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THE WILSONVILLE ADVANCED COAL LIQUEFACTION RESEARCH AND DEVELOPMENT FACILITY: RUN 254 WITH OHIO NO. 6 COAL

Technical Progress Report

February 1990
Date Published

Work Performed Under Contract No. AC22-82PC50041

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

and
Electric Power Research Institute
Palo Alto, California

By
Southern Electric International
Wilsonville, Alabama

and
Southern Company Services, Inc.
Birmingham, Alabama

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Printed in the United States of America, Office of Scientific and Technical Information, Oak Ridge , Tennessee

DOE/PC/50041--111

DE91 011277

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ADVANCED COAL LIQUEFACTION
RESEARCH AND DEVELOPMENT FACILITY

Wilsonville, Alabama

TECHNICAL PROGRESS REPORT

Run 254 with Ohio No. 6 Coal

Published: February 1990

Prepared by

SOUTHERN ELECTRIC INTERNATIONAL
Wilsonville, Alabama 35186

Subcontractor to

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

Prepared for

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

and

ELECTRIC POWER RESEARCH INSTITUTE
Palo Alto, California 94303

Contract No. RP1234-1-2

PREFACE

Operation of the Advanced Coal Liquefaction R & D Facility at Wilsonville, Alabama, is funded by the U.S. Department of Energy (DOE) and the Electric Power Research Institute (EPRI). Southern Company Services, Inc. (SCS) manages the Wilsonville program on behalf of DOE and EPRI. Catalytic, Inc. operated the facility during Run 254 under a subcontract with SCS.

The Wilsonville R & D Facility combines two process units: a Close Coupled Reactor (CCR) unit and a Critical Solvent Deashing (CSD) 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 operations. The modification primarily consisted of adding a new reactor in close proximity to a pre-existing reactor. These close-coupled reactors can be used for both thermal-catalytic and catalytic-catalytic modes of operation. The CSD unit uses a proprietary solid-liquid separation process developed by the Kerr-McGee Corporation. The process separates ash and unreacted coal (UC) from resid as a heavy fluid phase, termed ash concentrate, using a deashing solvent near its critical point. The combined three-unit system is generally known as a Two-Stage Liquefaction (TSL) process.

The TSL process is an advanced coal liquefaction concept, where the severities in the first and second stages may be independently varied, allowing for improvement in product slate flexibility. Emphasis has been placed on maximum production of low nitrogen distillate products with efficient hydrogen utilization and maximum coal conversion.

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ABSTRACT

This report presents the operating results for Run 254 at the Advanced Coal Liquefaction R & D Facility in Wilsonville, Alabama. This run operated in the Close Coupled Integrated Two-Stage Liquefaction mode (CC-ITSL) using Ohio No. 6 bituminous coal with Shell 317 catalyst. Because this was the first run using Ohio No. 6 coal, it was considered a screening run. The primary test for this run was the demonstration of unit and system operability with Ohio No. 6 coal as compared to similar operation with Illinois No. 6 coal. Evaluation of overall TSL yield structures with both low ash and high ash Ohio No. 6 coals with Shell 317 catalyst were evaluated. Other evaluations made in Run 254 included process solvent composition changes, multiple space velocity tests, second reactor temperature tests, and Amocat 1B catalyst ebullation tests.

Run 254 began on 23 August 1987 and continued through 1 March 1988. During this period 491.6 MF tons of Ohio No. 6 coal were fed in 2,625 hours of operation.

1.0 INTRODUCTION

Run 254 was in the Close-Coupled Integrated Two-Stage Liquefaction (CC-ITSL) mode. Prior runs in the CC-ITSL mode include Runs 250-253 (Ref. 1-4).

In the CC-ITSL mode the product from the first stage reactor is sent to the second stage reactor without prior fractionation or deashing. Interstage cooling and gas separation are possible. The second stage bottoms product is sent to the CSD unit. The deashed resid from the CSD is recycled to the slurry mix tanks along with second stage distillate solvent and second stage ash recycle material. In the reactor section, catalyst in the second reactor undergoes batch aging while catalyst in the first reactor incorporates a catalyst addition/withdrawal program to keep the catalyst activity high. A block flow diagram of CC-ITSL is shown in Figure 1.

A brief summary of recent and future Two-Stage Liquefaction (TSL) runs at Wilsonville is given below:

Run	Coal	Mode	Comments
242	Illinois	ITSL	SCT(1)
243	Illinois	ITSL	
244	Illinois	ITSL	
245	Illinois	ITSL	Continuous HTR catalyst addition/withdrawal
246	Wyoming	DITSL/ITSL	FeS catalyst in TLU(2)
247	Illinois	RITSL	TLU dissolver tracer study
248	Illinois	DITSL/ITSL	Low Contact Time Liquefaction(3)
249	Wyoming	RITSL	Forced Back-mixed Dissolver
250	Illinois	CC-ITSL	Thermal-Catalytic
251	Ill/Wyo.	CC-ITSL	Catalytic-Catalytic and Thermal-Catalytic
252	Illinois	CC-ITSL	Catalytic-Catalytic
253	Illinois	CC-ITSL	Catalytic-Catalytic
254	Ohio #6	CC-ITSL	Catalytic-Catalytic
Future:			
255	Texas Lignite	CC-ITSL	Thermal-Catalytic and Catalytic-Catalytic

(1) SCT denotes Short Contact Time Liquefaction. A dissolver was not used.
(2) Iron oxide and dimethyl disulfide added, which formed FeS catalyst in TLU.
(3) A 5.2" ID dissolver was used.
(4) In Runs 242-250 the first stage is a thermal liquefaction stage and the second stage hydrotreater is a catalytic stage.
(5) In Runs 251-254 both stages contain catalyst.

Figure 2 shows, in block diagram form, all of the TSL operating modes tested at Wilsonville since Run 242. Figures 3-6 are flow diagrams of coal feed operation, the CCR unit, the CSD unit, and the Distillation System.

Objectives

The objectives of Run 254 were:

1. To evaluate unit and overall TSL operating performance and yield structure in the ash recycle mode of operation with 1/20" Shell 317 trilobe catalyst and low ash Ohio No. 6 coal.
2. To evaluate the system performance with Ohio No. 6 coal relative to that with Illinois No. 6 coal in previous operations.
3. To compare the effects of high ash and low ash Ohio No. 6 coals on system performance.
4. To evaluate initial batch catalyst deactivation in both stages.
5. To determine catalyst activation energies in both reactors at a high space velocity.
6. To demonstrate the operating conditions at which an all-distillate product slate can be achieved using low ash Ohio No. 6 coal.

A summary of base operating conditions for the first and second stages during Run 254 is shown below:

Run no.	254
Start date	23 August 1987
End date	1 March 1988
Coal	Ohio No. 6
Mine	Crooksville
I. CCR Unit	
Slurry conc., wt % MF	32.9(a)(g),30(f),29.0(h), 31.9 (i),30.9(j)
Coal feed rate, MF lb/hr	493(a),205(b),380(c), 349(d),320(h),485(i), 444(j),363(k),300(n), 424(o),384(l),355(r)
Process solvent, wt %	
CI	12
Resid	39(a),(51)(g),(45.7-49.5)(h), (41.7-49.2)(i),48.8(j),49.3(l)
1st Stage, R1235 Reactor	
Inlet hydrogen partial pressure, psi	2705,(a)2566(b),2600(c), 2643(d),2650(i),2725(k) 2700(n)(p),2745(o),2760(q),2750(r)
Temperature, °F	810
Space velocity, WHSV, hr^{-1}	5.1(a), 2.2(b),4.0(c), 3.8(d),5.2(i),4.2(k) 3.5(n),5.1(o),4.3(q)
2nd Stage, R1236 Reactor	
Temperature, °F	761(a)(h),788(e),790(m),810(p)
Space velocity, WHSV, hr^{-1}	4.9(a),2.2(b),3.9(c),3.8(d), 5.2(i),4.3(k)(q), 3.5(n),5.1(o)
II. CSD Unit	
DAS type	4100;2054;2104;2254;2304; 2454;2504;2554;2354;2604;2654;2704

Note: The operation mode for Run 254 was CC-ITSL with a fresh sulfided Shell 317 catalyst charged to both reactors. The interstage separator was not in use during this period.

(a) Startup on 23 August at 500 MF lb/hr.
(b) Startup 16 September at 200 MF lb/hr.
(c) Raised feed rate to 385 MF lb/hr on 20 September.
(d) Lowered coal feed rate to 373 MF lb/hr on 9 October.
(e) Raised R1236 average target temperature to 790°F on 14 October.
(f) Used 30% coal in slurry for four hours of startup operation on 14 October.
(g) Diluted process solvent to 51% resid on 27 October prior to shutdown.
(h) Startup on 3 December at 500 MF lb/hr with 30% coal in slurry. Diluted process solvent resid concentration.
(i) Startup on 10 December increased to 33% coal in slurry. Diluted process solvent resid concentration.
(j) Lowered coal feed rate to 420 MF lb/hr on 14 December. Diluted slurry to 30% coal and process solvent to 55 wt % resid.
(k) Lowered coal feed rate to 375 MF lb/hr on 18 December.
(l) Changed process solvent resid target to 50 wt % resid on 22 December.
(m) Raised R1236 average target temperature to 790°F on 31 December.
(n) Lowered coal feed rate to 300 MF lb/hr on 20 January.
(o) Increased coal feed rate to 450 MF lb/hr on 31 January.
(p) Raised R1236 average target temperature to 810°F on 10 February.
(q) Lowered coal feed rate to 375 MF lb/hr on 17 February.
(r) Lowered coal feed rate to 363 MF lb/hr on 26 February for Ebullation Tests 254X.

2.0 CONCLUSIONS AND RECOMMENDATIONS

2.1 Conclusions

- Successful operations processing Ohio No. 6 coals were demonstrated by using Shell 317 catalyst in the close-coupled ITSL catalytic-catalytic configuration.

- Best process performance was achieved processing low ash (6 wt %) Ohio No. 6 coal. The distillate yield was 78 wt % MAF coal, the coal conversion was 97 wt % and the organic rejection was 7 wt %. These excellent process achievements can be attributed to: (1) high coal conversion activity of the low ash Ohio coal, (2) high distillate selectivity of the second stage reaction temperature at 790°F, and (3) additional resid recycle.

- The resid from Ohio No.6 coal is more difficult to convert to distillate than the resid from Illinois No. 6 coal. Recycle streams for Ohio No. 6 coal contained more heavy distillate. (More than 97% of V1067 indicated a boiling point above 650°F.)

- Cleaning the Ohio No. 6 coal from 9-11 wt % ash to 6-7 wt % ash with heavy media reduced the amount of organics rejected from the CSD unit by 2 lb organics/lb ash in coal. The reduction in organic loss to ash concentrate increased the potential distillate yield from 70 to 78 wt % MAF coal. However, the additional distillate yield comes at the expense of higher reactor temperatures, and/or lower throughput rates, and/or higher catalyst replacement rates.

- The high second stage reaction temperatures which were investigated, 790°F and 810°F, resulted in increased coal throughput and increased distillate production. The hydrogen content of the resid was reduced significantly.

- When the amount of resid in the recycle stream increased, the distillate production improved. When the 50 wt % resid recycle replaced the 40 wt %, the C_4+ distillate yield increased by 6 wt % MAF coal and the hydrogen efficiency improved from 10.3 to 11.1 lb (C_4+ dist/lb H_2 consumed). The quality of the distillate product was effected with a lower hydrogen content.

- The increased gas-oil recycle produced lighter and better quality distillates. All distillate product streams are low in sulfur (<0.06 wt % sulfur).

- The activation energy of Ohio No. 6 coal was 47,800 Btu/lb-mole for the second stage catalyst in the temperature range of 760-790°F. This value was slightly higher than the 42,300 Btu/lb mole which was calculated for Illinois No. 6 coal. The activation energy for Ohio No.6 in the temperature range of 790-810°F was much higher, 69,000 Btu/lb-mole.

- The higher second stage temperature lowered the hydrogen efficiency and distillate selectivity by increasing the C_1-C_3 gas yield by 2 wt % MAF coal.

- High ash (10 wt %) Ohio No. 6 coal had a higher potential liquid yield than high ash (11 wt %) Illinois No. 6 coal. The 2-4 wt % MAF coal difference in C_4+ resid is primarily due to the lower gas make (H_2S, CO_x and C_1-C_3).

- Reactor ebullating performance test results showed that better reactor ebullation can be achieved with the larger Amocat 1B (1/10") catalyst than with the Shell 317 (1/20") catalyst. However, the larger catalyst seems to be significantly damaged when subjected to coal slurry in the reactor.

2.2 Recommendations

- Investigate low ash Illinois No. 6 coal or the feasibility of cleaning Illinois No. 6 coal to a low ash content. The lower ash would increase the potential liquid yield and the use of Illinois No. 6 coal would produce more distillate.

- Due to the number of problems associated with reactor plugging in the plenum chamber (HRI proprietary design) and the resulting catalyst breakage, the design of the plenum chamber should be improved to eliminate catalyst backflow through the distributor.

- Investigate larger and denser catalyst to improve reactor ebullating performance. For Run 254 the ebullating flow was too low for the 1/20" Shell 317 catalyst. With the high CI recycle (12% CI and 50% resid in the process solvent) a lower ebullation rate was maintained to prevent catalyst carryover. The low flow rate caused high temperature rises within the catalyst bed and poor catalyst distribution. Since the Shell 317 catalyst has shown little breakage even at high catalyst ages, acquisition of a larger Shell 317 catalyst should be explored.

- Compare Shell 324 unimodal catalyst to Shell 317 bimodal catalyst.

- Develop kinetic expressions to interpret the apparent second stage catalyst deviation at low coal feed rates from the CSTR first order kinetics. Three possible explanations for this deviation are: (1) interaction of feed reactivity, (2) second order kinetics, and (3) two different coal feeds.

- Determine steady-state catalyst performance with catalyst addition/withdrawal in both stages. Test catalyst cascading to determine potential catalyst savings.

- Develop close-coupled ITSL process models and evaluate process economics.

3.0 OPERATING DATA AND PROCESS PERFORMANCE

Run 254 began on 23 August with Ohio No. 6 bituminous coal in the CC-ITSL mode of operation. Three hundred pounds of fresh sulfided Shell 317 catalyst was charged to each reactor. The CSD unit was used to purge ash from the system and to recover distillate solvent and resid for blending in the process solvent. Because the ash recycle operation was continued, the V1067 atmospheric bottoms containing the ash and unconverted coal (UC) that were not purged were recycled back to the coal feed system. R1235 reactor was used as the first stage and R1236 reactor was used as the second stage. The addition/withdrawal procedure for the first stage reactor started on 9 October at a level of 3 lb catalyst/ton MF coal. Fresh sulfided Shell 317 catalyst was added.

Run 254 continued with Ohio No. 6 coal on 3 December in the same configuration. The purpose of the continuation was to obtain a set of operating conditions that would yield an all-distillate product slate using Ohio No. 6 coal. Three hundred pounds of fresh sulfided Shell 317 catalyst was charged to each reactor and the catalyst replacement program resumed on 20 December.

During Run 254, the Close-Coupled Reactor unit operated 2625 hours out of a total of 3112 hours, for an on-stream factor of 84.4%.

3.1 TSL System Stability

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

- Mass balance closure errors for TSL, CCR (first and second stages), and CSD must be less than 10 wt % MAF coal.

- The sum of absolute values of inventory changes including drum-outs must be less than 15 wt % MAF coal for the following locations:

    a) Between the second stage and CSD unit
    b) Between the CSD unit and the first stage
    c) Between the second stage and the first stage

A description of the elemental balancing procedure and a more detailed description of the selection criteria are given in Appendix C, Material Balance Methodology. Yields for stable days with similar operating conditions were averaged to obtain the final characteristic yields for each set of operating conditions. Forty elemental balances were included in the characteristic yields. Periods (254A, 254C, 254E) that did not meet the selection criteria for plant performance stability are labeled as transitional. The operating conditions for each material balance period are shown below:

Run 254 Operating Periods

Operating Period	Description	Number of Selected Days	Elemental Balance Days (1987-88)
254A	Transitional Coal: Ohio No. 6 MF wt % ash = 10.2 MF coal feed rate, lb/hr = 495 Coal concentration, wt % = 32.9 Recycle process solvent: Wt % resid = 39 Wt % CI = 12 Reaction temperature (°F): 1st stage temp. = 810 2nd stage temp. = 760 No catalyst addition/withdrawal CSD DAS type = 4100	2	Aug 29,30
254B	Coal: Ohio No. 6 MF wt % ash = 9.5 MF coal feed rate, lb/hr = 385 Coal concentration, wt % = 32.8 Recycle process solvent: Wt % resid = 38 Wt % CI = 12 Reaction temperature (°F): 1st stage temp. = 811 2nd stage temp. = 760 No catalyst addition/withdrawal CSD DAS type = 2104-2504	6	Sep 23,24,25,28 29,30
254C	Transitional Coal: Ohio No. 6 MF wt % ash = 6.0 MF coal feed rate, lb/hr = 365 Coal concentration, wt % = 32.4 Recycle process solvent: Wt % resid = 39 Wt % CI = 12 Reaction temperature (°F): 1st stage temp. = 811 2nd stage temp. = 761 1st stage catalyst addition/withdrawal CSD DAS type = 2554-2604	2	Oct 9,10

Run 254 Operating Periods (continued)

254D	Coal: Ohio No. 6 MF wt % ash = 6.2 MF coal feed rate, lb/hr = 375 Coal concentration, wt % = 32.7 Recycle process solvent: Wt % resid = 39 Wt % CI = 12 Reaction temperature (°F): 1st stage temp. = 811 2nd stage temp. = 789 1st stage catalyst addition/withdrawal CSD DAS type = 2554-2604	4	Oct 16,18,19,20
254E	Transitional Coal: Ohio No. 6 MF wt % ash = 6.4 MF coal feed rate, lb/hr = 485 Coal concentration, wt % = 32.2 Recycle process solvent Wt % resid = 50 Wt % CI = 11 Reaction temperature (°F): 1st stage temp. = 810 2nd stage temp. = 761 No catalyst addition/withdrawal CSD DAS type = 2304	2	Dec 13,14
254F	Coal: Ohio No. 6 MF wt % ash = 7.7 MF coal feed rate, lb/hr = 365 Coal concentration, wt % = 29.9 Recycle process solvent: Wt % resid = 50 Wt % CI = 12 Reaction temperature (°F): 1st stage temp. = 811 2nd stage temp. = 760 1st stage catalyst addition/withdrawal CSD DAS type = 2304	4	Dec 26,27,28,29

Run 254 Operating Periods (continued)

254G	Coal: Ohio No. 6 MF wt % ash = 6.3 MF coal feed rate, lb/hr = 367 Coal concentration, wt % = 29 Recycle process solvent: Wt % resid = 49 Wt % CI = 12 Reaction temperature (°F): 1st stage temp. = 811 2nd stage temp. = 790 1st stage catalyst addition/withdrawal CSD DAS = 2304	4	Jan 1,2,3,4
254H	Coal: Ohio No. 6 MF wt % ash = 6.5 MF coal feed rate, lb/hr = 364 Coal concentration, wt % = 29 Recycle process solvent: Wt % resid = 49 Wt % CI = 12 Reaction temperature (°F): 1st stage temp. = 811 2nd stage temp. = 790 1st stage catalyst addition/withdrawal CSD DAS = 2454-2504	3	Jan 15,16,17
254I	Coal: Ohio No. 6 MF wt % ash = 5.9 MF coal feed rate, lb/hr = 297 Coal concentration, wt % = 29 Recycle process solvent: Wt % resid = 51 Wt % CI = 12 Reaction temperature (°F): 1st stage temp. = 811 2nd stage temp. = 790 1st stage catalyst addition/withdrawal CSD DAS type = 2604	2	Jan 29,30

Run 254 Operating Periods (continued)

254J	Coal: Ohio No. 6 MF wt % ash = 5.6 MF coal feed rate, lb/hr = 438 Coal concentration , wt % = 29 Recycle process solvent: Wt % resid = 49 Wt % CI = 12 Reaction temperature (°F): 1st stage temp. = 810 2nd stage temp. = 790 1st stage catalyst addition/withdrawal CSD DAS type 2654-2704	3	Feb 6,7,8
254K	Coal: Ohio No. 6 MF wt % ash = 5.4 MF coal feed rate, lb/hr = 440 Coal concentration, wt % = 29 Recycle process solvent: Wt % resid = 49 Wt % CI = 12 Reaction temperature (°F): 1st stage temp. = 810 2nd stage temp. = 810 1st stage catalyst addition/withdrawal CSD DAS type = 2654	5	Feb 11,13,14,15,16
254L	Coal: Ohio No. 6 MF wt % ash = 5.5 MF coal feed rate, lb/hr = 373 Coal concentration, wt % = 30 Recycle process solvent: Wt % resid = 50 Wt % CI = 12 Reaction temperature (°F): 1st stage temp. = 810 2nd stage temp. = 810 1st stage catalyst addition/withdrawal CSD DAS type 2704	3	Feb 20,21,23

3.2 Feed Coal

In Run 254, Ohio No. 6 high ash coal and Ohio No. 6 (cleaned) low ash coal were evaluated. The process solvent used for slurry preparation was V1074 distillate solvent, full range CSD resid, and V1067 atmospheric bottoms. This mixture was blended in V101A (the Slurry Blend Tank), mixed with coal, and fed to the first stage reactor. The coal slurry blend started with 39% resid in the process solvent and a 2.3:1 solvent-to-coal ratio. The process solvent composition was gradually increased to 50% resid and the solvent-to-coal ratio was strengthened to 2:1.

Feed coal introduced to the process after 29 September was ground in a fluid energy mill. About 20% of the coal was larger than 200 mesh. The previous grinding method used a roller mill and produced a grind 93-95% finer than 200 mesh.

Averaged data for Run 254 are presented below:

Run Period	254A-B 29 Aug-30 Sep 1987	254C-L 9 Oct-23 Feb 1988
Coal analysis, wt % MF		
Carbon	72.95±0.98	75.36±1.32
Hydrogen	4.94±0.25	5.32±0.07
Nitrogen	1.19±0.25	1.51±0.04
Sulfur	3.08±0.40	2.56±0.32
Oxygen (by difference)	8.02±1.39	9.11±1.15
Ash	9.85±0.53	6.17±0.66
Sulfur forms, wt % MF		
Pyritic	1.94	1.54
Sulfate	0.19	0.02
Organic	1.63	1.07
Chlorine wt % MF	0.02	0.02
Volatile matter as-is wt %	35.84	38.80
Ferric oxide in ash, %	28.02	29.74
H/C atomic ratio	0.77-0.84	0.83-0.86

Coal conversion averaged 94.0±0.6% with high ash coal (8.8-11.4% ash) and 96.5±0.4% with low ash coal (5.2-6.8% ash). The total sulfur content was 2.11-3.62 wt %. Detailed analyses of Ohio No. 6 coal including minerals, sulfur forms, proximate, and ultimate analyses are presented in Table 1. Additional elemental analysis for process solvent used in coal slurry mixing is presented in Table 2.

3.3 Close-Coupled Reactor Unit (CCR)

The primary objectives of CCR unit operations during Run 254 were:

1) To evaluate unit operating performance and yield structure while processing Ohio No. 6 coal with 1/20" Shell 317 trilobe catalyst in both reactors.

2) To compare unit performance with high ash Ohio No. 6 coal and (cleaned) low ash Ohio No. 6 coal relative to performance with Illinois No. 6 coal.

3) To evaluate both the initial batch catalyst deactivation in both stages and the catalyst addition/withdrawal in the first stage at a rate of three pounds of fresh sulfided catalyst per ton of MF coal feed.

4) To determine a set of operating conditions at which an all-distillate product slate could be achieved using low ash Ohio No. 6 coal.

Coal slurry composition, coal feed rate, and reactor temperatures were adjusted to produce a minimum resid yield. The coal conversion, hydrogen consumption, and resid yield were monitored on a frequent basis as performance indicators. Catalyst samples were taken on a routine basis to monitor the catalyst integrity.

Operations

Run 254 with Ohio No. 6 coal began on 23 August with fresh Shell 317 catalyst in both reactors. Unit startup conditions were:

MF Coal feed rate = 500 lb/hr
Slurry composition = 33% coal
67% process solvent
Process solvent = 38% resid
12% CI
50% heavy distillate solvent
1st stage average temperature = 810°F
1st stage inlet H_2 partial pressure = 2650 psia
2nd stage average temperature = 760°F

First startup attempts were hampered by a leaking flange on the second stage reactor inlet. Repairs were made and coal feed resumed on 25 August. This first period of operation served as an Ohio No. 6 solvent equilibration period and as an initial catalyst deactivation period. Period 254A was chosen during 29-30 August to characterize the transition period. Operation was interrupted on 31 August after an emergency shutdown situation required maintenance to open and inspect the ebullation pump on the second reactor. The catalyst was recharged to the second reactor and all repairs were made by 5 September but coal feed was not started until 16 September due to an interruption in the coal supply. Operation resumed at an MF coal feed rate of

200 lb/hr until the coal inventory was fully available. The coal feed rate was increased slowly over 19-20 September to 385 lb/hr. Period 254B was chosen during 23-25 and 28-30 September as a stable operation period on high ash coal.

An orderly shutdown was required on 1 October to repair the interstage sampler. A larger sampler had been installed at the start of Run 254, making the sample easier to collect and the results more reliable. However, the hand valves were easily eroded by the high velocity resulting from initial pressure surges between the process line and the sampler. Since the leaking valves posed a safety problem, the sample was not collected from 28 September through 1 October. Similarily the sample was not collected 22 October through 27 October. Interstage sample analysis is shown in Table 5.

Startups were attempted on a daily basis 3-6 October. Operations were hampered by flow restrictions in first one reactor and then the other. On 6 October, the recycle compressor (C1206) pressure safety valve lifted, resulting in a loss of gas flow to the first stage reactor. A restriction formed in the plenum chamber when the gas was lost and temperatures in the 10'-16' range went as high as 950°F. The CCR unit went on solvent, the restriction was cleared, and coal feed was re-established in less than seven hours.

Low ash Ohio No. 6 coal feed began on 6 October and the catalyst addition/withdrawal program began on 9 October. The Ohio No. 6 coal produced more resid than the Illinois No. 6 produced at the same conditions. To decrease the Ohio No. 6 resid yield, the MF coal feed rate was lowered to 373 lb/hr on 9 October. The third period, 254C, was chosen during 9-10 October. It is considered a transitional period for operation with low ash Ohio No. 6 coal.

Several nuclear scans of the catalyst bed prior to 11 October indicated that a section just above the 6 October high temperature area had very low catalyst density. Ebullating pump speed was adjusted but no response was evident. Since a partial restriction was probable, an orderly shutdown was called and R1235 was opened and cleaned. An area of high density was found after the bottom head of the reactor was dropped. This area was cleaned with a steam lance, the plenum chamber was inspected, and startup began on 14 October.

At startup the feed slurry was temporarily changed to 30 wt % coal to reduce the high viscosity and to improve the chances of a successful startup. The feed slurry of 33 wt % coal was resumed directly after startup. The second reactor average temperature was increased from 760°F to 790°F on 15 October to encourage resid conversion. Period 254D was chosen during 16, 18-20 October and is a stable operation period with low ash coal.

The high space velocity test began on 21 October when the MF coal feed rate was increased to 420 lb/hr. R1236 experienced temperature swings that influenced gas flows at the higher feed rate. Stable operations were achieved on 23 October but a material balance period was not selected because an all-distillate slate was not achieved. Resid yields 23-27 October exceeded acceptable levels at this higher coal feed rate. The shutdown on 27 October was planned to be the end of Run 254.

The scheduled routine maintenance program began and the V1082 vacuum vessel was elevated. To optimize the Ohio No. 6 coal conversion and yield slate, Run 254 was extended in November. Two additional batches of Shell 317 catalyst were presulfided and fresh catalyst was charged to both reactors. Unit conditions for 3 December startup were:

MF coal feed rate = 500 lb/hr
Slurry composition = 30.0% coal
70.0% process solvent
Process solvent composition = 38% resid
12% CI
50% heavy distillate solvent
1st stage average temperature = 810°F
1st stage inlet H_2 partial pressure = 2650 psia
2nd stage average temperature = 760°F

There were several small interruptions in the startup attempts 3-5 December which were primarily caused by minor restrictions in R1235. Due to the excessive height of the first stage catalyst bed, the targeted MF coal feed rate of 500 lb/hr could not be obtained. On 6 December catalyst carryover in the first stage reactor required a shutdown. The catalyst was removed and inspected. Upon weighing the catalyst it was found that an excessive amount had been charged to the reactor. All repairs were made by 10 December and coal feed was resumed with a higher coal concentration in the slurry. After stable operations were established, 25 pounds of catalyst was returned to the reactor bringing the total charge to 300 pounds.

Period 254E was chosen during 13-14 December with the highest MF coal feed rate. The higher throughput was planned to age the fresh catalyst. Since the resid yield was unacceptably high, the process solvent resid composition was gradually increased from 50% resid, 12% CI, and 38% solvent to 55% resid, 12% CI, and 33% solvent. An interruption in operation occurred during a power outage on 15 December in which the first reactor formed a restriction immediately after ebullation flow stopped. An emergency situation resulted in which the excursion temperatures reached as high as 930°F. Just prior to this power failure the process solvent had been diluted to meet a target 55% resid. The viscosity increase due to the compositional change is considered to be a contributing factor in the plug formation.

While off coal, the plant remained down to repipe the process heater, B1201, between the reactors. Installation of this heater was important because it would act as an interstage heater if second stage conditions required higher temperatures.

Startup on 18 December included a lower MF coal feed rate of 375 lb/hr. Due to the slurry viscosity increase, the process solvent was held at 50% resid, 12% CI, and 38% solvent. Also for this startup, 25 pounds of the first stage catalyst was removed to prevent catalyst carryover. The catalyst replacement program began on 20 December at 3 lbs fresh catalyst per ton MF coal. Period 254F was chosen during 26-29 December and reflects operation during a baseline period.

On 30 December, the second reactor temperature was increased to 790°F. Although increasing this temperature improved the resid conversion, it also allowed a direct comparison to to 254D. Period 254G was chosen during 1-4 January. On 7 January the plant was intentionally taken off feed during a winter storm to prevent problems due to power outages. Plant operations resumed on 11 January and Period 254H was chosen during 15-17 January.

The MF coal feed rate was reduced to 300 lb/hr on 20 January to study the effect of space velocity on CCR unit yields. Period 254I was chosen during 29-30 January. The MF coal feed rate was increased again on 31 January to 450 lb/hr, but operation was interrupted on 1 February to repair the catalyst addition valves. Solids (suspected to be ammonia salts) in the recycle gas line corroded the valves and made it impossible to make the routine catalyst addition on 31 January. Coal feed resumed on 3 February and Period 254J was chosen as 6-8 February.

The second reactor temperature was increased to 810°F on 10 February to improve resid conversion and period 254K was chosen during 11 and 13-16 February. The temperature increase had only marginal effects on conversion. The age of the R1236 catalyst was considerably high during this period, (3124-3467 lb resid + CI/lb catalyst) and was considered to be the inhibiting factor on additional conversion. To obtain an additional space velocity point at 810°F, the MF coal feed rate was lowered to 375 lb/hr on 17 February. Period 254L was chosen during 20-21 and 23 February.

Special ebullation tests began on 24 February and included 79.4 hours of coal feed. During this test, the ebullating characteristics for 1/20" Shell 317 were compared to 1/10" Amocat 1B. Catalyst samples were taken each day of the run. The results of this test are discussed in Section 7.3, Amocat 1B Ebullation Test Results.

Performance

A summary of first and second stage operating data for Run 254 is shown in Table 3. Because the reactor exotherm is proprietary, the relative exotherm was reported based on the lowest exotherm across the first reactor. All exotherm values are relative to period 254C. The exotherm on the first reactor decreased during 254A-254C as the coal feed rate (and WHSV) was lowered and as the catalyst age increased.

The second stage exotherm continued to drop through periods 254A-254D since there was no catalyst replacement in the second reactor. A similar trend can be noted for periods 254E-254I. The second reactor temperature increase from 760°F to 790°F which was seen twice, once between 254C and 254D and secondly between 254F and 254G, did not significantly effect the second stage relative exotherm. It should be noted that the exotherm across the second reactor was significantly lower than across the first reactor; thus, changes in the second reactor relative exotherm appear to be much smaller.

The CCR conversion data are shown in Table 4. The Phase 2 coal conversions were calculated using a steady state forced ash balance method. In this method, the interstage ash flow rate is adjusted to equal the coal ash and recycle ash entering the first stage. The coal conversion is then calculated for the first and second stages based on the interstage analysis and stream balances. Table 4 shows how the overall conversions increased during period 254A-254C as the coal feed rate (and WHSV) was decreased. A 2.0% increase in overall conversion is shown between periods 254C-254D and periods 254B-254C when the switch to low ash (cleaned) coal was made. Another significant increase in overall coal conversion and resid + UC conversion was shown between periods 254C-254D and periods 254F-254G when the second reactor temperature was increased from 760°F to 790°F. However, very little difference in coal conversion resulted from the second reactor temperature change from 790°F to 810°F. The first stage coal conversion is calculated using the interstage sample analysis which is shown in Table 5.

Similar to the coal conversion, responses to the second stage temperature were experienced in C_4+ distillate and resid yields before elemental balancing. CCR unit yields before elemental balancing are shown in Table 6. Increasing the second stage temperature between 254C and 254D decreased the resid yield by 10% MAF coal and increased the C_4+ distillate yield by 13.3% MAF. The same temperature increase from 254F to 254G decreased the resid yield by 6.0% MAF and increased the C_4+ distillate yield to the highest for Run 254, 74.6% MAF coal. It should be noted that raising the second reactor temperature from 760°F to 790°F increased the gas yield from 7.6% MAF in 254F to 8.4% MAF in 254G. Another temperature increase to 810°F raised the gas yield from 8.1% MAF in 254J to 9.7% MAF in 254K.

3.4 Critical Solvent Deashing Unit (CSD)

The primary objectives of CSD unit operations during Run 254 were:

1) To establish acceptable deashing performance and to recover full range resid for recycle to coal slurry.

2) To produce an optimum resid recovery with minimal energy rejection to the ash concentrate (less than 20% of the feed coal heating value).

3) To evaluate CSD operations in the catalytic-catalytic CC-ITSL mode of operations with Ohio No. 6 coal undergoing batch catalyst aging and catalyst addition/withdrawal.

DAS type and first stage operating conditions were adjusted to produce efficient CSD operations. The CSD feed solvency index, resid recovery, energy rejection, and resid content of the ash concentrate were monitored on a frequent basis as performance indicators.

Operations

For Run 254, the overall CSD performance was good. There were instances of lost on-stream time, with the only major operational problem occurring with the CSD feed tank agitator. The first stage level controller was adjusted six times (28 September, 21 December, 28-29 December, 20 January, and 4 February) to improve first stage operability after the DAS was changed. Incidents of ash carryover were promptly corrected by adjusting unit operating conditions to obtain maximum separation. The CSD was on-stream a total of 2304.0 hours for an on-stream factor of 86.2%. The operational upsets and changes are shown in Appendix B.1.

DAS type 4100 was used at the start of Run 254 on 24 August 1987. The DAS type was changed throughout the run to minimize the amount of organic material rejected with the ash concentrate. A summary of DAS changes for Run 254 is shown in Appendix B.2.

The performance of the CSD unit, outlined in Table 7, was good for Run 254. The CSD feed solvency index ranged from 0.67 to 0.89 and the preasphaltene content of the CSD feed ranged from 0.0 to 11.5 wt %. Table 8 shows the feed composition averaged 15.0 wt % ash, 8.0 wt % UC, and 4.6 wt % solvent. The ash concentrate contained an average of 42.6 wt % ash and 18.8 wt % toluene soluble material. The ash concentrate ranged in consistency to include gummy, chunky, extruded, grainy, and powder.

Observations

During the period of 22-26 January and 29-30 January, the CSD unit was producing ash concentrate which had a higher average ash (47-48 wt %) and cresol insolubles(73-76 wt %) than the remainder of Run 254 where the ash averaged 43-45 wt % and the cresol insolubles averaged 67-70 wt %. While producing the high CI ash concentrate, the CSD had the lowest energy and organic rejection of Run 254. The energy rejection averaged 7.7% and the organic rejection averaged 6.7 wt %, as compared to an average energy rejection of 9.9% and an average organic rejection of 8.6 wt % for the 4 January - 1 February period of operation. Also, during this time, the toluene soluble content of the CSD ash concentrate reached its lowest point during Run 254.

An investigation into the relationship of the amount of ash in the coal feed and the CSD organic rejection was conducted using the data points from the material balance periods of Run 254. As can be seen in Figure 7, the organic rejection has a near linear relationship to the percentage of ash in the coal feed. Also, the organic rejection is inversely dependent upon the percent ash in the CSD ash concentrate stream, but not to the degree that it is on the percent ash in the MF coal feed. To lower the organic rejection at the CSD unit, the amount of ash in the coal feed would need to be reduced or the amount of ash in the CSD ash concentrate stream would need to be increased. An operational limit of 77 wt % cresol insolubles has been observed, meaning the organic rejection can only be further reduced by reducing the ash in the coal feed. During Run 254 the lowest energy and organic rejections to date were recorded when the amount of ash was reduced in the coal feed by switching to the low ash coal.

An investigation into the relationship of the CCR feed rate, second stage reactor temperature, and catalyst age on the amount of preasphaltenes in the CSD feed was also done. From Figure 8, there seems to be no major dependent variable (in fact, all correlation coefficients are below 0.10), but in general, as the reaction severity increases (increase in temperature, decrease in feed rate, or a decrease in catalyst age), the amount of preasphaltenes decreases from the previous point.

CSD Unit Yield Structure

Period	254A	254B	254C	254D	254E	254F	254G	254H	254I	254J	254K	254L
Date	29-30 Aug	23-25,28-30 Sep	9-10 Oct	16,18-20 Oct	13-14 Dec	26-29 Dec	1-4 Jan	15-17 Jan	29-30 Jan	6-8 Feb	11,13-16 Feb	20-21,23 Feb
DAS	4100	2104,2204 2304,2504	2504,2604	2554	2304	2304	2304	2454,2504	2604	2654,2704	2654	2704
Input, wt % CSD feed												
Dist. solv.	4.4	1.7	2.4	4.3	9.1	4.0	8.0	3.0	4.7	7.5	3.9	3.5
Resid	63.9	69.7	74.1	71.5	70.0	72.5	70.6	75.4	73.7	72.7	75.0	74.1
UC	11.6	10.2	8.0	8.1	6.3	8.1	7.0	7.1	7.2	7.3	7.9	8.6
Ash	20.1	18.4	15.1	16.1	14.6	15.4	14.4	14.5	14.4	12.5	13.2	13.8
Yield, wt % CSD feed												
Ash Concentrate	47.1	46.6	36.0	35.4	29.7	34.2	31.3	33.6	29.5	27.5	31.4	31.1
Dist. solv.	1.3	0.7	0.1	0.3	0.4	0.6	0.9	0.7	0.2	0.1	0.3	0.2
Resid	14.0	16.5	12.0	9.5	9.3	10.0	8.9	10.5	7.5	6.7	8.3	7.3
UC	11.8	11.0	8.7	9.5	6.6	8.6	7.7	8.0	7.8	8.3	9.4	9.5
Ash	20.0	18.4	15.2	16.1	13.4	15.0	13.8	14.4	14.0	12.4	13.4	14.1
CSD resid	52.9	53.4	64.0	64.6	70.3	65.8	68.7	66.4	70.5	72.5	68.6	68.9
Dist. solv.	2.6	3.2	9.5	4.1	8.5	3.8	10.1	7.3	6.2	9.1	5.9	4.5
Resid	50.3	50.2	54.5	60.5	61.3	61.9	58.6	59.1	64.1	63.4	62.7	64.4
Ash	0.0	0.0	0.0	0.0	0.5	0.1	0.0	0.0	0.2	0.0	0.0	0.0
Resid rec., wt % cresol solubles	76.9	74.8	83.0	85.3	87.0	84.9	86.7	84.6	88.8	90.0	87.5	89.4
Energy rej., % of coal heating value	16.9	17.5	9.8	8.9	9.8	12.5	9.9	10.5	7.8	8.2	8.6	10.3
Organic rejection, % of adjusted ash concentrate (2nd Phase Balance Data)	15.3	16.1	8.8	8.0	8.3	10.2	8.3	9.2	6.6	7.1	7.9	8.9

4.0 OVERALL TWO-STAGE LIQUEFACTION YIELDS

The TSL yields are the result of averaged elemental balances around all units. The analytical data used for these balances are presented in Tables 9-12. Operating conditions, averaged elementally balanced overall yields, and unit contributions are summarized on the following pages for all material balance periods. The yield contributions are also shown schematically in material balance flow diagrams (Figures 9-20).

Overall TSL Yields
(Phase 3 Data)

Operating Period	254A (Transitional)	254B	254C (Transitional)	254D	254E (Transitional)	254F
Date, 1987	8/29-8/30	9/23-30/87	10/9-10/87	10/16-20/87	12/13-14/87	12/26-29/87
Days selected	8/29, 8/30	9/23, 9/24, 9/25 9/28, 9/29, 9/30	10/9, 10/10	10/16, 10/18 10/19, 10/20	12/13, 12/14	12/26, 12/27 12/28/12/29
Catalyst addition/withdrawal (1st stage)	No	No	Yes	Yes	No	Yes
Interstage separator	No	No	No	No	No	No
Coal feed rate, MF lb/hr	492	385	362	371	485	365
Ash in coal, wt % MF	10.5	9.5	6.0	6.2	6.4	7.7
Coal conc. in slurry, wt % MF	32.9	32.8	32.4	32.7	32.2	29.7
Process solvent, wt %						
Resid (a)	39.1	39.0(44)	40.9(46.5)	39.8(45)	49.4(56)	49.6(56)
CI	12.4	12.2	12.2	12.2	11.8	12.2
1st stage						
Reaction temp., °F (avaerage)	810	811	811	811	810	811
Inlet H_2 part. press., psia	2640	2620	2585	2630	2664	2726
Space velocity, lb feed/lb cat-hr	5.1	4.0	3.8	3.9	5.2	4.3
lb MF coal/hr-ft^3	60.7	47.5	44.6	45.7	59.8	45.0
Catalyst type	Shell 317	Shell 317	Shell 317	Shell 317	Shell 317	Shell 317
Catalyst age, lb (resid+CI)/lb cat	398-480	865-1302	1171-1229	1138-1200	384-474	830-967
lb MF coal/lb cat	196-236	425-637	571-599	554-604	171-210	347-400
2nd stage						
Reaction temp., °F (average)	761	760	761	789	761	760
Space velocity, lb feed/lb cat-hr	4.9	3.8	3.7	3.7	3.0	4.2
lb MF coal/hr-ft^3	60.7	47.5	44.6	45.7	59.8	45.0
Catalyst type	Shell 317	Shell 317	Shell 317	Shell 317	Shell 317	Shell 317
Catalyst age, lb (resid+CI)/lb cat	296-358	578-914	1113-1159	1258-1449	307-379	833-999
lb MF coal/lb cat	198-240	383-595	723-752	816-935	180-218	465-552
CSD						
DAS type	4100	2104-2504	2554-2604	2604-2554	2304	2304
H_2 consumption, wt % MAF	6.3±0.0	6.1±0.1	6.1±0.2	6.7±0.2	6.3±0.035	6.73±0.0
Energy rejection, %	16.9±0.1	17.5±1.2	9.0±0.1	8.9±0.5	9.8±	12.5±
Yield, wt % MAF Coal						
Water	8.4±0.4	10.5±0.4	10.7±0.2	10.6±0.3	13.9±0.2	10.5±0.3
H_2S, CO, CO_2, NH_3	3.5±0.1	3.5±0.2	3.6±0.1	3.0±0.1	3.5±0.1	4.0±0.2
C_1-C_3 gas	5.6±0.3	6.1±0.7	7.6±0.8	8.3±0.1	6.5±0.5	7.6±0.4
C_4+ distillate	68.4±2.3	60.2±2.0	57.8±0.5	68.7±2.2	67.6±4.9	69.7±3.5
C_4+ naphtha	18.2±0.6	18.7±1.3	17.8±1.3	20.9±1.1	16.1±0.7	20.8±2.4
Middle distillate	7.6±0.1	7.5±0.4	7.5±0.5	8.7±0.3	7.2±0.4	8.0±0.4
Distillate solvent	42.6±3.2	34.0±2.6	33.3±1.8	39.2±1.2	44.4±3.6	40.9±3.4
Resid (b)	5.2±2.8	9.8±2.5	17.7±0.2	7.3±2.3	6.6±3.8	4.6±2.3
Ash concentrate	15.3±0.3	16.1±0.7	8.8±0.0	8.0±0.4	8.3±6.7	10.3±0.8
H_2 efficiency						
lb C_4+ dist/lb H_2 cons.	10.8±0.3	9.9±0.4	9.5±0.4	10.3±0.2	10.6±0.7	10.4±0.8
C_1-C_3 selectivity						
to C_4+ distillate	0.08±0.0	0.10±0.01	0.13±0.01	0.12±0.01	0.10±0.01	0.11±0.01
Coal conversion, wt % MAF (c)						
1st stage (d)	91.2±1.0	93.3±0.5	93.2±0.0	-(f)	95.6±0.7	95.4±0.4
1st and 2nd stages	93.0±0.1	94.0±0.2	96.4±0.2	97.1±0.4	97.1±0.1	95.8±0.5
Overall TSL	92.9±0.4	93.5±0.2	95.9±0.1	96.5±0.3	96.7±0.3	95.2±0.5
Resid + UC conversion, wt % feed (d)						
1st stage (e)	30.3±1.8	26.7±0.5	23.9±1.2	-(f)	25.6±6.2	25.8±3.3
2nd stage	12.0±0.0	13.9±0.6	16.1±0.1	-(f)	14.2±7.3	11.5±3.5

(a) Data in parentheses on CI-free basis.
(b) Includes TSL system UC accumulation.
(c) Cresol solubles.
(d) Based on Phase 2 data, by the forced ash balance method.
(e) MAF coal as 100 wt % UC.
(f) Unavailable due to absence of interstage analysis.

Operating Period	254G	254H	254I	254J	254K	254L
Days, 1988	1/1-1/5	1/13-1/18	1/25-1/30	2/5-2/9	2/11-2/16	2/18-2/23
Days selected	1/1, 1/2, 1/3, 1/4	1/15, 1/16, 1/17	1/29, 1/30	2/6, 2/7, 2/8	2/11, 2/13 2/14, 2/15, 2/16	2/20, 2/21, 2/23
Catalyst addition/withdrawal (1st stage)	Yes	Yes	Yes	Yes	Yes	Yes
Interstage separator	No	No	No	No	No	No
Coal feed rate, MF lb/hr	367	364	297	438	440	373
Ash in coal, wt % MF	6.30	6.50	5.91	5.61	5.4	5.5
Coal conc. in slurry, wt % MF	29.2	29.2	29.4	29.2	29.4	30.0
Process solvent, wt %						
Resid (a)	48.8(55)	48.5(55)	50.5(58)	49.1(56)	49.3(56)	49.5(56)
CI	11.8	11.9	12.2	12.1	12.1	12.1
1st stage						
Reaction temp., °F (average)	811	811	811	810	810	810
Inlet H_2 part. press., psia	2,730	2,708	2,701	2,739	2,713	2,764
Space velocity, lb feed/lb cat-hr	4.3	4.3	3.5	5.1	5.1	4.3
lb MF coal/hr-ft^3	45.3	44.9	36.6	54.0	54.3	46.0
Catalyst type	Shell 317	Shell 317	Shell 317	Shell 317	Shell 317	Shell 317
Catalyst age, lb (resid+CI)/lb cat	1003-1124	1261-1332	1393-1452	1451-1538	1464-1558	1482-1556
lb MF coal/lb cat	415-462	514-543	571-595	591-626	595-635	604-631
2nd stage						
Reaction temp., °F (average)	790	790	790	790	810	810
Space velocity, lb feed/lb cat-hr	4.2	4.1	3.4	4.9	4.8	4.0
lb MF coal/hr-ft^3	45.3	44.9	36.6	54.0	54.3	46.0
Catalyst type	Shell 317	Shell 317	Shell 317	Shell 317	Shell 317	Shell 317
Catalyst age, lb (resid+CI)/lb cat	1166-1334	1703-1817	2392-2437	2774-2912	3124-3467	3690-3860
lb MF coal/lb cat	641-729	918-976	1276-1300	1464-1534	1638-1814	1936-2026
CSD						
DAS type	2304	2454-2504	2604	2654-2704	2654	2704
H_2 consumption, wt % MAF	6.94±0.09	6.59±0.53	7.10±0.12	6.05±0.05	6.47±0.11	6.56±0.06
Energy rejection, %	9.9	10.5	7.8	8.2	8.6	7.9
Yield, wt % MAF coal						
Water	9.17±0.25	9.67±0.70	10.47±0.07	9.64±0.33	10.82±0.14	10.53±10.10
H_2S, CO, CO_2, NH_3	3.45±0.19	3.39±0.06	3.55±0.08	3.32±0.08	3.10±0.12	3.17±0.12
C_1-C_3 gas	8.49±0.18	8.18±0.22	8.41±0.47	8.07±0.58	9.72±0.64	10.03±0.69
C_4+ distillate	78.39±1.34	71.73±0.45	75.95±1.30	63.49±1.46	64.36±0.06	66.98±0.31
C_4+ naphtha	21.09±1.19	19.74±0.63	21.32±0.66	18.37±0.82	20.60±0.47	20.90±0.57
Middle distillate	8.89±0.26	8.09±0.14	9.19±1.27	7.88±0.15	7.77±0.54	8.58±0.34
Distillate solvent	48.41±2.28	43.89±1.01	45.44±2.93	37.24±0.59	36.00±1.41	37.50±0.54
Resid (b)	-0.72±1.53	4.45±1.06	2.04±0.72	14.32±1.30	10.46±0.02	8.45±0.90
Ash concentrate	8.16±0.79	9.15±0.32	6.68±0.08	7.21±0.16	7.99±0.49	7.39±0.31
H_2 efficiency						
lb C_4+ dist/lb H_2 cons	11.30±0.26	10.89±0.42	10.78±0.36	10.50±0.16	9.96±0.24	10.22±0.10
C_1-C_3 selectivity						
to C_4+ distillate	0.11±0.00	0.12±0.01	0.12±0.01	0.13±0.01	0.15±0.01	0.15±0.01
Coal conversion, wt % MAF (c)						
1st stage (d)	94.98±0.71	95.86±0.36	96.98±0.00	95.99±0.76	96.23±0.26	96.30±0.16
1st and 2nd stages	96.72±0.44	96.49±0.22	97.02±0.64	96.43±0.31	96.37±0.18	96.29±0.50
Overall TSL	96.46±0.32	96.19±0.14	96.46±0.01	96.03±0.09	95.80±0.18	95.83±0.18
Resid + UC conversion, wt % feed (d)						
1st stage (e)	26.33±1.08	23.03±2.82	27.20±0.99	21.03±3.25	23.23±2.60	25.13±1.81
2nd stage	15.68±1.88	17.0±3.48	14.90±2.83	14.37±2.60	14.25±2.43	14.03±2.39

(a) Data in parentheses on CI-free basis.
(b) Includes TSL system UC accumulation.
(c) Cresol solubles.
(d) Based on Phase 2 data, by the forced ash balance method.
(e) MAF coal as 100 wt % UC.

4.1 Discussion of TSL Performance

Several conditions were varied during Run 254 to determine their effect on overall yields:

1) Coal ash: 5.4-10.5 wt % MF
2) 1st stage space velocity: 3.5-5.2
3) 2nd stage space velocity: 3.4-5.0
4) Coal concentration in feed: 30-33 wt %
5) Resid in process solvent: 40-50 wt %
6) 2nd stage average temperature: 760-810°F
7) Catalyst addition/withdrawal in the 1st stage

Process performance parameters and overall yields varied significantly due to these changes in process variables as shown below:

H_2 efficiency, lb C_4+ dist/lb H_2 consumed	9.5-11.3
C_1-C_3 selectivity to C_4+ distillate	0.08-0.15
Coal conversion, wt % MAF coal	92.9-96.7

Overall yield, wt % MAF coal	
H_2 consumption	6.1-7.1
C_1-C_3 gas	5.6-10.0
C_4+ distillate	57.8-78.4
Resid (and UC internal accumulation)	(-0.7)-(17.7)
Ash concentrate (ash-free)	6.7-16.1

The main achievement of Run 254 was an increase in distillate yield to 78% MAF. Figures 21-23 show distillate yield trend data. The increase in potential liquid yield was the result of using lower ash cleaned coal. The coal conversion of the cleaned coal was higher by 3% MAF. The organic rejection with the low ash coal was 8% MAF lower than with high ash coal. The relationship between organic rejection and coal ash content is discussed further in Section 3.4, Critical Solvent Deashing Unit.

In 254B and 254C the coal ash content was 9.5 and 6.0%, respectively. The organic rejection decreased by 7.3% with the lower coal ash. The potential liquid yield increased by 6% MAF.

Since the resid yield was high (17.7% MAF) in period 254C, the second stage reactor temperature was increased from 760 to 790°F to convert more resid. As shown in Figure 22 the higher second stage temperature increased the distillate yield by 11% MAF. The first stage catalyst age was the same for 254C and 254D due to the catalyst replacement program, but the second stage catalyst age was higher in 254D by 140 (lb resid + CI)/lb catalyst.

The distillate yield was 12% MAF higher in 254F than in 254C. The resid content of the process solvent was 40 wt % in 254C and 50 wt % in 254F. The coal concentration was 33 and 30 wt % respectively in 254C and 254F. The catalyst age in 254F was lower by 350 and 270 (lb resid+CI)/lb catalyst in the first and second stage reactors, respectively.

An all-distillate product slate was achieved with low ash Ohio No. 6 coal in period 254G. The second stage temperature was 790°F in 254G compared to 760°F in 254F. The temperature increase resulted in a distillate yield of 78.4% MAF -- an 8.7% MAF increase in distillate yield. The potential liquid yield increase of 3.4% MAF in 254G compared to 254F can be attributed to a decrease in coal ash from 7.7 to 6.3 wt % MF. The lower ash coal had a lower oxygen content and thus a lower water yield. The lower ash content of the coal also reduced the organic rejection by 2.2% MAF. The higher temperature increased the C_1-C_3 yield by 0.9% MAF. The catalyst age was higher in 254G by 166 and 335 (lb resid + CI)/lb catalyst in the first and second stage reactors, respectively.

Period 254H was at the same conditions as 254G with the exception of catalyst age. The catalyst age was higher by 230 and 510 (lb resid + CI)/lb catalyst in the first and second stage reactors, respectively. The catalyst aging lowered the distillate yield by 6.7% MAF to 71.7% MAF. The potential liquid yield was 1.5% lower in 254H than in 254G due in part to a 1% MAF increase in organic rejection.

In Period 254I the coal feed rate was reduced to 300 MF lb/hr to achieve resid extinction at a higher second stage catalyst age. The 65 lb/hr decrease in coal feed rate was the only change from 254H with the exception of the catalyst age, which was higher by 128 and 655 (lb resid + CI)/lb catalyst in the first and second stage reactors, respectively. The potential liquid yield was 78% MAF with a distillate yield of 76% MAF. A stronger DAS type was used in 254I than in 254H and the coal ash was 0.5 wt % lower. These differences reduced the organic rejection by 2.5% MAF to a new low of 6.68% MAF.

The coal feed rate was increased to 440 MF lb/hr in 254J to simulate ash removal by purging the vacuum bottoms. The first stage catalyst age was higher by only 86 (lb resid + CI)/lb catalyst and the second stage catalyst age was higher by 415 (lb resid + CI)/lb catalyst. The resid yield increased by 12.27% MAF to 14.32% MAF. Figure 23 shows the corresponding decrease (by more than 12% MAF) in distillate yield.

In 254K the second stage temperature was increased to 810°F. The temperature increase to 810°F decreased resid yield by 3.9% MAF, but only an additional 0.9% MAF of the resid was converted to distillate. The potential liquid yield decreased by 3.0% MAF and the gas yield increased by 1.7% MAF.

The decrease in coal feed rate to 375 MF lb/hr in 254L decreased the resid yield by 2% MAF. The catalyst age was higher by 450 (lb resid + CI)/lb catalyst in the second stage and essentially was unchanged in the first stage.

4.2 Comparison to Run 253

In Run 253 and Run 254, 300 pounds of Shell 317 catalyst was used in the first and second stage reactors. A comparison of Run 254 to Run 253 shows the difference between yields obtained with Illinois No. 6 and Ohio No. 6 coals.

Table 13 compares period 253D and period 254B. All reactor conditions and feed compositions were similar in these periods except the second stage catalyst age. The second stage catalyst age ranged between 578-914 (lb resid + CI)/lb catalyst in 254B and 1601-1738 (lb resid + CI)/lb catalyst in 253D. Another difference between the two periods was that interstage separation was used in 253D but was not used in 254B. However, removal of the interstage separator did not significantly change reaction yields in Run 253. Despite the higher second stage catalyst age in 253D, 11.4% MAF more distillate was produced in 253D than in 254B. The potential liquid yield was higher by 2.3% MAF in 254B due to higher coal conversion and lower organic rejection.

Table 14 compares 253E to 254H. The potential liquid yield in 254H was 7.3% MAF higher than in 253E. The distillate yield was 4.1% MAF higher in 254H than 253E due to more severe reaction conditions in 254H. Several conditions were different between these two periods: lower coal concentration, higher resid concentration, higher second stage temperature, and lower second stage catalyst age in 254H.

In general the potential liquid yield from the Ohio No. 6 coal is higher than for the Illinois No. 6 coal, but the resid from the Ohio No. 6 coal is more difficult to convert to distillate than the resid from the Illinois No. 6 coal. The higher potential liquid yield with the Ohio coal was the result of coal cleaning before liquefaction. The organic rejection for the cleaned Ohio coal was 7-8% MAF lower than the organic rejection for the as-is Ohio coal. The potential liquid yield for Illinois No. 6 coal could also be increased by cleaning prior to liquefaction.

4.3 Hydrogenation of Process Solvent

The difference in hydrogen content of the interstage sample and the process solvent was generally greater during the periods without catalyst replacement as shown in Figures 24 and 25. This difference is due to the increase of catalytic hydrogenation in the first stage during catalyst replacement periods.

After the second stage temperature was increased to 790°F and as the second stage catalyst aged, the wt % hydrogen in the interstage stream became larger than the wt % hydrogen in the process solvent. From Period 254F to the end of the run, the hydrogen content of the process solvent deteriorated from 7.7 to 6.8 wt %. Due to catalyst replacement in the first stage, the hydrogen content of the interstage stream decreased by only 0.5 wt % despite the 0.9 wt % decrease in the process solvent hydrogen content.

A similar trend was observed for the resid portion of the process solvent stream and interstage streams. The hydrogen content of the resid portion of the interstage stream became greater than the resid portion of the process solvent when the second stage temperature was raised to 810°F. This lower hydrogen content of the resid from the second reactor may be due to increased cracking of the resid at the higher temperature and equilibrium conditions for the hydrogenation reactions.

A decrease in coal conversion did not occur as the hydrogen content of the process solvent decreased.

4.4 TSL Process Severity (Hydrogenation and Heteroatom Removal)

The overall TSL process severity can be studied with respect to TSL hydrogenation and heteroatom removal. This section studies three cases:

1. Coal Types: Ohio Coal vs Illinois Coal
2. Process Solvent Composition: 40 wt % vs 50 wt % resid
3. Second Stage Temperature: 790°F vs 810°F

4.4.1 Process Severity of Coal Types: Ohio No. 6 Coal vs Illinois No. 6 Coal

The processing severity effects of processing Ohio No. 6 coal and Illinois No. 6 coal were compared using periods 253E-H and 254B-E. Several process operating conditions for these periods were similar, as shown below:

Run	253E-H	254B-E
Coal type	Illinois No. 6	Ohio No. 6
Ash in feed coal, wt %	11-12	6-10
Sulfur in feed coal, wt %	3.4-3.6	2.5-2.7
Resid in process solvent, wt %	40	40
Catalyst type		
1st stage	Shell 317	Shell 317
2nd stage	Shell 317	Shell 317
Catalyst age		
lb (res+CI)/lb cat		
1st stage	1241-1651	865-1200
2nd stage	2163-3322	578-1449
Coal feed rate, MF lb/hr	385	365-385
Reaction temp., °F		
1st stage	810	810
2nd stage	760-770	760-790

The heteroatom yields were correlated with the TSL hydrogen consumption and are shown in Figure 26. Major observations between these periods are:

- Run 254 with Ohio coal showed a 2-4 wt % MAF higher potential liquid yield (C_4+ resid) than Run 253 with Illinois coal. This was due to a reduced gas make (H_2S, CO_x, and C_1-C_3) with an organic rejection in the range of 15-20 wt %.
- Both runs showed similar hydrogen efficiency and product quality.

- Low ash Ohio coal (6 wt % of ash) indicated an 8% MAF a lower organic rejection compared to either high ash Ohio or Illinois coals with 10-11 wt % ash.

- Nitrogen and oxygen removal was similar for both runs. But, Run 254 with Ohio coal showed a lower H_2S yield by 1 wt % MAF coal than Run 253 with Illinois coal. The lower H_2S can be attributed to a lower sulfur content of the feed coal.

- Run 254 with Ohio coal indicated a lower hydrogen consumption contribution to the C_4+ distillate make than Run 253 with Illinois coal. The higher second stage temperature (790°F) employed during Run 254 to convert the excess resid produced, may have inhibited second stage hydrogenation. During this same period the hydrogen consumption contribution in C_1-C_3 gas make significantly increased from 0 to 17%.

The following table highlights the linear regression results of Figure 26:

Run	Product	Linear regression slope (product/H_2 cons)	H_2 required theoretically (% MAF coal)	Contribution (%)
253E-H	C_1-C_3	0(a)	0	0
	H_2O	0(a)	0.03(c)	3
	C_4+ dist	14.6	0.95(b)	95
	H_2S	0(a)	0	0
	NH_3	0(a)	0.02(d)	2
				=100
254B-E	C_1-C_3	2.85	0.17	17
	H_2O	0(a)	0	0
	C_4+ dist	14.16	0.79(b)	79
	H_2S	0(a)	0	0
	NH_3	0.24	0.04	4
				=100

(a) The slope in the linear regression analysis is assumed to be zero because of a poor correlation ($r^2 < 0.4$).
(b) By difference.
(c) Based on the calculated slope for CO-CO_2 gas make, 0.21 ($r^2 = 0.49$).
(d) Based on the calculated slope, 0.13 ($r^2 = 0.10$).

4.4.2 Process Severity of Process Solvent Composition: 40 wt % vs 50 wt % Resid

The process severity effects of 50 wt % resid in the process solvent were compared to the effects of 40 wt % resid in the process solvent in Figure 27. Several process operating conditions for these periods were similar, as listed below:

Run	254B-D	254E-J
Coal type	Ohio No. 6	Ohio No. 6
Ash in feed coal, wt %	6.0-9.5	5.6-7.7
Resid in process solvent, wt %	40	50
Catalyst type		
1st stage	Shell 317	Shell 317
2nd stage	Shell 317	Shell 317
Reaction temperature, °F		
1st reactor	810	810
2nd reactor	760-790	760-790
Catalyst age lb (resid+CI)/lb cat		
1st stage	865-1200	384-1538
2nd stage	578-1449	307-2912

Observations for these periods are:

- Operation with 50 wt % resid in the process solvent showed a slightly higher selectivity for the potential liquid yield (C_4+ resid) by 1-2 wt % MAF coal, compared to operation with 40 wt % resid.

- A higher hydrogen efficiency and a lower product quality were observed with 50 wt % resid in the recycle process solvent.

- Heteroatom removal and C_1-C_3 gas make were similar for both periods.

4.4.3 Effect of Increased Second Stage Temperature from 790 to 810°F

The process severity effects of 810°F second stage reaction temperature for period 254K-L were compared to 790°F for period 254E-J in Figure 28. Conditions for these periods were very similar as listed below:

Run	254E-J	254K-L
Coal type	Ohio No. 6	Ohio No. 6
Ash in feed coal, wt %	5.6-7.7	5.5
Resid in process solvent, wt %	50	50
Catalyst type		
1st stage	Shell 317	Shell 317
2nd stage	Shell 317	Shell 317
Catalyst age		
lb (resid+CI)/lb cat		
1st stage	384-1538	1464-1558
2nd stage	307-2912	3124-3860
Coal feed rate, MF lb/hr	300-485	440-373
Reaction temperature, °F		
1st reactor	810	810
2nd reactor	760-790	810

Observations are:

- The higher second stage reaction temperature of 810°F resulted in a lower selectivity for the potential liquid yield (C_4+ resid) by 2 wt % MAF coal. The lower potential liquid yield was attributed to the 2 wt % MAF higher C_1-C_3 gas make at 810°F than operation at 790°F.

- A lower hydrogen efficiency was observed during period 254K-L at 810°F than period 254E-J at 790°F, primarily due to a higher C_1-C_3 gas make.

- Heteroatom removal was similar for both operation temperatures at 810 and 790°F. Therefore, product quality may be similar for both periods whether the operation temperature is 810 or 790°F.

5.0 CATALYST

As planned in Run 254, both reactors were charged with three hundred pounds of 1/20" Shell 317 trilobe catalyst. Periodically, a one pound sample of catalyst was removed from the first stage reactor for characteristic studies. An equivalent amount of catalyst was returned to the reactor. Since the second stage reactor has no catalyst withdrawal system, a grab sample could only be obtained during a shutdown period when it could be taken from the top of the catalyst bed.

5.1 Catalyst Sulfiding Procedure and Results

The fresh catalyst was sulfided with dimethyl disulfide (DMDS) at a 1.5 wt % initial concentration in recirculating No. 2 diesel (fuel) oil. The feed gas rate was maintained at 3,000 SCFH, and catalyst bed ebullation began when the reactor temperature reached 250°F. The reactor was heated stepwise by 50°F from 250 to 400°F. Each temperature was held until the hydrogen sulfide "breakthrough" occurred, indicating the end point of the sulfiding at that temperature. During the sulfiding, the hydrogen-rich vent gas was recycled and DMDS was added to the diesel oil at a rate of 6 lbs/hr. The reactor temperature was increased stepwise by 50°F again and was held at 500°F, 600°F and 700°F until a breakthrough occurred. At the maximum temperature (700°F) the reactor was held until the analysis of Shell 317 catalyst samples indicated a sulfur content (wt % S = "as is" wt % S/wt % ash x 100) of at least 8.2 wt %. The reactor was cooled at a maximum rate of 100°F/hr until it was less than 250°F. The catalyst was withdrawn and stored in drums at ambient conditions.

Nine separate batches of 1/20" Shell 317 catalyst were presulfided for Run 254. The analytical results are shown in Table 15. Fresh, presulfided catalyst had an average naphthalene activity of 198 mmoles H_2 consumed. The highest carbon buildup for presulfiding was 2.54 wt % in Batch #9. Composite samples were not taken for Batch #6 or Batch #9 because the catalyst was not removed from R1235 after sulfiding. Very little damage was done to the catalyst during the sulfiding process and recovery was calculated for each available batch:

		American Standard Screen Size Distribution			
Batch #	Recovery	#14	#18	#25	-25
Unsulfided	-	83.4	16.5	0.1	0.1
1	95.2	81.5	18.1	0.4	0.0
2	98.1	78.3	21.1	0.2	0.5
3	97.2	82.2	17.7	0.1	0.0
4	98.1	83.1	16.7	0.1	0.1
5	99.8	81.9	18.0	0.1	0.1
6	Not available	84.5	15.3	0.1	0.1
7	90.2	78.0	21.8	0.1	0.1
8	88.0	77.9	20.4	0.4	1.4
9	Not available	85.3	14.2	0.2	0.3

5.2 Analytical Results and Recovery

Analytical results of each catalyst sample include: an elemental analysis, a screen analysis, an ash evaluation, and a naphthalene activity test. The carbon content is evaluated to determine buildup that would inhibit catalyst activity. The naphthalene activity test measures activity in units of mmoles of hydrogen consumed. The test determines the hydrogen consumption during the hydrogenation of naphthalene in the presence of a fixed volume of catalyst. Naphthalene is the model compound used to measure relative catalyst activity independent of reactor or TSL system performance.

Recovery of catalyst is calculated when the entire catalyst bed is removed from the reactor. Since the reported weight is always a dry weight, the ash analysis is used to determine the difference in the actual wet weight and the theoretical dry weight. Due to the additional process ash remaining in the reactor after a shutdown, the optimum recovery is approximately 106%.

5.2.1 First Stage Catalyst

The first stage catalyst charge was 300 pounds of fresh, sulfided Shell 317 catalyst. Batch deactivation continued until 9 October when the catalyst addition/withdrawal program began. The analytical results of first stage catalyst are shown in Table 16. During batch deactivation, the first stage catalyst naphthalene activity dropped to 46 mmoles of H_2 consumed and the carbon build-up was as high as 14.3 wt %. Catalyst replacement continued at a rate of 3 lbs of catalyst/ton of MF coal until the scheduled shutdown on 27 October. The screen analysis of most catalyst samples during both periods indicated a gradual breakdown of catalyst.

The catalyst was removed from the first stage during the 11 October shutdown. Catalyst recovery was excellent at 105.7%. The shutdown had been called to clear a restriction in the catalyst bed. The nuclear detector which is used to determine catalyst bed height indicated an uneven ebullation distribution on 7 October and again on 9 October. The screen analysis of the composite sample was:

69.5 wt % Screen #14
28.2 wt % Screen #18
2.2 wt % Screen #25
0.1 wt % Smaller than #25

Although the composite sample reinforced concern about a gradual catalyst breakdown, replacement was not considered because plans did not include operation past the end of October. Thirty pounds of fresh catalyst was added to replace the catalyst lost in the restriction formation and the recovered catalyst was added back to the reactor.

The catalyst was removed from both reactors following the 27 October shutdown. First stage catalyst recovery was low at 92%. The screen analysis of the R1235 catalyst composite sample was much like the previous results:

70.2 wt % Screen #14
24.4 wt % Screen #18
2.3 wt % Screen #25
3.1 wt % Smaller than #25

This composite reflects an even more significant shift to fine particle sizes from Screen #18. The seven additions of fresh catalyst between 11 October and the shutdown did not make a significant contribution to the screen size distribution even though they accounted for one third of the total catalyst bed. It is suspected that the catalyst was more easily broken after the high temperature excursion on 6 October. See Section 3.3, Close Coupled Reactor Unit. The low catalyst recovery could be a result of the smaller pieces being carried over to the second stage reactor.

When Run 254 was continued, additional catalyst was sulfided and fresh catalyst was charged to both reactors. Not long after the 3 December startup, the catalyst was removed from R1235 again. The plant operability had been very difficult due to an excessive bed height and an increased occurrence of voids within the bed. Approximately 355 pounds of catalyst was removed from R1235 at shutdown. The catalyst weight prior to shutdown was estimated based on the calculations using the weights and lab analysis from presulfiding. Only 275 pounds of this catalyst was charged to R1235 for startup and an additional 25 pounds was added after smoother operation allowed the ebullation and bed height to become stable.

The catalyst addition/withdrawal program began on 20 December with a replacement rate of 3 lbs of catalyst/ton of MF coal. The addition/withdrawal weight was adjusted with coal feed rate changes and a catalyst sample was collected at each withdrawal.

Run 254 offered a unique opportunity to compare results of two different startup periods with the same type of catalyst. Figures 29 and 30 compare the carbon buildup and the naphthalene activity for the catalyst in the August-October operation and the December-March operation. These plots illustrate basis of concern for the first stage catalyst in October. At similar ages, the December-March catalyst showed about 13.5 wt % carbon buildup but the August-October catalyst had 14.4 wt %. Also, at the end of operation for the August-October period, the composite had 18.3 wt % carbon while the December-March period had 12.5 wt % carbon. A grab sample from the August-October period of operation was also analyzed and had 16.1 wt % carbon. The higher carbon content could be attributed to the October startup when a temperature excursion went to 950°F.

Similar observations can be made using the naphthalene activity test. The naphthalene activity of fresh, sulfided Shell 317 catalyst averaged 198 mmoles H_2 consumed. The deactivation rate shown is typically, rapid deactivation from fresh to 200 (lb resid + CI)/lb catalyst and then tapering off to an "equilibrium activity" with catalyst replacement. The naphthalene activities were similar for both periods of operation after catalyst replacement. However, the August-October composite sample had a much lower activity (32 mmoles H_2 consumed) than the December-March composite (45 mmoles H_2 consumed). It should be noted that since the naphthalene activity test utilizes a very small amount of catalyst, a representative sample is difficult to obtain and large variations in results is very common.

5.2.2 Second Stage Catalyst

The second stage reactor was also charged with 300 pounds of fresh, sulfided Shell 317 catalyst. Samples from the second stage were not regularly obtained since R1236 does not have an addition/withdrawal system. Table 17 shows the analytical results of each sample taken from R1236 in Run 254.

The catalyst was removed from R1236 following the 31 August shutdown. Although catalyst carryover was the cause of the shutdown, catalyst recovery was good at 102.1%. Since the naphthalene activity of the catalyst was still high and the screen analysis for the catalyst was satisfactory, as much catalyst as possible was returned to the reactor. Approximately 83 pounds of fresh catalyst was added to bring the total back to 300 pounds for startup.

Multiple shutdowns in October allowed more samples of the second stage catalyst to be collected than in past runs. It is clear from these samples that the high temperature first stage catalyst bed has a more severe environment than the lower temperature second stage catalyst. The sample on 12 October had a low carbon buildup, 11.33 wt %, and a high naphthalene activity, 56 mmoles H_2 consumed. At the same (resid + CI) catalyst age and after five additions of fresh catalyst, the first stage had a higher carbon buildup at 14.48 wt % and a lower naphthalene activity of 44 mmoles H_2 consumed. The naphthalene activity level was similar for the composite sample of both reactors even though the second stage catalyst had a much lower carbon buildup (13.8 wt % vs 18.3 wt %).

The catalyst was removed from both reactors following the 27 October shutdown. Catalyst recovery in R1236 was good at 104.25%. When Run 254 was extended, 300 pounds of fresh sulfided 1/20" Shell 317 was charged to R1236. This catalyst was sampled twice, 8 December and 2 February, and remained in the second stage through the Amocat 1B ebullation tests. The composite sample had a very high age of 4034.9 lb (resid + CI)/lb catalyst or 2118.4 on lb MF coal/lb catalyst. Although the carbon buildup was high at 16.61 wt %, the naphthalene activity was good at 63 mmoles H_2 consumed. After the ebullation test, 316.2 pounds of catalyst were removed from R1236 for a 105.4% recovery. The second stage catalyst integrity is very difficult to monitor during the run. Grab samples taken during a shutdown are pulled from the top of the catalyst bed where smaller pieces naturally float in ebullation. The screen analysis of this composite is shown below:

62.4%	Screen #14
34.1%	Screen #18
2.6%	Screen #25
2.8%	Pan

For the age of this sample, this distribution is much better than expected.

5.3 Catalyst Activity Analysis

Catalyst activities for catalytic reactors were calculated, by 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).

$$K = WHSV \frac{\varepsilon}{1-\varepsilon}$$

The dependence of the conversion rate constant on temperature (T) is described by the Arrhenius equation and the decrease in rate constant due to catalyst age (t) is described by a separable 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 (Keq) with catalyst replacement is determined by using the residence time distribution function, RTD(t).

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

During the first part of Run 254 only a few catalyst activity data points were obtained for either stage because of interstage sampling problems. Catalyst activity trends for the second part of Run 254 were analyzed for the overall two-stage system, first stage, and second stage based on the Phase 2 resid yield and resid + UC conversion.

5.3.1 Overall Two-Stage Catalyst Activity and Deactivation

Although first order reaction yield logarithmic by definition, a linear regression technique is used as an accepted mode for the empirical fit over short periods of rapid deactivation or for long periods of equilibrium activity. Overall two-stage catalyst activity and deactivation trend data for period 254F-L are plotted in Figure 31. Phase 2 TSL resid yields (assuming 8 wt % MAF coal organic rejection) were analyzed by using a linear regression analysis technique for selected periods. Results are summarized in the following table:

Period	Y = aX + b	r^2
254F	Y = 0.011 x -0.111	0.505
254GH	Y = 0.007 x -4.968	0.422
254IJ	Y = 0.004 x -6.169	0.505
254KL	Y = 0.013 x -31.335	0.859

Typically as the catalyst deactivates, the C_4+ distillate yield declines by a particular amount and the TSL resid yield increases a proportional amount. The reciprocal of the deactivation slope (1/a) represents the TSL resid yield relationship to catalyst deactivation. Therefore a relationship can be established to determine how far the catalyst will age (on a resid + CI basis) before the TSL resid yield will increase 1% MAF. This relationship was calculated for selected periods:

Period	Age/Resid (1/a) (lb res+CI/lb cat/wt % MAF)
254E	45 (est.)
254F	95
254GH	155
254IJ	235
254KL	80

A similar relationship was calculated to determine how far the catalyst will age (on a resid + CI basis) before the TSL distillate yield will decrease 1% MAF. This relationship was calculated for the same periods where a' is estimated by using experimental values:

Period	Age/C_4+ dist (1/a') (lb res+CI/lb cat/wt % MAF)
254E	55 (est.)
254F	115
254GH	185
254IJ	280
254KL	95

It can be noted from both relationships that as Run 254 progressed, the catalyst had to age much more to effect the TSL yield structure. This observation can be the result of the initial batch deactivation or of the equilibrium catalyst activity. Detailed linear regression analyses of catalyst activity for both stages are discussed in the following sections 5.3.2 and 5.3.3.

Activation Energy

The activation energy for the catalyst was calculated based on the C_4+ distillate response during 254C-D. The activation energy ranged between 43,300 and 53,750 Btu/lb-mole for the second stage reaction temperature range of 760-790°F. A similar value of 47,800 Btu/lb-mole was also observed during the periods 254F-G which were periods with 50 wt % resid in the process solvent. The activation energy was calculated similarly for the temperature change from 790°F to 810°F and was 69,000 Btu/lb-mole. These activation energy values for Ohio No. 6 coal were slightly higher than for Illinois No. 6 coal (42,300 Btu/lb-mole for the temperature range of 720°F to 770°F). Activation energy values calculated from conversion activity values are shown below:

Period	2nd Stage Temperature, °F	Activation Energy* Btu/lb-mole
254C-D	760-790	43,300-53,750
254F-G	760-790	47,800
254J-K	790-810	69,000

*Activation energy calculated from TSL C_4+ distillate responses.

Period	2nd Stage Temperature, °F	ln(K)	Activation Energy* Btu/lb-mole
254F-J	760-790	0.3701	37,400
254G-L	790-810	0.3464	54,600

*Activation energy calculated from catalyst conversion activity (K). Lower values were obtained due to data scattering.

5.3.2 First Stage Catalyst Activity

The batch deactivation curve was estimated for Run 254 and is shown in Figure 32. A new batch deactivation curve was not developed for Run 254 because data were already available with Shell 317 catalyst from Run 253. The limited data available during Run 254 were used to create an "Estimated Batch Deactivation Curve" for the same type catalyst but Ohio No. 6 coal.

The experimental rate constants for the first stage catalyst were calculated and are presented as data points in Figure 33. The theoretical line shown in the plot is calculated using the "Estimated Batch Deactivation Curve" and illustrates the activity level which the catalyst was expected to be able to maintain at a stable catalyst addition/withdrawal rate of 3 lb/ton. Although the theoretical and the experimental rate constants are very close, the experimental values were higher during 254J-L than theoretically projected. It should be noted that unlike the overall analysis, the first stage catalyst activity analysis and the second stage catalyst analysis are totally dependent on the interstage sample for resid + UC conversion. Because this sample experiences a wide range of variability, data scattering in the catalyst data analysis is very common.

Figure 34 is a similar plot, but presents an analysis of the first stage rate constant based on equilibrium activity. Although previous catalyst activity analysis have been performed using only the second stage resid + CI catalyst age to indicate time, Figure 34 illustrates these regressions with run time based on second stage resid + CI age and with run time based on operational days.

Equilibrium activity was achieved relatively early in the run. Figure 34 analysis indicates that catalyst activity was within the standard deviation of equilibrium as early as 20 run operation time days. As shows on the plot, the equilibrium rate constant was calculated to be $0.353 \pm 0.085 hr^{-1}$. A linear regression was done on these points to determine only a slight deactivation of 0.000009 (slope). As expected for a horizontal line the coefficient of determination, r^2, was also low at 0.011. Data prior to 20 days of operation was also analyzed using the linear regression technique. However, due to the scatter of the data and poor correlation coefficient, the estimated batch deactivation curve was used to predict catalyst activity.

It should be noted that the first stage rate constant is an overall value that includes both thermal and catalytic effects on first stage conversion.

5.3.3 Second Stage Catalyst Activity

A similar analysis was conducted to compare the second stage catalyst activity. Figure 35 shows the second stage catalyst deactivation trends which have the following results:

	254F	254G,254H,254J	254K-254L
MF Coal Feed Rate (lb/hr)	370	370,300,440	440,375
Temperature, °F	760	790	810
Slope	$1.3\text{x}10^{-4}$	$0.9\text{x}10^{-4}$	$4.0\text{x}10^{-4}$
Intercept	-0.3604	-0.0303	1.2461
r^2	0.014	0.345	0.414

Using the results from Figure 35 the second stage deactivation coefficients were also calculated for selected periods.

	2nd stage ln(K)	Deactivation Coefficient (α)
254F	-0.360-0.00013t	0.00013
254G-254J	-0.030-0.00009t	0.00009
254K-254L	1.246-0.00040t	0.00040

The second stage reaction temperature increases from 760°F to 790°F, and further to 810°F significantly increased catalyst activity levels. However the rate of deactivation for the second stage catalyst at 810°F was signficantly higher than at 790°F or 760°F. Figure 36 shows an extended plot of batch catalyst aging data where deactivation rates were projected back to compare deactivation trends at different second stage temperatures. Comparing the extrapolated data for the second stage at 810°F to the first stage experimentally measured data, the second stage catalyst activity is projected to be higher and the second stage deactivation rate (slope) is projected to be slightly lower. These observations at 810°F second stage reaction temperature need to be studied further due to the limited data points at the high catalyst ages. Second stage equilibrium catalyst age was 1600-2100 lb MF coal/lb catalyst.

It is noteworthy that a decrease of the second stage catalyst activity for period 254I at 300 MF lb/hr low coal feed rate was observed, when compared to those for periods 254G-H and 254J (Figure 35). The first stage catalyst activity was not significantly changed due to coal feed rate variations (Figure 34). Further studies are necessary to better understand and explain this apparent deviation from the assumption of CSTR first order kinetics used for catalyst evaluations. Three possible explanations are: (1) interaction of feed reactivity, (2) second order kinetics, and (3) two different types of feeds. Future modeling studies will include these new concepts in development of reaction kinetics and reactor design expressions.

5.4 Parity Plots and Catalyst Requirements Calculations

Parity plots and catalyst addition/withdrawal requirements are calculated using a first order kinetics model for the following conversions:

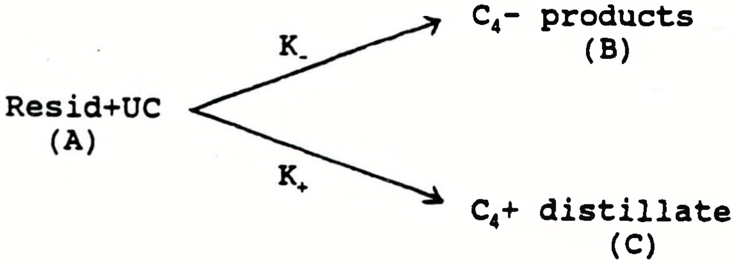
First stage conversion:

$$\text{Resid+UC} \xrightarrow{K_1} C_4\text{+ distillate} + C_4\text{- products}$$

Second stage conversion:

$$\text{Resid+UC} \xrightarrow{K_2} C_4\text{+ distillate} + C_4\text{- products}$$

Combined first and second stage conversions:



$$\begin{aligned} -r_A &= KC_A = (K_+ + k_-)C_A = r_B + r_C \\ r_B &= k_-C_A \\ r_C &= K_+C_A \end{aligned}$$

In these equations, K_1 and K_2 are the first and second stage resid + UC conversion rate constants, respectively. The rate constants K_+ and K_- respectively represent the production of C_4+ distillate and C_4- products (gases + H_2O) in the combined first and second stage conversion scheme.

In the combined first and second stage conversion, the fraction of C_4+ distillate produced per pound of resid + UC converted is defined as f (experimental value of f ranges from 0.768 to 0.839), and so the rate constants K_+ and K_- are given by

$$\begin{aligned} K_+ &= fK \\ K_- &= (1-f)K \end{aligned}$$

The rate constant K_+ can be related to the overall C_4+ distillate yield, Y_C' (weight % MAF/100), using

$$K_+ = f\ \text{WHSV}_1 \ \frac{Y_C'}{fC - Y_C'}$$

where C is a unit conversion factor of resid + UC conversion from weight % feed to weight % MAF coal (2.34-2.41). Rearranging the above equation for Y_C' and substituting $K_+=fK$, the distillate yield is given by

$$Y_C' = C\,f\,\frac{K}{WHSV_1 + K}$$

$$= C\,f\,\epsilon$$

where ε is the overall resid + UC conversion (= $\varepsilon_1+(1-\varepsilon_1)\,\varepsilon_2$). In terms of the first and second order resid + UC conversion rate constants, the overall C_4+ distillate yield is given by

$$Y_C' = C\,f\,\left[\frac{K_1}{WHSV_1 + K_1} + \frac{WHSV_1}{(K_1 + WHSV_1)}\,\frac{K_2}{(WHSV_2 + K_2)}\right]$$

Using the rate constant equations obtained by correlating the experimental data, the distillate yield is calculated and compared with experimental yield in the following table. A parity plot involving the distillate yields is given in Figure 37. It should be noted here that these comparisons are made using Phase 2 data since in this run an interstage separator was not used. First and second stage conversions could not be obtained using Phase 3 calculations since complete analysis of interstage process stream was not available.

This table also compares experimental coal feed rates with those predicted using the rate constant equations. For a run period, the predicted feed rate corresponds to the experimentally obtained distillate yield. The feed rate can be calculated using the above equation and the experimental value for the ratio of $WHSV_2/WHSV_1$. A parity plot involving coal feed rates is given in Figure 38.

The comparisons in the table and the parity plots give an indication of how well the experimental rate constant data are correlated using the first order kinetics. Because of large scatter in data, the errors obtained in correlating rate constants may not be sufficient to judge the accuracy of the correlation. Alternatively, the parity plots in Figures 37 and 38 and the errors in distillate yield and coal feed rates reported in the table, suggest that the rate constant correlations are accurate except for run period 254I. Thus, first order kinetics model is sufficiently accurate in the range of experimental data.

Experimental and Model Predicted Coal Feed Rate and Distillate Yield Comparisons

					Model Predicted		Experimental		C_4+ dist. yield wt % MAF coal			Coal feed MF lb/hr		
Run	Stage	Temp. (°F)	Rate constant (K), hr^{-1} $\ln K = [\ln A - E/RT] - \alpha t$	Catalyst age (t) lb res+CI/lb cat	Rate constant (K), 1/hr	Conversion wt % feed	Rate constant (K) 1/hr	Conversion wt % feed	Model (a)	Expt.	Error % (c)	Model (b)	Expt.	Error % (c)
254F	1st	810	0.74-0.00039t(d)	899	1.48	25.6	1.50	25.8	71.0	69.4	2.3	378	370	2.1
	2nd	760	-0.43-0.00005t(d)	916	0.62	12.9	0.55	11.5						
254G	1st	810	0.35	1064	1.42	24.8	1.53	26.3	74.2	74.6	-2.2	362	370	-2.2
	2nd	790	-0.03-0.00009t	1250	0.87	17.2	0.78	15.7						
254H	1st	810	0.35	1294	1.42	24.8	1.28	23.0	73.9	71.5	3.3	382	370	3.1
	2nd	790	-0.03-0.00009t	1760	0.83	16.8	0.84	17.0						
254I	1st	810	0.35	1423	1.42	28.9	1.31	27.2	82.2	74.1	9.9	344	300	12.8
	2nd	790	-0.03-0.00009t	2415	0.78	18.7	0.60	14.9						
254J	1st	810	0.35	1508	1.42	21.8	1.36	21.0	61.1	61.5	-0.7	429	440	-2.6
	2nd	790	-0.03-0.00009t	2843	0.75	13.3	0.82	14.4						
254K	1st	810	0.35	1505	1.42	21.8	1.54	23.2	62.9	62.4	0.8	441	440	0.2
	2nd	810	1.25-0.00040t	3316	0.93	16.2	0.80	14.3						
254L	1st	810	0.35	1508	1.42	24.8	1.44	25.1	66.3	64.1	3.3	392	375	4.3
	2nd	810	1.25-0.00040t	3766	0.77	16.1	0.65	14.0						

(a) C_4+ distillate yield predicted for the experimental coal feed rate.
(b) Coal feed rate predicted for the experimental C_4+ distillate yield.
(c) % error = (1 - experimental/predicted) x 100.
(d) Estimated batch deactivation rate (using Run 253 Illinois No. 6 coal data).

The coal feed rates projected to achieve resid extinction with a common organic rejection of 8 wt % MAF coal are given in the next table along with mean catalyst ages. The projected coal feed rates for the given achieveable distillate yields are calculated as described above. The catalyst addition/withdrawal rates necessary to maintain catalyst ages at a steady-state at the projected coal feed rates are also given in the next table along with the experimental addition/withdrawal rates. For example, for period 254L, the addition/withdrawal rates necessary to maintain catalyst ages at 618 and 1981 lb MF coal/lb catalyst in the first and second stages are 2.97 and 0.98 lb cat/ton MF, respectively. With these addition/withdrawal rates and a coal feed rate of 351 lb MF/hr, the distillate yield will be 69.2 wt % MAF coal with an organic rejection of 8 wt % MAF coal and resid extinction. It is important to note that the addition/withdrawal rates calculated are only valid for the experimental run conditions of catalyst age distribution, catalyst activity, and the deactivation rate.

The above addition/withdrawal rate calculations are based on the assumption that the rate equations are valid at the projected coal feed rates. This is a good assumption unless the coal feed rates are extremely low as in the case of 254I. Again, these calculations are only valid for the experimental run conditions of catalyst age distribution, activity and deactivation rate. Catalyst ages can be maintained with the addition/withdrawal rates calculated above, but the catalyst activity is continuously changing since the age distribution changes with every addition/withdrawal. For design calculations as well as for comparison between various runs, it is useful to calculate addition/withdrawal rates necessary to maintain an equilibrium activity level. Such calculations take into account an equilibrium catalyst age distribution and activity levels and deactivation rates over a broad range of catalyst ages. If the experimental data is available over a wide range of catalyst ages, then addition/withdrawal rates based on equilibrium activity levels is more accurate and useful in design calculations.

Projection of Coal Feed Rate for Resid Extinction, and Calculation of Steady State Catalyst Addition

Run	Stage	Temp (°F)	Coal feed lb MF/hr	Yield (wt % MAF coal)			Achievable C_4+ dist yield (a) (wt % MAF coal)	Projected coal feed rate (MF lb/hr)	Catalyst age (lb MF coal)/lb cat	Calculated steady-state catalyst A/W (lb/ton MF)	
				C_4+ dist	Organic rejection(b)	Resid(b)				(c)	Total
254F	1st	810	370	69.4	10.3	4.6	74.2	344	368	4.73(3.0)	8.28
	2nd	760							509	3.55	
254G	1st	810	370	74.6	8.2	-0.7	74.2	364	439	4.02(3.0)	6.71
	2nd	790							685	2.69	
254H	1st	810	370	71.5	9.2	4.5	75.6	353	529	3.42(3.0)	5.41
	2nd	790							947	1.99	
254I	1st	810	300	74.1	6.7	2.0	74.6	341	583	3.14(3.0)	4.63
	2nd	790							1288	1.49	
254J	1st	810	440	61.5	7.2	14.3	69.8	360	609	3.0(3.0)	4.29
	2nd	790							1499	1.29	
254K	1st	810	440	62.4	8.1	10.5	69.0	383	615	2.96(3.0)	4.08
	2nd	810							1726	1.12	
254L	1st	810	375	64.1	7.4	8.5	69.2	351	618	2.97(3.0)	3.95
	2nd	810							1981	0.98	

(a) Assuming 8 wt % MAF as achievable organic rejection.
(b) From Phase 3 analysis.
(c) Catalyst A/W experimentally conducted at the plant are shown in parentheses.

6.0 DISTILLATE PRODUCT QUALITY AND UNIT SOLVENTS

The primary products produced in Run 254 were distillates defined by gas chromatography as naphtha (IBP-350°F), light middle distillate (350-450°F), heavy middle distillate (450-650°F) and distillate solvent (650°F-EP). These distillates were also analyzed on an elemental basis to determine carbon, hydrogen, nitrogen, and sulfur. Oxygen was found by difference.

For Run 254 the interstage separator was not in use and the entire effluent stream from the first stage reactor was fed to the second stage reactor. Therefore, no distillate product was obtained from the first stage. The product distillate in this mode of operation consisted of distillate from the second stage only:

1) overhead flashed distillate from the second stage reaction product and
2) overhead flashed distillate from the vacuum flash system for second stage reaction product.

These two distillate streams are combined and processed in the distillation system of the pilot plant. The distillation section consists primarily of an atmospheric distillation column and a vacuum distillation column. A flow diagram of the distillation section is shown in Figure 6.

6.1 Distillate Product Quality

Run 254G was selected for a product quality analysis period since the plant operated at conditions which produced essentially an all-distillate yield slate. A modified product quality analysis procedure was used due to a poor material balance across the distillation system.

Based on the Phase III material balance for Run 254G, 27.4% of the solvent fed to the distillation section came from Vacuum Flash Overhead (V1072) and 72.6% came from Flashed Distillate Bottoms (V1078). An elemental analysis of the laboratory blend which was prepared in these proportions is shown below:

	Wt %				
	C	H	N	S	O(diff.)
27.4%/72.6% blend	88.2	11.0	0.38	0.01	0.41

The specific gravity (60/60°F) of the blend was 0.968 and the boiling point range was between 69.5°F and 937.4°F.

The distillate blend was fractionated in a laboratory scale Oldershaw column to simulate results expected in the distillation section of the plant. The amount and elemental analysis of each distillate fraction are shown below:

Distillate Fraction	Wt % Produced	Wt % C	H	N	S	O(diff.)
IBP-350°F	19.1	86.0	13.8	0.10	0.01	0.09
350-450°F	6.7	87.6	12.0	0.20	0.01	0.19
450-650°F	27.1	88.8	10.9	0.27	0.01	0.02
650°F-EP	47.1	90.0	9.5	0.45	0.01	0.04

The specific gravity (60°F/60°F) of each fraction was also analyzed.

IBP-350°F	0.791
350-450°F	0.911
450-650°F	0.970
650°F-EP	1.067

It is interesting to note that both the specific gravity and the elemental analysis of the original blend is closest to the 450°F-650°F fraction.

Of the total distillate yield, 22 wt % was required for coal slurry preparation in plant operation. To account for this recycle requirement in the laboratory, 22.2 wt % of the 650°F-EP fraction was removed and the product blend was normalized to an overall distillate yield distribution of:

	Overall Distillate Yield % MAF
IBP-350°F	24.6
350-450°F	8.6
450-650°F	34.8
650°F-EP	32.0

The total blend of product distillate was 88.2% carbon, 11.3% hydrogen, and 0.33% nitrogen. The endpoint of the G.C. distillation was 904.9°F. A refinement on the endpoint calculations based on the gas chromatograph distillation can be made. The vacuum distillation column conditions are set to control the bottoms rate at that point which just satisfies the coal feed recycle requirements. Using the gas chromatograph analysis from the original product quality blend, an endpoint in the range of 750°F for product distillates is estimated.

A comparison of the product quality analysis between 254G on Ohio No. 6 coal and 253E-G on Illinois No. 6 coal is shown in Table 18. Table 18 reports only calculated product quality results based on a method which utilizes Phase III material balance results and daily GC analysis for 254G and 253E-G. This method was derived to compare these similar periods for which formal product quality data was not developed. Therefore, Table 18 was prepared for convenience of comparison and does not reflect actual product quality results for these periods. These periods in Run 253 are similar to 254G because operation was without the interstage separator and with catalyst relacement. The catalyst used in both runs was Shell 317. Although the resid content of the process solvent and the second stage temperature were both higher for 254G, the product distribution looks very similar to 253E-G.

6.2 Unit Solvents

Many different meanings are associated with the term "solvent" within this pilot plant. For example, pasting solvent is used in the coal slurry at start-up until process solvent is generated. Also, a proprietary deashing solvent is used in the CSD unit. A discussion of the different solvents has been broken down for each unit.

6.2.1 CCR Unit

The solvent fed to the CCR unit was the process derived solvent blend which was mixed in coal slurry preparation. The process solvent composition began with:

50% heavy distillate solvent,
12% CI's, and
38% resid

Prior to the 27 October shutdown, two short tests were performed to determine the unit operability with additional resid in the coal slurry. The first test was to dilute the slurry from 67% to 70% process solvent. This test was performed during the 14 October startup. Slurry viscosity was monitored and remained within current operating limits. The second test was to dilute the process solvent mixture to 51% resid just before the scheduled 27 October shutdown. Since both tests were considered successful, the continuation of Run 254 (period 254E-L) included the process solvent composition as :

38% heavy distillate solvent,
12% CI's, and
50% resid

A 70 wt % process solvent mixture was used in the coal slurry during 254F-L. Another attempt was made to increase the resid composition on 15 December, when the process solvent was changed to:

33% heavy distillate solvent,
12% CI's, and
55% resid

Unfortunately an interruption in operation resulted in a first stage reactor plug which was attributed to the increase in slurry viscosity. The process solvent composition was returned to 50% resid.

Since Run 254 operated without interstage separation, the feed to the second stage was the total effluent from the first stage. The coal slurry composition changes were also reflected in the interstage analysis below:

composition wt %	Interstage Analysis 254A-D	254E	254F
Solvent	44.8	39.6	38.4
CI	13.9	12.9	13.6
Resid	41.3	47.5	48.0

Period 254A-D is prior to any change in process solvent and 254E is with 50% resid in the process solvent. The additional 12% resid in the slurry increased the resid in the interstage sample by 6.2 wt %. Only a slight change in the interstage analysis was noted in 254F when the coal slurry was diluted to 70 wt % process solvent.

6.2.2 CSD Unit

The CSD unit uses proprietary deashing solvents to process material containing ash. These deashing solvents are identified by numerical designations for reporting purposes to protect the proprietary agreement. The deashing solvent was strengthened when possible to optimize resid recovery and energy rejection to the ash concentrate and yet to maintain efficient, stable first stage deashing. During Run 254, the CSD unit operated in Mode II of the DAS Recycle System. A summary of DAS losses is shown below:

DAS Losses

Operating Period	Total DAS Loss lb/hr	 Wt % Feed	Loss to Products lb/hr	 Wt % Feed	 Wt % Total Loss	DAS Type
254A	10.1	3.8	1.0	0.4	9.5	4100
254B	6.0	3.0	1.8	0.9	29.3	2104-2504
254C	9.2	5.4	0.5	0.3	5.1	2554-2604
254D	9.5	5.5	1.1	0.6	11.3	2604-2554
254E	8.7	4.0	1.3	0.6	14.5	2304
254F	7.0	3.9	1.9	1.1	27.4	2304
254G	6.3	3.2	1.8	0.9	28.5	2304
254H	2.2	1.4	0.3	0.2	12.1	2454-2504
254I	5.5	3.7	0.8	0.5	14.2	2604
254J	4.4	2.1	2.0	0.9	44.4	2654-2704
254K	5.5	3.3	0.9	0.6	16.8	2654
254L	4.6	3.0	1.4	0.9	30.7	2704

7.0 SPECIAL TESTS FOR RUN 254

This section was written to outline special tests performed during Run 254. All tables and figures are in the text of this section and are listed alphabetically to distinguish them from Tables and Figures in the body of the main Run Report. The following data was collected or analyzed in conjunction with the Advanced Coal Liquefaction Research and Development Facility.

7.1 Consol Data

Background

Forty-five oils from Run 254 were sent to Consolidation Coal Company for characterization. The set constituted fifteen samples of: the first stage product (R1235), the second stage atmospheric bottoms product (V1067), and the recycle (pasting) solvent (V131B). The recycle solvent is adjusted for resid content by combining vacuum oil, second stage atmospheric bottoms, and deashed resid from the critical solvent deashing unit. Samples were taken during stable operating periods. All of the oils contain ash, unconverted carbon, resid, and a significant distillate material that boils above 650°F. These oils are not net products but rather the major part of them exhibit their influence through recycling in the process. Hence, their composition and characteristics are indicative of process performance parameters such as catalytic hydrogenation and solvent quality. The composition of these oils can be influenced by a single or even a combination of process operating parameters such as catalyst age, reactor temperature, and space velocity. Table A contains a summary of process operating conditions that are most likely to influence the composition of the oils.

TABLE A. SUMMARY OF OPERATING CONDITIONS FOR RUN 254

				1st Stage				2nd Stage		
Period	Run day	Coal ash, % MF	Resid in process solvent, wt %	Catalyst addition withdrawal	WHSV lb feed/hr/ lb/cat	Catalyst age lb(res+CI)/ lb/cat	Temp., °F	Temp., °F	Catalyst age lb(res+CI)/ lb/cat	WHSV lb feed/hr/ lb/cat
254A	4.5	10.5	39.1	No	5.1	398-480	810	761	296-358	4.9
254B	14.1	9.5	39.0	No	4.0	865-1302	811	760	578-914	3.8
254C	26.6	6.0	40.9	Yes	3.8	1171-1229	811	761	1113-1159	3.7
254D	28.8	6.2	39.8	Yes	3.9	1138-1200	811	789	1258-1449	3.7
254E	43.8	6.4	49.4	No	5.2	384-474	810	761	307-379	3.0
254F	53.3	7.7	49.6	Yes	4.3	830-967	811	760	833-999	4.2
254G	59.3	6.3	48.8	Yes	4.3	1003-1124	811	790	1166-1334	4.2
254H	68.8	6.5	48.5	Yes	4.3	1261-1332	811	790.	1703-1817	4.1
254I	82.8	5.9	50.5	Yes	3.5	1393-1452	811	790	2392-2437	3.4
254J	88.5	5.6	49.1	Yes	5.1	1451-1538	810	790	2774-2912	4.9
254K	93.5	5.4	49.3	Yes	5.1	1464-1558	810	810	3124-3467	4.8
254L	102.5	5.5	49.5	Yes	4.3	1482-1556	810	810	3690-3860	4.0

TABLE B. ^{1}H-NMR ANALYSES OF FIRST STAGE OIL (R1235)

	Whole Sample, %							Distillate, %							Resid, %						
	Aromatic		Cyclic		Alkyl			Aromatic		Cyclic		Alkyl			Aromatic		Cyclic		Alkyl		
Period	Cond.	Uncond	Alpha	Beta	Alpha	Beta	Gam	Cond.	Uncond	Alpha	Beta	Alpha	Beta	Gam	Cond.	Uncond	Alpha	Beta	Alpha	Beta	Gam
254A	14.6	5.8	14.0	17.2	8.8	23.1	16.5	10.4	5.7	15.7	19.5	9.2	25.5	13.9	22.3	2.7	21.4	17.7	9.3	18.0	8.6
254B	16.8	3.8	18.1	18.6	9.6	21.7	11.2	8.8	5.8	15.8	20.3	9.4	26.0	13.9	22.7	2.9	21.4	17.3	9.2	17.8	8.6
254C	167.0	5.9	16.6	17.4	9.8	20.8	13.5	10.3	6.3	14.9	19.3	9.7	25.3	14.2	23.0	4.3	21.2	16.7	9.5	16.1	9.0
254D	20.1	4.9	17.5	16.2	9.3	20.3	11.7	12.9	7.3	13.8	17.9	9.4	24.6	14.2	25.7	4.2	20.9	15.0	9.0	16.3	8.9
254E	18.1	5.2	17.0	16.7	10.0	20.2	12.7	10.9	6.6	14.7	18.8	10.3	24.6	14.2	24.9	3.6	21.0	6.3	9.5	16.3	8.5
254F	17.2	4.5	18.8	17.3	9.5	20.4	12.3	10.2	6.0	15.8	20.2	9.7	24.3	13.7	22.4	3.5	20.2	17.0	9.0	17.6	10.2
254G	18.8	3.6	18.2	17.4	9.5	20.4	12.1	10.3	6.4	15.8	19.8	9.8	23.8	14.2	23.3	4.7	19.0	15.8	8.8	17.8	10.6
254H	19.5	5.2	17.8	16.7	9.4	19.2	12.3	10.6	6.9	15.6	19.3	10.1	23.7	13.8	25.5	3.9	21.6	15.6	9.6	15.4	8.4
254I	19.7	4.7	17.8	16.9	9.2	19.3	12.3	10.4	6.8	14.7	19.4	10.0	24.1	14.6	27.1	3.3	21.6	15.9	9.1	15.3	7.7
254J	21.5	5.1	18.6	16.0	9.8	18.1	10.9	11.6	7.3	14.5	18.2	10.4	23.7	14.4	28.0	4.3	21.8	14.7	9.5	14.2	7.6
254K	23.5	5.0	18.9	15.6	9.8	16.8	10.4	13.5	7.4	15.9	17.7	10.6	22.1	12.7	29.4	3.1	22.6	14.9	9.7	13.4	6.9
254L	22.9	5.3	19.1	15.4	9.8	17.1	10.5	13.0	7.4	15.7	18.1	10.5	21.9	13.3	28.6	6.1	20.4	13.8	9.5	13.6	8.0

TABLE C. ^{1}H-NMR ANALYSES OF SECOND STAGE ATMOSPHERIC FLASH BOTTOMS (V1067)

	Whole Sample, %							Distillate, %							Resid, %						
	Aromatic		Cyclic		Alkyl			Aromatic		Cyclic		Alkyl			Aromatic		Cyclic		Alkyl		
Period	Cond.	Uncond	Alpha	Beta	Alpha	Beta	Gam	Cond.	Uncond	Alpha	Beta	Alpha	Beta	Gam	Cond.	Uncond	Alpha	Beta	Alpha	Beta	Gam
254A	14.9	3.2	17.4	20.3	8.7	22.9	12.4	9.2	5.5	15.2	20.6	9.0	25.8	14.6	19.2	2.5	20.3	19.3	8.8	19.9	9.8
254B	15.3	4.1	17.1	19.0	9.0	22.4	123.0	9.0	5.4	15.5	21.0	9.0	25.7	14.4	22.7	2.9	21.4	17.3	9.2	17.8	8.6
254C	15.5	5.2	16.5	18.3	9.3	21.7	13.5	9.5	5.7	15.6	20.8	9.2	24.7	14.3	23.0	4.3	21.2	16.7	9.5	16.1	9.0
254D	18.0	4.8	17.8	17.2	9.7	20.8	11.8	10.7	7.4	13.7	19.2	9.1	24.9	15.0	24.3	3.3	21.1	16.4	9.1	17.0	8.7
254E	18.2	4.0	19.1	16.8	10.0	21.1	10.9	10.3	6.5	16.2	19.8	9.9	24.1	13.2	22.3	3.0	21.3	17.9	9.4	16.7	9.3
254F	17.3	3.6	18.7	18.7	9.7	20.3	11.6	10.4	5.0	17.1	21.6	9.6	23.9	12.3	21.7	2.9	20.9	17.9	9.2	18.1	9.3
254G	18.1	5.0	17.2	17.2	9.8	20.2	12.6	10.8	6.4	15.8	19.7	9.5	23.8	14.0	23.8	3.0	20.7	16.7	9.1	17.3	9.4
254H	20.2	5.0	17.5	16.7	9.5	18.9	12.2	11.6	6.0	16.7	19.7	9.8	23.2	13.0	24.7	3.7	21.0	15.3	9.0	15.9	9.1
254I	21.7	4.2	18.6	16.6	9.1	18.7	11.1	11.8	6.2	16.2	19.8	9.5	23.0	13.5	26.5	3.1	21.4	16.1	8.8	15.8	8.4
254J	22.1	4.9	18.8	16.2	9.6	17.7	10.7	13.7	5.3	17.0	18.9	9.7	22.5	12.9	28.5	3.4	21.9	15.0	9.3	14.3	7.6
254K	25.1	5.4	18.3	15.1	9.6	16.4	10.1	15.6	6.6	16.5	17.2	10.1	21.5	12.5	30.4	4.3	21.2	14.2	9.2	13.6	7.2
254L	25.2	5.2	18.4	15.2	9.9	16.2	9.9	16.8	6.0	18.3	18.1	9.8	20.6	10.5	30.9	4.7	20.8	14.1	9.0	13.3	7.2

TABLE D. ^{1}H-NMR ANALYSES OF RECYCLE (PASTING) SOLVENT (V131B)

	Whole Sample, %							Distillate, %							Resid, %						
	Aromatic		Cyclic		Alkyl			Aromatic		Cyclic		Alkyl			Aromatic		Cyclic		Alkyl		
Period	Cond.	Uncond	Alpha	Beta	Alpha	Beta	Gam	Cond.	Uncond	Alpha	Beta	Alpha	Beta	Gam	Cond.	Uncond	Alpha	Beta	Alpha	Beta	Gam
254A	14.0	4.2	16.1	19.3	9.1	23.3	13.9	9.5	5.6	15.2	20.2	8.9	25.9	14.6	18.8	2.7	18.8	19.0	8.4	21.2	11.1
254B	14.8	4.1	17.0	19.1	8.9	23.1	13.1	8.8	5.4	15.4	20.9	9.0	26.0	14.4	18.4	4.0	16.9	17.5	8.1	22.0	13.0
254C	15.5	4.4	17.3	18.6	9.2	21.8	13.1	9.7	5.2	16.2	20.3	9.2	25.5	13.9	21.5	3.7	20.5	17.3	9.3	18.1	9.6
254D	19.3	6.1	16.5	16.7	8.9	20.8	11.6	14.9	10.2	13.4	17.7	8.2	22.6	12.9	22.9	3.9	19.4	16.7	8.4	18.4	10.2
254E	17.7	4.5	18.6	17.8	9.9	20.0	11.6	10.3	6.3	15.5	19.3	9.7	24.1	14.9	22.6	2.7	21.4	17.8	9.6	17.3	8.7
254F	16.3	4.9	17.3	17.9	9.6	21.2	12.8	10.1	5.6	17.0	20.7	9.7	24.2	12.8	21.4	2.5	21.2	18.4	8.9	18.2	9.5
254G	20.4	3.4	19.5	18.0	9.0	19.1	10.6	10.4	6.4	15.6	19.1	9.6	24.0	15.0	24.1	3.7	20.1	16.7	8.7	17.2	9.6
254H	21.8	3.6	19.5	17.0	9.3	18.6	10.2	11.0	6.4	15.4	19.1	9.3	24.5	14.9	24.5	4.7	19.5	16.0	8.8	16.5	9.9
254I	23.1	3.0	20.2	17.0	8.9	18.4	9.4	12.7	6.2	17.3	20.0	9.4	23.4	12.1	26.6	4.1	20.0	15.7	8.8	15.7	9.2
254J	23.1	4.1	19.8	15.9	9.5	17.7	9.9	13.3	6.5	15.8	18.0	9.4	23.1	13.9	27.3	4.4	20.5	15.3	9.1	15.0	8.4
254K	26.4	3.6	20.1	15.4	9.1	16.8	8.8	15.6	6.4	16.2	16.9	9.3	22.4	13.3	29.4	5.1	19.7	14.1	9.3	14.1	8.3
254L	26.4	4.0	20.0	15.5	9.3	16.2	8.6	16.4	5.6	18.9	17.9	10.0	20.2	11.1	29.8	4.6	20.3	14.1	9.1	13.9	7.7

Analyses

Consol performed ^{1}H-NMR analyses on all of whole sample oils and their respective distillates and resids. Table B, C, and D contain data for the first reactor product (R1235), second reactor atmospheric bottoms product (V1067), and the recycle solvent (V131B) respectively. Consistent with Consol reporting, the protons are lumped into three groups: aromatics, cyclics, and alkyls which are determined by signal location in the spectrum. To separate the distillates and resids, Consol distilled each oil under vacuum to an 850°F atmospheric equivalent end point. Resids and distillates were tested for phenols by FTIR (Fourier Transform Infra-Red). The distillates were evaluated under microautoclave coal conversions (modified equilibrium). Since similar tests were performed on whole sample oils, resid influence on coal conversion was judged by difference. Tables E, F, and G contain, excluding ^{1}H-NMR, the conversion data information for the interstage sample, the second stage product, and the process solvent sample. The data in Tables E, F, and G are complimented with Wilsonville laboratory analysis for hydrogen content on the respective oils, their distillates and resids and microautoclave equilibrium tests on the recycle solvent, V131B.

TABLE E. COMPONENT DISTRIBUTION AND PROPERTIES OF FIRST STAGE PRODUCT (R1235)

Period	Wt % of Sample				Phenolics, meq/g		Coal Conversion		Hydrogen %		
	850°F	THF Sol. Resid	IOM	Ash	Dist	Resid	Dist	Whole	Dist	Whole	Resid(a)
254A	32.1	48.1	7.1	9.0	0.24	0.69	70.8	68.6	9.73	7.53	7.17
254B	27.5	55.8	5.6	9.2	0.29	0.88	69.1	67.3	10.10	7.79	7.24
254C	32.3	52.5	4.9	8.0	0.39	1.05	68.8	71.4	9.92	7.61	6.80
254D	32.8	50.8	4.5	9.3	0.38	0.82	67.3	65.1	9.58	7.35	6.78
254E	27.5	57.0	4.5	8.1	0.42	0.81	68.9	58.5	9.47	7.37	6.97
254F	23.1	59.8	5.3	8.9	0.29	0.66	70.9	69.7	9.73	7.44	6.88
254G	24.8	60.8	4.4	7.7	0.33	0.66	71.2	73.1	9.58	7.45	6.15
254H	24.3	60.6	4.1	8.0	0.39	0.84	72.8	68.6	9.29	7.25	6.74
254I	28.1	56.1	4.5	8.3	0.35	0.78	72.8	73.4	9.43	7.33	6.76
254J	22.9	61.2	5.2	6.6	0.50	0.94	72.8	58.0	9.05	7.08	6.61
254K	26.3	58.6	4.5	7.5	0.50	0.89	75.1	72.4	9.03	6.98	6.60
254L	22.7	63.7	4.2	6.8	0.45	0.85	74.3	49.0	9.02	6.98	6.64

(a) CI-free.

TABLE F. COMPONENT DISTRIBUTION AND PROPERTIES OF SECOND STAGE PRODUCT (V1067)

Period	Wt % of Sample				Phenolics, meq/g		Coal Conversion		Hydrogen %	
	850°F	THF Sol. Resid	IOM	Ash	Dist	Resid	Dist	Whole	Dist	Whole
254A	32.6	46.3	8.1	11.6	0.14	0.37	78.4	82.7	-	7.50
254B	26.2	55.0	6.1	10.6	0.16	0.67	76.9	75.8	-	7.52
254C	27.6	56.6	4.7	9.0	0.23	0.74	74.3	67.2	-	7.50
254D	30.5	53.1	4.6	9.6	0.22	0.71	74.1	71.7	-	7.37
254E	23.6	59.6	4.4	9.1	0.31	0.56	77.3	70.7	-	7.44
254F	24.0	58.3	5.4	9.9	0.22	0.46	76.2	63.5	-	7.40
254G	23.5	59.9	4.6	9.6	0.22	0.40	77.5	77.8	-	7.20
254H	22.8	60.3	4.3	9.9	0.26	0.69	77.5	58.3	-	6.98
254I	20.7	63.2	4.7	10.1	0.23	0.59	79.1	66.5	-	6.76
254J	21.8	62.2	4.5	8.4	0.35	0.81	78.8	56.9	-	6.83
254K	20.7	63.8	4.8	8.6	0.36	0.77	78.5	57.8	-	6.66
254L	22.1	62.2	4.6	9.0	0.34	0.75	81.8	67.4	-	6.60

TABLE G. COMPONENT DISTRIBUTION AND PROPERTIES OF RECYCLE SOLVENT (V131B)

	Wt % of Sample				Phenolics, meq/g		Coal Conversion		Hydrogen %		
Period	850°F	THF Sol. Resid	IOM	Ash	Dist	Resid	Dist	Whole(a)	Dist	Whole	Resid(b)
254A	28.8	55.0	4.8	8.1	0.15	0.36	76.9	-	9.84	7.94	7.75
254B	26.0	61.1	4.1	7.1	0.16	0.51	75.1	78.8	9.94	7.91	7.42
254C	34.6	52.8	3.9	7.0	0.21	0.77	75.5	65.0(79.1)	9.86	7.35	-
254D	32.1	56.2	3.3	6.8	0.17	0.62	70.8	72.4	9.43	7.52	7.15
254E	20.7	65.0	3.8	7.9	0.30	0.55	77.8	76.5(78.3)	9.31	7.54	7.54
254F	22.7	62.9	4.3	7.8	0.21	0.48	75.9	74.8(79.1)	9.52	7.63	7.50
254G	23.8	61.3	4.0	8.3	0.21	0.46	78.0	75.0(78.1)	9.38	7.35	7.20
254H	21.1	66.3	3.5	7.3	0.25	0.67	77.6	54.5(75.3)	9.15	7.22	6.68
254I	22.0	64.0	3.6	7.2	0.23	0.59	78.2	69.3(78.6)	9.24	7.14	6.60
254J	23.2	63.1	4.0	7.2	0.34	0.77	79.2	58.6(67.4)	8.99	6.98	6.91
254K	21.3	66.6	3.8	6.9	0.35	0.74		59.4(69.3)	8.88	6.83	6.58
254L	20.0	65.5	4.1	7.5	0.33	0.72	81.4	49.4(67.0)	8.75	6.78	6.51

(a) (Wilsonville).
(b) CI-free.

Data Analysis and Interpretation

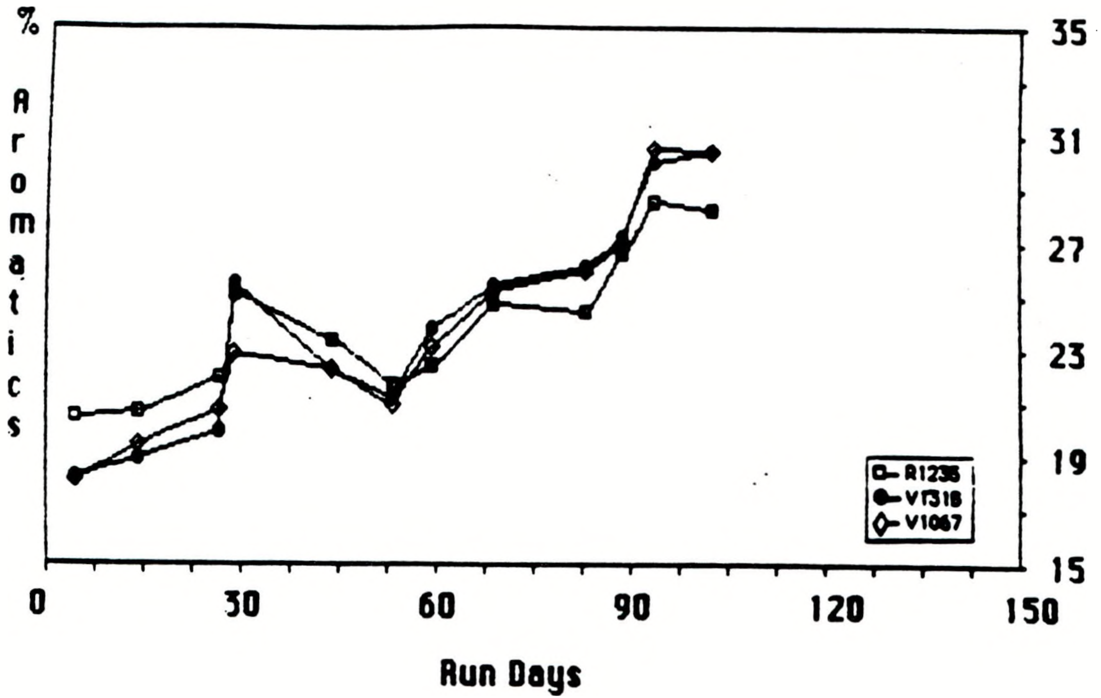
It is important to remember that Run 254 evolved into two parts. The first part of Run 254 covered about forty operating days and includes periods 254A-D. It is associated with mostly high ash coal, 40% resid in pasting solvent, fresh starting catalyst, and steady reactor temperatures. The second part of the run covered about sixty days and includes periods 254E-L. It is associated with low ash coal, 50% resid in pasting solvent, fresh starting catalyst, active catalyst replacement, second stage temperature changes, and high catalyst ages.

Most of the oil property data presented in this section was generated by Consol. Conclusions and data interpretation will relate closely to definitions and correlations of oil properties to process changes which have been defined in numerous Consol publications (5). Also, it should be noted that Consol's distillate end point (850°F) is about 200°F lower than Wilsonville's. Hence, the amount of distillate reported from the Wilsonville laboratory will be greater and the boiling components will be heavier. In a separate study, Consol compared typical properties (^{1}H-NMR, phenols, conversion) of their resids and distillates to a sample of resids and distillates from the Wilsonville laboratory. There was little difference and as predicted the resid and distillate from the Wilsonville sample was more aromatic and phenolic. In conclusion, the general trends observed in the Consol data about distillate and resid properties should apply to Wilsonville oils also.

7.1.1 ^{1}H-NMR Distribution in Oils

All of the oils increased in aromaticity as Run 254 progressed. This trend is shown in Figure A which includes aromaticity plots for all three whole sample oils. Compared to the aromatic levels at the start of the run, the first stage product (R1235) increased by 38.2% by the end of the run. The second stage product showed a large increase; it was 68.0% more aromatic at the conclusion of the run. The recycle solvent (V131B) increased 67.0% which was similar to second stage increase. The increase was evident in both parts of the run but more pronounced in the second part and was most likely due to the ever increasing second stage catalyst age associated with longer running time.

FIGURE A. AROMATICS IN WHOLE SAMPLES



However, there were periods in the second part of the run in which the increasing aromatic trend was temporarily reversed or held steady. The sharpest decrease happened between periods 254E (43.3 days) and 254F (53.3 days). The process changes which are suspected to cause this decrease include a lower space velocity for Period 254F and the beginning of catalyst replacement just prior to period 254F. Leveling trends are noted between periods 254H (68.8 days) and 254I (82.8 days) and also at the end of the run between periods 254K (93.3 days) and 254L (102.5 days). In both cases, the latter period had a lower space velocity which may have contributed to the leveling aromatic effect.

The sharpest increase in aromaticity for the entire run occurred in period 254D (28.8 days). This spike was found to be the result of a Dowtherm leak which was clearly identified in the Gas Chromatograph/Mass Spectrophotometry analysis. Dowtherm is a commercial heat transfer liquid commonly used in the process and is composed of biphenyl and biphenyl ether. The increasing aromaticity is prevalent in the first part of the run but the final level remains undetermined since Period 254D concluded the first segment.

The aromaticity for the distillate and resids from the respective oils are plotted in Figures B and C respectively. The resids show a greater increase in aromaticity than the distillates after comparison between the start of the run (254A) and the end of run (254L). The first stage resid increased 38.8%, the second stage resid increased by 64.1%, and the recycle solvent resid was similar at a 60.0% increase. Aromatic increases for the distillates were about 10% lower. The first stage distillate showed an overall increase of 26.7%, the second stage distillate increase was 55.1%, and the recycle distillate increased 45.7%. Therefore, the resids were more concentrated in aromatics than were the distillates, and resids showed a sharper increase in aromaticity as the run progressed. For whole oil aromaticity, the resid contribution was prevalent over the distillate but both contributed positively to the overall increasing trend.

FIGURE B. AROMATICS IN DISTILLATES

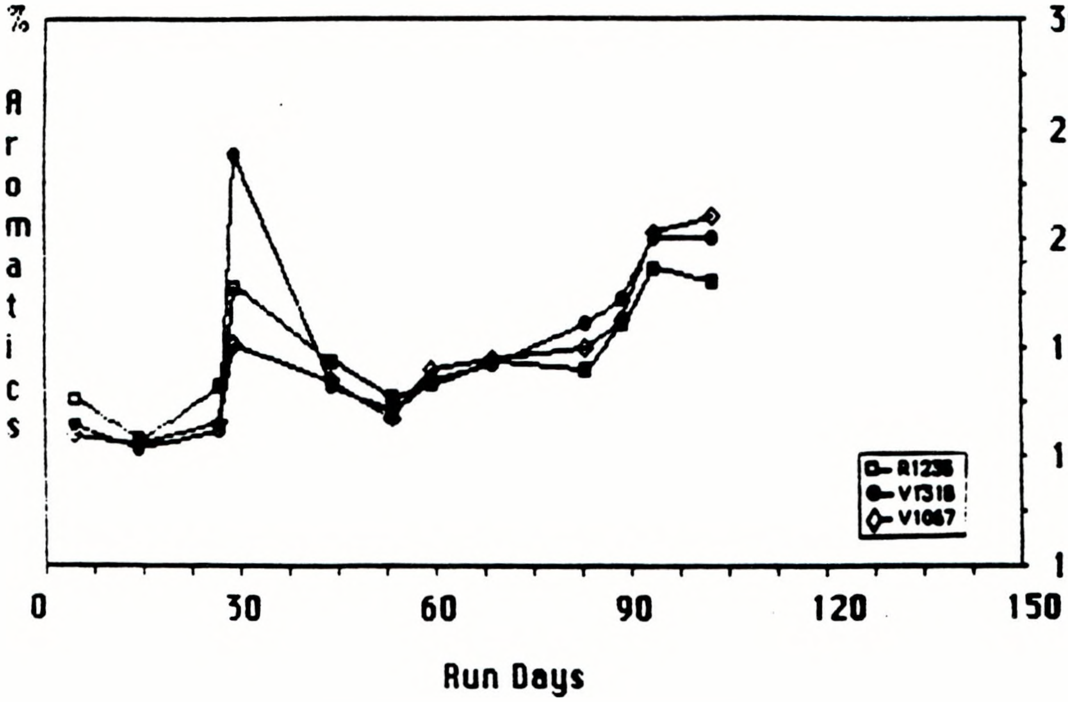
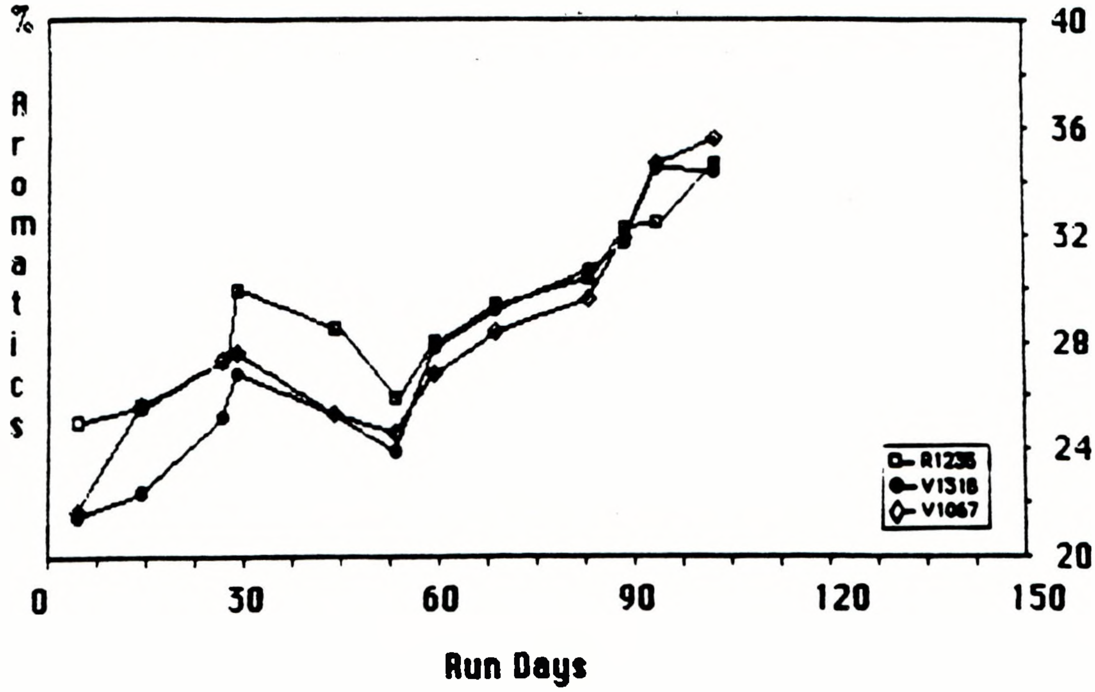


FIGURE C. AROMATICS IN RESIDS



The increased aromaticity of these oils indicated decreased hydrogenation due to lower catalyst activity as the run progressed. Also in support of this conclusion was the fact that the hydrogen content of all of the oils decreased as the run progressed. The trend for whole oil hydrogen content is shown in Figure D. Over the entire run, the first stage oil hydrogen decreased 7.3%, the second stage oil hydrogen decreased 12.0%, and the recycle solvent hydrogen decreased 14.6%. Period 254E (43.3 days) to 254F (53.3 days) was the only segment where steady hydrogenation was maintained. This period was mentioned previously for declining aromaticity and was associated with low space velocity and catalyst addition and withdrawal in the first stage.

Figure E shows similar trends in hydrogen plot for the distillates, and Figure F shows the hydrogen plot for the resids. The hydrogen content of the first stage distillate and resid decreased about 8.10% during Run 254. Distillate and resid in the second stage product was not isolated like the recycle solvent. However, the composition should be similar for the two streams. The distillate hydrogen for the recycle solvent decreased 11.1% and the resid decrease was 16.0%.

FIGURE D. HYDROGEN CONTENT OF WHOLE OILS

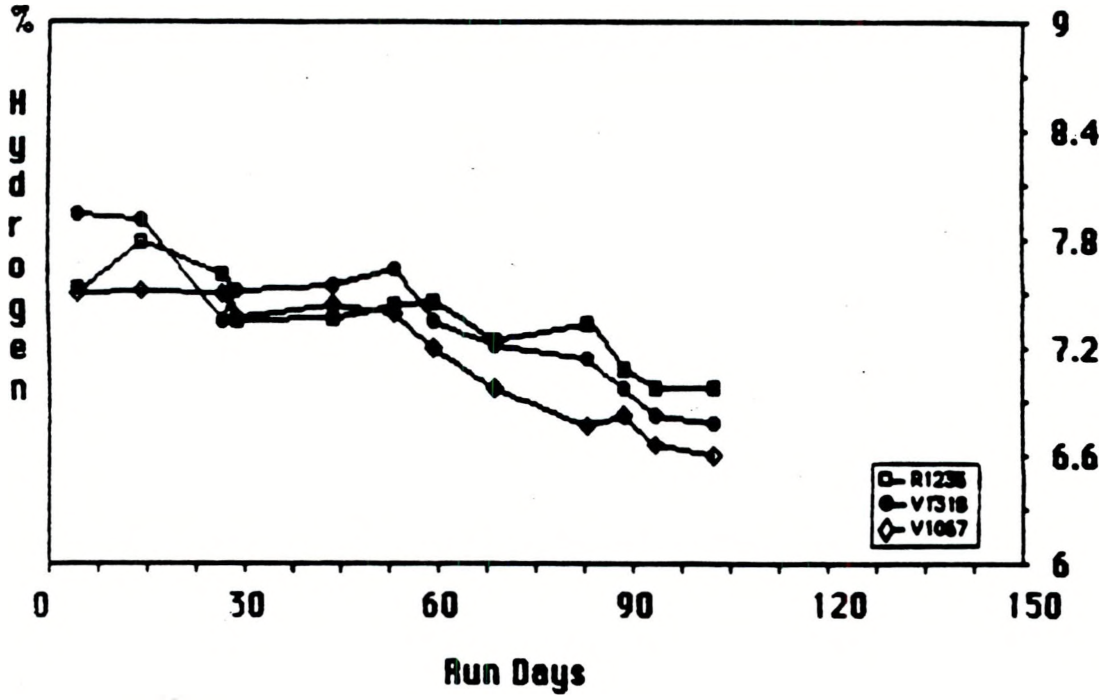


FIGURE E. HYDROGEN CONTENT OF DISTILLATES

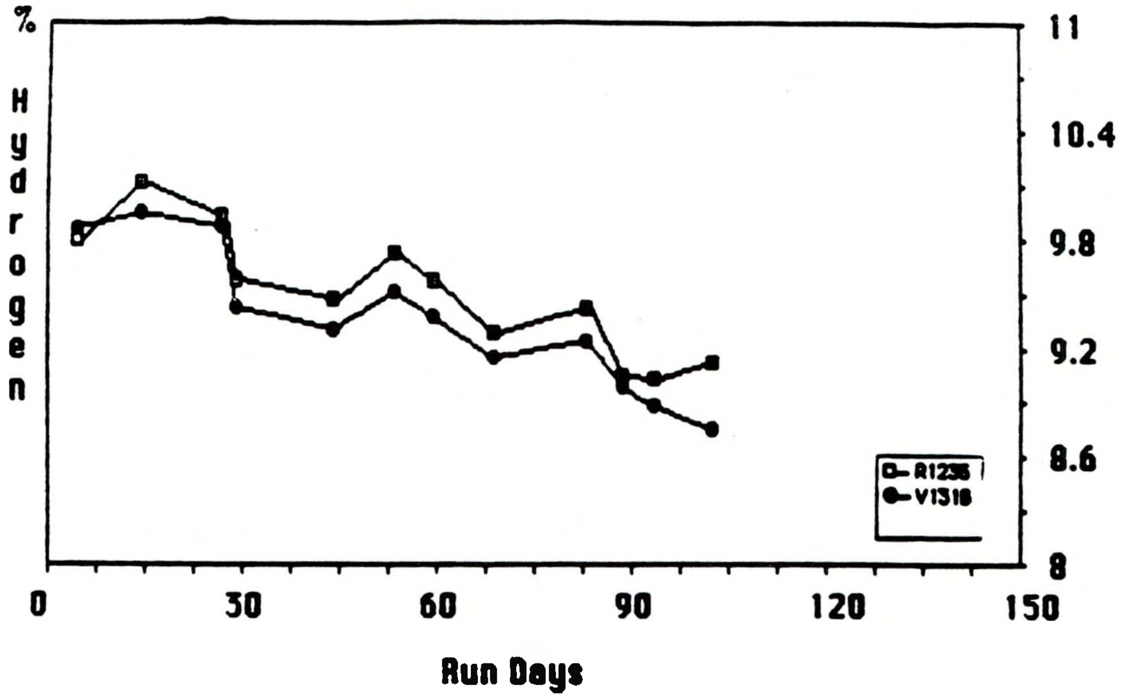
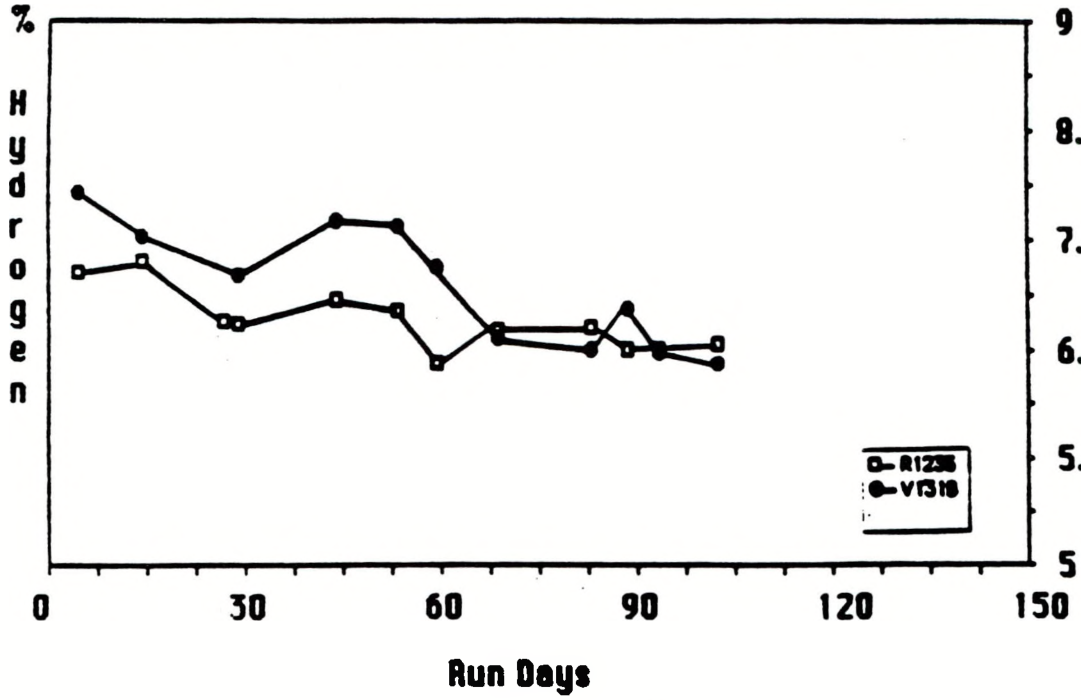


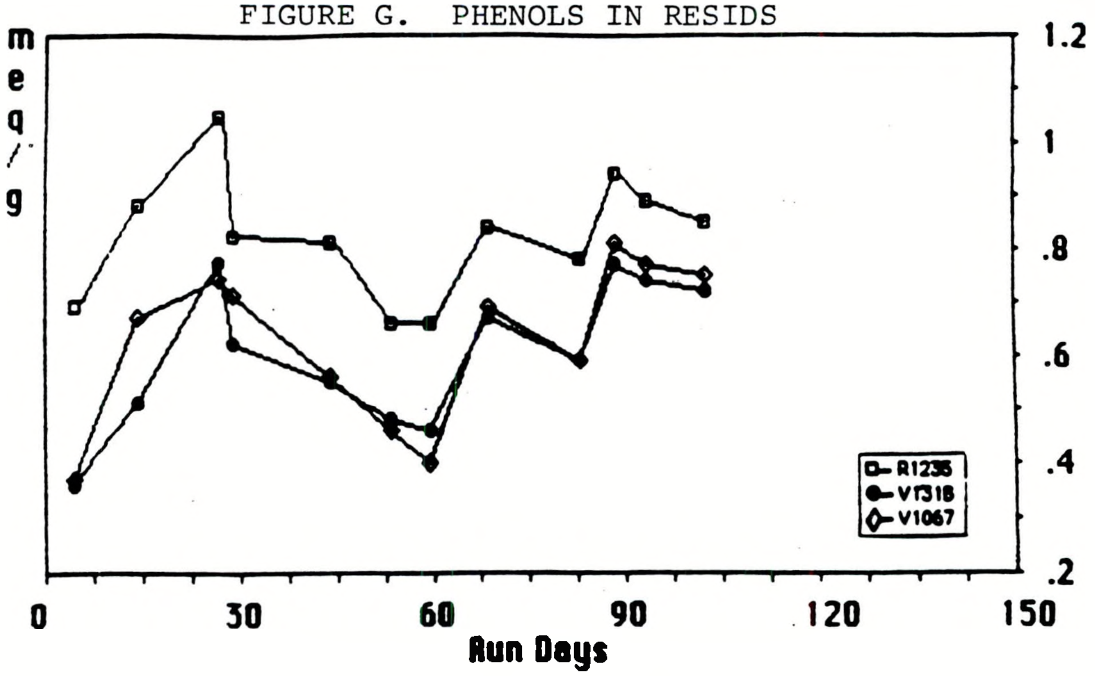
FIGURE F. HYDROGEN CONTENT OF RESIDS



7.1.2 Phenols in Oils

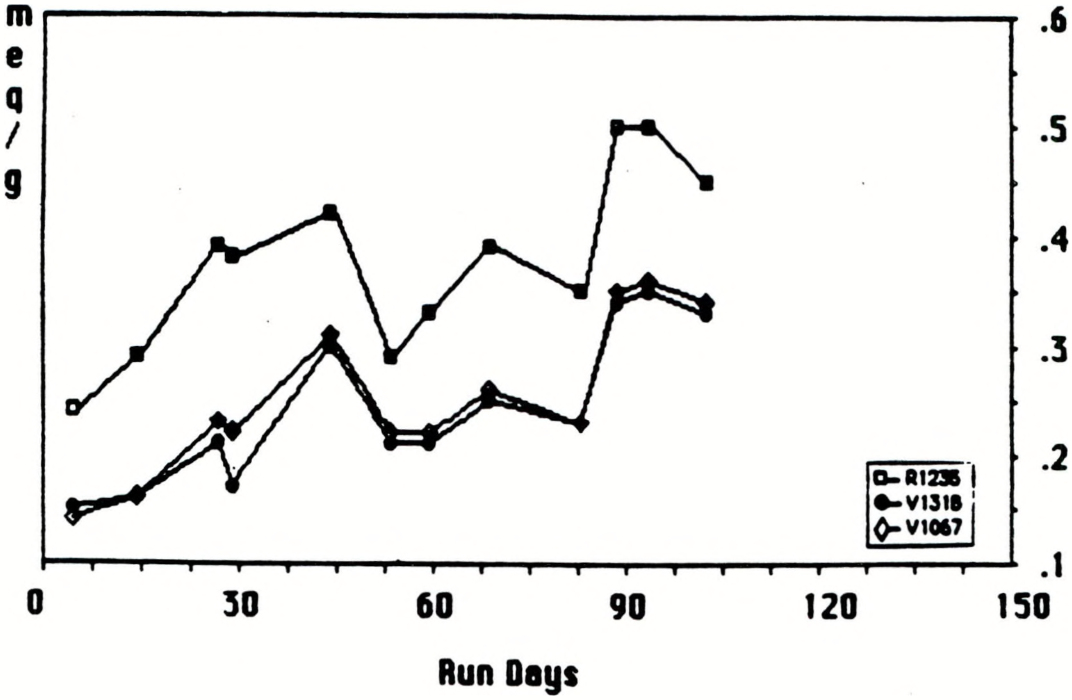
It is desirable to eliminate or minimize the presence of phenols in most all fuels due to its corrosive nature. An active catalyst, a low space velocity and a general trend of increasing reactor temperature was a good combination to remove phenols. Low (0.2-0.4 meq/g) to moderate (0.6-0.8 meq/g) describes the composition of phenolics in process oils for Run 254. The phenols analysis of the total sample was not determined, but could be approximated from the data in Tables E, F, and G (see Section 7.1.1). The average phenolics for the first stage product was 0.59 meq/g but the second stage product and recycle solvent were lower at 0.40 meq/g and 0.43 meq/g respectively. It is estimated that the phenolics level would have steadily increased without the first stage catalyst replacement program. Lower space velocity, first stage catalyst replacement, and second stage reactor temperature increases which worked toward diminishing the phenolic concentration also explained the lack of a dominating trend for Run 254 with regard to phenolics. Figure G shows the resid phenolics which constituted the major portion of phenolics for whole samples.

FIGURE G. PHENOLS IN RESIDS



From period 254C (26.6 days) to 254D (28.8 days) the decreasing trend related to a second stage temperature increase from 761°F to 789°F. A sustained decrease was also noted from Periods 254E (43.3 days) through 254G (59.3 days). Period 254F-G had positive factors for phenol removal which included decreasing space velocity, catalyst replacement, and increasing second stage temperature from 760°F to 790°F. Phenolics were more prevalent in all of the resids toward the end of the run due to higher space velocities. Increasing the second stage temperature from 790°F to 810°F for period 254J (88.5) to 254K (93.5 days) reduced the phenol content.

FIGURE H. PHENOLS IN DISTILLATES



Phenolics in the distillates, shown in Figure H, were about half the phenolics in respective resids. The changes in distillate phenols are similar to changes occurring in the resid phenols.

7.1.3 Solvent Quality

Donor solvent quality is determined by predominance of hydroaromatics in the recycle solvent. The ^{1}H-NMR data showed a fairly constant hydroaromatic concentration in the recycle solvent for Run 254. Coupled with increasing aromaticity and some periods of significant phenolic concentrations, the solvent quality would not have been predicted to show any improvement during Run 254. The analytical observation and the overall hydrogenation trend for Run 254 was toward poorer solvent quality as the run progressed. The Consol trend for solvent quality is shown in Figure I.

The microautoclave coal conversion indicated that the solvent quality was fair (70-80%) to poor (60-69%). Figure J shows the Wilsonville equilibrium microautoclave test values for recycle solvent with respect to run time. Although the trend was different from the Consol trend, poorer solvent quality toward the end of the run was indicated in both studies. The Wilsonville data indicated steady solvent quality (75-79%) until Period 254J (88.5 days). Afterwards, a significant drop was seen in the product quality with the average 68.0% for the remainder of the run. Obviously, with an already high catalyst age in the second reactor, increasing the space velocity from 3.5 to 5.1 between period 254I to 254J negatively influenced the solvent quality.

FIGURE I. COAL CONVERSION - WHOLE OIL

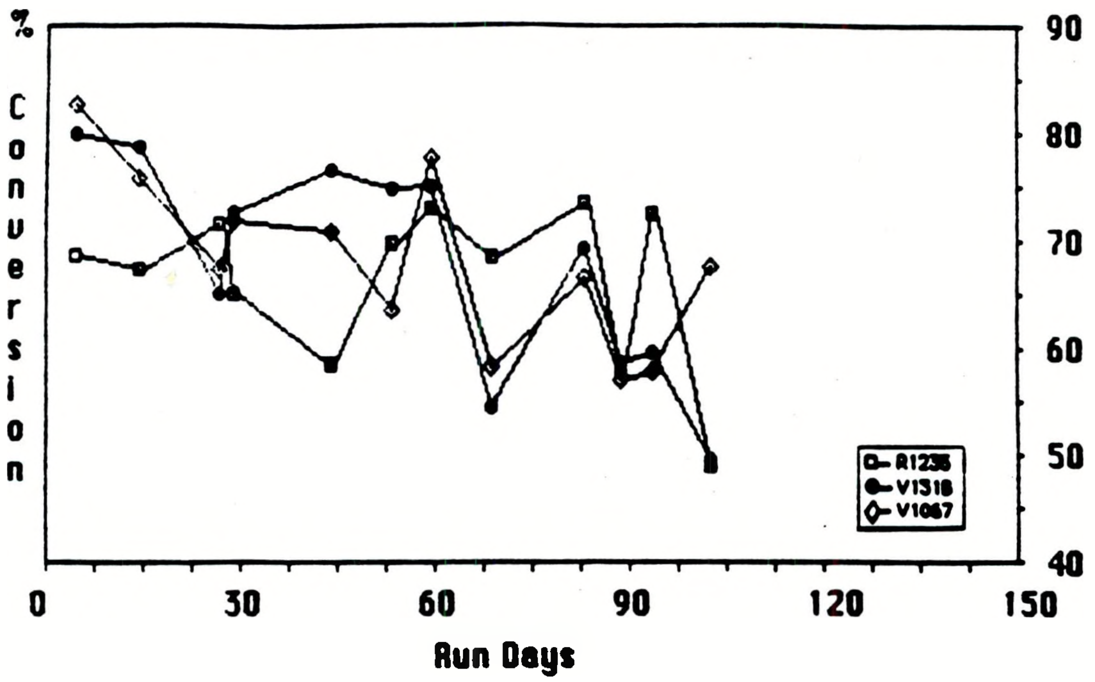
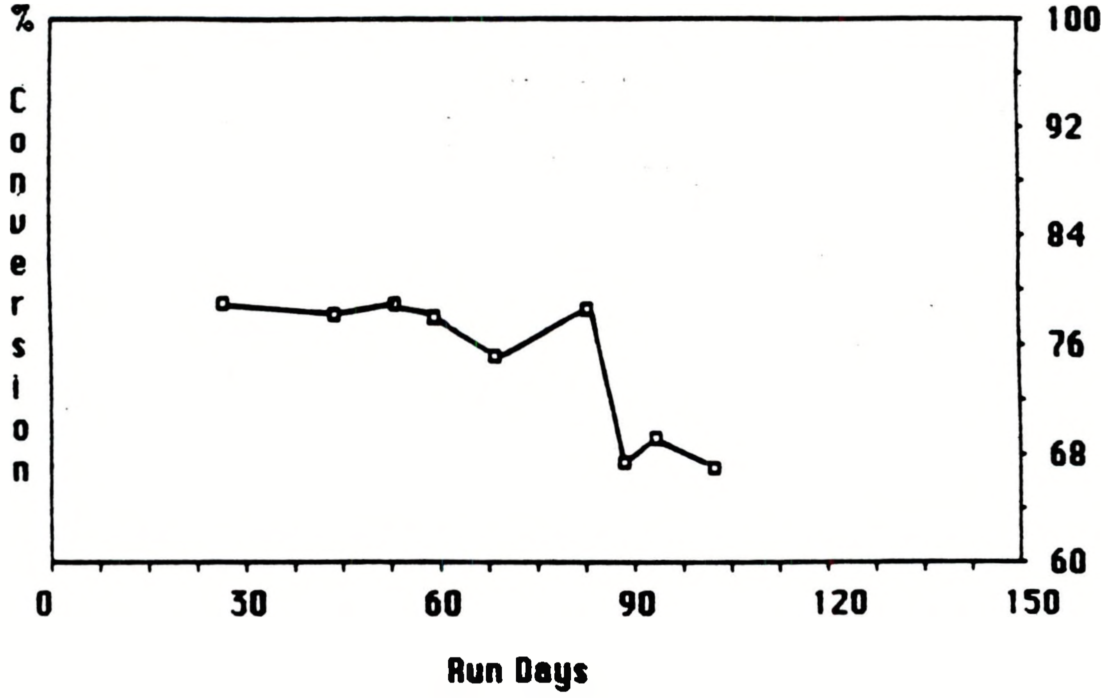


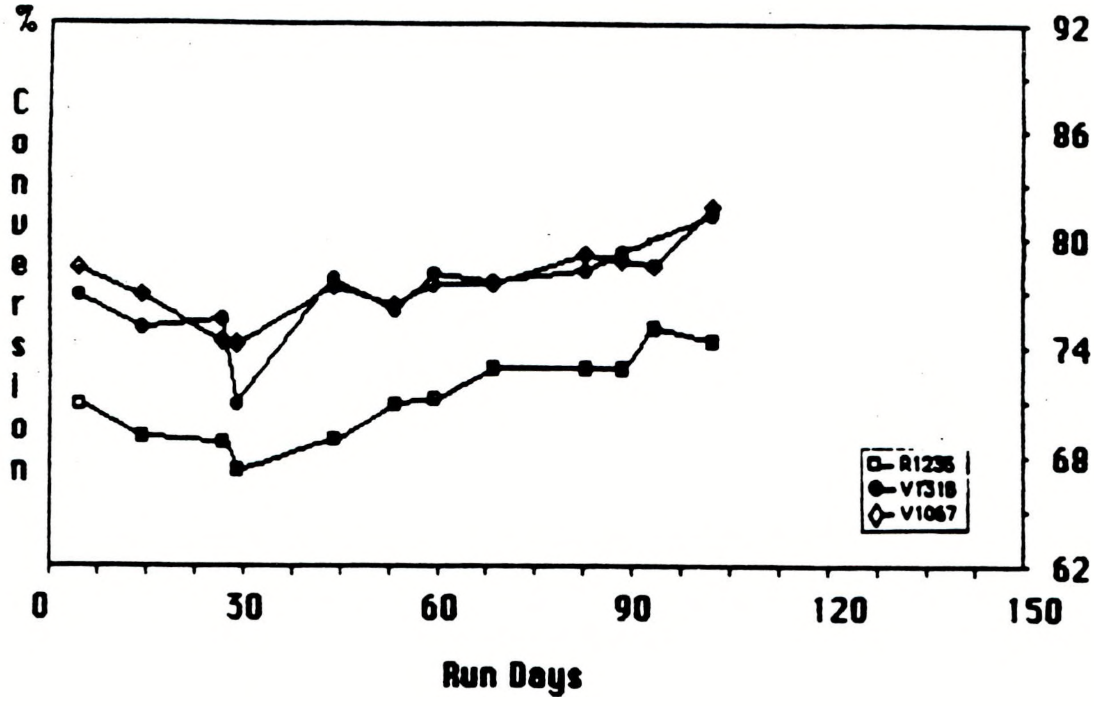
FIGURE J. COAL CONVERSION - WILSONVILLE (V131B)



Consol tested distillates for coal conversion which are plotted in Figure K. The solvent quality for the distillate portion was completely different from the whole sample and remained unchanged throughout the run. Good distillate solvent quality (80-85%) makes it apparent that the resid portion of the whole sample did not make a significant contribution to solvent quality. This was expected since the resid was more aromatic and phenolic than the distillate and the resid constituted the most significant part of the whole oil sample.

The effects of rather poor solvent quality were not that apparent. In thermal reactors, low solvent qualities indicated poor performance and even potential plugging problems. However, in Run 254, coal conversion was high, good distillate yields were achieved, and overall process performance was good. Poorer solvent quality does not affect operations as directly in the catalytic-catalytic close coupled mode. However, solvent quality makes the largest contribution to the yield structure. If the solvent quality had been higher for Run 254, the distillate yield may have been even better during periods of high space velocities.

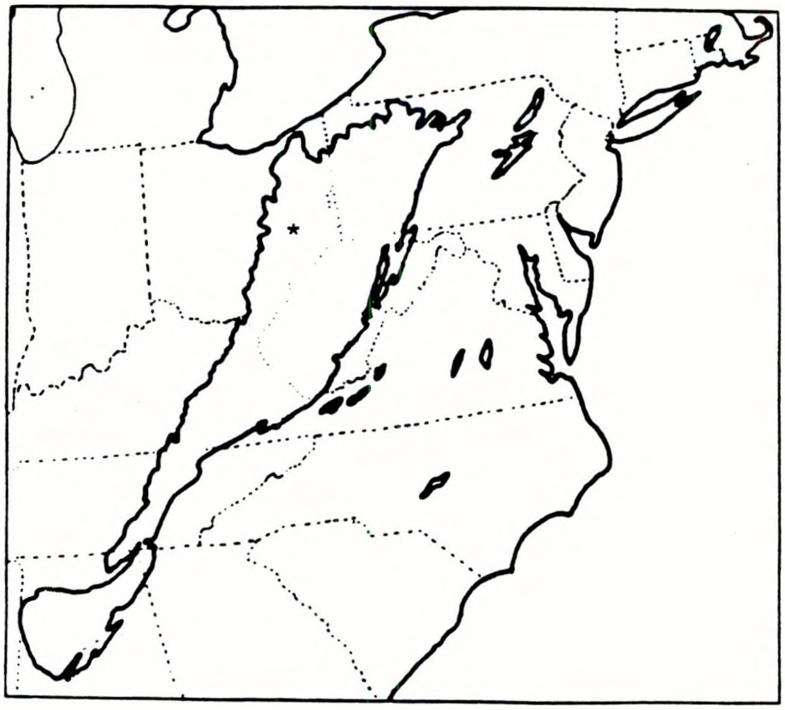
FIGURE K. COAL CONVERSION - DISTILLATES



7.2 Petrographic Analysis of Ohio No. 6 Coal

Coal utilized as feedstock in Run 254 originated from Crooksville, Ohio which is located in Perry County in Southeastern Ohio. The characteristics of the coal places it in the boundaries of one of the major coal regions of the U.S. known as the eastern province coal. Figure L shows this province which extends from Pennsylvania to central Alabama and includes portions of Rhode Island, Ohio, West Virginia, Kentucky, Tennessee, Georgia, Virginia, and minor regions of North Carolina and Maryland. The province is divided into three regions based on coal rank, physiography, age of strata, and structure. The anthracite region contains Pennsylvanian age coals of semianthracite and anthracite rank; the Atlantic Coast region contains minor traiassic age coals of bituminous rank. The Appalachian region is the most extensive and important region with mostly carboniferous coals of bituminous rank.

FIGURE L. THE EASTERN PROVENCE COAL BELT. SOURCE OF THE RUN 254 COAL SHOWN BY (*).



The Ohio No. 6 from Crooksville was in the Appalachian region and was part of the Alleghany formation. This formation includes the Kittanning and Freeport seams. The Ohio No. 6 coal which, in the Crooksville area, belongs to the middle Kittaning seam.

Within the Eastern Province, rank is the most important factor which controls the variability of the coal. Anthracite is the highest rank and high volatile B bituminous is the lowest. As rank continues to increase throughout the range, volatile matter and hydrogen decrease and reflectance increases. The data which is shown in Table H is for the Crooksville coal.

TABLE H. PETROGRAPHIC ANALYSES OF OHIO FEED COAL IN RUN 254[a]

Coal	Ohio No. 6	
Mine	Crooksville	
Designation	High ash[b]	Low ash[c]
Mean maximum reflectance (Ro), %	0.65	0.59
Maceral analysis, volume %		
Reactives	95.2	96.7
Vitrinite	86.2	90.1
Type 5	-	52.2
Type 6	86.2	36.1
Type 7	-	1.8
Exinite	8.2	6.0
Resinite	-	0.1
1/3 semifusinite	0.8	0.4
Inerts	4.8	3.3
2/3 semifusinite	1.6	0.8
Micrinite	2.2	1.2
Fusinite	1.1	1.2

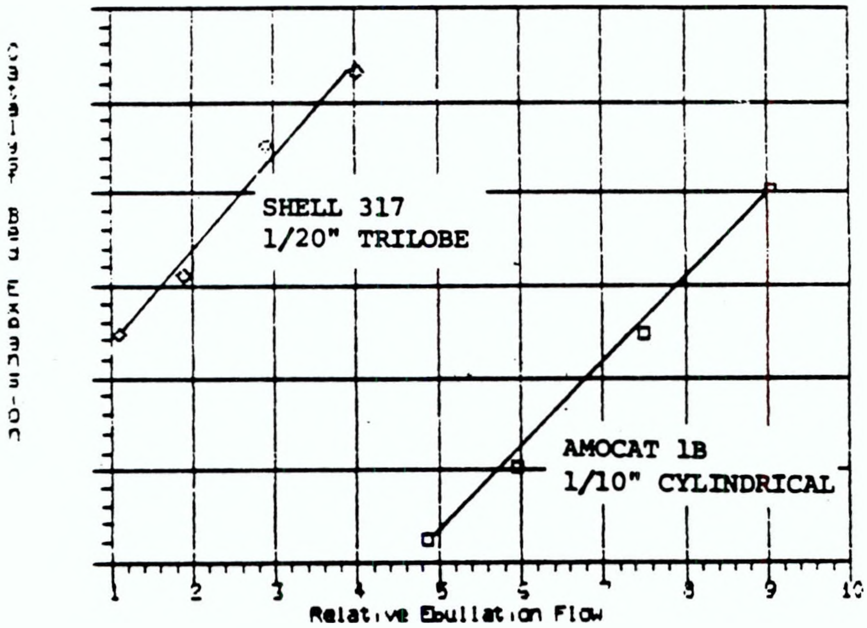
[a] Results reported on mineral free basis.
[b] Feed coal ash about 11.0% - used in first part of run.
[c] Feed coal ash about 6.0% - used in last part of run.

The petrography information generally places the coal in the high volatile B bituminous group. The reflectance value of 0.59-0.65 is more typical of high volatile B as opposed to an A or C high volatile. Additional data such as BTU value, carbon content, and hydrogen content also classify this coal as the high volatile B. The vitrimiter, 86.2 to 90.1%, were somewhat high for this coal and so were the itiniter (Elimate), which ranged from 6.0 to 8.2%. The inerts were unusually low and ranged from 3.3 to 4.8%. The petrographic analysis indicated this coal should be a good candidate for liquefaction.

7.3 Amocat 1B Ebullation Test Results

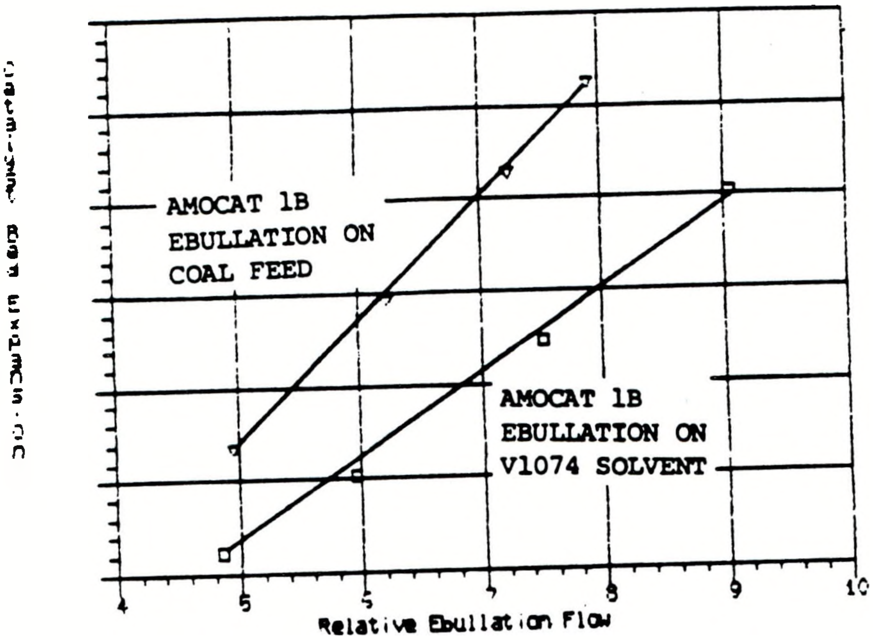
A short test with modified Amocat 1B catalyst was performed at the end of Run 254 to compare ebullation characteristics with the Shell 317 catalyst. The test was outlined in into several parts and test conditions were chosen to be similar to Period 254F (26-29 Dec). Part I of the test was performed on 24 February and established a solvent ebullation curve for the existing Shell 317 catalyst. The Shell 317 catalyst was removed from R1235 and the test catalyst was added. The total charge of 262 pounds was calculated using the relative bulk density of Shell 317 (37 lb/ft^3) and Amocat 1B (33 lb/ft^3). A quick sulfiding process followed the replacement of the ebullation pump which failed late on 24 February.

Part II of the test was conducted to establish a solvent ebullation curve for the modified Amocat 1B. This test was performed on 26 February and test conditions were the same as Part I. Both ebullation curves are shown below:



This plot illustrates the additional flow needed to ebullate the catalyst bed containing the larger Amocat catalyst. The ebullation curves seemed almost parallel; Shell 317 ebullation slope was 5.2 and Amocat 1B was 4.5. The linear correlation coefficient for both sets of data was excellent ranging between 0.991 and 0.998.

Part III of the ebullation test was performed after three days of operation on coal feed. On 29 February a coal feed ebullation curve was created for Amocat 1B. This plot was compared to the solvent ebullation curve for the same catalyst and is shown below:



This plot clearly illustrates how the density and viscosity of the feed affected the catalyst bed ebullation curve. The viscosity of the solvent was much lower than the viscosity of the coal feed. The heavier feed allowed the same ebullation flow to expand the bed as much as an additional ten percent. The increased ebullation flow even reduced the exotherm across the catalyst bed by as much as 50%.

The Amocat 1B catalyst elemental analysis is shown below and indicates a very quick carbon deposition from 6.62 to 17.19 wt % carbon. It should be noted that since the purpose of this test was to evaluate the ebullation characteristics of the larger catalyst, only a modified sulfiding procedure was completed prior to the introduction of coal feed.

		THF Extracted				
		Oil	C	H	N	S
Feb 26	Fresh Unsulfided	42.9	6.20	0.95	0.58	0.59
Feb 26	Fresh Sulfided	42.9	6.62	0.98	0.49	3.69
Feb 27	Routine Sample	40.4	9.43	1.17	0.52	4.67
Feb 28	Routine Sample	42.9	13.47	1.06	0.47	5.59
Feb 29	Routine Sample	43.5	15.06	1.11	0.45	-
Mar 02	Composite	30.5	17.19	1.29	0.49	5.16

The laboratory results indicate some catalyst damage during the test. The catalyst screen analysis is shown below:

		THF Extracted				
		# 8	# 10	# 12	# 14	Pan
Feb 26	Fresh Unsulfided	98.90	1.1	0	0	0
Feb 26	Fresh Sulfided	91.9	6.0	2.1	0	0
Feb 27	Routine Sample	89.9	7.1	3.0	0	0
Feb 28	Routine Sample	79.8	8.67	4.2	2.4	4.9
Feb 29	Routine Sample	94.2	2.1	0.8	0.9	2.0
Feb 29	Routine Sample Rerun	94.6	2.1	0.7	0.6	2.0
Mar 02	Composite	85.3	2.0	0.9	4.0	7.9
Mar 02	Composite Rerun	71.9	7.8	3.9	8.7	7.6

The most disturbing trend in this analysis was the change in catalyst size over such a short period of time. The breakup of catalyst seemed to follow a trend beginning with a 7% shift from screen size #8 during sulfiding. The routine sample on 27 February showed a 9% shift and on 28 February the routine sample indicated continued damage to 79.8% screen #8 from 98.9%.

The 29 February sample did not follow this trend. The original analysis and the rerun analysis both indicated better than 94% of the sample was larger than screen #8. However, the composite sample which was taken when the catalyst was removed from the reactor also indicated catalyst damage. When this sample was reanalyzed, only 71.9% of the catalyst was larger than screen #8 and the smaller particles which passed the #8 screen were sharp, jagged and split. It should be noted that particles in the screen #14 and smaller were heavily contaminated with Shell 317 trilobe catalyst which was used in the reactor previous to this test. Although the Shell 317 catalyst made it difficult to make a definitive screen analysis, the magnitude of breakage is too large for only three days of operation on coal feed.

8.0 ENVIRONMENTAL REGULATORY COMPLIANCE

The purpose of the wastewater treatment facilities is to maintain compliance with permit limitations established by the Alabama Department of Environmental Management (ADEM). Both the sanitary and process wastewater treatment facilities are operated under a National Pollutant Discharge Elimination System (NPDES) permit. This permit was issued by ADEM, which was authorized by the United States Environmental Protection Agency (EPA) to administer the NPDES program in Alabama. The permit was effective on 1 July 1983 and will expire on 30 June 1988. An application for renewal of the permit was filed with ADEM/USEPA on 22 December, 1987. The existing permit established average and maximum limits for specific parameters. The performance of the treatment facilities and the applicable permit limitations are shown in the following tables:

Process Wastewater Treatment Facility

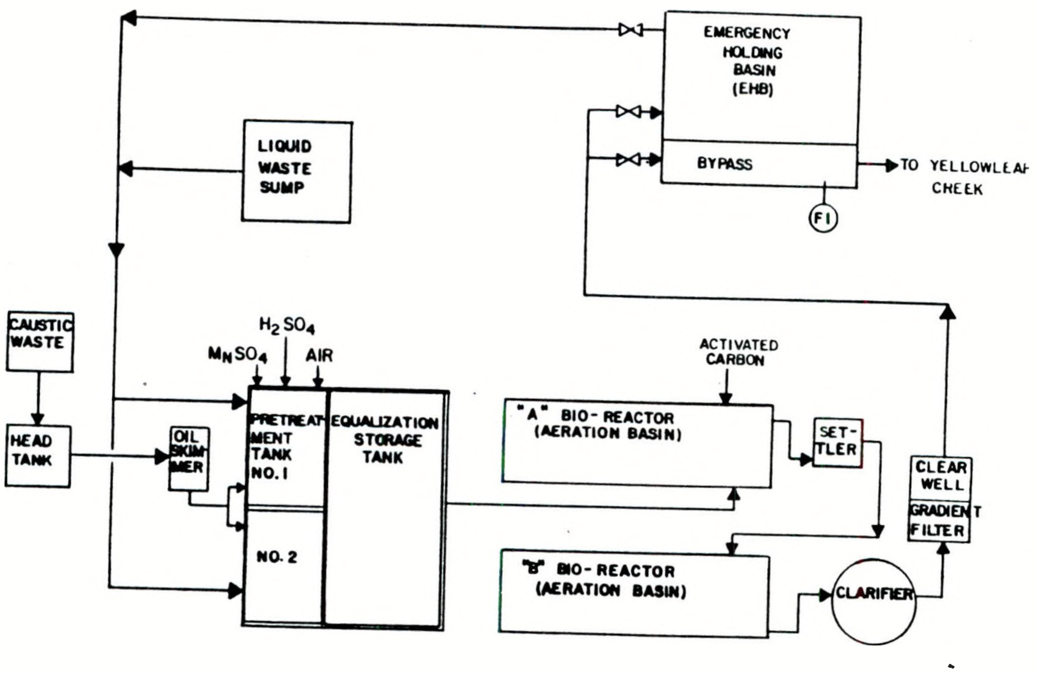
	Allowable Daily Average	Actual Daily Average 26 Aug 87 to 1 Mar 88	Allowable Daily Maximum	Actual Daily Maximum 26 Aug 87 to 1 Mar 88
Flow, gpd	-	17,344	-	34,240
BOD_5, mg/l	30	<1.5	45	8.3
Suspended solids, mg/l	30	<1.9	45	6.0
Phenolics, mg/l	0.25	<0.05	0.50	<0.05
Sulfides, mg/l	0.10	<0.05	0.20	<0.08
pH (range)	N/A	N/A	6-9	6.8-8.3

Sanitary Wastewater Treatment Facility

	Allowable Daily Average and Maximum	Actual Daily Average 26 Aug 87 to 1 Mar 87	Actual Daily Maximum 26 Aug 87 to 1 Mar 87
Flow, gpd	-	816	3701
BOD_5, mg/l	30	1.3	3.0
Suspended Solids, mg/l	30	3.7	9.0
Fecal Coliforms, N/100 ml	200	<0.1	1.0
Chlorine, mg/l	0.2	<0.1	<0.1
pH (range)	6-9	N/A	6.6-7.9

Most of the results shown are based on weekly (or monthly) samples collected by the plant operators and analyzed by an outside laboratory. As readily observed from the tables, all of the regulated parameters were in compliance with the allowable limitations.

The wastewater treatment facilities consists of oil removal, chemical oxidation for the removal of sulfides, equalization, two-stage activated sludge (with the option for addition of powdered activated carbon), and sand filtration. A schematic flow diagram of the wastewater treatment facilities is shown below:



WASTEWATER TREATMENT SYSTEM

ADEM was also authorized by EPA to administer a hazardous waste management program, as required by the Resource Conservation & Recovery Act (RCRA). Pursuant to this program, ADEM has issued a Part A Interim Status authorization for the hazardous waste management facilities at the Wilsonville plant.

9.0 REFERENCES

1. Catalytic, Inc., Technical Progress Report, "Run 250 with Illinois No. 6 Coal," DOE Contract No. DE-AC22-82PC50041, EPRI Contract No. RP1234-1-2, Document No. DOE/PC-50041-81, November 1987.

2. Catalytic, Inc., Technical Progress Report, "Run 251 with Illinois No. 6 and Wyodak Coal," DOE Contract No. DE-AC22-82PC50041, EPRI Contract No. RP1234-1-2, Document No. DOE/PC-50041-88, April 1988.

3. Catalytic, Inc., Technical Progress Report, "Run 252 with Illinois No. 6 Coal," DOE Contract No. DE-AC22-82PC50041, EPRI Contract No. RP1234-1-2, Document No. DOE/PC-50041-98, report not yet published.

4. Catalytic, Inc., Technical Progress Report, "Run 253 with Illinois No. 6 Coal," DOE Contract No. DE-AC22-82PC50041, EPRI Contract No. RP1234-1-2, Document No. DOE/PC/50041-99, report not yet published.

5. Winschel, R. A., and Robbins, G. A., and Burke, F. P., Fuel, 1986, Vol. 65, April, pp. 526-532.

APPENDIX A

Nomenclature and Definitions

Ash	Non-organic material obtained by muffle furnace burning at 800°C for 4 hours (adapted ASTM D-482).
Ash Concentrate	A product of the Kerr-McGee CSD unit first stage separator that is rich in cresol insolubles (ash and UC) with lesser amounts of resid and solvent.
Asphaltenes	A toluene-soluble and pentane-insoluble material which is non-distillable at 600°F and 0.1 mm Hg in the laboratory.
CC-ITSL	Close Coupled Integrated Two-Stage Liquefaction
CCR	Close Coupled Reactor Unit
CI	Material which is insoluble in hot cresol. This material is both ash and organic material.
CSD	Critical Solvent Deashing Unit.
DAS Deashing solvent	Deashing solvent in the CSD unit. A solvent used to deash the feed material.
Distillate solvent	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.
DITSL	Double Integrated Two-Stage Liquefaction
DMDS	Dimethyl disulfide
Energy rejection	The heating value lost to the ash concentrate as a percent of the feed coal heating value.

APPENDIX A (continued)

Nomenclature and Definitions

Feed solvency index	Laboratory analysis for fraction of CSD feed soluble in actual deashing solvent as compared to the solubility of a solvent standard.
HR	Hydrotreated resid
IBP	Initial Boiling Point
ITSL	Integrated Two-Stage Liquefaction
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).
Oils	A pentane-soluble material which is non-distillable at 600°F and 0.1 mm Hg in the laboratory.
Preasphaltenes	A cresol-soluble and toluene-insoluble product material which is non-distillable at 600°F and 0.1 mm Hg in the laboratory.
Process solvent	Material mixed with coal in slurry preparation which is normally a blend of distillate solvent, resid and CI at specified concentrations.

APPENDIX A (continued)

Nomenclature and Definitions

Resid	A cresol-soluble product of the coal liquefaction process which is non-distillable at 600°F and 0.1 mm Hg in the laboratory.
Resid + UC conversion	The fraction of the feed resid and UC that is converted to gases and liquids. $\text{Percent conversion} = \frac{\text{Resid+UC in} - \text{resid+UC out}}{\text{resid+UC in}} \times 100$
Resid recovery	The percent of CSD feed resid which is recovered in the deashed resid and not lost to the ash concentrate.
RITSL	Reconfigured Integrated Two-Stage Liquefaction
TR	Thermal Resid
Unconverted Coal (UC)	Organic material that is insoluble in hot cresol.
WHSV	Weight Hourly Space Velocity, lb/hr feed per lb catalyst

APPENDIX B

Operations Log

B.1 Run 254 Operations Log

Process Adjustments	Rationale/Explanation
● Sulfided six batches of Shell 317 catalyst between 6 August and 18 August.	● Catalyst for Run 254.
● Attempted startup on 23 August.	● Shutdown to repair leaking flange on R1236 inlet.
● On coal feed on 25 August.	
● On solvent on 31 August.	● Catalyst carryover into P1236 suction, plugged ebullation pump. Shutdown was caused by operator error.
● Repaired unit. Pressure checked, and ready for coal feed 5 September.	
● On coal feed on 16 September. Rate = 200 MF lb/hr.	● Rate was lower due to limited coal supply.
● Adjusted CSD first and second stage operating conditions from 17 September to 20 September.	● To correct CSD ash carryover.
● Increased coal feed rate to 250 lb/hr on 19 September.	● To compensate for drop in reactor temperature after heavy rain.
● Increased coal feed rate to 385 lb/hr on 20 September.	● Per run plan.
● Raised gas flows to be normal with coal feed rate on 23 September.	
● Adjusted gas flows on 26 September.	● To reduce swing caused by coal feed problems.
● Recalibrated CSD first stage level controller on 28 September.	● To improve separation and reduce energy rejection.
● First day of Ergon ground coal 29 September.	

Recalibrated CSD feed calibration pot on 29 September.	
On solvent on 1 October.	Orderly shutdown to repair interstage sampler.
Attempted startup on 3 October. Uneven distribution of catalyst ebullation was noted in R1236.	Shutdown due to restriction in R1235 plenum chamber and in R1236 bed.
Attempted startup on 4 October.	Shutdown due to flow restriction in R1235 (suspected to be above plenum chamber).
On coal feed on 5 October. Added 55 pounds (dry) of fresh catalyst to R1235 on 5 October.	To replace the catalyst which was crushed in an attempt to clear withdrawal tube.
Temporary shutdown on 6 October. Began feed with low ash Ohio No. 6 coal.	Lost pressure on R1235 when PSV lifted on C1206. Catalyst bed dropped and formed plenum chamber restriction.
Observed uneven distribution of catalyst in R1235 on 7 October.	
Began catalyst addition/withdrawal at 3 lb cat/ton feed on 9 October.	Per run plan. Catalyst addition/withdrawal continued until shutdown on 28 October.
Lowered coal feed rate to 373 lb/hr on 9 October. R1235 bed distribution showed gap during addition/withdrawal.	To improve conversion of resid produced by Ohio No. 6 coal.
On solvent on 11 October.	Shutdown to clear restriction in R1235 catalyst bed.
On feed on 14 October using 30% coal slurry.	To reduce slurry viscosity. Back to 33% coal slurry immediately after startup.
Raised R1236 temperature on 15 October. Note: Average temperature climbed to as high as 810°F before interstage cooler was put into service.	To increase conversion.

• Adjusted CSD first and second stage operating conditions from 16 October to 19 October.	• To correct CSD ash carry-over.
• Upset in gas flows on 21 October. Raised coal feed rate to 420 lb/hr.	• Repaired instrumentation problem. Adjusted feed rate to bring R1236 back to target temperature after gas flow upset.
• Adjusted coal feed rate and gas flows several times on 22 October.	• To achieve targeted stable run conditions.
• Smooth operation 23 October. Started product stability tests.	
• Power outage at 18:25 on 26 October. No shutdown, all equipment came back on-line.	
• On solvent on 27 October.	• Scheduled shutdown.
• Repaired unit. Pressure checked, and ready for catalyst presulfiding 25 November.	
• Presulfided Batch #7-#9 of Shell 317 catalyst during 27 November - 1 December.	• See Table 15 for lab analysis.
• Charged 300 pounds of catalyst from presulfided batch #7 to R1236 on 2 December.	
• Attempts at startup on 3, 4, 5, 6 December.	• Excessive catalyst bed height in R1235. Void indicated in R1235 catalyst bed. Lowered feed rate, but catalyst bed did not respond. Small plenum chamber restrictions.
• On solvent on 6 December.	• Catalyst carryover plugged P1222. R1235 had a restriction and catalyst bed indicated voids. Shutdown. Also cleared R1236 catalyst bed.
• Cleared resrictions. Unit was pressure checked and ready for coal feed on 9 December.	

● On coal feed on 10 December. Coal feed changes: 33% coal in slurry Process solvent: 38% Resid 12% CI	● Startup conditions included less catalyst in R1235 to reduce the chance of catalyst carryover.
● Returned 25 lbs of catalyst to R1235 on 11 December.	● To bring the total catalyst weight to 300 in R1235.
● Increased resid in process solvent 12-15 December.	● Per run plan.
● Adjusted CSD first and second stage operating conditions from 12 December to 14 December.	● To correct CSD ash carryover.
● Lowered coal feed rate to 420 MF lb/hr on 14 December.	● Per run plan.
● Recalibrated CSD feed calibration on 14 December.	
● On solvent on 15 December.	● Power failure. Restriction formed in R1235 during outage. Temperature excursion to 930°F.
● Remained down to repipe B1201 to act as interstage heater on 15 December.	
● Withdrew 25 lbs of catalyst from R1235 on 17 December.	● To prepare for startup.
● On coal on 18 December. MF coal feed rate 375 lb/hr. Process solvent 50% resid.	
● Returned 25 lbs of catalyst to R1235 on 19 December.	● To bring the total catalyst weight in R1235 to 300 lbs.
● Began catalyst addition/withdrawal program at 3 lbs/ton on 20 December.	
● Recalibrated CSD first stage level controller on 21 December.	● To improve separation and reduce energy rejection.
● Upset in coal feed on 24 December due to problems with feed pumps.	● Flushed approximately 700 lbs of solvent through feed pump and into process.

Adjusted CSD first and second stage operating conditions from 27 December to 3 January.	To correct CSD ash carry-over.
Recalibrated CSD first stage level controller on 28 December.	To improve separation and reduce energy rejection.
Small upset in recycle gas flows on 29 December.	Outside temperature low, valve froze.
Recalibrated CSD first stage level controller on 29 December.	To improve separation and reduce energy rejection
Increased R1236 temperature to 790°F on 30 December.	To improve conversion of resid produced by Ohio No. 6 coal.
Found a Dowtherm A leak into V1072 solvent tank on 4 January. Repaired.	
On solvent on 7 January.	Coal supplier on hold during winter storm.
B201 explosion on 7 January after shutdown.	No injuries.
Calibrated V1079 on 8 January.	For material balancing.
Calibrated V1078 on 9 January.	For material balancing.
On coal feed on 11 January.	
Resumed catalyst addition/withdrawal on 13 January.	Per run plan.
Reduced feedrate to 300 MF lb/hr on 20 January. Higher coal ash period (prior to 25 January).	Per run plan.
Recalibrated CSD first stage level controller on 29 January.	To improve separation and reduce energy rejection.
Adjusted CSD first and second stage operating conditions from 22 January to 28 January.	To correct CSD ash carry-over.
Problems with catalyst addition on 29 January.	Ammonia salts in recycle gas line.
Adjusted CSD first and second stage operating conditions from 29 January to 1 February.	To correct CSD ash carry-over.

Increased feedrate to 450 MF lb/hr on 31 January.	Per run plan.
Unit on solvent on 1 February to repair addition valves.	Unable to make catalyst addition on 31 January after withdrawal.
Found restriction in the line between R1235 and R1236 on 2 February.	
On coal feed on 3 February.	Restriction cleared.
Recalibrated CSD feed tank, CSD feed calibration pot, and CSD first stage level controller on 4 February.	To improve separation and reduce energy rejection.
Resumed catalyst addition/withdrawal on 5 February.	
Lost Pt #13 (6 ft) and Pt #2 (32 ft) thermocouples in R1236 on 5 February.	R1236 thermocouple corrosion.
Lost Pt #5 (22 ft) thermocouple in R1236 on 9 February.	R1236 thermocouple corrosion.
Increased R1236 average temperature to 810°F on 10 February.	Per run plan.
Adjusted CSD second stage operating conditions on 11 February.	Special request from EPRI.
Vacuum problems with V1082 14 February and 15 February.	Problems with B201 boiler.
Lost Pt #14 (2 ft) thermocouple in R1236 on 16 February. Temperature increase across the bed can no longer be calculated.	R1236 thermocouple corrosion.
Lowered feedrate to 375 MF lb/hr on 17 February.	Per run plan.
On solvent on 24 February. Began Ebullation Test 254X. Part I performed on solvent.	Per run plan.

● Depressured unit and replaced R1235 ebullation pump on 25 February. Also removed discharge valve on pump.	● P1222 had been running poorly for more than 2 weeks. Total failure on 24 February after Ebullation Test, Part I.
● Continued ebullation tests on 26 February. Part II performed on solvent.	● Per run plan.
● Lost Pt #9 (17 ft) thermocouple in R1236 on 28 February.	● R1236 thermocouple corrosion.
● Continued ebullation tests on 29 February. Part III performed on coal feed.	
● On solvent on 1 March.	● End of Run 254.

B.2 Summary of DAS Changes

The DAS type was changed throughout Run 254 to minimize the energy rejection to the ash concentrate. The DAS type is chosen to allow acceptable first stage deashing while minimizing the energy rejection. A summary of DAS changes for Run 254 is shown below:

Date	DAS Type
1987	
24 Aug	4100
31 Aug	2054
22 Sep	2104
24 Sep	2254
25 Sep	2354
26 Sep	2454
27 Sep	2504
8 Oct	2554
10 Oct	2604
7 Dec	4100
11 Dec	2204
12 Dec	2254
13 Dec	2304
1988	
6 Jan	2354
12 Jan	2404
15 Jan	2454
17 Jan	2504
18 Jan	2554
23 Jan	2604
4 Feb	2654
7 Feb	2704
11 Feb	2654
16 Feb	2704

APPENDIX C
MATERIAL BALANCE METHODOLOGY

C.1 Elemental Balancing of Yields

The mass and elemental balance 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. Weighting 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 CSD 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 rates.

The CCR unit balance is developed with the above procedure. Since the CSD unit 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 developed by combining the balances of the CCR and the CSD units.

C.2 Material Balance Methodology

In the past elementally balanced yields were calculated for several days each week and were reported within 2 to 5 days. Before this method was developed, elementally balanced yields were calculated only for 8 to 10 selected days each run. These calculations were done after run completion.

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. When elemental analyses are completed on all input and output streams for a given day, elementally balanced yields are calculated for that day (Phase 3). 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, averages of daily Phase 3 data (Phase 4) are calculated for stable operating periods. These are the yields that have been used throughout this report.

C.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. The averages and standard deviations of the Phase 2-3 Selection Criteria are reported for each Operating Period (Phase 4 Period).

Phase 3-4 Selection Criteria are related to element 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 element closure errors are then compared with yields from the other element 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 for each 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 has defined quantitative guidelines as it flows through the data phase system from Phase 2 to Phase 3 to Phase 4.

Table 1

SELECTED FEED COAL ANALYSIS

Coal	Ohio No. 6				
Mine	Crooksville Mine				
Run	254				
Date	8/23-9/17	9/24-28	10/15-18	12/12-1/4/88	1/1-6
Proximate analysis, wt % MF					
Volatile matter	37.40	39.21	39.84	40.14	40.25
Fixed carbon	50.35	51.47	53.77	53.27	53.86
Ash	12.26	9.31	6.40	6.59	5.89
Moisture	9.19	3.81	3.56	3.42	2.55
Ultimate analysis, wt % MF					
Carbon	70.75	71.88	74.77	75.34	75.68
Hydrogen	4.15	4.83	4.90	5.32	5.35
Nitrogen	0.53	1.35	1.43	1.40	1.45
Sulfur	4.61	2.89	2.78	2.63	2.48
Chlorine	0.02	0.02	0.02	0.02	0.03
Ash	12.26	9.31	6.40	6.59	5.89
Oxygen (by difference)	7.68	9.72	9.7	8.70	9.12
H/C atomic ratio	0.70	0.81	0.79	0.85	0.85
Dry heating value	-	-	-	13,620	13,543
Sulfur forms, wt % MF					
Pyrite	2.64	1.23	1.44	1.70	1.48
Sulfate	0.06	0.32	0.05	0.01	0.00
Organic	1.91	1.34	1.29	0.92	1.00
Mineral analysis, wt % (ignited basis)					
Phos. pentoxide, P_2O_3	0.036	0.0016	0.0046	0.053	0.35
Silica, SiO_2	40.78	42.17	41.92	36.94	36.59
Ferric oxide, Fe_2O_3	30.48	25.55	25.92	31.59	31.71
Alumina, Al_2O_3	24.20	27.39	27.10	25.61	27.36
Titania, TiO_2	1.00	0.90	0.89	0.75	0.68
Lime, CaO	1.22	1.42	1.29	1.72	1.55
Magnesia, MgO	0.64	0.62	0.61	0.46	0.48
Sulfur trioxide, SO_3	1.34	1.48	1.27	0.77	1.05
Potassium oxide, K_2O	1.51	1.49	1.26	1.51	1.13
Sodium oxide Na_2O	0.31	0.48	0.43	0.93	0.37
Undetermined	-1.52	-1.50	-0.69	-0.333	-1.27

Table 2

PROCESS SOLVENT ANALYTICAL DATA

	V131B					
Operating Period	CI Free-Residue, wt %			Distillate, wt %		
	C	H	N	C	H	N
254A	-	-	-	-	-	-
254B	89.77	7.45	0.94	89.72	9.94	0.23
254C	91.89	-	-	89.79	9.86	0.35
254D	90.36	7.15	1.05	90.03	9.43	0.34
254E	91.41	7.54	1.06	89.71	9.31	0.44
254F	91.56	7.50	0.93	89.88	9.52	0.53
254G	91.60	7.20	0.96	90.12	9.38	0.51
254H	89.97	6.68	0.96	90.15	9.15	0.68
254I	90.51	6.60	1.04	90.27	9.24	0.48
254J	90.75	6.91	1.23	89.98	8.99	0.61
254K	90.27	6.58	1.32	90.44	8.88	0.67
254L	91.56	6.51	1.31	90.33	8.75	0.63

Table 3

CLOSED-COUPLED REACTOR OPERATING DATA

Period	First Stage Reactor Data				Second Stage Reactor Data			
	Average Temperature °F	Relative Exotherm	Catalyst Age (Res+CI)	WHSV hr^{-1}	Average Temperature °F	Relative Exotherm	Catalyst Age (Res+CI)	WHSV hr^{-1}
254A	810	1.57	878	5.06	761	0.57	327	5.16
254B	811	1.24	1084	3.96	761	0.45	746	3.98
254C	812	1.00	1200	3.76	761	0.37	1136	3.83
254D	811	1.15	1167	3.86	790	0.35	1353	3.88
254E	810	1.62	429	5.13	761	0.46	343	5.13
254F	811	1.43	898	4.27	760	0.29	915	4.27
254G	811	1.43	1064	4.33	790	0.29	1250	4.34
254H	811	1.42	1294	4.26	790	0.26	1760	4.30
254I	811	1.41	1423	3.50	791	0.28	2415	3.51
254J	810	1.61	1508	5.14	790	0.39	2843	5.15
254K	810	1.82	1505	5.13	810	0.51	3316	5.14
254L	810	1.83	1508	4.29	810	Note(4)	3766	4.30

Notes:

1. Relative exotherm is the term for the exotherm across the catalyst bed compared to the lowest exotherm across the R1235 catalyst bed (254C). Reactor exotherm is proprietary.
2. WHSV (Weight Hourly Space Velocity) units are lb feed/hr/lb catalyst.
3. Catalyst age is measured two ways
   (Res+CI) age = lb (coal + resid + UC + ash)/lb catalyst
   MF coal age = lb MF coal/lb catalyst.
4. Exotherm could not be determined in R1236 after thermocouple failure.

Table 4

CCR CONVERSION DATA

Period	First Stage Coal Conversion*	Overall TSL Coal Conversion*	Overall TSL Resid+UC Conversion
254A	91.2	92.9	76.23
254B	93.3	94.0	73.50
254C	93.2	96.0	73.96
254D	95.8	97.4	84.27
254E	93.1	96.8	79.58
254F	94.5	95.9	81.78
254G	94.6	97.1	90.84
254H	92.7	96.4	86.42
254I	93.9	96.8	91.38
254J	92.8	96.3	77.81
254K	93.8	96.7	81.64
254L	95.2	97.1	83.95

Note: Coal conversions were calculated using a forced ash balance method.

Table 5

CCR UNIT ANALYTICAL DATA

	Interstage Sample wt %									
					CI Free Residue			Distillate		
Operating Period	Solv %	Resid %	UC %	Ash %	C %	H %	N %	C %	H %	N %
254A	44.3	39.8	6.2	9.9	-	-	-	-	-	-
254B	43.3	42.1	5.5	9.2	89.22	7.21	1.08	89.03	9.05	0.60
254C	45.6	42.0	4.7	7.7	89.21	6.80	1.36	89.43	9.92	0.46
254D	45.7	41.1	4.7	8.6	89.24	6.78	1.23	89.64	9.58	0.56
254E	39.6	47.5	4.8	8.2	90.94	6.97	1.24	88.41	9.47	0.58
254F	38.4	48.0	4.8	8.8	90.82	6.88	1.14	89.97	9.55	0.48
254G	42.5	45.3	4.5	7.8	90.73	7.08	1.17	89.78	9.58	0.50
254H	39.8	47.3	4.9	8.0	89.20	6.74	1.25	90.30	9.29	0.41
254I	41.5	45.9	4.8	8.0	90.25	6.76	1.14	89.60	9.43	0.47
254J	37.6	49.4	5.5	7.4	90.38	6.61	1.29	89.81	9.05	0.62
254K	38.2	49.2	5.1	7.4	89.61	6.60	1.37	89.74	9.03	0.64
254L	40.1	47.7	4.8	7.5	90.60	6.63	1.33	90.01	9.02	0.69

Table 6

CCR UNIT YIELDS BEFORE ELEMENTAL BALANCING
(WT % MAF COAL)

Operating Period	254A	254B	254C	254D	254E	254F	254G	254H	254I	254J	254K	254L
H_2 consumed	-6.4	-6.8	-6.7	-6.4	-6.1	-6.9	-7.4	-6.8	-7.7	-5.9	-6.0	-6.2
Sour water	12.0	12.0	12.0	13.3	12.0	12.2	12.0	12.0	12.0	12.0	12.6	12.7
Acid gases	0.1	0.3	0.4	0.2	0.3	0.3	0.3	0.3	0.1	0.5	0.2	0.3
C_1-C_3 gases	5.6	6.0	7.5	8.4	6.5	7.6	8.4	8.1	8.3	8.1	9.7	9.7
C_4+ distillate	65.6	56.9	54.7	68.0	66.9	69.5	74.6	71.5	74.0	61.6	61.7	64.4
C_4-C_6	1.9	1.9	1.4	2.0	1.2	1.8	1.7	2.0	1.7	1.6	2.6	1.8
IBP-350	16.4	17.7	15.3	19.3	14.7	18.8	19.1	17.4	18.9	16.3	17.4	18.4
350-450	7.6	7.2	7.7	9.0	7.3	8.0	8.9	8.1	9.1	7.9	7.6	8.5
450-650	39.7*	30.1*	18.4	23.9	18.6	21.0	22.3	20.3	21.4	18.9	19.7	20.0
650-EP	-	-	12.0	13.8	25.1	19.8	22.7	23.7	22.9	16.7	14.4	15.6
Resid	16.1	20.7	24.1	14.0	17.0	13.2	7.2	11.1	5.5	19.0	15.8	13.6
UC	7.1	6.0	4.0	2.6	3.2	4.1	3.1	3.5	3.2	3.7	3.6	3.3
Ash	0.8	0.8	0.0	-0.9	-0.2	0.8	-0.2	0.5	0.6	0.2	0.4	-0.2
Closure	-0.8	4.1	4.0	0.9	0.2	-0.6	2.0	-0.2	4.0	0.8	2.0	2.2
Second stage resid + UC conversion	12.1	13.9	16.1	13.2	14.4	11.5	15.7	17.0	14.9	14.3	15.4	14.0

*Material Balance (2nd Phase) done on a 650+ basis after 6 October 1987.

Table 7

SUMMARY OF CSD PERFORMANCE PARAMETERS

Operating period	DAS type	Resid recovery	Energy rejection	KMAC toluene solubles	FSI(a)	Ash consistency
254A	4100	76.9	16.9	25.5	0.74	Powder
254B	2104-2504	74.9	15.9	20.8	0.78	Grainy
254C	2554-2604	83.0	9.8	15.3	0.76	Grainy
254D	2604-2554	85.3	8.9	13.3	0.76	Powder
254E	2304	87.0	9.8	25.9	0.87	Grainy
254F	2304	84.9	12.5	21.6	0.83	Grainy
254G	2304	86.7	9.9	21.3	0.81	Powder
254H	2454-2504	84.6	10.5	16.9	0.77	Extruded
254I	2604	88.8	7.8	14.3	0.82	Powder
254J	2654-2704	90.0	8.2	9.0	0.79	Extruded
254K	2654	87.5	8.6	11.6	0.76	Extruded
254L	2704	89.4	10.3	10.2	0.78	Extruded

(a) Feed Solvency Index: Kerr McGee ratio of feed solubles in deashing solvent to feed solubles in cresol

Table 8

CSD UNIT ANALYTICAL DATA
RUN - 254

	254A	254B	254C	254D	254E	254F	254G	254H	254I	254J	254K	254L
CSD Feed Composition												
Wt % Ash	20.1	18.4	15.1	16.1	13.7	15.4	14.4	14.5	14.4	12.5	13.2	13.8
Wt % UC	11.5	10.2	8.0	8.1	6.3	8.1	7.0	7.1	7.2	7.3	7.9	8.4
Wt % Solvent	2.4	1.7	2.5	4.3	9.1	4.0	8.0	3.4	4.7	7.5	3.9	3.5
Wt % Carbon	71.11	71.93	75.17	74.71	77.60	75.56	76.98	77.01	77.52	78.26	77.20	76.21
Wt % Hydrogen	5.71	5.65	5.81	5.67	6.26	5.97	6.06	5.76	5.76	5.76	5.49	5.29
Wt % Nitrogen	0.67	0.85	1.02	0.92	0.84	0.90	0.79	0.94	0.91	1.11	1.15	1.12
Wt % Sulfur	-	2.13	-	2.23	1.65	2.47	2.25	2.10	-	1.80	1.73	1.85
Wt % Oxygen (a)	-	1.04	-	0.37	-0.05	-0.30	-0.48	-0.31	-	0.57	1.23	1.73
Soft. Pt. (°F)	159	166	168	146	170	168	160	133	159	169	159	200
Fusion pt. (°F)	176	188	192	168	181	203	198	182	204	186	195	241
Ash Concentrate Composition												
Wt % Ash (b)	42.5	39.4	42.8	45.6	45.0	44.0	44.2	43.0	47.4	44.9	42.7	45.4
Wt % UC (b)	25.0	23.6	24.3	26.9	22.2	25.2	24.6	23.7	26.6	30.3	30.0	30.5
Wt % Resid (b)	29.9	35.4	33.4	26.7	31.4	29.1	30.8	31.3	25.3	24.5	26.4	23.5
Wt % Solvent (b)	2.9	1.9	0.7	0.9	1.5	1.7	0.6	2.0	0.8	0.6	1.0	0.6
Wt % Ash	0.3	1.5	1.2	1.1	0.5	1.2	0.6	0.4	1.0	1.5	1.1	1.1
Wt % Carbon	47.69	51.37	47.99	45.08	46.75	49.16	47.23	47.86	43.75	46.32	48.18	45.63
Wt % Hydrogen	3.05	3.45	3.12	2.84	3.10	3.17	3.03	2.96	2.70	2.83	2.89	2.70
Wt % Nitrogen	0.61	0.85	0.86	0.75	0.65	0.69	0.65	0.80	0.66	0.83	0.88	0.80
Wt % Sulfur	4.54	4.20	5.13	5.95	5.33	6.39	6.55	6.34	5.45	5.23	5.33	5.73
Wt % Oxygen (a)	1.61	0.73	0.90	-0.22	-0.83	-3.41	-1.66	-0.96	0.04	-0.11	0.02	-0.26
Deashed Resid Composition												
Wt % Ash (b)	0.03	0.09	-	0.09	0.76	0.22	0.05	0.02	0.22	0.06	0.05	-
Wt % Solvent (b)	4.9	5.8	14.5	6.3	12.0	5.7	14.7	11.0	8.9	12.6	8.6	6.6
Wt % DAS	0.4	0.4	0.2	0.5	0.7	0.9	0.9	0.5	0.5	0.7	0.3	0.8
Wt % Sulfur	-	0.11	-	0.12	0.04	0.05	0.01	0.10	-	0.08	0.05	0.13
Soft. Pt. (°F)	125	117	146	117	99	144	94	91	142	140	95	171
Fusion Pt. (°F)	131	139	180	136	114	183	125	130	191	157	129	217
Wt % Carbon	91.04	90.48	90.18	91.15	90.95	90.91	91.00	91.76	91.00	91.01	90.85	91.17
Wt % Hydrogen	8.03	7.79	7.60	7.33	7.61	7.64	7.48	7.17	6.99	6.96	6.74	6.58
Wt % Nitrogen	0.73	0.83	0.98	1.00	0.96	0.91	0.87	0.99	0.98	1.18	1.24	1.28

(a) Results calculated by difference.
(b) Results are adjusted to a 'DAS-Free' basis.

Table 9

ANALYTICAL DATA OF STREAMS USED IN ELEMENTAL BALANCE (PART I)

Operating Period	Coal, wt % MF					
	C	H	N	S	O(a)	Ash
254A	73.64	4.76	1.01	3.36	7.01	10.22
254B	72.25	5.12	1.37	2.80	8.99	9.47
254C	75.21	5.31	1.50	2.58	9.41	5.99
254D	75.33	5.25	1.48	2.64	9.03	6.27
254E	72.54	5.19	1.42	2.60	11.31	6.44
254F	73.41	5.28	1.54	3.29	8.78	7.70
254G	76.43	5.38	1.51	2.64	7.71	6.33
254H	76.24	5.32	1.53	2.65	7.76	6.50
254I	76.13	5.41	1.54	2.32	8.66	5.94
254J	76.30	5.37	1.55	2.37	8.81	5.60
254K	75.79	5.38	1.51	2.19	9.73	5.40
254L	76.04	5.31	1.52	2.22	9.37	5.54

Operating Period	T102 Overhead, (V138) wt %				
	C	H	S	N	O(a)
254A	88.66	11.02	0.00	0.26	0.06
254B	88.69	11.08	0.00	0.25	-0.02
254C	89.74	10.98	0.00	0.29	-1.01
254D	88.97	10.43	0.00	0.31	0.29
254E	88.70	10.94	0.00	0.36	0.00
254F	88.53	11.09	0.00	0.38	0.00
254G	88.91	10.69	0.00	0.42	-0.02
254H	88.92	10.68	0.00	0.35	0.05
254I	88.75	10.68	0.00	0.43	0.14
254J	89.12	10.48	0.00	0.41	-0.01
254K	89.03	10.43	0.00	0.60	-0.06
254L	88.85	10.28	0.00	0.47	0.40

(a) Oxygen by difference.

Table 10

ANALYTICAL DATA OF STREAMS USED IN ELEMENTAL BALANCE (PART II)

Operating Period	Recycle Material (V1067)					
	C	H	N	S	O(a)	Ash
254A	79.78	7.40	0.59	1.19	-0.01	11.05
254B	79.56	7.51	0.62	1.10	0.60	10.61
254C	81.04	7.50	0.72	1.01	0.38	9.35
254D	81.22	7.39	0.72	1.24	-0.02	9.45
254E	81.38	7.44	0.75	1.14	-0.01	9.30
254F	80.68	7.45	0.64	1.52	-0.04	9.75
254G	80.78	7.16	0.69	1.51	0.44	9.42
254H	81.00	6.98	0.81	1.36	0.14	9.71
254I	81.23	6.80	0.78	1.13	-0.01	10.07
254J	81.71	6.83	0.95	1.23	0.46	8.82
254K	81.53	6.64	0.99	1.13	0.60	9.11
254L	81.54	6.64	0.96	1.24	0.62	9.00

Operating Period	Flashed Distillate (V1078), wt %				
	C	H	N	S	O(a)
254A	87.54	12.07	0.15	0.02	0.22
254B	87.52	12.32	0.13	0.01	0.02
254C	87.51	12.18	0.20	0.02	0.09
254D	87.87	11.71	0.26	0.04	0.12
254E	87.39	11.87	0.42	0.03	0.29
254F	87.36	12.20	0.28	0.03	0.13
254G	87.59	11.79	0.35	0.03	0.24
254H	87.80	11.73	0.35	0.01	0.11
254I	87.83	11.95	0.18	0.04	0.00
254J	87.51	11.57	0.31	0.01	0.60
254K	87.73	11.50	0.43	0.05	0.29
254L	87.65	11.38	0.35	0.05	0.57

Operating Period	T102 Bottoms, (V1074), wt %				
	C	H	N	S	O(a)
254A	89.89	9.74	0.37	0.00	0.00
254B	89.87	9.81	0.33	0.02	-0.03
254C	89.56	9.91	0.39	0.00	0.14
254D	89.80	9.73	0.44	0.00	0.03
254E	89.68	9.96	0.36	0.00	0.00
254F	89.94	9.69	0.55	0.00	-0.18
254G	90.10	9.41	0.50	0.00	-0.01
254H	89.74	9.62	0.44	0.00	0.20
254I	90.18	9.37	0.46	0.00	-0.01
254J	90.01	9.51	0.49	0.00	-0.01
254K	90.18	8.96	0.60	0.00	0.26
254L	90.57	8.62	0.68	0.00	0.13

(a) Oxygen by difference.

Table 11

ANALYTICAL DATA OF STREAMS USED IN ELEMENTAL BALANCE (PART III)

Operating Period	Vacuum Flash Overhead (V1072), wt %				
	C	H	N	S	O(a)
254A	89.43	9.79	0.36	0.01	0.41
254B	89.80	9.88	0.30	0.02	0.00
254C	89.72	9.89	0.37	0.02	0.00
254D	89.98	9.60	0.38	0.04	0.00
254E	89.77	9.61	0.57	0.06	-0.01
254F	89.70	9.79	0.45	0.06	0.00
254G	90.07	9.43	0.47	0.00	0.03
254H	90.15	9.25	0.43	0.01	0.16
254I	89.75	9.41	0.45	0.01	0.38
254J	89.81	9.20	0.57	0.01	0.41
254K	90.32	8.77	0.67	0.01	0.23
254L	90.27	8.62	0.66	0.01	0.44

Operating Period	CSD Feed, wt %					
	C	H	N	S	O(a)	Ash
254A	71.09	5.71	0.67	2.29	0.14	20.10
254B	71.91	5.65	0.87	2.09	1.14	18.34
254C	75.17	5.81	1.02	1.89	1.04	15.07
254D	74.71	5.67	0.92	2.17	0.42	16.11
254E	77.57	6.26	0.86	1.71	0.04	13.64
254F	75.23	5.97	0.90	2.40	0.10	15.40
254G	76.62	6.06	0.79	2.24	0.09	14.20
254H	76.61	5.76	0.94	2.11	0.11	14.47
254I	77.19	5.76	0.91	1.70	0.06	14.38
254J	78.26	5.76	1.11	1.82	0.60	12.45
254K	77.25	5.49	1.15	1.71	1.23	13.17
254L	77.37	5.38	1.12	1.89	0.46	13.78

Operating Period	CSD Resid, wt %					
	C	H	N	S	O(a)	Ash
254A	91.04	8.03	0.72	0.02	0.17	0.02
254B	90.51	7.79	0.83	0.09	0.71	0.07
254C	90.18	7.60	0.98	0.13	1.11	0.00
254D	91.15	7.33	1.00	0.10	0.42	0.00
254E	90.66	7.61	0.96	0.04	-0.02	0.75
254F	90.91	7.64	0.91	0.04	0.28	0.22
254G	91.00	7.48	0.87	0.01	0.59	0.05
254H	91.73	7.17	0.99	0.05	0.05	0.01
254I	91.00	6.99	0.98	0.04	0.77	0.22
254J	91.01	6.96	1.18	0.08	0.73	0.04
254K	90.90	6.75	1.24	0.06	1.03	0.02
254L	91.17	6.58	1.28	0.12	0.85	0.00

(a) Oxygen by difference.

Table 12

RUN 254 SOUR WATER ANALYSES (a)

Operating Period Date, 1988	2nd Stage V1080 254G 2 Jan
Total Organic Carbon	1,480
Kjeldahl Nitrogen	24,339
Sulfide Sulfur	22,153
Chloride	12
Phenols	18
Inorganic Carbon	608

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

Table 13

YIELD COMPARISONS RUN 253D AND RUN 254B

	253D	254B
Catalyst addition/withdrawal	No	No
Interstage separator	Yes	No
Coal		
MF wt % ash	11.6	9.5
MF coal feed rate, lb/hr	380	385
Coal concentration, wt %	32.9	32.8
Recycle process solvent		
Wt % resid	39	39
Wt % CI	12	12
1st stage reactor		
Average temperature (°F)	811	811
Space velocity, (hr^{-1})	3.9	4.0
Catalyst type	Shell 317	Shell 317
Catalyst age (lb resid + CI)/lb cat	741-927	865-1302
lb MF coal/lb cat	378-469	425-637
2nd stage reactor		
Average temperature (°F)	759	760
Space velocitry, (hr^{-1})	3.6	3.8
Catalyst type	Shell 317	Shell 317
Catalyst age (lb resid + CI)/lb cat	1601-1738	578-914
lb MF coal/lb cat	1151-1240	383-595
Yield		
C_4+ distillate	71.6	60.2
Resid	-3.9	9.8
Ash concentrate (ash-free)	18.4	16.1
Coal conversion	91.9	93.5
H_2 efficiency (lb C_4+ dist/lb H_2 consumed)	10.4	9.9

Table 14

YIELD COMPARISONS RUN 253E AND RUN 254H

	253E	254H
Catalyst addition/withdrawal	Yes	Yes
Interstage separator	No	No
Coal		
MF wt % ash	11.2	6.5
MF coal feed rate, lb/hr	381	364
Coal concentration, wt %	32.9	29.2
Recycle process solvent		
Wt % resid	39	49
Wt % CI	12	12
1st stage reactor		
Average temperature (°F)	809	811
Space velocity, (hr^{-1})	4.0	4.3
Catalyst type	Shell 317	Shell 317
Catalyst age (lb resid + CI)/lb cat	1241-1315	1261-1332
lb MF coal/lb cat	620-655	514-543
2nd stage reactor		
Average temperature (°F)	760	790
Space velocity, (hr^{-1})	3.8	4.1
Catalyst type	Shell 317	Shell 317
Catalyst type (lb resid + CI)/lb cat	2120-2277	1703-1817
lb MF coal/lb cat	1522-1642	918-976
Yield		
C_4+ distillate	67.6	71.7
Resid	1.3	4.5
Ash concentrate	15.3	9.2
Coal conversion	93	96.2
H_2 efficiency (lb C_4+ dist/lb H_2 consumed)	10.4	10.9

Table 15

PRESULFIDED CATALYST DATA

Date, 1987	Description of sample	As-is wt %		Analysis of THF-extracted catalyst wt %						Naphthalene activity
		S	Ash	Oil	C	H	N	S	Ash	
Aug										
10	Unsulfided Catalyst	-	-	-	0.85	0.70	0.12	0.0	93.2	-
9	Composite Batch #1	5.56	57.4	36.2	1.27	0.79	0.25	7.94	87.5	210
10	Composite Batch #2	5.42	56.8	36.1	0.94	0.76	0.16	8.24	88.5	175
12	Composite Batch #3	5.97	58.5	36.2	0.96	0.72	0.07	7.67	87.6	199
14	Composite Batch #4	6.20	57.0	34.8	0.59	0.75	0.07	7.70	85.1	207
16	Composite Batch #5	6.04	59.1	34.7	0.83	0.71	0.06	8.32	87.2	214
17	Batch #6	5.91	59.8	33.3	0.68	0.78	0.09	7.57	85.1	200
Nov										
28	Composite Batch #7	5.61	57.7	36.0	2.16	0.80	0.21	8.13	85.6	185
29	Composite Batch #8	5.25	58.7	33.7	1.71	0.73	0.14	8.15	88.2	203
Dec										
1	Batch #9	6.41	56.9	26.7	2.54	0.80	0.17	8.18	85.6	189

Table 16

FIRST STAGE SHELL 317 CATALYST

Date,	Average catalyst age			As-is wt %		Analysis of THF-extracted catalyst wt %						Naphthalene
1987-88	Res+CI	MF coal	Sample Description	S	Ash	Oil	C	H	N	S	Ash	activity
Aug												
17	0	0	Fresh catalyst	5.91	59.8	33.3	0.68	0.78	0.09	7.57	85.4	200
27	240	118	1st Routine Sample	5.70	54.8	33.4	11.70	1.01	0.32	7.12	79.8	92
29	398	196	2nd Routine Sample	6.67	56.3	30.1	12.39	0.91	0.30	6.95	81.0	118
31	560	275	3rd Routine Sample	6.37	56.3	40.6	13.51	0.92	0.32	6.32	79.7	74
Sep												
18	648	319	4th Routine Sample	5.41	55.6	32.5	12.42	0.93	0.31	6.35	79.7	64
23	864	425	5th Routine Sample	5.47	52.1	29.8	13.30	0.81	0.26	6.03	79.3	46
30	1301	637	6th Routine Sample	6.82	57.3	-	14.24	0.86	0.26	6.36	78.9	-
Oct												
5	1101	539	1st Addition/Withdrawal	5.46	55.7	30.4	-	-	-	-	79.7	-
16	1131	552	2nd Addition/Withdrawal	4.70	56.3	35.4	15.11	1.02	0.32	6.08	77.7	47
18	1137	554	3rd Addition/Withdrawal	5.28	56.6	28.8	14.92	0.98	0.31	6.21	78.3	38
20	1150	560	4th Addition/Withdrawal	4.51	57.0	29.2	13.88	0.92	0.33	6.17	79.4	43
22	1161	565	5th Addition/Withdrawal	4.49	57.0	28.8	14.48	0.96	0.32	6.21	80.9	44
24	1173	570	6th Addition/Withdrawal	4.54	57.4	29.5	13.48	0.91	0.34	6.12	79.4	66
26	1186	575	7th Addition/Withdrawal	4.56	57.7	29.7	13.83	0.91	0.36	6.23	80.3	48
27	1214	587	Final Composite	5.00	56.2	24.1	18.30	1.19	0.32	6.08	75.5	32
30	1214	587	Final Grab Sample	5.48	-	-	16.10	1.08	0.36	6.89	77.8	28

Table 16 (continued)

FIRST STAGE SHELL 317 CATALYST

Date,	Average catalyst age			As-is wt %		Analysis of THF-extracted catalyst wt %						Naphthalene
1987-88	Res+CI	MF coal	Sample Description	S	Ash	Oil	C	H	N	S	Ash	activity
Nov												
29	0	0	Fresh Sulfided Catalyst (used for addition)	5.25	58.7	33.7	1.71	0.73	0.14	8.15	88.2	203
Dec												
1	0	0	Fresh Sulfided	6.41	56.9	26.7	2.54	0.80	0.17	8.18	85.6	189
4	36.5	15.1	1st Routine Sample	5.32	54.0	34.7	7.30	0.87	0.42	7.67	81.1	142
6	81.5	33.0	Shutdown Composite	7.29	-	-	13.58	1.06	0.37	7.30	79.0	59
15	485.5	215.1	2nd Routine Sample	6.45	58.2	26.7	15.12	0.93	0.33	6.50	77.4	45
20	598.3	260.2	1st Addition/Withdrawal	5.07	55.5	29.6	13.96	0.92	0.30	6.43	79.4	44
22	686.2	292.3	2nd Addition/Withdrawal	5.13	55.4	32.2	13.51	0.81	0.33	6.39	80.1	40
24	761.8	320.8	3rd Addition/Withdrawal	4.97	55.9	31.7	13.24	0.81	0.37	6.58	80.2	55
26	829.6	347.0	4th Addition/Withdrawal	5.99	57.2	31.4	12.82	0.75	0.19	6.82	82.4	46
28	893.4	371.2	5th Addition/Withdrawal	5.06	57.0	30.7	13.33	0.80	0.24	6.85	77.5	52
30	951.7	393.6	6th Addition/Withdrawal	4.88	57.2	31.4	13.48	0.80	0.25	6.74	79.5	50
Jan												
1	1003.6	414.5	7th Addition/Withdrawal	5.54	56.7	29.9	12.95	0.89	0.25	6.85	78.4	46
3	1051.4	433.2	8th Addition/Withdrawal	5.46	53.6	33.0	13.33	0.82	0.29	6.76	78.4	42
5	1096.6	450.5	9th Addition/Withdrawal	5.01	55.4	30.7	14.48	0.91	0.33	6.74	79.4	47
13	1232.2	504.5	10th Addition/Withdrawal	5.17	55.6	31.0	13.05	0.78	0.27	6.17	81.0	38
15	1261.1	514.4	11th Addition/Withdrawal	5.66	54.4	33.5	13.64	0.76	0.27	6.22	79.9	26
17	1288.2	526.0	12th Addition/Withdrawal	4.77	55.5	31.1	13.72	0.80	0.29	6.30	79.1	36
19	1316.7	536.4	13th Addition/Withdrawal	4.64	56.6	32.2	13.28	0.76	0.29	6.17	79.5	50
21	1328.8	546.0	14th Addition/Withdrawal	4.66	57.3	28.4	13.14	0.80	0.29	6.15	79.5	50
23	1346.9	533.4	15th Addition/Withdrawal	4.74	56.9	29.1	13.10	0.82	0.28	6.14	81.4	48
25	1363.1	560.3	16th Addition/Withdrawal	4.72	57.7	28.5	13.21	0.80	0.27	6.17	82.5	49
27	1377.8	566.5	17th Addition/Withdrawal	5.02	58.0	27.6	13.90	0.85	0.28	5.86	80.4	61
29	1392.7	571.0	18th Addition/Withdrawal	4.80	59.9	27.4	13.51	0.82	0.28	6.46	80.9	50
31	1548.4	633.7	19th Addition/Withdrawal	4.92	60.2	25.6	13.98	0.87	0.30	6.02	80.5	53
Feb												
5	1446.3	590.4	20th Addition/Withdrawal	5.13	58.6	27.9	13.66	0.88	0.31	6.04	82.5	53
7	1451.2	591.3	21st Addition/Withdrawal	5.04	59.6	29.3	13.97	0.92	0.31	6.20	79.5	52
9	1457.7	593.4	22nd Addition/Withdrawal	4.57	55.1	30.6	13.37	0.85	0.33	6.12	80.8	43
11	1463.9	595.1	23rd Addition/Withdrawal	4.55	58.0	27.4	13.27	0.84	0.27	6.18	79.9	54
13	1471.8	597.8	24th Addition/Withdrawal	5.41	57.6	29.5	13.16	0.87	0.29	5.89	81.6	37
15	1471.2	599.7	25th Addition/Withdrawal	6.22	61.1	26.7	12.78	0.87	0.30	6.53	81.1	44
17	1468.4	597.7	26th Addition/Withdrawal	4.46	60.0	28.3	13.58	0.84	0.29	5.95	81.4	50
19	1475.9	601.3	27th Addition/Withdrawal	4.28	57.3	29.2	13.89	0.89	0.30	5.91	79.0	43
21	1482.1	604.1	28th Addition/Withdrawal	5.00	58.9	27.5	13.62	0.85	0.30	5.84	79.6	48
23	1491.9	607.2	29th Addition/Withdrawal	5.06	57.9	28.0	12.49	0.82	0.29	5.78	81.7	53
24	1492.7	607.5	R1235 Composite	4.36	58.7	27.6	12.45	0.92	0.31	6.18	81.9	45

Table 17

SECOND STAGE CATALYST

Date, 1987-88	Average Catalyst age Res+CI	Average Catalyst age MF coal	Description of sample	As-is wt % S	As-is wt % Ash	Analysis of THF-extracted catalyst wt % Oil	C	H	N	S	Ash	Naphthalene activity
Aug												
19	0	0	Fresh Catalyst	5.56	57.4	36.2	1.27	0.79	0.25	7.94	87.5	210
Sep												
2	426	279	Shutdown Composite	5.18	54.0	33.5	9.65	1.02	0.32	7.03	82.4	124
Oct												
1	915	595	Shutdown Grab Sample	5.20	54.8	-	10.10	0.88	0.30	6.88	81.4	-
12	1161	753	Shutdown Grab Sample	4.35	49.2	41.2	11.33	0.96	0.30	0.76	81.8	56
28	1727	1124	R1236 Composite (Catalytic Lab)	5.36	57.3	29.0	13.76	0.96	0.30	6.27	80.3	37
28	1727	1124	R1236 Composite (HRI Lab)	-	-	-	14.31	0.82	0.28	6.64	78.4	-
Nov												
30	0	0	Fresh, sulfided	5.61	57.7	36.0	2.16	0.80	0.21	8.13	85.6	185
Dec												
8	77.3	42.0	Shutdown Grab Sample	4.68	53.5	34.5	10.42	1.16	0.36	7.43	80.4	81
Feb												
2	2538.3	1351.4	Shutdown Grab Sample	4.54	59.3	29.8	12.93	0.88	0.26	-	81.7	53
Mar												
2	4034.9	2118.4	R1236 Composite	4.64	57.1	27.7	16.61	0.98	0.33	6.2	78.4	63

Table 18

PRODUCT QUALITY COMPARISON(e)

	253E	253F(a)	253G	254G
Coal type	Illinois No. 6	Illinois No. 6	Illinois No. 6	Ohio No. 6
Coal feed MF	385	385	385	370
Slurry	33	33	33	30
Process solvent				
Resid	38	38	38	50
CI	12	12	12	12
1st stage				
Temperature, °F	809	810	810	811
Space velocity, hr^{-1}	3.95	3.93	3.97	4.33
Catalyst type	Shell 317	Shell 317	Shell 317	Shell 317
Addition/withdrawal	Yes(b)	Yes(b)	Yes(b)	Yes(c)
Average catalyst age				
(resid+CI)	1265	1369	1527	1064
(MF coal)	631	680	755	438
Interstage separator	No	No	No	No
2nd stage				
Temperature, °F	760	760	770	790
Space velocity, hr^{-1}	3.73	3.76	3.80	4.21
Catalyst type	Shell 317	Shell 317	Shell 317	Shell 317
Average catalyst age				
(resid+CI)	2198	2435	2763	1250
(MF coal)	1582	1761	1997	685
TSL yield, wt % MAF				
H_2 consumption	6.5	6.5	6.5	6.9
C_1-C_3	7.1	7.0	7.4	8.5
Distillate	67.7	65.3	66.5	78.4
Resid	1.5	2.9	1.6	-0.7
Ash concentrate	15.3	14.9	15.9	8.2
Coal Conv.	93-94	93-94	93-94	95-97
Percent of Product				
IBP-350°F	24.9	23.9	24.1	25.4
350-450°F	13.0	11.3	13.5	11.4
450-650°F	35.4	35.4	34.8	36.7
650-EP(d)	26.7	29.4	27.6	26.5

Notes:

(a) Ground catalyst addition to coal slurry at 2 lb/ton of MF coal.

(b) Catalyst addition/withdrawal 2 lb/ton of MF coal.

(c) Catalyst addition/withdrawal 3 lb/ton of MF coal.

(d) In all cases the heavy fraction was found to have an end point below 850°F.

(e) This table reports CALCULATED product quality results based on Phase III material balance results and daily GC analysis to compare 254G to similar periods during 253 for which traditional product quality data was not developed. This table is for comparison purposes only and does not report actual product quality results for these periods.

Table 19

PHASE 2-3 CRITERIA
MASS BALANCE CLOSURES & INVENTORY CHANGES (a)(b)
STATISTICAL SUMMARY
(WT % MAF)

Operating Period	254A	254B	254C	254D	254E	254F	254G	254H	254I	254J	254K	254L
CCR closure	-0.78±2.24	4.11±1.66	3.99±0.39	0.93±1.14	0.21±2.00	0.59±1.61	2.01±1.59	-0.20±2.19	3.97±1.83	0.85±2.23	1.79±2.35	2.74±2.43
CSD closure	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00	0.00±0.00
Total closure	-0.78±2.24	4.11±1.66	3.99±0.39	0.93±1.14	0.21±2.00	-0.59±1.61	2.01±1.59	-0.20±2.19	3.97±1.83	0.85±2.23	1.79±2.35	2.74±2.43
Abs. tot. clo.	1.59±1.10	4.11±1.66	3.99±0.39	1.12±0.87	1.42±0.30	1.46±0.46	2.01±1.59	1.70±0.74	3.97±1.83	1.43±1.73	2.66±0.81	2.77±2.38

(a) Inv. changes include resid, UC and ash comp. only. Increases are positive.

(b) Mass Balance closure sign convention is that losses are negative.

(c) Absolute total is the absolute total of the inventory changes between both process units which gives a measure of overall plant and process stability.

Table 20

PHASE 3-4 CRITERIA
STATISTICAL SUMMARY
UNIT AND TSL ELEMENTAL CLOSURE ERRORS (A)
WT % MAF

OPERATING PERIOD	254A	254B	254C	254D
CCR UNIT DATA				
CARBON	0.29 ± 1.87	-0.97 ± 1.42	-0.92 ± 0.62	1.47 ± 0.98
HYDROGEN	-0.12 ± 0.16	-0.88 ± 0.30	-0.69 ± 0.09	0.29 ± 0.16
NITROGEN	0.11 ± 0.01	0.02 ± 0.06	0.01 ± 0.07	-0.13 ± 0.05
SULFUR	-0.32 ± 0.00	0.40 ± 0.21	0.37 ± 0.14	0.27 ± 0.15
OXYGEN	-0.34 ± 0.40	-0.23 ± 0.36	-0.33 ± 0.18	-0.44 ± 0.83
ASH	1.17 ± 0.58	0.35 ± 0.70	0.52 ± 0.34	-0.95 ± 0.63
CSD UNIT DATA				
CARBON	-0.30 ± 0.03	0.11 ± 0.38	-0.16 ± 0.15	0.00 ± 0.11
HYDROGEN	-0.04 ± 0.01	0.03 ± 0.08	0.07 ± 0.02	0.01 ± 0.01
NITROGEN	0.01 ± 0.01	-0.01 ± 0.02	-0.04 ± 0.04	0.00 ± 0.01
SULFUR	-0.07 ± 0.06	-0.05 ± 0.08	0.03 ± 0.03	0.02 ± 0.06
OXYGEN	0.47 ± 0.29	-0.10 ± 0.27	0.05 ± 0.12	-0.01 ± 0.07
ASH	-0.05 ± 0.32	0.02 ± 0.23	0.05 ± 0.04	0.00 ± 0.13
TOTALED DATA (B)				
CARBON	-0.01 ± 1.90	-0.86 ± 1.45	-1.08 ± 0.76	1.47 ± 1.00
HYDROGEN	-0.16 ± 0.17	-0.85 ± 0.33	-0.63 ± 0.11	0.30 ± 0.17
NITROGEN	0.11 ± 0.01	0.01 ± 0.05	-0.03 ± 0.03	-0.13 ± 0.06
SULFUR	-0.39 ± 0.07	0.35 ± 0.17	0.40 ± 0.12	0.28 ± 0.14
OXYGEN	0.13 ± 0.69	-0.32 ± 0.33	-0.28 ± 0.06	-0.45 ± 0.79
ASH	1.11 ± 0.91	0.37 ± 0.65	0.57 ± 0.37	-0.95 ± 0.62
ABSOLUTE SUM DATA (C)				
CARBON	1.62 ± 0.38	1.72 ± 0.71	1.08 ± 0.76	1.56 ± 0.98
HYDROGEN	0.16 ± 0.17	0.93 ± 0.29	0.76 ± 0.06	0.30 ± 0.17
NITROGEN	0.11 ± 0.01	0.07 ± 0.03	0.09 ± 0.05	0.13 ± 0.06
SULFUR	0.39 ± 0.07	0.47 ± 0.26	0.40 ± 0.12	0.31 ± 0.18
OXYGEN	0.81 ± 0.12	0.54 ± 0.26	0.42 ± 0.25	0.56 ± 0.78
ASH	1.40 ± 0.51	0.91 ± 0.21	0.57 ± 0.37	1.03 ± 0.62

(A) CLOSURE ERROR SIGN CONVENTION IS THAT LOSSES ARE NEGATIVE.
(B) TOTAL IS THE ARITHMETIC SUM OF THE INDIVIDUAL UNIT ERRORS.
(C) ABS. TOTAL IS THE SUM OF THE ABS. VALUES OF THE INDIVIDUAL UNIT ERRORS.

Table 20 (continued)

PHASE 3-4 CRITERIA
STATISTICAL SUMMARY
UNIT AND TSL ELEMENTAL CLOSURE ERRORS (A)
WT % MAF

Operating Period	254E	254F	254G	254H
CCR UNIT DATA				
Carbon	4.85 ± 0.63	3.38 ± 1.18	-0.17 ± 2.02	1.56 ± 1.51
Hydrogen	0.11 ± 0.17	-0.25 ± 0.35	-0.67 ± 0.25	-0.31 ± 0.24
Nitrogen	-0.13 ± 0.07	-0.17 ± 0.09	-0.19 ± 0.10	-0.09 ± 0.04
Sulfur	-0.13 ± 0.14	-0.03 ± 0.14	0.42 ± 0.26	0.48 ± 0.07
Oxygen	-4.86 ± 0.18	-2.49 ± 0.18	-1.35 ± 0.21	-1.82 ± 0.66
Ash	-0.07 ± 0.82	0.17 ± 0.51	-0.04 ± 0.84	0.39 ± 0.09
CSD UNIT DATA				
Carbon	-0.03 ± 0.04	0.16 ± 0.34	0.08 ± 0.38	0.01 ± 0.11
Hydrogen	-0.01 ± 0.01	0.03 ± 0.06	-0.02 ± 0.04	-0.01 ± 0.02
Nitrogen	0.00 ± 0.00	-0.02 ± 0.12	0.01 ± 0.00	0.00 ± 0.01
Sulfur	-0.05 ± 0.03	-0.10 ± 0.05	-0.10 ± 0.05	0.04 ± 0.04
Oxygen	0.03 ± 0.04	-0.01 ± 0.15	0.19 ± 0.15	-0.02 ± 0.06
Ash	0.04 ± 0.07	-0.11 ± 0.14	-0.15 ± 0.26	-0.01 ± 0.04
TOTALED DATA (B)				
Carbon	4.83 ± 0.67	3.53 ± 1.39	-0.09 ± 2.34	1.57 ± 1.43
Hydrogen	0.10 ± 0.17	-0.22 ± 0.32	-0.69 ± 0.25	-0.32 ± 0.25
Nitrogen	-0.12 ± 0.07	-0.20 ± 0.04	-0.18 ± 0.10	-0.10 ± 0.05
Sulfur	-0.18 ± 0.12	-0.13 ± 0.10	0.32 ± 0.27	0.52 ± 0.07
Oxygen	-4.83 ± 0.22	-2.50 ± 0.29	-1.16 ± 0.34	-1.84 ± 0.61
Ash	-0.03 ± 0.75	0.06 ± 0.51	-0.18 ± 1.09	0.38 ± 0.09
ABSOLUTE SUM DATA (C)				
Carbon	4.88 ± 0.59	3.63 ± 1.39	1.98 ± 0.54	1.65 ± 1.54
Hydrogen	0.13 ± 0.15	0.40 ± 0.22	0.71 ± 0.24	0.33 ± 0.25
Nitrogen	0.13 ± 0.07	0.27 ± 0.12	0.20 ± 0.10	0.10 ± 0.05
Sulfur	0.18 ± 0.12	0.21 ± 0.03	0.52 ± 0.27	0.53 ± 0.07
Oxygen	4.89 ± 0.14	2.59 ± 0.20	1.54 ± 0.12	1.85 ± 0.65
Ash	0.63 ± 0.17	0.55 ± 0.26	0.84 ± 0.53	0.42 ± 0.10

(A) Closure Error sign convention is that losses are negative.
(B) Total is the arithmetic sum of the individual unit errors.
(C) Abs. Total is the sum of the abs. values of the individual unit errors.

Table 20 (continued)

PHASE 3-4 CRITERIA
STATISTICAL SUMMARY
UNIT AND TSL ELEMENTAL CLOSURE ERRORS (A)
WT % MAF

Operating Period	254I	254J	254K	254L
CCR Unit Data				
Carbon	0.00 ± 0.73	0.16 ± 1.66	-0.58 ± 2.29	-1.35 ± 2.78
Hydrogen	-1.06 ± 0.38	-0.09 ± 0.16	0.20 ± 0.19	-0.10 ± 0.48
Nitrogen	-0.02 ± 0.21	-0.09 ± 0.04	-0.05 ± 0.08	0.05 ± 0.21
Sulfur	0.91 ± 0.34	0.59 ± 0.26	0.38 ± 0.14	0.89 ± 0.48
Oxygen	-3.62 ± 0.66	-1.67 ± 0.27	-2.11 ± 0.43	-2.48 ± 0.72
Ash	-0.22 ± 0.81	0.25 ± 0.50	0.41 ± 0.33	0.25 ± 0.92
CSD Unit Data				
Carbon	-0.13 ± 0.05	0.15 ± 0.07	0.00 ± 0.19	-0.23 ± 0.17
Hydrogen	-0.04 ± 0.06	0.01 ± 0.02	0.00 ± 0.02	-0.03 ± 0.00
Nitrogen	-0.01 ± 0.01	-0.01 ± 0.01	-0.01 ± 0.02	0.01 ± 0.01
Sulfur	-0.03 ± 0.00	-0.06 ± 0.01	0.01 ± 0.01	0.00 ± 0.06
Oxygen	0.30 ± 0.09	-0.04 ± 0.07	-0.14 ± 0.13	0.09 ± 0.22
Ash	-0.09 ± 0.03	-0.05 ± 0.03	0.09 ± 0.07	0.15 ± 0.10
Totaled Data (B)				
Carbon	-0.13 ± 0.68	0.31 ± 1.72	-0.58 ± 2.10	-1.58 ± 2.64
Hydrogen	-1.10 ± 0.32	-0.08 ± 0.17	0.20 ± 0.18	-0.13 ± 0.48
Nitrogen	-0.03 ± 0.19	-0.10 ± 0.02	-0.05 ± 0.06	0.06 ± 0.21
Sulfur	0.88 ± 0.34	0.53 ± 0.25	0.38 ± 0.14	0.89 ± 0.49
Oxygen	-3.32 ± 0.57	-1.71 ± 0.33	-2.24 ± 0.45	-2.39 ± 0.92
Ash	-0.31 ± 0.85	0.20 ± 0.51	0.51 ± 0.39	0.40 ± 1.00
Absolute Sum Data (C)				
Carbon	0.65 ± 0.06	1.25 ± 1.03	1.97 ± 1.31	2.81 ± 0.52
Hydrogen	1.10 ± 0.32	0.13 ± 0.13	0.25 ± 0.14	0.42 ± 0.09
Nitrogen	0.16 ± 0.01	0.10 ± 0.03	0.08 ± 0.06	0.19 ± 0.04
Sulfur	0.94 ± 0.34	0.65 + 0.27	0.39 ± 0.14	0.94 ± 0.50
Oxygen	3.93 ± 0.75	1.71 ± 0.33	2.28 ± 0.44	2.63 ± 0.56
Ash	0.67 ± 0.34	0.51 ± 0.14	0.57 ± 0.25	0.90 ± 0.40

(A) Closure Error sign convention is that losses are negative.
(B) Total is the arithmetic sum of the individual unit errors.
(C) Abs. Total is the sum of the abs. values of the individual unit errors.

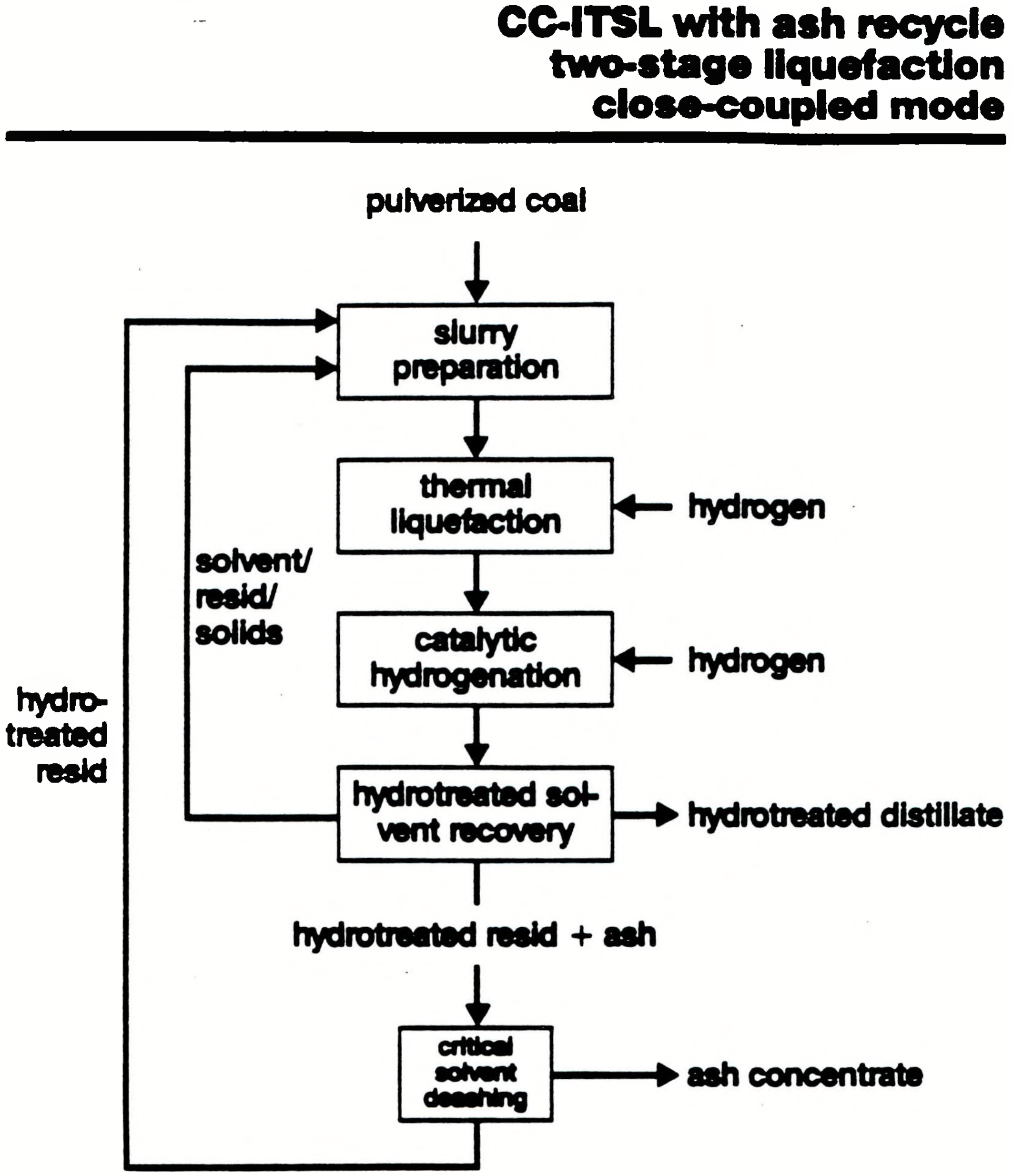
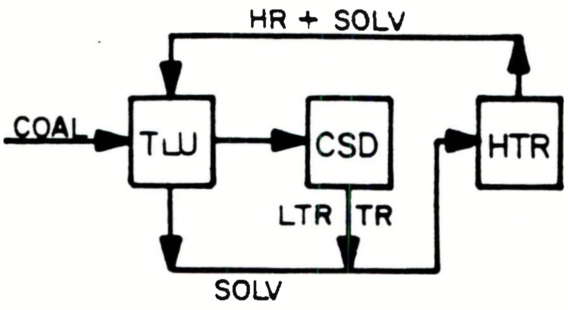
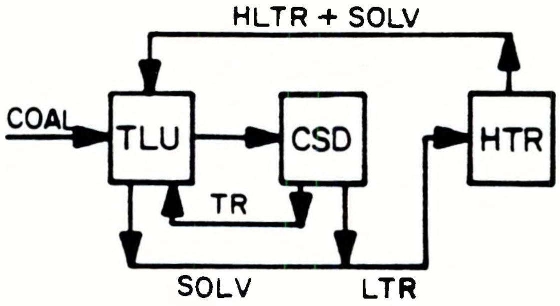


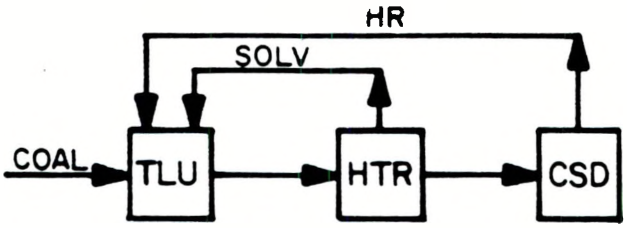
FIGURE 1. BLOCK FLOW DIAGRAM OF CC-ITSL OPERATION WITH SOLIDS RECYCLE



INTEGRATED TWO STAGE LIQUEFACTION
(RUNS: 243 1,2, 244,245,246)

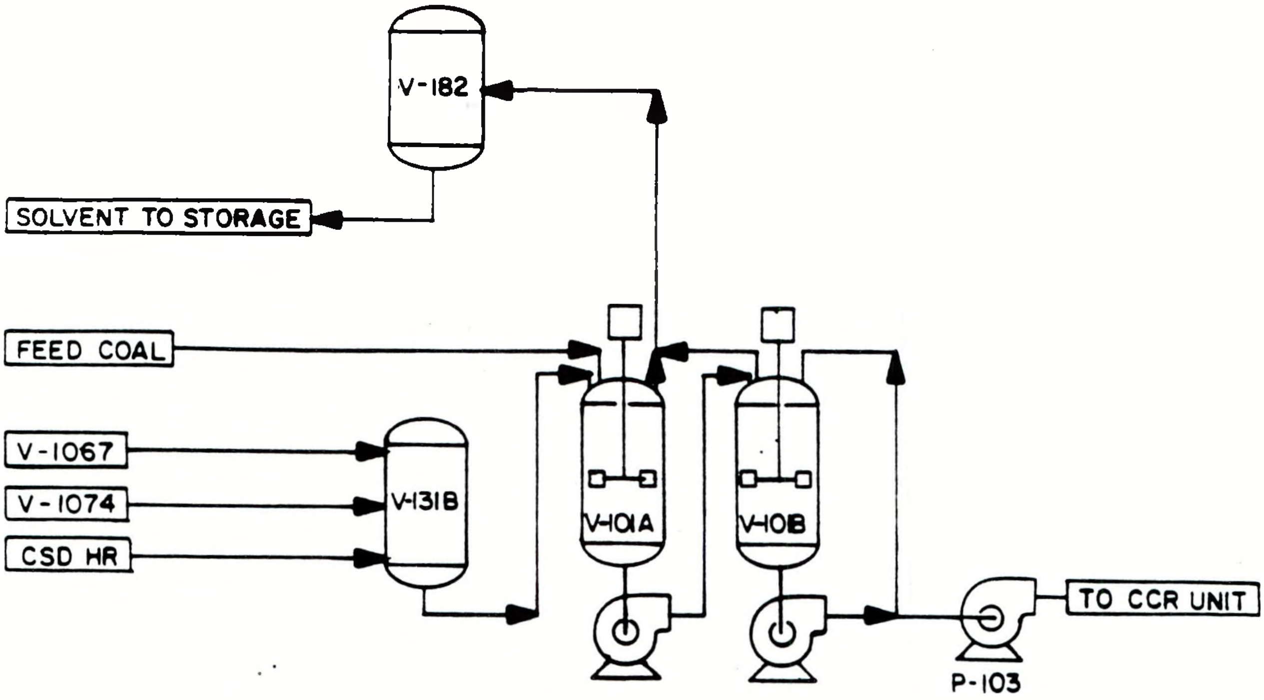


DOUBLE INTEGRATED TWO STAGE LIQUEFACTION
(RUNS: 246,248)



RECONFIGURED INTEGRATED TWO STAGE LIQUEFACTION
(RUNS: 247, 249)

FIGURE 2. TWO-STAGE LIQUEFACTION MODES TESTED SINCE RUN 242



V101A	SLURRY BLEND TANK
V101B	FEED TANK
V131B	RECOVERED PROCESS SOLVENT TANK
V182	SLURRY DRYING OVHD TANK
P103	HIGH PRESSURE FEED PUMP

FIGURE 3. SCHEMATIC FLOW DIAGRAM OF COAL FEED

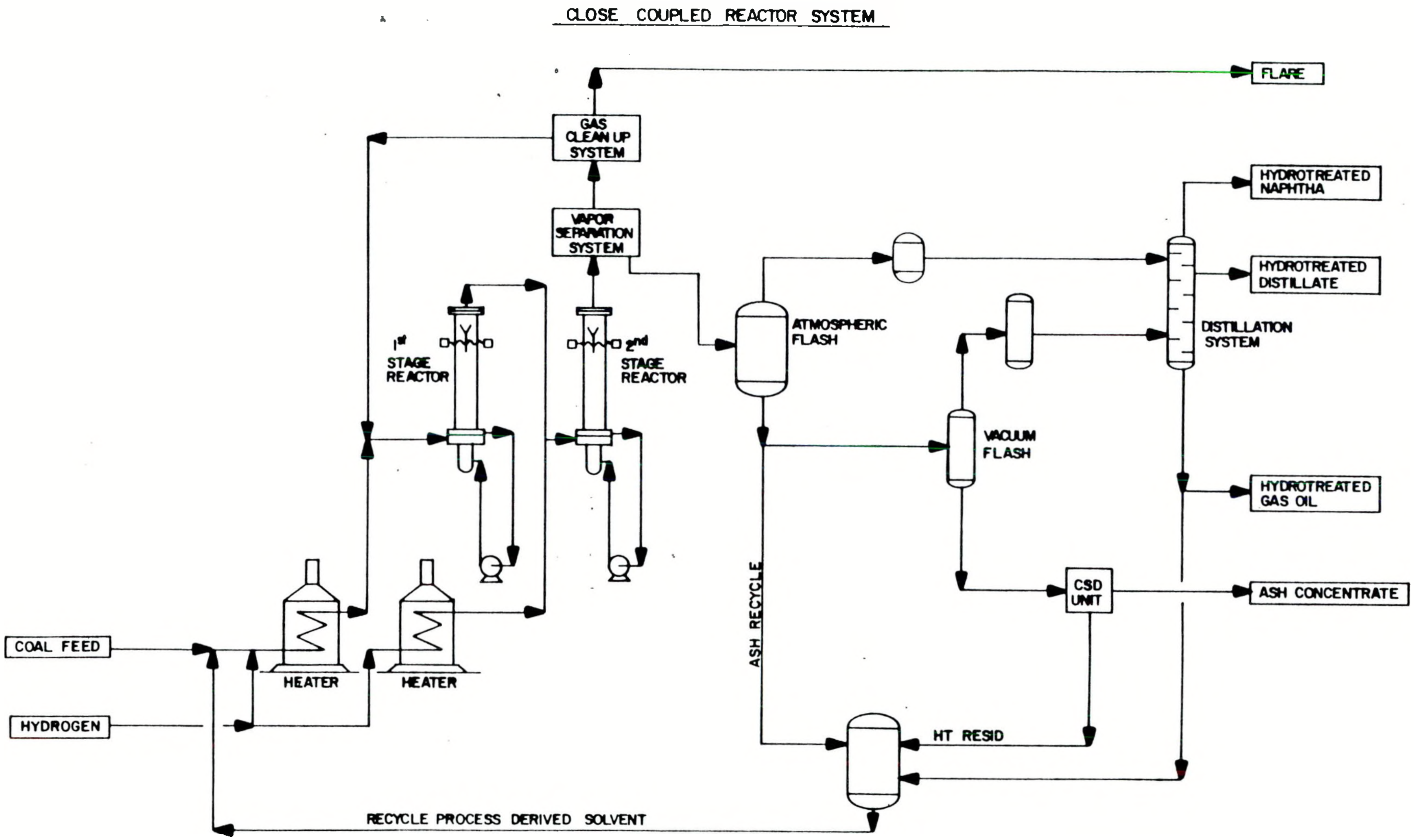


FIGURE 4. SCHEMATIC FLOW DIAGRAM OF THE CLOSE-COUPLED REACTOR (CCR) UNIT WITHOUT INTERSTAGE SEPARATION

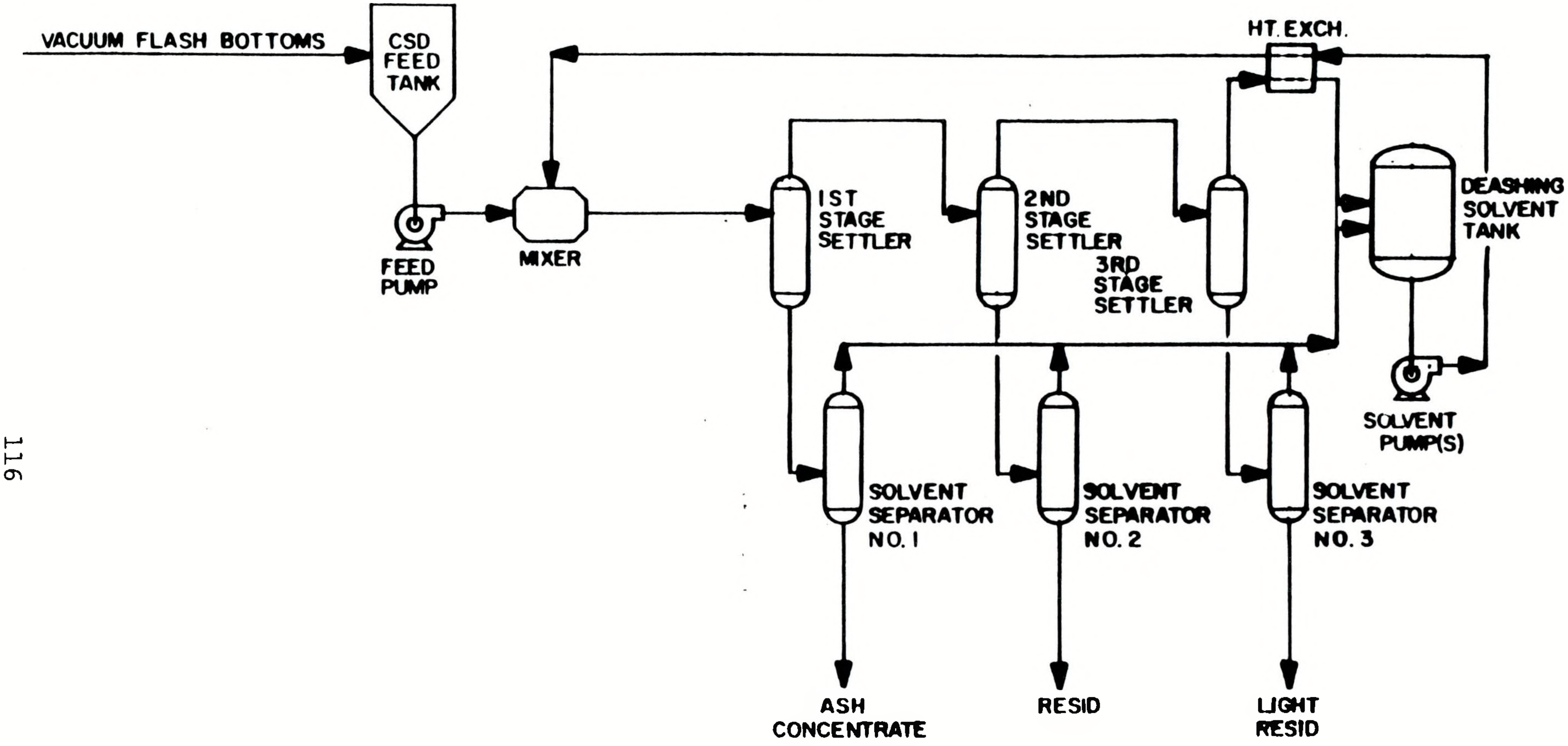


FIGURE 5. SCHEMATIC FLOW DIAGRAM OF THE CRITICAL SOLVENT DEASHING UNIT

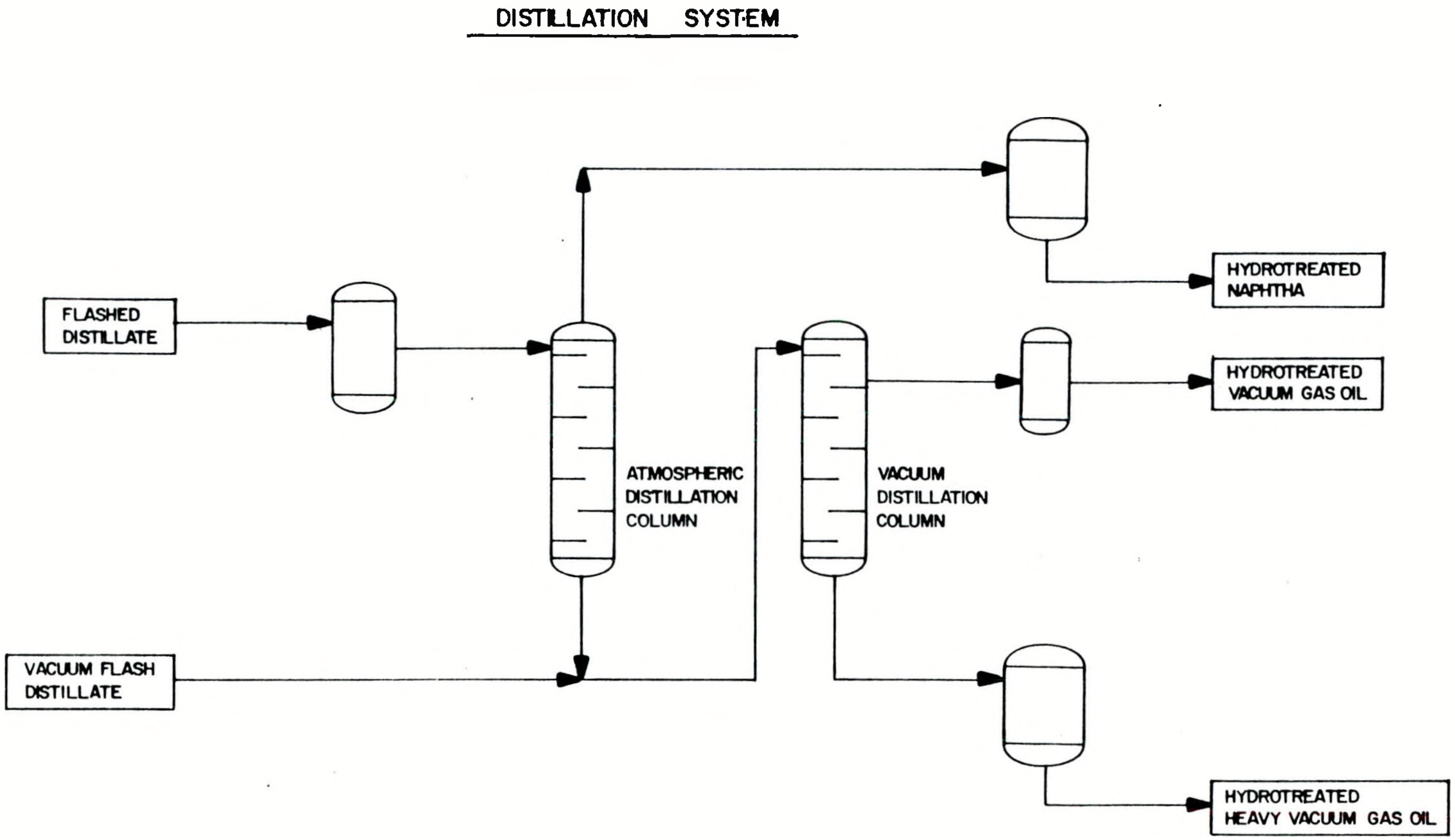


FIGURE 6. SCHEMATIC FLOW DIAGRAM OF THE DISTILLATION SYSTEM

FIGURE 7. ORGANIC REJECTION VS COAL ASH

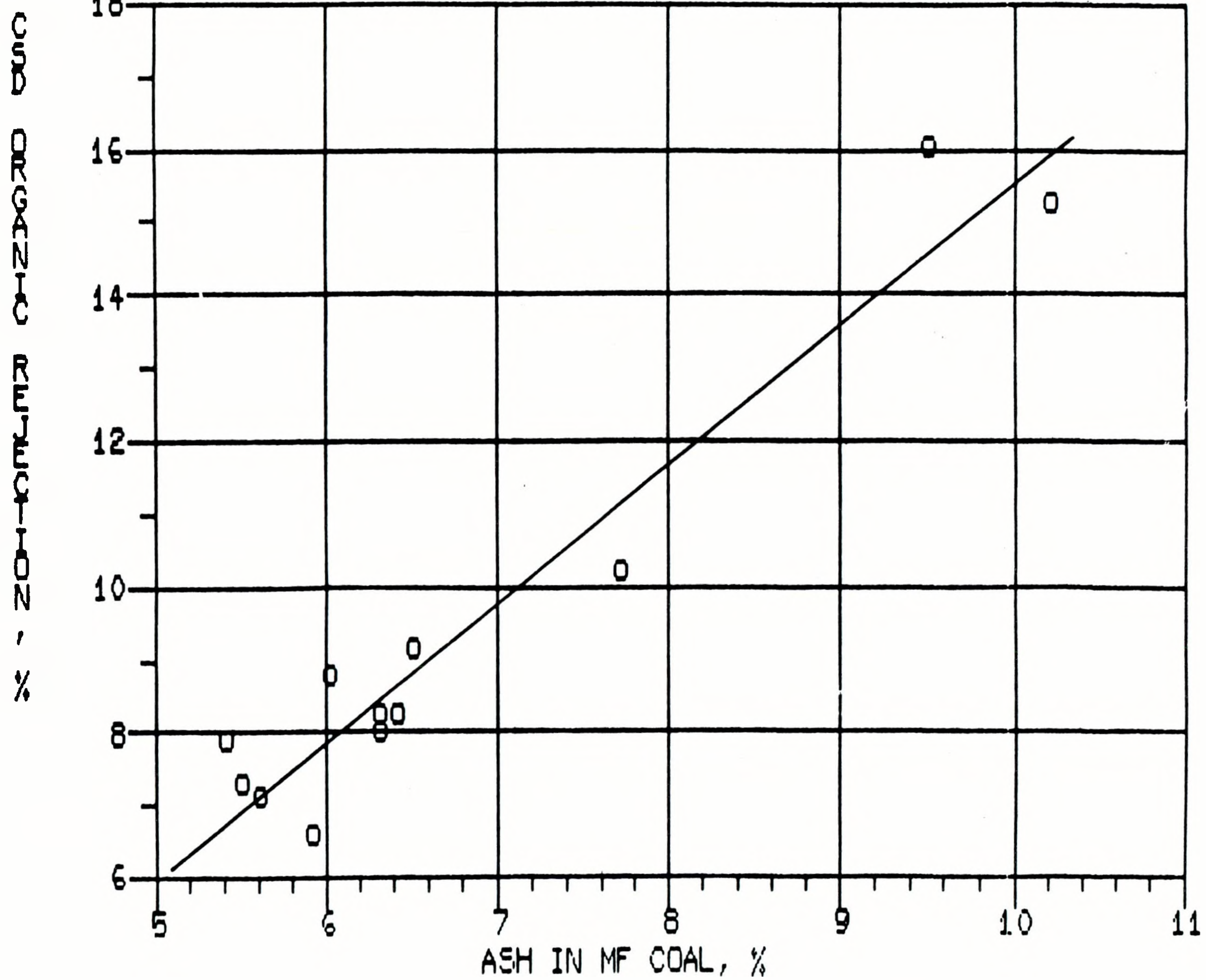
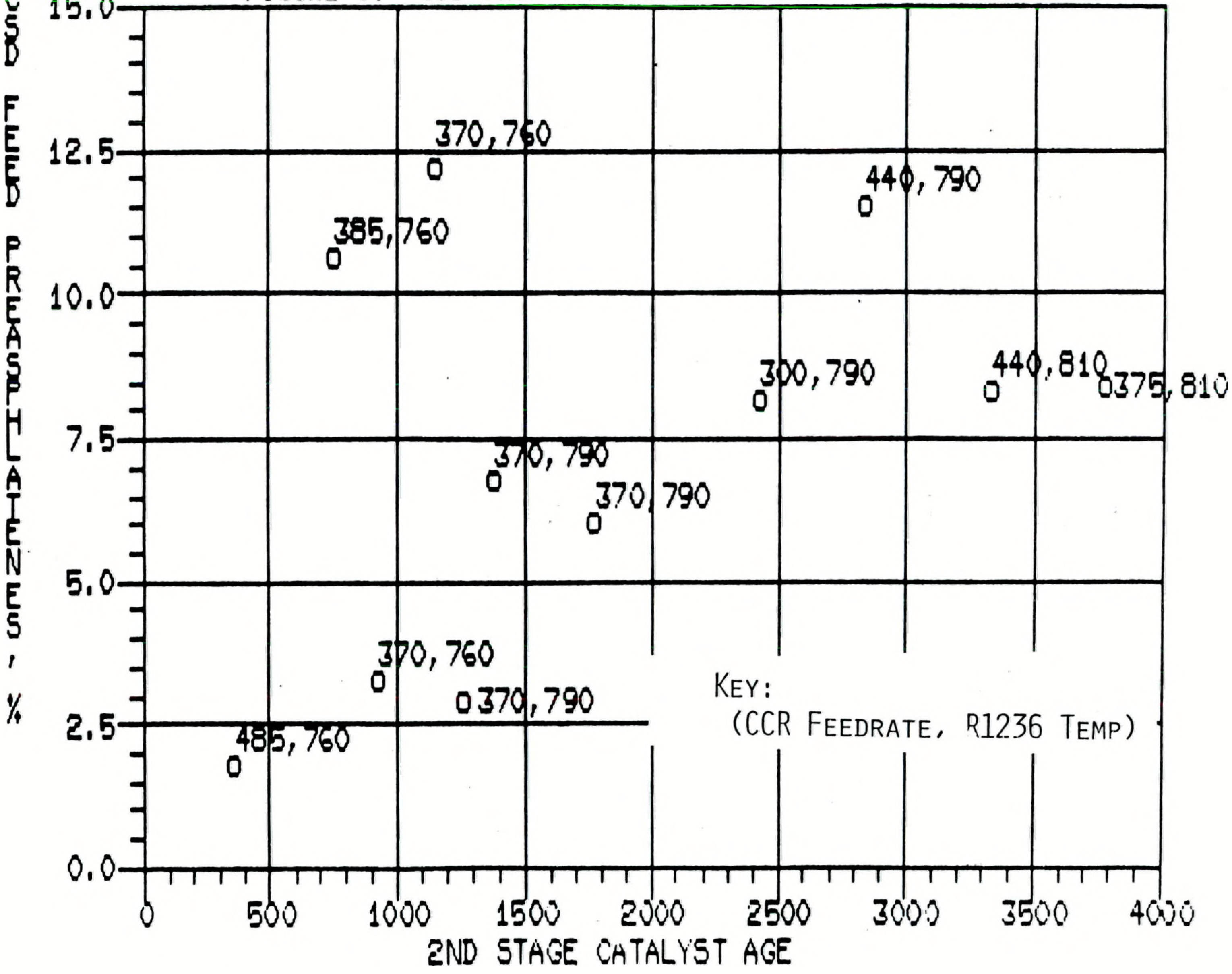


FIGURE 8. CSD PREASPHALTENES VS R1236 CATALYST AGE



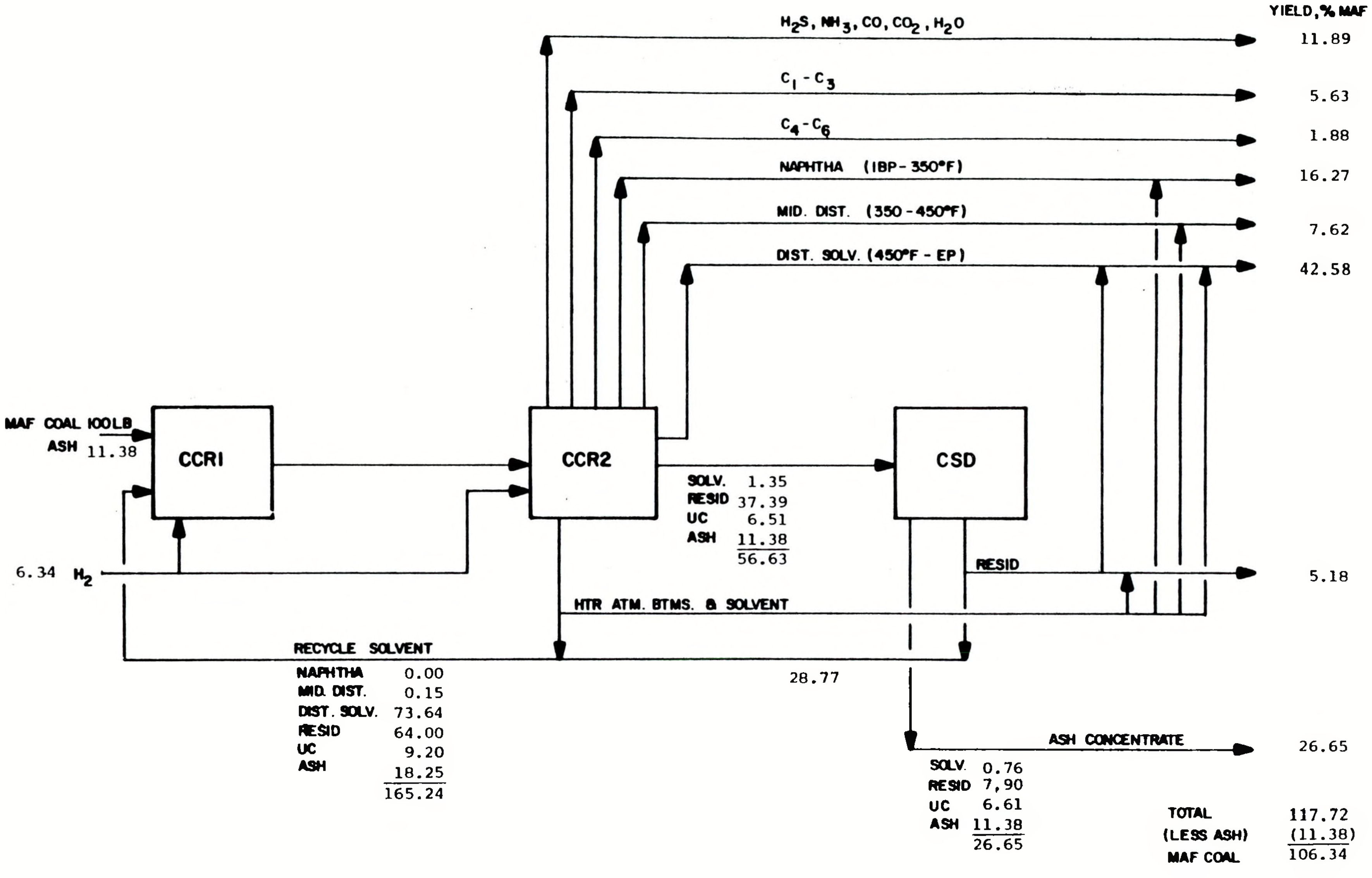


FIGURE 9. MATERIAL BALANCE FLOW DIAGRAM FOR 254A

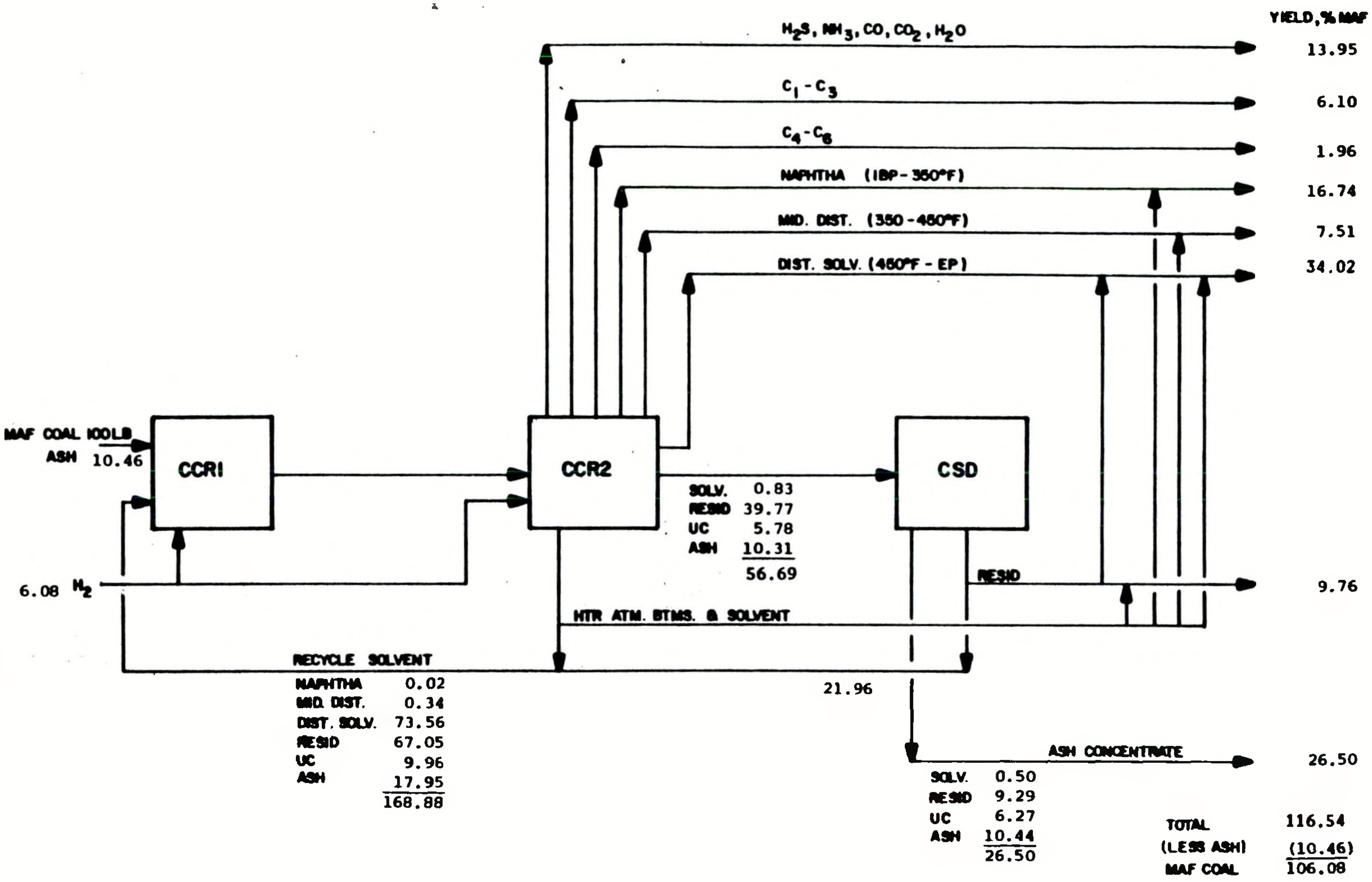


FIGURE 10. MATERIAL BALANCE FLOW DIAGRAM FOR 254B

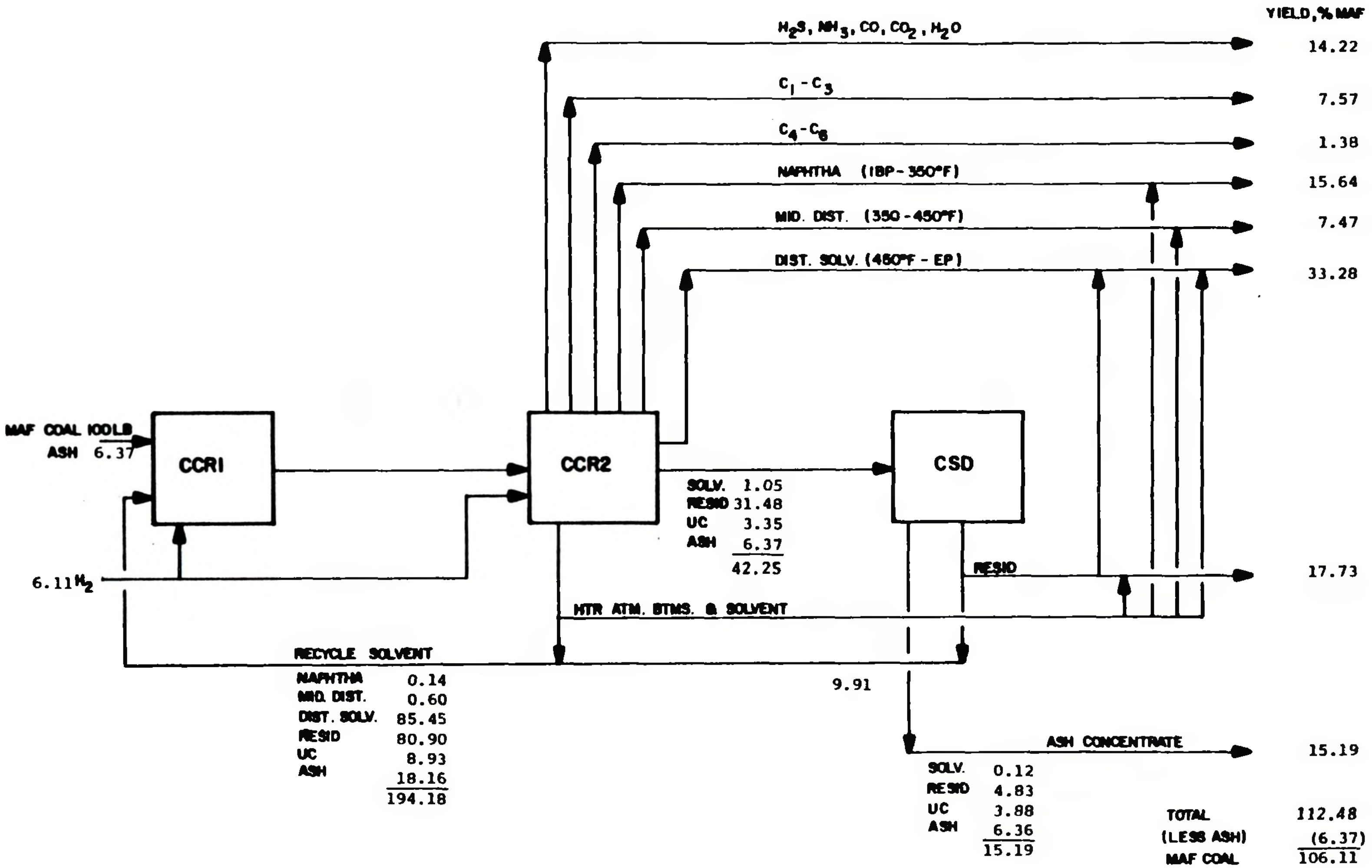


FIGURE 11. MATERIAL BALANCE FLOW DIAGRAM FOR 254C

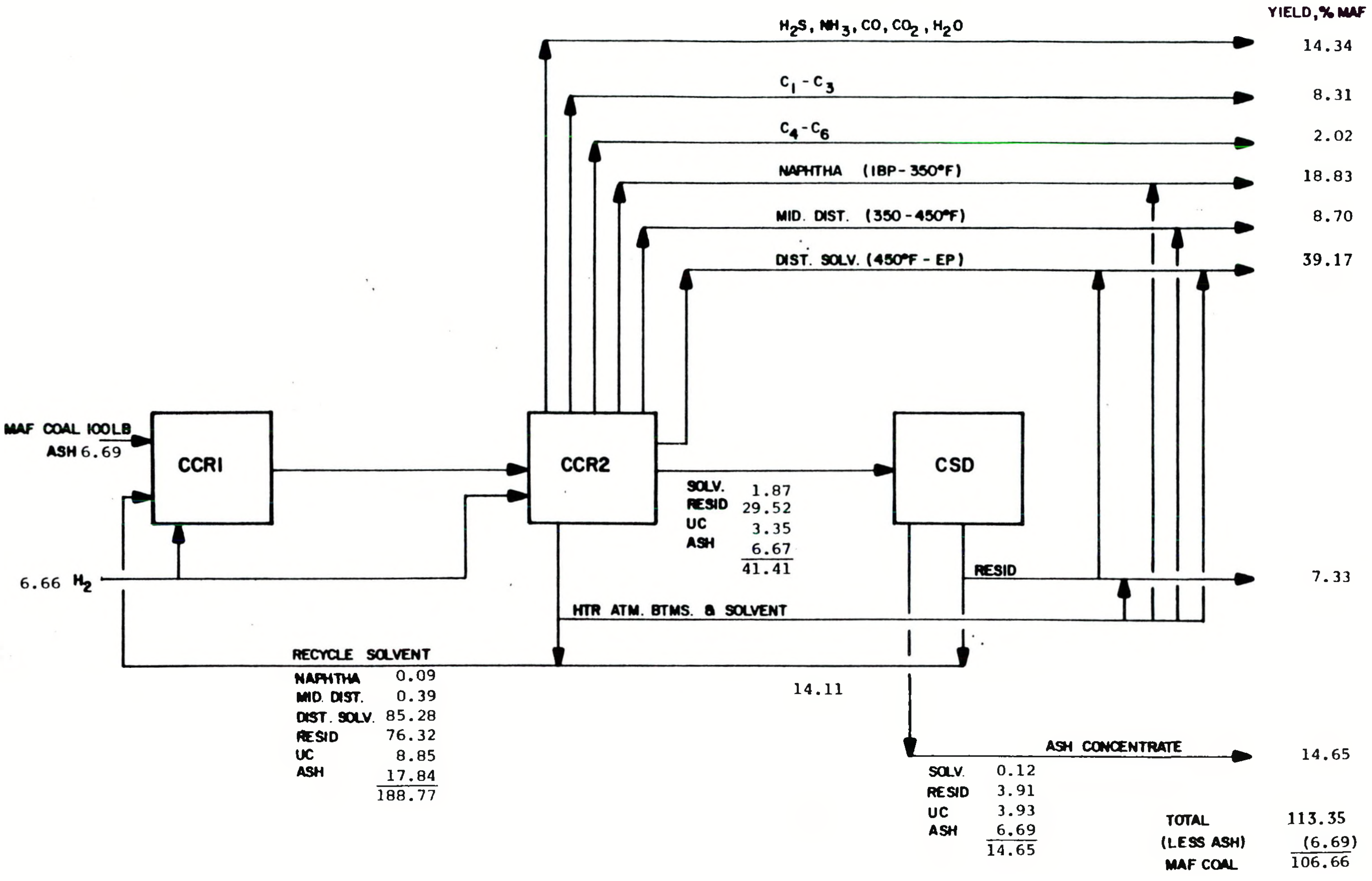


FIGURE 12. MATERIAL BALANCE FLOW DIAGRAM FOR 254D

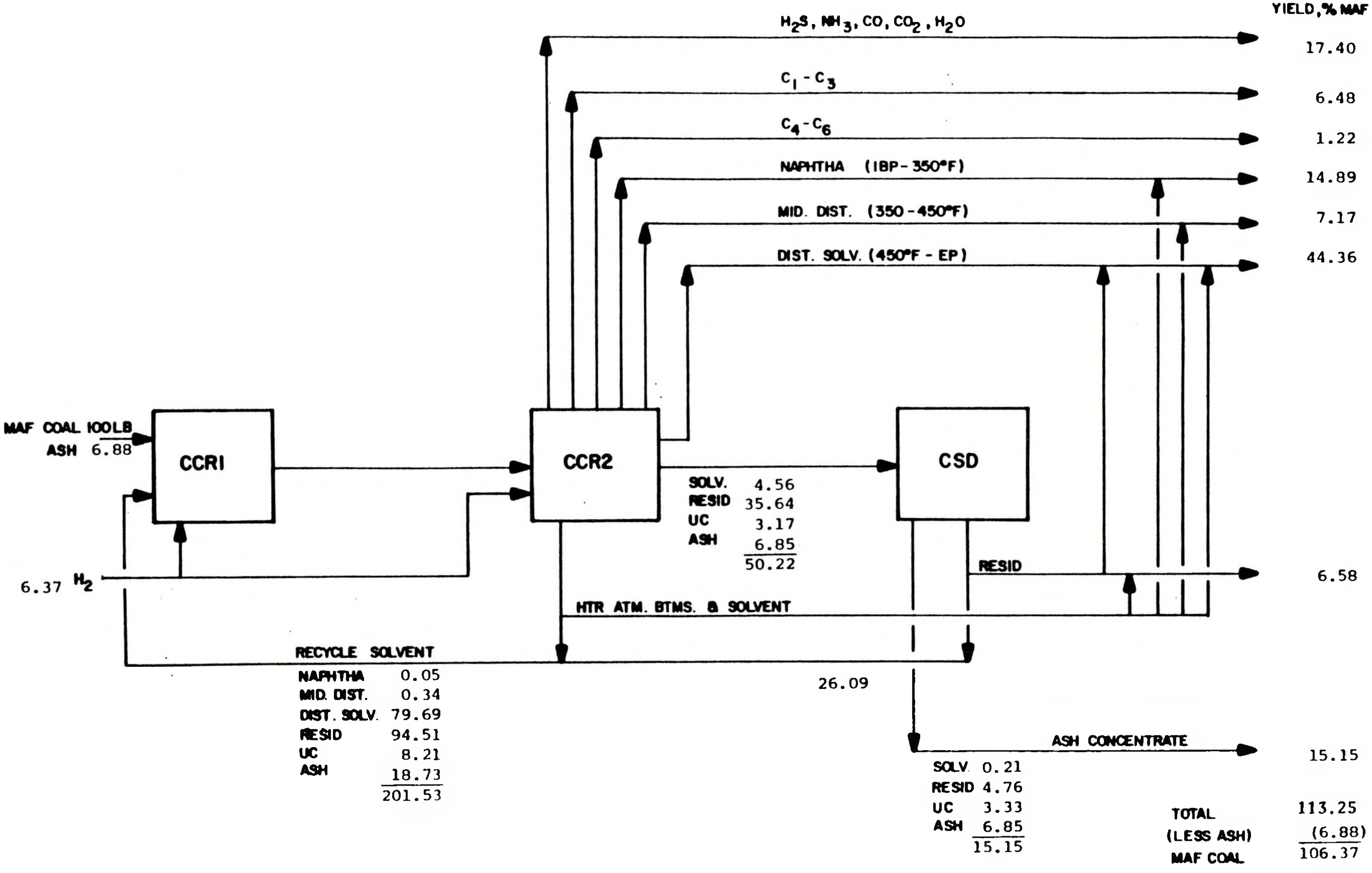


FIGURE 13. MATERIAL BALANCE FLOW DIAGRAM FOR 254E

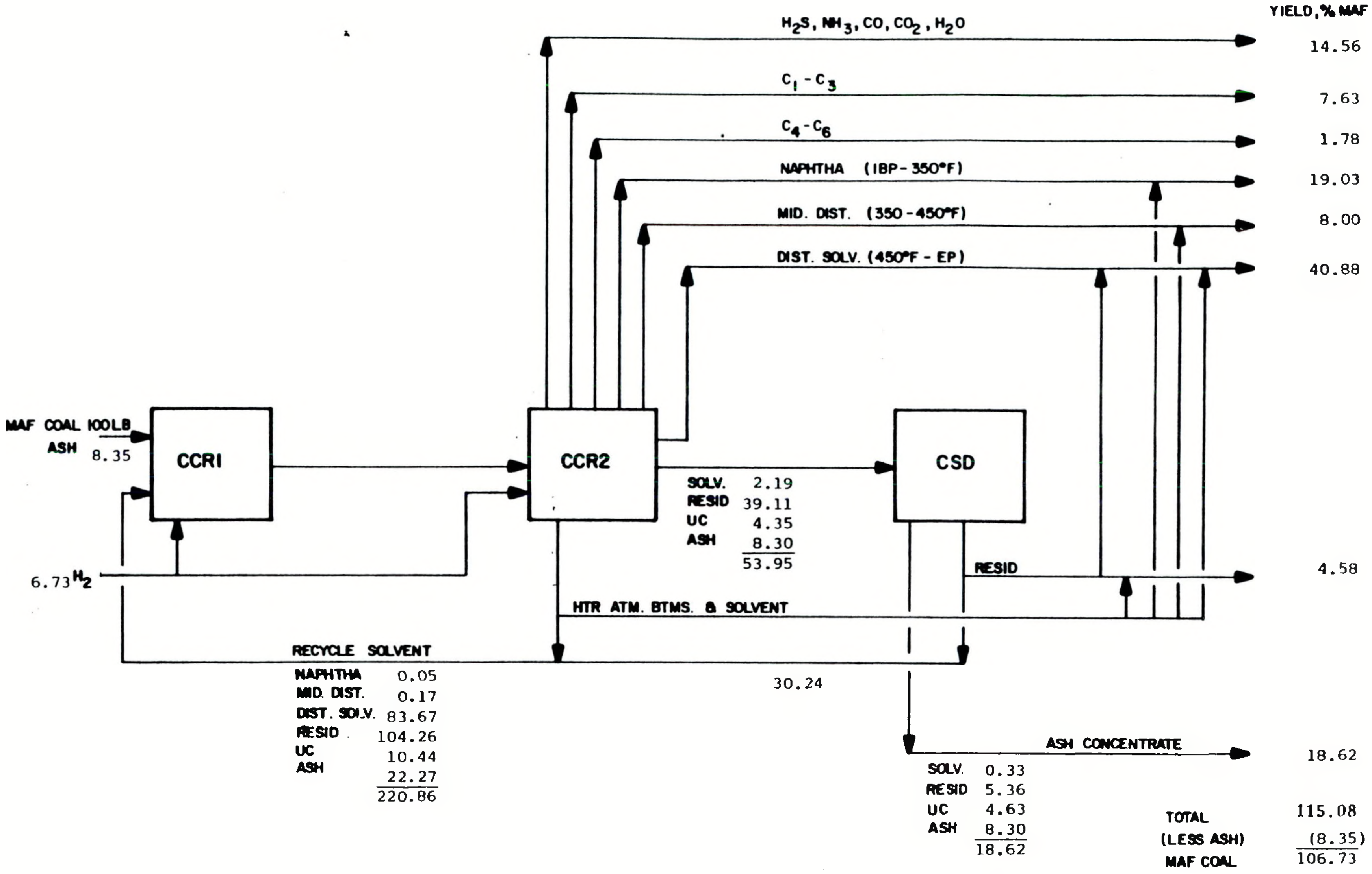


FIGURE 14. MATERIAL BALANCE FLOW DIAGRAM FOR 254F

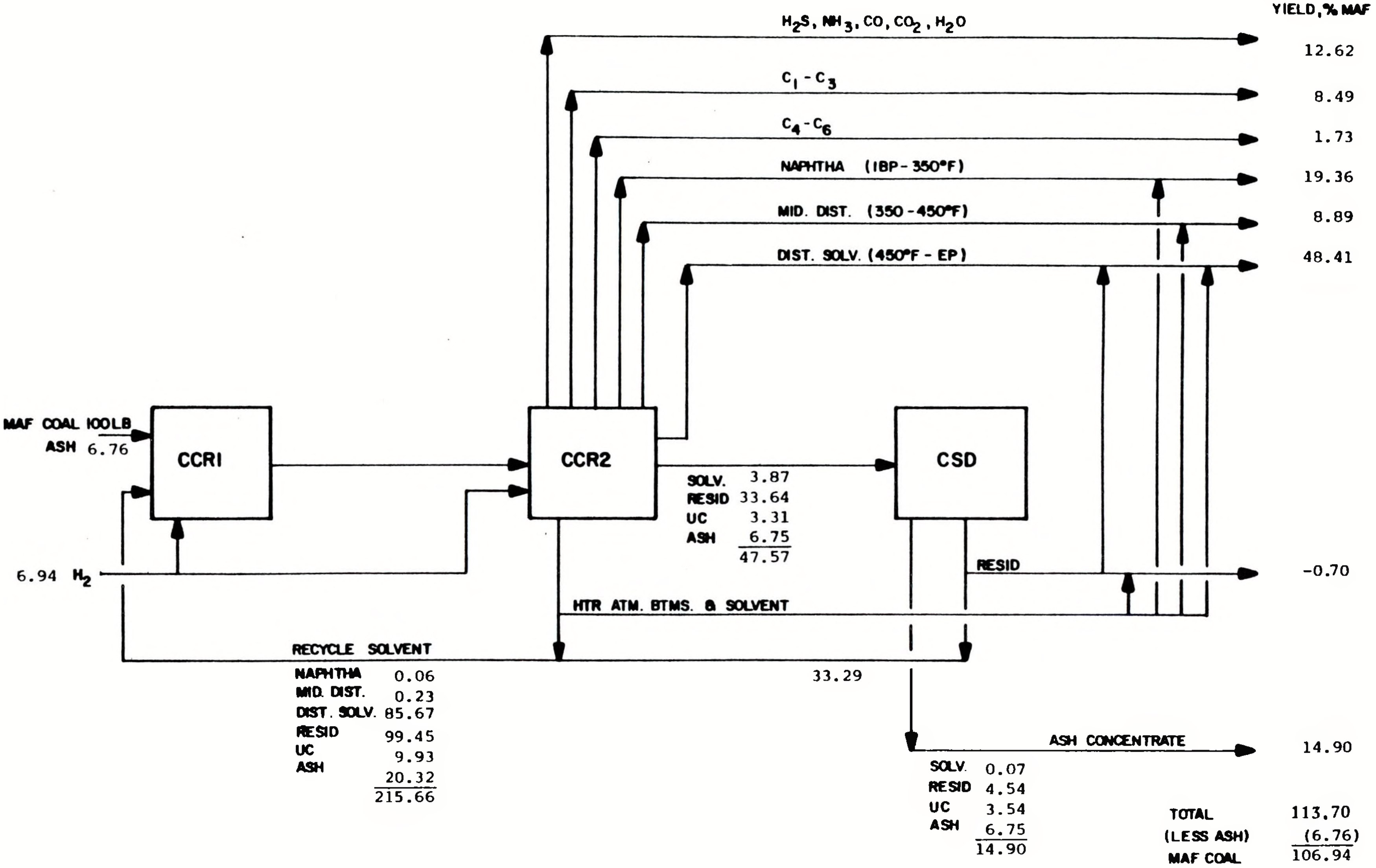


FIGURE 15. MATERIAL BALANCE FLOW DIAGRAM FOR 254G

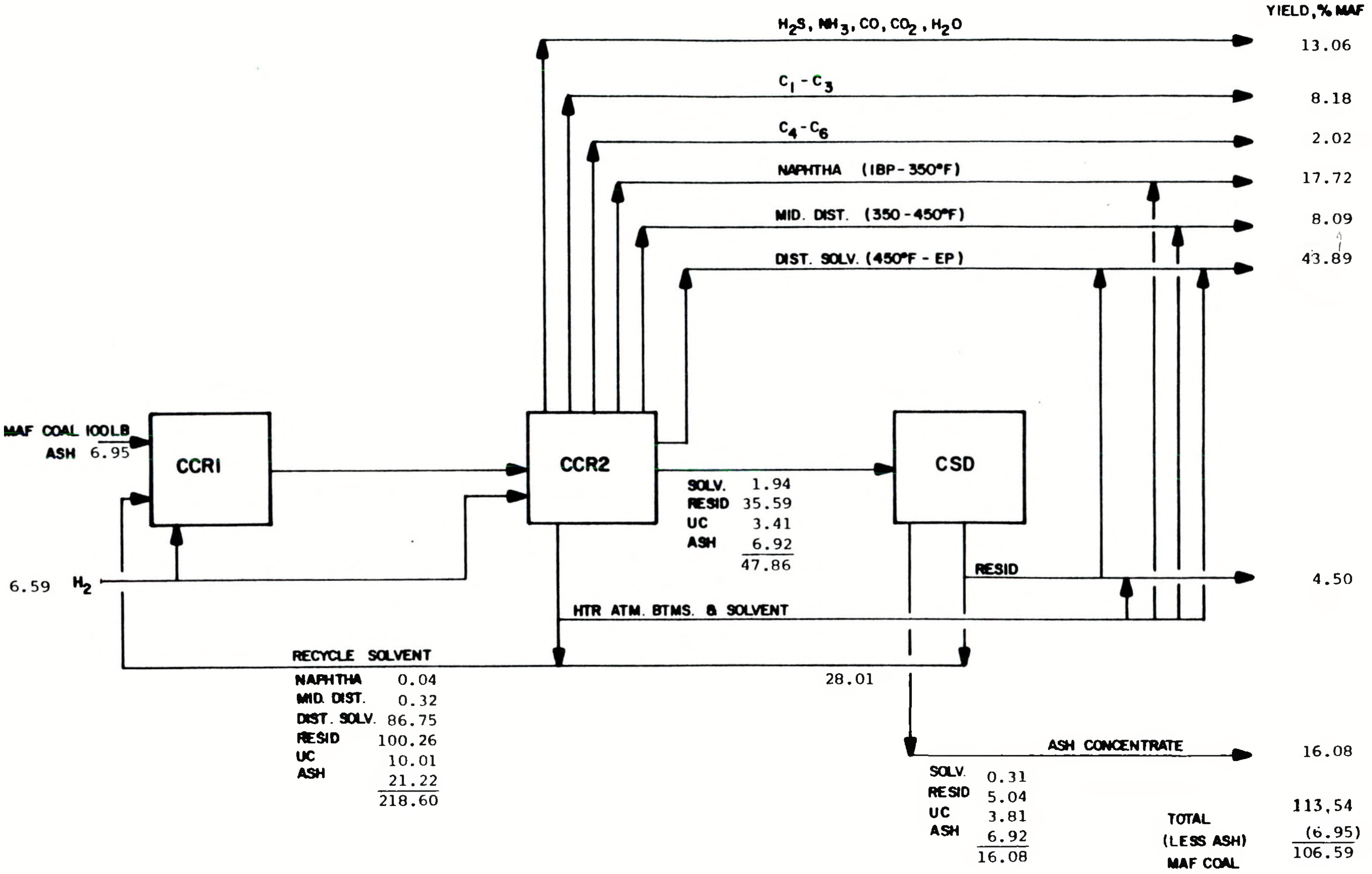


FIGURE 16. MATERIAL BALANCE FLOW DIAGRAM FOR 254H

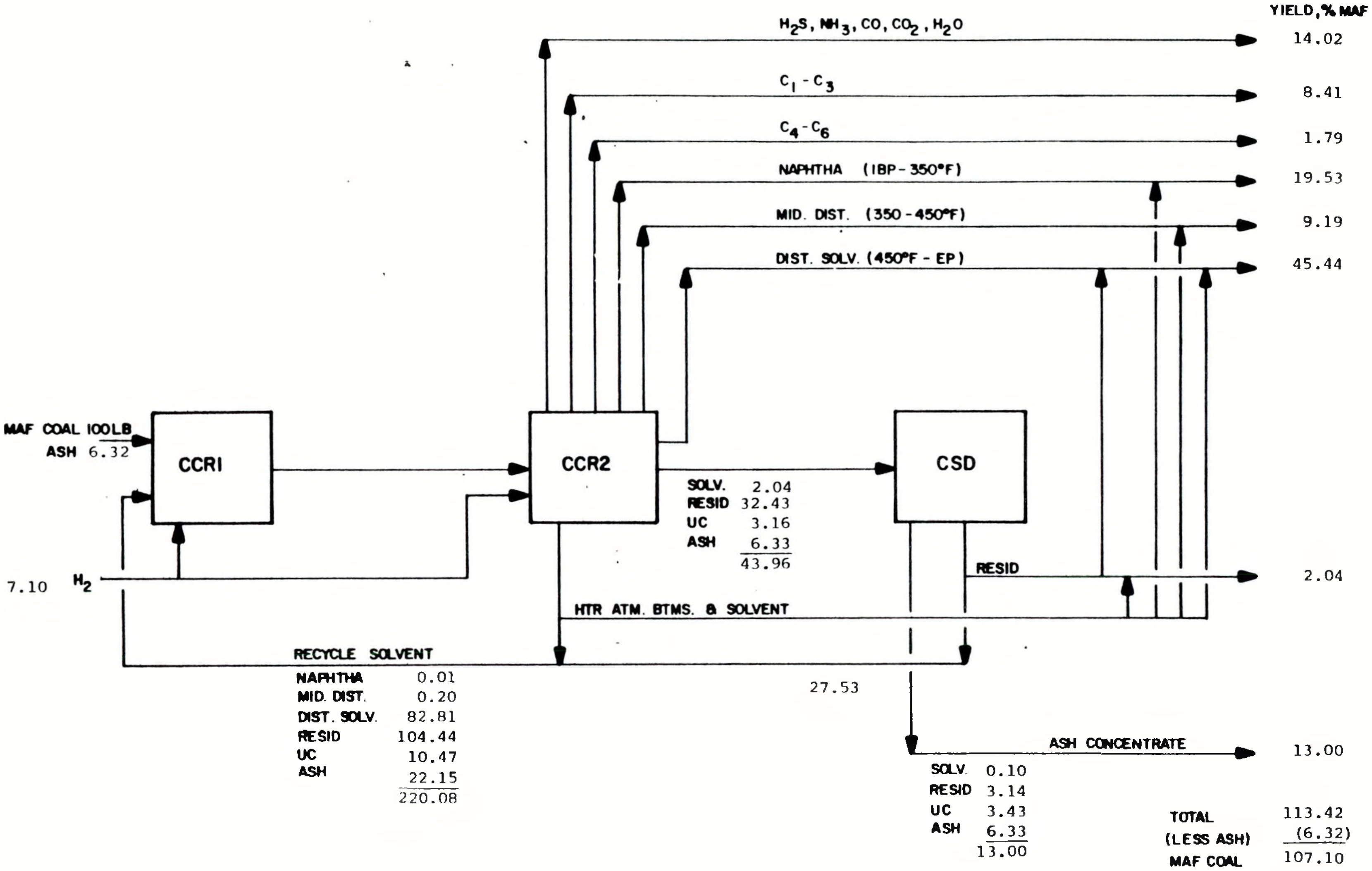


FIGURE 17. MATERIAL BALANCE FLOW DIAGRAM FOR 254I

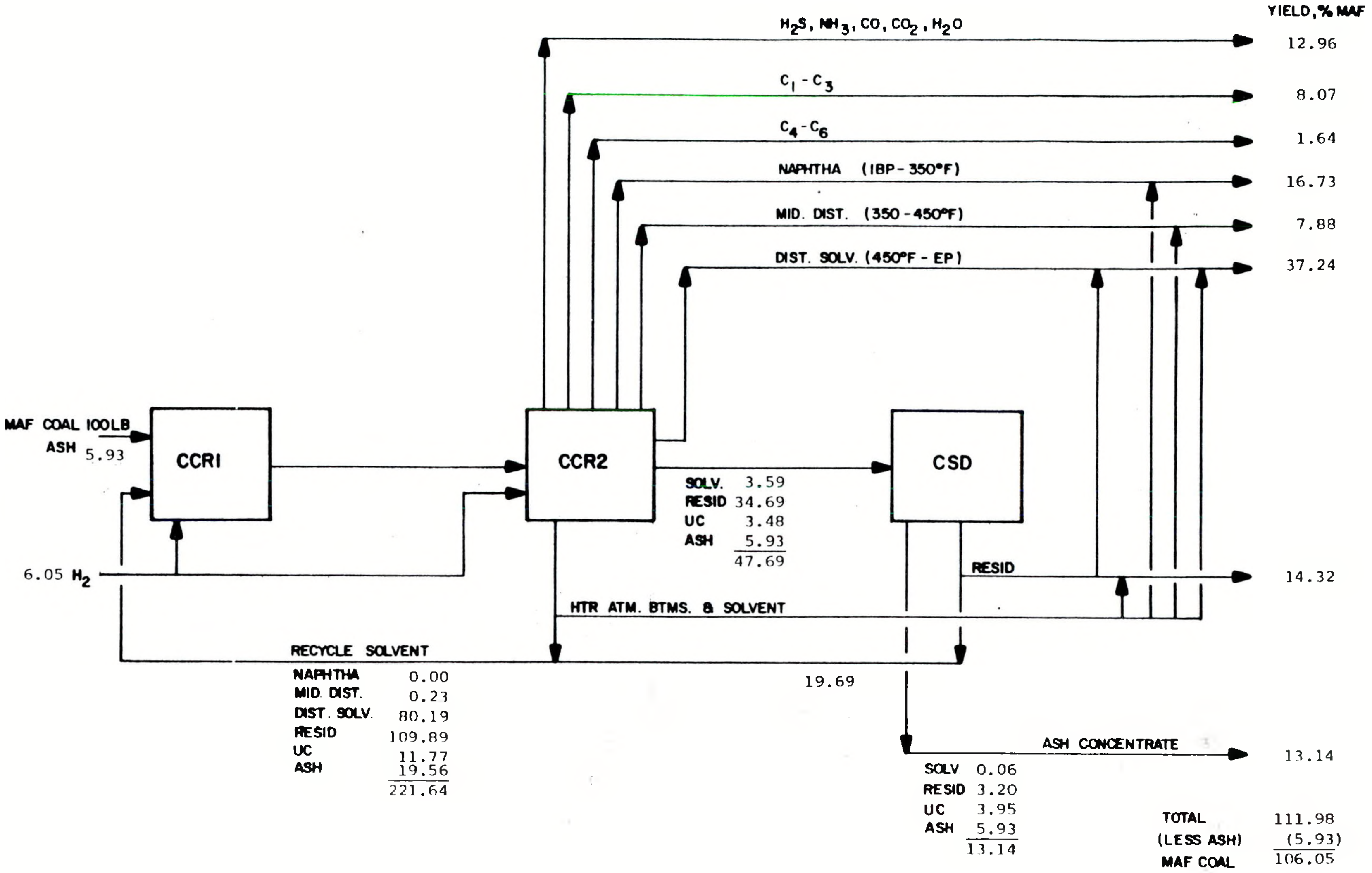


FIGURE 18. MATERIAL BALANCE FLOW DIAGRAM FOR 254J

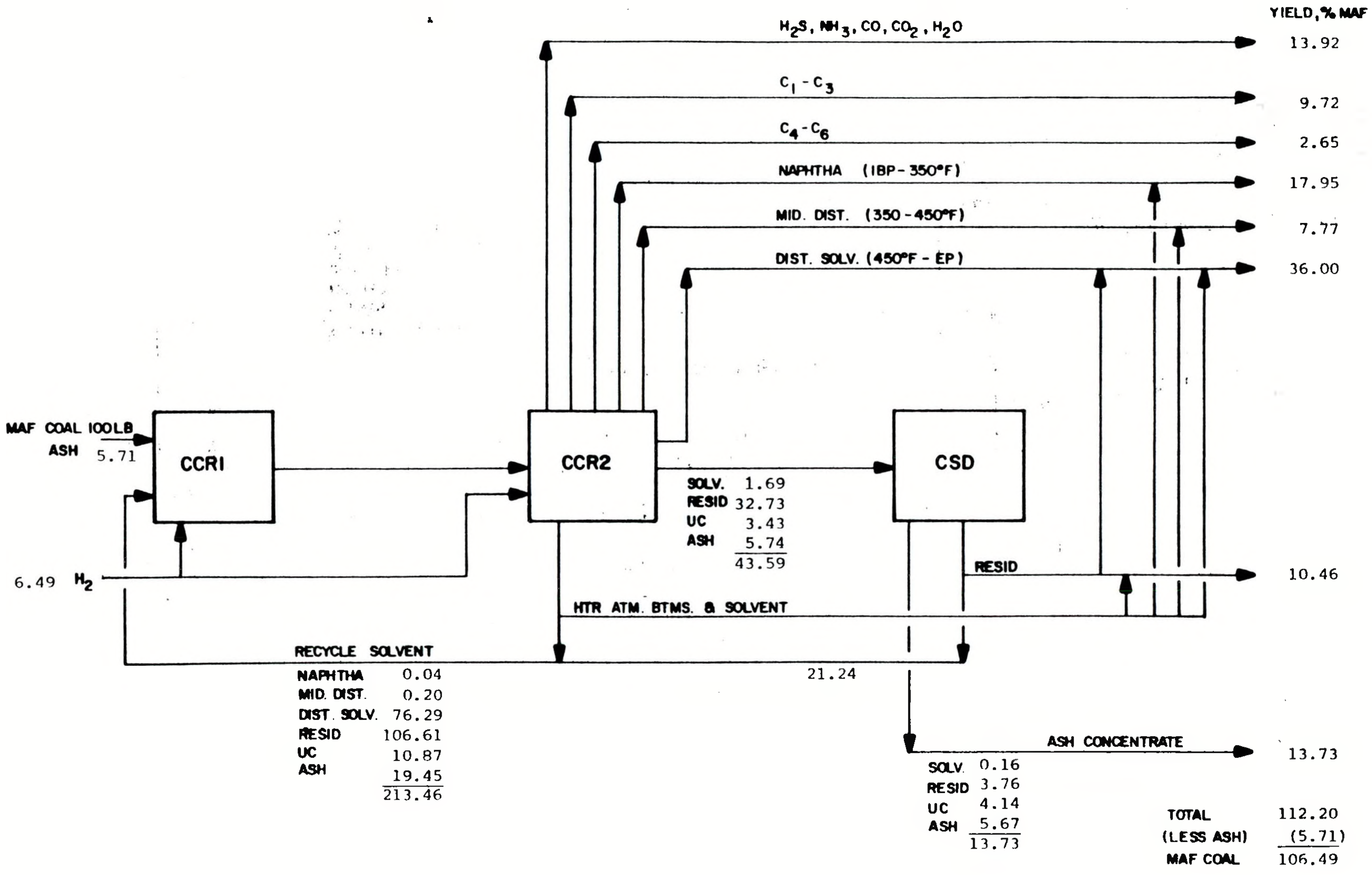


FIGURE 19. MATERIAL BALANCE FLOW DIAGRAM FOR 254K

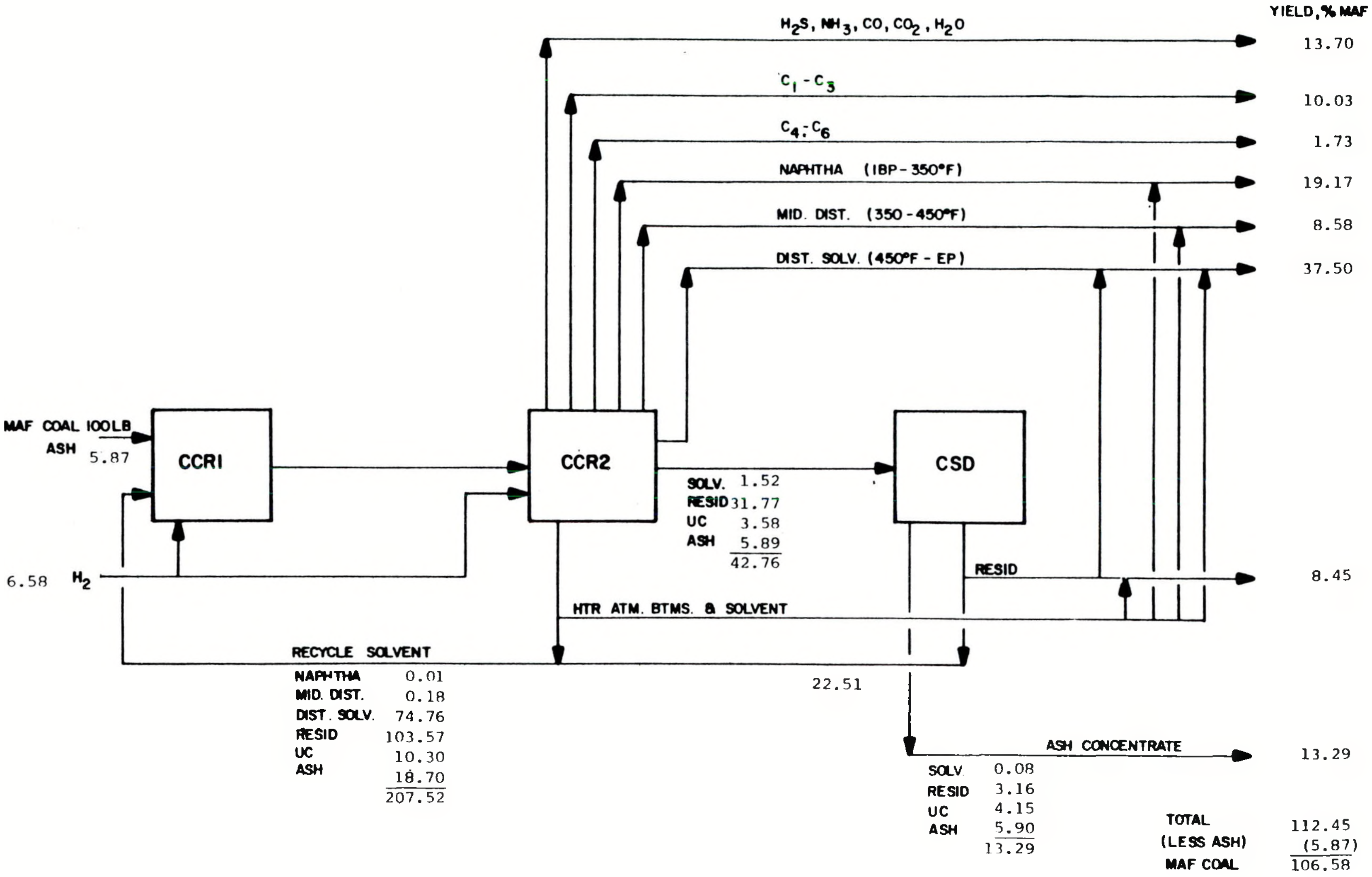


FIGURE 20. MATERIAL BALANCE FLOW DIAGRAM FOR 254L

FIGURE 21. OVERALL DISTILLATE YIELD RESPONSE 254A-B

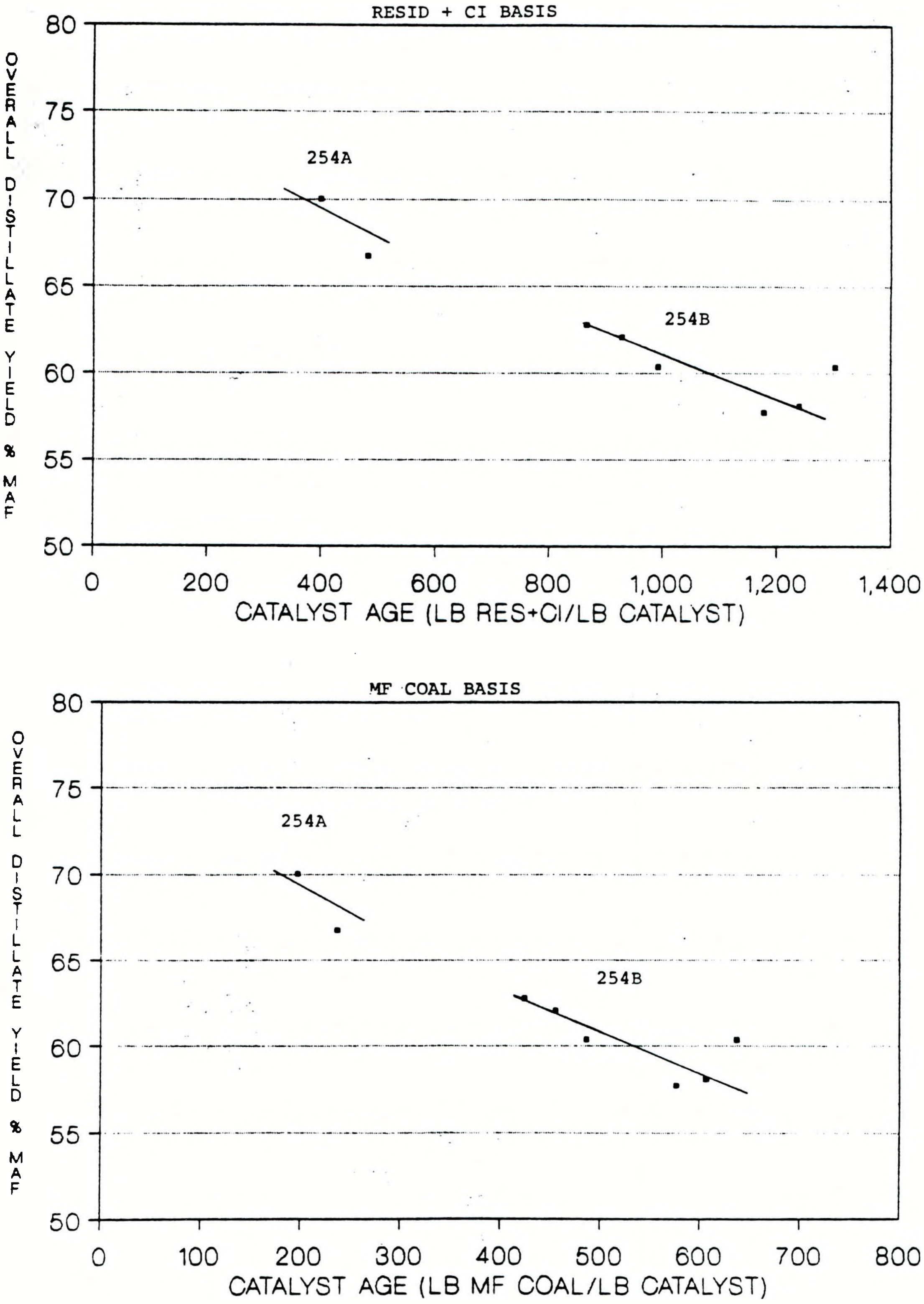
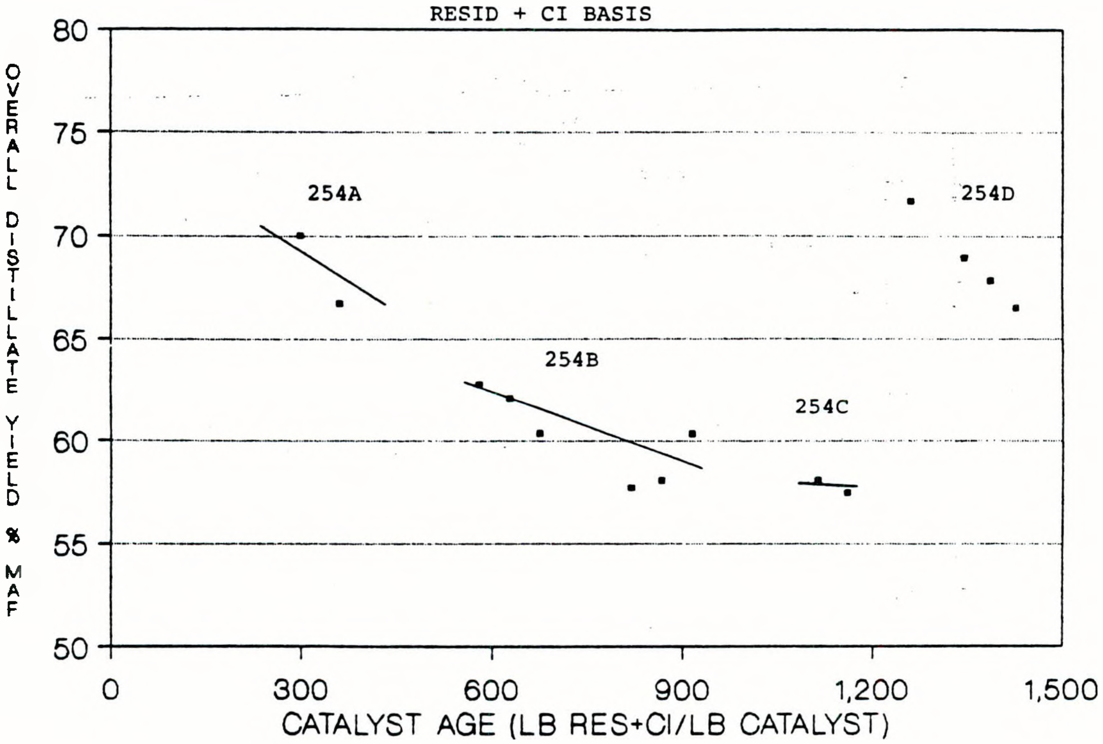


FIGURE 22. OVERALL DISTILLATE YIELD WITH RESPECT TO SECOND STAGE CATALYST AGE FOR 254A-D



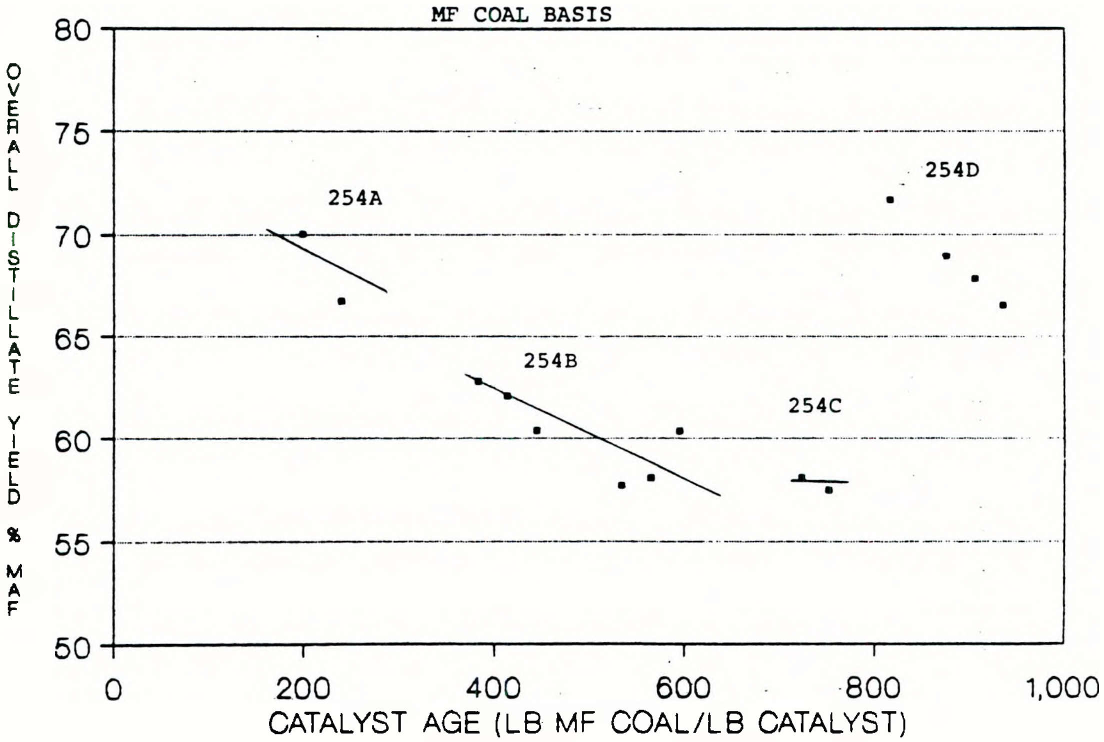


FIGURE 23. OVERALL DISTILLATE YIELD WITH RESPECT TO SECOND STAGE CATALYST AGE FOR 254E-L

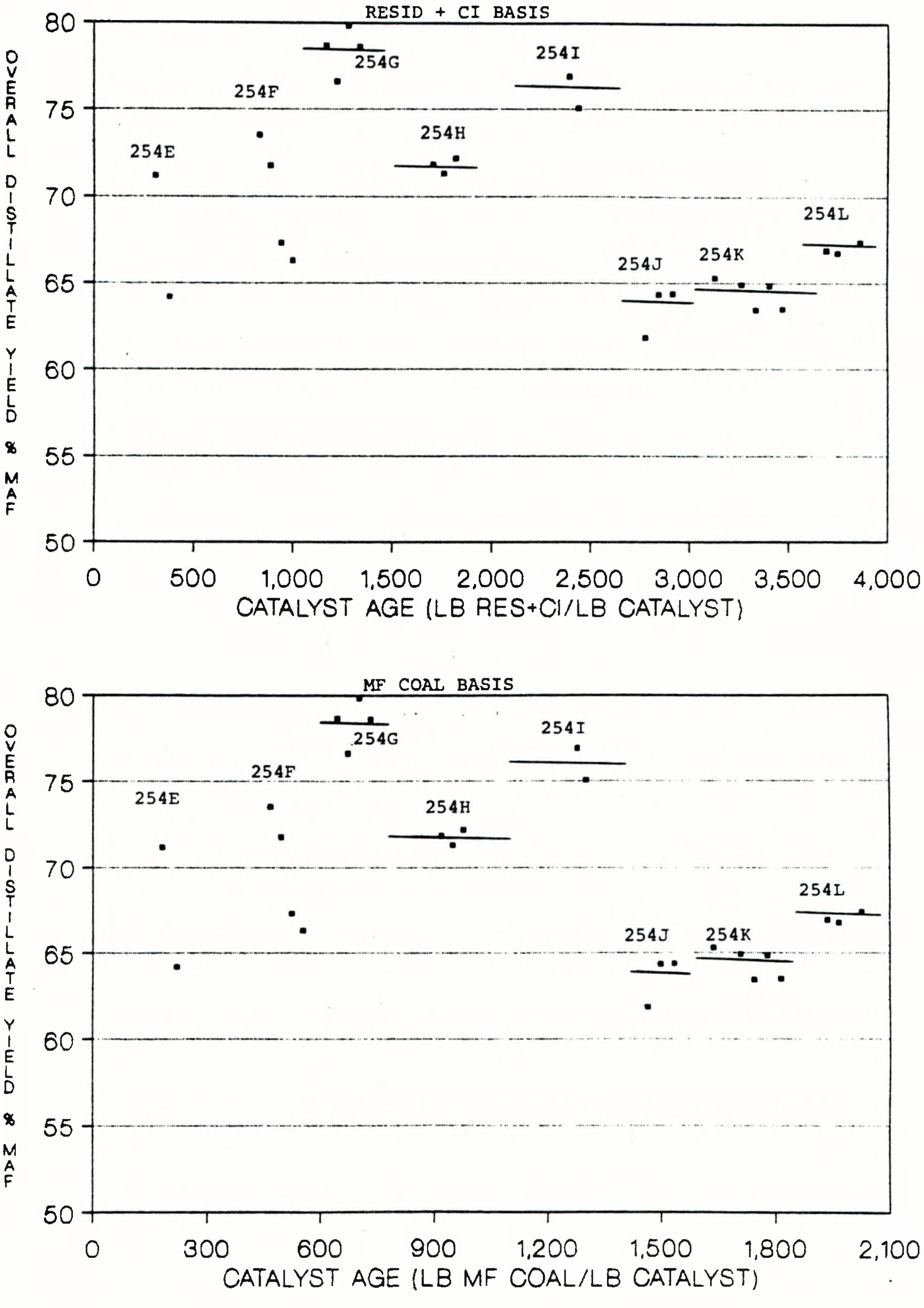


FIGURE 24. CI FREE RESID HYDROGEN CONTENT FOR THE RECYCLE PROCESS SOLVENT AND THE INTERSTAGE SAMPLES FOR RUN 254

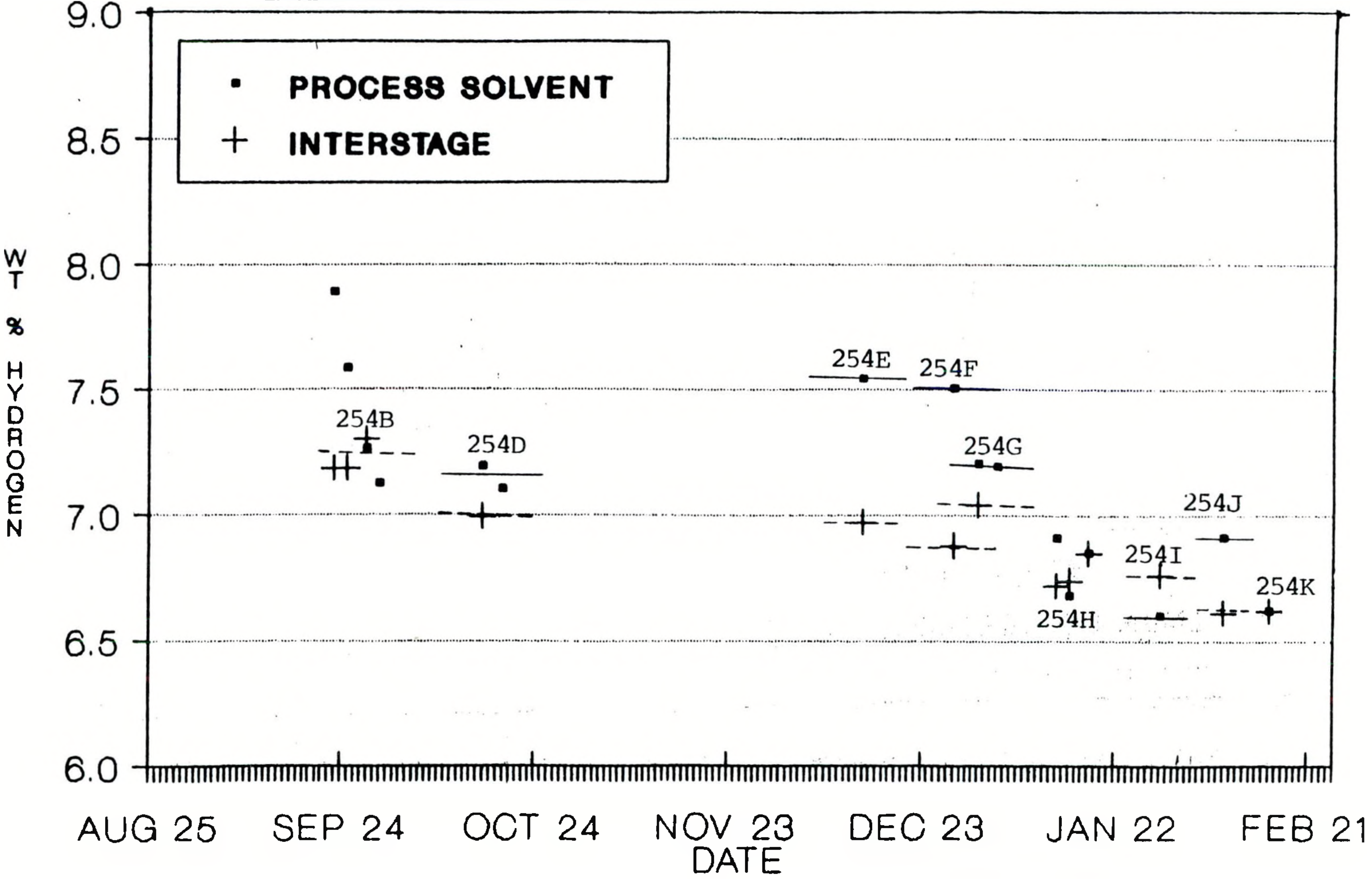


FIGURE 25. TOTAL HYDROGEN CONTENT OF THE RECYCLE PROCESS SOLVENT AND THE INTERSTAGE SAMPLES FOR RUN 254

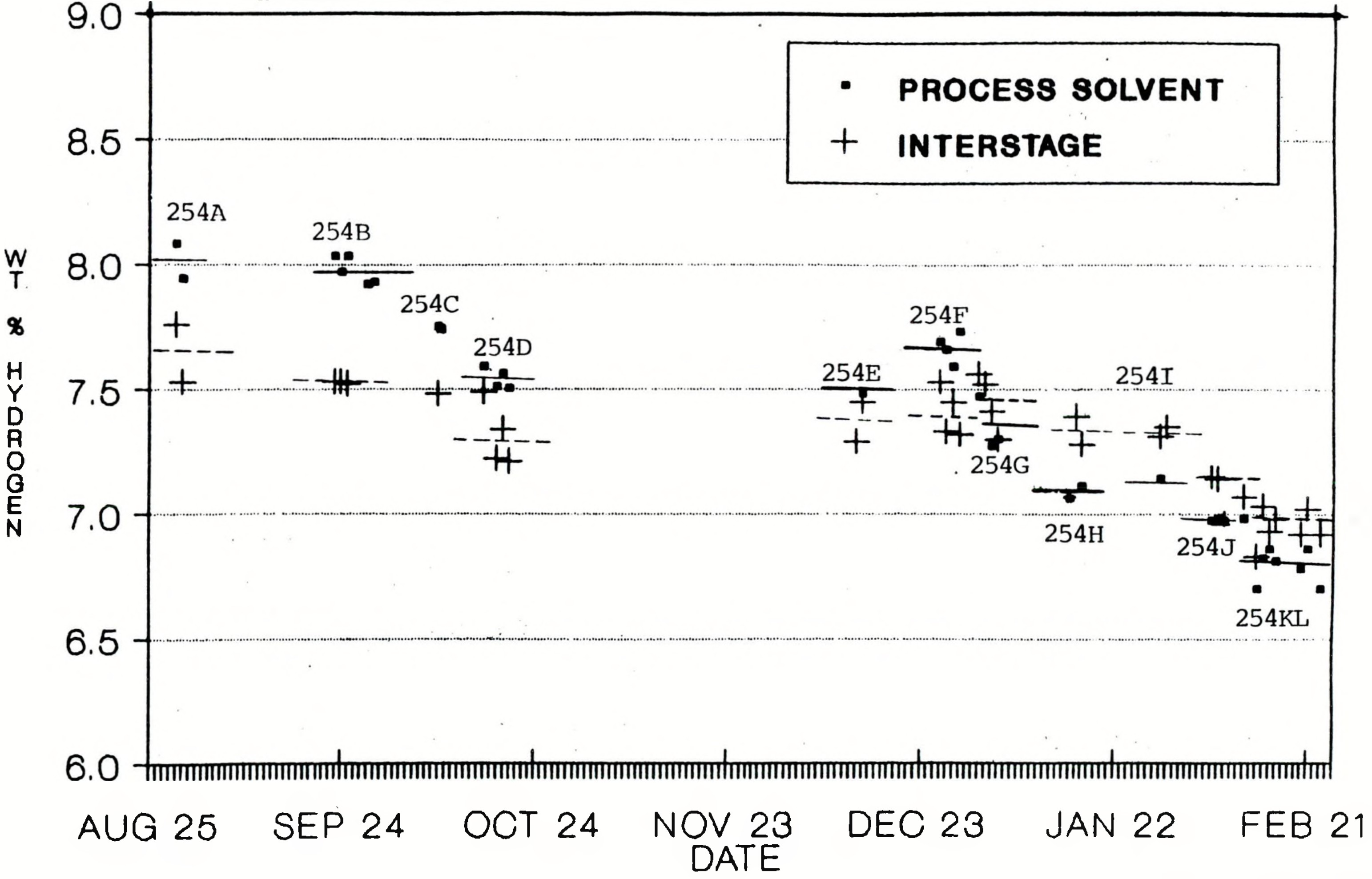


FIGURE 26. PROCESS SEVERITY STUDY OF COAL TYPES (HETEROATOM REMOVAL)

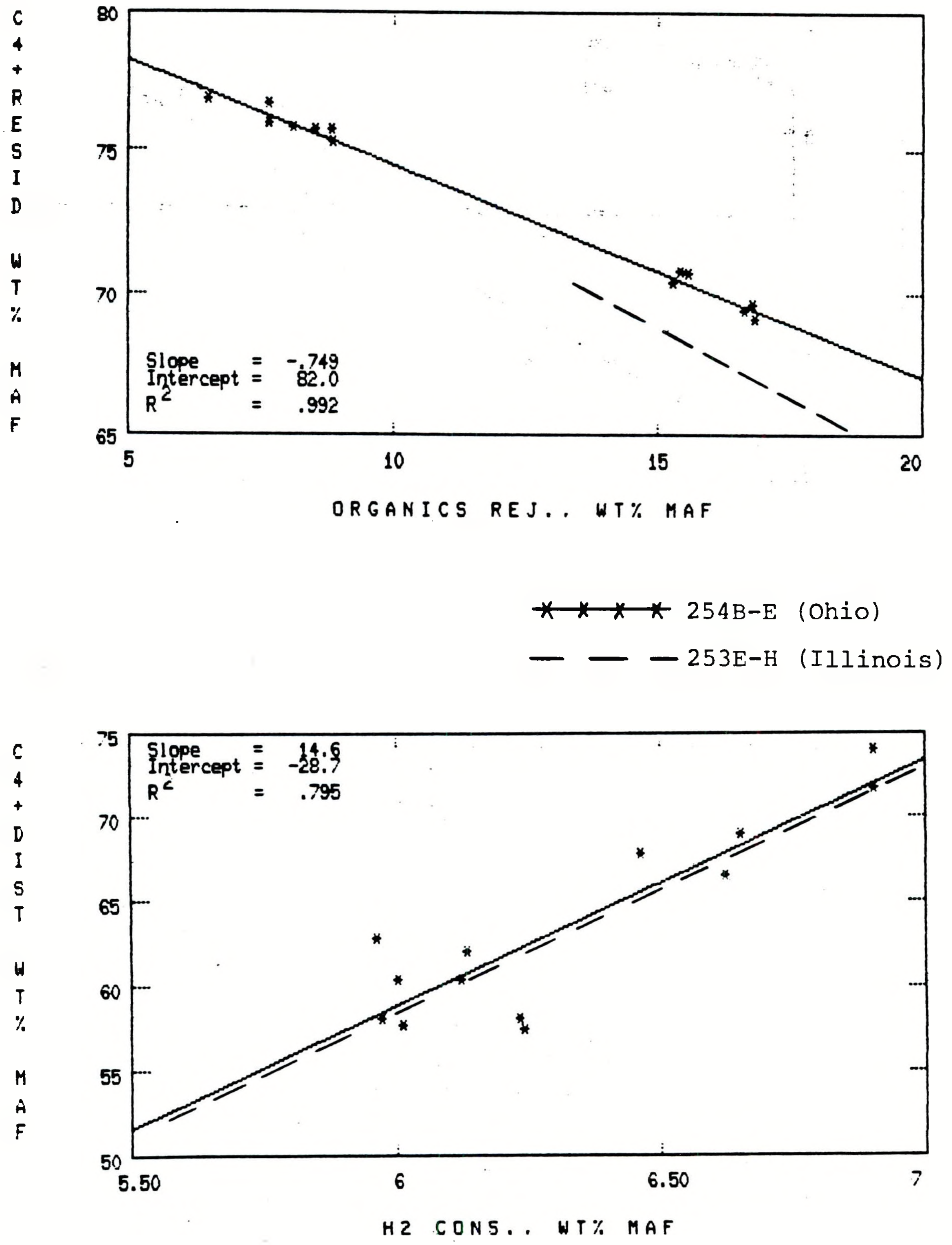


FIGURE 26. (continued)

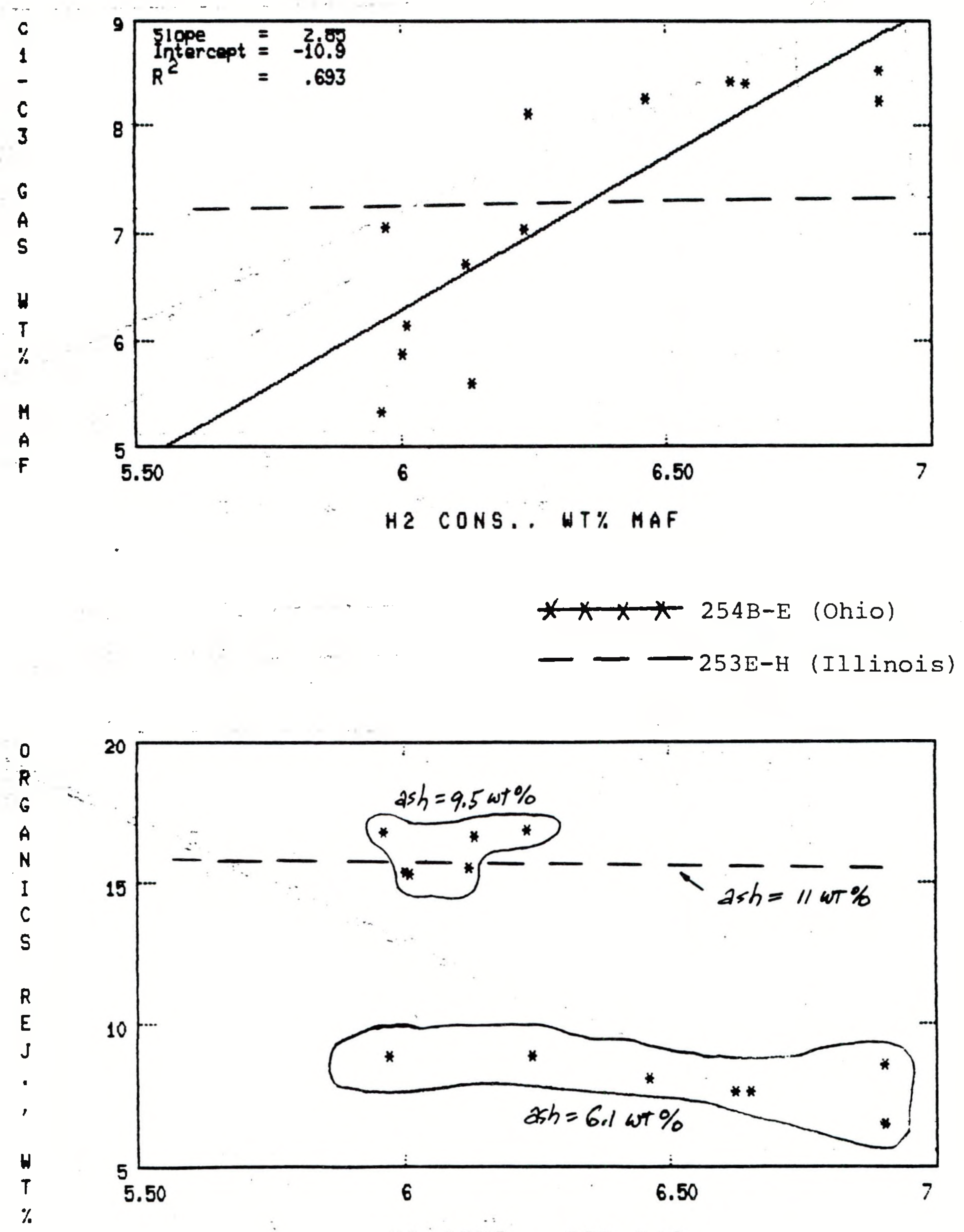


FIGURE 26. (continued)

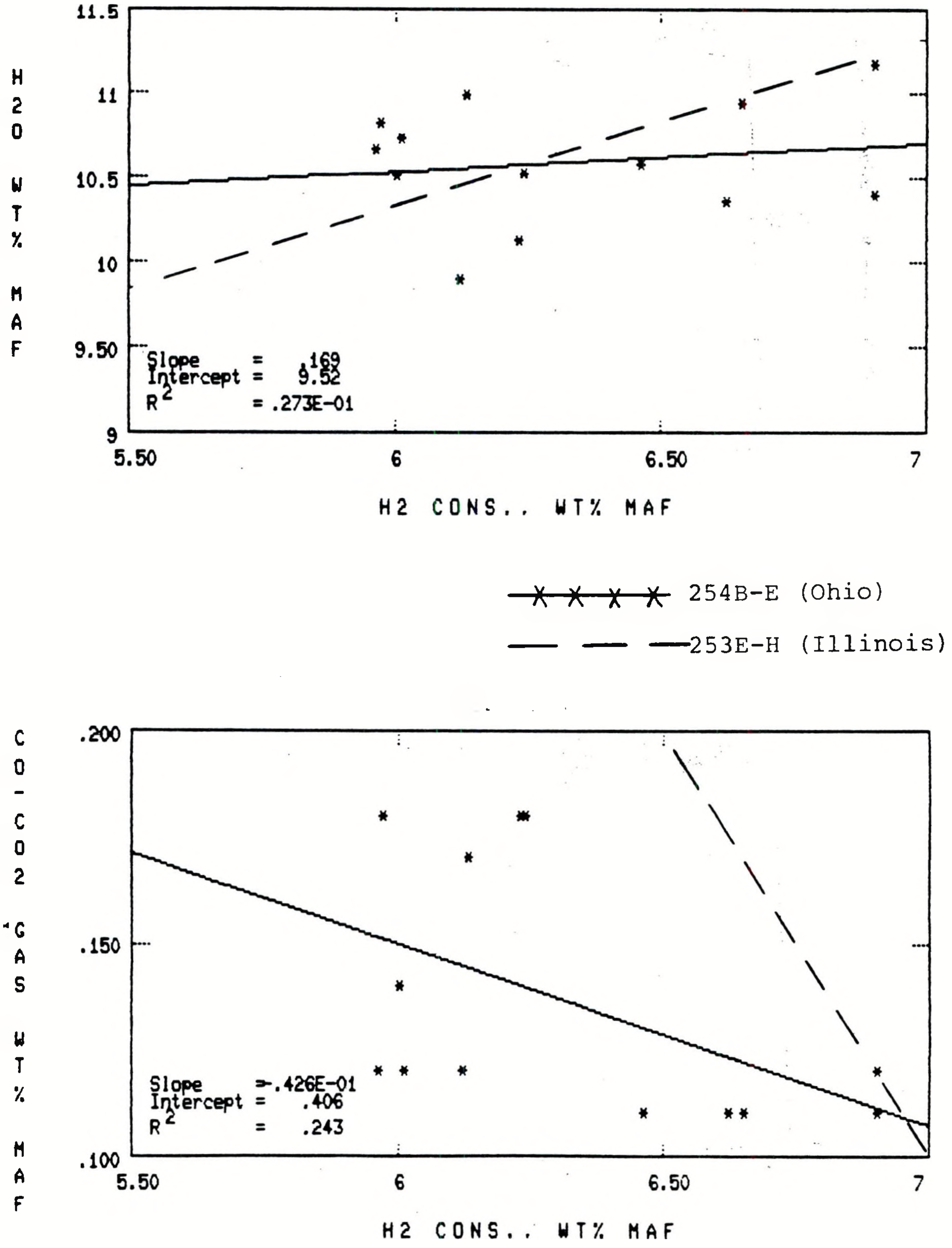
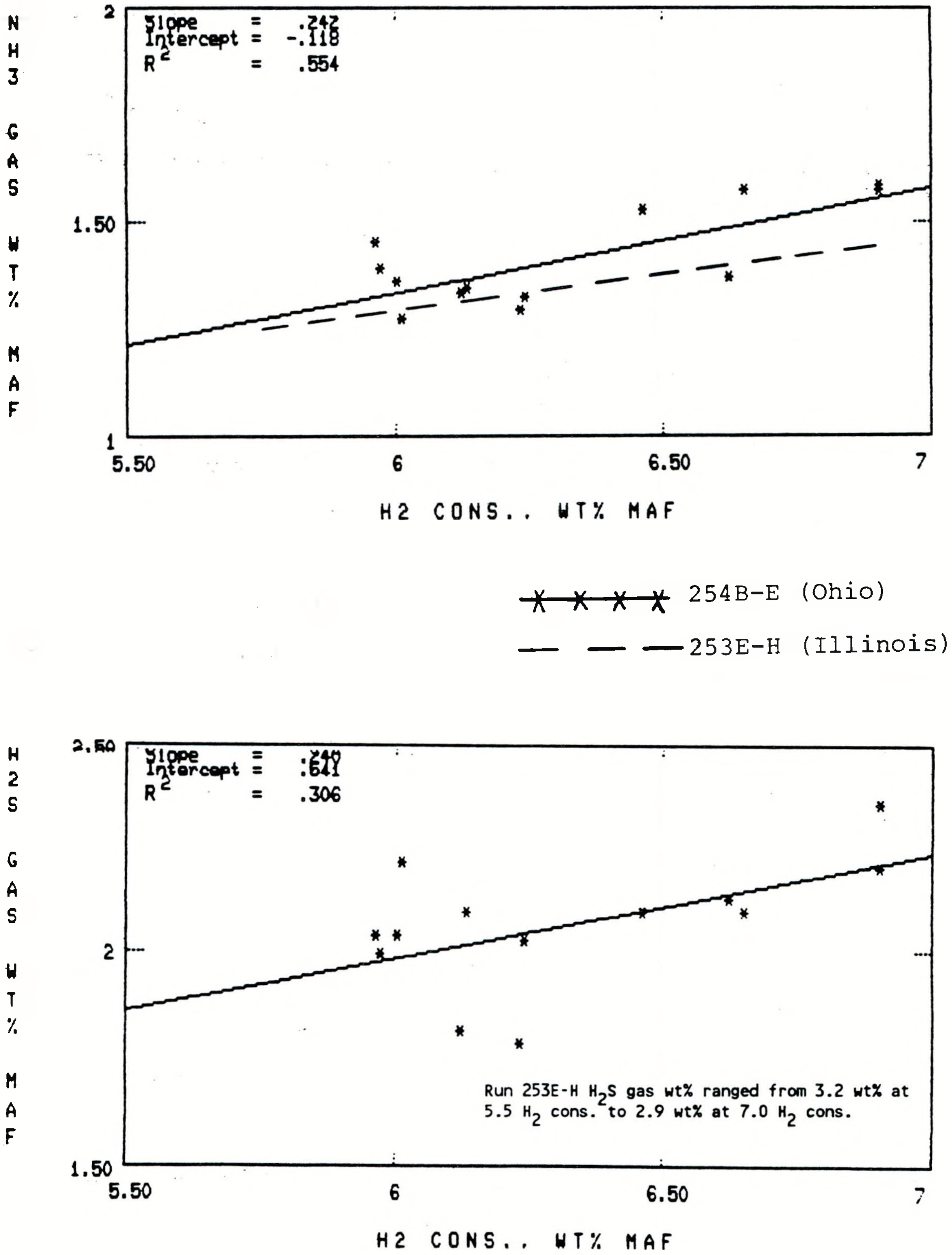


FIGURE 26. (continued)



Run 253E-H H_2S gas wt% ranged from 3.2 wt% at 5.5 H_2 cons. to 2.9 wt% at 7.0 H_2 cons.

FIGURE 27. PROCESS SEVERITY STUDY OF THE PROCESS SOLVENT CHANGES (HETEROATOM REMOVAL)

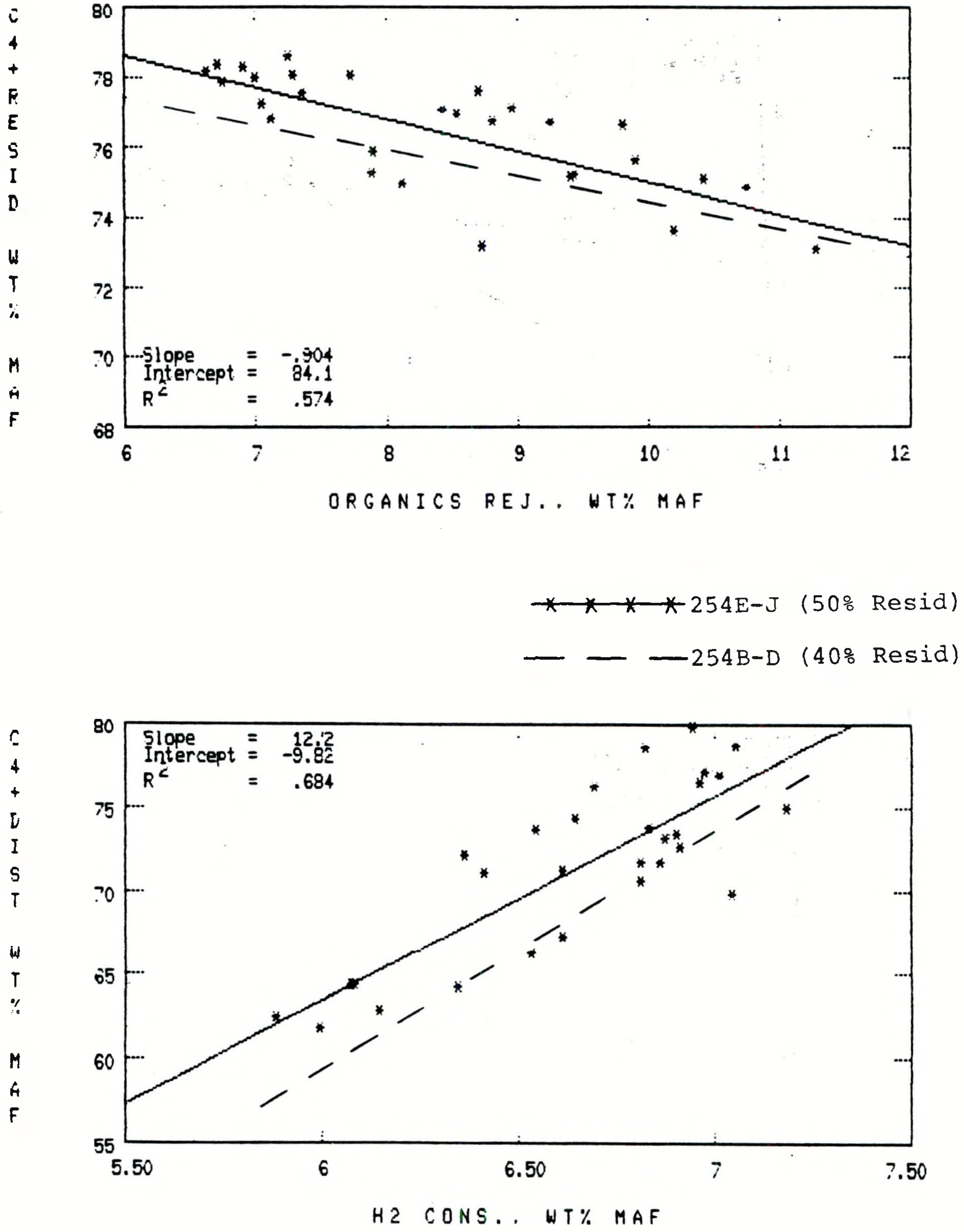


FIGURE 27. (continued)

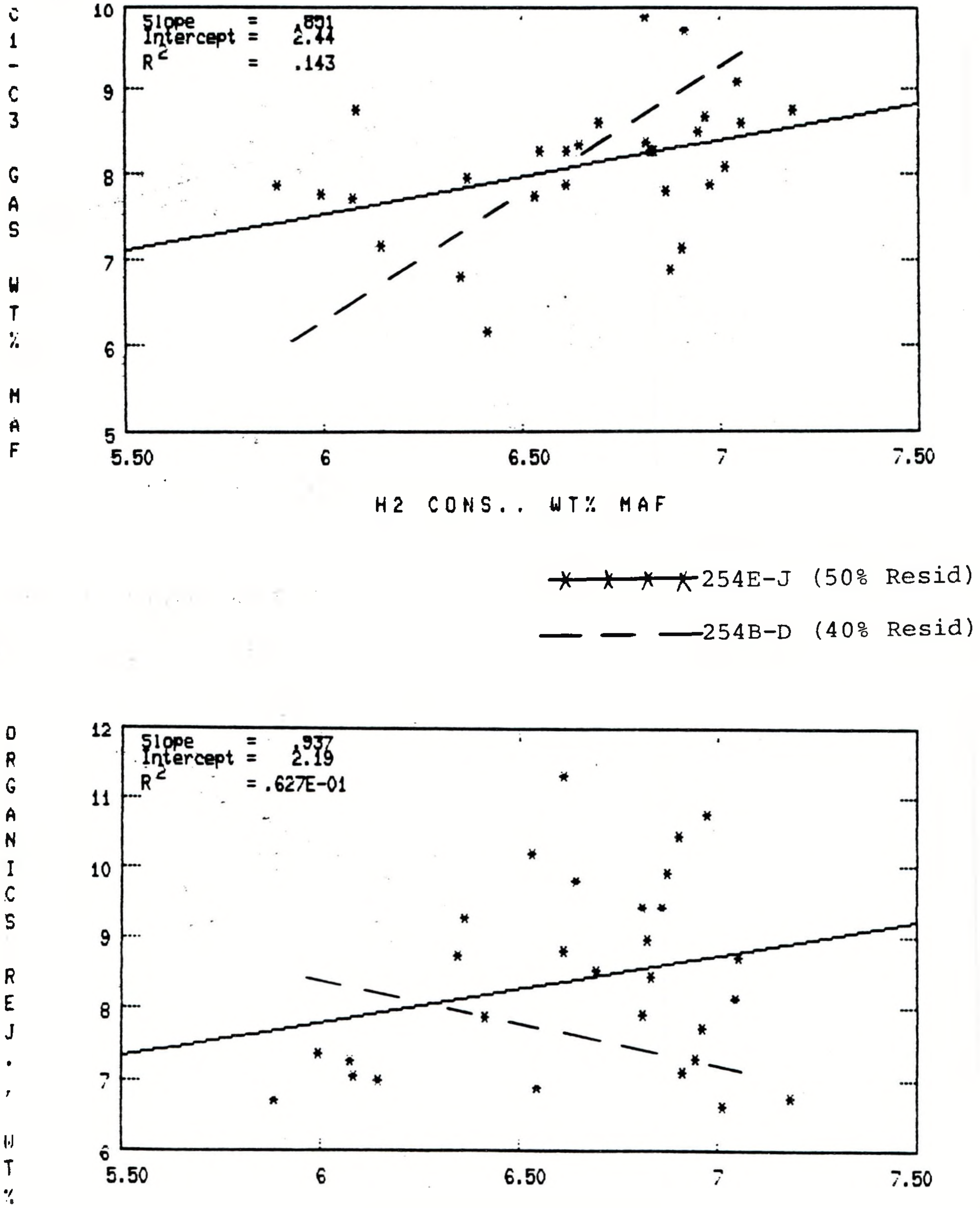


FIGURE 27. (continued)

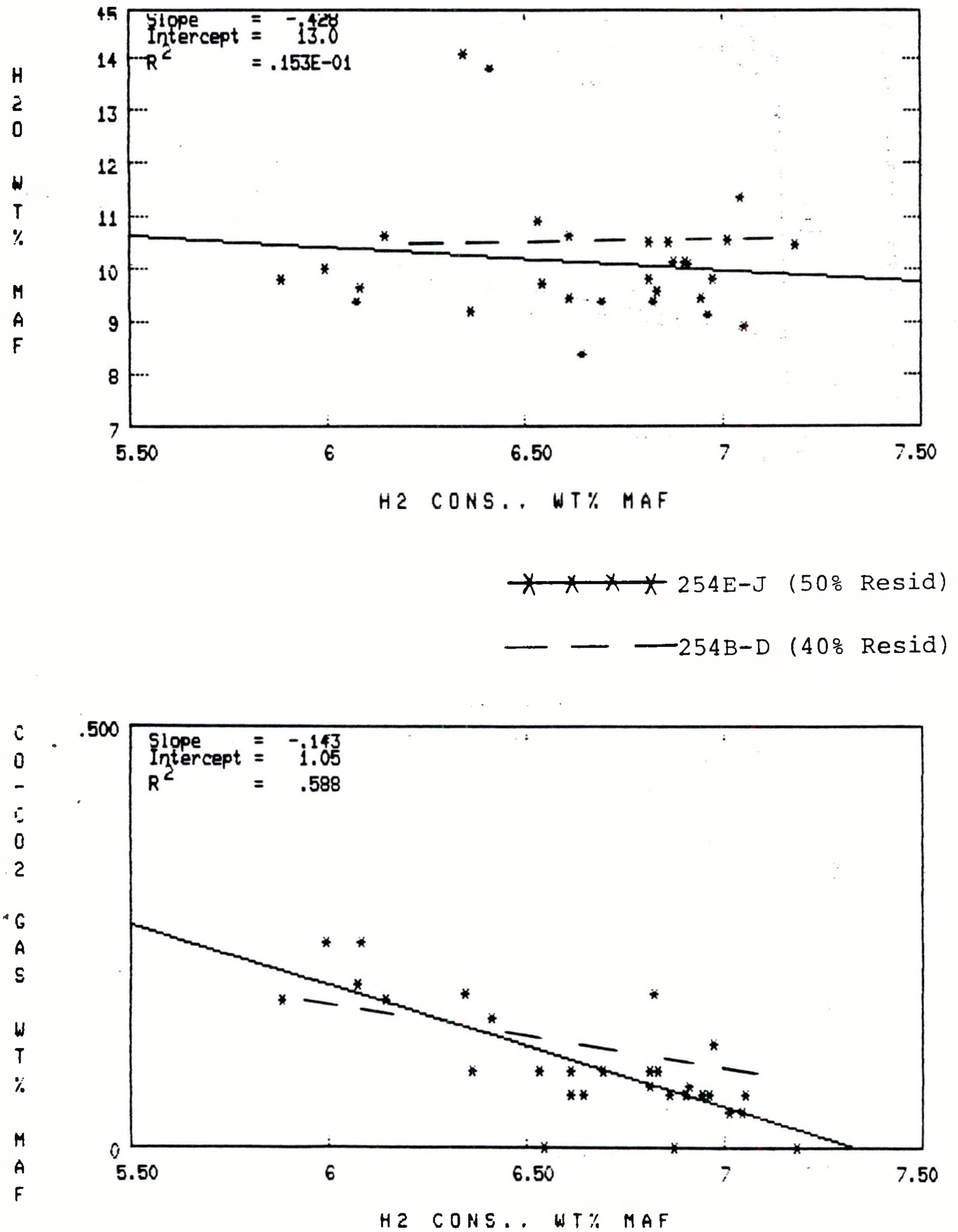


FIGURE 27. (continue)

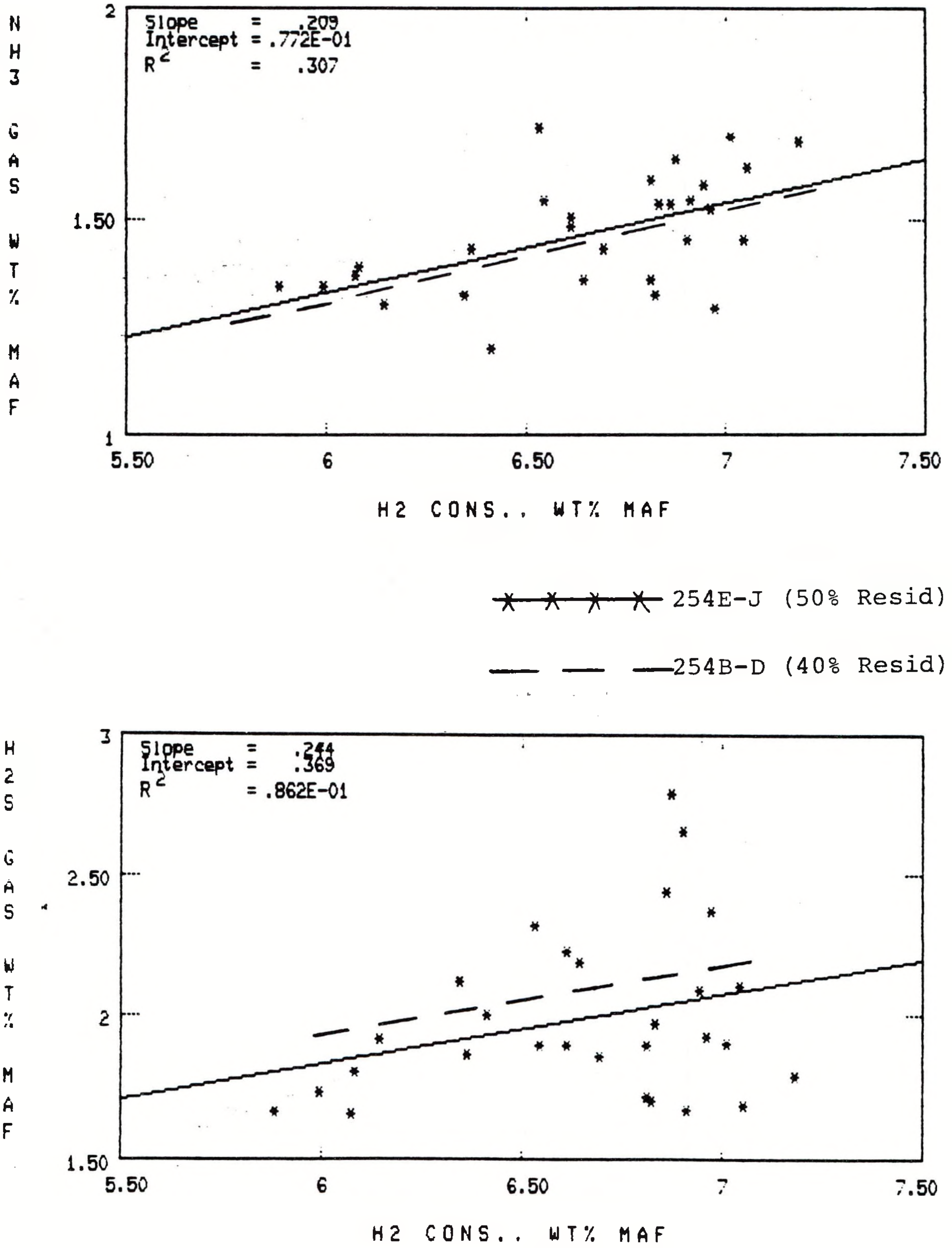


FIGURE 28. PROCESS SEVERITY STUDY OF THE SECOND STAGE TEMPERATURE CHANGES (HETEROATOM REMOVAL)

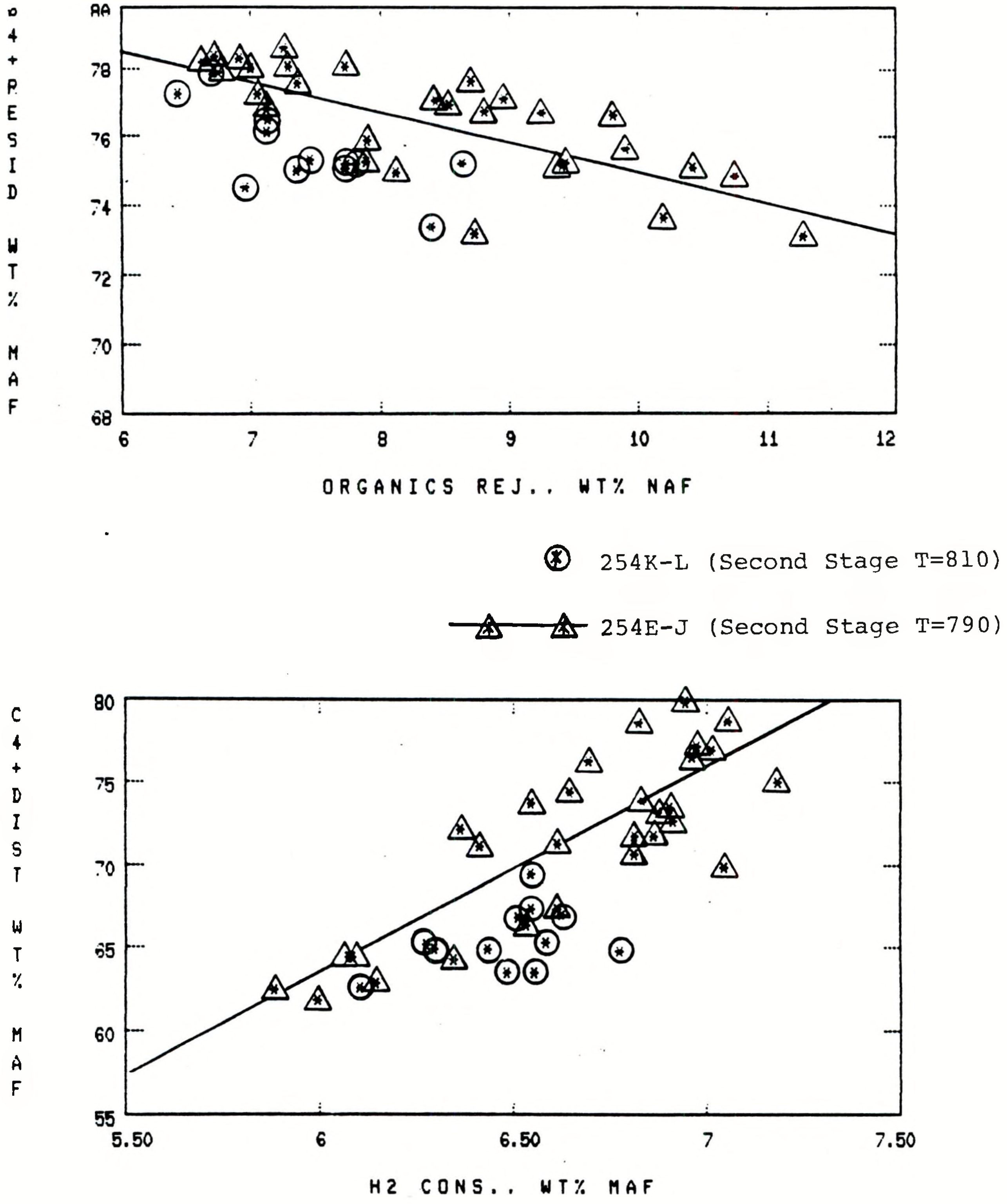


FIGURE 28. (continued)

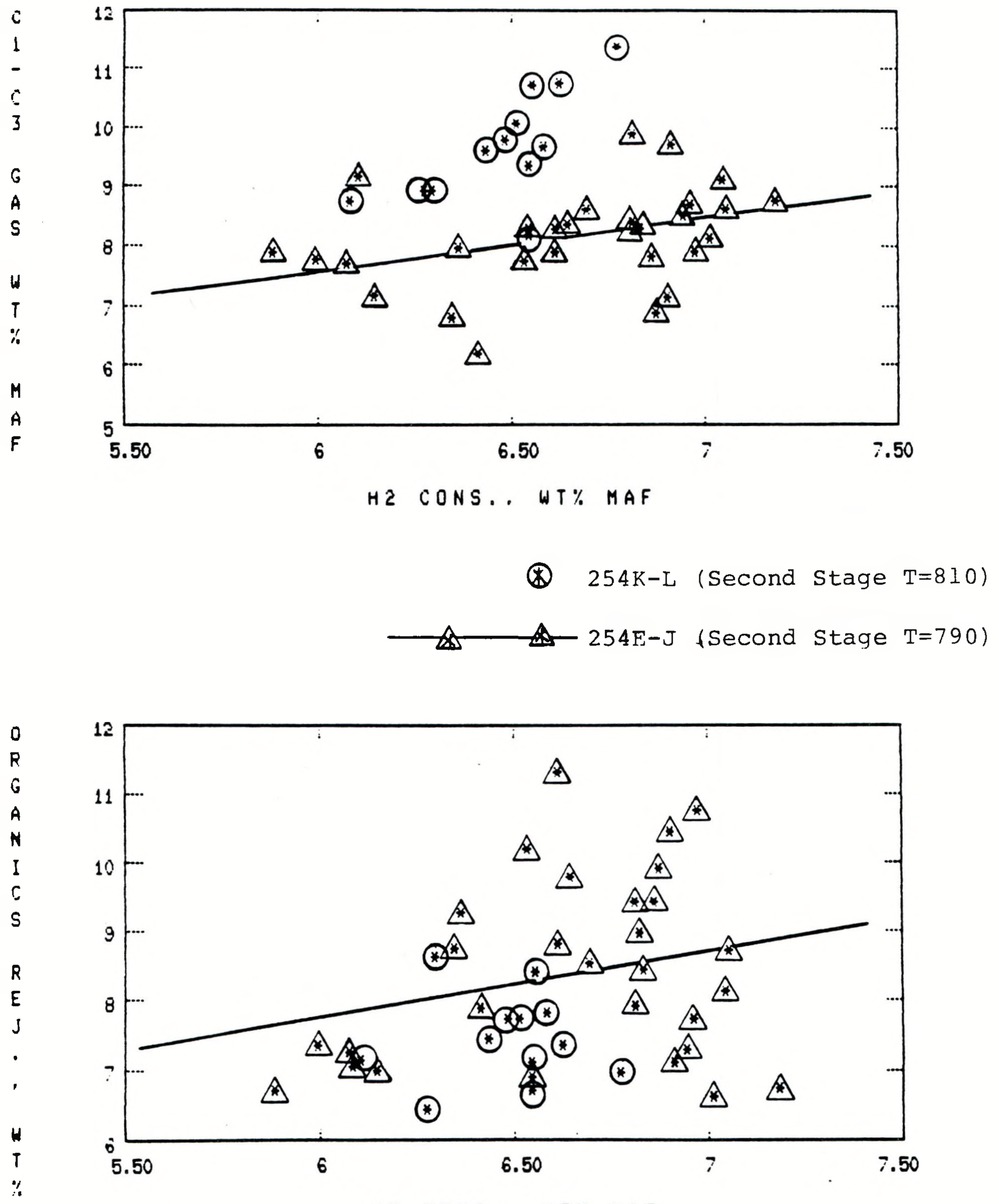


FIGURE 28. (continued)

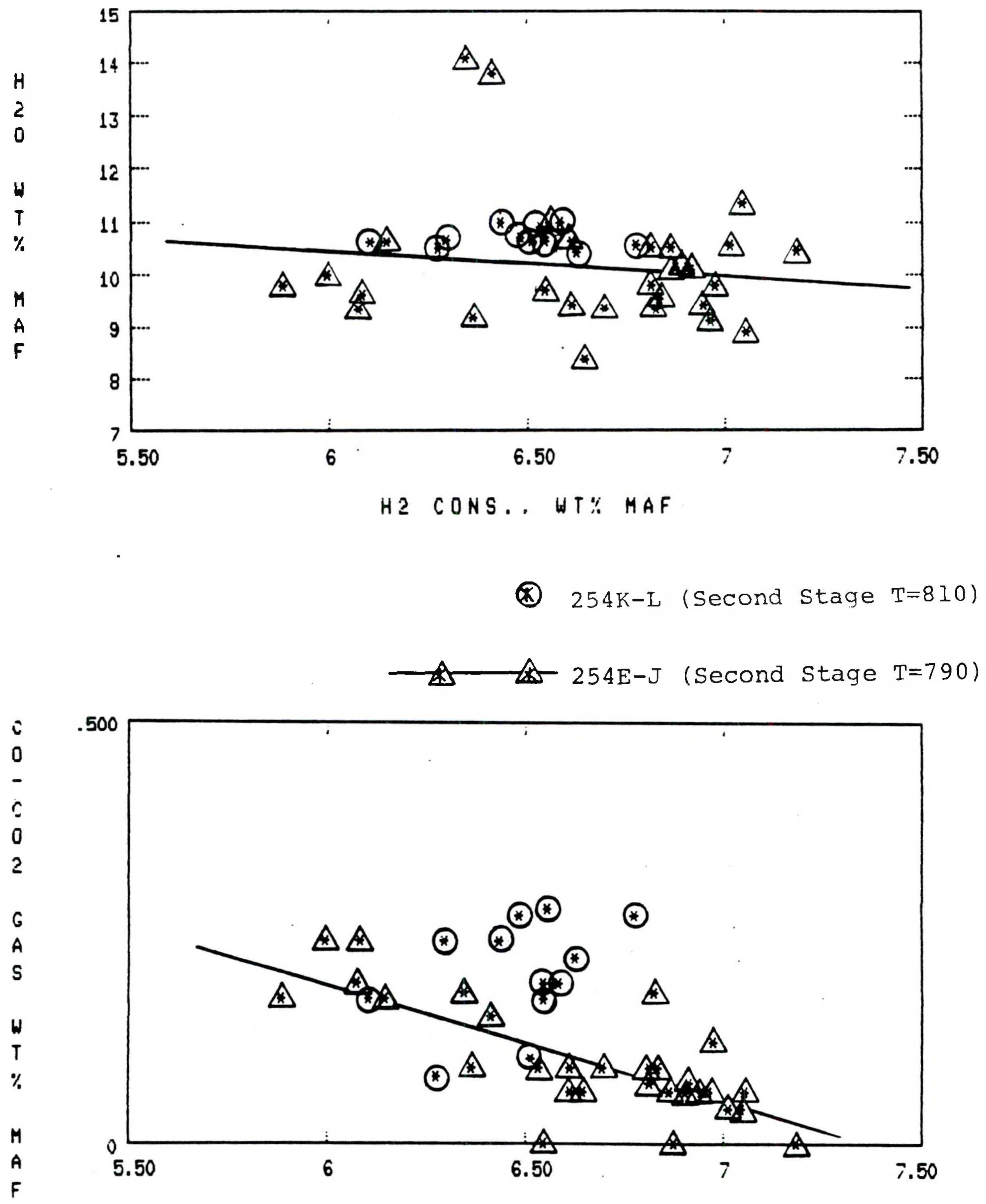


FIGURE 28. (continued)

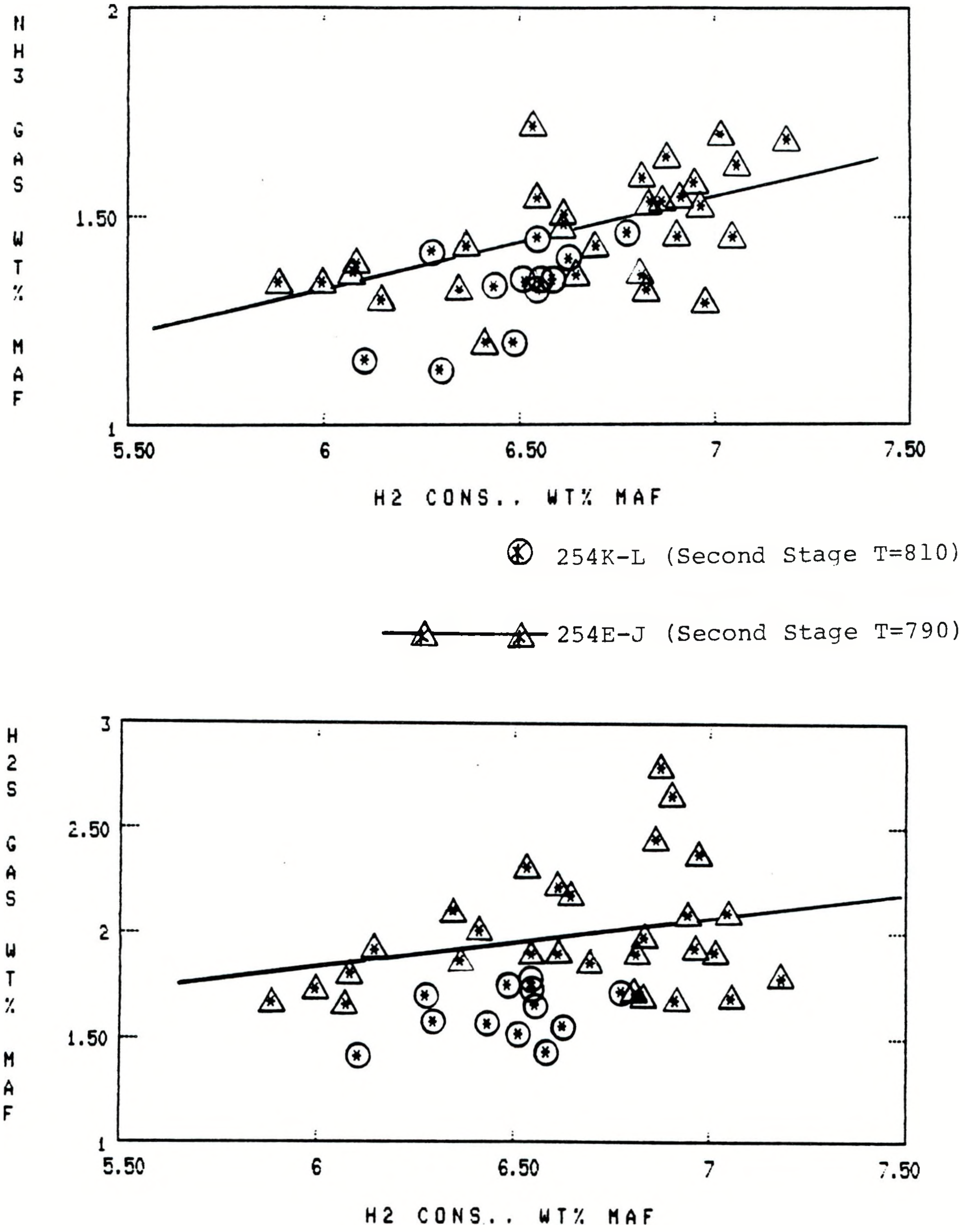


FIGURE 29. FIRST STAGE CATALYST CARBON BUILDUP

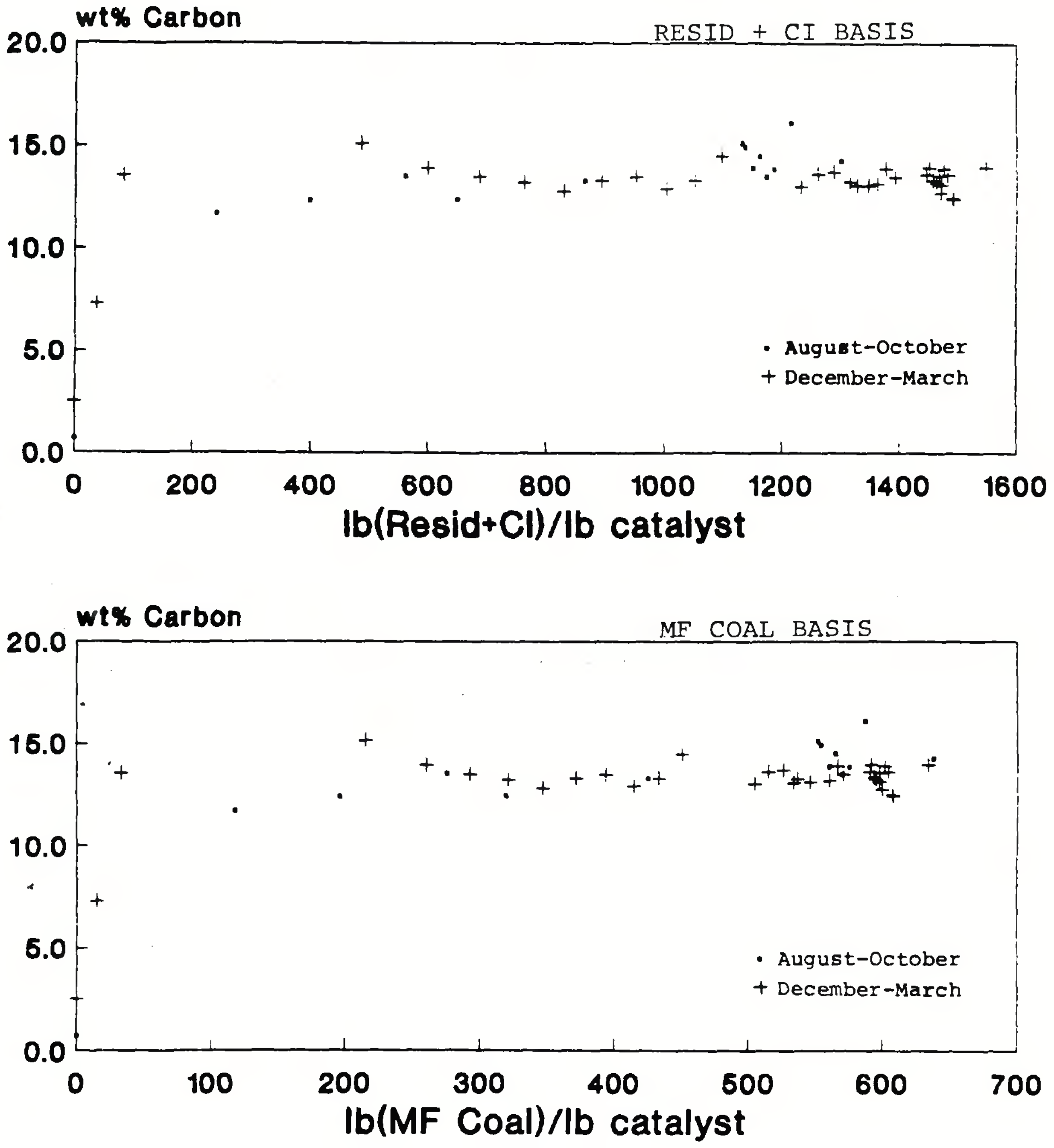
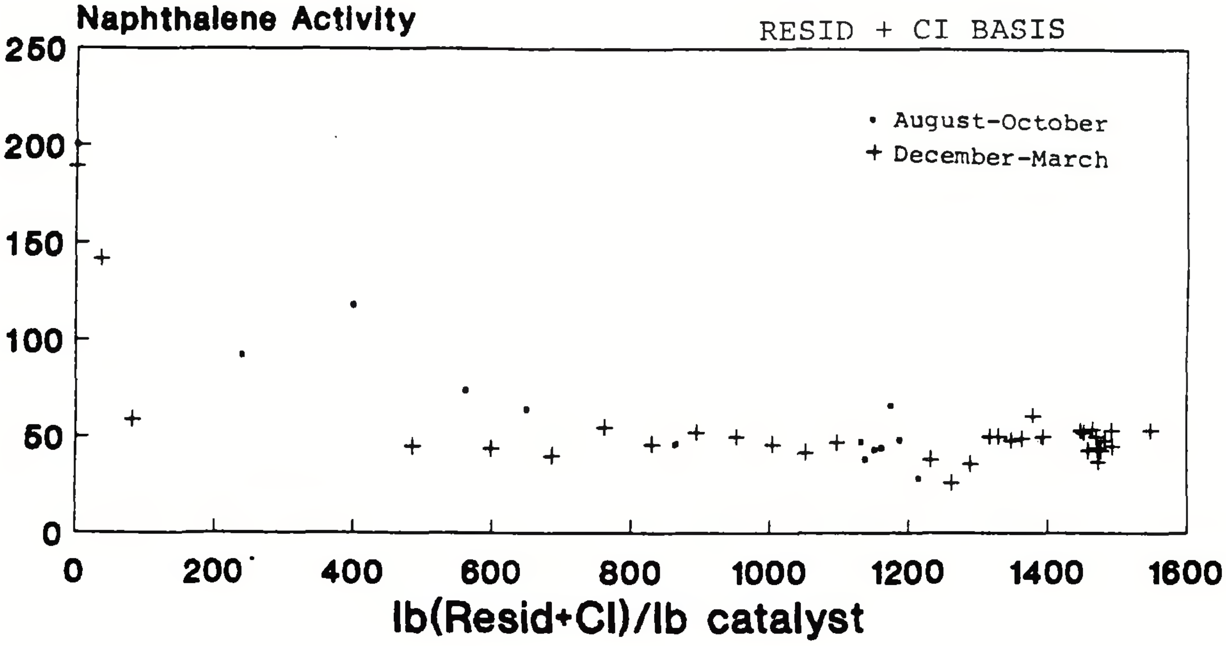


FIGURE 30. FIRST STAGE CATALYST NAPHTHALENE ACTIVITY



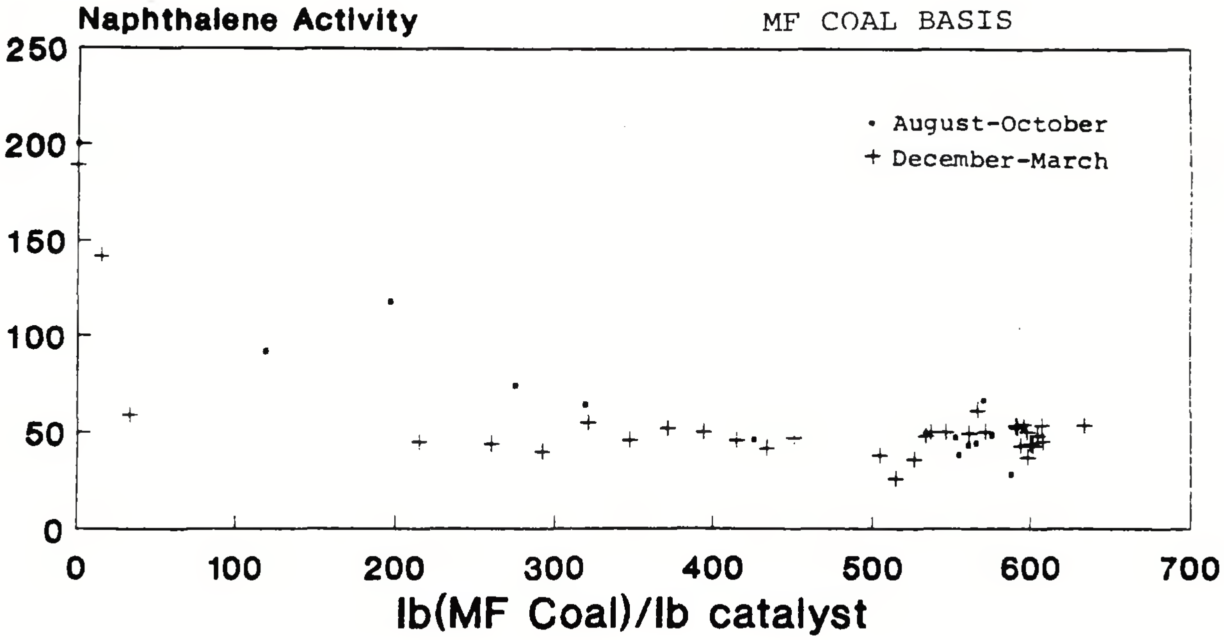


FIGURE 31. TSL RESID YIELD TRENDS FOR PERIODS 254E-L AS AN INDICATION OF TSL CATALYST ACTIVITY

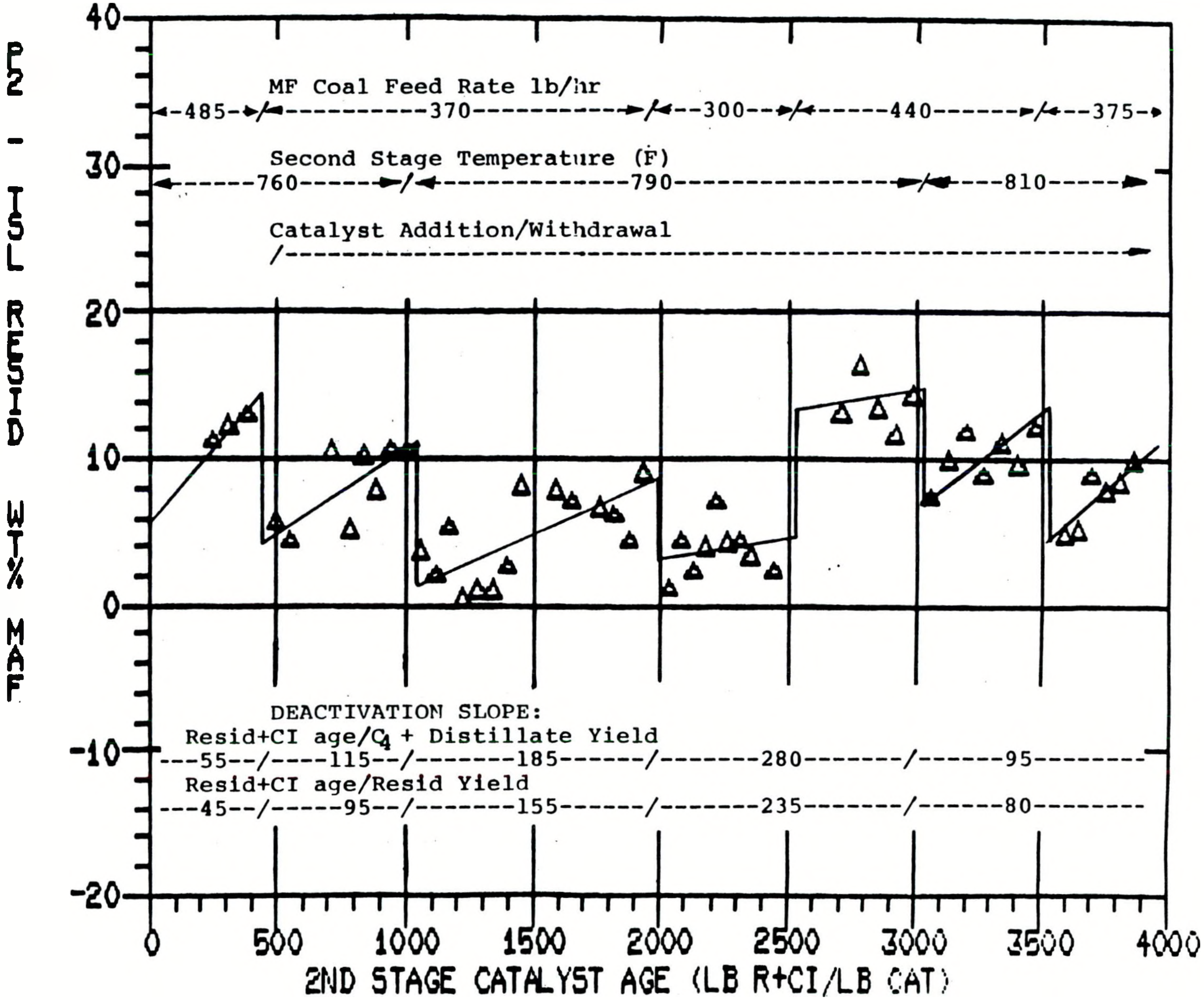
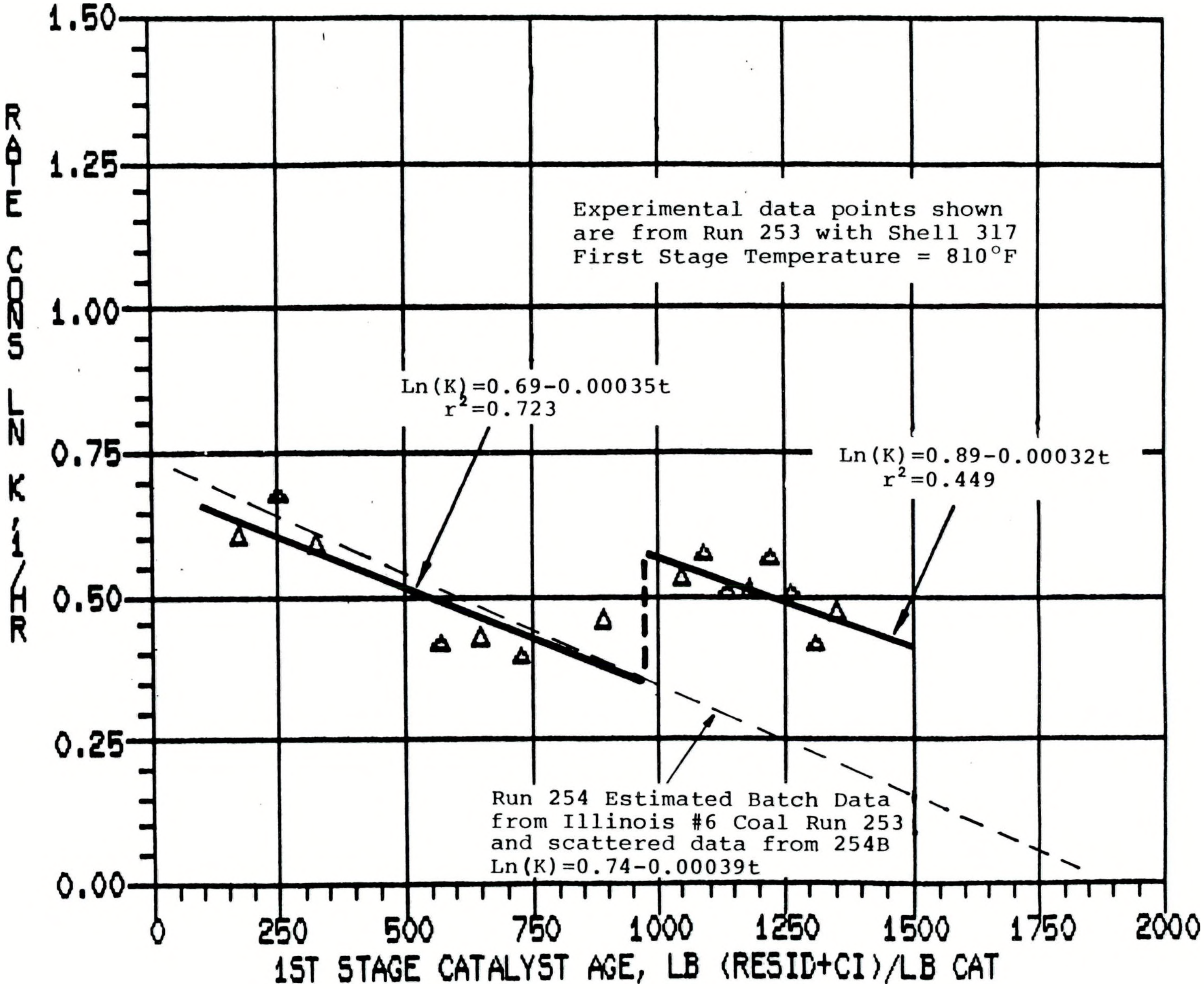


FIGURE 32. ESTIMATED BATCH DEACTIVATION CURVE FOR FIRST STAGE



Experimental data points shown are from Run 253 with Shell 317 First Stage Temperature = 810°F

$\mathrm{Ln}(K)=0.69-0.00035t$
$r^2=0.723$

$\mathrm{Ln}(K)=0.89-0.00032t$
$r^2=0.449$

Run 254 Estimated Batch Data from Illinois #6 Coal Run 253 and scattered data from 254B
$\mathrm{Ln}(K)=0.74-0.00039t$

FIGURE 33. FIRST STAGE CATALYST ACTIVITY EXPERIMENTAL DATA COMPARISON

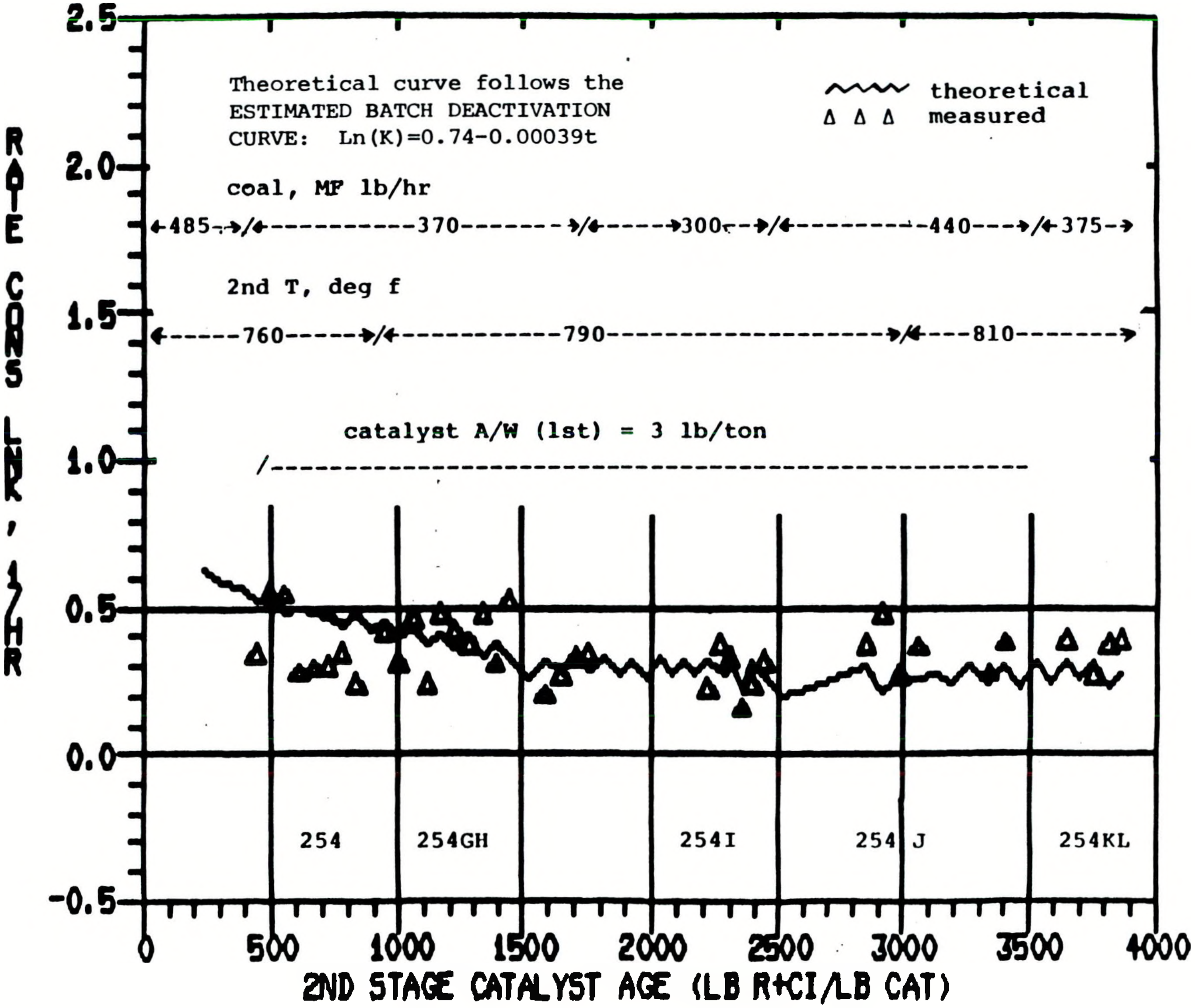


FIGURE 34. FIRST STAGE SHELL 317 CATALYST DEACTIVATION TRENDS FOR RUN 254

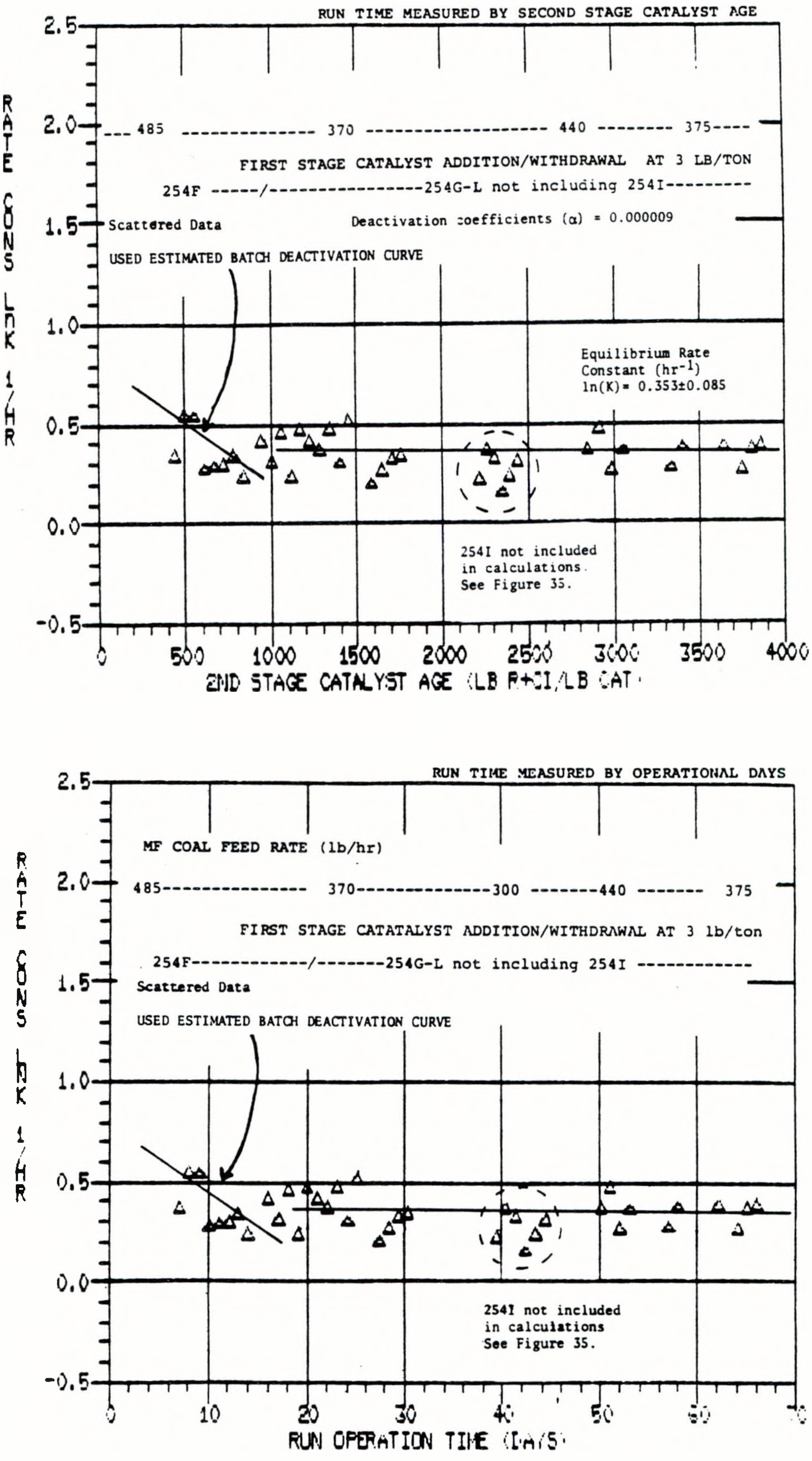


FIGURE 35. SECOND STAGE SHELL 317 CATALYST DEACTIVATION TRENDS FOR RUN 254

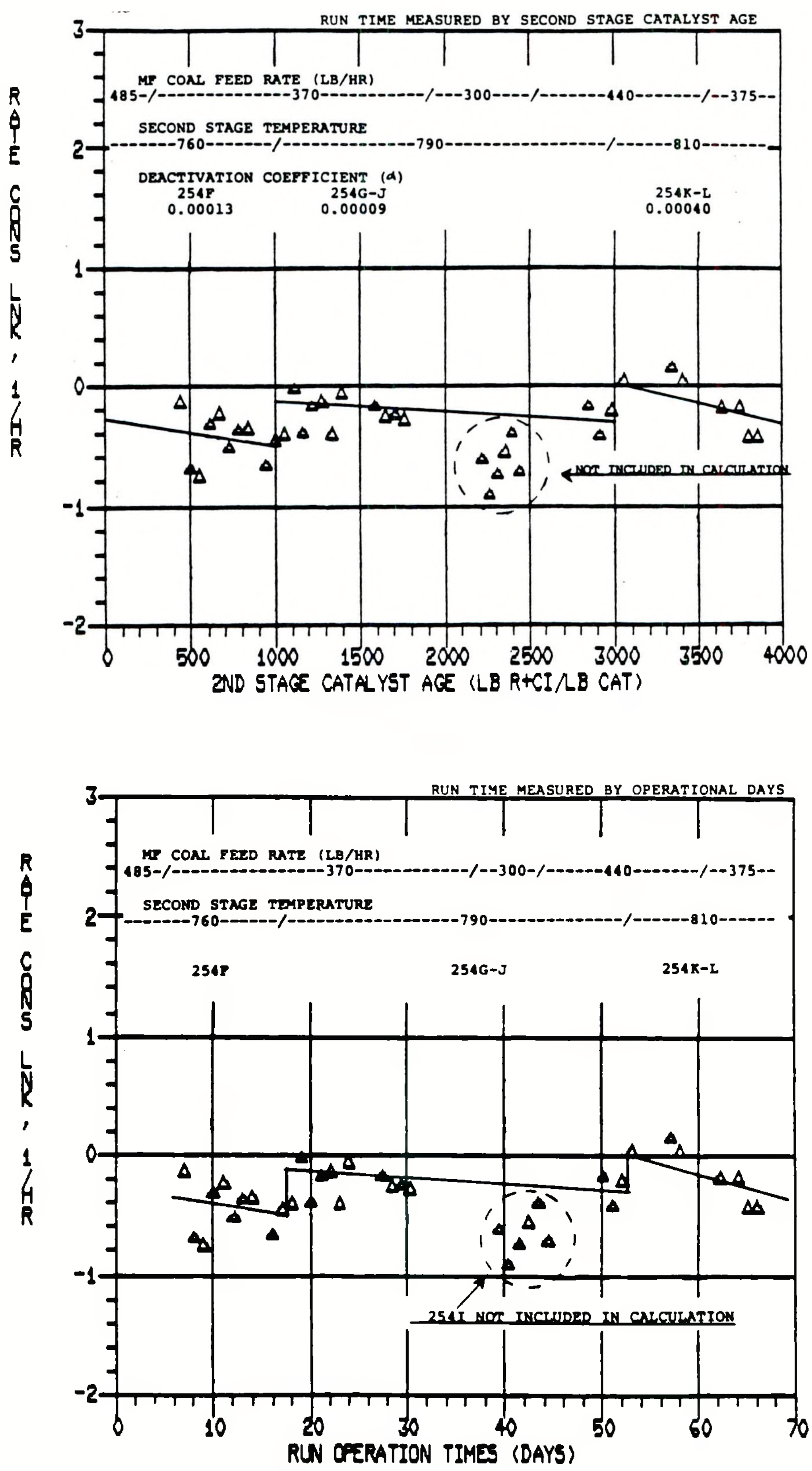


FIGURE 36. DEACTIVATION RATES AT DIFFERENT TEMPERATURES

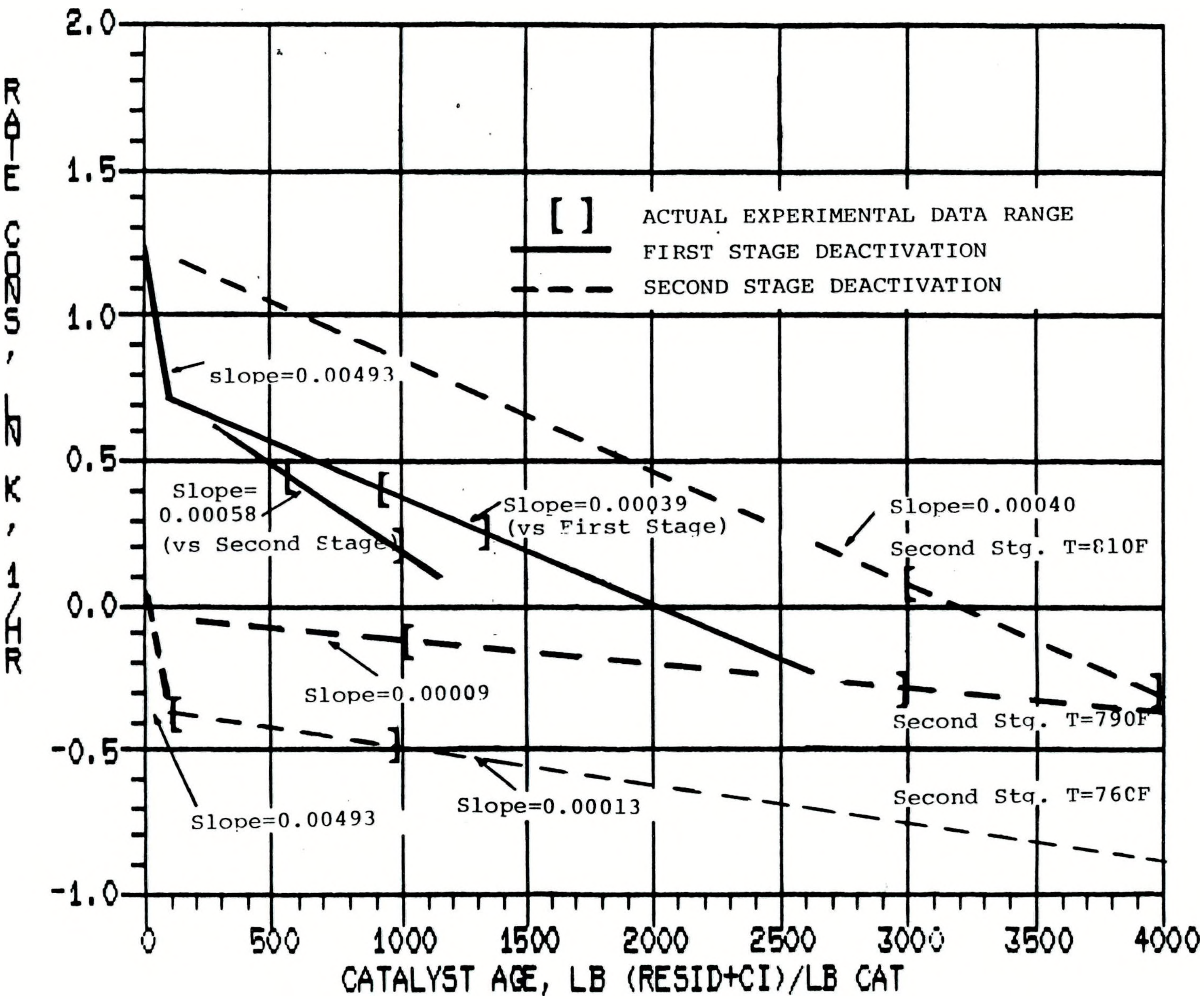


FIGURE 37. PARITY PLOT OF EXPERIMENTAL VS PREDICTED DISTILLATE YIELDS FOR RUN 254

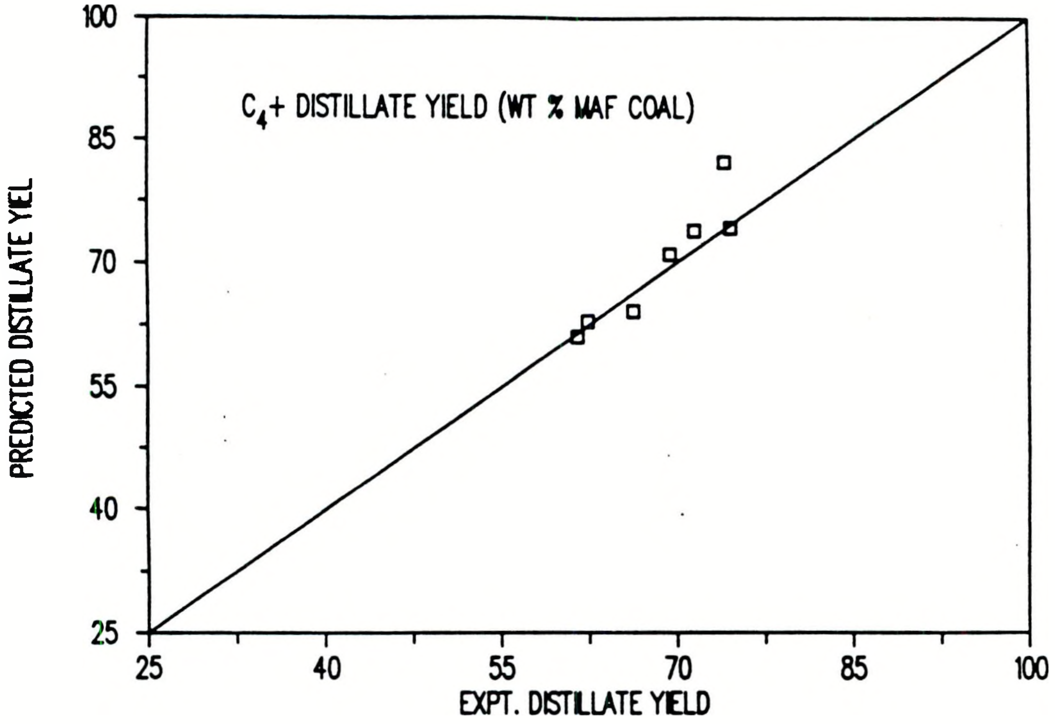
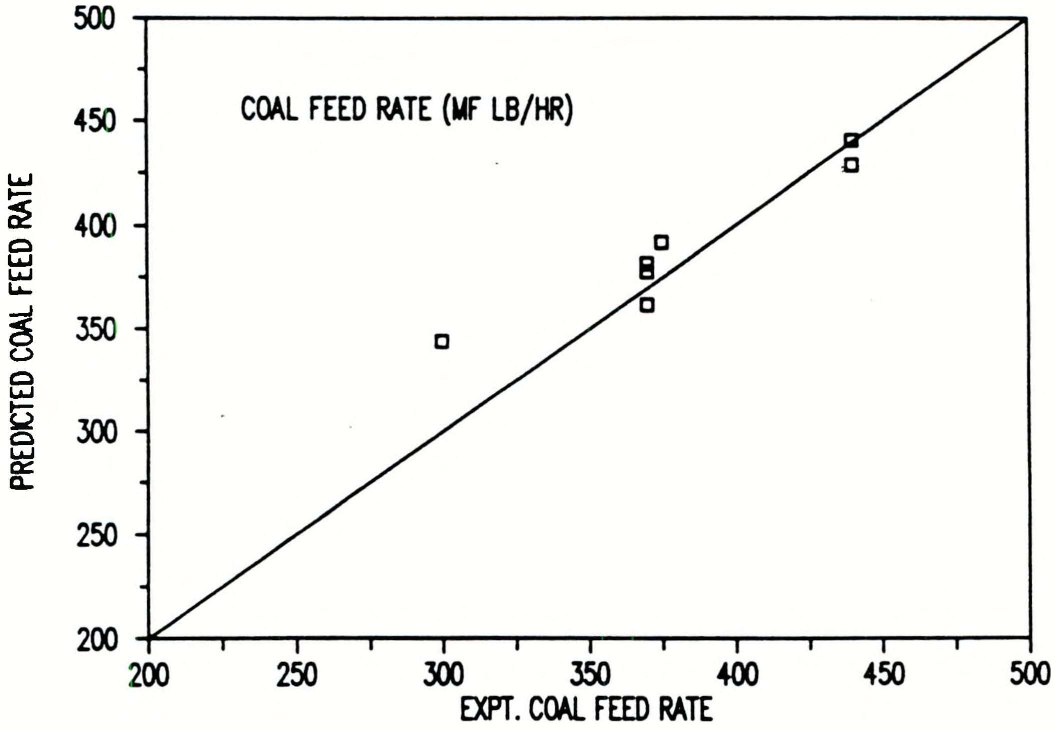


FIGURE 38. PARITY PLOT OF EXPERIMENTAL VS PREDICTED COAL FEED RATES FOR RUN 254



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