J using Amocat 1C catalyst in the catalytic/catalytic mode is also included for comparison.

run	catalyst	temper. (°F) 1st/2nd	cat. rep. (lb/t MF) 1st/2nd	recycle resid (wt %)	dist.prod. (lb/hr) exp.	the.[a]
(1) w/ supported catalyst in the second stage						
258H	Shell 324	840/790	-/1.5	40	127	126
258I	Shell 324	850/790	-/1.5	40	132	126
(2) w/ dispersed molybdenum catalyst						
262F[b]	Molyvan L	825/810	200[c]	40	152	137
263E	Molyvan L	840/810	100[c]	40	147	150
263H	Molyvan 822	840/810	100[c]	45	150	158
(3) w/ hybrid catalyst system						
262E	[d]	825/810	-/3[e]	40	185	181
263I	[d]	840/810	-/3[e]	45	195	207
263J	[d]	840/810	-/3[e]	40	188	197
(4) w/ Illinois coal in catalytic/catalytic mode						
257J	Amocat 1C	810/760	3/1.5	50	137	133

[a] Using CSTR 1st order kinetic model for resid extinction.
[b] Some Criterion catalyst was left in the second reactor.
[c] Unit: ppm based on MF coal.
[d] combination of Molyvan L and Criterion 324 catalysts.
[e] Molyvan L addition at 100 ppm based on MF coal.

5.3.2 Supported, Dispersed and Hybrid Catalyst Systems

Among the three catalyst systems tested, the hybrid catalyst system (Runs 262E and 263IJ) with a combination of dispersed molybdenum and Criterion supported catalysts showed the highest distillate production at 185-195 lb/hr. Corresponding coal space velocities for these runs were 59-62 MF lb/hr-ft^3-catalyst in the second stage or 24C-26C MF lb/hr-ft^3-reactors in CCR unit. The distillate production in the hybrid catalyst system was 30%

higher than in the dispersed molybdenum catalyst system (147-152 lb/hr in Runs 262F and 263EH at 20C coal space velocity); 50-60% higher than in the Criterion supported catalyst system (127-132 lb/hr in Run 258HI at 17C coal space velocity).

The hybrid catalyst system with Black Thunder mine subbituminous coal in the thermal/catalytic mode of operation produced 40-55% higher distillate than the Amocat 1C supported catalyst system with Illinois No. 6 bituminous coal in the catalytic/catalytic mode of operation (Run 257J at 18C coal space velocity). This result suggests that the Black Thunder subbituminous coal can produce more distillate than Illinois No. 6 bituminous coal, if process operating conditions are optimized for better coal and resid conversions, even though subbituminous coal has lower distillate yield and selectivity.

It seems that the addition of the dispersed molybdenum catalyst to the supported catalyst system enhances the distillate production with improved coal and resid conversions. The dispersed catalyst allows higher thermal severity operation compared to Run 257J with Illinois coal and Amocat 1C catalyst. However, as reported in Run 261 with Illinois bituminous coal using a new bimodal supported catalyst (EXP-AO-60) (Ref. 13) in the catalytic/catalytic mode with full-volume reactors in operation, the low/high severity operation significantly improved the distillate production by approximately 40-50%, employing higher thermal severity, increased recycle resid concentration and higher catalyst replacement. Further studies are required for better comparison with bituminous coals using half-volume reactors in operation.

5.3.3 Distillate Product Yield and Selectivity

The addition of a dispersed molybdenum allowed operation at lower thermal severity in the first stage, while the second stage operated at higher thermal severity compared to the supported catalyst system, resulting in higher C4+ distillate product yield and selectivity to resid+UC conversion [illegible] summarized below. The potential C4+ distillate yield is estimated by adjusting the resid yield for the "all-distillate" product slate with resid extinction by using an achievable common organic rejection (COR) and distillate selectivity measured in each run.

	catalyst	distillate yield, wt % MAF coal		distillate
run	system	experimental	theoretical (COR)	selectivity,%
258HI	supported	55-58	60 (14)	68-70
263EH	slurry	61	62-63 (14)	72-73
263IJ	hybrid	58-61	61-62 (13-14)	71-72

5.3.4 Effect of Molybdenum Concentration

Varying the molybdenum concentration from 100 to 1000 ppm (based on MF coal) seemed not to affect the resid+UC conversion, but increasing the molybdenum concentration slightly improved the coal conversion (Ref. 13). Good performance was observed at a low 100 ppm concentration in both slurry and hybrid catalyst systems in Runs 263 and 262. Distillate properties were generally the same within the range of molybdenum concentrations studied. The addition of Molyvan L or Molyvan 822 seemed to improve the overall two-stage resid and coal conversions by allowing lower temperature and higher space velocity operation. Mineral analysis of the vacuum flash bottoms ash indicated that the molybdenum content in the recycle ash had reached a reasonable level of equilibration after a change in molybdenum concentrations in the feed.

When compared at the same thermal reaction severity operation, in the hybrid catalyst system with a combination of dispersed and supported catalysts, the resid+UC conversion was 5-10 wt % MAF coal higher (Figure 92) and the coal conversion was 3-5 wt % higher than by using the catalysts separately (Figure 91). The use of just dispersed molybdenum catalyst (Molyvan L or Molyvan 822) or Criterion supported catalyst alone gave similar overall two-stage coal and resid+UC conversions, and the conversions improved by the combination of Criterion and Molyvan L or 822 catalysts.

Dispersed molybdenum seemed to catalyze thermal hydrogenation of coal and/or resid through hydrogen transfer from the process solvent (Figure 72), which resulted in a higher thermal conversion in the hybrid catalyst system, but seemed to have a lesser role in hydrogenation and hydrocracking of the process solvent compared to the supported catalyst (Figure 107). Black Thunder coal runs with Criterion supported and dispersed molybdenum catalysts produced distillate with less hydrogenation and heteroatoms removal with operations at higher space velocity and temperature with less catalyst replacement, compared to runs with Illinois No. 6 coal and Amocat 1C catalyst (Figure 107).

The effect of molybdenum addition rate varying from 100 to 1000 ppm was not significant in thermal conversion in the first stage, when tested at 825°F in Run 262 with the hybrid catalyst system (Figure 108). Also, the effect of changing molybdenum addition rate from 100 to 200 ppm was not detected at 825°F in Run 263 with the slurry catalyst system (Figure 108). However, the effect on second stage catalytic conversion in the hybrid catalyst system in Run 262 was very significant, with a more prominent impact at a higher reaction temperature (790 vs. 760°F) (Figures 74 and 109). At a low 100 ppm molybdenum addition rate in Run 263JM, the increase of molybdenum addition resulting from increasing the supported catalyst replacement rate from 2 to 3 lb/ton MF coal (1.5 lb/ton equivalent to 100 ppm molybdenum addition) improved conversion activity at 810°F, which was even higher than that seen at 790°F. Apparently dispersed molybdenum with supported catalyst produces a more reactive resid for cracking and thus diminishes the propensity for retrogressive reactions (Figure 109).

5.4 Thermal History in TSL Process System

Thermal histories during October 31, 1991 - January 2, 1992 time period in Run 263 are illustrated in Figures 110, 111, 116 and 117. Figures 112 to 115 and 118 to 121 show trends in system pressure, differential pressure, gas flow, and ebullation and backmixing pump flows for this period. For the above period, the outlet temperature of the B1201 interstage heater had to be increased above the second stage reactor temperature in order to maintain the targeted second stage reaction temperature of 810°F.

Figure 122 shows the change in the first stage reactor temperature profile that occurred after December 22, probably caused by a lower backmixing flow due to P1222 ebullation pump problems. The total reactor exotherm stayed the same, but the reactor temperature spread increased from 25-30 to 55-65°F. It is also possible that solids buildup in the reactor bottom might have affected the temperature profile or the reading of the bottom thermocouple might be erroneous.

Thermal histories during January 15 - February 22, 1992 in Run 263 are illustrated in Figures 123 to 125. Figures 126 to 130 show trends in system pressure, differential pressure, gas flow, and ebullation and backmixing pump flows for this period. Since the Criterion 324 supported catalyst was charged to the second stage on January 21 (od=72), the outlet temperature of the B1201 interstage heater no longer had to be increased above the second stage reactor temperature in order to maintain the targeted second stage reaction temperature of 810°F (Figure 125).
A significant increase in the second stage total exotherm was observed due to more hydrogenation by the supported catalyst in the second stage in period 263J (Figure 135), as was previously reported for Run 262E with a similar second stage temperature profile, even though the first stage reaction temperature was different, 840°F for 263J and 825°F for 262E. The bed exotherm increased from 2 to 6-7°F.

During February 4-9, at 350-400 psia lower hydrogen partial pressure in the first stage, the first stage total exotherm declined by 20°F due to less hydrogenation and cracking reactions (Figure 123). During February 9-16 (od=91-97) with catalyst batch deactivation, the second stage total exotherm continuously declined until 2 lb/ton MF coal catalyst replacement was started on February 17 (od=98), resulting in stabilization of the second stage total exotherm (Figure 125).

Figures 123 and 124 show that the first stage reactor temperature profile changed continuously for the entire period, probably caused by a lower backmixing flow from the P1222 ebullation pump due to solids buildup in the bottom of the reactor. The pumping problems started after December 22, gradually lowering the reactor bottom thermocouple reading, and became worse after February 13 (od=94). The total reactor exotherm varied much less, primarily depending upon the process operating conditions (partly affected by the low backmixing flow). The reactor temperature spread increased from 25-30 to 55-65°F, and then gradually to 110-115°F, and after February 13 to 250-315°F. It is possible that gradual solids buildup in the reactor bottom without a significant backmixing flow might have affected the temperature profile. It is also possible that the thermocouple reading was in error as indicated in period 263M with a large drop in process temperature from B1200 preheater to R1235 first stage reactor relative to the other periods (see Figure 137).

Thermal histories during periods 263A, B, C, H, J, K, and M are illustrated in Figures 131 to 137 by using temperature profiles and approximate relative residence times through the TSL system. Run 263 operated with half-volume reactors and an interstage separator. Process times for slurry blend and feed tanks, and process solvent storage tank were significantly reduced for illustration:

Key Equipment	Location Number	Description
V101A	6-10	slurry blend tank
V101B	10-14	slurry feed tank
B1200	16-18	preheater
R1235	19-29	1st stage reactor
V1258	31	interstage separator
B1201	33-35	interstage heater
R1236	37-47	2nd stage reactor
V1247	49	high press separator
V1069	51	atm. flash vessel
V1082	53	vac. flash vessel
V131B	55-59	process sol. storage

6. DISTILLATE PRODUCT QUALITY AND UNIT SOLVENTS

6.1 Product Quality

The distillates produced in Run 263 resulted from liquefying the subbituminous Black Thunder mine coal. Run 263 was similar to Run 262 since the same coal was used and the CC-ITSL operating modes were not all that different. The specific operating conditions have been documented elsewhere in this report. In terms of their influence on product quality, the operating conditions for Run 263 can be divided into two major segments. The first one is the operation with a dispersed molybdenum catalyst precursor, Molyvan L or Molyvan 822, only. The second segment is a hybrid operational case where both the dispersed molybdenum (Molyvan L or 822 precursor) and Criterion 324 supported catalysts were used. The supported catalyst was used in the second stage reactor and the first stage remained a thermal reactor. Distillate products were characterized for eleven material balance periods in Run 263. Eight were done for the dispersed molybdenum only case and include periods 263A through 263H. Three workups were performed for the hybrid case and include 263J, 263K, and 263M.

6.1.1 Methodology and Product Preparation

Tests indicative of product quality characteristics were performed for eleven material balance periods. The tests included carbon, hydrogen, nitrogen, and sulfur. All of these were done by combustion methods. Oxygen content was determined by difference. Simulated distillation was done on all of the distillates. This test was performed by gas chromatography and it gave the weight percent composition for various boiling ranges. The end point for the total product oil was determined by an actual laboratory distillation using ASTM Method D-1160. A pycnometer was used to determine specific gravity. Then, approximately 600 grams of each product oil was distilled on an Oldershaw column into typical petroleum boiling fractions of Naphtha (IBP-350°F), Middle Distillates (350-450°F and 450-650°F), and Vacuum Gas Oil (650°F+). This gave the percentage of each fraction present in the total oils and the yield of each fraction was sufficient to allow for their characterization using similar tests.

The total product oil was prepared from various product streams in proportion to their respective yields or flow rates on the material balance days. In all cases, except for 263F with V1067 atmospheric flash bottoms recycle, the products were prepared from a combination of distillation column overheads. Figure 138 is a schematic of the distillation separation system showing locations of various columns and vessels.

Table 28 summarizes the blending formulation for each product oil. The atmospheric column overhead is naphtha-like and this material collects in vessel V161. Comparatively, the stream has lower heteroatoms, contains about 12 wt % hydrogen, and boils in the 75-500°F range. It usually comprises 30-40 wt % of the total oil product. The other major component is the vacuum column overhead called V182 oil. It may comprise 40-70 wt % of the product. The oil has some properties similar to No. 2 diesel fuel. It boils in the 400-850°F range and contains from 10 to 11 wt % hydrogen. Sulfur is usually less than 0.10 wt % in the oil, but oxygen and nitrogen may total more than 3 wt %.

In period 263F, the atmospheric flash bottoms (V1067) was recycled instead of the vacuum flash bottoms (V1082). This resulted in 41.6 wt % of the T102 vacuum column bottoms (V1074) being used as a component of the total oil product. The bottoms product is very heavy and boils in the 650-1100°F range. It usually contains less than 10 wt % hydrogen and has a significant nitrogen content which may be from 5,000 to 10,000 ppm.

6.1.2 Product Quality and Results

Total Oil

Run 263 Black Thunder coal distillate properties are summarized in Table 29. The oil properties from operations using the dispersed molybdenum catalyst only differed from those produced with the hybrid catalyst system where both dispersed and supported catalysts were used. In general, the distillate properties from the latter operational cases were favored over the dispersed molybdenum catalyst only cases. Operations with the dispersed molybdenum catalyst only produced a distillate with lower hydrogen content and also higher heteroatoms content. For example, periods 263A through 263H were material balance periods with the dispersed molybdenum catalyst only. Hydrogen averaged 10.17 wt %, nitrogen 9,073 ppm, sulfur 0.05 wt % and oxygen by difference was 3.64 wt %. The product properties improved for the hybrid catalyst operational cases as the average hydrogen increased to 10.85 wt %, nitrogen decreased to 6,366 ppm, oxygen decreased to 1.88 wt % and sulfur was at 0.03 wt %.

Eight distillate products were evaluated for the dispersed molybdenum catalyst only operational cases. Within these periods, various operational changes were made which allows one to determine its effect on product quality. For example, at 100 ppm molybdenum concentration there was no difference in product properties using either Molyvan L or 822. This was evidenced by comparing product properties from 263E (using Molyvan L) with those from 263G (using Molyvan 822). The carbon, hydrogen, nitrogen, and sulfur values were about the same for both periods. Change in iron-oxide concentration in the coal feed apparently had no effect on product properties either. This conclusion was drawn by comparing the distillate properties from 263G (using 2 wt % iron-oxide) with those from 263H (using 1 wt % iron-oxide). The carbon, hydrogen, nitrogen, and sulfur values were so close they can be considered within experimental range of the instrument.

The utilization of the atmospheric flash bottoms (V1067) as recycle material did have an effect on product quality in that it elevated the end point of the product. This V1067 material was recycled in 263F and the end result was a distillate product with an end point of 963°F which was about 200°F greater than any of the other end points with V1082 vacuum flash bottoms recycle in place of V1067. The elevated end point could be explained by a large increase in the T102 vacuum column bottoms product (V1074) to the blending formula (see Table 28). Ordinarily this product was not a component of the total oil which means that in normal operations it was completely consumed in the recycle solvent preparation. It was very heavy and started to boil around 650°F and even 85 wt % of it boiled above 850°F.

Periods 263D and 263E allows for the comparison of resid concentration in recycle solvent on product quality. There was no apparent effect due to resid as it was increased from 41 wt % in 263D to 44 wt % in 263E. The increase was small but it might suggest that increasing it tends to elevate the end point of the product by producing heavier distillates. Within the ranges studied, space velocity and temperature had no great effect on product quality. Coal space velocity influence can be obtained by comparing product properties from 263C (77.6C MF lb/hr/ft^3-reactor) with those from 263D (68.6C MF lb/hr/ft^3-reactor). There were no significant changes in product properties.

The first stage thermal reactor temperature was increased by 15°F from 263B (825°F) to 263C (840°F). All properties were about equal except that the 840°F case did favor a slight reduction in nitrogen as it decreased from 10,698 ppm to 9,376 ppm. Decreasing the Molyvan L concentration from 200 to 100 ppm apparently caused a nitrogen increase in the distillate product. This was seen by comparing 263A when the Molyvan L concentration was 200 ppm and 263B where it was 100 ppm. The nitrogen increased from 8,475 ppm to 10,698 ppm from 263A to 263B.

The hybrid operational cases are for periods 263J, 263K, and 263M. As already mentioned, the overall distillate properties improved for these cases. Period 263J had the best product quality properties in terms of hydrogen content and heteroatoms content. Hydrogen decreased from 11.04 wt % in 263J to 10.69 wt % in 263K. The difference between these periods was the first stage inlet hydrogen partial pressure as it was decreased from 2744 psia in 263J to 2386 psia in 263K. In 263M, the hydrogen in the distillate increased to 10.82 wt % when the hydrogen partial pressure was restored to 263J conditions. However, the catalyst replacement rate for 263M was at 2 lb/ton MF coal feed while the rate for 263J was 3 lb/ton MF coal feed. Hence, higher hydrogen partial pressure and increased catalyst replacement rate all were more favorable for the distillate in 263J.

Excluding 263F with atmospheric bottoms recycle, the end points for the products averaged 744°F which was similar to product end points for Run 262 distillates. The 792°F end point for the 263K distillate was not a good representation since at that time the T102 vacuum column was not functioning properly. In the formulation, the laboratory had to distill out the lighter boiling portion of the T102 bottoms product (V1074) as indicated in Table 28. Some of the more heavier product could have come over in the distillation thus contaminating the final product.

Run 263 oil product composition was toward higher boiling components. From 70 to 85 wt % of the product boiled above 450°F. The lighter boiling naphtha fraction (IBP-350°F) accounted for about 11 wt% of the product. Heavier boiling components were consistent with a product having lower hydrogen content and usually higher heteroatom composition and especially nitrogen.

Naphtha (IBP-350°F)

Naphtha properties are shown in Table 30. The naphtha quality produced from hybrid operations was more favorable than the quality from the dispersed molybdenum catalyst only cases. Hydrogen content for the hybrid cases averaged 13.67 wt %, nitrogen 1,671 ppm, and oxygen by difference was 1.65 wt %. In the dispersed molybdenum only cases, hydrogen decreased to 13.10 wt % while nitrogen increased to 2,404 ppm and oxygen increased to 2.83 wt %. Sulfur in the distillate for dispersed molybdenum catalyst only operations was 0.07 wt % which was lower than the 0.14 wt for the distillates from hybrid operations. The °API gravities averaged 47.2 and 48.5 for the dispersed and hybrid cases, respectively.

Middle Distillate (350-450°F)

Middle distillate boiling in this range comprised 14.4 wt % of the total oil product. The product properties were better for the hybrid cases than for the dispersed molybdenum catalyst only cases. For the hybrid cases, the distillate hydrogen averaged 11.31 wt %, nitrogen 1,889 ppm, and oxygen 2.78 wt %. Hydrogen decreased to 10.23 wt % for the dispersed molybdenum catalyst only distillates while oxygen increased to 5.73 wt % and nitrogen increased to 4,195 ppm. Sulfur was about the same in the distillates for both types of operation and averaged 0.04 wt %.

The middle distillate properties are summarized in Table 31.

Middle Distillate (450-650°F)

This boiling fraction comprised 37.1 wt % of the total oil products. Its properties are shown in Table 32. Again, the distillate properties differed in the hybrid operational cases compared to use of the dispersed molybdenum catalyst only. The quality was slightly better for hybrid distillates as hydrogen averaged 10.67 wt %, nitrogen 7,421 ppm, and oxygen by difference was 1.46 wt %. Oxygen increased to 3.60 wt % and nitrogen to 10,734 ppm for the dispersed molybdenum catalyst only periods while hydrogen content of the distillates decreased to 10.05 wt %. Sulfur remained about the same for both operational cases and averaged 0.03 wt %. The °API gravities increased from 12.2 for the dispersed molybdenum catalyst only distillates to 16.5 for the hybrid operational cases.

Vacuum Gas Oil (650+°F)

The vacuum gas oil properties are shown in Table 33. The gas oil comprised 37.1 wt % of the total oil product. Its properties for the hybrid cases were slightly better compared to those from the use of the dispersed molybdenum catalyst only. Hydrogen averaged 9.83 wt %, nitrogen 8,035 ppm, and oxygen 0.86 wt % for the hybrid operational material balance periods. For the dispersed molybdenum catalyst only cases, hydrogen in the distillate decreased to 9.67 wt % but nitrogen increased to 11,201 ppm and oxygen to 1.81 wt %. Sulfur averaged 0.02 wt % and was about the same in both types of operations.

6.1.3 Summary

From a product quality perspective, the major conclusion from Run 263 was that operations using a dispersed molybdenum catalyst along with a supported catalyst produced better quality distillates than just using a dispersed molybdenum catalyst only. Hydrogen content increased while heteroatoms generally decreased in the distillates from the hybrid operational cases. Some observations concerning product quality for Run 263 were as follows:

- Using either Molyvan L or 822 at 100 ppm molybdenum concentration did not change distillate product properties.
- Recycling the atmospheric flash bottoms (V1067) increased the end point of the distillate product by about 200°F.
- The end point of the distillate product averaged 744°F

recycling the vacuum flash bottoms (V1082) excluding the end point from the product using V1067 recycle.

- Increasing the resid concentration from 41 to 44 wt % in the recycle process solvent had no significant effects on distillate product quality.
- Decreasing the space velocity by only 12% had no apparent effects on distillate product quality.
- Increasing first stage reactor temperature by 15°F had no great effect on product properties.
- Decreasing the iron-oxide concentration in the coal feed from 2 to 1 wt % MF coal had no effect on distillate properties.

6.2 Unit Solvents

6.2.1 CCR Unit Recycle Solvent

The recycle solvent was made up of ROSE-SR^{SM} deashed resid, the V1082 vacuum flash bottoms providing the solids recycle material, and the vacuum distillation column bottoms (V1074). In period 263F, the V1067 atmospheric flash bottoms was used instead of V1082 vacuum flash bottoms.

The targeted recycle solvent composition for the run was 40-45 wt % resid, 20 wt % CI, and 35-40 wt % distillate. In most periods except for 263A and B (with 825°F first stage reaction temperature) and 263M (with 2 lb/ton MF coal catalyst replacement), there was no positive resid make and no extra resid was available to recycle back. The process system operation was balanced at 42-45 wt % recycle resid levels.

The catalyst withdrawal tube flush for both reactors was vacuum distillation column bottoms material (the V1074 heavy vacuum gas oil) for periods 263A-D. The flush was heated by dowtherm jackets before it entered the reactors. Due to pumping difficulties with V1074, the lighter V178 (same as V182) solvent was used since period 263E.

6.2.2 ROSE Unit Deashing Solvent

The ROSE-SR^{SM} unit uses a proprietary deashing solvent to process material containing ash. The 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^{SM} bottoms product and maintaining efficient, stable deashing in the first stage.

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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 solid 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.

Appendix A (continued)

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, measured as 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.

P1090: Pump used in the resid circulation system to inject deashed resid from the ROSE-SR^{SM} unit into the CCR unit feed line.

P1091: Pump used in the resid circulation system to inject the solids recycle stream (V1082 bottoms material) into the CCR unit feed line.

R1235 OTL: Interstage sample.

Appendix A (continued)

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 solid 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.

Solid Product: A product of the Kerr-McGee ROSE-SR^{SM} 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.

Appendix A (continued)

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

V1090: Calibration dump pot vessel used to collect and measure the flow rate of the deashed resid from the ROSE-SR^{SM} unit used in the resid circulation system.

V1091: Calibration dump pot vessel used to collect V1082 bottoms material (solids recycle stream) and to measure the flow rate when the resid circulation system is in service.

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, and 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 263 can be found in Table 5. 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 34 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

Supported 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

Black Thunder Coal Analysis for Runs 263 and 262

Period	263A	263B	263C	263D	263E	263F	263G	263H	263I	263J	263K	263L	263M	262A
Proximate (wt % MF)														
Volatiles	43.0	43.4	43.0	43.0	43.3	43.7	43.9	43.4	43.8	43.9	44.1	43.8	43.1	49.6
Fixed Carbon	49.3	49.6	49.3	49.8	49.6	49.4	49.0	49.8	49.5	49.4	49.4	49.6	50.4	44.1
Ash	7.7	7.0	7.6	7.2	7.1	6.9	7.1	6.8	6.7	6.7	6.5	6.6	6.5	6.2
Moisture	22.8	21.7	23.6	23.5	23.4	23.3	24.5	24.2	23.6	23.4	23.4	23.3	23.6	20.8
Ultimate (wt % MF)														
Carbon	69.5	69.1	67.5	68.2	69.3	68.6	67.9	68.9	68.8	69.4	68.5	68.8	67.5	69.6
Hydrogen	5.0	5.0	5.2	4.9	5.1	5.1	5.0	5.1	5.1	5.0	5.2	5.1	5.3	5.3
Nitrogen	1.1	1.1	1.1	1.0	1.1	1.2	1.0	1.0	1.0	1.1	1.0	1.0	1.0	1.0
Sulfur	0.5	0.5	0.5	0.4	0.4	0.4	0.5	0.4	0.4	0.5	0.5	0.5	0.4	0.6
Ash	7.7	7.0	7.6	7.2	7.1	6.9	7.1	6.8	6.7	6.7	6.5	6.6	6.5	6.2
Oxygen(dif.)	16.2	17.3	18.1	18.2	17.0	17.7	18.4	17.7	17.9	17.3	18.3	18.0	19.3	17.1
Coal Conversion*		60.0	59.9			61.4	57.2			65.5		62.8		
Chlorine (ppm)														49
Pyritic Sulfur														0.05
Dry Heat Value (BTU/lb)														11,794
Mineral Analysis (wt % ash)														
Silica														28.8
Ferric Oxide														5.3
Alumina														18.1
Lime														26.2
Magnesia														4.5
Potassium Oxide														1.1
Sodium Oxide														0.7

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* Wt % THF Soluble Conversion after 30 minutes reaction at 750°F, obtained in the laboratory microautoclave tests.

Table 2

RECYCLE PROCESS SOLVENT ANALYTICAL DATA

Operating Period	V131B					
	CI-Free Resid, wt %			Distillate, wt %		
	C	H	N	C	H	N
263A	86.94	6.53	1.38	88.67	9.12	0.79
263B	87.16	7.10	1.31	88.71	9.07	0.86
263C	88.01	6.40	1.53	88.08	8.99	0.89
263D	88.49	6.60	1.51	88.53	8.96	0.92
263E	89.11	6.30	1.54	88.45	8.88	0.94
263F	89.70	6.56	1.60	88.80	8.54	1.11
263G	89.27	6.50	1.38	88.15	8.56	0.78
263H	88.86	6.29	1.53	88.19	8.62	0.96
263I	89.40	6.71	1.33	88.39	9.19	0.83
263J	90.28	6.81	1.20	89.89	9.20	0.73
263K	89.56	6.99	1.16	89.64	9.20	0.68
263L	89.39	6.57	1.15	89.12	9.21	0.56
263M	90.04	6.78	1.28	90.03	9.04	0.72

Table 3

Metal Analysis of Solid Deposits
(December 19 Shutdown and End-of-Run Samples)

Run Sample No. Description	263 27004 Solids in V1258	263 27005 Semi-Solids in V1258	263 30229 Solids in V1258	263 30230 Near Top Deposit in V1258	263 30231 Near Bottom Deposit in V1258
Ash, wt%	52.7	43.5	48.8	39.4	38.0
CI, wt%	61.3	52.9	45.3	68.1	80.4
<u>Metal Oxides, wt%</u>					
Aluminum	12.4	11.2	13.4	14.2	16.0
Calcium	7.7	13.1	15.1	15.4	15.9
Iron	21.7	22.1	19.8	23.2	19.3
Magnesium	1.7	2.7	3.1	3.2	4.0
Manganese	0.3	0.2	0.1	0.1	0.1
Molybdenum	0.1	0.2	0.2	0.2	0.3
Potassium	0.3	0.4	0.4	0.4	0.3
Silicon	42.6	31.3	31.2	33.7	29.1
Sodium	0.3	0.3	0.4	0.4	0.4
Sulfur Trioxide	6.6	12.7	14.4	11.3	13.6
Titanium	0.6	0.9	1.0	1.0	1.3

Table 3 (Continued)

Metal Analysis of Solid Deposits
(December 19 Shutdown and End-of-Run Samples)

Run	263	263	263
Sample No.	30232	30240	30241
Description	Suction Solids in P1222	Plenum Chamber Solids in R1235	Bottom Head Solids in R1235
Ash, wt%	52.7	57.4	51.2
CI, wt%	61.3	64.7	80.2
<u>Metal Oxides, wt%</u>			
Aluminum	17.6	9.5	10.8
Calcium	36.4	10.2	14.1
Iron	8.5	41.4	38.6
Magnesium	6.0	2.2	3.6
Manganese	0.2	0.4	0.7
Molybdenum	0.1	0.3	4.7
Potassium	0.2	0.1	0.2
Silicon	10.1	20.7	8.5
Sodium	0.4	0.1	0.3
Sulfur Trioxide	11.9	6.8	9.4
Titanium	1.8	0.9	1.8

Table 4

Solid Deposits from Runs 263 and 262

Run	Sample No.	Location	Wt % Ash	Sample CI	Total Wt., lbs
262	24807	R1235 bottom wall	55.7	90.6	
262	24804	R1235 bottom solids	53.4	73.4	37
262	24852	V1258 bottom solids	21.2	49.6	17
262	24852	V1258 solid chunk	10.5	80.7	
December 19-21 Shutdown:					
263	27590	R1235 first withdrawal	62.4	67.8	
263	27591	R1235 second withdrawal	36.3	41.9	
263	27005	V1258 solids	52.7	61.3	100
263	27006	V1258 gummy material	43.5	52.9	
End-of-Run Inspection:					
263	30229	V1258 material	48.8	45.3	150
263	30230	V1258 deposit near top	39.4	68.1	
263	30231	V1258 deposit near bottom	38.0	80.4	
263	30232	P1222 suction	53.7	84.3	
263	30241	R1235 bottom head	51.2	80.2	45
263	30240	R1235 plenum chamber	57.4	64.7	

Table 5

TSL Yields Before Elemental Balancing
(Phase 2 Data)

	Operating Period						
Yield, wt % MAF	263A	263B	263C	263D	263E	263F	263G
H_2 consumed	-4.5	-4.1	-4.5	-4.9	-5.3	-5.0	-5.0
CO, CO_2	6.3	6.3	6.1	6.2	5.5	5.9	5.7
Sour Water	15.4	15.8	15.8	16.8	15.3	15.6	16.7
H_2S	-0.1	-0.2	-0.2	-0.3	-0.2	-0.1	-0.2
C_1-C_3 gases	6.3	6.4	8.3	8.5	8.5	8.6	8.3
C_4+ distillate	47.5	54.9	50.3	52.1	59.1	57.8	54.2
C_4-C_6	2.5	3.0	3.4	3.1	3.3	2.4	3.0
IBP-350°F	7.8	8.0	9.8	9.0	7.8	9.2	9.2
350-450°F	8.4	9.3	11.5	10.1	10.3	10.0	9.5
450-750°F	26.9	26.4	33.4	27.3	36.3	20.8	37.0
750-850°F	18.0	20.0	11.8	19.0	15.9	11.6	26.4
850°F-EP	-16.0	-11.7	-19.6	-16.3	-14.6	3.7	-31.0
Int. Accum.(Res. Free)	4.0	4.2	-0.3	-0.5	-1.5	-1.9	0.3
Resid	6.6	4.1	6.2	1.9	1.2	1.8	2.0
Solid Product(Ash-free)	20.7	16.9	16.7	15.1	13.0	14.3	13.2
Closure	-2.2	-4.3	1.6	5.1	4.4	3.2	4.4
Resid+UC conv. wt % feed							
1st stage	17.2	17.8	19.5	21.1	23.5	19.9	20.2
2nd stage	9.1	11.6	11.4	13.2	9.7	14.0	13.8
Coal Conversions % MAF							
1st stage	81.1	83.5	87.5	86.8	89.8	90.8	88.6
TSL	88.9	89.4	93.6	93.0	93.9	95.4	93.3

Table 5 (Continued)

TSL Yields Before Elemental Balancing
(Phase 2 Data)

	Operating Period					
Yield, wt % MAF	263H	263I	263J	263K	263L	263M
H_2 consumed	-5.2	-5.9	-5.7	-5.5	-6.0	-5.7
CO, CO_2	4.0	6.4	6.2	5.0	7.4	7.2
Sour Water	15.1	19.3	18.7	21.7	18.8	18.7
H_2S	-0.6	-0.3	-0.3	-0.3	0.1	0.0
C_1-C_3 gases	8.4	7.3	7.9	7.3	7.7	7.1
C_4+ distillate	59.7	61.7	61.2	58.5	57.1	52.9
C_4-C_6	3.2	2.7	3.4	3.3	3.7	2.9
IBP-350°F	7.9	12.1	12.0	11.7	11.5	10.2
350-450°F	9.8	8.7	8.3	8.2	8.4	7.7
450-750°F	40.6	34.2	31.7	30.2	30.2	32.4
750-850°F	9.1	19.1	17.6	17.6	16.5	15.0
850°F-EP	-10.9	-15.1	-12.0	-12.6	-13.1	-15.2
Int. Accum.(Res. Free)	-0.3	-2.4	-2.3	2.0	-0.5	1.9
Resid	-1.7	-2.1	2.4	3.0	4.3	6.5
Solid Product(Ash-free)	13.5	13.5	13.2	13.4	15.9	14.5
Closure	7.1	2.5	-1.2	-5.0	-4.3	-3.1
Resid+UC conv. wt % feed						
1st stage	21.4	22.9	22.4	22.7	26.0	24.0
2nd stage	12.7	18.2	20.0	18.3	12.5	14.4
Coal Conversions % MAF						
1st stage	88.9	92.2	89.1	87.8	91.9	91.1
TSL	94.1	94.1	95.3	92.2	93.4	92.4

Table 6

Close-Coupled Reactors Operating Data

Operating period	feed rate MF lb/hr	First Stage Avg temp., (°F)	First Stage Bed exotherm (°F)	Second Stage Avg temp., (°F)	Second Stage Bed exotherm (°F)
263A	299	826	15	810	2.2
263B	299	825	15	809	2.2
263C	294	840	29	810	1.9
263D	260	840	27	810	1.7
263E	261	840	30	809	1.6
263F	254	840	27	808	1.5
263G	264	841	28	809	1.6
263H	264	840	32	806	1.6
263I	343	840	42	810	6.1
263J	345	840	50	810	6.8
263K	344	842	39	809	7.0
263L	345	842	38	809	5.8
263M	348	840	37	810	6.8

TABLE 7

SUMMARY OF ROSE-SR^{SM} PERFORMANCE PARAMETERS

Operating Period	DAS type	Resid recovery (wt %)	Energy rejection (wt %)	Organic rejection (wt %)	Bottoms product toluene insolubles (wt %)	FSI(a)	Ash consistency
263A	2334	68.7	26.6	19.6	79.8	0.63	Powder
263B	2304	70.7	21.9	16.9	83.6	0.67	Powder
263C	2394	73.2	20.8	16.7	80.3	0.68	Powder
263D	2404	72.5	19.6	15.0	80.8	0.71	Powder
263E	2414	75.9	18.0	13.0	79.9	0.68	Chunky
263F	2364	71.4	19.7	14.3	75.4	0.67	Extruded
263G	2424	75.5	17.9	13.2	80.8	0.71	Grainy
263H	2424	72.4	17.4	13.5	77.7	0.70	Gummy
263I	2404	74.8	17.4	13.5	80.7	0.71	Powder
263J	2394	74.0	17.0	13.4	80.7	0.71	Powder
263K	2354	68.1	19.8	13.7	78.2	0.68	Chunky
263L	2374	69.0	20.0	15.9	78.7	0.67	Extruded
263M	2364	70.8	18.3	14.5	81.5	0.66	Powder

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

Table 8

ROSE Unit Analytical Data - Run 263

Operating Period	263A	263B	263C	263D	263E	263F	263G	263H	263I	263J	263K	263L	263M
Feed Comp (Wt%)													
Ash	19.3	19.4	19.6	20.8	21.1	19.3	21.0	19.8	19.3	20.2	20.3	18.9	19.5
UC	14.2	12.7	12.2	13.3	12.5	10.6	11.8	12.5	15.3	15.1	16.4	16.4	16.4
Resid (a)	62.5	62.6	65.6	62.0	64.4	59.9	62.5	61.6	64.8	62.1	60.4	62.6	60.8
Solvent	4.0	3.7	2.6	3.9	2.0	10.2	4.7	6.0	0.7	2.6	2.9	2.1	3.3
Carbon	72.3	71.9	71.3	70.7	71.6	73.4	71.5	71.6	72.7	72.3	71.5	72.5	72.3
Hydrogen	5.3	5.3	5.0	5.1	5.0	5.0	4.9	5.0	5.1	5.1	5.0	5.1	5.1
Nitrogen	1.2	1.2	1.1	1.2	1.2	1.3	1.2	1.3	1.0	1.0	1.0	1.0	1.0
Sulfur	1.8	2.0	2.1	2.1	2.3	2.3	2.2	1.8	1.6	1.6	1.5	1.4	1.6
Oxygen	0.1	0.2	0.9	0.2	-1.2	-1.2	-0.7	0.5	0.3	-0.2	0.7	1.2	0.5
Preasphaltenes	8.8	5.6	6.4	4.2	11.7	7.9	5.9	6.6	4.7	4.2	5.2	4.1	
Soft Point (deg F)	185	188	187	181	202	130	180	194	190	179	194	189	205
Fusion Pt (deg F)	216	232	208	200	215	159	199	235	205	202	235	230	235
Bottoms Prod Comp (Wt%)													
Ash (b)	34.7	36.0	39.0	39.8	42.4	38.9	42.6	38.9	37.8	38.3	35.5	34.1	35.7
UC (b)	24.0	30.1	24.0	26.8	24.2	21.9	24.7	25.6	30.8	29.9	30.5	30.8	31.8
Resid (a) (b)	39.5	32.1	36.0	33.1	32.8	38.8	31.1	34.6	31.2	30.9	33.3	34.5	31.5
Solvent (b)	1.7	1.8	0.9	0.3	0.7	0.6	1.6	1.0	0.3	0.9	0.7	0.7	1.0
DAS	1.9	0.7	1.0	0.8	1.0	1.2	1.1	0.7	1.7	1.7	0.4	0.6	0.8
Carbon	59.5	56.3	54.7	55.2	54.0	55.8	54.1	55.2	55.5	55.9	57.3	59.1	57.3
Hydrogen	3.8	3.5	3.3	3.5	3.2	3.2	3.2	3.4	3.3	3.4	3.4	3.6	3.5
Nitrogen	1.1	1.0	1.0	1.0	0.9	1.0	0.9	1.0	0.9	0.8	0.9	0.9	0.9
Sulfur	3.0	3.6	3.8	3.8	4.3	3.9	4.1	3.5	3.0	2.9	2.6	2.4	2.6
Oxygen (a)	-2.1	-0.5	-1.7	-3.2	-4.9	-2.8	-4.9	-2.0	-0.5	-1.3	0.3	-0.2	-0.0
Recycle Resid Comp (Wt%)													
Ash (b)	0.1	0.1	0.4	1.1	1.2	0.5	1.6	0.6	0.7	1.1	0.8	0.6	0.5
UC (b)	0.4	0.2	0.3	0.4	0.7	0.5	0.2	0.5	1.1	1.0	0.6	0.5	0.3
Resid (a) (b)	86.0	82.6	90.8	90.2	86.0	80.9	91.2	86.1	91.9	91.0	88.3	89.7	90.0
Solvent (b)	13.5	17.1	8.6	8.3	12.1	18.0	7.0	12.8	6.4	6.9	10.4	9.1	9.2
DAS	0.2	0.3	4.4	1.4	0.9	1.2	0.7	0.3	1.1	0.9	0.5	0.9	0.5
Carbon	89.1	88.9	88.7	88.6	89.1	90.4	88.9	88.9	90.0	89.9	89.7	88.2	87.5
Hydrogen	7.3	7.3	6.7	6.9	6.8	6.6	6.5	6.7	6.8	6.9	7.0	6.8	6.8
Nitrogen	1.3	1.4	1.4	1.5	1.5	1.6	1.4	1.5	1.3	1.2	1.1	1.1	1.2
Sulfur	0.1	0.1	0.1	0.2	0.2	0.1	0.2	0.1	0.0	0.1	0.1	0.6	0.4
Oxygen	2.2	2.3	2.8	1.7	1.2	0.8	1.4	2.1	1.2	0.8	1.4	2.6	3.6
Soft Point (deg F)	128	123	122	145	140	72	144	150	159	135	135	139	141
Fusion Pt (deg F)	149	167	141	162	157	98	166	196	185	154	182	180	184

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

Table 9

T102 OPERATING CONDITIONS SUMMARY

RUN PERIOD	263A	263B	263C	263D	263E	263F	263G	263H	263I	263J	263K	263L	263M
T102 Tray 2 temp, F	449	452	463	463	470	474	474	475	470	475	474	472	478
T102 Tray 3 temp, F	492	487	487	489	487	488	486	490	495	490	485	489	499
T102 Top pack temp, F	555	546	539	538	543	546	537	538	548	539	536	538	545
T102 Tray 8 temp, F	582	583	583	583	586	590	585	586	586	583	581	583	588
T102 Bottom temp, F	624	622	624	625	625	623	624	622	624	626	625	626	627
T102 TR3 Reflux temp, F	235	236	238	236	236	233	240	232	239	237	228	235	238
T102 Top Pres, psia	1.6	1.6	1.7	1.9	2.0	2.5	2.4	2.2	1.5	1.9	2.2	1.8	1.8
T102 Reflux flow, LB/HR	642	620	640	639	626	626	626	623	626	632	631	631	629
B102 Inlet temp, F	610	609	612	612	614	612	613	611	613	615	614	614	617
B102 Outlet temp, F	672	670	669	670	669	663	671	671	670	677	680	680	678
B102 Process flow, GPM	23	22	24	24	24	23	23	23	23	21	20	20	21
GC CUT POINT, F	804	797	795	776	780	745	760	762	778	781	763	778	799
STD	12	14	23	18	27	6	13	15	15	14	9	3	16
GC EQUAL WT% OVERLAP	9	10	12	8	8	10	8	10	9	6	7	8	8

Table 10

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
263A	32.3	43.5	12.2	12.0	88.23	6.59	1.46	87.97	9.32	0.81
263B	34.2	43.3	10.1	12.4	87.87	6.75	1.44	87.60	9.09	0.89
263C	35.3	43.0	8.4	13.3	87.89	6.43	1.53	87.11	8.96	0.93
263D	37.2	40.8	9.1	12.9	87.72	6.48	1.47	88.68	9.07	0.96
263E	36.7	42.0	8.3	13.0	88.95	6.43	1.55	88.17	8.90	0.97
263F	32.1	44.6	10.3	13.6	89.20	6.31	1.58	88.60	8.70	1.06
263G	34.8	43.9	7.8	13.5	88.70	6.14	1.43	87.52	8.96	1.06
263H	35.4	43.3	8.9	12.4	88.43	6.09	1.55	86.96	8.88	0.98
263I	33.8	46.0	8.5	11.7	86.65	6.56	1.36	87.44	9.26	0.84
263J	35.5	43.5	9.5	11.5	89.17	6.76	1.27	88.50	9.26	0.81
263K	36.5	41.7	10.5	11.3	87.61	6.31	1.28	88.57	9.38	0.76
263L	38.5	40.9	9.8	10.8	88.28	6.32	1.28	87.98	8.96	0.70
263M	37.1	41.8	10.1	11.0	88.48	6.65	1.30	87.99	8.97	0.80

Table 11

ANALYTICAL DATA OF STREAMS USED IN THE ELEMENTAL MATERIAL BALANCES

Operating	Coal, wt % MF					
Period	C	H	N	S	O(a)	Ash
263A	69.1	5.7	1.1	0.4	16.5	7.2
263B	69.0	5.3	1.0	0.4	17.4	6.9
263C	67.3	5.3	1.0	0.4	18.7	7.3
263D	69.0	5.1	1.0	0.4	17.4	7.1
262E	68.7	5.5	1.1	0.4	17.3	7.0
262F	68.7	5.6	1.1	0.4	17.3	6.9
262G	68.1	5.5	1.1	0.5	17.5	7.3
263H	68.8	5.5	1.0	0.4	17.6	6.7
263I	68.8	5.4	1.0	0.5	17.7	6.6
263J	69.4	5.3	1.0	0.5	17.1	6.7
263K	68.5	5.6	1.0	0.5	17.9	6.5
262L	68.8	5.5	1.0	0.5	17.5	6.7
262M	70.2	5.5	1.0	0.5	16.1	6.7

Operating	Recycle Material (V1082), wt %					
Period	C	H	N	S	O(a)	Ash
263A	71.9	5.3	1.2	1.8	0.5	19.3
263B	71.7	5.3	1.2	2.0	0.3	19.5
263C	71.0	5.0	1.1	2.1	0.4	20.4
263D	70.7	5.1	1.2	2.1	1.5	19.4
263E	72.2	5.0	1.2	2.3	-	21.4
263F	72.8	4.9	1.2	2.2	-	20.1
263G	71.3	4.9	1.2	2.2	-	21.3
263H	72.0	5.0	1.3	1.8	-	20.0
263I	72.3	5.1	1.0	1.6	0.6	19.4
263J	72.2	5.1	1.0	1.7	-	20.4
263K	71.6	5.0	1.0	1.3	0.9	20.2
263L	72.5	5.1	1.0	1.4	1.1	18.9
263M	72.6	5.2	1.0	1.5	0.7	19.0

(a) Oxygen by difference.

Table 11 (Continued)

ANALYTICAL DATA OF STREAMS USED IN THE ELEMENTAL MATERIAL BALANCES

Operating Period	Flashed Distillate (V1078), wt %				
	C	H	N	S	O(a)
263A	84.4	10.4	0.8	0.04	4.4
263B	84.6	10.4	0.8	0.05	4.1
263C	84.0	10.3	0.8	0.04	4.9
263D	83.8	10.4	0.8	0.05	4.9
263E	84.7	10.5	0.8	0.04	4.0
263F	85.7	10.3	0.9	-	3.1
263G	84.4	10.3	0.9	-	4.4
263H	84.3	10.4	0.9	-	4.4
263I	86.3	11.5	0.6	-	1.6
263J	86.7	11.7	0.5	-	1.1
263K	86.1	11.5	0.6	-	1.8
263L	86.1	11.4	0.5	0.04	2.0
263M	86.0	11.3	0.6	0.02	2.1

Operating Period	T102 Bottoms (V1074) + Withdrawal Tube Flush, wt %				
	C	H	N	S	O(a)
263A	88.2	9.2	0.9	0.06	1.6
263B	88.3	9.1	0.9	0.05	1.6
263C	87.9	9.1	0.9	0.05	2.0
263D	88.6	9.0	1.0	0.05	1.3
263E	88.4	8.8	0.9	0.04	1.9
263F	88.2	8.9	0.9	-	2.0
263G	87.9	8.9	0.9	-	2.3
263H	88.2	8.8	1.1	-	1.9
263I	89.0	9.3	0.8	-	0.9
263J	88.4	9.4	0.8	-	1.4
263K	89.6	9.3	0.7	-	0.4
263L	89.4	9.1	0.7	0.02	0.8
263M	89.6	9.1	0.8	0.00	0.5

(a) Oxygen by difference.

Table 11 (continued)

ANALYTICAL DATA OF STREAMS USED IN THE ELEMENTAL MATERIAL BALANCES

Operating Period	Vaccum Flash Overhead (V1072), wt %				
	C	H	N	S	O(a)
263A	88.2	9.1	0.9	0.03	1.8
263B	88.3	9.2	0.9	0.03	1.6
263C	87.5	8.8	0.9	0.05	2.6
263D	88.3	9.0	1.0	0.05	1.6
263E	88.2	8.9	1.0	0.04	1.9
263F	87.5	8.9	1.1	0.00	2.5
263G	88.0	8.9	0.9	-	2.2
263H	87.7	8.9	1.1	-	2.3
263I	89.2	9.5	0.7	-	0.6
263J	89.7	9.4	0.7	-	0.2
263K	89.5	9.4	0.7	-	0.4
263L	89.5	9.3	0.7	0.03	0.5
263M	89.3	9.4	0.8	0.00	0.5

Operating Period	ROSE-SR^{SM} Resid, wt %					
	C	H	N	S	O(a)	Ash
263A	89.0	7.3	1.3	0.07	2.1	0.2
263B	88.9	7.3	1.4	0.07	1.9	0.4
263C	88.3	6.8	1.4	0.09	2.0	1.4
263D	88.6	6.9	1.5	0.18	1.3	1.5
263E	89.2	6.7	1.5	0.23	1.5	0.9
263F	90.4	6.6	1.6	0.07	0.8	0.5
263G	88.8	6.5	1.4	0.23	1.5	1.6
263H	88.9	6.7	1.5	0.12	2.1	0.7
263I	90.0	6.8	1.3	0.04	1.4	0.5
263J	90.0	6.9	1.2	0.13	0.6	1.2
263K	89.7	7.0	1.1	0.12	1.3	0.8
263L	88.4	6.9	1.1	0.61	0.1	2.9
263M	87.5	6.8	1.2	0.28	0.2	4.0

(a) Oxygen by difference.

Table 12

Run 263 Sour Water Analysis

Period	1st Stage V105 Water (mg/liter)	2nd Stage V1080 water (mg/liter)
Kjeldahl nitrogen	9,016	2,217
Total organic carbon	21,755	8,763
Inorganic carbon	6,338	2,074
Phenols	5,850	6,800
Sulfide sulfur	5	14
Chloride	<25	<25

Note: Analysis data were for period 263A in the thermal/thermal mode of operation with addition of Molyvan L as a dispersed catalyst.

Table 13

COMPARISON OF CATALYST PROPERTIES

	Criterion 324 (Shell 324)	Shell 317	Amocat 1C	Amocat 1C	EXP-AO-60
Run	256,259,261 262, 263	253,254,256	257,259	251-252	261
Support	Unimodal	Bimodal	Bimodal	Bimodal	Bimodal
Size, inches	1/16	1/20	1/12	1/16	1/16
Shape	Cylindrical	Trilobe	Cylindrical	Cylindrical	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
Half-volume	170	..	110	..	..

Table 14

R1236 Catalyst Age and Analytical Data

Date	Operation Day	Description	Catalyst Age R+CI	Catalyst Age MF Coal	<-----As Is-----> S	Ash (wt %)	Oil	<-----THF Extracted-----> C	H	N (wt %)	S	Ash	Naph (%)	14 <------Screen Size------>	18	25	-25
18-Jan-92	Run 262	Aged Catalyst	1522	736	5.7	70.1	20.8	10.8	0.8	0.2	6.7	87.1	38	84.9	10.0	1.8	3.4
18-Jan-92	"	"			5.8	74.3	16.9	10.2	0.7	0.2	7.2	87.9	17	97.8	1.3	0.3	0.6
18-Jan-92	"	"			6.1	72.7	19.0	9.1	0.7	0.2	7.3	87.7	44	97.6	1.1	0.1	1.3
21-Jan-92	Presulfided	Before	0	0	6.6	69.0	23.9	1.2	0.6	0.2	6.9	89.3	126	99.4	0.5	0.0	0.1
23-Jan-92	74	1st A/W	1401	675	6.2	71.2	18.3	11.6	0.9	0.2	7.4	85.1	32	99.2	0.2	0.2	0.3
25-Jan-92	76	2nd A/W	1354	652	5.8	71.0	17.6	12.1	0.9	0.2	6.9	86.4	37	99.2	0.6	0.0	0.3
27-Jan-92	78	3rd A/W	1337	646	6.1	71.9	17.0	13.1	0.9	0.2	7.4	83.8	20	99.4	0.3	0.0	0.3
29-Jan-92	80	4th A/W	1313	637	6.2	73.2	16.8	13.0	0.8	0.2	7.5	84.4	25	99.2	0.4	0.1	0.3
31-Jan-92	82	5th A/W	1298	634	5.8	73.5	16.0	13.4	0.8	0.2	7.6	85.2	23	98.7	0.7	0.3	0.5
02-Feb-92	84	6th A/W	1365	671	5.8	70.8	15.7	12.9	0.8	0.2	7.2	83.9	24	98.7	1.0	0.0	0.3
04-Feb-92	86	7th A/W	1255	618	6.0	72.1	15.6	13.0	0.8	0.2	7.1	88.1	16	98.6	1.2	0.0	0.1
06-Feb-92	88	8th A/W	1250	616	5.9	77.8	15.6	13.6	0.8	0.2	6.9	84.6	22	98.1	1.8	0.0	0.1
08-Feb-92	90	9th A/W	1241	615	5.9	79.9	15.6	13.0	0.8	0.2	7.0	85.4	23	96.9	2.8	0.0	0.3
10-Feb-92	92	10th A/W	1427	712	6.1	73.2	14.5	14.4	0.9	0.2	7.0	84.2		97.8	2.1	0.0	0.1
12-Feb-92	94	11th A/W	1516	761	6.1	73.9	14.8	13.2	0.9	0.2	6.8	85.6		96.6	3.1	0.2	0.2
14-Feb-92	96	12th A/W	1745	883	5.8	73.0	15.5	13.4	0.9	0.2	6.9	82.8	30	93.2	6.6	0.2	0.0
16-Feb-92	98	13th A/W	1932	981	5.8	77.4	12.9	12.4	0.8	0.2	6.8	79.4		89.4	9.1	1.0	0.5
17-Feb-92	99	14th A/W	1836	932	6.1	69.9	12.8	14.3	0.9	0.2	6.7	80.8	29	95.4	4.3	0.2	0.0
19-Feb-92	101	15th A/W	1836	934	5.9	77.9	14.1	12.2	0.8	0.2	6.9	84.6	43	91.9	7.1	0.6	0.4
21-Feb-92	103	16th A/W	1834	935	5.9	75.7	15.0	12.0	0.8	0.2	6.8	83.8	34	93.9	5.8	0.0	0.3
23-Feb-92	104	17th A/W	1922	983	5.8	70.4	16.1	12.3	0.9	0.2	6.7	84.4	32	90.1	7.3	0.8	1.8
28-Feb-92		End of Run Composite				66.2	16.6					77.6	65.0	76.7	16.1	5.0	2.1

Table 15

Metal Analysis of Second Stage (R1236) Criterion 324 Catalyst
(Wt % Metal Oxides)

Sample No.	28360	28710	29087	29439	29752	30048
Date, 1992	10/8/91	Presul.	2/2	2/8	2/14	2/21
Period	262 End	Before	263J	263K	263L	263M
Metal Oxides						
Aluminum	67.8	71.4	63.8	65.7	67.1	62.8
Calcium	0.2	0.4	0.4	0.4	0.4	0.4
Iron	0.2	0.1	0.1	0.1	0.1	0.1
Magnesium	0.2	0.1	0.2	0.2	0.2	0.2
Molybdenum	17.4	17.5	16.9	17.3	16.4	16.8
Nickel	3.3	3.5	3.3	3.5	3.3	3.4
Titanium	0.1	0.1	0.1	0.1	0.1	0.1
Silicon	0.1	0.2	0.1	0.2	0.2	0.2

Table 16

TSL SYSTEM RESPONSES IN RESID + UC CONVERSION, EXCESS RESID, AND COAL CONVERSION
Run 263 with Black Thunder Coal, Oct. 31, 1991 - Feb. 23, 1992
Phase 2 data

unit: wt % MAF coal

Date	Days	resid+UC conversion First	Second	Overall	TSL resid	Org.Rej.	Resid w/ OR=15%	Resid w/ OR=20%	coal conversion First	Second
02-Nov-91	2	65.3	8.6	73.9	5.1	21.0	11.1	6.1	91.4	94.9
03-Nov-90	3	52.7	26.4	79.1	0.4	20.5	5.9	0.9	84.3	94.6
04-Nov-91	4	56.2	13.4	69.6	8.2	22.2	15.4	10.4	84.0	88.4
05-Nov-91	5	52.3	9.3	61.6	15.8	22.6	23.4	18.4	87.7	83.9
06-Nov-91	6	43.1	29.1	72.2	7.5	20.3	12.8	7.8	85.5	86.2
07-Nov-91	7	44.6	42.1	86.7	-7.0	21.0	-1.0	-6.0	83.6	88.4
08-Nov-91	8	42.0	20.5	62.5	16.7	20.8	22.5	17.5	85.0	86.7
09-Nov-91	9	42.2	21.8	64.0	13.1	22.9	21.0	16.0	80.3	83.3
10-Nov-91	10	60.4	8.1	68.5	10.4	21.1	16.5	11.5	82.7	89.9
11-Nov-91	11	52.8	22.2	75.0	1.4	23.6	10.0	5.0	81.3	87.9
12-Nov-91	12	40.1	21.9	62.0	17.4	20.6	23.0	18.0	81.9	89.2
13-Nov-91	13	38.0	29.5	67.5	12.7	19.8	17.5	12.5	78.3	88.3
14-Nov-91	14	46.5	23.9	70.4	11.2	18.4	14.6	9.6	81.3	89.1
15-Nov-91	15	44.9	37.9	82.8	1.3	15.9	2.2	-2.8	81.2	90.9
16-Nov-91	16	39.8	28.8	68.6	16.2	15.2	16.4	11.4	85.3	87.9
17-Nov-91	17	38.0	38.8	76.8	7.1	16.1	8.2	3.2	84.6	89.3
18-Nov-91	18	45.2	23.9	69.1	15.2	15.7	15.9	10.9	80.5	85.6
19-Nov-91	19	48.0	26.6	74.6	9.9	15.5	10.4	5.4	81.8	89.3
20-Nov-91	20	47.2	25.3	72.5	7.6	17.9	10.5	5.5	85.8	90.9
21-Nov-91	21	51.9	25.4	77.3	5.9	16.8	7.7	2.7	82.6	91.6
22-Nov-91	22	57.4	18.6	76.0	6.1	17.9	9.0	4.0	83.8	89.8
28-Nov-91	23	67.7	27.5	95.2	-13.6	18.4	-10.2	-15.2	87.8	92.5
29-Nov-91	24	54.3	31.6	85.9	-4.1	18.2	-0.9	-5.9	88.3	95.6
30-Nov-91	25	54.0	34.6	88.6	-6.5	17.9	-3.6	-8.6	86.5	92.5
03-Dec-91	26	59.9	14.9	74.8	8.7	16.5	10.2	5.2	85.0	94.1
04-Dec-91	27	62.9	23.0	85.9	-0.9	15.0	-0.9	-5.9	91.3	94.1
05-Dec-91	28	53.5	34.2	87.7	-2.0	14.3	-2.7	-7.7	84.8	93.3
06-Dec-91	29	51.5	35.4	86.9	-3.0	16.1	-1.9	-6.9	92.6	96.0
07-Dec-91	30	49.2	19.5	68.7	13.7	17.6	16.3	11.3	88.6	91.5
08-Dec-91	31	49.7	28.8	78.5	4.9	16.6	6.5	1.5	86.8	94.8
09-Dec-91	32	55.2	22.8	78.0	5.2	16.8	7.0	2.0	84.5	91.8
10-Dec-91	33	56.0	37.4	93.4	-9.9	16.5	-8.4	-13.4	85.6	95.7
11-Dec-91	34	62.7	34.0	96.7	-12.7	16.0	-11.7	-16.7	88.2	95.1
12-Dec-91	35	43.7	34.4	78.1	3.3	18.6	6.9	1.9	86.7	90.3
13-Dec-91	36	52.8	32.1	84.9	-0.4	15.5	0.1	-4.9	86.9	92.3
14-Dec-91	37	56.6	37.3	93.9	-8.7	14.8	-8.9	-13.9	88.7	91.8
15-Dec-91	38	51.3	29.0	80.3	4.0	15.7	4.7	-0.3	84.5	91.9
16-Dec-91	39	54.9	15.5	70.4	15.3	14.3	14.6	9.6	87.9	95.4
17-Dec-91	40	60.2	28.3	88.5	-3.0	14.5	-3.5	-8.5	86.0	93.6
18-Dec-91	41	54.3				13.6			85.3	91.3

Table 16 (continued)

23-Dec-91	43	57.8	36.7	94.5	-6.6	12.1	-9.5	-14.5	93.4	98.0
24-Dec-91	44	61.4	23.4	84.8	3.1	12.1	0.2	-4.8	89.7	94.6
25-Dec-91	45	38.1	42.4	80.5	4.0	15.5	4.5	-0.5	84.7	93.3
26-Dec-91	46	64.1	21.4	85.5	1.2	13.3	-0.5	-5.5	89.0	93.9
27-Dec-91	47	58.2	29.6	87.8	-1.1	12.3	-3.8	-8.8	90.2	91.6
28-Dec-91	48	69.4	20.7	90.1	-3.6	13.5	-5.1	-10.1	88.8	96.6
31-Dec-91	51	62.8	29.1	91.9	-4.4	12.5	-6.9	-11.9	93.5	93.0
01-Jan-92	52	53.7	26.7	80.4	6.3	13.3	4.6	-0.4	87.5	94.2
03-Jan-92	54	62.4	10.2	72.6	12.1	15.3	12.4	7.4	90.3	100.1
04-Jan-92	55	64.9	24.9	89.8	-3.6	13.8	-4.8	-9.8	94.8	97.6
05-Jan-92	56	46.4	44.9	91.3	-5.8	14.5	-6.3	-11.3	92.1	95.2
06-Jan-92	57	51.5	24.6	76.1	9.3	14.6	8.9	3.9	85.4	93.3
08-Jan-92	59	46.7	34.8	81.5	4.7	13.8	3.5	-1.5	88.2	94.9
09-Jan-92	60	45.8	42.7	88.5	-1.9	13.4	-3.5	-8.5	86.0	94.0
10-Jan-92	61	49.7	35.4	85.1	2.0	12.9	-0.1	-5.1	84.9	91.6
11-Jan-92	62	57.6	21.0	78.6	8.9	12.5	6.4	1.4	92.3	93.7
12-Jan-92	63	57.3	28.1	85.4	1.6	13.0	-0.4	-5.4	89.1	91.2
13-Jan-92	64	62.1	22.8	84.9	1.0	14.1	0.1	-4.9	90.9	96.2
14-Jan-92	65	55.0	30.6	85.6	0.2	14.2	-0.6	-5.6	89.9	95.8
15-Jan-92	66	56.3	46.8	97.1	-8.6	11.5	-12.1	-17.1	88.4	94.9
16-Jan-92	67	56.5	29.0	85.5	1.8	12.7	-0.5	-5.5	92.0	92.2
17-Jan-92	68	57.5	31.6	89.1	-2.1	13.0	-4.1	-9.1	89.7	92.6
18-Jan-92	69	65.8	11.8	77.6	9.1	13.3	7.4	2.4	88.3	96.5
19-Jan-92	70	65.4	28.0	93.4	-8.0	14.6	-8.4	-13.4	85.9	93.3
20-Jan-92	71	46.8	50.3	97.1	-10.8	13.7	-12.1	-17.1	88.4	96.0
21-Jan-92	72									
22-Jan-92	73	47.6	40.5	88.1	-1.6	13.5	-3.1	-8.1	89.6	92.3
23-Jan-92	74	52.0	37.8	89.8	-3.3	13.5	-4.8	-9.8	95.1	92.8
25-Jan-92	76	59.0	42.7	101.7	-14.6	12.9	-16.7	-21.7	92.6	95.3
26-Jan-92	77	55.6	33.8	89.4	-3.3	13.9	-4.4	-9.4	90.5	94.0
27-Jan-92	78	52.1	30.9	83.0	3.4	13.6	2.0	-3.0	90.7	94.3
28-Jan-92	79	58.5	29.2	87.7	-2.7	15.0	-2.7	-7.7	91.0	94.9
29-Jan-92	80	55.6	28.6	84.2	2.5	13.3	0.8	-4.2	94.8	96.7
30-Jan-92	81	50.8	34.8	85.6	1.0	13.4	-0.6	-5.6	88.1	91.0
31-Jan-92	82	50.1	38.4	88.5	-1.2	12.7	-3.5	-8.5	88.3	95.7
01-Feb-92	83	56.8	28.7	85.5	1.5	13.0	-0.5	-5.5	83.7	95.2
03-Feb-92	85	38.7	51.0	89.7	-3.4	13.7	-4.7	-9.7	90.4	97.8
04-Feb-92	86	46.1	38.1	84.2	2.2	13.6	0.8	-4.2	88.4	92.0
05-Feb-92	87	37.1	32.0	69.1	17.6	13.3	15.9	10.9	87.9	90.9
06-Feb-92	88	45.2	35.4	80.6	6.4	13.0	4.4	-0.6	87.5	90.6
07-Feb-92	89	46.3	35.7	82.0	4.9	13.1	3.0	-2.0	84.6	91.9
08-Feb-92	90	55.8	26.2	82.0	3.7	14.3	3.0	-2.0	89.9	91.7
09-Feb-92	91	53.3	28.7	82.0	4.8	13.2	3.0	-2.0	89.1	94.5
10-Feb-92	92	54.5	24.6	79.1	6.4	14.5	5.9	0.9	91.0	91.9
11-Feb-92	93	61.2	18.0	79.2	6.1	14.7	5.8	0.8	94.0	93.9
14-Feb-92	95	59.6	20.3	79.9	3.6	16.5	5.1	0.1	92.0	94.2
15-Feb-92	96	55.9	25.3	81.2	2.8	16.0	3.8	-1.2	90.1	94.2
16-Feb-92	97	57.1	26.0	83.1	0.5	16.4	1.9	-3.1	91.5	91.4
17-Feb-92	98	50.6	27.0	77.6	8.4	14.0	7.4	2.4	91.6	93.4
18-Feb-92	99	50.4	29.7	80.1	4.0	14.9	3.9	-1.1	91.1	92.8
19-Feb-92	100	51.8	26.1	77.9	9.2	12.9	7.1	2.1	91.0	93.5
20-Feb-92	101	46.2	22.6	68.8	16.8	14.4	16.2	11.2	89.1	90.4
21-Feb-92	102	58.0	18.9	76.9	8.0	15.1	8.1	3.1	91.6	92.1
22-Feb-92	103	60.3	20.6	80.9	4.0	15.1	4.1	-0.9	92.7	93.3

Table 16 (continued)

PERIOD 263A : November 10-14									
Average	47.6	21.1	68.7	10.6	20.7	16.3	11.3	81.1	88.9
Stan. Dev. (σn)	8.3	7.1	4.2	5.2	1.7	4.2	4.2	1.5	0.7
Stan. Dev. (σn-1)	9.2	7.9	4.7	5.8	1.9	4.7	4.7	1.7	0.8
PERIOD 263B : November 17-22									
Average	47.9	26.4	74.4	8.6	16.7	10.3	5.3	83.2	89.4
Stan. Dev. (σn)	5.9	6.1	2.8	3.2	1.0	2.7	2.7	1.8	1.9
Stan. Dev. (σn-1)	6.5	6.7	3.1	3.5	1.1	3.0	3.0	1.9	2.1
PERIOD 263C : December 3-9 including Dec. 4 & 5									
Average	54.6	25.5	80.1	3.8	16.1	4.9	-0.1	87.7	93.7
Stan. Dev. (σn)	4.8	7.0	6.6	5.7	1.0	6.6	6.6	3.0	1.5
Stan. Dev. (σn-1)	5.2	7.6	7.1	6.2	1.1	7.1	7.1	3.3	1.6
PERIOD 263D : December 13-17									
Average	55.2	28.4	83.6	1.4	15.0	1.4	-3.6	86.8	93.0
Stan. Dev. (σn)	3.1	7.2	8.0	8.1	0.5	8.0	8.0	1.5	1.4
Stan. Dev. (σn-1)	3.5	8.1	8.9	9.0	0.6	8.9	8.9	1.6	1.5
PERIOD 263E : December 26 - January 1									
Average	61.6	25.5	87.1	-0.3	13.0	-2.3	-7.3	89.8	93.9
Stan. Dev. (σn)	5.3	3.8	4.0	3.9	0.5	4.1	4.1	2.0	1.6
Stan. Dev. (σn-1)	6.0	4.2	4.5	4.3	0.5	4.5	4.5	2.3	1.8
PERIOD 263F : January 4-6									
Average	54.3	31.5	85.7	-0.0	14.3	-0.7	-5.7	90.8	95.4
Stan. Dev. (σn)	7.8	9.5	6.8	6.7	0.4	6.8	6.8	4.0	1.8
Stan. Dev. (σn-1)	9.6	11.6	8.4	8.2	0.4	8.4	8.4	4.8	2.2
PERIOD 263G : January 9-13									
Average	54.5	30.0	84.5	2.3	13.2	0.5	-4.5	88.6	93.3
Stan. Dev. (σn)	5.9	8.1	3.2	3.6	0.5	3.2	3.2	2.8	1.8
Stan. Dev. (σn-1)	6.6	9.0	3.6	4.0	0.6	3.6	3.6	3.1	2.0
PERIOD 263H : January 16-20									
Average	58.4	30.1	88.5	-2.0	13.5	-3.5	-8.5	88.9	94.1
Stan. Dev. (σn)	7.0	12.3	6.7	7.1	0.7	6.7	6.7	2.0	1.8
Stan. Dev. (σn-1)	7.8	13.7	7.5	7.9	0.7	7.5	7.5	2.2	2.0
PERIOD 263I : January 23-27									
Average	54.7	36.3	91.0	-4.5	13.5	-6.0	-11.0	92.2	94.1
Stan. Dev. (σn)	2.9	4.4	6.8	6.5	0.4	6.8	6.8	1.9	0.9
Stan. Dev. (σn-1)	3.3	5.1	7.8	7.5	0.4	7.8	7.8	2.1	1.0
PERIOD 263J : January 29 - February 3									
Average	50.4	36.3	86.7	0.1	13.2	-1.7	-6.7	89.1	95.3
Stan. Dev. (σn)	6.4	8.2	2.1	2.1	0.3	2.1	2.1	3.6	2.3
Stan. Dev. (σn-1)	7.2	9.2	2.3	2.4	0.4	2.3	2.3	4.0	2.6
PERIOD 263K : February 6-9									
Average	50.2	31.5	81.7	5.0	13.4	3.4	-1.7	87.8	92.2
Stan. Dev. (σn)	4.5	4.1	0.6	1.0	0.5	0.6	0.6	2.0	1.4
Stan. Dev. (σn-1)	5.2	4.8	0.7	1.1	0.6	0.7	0.7	2.3	1.7
PERIOD 263L : February 11-16									
Average	58.5	22.4	80.9	3.3	15.9	4.1	-0.9	91.9	93.4
Stan. Dev. (σn)	2.1	3.4	1.5	2.0	0.7	1.5	1.5	1.4	1.2
Stan. Dev. (σn-1)	2.4	3.9	1.7	2.3	0.8	1.7	1.7	1.6	1.4
PERIOD 263M : February 18-22									
Average	53.3	23.6	76.9	8.4	14.5	7.9	2.9	91.1	92.4
Stan. Dev. (σn)	5.1	3.9	4.3	4.7	0.8	4.5	4.5	1.2	1.1
Stan. Dev. (σn-1)	5.7	4.3	4.8	5.2	0.9	5.0	5.0	1.3	1.3

Table 17

TSL SYSTEM RESPONSES IN RESID+UC CONVERSION AND EXCESS RESID
Run 263 with Black Thunder Coal, Nov. 10, 1991 - Feb. 22, 1992
Phase 3 Data

unit: wt % MAF coal

Date	Days	resid+UC conversion First	Second	Overall	TSL resid	Org.Rej.	Resid w/OR=15%	w/OR=20%
10-Nov-91	10	60.5	6.6	67.2	11.8	21.0	17.8	12.8
11-Nov-91	11	56.3	19.5	75.8	0.4	23.9	9.3	4.3
12-Nov-91	12	41.8	25.9	67.8	11.3	21.0	17.3	12.3
13-Nov-91	13	37.3	28.4	65.9	14.2	19.9	19.1	14.1
14-Nov-91	14	47.6	19.1	66.7	12.8	20.4	18.2	13.2
17-Nov-91	17	37.8	35.1	72.8	8.8	16.4	10.2	5.2
18-Nov-91	18	44.4	26.6	71.1	12.7	16.3	14.0	9.0
20-Nov-91	20	49.4	25.5	74.9	7.0	18.1	10.1	5.1
21-Nov-91	21	41.0	35.5	76.5	7.2	16.3	8.5	3.5
22-Nov-91	22	36.5	35.5	72.0	10.1	17.9	13.0	8.0
03-Dec-91	26	52.9	24.7	77.6	4.3	18.1	7.4	2.4
06-Dec-91	29	56.4	17.0	73.3	9.3	17.4	11.7	6.7
07-Dec-91	30	49.3	24.8	74.1	8.8	17.1	10.9	5.9
08-Dec-91	31	53.2	22.5	75.6	7.4	17.0	9.4	4.4
09-Dec-91	32	55.3	21.0	76.3	5.6	18.1	8.7	3.7
13-Dec-91	36	49.6	27.4	77.1	7.2	15.8	8.0	3.0
14-Dec-91	37	52.8	30.3	83.1	0.4	16.5	1.9	-3.1
15-Dec-91	38	48.7	24.1	72.8	11.0	16.2	12.2	7.2
16-Dec-91	39	51.3	27.3	78.7	6.5	14.8	6.3	1.3
17-Dec-91	40	57.2	23.8	80.9	3.4	15.7	4.1	-0.9
26-Dec-91	46	65.0	19.7	84.7	0.8	14.5	0.3	-4.7
27-Dec-91	47	60.7	22.3	83.0	2.6	14.4	2.0	-3.0
28-Dec-91	48	64.5	20.0	84.5	1.8	13.7	0.5	-4.5
31-Dec-91	51	60.1	23.3	83.4	2.8	13.8	1.6	-3.4
01-Jan-92	52	60.2	23.4	83.6	2.2	14.3	1.5	-3.5
04-Jan-92	55	63.8	18.9	82.7	2.4	14.9	2.3	-2.7
05-Jan-92	56	61.1	26.2	87.3	-0.9	13.5	-2.4	-7.4
06-Jan-92	57	67.8	20.3	88.1	-3.2	15.1	-3.1	-8.1
09-Jan-92	60	63.8	18.8	82.6	3.8	13.6	2.4	-2.6
10-Jan-92	61	62.1	19.9	82.0	4.6	13.4	3.0	-2.0
11-Jan-92	62	59.5	21.7	81.3	5.1	13.6	3.7	-1.3
12-Jan-92	63	59.2	23.7	82.9	3.3	13.9	2.2	-2.8
13-Jan-92	64	60.0	19.7	79.8	5.0	15.3	5.3	0.3
16-Jan-92	67	62.2	20.1	82.3	4.6	13.1	2.7	-2.3
17-Jan-92	68	59.2	26.3	85.5	1.1	13.4	-0.5	-5.5
18-Jan-92	69	63.1	20.5	83.6	2.7	13.8	1.5	-3.5
19-Jan-92	70	63.1	19.1	82.2	3.4	14.4	2.8	-2.2
20-Jan-92	71	60.8	24.7	85.5	0.2	14.4	-0.4	-5.4

Table 17 (continued)

23-Jan-92	74	49.6	35.3	84.8	0.8	14.4	0.2	-4.8
25-Jan-92	76	61.4	25.8	87.2	-0.3	13.1	-2.2	-7.2
26-Jan-92	77	57.9	27.6	85.6	1.1	13.4	-0.5	-5.5
27-Jan-92	78	53.4	29.8	83.1	3.1	13.8	1.9	-3.1
29-Jan-92	80	53.1	29.1	82.2	4.5	13.3	2.8	-2.2
30-Jan-92	81	52.0	28.0	80.1	6.8	13.1	4.9	-0.1
31-Jan-92	82	51.0	31.5	82.5	4.4	13.1	2.5	-2.5
01-Feb-92	83	50.9	34.1	84.9	1.5	13.6	0.1	-4.9
03-Feb-92	85	52.1	30.4	82.5	3.7	13.7	2.4	-2.6
06-Feb-92	88	43.8	40.4	84.2	2.4	13.4	0.8	-4.2
07-Feb-92	89	48.3	33.3	81.6	4.8	13.6	3.4	-1.6
08-Feb-92	90	49.9	31.3	81.2	5.4	13.4	3.8	-1.2
09-Feb-92	91	49.4	30.5	80.0	6.5	13.5	5.0	0.0
11-Feb-92	93	55.2	26.2	81.4	3.7	14.9	3.6	-1.4
14-Feb-92	95	59.8	19.8	79.6	4.4	16.0	5.4	0.4
15-Feb-92	96	54.3	24.3	78.6	5.1	16.3	6.4	1.4
16-Feb-92	97	56.4	26.4	82.8	1.0	16.2	2.2	-2.8
18-Feb-92	99	55.6	23.8	79.4	5.7	14.8	5.5	0.5
19-Feb-92	100	54.1	27.4	81.5	4.5	14.0	3.5	-1.5
20-Feb-92	101	55.5	25.0	80.5	4.9	14.5	4.4	-0.6
21-Feb-92	102	57.0	22.3	79.3	5.3	15.4	5.7	0.7
22-Feb-92	103	57.8	21.9	79.7	5.1	15.2	5.3	0.3

Table 17 (continued)

PERIOD 263A : November 10-14							
Average	48.7	19.9	68.7		10.1	21.2	16.3	11.3
Stan. Dev. (σn)	8.7	7.6	3.6		5.0	1.4	3.6	3.6
Stan. Dev. (σn-1)	9.7	8.5	4.0		5.5	1.6	4.0	4.0
PERIOD 263B : November 17-22								
Average	41.8	31.6	73.5		9.2	17.0	11.2	6.2
Stan. Dev. (σn)	4.7	4.6	2.0		2.1	0.8	2.0	2.0
Stan. Dev. (σn-1)	5.2	5.1	2.2		2.3	0.9	2.3	2.3
PERIOD 263C : December 3-9								
Average	53.4	22.0	75.4		7.1	17.5	9.6	4.6
Stan. Dev. (σn)	2.4	2.9	1.5		1.9	0.5	1.5	1.5
Stan. Dev. (σn-1)	2.7	3.2	1.7		2.1	0.5	1.7	1.7
PERIOD 263D : December 13-17								
Average	51.9	26.6	78.5		5.7	15.8	6.5	1.5
Stan. Dev. (σn)	3.0	2.4	3.5		3.6	0.6	3.5	3.5
Stan. Dev. (σn-1)	3.3	2.7	3.9		4.0	0.6	3.9	3.9
PERIOD 263E : December 26 - January 1								
Average	62.1	21.7	83.8		2.0	14.1	1.2	-3.8
Stan. Dev. (σn)	2.2	1.6	0.7		0.7	0.3	0.7	0.7
Stan. Dev. (σn-1)	2.4	1.8	0.7		0.8	0.4	0.7	0.7
PERIOD 263F : January 4-6								
Average	64.2	21.8	86.0		-0.6	14.5	-1.1	-6.1
Stan. Dev. (σn)	2.8	3.2	2.4		2.3	0.7	2.4	2.4
Stan. Dev. (σn-1)	3.4	3.9	2.9		2.8	0.9	2.9	2.9
PERIOD 263G : January 9-13								
Average	60.9	20.8	81.7		4.4	14.0	3.3	-1.7
Stan. Dev. (σn)	1.8	1.7	1.1		0.7	0.7	1.1	1.1
Stan. Dev. (σn-1)	2.0	2.0	1.2		0.8	0.8	1.3	1.3
PERIOD 263H : January 16-20								
Average	61.7	22.1	83.8		2.4	13.8	1.2	-3.8
Stan. Dev. (σn)	1.5	2.8	1.5		1.6	0.5	1.4	1.4
Stan. Dev. (σn-1)	1.7	3.2	1.6		1.8	0.6	1.6	1.6
PERIOD 263I : January 23-27								
Average	55.6	29.6	85.2		1.2	13.7	-0.2	-5.2
Stan. Dev. (σn)	4.5	3.6	1.5		1.2	0.5	1.5	1.5
Stan. Dev. (σn-1)	5.2	4.1	1.7		1.4	0.6	1.7	1.7
PERIOD 263J : January 29 - February 3								
Average	51.8	30.6	82.4		4.2	13.4	2.5	-2.5
Stan. Dev. (σn)	0.8	2.1	1.5		1.7	0.2	1.5	1.5
Stan. Dev. (σn-1)	0.9	2.4	1.7		1.9	0.3	1.7	1.7
PERIOD 263K : February 6-9								
Average	47.9	33.9	81.8		4.8	13.5	3.3	-1.8
Stan. Dev. (σn)	2.4	3.9	1.5		1.5	0.1	1.5	1.5
Stan. Dev. (σn-1)	2.8	4.5	1.8		1.7	0.1	1.8	1.8
PERIOD 263L : February 11-16								
Average	56.4	24.2	80.6		3.6	15.9	4.4	-0.6
Stan. Dev. (σn)	2.1	2.7	1.6		1.6	0.6	1.6	1.6
Stan. Dev. (σn-1)	2.4	3.1	1.9		1.8	0.6	1.9	1.9
PERIOD 263M : February 18-22								
Average	56.0	24.1	80.1		5.1	14.8	4.9	-0.1
Stan. Dev. (σn)	1.3	2.0	0.8		0.4	0.5	0.8	0.8
Stan. Dev. (σn-1)	1.4	2.2	0.9		0.4	0.6	0.9	0.9

Table 18

TSL SYSTEM RESPONSES IN COAL CONVERSION AND RESID + UC CONVERSION
Run 263 with Black Thunder Coal, Nov. 10, 1991 - Feb. 22, 1992
Phase 3 Data

		unit :	wt % MAF coal		wt% feed	hr^{-1}	hr^{-1}	wt% feed	hr^{-1}	hr^{-1}
		coal conversion			1st stage rate constant			2nd stage rate constant		
Date	Days	First	Second	TSL	Conv.	K_Vol	LN K_Vol	Conv.	K_Vol	LN K_Vol
10-Nov-91	10	81.0	92.3	91.2	21.9	93.6	4.54	1.7	5.8	1.75
11-Nov-91	11	84.0	91.6	91.5	20.0	83.0	4.42	7.4	26.5	3.28
12-Nov-91	12	73.5	92.4	93.2	15.9	61.8	4.12	10.4	38.0	3.64
13-Nov-91	13	75.8	93.6	94.3	14.5	56.2	4.03	12.9	49.1	3.89
14-Nov-91	14	87.9	93.7	93.9	17.3	70.3	4.25	9.4	34.9	3.55
17-Nov-91	17	80.5	93.6	92.8	14.6	56.5	4.03	14.3	55.2	4.01
18-Nov-91	18	83.2	93.6	91.1	16.9	67.2	4.21	10.8	40.0	3.69
20-Nov-91	20	87.3	93.2	91.2	18.0	71.8	4.27	9.7	35.1	3.56
21-Nov-91	21	79.7	93.6	94.4	16.3	63.2	4.15	15.4	59.0	4.08
22-Nov-91	22	80.2	92.9	92.9	17.0	67.2	4.21	18.1	72.5	4.28
03-Dec-91	26	80.3	93.6	92.6	20.0	82.2	4.41	9.9	36.1	3.59
06-Dec-91	29	87.5	93.6	93.2	21.0	87.4	4.47	7.0	24.7	3.21
07-Dec-91	30	90.0	93.6	93.7	18.2	73.2	4.29	11.2	41.5	3.73
08-Dec-91	31	86.6	93.9	92.3	19.8	81.0	4.39	10.2	37.2	3.62
09-Dec-91	32	85.2	93.7	92.7	20.4	81.8	4.40	9.7	34.3	3.53
13-Dec-91	36	87.0	94.5	93.2	19.0	68.8	4.23	12.6	42.3	3.75
14-Dec-91	37	86.3	92.6	92.6	20.3	74.8	4.32	15.0	51.8	3.95
15-Dec-91	38	85.0	92.8	92.8	19.4	70.4	4.25	11.3	37.3	3.62
16-Dec-91	39	88.6	93.8	93.8	19.9	71.5	4.27	13.0	43.0	3.76
17-Dec-91	40	87.9	93.0	93.0	22.1	80.7	4.39	11.7	37.7	3.63
26-Dec-91	46	89.5	94.7	93.4	23.6	89.8	4.50	8.5	27.0	3.30
27-Dec-91	47	91.6	94.1	93.4	22.1	82.9	4.42	9.5	30.7	3.42
28-Dec-91	48	91.3	95.0	93.9	22.5	84.8	4.44	9.1	29.2	3.38
31-Dec-91	51	90.2	94.4	94.5	22.6	85.9	4.45	11.1	36.7	3.60
01-Jan-92	52	88.5	95.0	95.3	22.5	85.8	4.45	10.2	33.6	3.51
04-Jan-92	55	90.0	95.6	94.6	23.1	87.9	4.48	9.8	31.8	3.46
05-Jan-92	56	88.7	94.8	95.3	22.1	83.2	4.42	12.1	40.4	3.70
06-Jan-92	57	87.3	95.2	94.9	23.3	88.8	4.49	7.5	23.7	3.17
09-Jan-92	60	84.9	95.0	94.0	23.8	92.0	4.52	9.3	30.2	3.41
10-Jan-92	61	90.7	95.1	93.4	22.8	86.3	4.46	9.6	31.0	3.44
11-Jan-92	62	91.7	95.0	93.8	22.2	83.0	4.42	10.7	34.8	3.55
12-Jan-92	63	91.1	94.3	94.9	21.8	81.4	4.40	11.0	36.1	3.59
13-Jan-92	64	88.2	95.0	94.5	23.2	88.4	4.48	9.1	29.3	3.38
16-Jan-92	67	90.3	94.7	93.9	22.0	82.3	4.41	9.2	29.6	3.39
17-Jan-92	68	88.3	94.4	93.9	21.4	79.8	4.38	11.8	39.2	3.67
18-Jan-92	69	84.3	95.5	94.6	22.9	87.2	4.47	9.8	31.9	3.46
19-Jan-92	70	88.6	94.9	94.7	22.6	86.1	4.45	8.1	26.0	3.26
20-Jan-92	71	90.7	95.0	94.3	21.4	80.6	4.39	11.4	38.1	3.64

Table 18 (continued)

23-Jan-92	74	90.7	93.5	92.3	20.3	105.2	4.66	17.6	106.1	4.66
25-Jan-92	76	91.7	93.7	93.8	24.8	105.9	4.66	13.4	59.7	4.09
26-Jan-92	77	90.8	93.8	93.6	24.2	101.0	4.62	14.5	64.5	4.17
27-Jan-92	78	92.6	93.9	93.3	22.8	93.6	4.54	15.8	71.5	4.27
29-Jan-92	80	88.5	94.4	93.6	23.6	97.6	4.58	16.4	74.5	4.31
30-Jan-92	81	90.1	93.8	93.8	23.5	95.7	4.56	16.8	75.6	4.33
31-Jan-92	82	89.0	94.2	94.2	22.8	94.2	4.55	18.3	85.9	4.45
01-Feb-92	83	91.3	93.8	92.9	23.1	94.5	4.55	19.1	89.3	4.49
03-Feb-92	85	88.9	94.4	93.4	23.0	95.2	4.56	17.2	79.6	4.38
06-Feb-92	88	88.6	93.7	93.4	19.9	78.7	4.37	22.9	113.2	4.73
07-Feb-92	89	85.6	93.7	93.7	21.9	89.0	4.49	18.5	86.6	4.46
08-Feb-92	90	89.7	93.8	94.0	23.0	95.5	4.56	18.1	84.9	4.44
09-Feb-92	91	88.8	93.9	93.6	22.9	94.1	4.54	17.2	79.1	4.37
11-Feb-92	93	88.4	93.7	92.1	24.6	103.9	4.64	14.5	64.9	4.17
14-Feb-92	95	91.3	93.5	93.2	26.5	115.7	4.75	12.0	52.6	3.96
15-Feb-92	96	89.1	93.5	92.8	24.3	102.8	4.63	14.0	62.7	4.14
16-Feb-92	97	89.9	93.3	92.5	24.9	105.4	4.66	14.8	66.4	4.20
18-Feb-92	99	89.9	93.5	92.5	25.3	108.2	4.68	14.7	66.2	4.19
19-Feb-92	100	89.9	93.5	93.1	24.7	105.1	4.65	16.1	73.9	4.30
20-Feb-92	101	89.9	93.5	92.8	24.9	105.4	4.66	14.4	64.3	4.16
21-Feb-92	102	91.8	93.2	92.1	25.8	110.6	4.71	13.2	58.2	4.06
22-Feb-92	103	91.2	93.5	93.1	26.2	112.4	4.72	13.3	58.4	4.07

Table 18 (continued)

PERIOD 263A : November 10-14									
Average	80.4	92.7	92.8	17.9	73.0	4.27	8.4	30.8	3.22
Stan. Dev. (σn)	5.3	0.8	1.3	2.7	13.7	0.19	3.8	14.5	0.76
Stan. Dev. (σn-1)	5.9	0.9	1.4	3.0	15.3	0.21	4.2	16.2	0.85
PERIOD 263B : November 17-22									
Average	82.2	93.4	92.5	16.6	65.2	4.17	13.7	52.4	3.92
Stan. Dev. (σn)	2.8	0.3	1.2	1.1	5.1	0.08	3.1	13.5	0.26
Stan. Dev. (σn-1)	3.2	0.3	1.4	1.3	5.7	0.09	3.4	15.1	0.30
PERIOD 263C : December 3-9									
Average	85.9	93.7	92.9	19.9	81.1	4.39	9.6	34.8	3.53
Stan. Dev. (σn)	3.2	0.1	0.5	0.9	4.6	0.06	1.4	5.5	0.17
Stan. Dev. (σn-1)	3.6	0.1	0.6	1.0	5.1	0.06	1.6	6.2	0.20
PERIOD 263D : December 13-17									
Average	87.0	93.3	93.1	20.1	73.3	4.29	12.7	42.4	3.74
Stan. Dev. (σn)	1.3	0.7	0.4	1.1	4.2	0.06	1.3	5.3	0.12
Stan. Dev. (σn-1)	1.4	0.8	0.5	1.2	4.7	0.06	1.4	5.9	0.13
PERIOD 263E : December 26 - January 1									
Average	90.2	94.6	94.1	22.7	85.8	4.45	9.7	31.4	3.44
Stan. Dev. (σn)	1.1	0.3	0.7	0.5	2.2	0.03	0.9	3.4	0.11
Stan. Dev. (σn-1)	1.3	0.4	0.8	0.6	2.5	0.03	1.0	3.8	0.12
PERIOD 263F : January 4-6									
Average	88.7	95.2	94.9	22.8	86.6	4.46	9.8	32.0	3.44
Stan. Dev. (σn)	1.1	0.3	0.3	0.5	2.4	0.03	1.9	6.8	0.22
Stan. Dev. (σn-1)	1.4	0.4	0.4	0.6	3.0	0.03	2.3	8.3	0.27
PERIOD 263G : January 9-13									
Average	89.3	94.9	94.1	22.8	86.2	4.46	9.9	32.3	3.47
Stan. Dev. (σn)	2.5	0.3	0.5	0.7	3.8	0.04	0.8	2.7	0.08
Stan. Dev. (σn-1)	2.8	0.3	0.6	0.8	4.3	0.05	0.9	3.0	0.09
PERIOD 263H : January 16-20									
Average	88.4	94.9	94.3	22.1	83.2	4.42	10.1	32.9	3.48
Stan. Dev. (σn)	2.3	0.4	0.3	0.6	3.0	0.04	1.4	5.0	0.15
Stan. Dev. (σn-1)	2.5	0.4	0.4	0.7	3.3	0.04	1.5	5.6	0.17
PERIOD 263I : January 23-27									
Average	91.5	93.7	93.3	23.0	101.4	4.62	15.3	75.5	4.30
Stan. Dev. (σn)	0.8	0.1	0.6	1.7	4.9	0.05	1.6	18.2	0.22
Stan. Dev. (σn-1)	0.9	0.2	0.7	2.0	5.6	0.06	1.8	21.0	0.26
PERIOD 263J : January 29 - February 3									
Average	89.6	94.1	93.6	23.2	95.4	4.56	17.6	81.0	4.39
Stan. Dev. (σn)	1.0	0.3	0.4	0.3	1.2	0.01	1.0	5.8	0.07
Stan. Dev. (σn-1)	1.1	0.3	0.5	0.3	1.3	0.01	1.1	6.4	0.08
PERIOD 263K : February 6-9									
Average	88.2	93.8	93.7	21.9	89.3	4.49	19.2	91.0	4.50
Stan. Dev. (σn)	1.5	0.1	0.2	1.2	6.6	0.08	2.2	13.1	0.14
Stan. Dev. (σn-1)	1.8	0.1	0.2	1.4	7.6	0.09	2.5	15.2	0.16
PERIOD 263L : February 11-16									
Average	89.7	93.5	92.6	25.1	106.9	4.67	13.8	61.7	4.12
Stan. Dev. (σn)	1.1	0.1	0.4	0.8	5.2	0.05	1.1	5.4	0.09
Stan. Dev. (σn-1)	1.2	0.2	0.5	1.0	6.0	0.05	1.3	6.2	0.11
PERIOD 263M : February 18-22									
Average	90.5	93.4	92.7	25.4	108.3	4.68	14.3	64.2	4.16
Stan. Dev. (σn)	0.8	0.1	0.4	0.6	2.9	0.03	1.1	5.8	0.09
Stan. Dev. (σn-1)	0.9	0.1	0.4	0.6	3.2	0.03	1.2	6.5	0.10

Table 19

RUN 263 FIRST STAGE THERMAL ACTIVITY IN RESID + UC CONVERSION
Black Thunder Coal, Molyvan L (822 in Jan. 2-26) and Fe2O3 Addition
Oct. 31, 1991 - Feb. 23, 1992

Half-Volume Reactor
Phase 2 Data

Date	Days	Temp.	SV/C_Vol	Factor	Resid + UC Conv.	K/C	LN K/C	T_Inv.
02-Nov-91	2	811	78.4	4.180	24.9	108.7	4.69	0.000787
03-Nov-90	3	810	77.0	4.291	19.2	78.5	4.36	0.000787
04-Nov-91	4	810	74.7	4.415	20.3	84.0	4.43	0.000787
05-Nov-91	5	817	75.1	4.368	19.1	77.4	4.35	0.000783
06-Nov-91	6	825	75.0	4.389	15.4	59.9	4.09	0.000778
07-Nov-91	7	825	78.3	4.233	16.4	65.0	4.17	0.000778
08-Nov-91	8	825	78.2	4.239	15.7	61.8	4.12	0.000778
09-Nov-91	9	826	79.5	4.207	15.8	62.7	4.14	0.000778
10-Nov-91	10	826	79.3	4.208	21.2	89.8	4.50	0.000778
11-Nov-91	11	826	78.6	4.228	18.8	76.9	4.34	0.000778
12-Nov-91	12	824	77.8	4.206	14.6	55.9	4.02	0.000779
13-Nov-91	13	826	79.6	4.166	14.1	54.4	4.00	0.000778
14-Nov-91	14	827	79.8	4.210	17.1	69.3	4.24	0.000777
15-Nov-91	15	826	78.8	4.261	17.0	68.8	4.23	0.000778
16-Nov-91	16	826	78.9	4.213	14.6	56.8	4.04	0.000778
17-Nov-91	17	826	79.6	4.154	14.2	54.7	4.00	0.000778
18-Nov-91	18	824	80.1	4.122	17.0	67.7	4.21	0.000779
19-Nov-91	19	827	77.5	4.240	17.6	70.2	4.25	0.000777
20-Nov-91	20	826	78.1	4.187	17.4	68.9	4.23	0.000778
21-Nov-91	21	826	77.7	4.174	19.1	76.6	4.34	0.000778
22-Nov-91	22	825	78.4	4.182	21.4	89.3	4.49	0.000778
28-Nov-91	23	840	80.7	4.068	25.4	111.7	4.72	0.000769
29-Nov-91	24	840	82.2	4.002	20.8	86.4	4.46	0.000769
30-Nov-91	25	837	77.6	4.247	19.4	79.3	4.37	0.000771
03-Dec-91	26	841	77.6	4.233	21.6	90.6	4.51	0.000769
04-Dec-91	27	841	79.2	4.220	23.2	101.0	4.61	0.000769
05-Dec-91	28	840	76.5	4.322	19.4	79.6	4.38	0.000769
06-Dec-91	29	838	77.5	4.242	19.0	77.1	4.35	0.000770
07-Dec-91	30	839	77.4	4.251	18.2	73.2	4.29	0.000770
08-Dec-91	31	839	78.1	4.200	18.6	74.9	4.32	0.000770
09-Dec-91	32	840	77.2	4.133	20.2	80.8	4.39	0.000769
10-Dec-91	33	840	74.7	4.334	20.2	81.9	4.41	0.000769
11-Dec-91	34	841	70.7	4.246	23.1	90.1	4.50	0.000769
12-Dec-91	35	841	69.3	4.217	16.6	58.2	4.06	0.000769
13-Dec-91	36	841	69.7	4.208	20.4	75.2	4.32	0.000769
14-Dec-91	37	839	69.2	4.243	21.6	80.9	4.39	0.000770
15-Dec-91	38	840	68.5	4.270	19.8	72.2	4.28	0.000769
16-Dec-91	39	840	67.7	4.254	21.0	76.5	4.34	0.000769
17-Dec-91	40	840	67.9	4.191	22.9	84.5	4.44	0.000769
18-Dec-91	41	839	68.0	4.299	20.4	74.9	4.32	0.000770

Average Conversion, K/C, ln K/C
period Nov. 10-22 at T = 825 F

17.2	69.2	4.22
2.4	11.4	0.16
2.5	11.9	0.17

period Nov. 28 - Jan. 20 at T = 840 F

20.8	79.5	4.36
2.5	12.3	0.16
2.5	12.5	0.16

period Jan. 22 - Feb. 22 at T = 840 F

23.0	96.1	4.55
2.7	14.5	0.16
2.8	14.8	0.16

period Nov. 28 - Feb. 22 at T = 840 F

21.7	86.0	4.44
2.8	15.5	0.19
2.8	15.6	0.19

Table 19 (continued)

23-Dec-91	43	841	71.5	4.190	21.5	82.0	4.41	0.000769
24-Dec-91	44	840	68.9	4.227	22.5	84.6	4.44	0.000769
25-Dec-91	45	839	69.3	4.247	13.9	47.5	3.86	0.000770
26-Dec-91	46	839	68.7	4.230	23.3	88.3	4.48	0.000770
27-Dec-91	47	839	69.3	4.219	21.5	80.0	4.38	0.000770
28-Dec-91	48	839	67.7	4.317	25.5	100.0	4.60	0.000770
31-Dec-91	51	840	69.6	4.225	23.2	88.9	4.49	0.000769
01-Jan-92	52	840	69.5	4.253	24.1	93.8	4.54	0.000769
03-Jan-92	54	843	67.7	4.276	22.7	85.0	4.44	0.000767
04-Jan-92	55	841	67.4	4.341	23.7	90.9	4.51	0.000769
05-Jan-92	56	841	68.1	4.311	17.2	60.9	4.11	0.000769
06-Jan-92	57	840	66.2	4.419	18.7	67.2	4.21	0.000769
08-Jan-92	59	841	69.5	4.233	17.4	62.0	4.13	0.000769
09-Jan-92	60	841	70.1	4.203	17.1	60.8	4.11	0.000769
10-Jan-92	61	841	69.1	4.231	18.3	65.5	4.18	0.000769
11-Jan-92	62	840	68.3	4.258	21.5	79.6	4.38	0.000769
12-Jan-92	63	840	69.5	4.197	21.3	79.0	4.37	0.000769
13-Jan-92	64	840	71.0	4.123	23.4	89.4	4.49	0.000769
14-Jan-92	65	841	69.4	4.189	20.5	74.9	4.32	0.000769
15-Jan-92	66	840	69.5	4.218	18.6	67.0	4.20	0.000769
16-Jan-92	67	840	69.6	4.190	21.3	79.0	4.37	0.000769
17-Jan-92	68	841	69.8	4.200	21.0	77.9	4.36	0.000769
18-Jan-92	69	840	69.8	4.205	24.0	92.7	4.53	0.000769
19-Jan-92	70	840	69.9	4.216	23.6	91.0	4.51	0.000769
20-Jan-92	71	839	69.5	4.258	17.3	61.9	4.13	0.000770
21-Jan-92	72		68.9					
22-Jan-92	73	838	78.3	4.257	17.3	69.7	4.24	0.000770
23-Jan-92	74	840	89.1	4.637	21.3	111.8	4.72	0.000769
25-Jan-92	76	840	90.1	3.562	24.5	104.2	4.65	0.000769
26-Jan-92	77	841	90.7	3.488	23.5	97.2	4.58	0.000769
27-Jan-92	78	841	92.2	3.439	22.3	91.0	4.51	0.000769
28-Jan-92	79	840	84.0	3.518	24.5	95.9	4.56	0.000769
29-Jan-92	80	840	90.8	3.478	24.2	100.8	4.61	0.000769
30-Jan-92	81	840	92.2	3.380	22.7	91.5	4.52	0.000769
31-Jan-92	82	840	91.9	3.471	22.3	91.5	4.52	0.000769
01-Feb-92	83	841	89.8	3.502	25.2	105.9	4.66	0.000769
03-Feb-92	85	840	90.9	3.506	17.4	67.1	4.21	0.000769
04-Feb-92	86	841	86.8	3.506	20.8	79.9	4.38	0.000769
05-Feb-92	87	839	90.7	3.520	16.9	64.9	4.17	0.000770
06-Feb-92	88	841	90.2	3.513	20.4	81.2	4.40	0.000769
07-Feb-92	89	841	91.1	3.484	20.9	83.8	4.43	0.000769
08-Feb-92	90	842	91.6	3.487	25.2	107.7	4.68	0.000768
09-Feb-92	91	842	90.9	3.486	24.2	101.1	4.62	0.000768
10-Feb-92	92	841	91.0	3.521	24.2	102.3	4.63	0.000769
11-Feb-92	93	844	90.4	3.521	27.2	119.0	4.78	0.000767
14-Feb-92	95	842	91.5	3.508	26.4	115.2	4.75	0.000768
15-Feb-92	96	841	92.2	3.474	24.9	106.2	4.67	0.000769
16-Feb-92	97	840	90.8	3.501	25.3	107.6	4.68	0.000769
17-Feb-92	98	840	90.3	3.524	22.3	91.3	4.51	0.000769
18-Feb-92	99	841	92.2	3.466	22.8	94.4	4.55	0.000769
19-Feb-92	100	841	92.4	3.468	23.3	97.3	4.58	0.000769
20-Feb-92	101	835	91.7	3.467	20.8	83.5	4.42	0.000772
21-Feb-92	102	841	92.2	3.450	26.1	112.3	4.72	0.000769
22-Feb-92	103	842	91.2	3.471	27.1	117.7	4.77	0.000768

Table 19 (continued)

PERIOD 263A : November 10-14							
Average	826	79.0	4.204	17.2	69.3	4.22	0.000778
Stan. Dev. (σn)	1	0.8	0.020	2.6	13.2	0.19	0.000001
Stan. Dev. (σn-1)	1	0.8	0.023	3.0	14.8	0.21	0.000001
PERIOD 263B : November 17-22							
Average	826	78.6	4.177	17.8	71.2	4.26	0.000778
Stan. Dev. (σn)	1	1.0	0.036	2.2	10.4	0.15	0.000001
Stan. Dev. (σn-1)	1	1.1	0.039	2.4	11.4	0.16	0.000001
PERIOD 263C : December 3-9 including Dec. 4 & 5							
Average	840	77.6	4.229	20.0	82.4	4.41	0.000769
Stan. Dev. (σn)	1	0.8	0.053	1.7	9.2	0.11	0.000001
Stan. Dev. (σn-1)	1	0.8	0.057	1.8	9.9	0.11	0.000001
PERIOD 263D : December 13-17							
Average	840	68.6	4.233	21.1	77.9	4.35	0.000769
Stan. Dev. (σn)	1	0.8	0.029	1.1	4.3	0.06	0.000000
Stan. Dev. (σn-1)	1	0.9	0.033	1.2	4.8	0.06	0.000000
PERIOD 263E : December 26 - January 1							
Average	839	68.9	4.249	23.5	90.2	4.50	0.000770
Stan. Dev. (σn)	0	0.7	0.036	1.3	6.6	0.07	0.000000
Stan. Dev. (σn-1)	1	0.8	0.040	1.5	7.4	0.08	0.000000
PERIOD 263F : January 4-6							
Average	841	67.2	4.357	19.9	73.0	4.28	0.000769
Stan. Dev. (σn)	0	0.8	0.046	2.8	12.9	0.17	0.000000
Stan. Dev. (σn-1)	1	1.0	0.056	3.4	15.8	0.21	0.000000
PERIOD 263G : January 9-13							
Average	840	69.6	4.202	20.3	74.9	4.31	0.000769
Stan. Dev. (σn)	0	0.9	0.045	2.3	10.4	0.14	0.000000
Stan. Dev. (σn-1)	1	1.0	0.051	2.6	11.6	0.16	0.000000
PERIOD 263H : January 16-20							
Average	840	69.7	4.214	21.4	80.5	4.38	0.000769
Stan. Dev. (σn)	1	0.1	0.024	2.4	11.1	0.14	0.000000
Stan. Dev. (σn-1)	1	0.2	0.026	2.7	12.4	0.16	0.000000
PERIOD 263I : January 23-27							
Average	841	90.5	3.781	22.9	101.0	4.61	0.000769
Stan. Dev. (σn)	1	1.1	0.496	1.2	7.8	0.08	0.000000
Stan. Dev. (σn-1)	1	1.3	0.573	1.4	9.0	0.09	0.000000
PERIOD 263J : January 29 - February 3							
Average	840	91.1	3.467	22.4	91.4	4.50	0.000769
Stan. Dev. (σn)	0	0.8	0.046	2.7	13.3	0.16	0.000000
Stan. Dev. (σn-1)	0	0.9	0.051	3.0	14.9	0.18	0.000000
PERIOD 263K : February 6-9							
Average	842	90.9	3.492	22.7	93.5	4.53	0.000768
Stan. Dev. (σn)	1	0.5	0.012	2.1	11.2	0.12	0.000000
Stan. Dev. (σn-1)	1	0.6	0.014	2.4	12.9	0.14	0.000000
PERIOD 263L : February 11-16							
Average	842	91.2	3.501	26.0	112.0	4.72	0.000768
Stan. Dev. (σn)	1	0.7	0.017	0.9	5.3	0.05	0.000001
Stan. Dev. (σn-1)	2	0.8	0.020	1.0	6.1	0.05	0.000001
PERIOD 263M : February 18-22							
Average	840	91.9	3.464	24.0	101.0	4.61	0.000769
Stan. Dev. (σn)	3	0.4	0.007	2.3	12.4	0.12	0.000002
Stan. Dev. (σn-1)	3	0.5	0.008	2.6	13.9	0.14	0.000002

Table 20

RUN 263 SECOND STAGE THERMAL ACTIVITY IN RESID + UC CONVERSION
Black Thunder Coal, Oct. 31, 1991 - Feb. 23, 1992
Half-Volume Reactor, Criterion 324 Since Jan. 21 (170 lbs), 3 lb/t Replacement
in Jan. 23 - Feb. 8, Batch in Feb. 9-16, 2 lb/t Replacement in Feb. 17-21
Phase 2 Data

Date	Days	Temp.	SV/C_Vol	Factor	Resid + UC Conv.	K/C	LN K/C	T_Inv.
02-Nov-91	2	795	78.4	4.180	1.7	5.7	1.74	0.000797
03-Nov-90	3	797	77.0	4.291	11.7	43.8	3.78	0.000796
04-Nov-91	4	795	74.7	4.415	5.1	17.7	2.88	0.000797
05-Nov-91	5	794	75.1	4.368	1.7	5.7	1.74	0.000797
06-Nov-91	6	791	75.0	4.389	11.6	43.2	3.77	0.000799
07-Nov-91	7	809	78.3	4.233	17.1	68.4	4.22	0.000788
08-Nov-91	8	811	78.2	4.239	9.4	34.4	3.54	0.000787
09-Nov-91	9	811	79.5	4.207	10.2	38.0	3.64	0.000787
10-Nov-91	10	810	79.3	4.208	3.1	10.7	2.37	0.000787
11-Nov-91	11	810	78.6	4.228	8.0	28.9	3.36	0.000787
12-Nov-91	12	810	77.8	4.206	10.0	36.3	3.59	0.000787
13-Nov-91	13	810	79.6	4.166	14.1	54.4	4.00	0.000787
14-Nov-91	14	810	79.8	4.210	11.5	43.7	3.78	0.000787
15-Nov-91	15	826	78.8	4.261	17.0	68.8	4.23	0.000778
16-Nov-91	16	810	78.9	4.213	13.1	50.1	3.91	0.000787
17-Nov-91	17	810	79.6	4.154	15.7	61.6	4.12	0.000787
18-Nov-91	18	808	80.1	4.122	9.4	34.3	3.53	0.000789
19-Nov-91	19	808	77.5	4.240	10.0	36.5	3.60	0.000789
20-Nov-91	20	810	78.1	4.187	10.0	36.3	3.59	0.000787
21-Nov-91	21	808	77.7	4.174	13.5	50.6	3.92	0.000789
22-Nov-91	22	810	78.4	4.182	9.3	33.6	3.52	0.000787
28-Nov-91	23	807	80.7	4.068	10.8	39.7	3.68	0.000789
29-Nov-91	24	810	82.2	4.002	15.8	61.8	4.12	0.000787
30-Nov-91	25	809	77.6	4.247	15.0	58.1	4.06	0.000788
03-Dec-91	26	810	77.6	4.233	6.8	24.0	3.18	0.000787
04-Dec-91	27	810	79.2	4.220	10.8	40.5	3.70	0.000787
05-Dec-91	28	810	76.5	4.322	15.0	58.3	4.07	0.000787
06-Dec-91	29	808	77.5	4.242	19.2	78.1	4.36	0.000789
07-Dec-91	30	810	77.4	4.251	7.9	28.2	3.34	0.000787
08-Dec-91	31	811	78.1	4.200	12.6	47.3	3.86	0.000787
09-Dec-91	32	810	77.2	4.133	10.5	37.4	3.62	0.000787
10-Dec-91	33	809	74.7	4.334	17.3	67.7	4.22	0.000788
11-Dec-91	34	809	70.7	4.246	12.7	43.7	3.78	0.000788
12-Dec-91	35	810	69.3	4.217	13.1	44.1	3.79	0.000787
13-Dec-91	36	810	69.7	4.208	12.7	42.7	3.75	0.000787
14-Dec-91	37	808	69.2	4.243	17.3	61.5	4.12	0.000789
15-Dec-91	38	810	68.5	4.270	13.7	46.4	3.84	0.000787
16-Dec-91	39	810	67.7	4.254	7.7	24.0	3.18	0.000787
17-Dec-91	40	810	67.9	4.191	14.5	48.2	3.88	0.000787
18-Dec-91	41	810	68.0	4.299	17.2	60.7	4.11	0.000787

Average Conv.,K/C,ln K/C
period Nov. 10 - Jan.20
at T = 810 F

12.6	44.9	3.74
3.8	15.4	0.37
3.8	15.5	0.38

Table 20 (continued)

23-Dec-91	43	808	71.5	4.190	15.9	56.6	4.04	0.000789		
24-Dec-91	44	808	68.9	4.227	10.3	33.4	3.51	0.000789		
25-Dec-91	45	809	69.3	4.247	16.7	59.0	4.08	0.000788		
26-Dec-91	46	810	68.7	4.230	8.8	28.0	3.33	0.000787		
27-Dec-91	47	810	69.3	4.219	11.4	37.6	3.63	0.000787		
28-Dec-91	48	806	67.7	4.317	8.9	28.5	3.35	0.000790		
31-Dec-91	51	810	69.6	4.225	12.9	43.6	3.77	0.000787		
01-Jan-92	52	810	69.5	4.253	6.7	21.2	3.05	0.000787		
03-Jan-92	54	802	67.7	4.276	8.2	25.8	3.25	0.000792		
04-Jan-92	55	806	67.4	4.341	12.4	41.4	3.72	0.000790		
05-Jan-92	56	810	68.1	4.311	19.5	71.1	4.26	0.000787		
06-Jan-92	57	808	66.2	4.419	10.2	33.2	3.50	0.000789		
08-Jan-92	59	810	69.5	4.233	15.8	55.2	4.01	0.000787		
09-Jan-92	60	810	70.1	4.203	19.5	71.4	4.27	0.000787		
10-Jan-92	61	806	69.1	4.231	15.6	54.0	3.99	0.000790		
11-Jan-92	62	810	68.3	4.258	10.6	34.5	3.54	0.000787		
12-Jan-92	63	809	69.5	4.197	12.5	41.7	3.73	0.000788		
13-Jan-92	64	809	71.0	4.123	10.6	34.7	3.55	0.000788		
14-Jan-92	65	801	69.4	4.189	12.7	42.3	3.74	0.000793		
15-Jan-92	66	808	69.5	4.218	19.1	69.2	4.24	0.000789		
16-Jan-92	67	803	69.6	4.190	12.6	42.1	3.74	0.000792		
17-Jan-92	68	806	69.8	4.200	10.3	33.6	3.52	0.000790		
18-Jan-92	69	806	69.8	4.205	7.7	24.5	3.20	0.000790		
19-Jan-92	70	806	69.9	4.216	11.3	37.5	3.63	0.000790	Since Jan. 21 w/ Criterion	
20-Jan-92	71	810	69.5	4.258	21.8	82.5	4.41	0.000787	K and ln K values reported	
21-Jan-92	72		SV_Cat_Vol						MF_Age	Age_Vol
22-Jan-92	73	808	94.2	4.257	16.7	80.4	4.39	0.000789	736	3.97
23-Jan-92	74	809	107.1	4.637	18.0	109.0	4.69	0.000788	675	3.65
25-Jan-92	76	810	108.4	3.562	19.9	95.9	4.56	0.000787	652	3.52
26-Jan-92	77	810	109.1	3.488	18.1	84.1	4.43	0.000787	700	3.78
27-Jan-92	78	809	110.8	3.439	16.6	75.9	4.33	0.000788	646	3.49
28-Jan-92	79	809	101.0	3.518	15.8	66.7	4.20	0.000788	691	3.73
29-Jan-92	80	810	109.2	3.478	16.5	75.0	4.32	0.000787	637	3.44
30-Jan-92	81	810	110.8	3.380	19.9	93.1	4.53	0.000787	686	3.70
31-Jan-92	82	810	110.5	3.471	21.6	105.7	4.66	0.000787	634	3.42
01-Feb-92	83	810	108.0	3.502	15.6	69.9	4.25	0.000787	682	3.68
03-Feb-92	85	809	109.3	3.506	26.5	138.1	4.93	0.000788	670	3.62
04-Feb-92	86	809	104.3	3.506	20.9	96.6	4.57	0.000788	618	3.34
05-Feb-92	87	804	109.1	3.520	18.3	86.0	4.45	0.000791	666	3.60
06-Feb-92	88	809	108.5	3.513	20.2	96.5	4.57	0.000788	616	3.33
07-Feb-92	89	809	109.5	3.484	20.5	98.4	4.59	0.000788	664	3.59
08-Feb-92	90	810	110.2	3.487	15.9	72.6	4.29	0.000787	615	3.32
09-Feb-92	91	810	109.2	3.486	16.6	75.8	4.33	0.000787	664	3.59
10-Feb-92	92	807	109.4	3.521	14.6	65.9	4.19	0.000789	712	3.84
11-Feb-92	93	809	108.7	3.521	9.8	41.6	3.73	0.000788	760	4.10
14-Feb-92	95	810	110.0	3.508	12.2	53.6	3.98	0.000787	883	4.77
15-Feb-92	96	810	110.8	3.474	14.4	64.8	4.17	0.000787	933	5.04
16-Feb-92	97	810	109.1	3.501	13.6	60.2	4.10	0.000787	981	5.30
17-Feb-92	98	810	108.6	3.524	15.8	71.8	4.27	0.000787	932	5.03
18-Feb-92	99	810	110.9	3.466	17.8	83.2	4.42	0.000787	981	5.30
19-Feb-92	100	810	111.1	3.468	16.5	76.1	4.33	0.000787	934	5.04
20-Feb-92	101	810	110.2	3.467	13.6	60.2	4.10	0.000787	983	5.31
21-Feb-92	102	809	110.9	3.450	11.4	49.2	3.90	0.000788	935	5.05
22-Feb-92	103	810	109.7	3.471	12.9	56.4	4.03	0.000787	983	5.31

Table 20 (continued)

PERIOD 263A : November 10-14									
Average	810	79.0	4.204	9.3	34.8	3.42	0.000787		
Stan. Dev. (σn)	0	0.8	0.020	3.7	14.7	0.57	0.000000		
Stan. Dev. (σn-1)	0	0.8	0.023	4.1	16.5	0.63	0.000000		
PERIOD 263B : November 17-22									
Average	809	78.6	4.177	11.3	42.2	3.71	0.000788		
Stan. Dev. (σn)	1	1.0	0.036	2.4	10.4	0.23	0.000001		
Stan. Dev. (σn-1)	1	1.1	0.039	2.7	11.4	0.25	0.000001		
PERIOD 263C : December 3-9 including Dec. 4 & 5									
Average	810	77.6	4.229	11.8	44.8	3.73	0.000787		
Stan. Dev. (σn)	1	0.8	0.053	3.9	17.2	0.38	0.000001		
Stan. Dev. (σn-1)	1	0.8	0.057	4.3	18.6	0.41	0.000001		
PERIOD 263D : December 13-17									
Average	810	68.6	4.233	13.2	44.6	3.75	0.000788		
Stan. Dev. (σn)	1	0.8	0.029	3.1	12.1	0.31	0.000000		
Stan. Dev. (σn-1)	1	0.9	0.033	3.5	13.5	0.35	0.000001		
PERIOD 263E : December 26 - January 1									
Average	809	68.9	4.249	9.7	31.8	3.43	0.000788		
Stan. Dev. (σn)	2	0.7	0.036	2.2	7.9	0.25	0.000001		
Stan. Dev. (σn-1)	2	0.8	0.040	2.4	8.8	0.28	0.000001		
PERIOD 263F : January 4-6									
Average	808	67.2	4.357	14.0	48.6	3.83	0.000789		
Stan. Dev. (σn)	2	0.8	0.046	4.0	16.3	0.32	0.000001		
Stan. Dev. (σn-1)	2	1.0	0.056	4.9	19.9	0.39	0.000001		
PERIOD 263G : January 9-13									
Average	809	69.6	4.202	13.8	47.3	3.82	0.000788		
Stan. Dev. (σn)	1	0.9	0.045	3.4	14.0	0.28	0.000001		
Stan. Dev. (σn-1)	2	1.0	0.051	3.8	15.7	0.31	0.000001		
PERIOD 263H : January 16-20									
Average	806	69.7	4.214	12.7	44.1	3.70	0.000790		
Stan. Dev. (σn)	2	0.1	0.024	4.8	20.1	0.40	0.000001		
Stan. Dev. (σn-1)	2	0.2	0.026	5.4	22.4	0.45	0.000002		
PERIOD 263I : January 23-27									
Average	810	108.8	3.781	18.2	91.2	4.50	0.000788	668	3.61
Stan. Dev. (σn)	1	1.4	0.496	1.2	12.5	0.14	0.000000	21	0.11
Stan. Dev. (σn-1)	1	1.6	0.573	1.4	14.4	0.16	0.000000	25	0.13
PERIOD 263J : January 29 - February 3									
Average	810	109.5	3.467	20.0	96.4	4.54	0.000788	662	3.57
Stan. Dev. (σn)	0	1.0	0.046	3.9	24.5	0.25	0.000000	22	0.12
Stan. Dev. (σn-1)	0	1.1	0.051	4.4	27.4	0.27	0.000000	25	0.13
PERIOD 263K : February 6-9									
Average	810	109.4	3.492	18.3	85.8	4.44	0.000788	640	3.45
Stan. Dev. (σn)	1	0.6	0.012	2.1	11.7	0.14	0.000000	24	0.13
Stan. Dev. (σn-1)	1	0.7	0.014	2.4	13.5	0.16	0.000000	28	0.15
PERIOD 263L : February 11-16									
Average	810	109.7	3.501	12.5	55.0	3.99	0.000788	889	4.80
Stan. Dev. (σn)	0	0.8	0.017	1.7	8.7	0.17	0.000000	82	0.44
Stan. Dev. (σn-1)	1	0.9	0.020	2.0	10.1	0.19	0.000000	95	0.51
PERIOD 263M : February 18-22									
Average	810	110.5	3.464	14.4	65.0	4.16	0.000788	963	5.20
Stan. Dev. (σn)	0	0.5	0.007	2.4	12.7	0.19	0.000000	23	0.13
Stan. Dev. (σn-1)	0	0.6	0.008	2.6	14.2	0.22	0.000000	26	0.14

Table 21

TSL Process Performance Summary

Period	Reactor volume %	Recycle resid (wt %)	Reaction temp. (°F) 1st	Reaction temp. (°F) 2nd	Coal feed (MF lb/hr)	Yield (wt % MAF coal) C4+ dist	Yield (wt % MAF coal) Organic rejection	Yield (wt % MAF coal) Resid	Catalyst replacement (lb/ton MF)	Catalyst age (lb MF coal/ lb cat)	Coal space velocity (lb MF coal/hr /cuft rea) (x1/C)	Coal space velocity (lb MF coal/hr /cuft cat)	Interstage separator	Inlet H2 pp (psia) 1st	Inlet H2 pp (psia) 2nd
263A	50/50(b)	45	826	810	299	48	21	10	-	-	79	-	Yes	2760	2560
263B	50/50	44	825	809	299	52	17	10	-	-	79	-	Yes	2800	2530
263C	50/50	43	840	810	294	52	18	7	-	-	78	-	Yes	2710	2540
263D	50/50	41	840	810	260	55	16	6	-	-	69	-	Yes	2760	2580
263E	50/50	44	840	809	261	61	14	2	-	-	69	-	Yes	2720	2550
263F(a)	50/50	44	840	808	254	62	15	-1	-	-	67	-	Yes	2770	2530
263G	50/50	45	841	809	264	57	14	4	-	-	70	-	Yes	2770	2550
263H	50/50	45	840	806	264	61	14	2	-	-	70	-	Yes	2760	2550
263I(a)	50/50(c)	45	840	810	343	61	14	1	3	668	91	109	Yes	2770	2480
263J	50/50	41	840	810	345	58	14	4	3	662	91	110	Yes	2740	2540
263K	50/50	39	842	809	344	58	14	4	3	640	91	109	Yes	2390	2520
263L	50/50	40	842	810	345	56	16	4	0	889	91	110	Yes	2780	2540
263M	50/50	40	840	810	348	55	15	5	2	963	92	111	Yes	2790	2550

Note:
(a) Transitional.
(b) Thermal/thermal mode in periods 263A-H.
(c) Thermal/catalytic mode in periods 263I-M.
(d) Addition of Molyvan L as a precursor in periods 263A-E and J-M; addition of Molyvan 822 in periods 263F-I.

Table 22

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-rea) (x1/C)	Adjusted yield (wt % MAF coal) C4+ dist	Adjusted yield (wt % MAF coal) Common(b) organic rej.	Calculated steady-state catalyst replacement (lb/ton MF)
263A	252.2	66.6	55.3	21	-
263B	259.2	68.1	58.6	17	-
263C	284.2	75.0	56.6	18	-
263D	254.1	67.0	58.5	16	-
263E	260.0	68.6	62.3	14	-
263F(c)	234.6	67.4	60.7	15	-
263G	254.2	67.0	60.0	14	-
263H	270.8	71.5	62.5	14	-
263I(c)	360.4	95.1	61.5	14	2.6
263J	341.4	90.2	61.7	13	2.6
263K	318.5	84.1	60.8	14	2.7
263L	323.6	85.5	58.5	16	2.0
263M	304.1	80.3	57.9	15	1.9

Note:
(a) Projected by using CSTR 1st order kinetics.
(b) Estimated.
(c) Transitional.
(d) Thermal/thermal mode in 263A-H; thermal/catalytic mode in 263I-M.

Table 23

Distillate Production Comparison (Run 263 with Black Thunder Coal)

run(a)	263A	263B	263C	263D	263E	263F(c)	263G	263H	263I(c)	263J	263K	263L	263M
coal	Black Thunder												
ash content (wt % MF coal)	7.7	7.0	7.6	7.2	7.1	6.9	7.1	6.8	6.7	6.7	6.5	6.6	6.5
catalyst	None								Criterion 324				
iron-oxide (+DMDS) (wt % MF coal)	2.0	2.0	2.0	2.0	2.0	2.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0
molybdenum (ppm MF coal)(d)	200	100	100	100	100	100	100	100	100	100	100	100	100
recycle resid (wt % in process solvent)	45	44	43	41	44	44	45	45	45	40	40	40	40
coal concentration (wt % in slurry)	25	25	24	25	25	24	25	25	29	30	30	30	30
inlet hydrogen part. pres. (psia)													
1st stage	2760	2800	2710	2760	2720	2770	2770	2760	2770	2740	2390	2780	2790
2nd stage	2560	2530	2540	2580	2550	2530	2550	2550	2480	2540	2520	2540	2550
yield (wt % MAF coal)													
C4+ distillate	47.9	51.8	52.1	54.6	60.7	61.5	57.0	60.9	60.9	58.4	57.8	56.1	54.6
resid	10.1	9.6	7.0	5.8	2.1	-0.6	4.3	2.4	1.2	4.2	4.8	3.6	5.1
organic rejection	21.3	17.0	17.5	15.8	14.1	14.5	14.0	13.8	13.7	13.4	13.5	15.9	14.8
distillate selectivity to conversion	0.70	0.71	0.69	0.70	0.72	0.71	0.70	0.73	0.72	0.71	0.71	0.70	0.68
coal feed (MF lb/hr)	299	299	294	260	261	254	264	264	343	345	344	345	348
coal space velocity													
(MF lb/hr-cuft-cat per stage)	-	-	-	-	-	-	-	-	108.8	109.5	109.4	109.7	110.5
(relative to 251-IIB)	-	-	-	-	-	-	-	-	(250)	(251)	(251)	(252)	(253)
(MF lb/hr-cuft-reactors)(x1/C)	39.5	39.3	38.8	34.3	34.5	33.6	34.8	34.9	45.3	45.6	45.5	45.6	46.0
(relative to 251-IIB)	(181)	(180)	(178)	(157)	(158)	(154)	(160)	(160)	(208)	(209)	(209)	(209)	(211)
catalyst replacement (lb/ton MF coal)													
1st stage	-	-	-	-	-	-	-	-	-	-	-	-	-
2nd stage	-	-	-	-	-	-	-	-	3	3	3	2(b)	2
total	-	-	-	-	-	-	-	-	3	3	3	2	2
(relative to 251-IIB)	-	-	-	-	-	-	-	-	(100)	(100)	(100)	(67)	(67)
reaction temperature (deg F)													
1st stage	826	825	840	840	840	840	841	840	840	840	842	842	840
2nd stage	810	809	810	810	809	808	809	806	810	810	809	810	810
(dT total to 251-IIB)	(+74)	(+72)	(+88)	(+88)	(+87)	(+86)	(+88)	(+84)	(+88)	(+88)	(+89)	(+90)	(+88)
(dT average to 251-IIB)	(+37)	(+36)	(+44)	(+44)	(+44)	(+43)	(+44)	(+42)	(+44)	(+44)	(+45)	(+45)	(+44)
distillate production													
(lb/hr)	132	144	142	132	147	145	140	150	195	188	186	181	178
(lb/hr-cuft-cat per stage)	-	-	-	-	-	-	-	-	61.8	59.7	59.1	57.5	56.4
(relative to 251-IIB)	-	-	-	-	-	-	-	-	255	246	244	237	233
(lb/hr-cuft-reactors)(x1/C)	17.5	18.9	18.7	17.4	19.5	19.2	18.4	19.8	25.7	24.8	24.6	23.9	23.5
(relative to 251-IIB)	145	156	154	143	160	159	152	163	212	205	203	197	194

Note:
(a) Thermal/thermal mode of operation in periods 263A-H; thermal/catalytic mode in periods 263I-M. With interstage separation.
(b) Estimated at steady-state operation from batch aging data.
(c) Transitional.
(d) Addition of Molyvan L as a precursor in periods 263A-E and J-M; addition of Molyvan 822 in periods 263F-I.

Table 24

Distillate Production Comparison (Low-Rank Coals and Lignite)

run(a)	251-IIB	255B	258E	258H	258I	260C(c)	260D	262B	262C1	262D	262E	262F
coal	Wyodak	Tex. Lig.	Sp.Cr.	Black Thunder								
ash content (wt % MF coal)	8.8	12.1	5.6	7.3	7.3	6.2	6.2	6.3	6.5	6.4	6.3	6.5
catalyst	A1C	S324	S324	S324	S324	S324	S324	C324	C324	C324	C324	C324
iron-oxide (+DMDS) (wt % MF coal)	0.8	2.0	2.1	2.1	2.1	2.0	2.0	2.0	2.0	2.0	2.0	2.0
molybdenum (ppm MF coal)(e)	-	-	-	-	-	-	-	500	500	200	100	200
recycle resid (wt % in p. solvent)	25	25	40	41	41	40	39	38	39	36	38	41
coal concentration (wt % in slurry	33	29	30	30	30	30	30	30	30	30	30	30
inlet hydrogen part. pres. (psia)												
1st stage	2510	2530	2350	2450	2500	2780	2710	2710	2720	2800	2840	2860
2nd stage	2550	2600	2400	2520	2530	2110	2240	2500	2550	2530	2540	2530
yield (wt % MAF coal)												
C4+ distillate	61.0	50.1	55.3	55.4	58.1	54.1	59.8	58.0	64.7	60.8	57.2	47.5
resid	3.9	2.6	8.6	4.9	2.6	7.5	0.3	1.6	-4.1	0.3	-0.5	11.4
organic rejection	9.4	21.4	10.4	13.8	14.2	10.7	18.5	17.0	16.9	19.0	19.8	19.1
distillate selectivity to conversion	0.69	0.66	0.62	0.68	0.70	0.61	0.73	0.72	0.75	0.75	0.71	0.68
coal feed (MF lb/hr)	353	294	195	247	245	251	253	248	301	300	346	343
coal space velocity												
(MF lb/hr-cuft-cat per stage)	43.6	74.6	62.1	78.3	77.9	45.5	45.6	78.7	95.7	95.2	110.0	-
(relative to 251-IIB)	(100)	(171)	(142)	(180)	(179)	(104)	(105)	(181)	(220)	(218)	(252)	-
(MF lb/hr-cuft-reactors)(x1/C)	21.8	24.7	25.8	32.6	32.4	15.4	15.6	32.7	39.8	39.6	45.8	45.3
(relative to 251-IIB)	(100)	(113)	(118)	(150)	(149)	(71)	(72)	(150)	(183)	(182)	(210)	(208)
catalyst replacement (lb/ton MF coal)												
1st stage	-	-	-	-	-	-	3	-	-	-	-	-
2nd stage	3(b)	1(b)	1.5	1.5	1.5	3	-	3	3	3	3	-
total	3	1	1.5	1.5	1.5	3	3	3	3	3	3	-
(relative to 251-IIB)	(100)	(33)	(50)	(50)	(50)	(100)	(100)	(100)	(100)	(100)	(100)	-
reaction temperature (deg F)												
1st stage	819	840	840	841	852	825	790	825	825	825	824	824
2nd stage	743	730	788	790	790	790	774	761	790	790	810	809
(dT total to 251-IIB)	(base)	(+8)	(+66)	(+69)	(+76)	(+53)	(+2)	(+24)	(+53)	(+53)	(+72)	(+71)
(dT average to 251-IIB)	(base)	(+4)	(+33)	(+35)	(+38)	(+27)	(+1)	(+12)	(+27)	(+27)	(+36)	(+36)
distillate production												
(lb/hr)	196	129	102	127	132	127	142	135	182	171	185	152
(lb/hr-cuft-cat per stage)	24.3	32.9	32.4	40.2	42.0	23.1	25.6	42.8	57.9	54.2	59.0	-
(relative to 251-IIB)	100	135	134	166	173	95	105	176	239	223	243	-
(lb/hr-cuft-reactors)(x1/C)	12.1	10.9(16.4)(d)	13.5	16.7	17.5	7.8	8.8	17.8	24.1	22.5	24.5	20.1
(relative to 251-IIB)	100	90(136)(d)	111	138	144	64	72	147	199	186	202	166

Note: (a) Operation in thermal/catalytic mode except for 260D in catalytic/catalytic mode.
(b) Estimated at steady-state operation from batch aging data.
(c) Operation without interstage separation.
(d) Estimated for a half-volume reactor in the second stage considering low thermal conversion.
255B operated with half-/full-volume reactors with half catalyst charge in the second stage.
(e) Addition of Molyvan L as a precursor.

Abbreviations: Tex. Lig. - Texas Lignite; Sp. Cr. - Spring Creek; A1C - Amocat 1C, S324 - Shell 324; C324 - Criterion 324

Table 25

Projection for "All-Distillate" Product Slate by Using CSTR 1st Order Kinetics

Distillate Production Comparison (Run 263 with Black Thunder Coal)

run	263A	(proj)	263B	(Proj)	263C	(Proj)	263D	(Proj)	263E	(Proj)	263F	(Proj)	263G	(Proj)
coal	Black Thunder													
ash content (wt % MF coal)	7.7	7.7	7.0	7.0	7.6	7.6	7.2	7.2	7.1	7.1	6.9	6.9	7.1	7.1
yield (wt % MAF coal)														
C4+ distillate	47.9	55.3	51.8	58.6	52.1	56.6	54.6	58.5	60.7	62.3	61.5	60.7	57.0	60.0
resid	10.1	0.0	9.6	0.0	7.0	0.0	5.8	0.0	2.1	0.0	-0.6	0.0	4.3	0.0
organic rejection	21.3	21.0	17.0	17.0	17.5	18.0	15.8	16.0	14.1	14.0	14.5	15.0	14.0	14.0
coal feed (MF lb/hr)	299	252	299	259	294	284	260	254	261	260	254	235	264	254
coal space velocity														
(MF lb/hr-cuft-cat per stage)	-	-	-	-	-	-	-	-	-	-	-	-	-	-
(relative to 251-IIB)	-	-	-	-	-	-	-	-	-	-	-	-	-	-
(MF lb/hr-cuft-reactors)(x1/C)	39.5	33.3	39.3	34.1	38.8	37.5	34.3	33.5	34.5	34.3	33.6	33.7	34.8	33.5
(relative to 251-IIB)	181	1.67	180	1.71	178	1.88	157	1.68	158	1.71	154	1.69	160	1.68
distillate production														
(lb/hr)	132	129	144	141	142	149	132	138	147	150	145	133	140	142
(lb/hr-cuft-cat per stage)	-	-	-	-	-	-	-	-	-	-	-	-	-	-
(relative to 251-IIB)	-	-	-	-	-	-	-	-	-	-	-	-	-	-
(lb/hr-cuft-reactors)(x1/C)	17.5	17.0	18.9	18.6	18.7	19.6	17.4	18.2	19.5	19.9	19.2	19.0	18.4	18.7
(relative to 251-IIB)	145	1.48	156	1.62	154	1.71	144	1.58	161	1.73	159	1.66	152	1.62

run	263H	(proj)	263I	(Proj)	263J	(Proj)	263K	(Proj)	263L	(Proj)	263M	(Proj)
coal	Black Thunder											
ash content (wt % MF coal)	6.8	6.8	6.7	6.7	6.7	6.7	6.5	6.5	6.6	6.6	6.5	6.5
yield (wt % MAF coal)												
C4+ distillate	60.9	62.5	60.9	61.5	58.4	61.7	57.8	60.8	56.1	58.5	54.6	57.9
resid	2.4	0.0	1.2	0.0	4.2	0.0	4.8	0.0	3.6	0.0	5.1	0.0
organic rejection	13.8	14.0	13.7	14.0	13.4	13.0	13.5	14.0	15.9	16.0	14.8	15.0
coal feed (MF lb/hr)	264	271	343	360	345	341	344	319	345	324	348	304
coal space velocity												
(MF lb/hr-cuft-cat per stage)	-	-	108.8	114.3	109.5	108.3	109.4	101.2	109.7	103.0	110.5	96.6
(relative to 251-IIB)	-	-	249.5	2.86	251.1	2.71	250.9	2.53	251.6	2.57	253.4	2.41
(MF lb/hr-cuft-reactors)(x1/C)	34.9	35.8	45.3	47.6	45.6	45.1	45.5	42.1	45.6	42.8	46.0	40.2
(relative to 251-IIB)	160	1.79	208	2.38	209	2.25	209	2.11	209	2.14	211	2.01
distillate production												
(lb/hr)	150	158	195	207	188	197	186	181	181	177	178	165
(lb/hr-cuft-cat per stage)	-	-	61.8	65.6	59.7	62.3	59.1	57.5	57.5	56.3	56.4	52.3
(relative to 251-IIB)	-	-	254	2.85	246	2.71	243	2.50	237	2.45	232	2.27
(lb/hr-cuft-reactors)(x1/C)	19.8	20.9	25.7	27.3	24.8	26.0	24.6	23.9	23.9	23.4	23.5	21.8
(relative to 251-IIB)	164	1.81	213	2.38	205	2.26	203	2.08	197	2.03	194	1.89

Table 26

Projection for "All-Distillate" Product Slate by Using CSTR 1st Order Kinetics

Distillate Production Comparison (Run 262 with Black Thunder Coal)

run	251-IIB	(Proj)	262B	(Proj)	262C1	(Proj)	262D	(Proj)	262E	(Proj)	262F	(Proj)
coal	Wyodak		Black Thunder									
ash content (wt % MF coal)	8.8	8.8	6.3	6.3	6.5	6.5	6.4	6.4	6.3	6.3	6.5	6.5
yield (wt % MAF coal)												
C4+ distillate	61.0	63.0	58.0	60.6	64.7	62.5	60.8	61.0	57.2	57.0	47.5	55.4
resid	3.9	0.0	1.6	0.0	-4.1	0.0	0.3	0.0	-0.5	0.0	11.4	0.0
organic rejection	9.4	10.0	17.0	16.0	16.9	16.0	19.0	19.0	19.8	19.0	19.1	19.0
coal feed (MF lb/hr)	353	324	248	221	301	311	300	282	346	339	343	265
coal space velocity (MF lb/hr-cuft-cat per stage)	43.6	40.0	78.7	70.0	95.7	99.0	95.2	89.6	110.0	107.8	-	-
(relative to 251-IIB)	(100)	1.00	(181)	1.75	(220)	2.47	(218)	2.24	(252)	2.70	-	-
(MF lb/hr-cuft-reactors)(x1/C)	21.8	20.0	32.7	29.1	39.8	41.2	39.6	37.3	45.8	44.9	45.3	35.0
(relative to 251-IIB)	(100)	1.00	(150)	1.45	(183)	2.06	(182)	1.86	(210)	2.24	(208)	1.75
distillate production (lb/hr)	196	186	135	125	182	182	171	161	185	181	152	137
(lb/hr-cuft-cat per stage)	24.3	23.0	42.8	39.7	57.9	57.9	54.2	51.2	59.0	57.6	-	-
(relative to 251-IIB)	100	1.00	176	1.73	239	2.52	223	2.23	243	2.51	-	-
(lb/hr-cuft-reactors)(x1/C)	12.1	11.5	17.8	16.5	24.1	24.1	22.5	21.3	24.5	24.0	20.1	18.1
(relative to 251-IIB)	100	1.00	147	1.44	199	2.09	186	1.85	202	2.09	166	1.58

Table 27

Projection for "All-Distillate" Product Slate by Using CSTR 1st Order Kinetics

Distillate Production Comparison (Runs 258 and 260 with Black Thunder Coal and Run 255 with Texas Lignite)

run	251-IIB	(Proj)	255B	(Proj)	258E	(Proj)	258H	(Proj)	258I	(Proj)	260C(c)	(Proj)	260D	(Proj)
coal	Wyodak		Tex. Lig.		Sp.Cr.		Black Thunder							
ash content (wt % MF coal)	8.8	8.8	12.1	12.1	5.6	5.6	7.3	7.3	7.3	7.3	6.2	6.2	6.2	6.2
yield (wt % MAF coal)														
C4+ distillate	61.0	63.0	50.1	50.0	55.3	62.0	55.4	60.0	58.1	60.0	54.1	59.0	59.8	60.0
resid	3.9	0.0	2.6	0.0	8.6	0.0	4.9	0.0	2.6	0.0	7.5	0.0	0.3	0.0
organic rejection	9.4	10.0	21.4	23.0	10.4	10.0	13.8	14.0	14.2	14.0	10.7	11.0	18.5	19.0
coal feed (MF lb/hr)	353	324	294	288	195	170	247	227	245	226	251	224	253	252
coal space velocity														
(MF lb/hr-cuft-cat per stg)	43.6	40.0	74.6	73.0	62.1	54.0	78.3	72.0	77.9	72.0	45.5	40.6	45.6	45.5
(relative to 251-IIB)	(100)	1.00	(171)	1.83	(142)	1.35	(180)	1.80	(179)	1.80	(104)	1.02	(105)	1.14
(MF lb/hr-cuft-reacts)(1/C)	21.8	20.0	24.7	24.2	25.8	22.4	32.6	30.0	32.4	29.9	15.4	13.7	15.6	15.6
(relative to 251-IIB)	(100)	1.00	(113)	1.21	(118)	1.12	(150)	1.50	(149)	1.50	(71)	0.69	(72)	0.78
distillate production (lb/hr)	196	186	129	126	102	99	127	126	132	126	127	124	142	142
(lb/hr-cuft-cat per stage)	24.3	23.0	32.9	32.1	32.4	31.6	40.2	40.0	42.0	40.0	23.1	22.5	25.6	25.6
(relative to 251-IIB)	100	1.00	135	1.40	134	1.38	166	1.74	173	1.74	95	0.98	105	1.11
(lb/hr-cuft-reactors)(x1/C)	12.1	11.5	10.9(16.4)(a)	10.6(15.9)(a)	13.5	13.1	16.7	16.7	17.5	16.7	7.8	7.6	8.8	8.8
(relative to 251-IIB)	100	1.00	90(136)(a)	0.88(1.39)(a)	111	1.14	138	1.45	144	1.45	64	0.66	72	0.76

Note: (a) Estimated for a half-volume reactor in the second stage considering low thermal conversion.
255B operated with half-/full-volume reactors with half catalyst charge in the second stage.

Abbreviations: Tex. Lig. - Texas Lignite; Sp. Cr. - Spring Creek

Table 28

COMPOSITION OF RUN 263 TOTAL OIL BLENDS

		T102 Vacuum Column	
Period	Atm. Overheads V161 wt %	Overheads V182 wt %	Bottoms V1074 wt %
263A	27.2	72.8	
263B	29.3	70.7	
263C	30.7	69.3	
263D	34.5	65.5	
263E	40.6	59.4	
263F	47.9	10.5	41.6
263G	43.6	56.4	
263H	41.9	58.1	
263J	45.9	54.1	
263K	38.8	43.7 + 17.5(*)	
263M	38.1	61.9	

(*) Distilled in Lab from V1074 due to T102 vacuum column problems. Distilled 9.2 wt% from V1074.

Table 29

PROPERTIES OF TOTAL OIL PRODUCTS

PERIOD	263A	263B	263C	263D	263E	263F	263G	263H	263J	263K	263M
Elemental wt %											
C	85.33	85.72	85.29	84.50	85.10	85.83	85.06	85.09	86.26	86.70	86.85
H	10.16	10.23	10.41	9.93	10.07	9.97	10.23	10.36	11.04	10.69	10.82
N (ppm)	8475	10698	9376	9159	8819	8827	8644	8588	6055	6357	6597
S	0.07	0.06	0.04	0.06	0.02	0.02	0.04	0.05	0.04	0.02	0.03
O (diff.)	3.59	2.92	3.32	4.59	3.93	3.30	3.81	3.64	2.05	1.95	1.64
Gravity (°API)	15.3	14.4	14.5	15.8	15.3	14.7	17.7	17.6	22.1	19.6	19.0
G.C. Sim. Dist., °F											
IBP	105	134	133	97	97	88	97	97	97	97	97
10%	359	359	358	355	344	240	286	293	264	306	332
20%	460	428	451	405	400	347	368	370	359	405	424
30%	513	486	503	465	465	359	455	451	435	465	492
40%	590	554	563	515	501	415	496	496	486	531	549
50%	662	624	614	570	564	462	549	558	531	598	601
60%	712	676	663	626	628	496	606	607	591	655	663
70%	745	722	713	665	694	568	643	657	643	711	711
80%	775	771	747	712	740	793	676	686	685	740	740
90%	807	803	783	758	778	850	721	740	740	800	771
95%	841	825	813	783	820	876	747	760	755	828	796
EP	871	862	862	856	867	1049	827	814	814	885	866
Distribution, Wt % (*)											
IBP-350°F	10.2	7.6	7.4	7.8	9.7	19.2	12.4	12.3	15.7	11.9	10.7
350-450°F	10.8	14.4	11.2	15.0	14.3	24.5	15.6	16.6	13.9	12.0	10.6
450-650°F	36.5	32.7	37.2	42.2	38.3	28.7	43.1	39.7	40.3	34.1	35.8
650-EP °F	42.5	45.3	44.2	35.0	37.7	27.6	28.9	31.4	30.1	42.0	42.9
EP °F (D1160)	762	759	754	728	755	963	708	719	717	792	745

(*) preformed by laboratory distillation

Table 30

PROPERTIES OF NAPHTHA (IBP-350°F)

PERIOD	263A	263B	263C	263D	263E	263F	263G	263H
Elemental wt %								
C	84.43	83.24	82.98	84.62	82.69	84.01	83.62	
H	12.80	12.91	12.89	13.55	12.82	13.34	12.91	
N (ppm)	1727	2203	2257	2139	3145	2759	2751	2227
S	0.12	0.06	0.08	0.07	0.05	0.05	0.07	0.06
O (diff.)	2.48	3.57	3.82	1.55	4.12	2.32	3.12	
Gravity (°API)	42.6	45.2	47.5	48.6	46.2	48.9	48.5	50.4
G.C. Sim. Dist., °F								
IBP	146	70	91	71	72	78	71	71
10%	175	156	156	156	150	145	133	144
20%	217	176	176	190	176	170	165	165
30%	240	231	218	234	228	209	191	209
40%	245	241	241	243	241	231	237	240
50%	273	267	266	265	265	248	247	246
60%	300	295	291	291	294	281	281	281
70%	329	330	330	339	335	305	308	303
80%	329	357	350	358	357	336	354	335
90%	350	359	358	359	358	358	358	356
95%	364	366	359	364	360	359	360	359
EP	386	405	382	383	383	383	383	375

Table 31

PROPERTIES OF MIDDLE DISTILLATE (350-450°F)

PERIOD	263A	263B	263C	263D	263E	263F	263G	263H
Elemental wt %								
C	83.82	82.71	83.68	84.07	83.66	83.91	83.44	
H	10.28	9.94	10.55	10.48	10.27	10.38	10.17	
N (ppm)	3480	4472	4031	3974	4252	4292	4426	4634
S	0.06	0.03	0.03	0.02	0.11	0.01	0.02	0.02
O (diff.)	5.49	6.87	5.34	5.03	5.53	5.27	5.93	
Gravity (°API)	20.3	16.9	19.4	19.2	19.1	18.6	18.6	19.6
G.C. Sim. Dist.,°F								
IBP	291	311	312	312	292	310	341	309
10%	359	360	359	360	360	360	360	360
20%	359	368	360	367	365	370	370	370
30%	381	394	376	387	383	385	385	385
40%	405	400	394	394	405	405	405	405
50%	424	405	400	405	424	414	424	418
60%	433	435	420	427	435	424	433	433
70%	447	457	448	456	458	458	458	452
80%	460	465	460	460	460	460	460	460
90%	476	492	475	473	476	477	470	477
95%	487	496	488	487	488	488	489	488
EP	496	568	496	496	496	496	496	496

Table 32

PROPERTIES OF MIDDLE DISTILLATE (450-650°F)

PERIOD	263A	263B	263C	263D	263E	263F	263G	263H
Elemental wt %								
C	84.72	85.00	85.71	85.39	85.54	84.69	85.44	
H	9.98	10.13	10.47	10.16	9.84	9.93	9.96	
N (ppm)	9981	11152	10656	10868	10185	11495	10635	10899
S	0.07	0.05	0.05	0.03	0.01	0.01	0.04	0.03
O (diff.)	4.23	3.70	2.70	3.33	3.59	4.22	3.50	
Gravity (°API)	11.6	11.6	12.5	12.3	11.7	12.9	12.5	12.1
G.C. Sim. Dist., °F								
IBP	405	388	424	405	395	385	365	360
10%	475	466	492	466	466	460	466	465
20%	502	492	510	496	494	492	492	492
30%	531	507	531	531	500	496	508	508
40%	555	548	557	555	515	511	549	549
50%	581	568	578	575	551	531	573	571
60%	614	576	620	619	572	555	590	600
70%	637	604	643	643	603	576	614	619
80%	660	632	663	660	627	622	631	637
90%	676	663	676	676	643	663	643	660
95%	696	676	685	685	676	676	657	676
EP	721	722	722	722	722	722	676	685

Table 33

PROPERTIES OF VACUUM GAS OIL (650°F+)

PERIOD	263A	263B	263C	263D	263E	263F	263G	263H
Elemental wt %								
C	87.56	87.47	87.54	87.04	87.57	88.55	86.31	
H	10.25	9.90	9.79	9.44	9.58	9.13	9.59	
N (ppm)	8881	11110	11076	12267	10953	10739	12449	12132
S	0.05	0.02	0.03	0.03	0.01	0.01	0.03	0.03
O (diff.)	1.25	1.50	1.53	2.26	1.74	1.24	2.82	
Gravity (°API)	12.4	7.1	6.7	5.4	3.3	2.7	8.2	2.1
G.C. Sim. Dist., °F								
IBP	722	733	740	525	740	740	685	740
10%	722	733	740	720	740	775	680	740
20%	752	745	751	740	746	807	685	740
30%	771	759	762	740	759	838	706	740
40%	771	771	783	760	777	855	740	750
50%	788	780	795	778	789	874	748	760
60%	813	790	810	789	799	895	753	778
70%	813	810	838	797	813	920	770	790
80%	838	830	846	821	823	951	794	799
90%	852	856	860	838	838	1027	803	822
95%	868	874	874	851	867	1104	817	838
EP	937	959	937	937	937	1213	925	928

Table 34

PHASE 3-4 CRITERIA
UNIT AND ELEMENTAL CLOSURE ERRORS
WT % MAF COAL

PERIODS ->	263A	263B	263C	263D	263E	263F	263G	263H
CCR1 UNIT DATA								
CARBON	1.69±2.24	-0.40±3.35	-0.13±1.93	0.56±1.41	4.50±2.82	3.61±2.01	4.00±2.79	2.40±1.26
HYDROGEN	-0.24±0.65	-0.33±0.66	-1.11±0.86	-0.52±0.73	-0.46±0.60	-0.09±0.53	-0.26±0.42	0.21±0.46
NITROGEN	-0.09±0.03	-0.09±0.15	-0.16±0.28	-0.18±0.11	-0.06±0.30	-0.19±0.03	-0.18±0.54	-0.03±0.18
SULFUR	0.01±0.16	-0.70±2.26	0.28±0.34	0.29±0.30	0.13±0.12	0.30±0.07	0.08±0.25	0.63±0.29
OXYGEN	-0.45±0.70	0.48±3.44	-0.05±0.67	-2.45±2.55	-3.55±1.99	-2.19±3.16	-3.50±0.28	-3.19±1.78
ASH	-0.03±2.00	2.05±1.39	1.53±2.22	2.81±1.91	0.49±1.33	-0.40±1.47	0.44±1.95	1.10±2.45
CCR2 UNIT DATA								
CARBON	-2.21±3.74	-4.07±7.75	-5.27±14.2	-7.93±12.6	-7.69±7.36	2.68±16.1	-7.16±6.23	-12.0±4.54
HYDROGEN	0.33±0.41	1.11±1.03	1.12±1.56	0.51±1.24	0.40±0.52	0.64±2.01	0.18±0.99	-0.89±0.68
NITROGEN	0.13±0.09	0.06±0.17	-0.07±0.26	-0.10±0.15	-0.05±0.22	0.00±0.09	-0.08±0.63	-0.22±0.17
SULFUR	0.40±0.21	0.74±2.23	-0.02±0.41	-0.07±0.24	0.24±0.29	0.16±0.33	0.32±0.21	-0.42±0.18
OXYGEN	2.62±1.68	0.64±3.70	0.73±0.55	4.17±2.42	3.82±1.15	0.91±1.03	4.46±1.61	2.01±1.95
ASH	1.24±2.09	-2.95±1.26	-3.07±3.00	-4.16±3.49	-1.86±1.86	-0.36±0.70	-0.93±3.43	-1.94±1.37
ROSE-SR UNIT DATA								
CARBON	-0.54±0.30	-0.43±0.27	-0.68±0.39	-1.04±0.66	-0.95±0.46	-0.13±1.10	-0.88±0.47	-0.58±0.21
HYDROGEN	-0.08±0.04	-0.06±0.05	-0.20±0.06	-0.07±0.17	-0.04±0.05	-0.10±0.06	-0.06±0.02	-0.05±0.03
NITROGEN	0.00±0.02	-0.01±0.02	0.03±0.03	0.00±0.04	0.00±0.02	0.02±0.04	0.00±0.01	0.00±0.02
SULFUR	0.01±0.04	0.18±0.50	0.02±0.04	0.03±0.08	-0.05±0.07	-0.05±0.05	-0.04±0.05	0.04±0.03
OXYGEN	0.22±0.32	0.24±0.65	0.41±0.27	0.23±0.58	0.41±0.33	0.24±0.22	0.43±0.24	0.27±0.19
ASH	0.40±0.47	0.09±0.52	0.44±0.12	0.26±0.23	0.43±0.06	-0.08±0.81	0.50±0.34	0.31±0.31
TOTALED DATA								
CARBON	-1.06±4.60	-4.90±8.43	-6.07±14.2	-8.41±12.6	-4.13±7.87	6.17±15.1	-4.04±7.94	-10.2±4.45
HYDROGEN	0.02±0.87	0.72±0.91	-0.19±1.85	-0.08±0.65	-0.10±0.72	0.45±1.44	-0.14±1.02	-0.73±0.65
NITROGEN	0.04±0.08	-0.05±0.10	-0.20±0.11	-0.28±0.19	-0.11±0.23	-0.17±0.09	-0.25±0.10	-0.24±0.08
SULFUR	0.43±0.11	0.22±0.25	0.28±0.23	0.25±0.43	0.32±0.17	0.40±0.26	0.36±0.27	0.25±0.25
OXYGEN	2.39±1.67	1.36±1.20	1.09±0.75	1.95±1.33	0.69±1.30	-1.04±3.32	1.39±1.15	-0.91±0.72
ASH	1.61±1.51	-0.81±1.19	-1.11±1.94	-1.08±3.71	-0.94±1.49	-0.84±2.97	0.01±1.89	-0.53±1.30
ABSOLUTE SUM DATA								
CARBON	5.94±2.95	9.88±5.62	14.99±6.60	14.30±7.81	13.14±7.45	16.59±8.99	12.04±4.99	15.02±5.29
HYDROGEN	1.11±0.26	1.76±1.08	2.53±1.61	1.69±1.46	1.14±0.69	2.26±0.51	1.22±0.30	1.35±0.59
NITROGEN	0.22±0.11	0.27±0.22	0.47±0.27	0.35±0.15	0.39±0.27	0.27±0.06	0.87±0.69	0.40±0.15
SULFUR	0.57±0.23	2.91±3.63	0.68±0.37	0.59±0.21	0.50±0.20	0.55±0.31	0.58±0.14	1.09±0.42
OXYGEN	3.61±2.02	6.63±2.29	1.68±0.65	7.14±4.58	7.78±3.07	4.38±1.66	8.40±1.65	5.66±3.32
ASH	3.59±2.09	5.44±2.48	5.41±4.51	7.29±4.26	3.62±1.72	2.52±0.60	4.74±2.54	4.63±1.84

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

Table 34 (Continued)

PHASE 3-4 CRITERIA
UNIT AND ELEMENTAL CLOSURE ERRORS
WT % MAF COAL

PERIODS ->	263I	263J	263K	263L	263M
CCR1 UNIT DATA					
CARBON	0.14±1.76	-1.80±1.48	-1.35±2.15	-1.06±0.54	-0.66±1.02
HYDROGEN	-0.33±0.18	-0.24±0.55	-0.53±0.09	-0.20±0.39	-0.83±0.32
NITROGEN	0.20±0.28	-0.01±0.19	0.04±0.02	0.00±0.30	0.03±0.10
SULFUR	0.53±0.25	-0.17±0.22	0.19±0.26	0.19±0.14	0.32±0.12
OXYGEN	0.26±2.21	3.07±1.46	1.40±3.18	2.02±1.53	1.46±1.11
ASH	0.08±0.92	-0.03±1.71	1.13±1.10	-0.12±1.52	0.50±1.04
CCR2 UNIT DATA					
CARBON	-5.24±11.4	6.16±6.77	-3.85±4.12	1.74±6.33	0.85±2.01
HYDROGEN	0.13±1.52	1.04±0.62	0.89±0.55	0.29±0.72	0.62±0.30
NITROGEN	-0.66±0.10	-0.40±0.22	-0.46±0.04	-0.30±0.37	-0.33±0.07
SULFUR	-0.81±0.26	0.02±0.28	-0.55±0.10	-0.19±0.19	0.04±0.31
OXYGEN	3.49±2.27	-0.49±1.47	1.98±1.22	1.65±1.52	0.43±0.99
ASH	-2.32±1.31	0.33±1.94	-2.88±0.87	-0.49±2.76	-0.79±1.59
ROSE-SR UNIT DATA					
CARBON	0.05±0.64	-0.41±0.38	0.63±0.70	0.01±0.14	-0.12±0.08
HYDROGEN	-0.11±0.08	-0.09±0.05	0.02±0.07	-0.08±0.10	-0.11±0.07
NITROGEN	0.01±0.01	0.01±0.01	0.03±0.04	0.00±0.02	0.00±0.01
SULFUR	-0.02±0.07	-0.02±0.05	0.08±0.21	-0.02±0.02	-0.01±0.07
OXYGEN	0.16±0.24	0.26±0.17	0.00±0.32	-0.11±0.16	0.10±0.19
ASH	0.27±0.59	0.25±0.29	-0.20±0.48	0.20±0.14	0.17±0.26
TOTALED DATA					
CARBON	-5.06±12.7	3.95±7.33	-4.56±4.59	0.69±6.78	0.07±2.78
HYDROGEN	-0.31±1.38	0.72±0.37	0.38±0.60	0.01±0.88	-0.32±0.43
NITROGEN	-0.44±0.20	-0.40±0.18	-0.39±0.07	-0.30±0.08	-0.29±0.07
SULFUR	-0.30±0.11	-0.17±0.12	-0.28±0.06	-0.02±0.07	0.35±0.37
OXYGEN	3.90±2.75	2.84±0.67	3.38±2.21	3.56±0.66	2.00±0.63
ASH	-1.97±1.33	0.55±0.83	-1.95±0.89	-0.41±1.33	-0.13±0.85
ABSOLUTE SUM DATA					
CARBON	11.54±6.77	9.31±5.85	5.97±3.83	5.07±4.75	2.74±1.38
HYDROGEN	1.54±1.00	1.55±0.89	1.47±0.54	0.77±0.63	1.56±0.48
NITROGEN	0.88±0.37	0.54±0.24	0.54±0.05	0.57±0.42	0.42±0.08
SULFUR	1.39±0.52	0.41±0.40	0.96±0.16	0.41±0.31	0.61±0.21
OXYGEN	5.32±3.06	4.47±2.23	4.77±1.12	3.93±0.53	2.48±0.75
ASH	3.38±1.47	2.81±2.51	4.42±2.02	3.02±2.63	2.13±2.07

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

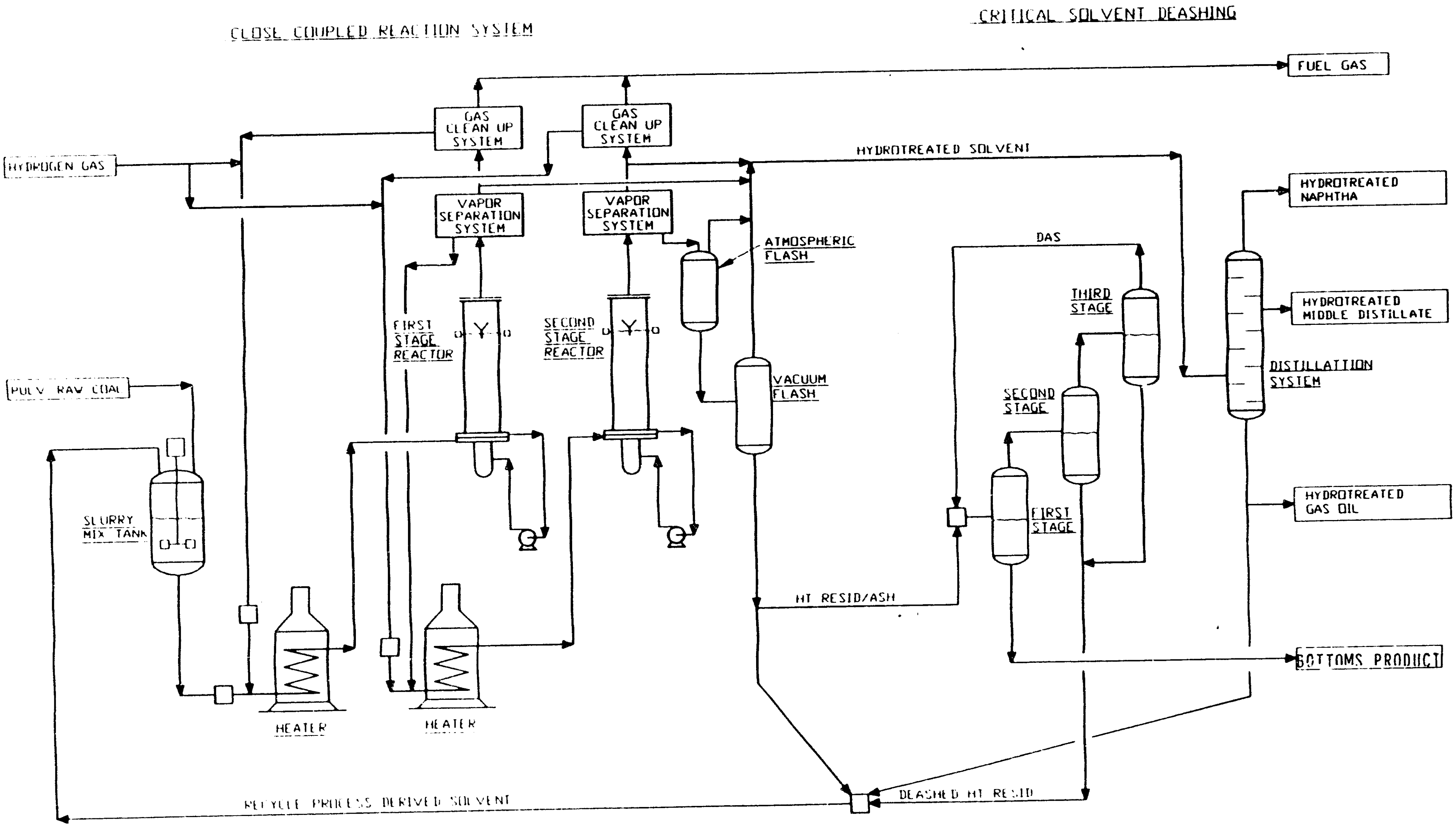
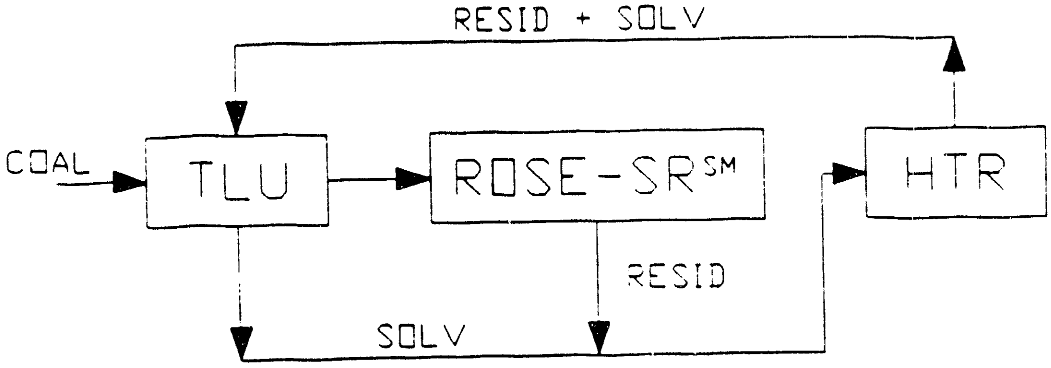
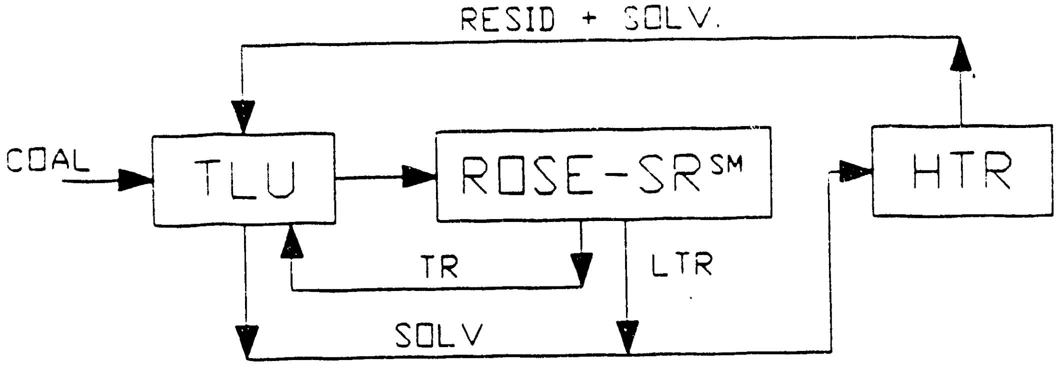


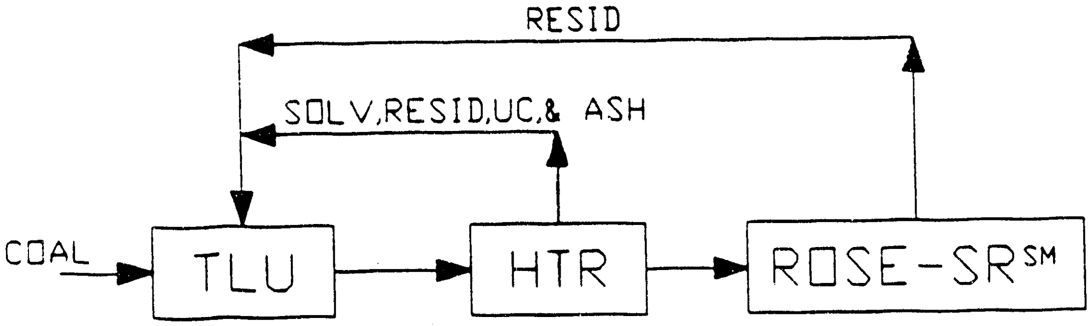
FIGURE 1. SIMPLIFIED FLOW DIAGRAM OF THE CLOSE-COUPLED ITSL COAL LIQUEFACTION SYSTEM WITH INTERSTAGE SEPARATION



INTEGRATED TWO-STAGE LIQUEFACTION

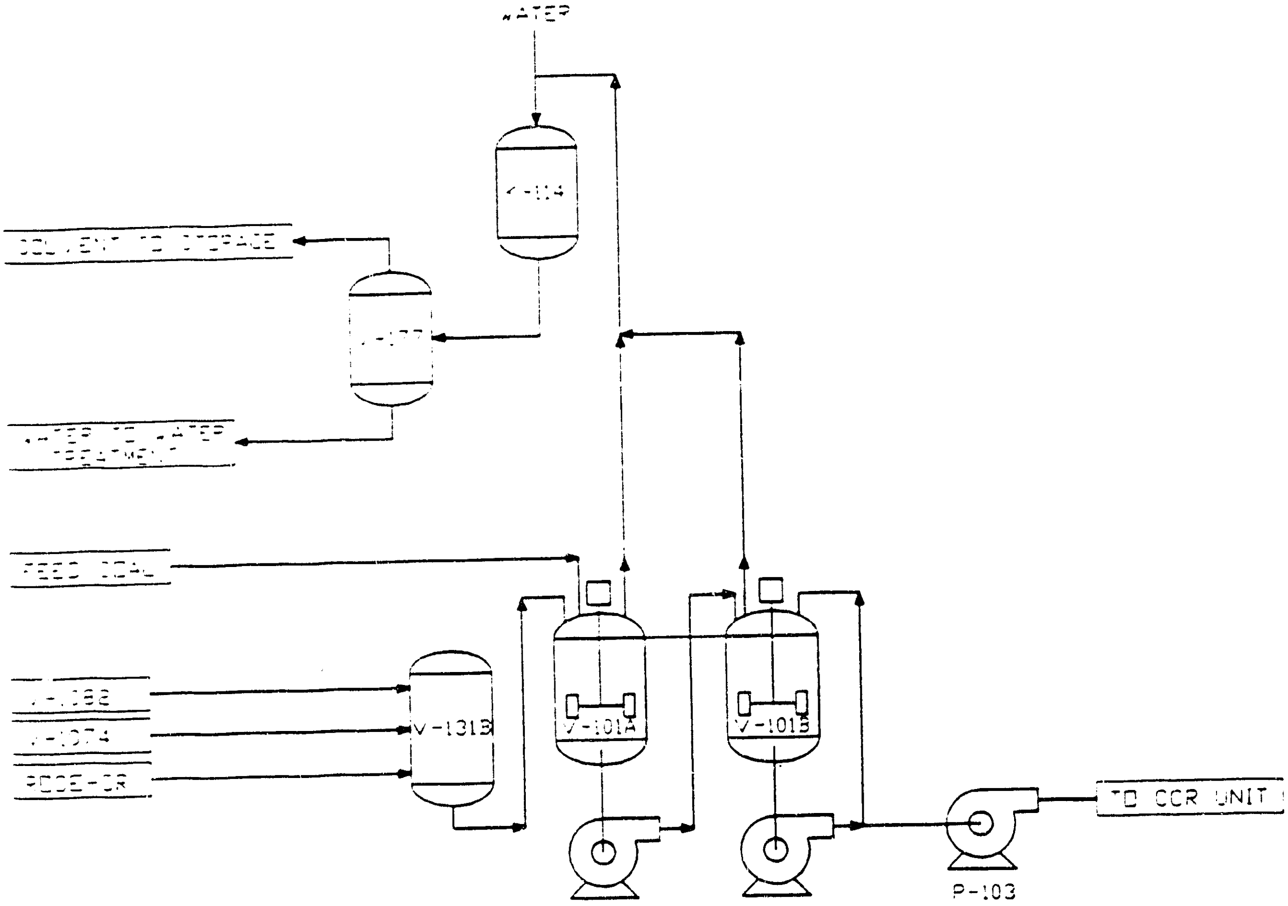


DOUBLE INTEGRATED TWO-STAGE LIQUEFACTION



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
P103	HIGH PRESSURE FEED PUMP
K-114	WATER CONTACT SCRUBBER
V-177	SETTLING TANK

FIGURE 3. FLOW DIAGRAM OF COAL SLURRY PREPARATION SYSTEM

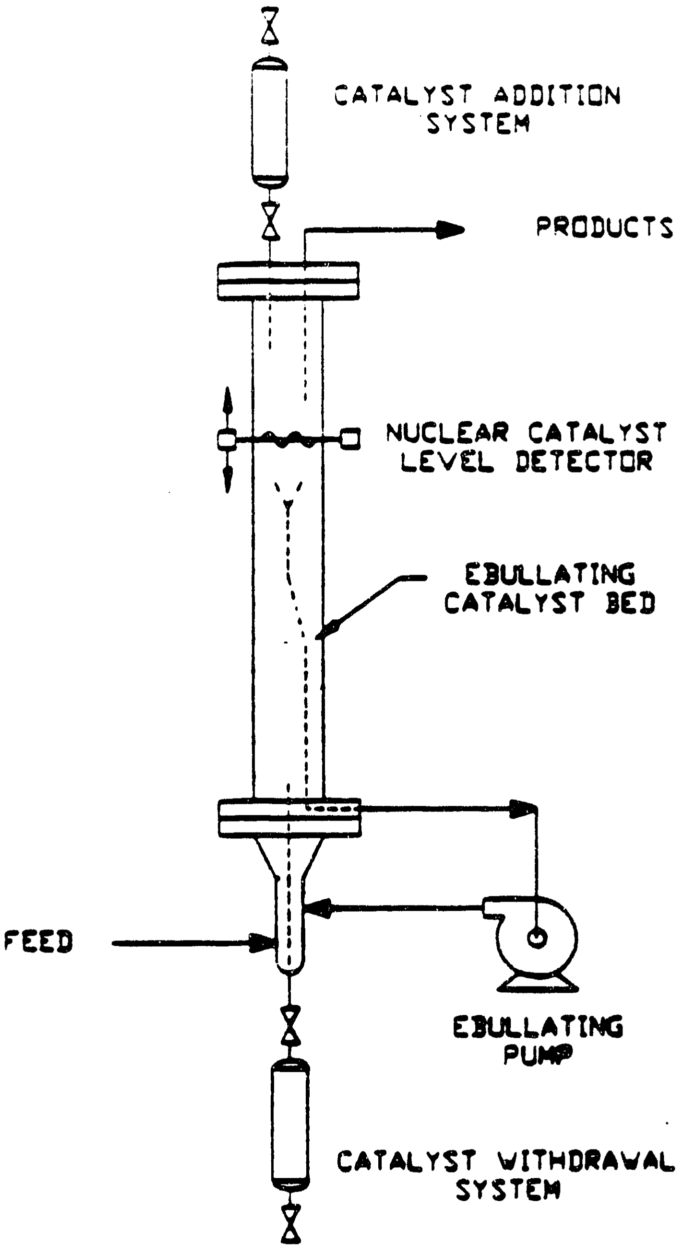


FIGURE 4. H-OIL® EBULLATED BED REACTOR

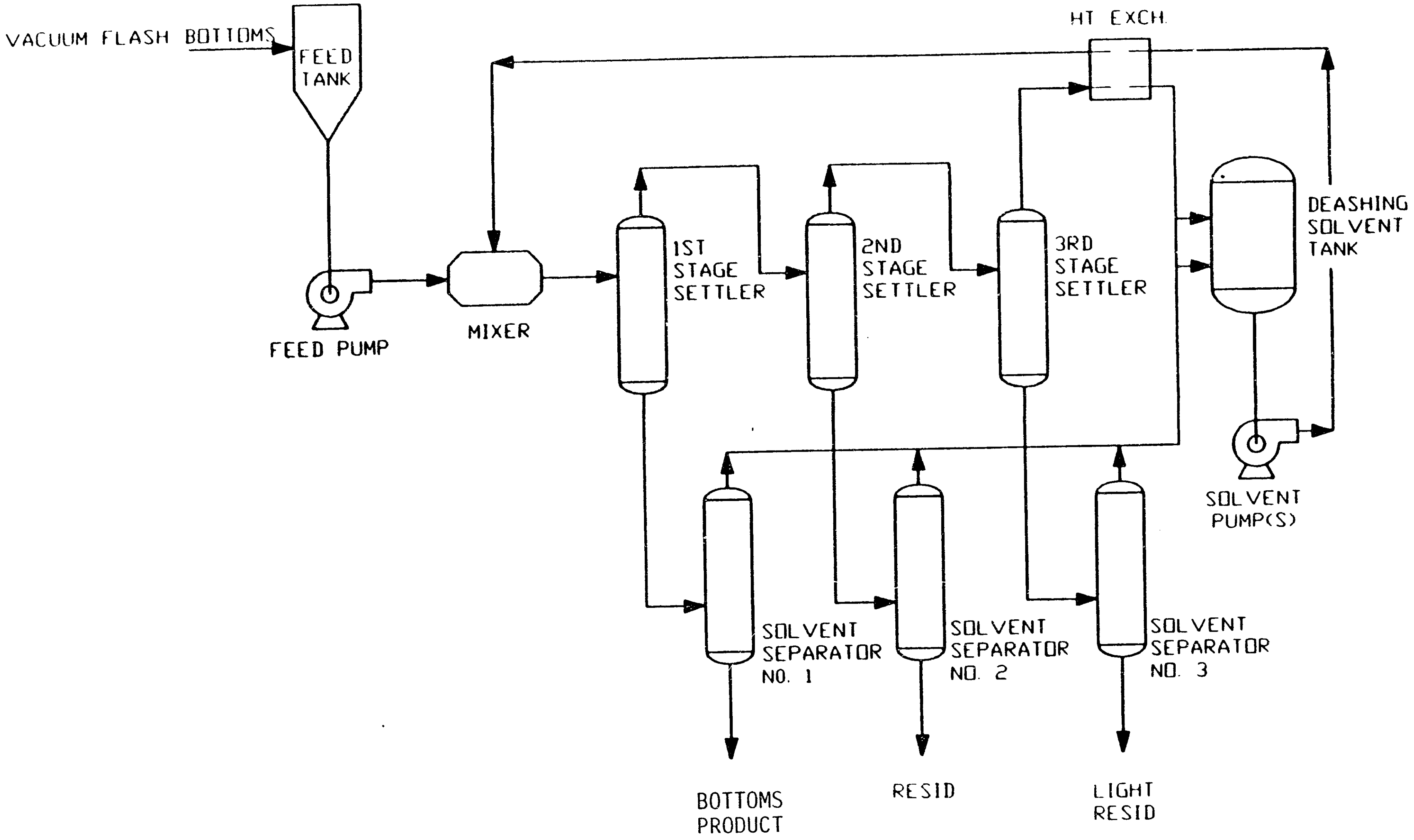


FIGURE 5. FLOW DIAGRAM OF THE RESIDUUM OIL SUPERCRITICAL EXTRACTION - SOLIDS REJECTION UNIT

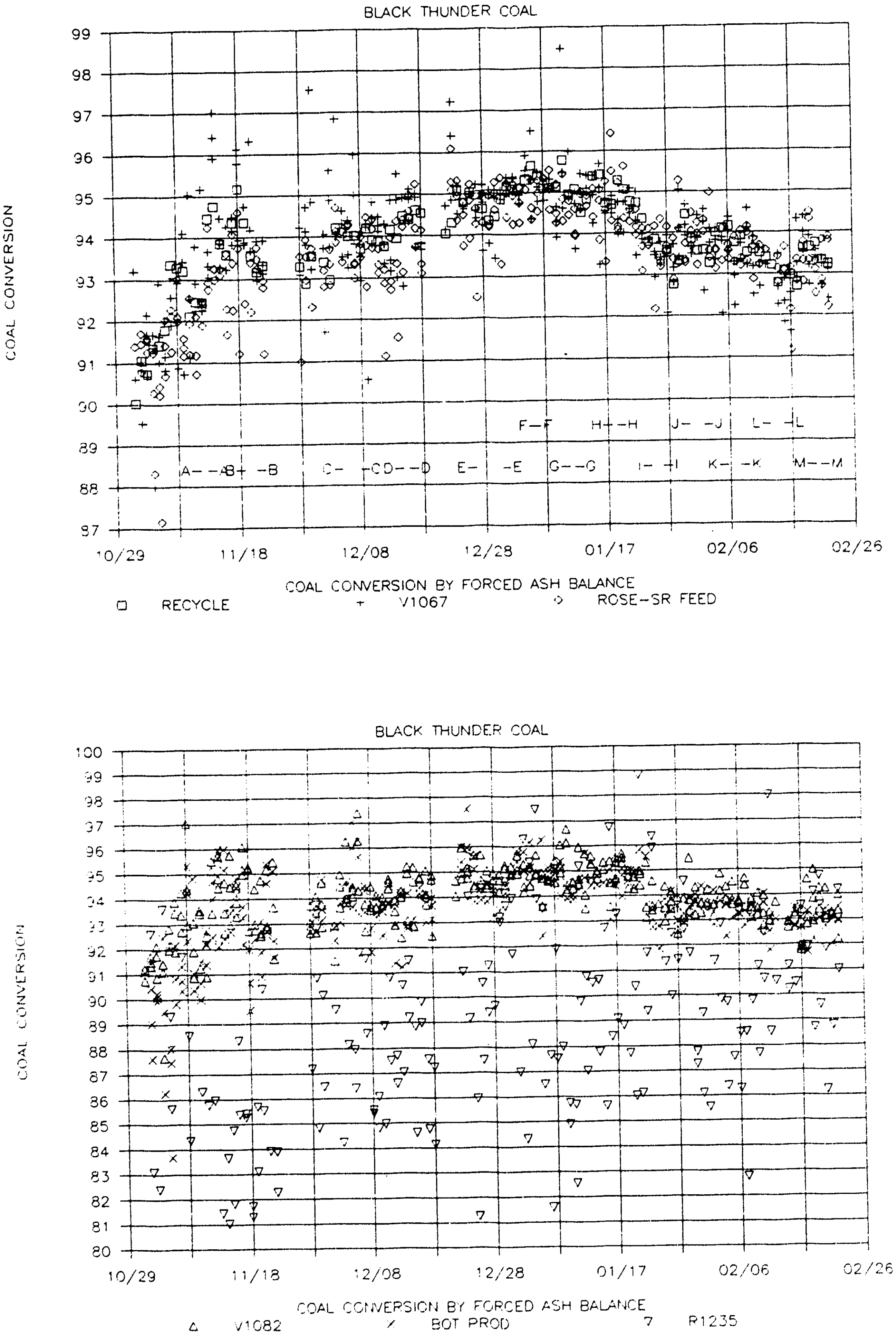


FIGURE 6. COAL CONVERSION BY FORCED ASH BALANCE

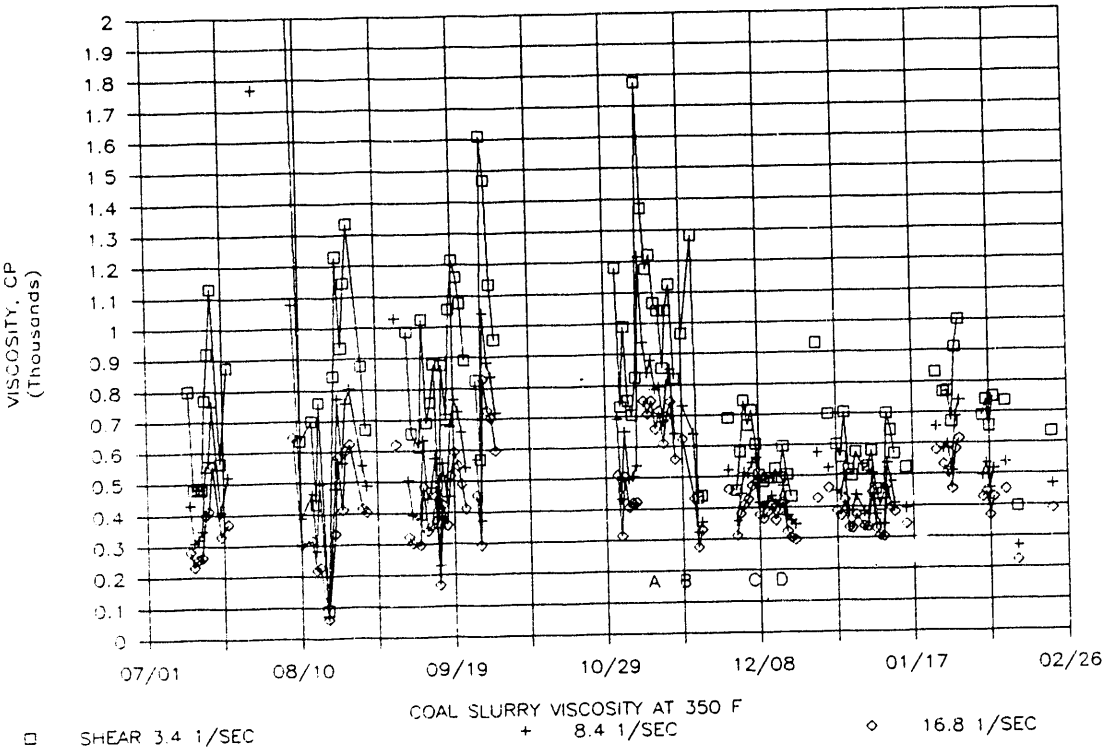
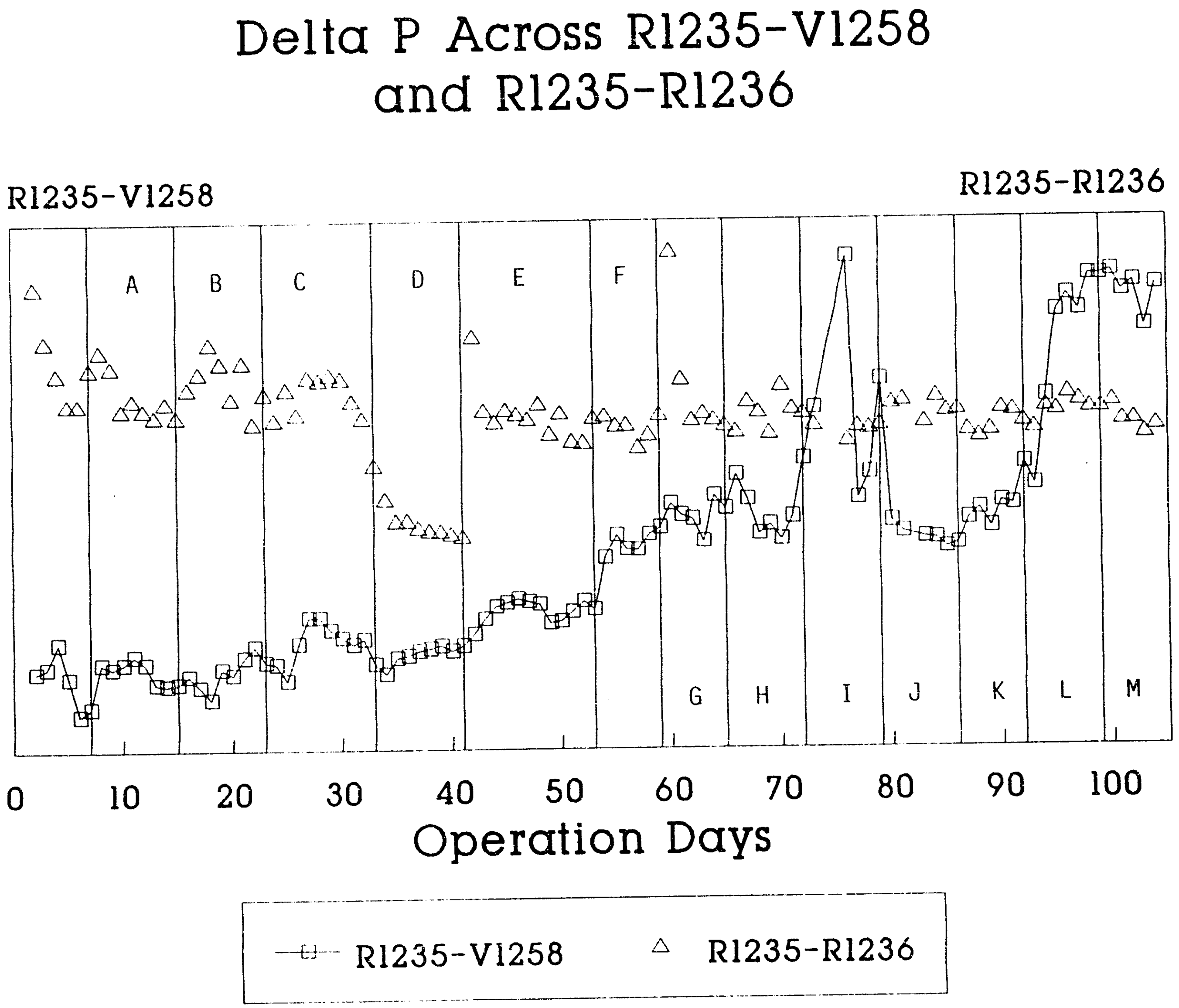


FIGURE 7. COAL SLURRY VISCOSITY



Day 1 = 11/01/91; Day 70 = 01/19/92
Day 30 = 12/07/91; Day 90 = 02/08/92
Day 50 = 12/30/91;

FIGURE 8. PRESSURE DROP BETWEEN FIRST REACTOR AND INTERSTAGE SEPARATOR AND BETWEEN THE REACTORS FOR RUN 263

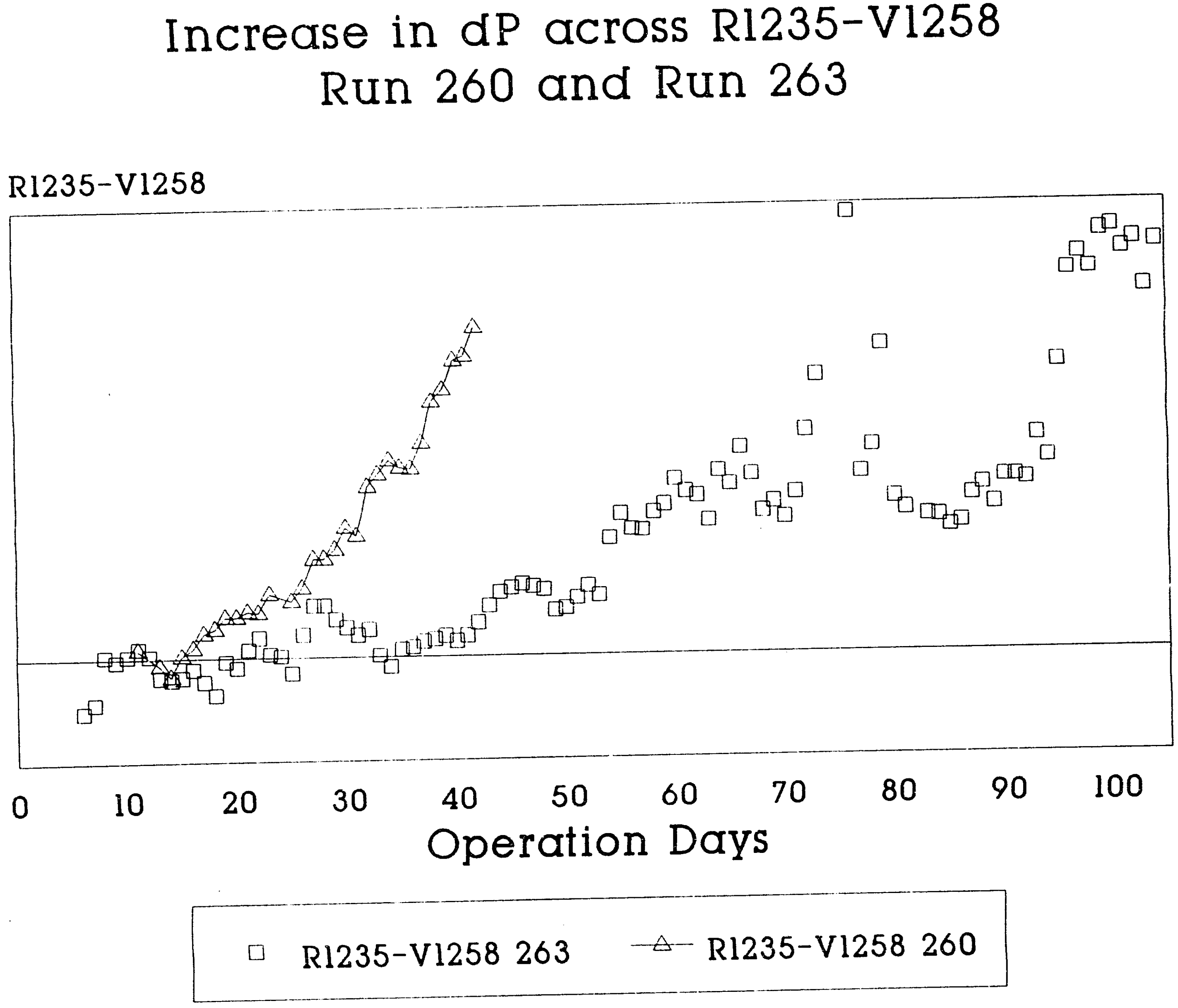


FIGURE 9. PRESSURE DROP COMPARISON BETWEEN FIRST REACTOR AND INTERSTAGE SEPARATOR FOR RUNS 263 and 260

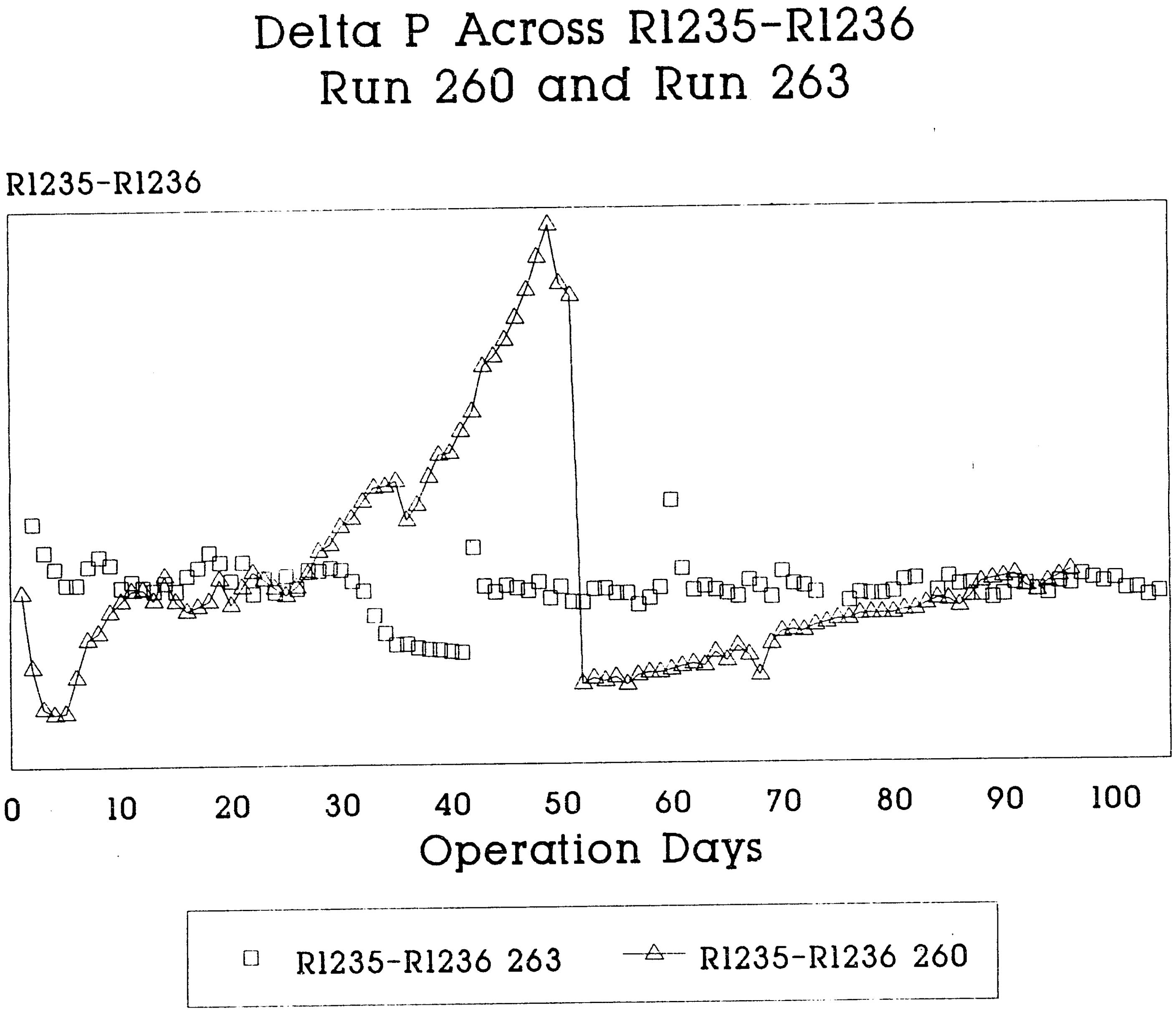
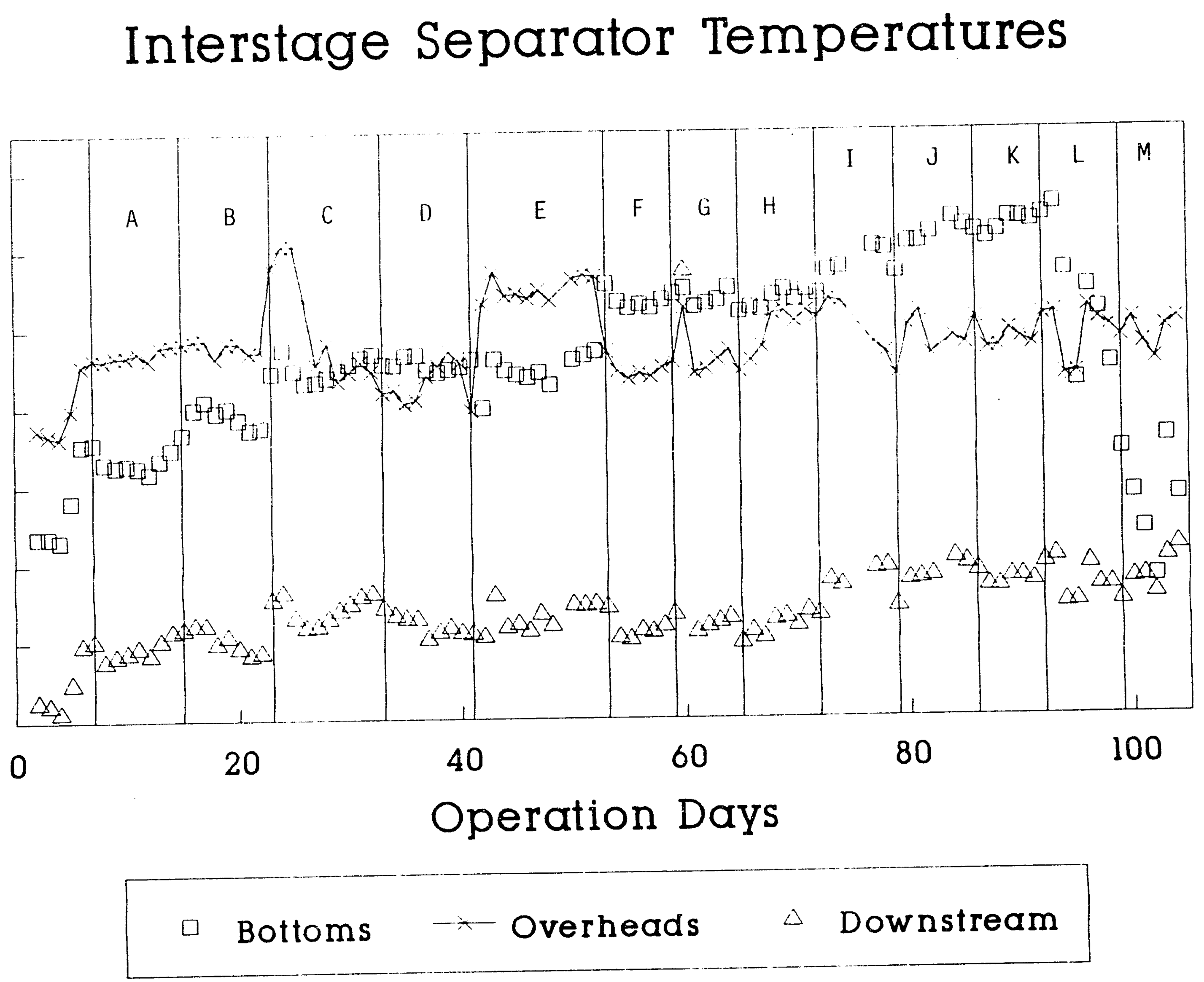
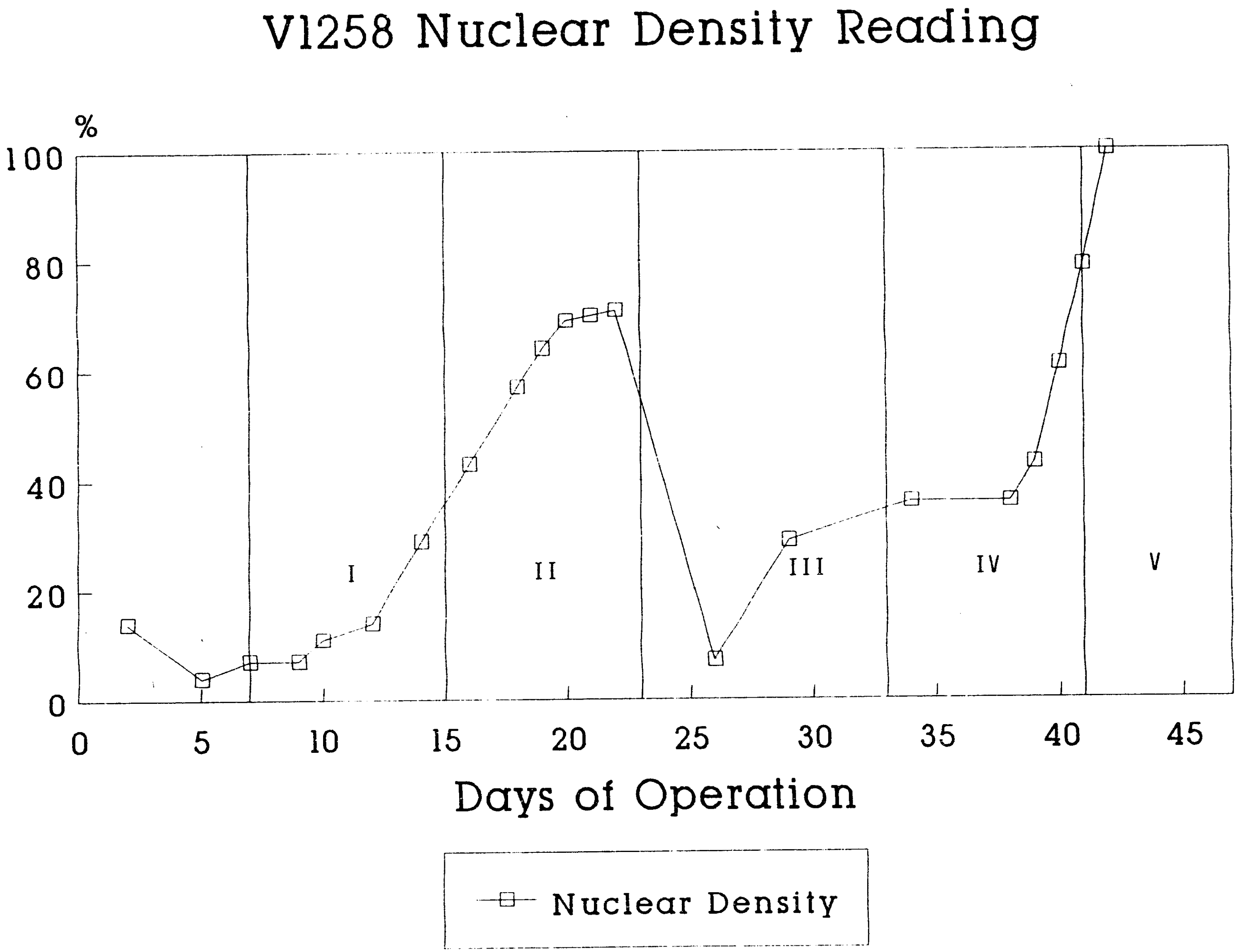


FIGURE 10. PRESSURE DROP COMPARISON BETWEEN THE REACTORS FOR RUNS 263 AND 260



Day 1 = 11/01/91; Day 70 = 01/19/92
Day 30 = 12/07/91; Day 90 = 02/08/92
Day 50 = 12/30/91

FIGURE 11. INTERSTAGE SEPARATOR TEMPERATURE TREND DATA FOR RUN 263



Day 1 = 11/01/91; Day 30 = 12/07/91
Day 10 = 11/10/91; Day 40 = 12/17/91
Day 20 = 11/20/91

FIGURE 12. INTERSTAGE SEPARATOR NUCLEAR DENSITY GAUGE READING TREND

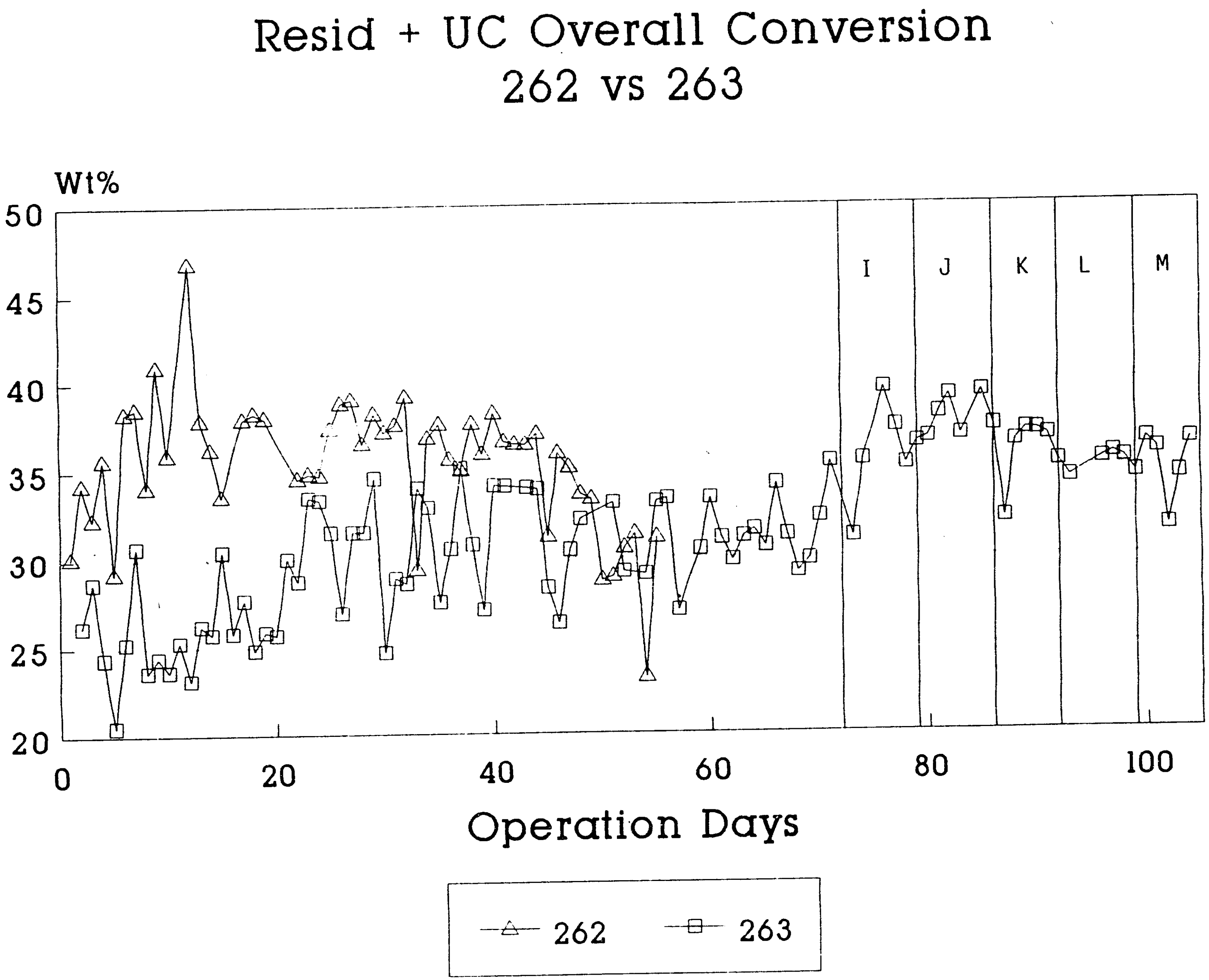
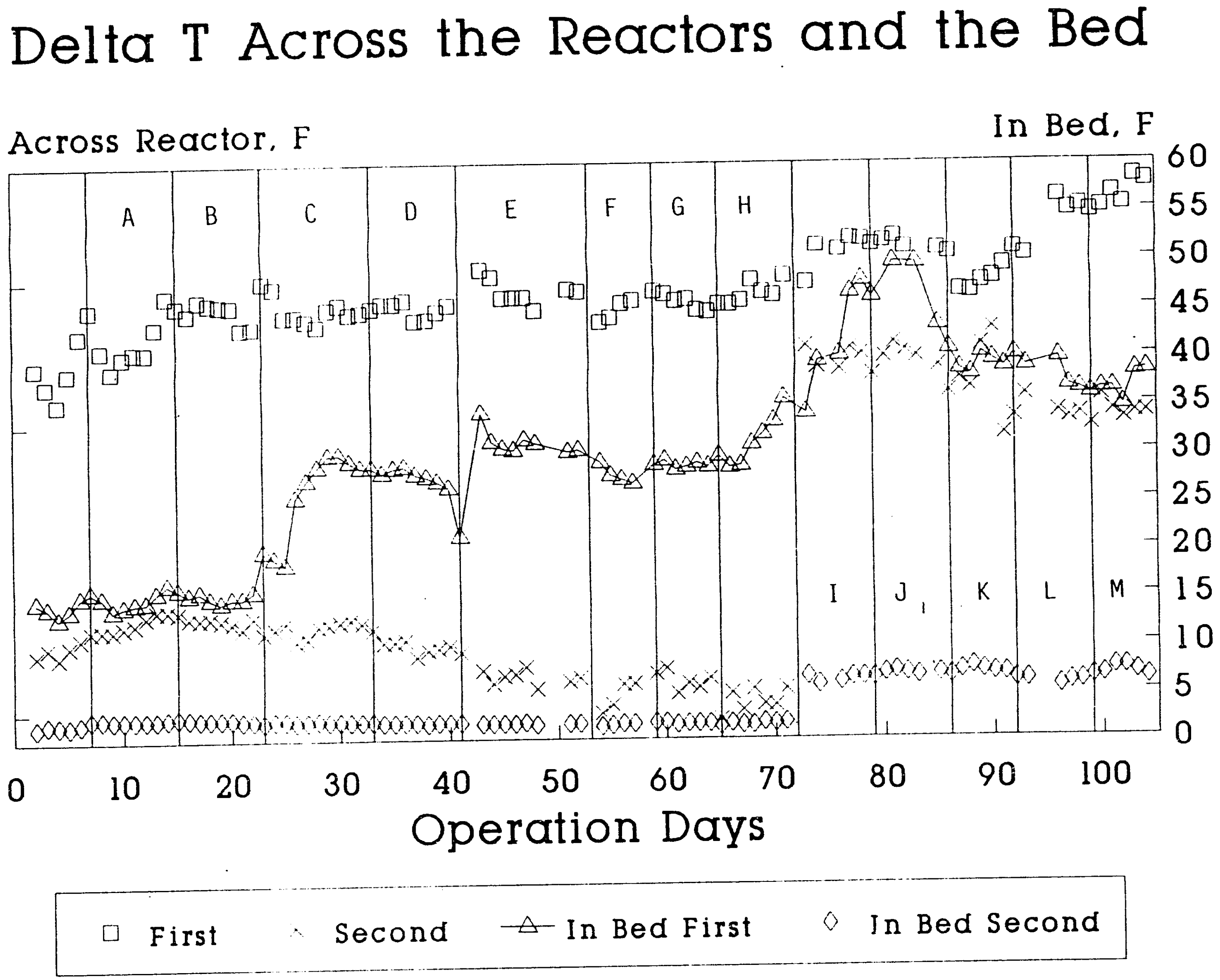
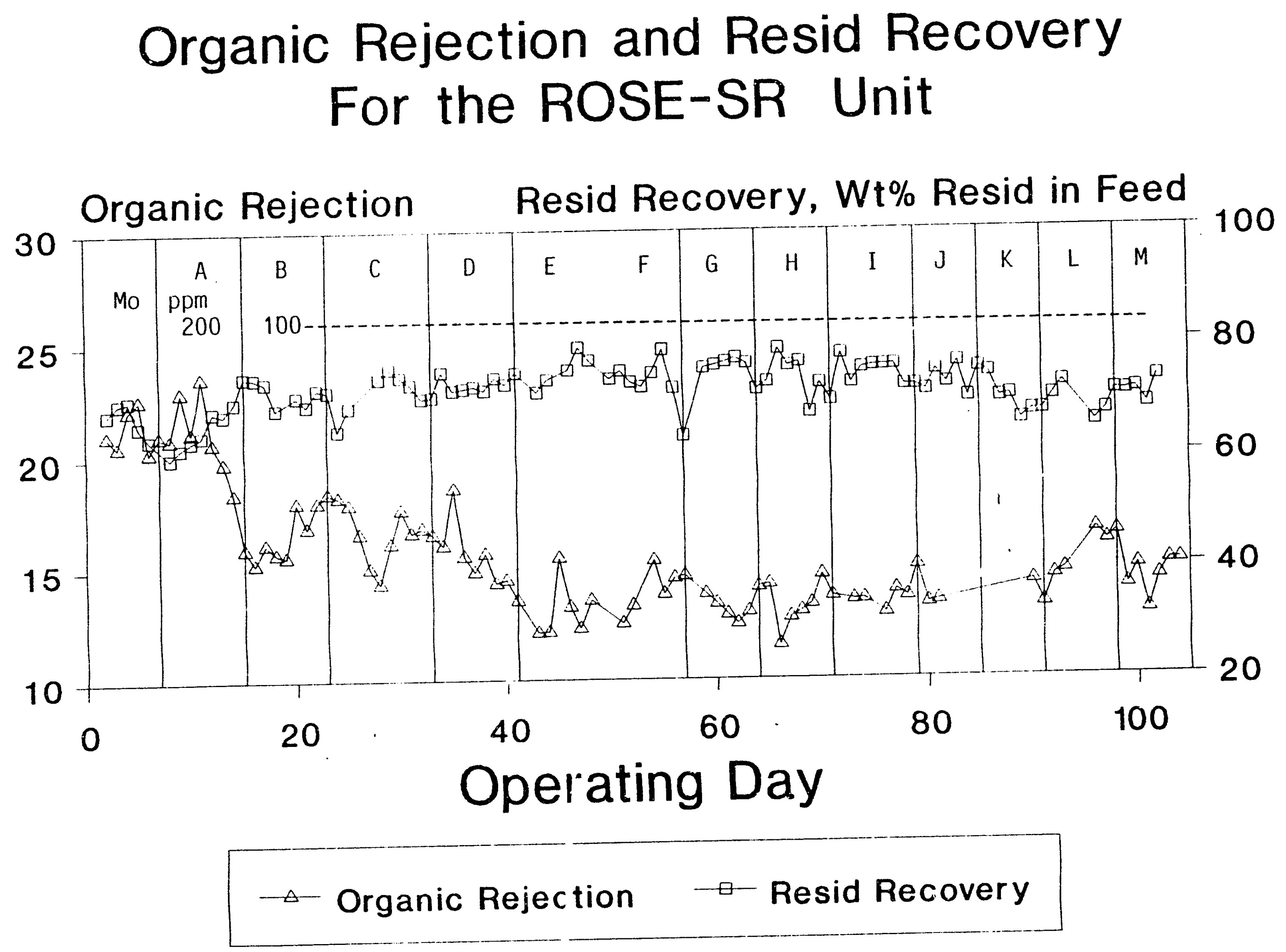


FIGURE 13. RESID + UC CONVERSION COMPARISON IN RUNS 263 and 262



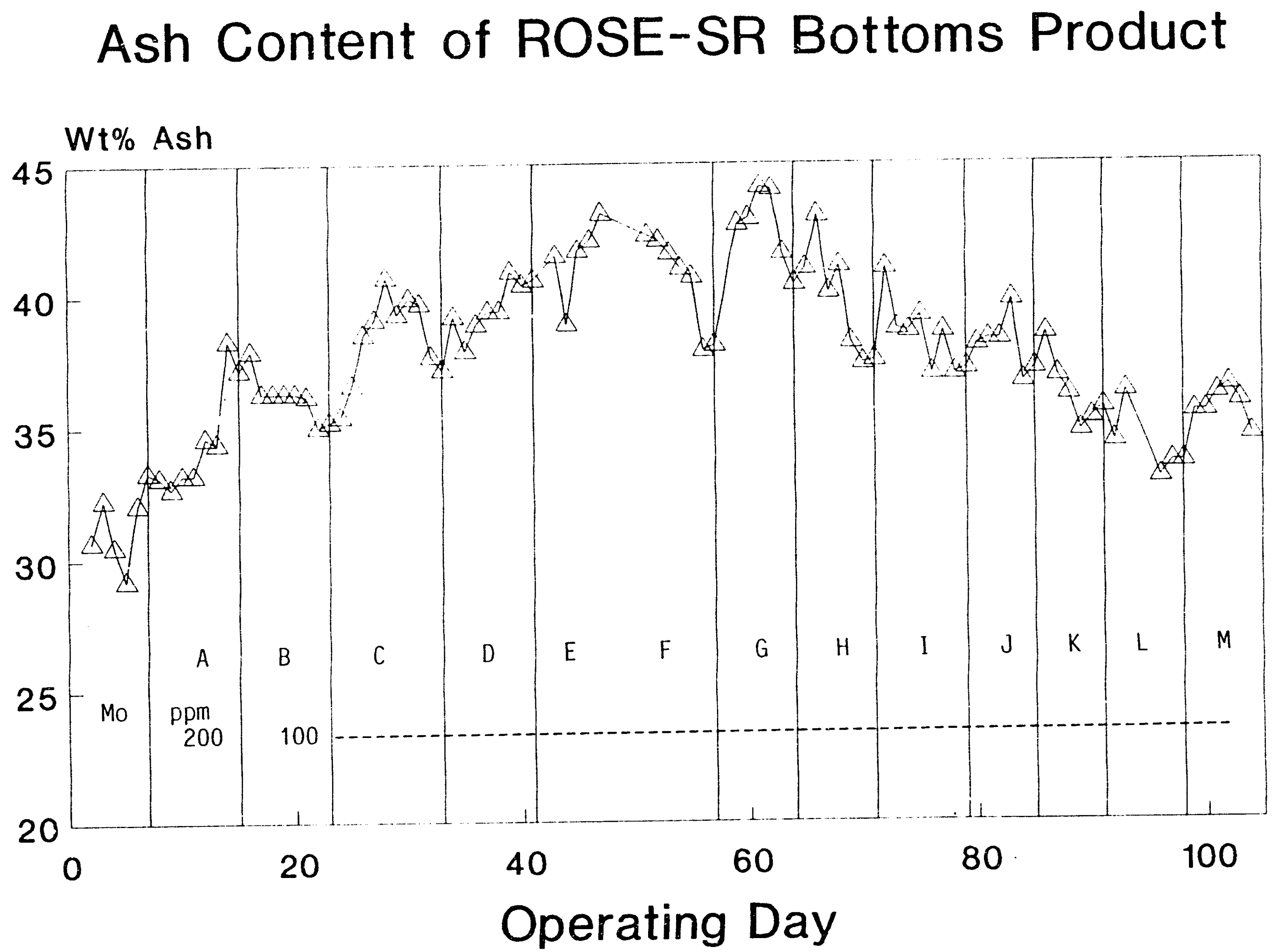
Day 1 - 11/01/91; Day 70 - 01/19/92
Day 30 - 12/07/91; Day 90 - 02/08/92
Day 50 - 12/30/91

FIGURE 14. FIRST AND SECOND STAGE DELTA T ACROSS THE REACTOR AND IN THE BED



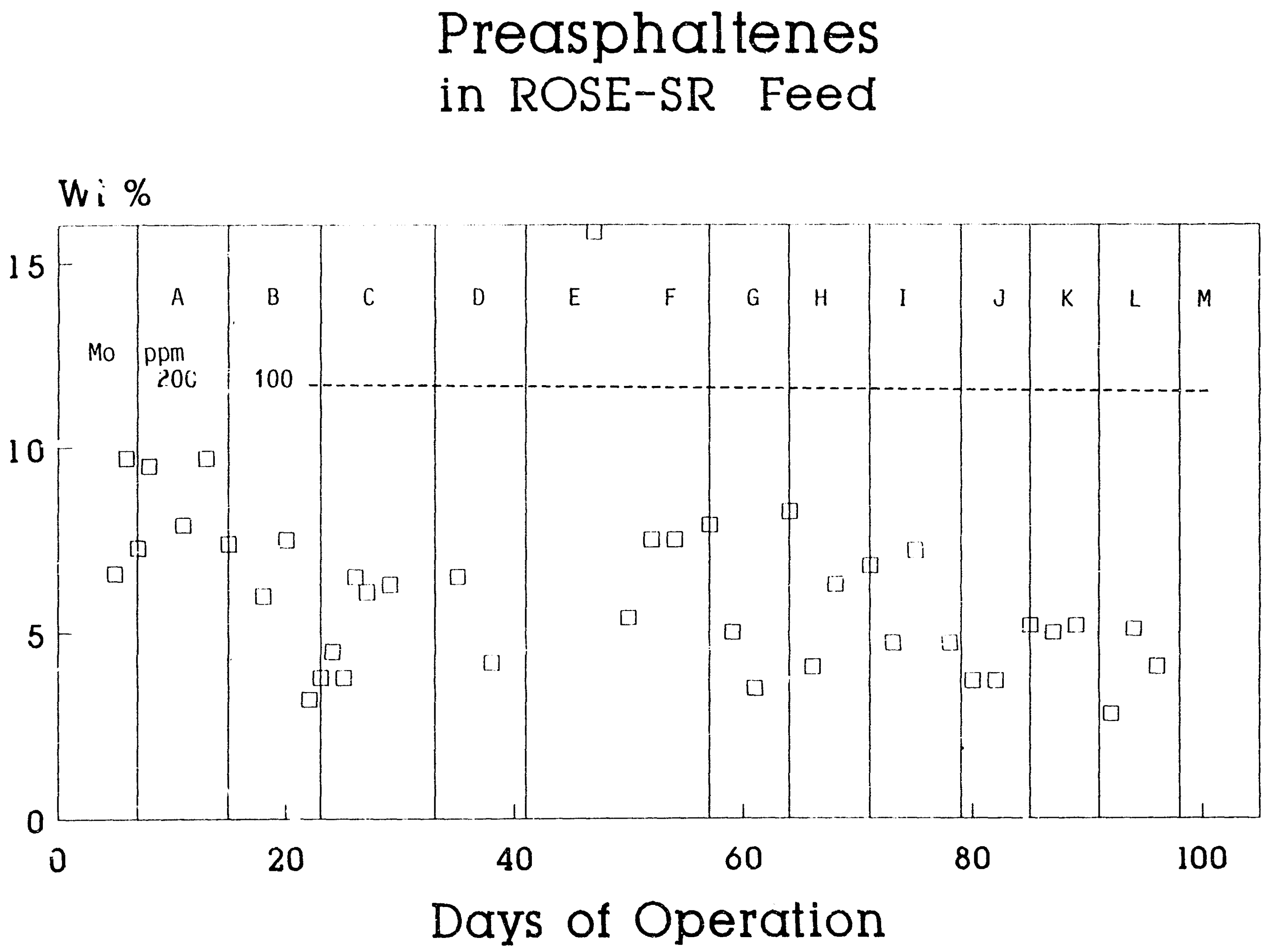
Day 1 = 11/01/91; Day 70 = 01/19/92
Day 30 = 12/07/91; Day 90 = 02/08/92
Day 50 = 12/30/91

FIGURE 15. ORGANIC REJECTION AND RESID RECOVERY FOR THE ROSE-SR^{sm} UNIT



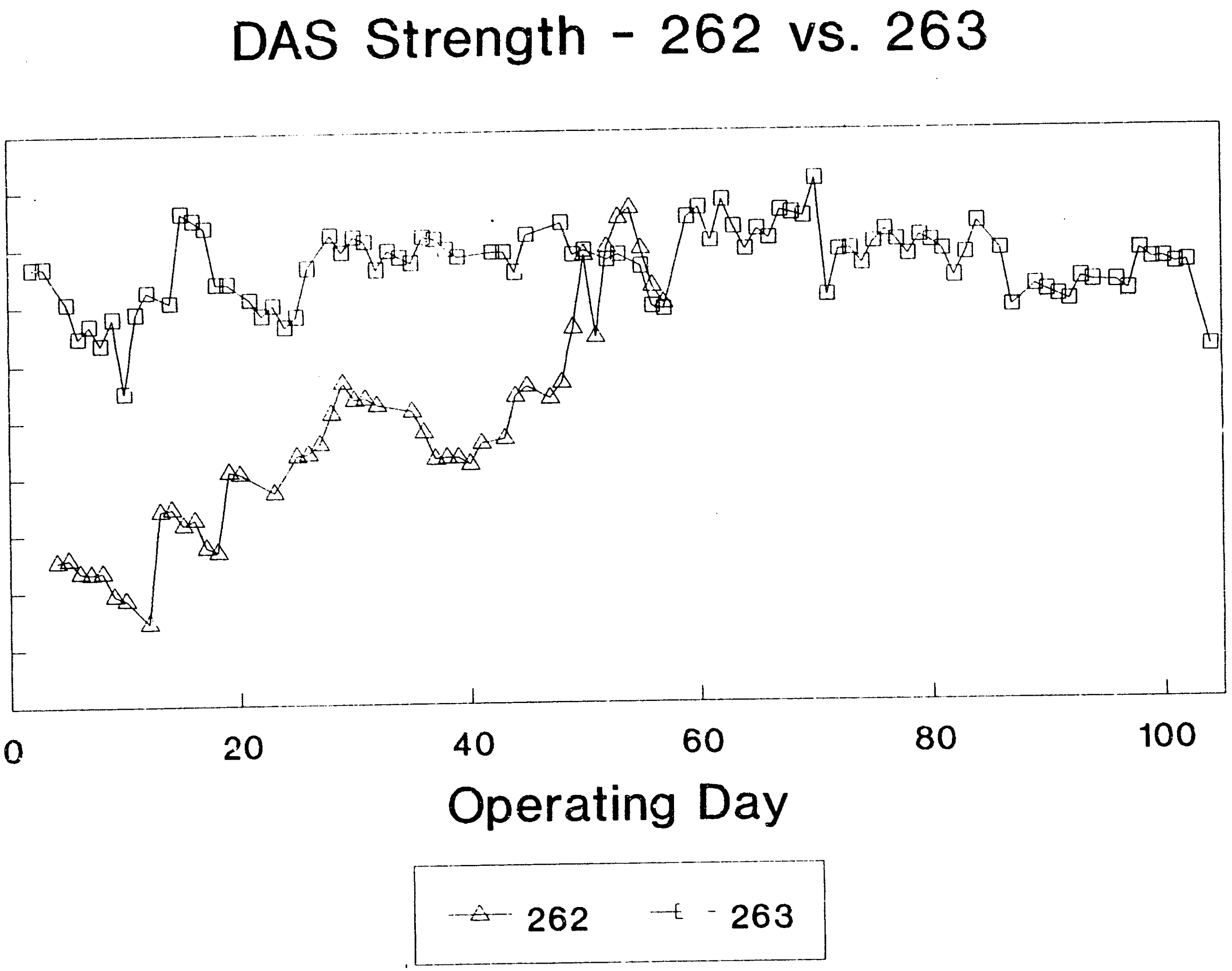
Day 1 = 11/01/91: Day 70 = 01/19/92
Day 30 = 12/07/91; Day 90 = 02/08/92
Day 50 = 12/30/91

FIGURE 16. ASH CONTENT IN THE ROSE-SR^{sm} BOTTOMS PRODUCT



Day 1 = 11/01/91; Day 70 = 01/19/92
Day 30 = 12/07/91; Day 90 = 02/08/92
Day 50 = 12/30/91

FIGURE 17. PREASPHALTENE CONTENT IN THE ROSE-SR^{sm} FEED



Day 1 = 11/01/91: Day 70 = 01/19/92
Day 30 = 12/07/91; Day 90 = 02/08/92
Day 50 = 12/30/91

FIGURE 18. COMPARISON OF DEASHING SOLVENT (DAS) STRENGTH FOR RUNS 263 AND 262

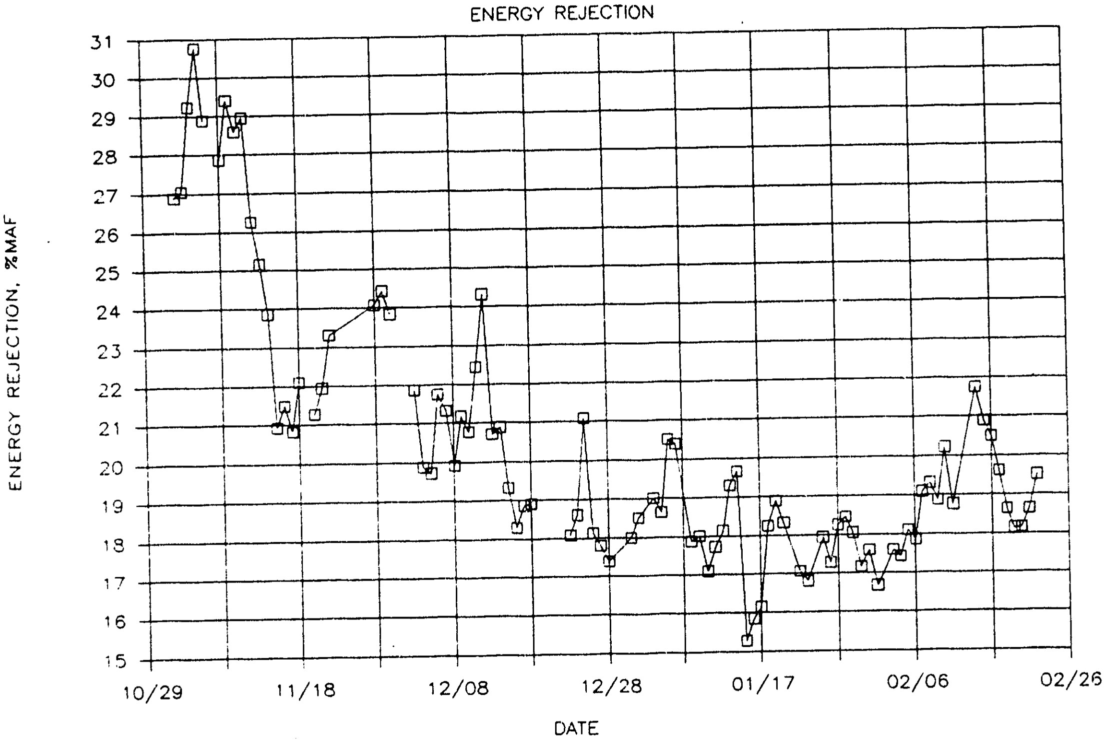


FIGURE 19. ENERGY REJECTION FOR THE ROSE-SR^{sm} UNIT

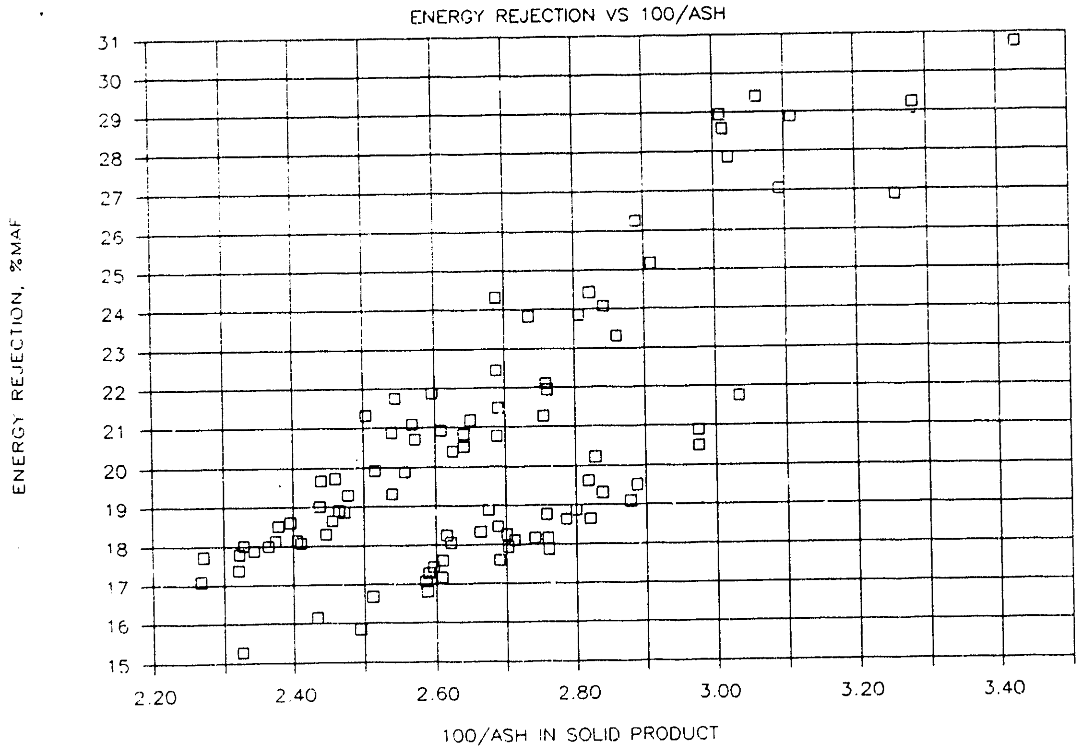


FIGURE 20. ENERGY REJECTION VS. ASH CONTENT IN THE ROSE-SR^{sm} BOTTOMS PRODUCT

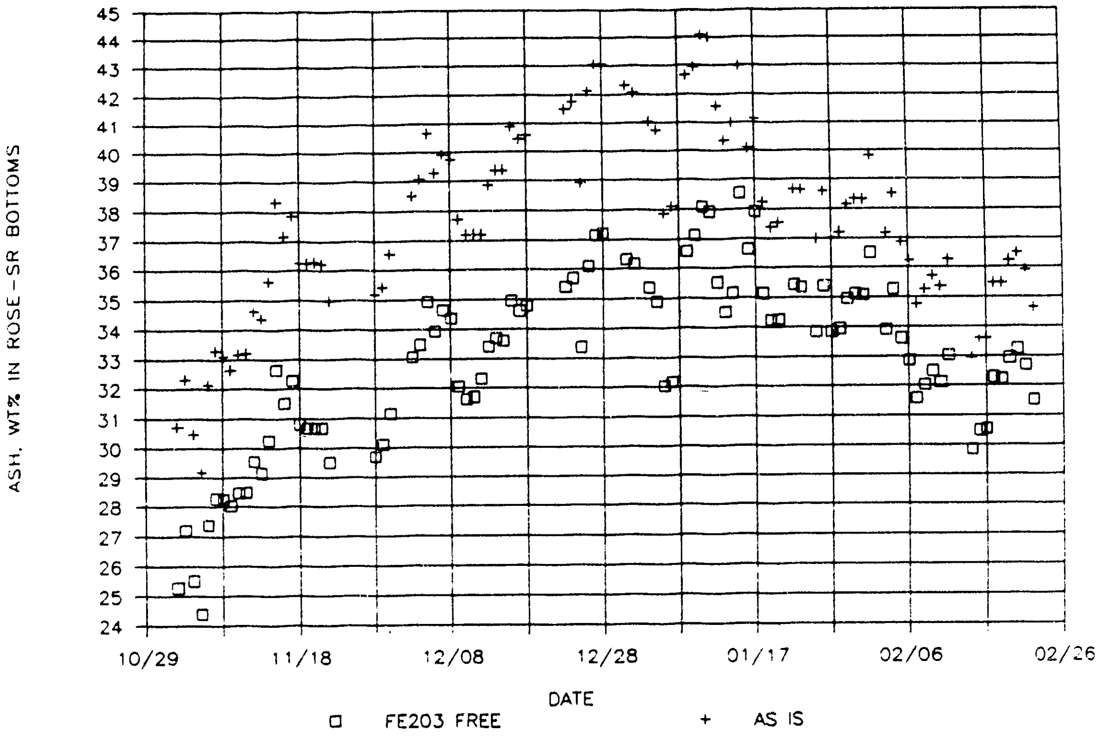


FIGURE 21. AS-IS AND FE_2O_3-FREE ASH CONTENTS IN THE ROSE-SR^{sm} BOTTOMS PRODUCT

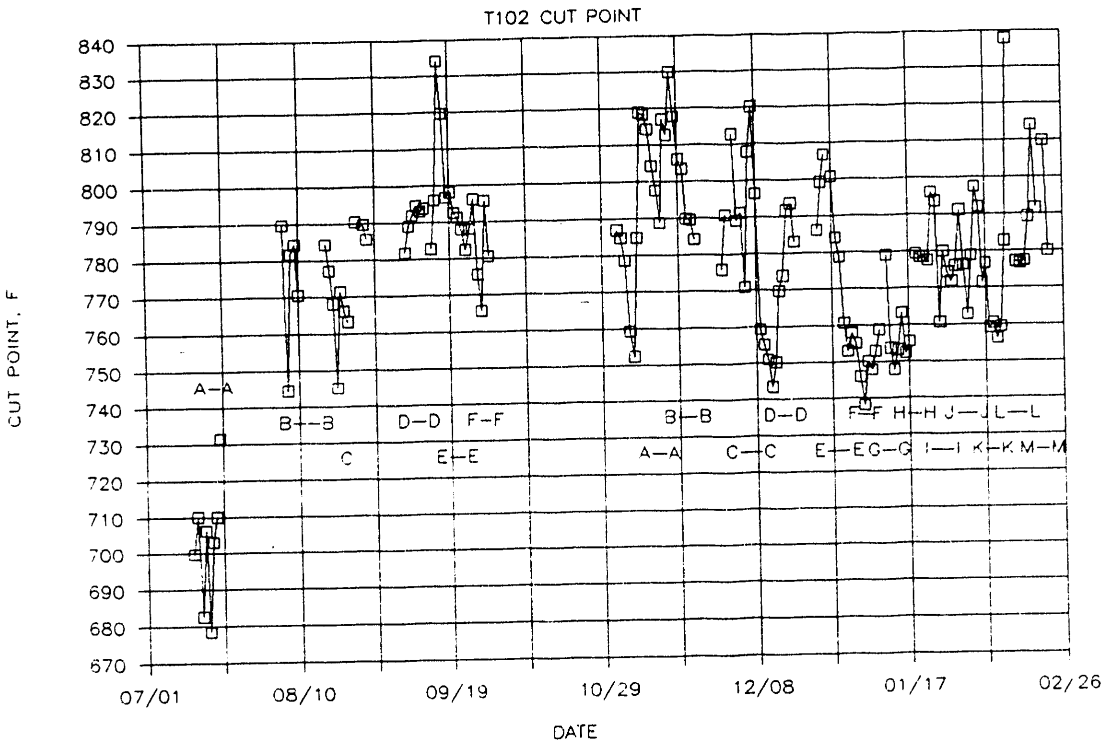


FIGURE 22. T102 VACUUM COLUMN CUT POINT BY GC

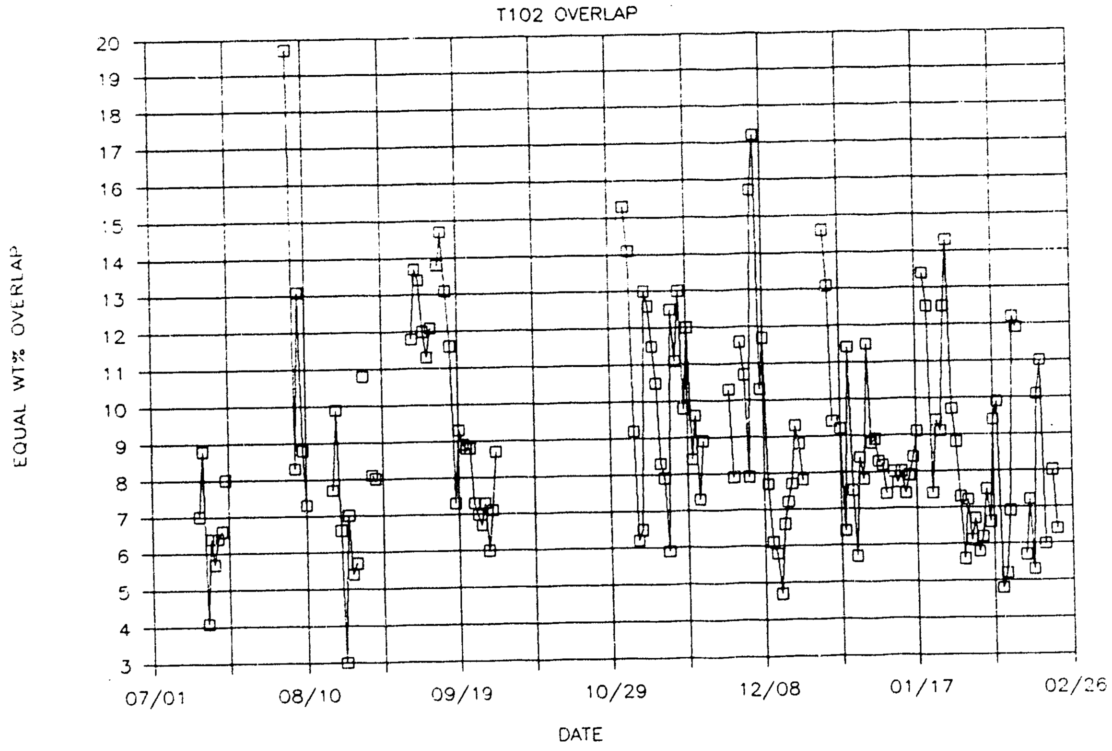


FIGURE 23. T102 VACUUM COLUMN OVERLAP BETWEEN OVERHEADS AND BOTTOMS

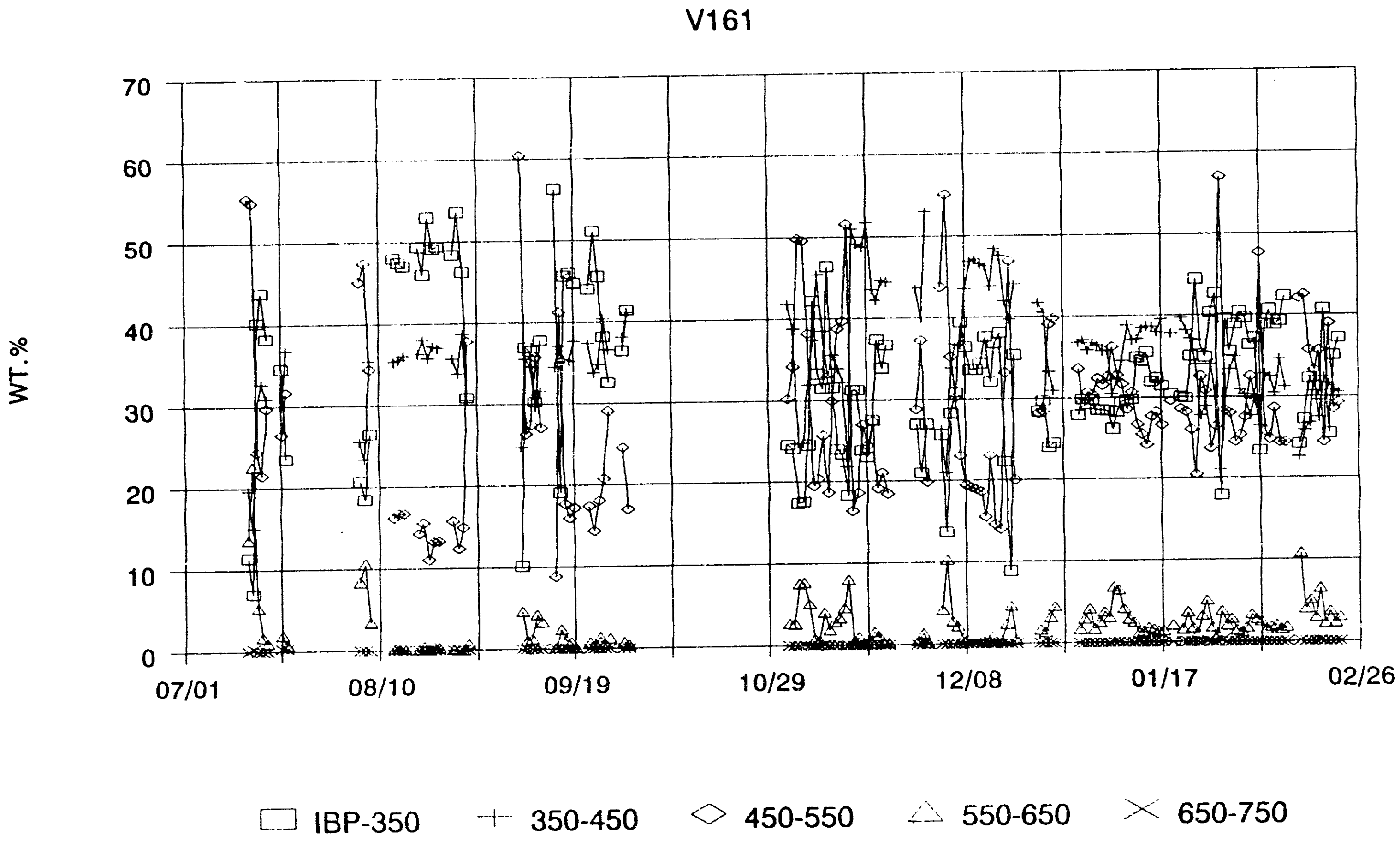


Figure 6-3

FIGURE 24. T105 OVERHEADS DISTRIBUTION

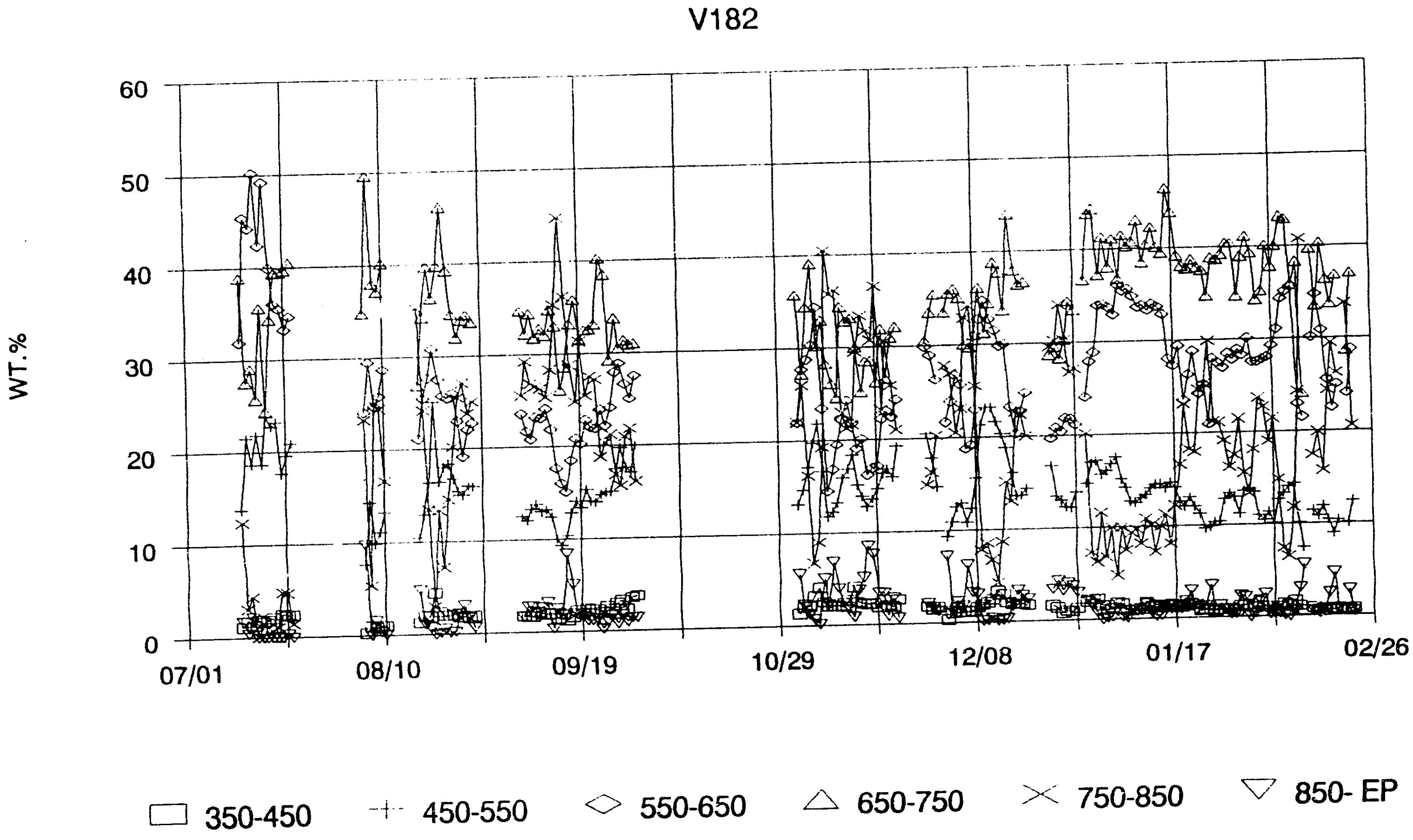


Figure 6-4

FIGURE 25. T102 OVERHEADS DISTRIBUTION

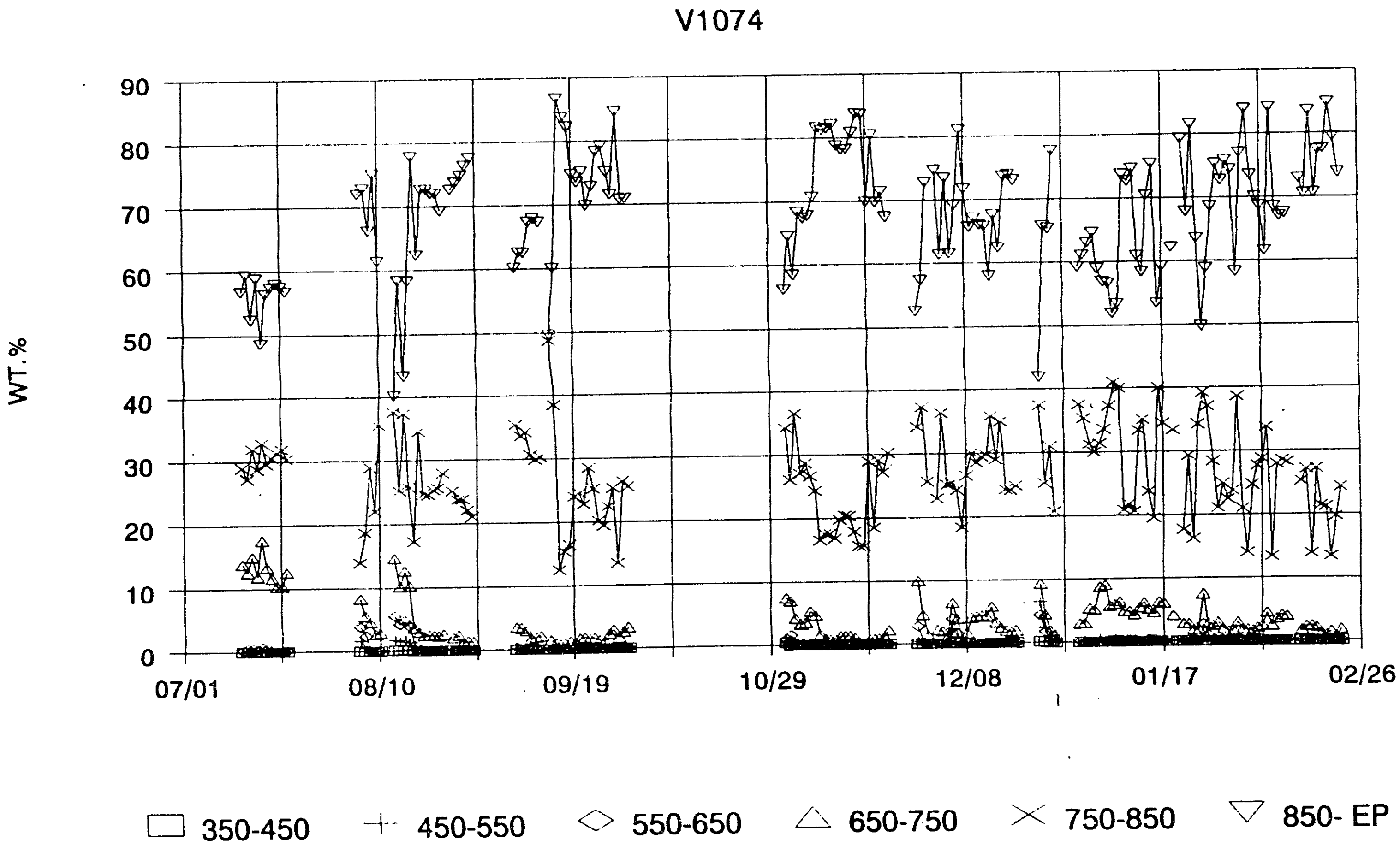
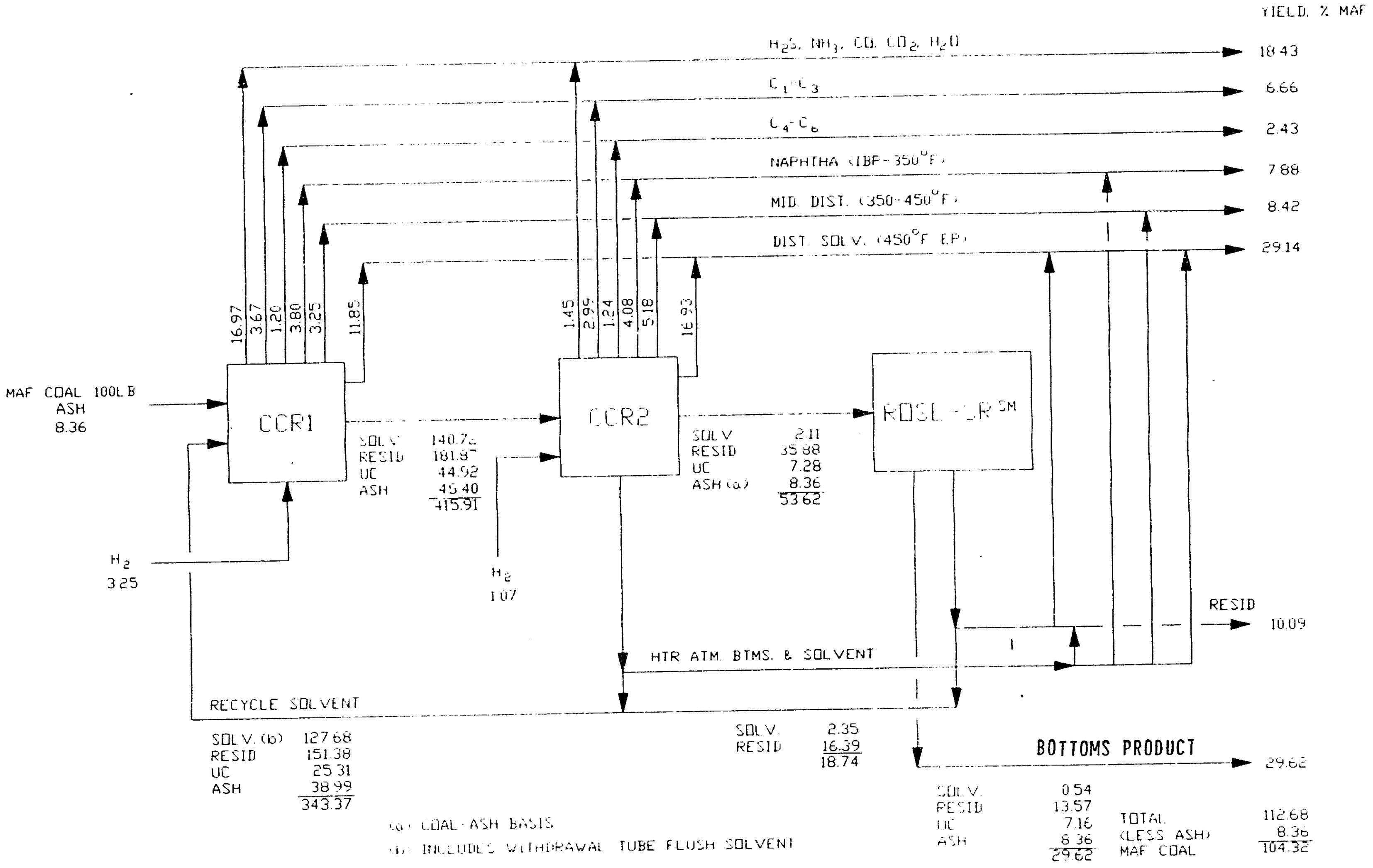


Figure 6-5

FIGURE 26. T102 BOTTOMS DISTRIBUTION



(a) COAL-ASH BASIS

(b) INCLUDES WITHDRAWAL TUBE FLUSH SOLVENT

FIGURE 27. MATERIAL BALANCE FLOW DIAGRAM FOR PERIOD 263A

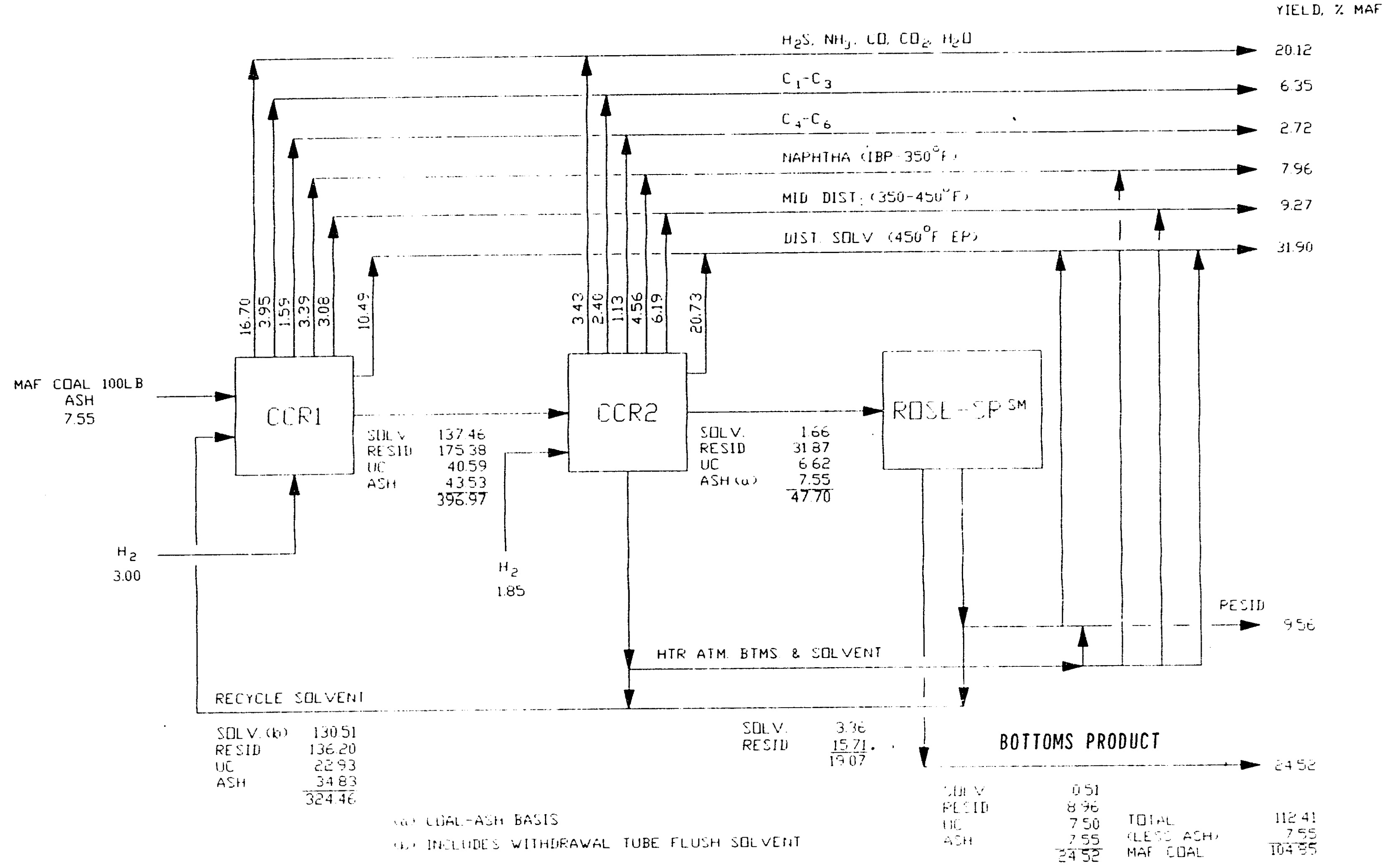


FIGURE 28. MATERIAL BALANCE FLOW DIAGRAM FOR PERIOD 263B

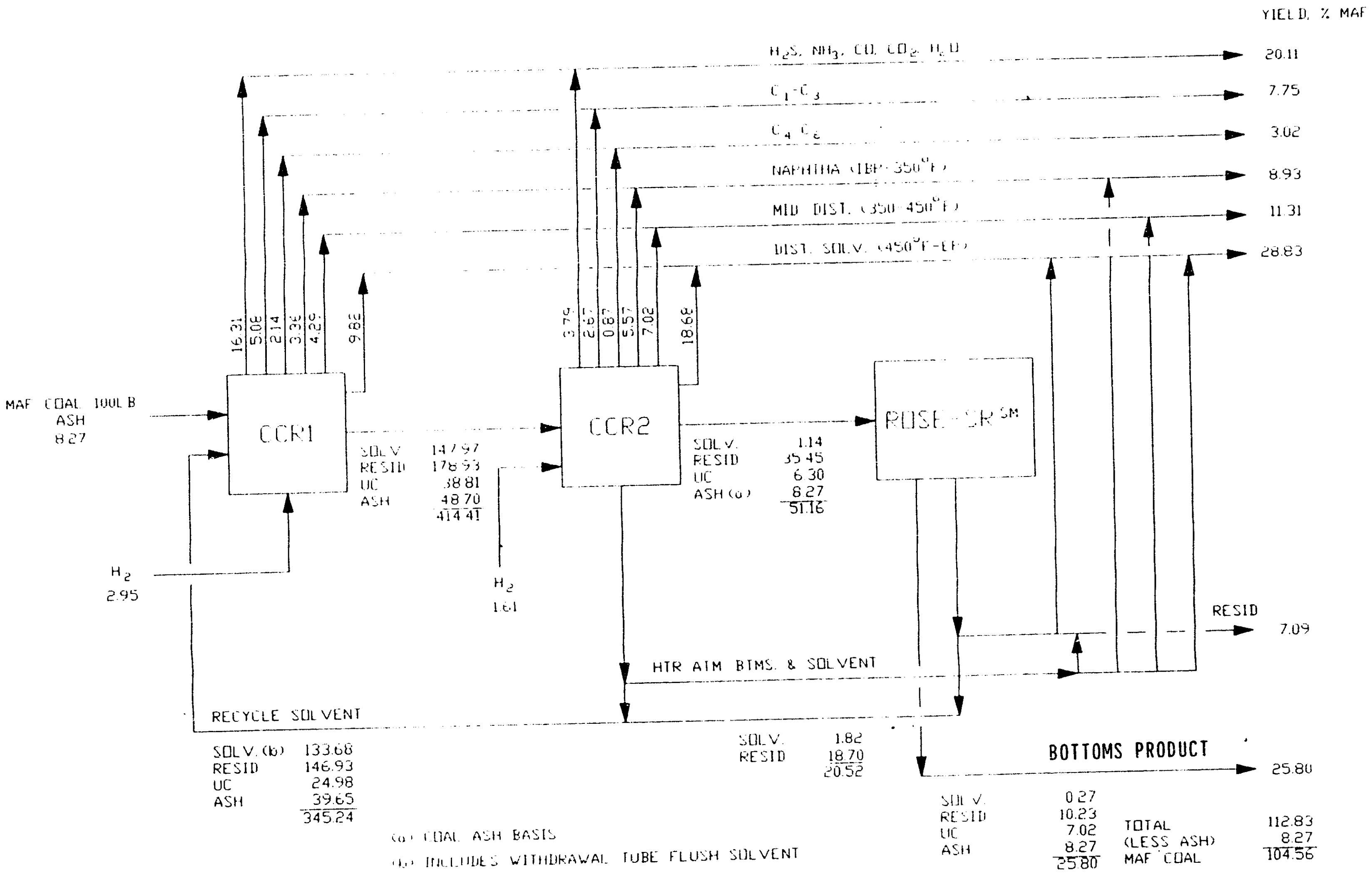


FIGURE 29. MATERIAL BALANCE FLOW DIAGRAM FOR PERIOD 263C

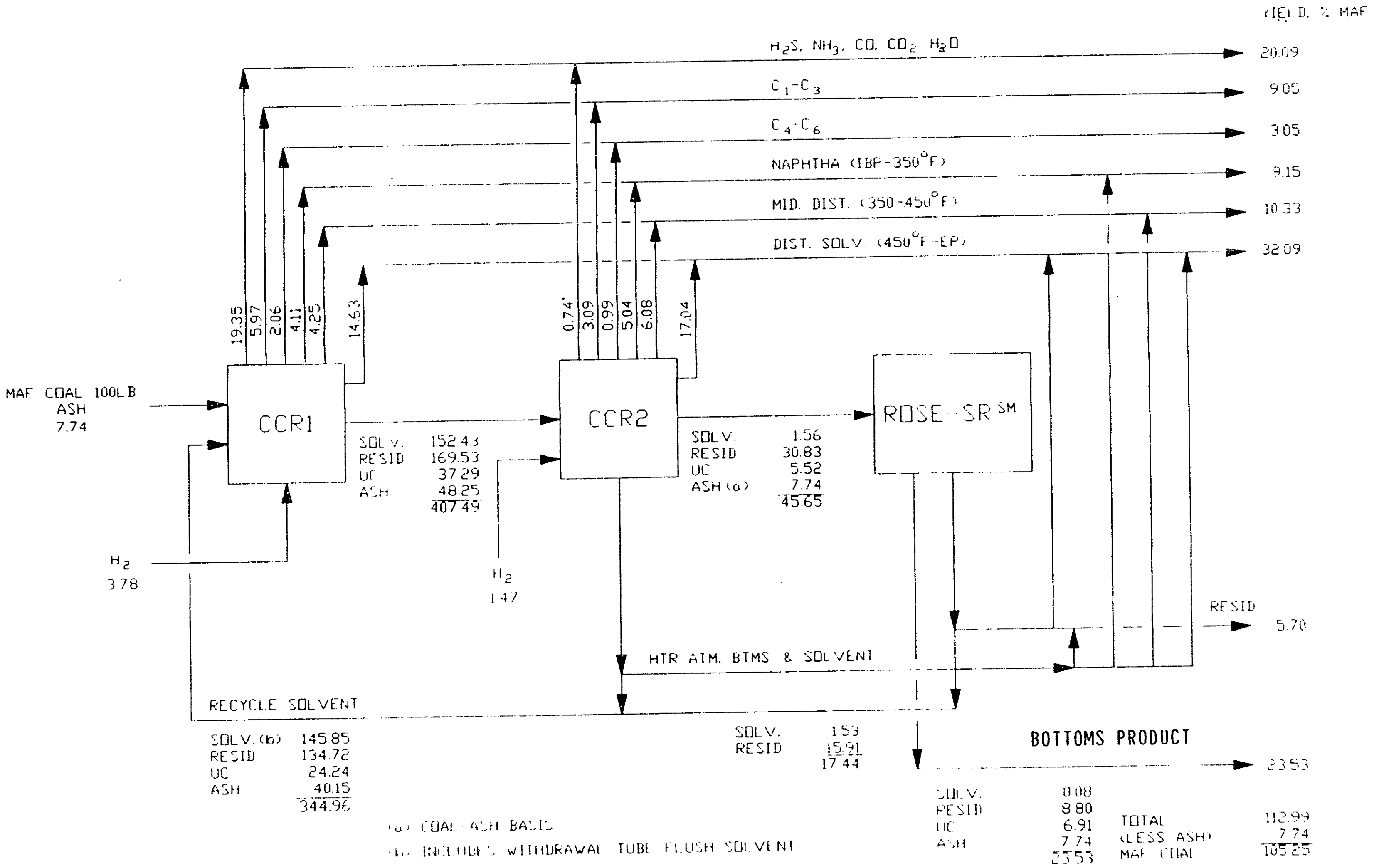


FIGURE 30. MATERIAL BALANCE FLOW DIAGRAM FOR PERIOD 263D

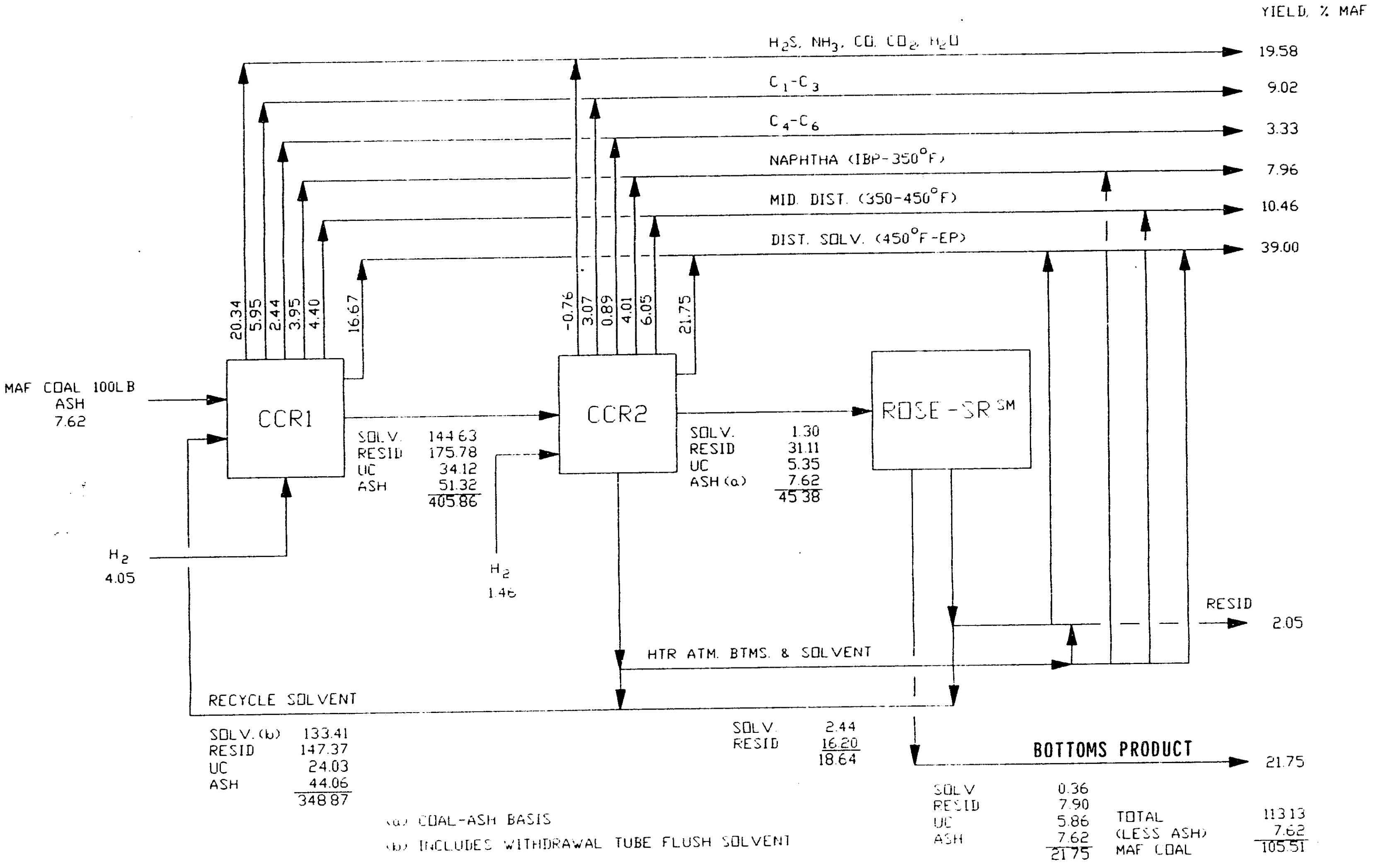
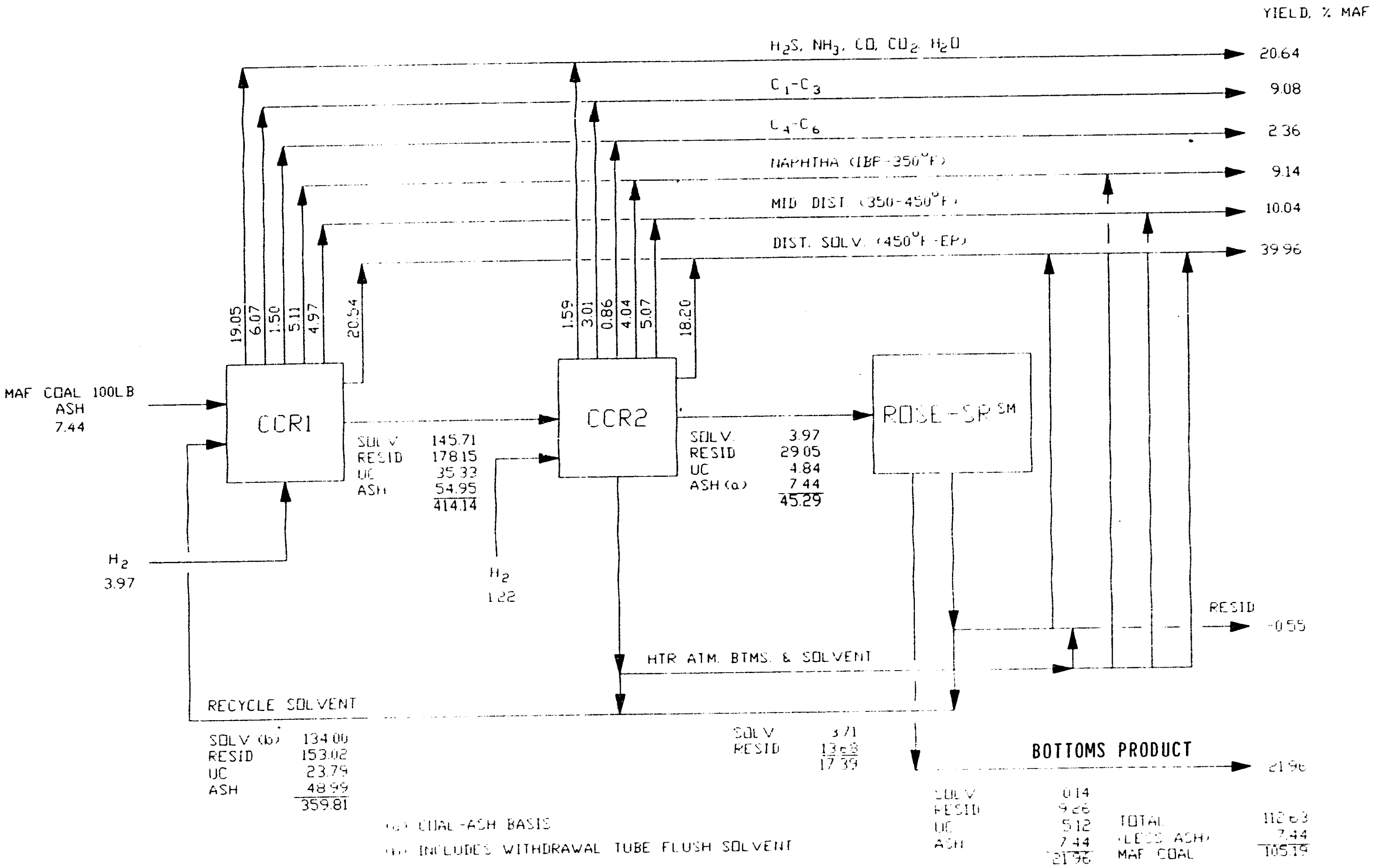


FIGURE 31. MATERIAL BALANCE FLOW DIAGRAM FOR PERIOD 263E



(a) COAL-ASH BASIS

(b) INCLUDES WITHDRAWAL TUBE FLUSH SOLVENT

FIGURE 32. MATERIAL BALANCE FLOW DIAGRAM FOR PERIOD 263F

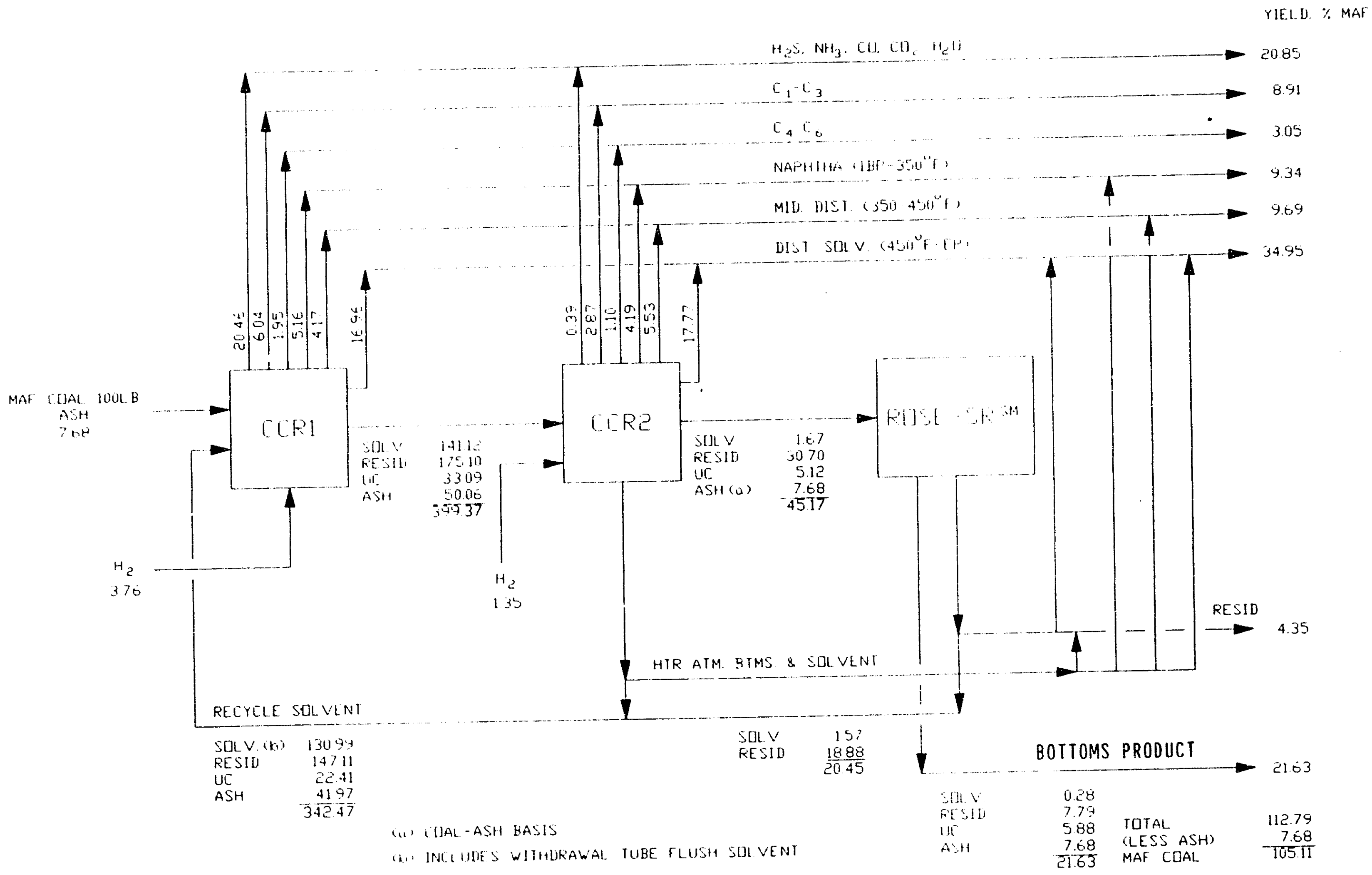


FIGURE 33. MATERIAL BALANCE FLOW DIAGRAM FOR PERIOD 263G

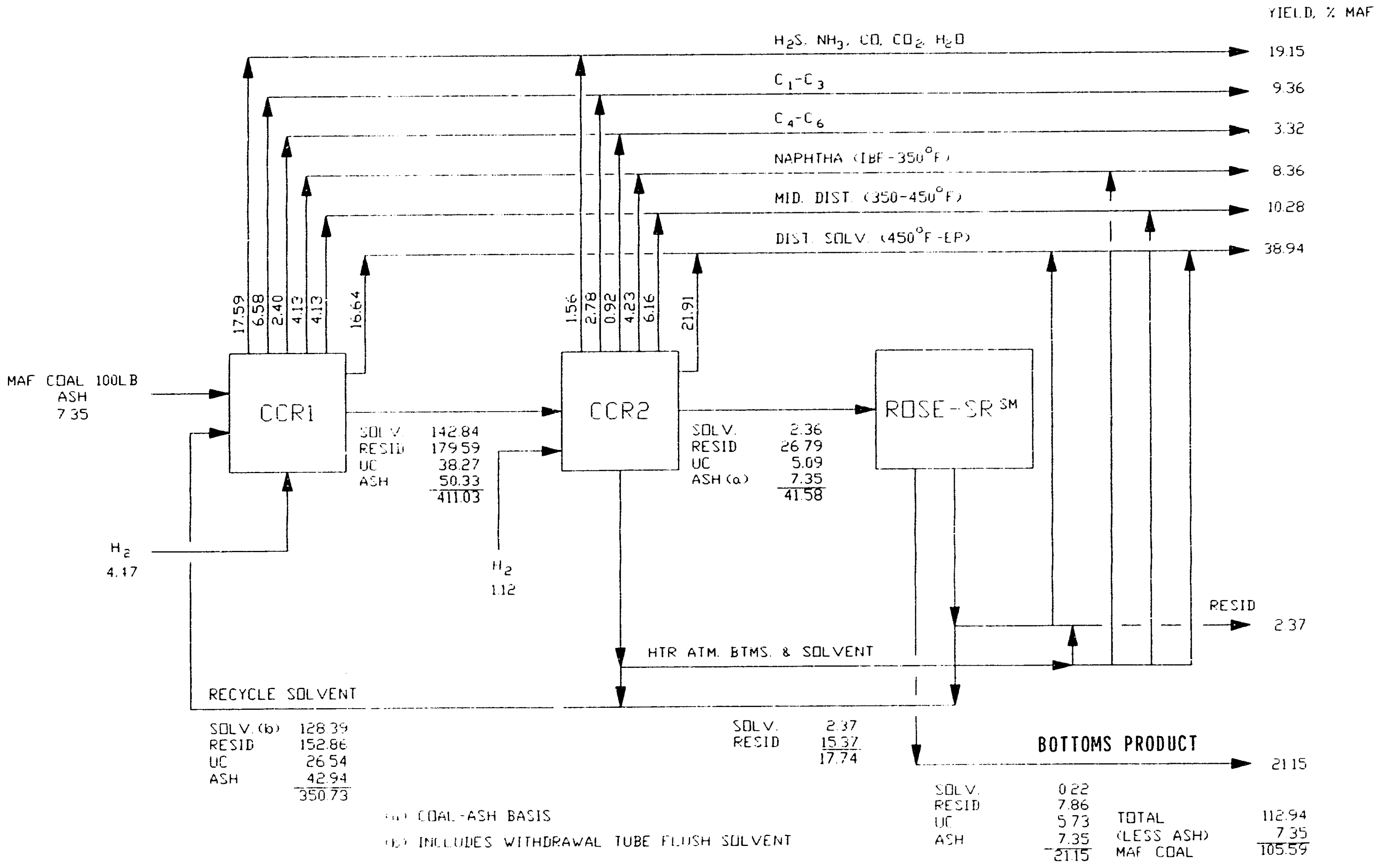


FIGURE 34. MATERIAL BALANCE FLOW DIAGRAM FOR PERIOD 263H

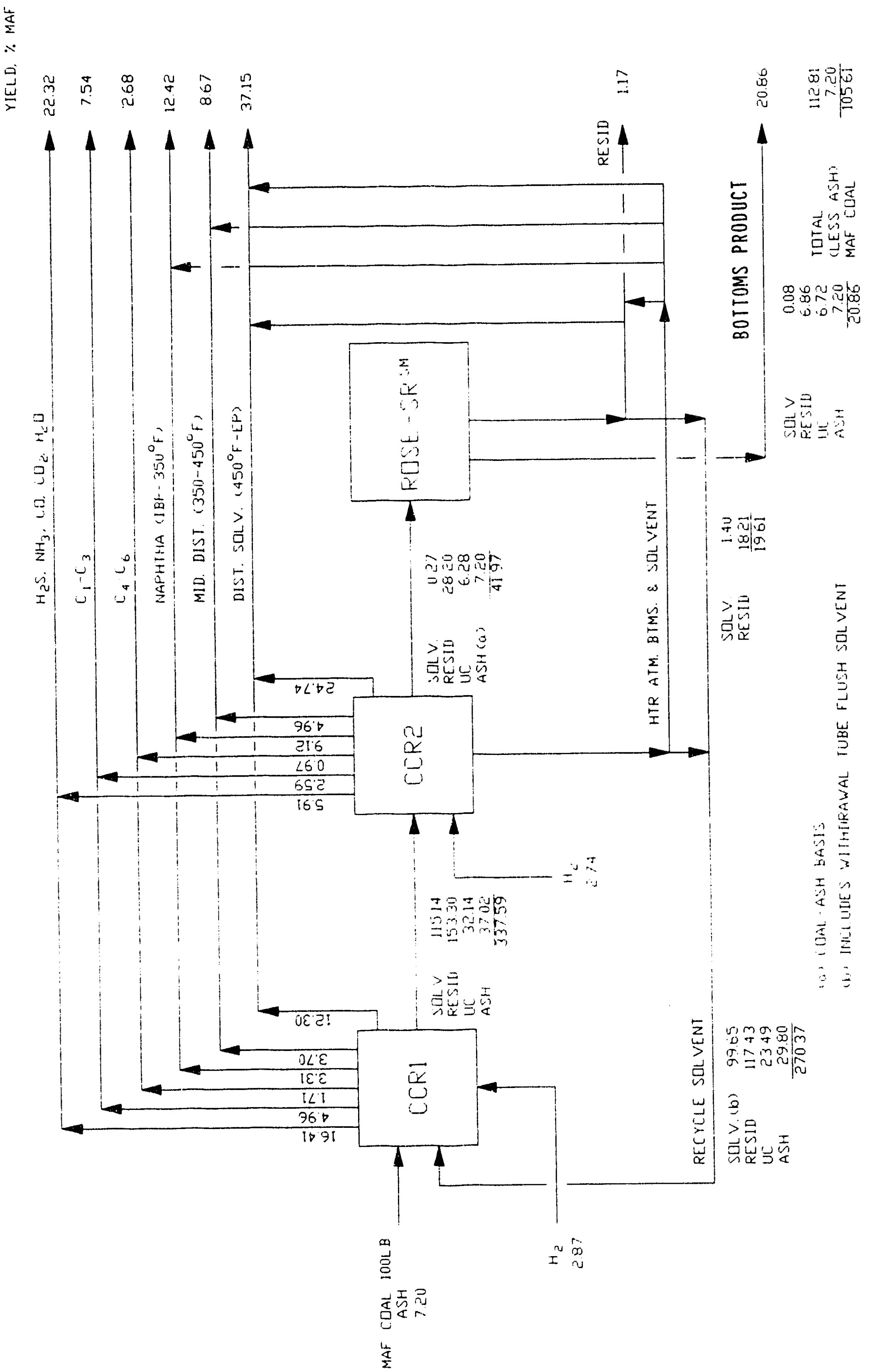


FIGURE 35. MATERIAL BALANCE FLOW DIAGRAM FOR PERIOD 263I

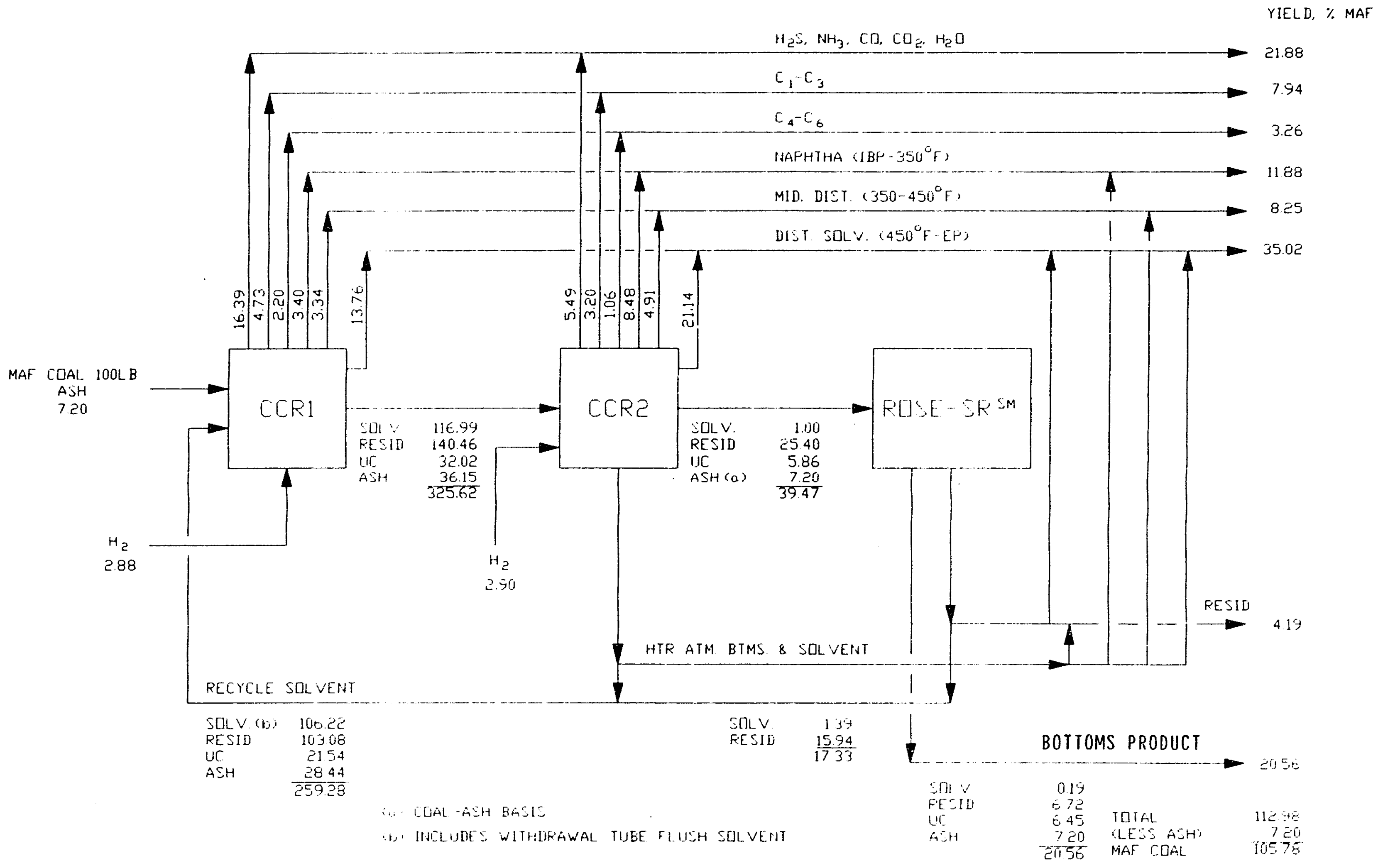


FIGURE 36. MATERIAL BALANCE FLOW DIAGRAM FOR PERIOD 263J

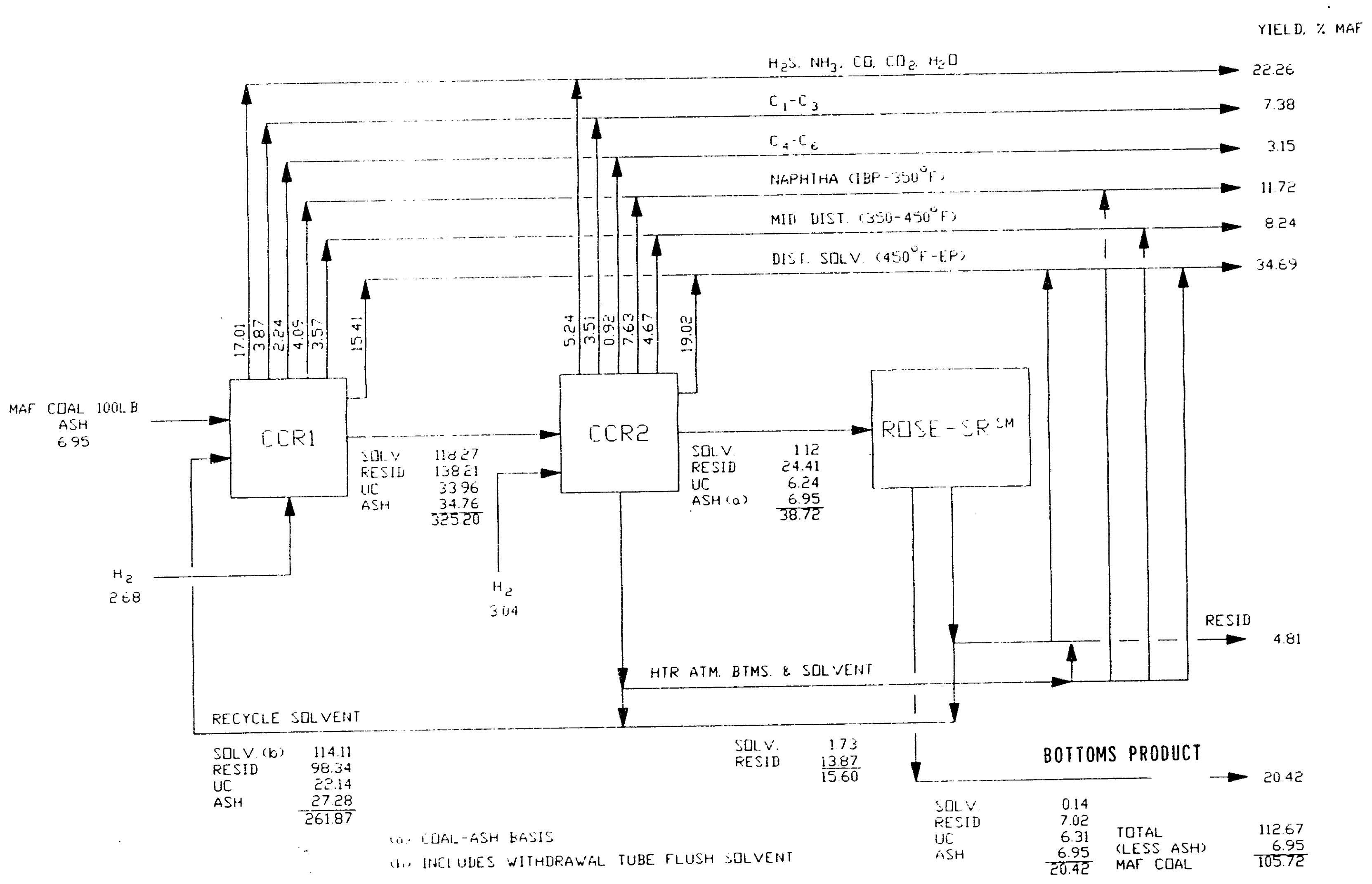


FIGURE 37. MATERIAL BALANCE FLOW DIAGRAM FOR PERIOD 263K

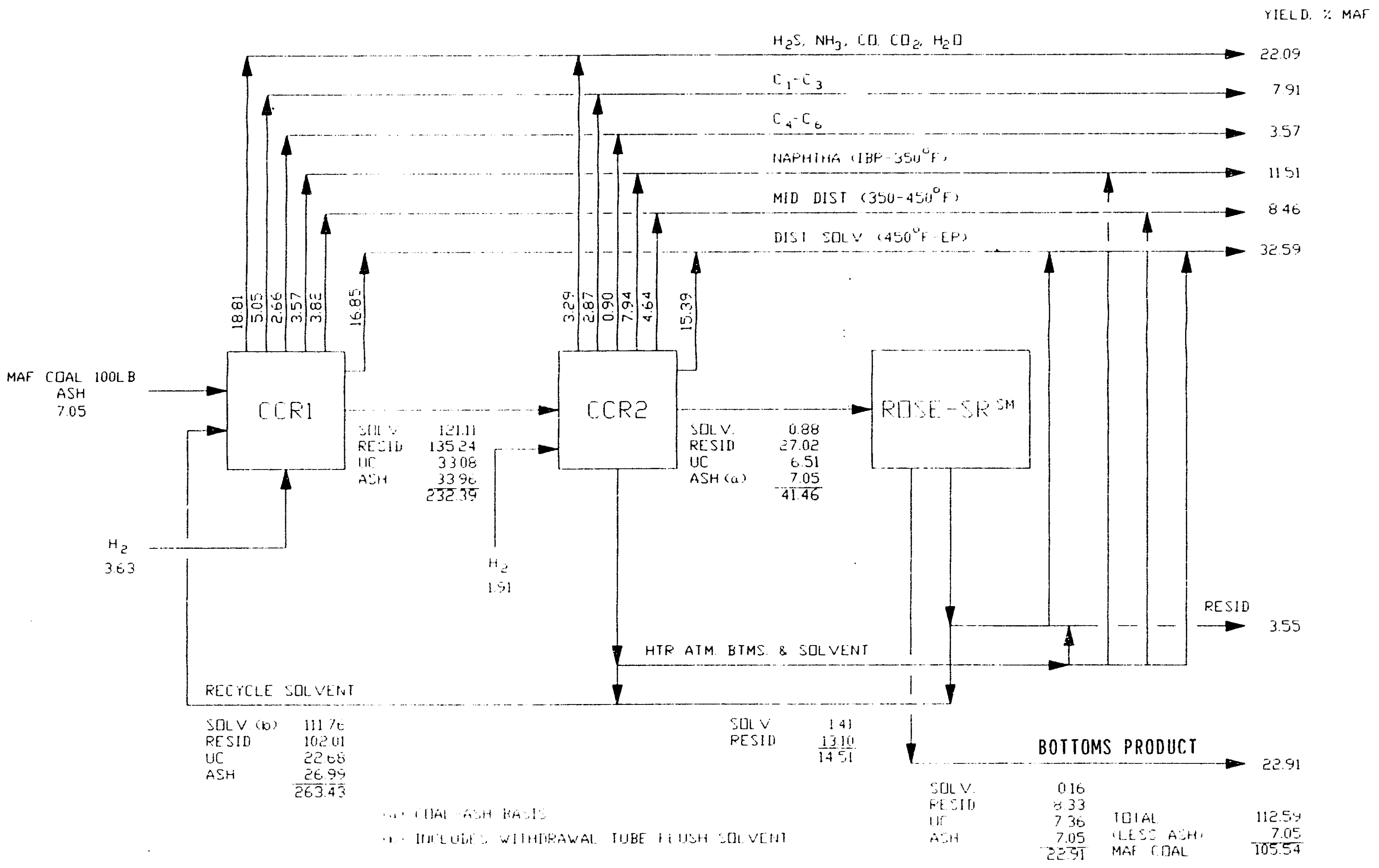
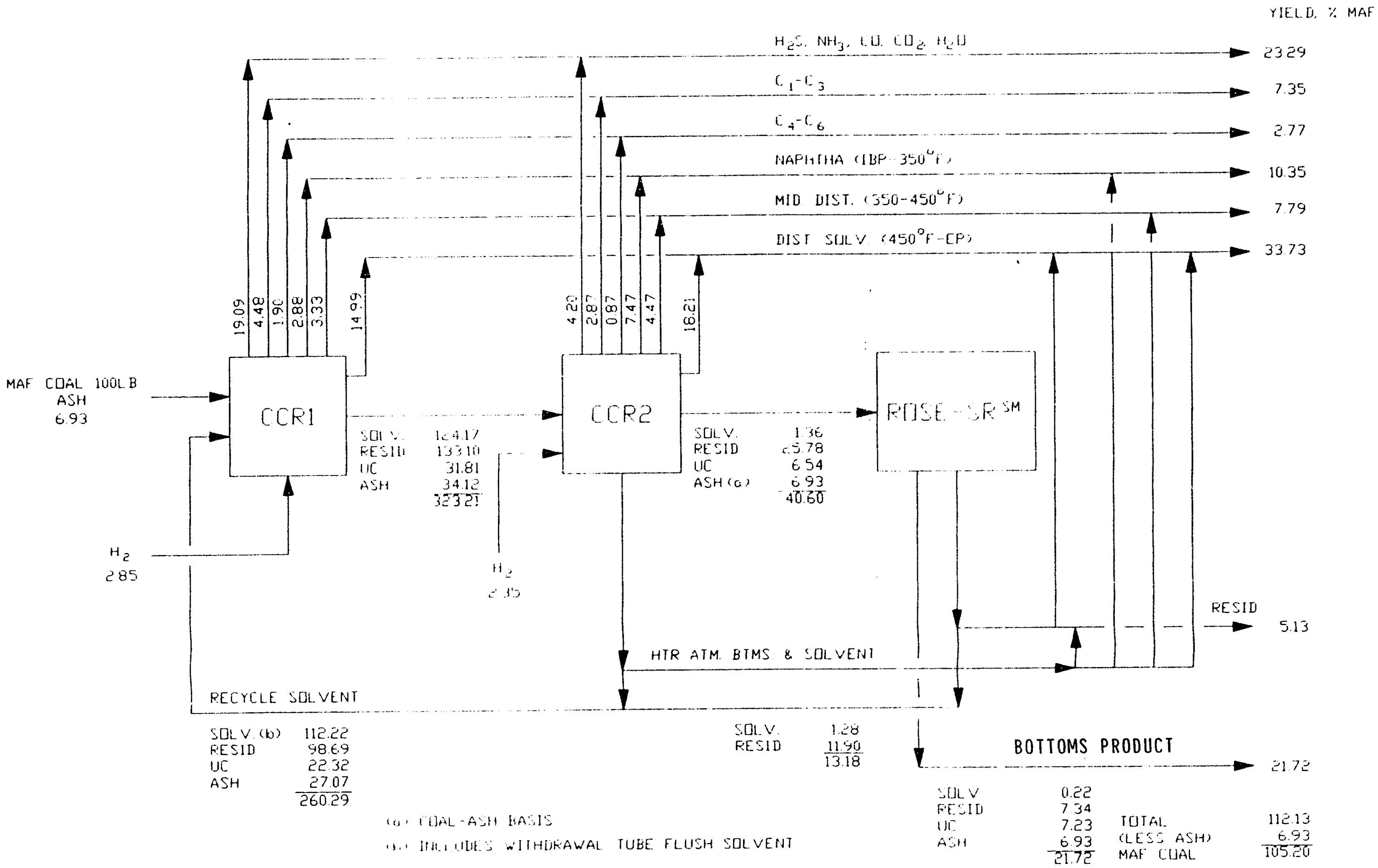


FIGURE 38. MATERIAL BALANCE FLOW DIAGRAM FOR PERIOD 263L

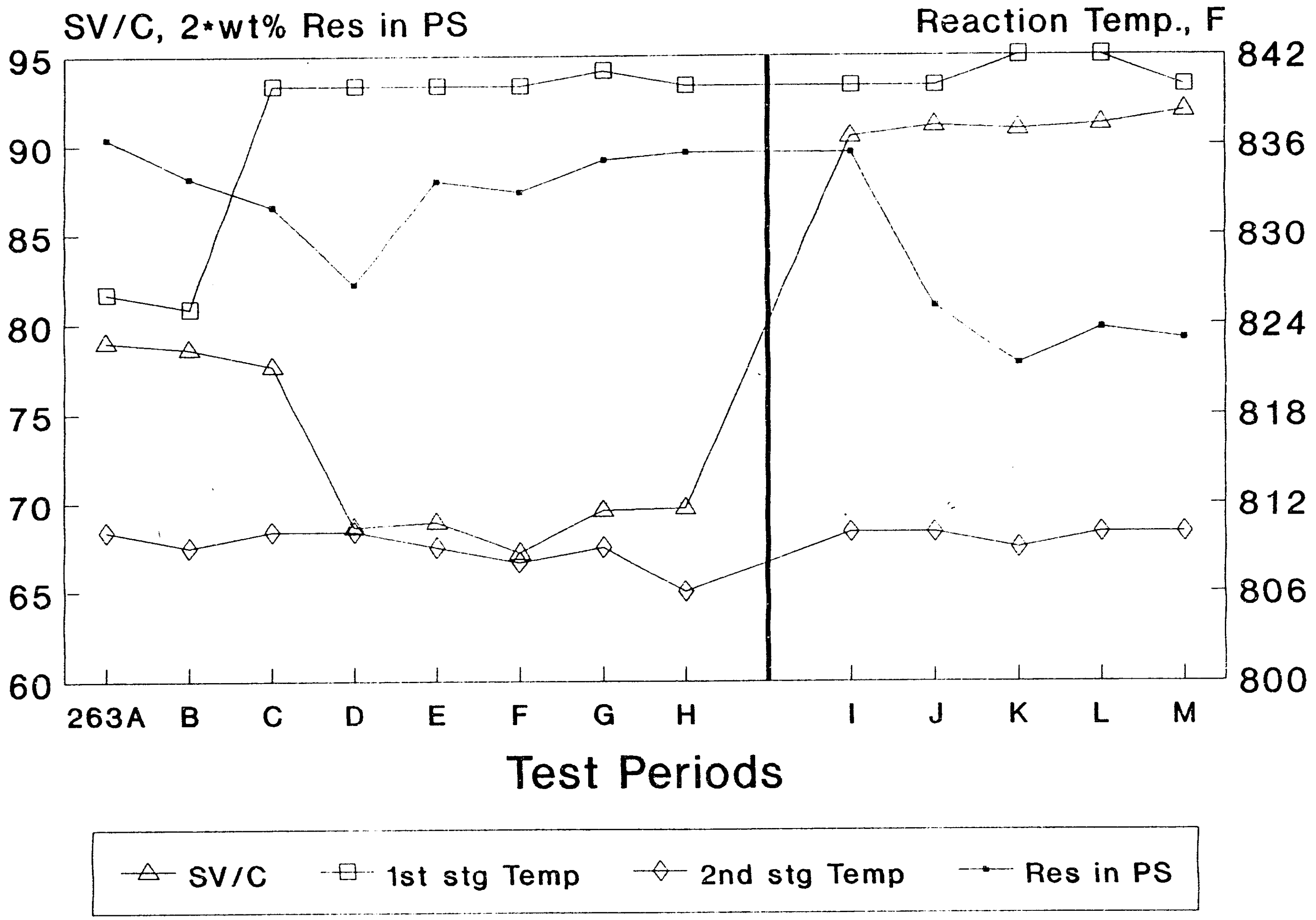


(a) COAL-ASH BASIS

(b) INCLUDES WITHDRAWAL TUBE FLUSH SOLVENT

FIGURE 39. MATERIAL BALANCE FLOW DIAGRAM FOR PERIOD 263M

Run 263: Test Conditions



SV/C - lb MF coal/hr/cu ft reactor

FIGURE 40. TEST CONDITIONS FOR PERIODS 263A-M

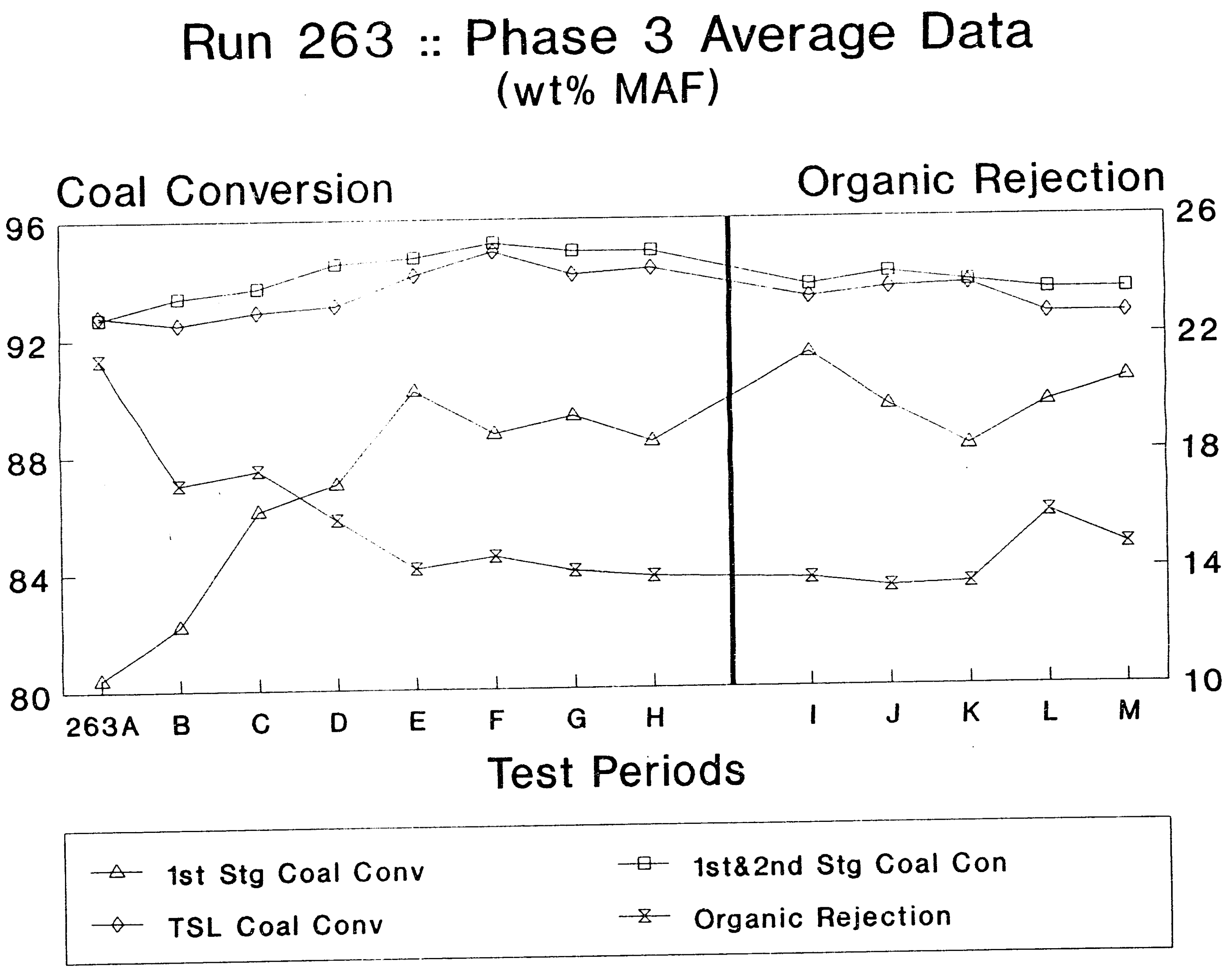


FIGURE 41. COAL CONVERSION AND ORGANIC REJECTION (PERIODS 263A-M)

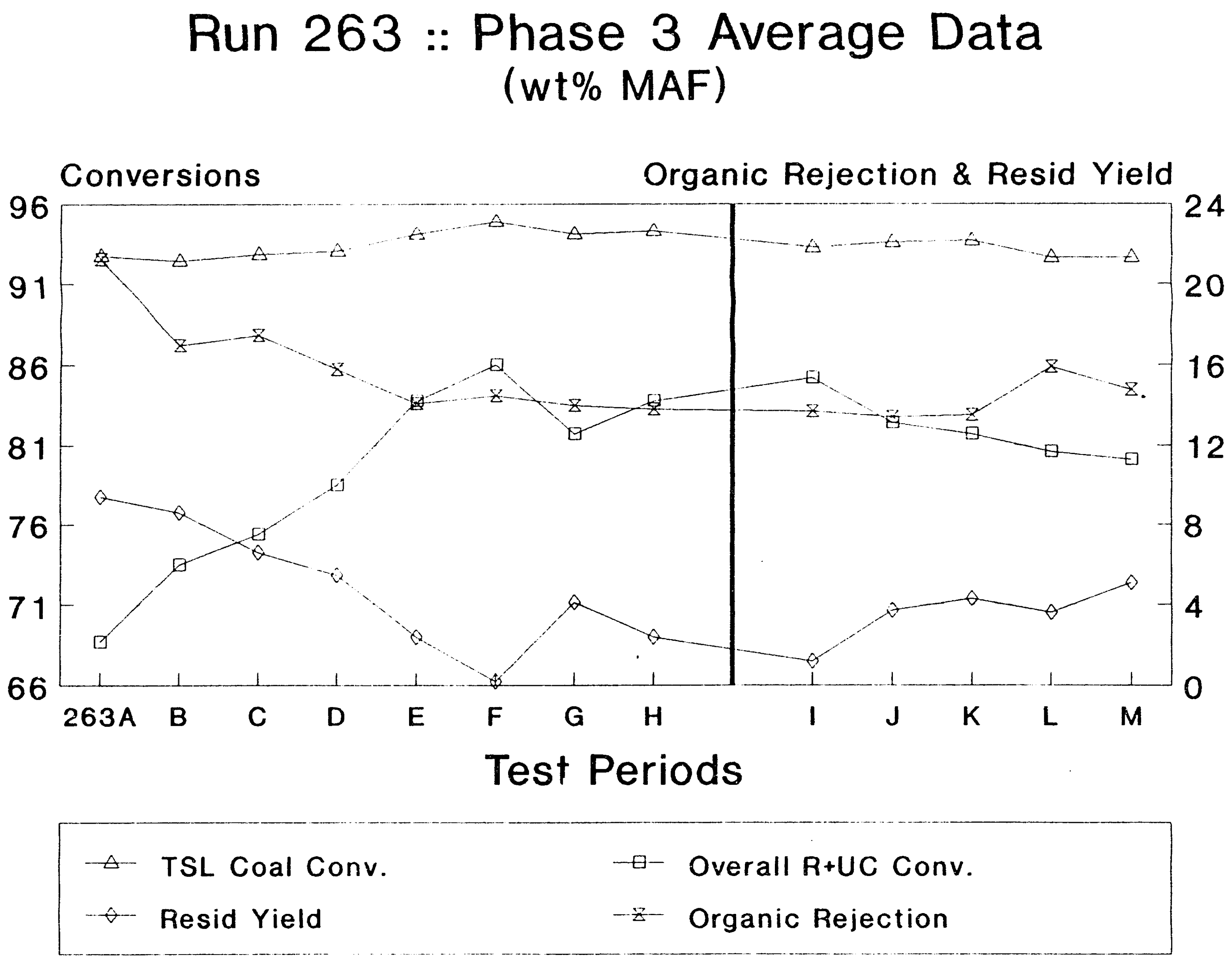


FIGURE 42. RESID YIELD AND OVERALL RESID + UC CONVERSION (PERIODS 263A-M)

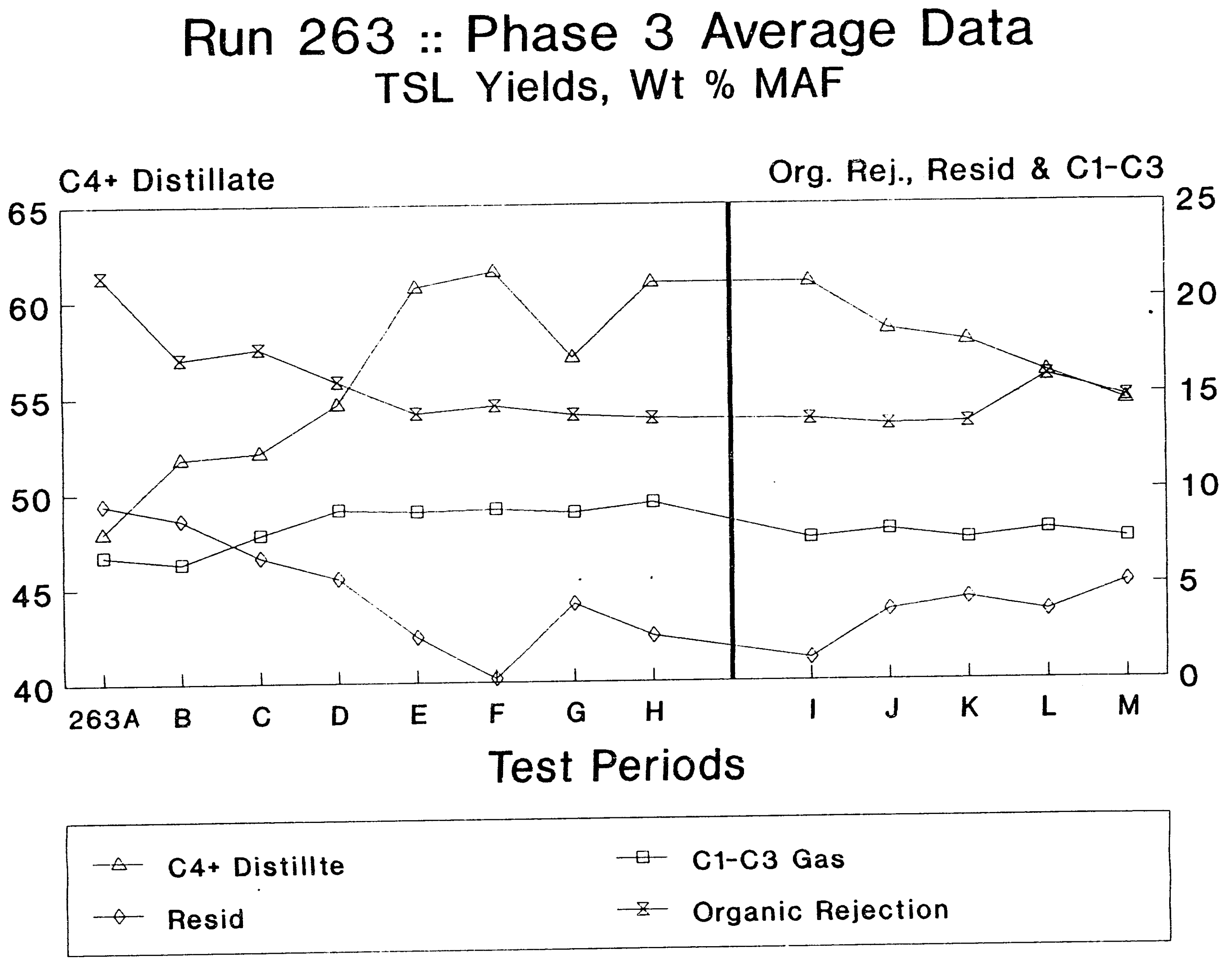


FIGURE 43. OVERALL YIELDS TREND (PERIODS 263A-M)

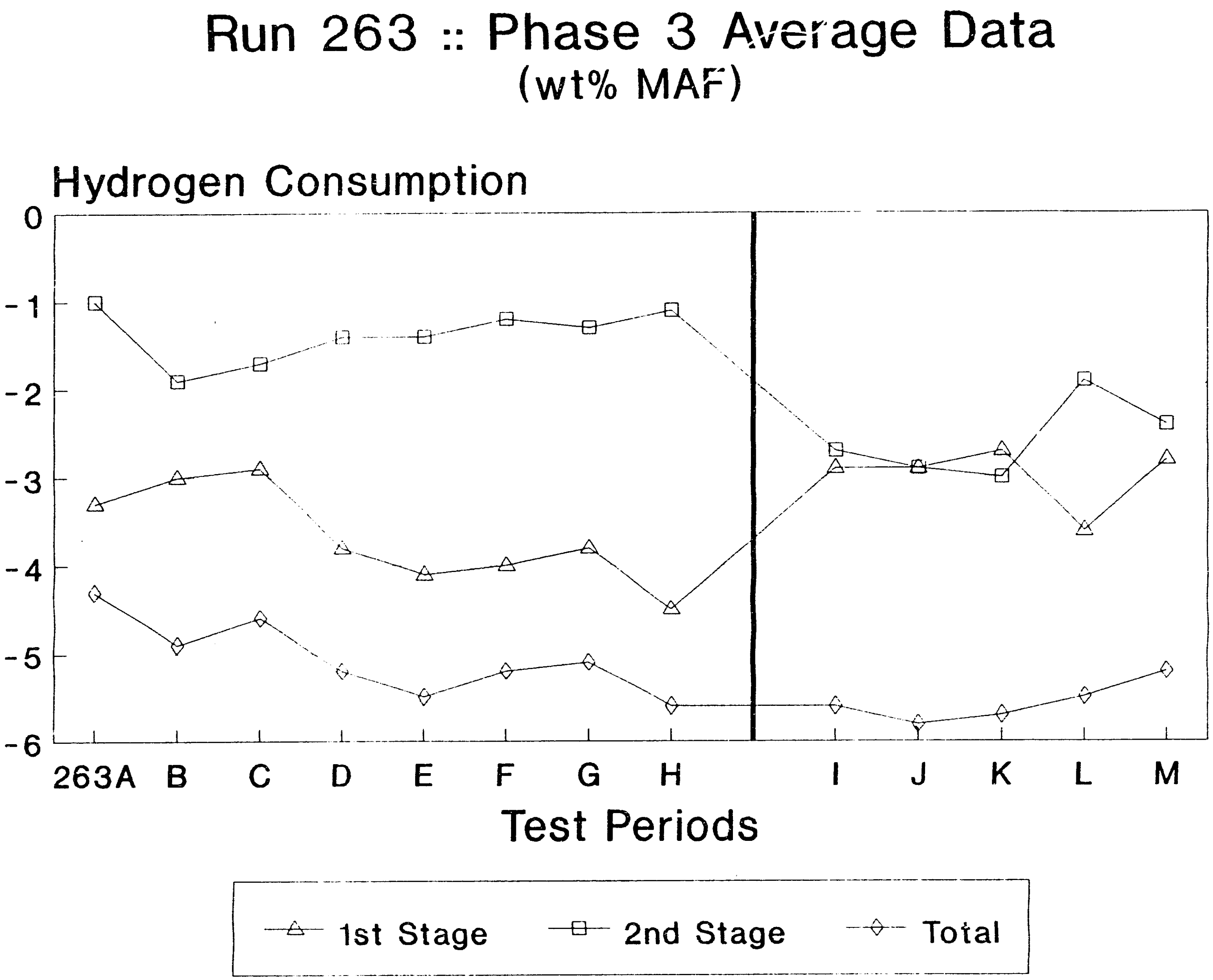
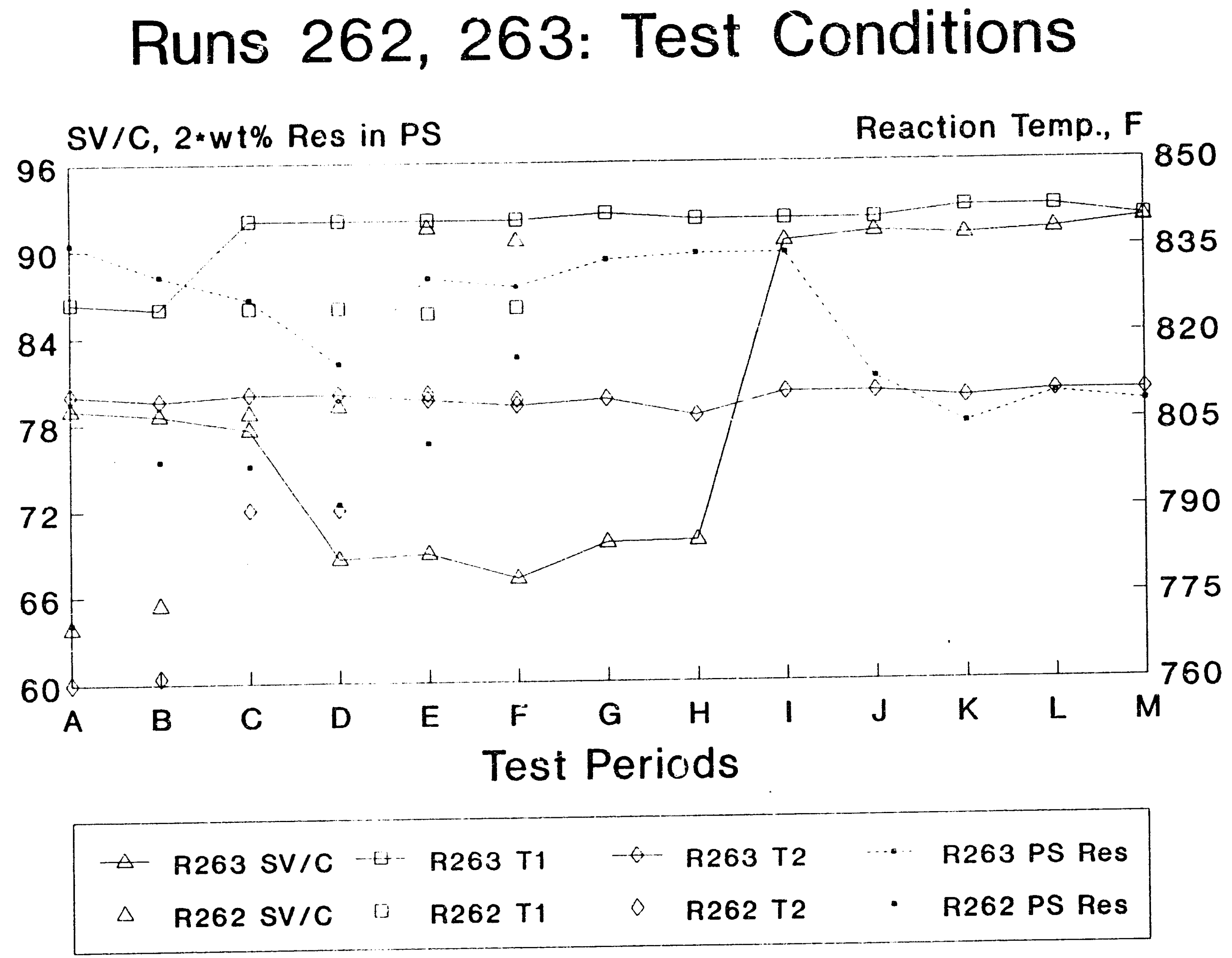


FIGURE 44. HYDROGEN CONSUMPTION TREND (PERIODS 263A-M)



SV/C - lb MF coal/hr/cu ft reactor vol.

FIGURE 45. COMPARISON OF TEST CONDITIONS FOR RUNS 263 AND 262

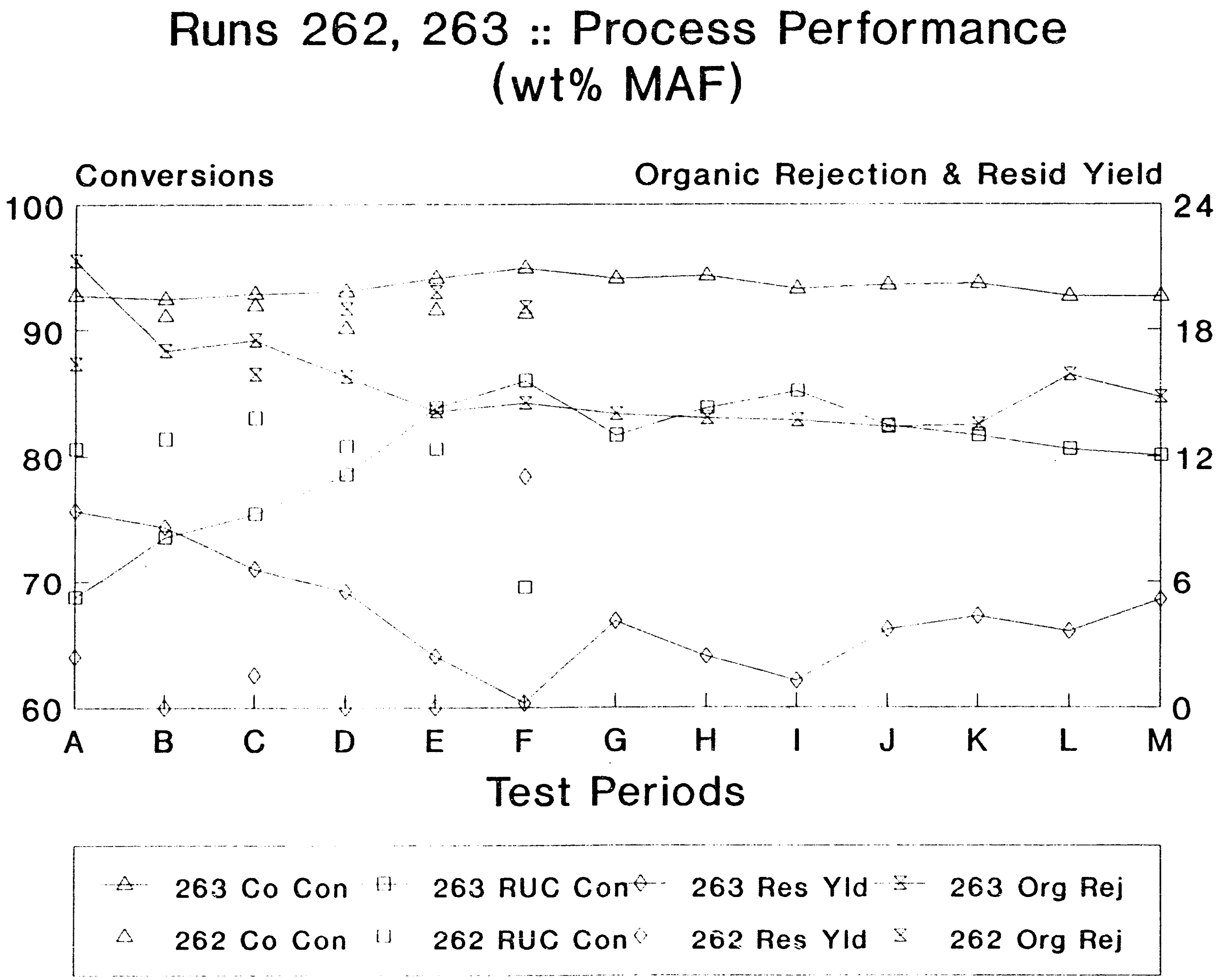


FIGURE 46. COMPARISON OF COAL CONVERSION AND ORGANIC REJECTION (RUNS 263, 262)

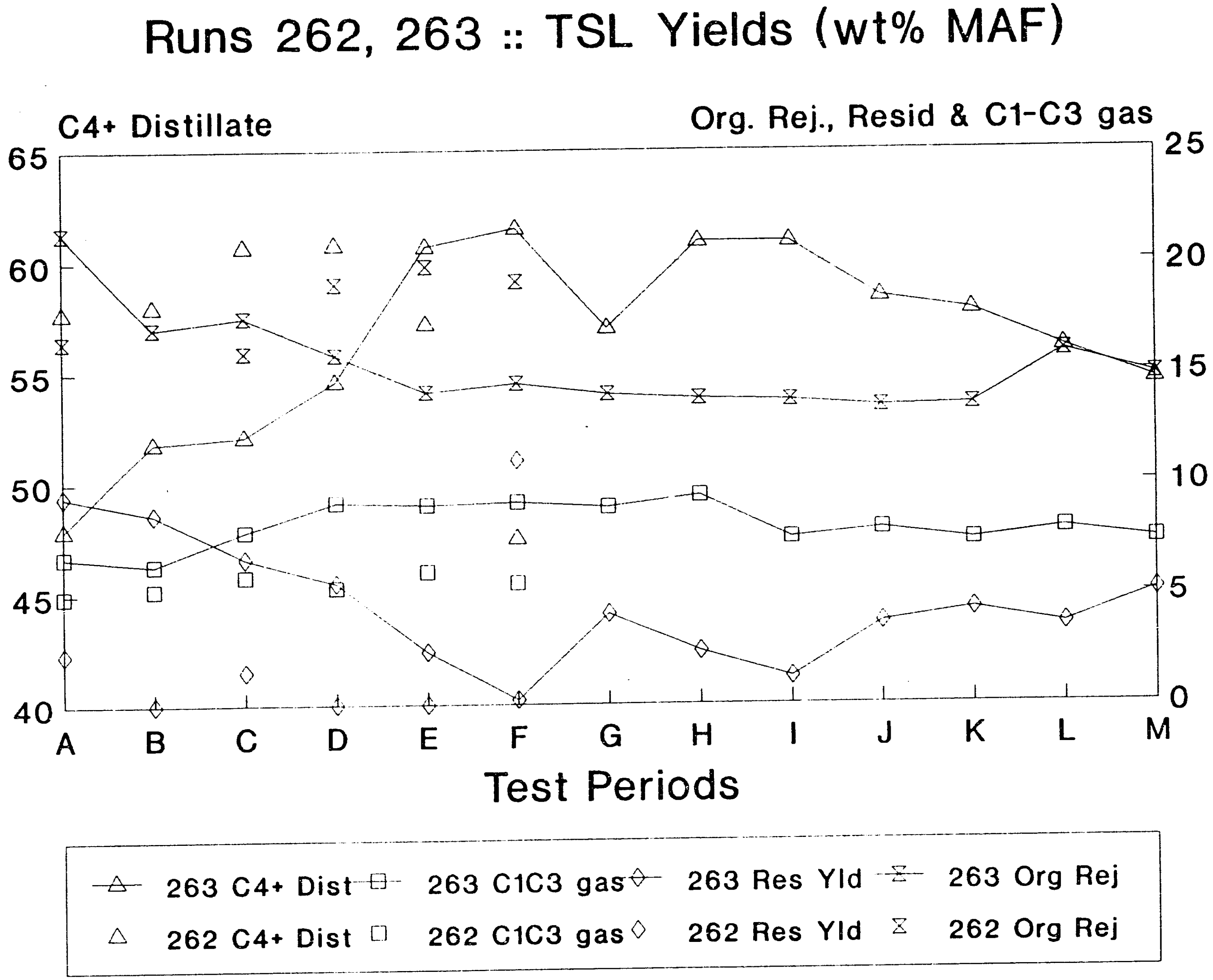
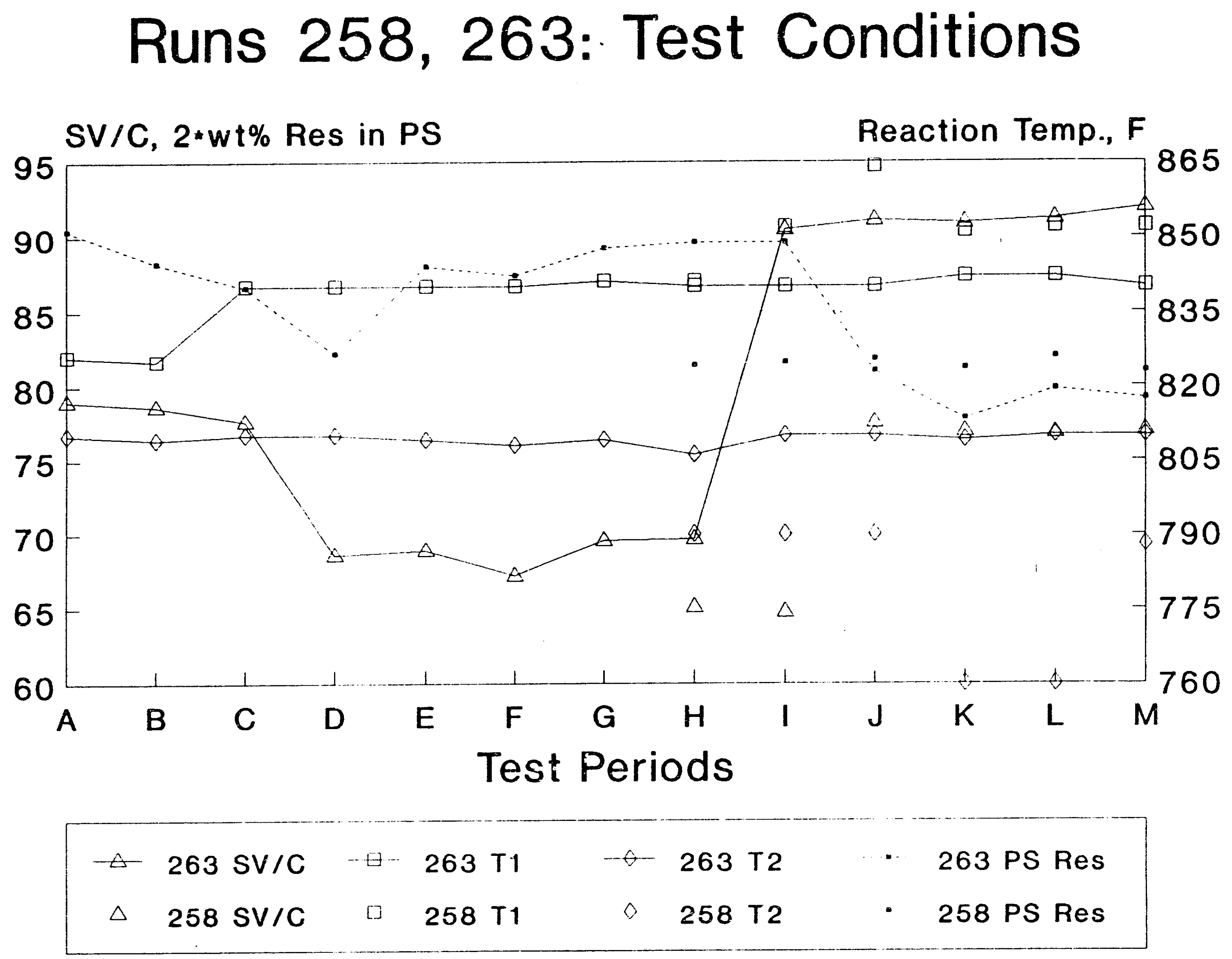


FIGURE 47. COMPARISON OF OVERALL YIELDS (RUNS 263, 262)



SV/C - lb MF coal/hr/cu ft reactor vol.

FIGURE 48. COMPARISON OF TEST CONDITIONS FOR RUNS 263 AND 258

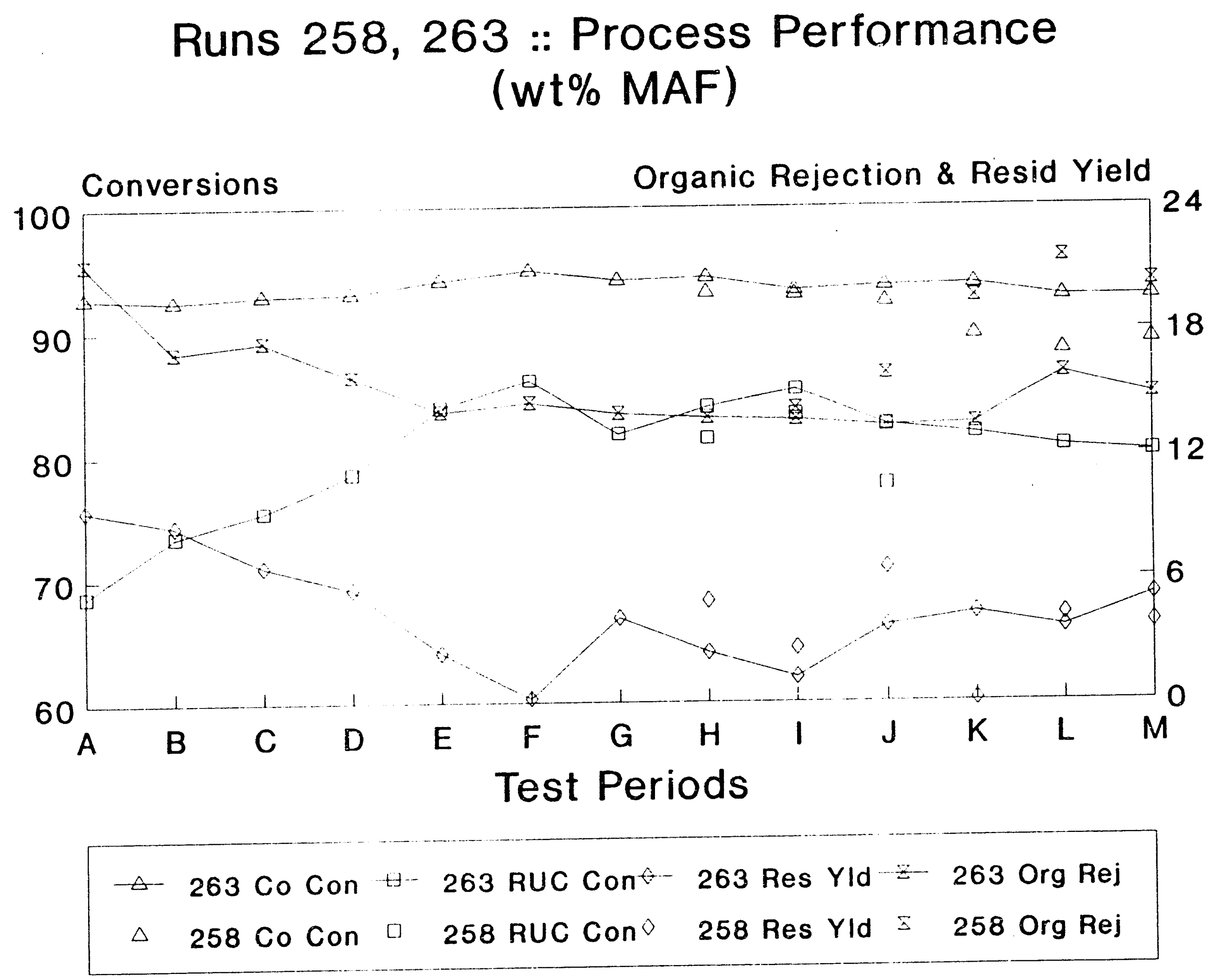


FIGURE 49. COMPARISON OF COAL CONVERSION AND ORGANIC REJECTION (RUNS 263, 258)

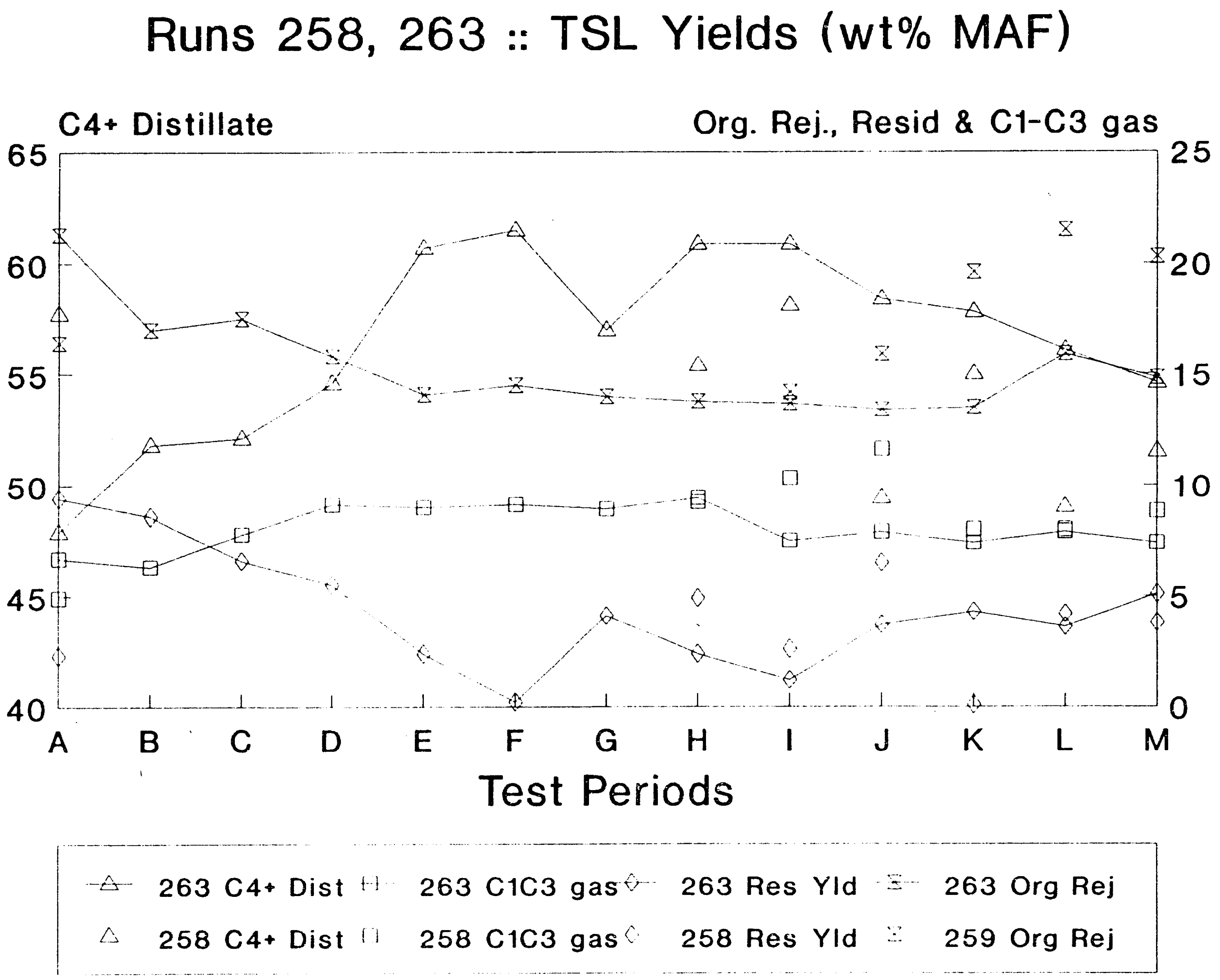
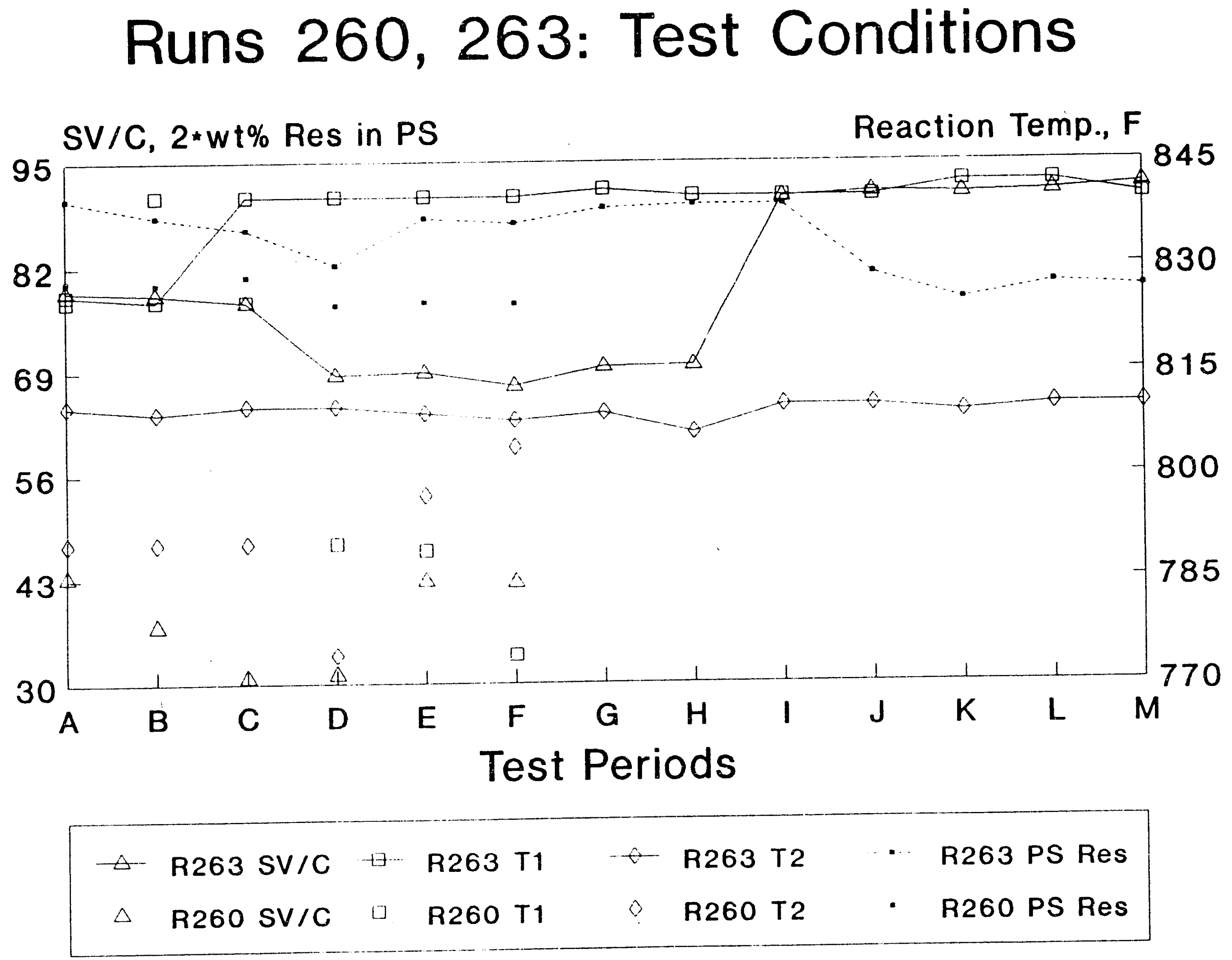


FIGURE 50. COMPARISON OF OVERALL YIELDS (RUNS 263, 258)



SV/C - lb MF coal/hr/cu ft reactor vol.

FIGURE 51. COMPARISON OF TEST CONDITIONS FOR RUNS 263 AND 260

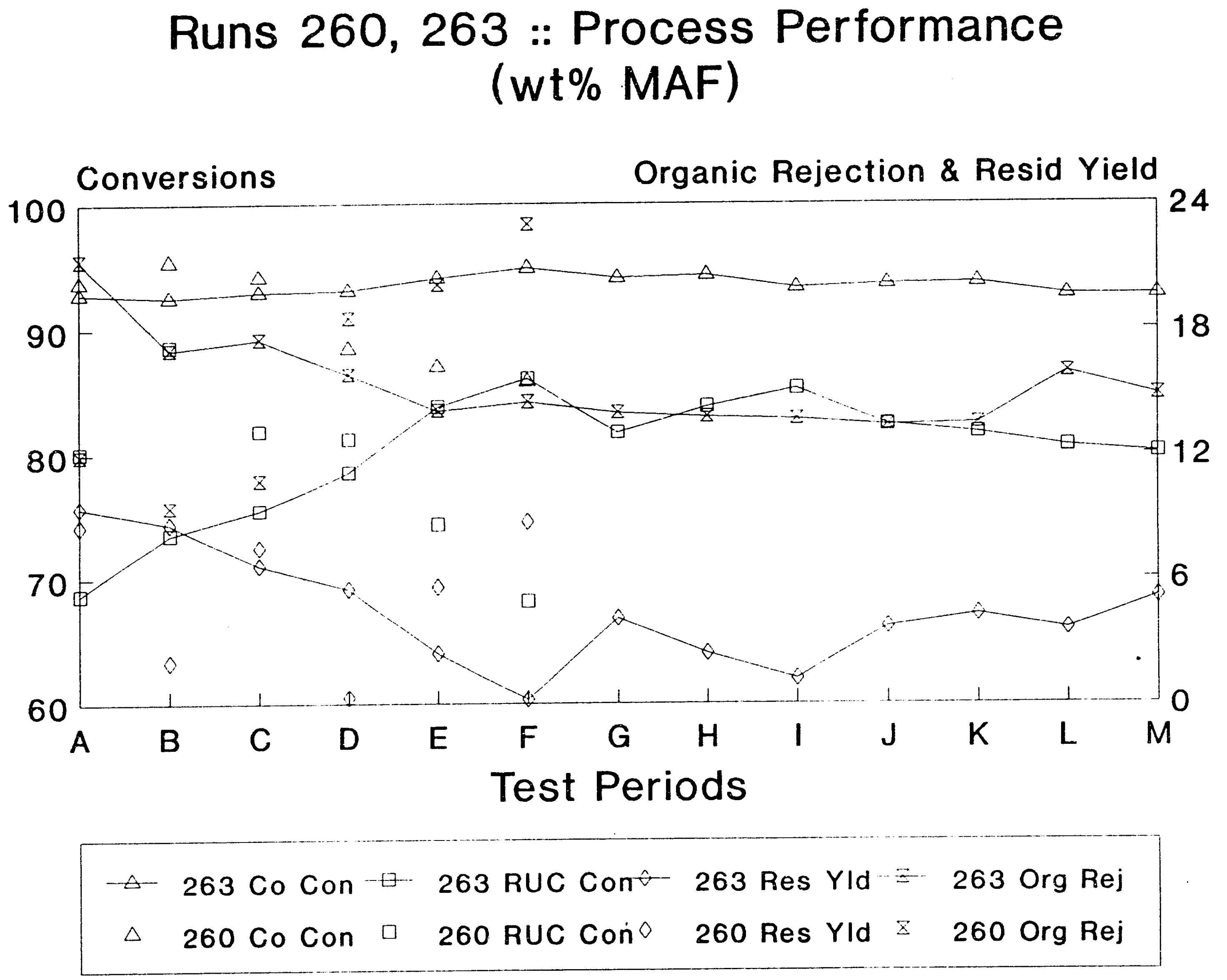


FIGURE 52. COMPARISON OF COAL CONVERSION AND ORGANIC REJECTION (RUNS 263, 260)

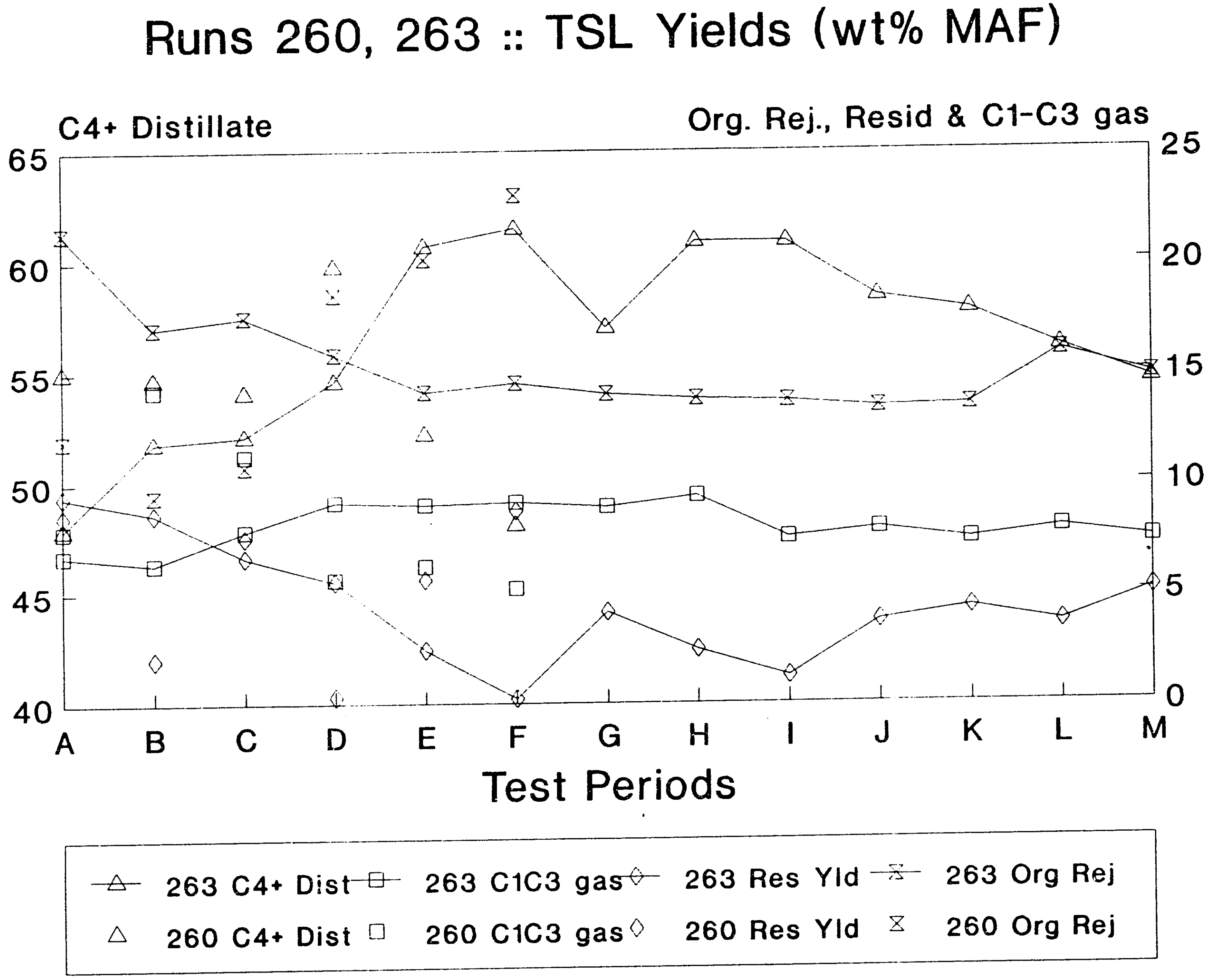
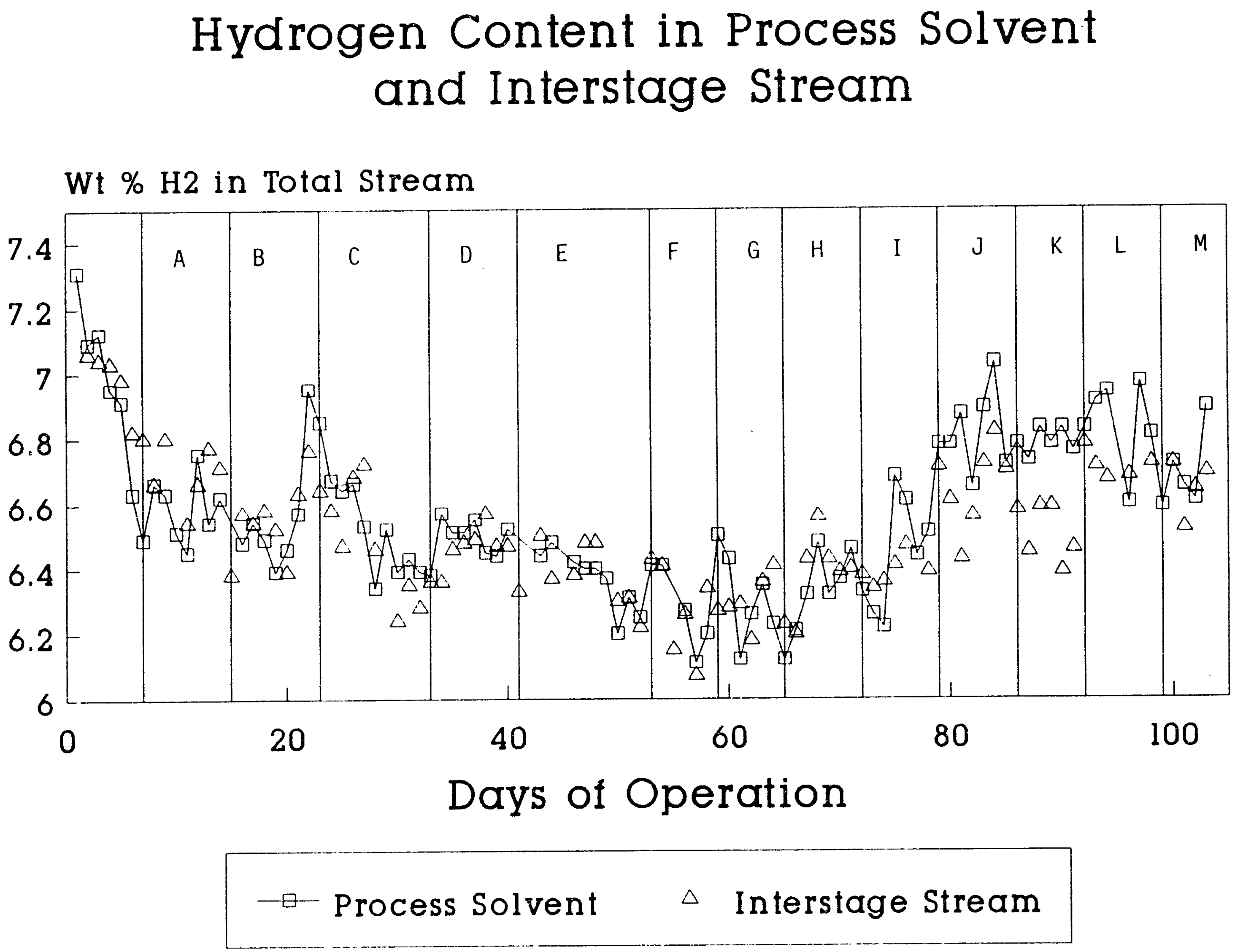
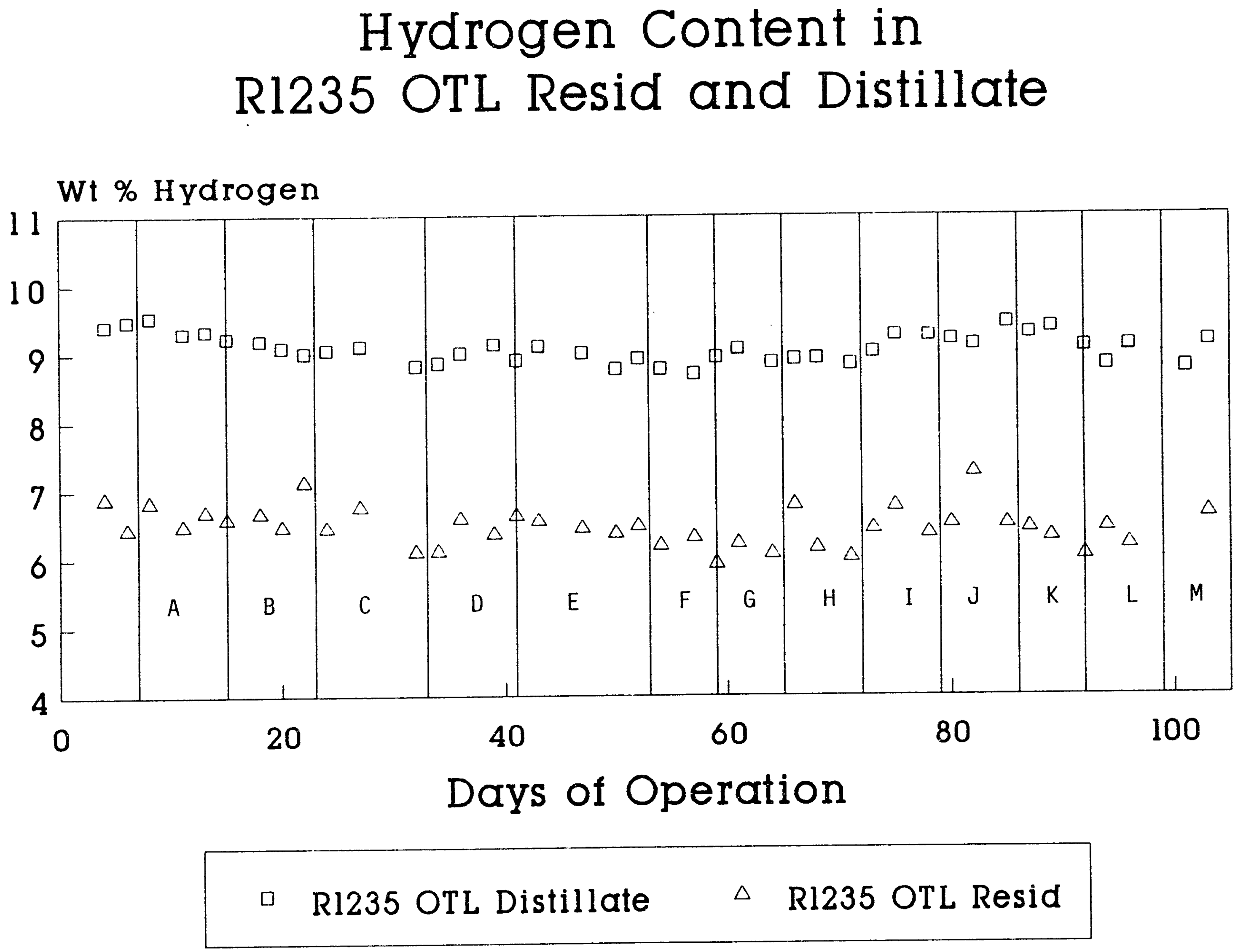


FIGURE 53. COMPARISON OF OVERALL YIELDS (RUNS 263, 260)



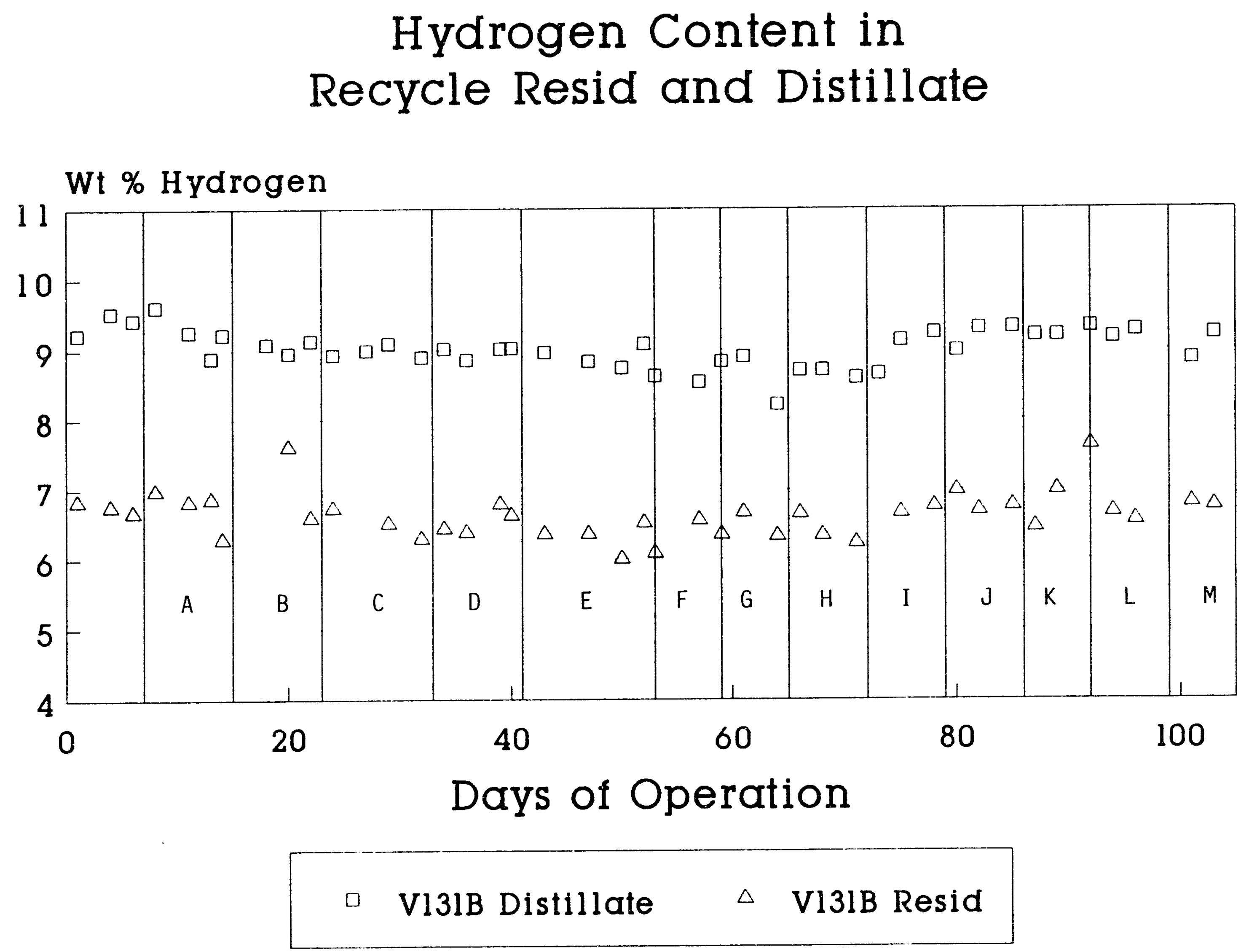
Day 1 - 11/01/91; Day 70 - 01/19/92
Day 30 - 12/07/91; Day 90 - 02/08/92
Day 50 - 12/30/91

FIGURE 54. HYDROGEN CONTENT IN THE PROCESS SOLVENT AND INTERSTAGE STREAM



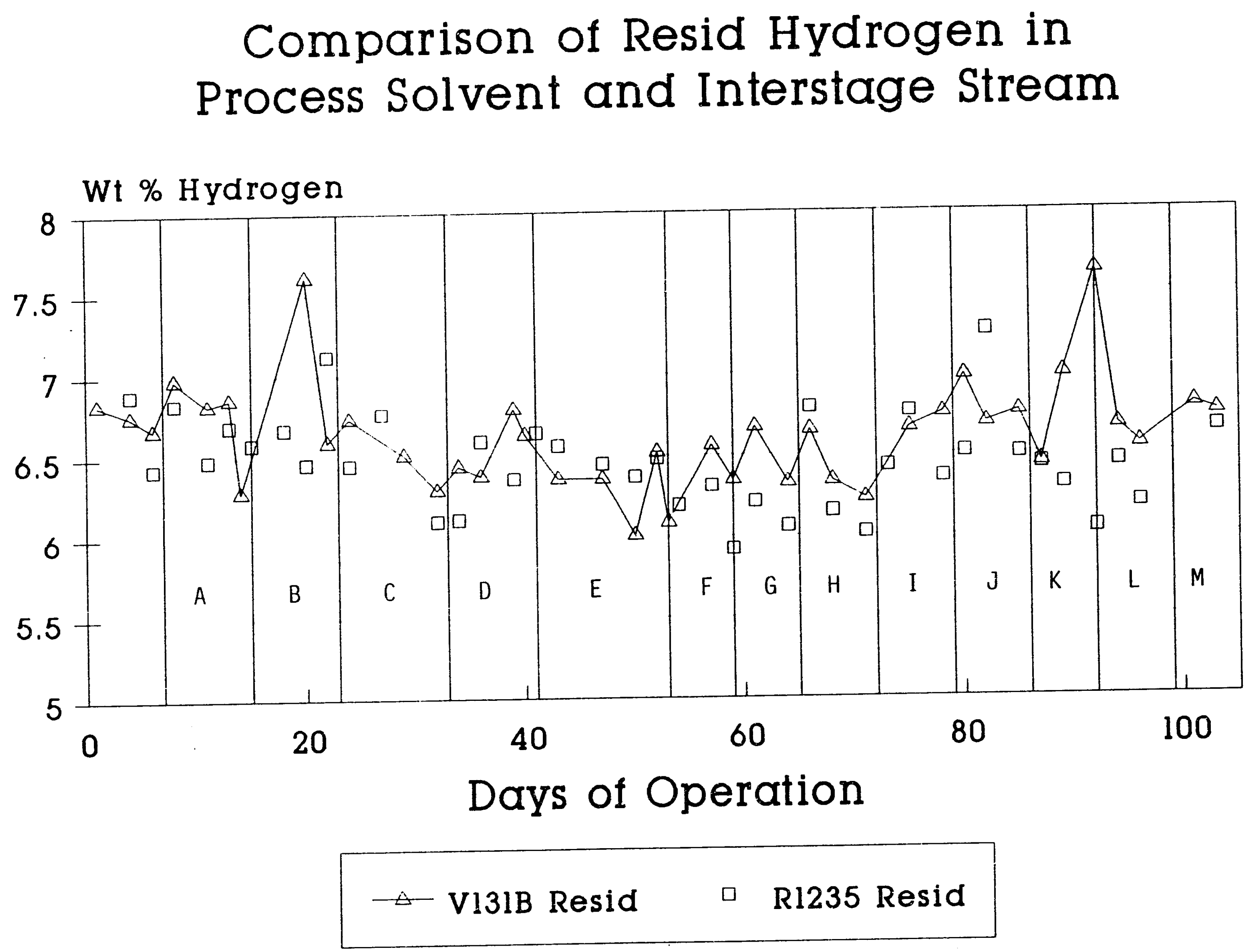
Day 1 = 11/01/91; Day 70 = 01/19/92
Day 30 = 12/07/91; Day 90 = 02/08/92
Day 50 = 12/30/91

FIGURE 55. HYDROGEN CONTENT IN THE DISTILLATE AND RESID PORTIONS OF THE INTERSTAGE STREAM



Day 1 - 11/01/91; Day 70 - 01/19/92
Day 30 - 12/07/91; Day 90 - 02/08/92
Day 50 - 12/30/91

FIGURE 56. HYDROGEN CONTENT IN THE DISTILLATE AND RESID PORTIONS OF THE PROCESS SOLVENT



Day 1 = 11/01/91; Day 70 = 01/19/92
Day 30 = 12/07/91; Day 90 = 02/08/92
Day 50 = 12/30/91

FIGURE 57. COMPARISON OF RESID HYDROGEN IN THE PROCESS SOLVENT AND INTERSTAGE STREAM

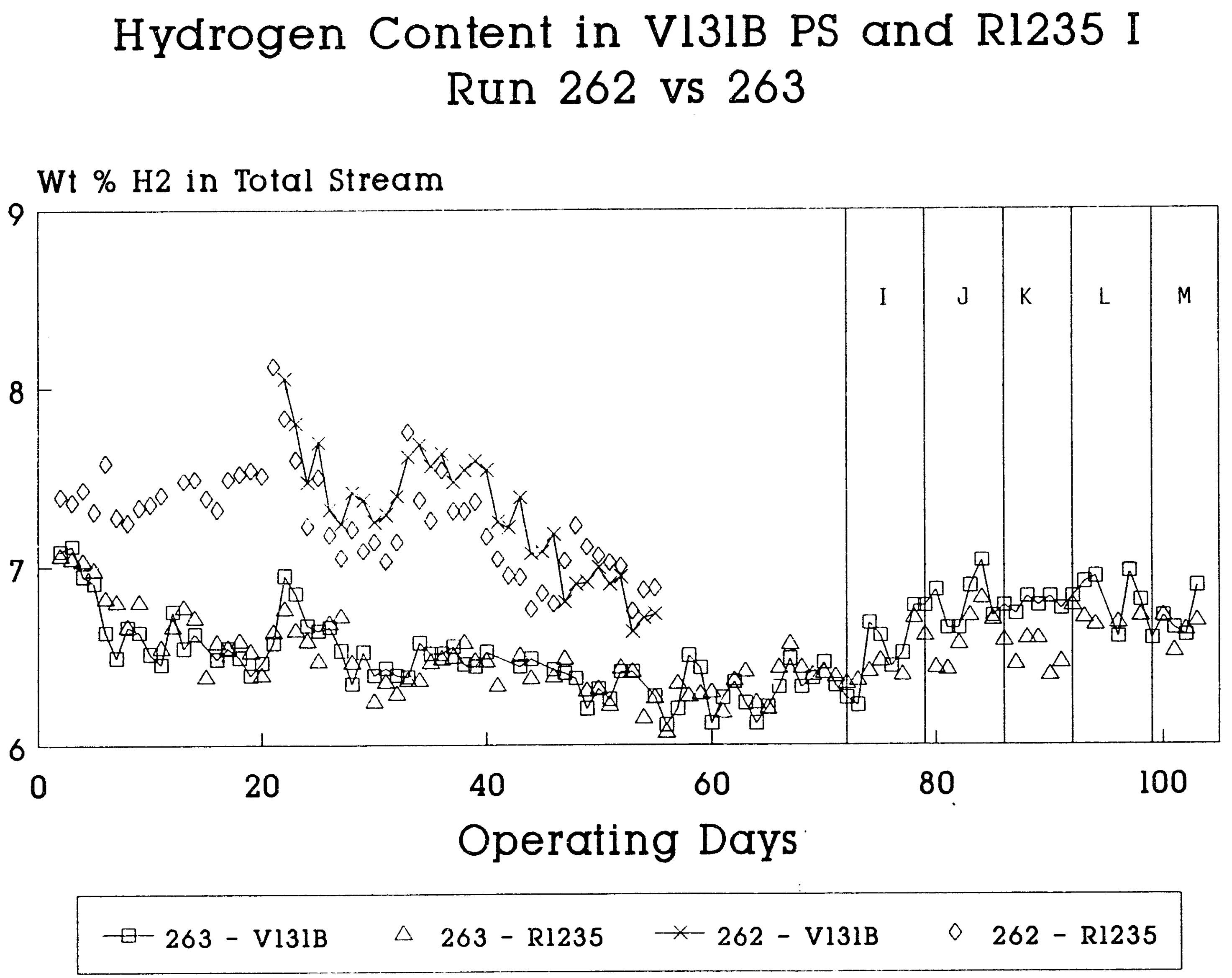
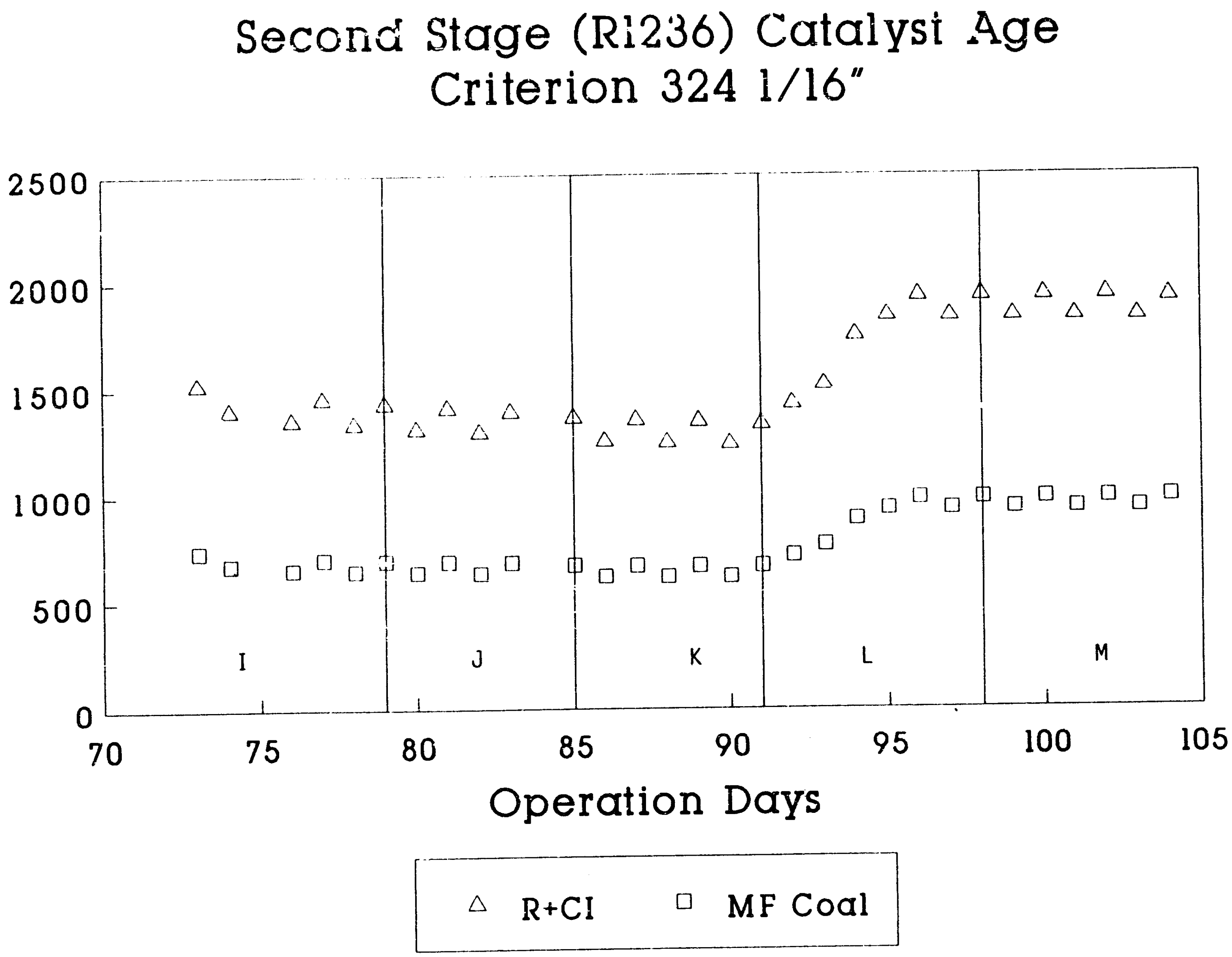
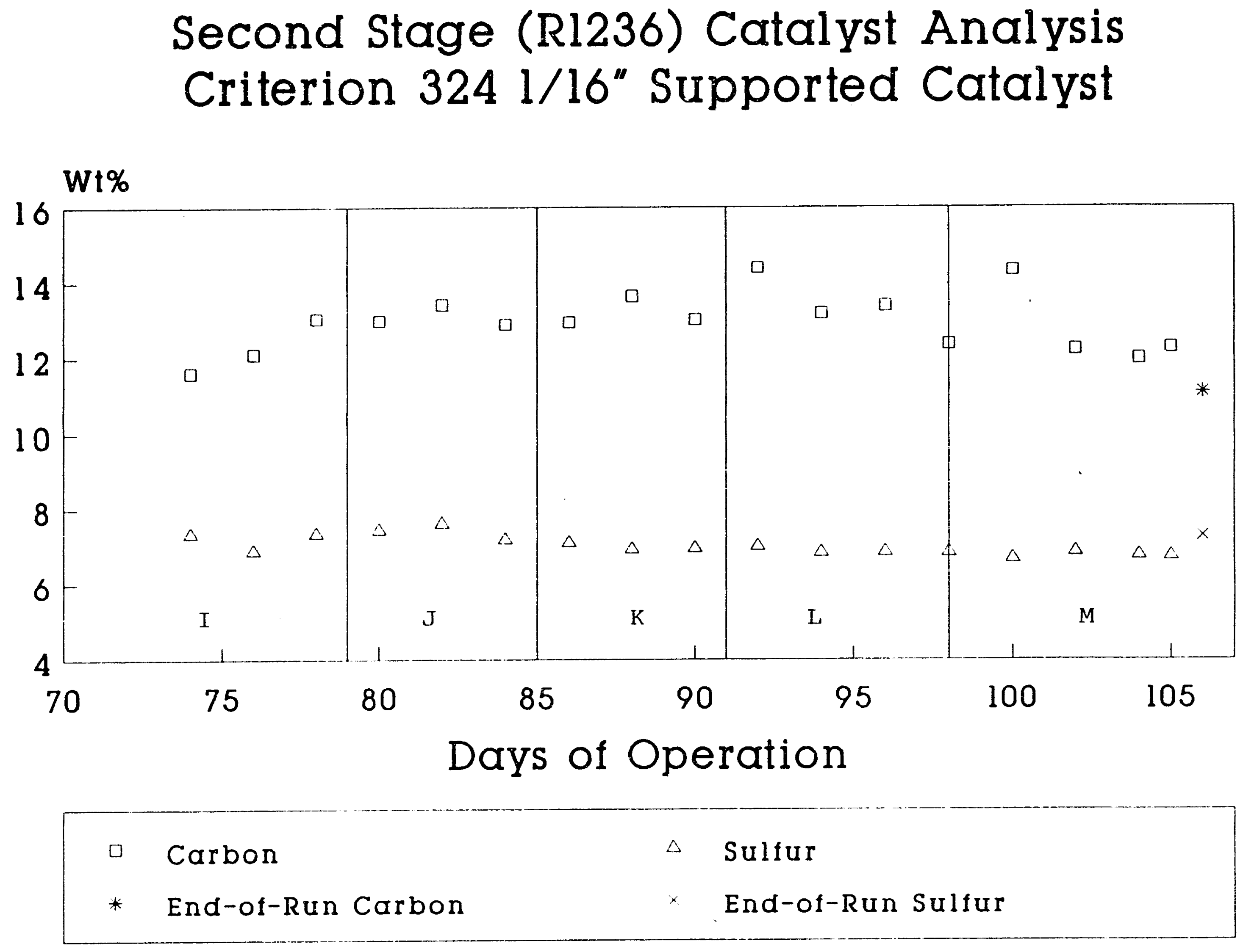


FIGURE 58. COMPARISON OF HYDROGEN CONTENT IN THE PROCESS SOLVENT AND INTERSTAGE STREAM FOR RUNS 263 AND 262



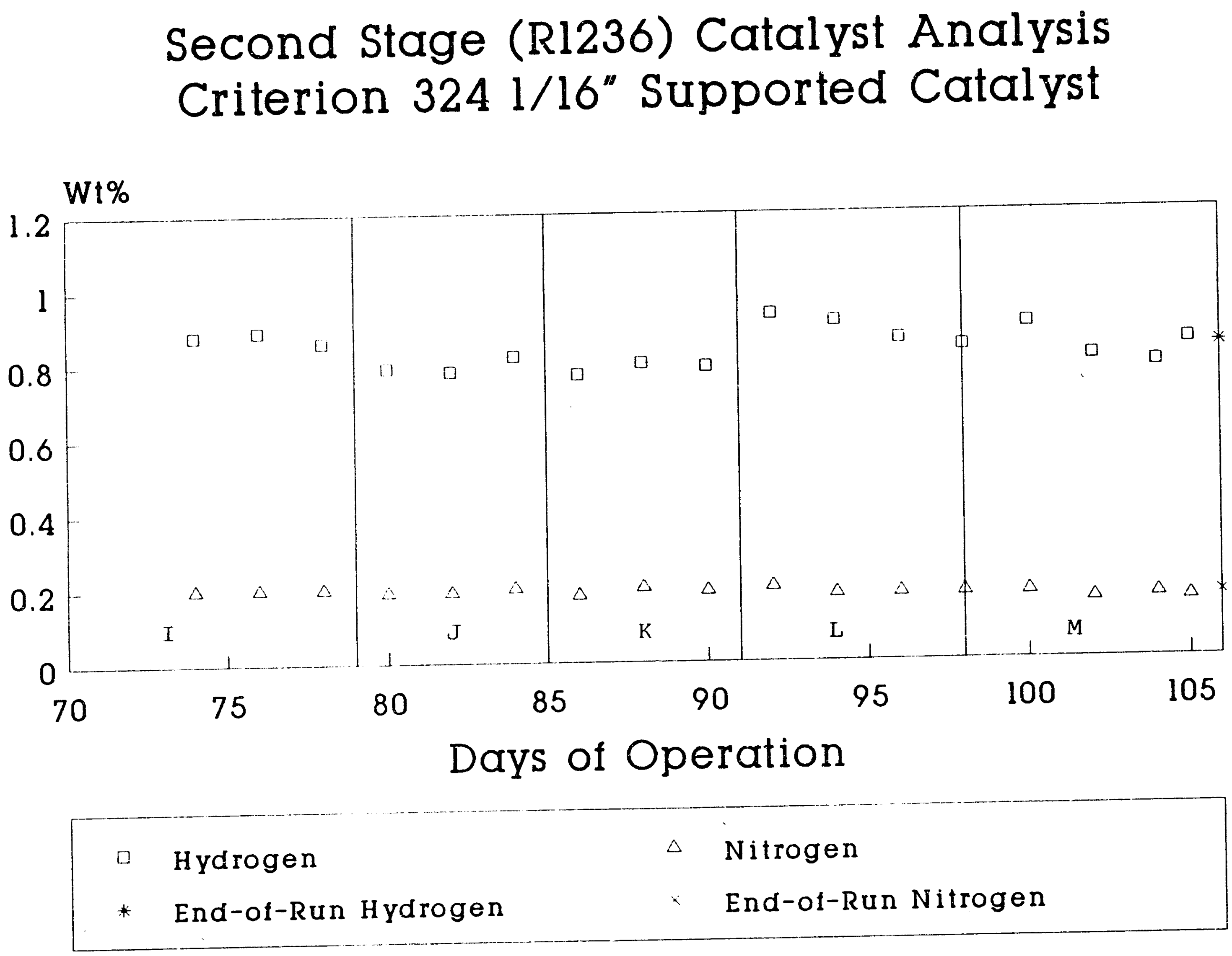
Day 70 = 1/19/92; Day 80 = 1/29/92
Day 90 = 2/08/92; Day 100 = 2/18/98

FIGURE 59. SECOND STAGE SUPPORTED CATALYST AGE



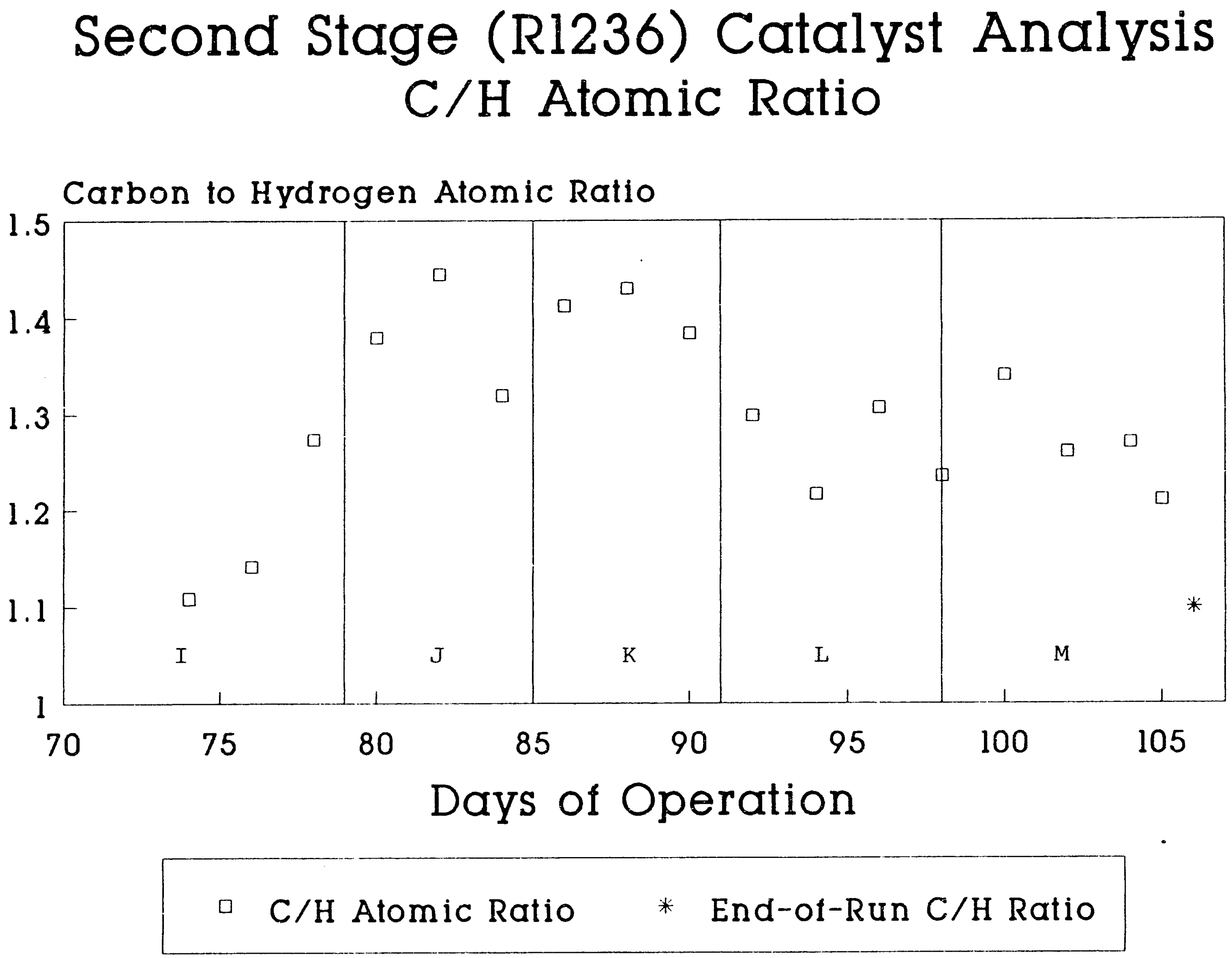
Day 70 = 1/19/92; Day 80 = 1/29/92
Day 90 = 2/08/92; Day 100 = 2/18/92

FIGURE 60. CARBON AND SULFUR DEPOSITS ON THE SECOND STAGE CATALYST



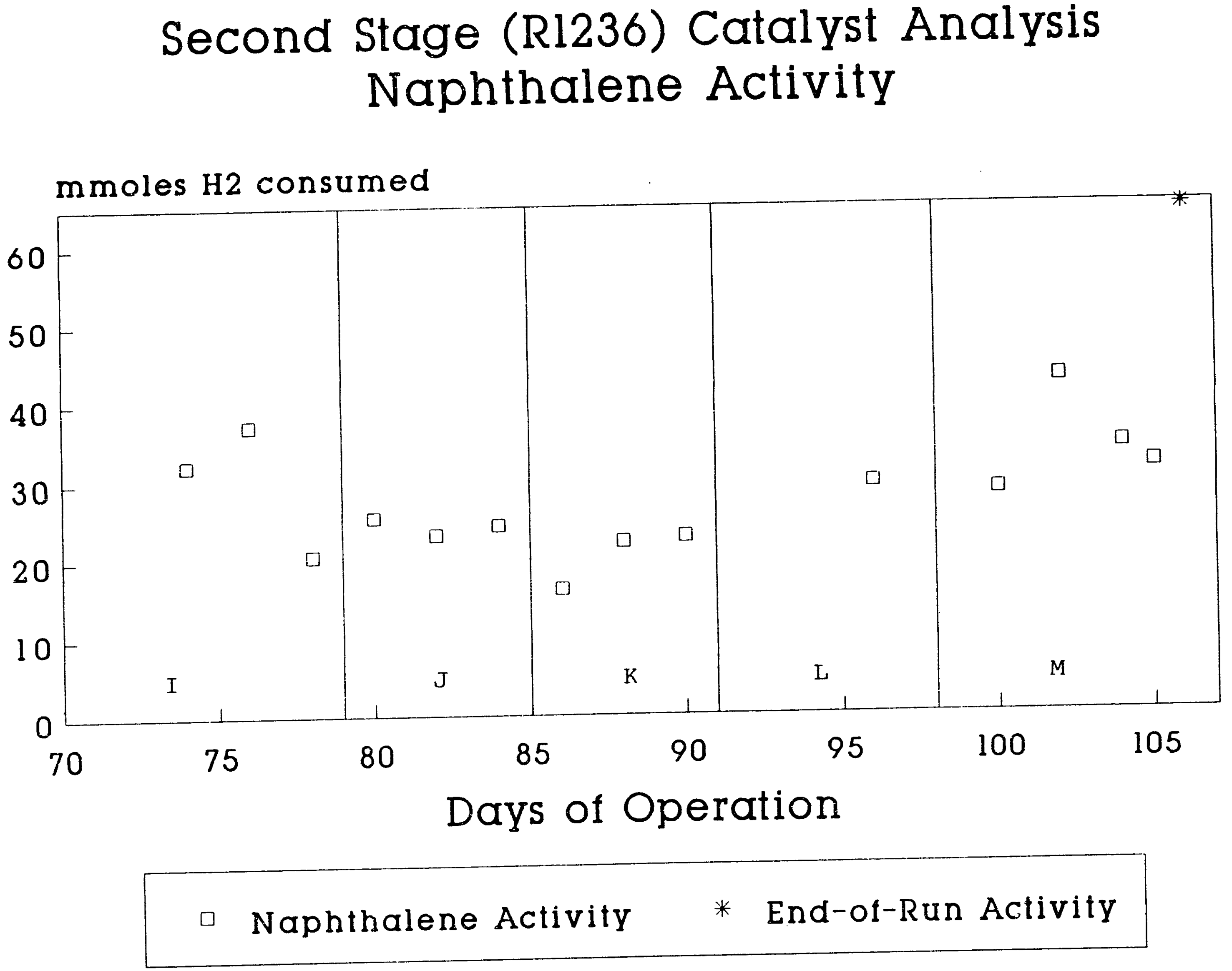
Day 70 = 1/19/92; Day 80 = 1/29/92
Day 90 = 2/08/92; Day 100 = 2/18/92

FIGURE 61. HYDROGEN AND NITROGEN DEPOSITS ON THE SECOND STAGE CATALYST



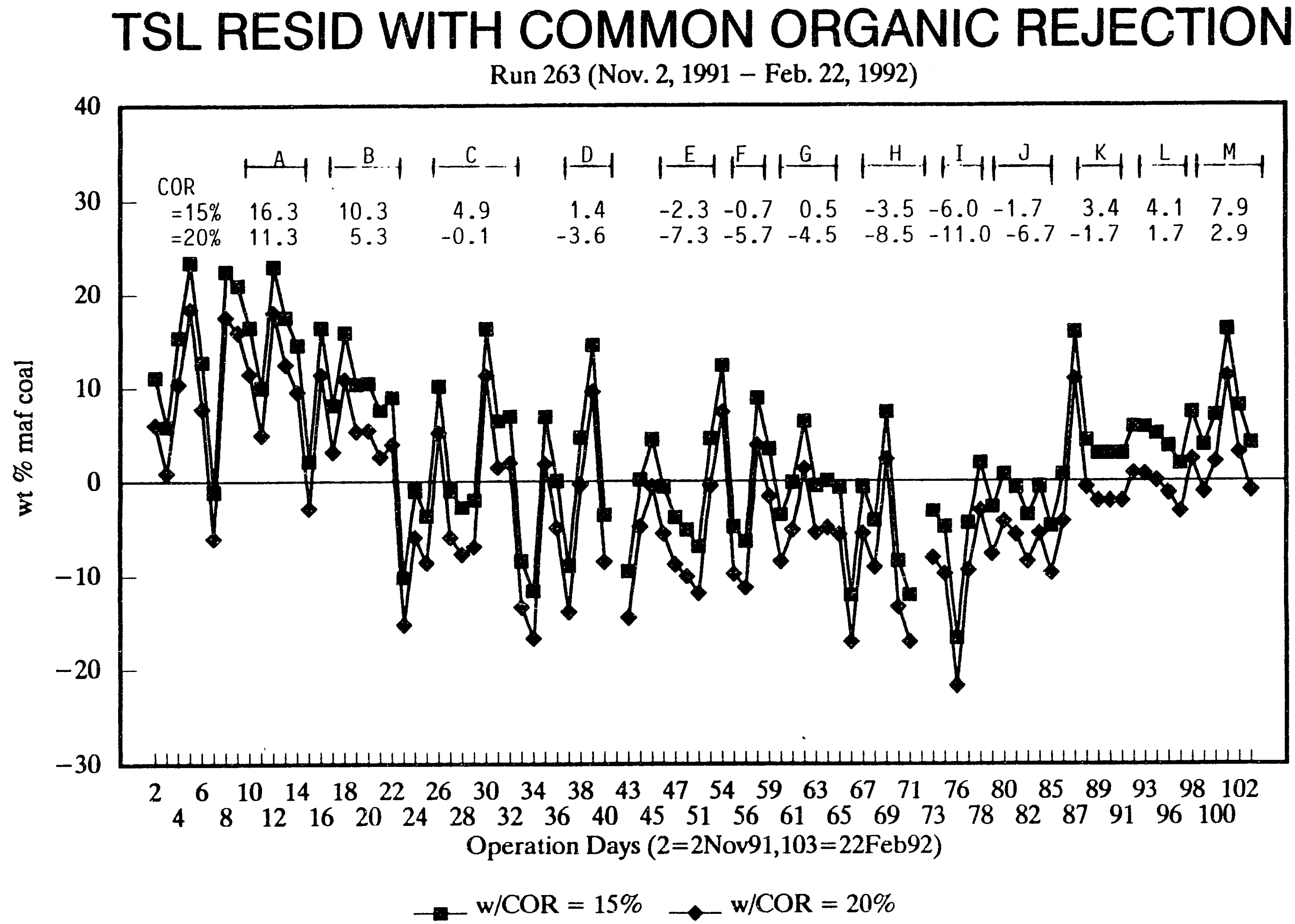
Day 70 = 1/19/92; Day 80 = 1/29/92
Day 90 = 2/08/92; Day 100 = 2/18/92

FIGURE 62. CARBON TO HYDROGEN ATOMIC RATIO ON THE SECOND STAGE CATALYST



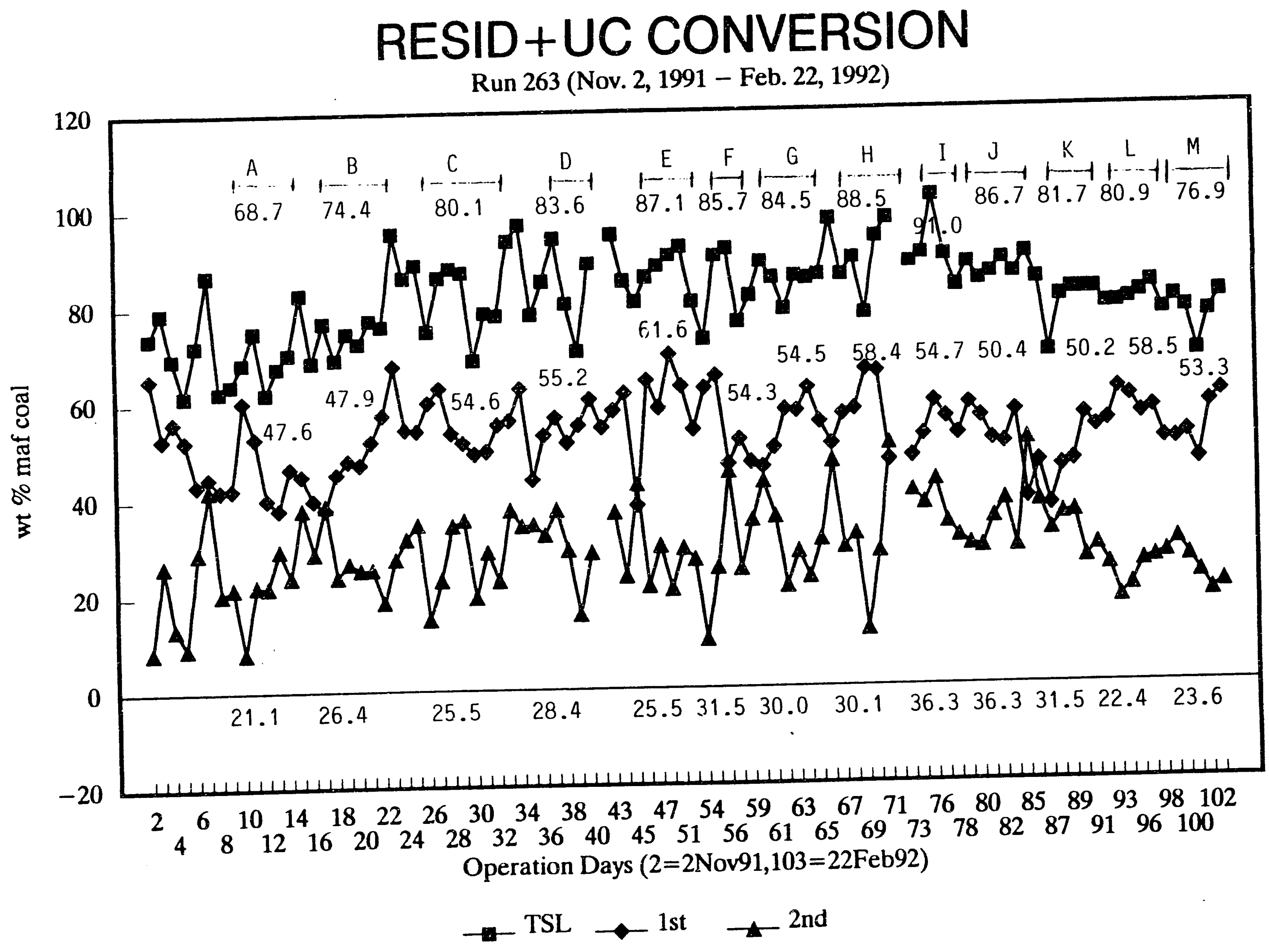
Day 70 = 1/19/92; Day 80 = 1/29/92
Day 90 = 2/08/92; Day 100 = 2/18/92

FIGURE 63. NAPHTHALENE ACTIVITY OF THE SECOND STAGE CATALYST



* Black Thunder in thermal/thermal (od=1−71); thermal/catalytic since Jan. 21 (od=72) w/ 3 lb/t cat rep (od=74−90) batch (od=91−97), 2 lb/t (od=98−103); Molyvan L addition (Molyvan 822 in Jan. 1−26 (od=52−77))

FIGURE 64. TREND DATA OF OVERALL TWO-STAGE EXCESS RESID YIELD WITH COMMON ORGANIC REJECTION



* Phase 2 data measured

FIGURE 65. TREND DATA OF OVERALL TWO-STAGE RESID + UC CONVERSION

]

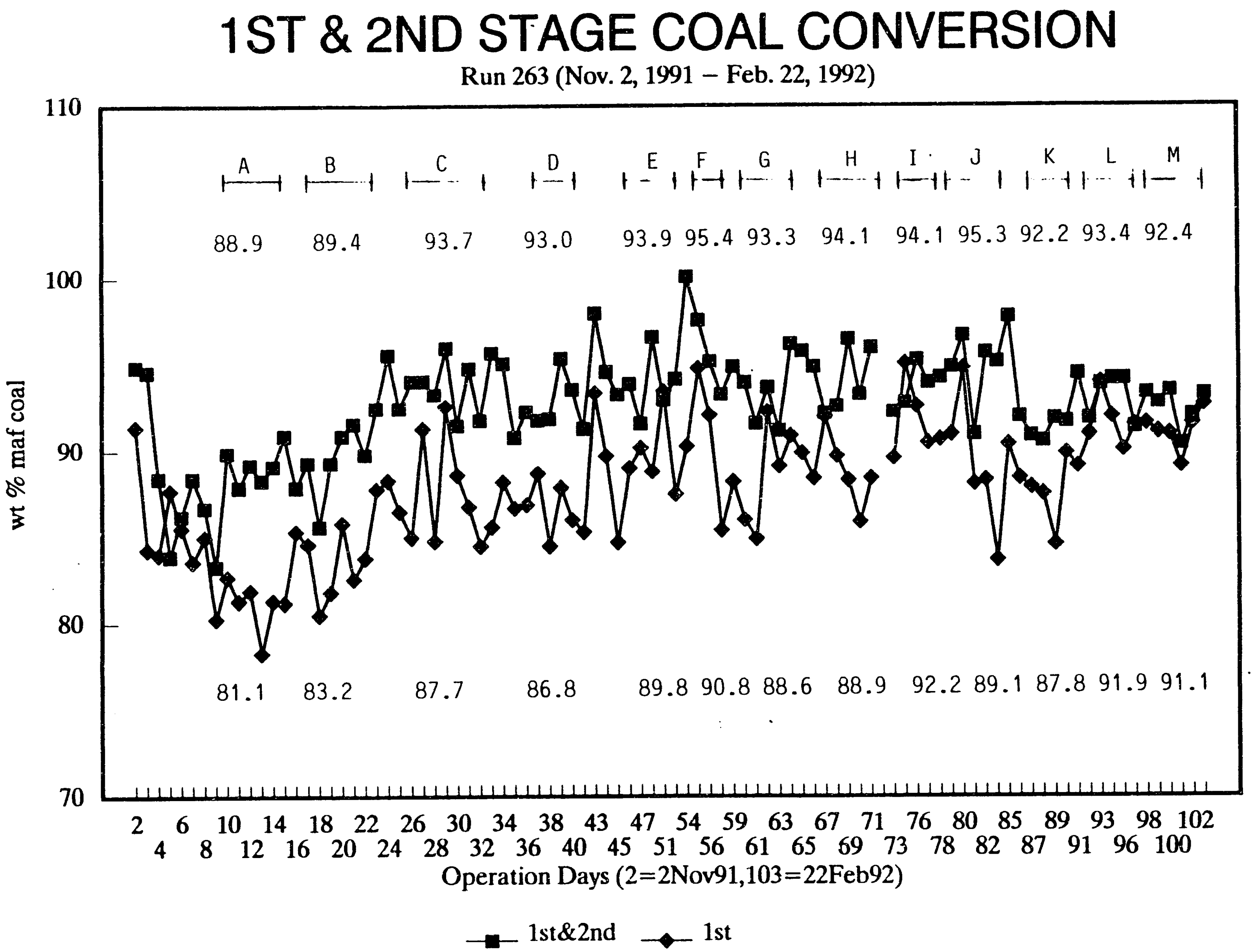


FIGURE 66. TREND DATA OF COAL CONVERSION

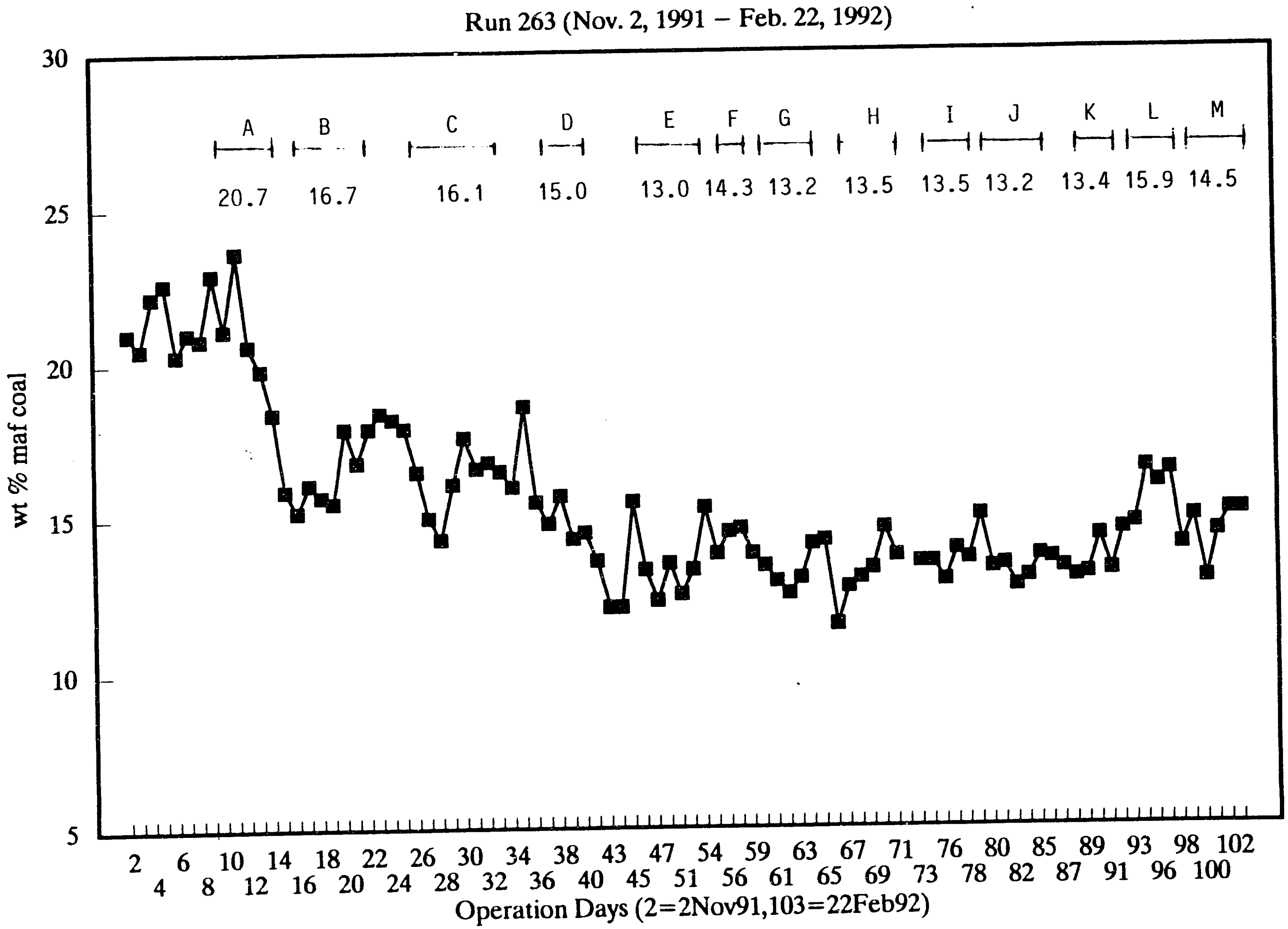


FIGURE 67. TREND DATA OF ORGANICS IN ROSE-SR^{SM} BOTTOMS PRODUCT

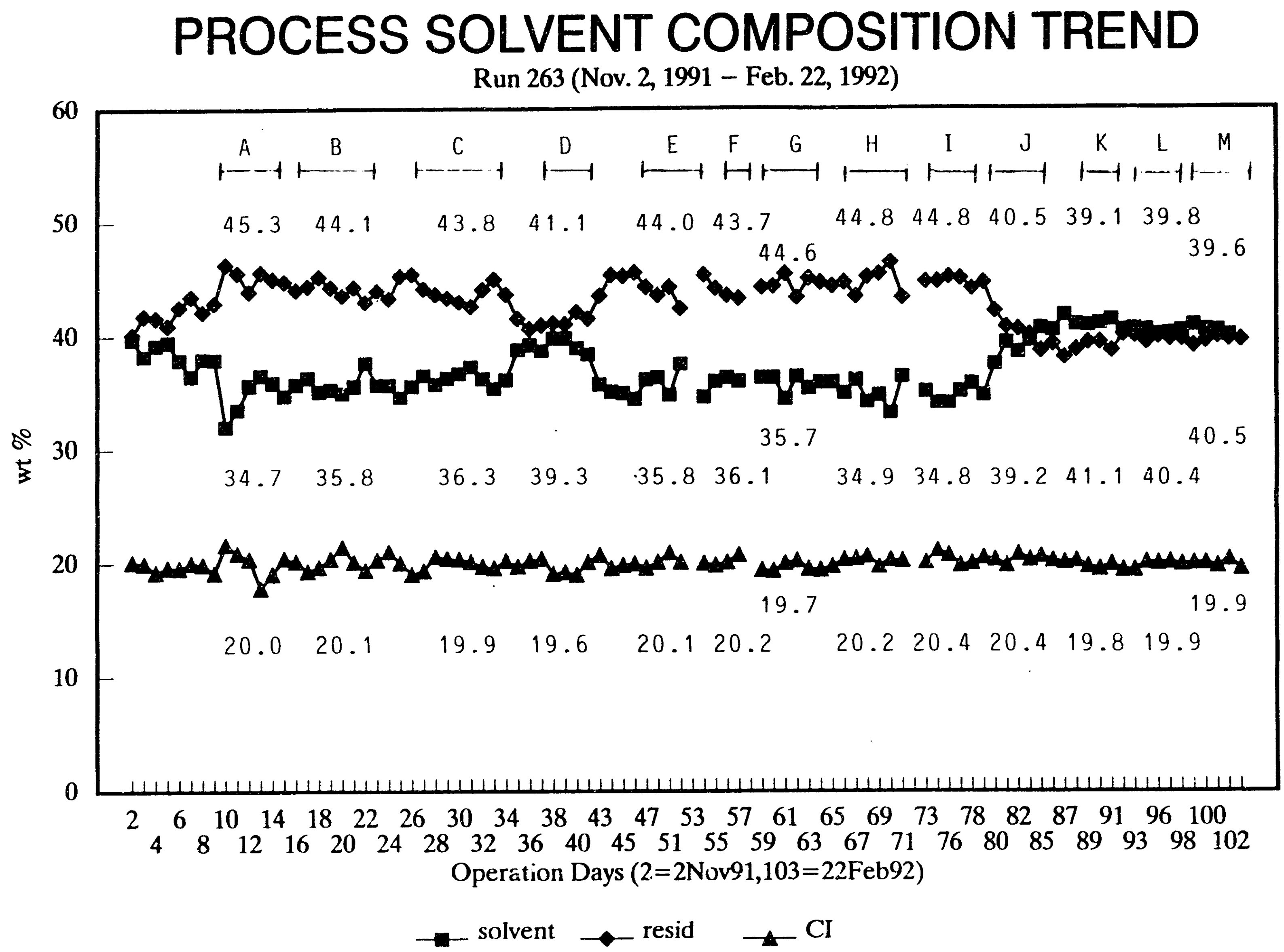


FIGURE 68. TREND DATA OF PROCESS SOLVENT COMPOSITION

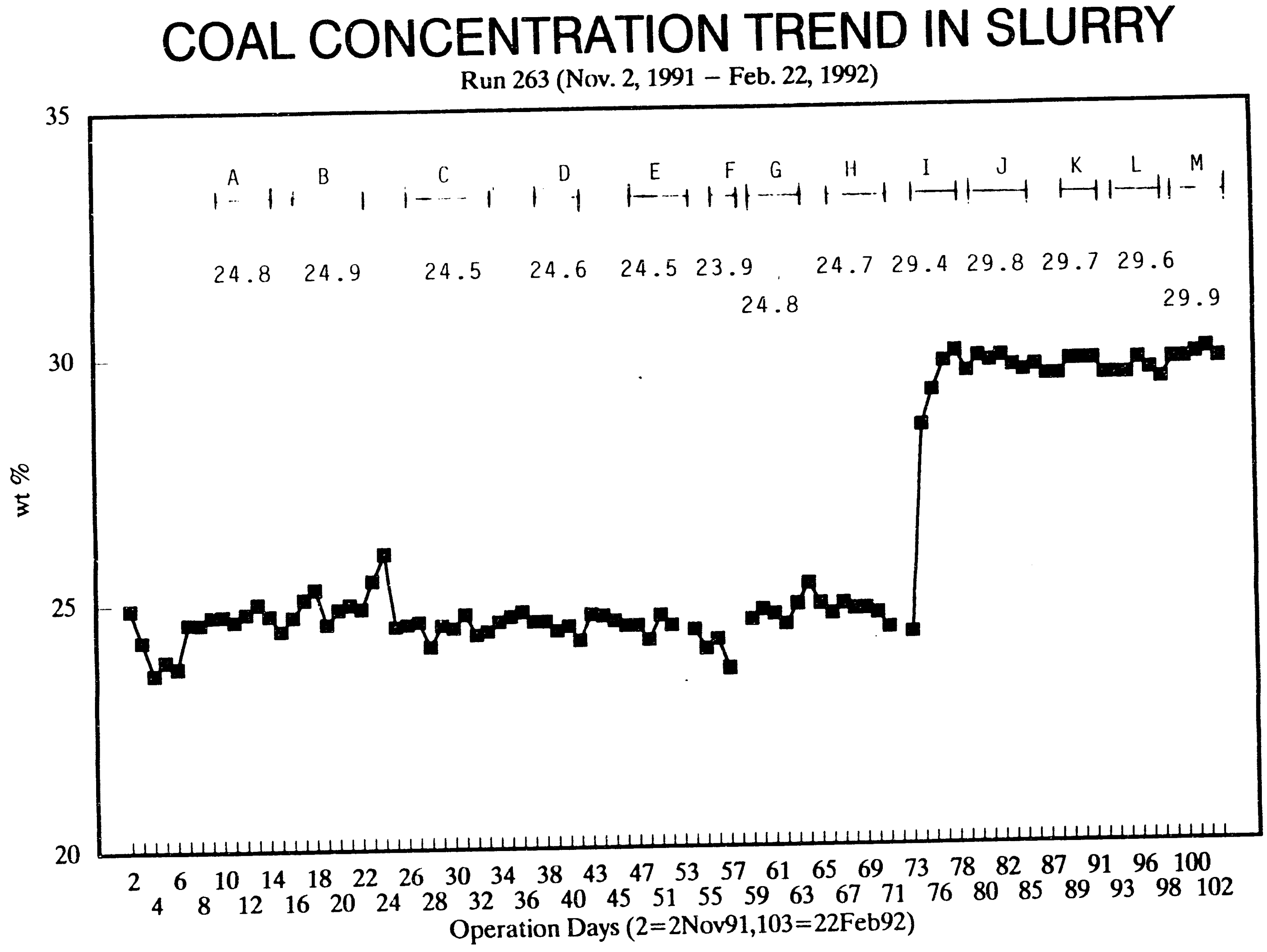


FIGURE 69. TREND DATA OF COAL CONCENTRATION IN SLURRY

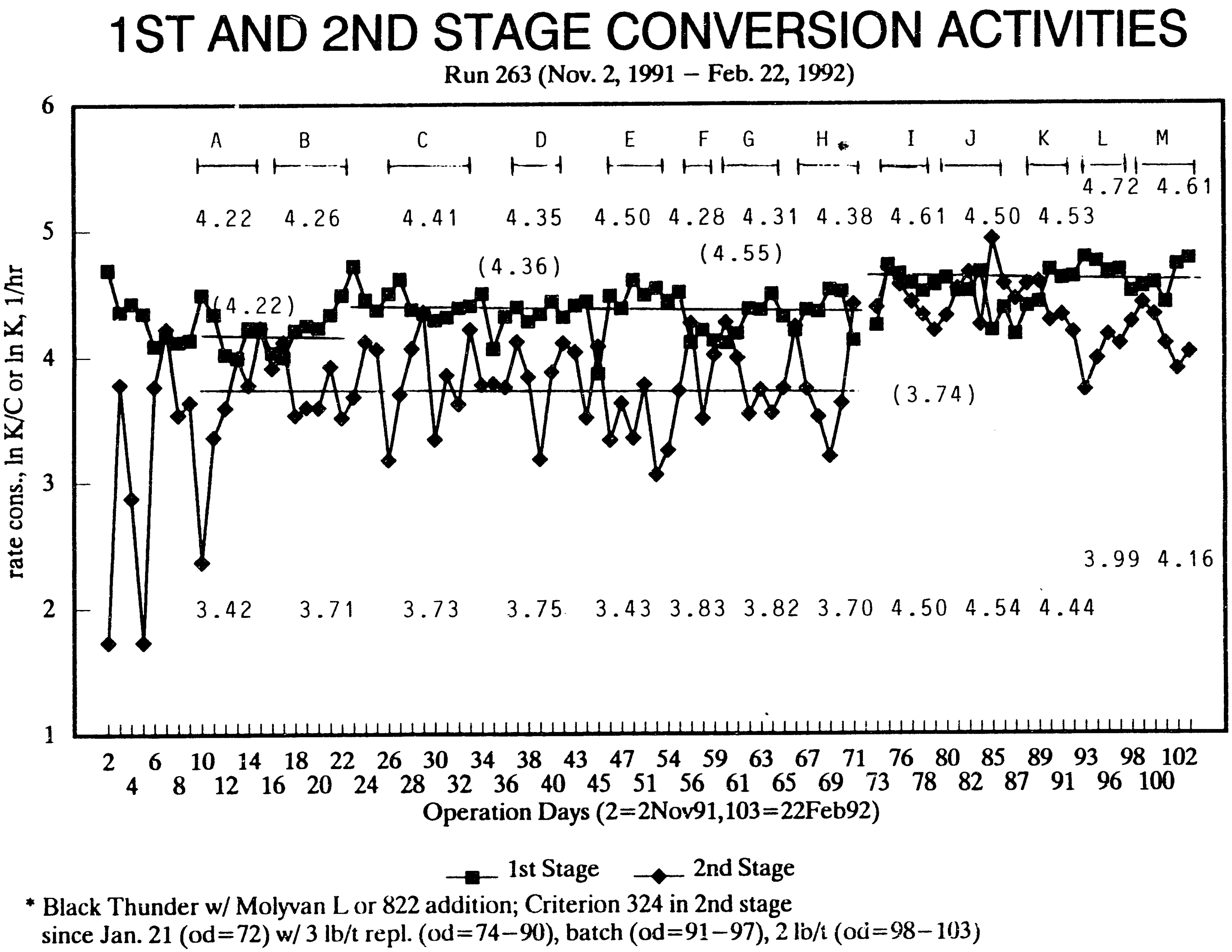


FIGURE 70. FIRST STAGE THERMAL AND SECOND STAGE CATALYTIC CONVERSION ACTIVITY TRENDS

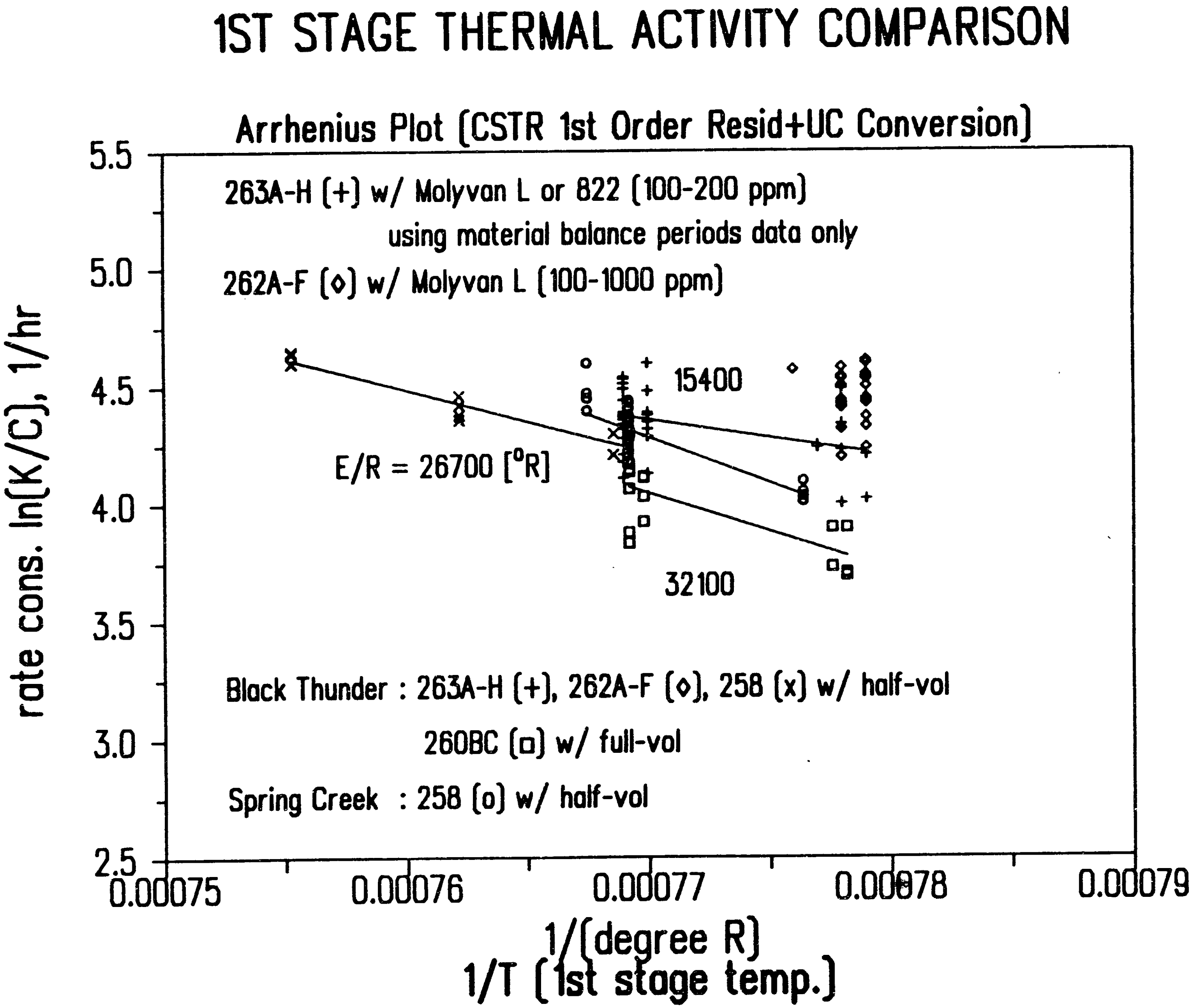


FIGURE 71. FIRST STAGE THERMAL ACTIVITY COMPARISON AND ARRHENIUS PLOT FOR THERMAL ACTIVATION ENERGY (RUNS 263A-H, 262, 260, 258)

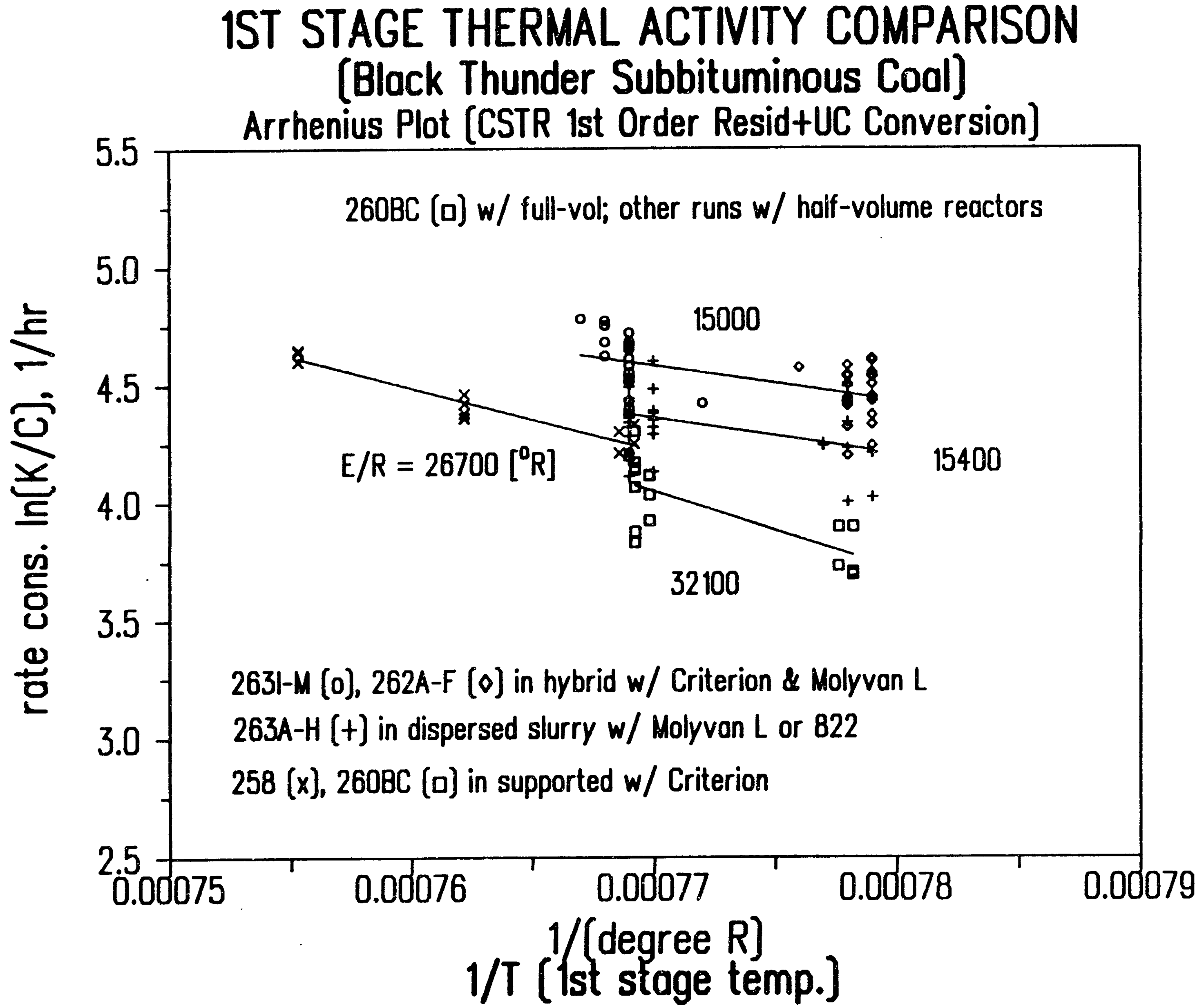


FIGURE 72. FIRST STAGE THERMAL ACTIVITY COMPARISON AND ARRHENIUS PLOT FOR THERMAL ACTIVATION ENERGY (RUNS 263A-M, 262, 260, 258)

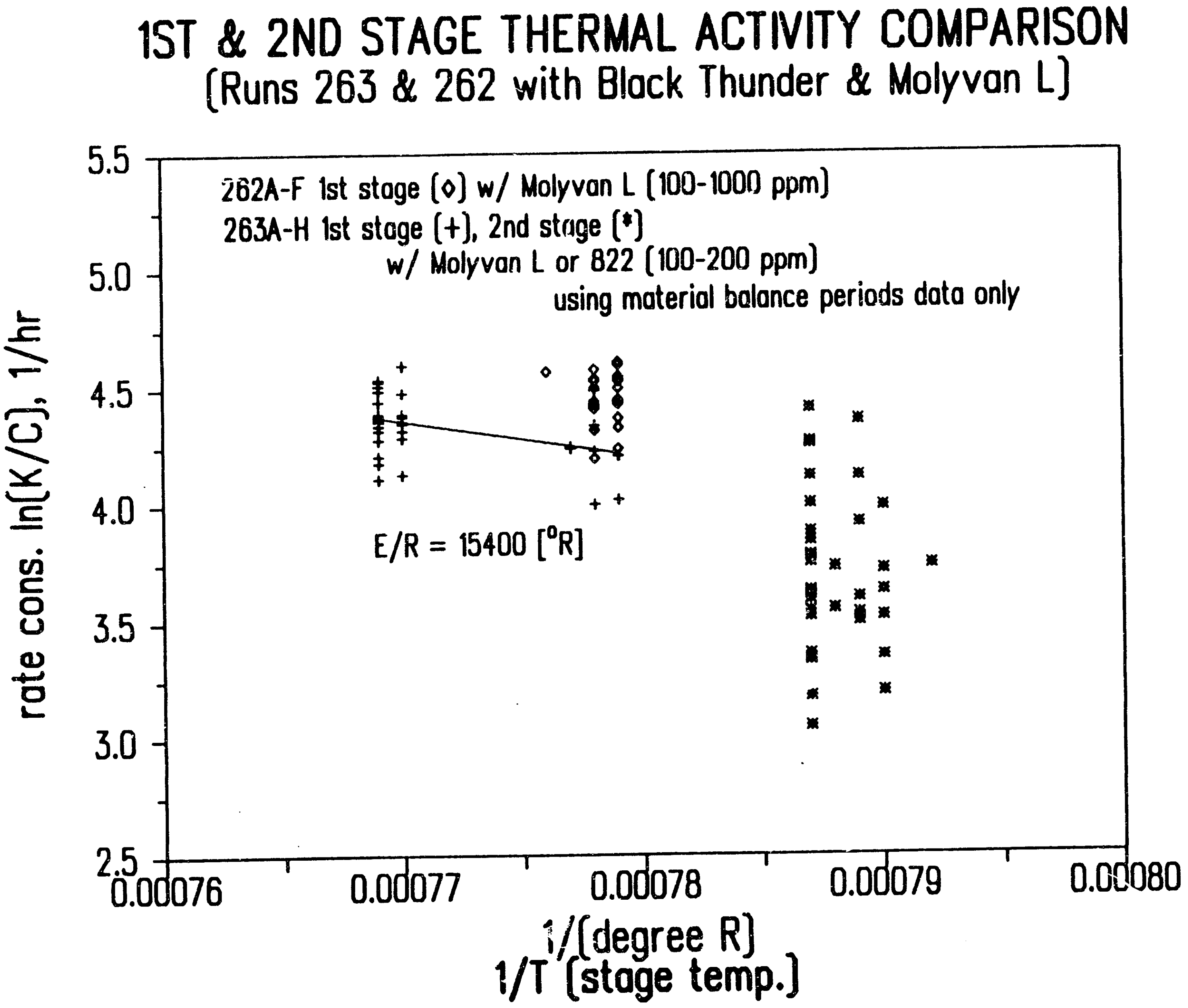


FIGURE 73. COMPARISON OF FIRST AND SECOND STAGE THERMAL ACTIVITY (263A-H)

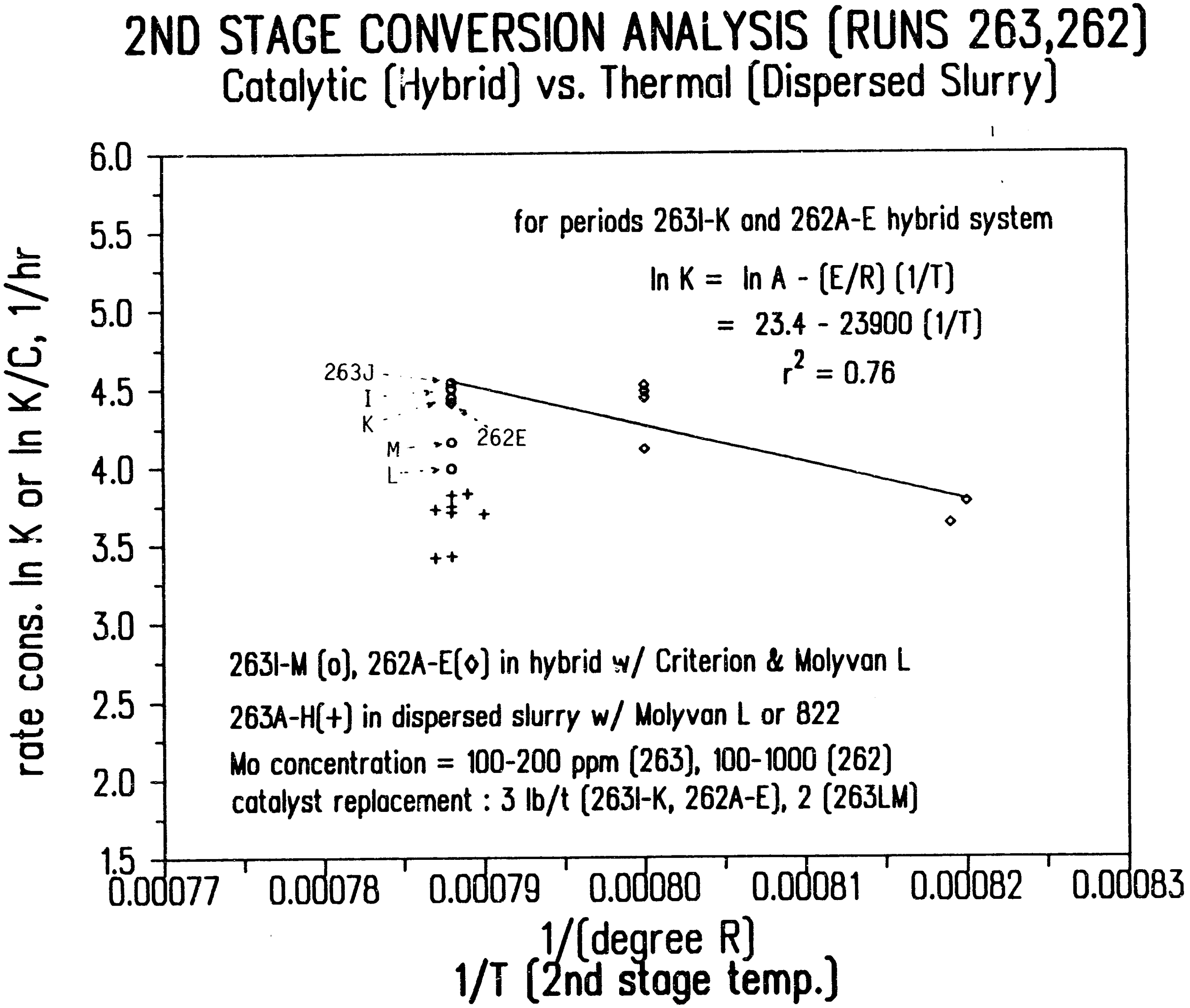


FIGURE 74. SECOND STAGE CATALYST ACTIVITY COMPARISON AND ARRHENIUS PLOT FOR CATALYTIC ACTIVATION ENERGY (RUNS 263A-M, 262A-E)

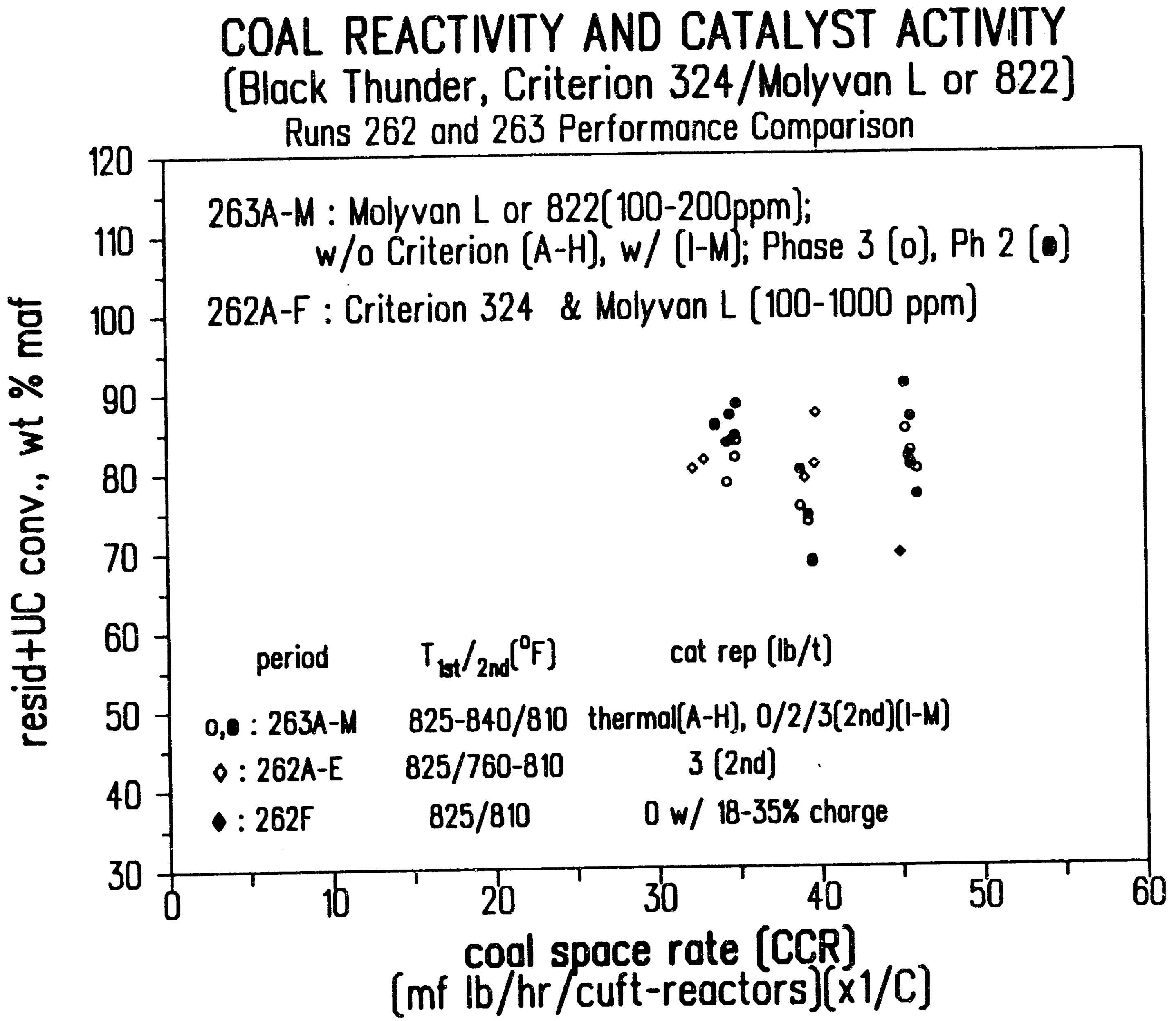


FIGURE 75. OVERALL TWO-STAGE RESID + UC CONVERSION COMPARISON IN LB MF COAL/HR/CU FT REACTORS (CCR) (x1/C) (RUNS 263, 262)

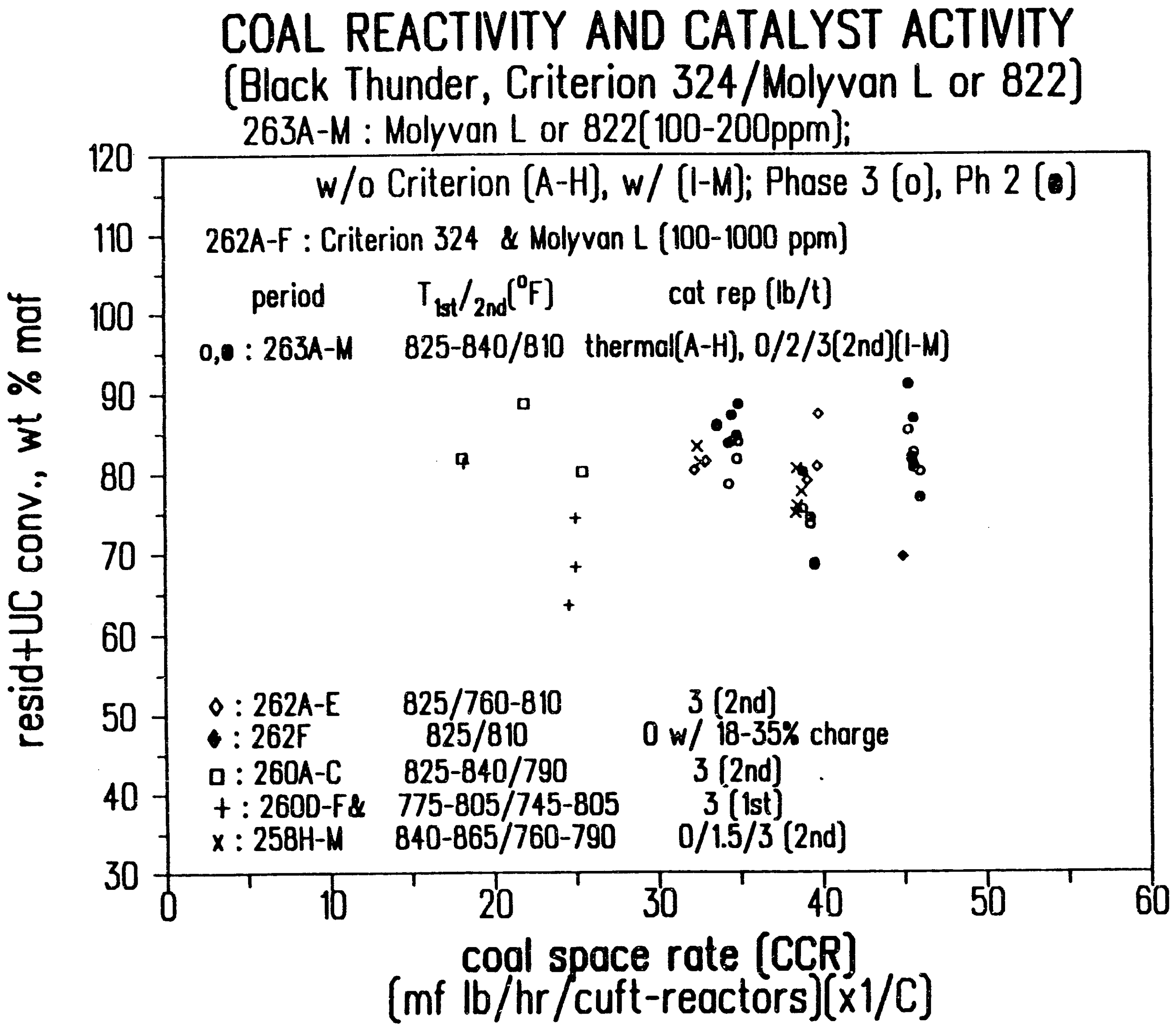


FIGURE 76. OVERALL TWO-STAGE RESID + UC CONVERSION COMPARISON IN LB MF COAL/HR/CU FT REACTORS (CCR) (x1/C) (RUNS 263, 262, 260, 258)

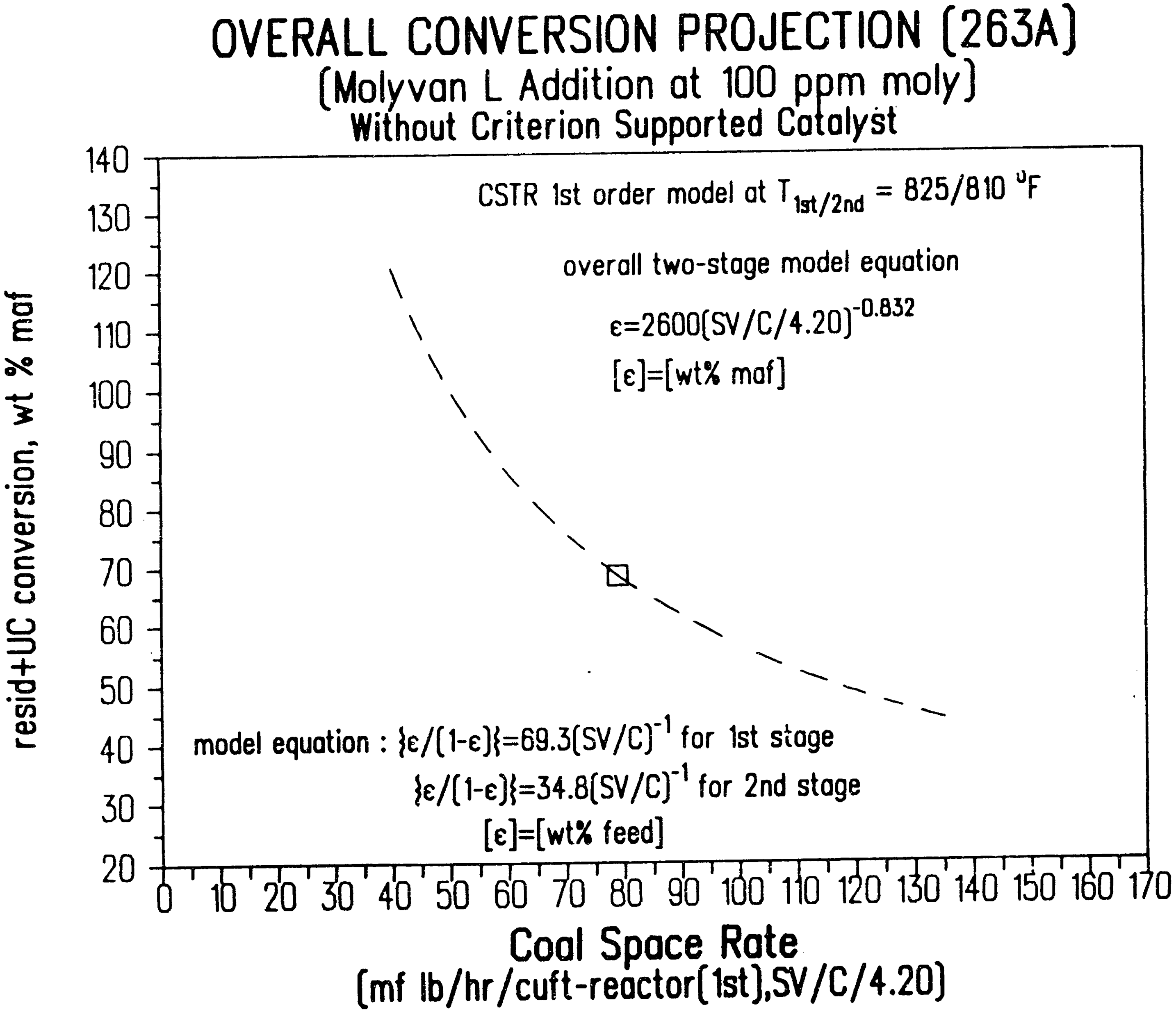


FIGURE 77. PROJECTION USING CSTR FIRST-ORDER RESID + UC CONVERSION MODEL (263A) IN LB MF COAL/HR/CU FT REACTOR (x1/C)

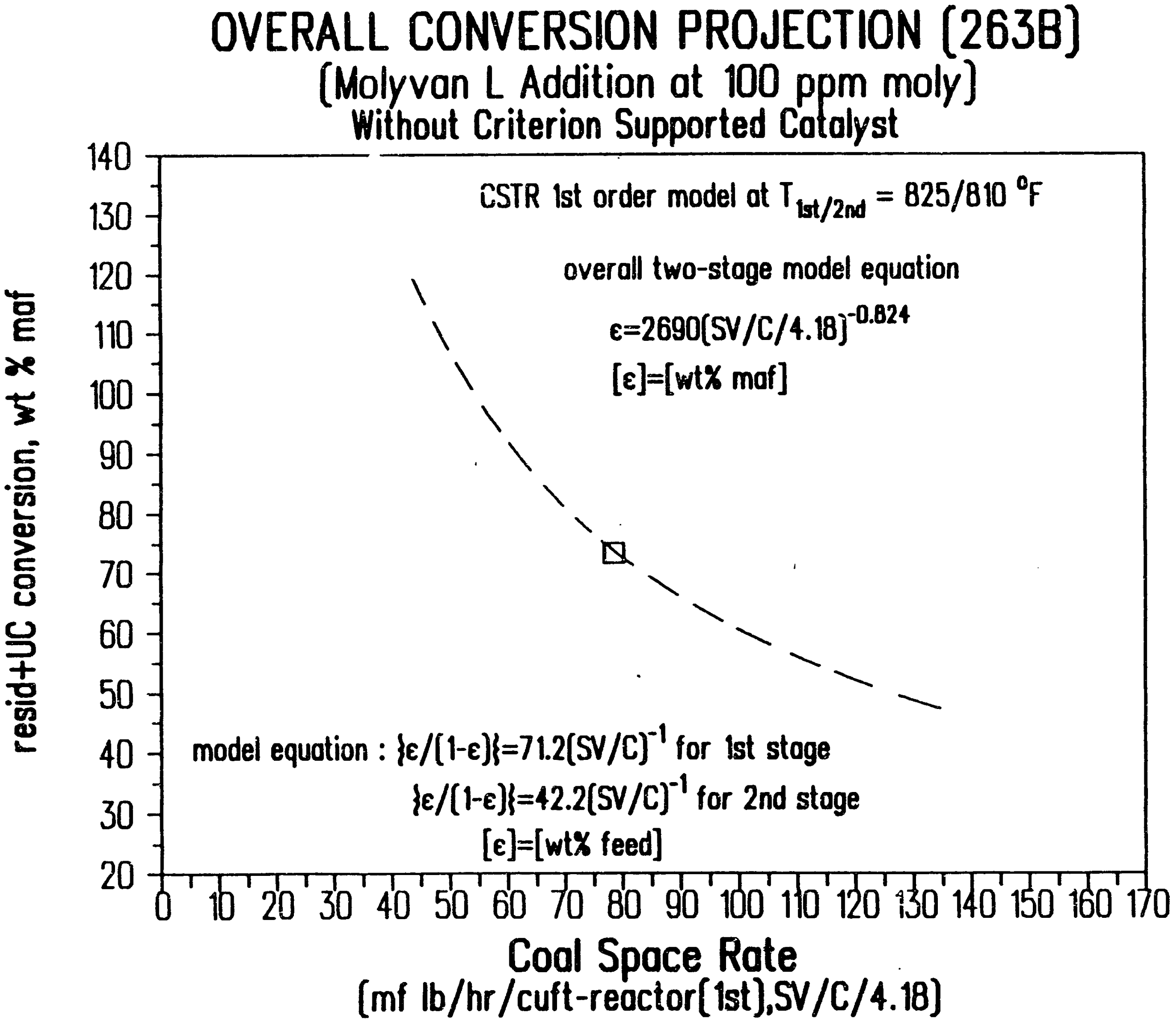


FIGURE 78. PROJECTION USING CSTR FIRST-ORDER RESID + UC CONVERSION MODEL (263B) IN LB MF COAL/HR/CU FT REACTOR (x1/C)

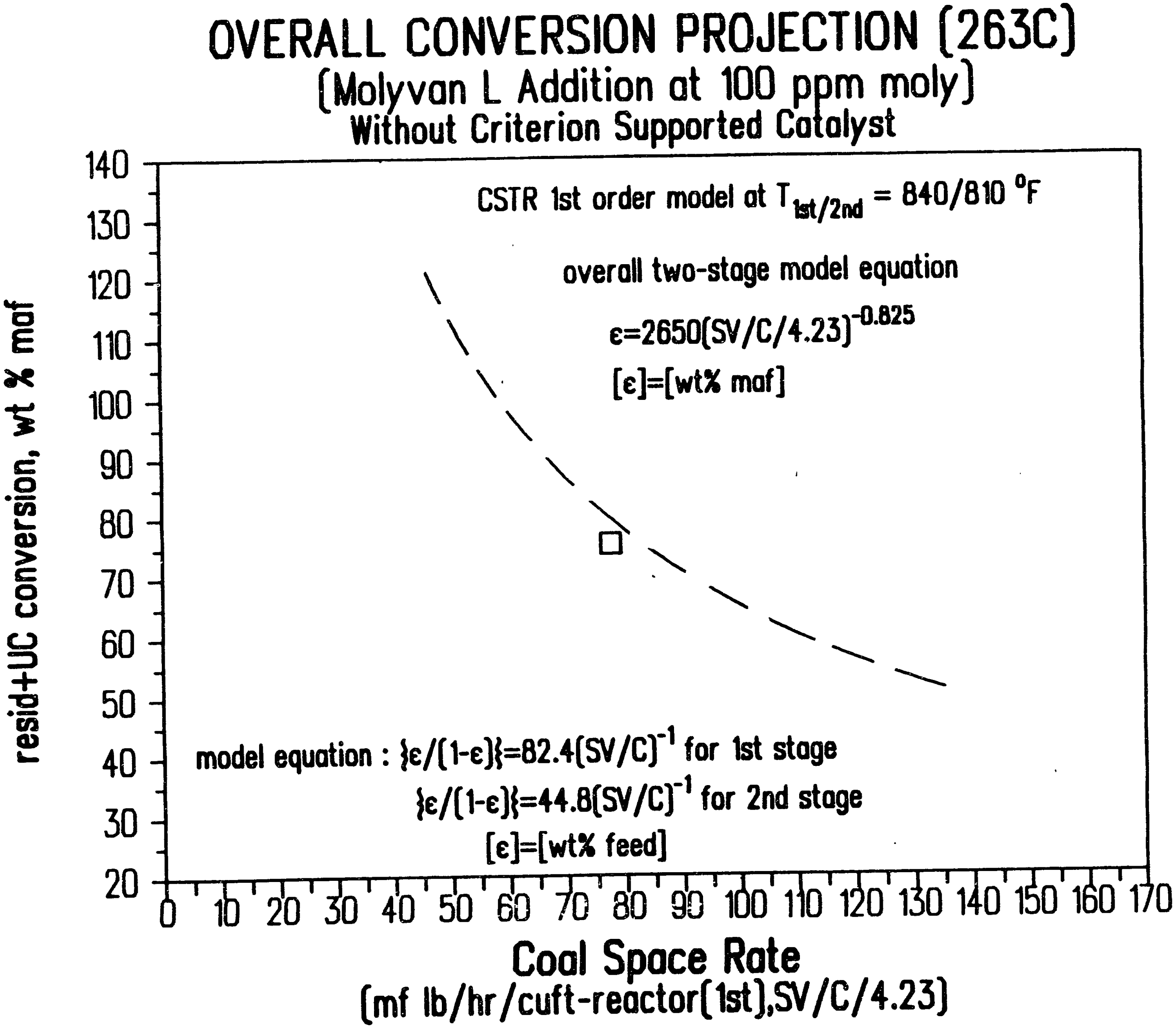


FIGURE 79. PROJECTION USING CSTR FIRST-ORDER RESID + UC CONVERSION MODEL (263C) IN LB MF COAL/HR/CU FT REACTOR (x1/C)

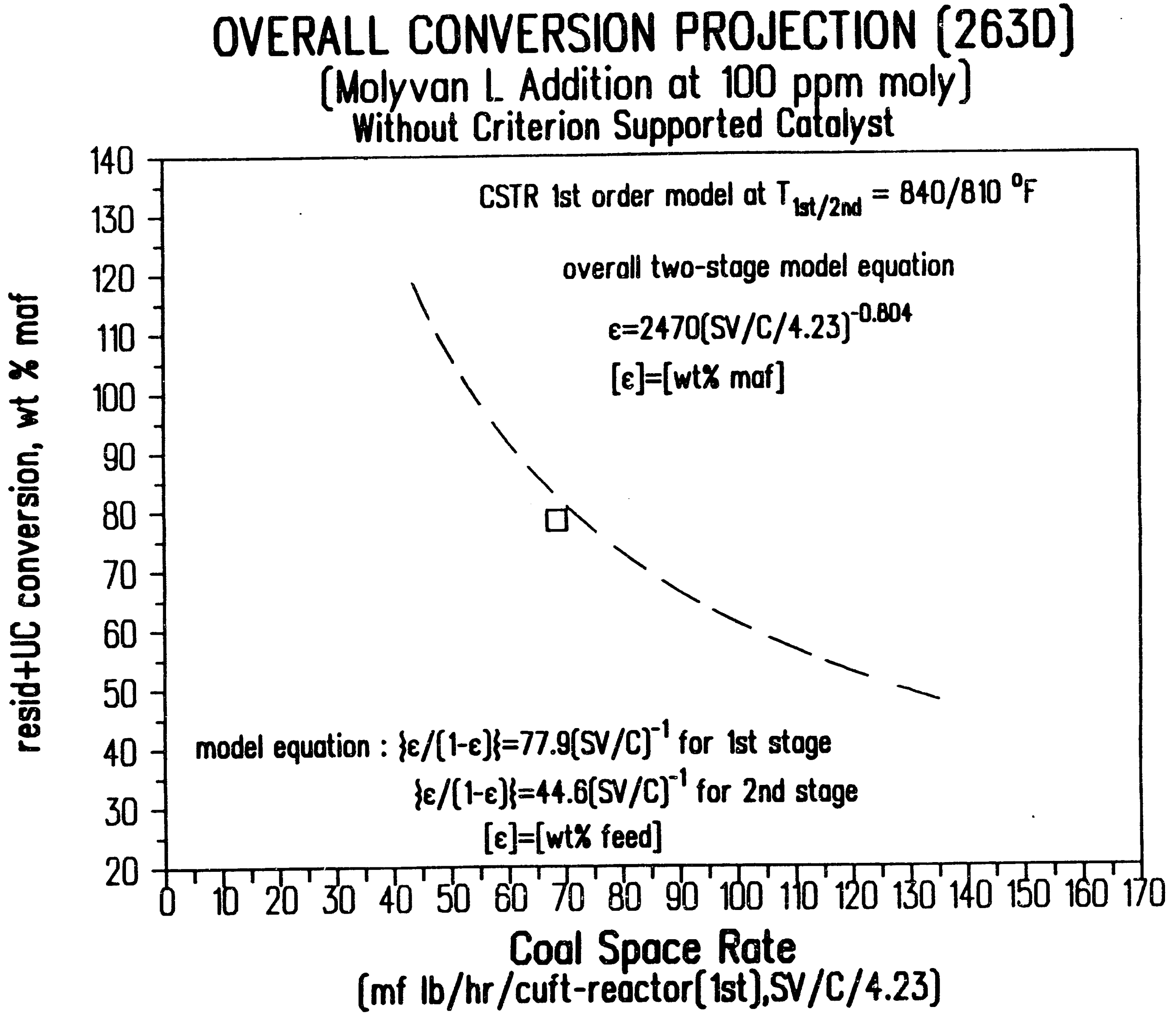


FIGURE 80. PROJECTION USING CSTR FIRST-ORDER RESID + UC CONVERSION MODEL (263D) IN LB MF COAL/HR/CU FT REACTOR (x1/C)

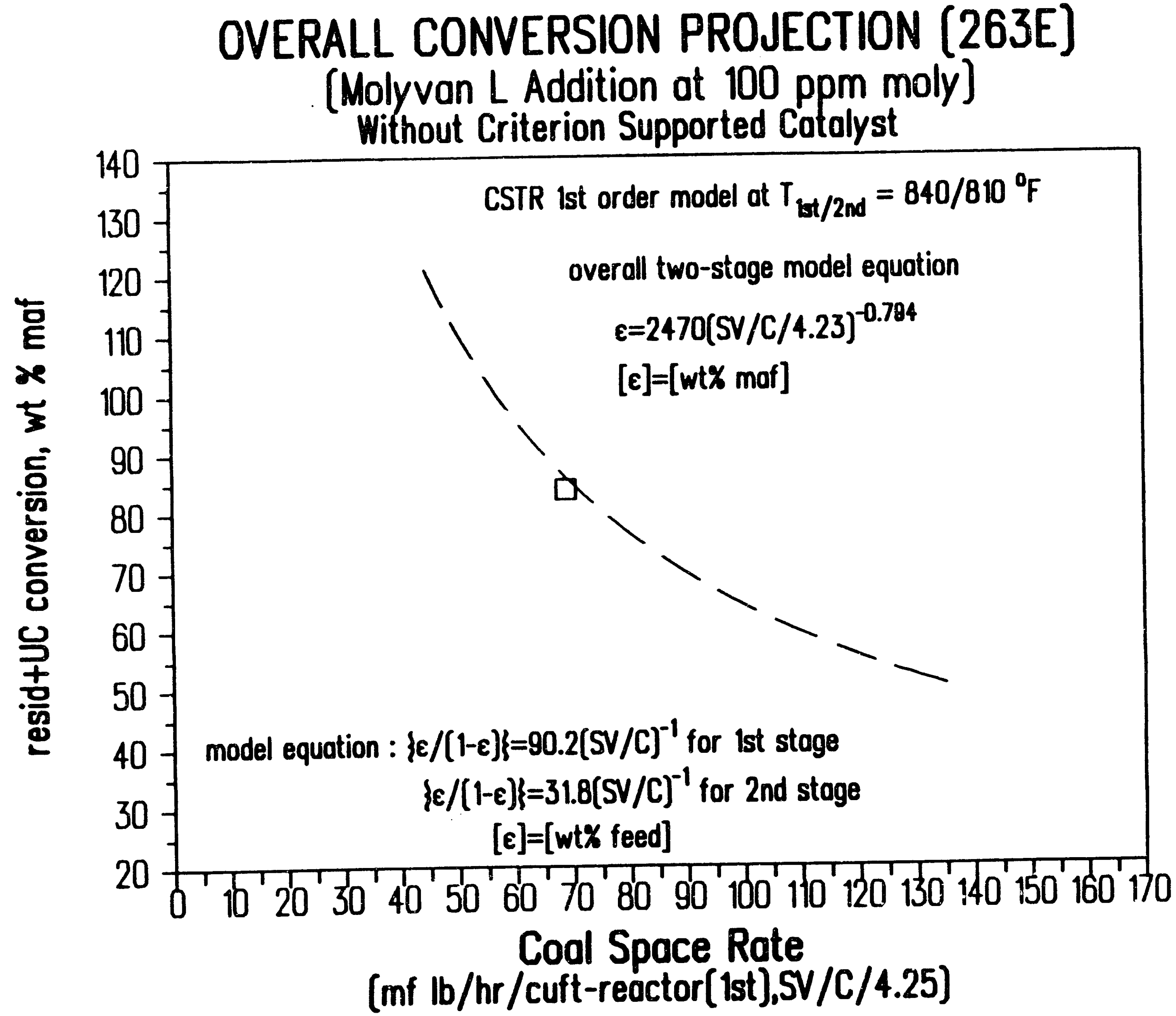


FIGURE 81. PROJECTION USING CSTR FIRST-ORDER RESID + UC CONVERSION MODEL (263E) IN LB MF COAL/HR/CU FT REACTOR (x1/C)

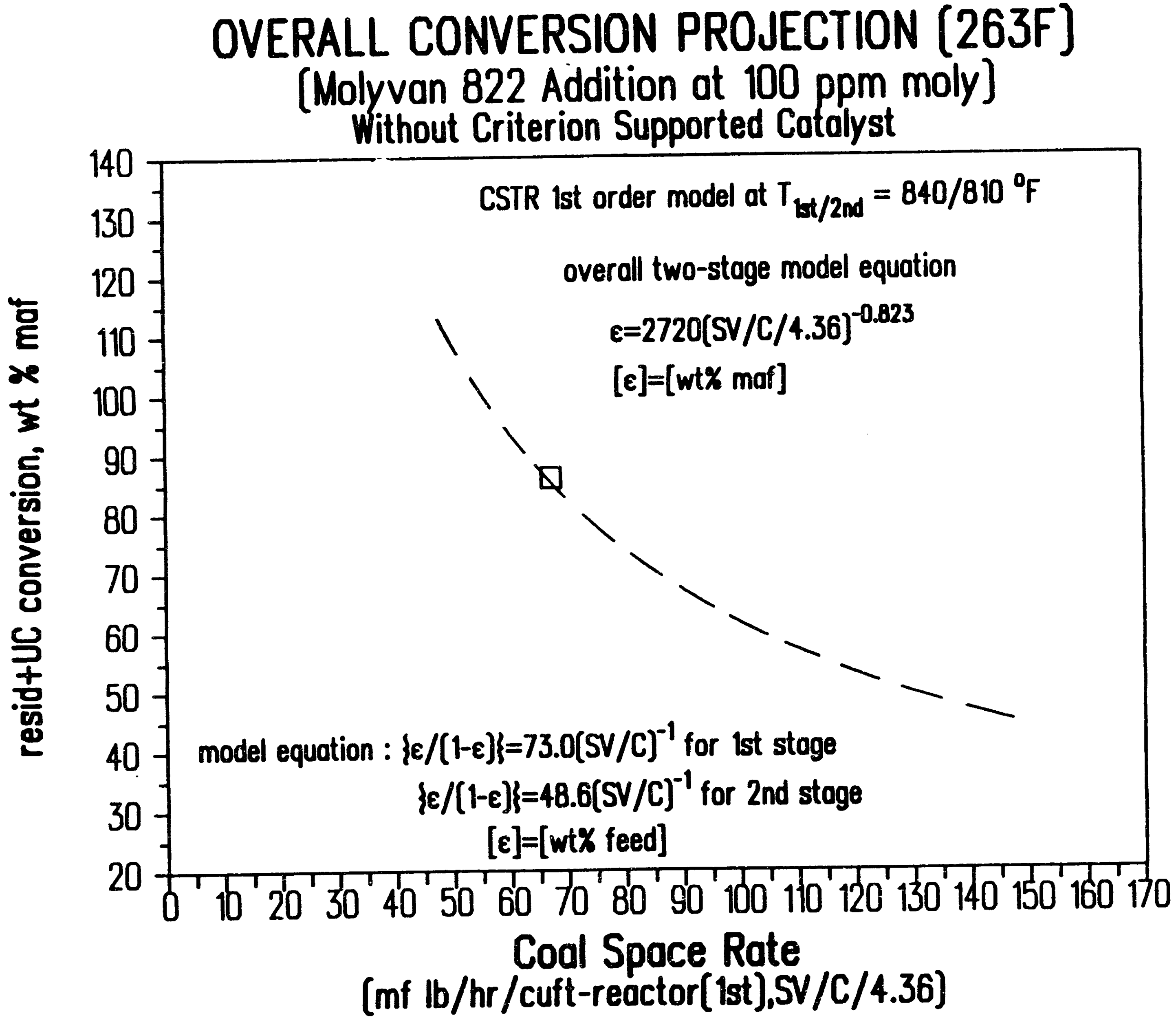


FIGURE 82. PROJECTION USING CSTR FIRST-ORDER RESID + UC CONVERSION MODEL (263F) IN LB MF COAL/HR/CU FT REACTOR (x1/C)

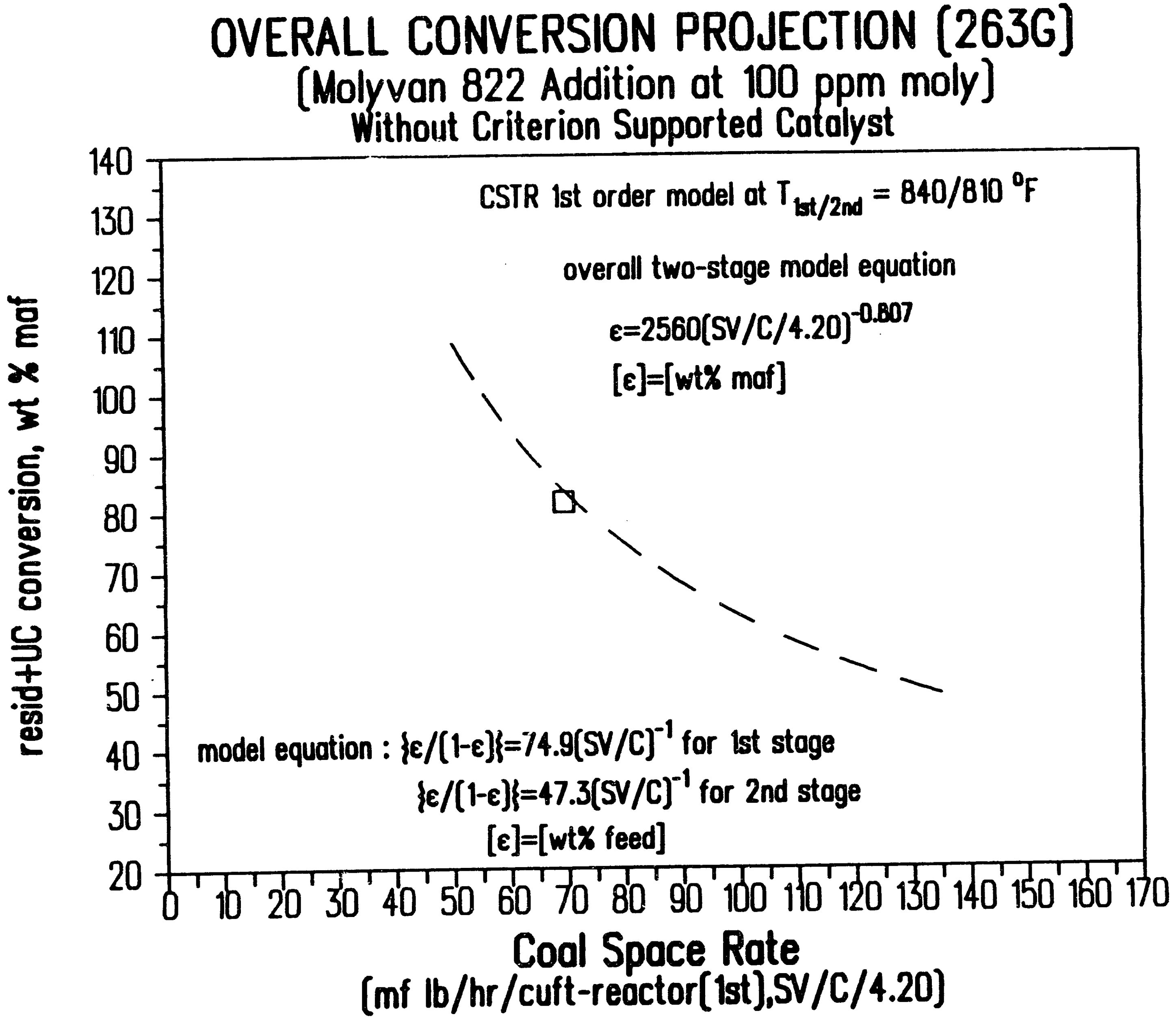


FIGURE 83. PROJECTION USING CSTR FIRST-ORDER RESID + UC CONVERSION MODEL (263G) IN LB MF COAL/HR/CU FT REACTOR (x1/C)

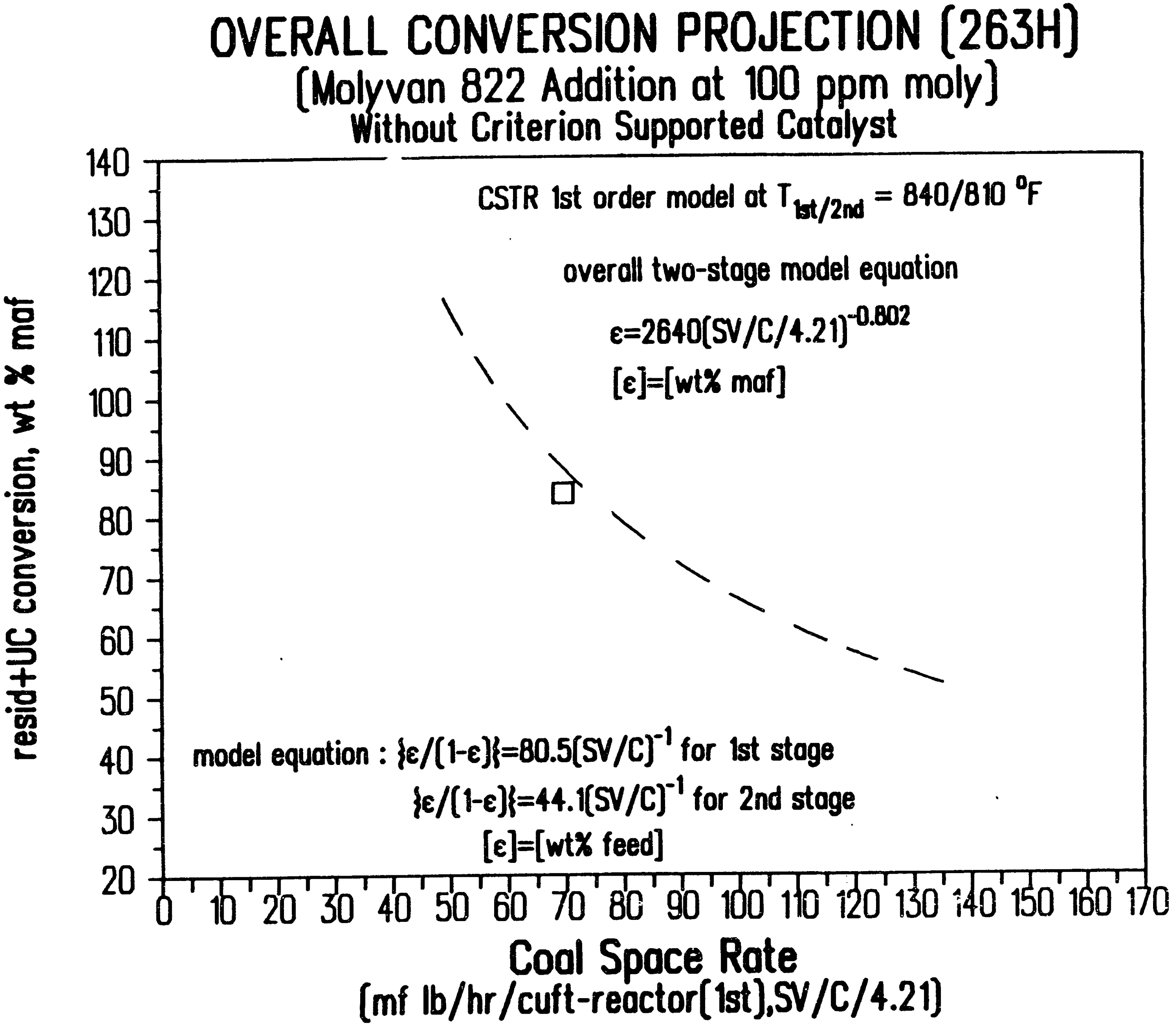


FIGURE 84. PROJECTION USING CSTR FIRST-ORDER RESID + UC CONVERSION MODEL (263H) IN LB MF COAL/HR/CU FT REACTOR (x1/C)

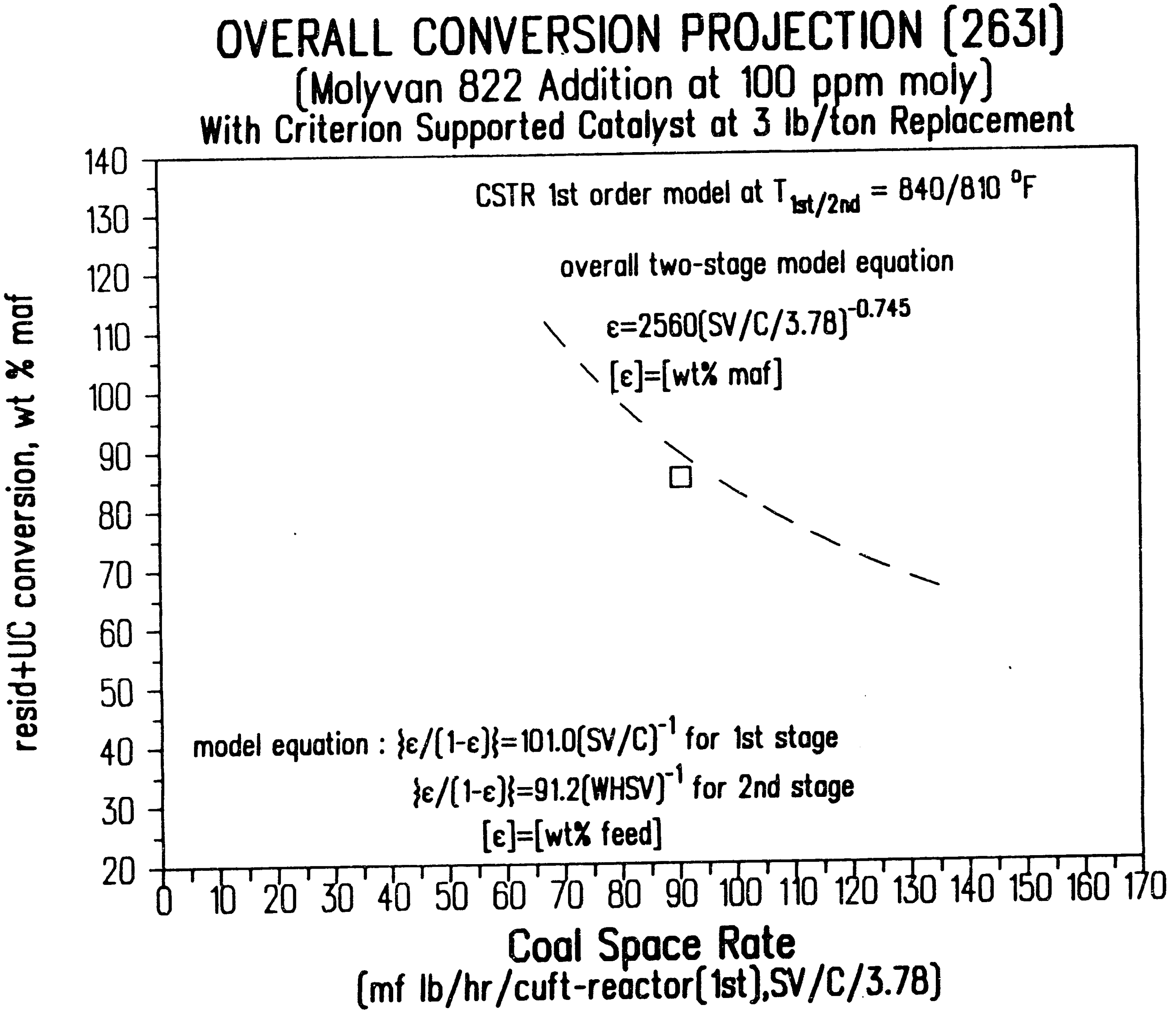


FIGURE 85. PROJECTION USING CSTR FIRST-ORDER RESID + UC CONVERSION MODEL (263I) IN LB MF COAL/HR/CU FT REACTOR (x1/C)

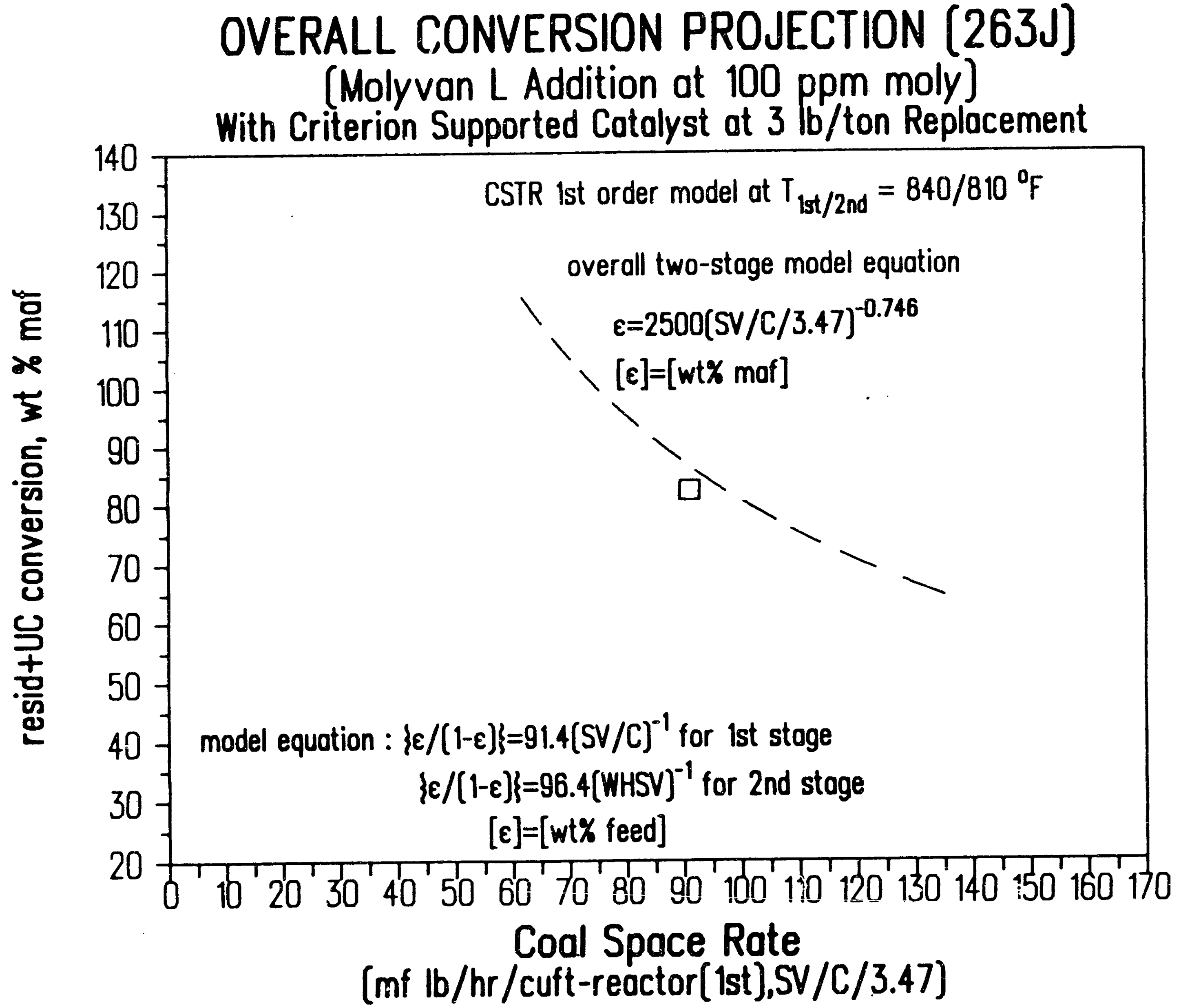


FIGURE 86. PROJECTION USING CSTR FIRST-ORDER RESID + UC CONVERSION MODEL (263J) IN LB MF COAL/HR/CU FT REACTOR (x1/C)

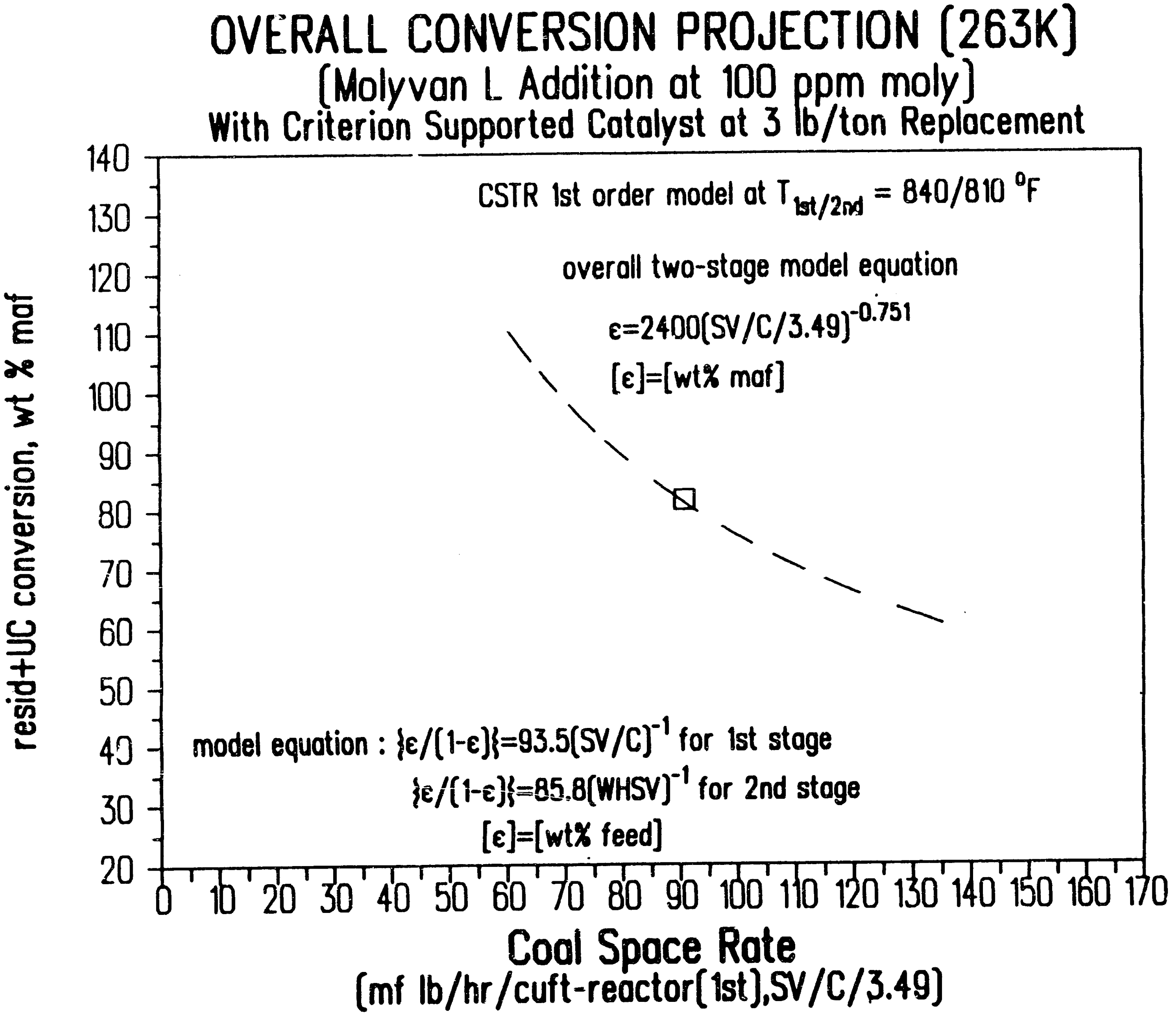


FIGURE 87. PROJECTION USING CSTR FIRST-ORDER RESID + UC CONVERSION MODEL (263K) IN LB MF COAL/HR/CU FT REACTOR (x1/C)

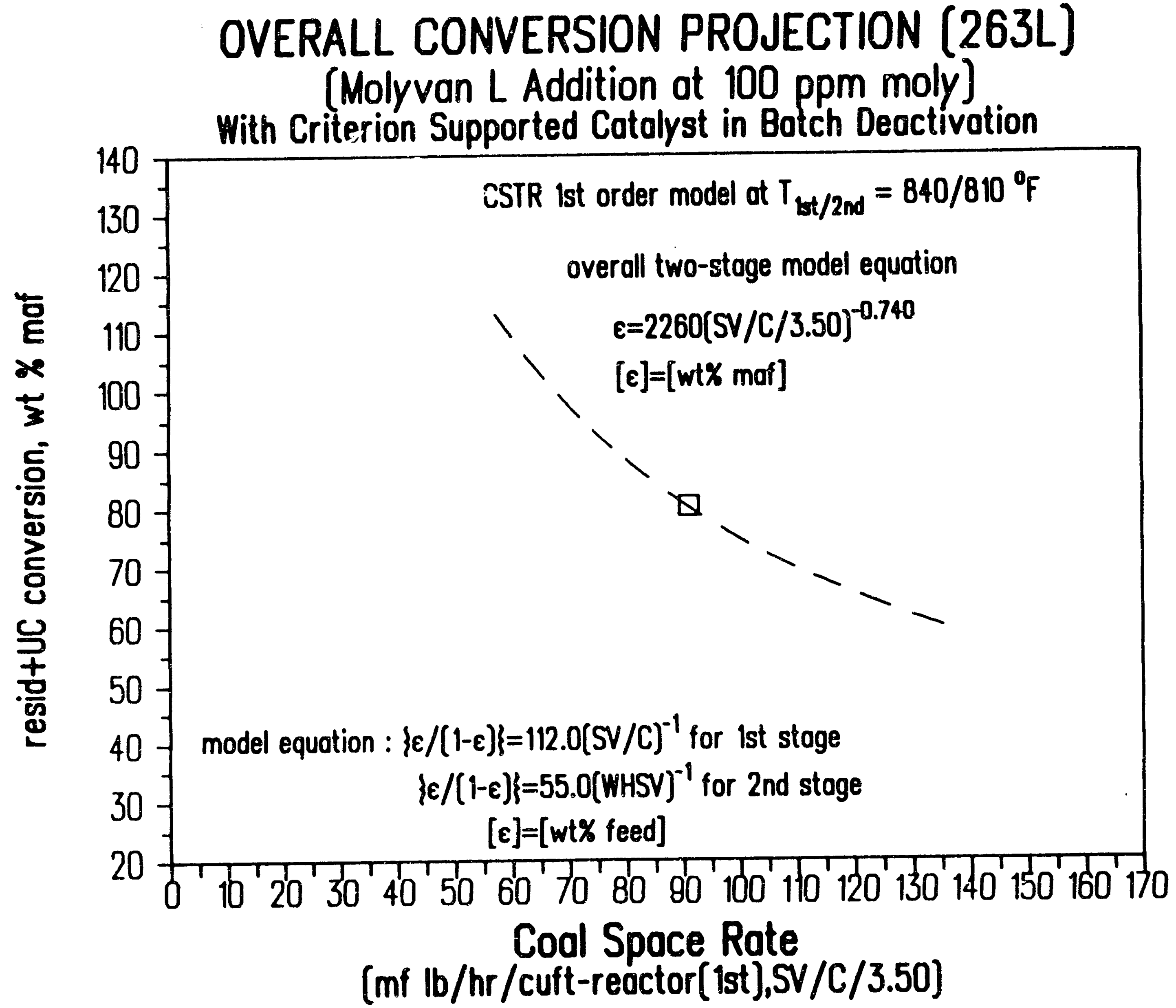


FIGURE 88. PROJECTION USING CSTR FIRST-ORDER RESID + UC CONVERSION MODEL (263L) IN LB MF COAL/HR/CU FT REACTOR (x1/C)

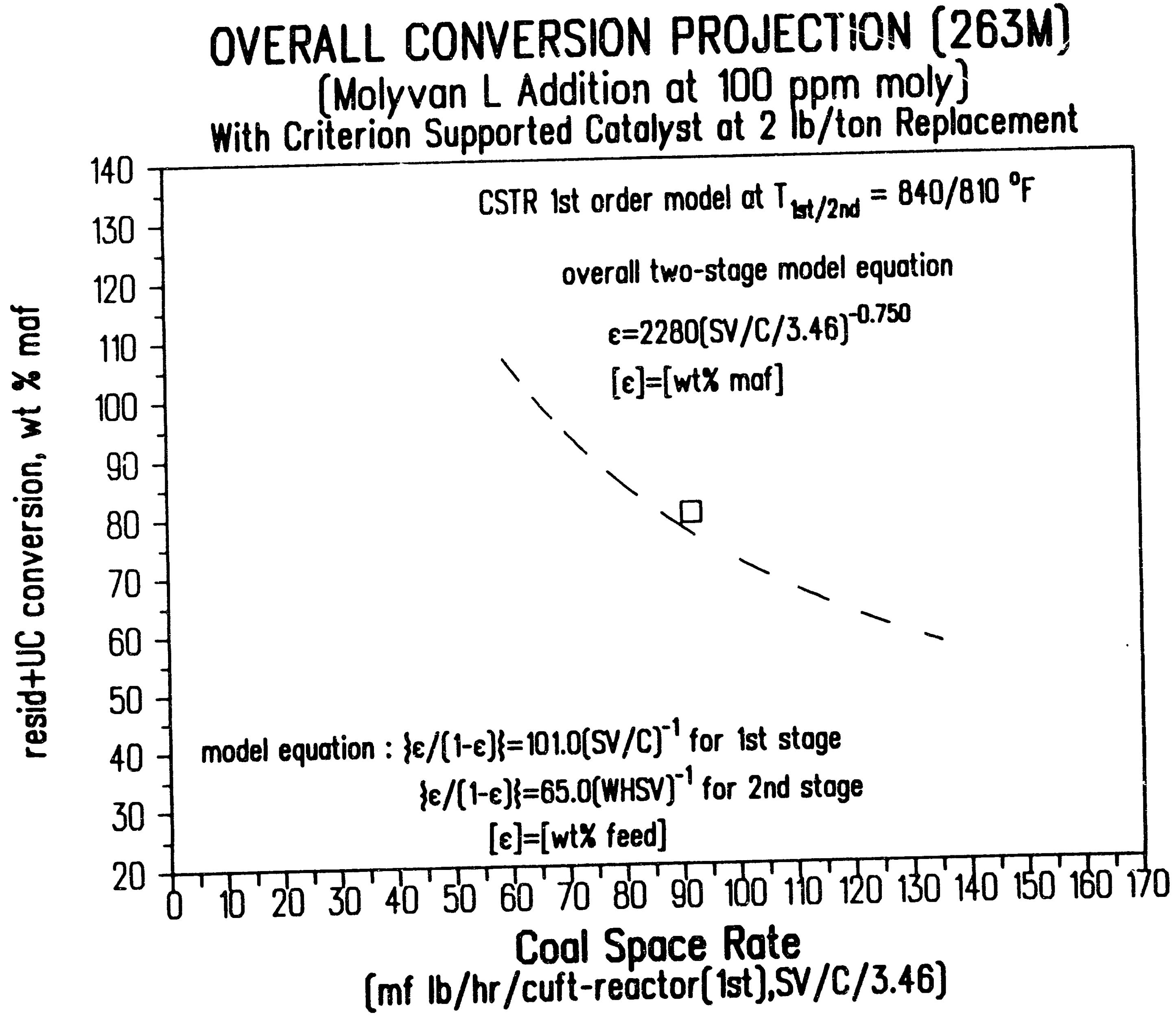


FIGURE 89. PROJECTION USING CSTR FIRST-ORDER RESID + UC CONVERSION MODEL (263M) IN LB MF COAL/HR/CU FT REACTOR (x1/C)

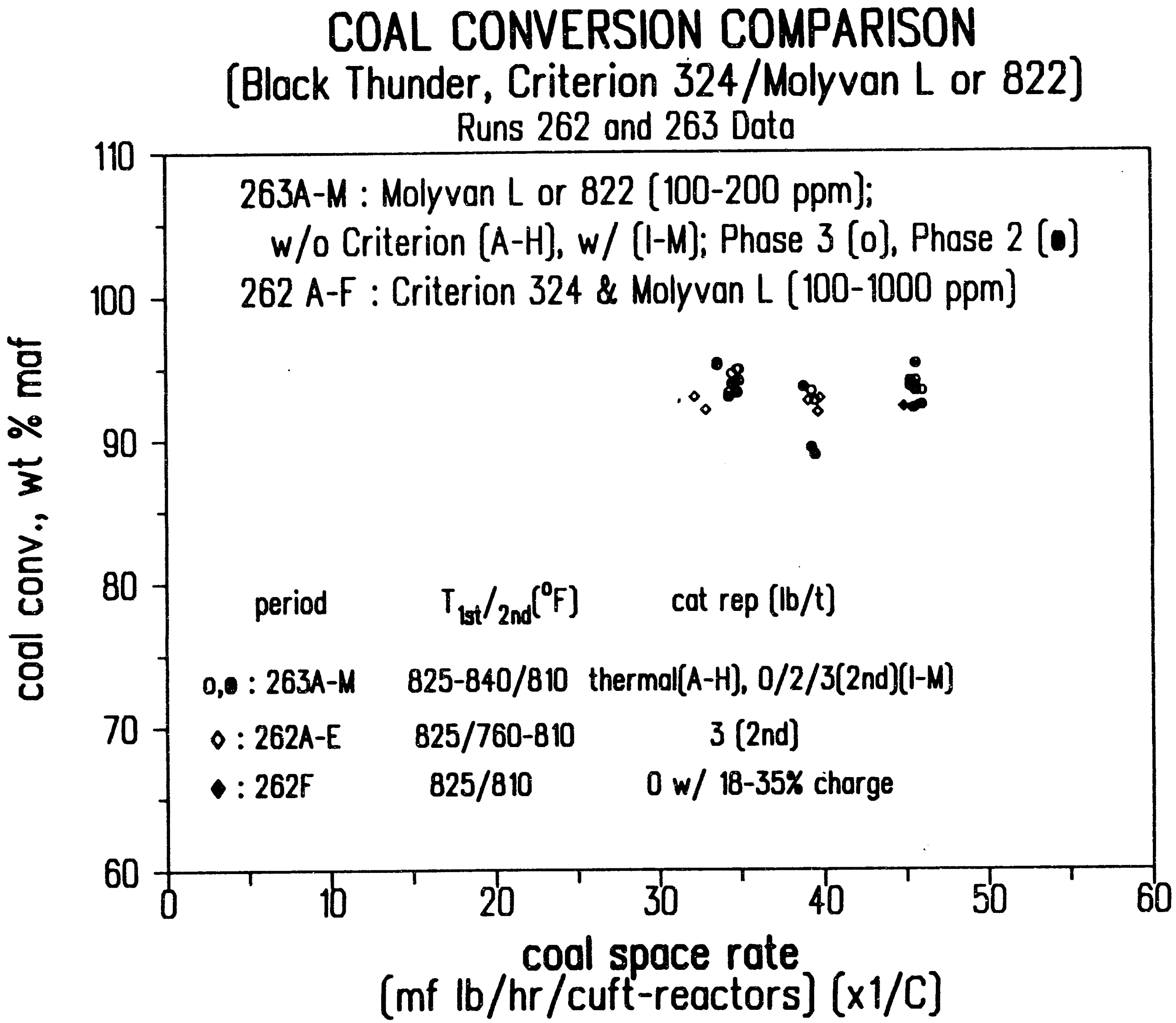


FIGURE 90. COAL CONVERSION COMPARISON IN LB MF COAL/HR/CU FT REACTORS (CCR) (x1/C)

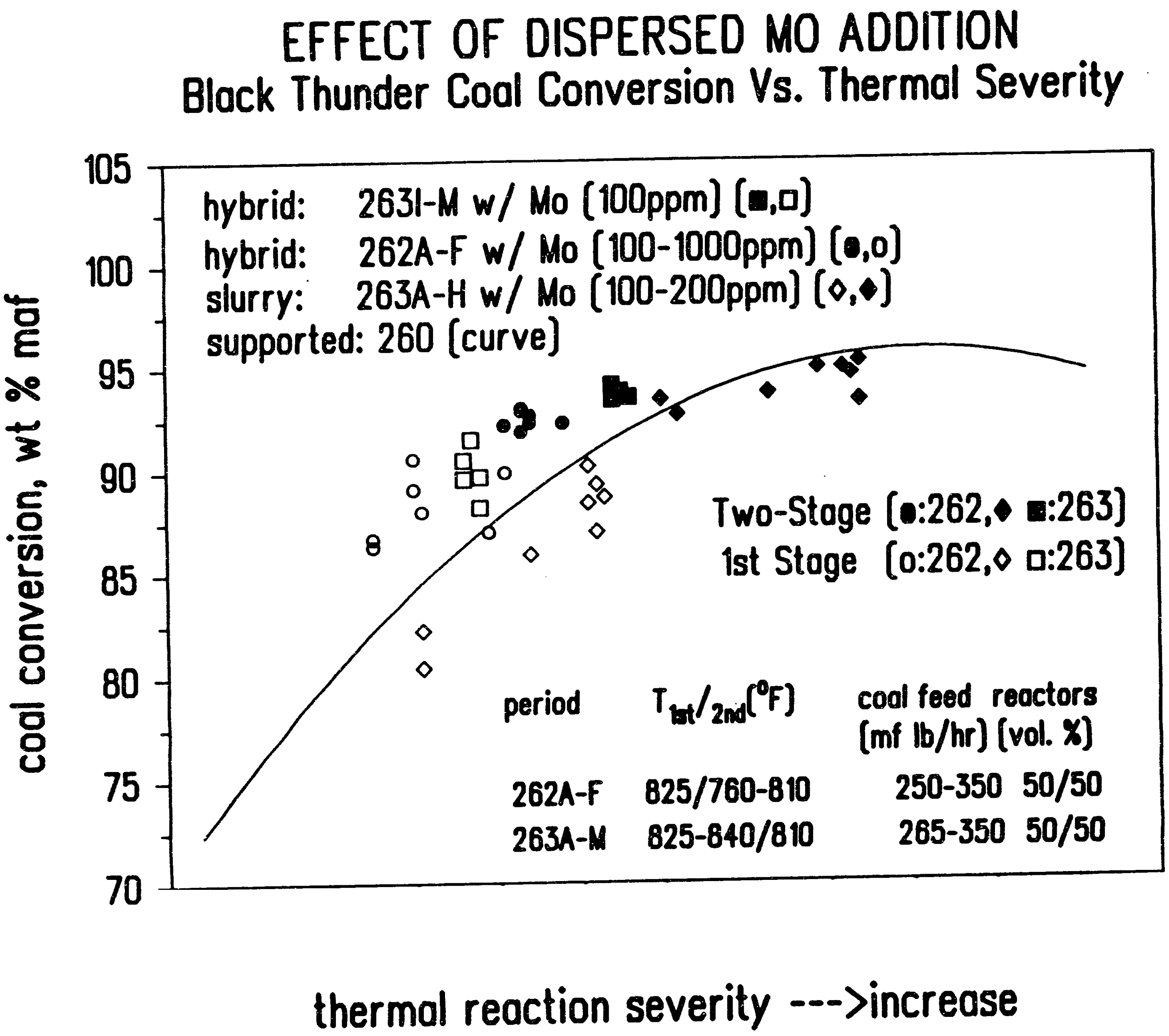


FIGURE 91. EFFECT OF THERMAL REACTION SEVERITY ON COAL CONVERSION

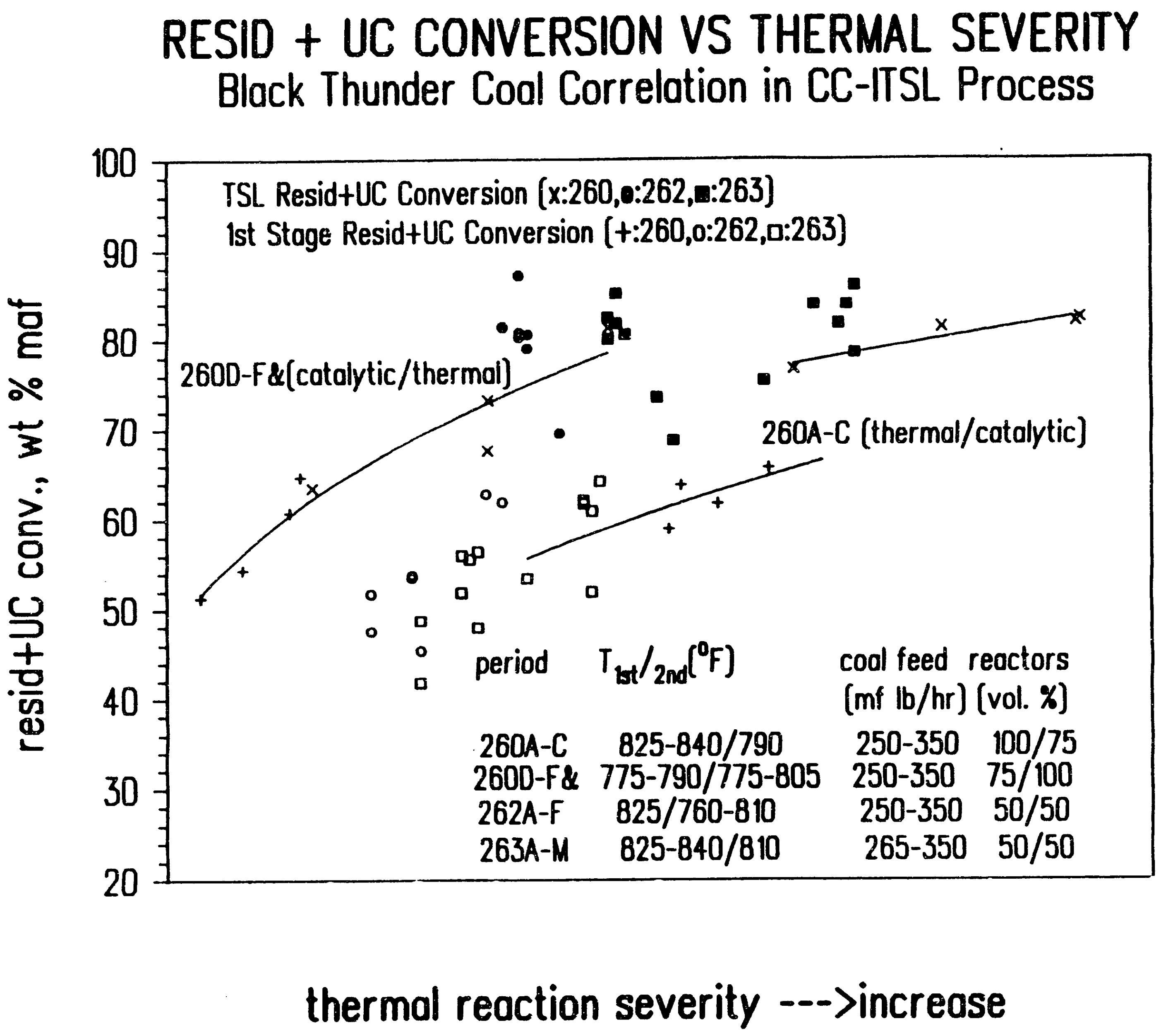


FIGURE 92. EFFECT OF THERMAL REACTION SEVERITY ON RESID + UC CONVERSION

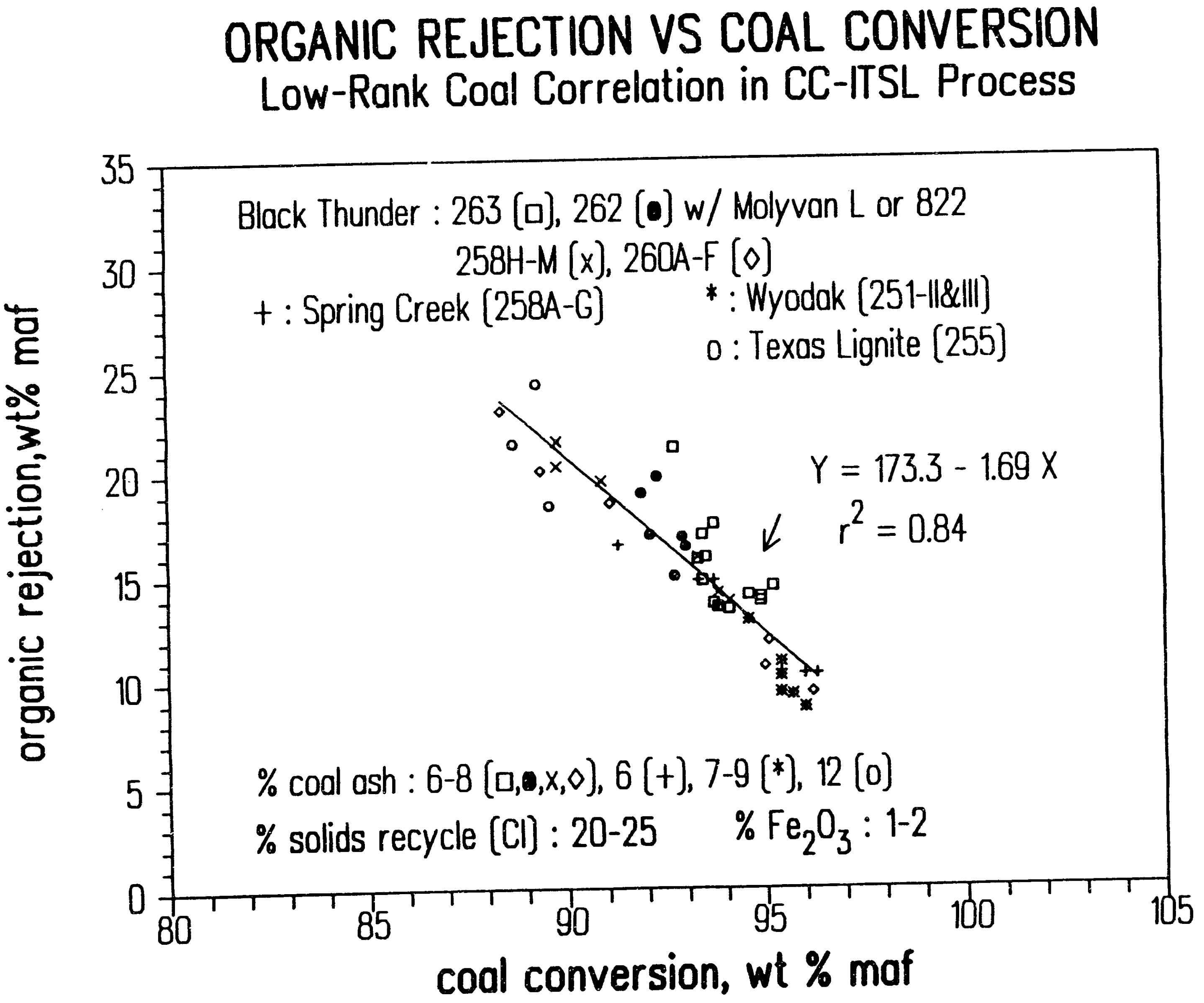
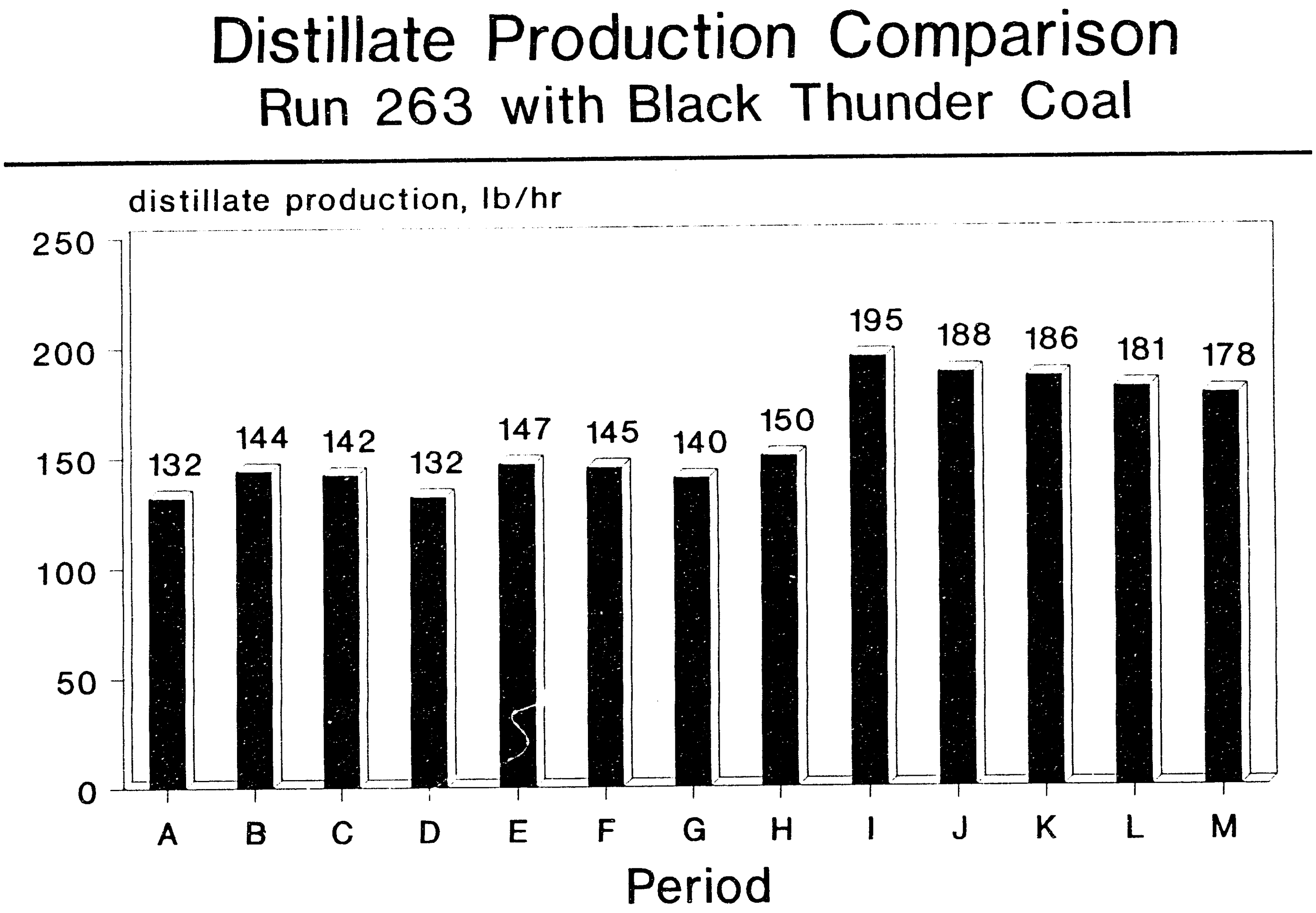


FIGURE 93. LINEAR CORRELATION BETWEEN ORGANIC REJECTION AND COAL CONVERSION IN LOW RANK COALS

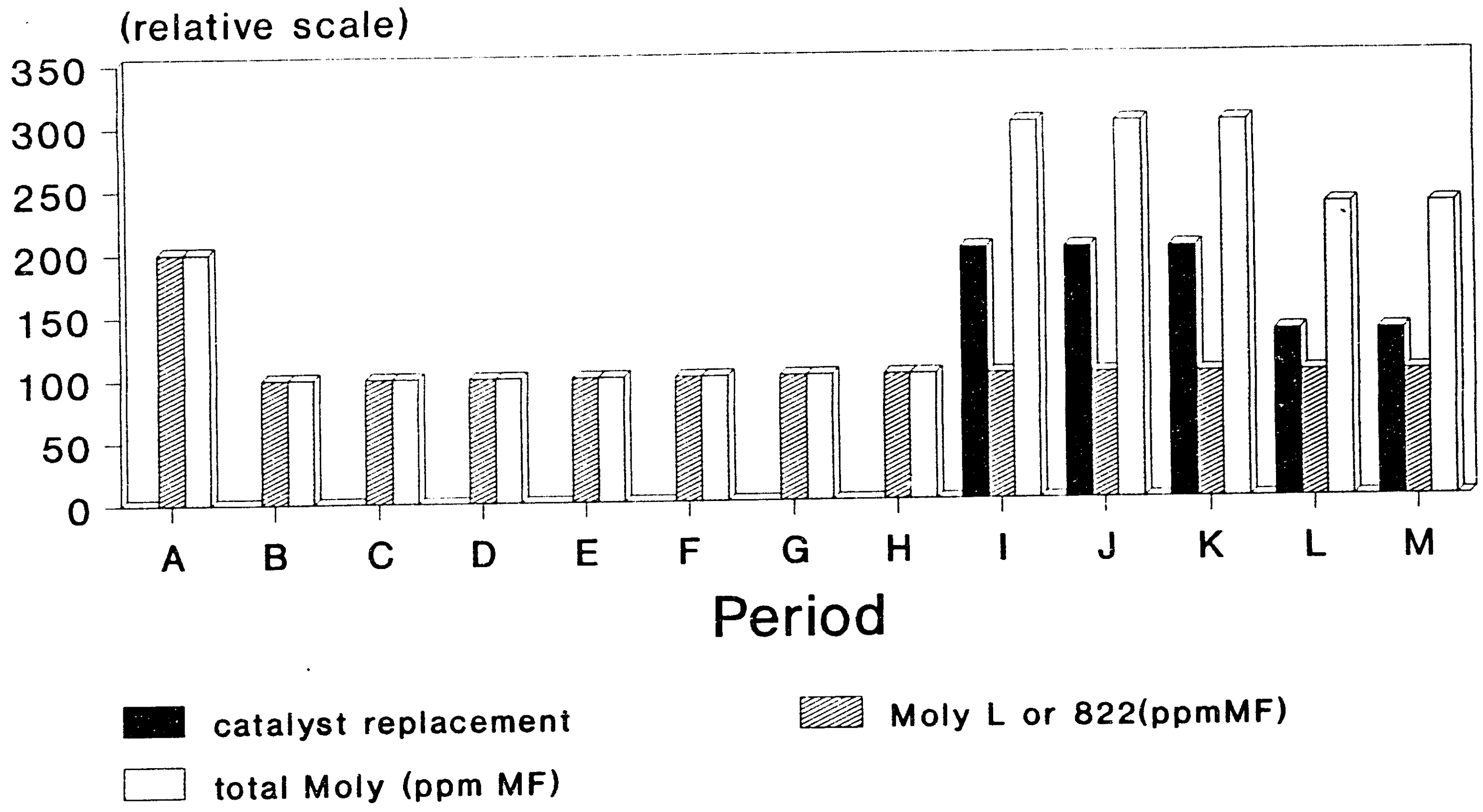


half-volume reactor operation; 263A-H
(slurry), 263I-M (hybrid); periods A-E,
J-M w/ Molyvan L; F-I w/ Molyvan 822

FIGURE 94. DISTILLATE PRODUCTION WITH BLACK THUNDER COAL (RUN 263-A-M)

Catalyst & Molybdenum Requirement

Run 263 with Black Thunder Coal



supported catalyst replacement -
100 = 1.5 lb/t; Moly L - Molyvan L;
total Moly - total molybdenum

FIGURE 95. CATALYST AND MOLYBDENUM REQUIREMENT WITH BLACK THUNDER COAL (RUN 263A-M)

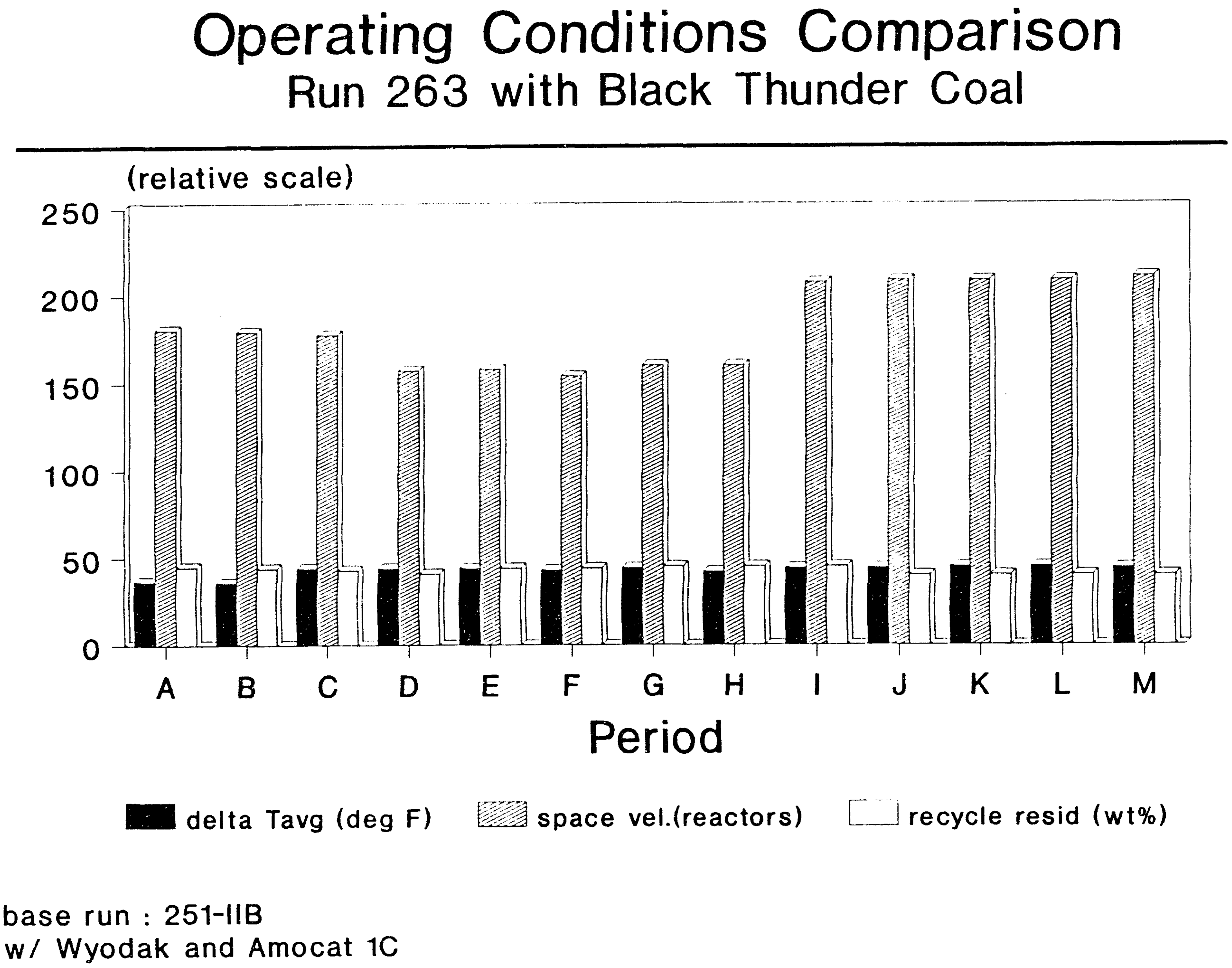
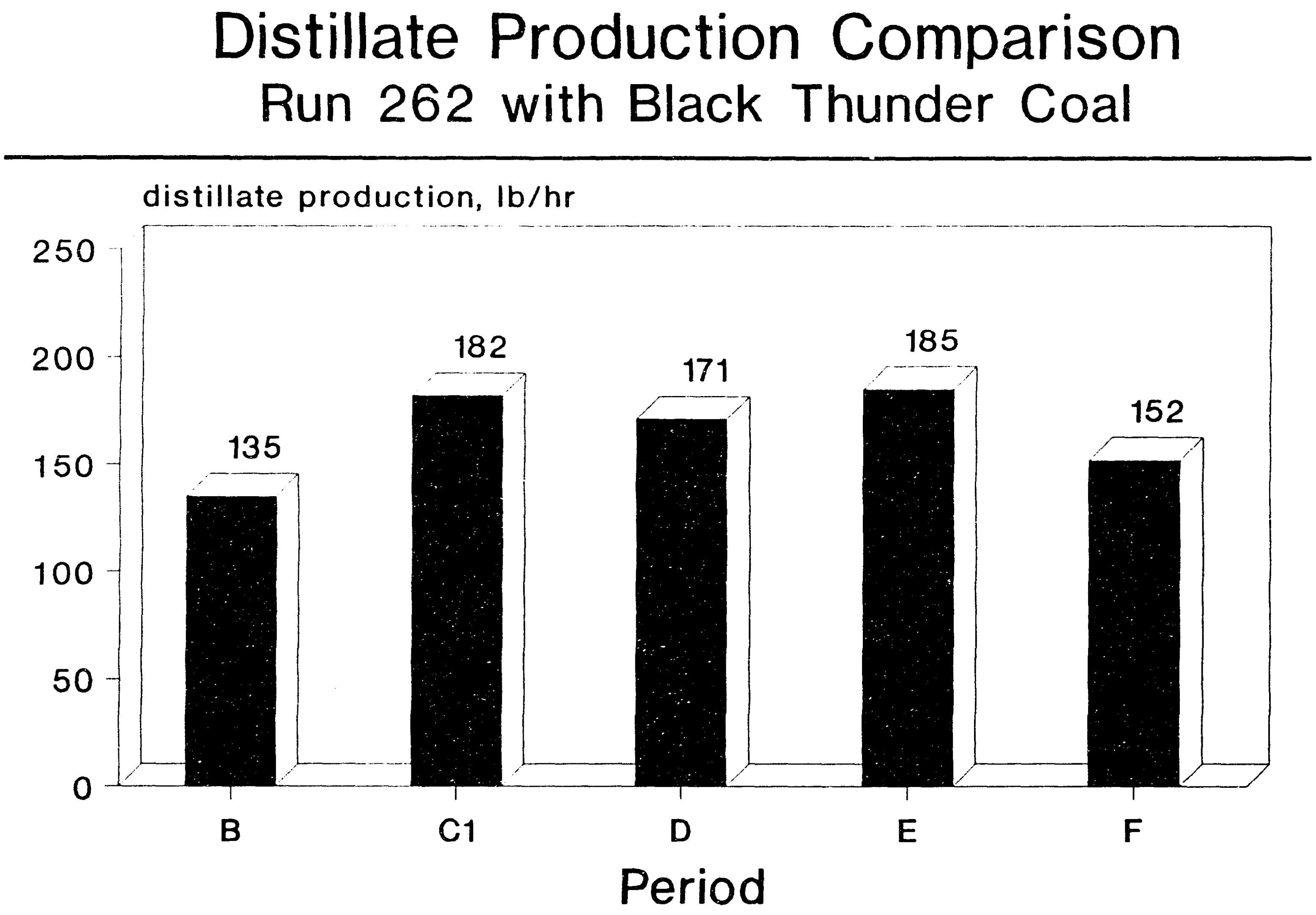
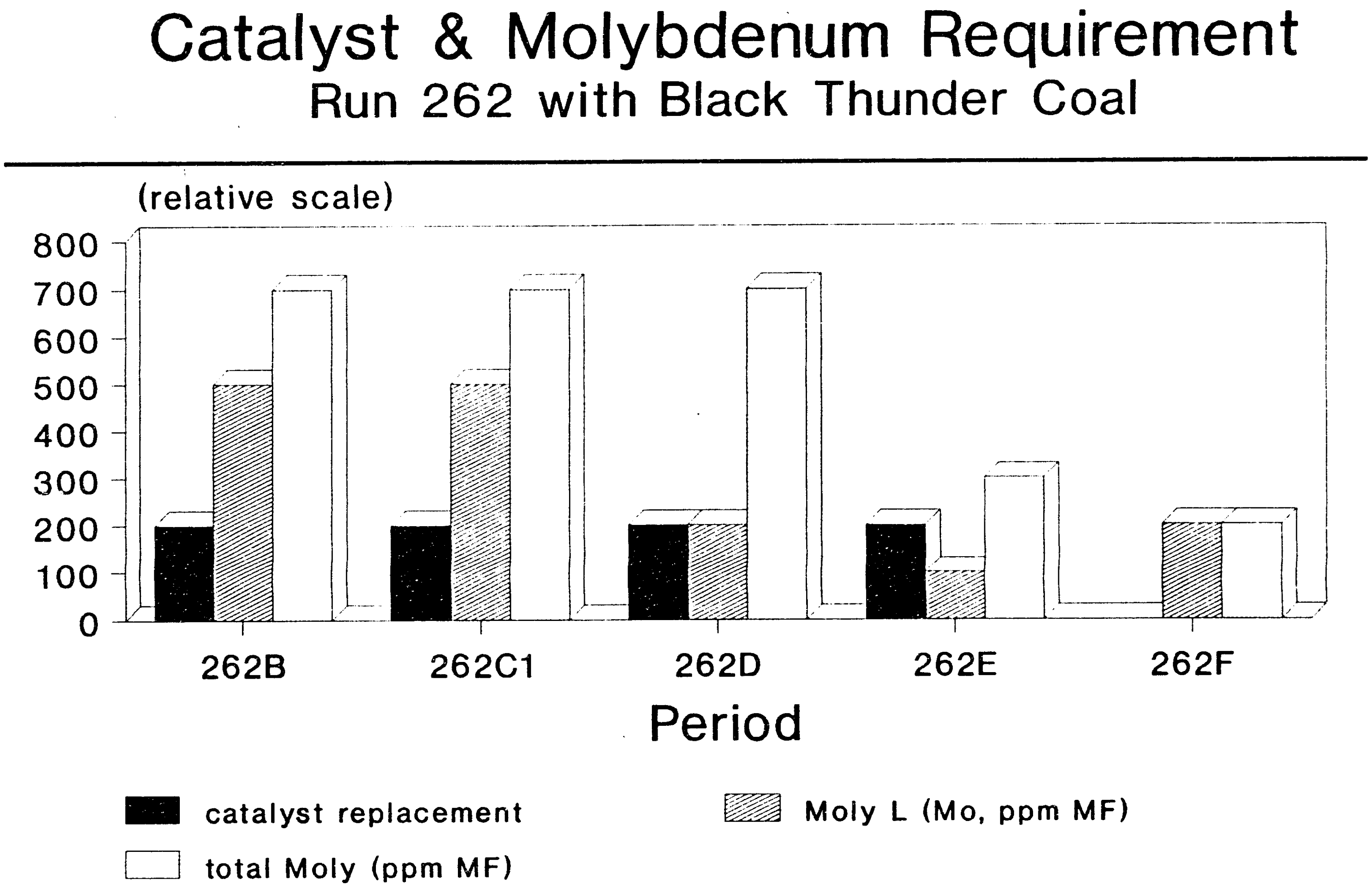


FIGURE 96. OPERATING CONDITIONS WITH BLACK THUNDER COAL (RUN 263A-M)



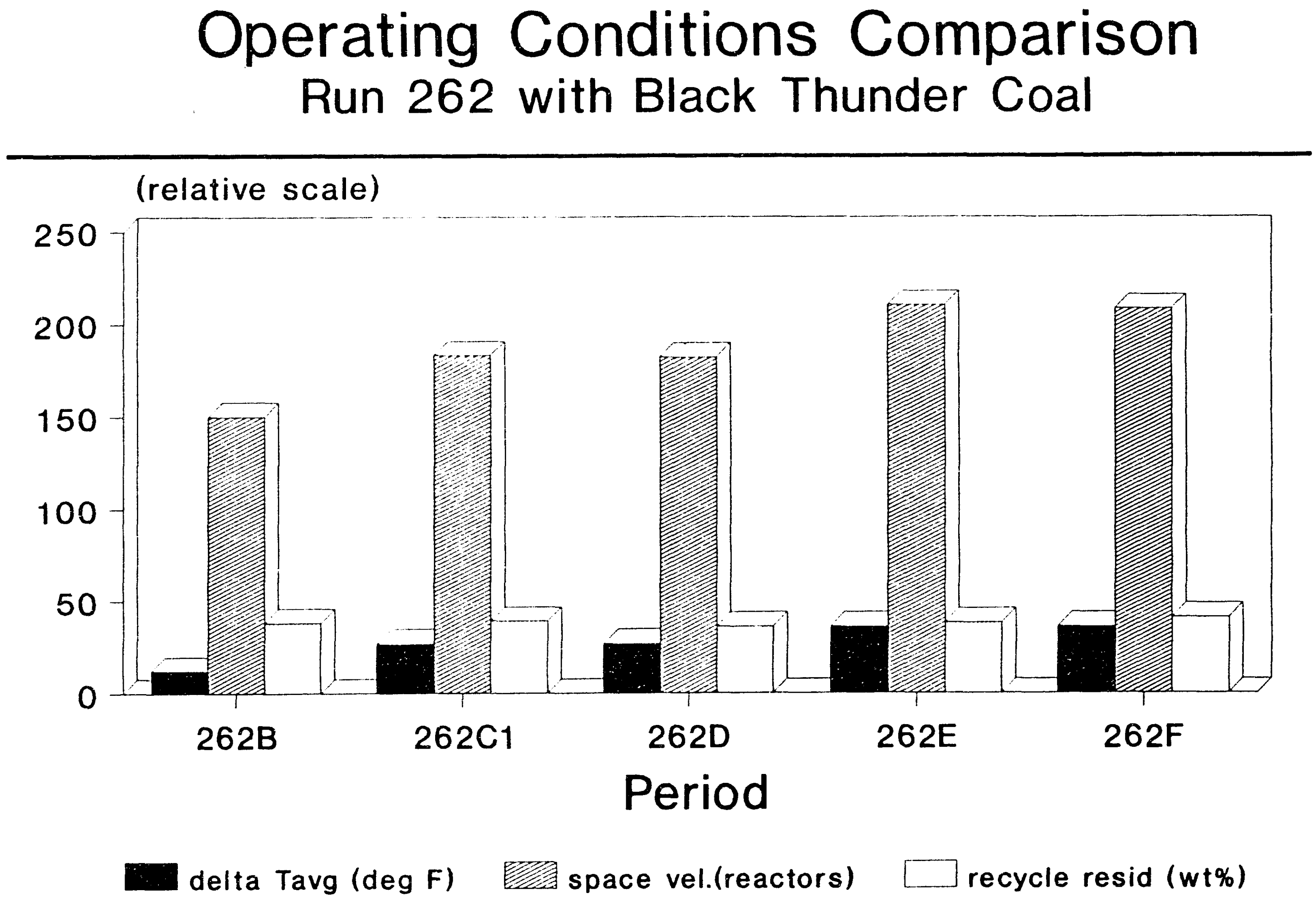
half-volume reactor operation;
262B-E (hybrid), 262F (slurry);
Molyvan L addition

FIGURE 97. DISTILLATE PRODUCTION WITH BLACK THUNDER COAL (RUN 262B-F)



supported catalyst replacement -
100 = 1.5 lb/t; Moly L - Molyvan L;
total Moly - total molybdenum

FIGURE 98. CATALYST AND MOLYBDENUM REQUIREMENT WITH BLACK THUNDER COAL (RUN 262B-F)

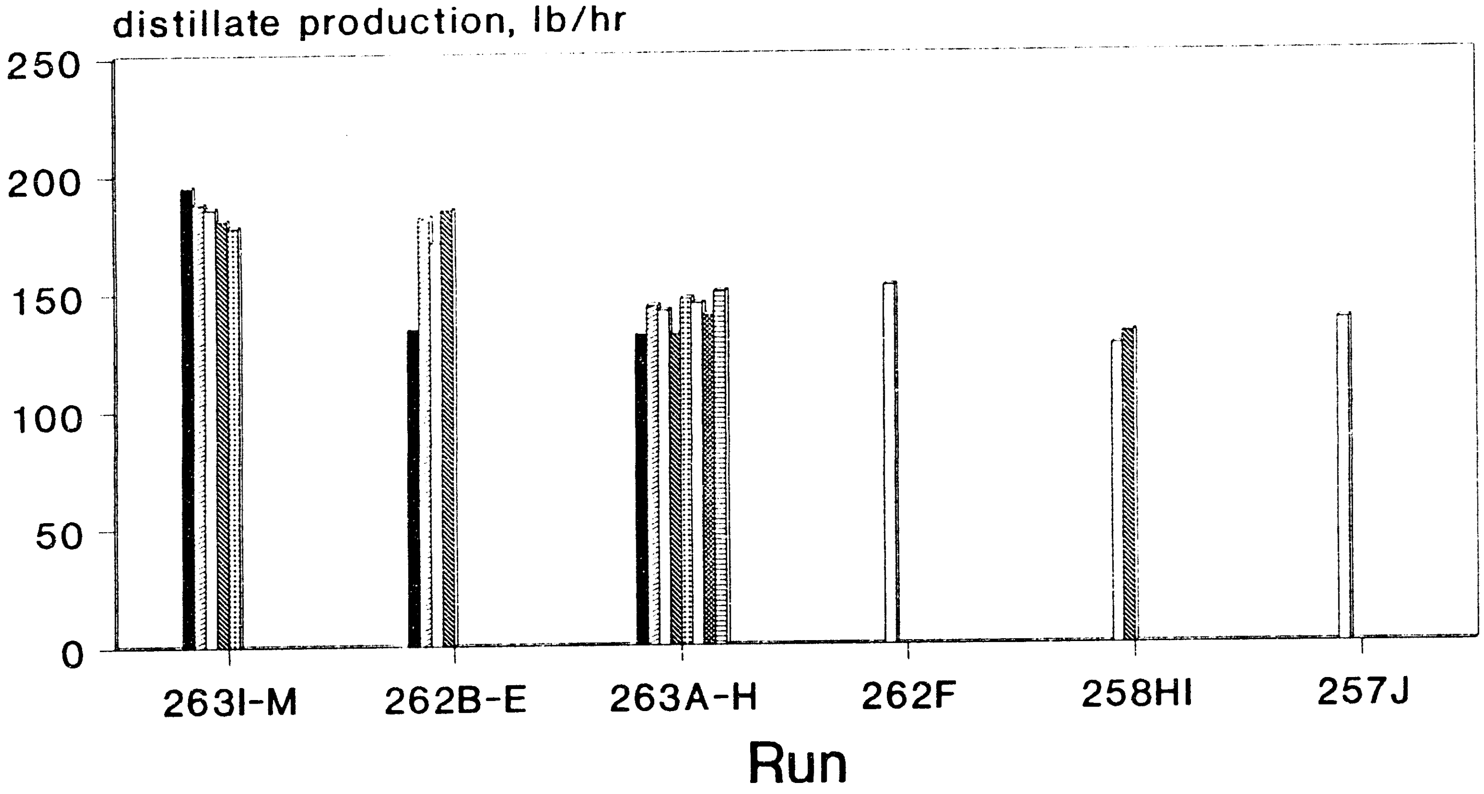


base run : 251-IIB
w/ Wyodak and Amocat 1C

FIGURE 99. OPERATING CONDITIONS WITH BLACK THUNDER COAL (RUN 262B-F)

Distillate Production Comparison

Black Thunder Coal (Runs 258, 262 & 263) with Half-Volume Reactor Operation

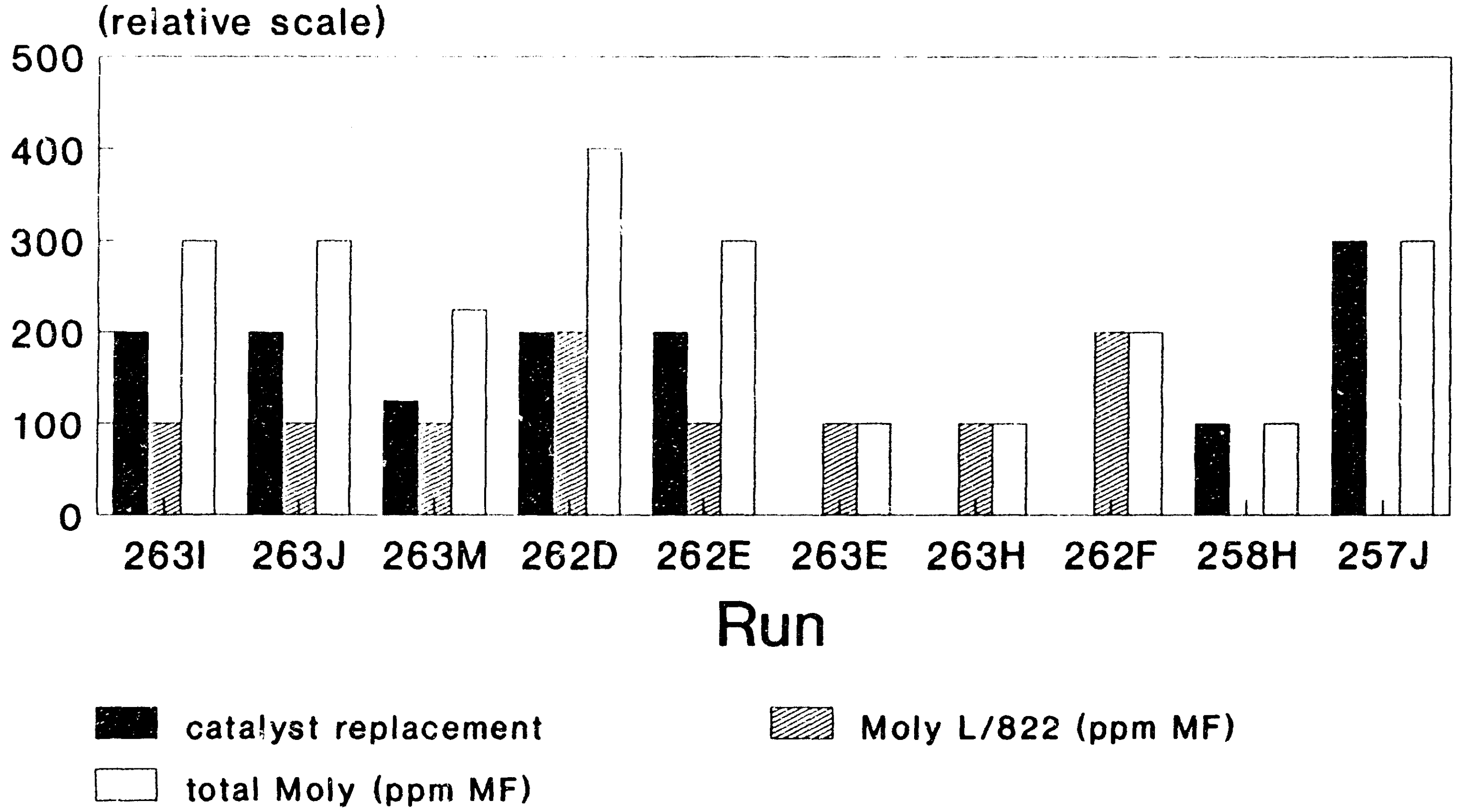


hybrid: 263I-M, 262B-E w/ Criterion and Molyvan L or 822
slurry: 263A-H, 262F w/ Molyvan L or 822
supported: 258HI w/ Shell 324, 257J w/ Illinois No. 6 and Amocat 1C

FIGURE 100. COMPARISON OF DISTILLATE PRODUCTION WITH BLACK THUNDER AND ILLINOIS NO. 6 COALS (RUNS 263, 262, 258HI, 257J)

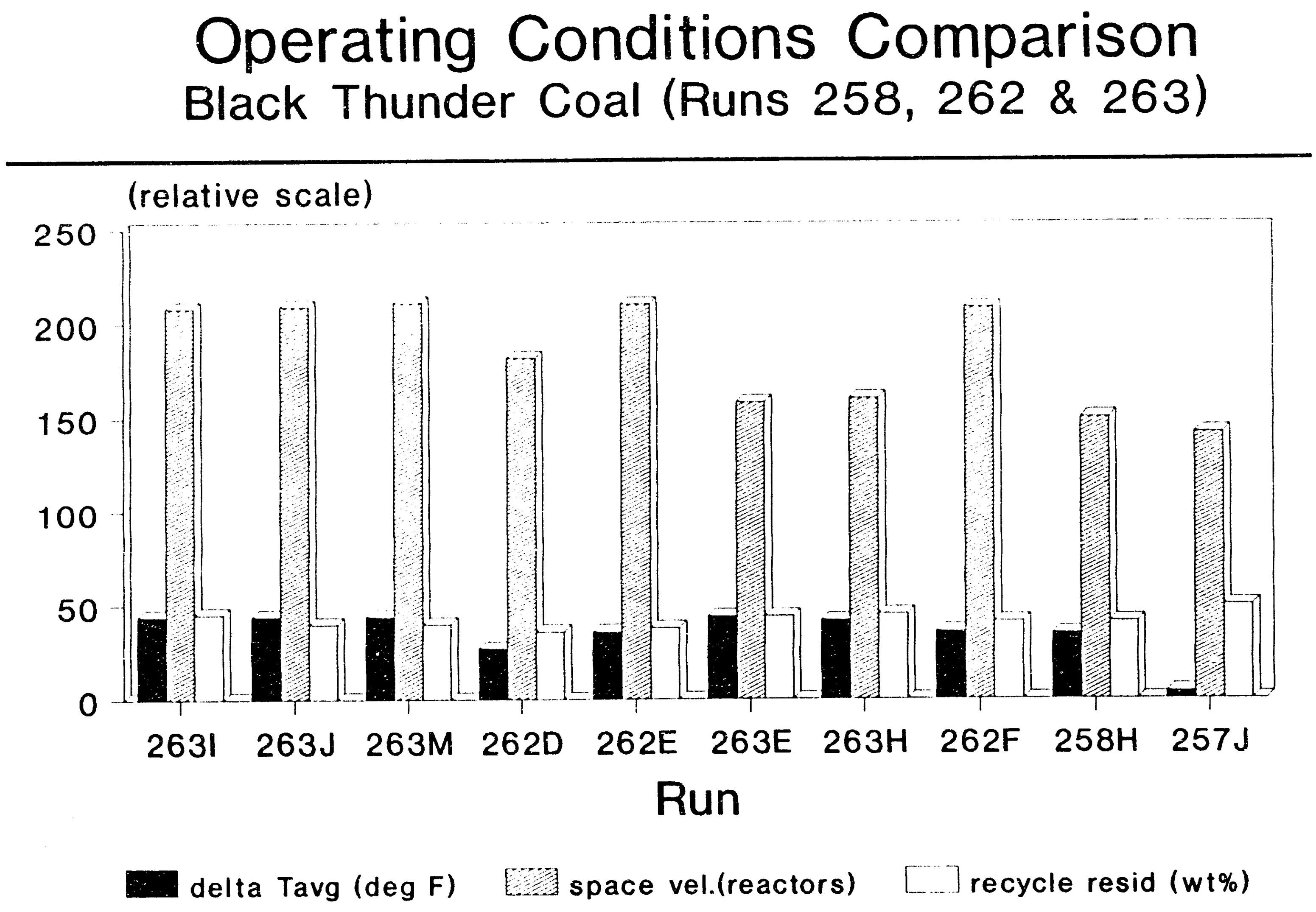
CATALYST & MOLYBDENUM REQUIREMENT

Black Thunder Coal (Runs 258, 262 & 263)



supported catalyst replacement - 100 = 1.5 lb/t; Moly L - Molyvan L; total Moly - total molybdenum

FIGURE 101. COMPARISON OF CATALYST AND MOLYBDENUM REQUIREMENT WITH BLACK THUNDER AND ILLINOIS NO. 6 COALS (RUNS 263, 262, 258HI, 257J)



base run : 251-IIB
w/ Wyodak and Amocat 1C

FIGURE 102. COMPARISON OF OPERATING CONDITIONS WITH BLACK THUNDER AND ILLINOIS NO. 6 COALS (RUNS 263, 262, 258HI, 257J)

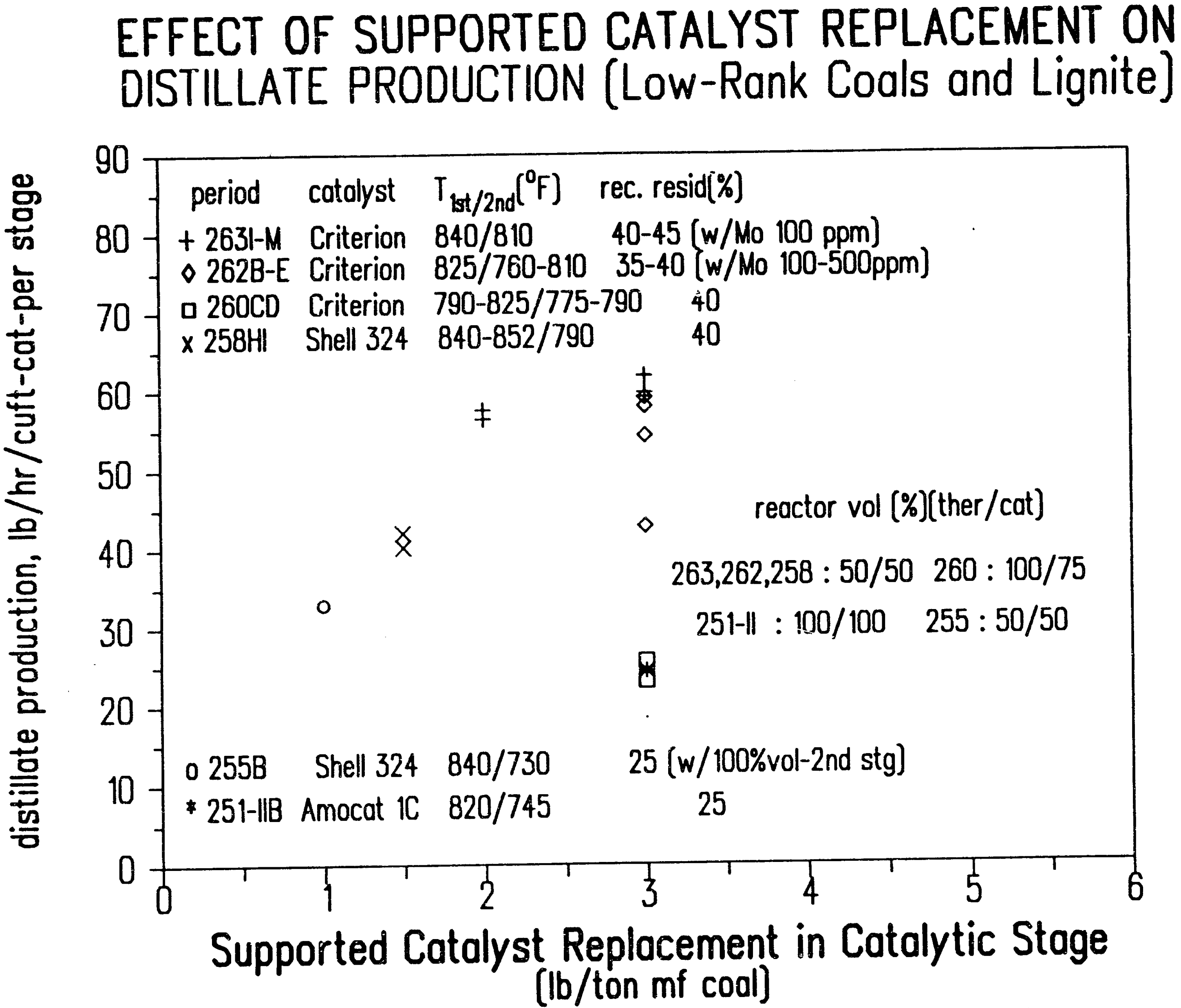


FIGURE 103. EFFECT OF SUPPORTED CATALYST REPLACEMENT ON DISTILLATE PRODUCTION - LOW-RANK COALS AND LIGNITE (RUNS 263, 262, 260, 258, 255, 251-II)
UNIT: LB/HR/CU FT-CATALYST PER STAGE

EFFECT OF SUPPORTED CATALYST REPLACEMENT ON DISTILLATE PRODUCTION (Low-Rank Coals and Lignite)

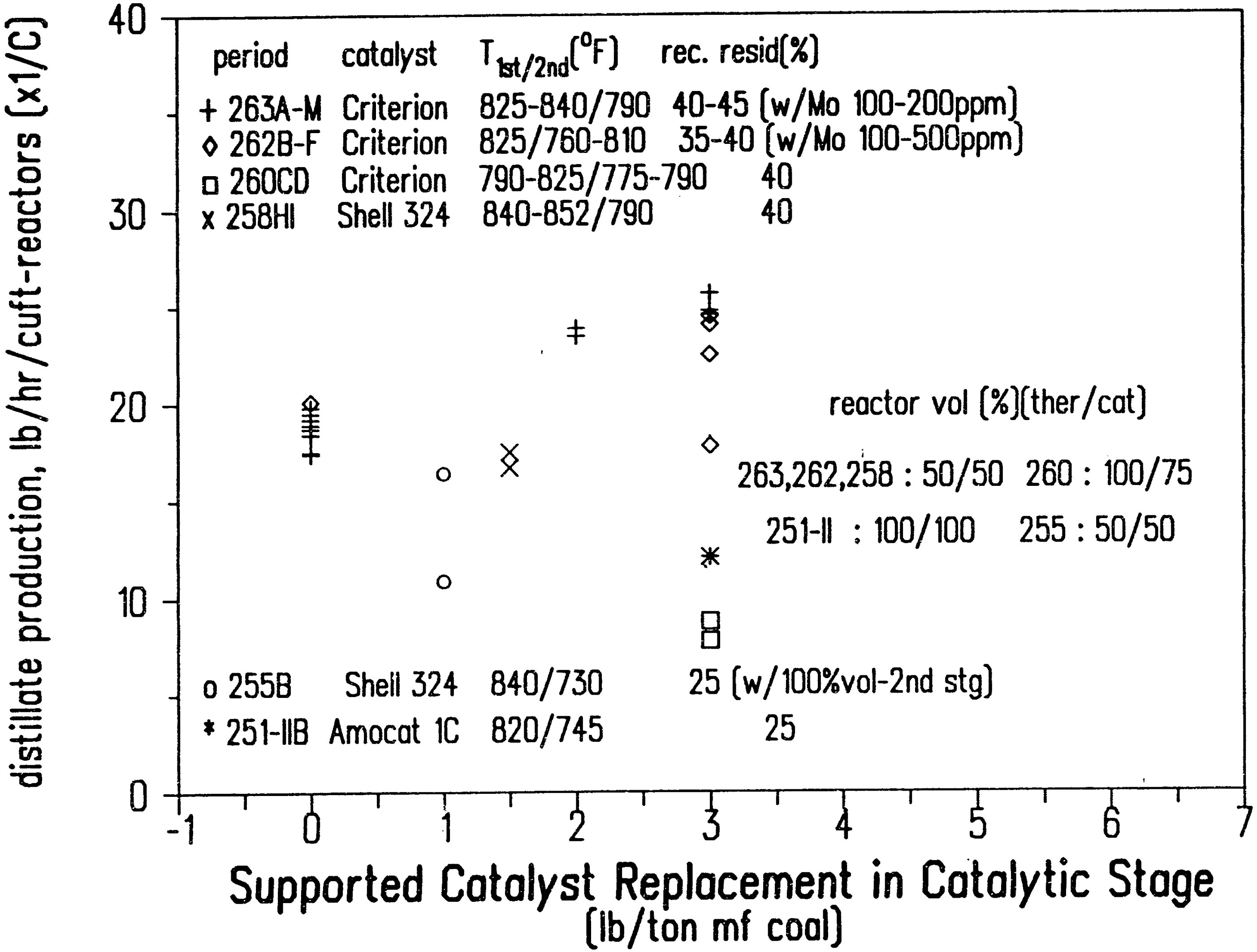
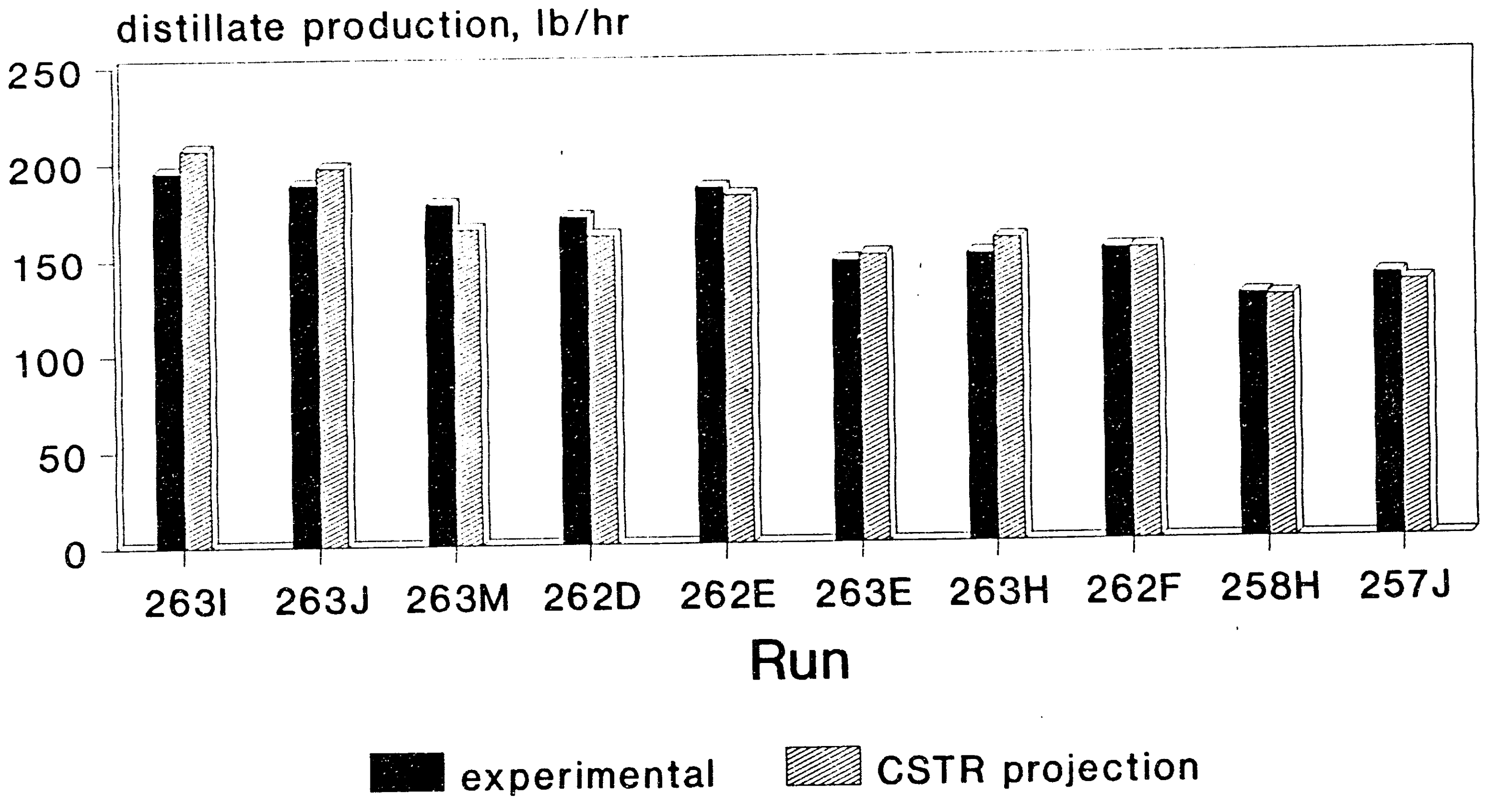


FIGURE 104. EFFECT OF SUPPORTED CATALYST REPLACEMENT ON DISTILLATE PRODUCTION - LOW-RANK COALS AND LIGNITE (RUNS 263, 262, 260, 258, 255, 251-II) UNIT: LB/HR/CU FT-CCR REACTORS (x1/C)

Experimental Vs. CSTR Projection
Black Thunder Coal (Runs 258, 262 & 263)
with Half-Volume Reactor Operation



* CSTR 1st order projection
for "all-distillate" product slate

FIGURE 105. DISTILLATE PRODUCTION COMPARISON BETWEEN EXPERIMENTAL AND CSTR PROJECTION (RUNS 263, 262, 258HI, 257J)

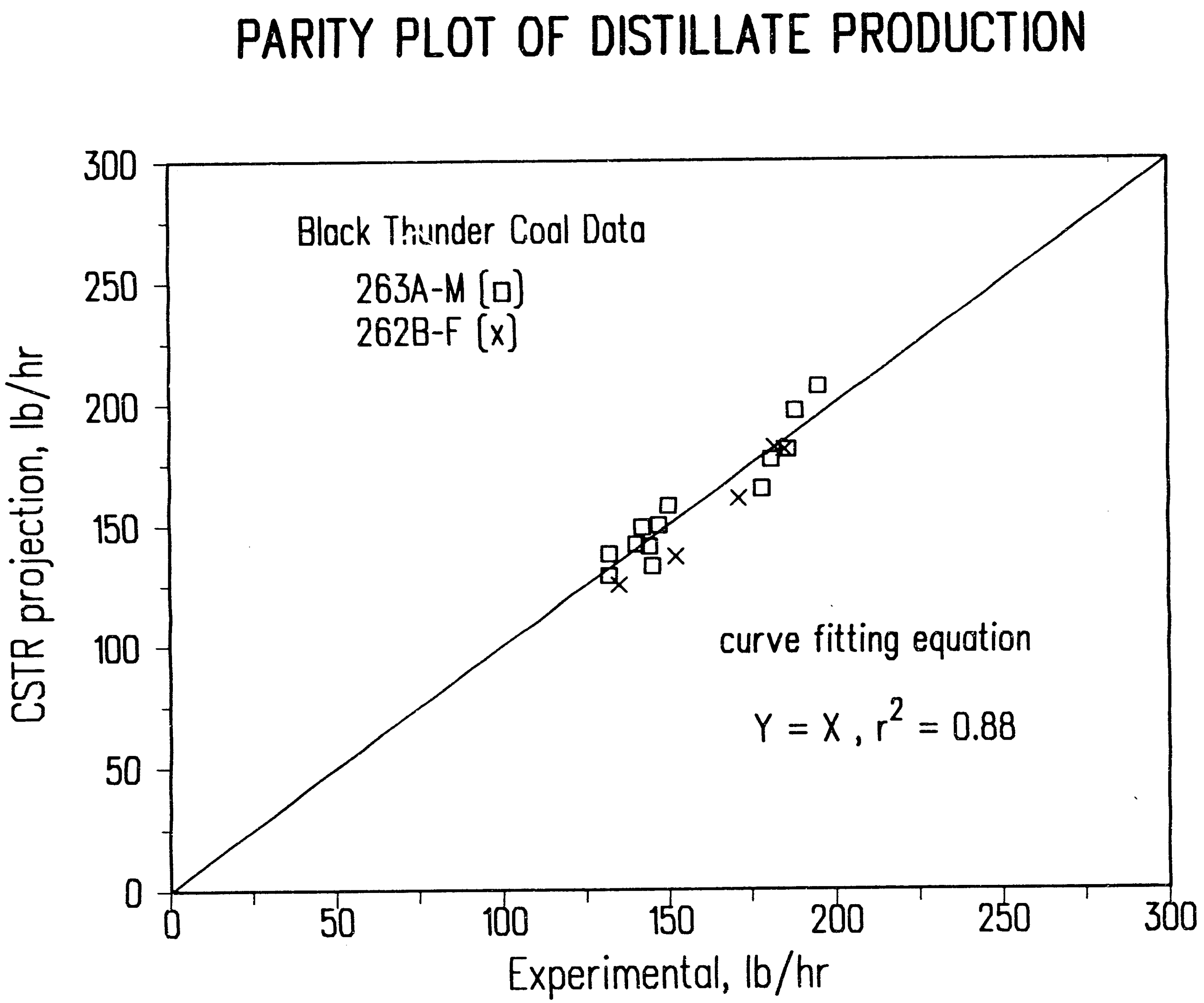
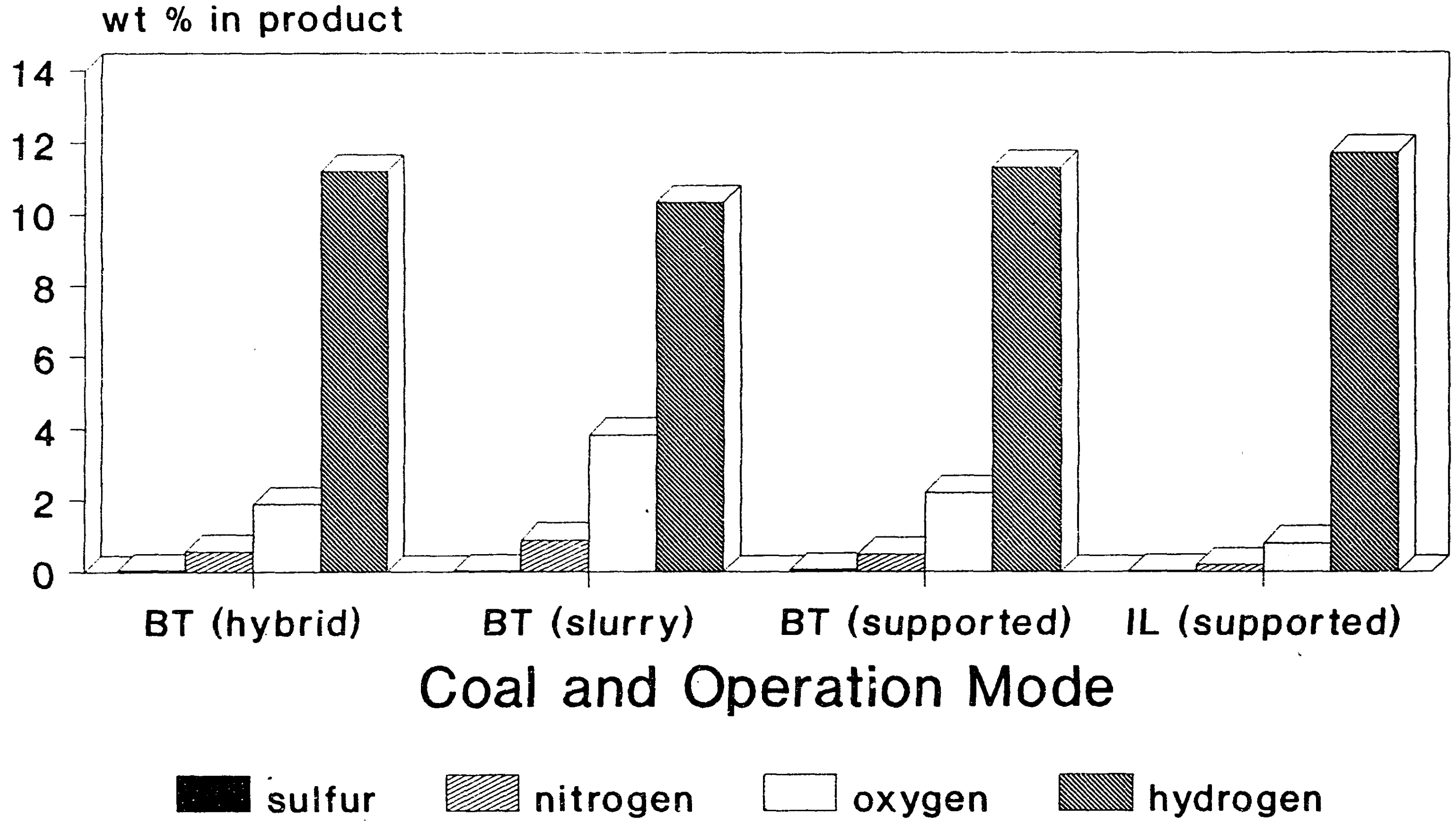


FIGURE 106. PARITY PLOT OF DISTILLATE PRODUCTION WITH BLACK THUNDER COAL - EXPERIMENTAL VS. CSTR PROJECTION (RUNS 263, 262)

DISTILLATE PRODUCT QUALITY COMPARISON
Black Thunder Coal Vs. Illinois Coal



BT - Black Thunder, IL - Illinois;
hybrid (262E,263J); slurry (263EH);
supported (BT-258HI, IL-257J,261BD)

Operating Conditions

BT runs	IL runs
thermal/catalytic	catalytic/catalytic
Shell, Criterion 324 Molyvan L or 822	Amocat 1C, EXP-AO-60
SV/C=65-92	SV/C=44-68 lb/hr/cuft-reactor
T=790-850F	T=760-810F

FIGURE 107. COMPARISON OF DISTILLATE PRODUCT QUALITY - BLACK THUNDER VS. ILLINOIS COAL

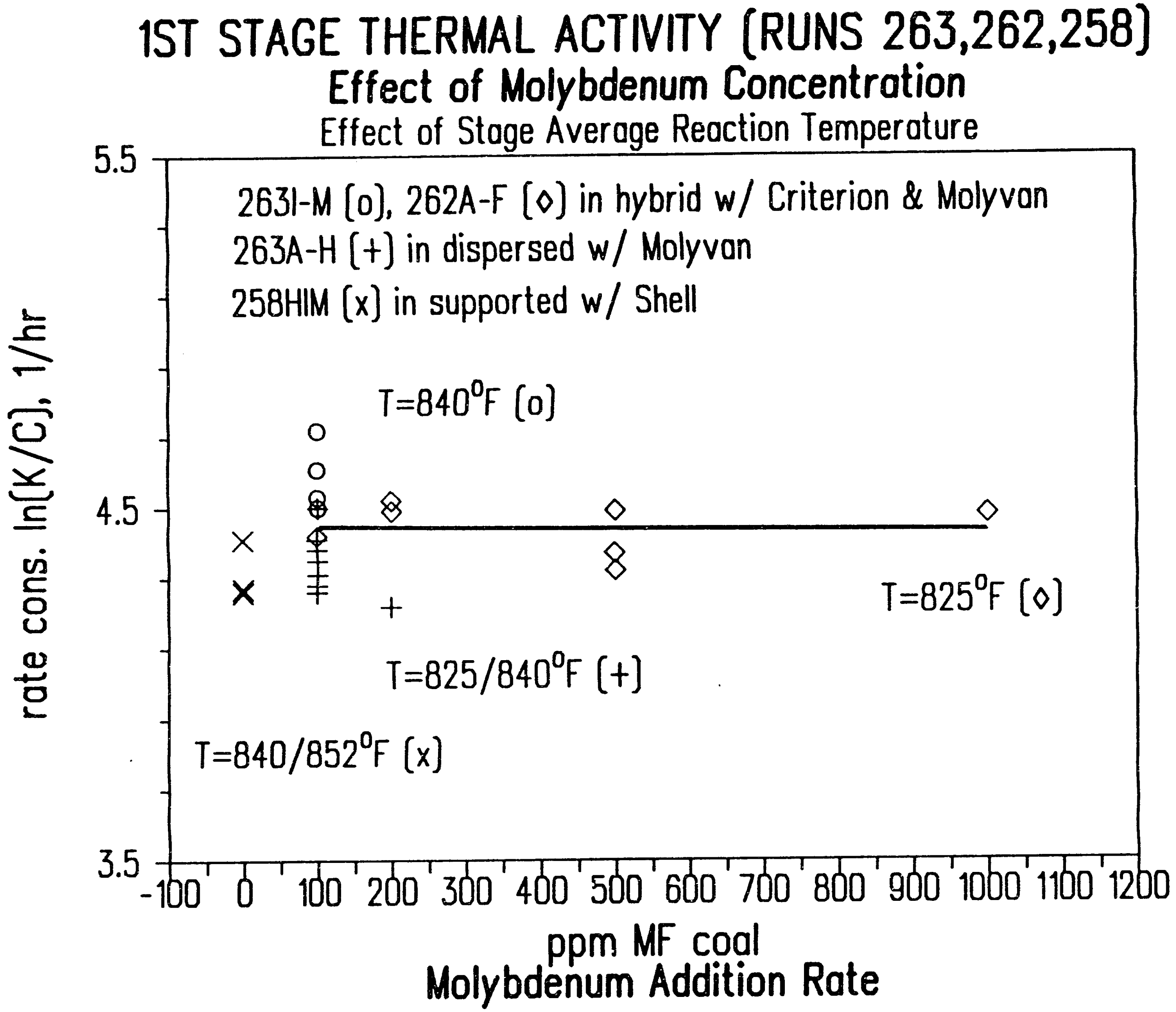


FIGURE 108. EFFECTS OF FIRST STAGE TEMPERATURE AND MOLYBDENUM ADDITION RATE ON FIRST STAGE THERMAL ACTIVITY IN RESID+UC CONVERSION (RUNS 263, 262,258)

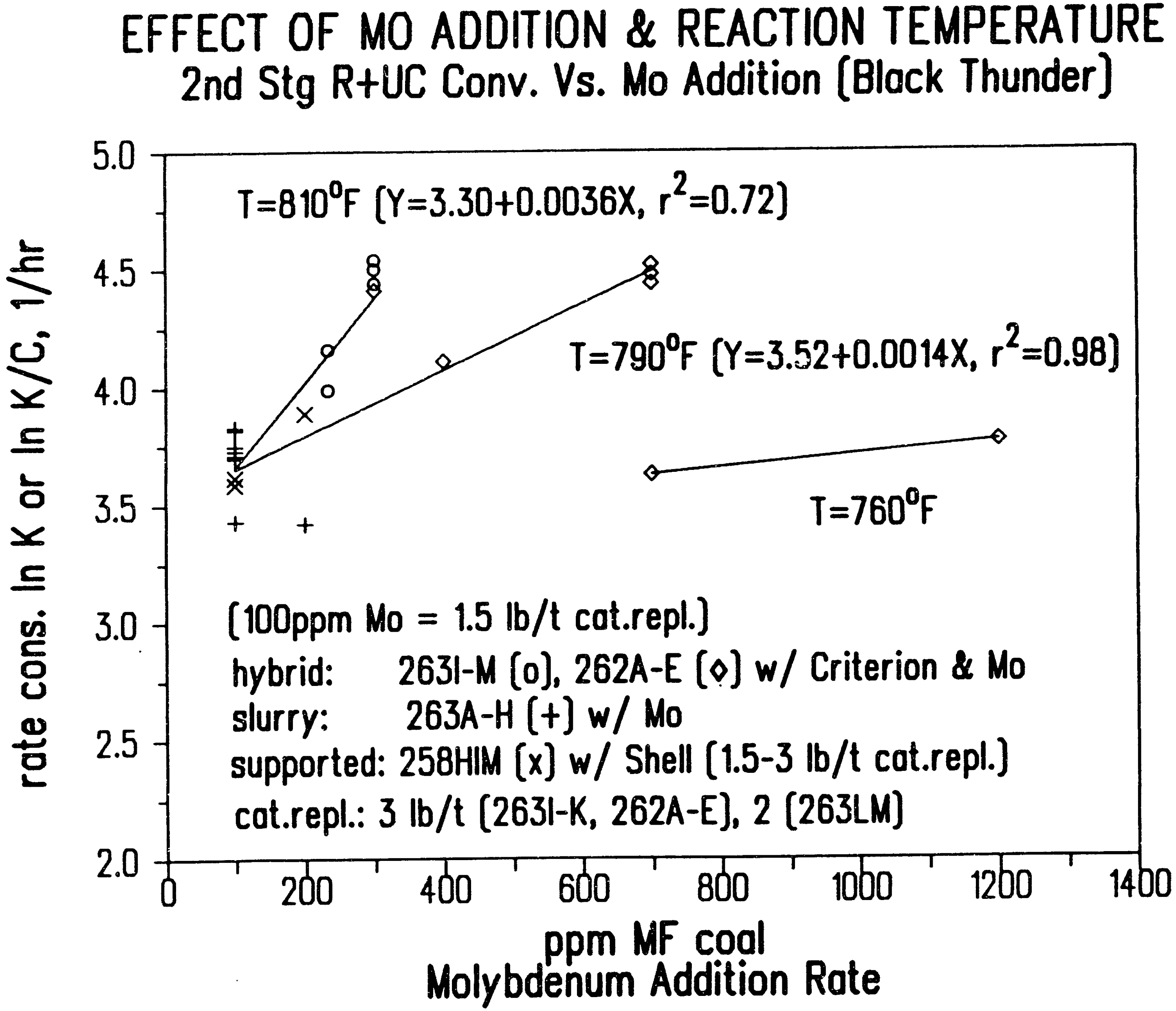


FIGURE 109. EFFECTS OF SECOND STAGE TEMPERATURE AND MOLYBDENUM ADDITION RATE ON SECOND STAGE ACTIVITY IN RESID+UC CONVERSION (RUNS 263, 262, 258)

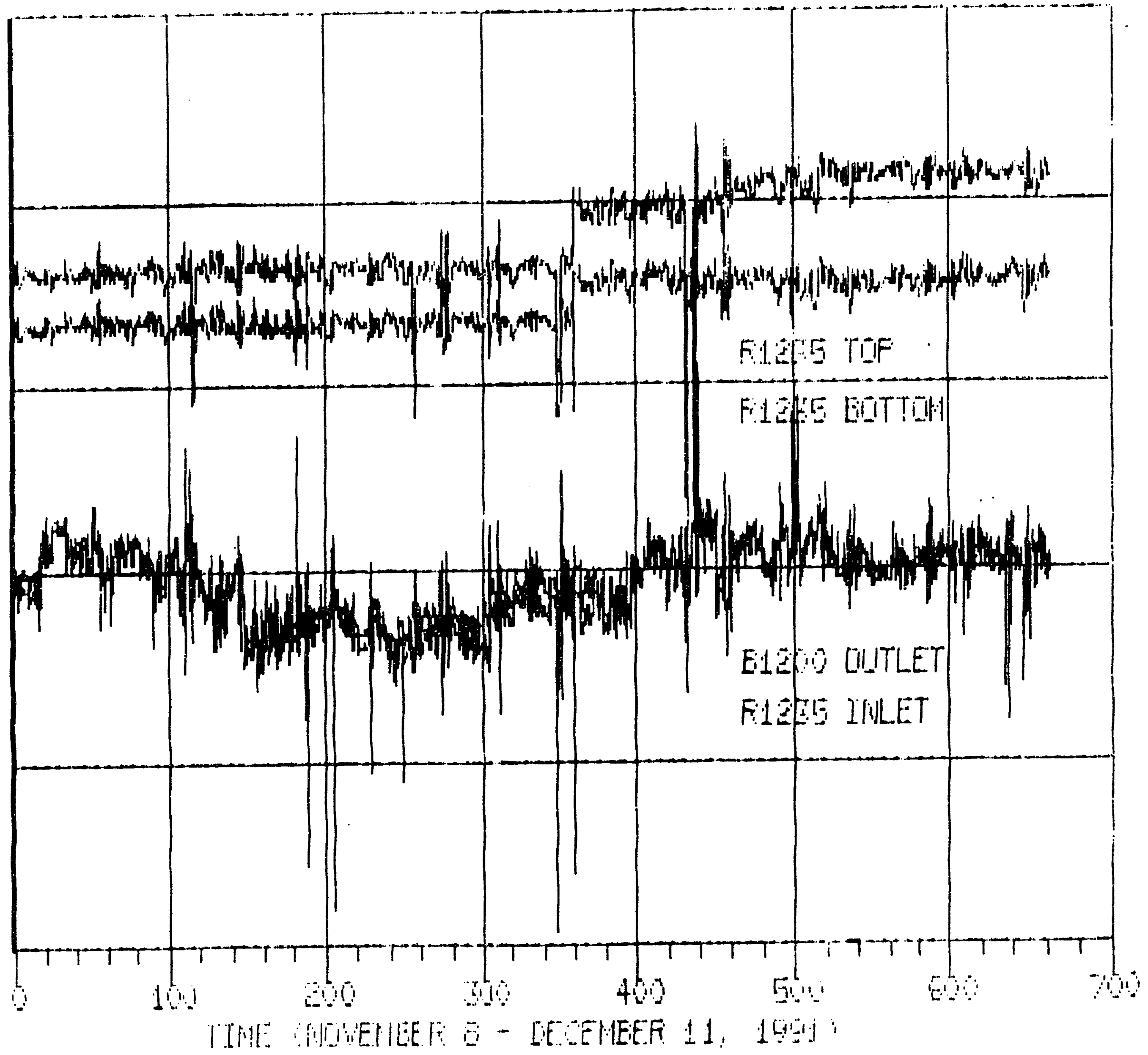


FIGURE 110. PREHEATER OUTLET AND FIRST STAGE REACTOR TEMPERATURE TREND (NOVEMBER 8 - DECEMBER 11)

B1201 - R1236 2ND STAGE

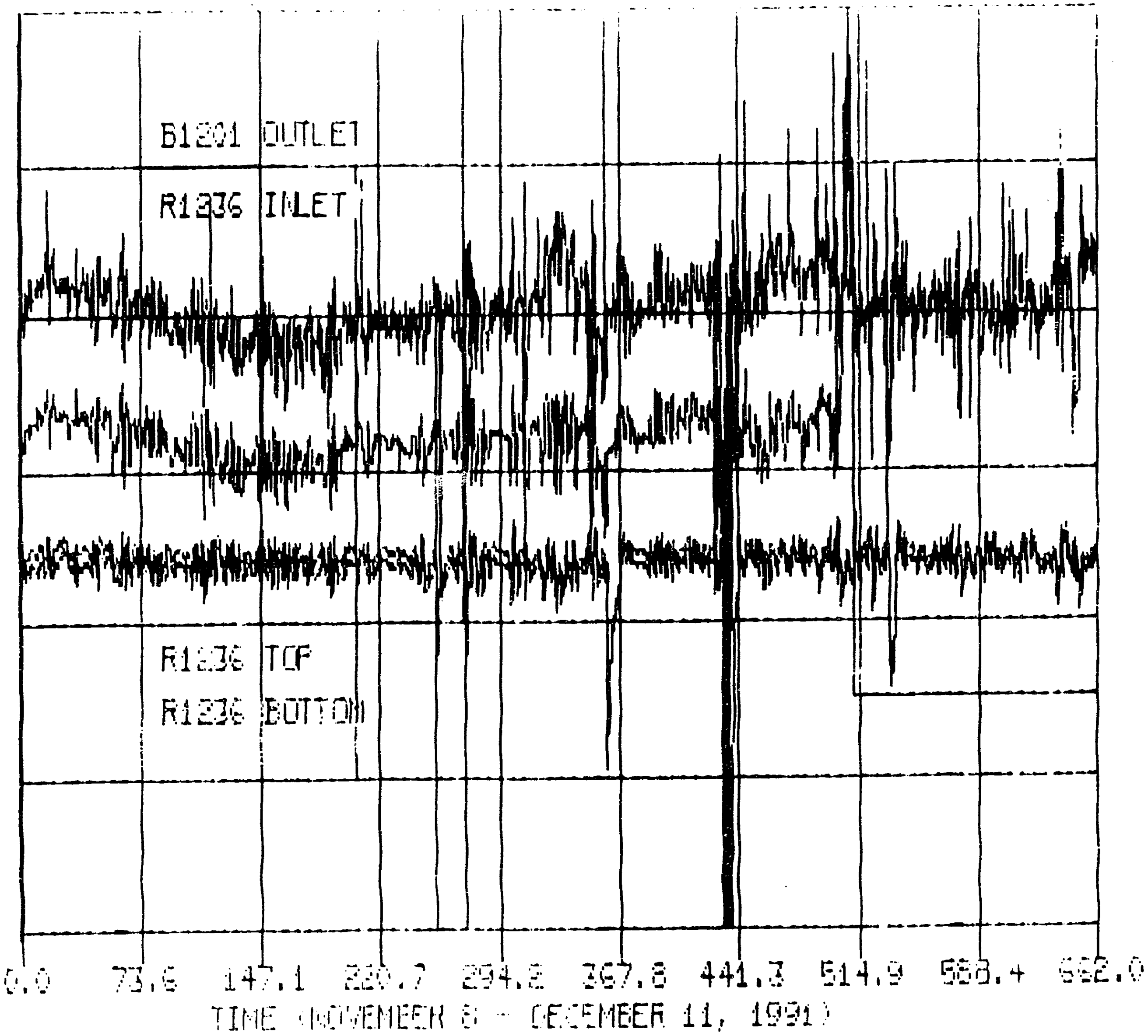


FIGURE 111. INTERSTAGE HEATER OUTLET AND SECOND STAGE REACTOR TEMPERATURE TREND (NOVEMBER 8 - DECEMBER 11)

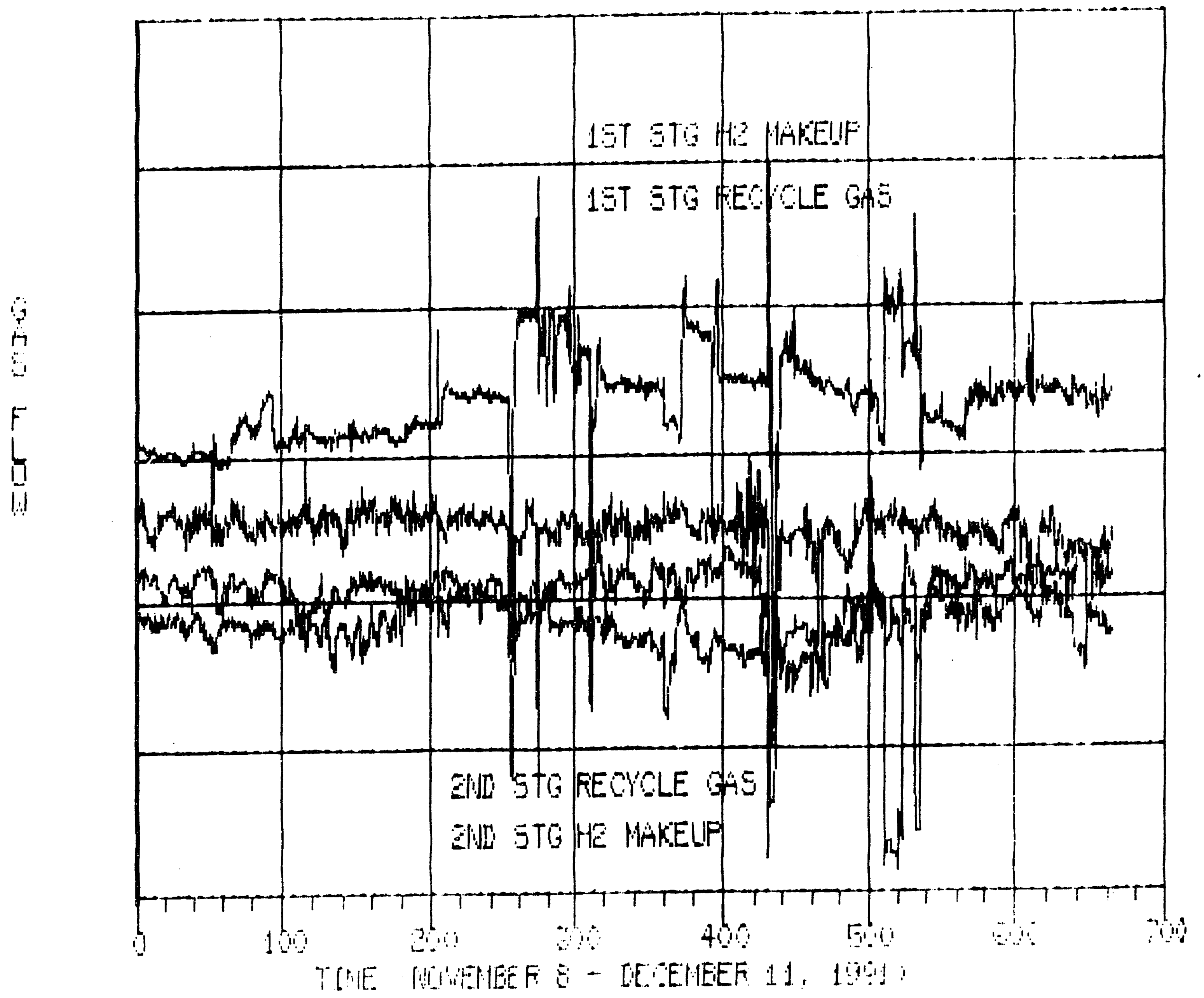


FIGURE 112. GAS FLOW RATE TREND (NOVEMBER 8 - DECEMBER 11)

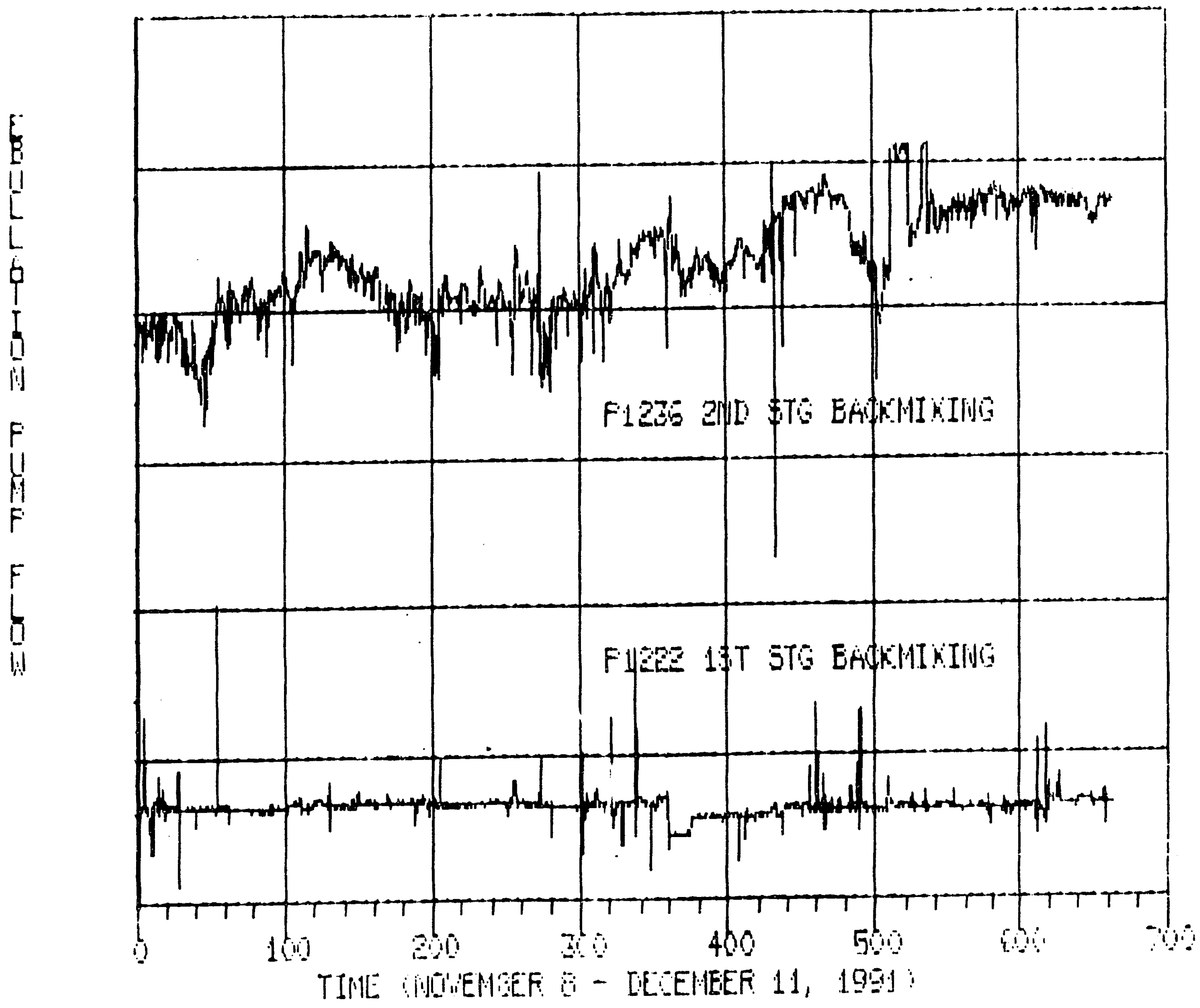


FIGURE 113. EBULLATING AND BACKMIXING PUMP FLOW RATE TREND (NOVEMBER 8 - DECEMBER 11)

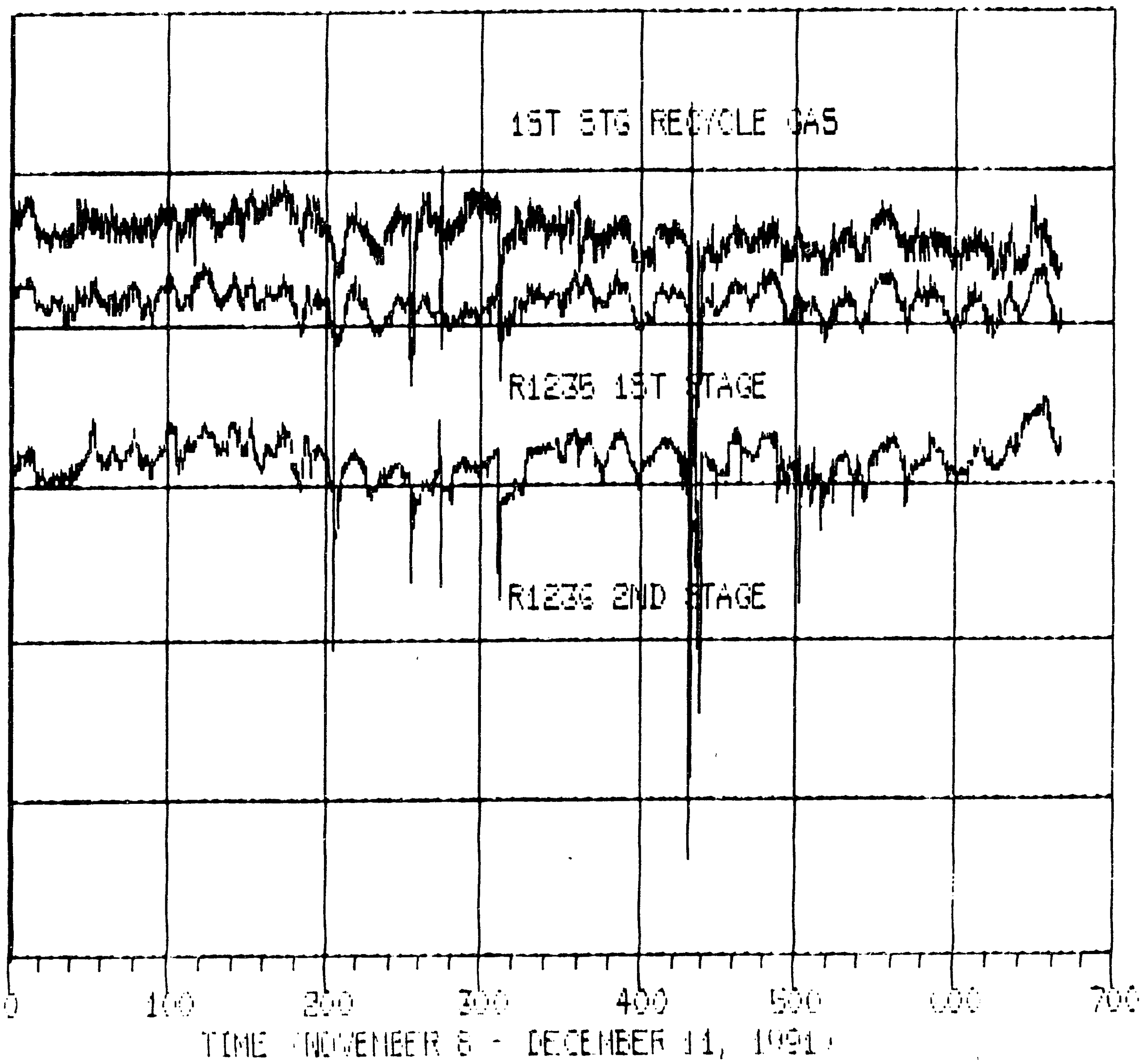


FIGURE 114. SYSTEM PRESSURE TREND (NOVEMBER 8 - DECEMBER 11)

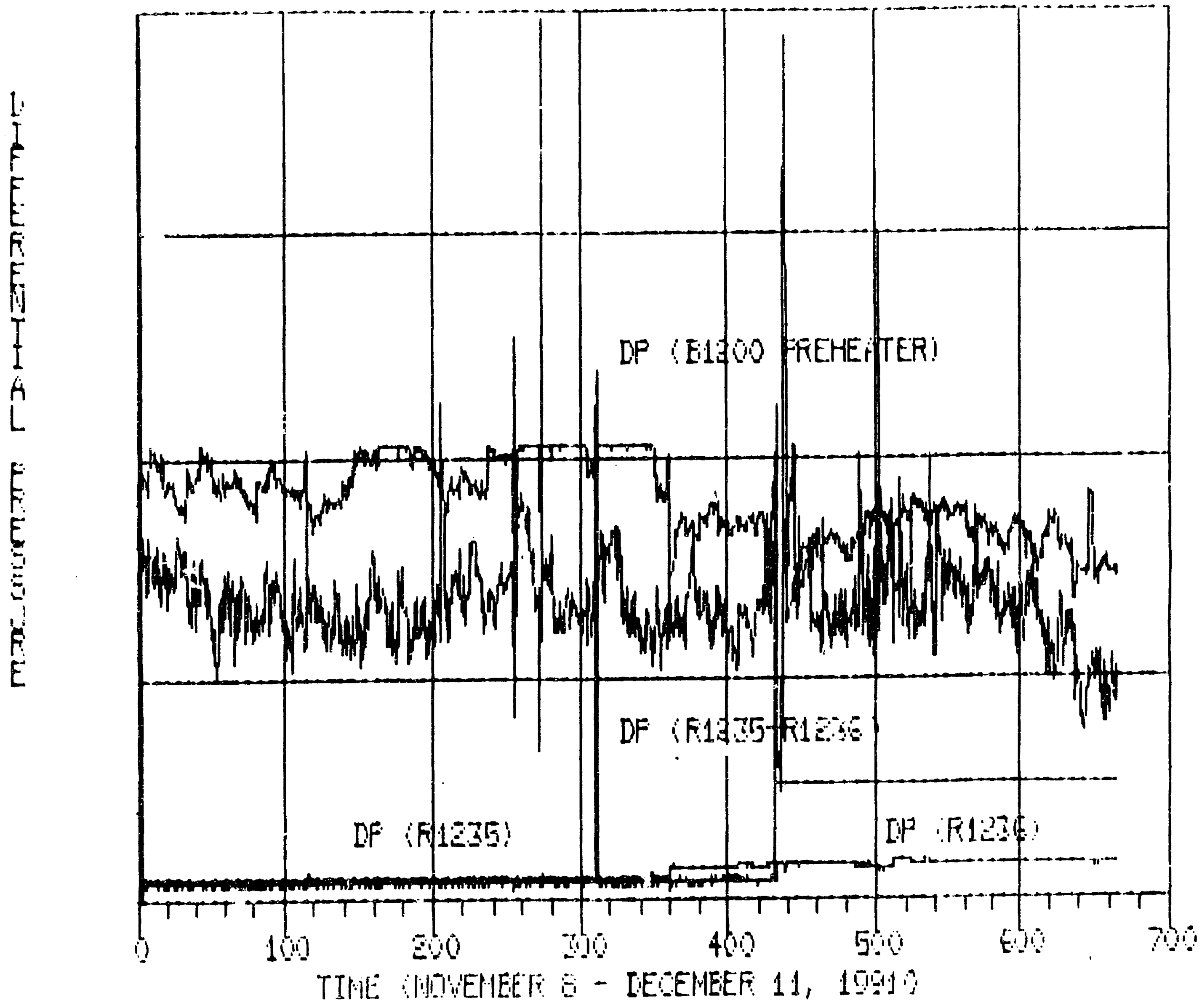


FIGURE 115. DIFFERENTIAL PRESSURE TREND (NOVEMBER 8 - DECEMBER 11)

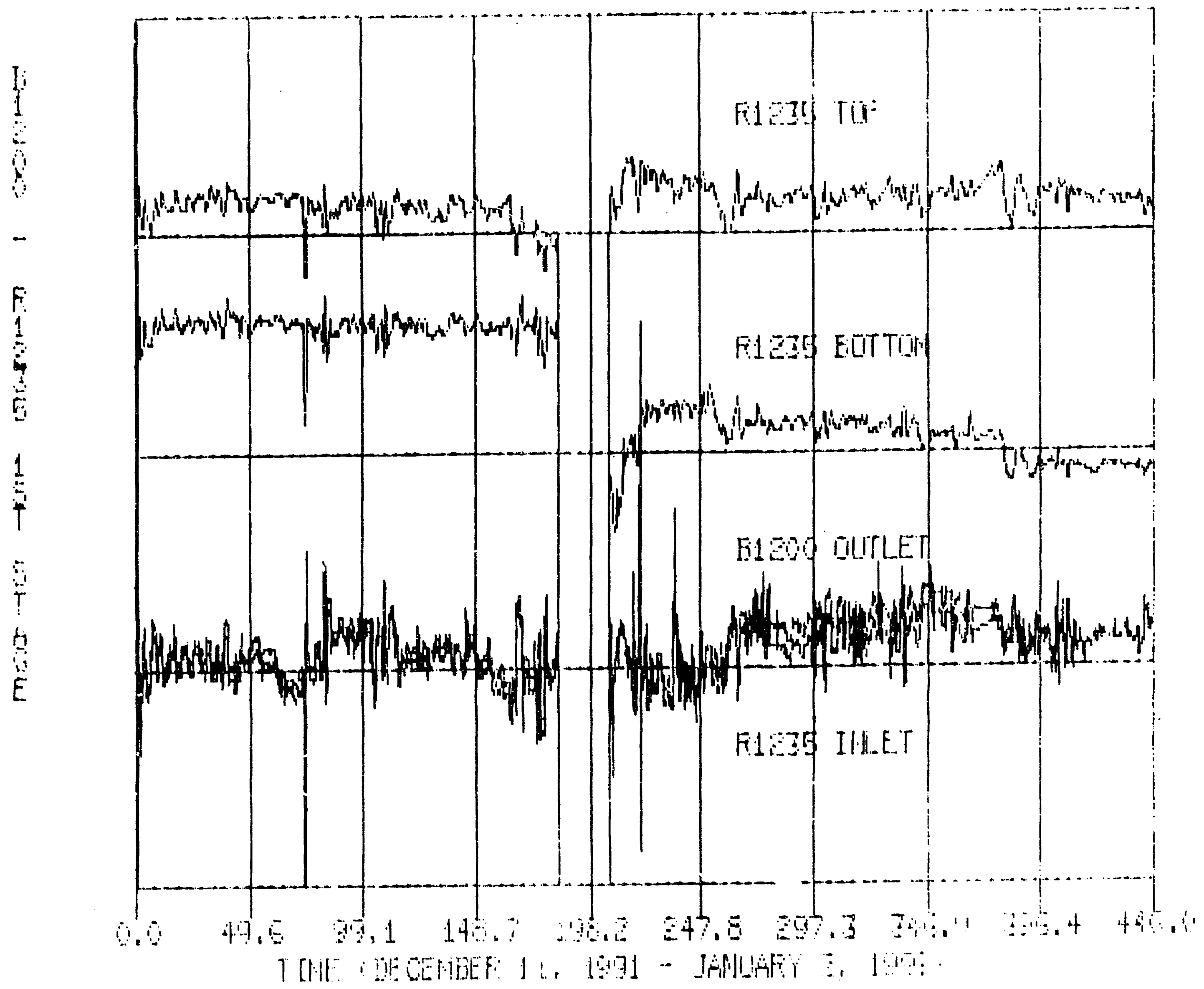


FIGURE 116. PREHEATER OUTLET AND FIRST STAGE REACTOR TEMPERATURE TREND (DECEMBER 11 - JANUARY 2)

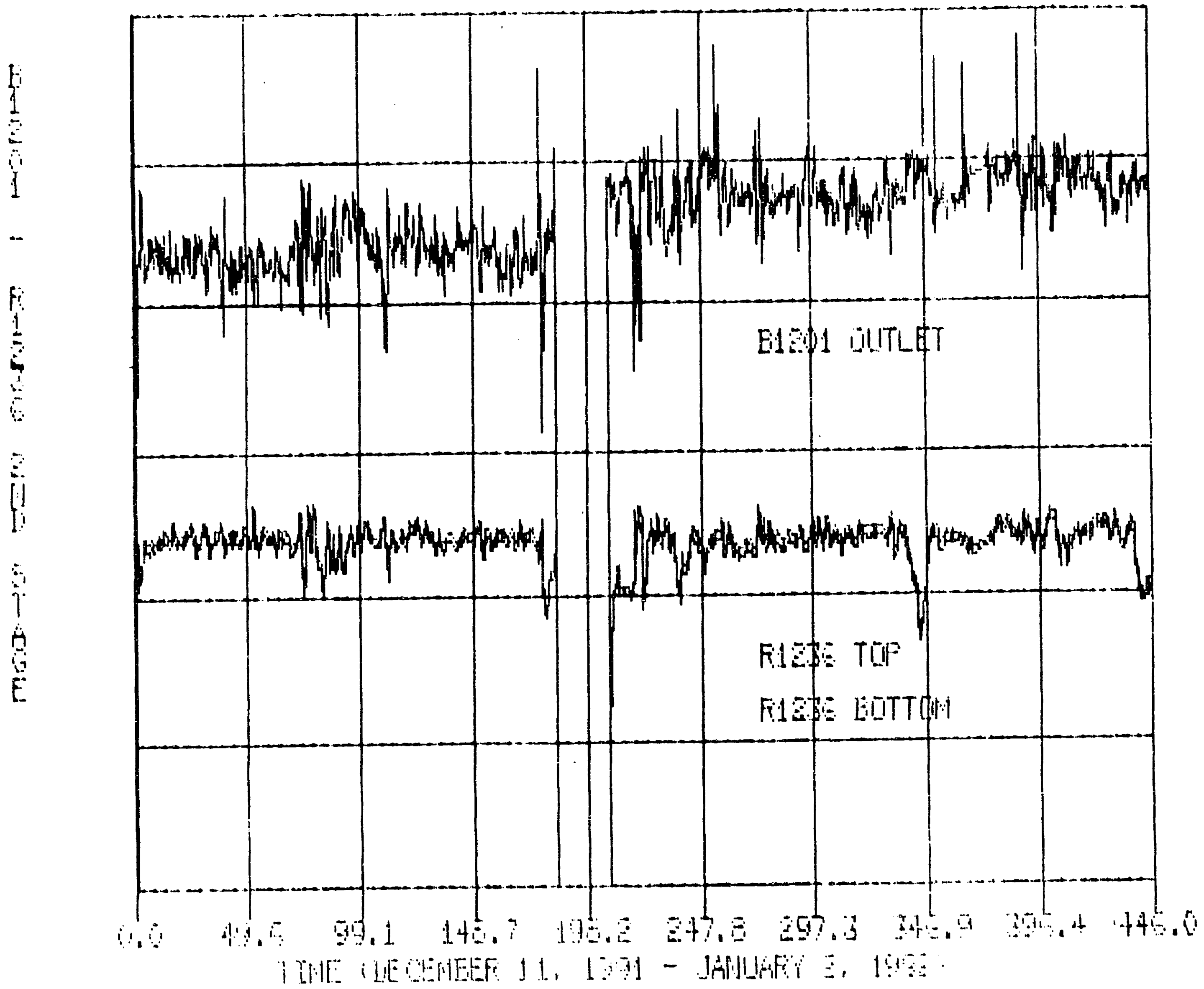


FIGURE 117. INTERSTAGE HEATER OUTLET AND SECOND STAGE REACTOR TEMPERATURE TREND (DECEMBER 11 - JANUARY 2)

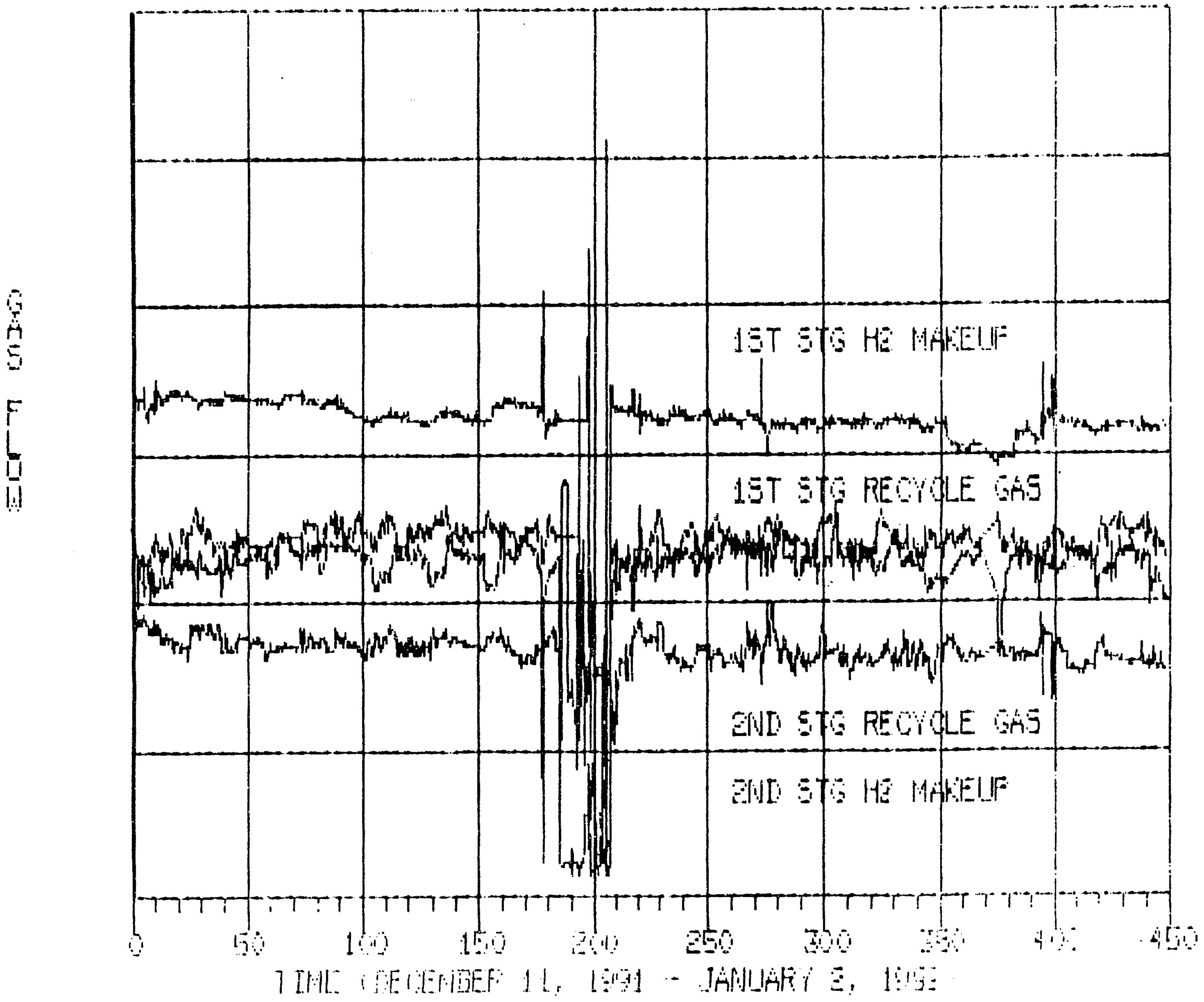


FIGURE 118. GAS FLOW RATE TREND (DECEMBER 11 - JANUARY 2)

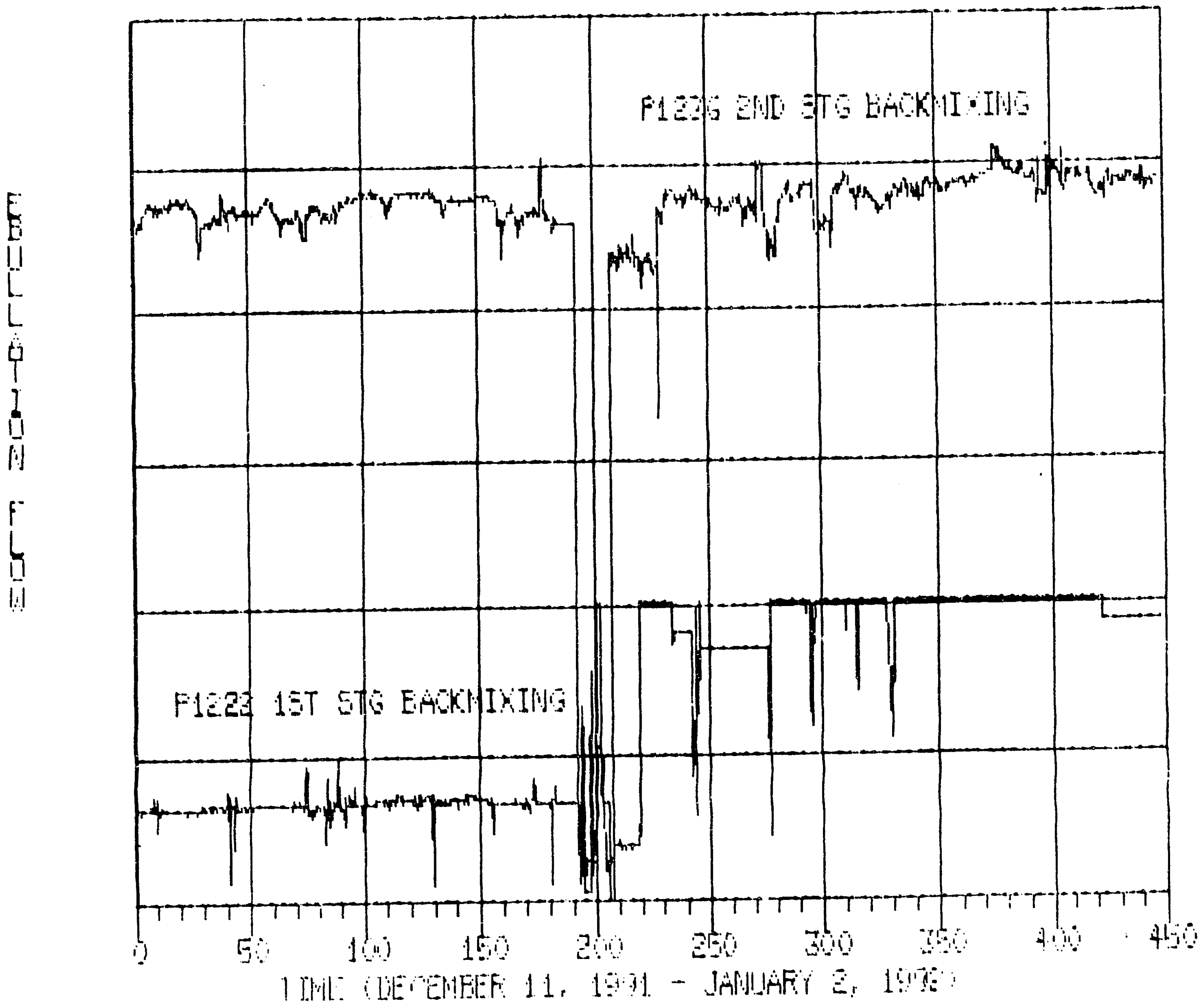


FIGURE 119. EBULLATING AND BACKMIXING PUMP FLOW RATE TREND (DECEMBER 11 - JANUARY 2)

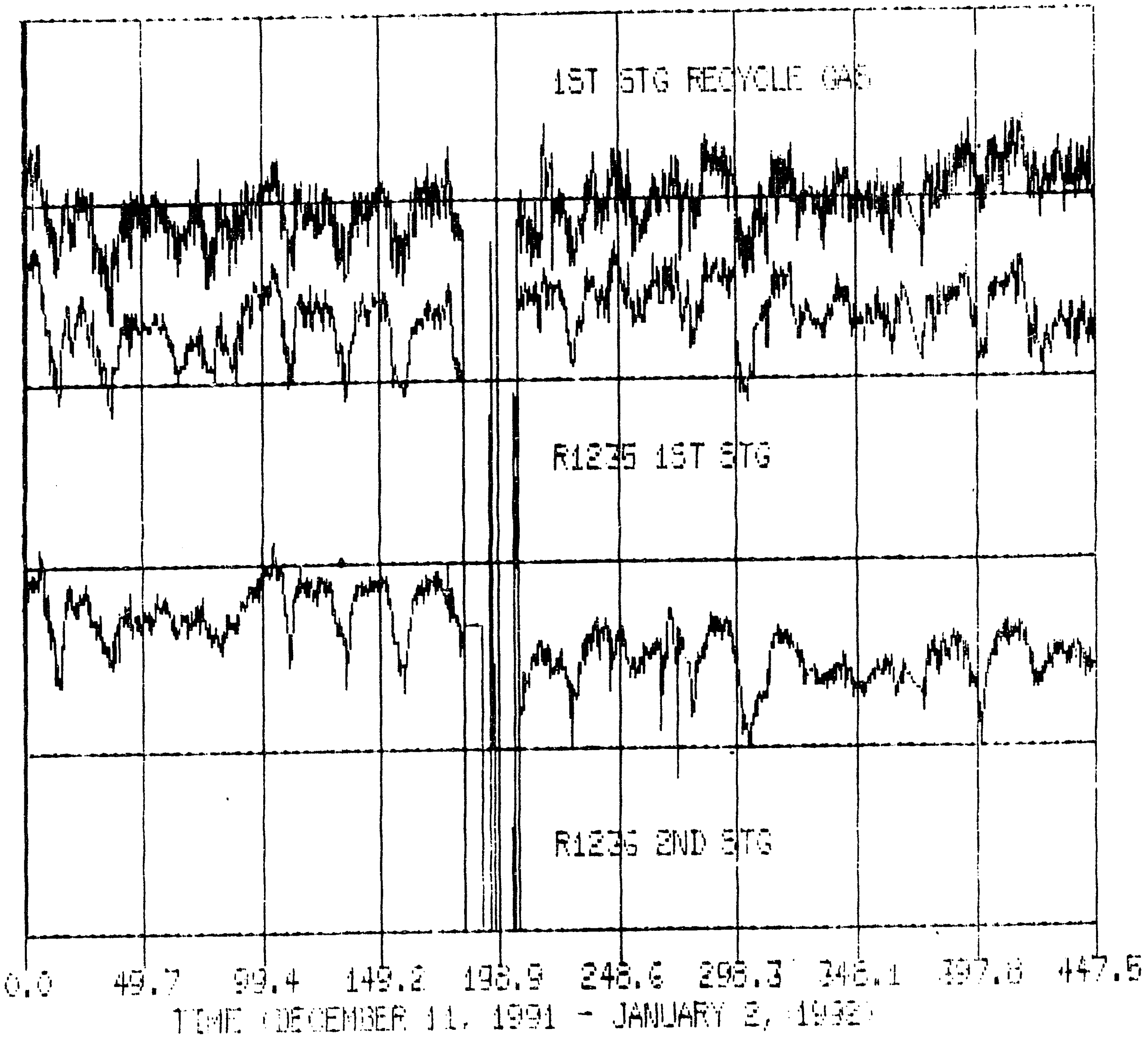


FIGURE 120. SYSTEM PRESSURE TREND (DECEMBER 11 - JANUARY 2)

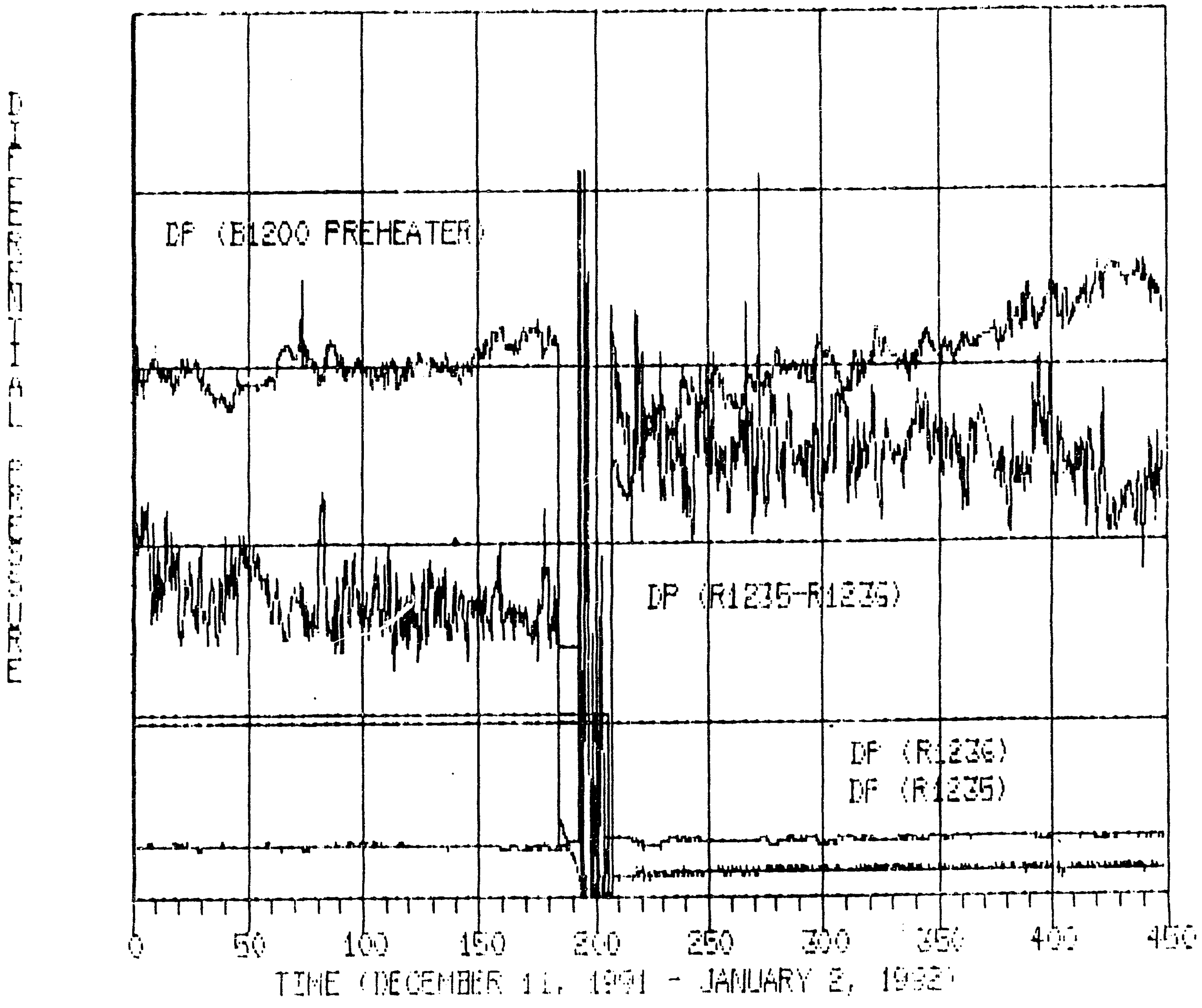


FIGURE 121. DIFFERENTIAL PRESSURE TREND (DECEMBER 11 - JANUARY 2)

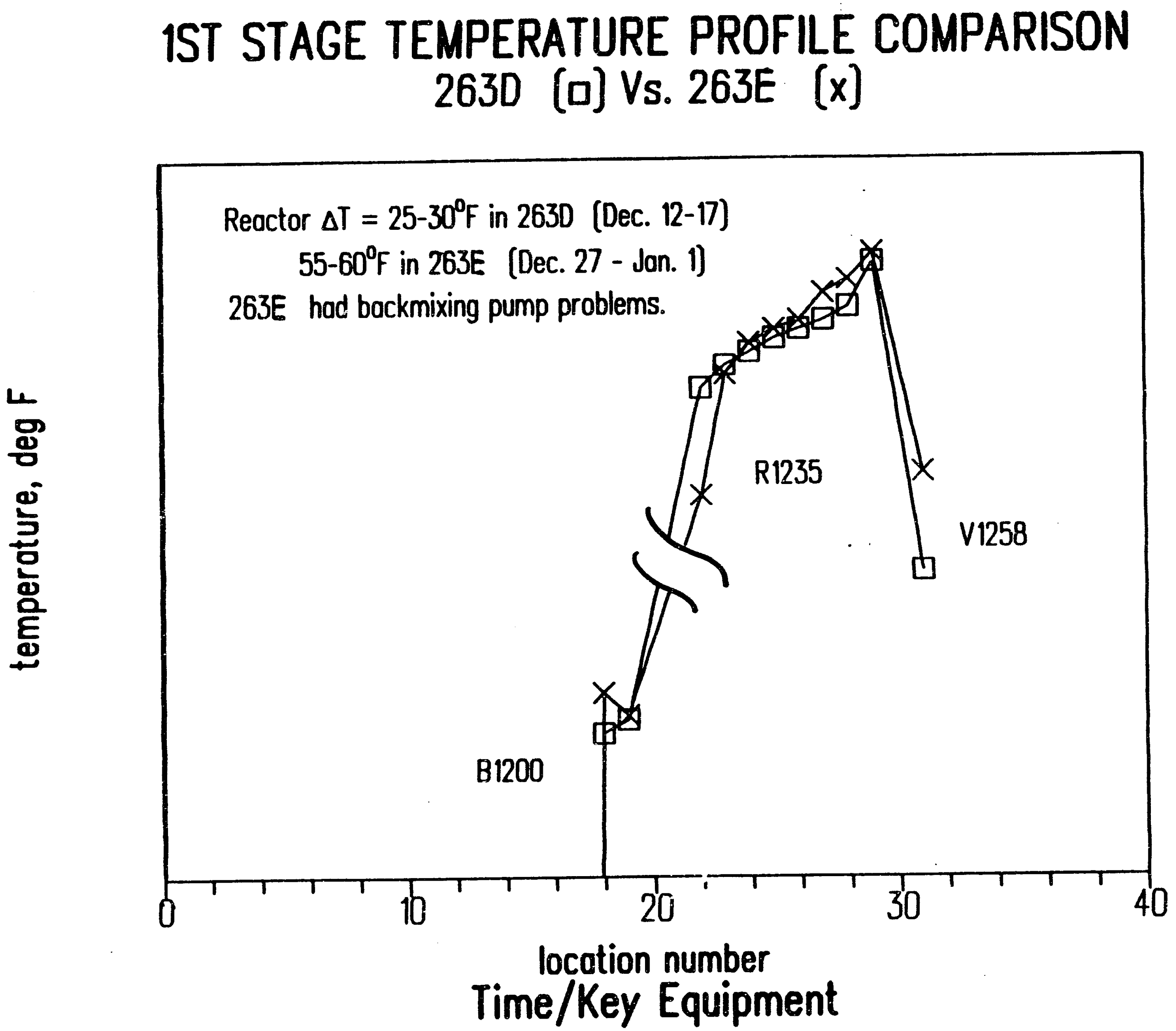


FIGURE 122 . COMPARISON OF FIRST STAGE TEMPERATURE PROFILE IN PERIODS 263D AND E

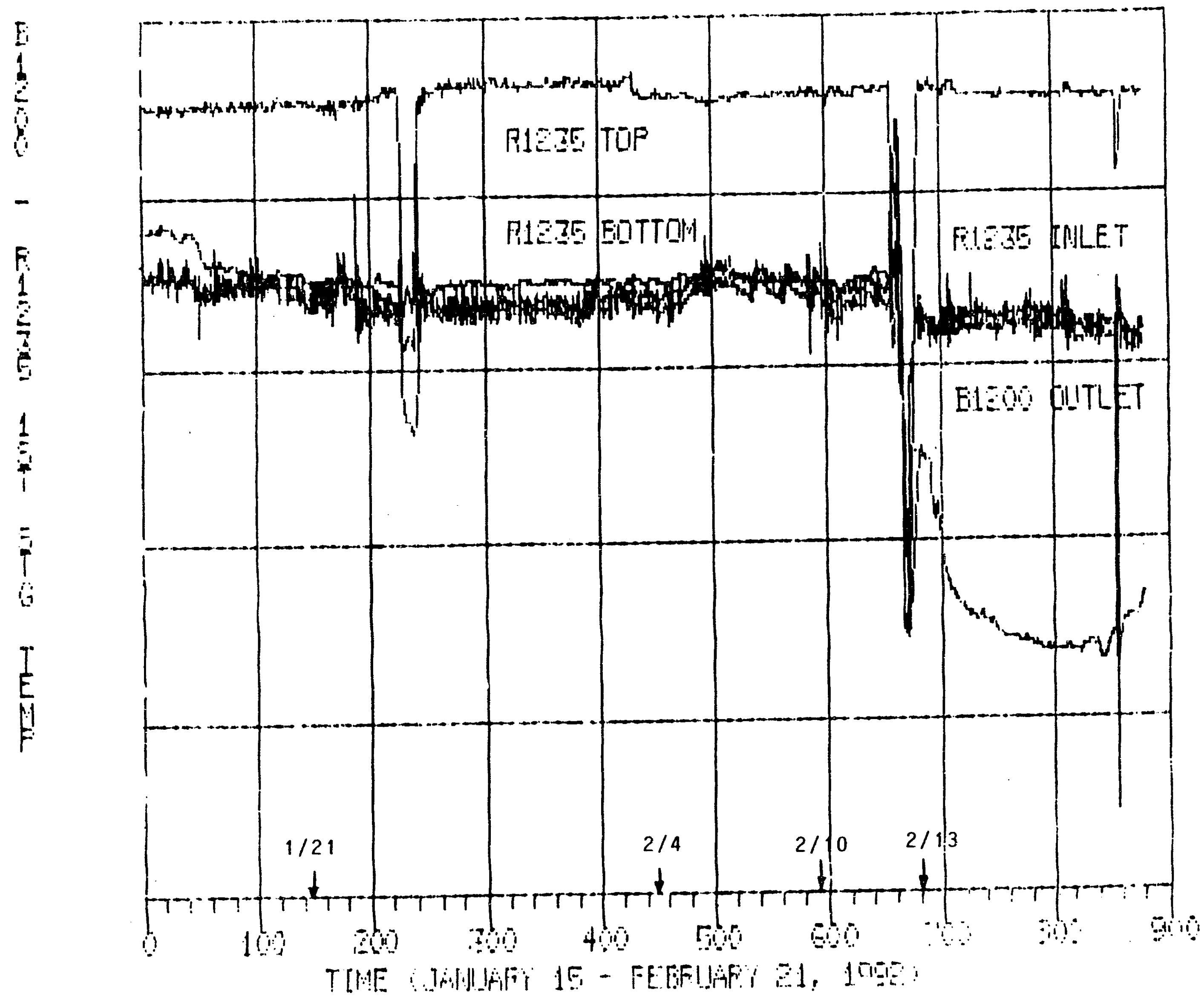


FIGURE 123. PREHEATER OUTLET AND FIRST STAGE REACTOR TEMPERATURE TREND (JANUARY 15 - FEBRUARY 21)

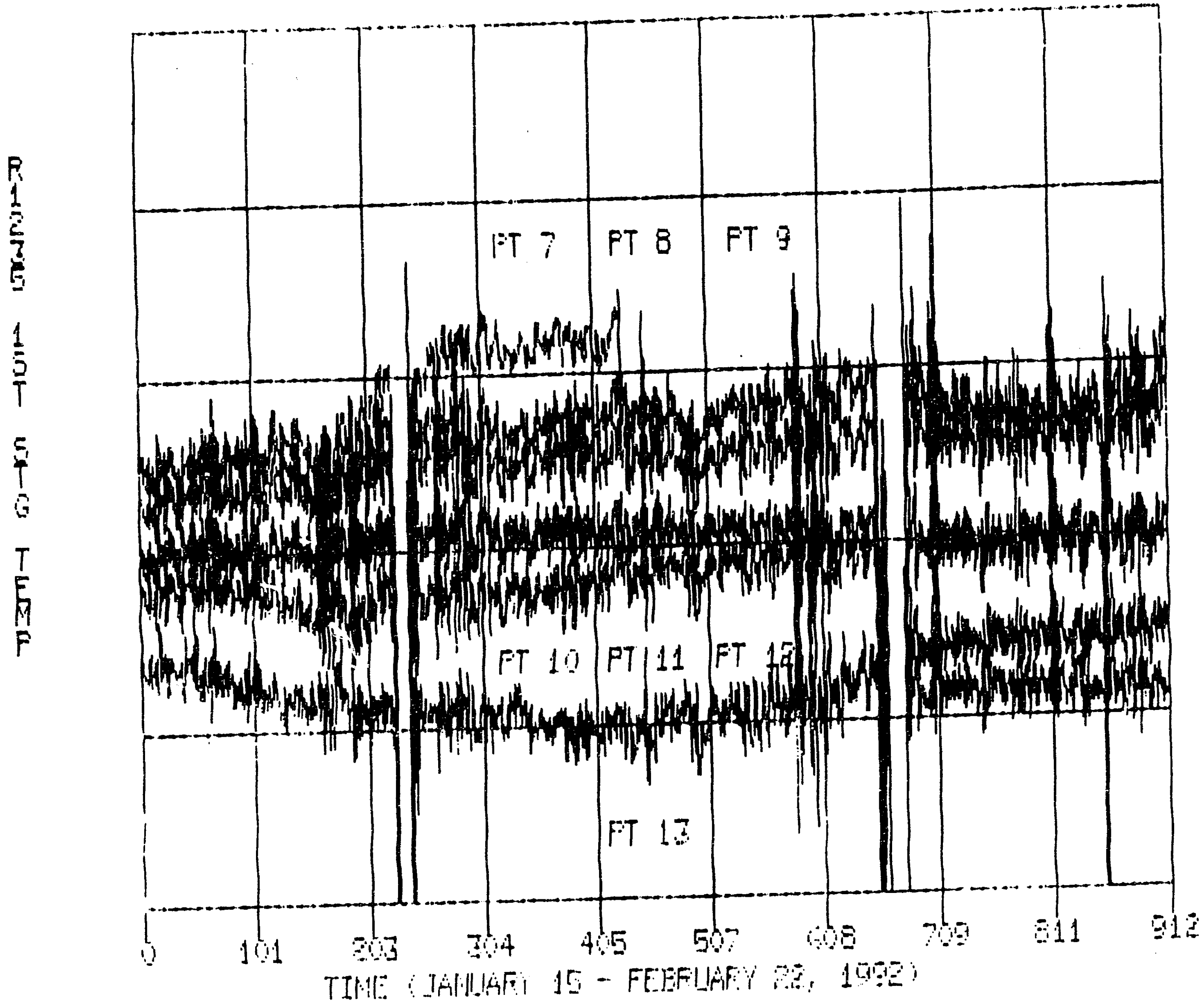


FIGURE 124. FIRST STAGE REACTOR BED TEMPERATURE TREND (JANUARY 15 - FEBRUARY 22)

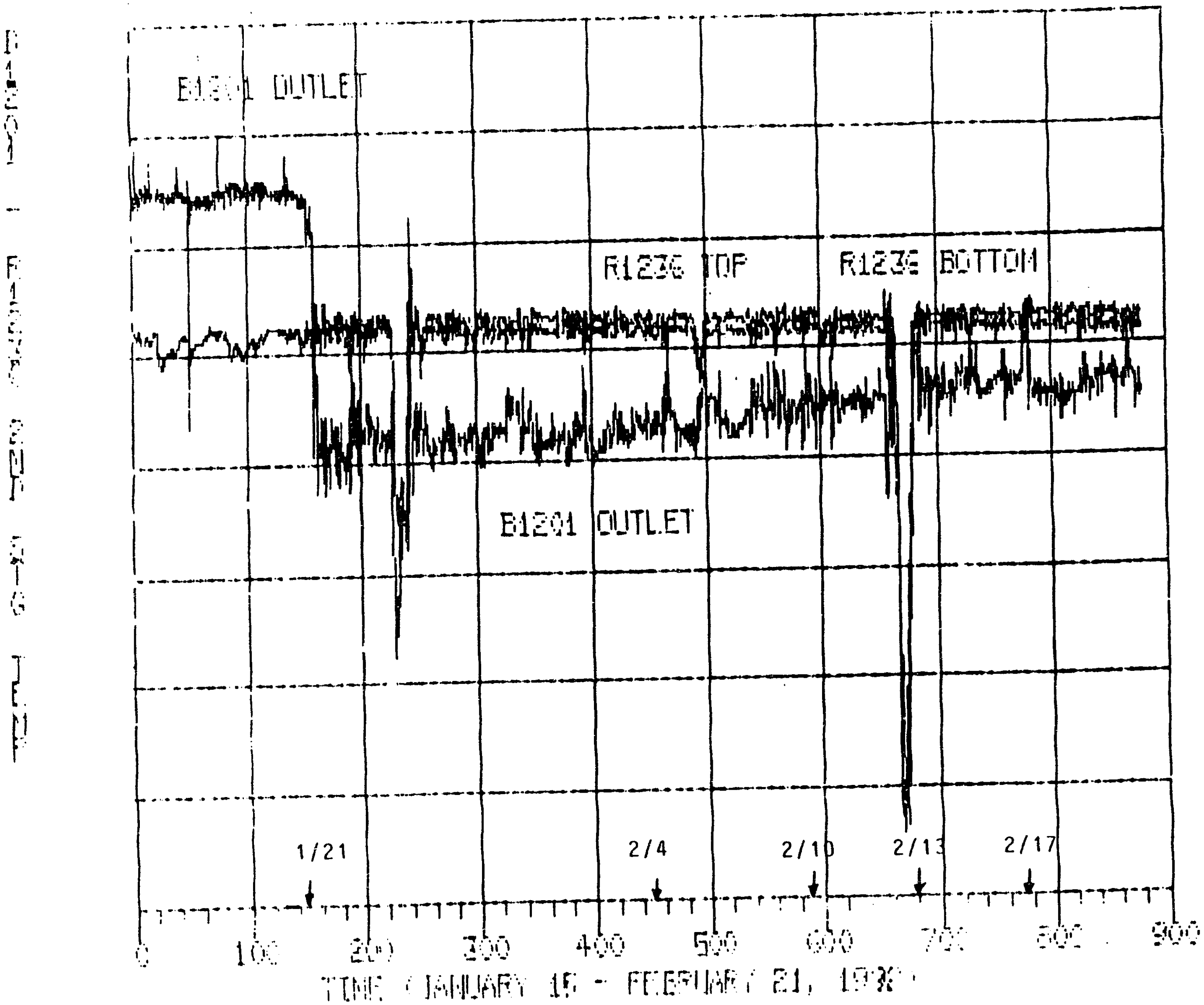


FIGURE 125. INTERSTAGE HEATER OUTLET AND SECOND STAGE REACTOR TEMPERATURE TREND (JANUARY 15 - FEBRUARY 21)

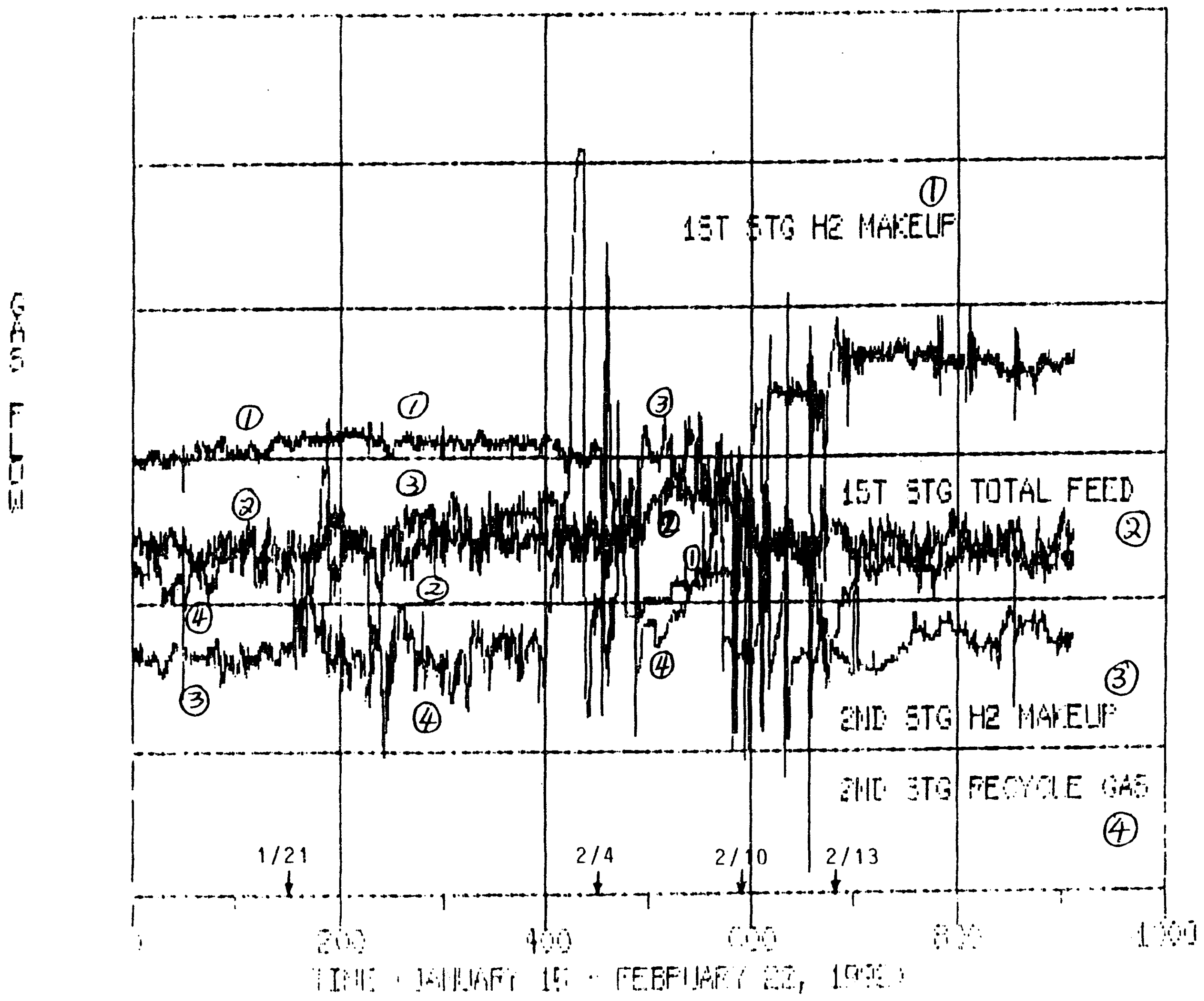


FIGURE 126. GAS FLOW RATE TREND (JANUARY 15 - FEBRUARY 22)

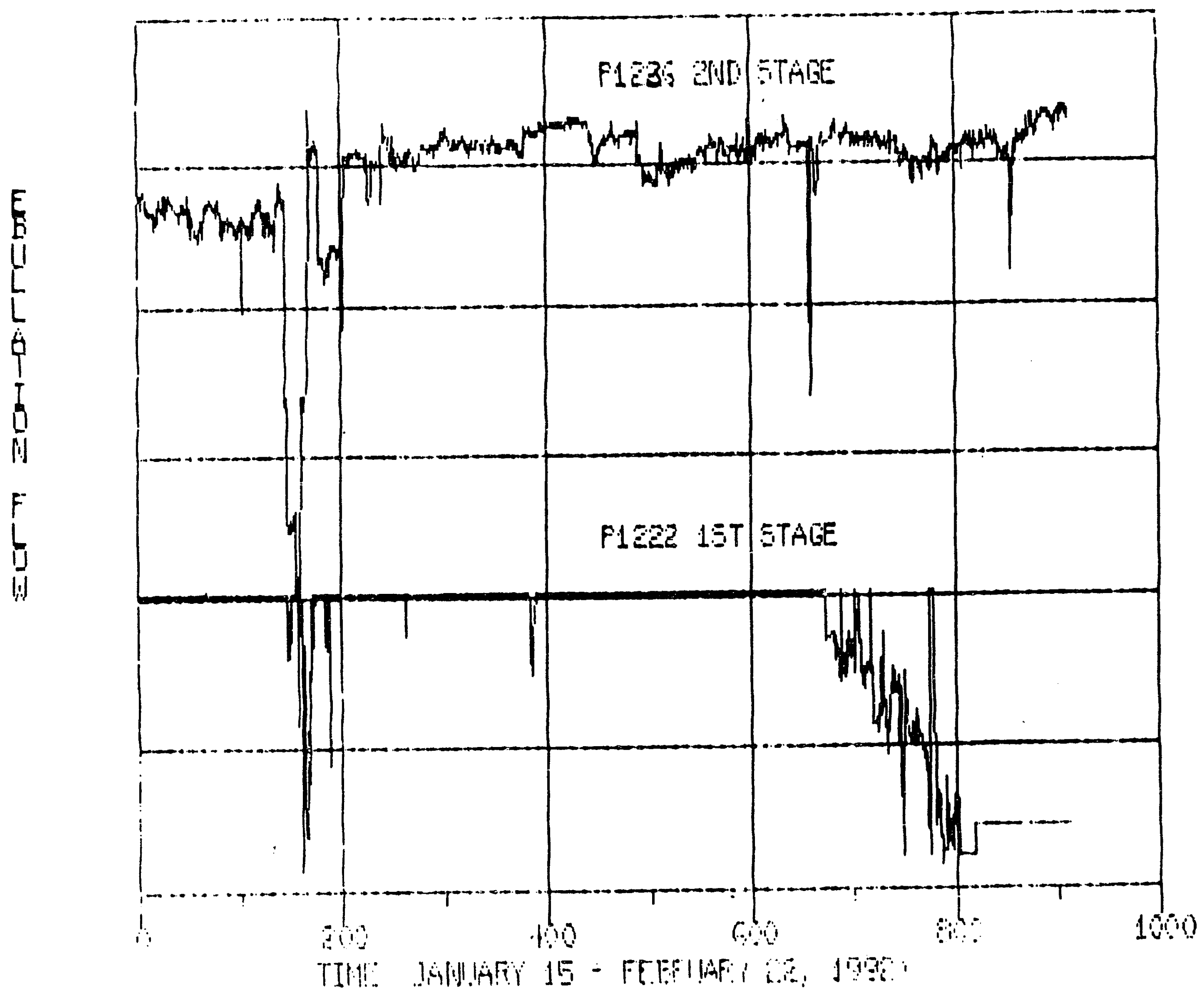


FIGURE 127. EBULLATING AND BACKMIXING PUMP FLOW RATE TREND (JANUARY 15 - FEBRUARY 22)

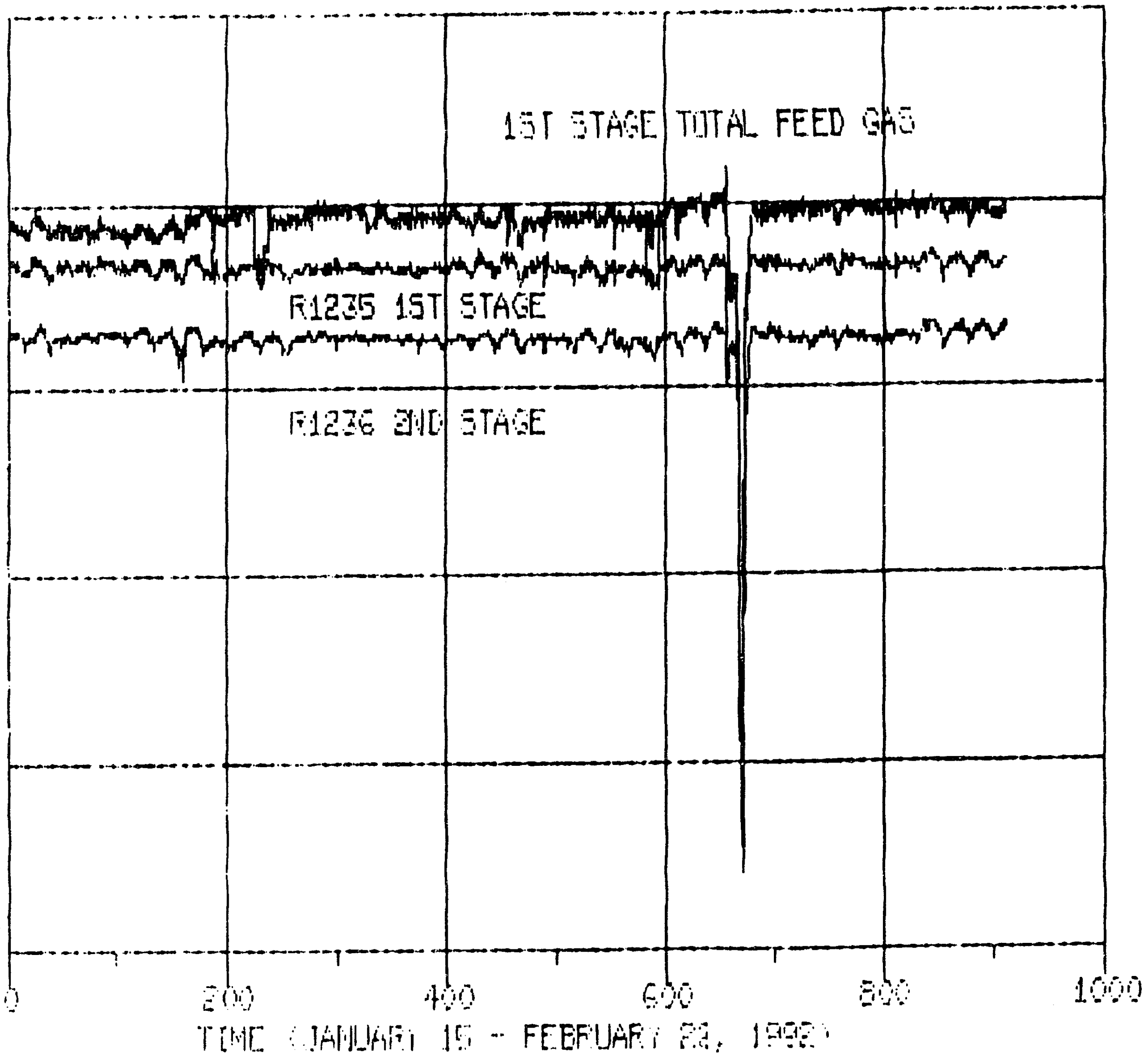


FIGURE 128. SYSTEM PRESSURE TREND OF FEED AND REACTORS (JANUARY 15 - FEBRUARY 22)

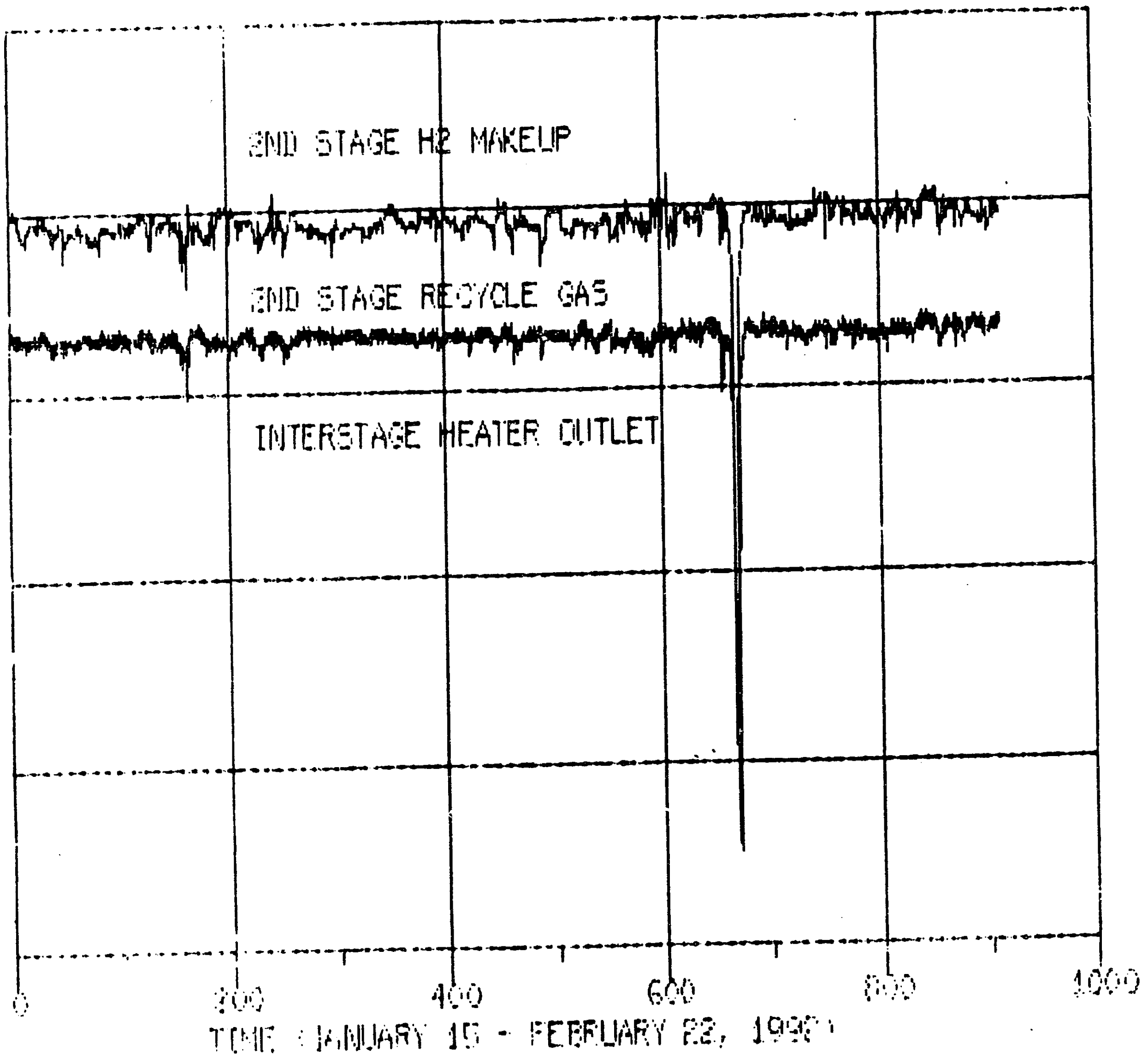


FIGURE 129. SYSTEM PRESSURE TREND OF INTERSTAGE HEATER AND SECOND STAGE FEED (JANUARY 15 - FEBRUARY 22)

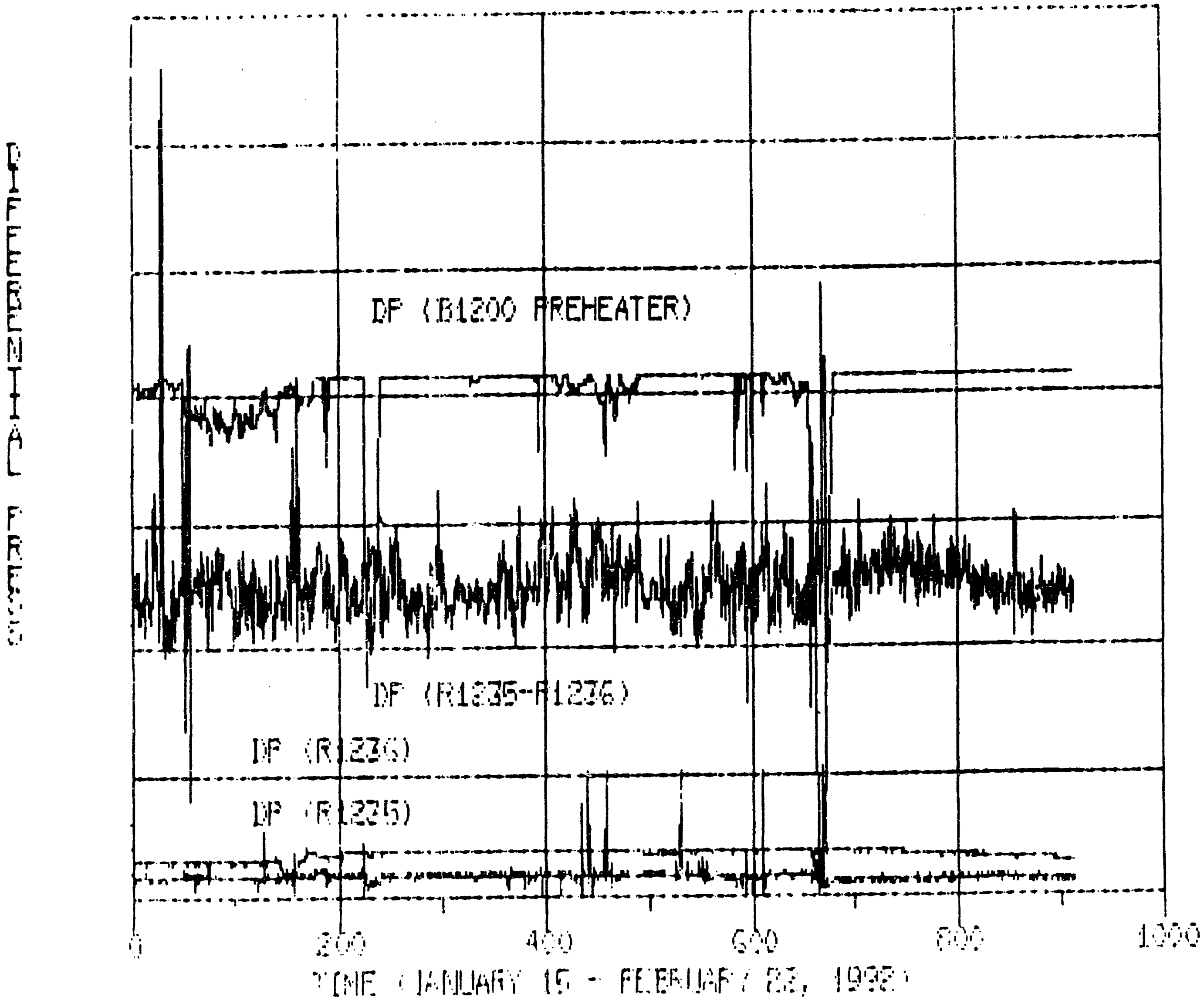


FIGURE 130. DIFFERENTIAL PRESSURE TREND (JANUARY 15 - FEBRUARY 22)

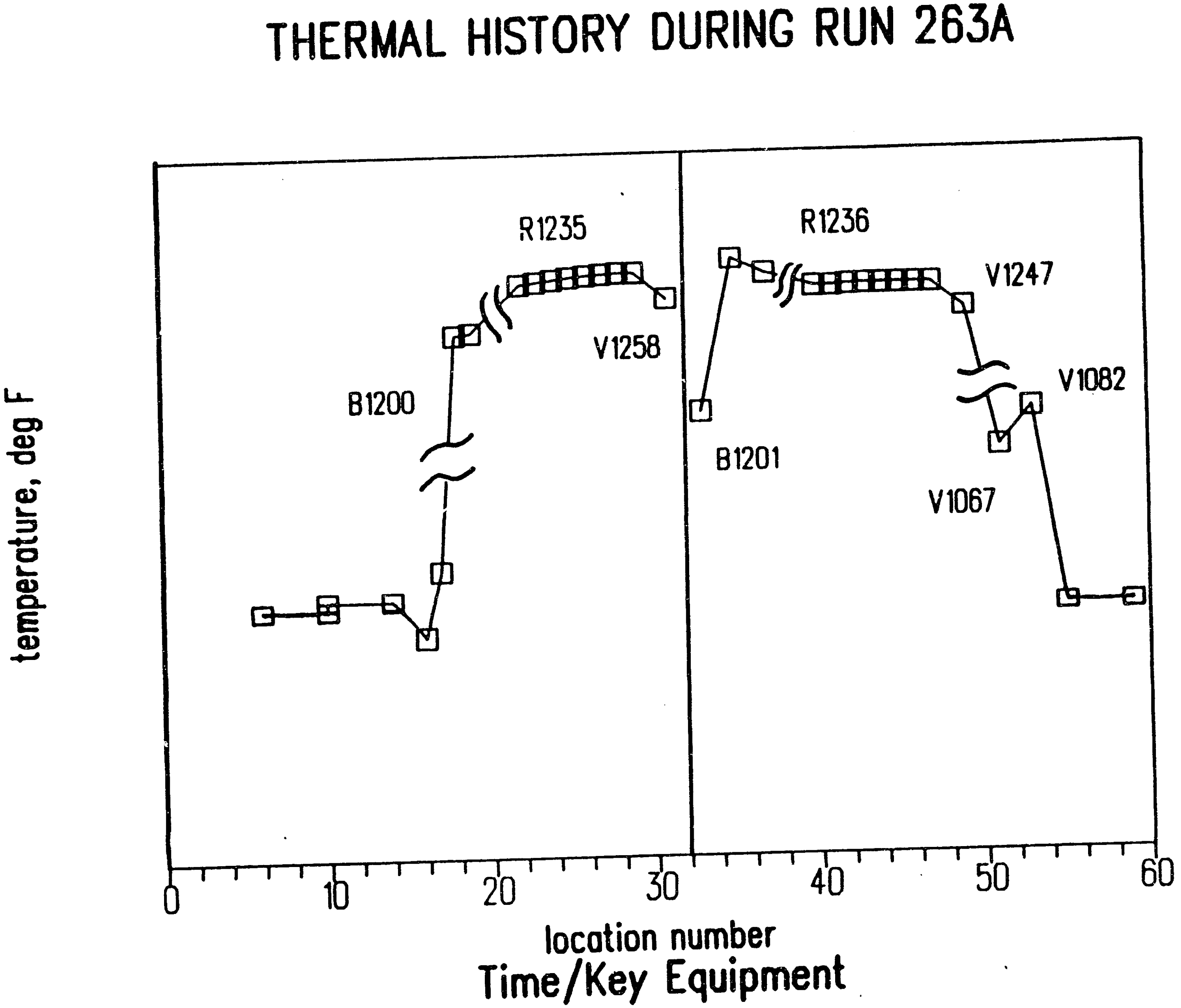


FIGURE 131. TSL THERMAL HISTORY DURING PERIOD 263A

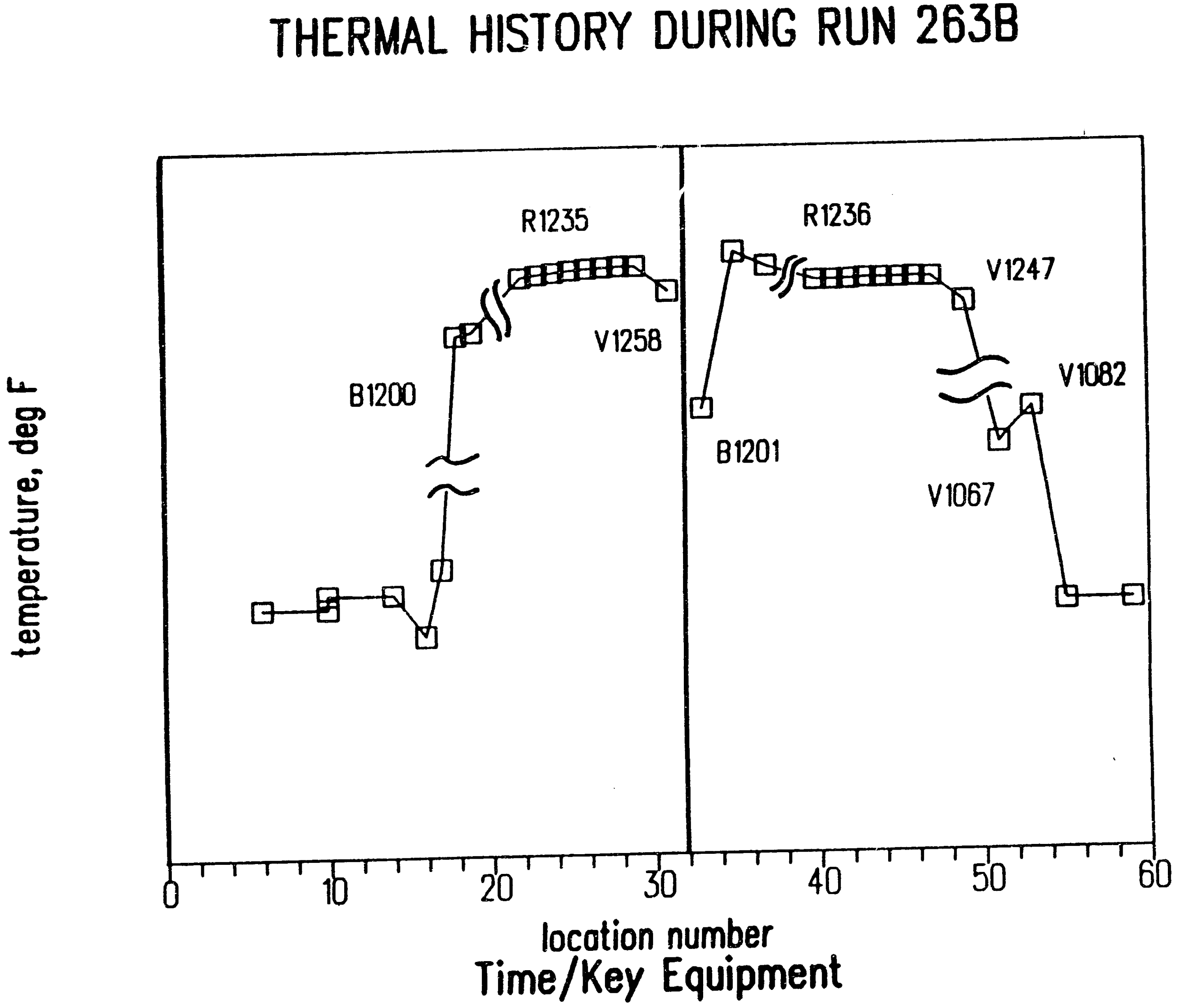


FIGURE 132. TSL THERMAL HISTORY DURING PERIOD 263B

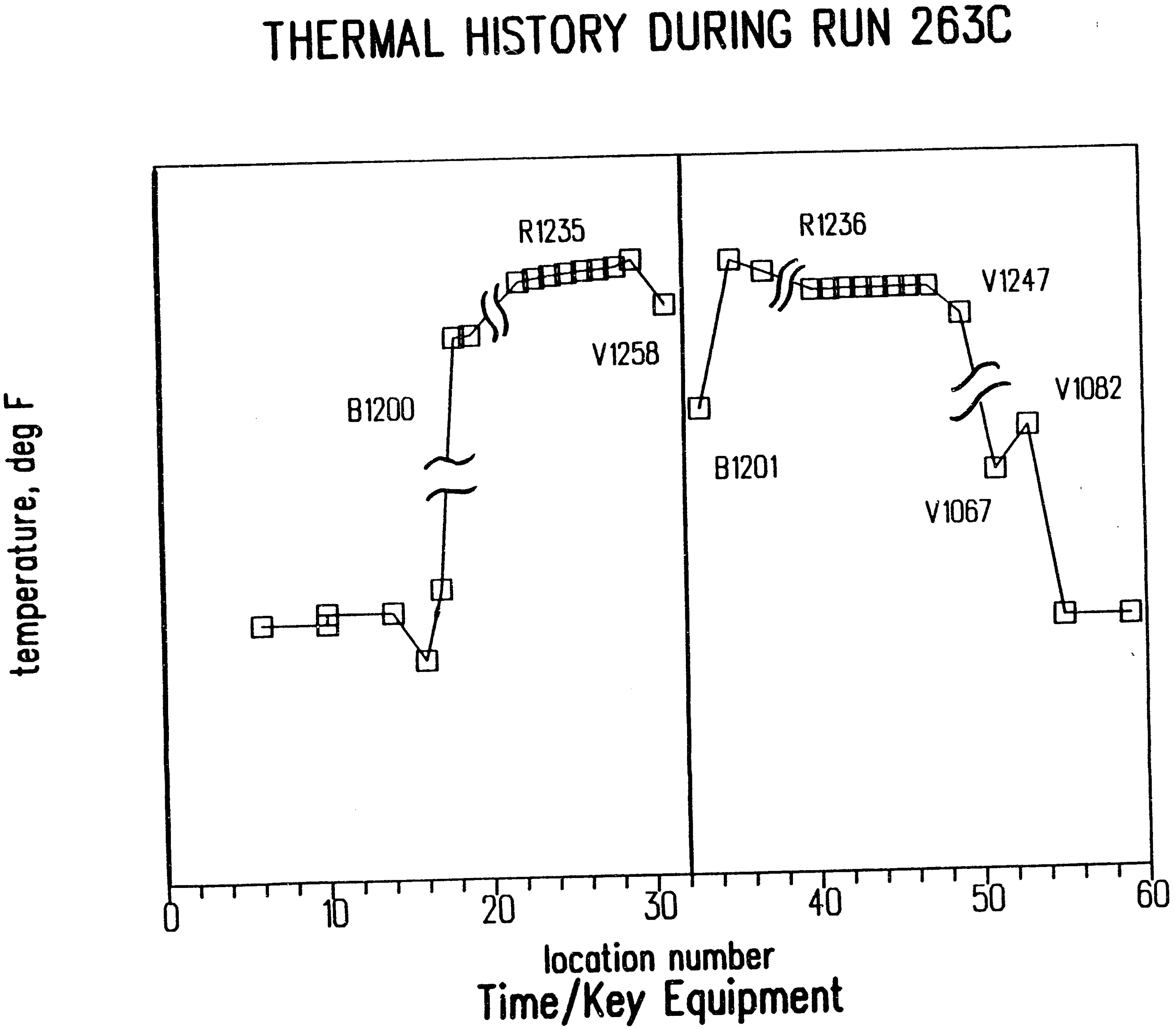


FIGURE 133. TSL THERMAL HISTORY DURING PERIOD 263C

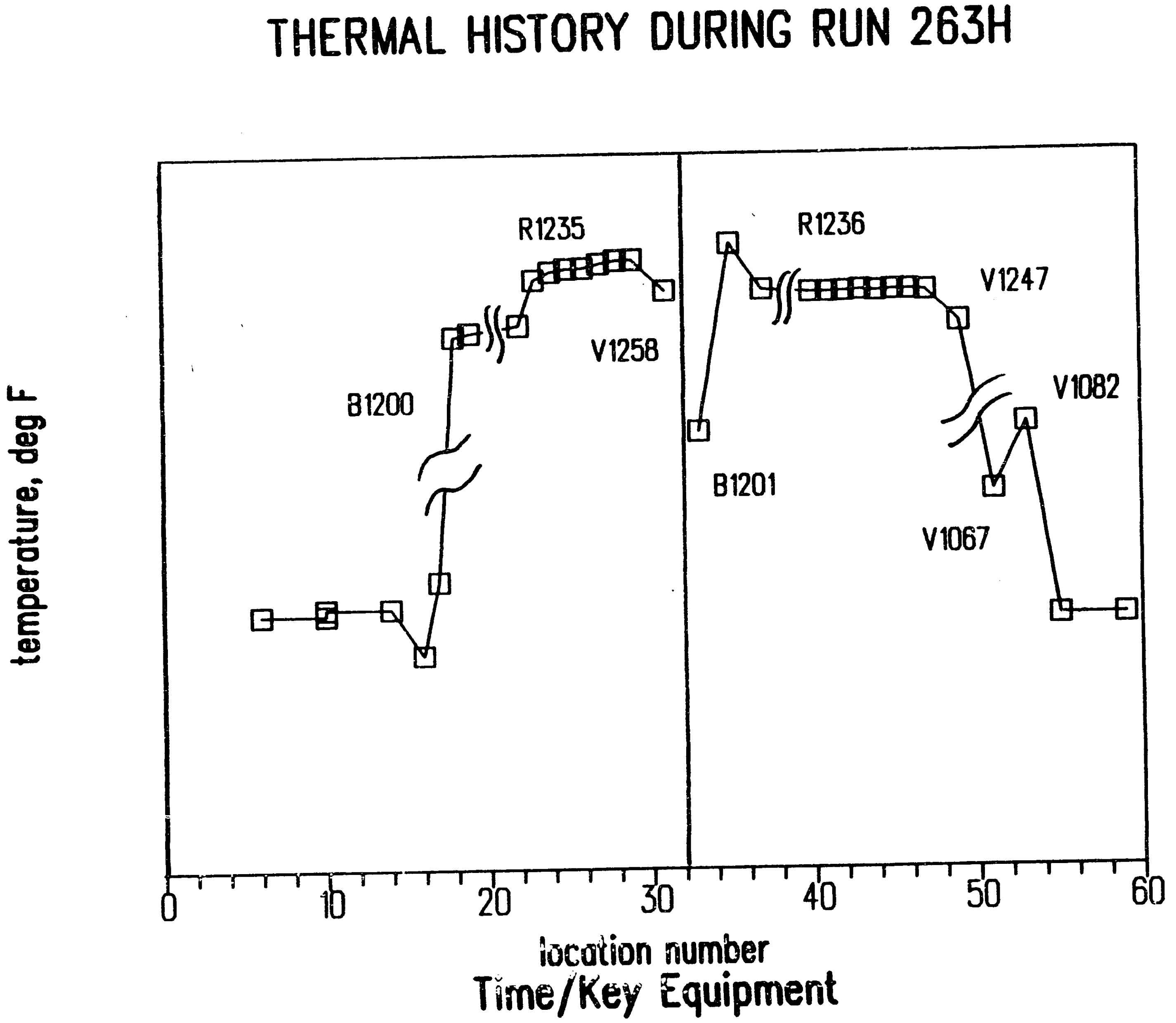


FIGURE 134. TSL THERMAL HISTORY DURING PERIOD 263H

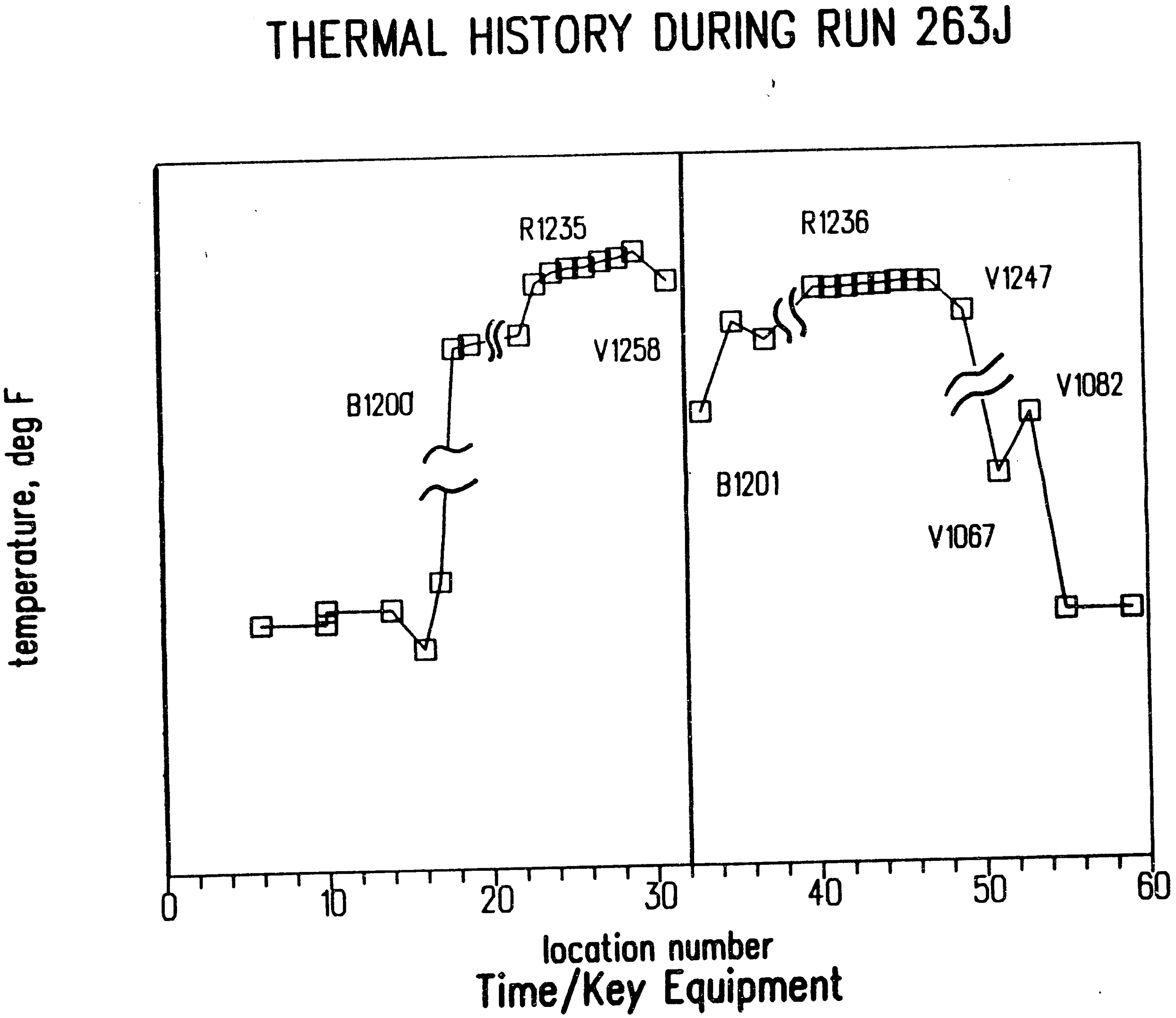


FIGURE 135. TSL THERMAL HISTORY DURING PERIOD 263J

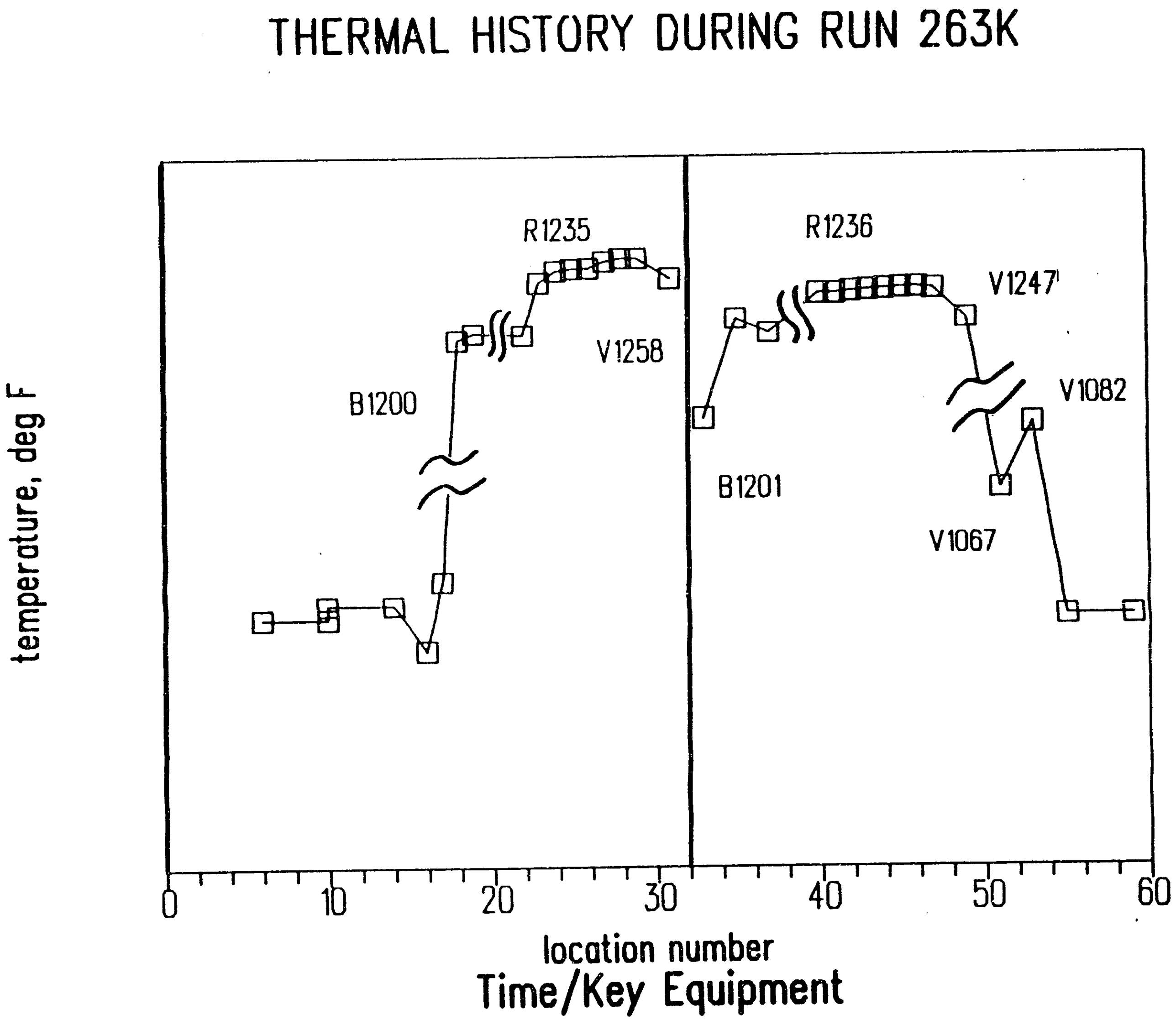


FIGURE 136. TSL THERMAL HISTORY DURING PERIOD 263K

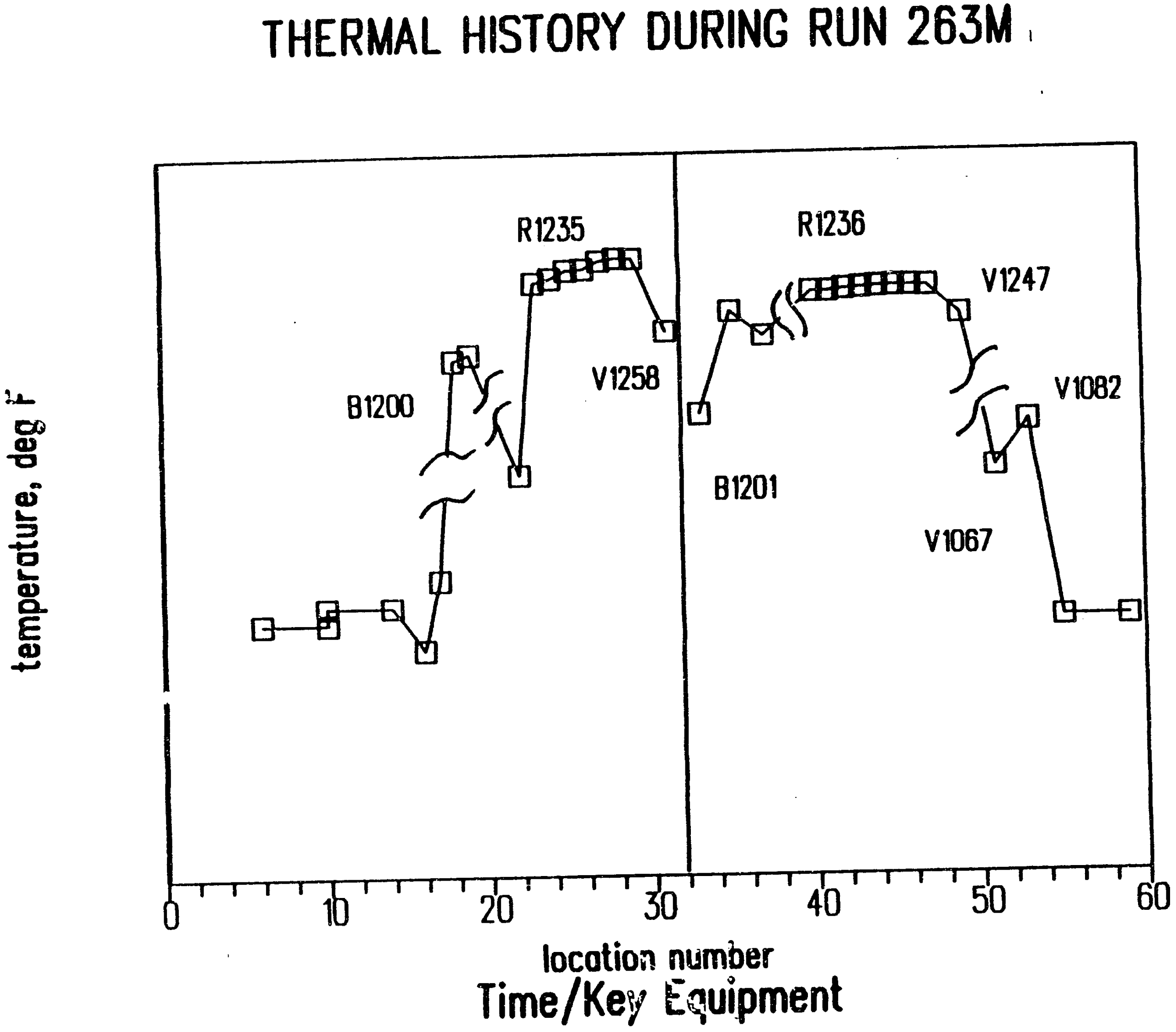


FIGURE 137. TSL THERMAL HISTORY DURING PERIOD 263M

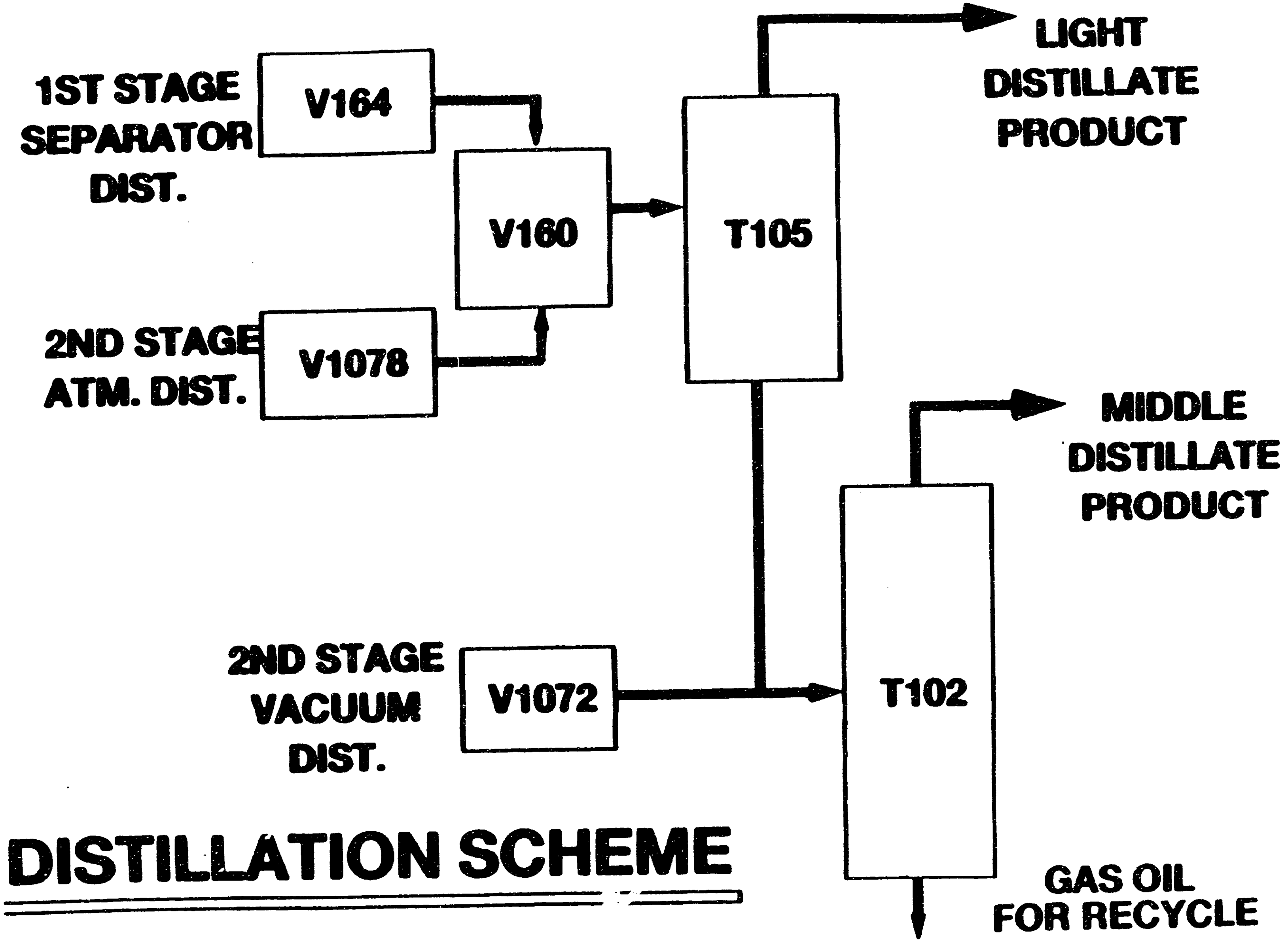


FIGURE 138. SCHEMATIC SHOWING THE FEED AND PRODUCT STREAMS OF THE DISTILLATION COLUMNS

END

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7/22/93