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DIRECT LIQUEFACTION PROOF-OF-CONCEPT PROGRAM
Hydrocarbon Technologies, Inc., Lawrenceville, N.J.

A.G. Comolli
L.K. (Theo) Lee
V.R. Pradhan
R.H. Stalzer
W.F. Karolkiewicz
R.M. Pablacio

FINAL

Topical Report
POC Run 02 (260-05)

Work Performed Under Contract No. AC22-92PC92148

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

By

Hydrocarbon Technologies Inc., Lawrenceville, NJ,

and

Kerr-McGee Corporation, Oklahoma City, Oklahoma

DECEMBER 1996

DISCLAIMER

DE-92148-TOP-02

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ABSTRACT

This report presents the results of work conducted under the DOE Proof-of-Concept Program in direct coal liquefaction at Hydrocarbon Technologies, Inc[1]. in Lawrenceville, New Jersey, from February 1994 through April 1995. The work includes modifications to HRI's existing 3 ton per day Process Development Unit (PDU) and completion of the second PDU run (POC Run 2) under the Program. The 45-day POC Run 2 demonstrated scale up of the Catalytic Two-Stage Liquefaction (CTSL Process) for a subbituminous Wyoming Black Thunder Mine coal to produce distillate liquid products at a rate of up to 4 barrels per ton of moisture-ash-free coal. The combined processing of organic hydrocarbon wastes, such as waste plastics and used tire rubber, with coal was also successfully demonstrated during the last nine days of operations of Run POC-02.

Prior to the first PDU run (POC-01) in this program, a major effort was made to modify the PDU to improve reliability and to provide the flexibility to operate in several alternative modes. The Kerr McGee Rose-SR[SM] unit from Wilsonville, Alabama, was redesigned and installed next to the U.S. Filter installation to allow a comparison of the two solids removal systems. The facility upgrade also included a new enclosed reactor tower, upgraded computer controls and a data acquisition system, an alternate power supply, a newly refurbished reactor, an in-line hydrotreater, an interstage sampling system, a coal handling unit, a new ebullating pump, load cells and improved controls and remodeled preheaters.

The 45-day CTSL Wyoming Black Thunder Mine coal demonstration run achieved several milestones in the effort to further reduce the cost of liquid fuels from coal. The primary objective of PDU Run POC-02 was to scale-up the CTSL extinction recycle process for subbituminous coal to produce a total distillate product using an in-line fixed-bed hydrotreater. Of major concern was whether calcium-carbon deposits would occur in the system as has happened in other low rank coal conversion processes. An additional objective of major importance was to study the co-liquefaction of plastics with coal and waste tire rubber with coal. This was a direct scale-up from microautoclaves and one liter reactors to a 3 ton per day unit. In the first 36 days of coal liquefaction operations, typical coal conversions of 92-94 W% were obtained with 84-86 W% resid conversions and as high as 66 W% light

[1]Hydrocarbon Technologies, Inc. is the successor to Hydrocarbon Research, Inc.

distillate yields (all maf basis). The distillate products from the in-line fixed bed hydrotreater were very clean, containing as low as 20 ppm nitrogen and 5 ppm sulfur, with high H/C atomic ratio (1.78). The effects of feed space velocity, reaction severity, addition of a dispersed catalyst (molybdenum), and recycle stream composition on process performance were studied. In the final nine days of POC-02, pulverized waste tires and mixed plastics were processed with coal at 25 W% tire rubber with coal and 30 W% mixed plastics with coal. A total of about 15 tons of coal, tire rubber, and plastics were converted to light distillates containing less than 10 ppm sulfur and 25 ppm nitrogen, and with a cetane index of over 40.

This was the first successful demonstration of Catalytic Two Stage Liquefaction technology at the 3 ton per day scale for a subbituminous coal and featured many improvements over the earlier testing conducted at the Wilsonville, Alabama, Pilot Plant. Distillate yields of 4.3 barrels per ton of moisture ash free coal (about 66 wt% on MAF coal) were achieved. Solvent recycle rates were reduced from the 2-2.5 to 1 ratio demonstrated at Wilsonville to as low as 1.2 to 1 during the recent test. This greatly improves process economics. Coal feed rates were increased during the test by 50 - 100 % while maintaining process performance at a marginally higher reactor severity. This offers the potential for further reduction of commercial plant investment per unit of coal feed. More than 3,200 gallons of hydrotreated distillate fuels were collected for end use evaluation and upgrading studies by DOE and their contractors. The ROSE-SR^{SM} Process was operated successfully using a pentane solvent in a steady-state mode. The energy rejection of the ash concentrate was consistently below prior data, being as low as 13%, allowing improved liquid yields and recovery.

EXECUTIVE SUMMARY

This report is prepared under a multi-year Proof-of-Concept direct coal liquefaction program funded by the U.S. Department of Energy's Pittsburgh Energy Technology Center, Kerr-McGee Corporation, and Hydrocarbon Technologies, Inc. (HTI). The program is directed toward scaling up and demonstrating new liquefaction concepts that can potentially lower the cost of synthetic liquid fuels to less then $30 per barrel. The work reported herein includes modifications to the Proof-of-Concept facility at HTI's Lawrenceville, New Jersey, R&D Center and completion of a 45-day demonstration run with Wyoming Black Thunder Mine subbituminous coal in a Catalytic Two-Stage Liquefaction mode. Operations at the 3 ton of coal per day facility produced yields of over four barrels of clean distillate products per ton of maf coal. The high quality liquid products can be readily refined into gasoline and diesel fuel.

The 45 day demonstration run fed a subbituminous coal alone for the first 36 days of operation and the same coal with either 25% used tire rubber or with 30% mixed plastics during the last nine days of operation. In the conversion process, the first stage reactor operates at a lower temperature (385-415°C) to hydrogenate the coal and recycle oil, while the second stage operates at a higher temperature (425-440°C) to convert the coal and heavy oils to clean distillate products. The products can be utilized for gasoline, jet fuel, or diesel fuels, or as home heating oil or combustion turbine fuel. Unconverted coal and ash are separated from recycle oils and valuable products using solids separation techniques such as filtration or solvent extraction. Kerr-McGee's ROSE-SR^{SM} solids separation technology was also demonstrated during this Black Thunder Mine coal demonstration run.

The scale-up of the CTSL process in POC Run 02 with Wyoming Black Thunder Mine coal was the culmination of a ten year effort devoted to the development of this two stage ebullated-bed reactor system using a low-to-high temperature sequence. POC-02, the second PDU Run of this program, was completed on July 28,1994, after 45 days of on-stream coal liquefaction and coal/waste co-liquefaction operations. The effects of feed space velocity, reaction severity, addition of a dispersed catalyst (molybdenum), recycle stream composition, and the combined processing of plastics and rubber with coal were studied.

The general objectives of PDU Run POC-02 were:

- To demonstrate the scale-up of the CTSL Process with a Wyoming sub-bituminous coal

- To demonstrate in-line hydrotreating to provide clean distillate liquid products which need minimal additional refining

- To demonstrate the CTSL Process with integrated solids separation

- To study the combined processing of mixed plastics and used tire rubber with coal in the CTSL Process mode.

The major accomplishments from POC Run No. 2 were:

- Successfully demonstrated the catalytic two-stage liquefaction of Wyoming subbituminous coal in a recycle extinction mode yielding a total distillate product

- Processed 350 tons of Black Thunder Coal over 36 days of solvent-balanced operation, with no evidence of calcium-carbon deposition in the reaction and separation train

- Demonstrated the reliability of low/high temperature staging, operating at a low solvent ratio of 1-1.2 without the use of external (make-up) solvent

- Attained high coal conversion of 91-93% and distillate yield of up to 4 barrels/ton of maf coal at a space velocity of 320 $kg/m^3/h$. Increasing the coal throughput to 600 $kg/m^3/h$ only reduced the distillate yield to about 3.5 barrels/ton.

- Operated the ROSE-SR^{SM} unit with mixed solvent and achieved organic rejections as low as 13 W% MAF coal.

- Demonstrated the concept of combined processing of coal and waste hydrocarbons; processed about 9 tons of waste plastics (a mixture of polystyrene, and polyethylene terephthalate) and waste tire rubber.

- Successfully operated an in-line hydrotreater to produce premium distillate (IBP-343°C) containing 25-50ppm nitrogen and 1-10ppm sulfur.

- Produced large quantities (3200 gal) of distillate product for end-use and upgrading studies (DOE end-use Contract).

Conclusions

Conclusions from the run are:

- POC scale demonstration of the liquefaction of Wyoming Black Thunder Mine coal was successfully achieved.

- A clean, IBP-360°C (IBP-680°F) distillate (sulfur content of 1-10 ppm and nitrogen content of 20-40 ppm) can be produced with in-line hydrotreating.

- The CTSL Process with subbituminous coal is operable at slurry oil/coal ratios as low as 1.2 to 1.

- ROSE-SR^{SM} Process separation efficiency is highly dependent on the asphaltene content of the feed and on the solvent utilized.

- Within the limitations of the ROSE-SR^{SM} unit to recover resid, extinction recycle can be achieved.

- Liquefaction of Wyoming Black Thunder Mine coal resulted in lower total conversion levels and distillate yields than the Illinois No. 6 coal used in POC-01.

- Equilibrated Akzo AO-60 catalyst, used during POC-02 operations, was found to undergo some attrition probably as a result of increased water vapor concentration in the reactor with high oxygen-containing sub-bituminous coal; the extent of catalyst attrition increased during the last nine days of coal/waste coprocessing operations.

- ROSE-SR^{SM} unit efficiency is unaffected by whether the liquefaction recycle system is operated with or without ashy recycle.

- Analyses indicate that during the coal/waste coprocessing operations, some retrograde reactions were occurring in the ROSE-SR^{SM} unit as observed previously with higher boiling ROSE-SR^{SM} solvents.

- Addition of 150 ppm of a soluble molybdenum dispersed slurry catalyst improved total coal conversion by over a percent; the molybdenum from the soluble precursor deposited at a low level on the supported extrudate catalyst.

- Although process equilibration was never achieved during coal/plastics and coal/rubber coprocessing, resulting in the use of significant amounts of make-up oil (negative solvent-balance), coprocessing Periods resulted in higher light distillate yields, lower gas makes and reduced chemical hydrogen consumptions.

Recommendations

- CTSL type processing of low rank coals using either a combination of dispersed slurry and supported extrudate catalysts or dispersed slurry catalysts only should be studied to further improve overall liquefaction economics.

- Combined processing of organic hydrocarbon wastes with coal should be studied under conditions of a net positive recycle solvent balance, i.e., under steady-state conditions.

- The reliability of the catalyst addition system needs to be improved.

- Other unit operations that require redesign for improved operability are:

  Oil/Water Separation, External Separation, Let-down Valves, Slurry Heat Exchange, the U.S. Filter, the ROSE-SR^{SM} Bottoms Removal and Heat Exchange, Coal Feed System and the Interstage Sampling System.

- Operations feeding subbituminous coal with in-line hydrotreating and interstage products separation should test synthesis gas as the reducing gas in the first stage reactor.

SECTION I

INTRODUCTION

As a part of the National Energy Strategy an Advanced Research Strategic Thrust is identified as Advanced Research for Coal-Derived Liquid Fuels with a primary objective "To evaluate novel concepts and establish the technology base for producing high quality hydrocarbon-based transportation fuels from coal to cost in the range of $25-$30/barrel of Crude Oil Equivalent". The advanced research thrusts focus on achieving objectives that support adaption of new technology into commercial practice in 5-10 years with some application in the near term (up to 5 years) as well. The Proof-of-Concept Program is the initial scale-up for direct coal liquefaction that establishes the basis of design for commercialization and provides process economics. Under the Proof-of-Concept Program HTI was chosen to operate a two-stage Process Development Unit for a period of 3 years followed by two optional years.

The Department of Energy and Electric Power Research Institute (DOE & EPRI) operated a facility in Wilsonville, Alabama, for over 10 years processing coal in various modes with single and two-stage reactors using dispersed and supported catalysts. In 1992 the DOE decided to close the Wilsonville facility (6 tons/day) and chose the smaller (3 tons/day) HTI PDU facility, a less costly, more flexible system that could be operated part time. In September 1992 HTI was awarded a 3 year contract to modify and operate the existing 3 ton/day unit on a cost shared basis with Kerr-McGee as a participant. In April 1995, the contract was subsequently assigned to Hydrocarbon Technologies, Inc., an employee owned company that assumed the ownership of the PDU facility at Lawrenceville, NJ.

Research and development objectives include scale-up of advanced direct liquefaction technology involving two stage reactions, co-processing of crude oils with coal, studies of alternative processing modes, evaluation of materials and equipment, improving product quality and reducing product cost. By the use of strategic feedstocks, commercially available catalysts, prototype equipment and improved design techniques and materials of construction, efforts have been and will be focused on improving process economics. The PDU produces hydrocarbon distillates and by-products in sufficient quantity to allow various research activities, such as product fractionation, upgrading, engine testing, storage stability, small scale combustion testing, and refining into chemical feedstocks.

Modifications were made to the PDU to improve reliability and to provide flexibility for operation in several alternative modes. Included were upgraded computer controls for automation and an alternative power supply to provide additional back-up in case of power failure. The Kerr McGee ROSE-SR^{SM} unit from Wilsonville was modified to be a single-stage unit using a pentane solvent and installed next to the U.S. Filter system to allow for direct comparison of the two types solid separation systems. A new reactor, hydrotreater, interstage sample system, a coal handling system to receive pulverized coal, a new ebullating pump, and improved instrumentation were installed over a period of about one year. A major part of this installation was a new reactor tower, enclosing the high pressure, high temperature vessels, and upgraded preheaters.

The PDU is a totally integrated two-reactor coal and oil hydrogenation process development unit. It includes coal and oil handling systems, slurry mixing, high pressure pumping, preheating, reaction, product separation, atmospheric and vacuum fractionation, naphtha stabilization, bottoms separation, product storage, data acquisition, and computer control. The PDU has been used to develop and scale-up the H-Oil® Process, H-Coal Process, Coal/Oil Co-Processing and CTSL processes. For this operation the PDU was equipped to remove solids via the ROSE-SR^{SM} critical solvent process, vertical leaf pressure filtration, or vacuum distillation. The simplified schematic of the unit for POC-02 operations is depicted in Figure 1.1.

Phase I of the Proof-of-Concept Program, consisted of four PDU Runs preceded by equipment modifications. The first PDU Run in this Program was completed during 1993 in a 58 day operation demonstrating the CTSL Processing of a high volatile bituminous Illinois No. 6 coal from Crown II mine. This report documents the results from POC Run 2, a 45 day on-stream coal operation processing Wyoming Black Thunder Mine Subbituminous coal in the catalytic two-stage liquefaction mode (CTSL). A major objective was to operate with extinction recycle of the 370°C+ fraction using the ROSE-SR^{SM} process for solid separation. During the final nine days of operation, steel and fiber-free used rubber tire material and mixed plastics were coprocessed with coal under similar reaction conditions. Three thousand two hundred gallons of 60°C (140°F) to 349°C (660°F) equilibrium product that had passed through the in-line hydrotreater was collected for upgrading and end use studies. The typical process performance data is listed in Table 1.1. Results from this POC scale-up operations are reported and compared with prior bench scale and Wilsonville pilot plant data.

1. Program Objectives

The following are the objectives of the Proof-of-Concept Direct Coal Liquefaction Program.

Develop direct coal liquefaction and associated transitional technologies capable of producing premium liquid fuels, which are economically competitive with petroleum and can be produced in an environmentally acceptable manner.

Focus on continued development two-stage liquefaction by utilizing geographically strategic feedstocks, commercially feasible catalysts, and prototype equipment. Include testing of co-processing or alternative feedstocks and improved process configurations.

- Demonstrate operation of a two-stage catalytic ebullated-bed reactor system with bituminous and subbituminous coals (or lignite) using commercially available supported catalysts having good physical strength and activity for comparison with a slurry reactor system using dispersed catalysts and with prior bench scale and Wilsonville PDU results.

- Demonstrate variant liquefaction schemes, especially coal/oil co-processing, utilizing appropriate feedstocks (such as petroleum resids, waste plastics, used tire rubber, and waste oils) with the scope of development depending on preliminary technical and economic evaluations. Co-processing may enable early commercialization of coal liquefaction technology due to more favorable economics.

- Demonstrate satisfactory operation with alternative feedstocks. (Selection of another Illinois No. 6 coal and a lignite for pilot-scale tests is necessary, as Burning Star #2 coal and Martin Lake lignite that were used in the past may not be readily available in the future.)

- Focus on development of data to allow scale-up from PDU to a commercial size unit by establishing operating parameters, such as coal space velocity, bed exotherms, hydrogen gas rates/consumption, and reactor geometry/hydrodynamics.

- Prioritize process development for low-cost feedstocks based on distillate production rate and coal reactivity.

- Demonstrate suitable low-rank coal liquefaction conditions for obtaining low heteroatom and hydrocarbon gas yields and high coal conversions while eliminating potential solids deposition in process units/lines.

- Obtain high distillate yields having good quality under low-severity conditions.

- Demonstrate the economic viability of well dispersed, highly active catalyst (disposable as well as recoverable) for slurry reactor applications in two-stage liquefaction.

- Demonstrate optimum supported catalyst replacement rates under steady-state catalyst activity conditions. Elucidate catalyst pore structure effects on conversion and hydrogenation. Evaluate improved catalyst utilization concepts (e.g., regeneration, cascading).

- Produce premium products by in-line hydrotreating of distillate.

- Demonstrate improved hydrogen utilization in two-stage liquefaction by removing heteroatoms using pretreatment/preconversion methods (proven at bench-scale), especially for low-rank coals. $CO+H_2O$ is a possible candidate.

- Define and demonstrate two-stage liquefaction product properties (e.g., end-point) for economic upgrading and refining to make specification-grade products.

- Perform process development with strategically important high- and low-rank coals. When appropriate, select readily available low-ash coals that have good reactivity.

- Facilitate process development by studying the interaction between the first and second stages by developing appropriate sampling and analytical methods (e.g., evaluate conversions at preheater outlet, interstage, etc.).

- Demonstrate efficient and economic solids separation methods for different ranks of coal. Evaluate vacuum bottoms to determine the merits of schemes involving fluid or delayed coking.

- Study the merits of integrating advanced coal cleaning methods (e.g., agglomeration, acid washing/coal beneficiation, etc.) with two-stage liquefaction.

- Improve overall process operability by selecting and monitoring advanced equipment and instrumentation that have improved tolerance for material degradation while handling slurries containing fine particulates, heavy resids, and corrosive streams under high severity conditions.

2. Proof-of-Concept Run 2 Objectives

The following are the objectives of the second PDU Run, POC-02, under the Proof-of-Concept Program:

- **To Ascertain Equipment Operability.**

  New installations included another reactor rebuilt from a salvaged high-pressure vessel, an in-line hydrotreater, remodeled preheaters, a new coal handling and storage system, a redesigned and newly installed Kerr-McGee ROSE-SR^{SM} Unit, a repaired U.S. Filter System, an expanded computer control & data acquisition system, an on-line sampling system, a larger hot separator, rebuilt hydrogen compressors, new catalyst addition valves, and a new flare system.

- **To Provide a Tie Point with Wilsonville Data (Runs 263J and 262 E).**

  Wilsonville Runs 262 and 263 fed Wyoming Black Thunder Mine Coal to a two-stage, reactor system employing dispersed slurry catalysts, based on iron and molybdenum, in the first stage reactor and on supported catalyst in the second stage reactor(4,5). Condition 263 J gave the best performance in the run and was used as the basis of design for an economic study by Bechtel for DOE.

- **To test the ROSE-SR^{SM} process and filtration for solid/liquid separation.**

  A redesigned Kerr McGee ROSE-SR^{SM} Critical Solvent Deasher from Wilsonville was to be tested using pentane solvent. A U.S. vertical leaf pressure filter, similar to that in use at the British Coal LSE Pilot Plant, was also available for study.

- **To obtain data on the effects of coal throughput, reactor severity, dispersed catalyst addition, and combined processing of waste plastics and used tire rubber with coal.**

  It was planned to study the performance of Wyoming Black Thunder Mine coal in a CTSL process at different coal space velocities, reactor temperatures, and in the presence of an added 100 ppm of molybdenum slurry catalyst. In the final two run conditions, it was also planned to study the effects of coprocessing mixed waste plastics and used tire material with coal.

- **To Obtain Data on In-Line Hydrotreating.**

  Based on favorable bench-scale data, a hydrotreater was designed and installed on the PDU to refine the hot separator light hydrocarbon overhead stream. Due to problems in the design of this hydrotreater, the operation was not successful during the first PDU Run, POC-01. Thus, study of the modified design of the in-line hydrotreater became an important objective during PDU Run POC-02. It was desired to achieve IBP-360°C product qualities of less than 20 wppm sulfur, less than 40 wppm nitrogen, and with the cetane number of the middle distillate fraction approaching 40.

- **To Collect Products for Evaluation and for other DOE Programs.**

  The DOE sponsored an upgrading study by Bechtel and others to produce transportation fuels from the distillate products of a coal liquefaction facility. A goal of collecting 2500 gallons of naphtha and distillate produced from extinction recycle operation was set. Other samples of distillate, heavy ends and bottoms products were also scheduled for collection for other DOE Programs.

- **To Evaluate Materials of Construction.**

  The Oak Ridge National Laboratory and the Japanese New Energy Development Organization (NEDO) supplied coupons of various materials to study exposure to coal liquefaction environments. Coupons were placed in the reactors, separators and fractionators.

- **To Obtain Data for Commercial Design and Technical Assessment.**

  During the run and prior to shutdown, extensive data were collected including process yields, product qualities, stream properties, equipment performance, catalyst properties, solids-liquid separation performance, and effects of process operating conditions. These data form the basis for future commercial plant design and technical assessment.

TABLE 1.1 POC-02 Process Performance

Coal: Wyoming Black Thunder Mine (6.2 wt% Dry Ash)
Catalyst: Spent Akzo AO-60 1/16" NiMo Extrudates in both Reactors (From POC-01 Operation)

CONDITION (Periods)		2 (20-21)
Solids-Separation		ROSE-SR
Recycle Type		Ashy
Coal Space Velocity, kg/h/m^3 (Stage)		458
	Lb/hr/ft^3	28.6
K-1:	Temperature, °C (°F)	413 (775)
	Cat Replace. Rate, kg/kg Ton MF Coal	0.45
K-2:	Temperature, °C (°F)	444 (831)
	Cat Replace. Rate, kg/kg Ton MF Coal	0.90
Flow Rates		
Coal Feed, kg/h		102
Solvent/Coal Ratio, kg/kg		1.30
Material Balances		
Liquefaction Section Recovery, wt%		100.3
Overall Material Recovery, wt%		97.8
YIELDS, W% MAF COAL (Based on Liquefaction Section)		
H2S		0.47
NH3		0.94
H2O		19.08
CO2		0.32
CO		0.12
C1		5.23
C2		2.64
C3		1.56
C4-177 °C (C4-350 °F)		19.33
177-288 °C (350-550 °F)		23.57
288-343 °C (550-650 °F)		11.07
343-524 °C (650-975 °F)		7.85
524 °C+ (975 °F+)		8.70
Unconverted Coal		7.40
Hydrogen Consumption		8.26

TABLE 1.1 (cont'd)
POC-02 Process Performance

Coal:	Wyoming Black Thunder Mine (6.2 wt% Dry Ash)
Catalyst:	Spent Akzo AO-60 1/16" NiMo Extrudates in both Reactors (From POC-01 Operation)

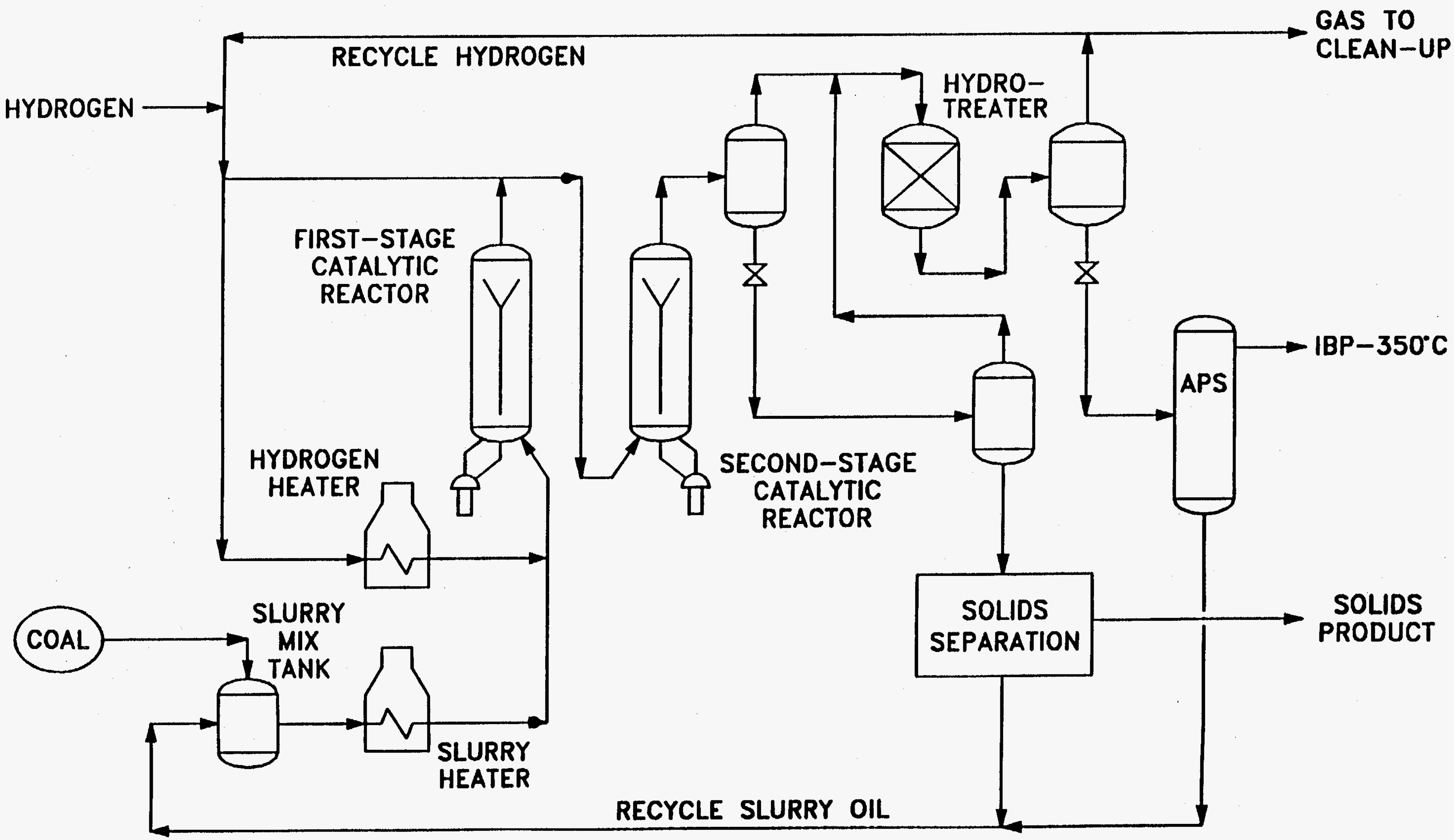
PROCESS PERFORMANCE, Wt% MAF COAL

Coal Conversion	92.6
524°C+ Conversion	83.9
Desulfurization (Organic), W%	96.5
Denitrogenation, W%	87.7
C_4-343°C Net Distillates	54.0
C_4-524°C Distillates	61.9
(Barrels/MAF Ton)	(4.2)
C_1-C_3 Selectivity, kg/kg of C_4-524°C (X 100)	15.2
H_2 Efficiency,kg C4-524°C/kg H_2	7.5

DEASHER PERFORMANCE

Organic Rejection, W% MAF	22.0
Energy Rejection, %	21.0
Deasher Coal Conversion, W% MAF	91.5

FIGURE 1.1
Simplified Flow Diagram of Catalytic Two-Stage Process



SECTION II

FEEDSTOCKS AND OPERATING SUMMARY

A. PROCESS DESCRIPTION OF THE PROOF-OF-CONCEPT RUN POC-02

The POC-02 Run in the Proof-of-Concept Program was carried out in HTI's Process Development Unit (Unit 260), which consists of two close-coupled ebullated bed reactors in series to convert coal and/or residual oil to high quality distillate fuels (*Figure 2.1*). The PDU is capable of operating at temperatures of up to 465°C (865°F) and pressures up to 20.7 MPa (3000 psig). Feed capacity can be as high 4 tonnes of coal per day. HTI's PDU is a totally integrated coal liquefaction/oil hydrogenation process development unit that includes coal and oil feed handling systems, slurry mixing (P-4), pumping (J-1), preheating sections (L-1 and L-2) besides two close-coupled ebullated bed reactors, product separators, atmospheric and vacuum fractionators, naphtha stabilizer, bottoms handling/recovery units, product storage, and on-line data acquisition (with computer control)/storage/reporting facilities. The HTI PDU facilities have been used in the past for the development of the H-Oil and the H-Coal Processes, for coal-oil coprocessing operations, and for jet fuel production from shale oil for the U.S. Air Force.

One of the main objectives of the PDU Run POC-02 was to demonstrate HTI's CTSL Technology with extinction recycle operations (recycle of all 400°C+ heavy oil) while processing a subbituminous Powder River Basin Coal and show the effectiveness of in-line hydrotreating. During Run POC-02, the unit was configured with ebullating pumps for both reactor stages, catalyst addition/withdrawal systems, and an in-line fixed bed hydrotreater. Three different solid separation options were utilized during this run to determine steady-state on-line operability and rejection of the organic material. The solid separations schemes used during POC-02 are discussed later as a part of the unit flow scheme.

Before startup, both reactors (K-1 and K-2) were charged with a predetermined amount of spent Akzo AO-60 catalyst extrudates, which were obtained as catalyst withdrawals during PDU Run POC-01. During continuous operations, the feed mixture, consisting of coal, recycle slurry oil, and hydrogen is pressurized and preheated prior to being introduced into the reactor. Recycle gas (about 80-85% hydrogen), pressurized and preheated, is also introduced into the reactor. This mixture then enters the first ebullated catalyst bed; the effluent from the first reactor stage flows directly to the second stage reactor. An on-line sample withdrawal system, installed on the ebullating line (internal recycle line) of the stage one reactor, can be activated to collect reactor slurry samples for characterizing the performance of the stage one reactor. Additional recycle gas is charged to the second stage reactor to control the inlet slurry temperature and to maintain an adequate hydrogen partial pressure. Second-stage reactor effluent, consisting of various liquid and gaseous products, unconverted feed and unreacted hydrogen, enters a hot separator (O-1). The operating conditions for the reactor stages and separators are addressed in later sections.

The overhead effluent from the hot separator, consisting primarily of unreacted hydrogen, gaseous reaction products (such as C_1-C_3, CO_x, H_2S, and H_2O), and light distillates, passes through an in-line hydrotreater (K-3) that employed fixed beds of Criterion 411 catalyst with an intermediate quenching zone. Hydrotreater effluent is mixed with water to prevent plugging due to (ammonium chloride/ammonium sulfide) salt formation and cooled in a heat exchanger (M-2) before entering another flash vessel (Cold Separator, O-5). This vessel separates the hydrogen and gaseous products from the condensed light distillates and water. The overhead gases are scrubbed with No. 2 fuel oil (N-1), and the resulting hydrogen-rich gas (mol. wt. between 3-5) is pressurized and recycled to the reactor. The liquid flash bottoms are depressurized and sent to an oil-water separator (O-45).

O-1 separator bottoms, (heavy slurry product) is depressurized and undergoes a flash separation in the reactor liquid flash vessel (O-13). The resultant bottoms product is partly sent to the recycle oil tank (O-43) in the ashy-recycle mode of operation. The rest of the bottoms slurry goes to a solids separation section which removes solids and recovers oil for recycle. In the ash-free recycle mode of operation, the entire O-13 bottoms go to the solids separation section for recovery of solids-free recycle oil.

The vapors from O-13 are cooled and flashed in vessel O-12, with the condensate re-introduced to the inlet of the hydrotreater for further processing. The feed stream to the atmospheric still is the cold separator O-5 bottoms (after passing through an oil-water separator, O-45). Atmospheric still bottoms are primarily sent back to recycle tank O-43 to generate enough slurrying oil for the desired solvent-to-coal ratio. Any atmospheric still bottoms produced in excess of that needed for recycle is stored as a net process product. The atmospheric still overheads pass through a stabilizer (N-5) which strips dissolved gasses, such as hydrogen sulfide and light hydrocarbons. Liquid from the stabilizer column is collected as naphtha product from the process. All noncondensables from the unit are flared.

During Run POC-02, the PDU had the flexibility to be operated in three different solids separation modes:

- Vacuum Distillation
- ROSE-SR^{SM}
- Filtration (vertical leaf U.S. Filter)

In general, as described earlier, O-13 flash vessel bottoms product is sent to a surge drum (O-46), from which the solids containing stream is sent to one of the three solids separation options.

Vacuum Still Mode (*Figure 2.2*): For the vacuum still option, a portion of the material from O-46 is sent through the recycle holding drum, O-41, and the recycle weigh drum, O-43, to the slurry mix tank, P-4. The remainder of the material from O-46 is sent to the vacuum still feed accumulator, O-50, and then to the vacuum still, N-3. The solids containing vacuum still bottoms stream goes to storage, while the vacuum still overhead material goes to the flush/purge oil storage drum, O-42, and any excess overheads go to the clean oil tank, P-3.

ROSE-SR^{SM} Mode (*Figure 2.3*): For this route of solid separation, the slurry product from O-13 bottoms is topped to remove 414°C- material prior to the ROSE-SR operation. The overheads from the vacuum still go to the flush/purge oil storage drum, O-42, and then to either purge oil day tank, O-40, or to the clean oil tank, P-3. The vacuum still bottoms stream goes to the ROSE-SR^{SM} section, where the solids concentrate stream is sent to storage after separation and the solids-free oil goes to the recycle oil receiver (deasphalted oil).

Filtration Mode (*Figure 2.4*): During this mode of solid separation, the material from O-46 bottoms is sent to the filter feed drum, O-47, and then to filter, XF-1. From the filter, the solids concentrated stream is sent to storage. The solids-free stream is sent through the filtrate receiver, O-48, and the vacuum still feed accumulator, O-50, to the vacuum still, N-3.

FIGURE 2.1
PDU-260 Simplified Flow Diagram

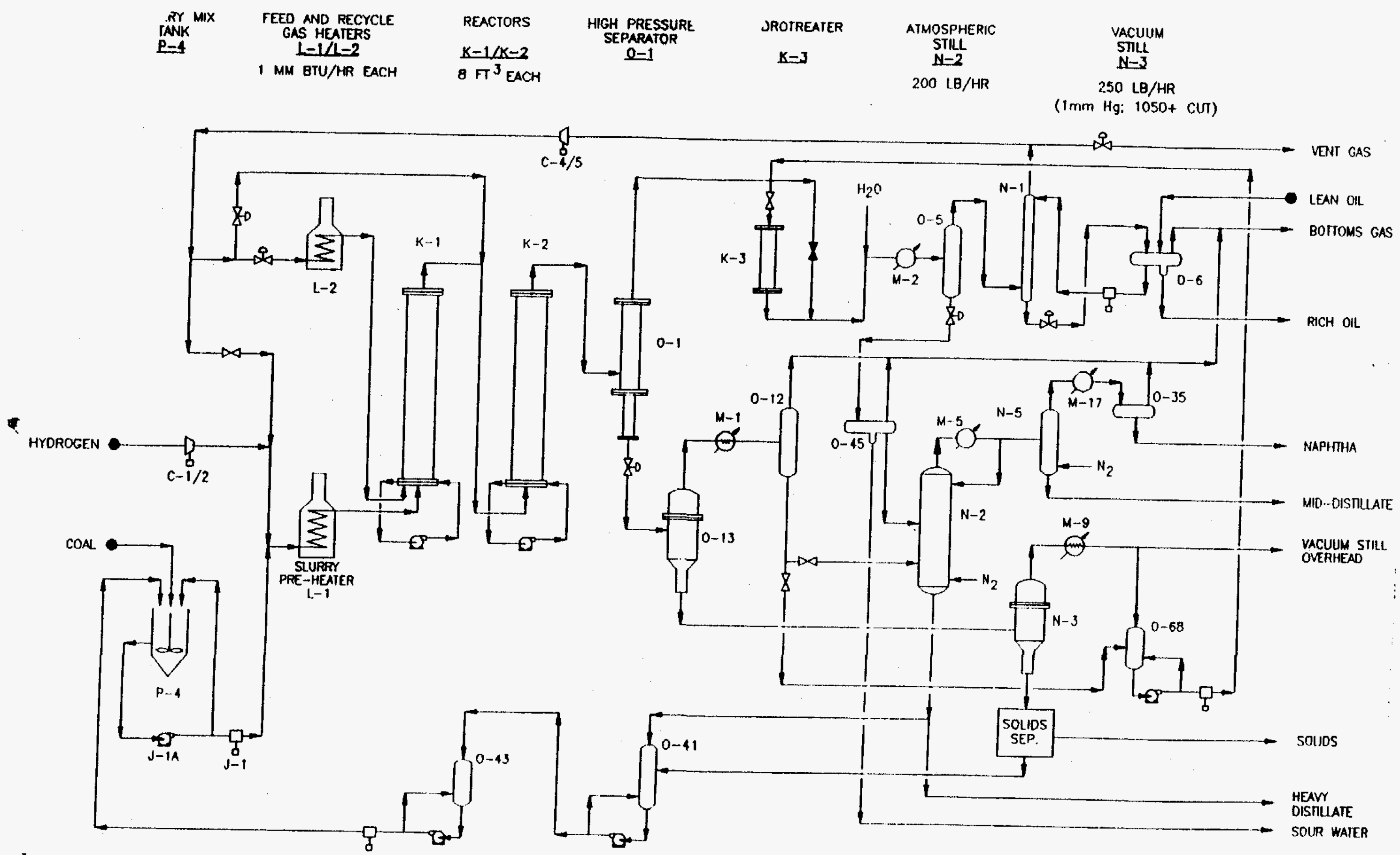


FIGURE 2.2
PDU-260 Simplified Flow Diagram: Vacuum Still Solids Removal

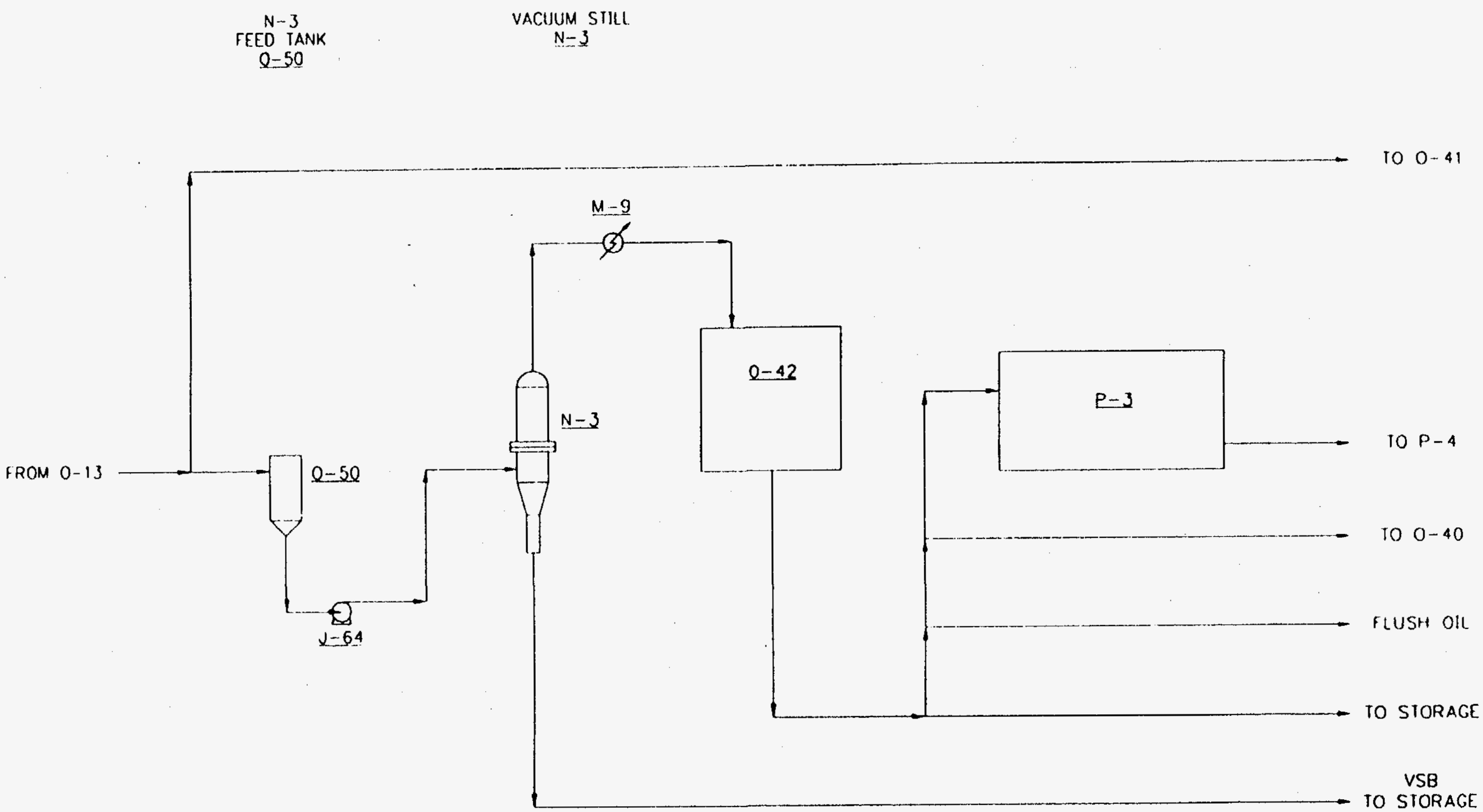


FIGURE 2.3
PDU-260 Simplified Flow Diagram: ROSE-SR℠ Unit

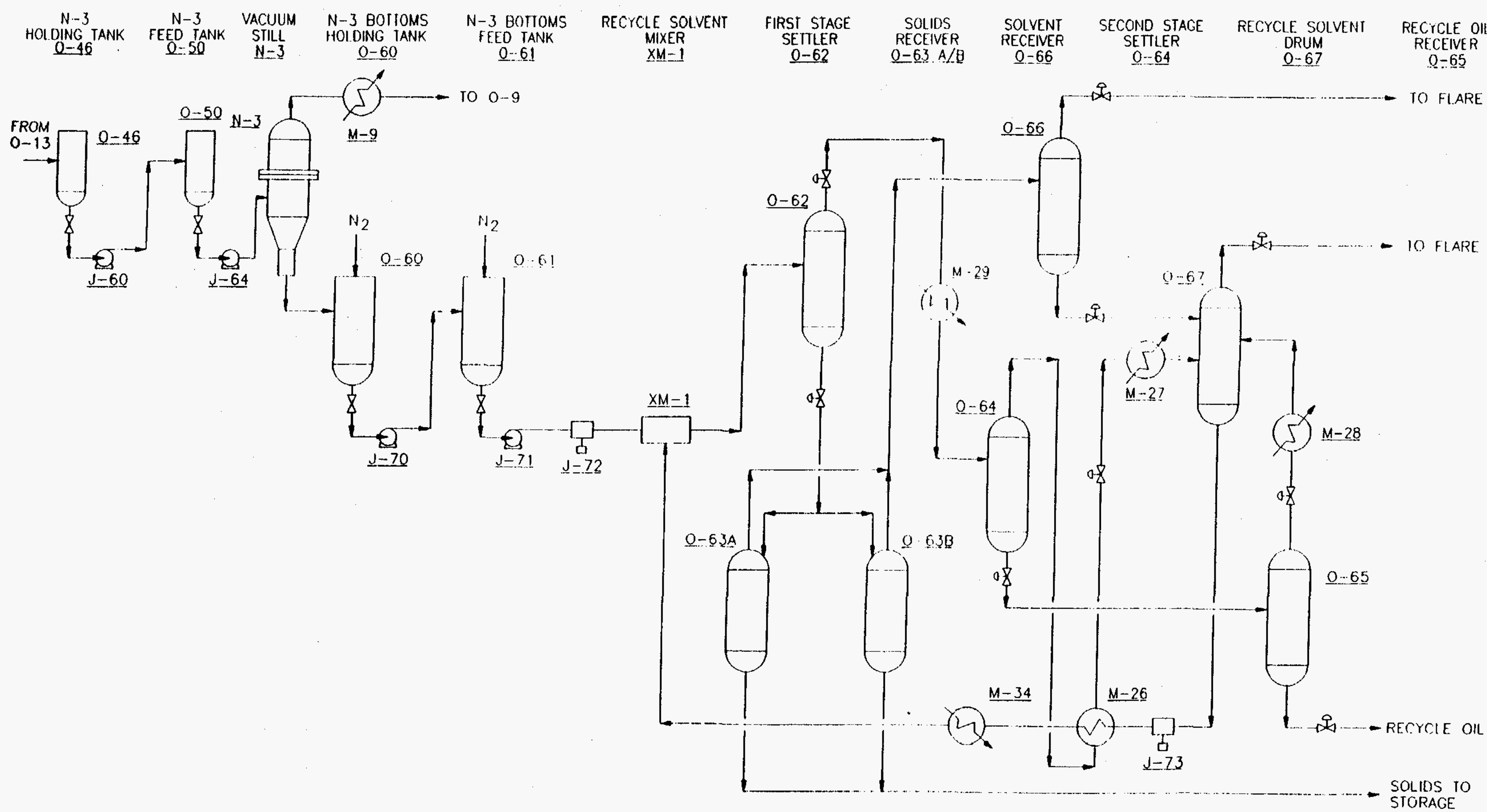
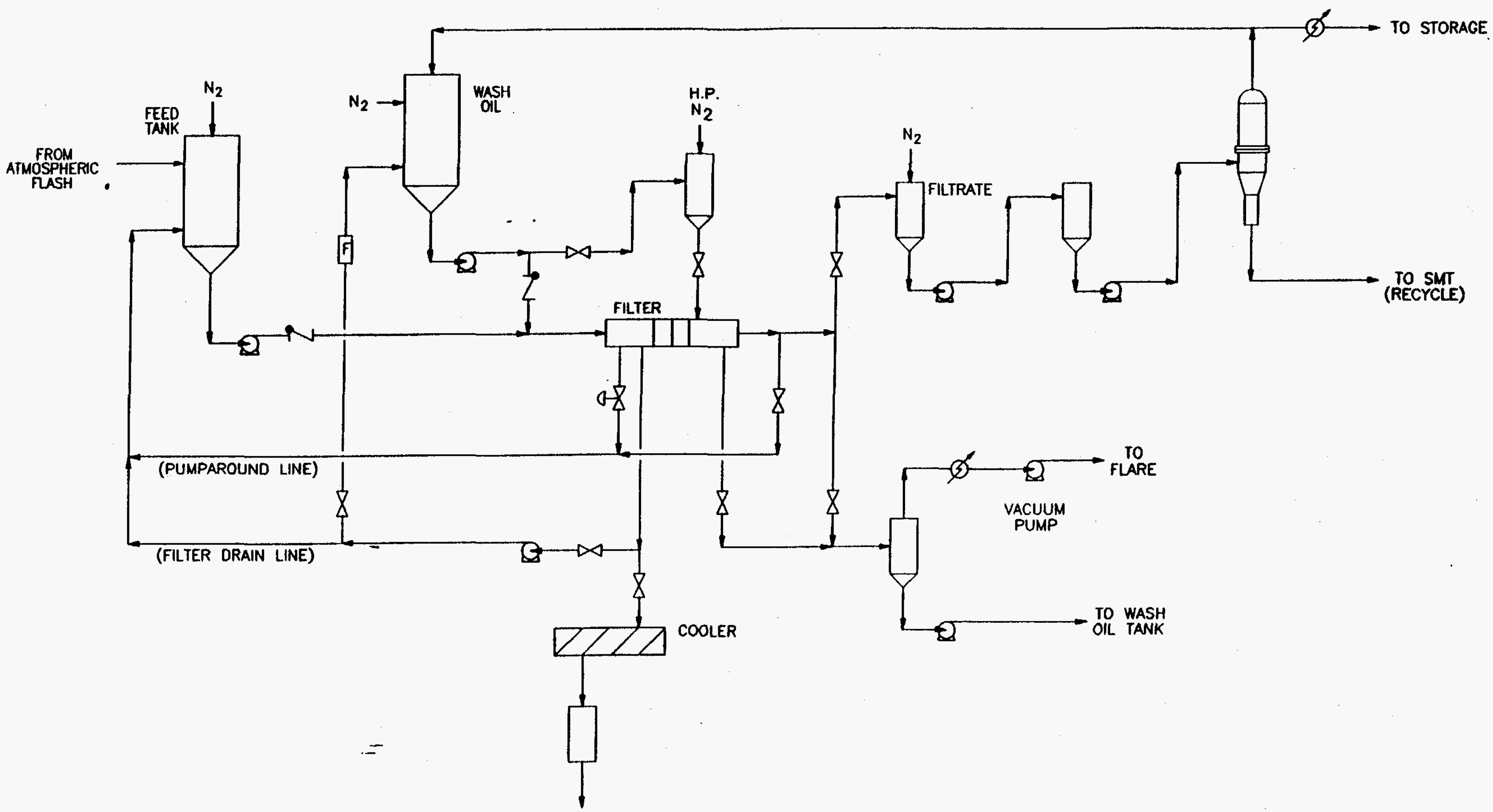


FIGURE 2.4
PDU-260 Simplified Flow Diagram: Filtration Section



B. FEEDSTOCKS

B.1 Feed Coal

The second PDU Run in the Proof-of-Concept program, POC-02, was a pilot-scale demonstration of HTI's CTSL Technology carried out with a subbituminous Wyoming Black Thunder Mine coal. The selection of a candidate low-rank coal for the PDU run was based on several criteria, mainly concerning coal-reactivity, cost, and ash-content. The following were the specifications on feed coal for PDU Run POC-02:

- Moisture Content: 10-12 Wt%
- Ash Content (Dry): 5-7 W%
- Chlorine Content: < 0.10W%
- Particle Size: < 1W% on 50 mesh screen and < 35W% through 350 mesh screen
- Drying & Grinding: Atmosphere containing < 3W% Oxygen

The feed coal was obtained at an "as is" moisture content of between 28-30 w% from the Black Thunder Mine in Wyoming. This coal was predried and pulverized to meet the above specifications for the POC-02 operations. Two random samples of the dried and pulverized coal were obtained from the Empire Coke Company. The qualification tests to find the reactivity of these coal samples were conducted at 427°C and 13.6 MPa for one hour using a 20 cc microautoclave. As shown in Table 2.1, total coal conversions, based upon the THF solubility, were high (92-93 % maf); 524°C+ resid conversions were also high (63-65 % maf). These tests were carried out in the presence of a supported Akzo AO-60 presulfided catalyst.

Table 2.1 Qualification of Two Coal Samples from Empire Coke Company

Coal Sample	W% Moisture	W% Ash, Dry	% Coal Conv.	% Resid Conv.
HTI-6208	9.6	6.3	92.5	63.6
HTI-6209	11.5	6.0	93.0	65.1

The actual coal that was used during POC-02 operations was designated as HTI-6213. Although essentially the same coal as the samples in Table 2.1, it was given a different designation as it arrived later in larger amount. The detailed analysis of the feed coal is shown in Table 2.2.

B.2 Coal Preparation

The run-of-mine coal obtained from the Black Thunder Mine was pulverized and dried to meet the desired specifications at the Empire Coke Company in Alabama. Even though it was desired to conduct the coal grinding operations in the presence of less than 3 % oxygen in order to avoid significant coal surface oxidation, oxygen levels up to 15% were recorded during the drying operations. The coal from these operations was typically -200 mesh and had between 8-10 % moisture. The detailed logsheets from the grinding operations and the proximate analysis of the coal are listed in Tables 2.3 and 2.4.

B.3 Start-up/Make-up Oil

Startup operations for the PDU involve preheating, catalyst presulfiding, and initial catalyst bed ebullation. All these steps require continuous passage of an oil of appropriate physical and chemical properties through the unit. The oil is also needed for slurrying the initial batch of the feed coal. Any emergency unit shutdowns also need oil for flushing. This oil, called the start-up oil, can also be used later during the run as a make-up oil that is mixed with the process-generated recycle solvent to achieve the desired solvent-to-coal ratio. The start-up/make-up solvent (L-814) for POC-02 operations was a mixture of L-809 or Tank-4 oil and a hydrotreated cat cycle oil from a Mobil refinery. The Tank-4 oil (L-809) was a combination of the hydrotreated cat cycle oil and coal-derived materials from POC-01 operations conducted earlier. The detailed analysis of L-814 startup solvent is given in Table 2.5.

TABLE 2.2
Feed Coal Analysis

Proximate Analysis, W% (Empire Coke Lab.)

Moisture	10.28	10.85
Volatile Matter (dry)	39.46	43.17
Fixed Carbon (dry)	52.40	50.16
Ash (dry)	8.14	6.67
Sulfur (dry)	0.47	0.40

Commercial Testing & Eng. Co.
Ultimate Analysis, W%

Moisture	7.89
Carbon	64.59
Hydrogen	4.71
Nitrogen	0.91
Sulfur	0.32
Ash	5.70
Oxygen (diff)	15.88
	100.00

Forms of Sulfur, W% (dry)

Pyritic	0.05
Sulfate	0.01
Organic (diff)	0.29
Total	0.35

Mineral Analysis, W% (Ignited)

SiO2	29.52
Al2O3	16.08
TiO2	1.41
Fe2O3	5.25
CaO	23.98
MgO	4.87
K2O	0.33
Na2O	1.46
SO3	14.41
P2O5	1.03
SrO	0.31
BaO	0.48
Mn3O4	0.07
Undetermined	0.80
	100.00

TABLE 2.3
Grinding Results Used For Process Control

Time	11:30	2:30	5:30	8:30	11:30	2:30	5:00	8:30
Raw Coal Moisture, W%	28.9	28.8	28.7	28.6	28.6	28.8	28.6	
Ground Coal Moisture, W%	10.0	11.1	11.4	9.6	10.2	12.2	11.8	11.4
Screen Analysis, W%								
+50 mesh	0	0	0	0	0	0	0	0
+100 mesh	8.0	6.4	10.2	8.8	10.2	8.2	8.8	8.4
+200 mesh	53.2	48.2	56.0	59.0	56.2	48.2	48.2	49.1
+325 mesh	66.6	71.2	71.6	70.8	71.8	68.8	68.4	67.1

TABLE 2.4
Empire Coke Analysis of Ground Coal Batches

Moisture, W%	8.57
Volatile Matter, W% MF	43.30
Fixed Carbon, W% MF	50.04
Ash, W% MF	6.66
Sulfur Content, W% MF	0.39

TABLE 2.5

Inspection of Hydrotreated Startup Oil - Sample No. L-814

ASTM D-1160 Distillation, Temp [°C]		Distribution, W%	
IBP	309	IBP-454°C	58.99
3V%	343	454-524°C	22.18
5V%	351	524°C+	18.36
10V%	374	Loss	0.47
20V%	394		
30V%	409	Elemental Analysis, W%	
40V%	426	Carbon	88.47
50V%	437	Hydrogen	10.54
60V%	449	Nitrogen	0.83
62V%	454	Sulfur	0.11
70V%	467	Oxygen (diff)	0.05
80V%	507		
84V%	524	H/C Ratio	1.43

B.4 Waste Rubber and Plastics Feeds

During the last nine days of operations of POC-02, coprocessing of used tire rubber material and of mixed plastics with coal was carried out. The used rubber tire material was acquired from Baker Rubber Inc. This material, designated by Baker as Granulite WRF-30, is -30 mesh steel and fiber-free material. The waste plastic feed was simulated by using virgin plastics, such as high density polyethylene (HDPE), polystyrene (PS), and polyethylene terphthalate (PET), in a co-mingled form. All the plastics were supplied by the Aamco Plastics Corporation. The detailed analyses of the rubber tire material and plastics are shown in Tables 2.6 and 2.7, respectively.

C. CATALYSTS FOR POC-02

The POC-02 PDU Run was carried out with two ebullated catalyst bed reactor stages. The initial batch of catalyst used in both the stages was a supported Ni-Mo on alumina catalyst, manufactured by Akzo (AO-60). This catalyst was in the form of 1/16" extrudates, recovered as spent catalyst during the daily withdrawals from POC-01 operations. The spent catalyst from the first PDU run was washed with toluene to extract most of the oil and dried. The initial charge to Reactor #1 was catalyst withdrawn from the first stage during POC-01. This catalyst had an average estimated age of about 580 kg coal/kg catalyst. Initially, the second stage reactor was charged with POC-01 second stage reactor withdrawals with an average age of 500 kg coal/kg catalyst. The average analyses of these two catalysts are shown in Table 2.8. The hydrotreater unit, K-3, operated as a trickle bed, was charged with a trilobe-shaped Criterion C-411 hydrotreating catalyst. During one of the run conditions, an oil-soluble molybdenum additive was employed to mitigate thermal reactions. This was a compound called Molyvan 822, commonly used as an anti-wear additive in lubricating oils; it contained about 4.9 w% molybdenum (in an organometallic complex structure). Molyvan 822 was supplied by R. T. Vanderbilt Company, Norwalk, CT. During the run, 150 ppm of molybdenum relative to feed coal was employed.

TABLE 2.6
Analysis of Feed Plastics

Analysis Type	PS	HDPE	PET
Solubility Test, W%			
Cyclohexane Insolubles	0.29	100	100
CI Ash	0.02	0	0.04
Quinoline Insolubles	0.24	100	100
QI Ash	0.04	0.04	0
Sulfur, W%	0.006	0.46	0.006

TABLE 2.7
Analysis of Feed Rubber

Supplier: Baker Rubber Inc.
Code: Granulite WRF-30, Lot# 9509

Feature: Ground Rubber; Steel and Fibre Free

Baker Test Results		HTI Analysis	
Composition, W% MF		TGA Analysis, W% MF	
Acetone Extraction	11.9	Extender	10.44
RHC	46.5	Polymer	53.80
Carbon Black	36.6	Carbon Black	30.67
Ash	5.0	Ash	5.09
Moisture, W%	0.39	Moisture, W%	0.61
		Elemental Analysis, W% As Is	
		Carbon	82.94
		Hydrogen	7.12
		Nitrogen	0.29
		Sulfur	1.88
		Oxygen (by diff)	2.71
		Ash	5.06

TABLE 2.8
Analytical Data on Catalysts used for Start-up

	------------HTI 6043---------			Equilibrium Catalyst from POC-01	
	Fresh	DMDS Presulfided	TNPS Presulfided	Stage 1	Stage 2
Particle Density, gm/cc	0.872			1.226	1.228
Ignition Loss, W%	3.08			17.08	24.92
Carbon, W%		2.8	2.32	11.3	21.5
Hydrogen, W%		0.46	0.43	0.60	0.57
Nitrogen,W%		0	0	0.14	0.15
Sulfur, W%		6.07	7.54		
Molybdenum, W%	11.31			6.52	7.72
Nickel, W%	2.48			1.38	1.56
Iron, W%	0.01			0.66	0.16
Sodium, W%	0.07			0.64	1.04
Calcium, W%	0.00			0.10	0.05
Molybdenum, gm/cc	0.0986			0.0799	0.0948
Nickel, gm/cc	0.0216			0.0169	0.0192
Fresh Catalyst Content, W%					
Basis Particle Density				71.1	71.0
Basis Ignition Loss				80.8	73.0
Basis Contaminants		93.7	93.5	85.2	74.9
Basis Molybdenum Content				57.6	68.3
Basis Nickel Content				55.6	62.9

*Toluene extracted catalyst from the end of POC-01

D. UNIT OPERATIONS

D.1 Run Plan

The following were key parameters and operating conditions during POC-01 operations:

- Feed Coal: Wyoming Black Thunder Mine Subbituminous Coal
- Other Feeds: Used Tire Rubber and Plastics
- Space Velocity: 320-720 kg feed/h/m^3 reactor volume
- Temperatures: 1st Stage: 399-432°C
2nd Stage: 435-446°C
In-line HTU: 357-382°C
- Catalysts: Spent Akzo AO-60 Catalyst from POC-01 Withdrawals/Molyvan 822 as molybdenum additive
- Solids Separation: Vacuum Still for Start-up, ROSE-SR (on-line), and Filter (off-line)
- Ashy Recycle: 399°C+ Extinction mode
- Recycle stream: O-13 Bottoms, DAO, ASB, VSOH, and Make-up Oil

The primary objective of PDU Run POC-02 was to demonstrate the liquefaction of Black Thunder Subbituminous Coal using CTSL Technology. A total of five operating conditions were initially planned. The original run plan (Revision #3), shown in Table 2.9, was designed to assess the impact of coal space velocity on the yield structure and process performance. As the coal space velocity is increased from about 320 to 640 kg/h/m^3 reactor, temperatures are raised in order to maintain a relatively constant process severity index (5.16 to 5.33). In view of the success observed at low solvent/coal ratio (CMSL-02 and CMSL-05 Bench Runs), a solvent to coal ratio of 1.0 was chosen for the entire run. Both ashy and solids-free recycle modes were to be studied during the POC-02 run.

Two decision points were planned for this run. The first one at the beginning of Condition 3, which starts from Period 19. At this point, the performance of the ROSE-SR was to be reviewed. If ROSE-SR performance was below expectations, filtration would be brought on line to replace the ROSE-SR unit. The second decision point involved the maximum coal throughput to be demonstrated. Based upon process performance and system operability at the end of Condition 4, a decision was to be made whether to increase space velocity to 720 kg/h/m^3. In the last condition, it was planned to evaluate the impact of adding 100-200 wppm of molybdenum precursor. The Mo-additive, which was in the form of an organometallic complex, was to be predispersed in coal-derived solvent and injected before the slurry preheater.

The run was to be started with a coal space velocity of 320 kg/h/m^3, which was to serve as a tie-point with previously obtained bench data. As shown in the Table 2.9 Run Plan, reactor temperatures and space velocities were changed from condition to provide almost identical thermal severities. Starting with reactor temperatures of 400/432°C at the lower space velocities, temperatures were progressively increased to 432/446°C at the higher space velocities. The in-line hydrotreater was initially to be operated at 357°C. This temperature was planned to be raised to 379°C in the later part of the run in order to compensate for the aging of the catalyst. Under these operating conditions sulfur and nitrogen contents of around 20 wppm were expected for the distillates.

In order to shorten the time required to bring the catalysts to steady state, withdrawn catalyst from POC-01 operations was to be used as the initial charge for POC-02 operations. The activity of these spent catalysts was determined in microautoclave tests. The activity data in reference to fresh Akzo AO-60 catalyst is depicted in Figure 2.5. Catalyst replacement rates of 0.5 and 1.0 kg/Mg of coal were selected for the first and second stage reactors respectively. The higher replacement rate for the second stage is essential to maintain performance of the process as the catalyst is expected to age at a much higher rate than in the lower temperature first stage.

Performance, as per the original run plan shown in Table 2.9, was simulated using a kinetic model based upon bench unit data. This projected performance, is summarized in Table 2.10. The two types of solid separation systems considered in these simulations are ROSE-SR and filtration. Estimated performance is presented in Figures 2.6 through 2.13. The model assumed that the resid content of the solid rejects was 5 and 30 w% for filtration and ROSE-SR, respectively. The higher resid content (30 w%) for ROSE-SR operations was based upon Kerr-McGee's experience for effective agglomeration of unreacted coal and mineral matter. On the other hand, the lower resid content for filtration was a projected performance assuming effective cake washing using a low boiling process stream. Since the system is assumed to be operated in resid extinction mode, the lower resid content in the filter cake results in 5-6 w% improvement in distillate yields. As a result, hydrogen consumption also increased by 0.2 to 0.3 w% when using filtration. The starting solvent for ROSE-SR operations was to be n-pentane. It was planned that this would be changed during the course of the run to a mixed solvent comprising n-pentane and toluene to improve recovery of asphaltenic materials.

The original run plan for POC-02 had to be modified several times. The final modified Run Plan (Revision 11), shown in Table 2.11, includes nine additional operating days to the original run plan shown in Table 2.9. A detailed listing of various internal samples from the POC-02 operations is shown in Tables 2.12 and 2.13; these two tables show the forms prepared by HTI to record the intended samples. The additional nine days were designed to study combined processing of coal and organic wastes, such as used tire rubber and plastics. The solvent/coal ratio was increased from 1.5 to 2.0 during these periods to ensure pumpability of the high viscosity slurries formed from coal and wastes. Temperatures and space velocities during these periods were similar to the last 'coal-only' feed condition. A -30 mesh crumb rubber (steel and fiber free) was used for the waste tire material, while virgin plastics, such as HDPE (50 w%), polystyrene (35 w%), and PET (15 w%), were mixed to represent a typical municipal solid waste plastic composition.

TABLE 2.9
Proposed Run Plan

Condition No.	Period No.	No. of Days	Deasher Type	Recycle Mode	Solv/Coal Ratio	Space Velocity	------ Temperature [°C] ---- K1	K2	K3	Catalyst Replacement K1	K2	Severity Index
L/O	1-2	2	Vac. Still	Ashy	1.5-1.2	320	393-399	393-432	343	0.5	1.0	
L/O	3-4	2	Vac. Still	Ashy	1.0	320	399	435	357	1.0	2.0	
1	5-11	7	Rose	Solid-free	1.0	320	399	435	357	1.0	2.0	5.33
2	12-18	7	Rose	Solid-free	1.0	481	413	443	TBA	1.0	2.0	5.16
3	19-25	7	ROSE/Filter*	Solid-free	1.0	561	427	443	TBA	1.0	2.0	5.19
4	26-32	7	ROSE/Filter	Solid-free	1.0	641	432	446	TBA	1.0	2.0	5.25
5	33-40	8	ROSE/Filter	Ashy	1.0	641**	432	446	TBA	1.0	2.0	5.25
		(or)	ROSE/Filter	Ashy	1.0	721	432	446	TBA	1.0	2.0	4.67

Note:

1. Coal Space Velocity: Kg dry coal/h/m^3 reactor
2. In-line hydrotreater to process second stage overheads and O-13 overheads (O-12).
3. Catalyst: Akzo AO-60 1/16" extrudates used in POC-01.

* Decision point for bringing the filter on-line for removing solids.

** Decision point for increasing the coal space velocity to 721 Kg dry coal/h/m^3 reactor.

TABLE 2.10
Projected Yields and Performance

Solids Separation System	ROSE-SR System					Filtration System				
Case No.	1	2	3	4	5	1	2	3	4	5
Coal Space Velocity, Kg dry coal/h/m3	320	481	561	641	721	320	481	561	641	721
Temperatures, °C										
First Stage	399	413	427	432	432	399	413	427	432	432
Second Stage	435	443	443	446	446	435	443	443	446	446
Severity Index (ref: 399°C, 320 Kg dry coal/h/m3)										
First Stage	1.00	1.19	1.79	1.94	1.73	1.00	1.19	1.79	1.94	1.73
Second Stage	4.33	3.97	3.40	3.31	2.94	4.33	3.97	3.40	3.31	2.94
Total	5.33	5.16	5.19	5.25	4.67	5.33	5.16	5.19	5.25	4.67
Catalyst Replacement Rate, Kg/Ton										
First Stage	0.45	0.45	0.45	0.45	0.45	0.45	0.45	0.45	0.45	0.45
Second Stage	0.91	0.91	0.91	0.91	0.91	0.91	0.91	0.91	0.91	0.91
Yields, W% Dry Coal										
C_1-C_3	8.55	8.33	8.37	8.44	7.74	8.55	8.33	8.37	8.44	7.74
C_4-199°C	22.58	22.00	22.12	22.29	20.44	22.58	22.00	22.12	22.29	20.44
199°C-524°C	32.88	34.45	34.38	34.40	35.62	39.40	40.28	40.66	40.61	42.20
Water	17.41	17.42	17.54	17.57	17.37	17.41	17.42	17.54	17.57	17.37
CO_X	0.76	0.76	0.76	0.76	0.75	0.76	0.76	0.76	0.76	0.75
NH_3	0.59	0.66	0.69	0.71	0.71	0.59	0.66	0.69	0.71	0.71
H_2S	0.40	0.40	0.40	0.40	0.40	0.40	0.40	0.40	0.40	0.40
Resid	7.11	6.45	6.85	6.79	7.19	0.87	0.87	0.84	0.83	0.88
Unconverted Coal	10.00	9.95	9.39	9.23	10.17	10.00	9.95	9.39	9.23	10.17
Ash	6.60	6.60	6.60	6.60	6.60	6.60	6.60	6.60	6.60	6.60
Total	106.88	107.02	107.10	107.19	106.99	107.16	107.27	107.37	107.44	107.26
Resid in DAO or PFL	15.46	16.41	14.81	15.16	16.30	23.07	23.30	22.15	22.48	24.21
Hydrogen Consumption	6.88	7.02	7.10	7.19	6.99	7.16	7.27	7.37	7.44	7.26
Process Performance, W% Dry Coal										
C_4-524°C	55.46	56.45	56.50	56.69	56.06	61.98	62.28	62.78	62.90	62.64
Coal Conversion	89.3	89.3	89.9	90.1	89.1	89.3	89.3	89.9	90.1	89.1
Resid Conversion	81.7	82.4	82.6	82.8	81.4	88.4	88.4	89.0	89.2	88.2

TABLE 2.11
Modified Run Plan

Condition No.	Period No.	No. of Days	Deasher Type	Recycle Mode	Recy/Feed Ratio	Space Velocity	------ Temperature [C] ------ K-1	K-2	K-3	Catalyst Repl. Rate K-1	K-2	Severity Index	Remarks
L/O-1	1-2	2	Vac. Still	Ashy	1.5-1.2	320	393-399	393-432	343	0.5	1.0		
1A	3-6	4	ROSE	Solid-free	1.0	320	394	435	357	1.0	2.0		
L/O-2	7-8	2	Vac. Still	Ashy	1.5-1.2	320	393-399	393-432	357	0.5	1.0		
1B	9-15	7	ROSE	Ashy	1.2	320	399	435	371	1.0	2.0	5.33	
2A	16-17	2	ROSE	Ashy	1.2	320	399	435	371	1.0	2.0	5.33	
2B	18-22	7	ROSE	Ashy	1.2	481	413	443	374	1.0	2.0	5.16	
3	(deleted)												
L/O-3	23-25A	3	V.S./ROSE	Ashy	1.5-1.2	320-481	393-416	393-435	377	1.0	2.0		
L/O-4	26A	1	ROSE	Ashy*	1.5-1.2	320-481	393-416	393-435	377	1.0	2.0		
L/O-5	27	1	ROSE	Ashy*	1.5	320	393-416	393-435	377	1.0	2.0		
L/O-5	28	1	ROSE	Ashy*	1.2	320-481	416	435-443	377-382	1.5	2.5		
4A	29	1	ROSE	Ashy*	1.2	481-561	416	443	382	1.5	2.5		
4B	30-31	2	ROSE	Ashy*	1.2	561-641	416-432	443-446	382	1.5	2.5		
4C	32-33	2	ROSE	Ashy*	1.2	641	432	446	382	1.5	2.5	5.25	
5	34-36	3	Filter	Ashy*	1.2	641	432	446	382	1.5	2.5	5.25	Mo Additive (150 ppm)
Co-Liquefaction of Coal and Waste Hydrocarbons													
6A**	37-38A	1.5	ROSE/V.Still	Solid-free	2.0	481	432	443	382	1.5	2.5	5.16	Coal/Used Tire=75/25
7A+	38B-40	2.5	ROSE/V.Still	Solid-free	2.0	481	432	443	382	1.5	2.5	5.16	Coal/Plastics=90/10
7B++	41-42	2	ROSE/V.Still	Solid-free	2.0	481	432	443	382	1.5	2.5	5.16	Coal/Plastics=70/30
7C+++	43	1	ROSE/V.Still	Solid-free	2.0	481	432	443	382	1.5	2.5	5.16	Coal/Plastics=70/30
6B**	44-45	2	ROSE/V.Still	Solid-free	2.0	481	432	443	382	1.5	2.5	5.16	Coal/Used Tire=75/25

Note:
1. Coal Space Velocity: Kg dry coal/h/m3 reactor
2. In-line Hydrotreater to process 2nd stage Overheads and O-13 Overheads (O-12).
3. Catalyst: Akzo AO-60 1/16" Extrudates used in POC-01.
4. Hydrotreator to be taken off-line in Periods34A and 43A so that O-1 Overheads can be collected.
5. Conditions 6 & 7: K1 temperature of 432C is selected; contingent operability and yield structure.
6. Condition 7C: Targart feed rate of 50% coal and 50% plastics (without PET); contingent on operability and discussion with DOE.

* O-13 Bottom in recycle solvent is 20 W% of the dry coal rate.
** Co-liquefaction of coal (75 W%) and Used Rubber Tire (25 W%); recycle/coal/tire=8/3/1
\+ Co-liquefaction of coal (90W%), Polyethylene (6 W%) and Polystyrene (4 W%); recycle/coal/plastic=8/3.6/0.4
++ Co-liquefaction of coal (70 W%), Polyethylene (18 W%) and Polystyrene (12 W%); recycle/coal/plastic=8/2.8/1.2
+++ Co-liquefaction of coal (70 W%), Polyethylene (15 W%),Polystyrene (10 W%) and PET(5W%); recycle/coal/plastic=8/2.8/1.2

TABLE 2.12
Daily Samples Sheet: Rose-SR^{SM} Solid Separation Mode

PDU Run 260-05 Daily Samples

Solids Separation: ROSE-SR
Periods 5 to 45

Period No: ________ By: ______________ Date: __________

Sample Type	Quantity	Sub-period A	Sub-period B
Gases			
Vent Gas	Flow Bottle	______	______
Bottoms Gas	Flow Bottle	______	______
Liquids			
Naphtha Stabilizer (N-5) Bottoms	1/2 Gallon		______
Atmospheric Still (N-2) Bottoms	1/2 Gallon		______
Cold Separator (O-5) Bottoms (+)	1/2 Gallon		______
RLFD Overheads (O-12) (+)	Quart		______
Atmospheric Still (N-2) Feed (+)	Quart		______
Vacuum Still (N-3) Overheads	Quart		______
ROSE Recycle Oil Receiver (O-65)	Quart		______
ROSE Recycle Solvent Drum (O-67)	Pint		______
Clean Oil Tank (P-3)	Pint		______
Sour Water (O-44)	Pint		______
Slurry Tank Condensate (P-4)	Pint		______
Slurries			
Feed Slurry (P-4)	Pint		______
Reac. Liquid Flash Drum Btms. (O-46)	Quart		______
Recycle Holding Tank (O-43)	Quart		______
Settler Feed Tank (O-61)	Quart		______
Vacuum Still (N-3) Bottoms	Quart		______
Reactor #1 Slurry (*)	All		______
Solids			
Feed Coal	100 Grams		______
Solid Receiver (O-63 A/B)	Quart (Lab)		______
	Gallon (Storage)		______
1st Stage Catalyst			
Drained	All	______	______
Draining	Storage	______	______
2nd Stage Catalyst			
Drained	All	______	______
Draining	Storage	______	______

(*) Periods 14, 23, 30, 38, 41 & 44.
(+) Special O-5 and O-12 liquid Samples to be taken in Periods 4,15,24,32,39, 42 & 45

TABLE 2.13
Daily Samples Sheet: Vacuum Tower Mode

PDU Run 260-05 Daily Samples
Solids Separation: Vacuum Tower
Periods1 to 4

Period No: ________ By: ______________ Date: __________

Sample Type	Quantity	Sub-period A	Sub-period B
Gases			
Vent Gas	Flow Bottle	______	______
Bottoms Gas	Flow Bottle	______	______
Liquids			
Naphtha Stabilizer (N-5) Bottoms	1/2 Gallon		______
Atmospheric Still (N-2) Bottoms	1/2 Gallon		______
Vacuum Still (N-3) Overheads	1/2 Gallon		______
Cold Separator (O-5) Bottoms	1/2 Gallon		______
RLFD Overheads (O-12)	Quart		______
Clean Oil Tank (P-3)	Pint		______
Sour Water (O-45)	Pint		______
Slurry Tank Condensate	Pint		______
Slurries			
Feed Slurry (P-4)	Pint		______
Reac. Liquid Flash Drum Btms (O-46).	Quart		______
Vac. Still (N-3) Bottoms	1/2 Gallon		______
Recycle Holding Tank (O-43)	Pint		______
Solids			
Feed Coal	100 Grams		______
1st Stage Catalyst			
Drained	All	______	______
Draining	Storage	______	______
2nd Stage Catalyst			
Drained	All	______	______
Draining	Storage	______	______

FIGURE 2.5
Activity of Spent Catalysts from POC-1 Withdrawals

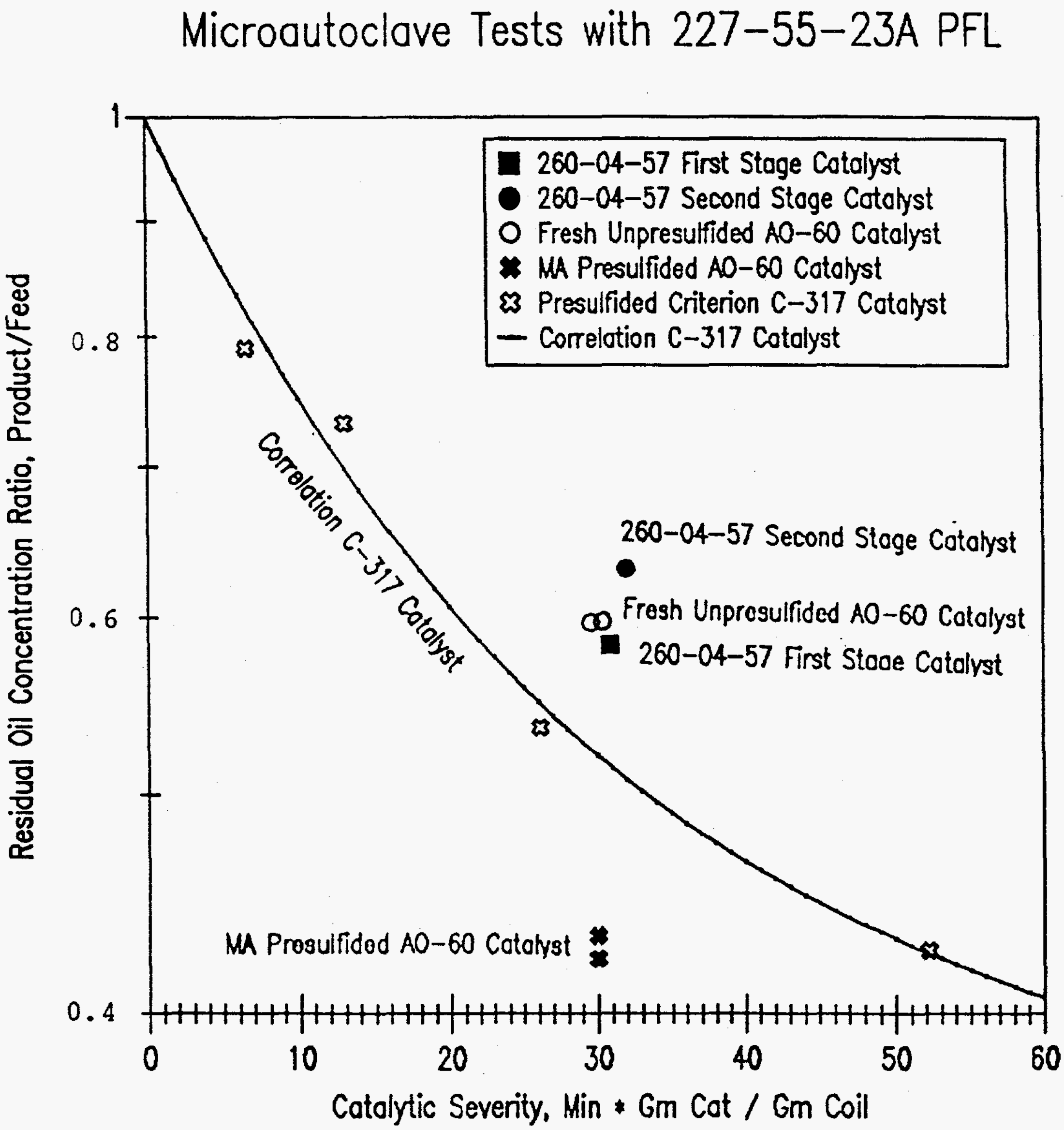


FIGURE 2.6
Target vs. Actual Catalyst/Process Equilibration

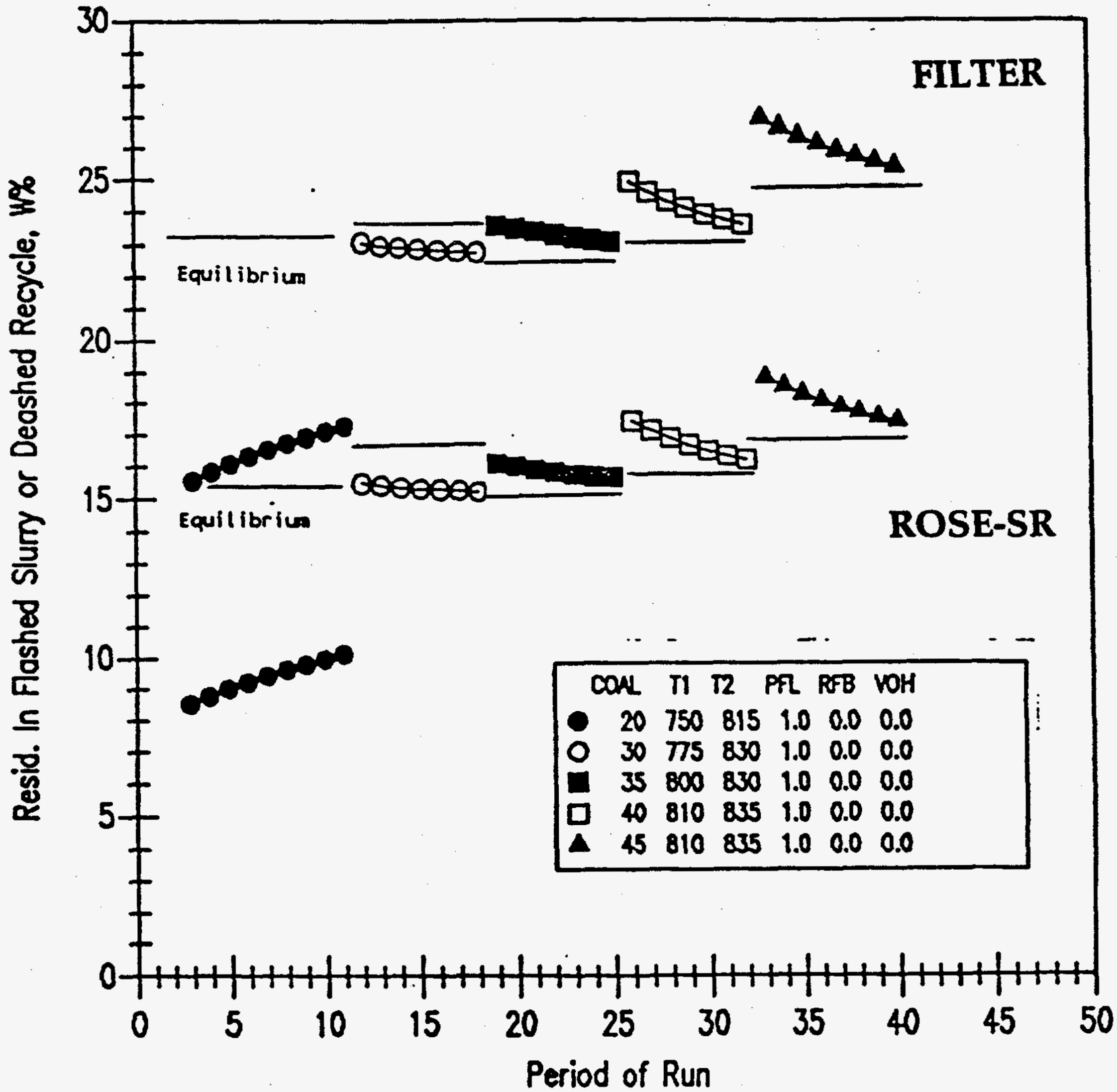


FIGURE 2.7
Resid Content in Recycle Solvent

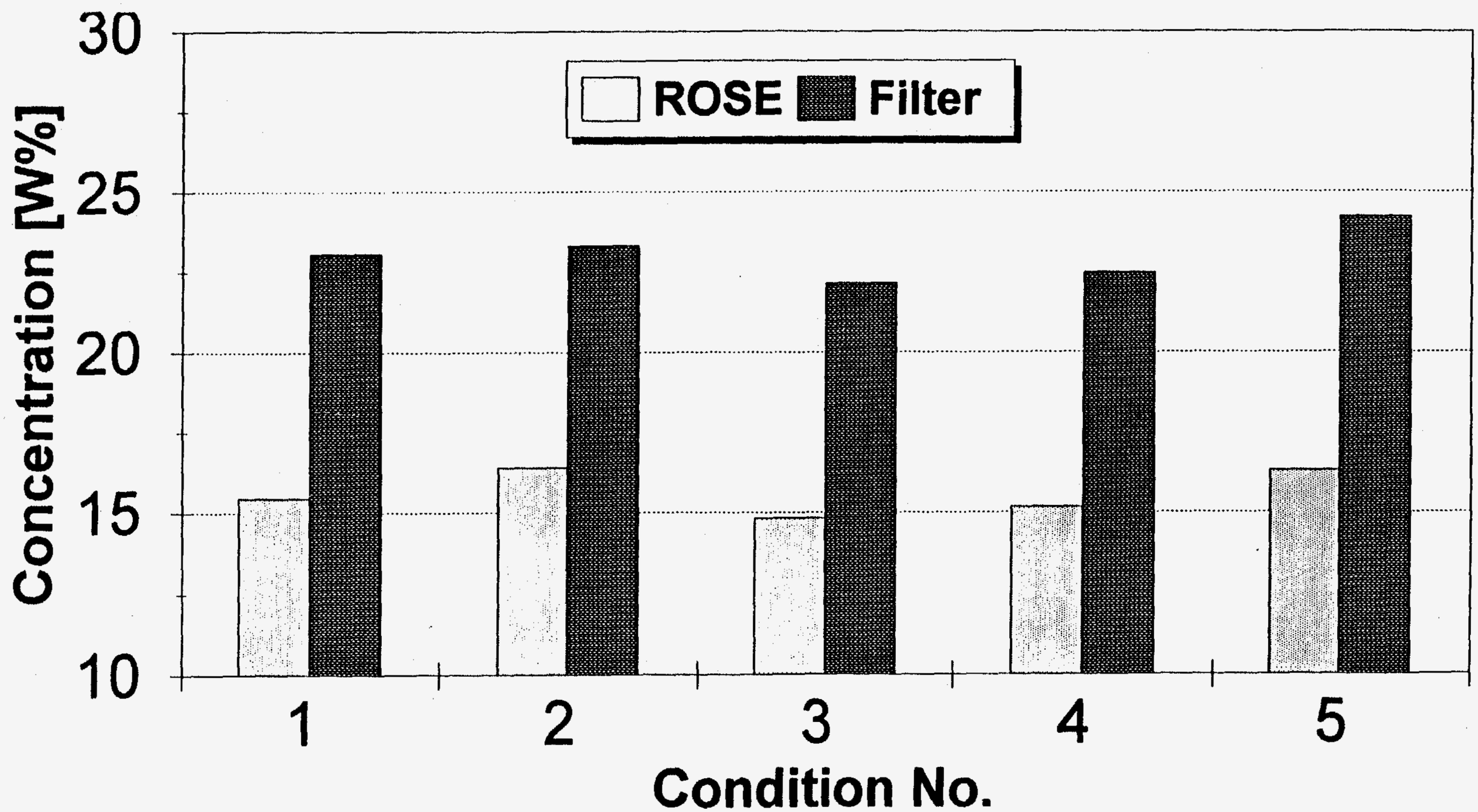


FIGURE 2.8
Gas Make & Hydrogen Consumption for ROSE-SR^{SM} Operations

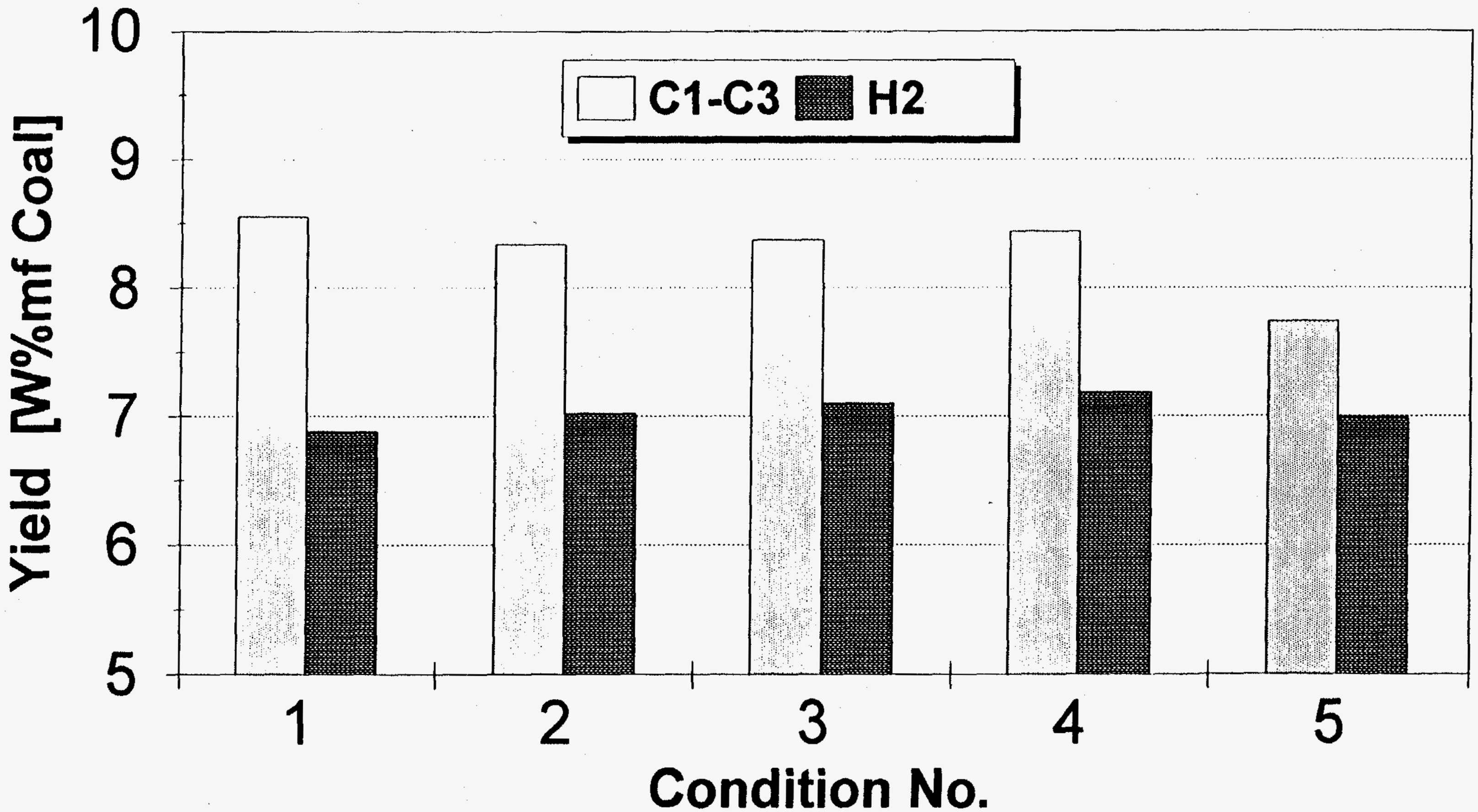


FIGURE 2.9
Coal and Resid Conversions for ROSE-SR^{SM} Operations

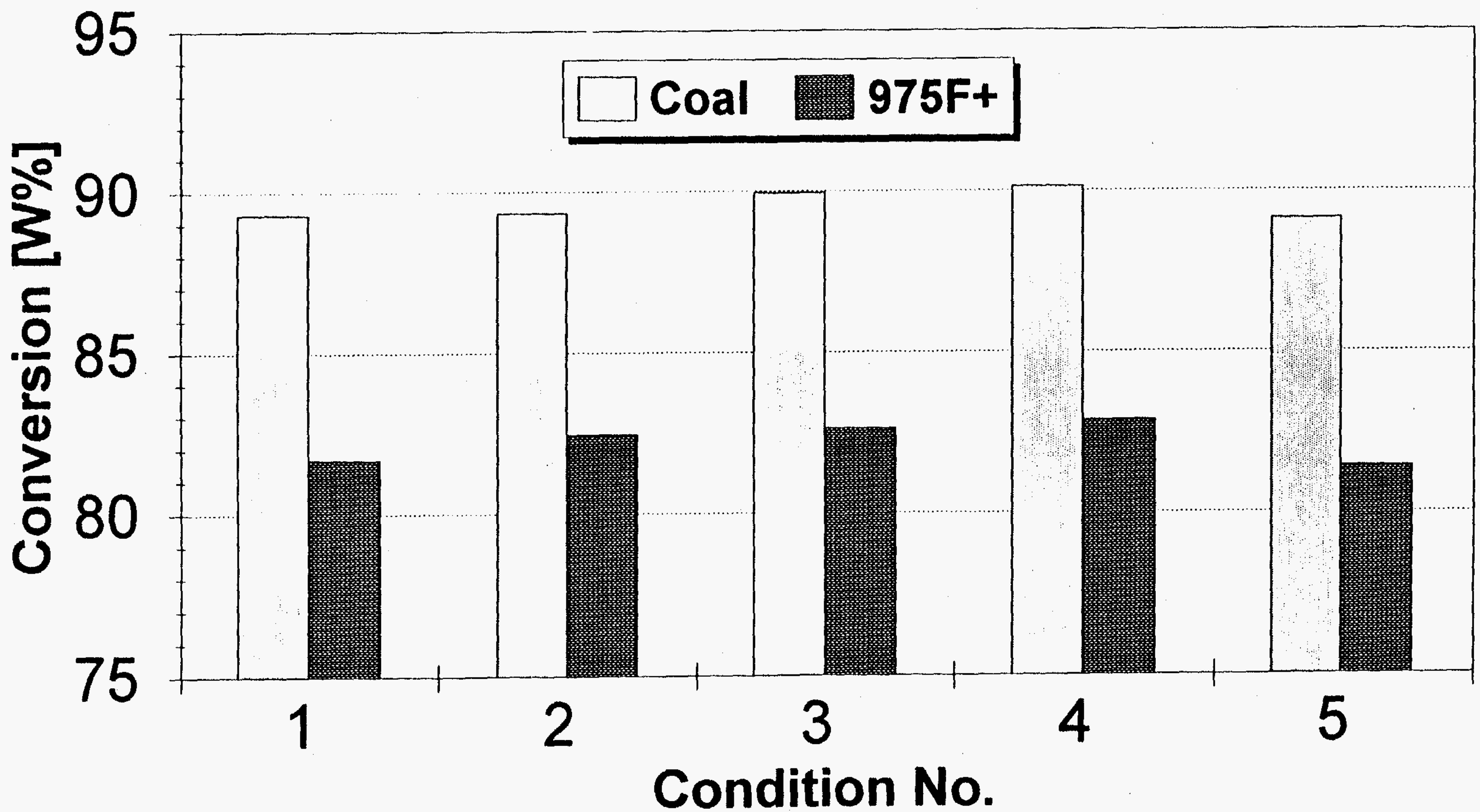


FIGURE 2.10

Distillate and Resid Yields for ROSE-SR^{SM} Operations

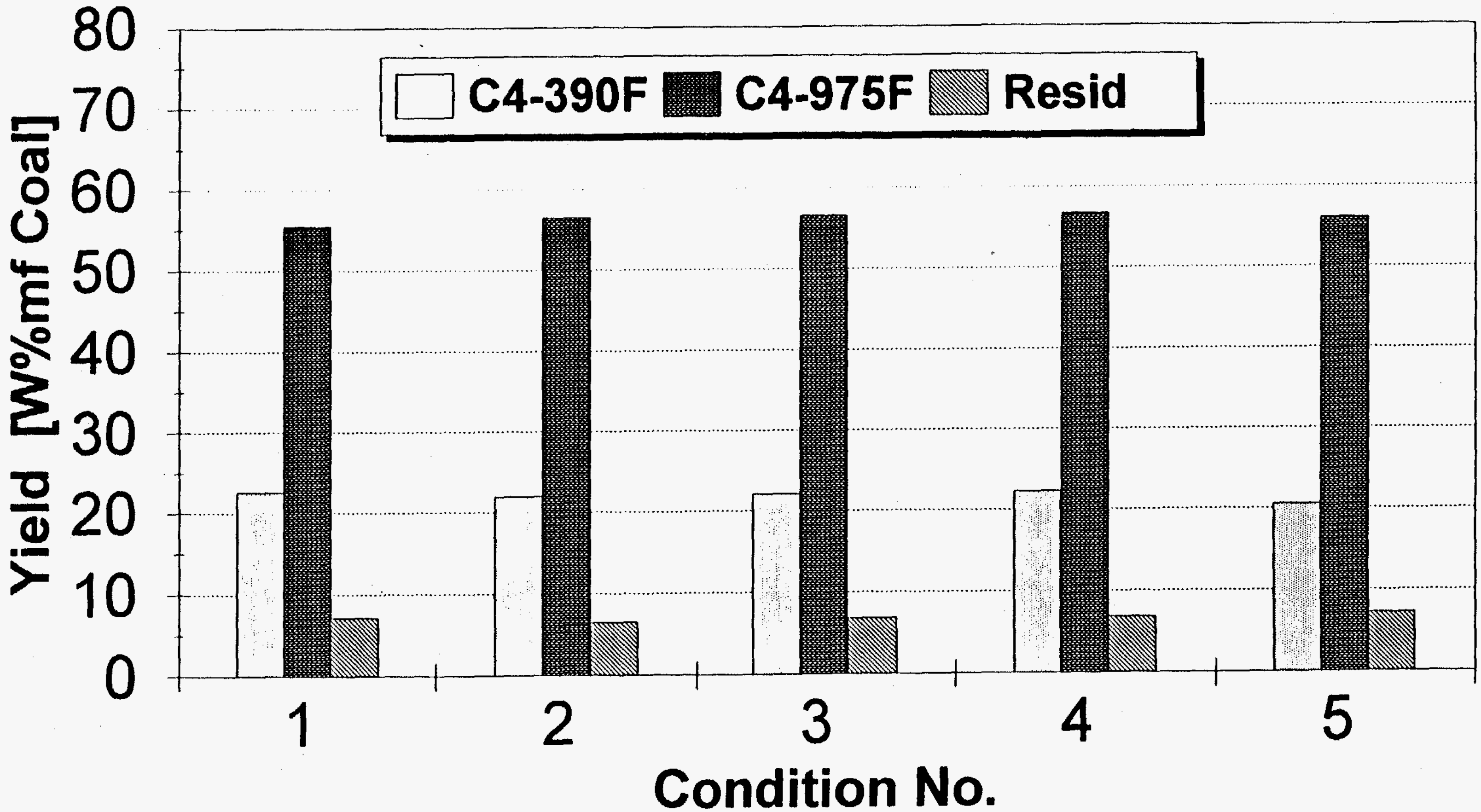


FIGURE 2.11
Coal and Resid Conversions for Filter Operations

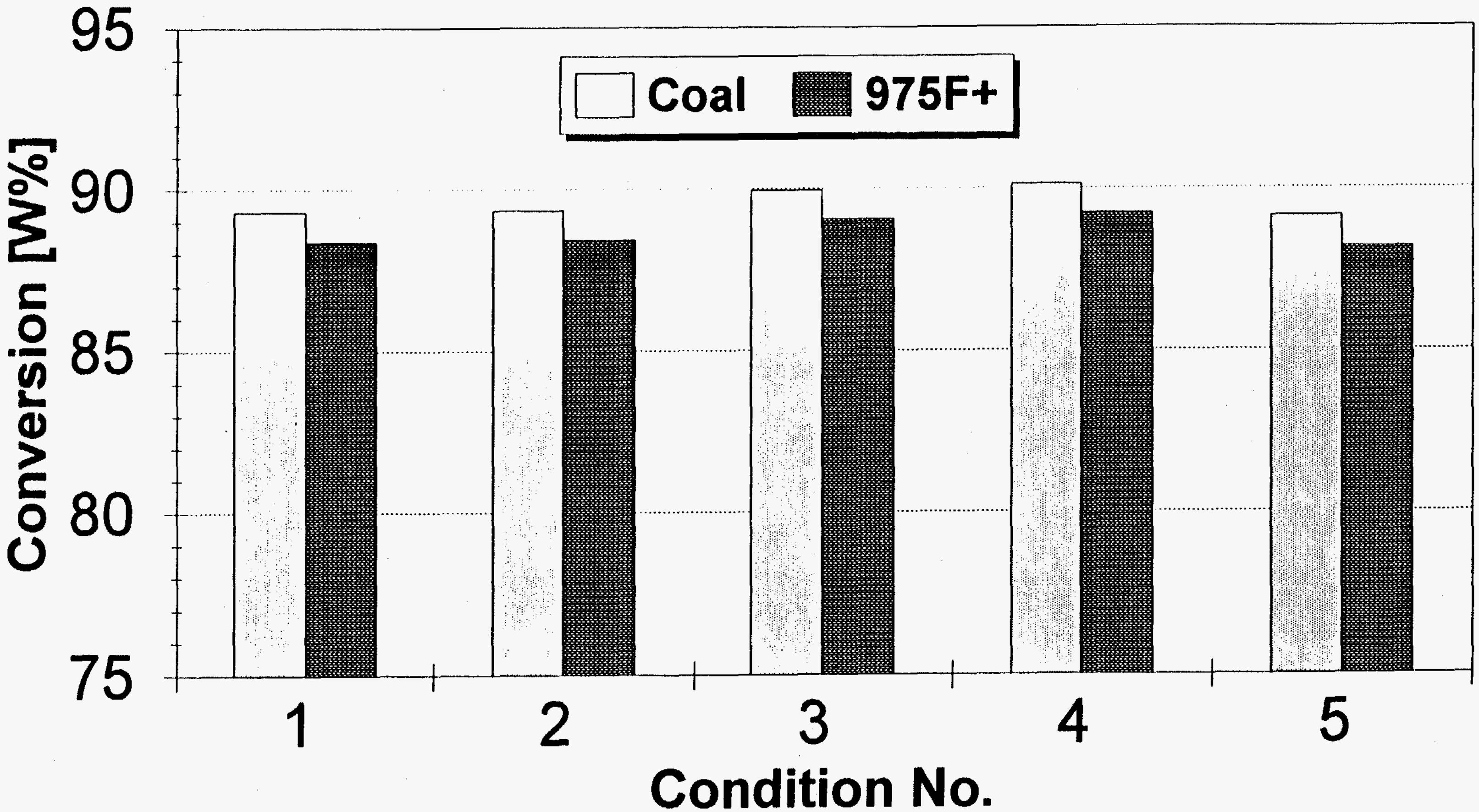


FIGURE 2.12
Distillate and Resid Yields for Filter Operations

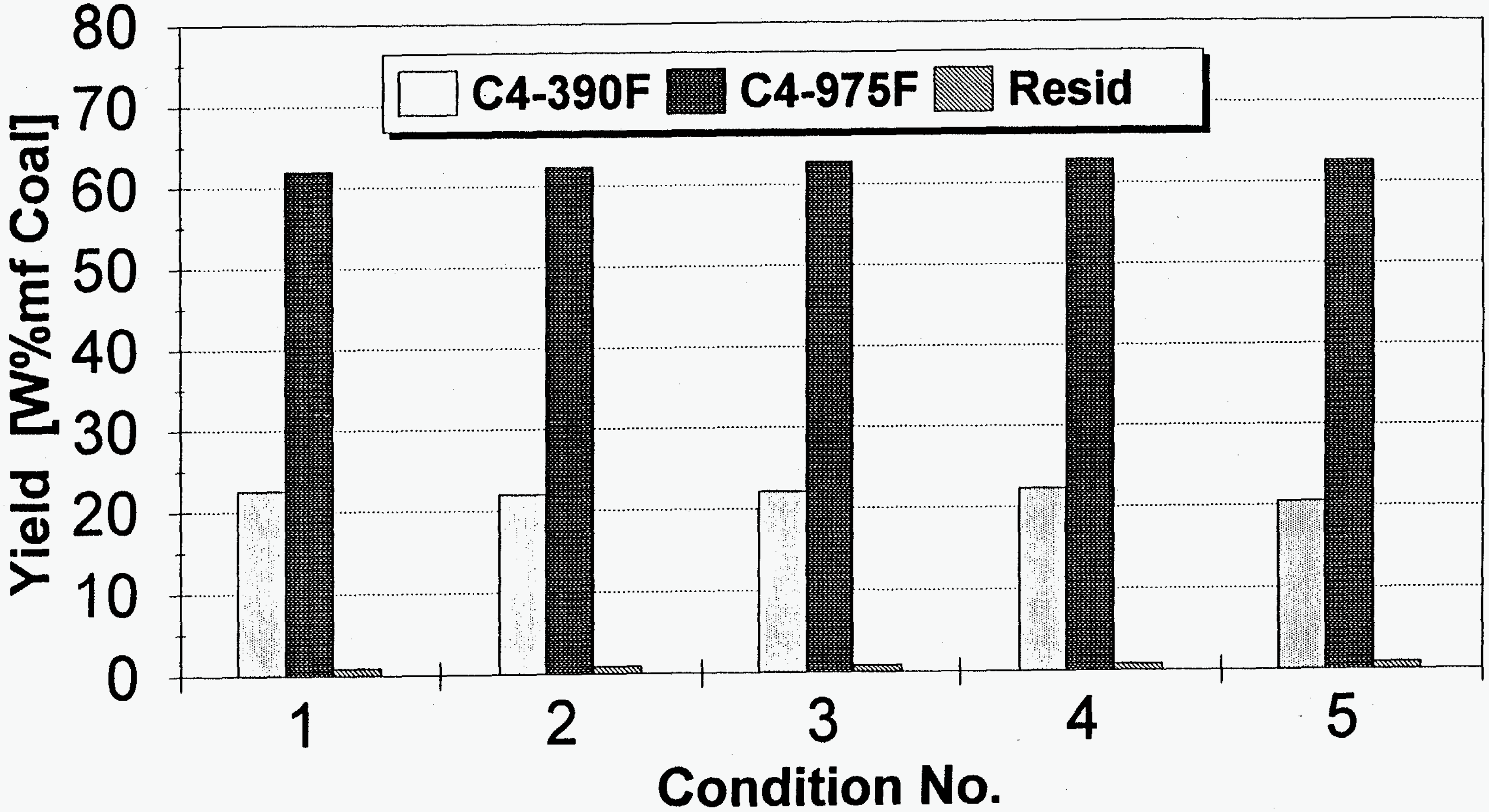
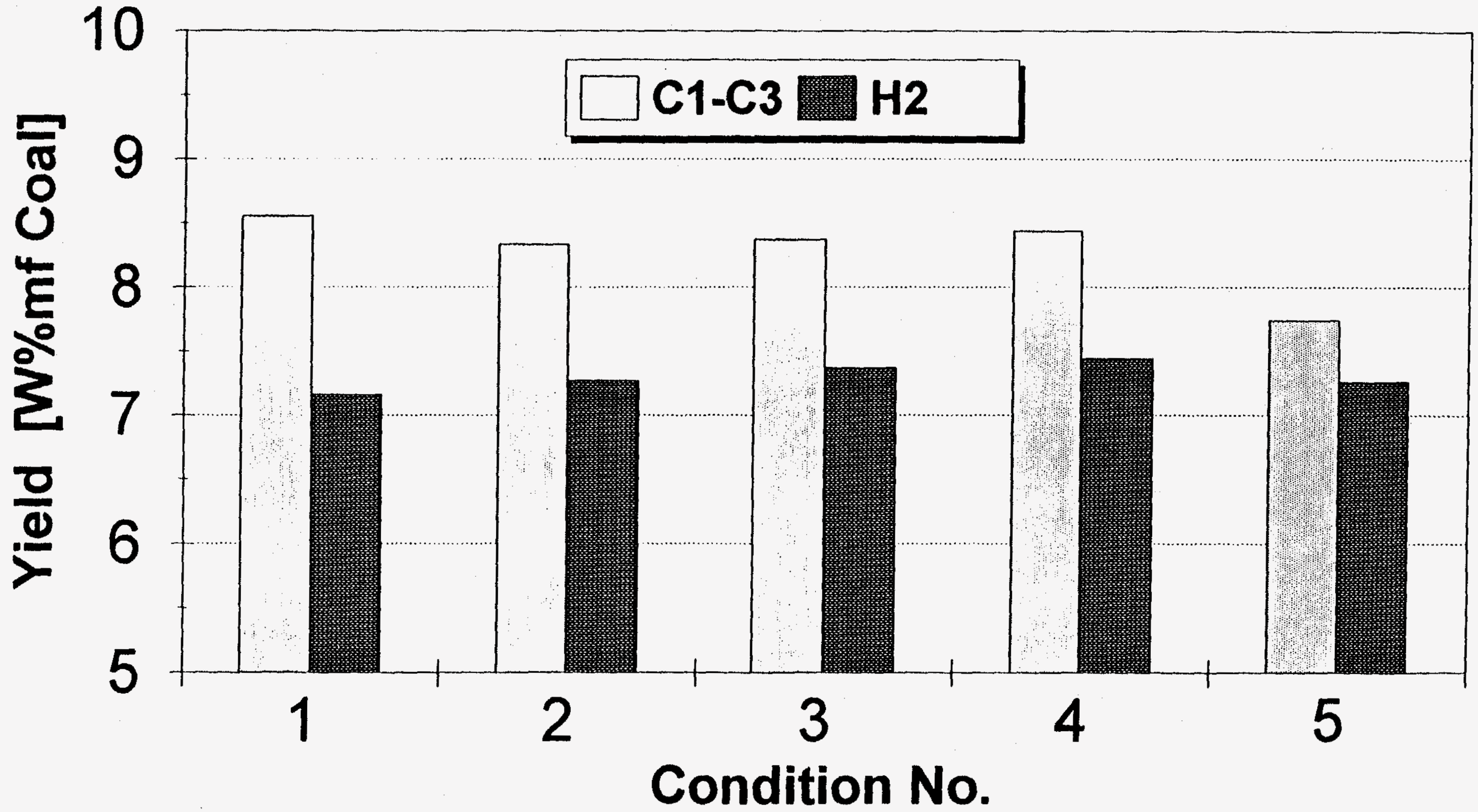


FIGURE 2.13
Gas Make and Hydrogen Consumption for Filter Operations



D.2 OPERATIONS SUMMARY

I. Overview

A number of modifications were made to the 260 Unit prior to the second proof-of-concept operation, POC-02 (260-05). These modifications included:

- Installation of hydrogen purge meter coalescers
- Installation of O-41 scale
- Installation of O-46 scale
- Connection of VSO, sour water scales to the process control system
- Routing recycle gas compressor relief line to flare
- Installing an emergency block valve in the recycle gas heater to the second stage reactor line
- Modifying the ROSE-SR^{sm} first stage settler to bottoms receiver line
- Installing a larger oil/water separator
- Installing a purge oil connection to the K-1 sample system inlet.
- Installing a ROSE solvent trim heater
- Routing ASBs to COT, VSO to O-41
- Installing a ceramic shell around the flare
- Establishing a reactor thermocouple repair method
- Installing new hydrotreater internals
- Installing a coal hopper rotary valve
- Replacing the secondary oil/water with a metal vessel.
- Routing the hot separator inlet to the larger diameter vapor zone.
- Installing a waterboot on the naphtha stabilizer feed accumulator
- Relocating the catalyst addition valves to the top of the reactor

These modifications were completed between March and June 1994. In general, these modifications improved the unit operations and data processing. Additional details on each of these modifications are given later in this section.

The primary objective of the second proof-of-concept operation was to demonstrate the CTSL process with Black Thunder coal. Other objectives include:

- confirming the operability of the modified unit,
- evaluating ROSE-SR solid separation technology,

- evaluating filtration solid separation technology,
- obtaining process performance data at different operating conditions regarding:
  - conversion and yields,
  - catalyst consumption,
  - extinction recycle and
  - in-line hydrotreating,
- generating samples for outside analysis,
- demonstrating coal/rubber processing,
- demonstrating coal/plastic processing and
- obtaining environmental air monitoring data.

Efforts were expended to achieve each of these objectives. In general, the new and modified equipment performed well. Additional equipment and procedural modifications have been identified to further improve unit operation. (Refer to Sections IV and V of this report for additional details.)

Several accomplishments and highlights of this program are:

- hydrotreating about 30,280 liters (8,000 gallons) of start-up oil prior to the run
- processing Black Thunder coal for a total of 45 days
- completing 13 different operating conditions (1A, 1B, 2A, 2B, 4A, 4B, 4C, 5, 6A, 6B, 7A, 7B and 7C)
- obtaining 2 first stage reactor liquid samples
- obtaining sour water samples to evaluate commercial waste water requirements
- demonstrating successful hydrotreater process performance
- testing the filter to generate recycle solvent
- hydrotreating and collecting over 11, 355 liters (3,000 gallons) of net process distillate
- obtaining special samples for outside testing and processing at:
  - SouthWest Research Institute
  - Consol, Inc.
  - PETC
  - Consortium for Fossil Fuel Liquefaction Science
- exposing corrosion coupons (provided by Oak Ridge National Laboratory) to the process in:
  - the first stage reactor
  - the second stage reactor
  - the hot separator vapor zone

+ the hydrotreater
+ the top of the atmospheric still
+ the bottom of the atmospheric still
+ the vacuum still

- completing an environmental air monitoring program
- operating the ROSE section off-line
- operating the ROSE section for extended periods during PDU operations
- co-processing coal and rubber
- co-processing coal and plastics
- processing coal smoothly at a space velocity of 640 kg/h/m3 (40 lb/hr/ft3)
- demonstrating dual solvent ROSE operation
- producing ROSE bottoms material with less than 10 W% pentane solubles
- discovering a method for removing ammonium salt deposits without shutting down the unit.
- demonstrating a positive solvent balance of 53 kg (117 pounds) over the first 36 periods of the run.

There were three coal outages, which lasted more than one day. The first outage, after Period 6, was related to a high pressure drop between the second stage reactor and the high pressure overheads cooler. This was probably an ammonium salt deposit. In Period 23, catalyst was carried over into the second stage ebullating cup, restricting flow and causing the second shutdown. The unit was shutdown a third time to repair the alternative hydrotreater feed line. These three outages accounted for 324 of the total 361 hours coal was not feed during this run.

Over 98,000 kg (108 tons) of as-received coal were processed during this 45 day campaign. During the last seven days, 12,700 kg (14 tons) of coal were processed with 3,200 (3.5 tons) of various mixtures of plastics or rubber. This run included two extended operations of 17 and 19 days. The overall operating performance data during this campaign are:

Periods	All	1-38
average liquefaction section liquid closure:	99.52%	98.14%
average liquefaction section material balance:	100.17%	100.99%
on-stream factor including all outages:	73.61%	69.63%
capacity utilization factor:	68.69%	65.51%

daily capacity utilization factor:	93.33%	94.09%

The target operating conditions for run POC-02 (260-05) are summarized in Table 2.14.

D.3. Operating History

After the modifications were completed, the unit and the corresponding drawings were reviewed to confirm conformance. The unit was insulated, loaded with catalyst from the end of the first proof-of-concept run and pressure checked. An appropriate amount of catalyst was then charged to each of the three reactors. Operating procedures were written, reviewed and issued.

At the end of April 1994, withdrawn catalysts from POC-01 were charged to reactors one and two, while fresh Criterion C-411 (HRI-6135) was loaded into the hydroteater.

A total of 18,225 liters (4,815 gallons) of No. 6 fuel oil from Apex Oil Co. of St. Louis, MO, arrived March 31, 1994. This was blended in Tank 5 with the remaining unhydrotreated start-up oil from the previous POC run to make a single 30,280 liters (8,000 gallon) batch of start-up oil for this run. The target operating conditions are listed in Table 2.14.

Start-Up

Oil flows to the unit were started May 19, 1994, with the first and second stage reactors ebullated that day. The unit was then lined out with the reactors at about 343 °C (650°F) and 18.6 MPa (2,700 psig). These conditions were maintained until May 26, 1994, when all of the available start-up oil had been hydrotreated and distilled, making a 343°C+ (650°F+) hydrotreated make-up oil.

The catalyst from both the first and second stage reactors was then removed via the catalyst withdrawal system. Catalyst from the first proof-of-concept run daily catalyst withdrawals were then added to these reactors.

Oil flows to the unit were resumed May 30, 1994, with the new catalyst beds ebullated May 31. DMDS injection was begun May 31 and continued throughout the run except during occasional repairs to the pump. Sulfur breakthrough at 232 °C (450°F) and 343 °C (650°F) was confirmed before coal processing began at 0400 hours June 1, marking the beginning of Period 1.

The filter was successfully operated May 30, June 1 and June 2, 1994. Approximately 1,860 kg (4,100 pounds) of vacuum bottoms from Run 4 Periods 3-5 were processed with 145 kg (320 pounds) of a 50 W% filter aid solution, May 30. Additional filter aid was added to the second and third filtration cycles. A definitive cake and more than 454 kg (1,000 pounds) of filtrate were produced from each cycle. Definitive cakes were produced in each cycle with the first cake in excess of 227 kg (500 pounds) and the third cake equaling 103 kg (227 pounds).

Condition 1A, Periods 1-6

Unit conditions were adjusted during Periods 1 and 2 for Condition 1A. These were a 320 $kg/h/m^3$ (20 $lb/hr/ft^3$) space velocity, 399 °C (750°F) first stage, 435 °C (815°F) second stage temperature, with the hydrotreater on-line. Ashy recycle was used in Periods 1 and 2 to help build up the ash concentration in the system.

There were 4 minor coal outages during Condition 1A, totaling about 5.5 hours. In each case, when the oil to coal ratio was lowered much below 1.1, the slurry mix tank circulating pumps had trouble providing slurry to the reactor feed pumps. The initial pumping issues were related to the new recycling scheme which has the ASBs recycling to the COT instead of O-41. Only O-46 recycle (ashy recycle) was entering O-41 until Period 3, because all VSOs from the unit were going to O-42 to build-up the purge/flush oil inventory. Then in Period 3, VSO and DAO both became available at a rate sufficient to meet the unit's recycle slurry needs. However, the time lag in O-41 and O-43 meant that for a couple of hours, the recycle solvent was high in solids, since the material in O-43 actually came from O-46 and had little or no VSO or DAO. A procedural change to prefill O-42 and to initialize VSO recycle in Period 1 should help this issue.

Regular scheduled catalyst additions and withdrawals were conducted during Condition 1. Catalyst was added to the first stage reactor in Periods 1, 3 and 5 while the second stage reactor received catalyst in Periods 2, 4 and 5. Catalyst was withdrawn from the first stage reactor in Periods 1 and 3 and from the second stage in Periods 2 and 4. In general, additions and withdrawals proceeded normally throughout the entire campaign, albeit at times the amount of withdrawn catalyst was smaller than expected.

The number one make-up hydrogen compressor failed at 0547 hours of Period 3. A bolt inside the gear box broke and had to be replaced. This compressor was returned to service at 2244 hours of Period 3 after a new bolt was obtained from Norwalk and the repairs were completed. During this compressor outage, the coal rate to the unit was lowered to 61 kg/h (135 pph) (MF basis) to the hydrogen consumption to the supply.

The hydrotreater inlet temperature was initially above the target of about 327 °C (620°F). Hydrotreater feed (O-12 liquid) was established at 1130 hours of Period 2. This flow was not sufficient to lower the hydrotreater internal temperature to 357°C (675°F). A new recycle loop, recycling NSBs to the alternate hydrotreater feed tank, was installed and commissioned at 1715 hours of Period 3, which can now permits easy control of the hydrotreater temperatures. Nitrogen and sulfur levels in the Period 2 sample were below 1 ppm and 17 ppm, respectively.

The recycle gas flow to the second stage reactor was accidently shut off at 0847 hours of Period 4. This was discovered during Period 4B.

The hot separator level control valve failed early in Period 5. The outlet nozzle, which is equipped with tungsten carbide inserts, appears to have eroded through after several years of service. Operations put the on-line spare valve into service and replaced the failed valve with the off-line spare.

The unit was shutdown at 0900 hours of Period 6 (June 6, 1994) after a nearly 2.8 MPa (400 psi) pressure drop developed during Period 5 in the high pressure section after the first stage reactor. Inspections and later operations in this run indicated that this pressure was probably due to ammonium salt deposit. A new water injection port was installed at the hydrotreater exit during this shutdown.

<u>Condition 1B, Periods 7-15</u>

Unit operations were resumed June 9 after completing inspections of the high pressure section, reassembly, insulation and pressure checking the unit. Coal processing resumed at 0400 hours on June 11. Operating conditions were adjusted during Periods 7 and 8 for Condition 1B. Condition 1B was a repeat of Condition 1A , but with ashy recycle.

The transfer line between the ROSE first stage settler and the bottoms receiver plugged in Period 7B. This area was cleared and returned to service June 13. The ROSE section was onstream during the remainder of this condition, except for a two hour and a 5 hour outage in Periods 8 and 15, respectively. Approximately 41 kg/h (90 lb/hr) of feed were processed. The solids product was mostly a fine powder.

During period 9 (0400 June 13 to 0400 June 14) the following significant items occurred:

- The on-line recycle gas compressor coupling broke, causing a slight upset to the unit pressure until the spare compressor was placed on-line.
- The hot separator letdown valve trim failed. When the backup valve was put into service it leaked, and the separator level had to be controlled manually from 1130 to 1520 hours while repairs were made.
- An electrical power failure occurred at 1500 hours. The spare electrical service picked up automatically.
- The fresh feed preheater would not restart after power had been restored. It was returned to service at 1611 hours.
- Coal feed was suspended from 1610 to 1807 hours due to the preheater status.
- Feed to the vacuum tower was interrupted from 1800 to 2117 hours due to a plugged downleg.

A slow increase in the unit overall differential pressure was observed until Period 11, June 15. At that time, the differential pressure rose to over 1.7 MPa (250 psi) versus 0.69 MPa (100 psi) at the beginning of Period 11. A restriction appeared to be located downstream of the hydrotreater. The issue was resolved by lowering the pH of the injected water and switching the source of the injected water from city water to boiler discharge water.

The hydrotreater temperature was raised from 360 °C (680°F) to about 377 °C (710°F) during Condition 1B. This change was made to improve the hydrotreater performance.

A first stage reactor liquid sample was taken in Period 15. However, the drain line between the isolation vessel and the sample receiver was restricted. A total of 2,139 grams was recovered after Period 22.

The number 1 hydrogen compressor failed at the beginning of Period 15, June 19. Because only one compressor was available, the coal feed rate was reduced 10% to 72.6 kg/h (160 pounds per hour) (wet basis). The second to third stage interstage valves had to be replaced. These repairs were completed in Period 17.

All catalyst additions and withdrawals were smooth and on schedule throughout Condition 1B.

Condition 2A, Periods 16-17

Condition 2A was a two day extension of Condition 1B with a reduced feed rate of 72.6 kg/h (160 pph) wet. The above mentioned make-up compressor was repaired during this extension.

At 0030 hours of Period 17, the vacuum still downleg became restricted with heavy coal-derived material, probably due to the low throughput. This line was cleared and the vacuum still was returned to service in about 4 hours.

The ROSE section operated continuously throughout Condition 2A, except for about 6 hours from 0010 hours to 0610 hours June 22. In general, the bottoms product continued to be a dry powder.

Condition 2B, Periods 18-22

Once the make-up compressor became available, the coal rate was returned to a 320 $kg/h/m^3$ (20 $lb/h/ft^3$) space velocity from about 18. The transition also included raising the first stage temperature 11°C (20°F) to 413°C (775°F) and the second stage temperature 8°C (15°F) to 443°C (830°F). This condition change was completed smoothly in Period 18.

The PPI recycle gas compressor broke a diaphragm in Period 18. This compressor was repaired and returned to service without adversely affecting the unit operation. The Joy recycle gas compressor was utilized to maintain recycle gas flows while repairs to the PPI were completed. The overall unit pressure drop increased from about 275 Pa (40 psi) to about 550 Pa (80 psi) during Period 18. Acid treating the injected water in Period 19 returned this pressure drop to about 275 Pa (40 psi).

The ROSE section operated continuously from 1400 hours Period 19 to 1505 hours Period 21 June 25. The solvent-feed mixer and downstream transfer line became restricted at that time with carbonaceous material. The mixer and the transfer line were replaced June 27.

The hot separator level control was inconsistent during Period 22A. Operations switched over to the spare valve at 1845 hours Period 22. Level control then stabilized.

To compensate for the loss of resid in the recycle stream while the ROSE section was off-line, the amount of ashy recycle was increased from 20% to 30 W% of the coal feed in Period 22. This reduced the amount of material which was sent to the vacuum still to the point that it was decided to batch operate the vacuum still until the vacuum tower feed rate increased.

Coal processing was suspended at 0700 hours June 27 after the second stage reactor ebullating oil flow was lost. This flow initially decreased at about 2230 hours Period 22. It returned to normal within two hours, but then was lost at about 0630 hours Period 23. The first stage was not affected by these events.

Periods 23-26A

Coal processing was resumed at 2100 hours July 3, 1994, after the second stage reactor ebullating line was cleared. Refer to Section IV for additional details regarding this shutdown. For accounting purposes this was considered Period 23. The restart proceeded well with the two ebullated bed reactors at 413 °C (775°F) and 427 °C (800°F), respectively, processing Black Thunder coal at a 320 kg/h/m^3 (20 lb/h/ft^3) space velocity. During the restart, approximately 10% of the first and second stage catalyst beds was removed to reduce the reactor catalyst inventory to a 90% loading.

The ROSE section was restarted about 1300 hours July 4, 1994, and operated well throughout these 4 periods.

It was originally planned to operate the filter in Period 23 for an extended operating period. Two filter operations were conducted between 1730 hours Period 23 and 0500 hours Period 24. The filtration rate was very slow in both operations, i.e., about 91 kg (200 pounds) of filtrate per hour was recovered. Sluicing of the filter leaves was performed to no avail. It appears that material restricted the filter leaves, probably from the previous operation (prior to Period 1), since several of the filter transfer lines also needed to be cleaned before this testing could be accomplished.

Period 25 was proceeding well when at 1639 hours, HTI experienced an electrical power failure. The automatic switch gear to the facility changed over to the reserved power supply, limiting the power failure to approximately 10 seconds. Per the emergency power failure procedure, coal feed to the unit was discontinued until unit conditions were lined out. Operations was able to restart the major rotating equipment, but ebullation to the second stage was not recovered immediately. This was probably due to the

degassing effect of the second stage reactor which occurs on loss of the make-up or recycle gas compressor. The second stage reactor was re-ebullated at 1930 hours, and coal operations were resumed at 0400 hours Period 26.

The alternate hydrotreater feed line became restricted at 1045 hours Period 26, July 6, when an electrical resistance winding shorted out, causing material in this line to coke. It was then decided to shut down the unit, depressure, repair this line and then restart. The other option would have been to discontinue hydrotreating the alternate hydrotreater feed material. This shutdown was completed 0325 hours July 7, 1994, with the repairs completed by 1635 hours the same day.

The ROSE section operated throughout this reporting period utilizing pentane solvent to recover heavy oils from our vacuum bottoms stream. Approximately 113 kg (250 pounds) of solvent was being lost each day in this section. Prior to Period 23, it was thought that the PSV on O-67 was prematurely opening, venting solvent to the flare header. This PSV was inspected during the Period 23 shutdown. It did not show signs of wear and it did not operate prematurely on the bench. Pentane was later found in the filter/ROSE section vacuum pump. This evidence suggests that pentane was being vaporized by the vacuum pump and not being recovered.

Conditions 4A, 4B and 4C, Periods 27-33

Unit operations were resumed after completing repairs to the hydrotreater alternate feed line. Coal processing was resumed at 1400 hours July 8, 1994. Operating conditions were adjusted gradually during Periods 27 to 33 with lined out operations of 12 to 24 hours per Table 2.14. These lined-out operations were considered Conditions 4A, 4B and 4C. These conditions provided process performance data between 480 kg/h/m^3 (30 lb/h/ft^3) and 640 kg/h/m^3 (40 lb/h/f^{t3}) space velocity. The hydrotreater temperature was raised to 382°C (720°F) in Period 29.

The fresh feed preheater was lost at 1430 hours Period 30, when the high range for the thermal cutout device was being adjusted. This device was set to shut down the heater at 593°C (1,100°F), and the thermocouple was reading 574°C (1,066°F) just about an hour before the effort to reset the device was made. None of the heater coil temperatures were above 427 °C (800°F) during the incident. The heater was returned to service at 1700 hours. Coal feed to the unit was suspended during this outage and not resumed until 2100 hours. Initially coal processing was at a 320 kg/h/m^3 (20 lb/h/ft^3) space velocity, but it gradually increased to a 640 kg/h/m^3 (40 lb/h/ft3) space velocity by Period 32B.

A heavy rain storm caused water to enter the heater control box in Period 32, causing the fresh feed preheater to shut down at 2100 hours. This control was bypassed, and the heater relit by 2200 hours. Operations then returned to the target operating conditions. The control box was replaced at 0800 hours Period 33. The heater had to be shutdown for about 15 minutes during this replacement.

Black Thunder coal was being processed smoothly at approximately 640 kg/h/m^3 (40 lb/hr/ft^3) space velocity in Period 33. Conditions were stable with the recycle gas rates set at about 2/3 thirds of the respective targets. Higher gas rates caused the second stage ebullating horsepower to fluctuate. The unit back pressure had been lowered to 17.6 MPa (2,550 psig) from 18.6 (2,700psig) to aid the injection of make-up hydrogen. Efforts to distribute more of the recycle gas to the second stage reactor were made in Period 34, allowing us to increase the unit back pressure by reducing the fresh feed preheater pressure drop.

The hydrotreater temperature was raised to 388°C (730°F) from 377°C (710°F) during Periods 27-33. This was done to maintain the hydrotreater performance with the increasing unit space velocity.

A 1,282 gram first stage reactor liquid sample was taken in Period 33B. This was recovered after the run was completed.

The ROSE section operated throughout Conditions 4A, 4B and 4C, except for 2.6 hours in Period 32B, when the first stage settler bottom line required a cleaning. This line was flushed with vacuum gas oil about once every 2 to 4 hours in order to maintain operability during Period 33. Initially, pentane was used as the solvent; then, at 1200 hours of Period 31, toluene was added to make a 10% solution with pentane. The addition of toluene required increasing the first stage settler temperature by about 6.7 °C (12°F) in order to operate smoothly. The unit produced a dry powdery product with the mixed solvent.

The filter was reassembled and pressure checked. However, the associated vacuum pump was found to need repairs. This pump was not repaired until Run POC-02 was completed. Hence, no further filtration was conducted in this campaign.

Condition 5, Periods 34-36

The transition to Condition 5 was performed at the start of Period 34. The only operating change was to add 150 ppm of Molyvan 822 to the reactor feed. In general, operations were smooth during this condition.

The hydrotreater was taken off-line at the start of Period 34. This was done so that samples of non-hydrotreated material (O-5 and O-12) could be obtained at the end of subperiod 34A. The hydrotreater was returned to service at 393 °C (740°F) once the samples were taken.

The ROSE section operated throughout this reporting period, although the first stage settler bottoms drawoff line required several flushes, between once every 4 hours to twice an hour. Analysis of the solids indicates that the pentane insoluble content in the bottoms was consistently above 88 W% after toluene was added to the solvent.

Special sour water samples were taken in Period 36.

An additional 0.7 to 1.4 MPa (100 to 200 psi) pressure drop across the high pressure section occurred at approximately 1630 hours Period 36 and 1130 hours Period 37. Acid treatment was performed on the line between the hydrotreater and overheads cooler after each of these incidents, reducing this pressure drop to normal.

Condition 6A, Periods 37-38

The transition from Condition 5 to Condition 6 included reducing the space velocity from 640 to 480 kg/h/m^3 (40 to 30 lb/h/ft^3). By the end of Period 37, rubber was planned for up to 10% of the reactor feed. However, the Vac-u-Max transfer system malfunctioned. In essence no rubber was fed until Period 38A. This transfer system appeared to work well at first, transporting rubber into blending tank O-41 during Period 37. However, the associated transfer lines were found to be plugged with rubber at the start of Period 38. It is doubtful that any rubber got into the system during Period 37. Inspection of the device indicated that the associated air regulator needed to be replaced. Rubber and plastic slurries were then prepared manually for the duration of the run.

Condition 6 was originally scheduled as a rubber/coal co-processing demonstration during Periods 37 - 39. This was the first demonstration of this kind for HTI. The original plan was to slurry 1/4-inch crumb rubber at about 177-204°C (350°F-400°F) in a 4:1 coal:coal derived oil mixture. However, when this material was first added to the slurry mix tank, between 1000 hours and 1048 hours Period 38A, the feed pump suction lines became restricted. This was at least partly due to rapid transfer of rubber into the slurry mix tank, over 91 kg (200 pounds) in less than 10 minutes. The flow control valve between O-43 and the slurry mix tank needed a stem position adjustment to pass the rubber particles. While this valve was being adjusted this rapid transfer occurred.

It was then decided to dump the contents of the mix tank and proceed to Condition 7. Rubber processing was resumed after the completion of Condition 7C with minus 30 mesh pulverized rubber.

Conditions 7A, 7B and 7C, Periods 39-43

Plastic processing began at 1426 hours Period 39. In general, Periods 39B to 41 were used to debug plastic process operating procedures. Plastic concentrations as high as 50 W% were attempted. At this point, the plastics concentration appears to be limited by the time required to prepare blends, the rise in the SMT temperature, the associated foaming and reduction in feed pump capacities. During one foaming incident, pump capacities were less than 50% of their rated values. Foaming in the SMT was an issue throughout the plastic operation. The high temperature required to melt the plastics and the associated slurry viscosity caused coal moisture vaporization and foaming in the

SMT. This has been controlled since Period 42 by lowering the SMT temperature to about 129°C (265°F) from nearly 138°C (280°F) and by lowering the inventory in this vessel.

Unit operations were smooth after the beginning of Period 42. During Period 42, the reactor feed was approximately:

-	dry coal	73 W%
-	polyethylene	16 W%
-	polystyrene	11 W%

PET was added to the feed blend at 0600 hours Period 43, and the plastics concentration was then increased to slightly more than a combined 30% as follows:

-	dry coal	69 W%
-	polyethylene	15 W%
-	polystyrene	11 W%
-	PET	5 W%

Plastic feed to the slurry mix tank was terminated at 0300 hours Period 43 after processing coal, polyethylene, polystyrene and PET for nearly 24 hours. The total plastic content of the net reactor feed exceeded 30% during Period 43B. Four to one oil:plastics blends were first prepared in O-47 and O-51, then shipped to O-43 where this material was metered into the slurry mix tank. Operations during Period 43 with the PET plastic mixture were smooth.

The hydrotreater temperature was lowered in Period 42 from 393 °C (740°F) to 382 °C (720°F). It appears that the mid-distillate sulfur and nitrogen contents decreased after the coal space velocity was reduced from 640 $kg/h/m^3$ (40 $lb/hr/ft^3$) in Period 36 to 320 in Period 37. Naphtha was not needed to control the hydrotreater temperature since 1130 hours Period 41. The mid-distillate stream was then heated to 260 °C (500°F) to help achieve the targeted reaction temperature.

Acid was added to the wash water stream twice in Period 39, once in Period 41 and once more in Period 43 to control pressure drop across the high pressure section. Each time this pressure drop returned to normal shortly thereafter.

The bottom outlet of O-41 became plugged at the end of Period 39. Two windings on the cone section failed, while a blend of plastics was being prepared. Partially melted plastics settled out and plugged the bottom of this tank. A major maintenance effort to clean this tank was required because there are several pipe connections to it. Also, the agitator needs to be repaired. When used during the first rubber blend preparation the agitator hit the internal baffles. O-51, wash oil tank, and O-47, filter feed tank, were used to prepared plastic slurries after O-41 was taken out of service.

A Eurotherm representative was on-site during Period 37 to debug the automatic rate of change control on O-43. This control loop operated well during the remainder of this run after it was put into service at 1426 hours Period 39.

The first stage reactor catalyst withdrawal valve, G, had to be replaced in Period 40. The same valve on the second stage reactor had to be replaced in Period 43. In both cases, the stem broke where it connected to the ball.

The ROSE section operated throughout this condition. Toluene was no longer added to the unit after 1610 hours Period 40. The first stage settler temperature was lowered from 214 °C (418°F) to about 197 °C (386°F). The bottoms material was hard and dry, but not powdery.

Condition 6B, Periods 44-45

Only coal and slurry oil were fed to the slurry mix tank between 0300 hours Period 43 and 1112 hours Period 44. Rubber slurries were then recycled to the slurry mix tank from O-43. These were prepared in O-51. The total rubber content of the reactor feed exceeded 25% of the net feed during Period 45. Although the feed pumps did not operate flawlessly during this rubber operation, they did perform much better than in Period 38. The two principal differences were:

- more uniform rate of charging rubber to the slurry mix tank
- the source of the rubber.

In the first attempt to feed rubber, approximately 91 kg (200 pounds) of rubber slurry was added to the mix tank in about 10 minutes. This preceded the pumping problems which occurred in Period 38.

In Period 38, nominal 0.6 cm (1/4-inch) rubber with fibers (from Colletta) was used while the rubber in Period 45 was minus 30 mesh and fiber free (from Baker).

The hydrotreater was taken off-line at 0900 hours Period 44 and returned to service at 0400 hours Period 45. This was done in order to obtain samples of non-hydrotreated materials.

The ROSE section operated throughout Condition 6B except for a 30 minute span in Period 45 and a 4 hour duration in the shutdown. An apparent first stage settler false level occur in Period 45. The second settler level control valve failed during the shutdown. ROSE operations were easily resumed after each of these outages. First stage settler temperatures as low as 191 °C (376°F) were successfully demonstrated.

D.4. Unit Inspections

<u>After Period 6</u>

The measured settled bed height was within 90% of the estimated bed height based on daily addition and withdrawal.

The recycle gas heater and the line to the first stage reactor outlet was cleared. A small amount of carbon build-up was found in the coil and in the heater outlet check valve.

Transfer lines to and from the hydrotreater were found clear. The transfer line between the first and second stage reactor was also clear.

After Period 23

Second stage reactor ebullating flow was lost at about 0630 hours June 27. The second stage reactor plenum, the ebullating pump suction and discharge lines were cleaned and flushed. Material, including catalyst, was removed from the pump suction line, pump discharge line, the associated cup and plenum chamber. This was done without removing either reactor head. The number 2 ebullating pump was pulled and replaced with the number 3 pump to allow the number 2 pump to be inspected without delaying the unit restart.

Prior to Period 23, it was thought that the PSV on O-67 was prematurely opening venting solvent to the flare header. This PSV was inspected during the Period 23 shutdown. It did not show signs of wear and it did not operate prematurely on the bench.

The filter was disassembled and inspected. Approximately 23 kg (50 pounds) of hard carbonaceous material was found on the bottom near the cake exit. Between 0.16 cm (1/16-inch) and 0.32 cm (1/8-inch) thick layer of hard carbonaceous material was found covering each of the 3 filter leaves. Five of the six sluice nozzles were found plugged with solid material. Each inter-leaf gasket was found intact and in place.

After Period 45

During shutdown, catalyst was removed from both ebullated bed reactors via their respective catalyst withdrawal systems. Only 23 kg (50 lbs) and 82 kg (180 lbs) of oil soaked catalyst were recovered from the first and second stage reactors, respectively.

D.5. Mechanical History

After Period 6

There was approximately a 6.1 m (20-foot) portion of transfer line between the hydrotreater and the overheads cooler which could not be flushed with water to prevent salt formation. A new water injection tap was added at the outlet of the hydrotreater during this shutdown which allowed this section of line to be flushed with water.

A bad computer board in the process control system was replaced by DEC June 10, 1994. There were several computer outages June 10 and 11, 1994, related to this matter. Unit operations were not adversely affected by this issue.

After Period 23

Two new valve bodies were made for the hot separator level control valve. These were placed in service during this shutdown.

Period 37

A meeting was held with our heater control suppliers to discuss the heater operation and the thermal cut-out device. After this run HTI relocated the heater control box into a weatherproof enclosure. The supplier will modified one heater to operate with fixed air and throttled fuel gas. This should provide more uniform heater operation since the flame height should be more constant.

Regarding the preheater thermal cut out device, the high range is the maximum temperature at which the high temperature shutdown switch can be set. Hence, this parameter, which was currently set at 760 °C (1,400°F) after Period 30, can be set at any temperature up to the limit of the thermocouple, about 871 °C (1,600°F) in this case. The shutdown alarm was reset to 649 °C (1,200°F). These settings were adequate to protect the heater box and not cause premature heater shutdowns during the remainder of the run.

D.6. Recommendation on Procedural and Unit Modification

Recommended Maintenance Items for Run POC-03 and Beyond

Item	Description
1	The slurry mix tank mistkop is very difficult to get to and can not be cleaned without disassembly. It will be cleaned, possibly relocated and modified to facilitate cleaning. Installation of a inlet filter which can be easily remove should resolve this issue.
2a	The slurry mix tank circulating pumps will be maintenanced.
2b	Replacement pumps for the slurry mix tank circulating pumps will be specified. New pumps will not be purchased at this time.
3	The slurry mix tank agitator will be evaluated and potentially replaced after Run 6.
4a	Modified preheater controls will be installed per the Period 37 meeting with the vendor (See section IV).
4b	The fresh feed preheater pressure data from Run 5 will be evaluated to determine the coil diameter for Run 6.
5	The first stage sample tap will be relocated to the side of ebullating oil line. The sample vessel drain line will be relocated to remove an approximate 6-inch dead line.
6	Mogas bi-directional valves have been specified to replace the Rockwell plug valves in ebullating pump discharge line. Purchase of these valves were not to occur before run 6.
7	An evaluation of installing a plenum drain line has been conducted. This installation is not currently recommended.

8 A first stage reactor emergency depressuring line will be engineered and probably installed before the next run.

9 Recycle gas impulse lines on hydrotreater pressure sensing lines will be installed. This will facilitate clearing these lines of accumulated liquid as-needed to minimize false pressure data.

10 The ebullating pumps used in Run POC-02 will be inspected and repaired, as-required.

11 The water injection pumps will be rebuilt with diaphragms. Acetic acid in water increased the maintenance that the piston pump heads required.

12 The catalyst addition and withdrawal valves will be inspected and repaired as-required before the next run.

13 The direct piping route between O-13 and N-3 (vacuum still) will be reactivated for the next run.

14 Several vessels will be opened and inspected including O-1, O-5, O-41, O-43, O-47, O-50, O-51, O-60, O-61, N-1 and N-3.

15 The recycle oil system needs to be de-bottlenecked for future runs. This may include replacing O-43 with larger vessel with conical bottom, agitator and centrifugal pump (probably Durco).

16 Oil soaked insulation on L-1, N-3, O-41, O-43, O-46, O-50, O-51, O-60, O-61, P-3 and P-4 will be replaced.

17 The in-line Cuno filter in flush oil system between O-42 and O-40 will be repaired.

18 Tungsten carbide trims have been ordered and will be installed in the second stage settler LCV.

19 The vacuum still vacuum pump will be replaced.

20 A evaluation of the ROSE section solvent vapor recovery will be conducted. The solution will probably include a solvent vapor condenser and a recommendation to install the same for POC-4.

21 The transfer line between O-60 and O-61 needs to be repiped so that it self drains.

22 An automatic plastic/rubber feed system for O-47 will be engineered. This will not be installed until plans for a major plastic/rubber with coal test is scheduled.

23 Ventilation fans will be installed in the O-81 area.

24 O-44 control instrumentation has finally arrived and will be installed.

25 The current O-1 letdown valve body design has been evaluated. A new valve body design has been engineered and will be incorporated after appropriate approvals are received. This should be completed before the next run.

TABLE 2.14

TARGET OPERATING CONDITIONS - POC-02 (260-05)

PERIODS	CONDITION	SPACE VELOCITY KG/HR/M³ (LB/HR/FT³)	REACTOR TEMP °C (°F) K-1	REACTOR TEMP °C (°F) K-2	COAL RATIO	METHOD OF RECYCLE TO REMOVAL	CATALYST REPLACEMENT SOLIDS K-1	CATALYST REPLACEMENT RATE, #/TON K-2
3-6	1A	320 (20)	399 (750)	435 (815)	1.0	ROSE	1.0	2.0
9-15	1B	320 (20)	399 (750)	435 (815)	1.2	ROSE	1.0	2.0
16-17	2A	320 (20)	399 (750)	435 (815)	1.2	ROSE	1.0	2.0
18-22	2B	480 (30)	413 (775)	443 (830)	1.2	ROSE	1.0	2.0
29	4A	480 (30)	416 (780)	443 (830)	1.2	ROSE	1.5	2.5
30-31	4B	560 (35)	432 (810)	446 (835)	1.2	ROSE	1.5	2.5
32-33	4C	640 (40)	432 (810)	446 (835)	1.2	ROSE	1.5	2.5
34-36(1)	5	640 (40)	432 (810)	446 (835)	1.2	ROSE	1.5	2.5
37-38(2)	6A	480 (30)	432 (810)	443 (830)	1.2	ROSE	1.0	2.0
39-40(3)	7A	480 (30)	432 (810)	443 (830)	1.2	ROSE	1.0	2.0
41-42(4)	7B	480 (30)	432 (810)	443 (830)	1.2	ROSE	1.0	2.0
43(5)	7C	480 (30)	432 (810)	443 (830)	1.2	ROSE	1.0	2.0
44-45(1)	6B	480 (30)	432 (810)	443 (830)	1.2	ROSE	1.0	2.0

Notes:

1. 150 ppm Molyvan 822 was added to the feed during these condition.
2. Target 3:1 Coal: crumb rubber
3. Target 90W% Coal, 6W% Polyethylene and 4W% Polystyrene, Recycle: feed of 2:1
4. Target 70W% Coal, 18W% Polyethylene and 12W% Polystyrene, Recycle: feed of 2:1
5. Target 70W% Coal, 15W% Polyethylene and 10W% Polystyrene, 5W% PET, Recycle: feed of 2:1
6. 20% ashy recycle from O-46 done in Period 1,2,7-36.

E. Summary of POC-02 Material Balance

E.1. Material Balance Methods

The material balance is calculated on two bases: as an overall balance and as a liquefaction section balance. The overall balance is calculated on a net basis after all recycle streams and inventory changes are included. The streams out are actual net balances; however, it is possible for a net balance to be negative. This is caused by using more of a particular stream for recycle than was produced due to the inventory reserves of the various vessels. The liquefaction balance is from the slurry mix tank to the bottoms from O-13 flash drum. The streams that are being recycled, such as VSOH, VSB, ROSE-SR DAO and ASB, are included in this section as input and product streams (Figure 2.14).

The solvent balance calculation includes all the inventory changes in the vacuum still feed vessel, O-13 bottoms vessel, recycle oil vessels, ROSE-SR feed vessels and ROSE-SR DAO vessel, as well as the net VSOH, net ASB and makeup oil. The solvent to coal ratio is based on the dry coal feed and the oil streams that are fed through the SMT (ie. O-43 recycle, ASB from the COT, and makeup oil).

The recycle rates presented in the material balance summary require an additional explanation. The ASB's are recycled through the COT and are handled separately from the rest of the recycle streams. The other four recycle streams are all sent to O-41 then O-43 and finally to the SMT. Since some of these streams are batch transferred into O-41 and some are continuous and O-41 is batch transferred to O-43 which feeds continuously to the SMT, it is extremely cumbersome to calculate the exact composition of the flow from O-43 to the SMT. The recycle rates for these four streams sum to the actual total recycle rate measured for O-43; however, the individual component rates out of O-43 are taken as their proportional rate fed into O-41. During smooth operations, line-outs and work-up periods, this estimation should be reasonably accurate.

For periods 39-45 waste was also fed to the reactors as part of the recycle stream through tank O-43. This waste, consisting of either plastics or rubber tires, was initially batch slurried in tank O-41, but due to difficulties these slurries were later mixed in tanks O-47 and O-51. Material from O-47 was batch transferred to O-51 and then batch transferred to O-43, where it was continuously fed to the SMT. The oil used to form these slurries came from tank O-42, the COT; make-up oil or various blends of these three oils. The COT normally contains only ASB; however, during these periods frequent transfers of both O-42 oil and make-up oil were made into the COT. Tank O-42 normally contains only VSO; however, during this period all DAO produced was sent here as well as occasional influxes of make-up oil. Since all these different blends of oil were used and mixed to make the waste/oil slurries, it becomes very cumbersome to keep track of the changing concentrations of not only the plastics and rubber being fed from O-43 to the SMT but of the various oil streams as well. Additionally some data was missing from the logbook and logsheets concerning these feed operations and some assumptions were made based upon the two minute data available from the database. The total coal feed net value is actually a composite value consisting of the total coal feed as well as the total waste feed. The oil feed values are correct; however, due to the way the slurry batches were made they do not necessarily relate to the vessels that they normally do. For example, the total makeup oil is the correct value but not all of this would have flowed through the COT to the SMT as indicated in the material balance output; some actually flowed through O-47 to O-51 to O-43 to the SMT. This is also true for the VSO, DAO and ASB that are recycled.

E.2. End of Run Material Balance Summary

A summary of the entire run is presented in Tables 2.15 through 2.18. The first two Tables (2.15 and 2.16) cover the entire run (Periods 1-45). The overall material balance for the run is shown as 99.56 wt%. This value is based on the actual total feed and product values. The value of 100.33 wt% presented as the average in the figure is really the average of the individually daily calculated overall recoveries. The total wet coal fed to the unit is 98,000 kg (108 tons), or 87,000 kg (96 tons) on a dry basis. The solvent balance over the course of the run is -7 tons. The liquefaction section balance shows an overall recovery of 100.17 wt%, based on actual total feed and product weights as opposed to the average of the individually calculated recoveries presented in the figure of 100.5 wt%.

The next two tables (2.17 and 2.18) present the same data but only for Periods 1-38 the periods when only coal was fed to the unit. The overall balance is 100.89 wt%, while the liquefaction balance is 100.99 wt%. The net solvent balance for these periods is 52 kg (114 lbs), showing a virtually perfect solvent balance for these coal periods.

E.3. Summary of Operating Conditions

Figure 2.15 shows reactor temperatures during the course of POC-02 operations. As can be seen from the figure, the periods where the temperature dropped (i.e., Periods 6, 22 and 26) are when shutdowns occurred; otherwise, reactor temperature profiles were fairly smooth. Figure 2.16 shows the space velocity, which reached a maximum of 619 kg/h/m^3 (38.7 lb/h/ft^3) in Period 35. The material balance recovery, as shown in Figures 2.17 and 2.18, for both the overall plant and the liquefaction section, is excellent for most of the run after an initial few days of settling down. The extremely high recovery in Period 38 is due to the problems that arose from attempting to first run the large particle size rubber feed, which caused numerous upsets and required flushing parts of the feed and recycle systems. The solid separation material recovery Figure 2.19 shows that the ROSE-SR recovery was generally slightly higher than 100%, probably due to the small amount of solvent trapped in the ROSE-SR bottoms. The solvent to coal ratio was normally in the range of 1.2 to 1.3 during the coal-only feed periods (Figure 2.20). During the waste feed periods this was actually the solvent to coal and waste ratio, which was typically 2.4 to 2.8. The solvent balance for the entire run was -6,400 kg (-14,128 lbs). This extremely negative balance is entirely due to the waste feed periods, when the unit was far from solvent balanced. At the end of the coal only feed periods, the solvent balance was +52 kg (+114 lbs), virtually perfect solvent balance. Until the end of Condition 2A, the solvent balance continually decreased; at the beginning of Condition 2B the solvent balance increased until it finally balanced. The overall operating history for the POC-02 operations is depicted in Figure 2.21.

Following is a list of assumptions that were made in the process of performing the daily material balance calculations.

Period	Assumptions
1.	Gas Analysis not available so gas analysis from POC-01 period 1 was used.
2.	Due to problems with corrupted data in the HTA 2 minute data, logsheet entries and estimates were used to calculate flows.
3.	HTA 2 minute data for all of period 3A and the early part of 3B did not exist so results are based on logsheet entries and estimates of flows. ROSE-SR was brought on-line but since no DAO was recycled this was handled as a Vacuum Still period with the VSB to the ROSE-SR entered as VSB production. The K-1 Recycle gas low range point was not carried over from DAS to the VAX database, used period 2T value.
4.	The K-1 Recycle gas low range point was not carried over from DAS to the VAX database, used period 2T value.
5.	Handled as a ROSE-SR period even though ROSE-SR feed was cut during 5B, this is necessary so that the DAO that is recycled is handled properly. ROSE-SR bottom weights required more estimation than usual due to the large overlap in periods for drum collection of bottoms. The K-1 Recycle gas low range point was not carried over from DAS to the VAX database, used period 2T value.
6.	Shutdown started in Period 6A. Results are based on logsheet entries up to 1000 hours and on period 5B gas analysis.
7.	Unit restarted, no assumptions.

8. ROSE-SR feed tanks being filled but no recycle of DAO, handled as a Vacuum Still period with the VSB's into the ROSE-SR feed tanks handled as a VSB product.

9. Problems with the ROSE-SR system, handled as a Vacuum Still period. COT was filled twice with Tank-4 oil for a total make-up oil addition of 901 kg (1,986 lbs). The COT was monitored closely this period to evaluate a running total of make-up oil and ASB's that were sent to the SMT. Of the total make-up oil added to the COT only 534 kg (1,177 lbs) were sent to the SMT.

10. Even though no additional make-up oil was brought into the COT during this period there was a residual concentration of make-up oil left from Period 9. This was followed through the COT during the period and took until nearly the end of the period for it to flush through.

11. Two major holes in the HTA 2 minute data require that the logsheet entries and estimates be used for some of the flows. This is especially true for the DAO being recycled to O-41.

12. Due to a change in the correlation equations that was made Thursday the weigh scale values in the HTA 2 minute data are not reliable. This predominantly effected calculations concerning O-46, O-60 and O-61. Some of these values were estimated for this period.

13. The COT was filled during the B period with 251 kg (553 lbs) of Tank 4 oil of which only 58 kg (128 lbs) was introduced to the SMT.

14. The COT was filled during the B period with 263 kg (580 lbs) of Tank 4 oil. From the previous period there was 193 kg (425 lbs) of makeup oil still in the SMT for a total of 456 kg (1,005 lbs) this period. 254 kg (559 lbs) of the makeup oil was introduced to the SMT.

15. 202 kg (446 lbs) of makeup oil was still in the COT at the beginning of this period left from the previous periods. 123 kg (271 lbs) of this

makeup oil was introduced to the SMT. One drum of ROSE-SR bottoms collected was flush oil and was not included in the ROSE-SR balance calculations. K-1 ebullating oil flow rate from the high range transmitter was abnormal, forced material balance program to use the low range transmitter signal.

16. The 79 kg (175 lbs) of makeup oil left in the COT from previous periods finally flushed through and was introduced to the SMT. K-1 ebullating oil flow rate from the high range transmitter was abnormal, forced material balance program to use the low range transmitter signal.

17. ROSE-SR bottoms drums are now being removed exactly at the period breaks and not a few hours before or after so the bottoms no longer need to be pro-rated to cover those drums that split periods.

18. Weigh scales for O-46 to O-50 batch transfers no longer agree reasonably well while for O-46 to O-41 batch transfers the weigh scales still agree reasonably well. Using the O-46 weigh scale to measure transfers and not the O-50 weigh scale.

19. Another drum of flush oil was removed as ROSE-SR bottoms and was not included in the balance calculations. K-1 and K-2 recycle gas low range points are no longer being carried over from the DAS to the VAX database, using the numbers from period 18T.

20. K-1 and K-2 recycle gas low range points are no longer being carried over from the DAS to the VAX database, using the numbers from period 18T.

21. K-1 and K-2 recycle gas low range points are no longer being carried over from the DAS to the VAX database, using the numbers from period 18T. K-1 ebullating oil flow rate from the high range transmitter was abnormal, forced material balance program to use the low range transmitter signal.

22. K-1 and K-2 recycle gas low range points are no longer being carried over from the DAS to the VAX database, using the numbers from period 18T. Shutdown at the end of the period. K-1 ebullating oil

flow rate from the high range transmitter was abnormal, forced material balance program to use the low range transmitter signal.

23. Coal was introduced to the unit at 9:00 PM so only a B period was calculated. No gas analysis was available so Period 24 gas analysis was used. 311 kg (686 lbs) of makeup oil was brought into the COT at the beginning of the period, and since the COT rate was so high and this was a restart it was simply assumed that all 311 kg (686 lbs) passed through to the SMT during this period.

24. ROSE-SR was started this period but since the O-65 recycle back to O-41 was insignificant, this period was handled as a Vacuum Still period so that the recycle flows would be correctly calculated. The material transferred to the ROSE-SR feed tanks was taken as the VSB product. K-1 ebullating oil flow rate from the high range transmitter was abnormal, forced material balance program to use the low range transmitter signal.

25. Due to operation difficulties, coal was cut to the unit beginning at 34 minutes into the B period; so this was handled as an A period only. Gas analysis from period A was used. K-1 recycle gas high range signal was not carried over from DAS to the VAX database, used Period 24T value. K-1 ebullating oil flow rate from the high range transmitter was abnormal, forced material balance program to use the low range transmitter signal.

26. Unit shutdown began during the A period; so no B period was included. Period 25A gas analysis was used. Unit was restarted a few days later as Period 26B; so that Period 26T is actually a composite period based on these two different half days of operation.

27. No assumptions.

28. No assumptions.

29. K-1 recycle gas high range signal was not carried over from DAS to the VAX database, used Period 28T value.

30. 1,030 kg (2,271 lbs) of Tank 4 makeup oil was added to the COT; 547 kg (1,206 lbs) of which were introduced to the SMT during this period.

K-1 recycle gas low range signal was off for this period, used the high range signal instead.

31. The rest of the makeup that was added during Period 30 was introduced to the SMT during this period.

32. K-1 recycle gas low range signal was off for this period, used the high range signal instead.

33. 971 kg (2,140 lbs) of Tank 4 makeup oil was added to the COT; 639 kg (1,408 lbs) of which were introduced to the SMT during this period. K-1 recycle gas low range signal was off for this period, used the high range signal instead.

34. 332 kg (732 lbs) of makeup oil originally brought into the COT during Period 33 was introduced to the SMT. The VSB'S collected were included as an artificial level increase in the holding tanks for the ROSE-SR feed.

35. The VSB'S collected were included as an artificial level increase in the holding tanks for the ROSE-SR feed.

36. The VSB'S collected were included as an artificial level increase in the holding tanks for the ROSE-SR feed.

37. 856 kg (1,887 lbs) of Tank 4 makeup oil was added to the COT; 757 kg (1,668 lbs) of which were introduced to the SMT during this period.

38. From 13:52 to 23:06 hours there was no coal feed to the unit. NSB, ASB, VSO and Sour H_2O rates were only available for 8 hours in Period A; so this was prorated for the other 4 hours. The VSB'S collected were included as an artificial level increase in the holding tanks for the ROSE-SR feed.

39-45. Waste was fed to the unit during these periods. The wet coal feed value entered into the database is actually a composite number consisting of both the coal feed and the waste feed. The calculated flow for the various waste feeds is given in Table 2.19.

TABLE 2.15
Overall Material Balance Recovery

	Kilograms
STREAMS IN	
Coal Feed Wet	98502
Make-Up Oil	15893
Mix Tank Inv Loss	298
Seal Oil	2200
Water Injected	55761
Fresh Hydrogen	8907
DMDS	1606
TOTAL FEED	183165
STREAMS OUT	
Vent Gas	2546
Bottoms Gas	13051
Mix Tank Vent Drain	92
Unit Knockouts	1283
NSB	48001
ASB & COT Inv	953
Sour Water	87152
VSOH & O-42 Inv	665
VSB	3718
ROSE-SR DAO	71
ROSE-SR Bottoms	17037
ROSE-SR Inv	5573
Recycle Oil Inv	521
VS Feed Inv	1001
RLFVB Inv	700
TOTAL PRODUCT	182365
OVERALL RECOVERY, WT%	99.56
SOLVENT BALANCE	-6408

TABLE 2.16
Liquefaction Material Balance Recovery

	Kilograms
STREAMS IN	
Coal Feed Wet	98502
Oil Streams to SMT	0
O-43 Recycle	65089
Make-Up Oil	15893
ASB	59821
Mix Tank Inv Loss	298
Seal Oil	2200
VSO to Purge Pump	8857
Make-Up to Purge	0
Water Injected	55761
Fresh Hydrogen	8907
DMDS	1606
TOTAL FEED	316933
STREAMS OUT	
Vent Gas	2546
Bottoms Gas	13051
Mix Tank Vent Drain	92
Unit Knockouts	1283
NSB	48001
ASB	60774
Sour Water	87152
RLFVB	104580
TOTAL PRODUCT	317480
LIQUEFACTION RECOVERY, WT%	100.17

TABLE 2.17
Overall Material Balance Recovery From 'Coal-Only' Feed Periods (1-38)

	Kilograms
STREAMS IN	
Coal Feed Wet	82402
Make-Up Oil	7265
Mix Tank Inv Loss	195
Seal Oil	1803
Water Injected	44055
Fresh Hydrogen	7243
DMDS	1347
TOTAL FEED	144310
STREAMS OUT	
Vent Gas	2050
Bottoms Gas	10382
Mix Tank Vent Drain	71
Unit Knockouts	1077
NSB	36231
ASB & COT Inv	1868
Sour Water	70835
VSOH & O-42 Inv	1712
VSB	3701
ROSE-SR DAO	25
ROSE-SR Bottoms	12838
ROSE-SR Inv	2194
Recycle Oil Inv	311
VS Feed Inv	728
RLFVB Inv	479
TOTAL PRODUCT	144502
OVERALL RECOVERY, WT%	100.89
SOLVENT BALANCE	52

TABLE 2.18
Liquefaction Balance Recovery From 'Coal-Only' Feed Periods (1-38)

	Kilograms
STREAMS IN	
Coal Feed Wet	82402
Oil Streams to SMT	
O-43 Recycle	51128
Make-Up Oil	7265
ASB	47269
Mix Tank Inv Loss	195
Seal Oil	1803
VSO to Purge Pump	7251
Make-Up to Purge	0
Water Injected	44055
Fresh Hydrogen	7243
DMDS	1347
TOTAL FEED	249958
STREAMS OUT	
Vent Gas	2050
Bottoms Gas	10382
Mix Tank Vent Drain	71
Unit Knockouts	1077
NSB	36231
ASB	49137
Sour Water	70835
RLFVB	82158
TOTAL PRODUCT	251941
LIQUEFACTION RECOVERY, WT%	100.99

TABLE 2.19
Break-down of Wet Total Feed to PDU During Periods 39-45

Period	39	40	41	42	43	44	45
Entered Wet Coal Feed, kg (lbs)	**1947 (4292)**	**2113 (4659)**	**2417 (5328)**	**2392 (5274)**	**2663 (5871)**	**2135 (4707)**	**2432 (5362)**
Actual Wet Coal	1936 (4048)	1907 (4205)	1807 (3983)	1718 (3788)	1949 (4297)	1817 (4006)	1853 (4086)
HDPE	67 (147)	124 (274)	366 (806)	404 (890)	360 (793)	27 (60)	0 (0)
PS	44 (97)	82 (180)	244 (539)	270 (596)	251 (554)	19 (42)	0 (0)
PET	0 (0)	0 (0)	0 (0)	0 (0)	103 (227)	8 (18)	0 (0)
Rubber	0 (0)	0 (0)	0 (0)	0 (0)	0 (0)	264 (581)	579 (1276)

FIGURE 2.14
Material Balance Flow Diagram

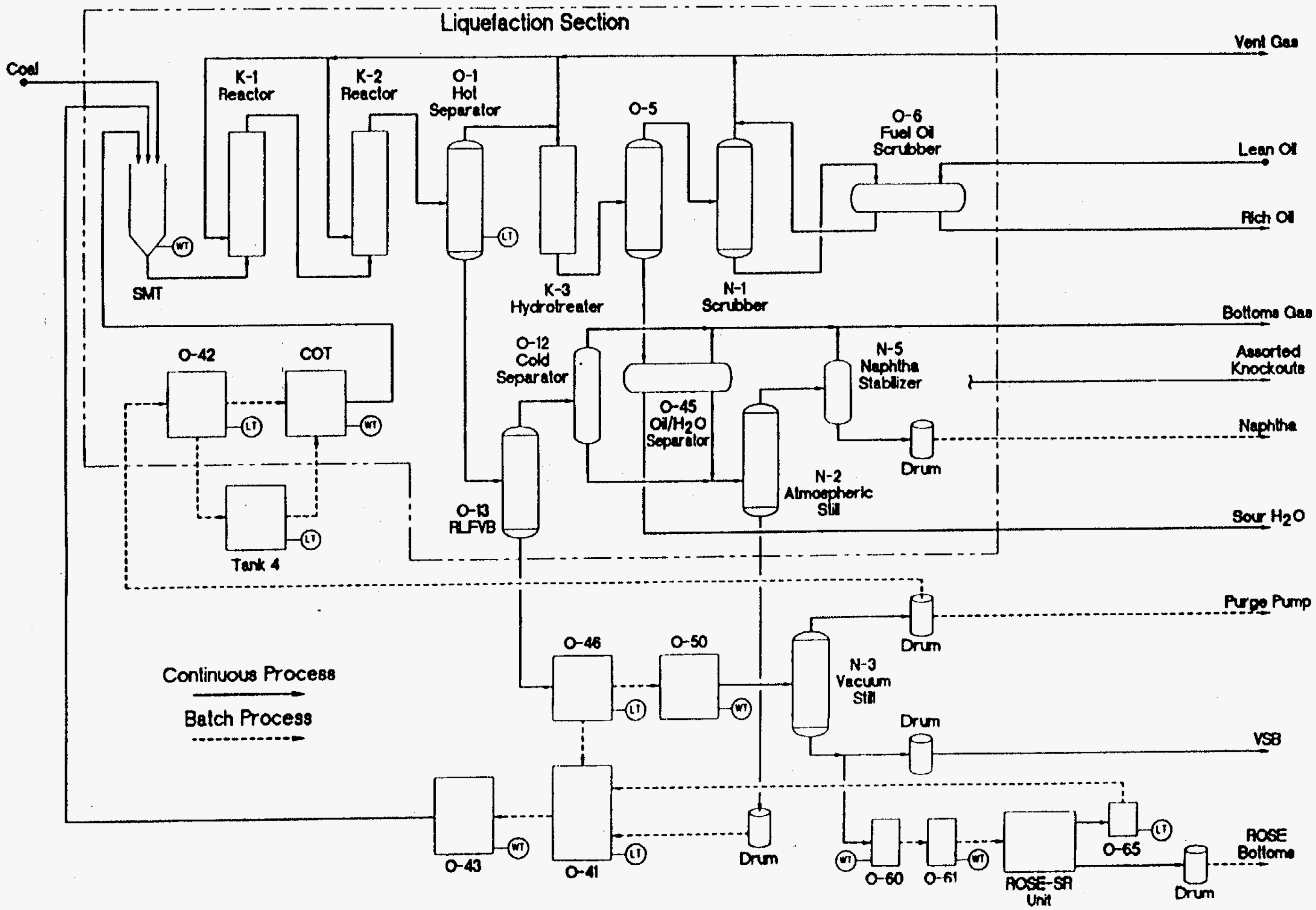


FIGURE 2.15
Average Reactor Temperatures

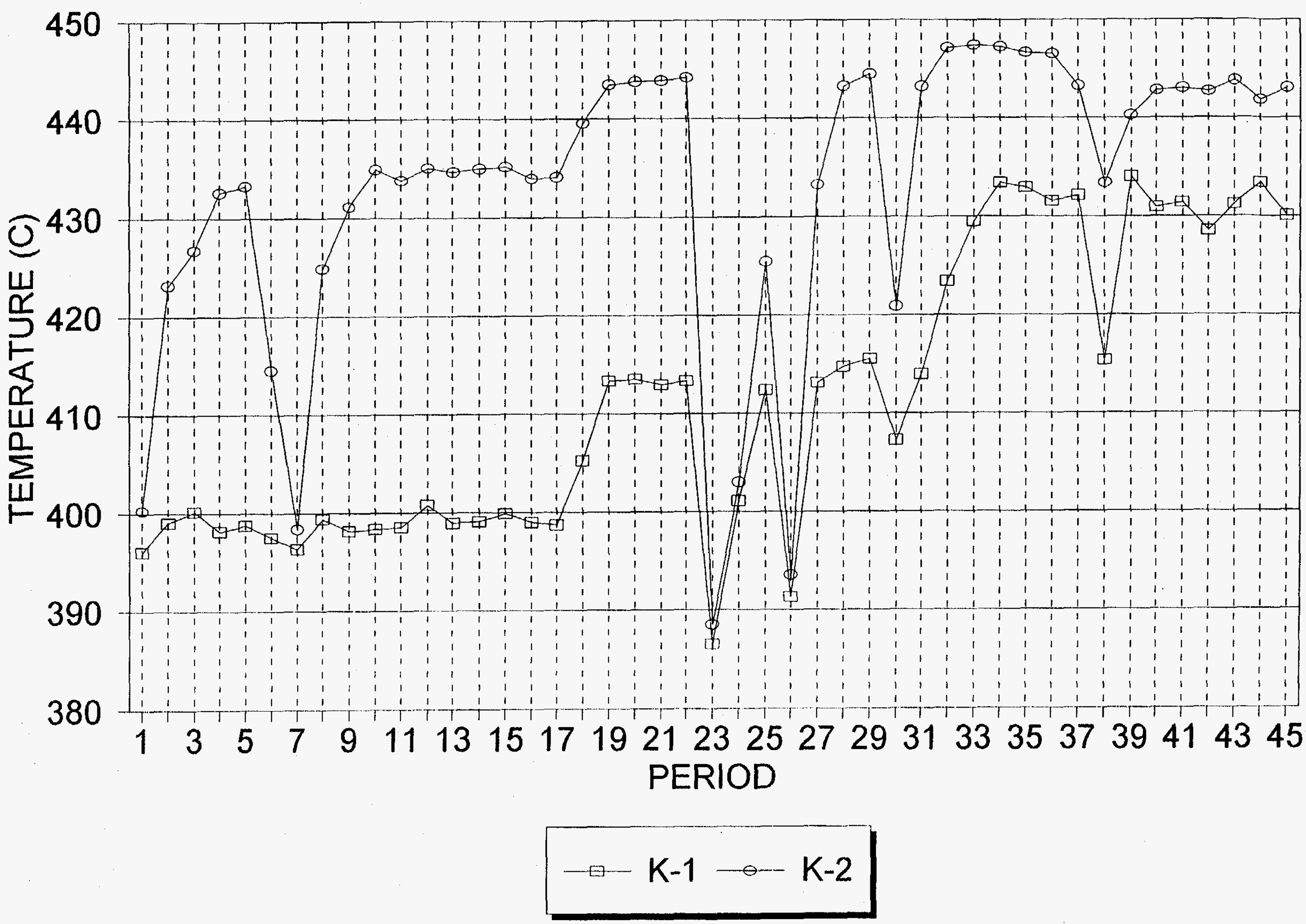


FIGURE 2.16
Feed Space Velocity (Dry Basis)

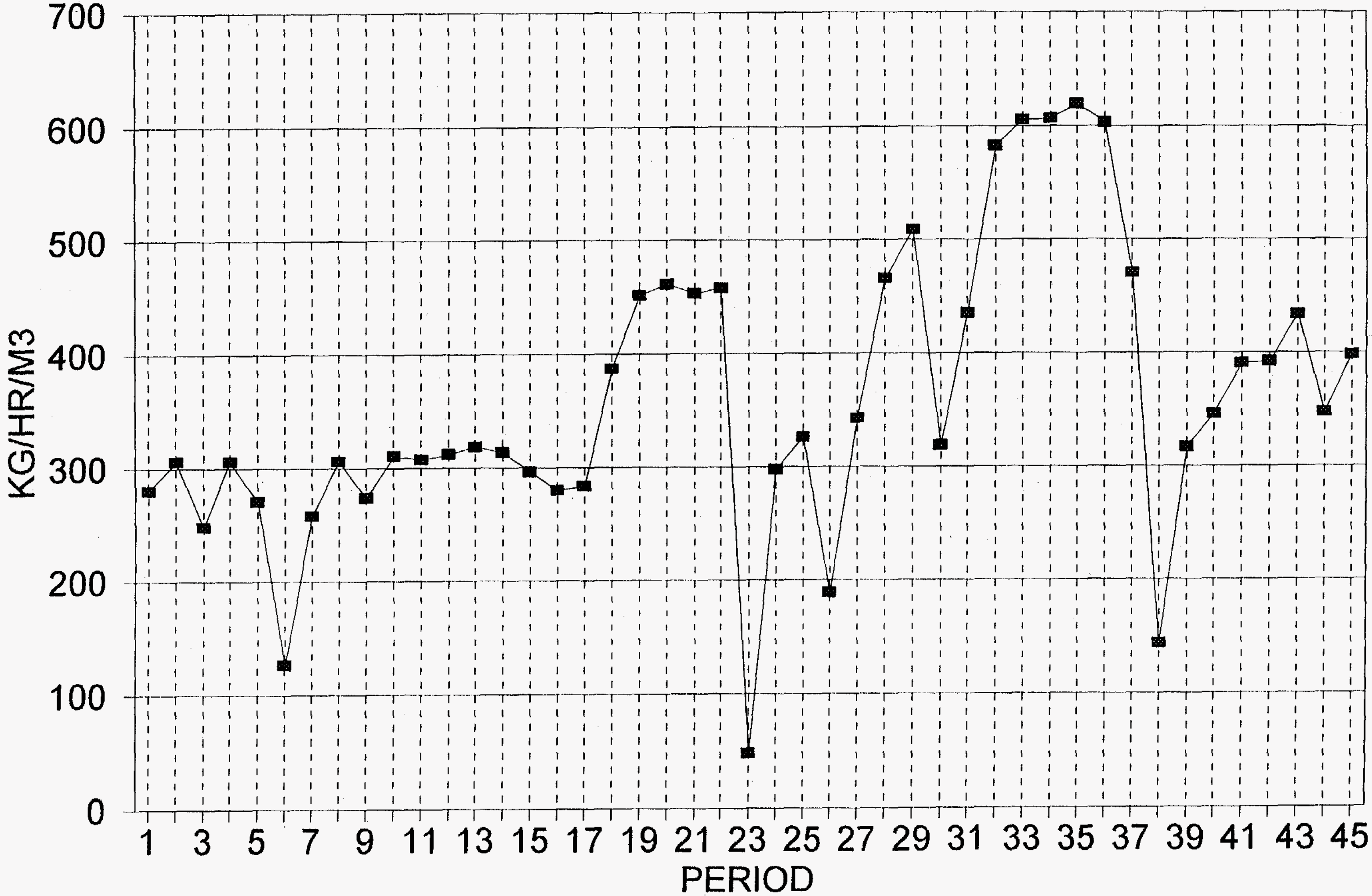


FIGURE 2.17
Daily Material Balance Recovery

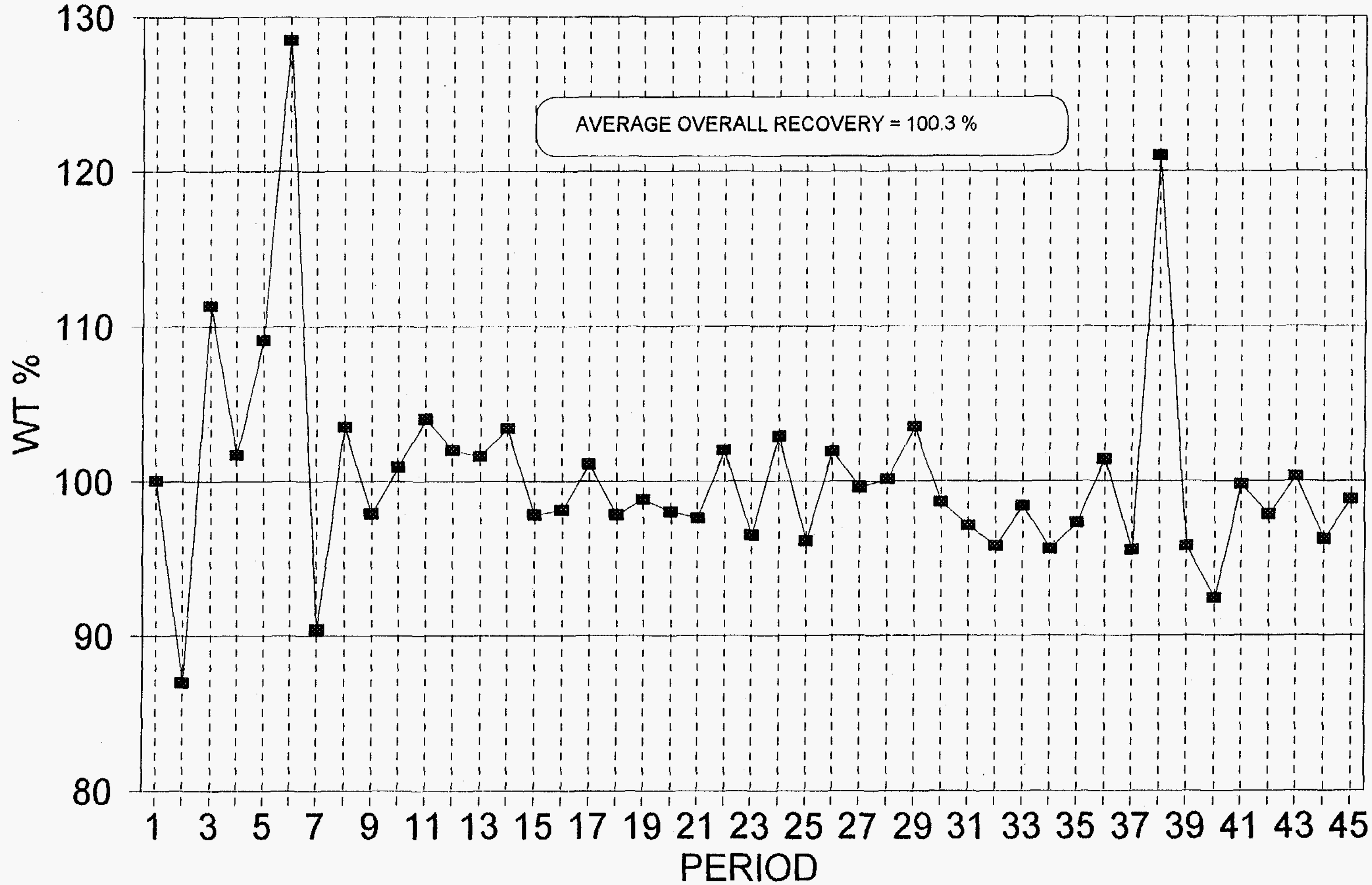


FIGURE 2.18
Liquefaction Material Balance Recovery

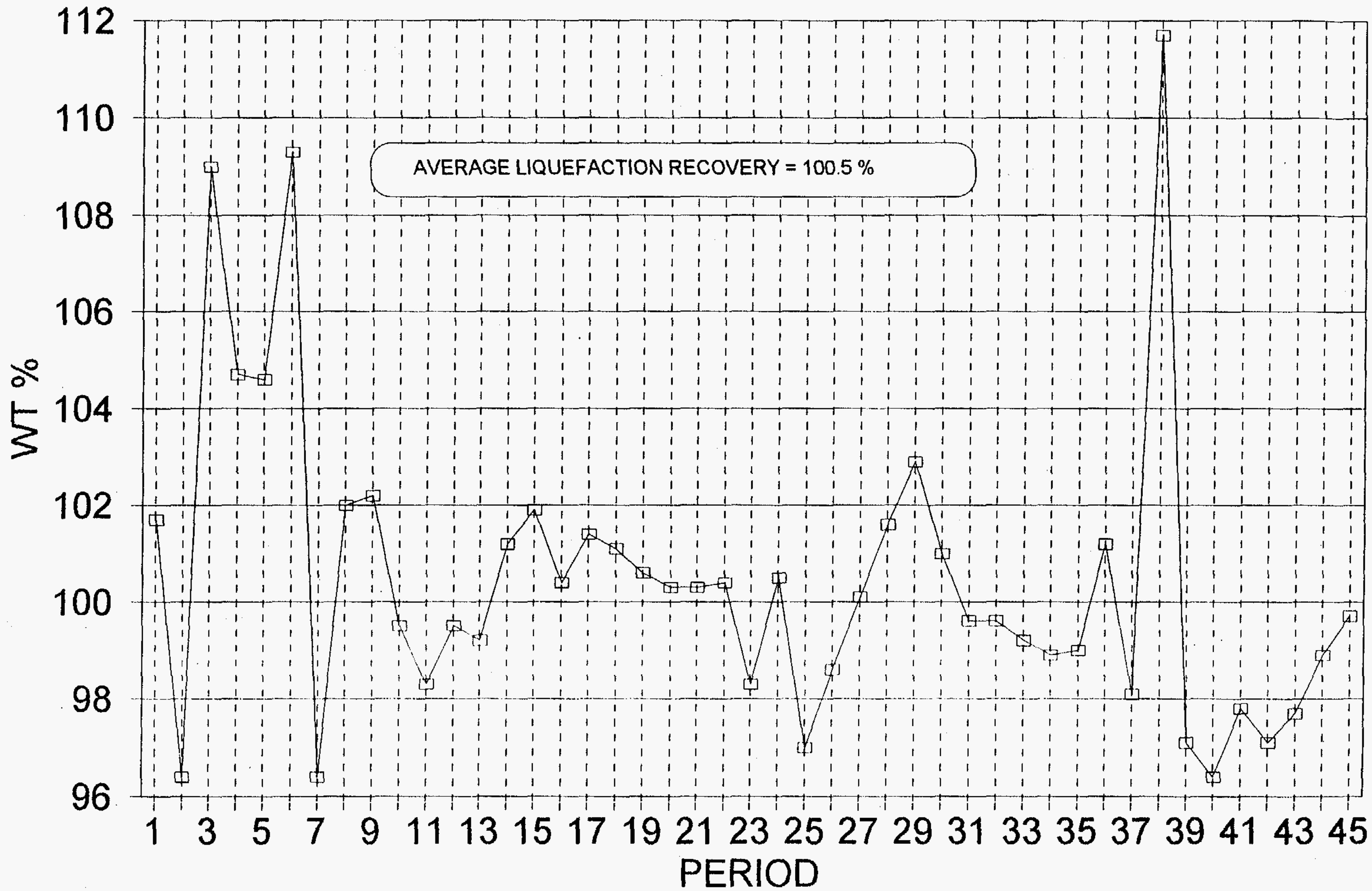


FIGURE 2.19
Solid Separation System Material Balance Recovery

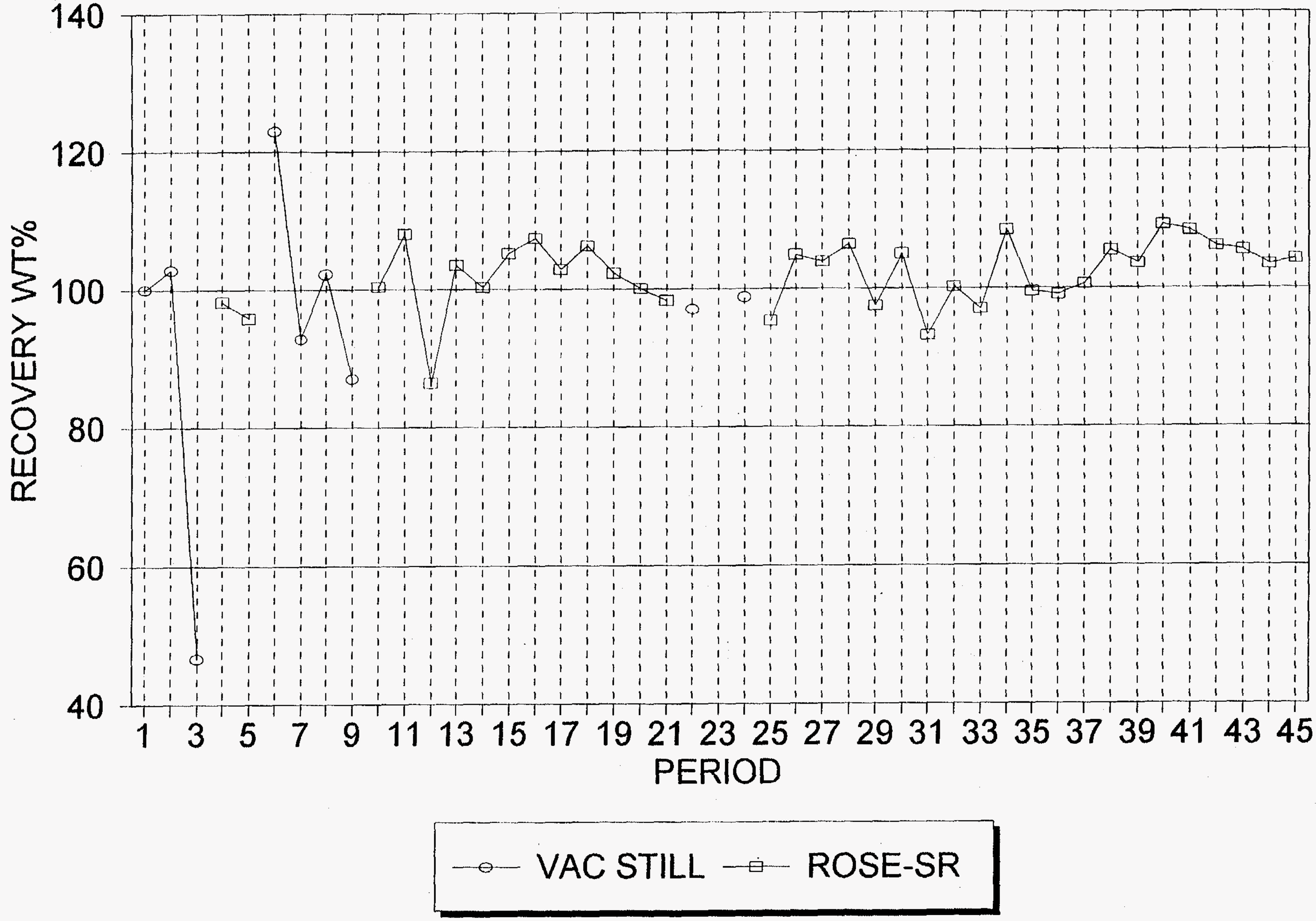


FIGURE 2.20
Solvent To Coal Ratio (MF)

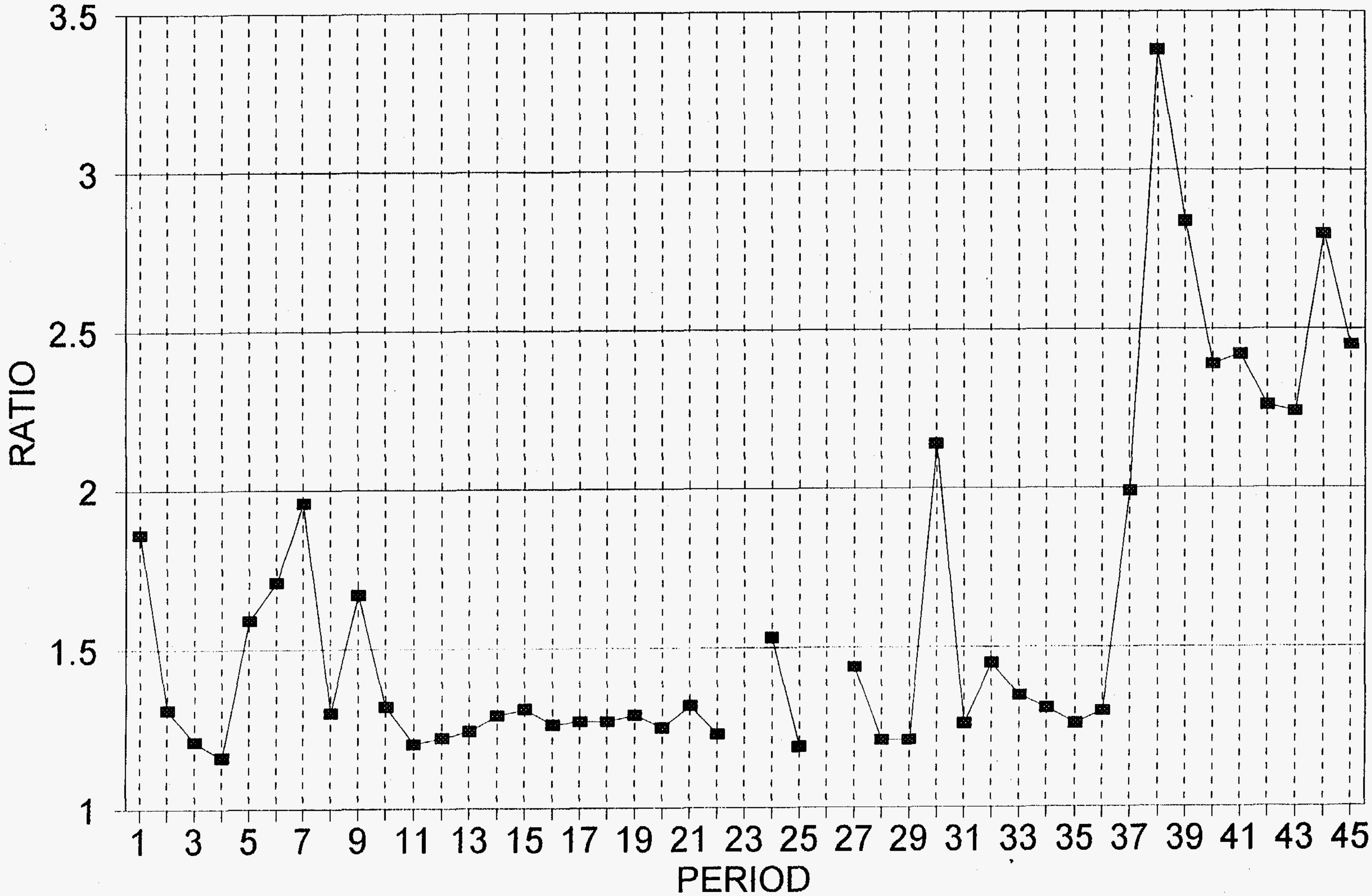
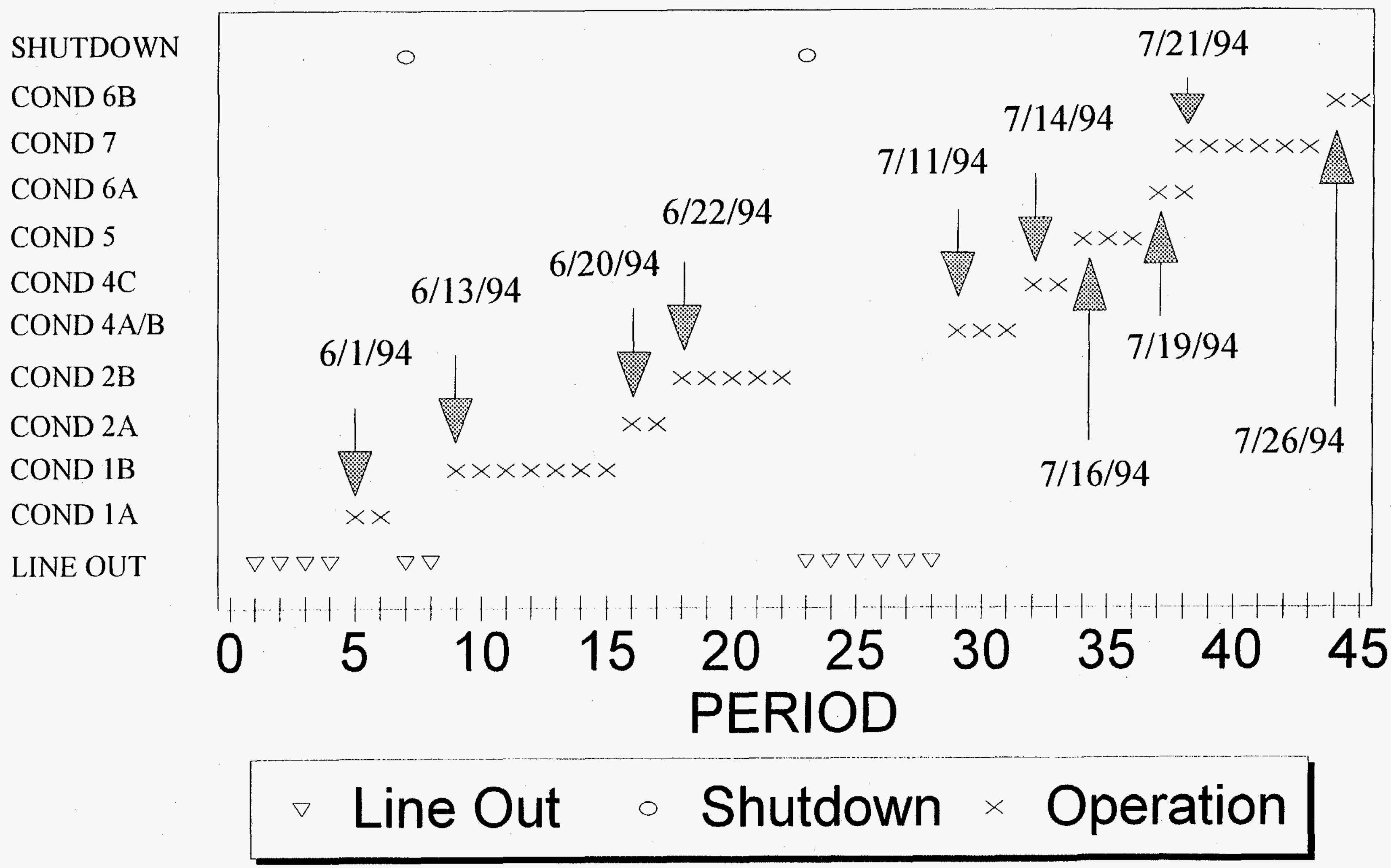


FIGURE 2.21
Operation History Summary



SECTION III

PROCESS PERFORMANCE

PDU run POC-02 was a successful scale-up from a 25 kg/day bench scale operation of HTI's CTSL technology. It was a 45 day run with five steady state work-up conditions (Line-Out, 1B, 2B, 4C, and 5) during the 'coal-only' part of the run and three work-up periods (which, due to short duration of operations, had not reached equilibration) during the 'co-liquefaction' part of the run (Periods 42, 43, and 45). Process yields were normalized based upon the material balance and the elemental analyses of streams in the on coal liquefaction section shown in the Mass Balance Flow Diagram. These yields are elementally balanced, and the ash-balances are also close to 100 W%. The reason for normalizing process yields across the liquefaction section was the ability to better handle the solids containing stream in the form of reactor flash vessel bottoms (O-13 bottoms) rather than handling streams derived from the solids-separation section. The process performance parameters, such as coal and 524°C+ resid conversions, light distillate yields, and heteroatom removals, are best represented by the yields across the liquefaction section. The performance of the solids-separation and recycle solvent-recovery section has been evaluated separately in terms of the percentage organic and energy rejections, overall coal conversion, and the weight percent rejection of light material (524°C- distillates) through the bottoms of the solids-separation section. Therefore, the overall C_4-524°C distillate yield from the process was obtained by adjusting the liquefaction section distillate yields by the amount of light material rejected with the solids in the bottoms.

A. Process Performance Normalized Yields - Impact of Space Velocity and Dispersed Molybdenum Catalyst Addition

Process performance results for POC-02 were calculated as the average of two adjacent steady-state periods. As shown in Table 3.1, for the 'coal-only' conditions, the Wyoming coal from Black Thunder Mine was fairly reactive, giving over 90 Wt% maf conversions for most of the conditions. C_4-524°C distillate yields as high as 66 Wt% maf coal were obtained; these yields ranged between 57 to 66 Wt% maf coal. More than 80 Wt% of these distillates boiled below 343°C, as a result of the extinction recycle mode of operations during POC-02 (all the material boiling above 398°C was recycled). High levels of organic desulfurization (94-98 Wt%) and denitrogenation (over 85 Wt%) were achieved with the in-line hydrotreater unit; typically distillates contained less than 20 ppm nitrogen and sulfur.

The C_1-C_3 gas yields were on the high side (C_1-C_3 selectivities of between 12-18 %) and high hydrogen efficiencies (7-8.5) were also obtained. The space velocity of coal was varied from about 300 to about 615 kg/h/m^3 reactor. Increased coal space velocity resulted in a decline in resid conversion and distillate yields, while overall coal conversion was not much affected. The addition of 200 ppm of molybdenum as Molyvan-A during Condition 5 was found to increase chemical hydrogen consumption and shift the product distribution in favor of lighter distillates. Figures 3.1 through 3.3 show the process performance during POC-02 PDU run in terms of the light distillate yields vs. process severity and hydrogen consumption, heteroatom removals, and deasher performance in terms of organic and energy rejections.

For most of Run POC-02, the solids-separation unit consisted of the ROSE-SR system using initially n-pentane and in the later part of the run, a mixture of n-pentane and toluene, as the extraction solvent. As indicated in Table 3.1, the on-line operation of the ROSE-SR unit was very successful, achieving as low as 12-24 W t% organic rejection, 12-23% energy rejection, and absolutely no degradation of the products (maintaining coal conversion value around that based upon the liquefaction section). Moreover, the bottoms products from the ROSE-SR^{SM} operations were dry and powdery in physical appearance and contained as low as 1-3 Wt% of light (524°C-) material.

B. Co-Liquefaction of Coal and Waste Organic Materials

As mentioned earlier, the feasibility of the co-liquefaction of coal with plastics/rubber wastes was investigated at a 3.0 tpd scale during POC PDU Run No.2. Nine days were run, in co-liquefaction mode towards the end of POC-02, with six days of mixed plastics (as much as 30 W% of solid feed) and two days with used tire crumb rubber (about 26 W% of solid feed). The composition of the net feed during co-liquefaction conditions is shown in Figure 3.4. Recycle solvent-to-feed ratios of about 2.25 had to be employed to keep the plastics/rubber in suspension and render the slurries pumpable. More detailed operating conditions are shown in Table 3.2. Also shown in Table 3.2 are the process performance summaries for the coal-only condition, Period 36, two plastics/coal co-liquefaction conditions, Periods 42 and 43, and one rubber/coal co-liquefaction condition, Period 45. Mixed plastics containing 50 W% HDPE, 35 W% polystyrene, and 15 W% PET were processed with Wyoming Black Thunder Mine subbituminous coal during Periods 42 and 43. Crumb rubber was part of the feed with coal during Period 45. These materials were successfully converted to light liquids and gases. The overall material balances were tight during the continuous operations.

Compared to the "coal-only" condition, higher gas and light (IBP-343°C) distillates were obtained at a reduced overall hydrogen consumption. Since not enough time was available to reach steady-state, with either plastics or rubber as part of the feed, recycle solvent-balance was never achieved. Significant portions of a light, petroleum-based, make-up oil had to be used to realize the high solvent/feed requirements for plastics-containing/slurry preparation and pumping. The composition of the recycle solvent stream is addressed in Section VI; O-13 bottoms, DAO, VSOH, and ASB are all process-derived streams, while make-up oil is an external oil stream. As much as 25-30 W% of the total recycle stream was make-up oil this, coupled with high process severity, resulted excessive hydrocracking of the make-up oil and high gas-makes and light distillate yields.

With either plastics or rubber as part of the feedstock, chemical hydrogen consumption was noticeably lower. With an in-line hydrotreater, net process distillates (naphtha stabilizer bottoms) contained less than about 50 ppm nitrogen and 25 ppm sulfur. For the performance of the ROSE-SR system, both organic and energy rejections increased during co-liquefaction conditions (the extraction solvent for the ROSE-SR operations during the co-liquefaction condition was n-pentane). Surprisingly, coal conversion, based on quinoline solubility, dropped significantly across the ROSE section, indicating the possibility of some retrograde reactions occurring as a result of the different (from coal-only condition) nature of the liquefaction bottoms products.

The process mass-balance/yield flow diagrams (with individual stream flow-rates and compositions) for Periods 15, 20, 34, and 36 are indicated in Figures 3.5 through 3.8. Good material balances were obtained during these steady-state periods which required at most low amounts of make-up oil. Since the length of the co-liquefaction conditions was short, the line-out was never achieved. As a result, a significant amount of make-up oil was used during line-out Period 4 and 'co-liquefaction' Periods 43 and 45, while Periods 15, 20, 34, and 36 used small to negligible amounts of make-up oil with ROSE-SR^{SM} as an on-line solids-separation and recycle solvent-recovery unit. The flow rate of deasher bottoms on all these figures is adjusted for ash-balanced yields. The overall material recovery for all four representative periods, is very good (between 97-102 Wt%). It is important to note that the data presented in Figures 3.5 through 3.8 is based on normalized (by mass and elemental balances) stream flow rates, as is the data in Table 3.1. The only difference is that the values in Table 3.1 are averages for two periods, chosen to represent a particular run condition, while Figures 3.5 through 3.8 show actual values for an individual work-up period.

C. Comparison Between POC-02 PDU and CC-01 Bench Runs

PDU Run POC-02 was similar in some respects to an earlier Bench Run, CC-01, conducted by HRI during the CTSL DOE sponsored Bench Program (1988-92). This particular bench, like POC-02, fed a subbituminous coal, (Wyoming Black Thunder Mine coal). Supported $NiMo/Al_2O_3$ catalyst (Shell 317) was used during CC-01, and simlilar reactor temperatures were also employed. Pressure filtration was used for solids separation, and the catalyst in the reactors underwent batch deactivation (as the bench unit does not allow on-stream catalyst replacement). The details of operating conditions during Period 19 of CC-01 are compared with Condition 1B of POC-02 in Table 3.3 and Figure 3.9. Space velocities based upon coal feed are very similar and so are the reactor temperatures (except that during CC-01, reactor K-2 temperature was about 5°C higher than POC-02) . Average catalyst ages are slightly higher for the PDU operations.

About 2 W% maf higher total coal conversion and about 1.5 W% maf higher 524°C+ resid conversion were obtained during the bench run, but the C_4-524°C distillate yields were about 3 W% higher during the PDU run. The yield of the C_4-343°C distillate fraction was also significantly higher (8 W% maf) for the PDU operation. Chemical hydrogen consumption was slightly higher for the PDU operation, which could be due to the use of an in-line hydrotreater in the PDU operation but not with the bench unit. Very similar C_1-C_3 light gas yields were obtained.

D. Comparison Between POC-02 and Wilsonville Runs 262-E and 263-J

An attempt has been made to compare process performance during POC-02 with results reported for Wilsonville Runs 262-E and 263-J[4,5]. Both the Wilsonville runs were conducted in a thermal/catalytic mode with a high/low reactor temperature sequence. The results under similar operating severity (the Wilsonville runs were at higher average temperatures but at much higher space velocities than the PDU Run POC-02) are composed in Table 3.4 and Figure 3.10. The Wilsonville data indicate higher coal conversions, but resid conversions and C_4-454°C distillate yields are lower for the Wilsonville runs. The yields of individual boiling fractions strongly indicate that POC-02 resulted in the production of higher light distillate (C_4-288°C) yields than those projected for the Wilsonville operations. The light gas C_1-C_3 selectivity, and H_2 efficiency were better during the Wilsonville operations than the POC-02 operations, probably due to the much higher space velocities employed during the Wilsonville operations.

TABLE 3.1
Process Performance - Averaged Per Condition

Process Conditions	L/O Rose-SR	1B Rose-SR	2B Rose-SR	4C Rose-SR	5* Rose-SR
Period/s	4 & 12	14-15	20-21	33-34	35-36
Recycle Type	Ashy	Ashy	Ashy	Ashy	Ashy
Space Velocity, Kg/hr/m3	308.50	305.00	458.00	608.30	614.50
K-1:					
Temperature, Deg. C	399.30	399.50	413.00	431.20	432.20
Cat Replac. Rate, Kg/Kg Ton MF Coal	0.10	0.10	0.45	0.75	0.75
Catalyst Age, Kg MF Coal/Kg Cat	720.00	936.00	1042.00	1040.00	1026.00
K-2:					
Temperature, Deg. C	434.00	435.00	444.00	447.20	443.50
Cat Replac. Rate, Kg/Kg Ton MF Coal	0.16	0.16	0.90	1.25	1.25
Catalyst Age, Kg MF Coal/Kg Cat	626.00	702.00	721.00	618.00	626.00
Flow Rates					
Coal Feed, Kg/hr	69.35	67.75	102.00	137.05	138.40
Oil Streams to SMT					
O-43 Recycle to SMT, Kg/hr	51.08	53.00	43.85	65.85	67.85
Make up Oil, Kg/hr	0.00	7.75	0.00	20.20	0.00
ASB (thru' COT) to SMT, Kg/hr	31.50	27.30	87.53	95.90	108.85
Solvent/Coal Ratio, Kg/Kg	1.19	1.30	1.30	1.30	1.30
Material & Ash Balances					
Liquefaction Section Recovery, W%	102.10	101.55	100.30	99.05	100.10
Overall Material Recovery, W%	101.85	100.60	97.80	97.00	99.35
Normalization Factor	0.99	0.99	0.99	1.01	1.00
Ash Balance, W%	95.00	102.65	106.53	107.70	103.70
NORMALIZED YIELDS, W% MAF COAL [Based on Liquefaction Section: O-13 Bottoms]					
H2S	0.48	0.84	0.47	0.74	0.56
NH3	1.22	1.09	0.94	1.13	1.03
H2O	19.44	18.11	19.08	21.45	19.26
COx	1.24	0.24	0.44	1.13	1.16
C1-C3	9.46	8.13	9.43	9.68	10.11
C4-C6	4.41	4.12	4.40	3.04	4.50
IBP-177 C	12.65	12.36	14.93	15.37	18.28
177-288 C	21.61	22.17	23.57	26.72	24.91
288-343 C	17.65	19.76	11.07	10.36	1.32
343-524 C	8.66	7.78	7.85	1.08	8.42
524 C+ (Solids-free)	3.00	3.58	8.46	7.63	11.67
524 C+ (Tol. Insols)	0.22	0.22	0.24	0.34	0.34
Unconverted Coal	8.85	10.33	7.40	8.22	6.51
PROCESS PERFORMANCE*					
Chemical H2-Consumption, W% MAF	7.72	8.60	8.26	6.86	8.05
Coal Conversion, W% MAF	91.15	89.65	92.60	91.80	93.50
524 C+ Conversion, W% MAF	87.95	85.85	83.90	83.85	81.50
Denitrogenation, W%	86.50	82.50	87.70	85.70	86.25
C4-343 C Net Distillates, W% MAF	55.40	58.35	53.95	55.50	49.00
C4-524 C Distillates, W% MAF	64.05	66.10	61.80	56.60	57.45
C1-C3 Selectivity, Kg/Kg of C4-524 C (X 100)	14.65	12.25	15.25	17.10	17.60
H2 Efficiency, Kg C4-524 C/Kg H2	8.30	7.70	7.50	8.30	7.15
DEASHER PERFORMANCE					
Organic Rejection, W% MAF	24.15	24.35	22.00	12.70	13.80
Energy Rejection, %	23.05	23.10	21.00	11.95	13.60
Deasher Coal Conversion, W% MAF	88.25	89.80	91.50	89.80	90.90
Deasher Rejection of 524 C- Material, W% MAF	5.10	3.70	3.00	1.70	1.85

* Molyvan-822 was a moly-additive used in Condition 5 at 150 ppm Mo relative to coal

TABLE 3.2
Operating Summary and Process Performance During 'Co-Liquefaction' Conditions

Process Conditions		5 Rose-SR	6 Rose-SR	6 Rose-SR	7 Rose-SR
Period/s		35-36	42	43	45
Recycle Type		Ashy	Solids-free	Solids-free	Solids-free
Feed*, W%:	Coal	100.00	68.00	70.00	74
	Plastics (42,43) & Rubber (45)	0.00	32.00	30.00	26
	HDPE	n/a	19	15	n/a
	PS	n/a	13	10	n/a
	PET	n/a	0	5	n/a
	Ground Rubber	n/a	n/a	n/a	26
Space Velocity, Kg/hr/m3		614.50	379.00	433.60	398.40
K-1:					
	Temperature, Deg. C	432.20	428.30	431.10	430.00
	Cat Replac. Rate, Kg/Kg Ton MF Coal	0.75	0.45	0.45	0.00
	Catalyst Age, Kg MF Coal/Kg Cat	1026.00	1044.00	1056.00	1072.00
K-2:					
	Temperature, Deg. C	443.50	442.80	443.80	442.80
	Cat Replac. Rate, Kg/Kg Ton MF Coal	1.25	0.90	0.90	0.00
	Catalyst Age, Kg MF Coal/Kg Cat	626.00	632.00	641.00	660.00
Flow Rates					
Coal Feed, Kg/hr		138.40	60.00	68.80	66.40
	Plastics/Rubber	0.00	28.24	29.50	23.30
Oil Streams to SMT					
	O-43 Recycle to SMT, Kg/hr	67.85	103.50	64.88	71.56
	Make up Oil, Kg/hr	0.00	40.00	58.53	93.52
	ASB (thru' COT) to SMT, Kg/hr	108.85	55.67	96.20	54.49
Solvent/Coal Ratio, Kg/Kg		1.30	2.26	2.24	2.45
Material & Ash Balances					
Liquefaction Section Recovery, W%		100.10	97.10	97.70	99.70
Overall Material Recovery, W%		99.35	97.80	100.30	98.80
Normalization Factor		1.00	1.03	1.02	1.00
Ash Balance, W%		103.70	118.99	108.90	116.44
NORMALIZED YIELDS, W% MAF FEED* [Based on Liquefaction Section: O-13 Bottoms]					
	H2S	0.56	1.61	1.25	2.25
	NH3	1.03	0.68	0.63	0.86
	H2O	19.26	14.72	16.56	16.30
	COx	1.16	0.49	0.99	0.85
	C1-C3	10.11	14.39	10.28	11.94
	C4-C6	4.50	7.77	4.17	6.61
	IBP-177 C	18.28	27.81	22.04	22.05
	177-288 C	24.91	39.31	31.08	40.16
	288-343 C	1.32	15.39	15.94	24.27
	343-524 C	8.42	-22.62	-3.73	-26.92
	524 C+	12.01	-0.42	0.10	3.79
	Unconverted Coal	6.51	7.20	7.20	5.32
PROCESS PERFORMANCE (Combined Feed Basis)					
Chemical H2-Consumption, W% MAF		8.05	6.34	6.50	7.47
Total Feed Conversion, W% MAF		93.50	92.80	92.80	94.68
524 C+ Conversion, W% MAF		81.50	93.20	92.70	90.90
Denitrogenation, W%		86.25	77.40	78.00	74.60
C4-343 C Net Distillates, W% MAF		49.00	90.30	73.20	93.10
C4-524 C Distillates, W% MAF		57.50	67.70	69.50	66.20
C1-C3 Selectivity, Kg/Kg of C4-524 C (X 100)		17.60	21.30	14.80	18.00
H2 Efficiency, Kg C4-524 C/Kg H2		7.16	10.70	10.70	8.90
DEASHER PERFORMANCE					
Organic Rejection, W% MAF		13.8	29.50	25.60	28.50
Energy Rejection, %		13.6	28.30	25.70	29.00
Deasher Coal Conversion, W% MAF		90.9	79.00	85.50	85.50
Deasher Rejection of 524 C- Material, W% MAF		1.85	5.30	4.80	3.60

*"Fresh Feed" is a combination of coal and plastics or coal and crumb rubber for Periods 42,43, & 45;

TABLE 3.3
Process Performance Comparison Between Bench Run CC-01 and POC-02

Coal : Wyoming Black Thunder Mine
Catalyst: Aged Akzo AO-60 1/16" NiMo Extrudates in both Reactors

Run		POC-02	CC-01
COAL		Black Thunder	Black Thunder
Process		CTSL	CTSL
Period/s		14-15	19
Solids-Separation		ROSE-SR	FILTER
Recycle Type		Ashy	Ash-free
Space Velocity, Kg/hr/m3 (Stage)		305.0	323.2
K-1:	Temperature, Deg. C	399.5	399.0
	Cat Replac. Rate, Kg/Kg Ton MF Coal	0.1	n/a
	Cat. Age, Kg MF Coal/Kg Cat.	936.0	541.0
K-2:	Temperature, Deg. C	435.0	440.0
	Cat Replac. Rate, Kg/Kg Ton MF Coal	0.2	n/a
	Cat. Age, Kg MF Coal/Kg Cat.	702.0	541.0
Flow Rates			
Coal Feed, Kg/hr		67.8	0.8
Solvent/Coal Ratio, Kg/Kg		1.3	1.2
Material Balances			
Liquefaction Section Recovery, W%		101.6	n/a
Overall Material Recovery, W%		100.6	96.1
YIELDS, W% MAF COAL			
[Based on Liquefaction Section]			
	H2S	0.8	0.4
	NH3	1.1	1.0
	H2O	18.1	20.1
	COx	0.2	0.9
	C1-C3	8.1	9.5
	C4-177 C	16.5	21.2
	177-288 C	22.2	10.8
	288-343 C	19.8	18.2
	343-524 C	7.8	13.3
	524 C+	3.8	4.4
	Unconverted Coal	10.3	8.2
PROCESS PERFORMANCE			
Chemical H2-Consumption, W% MAF		8.6	8.1
Coal Conversion, W% MAF		89.7	91.8
524 C+ Conversion, W% MAF		85.9	87.4
Denitrogenation, W%		82.5	75.7
C4-343 C Net Distillates, W% MAF		58.5	50.2
C4-524 C Distillates, W% MAF		66.3	63.5
C1-C3 Selectivity, Kg/Kg of C4-524 C (X 100)		12.2	14.9
H2 Efficiency, Kg C4-524 C/Kg H2		7.7	7.9
DEASHER PERFORMANCE			
Organic Rejection, W% MAF		24.4	n/a
Energy Rejection, %		23.1	n/a
Deasher Coal Conversion, W% MAF		89.8	n/a

TABLE 3.4
Process Performance Comparison Between POC-02 & Wilsonville Operations Run 262 E and 263 J

Run	POC-02	POC-02	Wilsonville	Wilsonville
COAL	Black Thunder	Black Thunder	Black Thunder	Black Thunder
Ash %	5.7	5.7	6.3	11.2
Catalyst	AO-60; 1/16"	AO-60; 1/16"	Criterion 324	Criterion 324
Period/s	14-15	20-21	262E	263J
Solids-Separation	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR
Recycle Type	Ashy	Ashy	Ashy	Ashy
Space Velocity, Kg/hr/m3 (Stage)	305.0	458.0	n/a	n/a
K-1: Temperature, Deg. C	399.5	413.0	440.0	440.0
Cat Replac. Rate, Kg/Kg Ton MF Coal	0.1	0.5	n/a	n/a
Cat. Age, Kg MF Coal/Kg Cat.	936.0	1042.0	n/a	n/a
K-2: Temperature, Deg. C	435.0	444.0	432.0	432.0
Cat Replac. Rate, Kg/Kg Ton MF Coal	0.2	0.9	1.5	1.5
Cat. Age, Kg MF Coal/Kg Cat.	702.0	721.0	674.0	662.0
Flow Rates				
Coal Feed, Kg/hr	67.8	102.0	n/a	n/a
Solvent/Coal Ratio, Kg/Kg	1.3	1.3	2.7	2.7
Material Balances				
Liquefaction Section Recovery, W%	101.6	100.3	n/a	n/a
Overall Material Recovery, W%	100.6	97.8	n/a	n/a
YIELDS, W% MAF COAL [Based on Liquefaction Section]				
H2S+H2O+NH3+COx	20.3	20.9	22.9	21.9
C1-C3	8.1	9.4	6.0	7.9
C4-177 C	16.5	19.3	10.3	10.1
177-288 C	22.2	23.6	26.0	26.7
288-454 C	23.8	16.1	20.9	21.7
454 C+ (+ unconverted coal)	17.9	19.0	19.4	17.6
PROCESS PERFORMANCE				
Chemical H2-Consumption, W% MAF	8.6	8.3	5.5	5.8
Coal Conversion, W% MAF	89.7	92.6	92.3	94.1
454 C+ Resid Conversion, W% MAF	82.1	81.0	80.6	82.4
Desulfurization, W%	86.6	79.3	n/a	n/a
Denitrogenation, W%	82.5	87.7	n/a	n/a
C4-454 C Net Distillates, W% MAF	62.5	59.0	57.2	58.5
Deasher Organic Rejection, W% MAF	22.2	15.2	19.8	13.4

FIGURE 3.1
Process Performance During POC-02 Coal & Resid Conversions

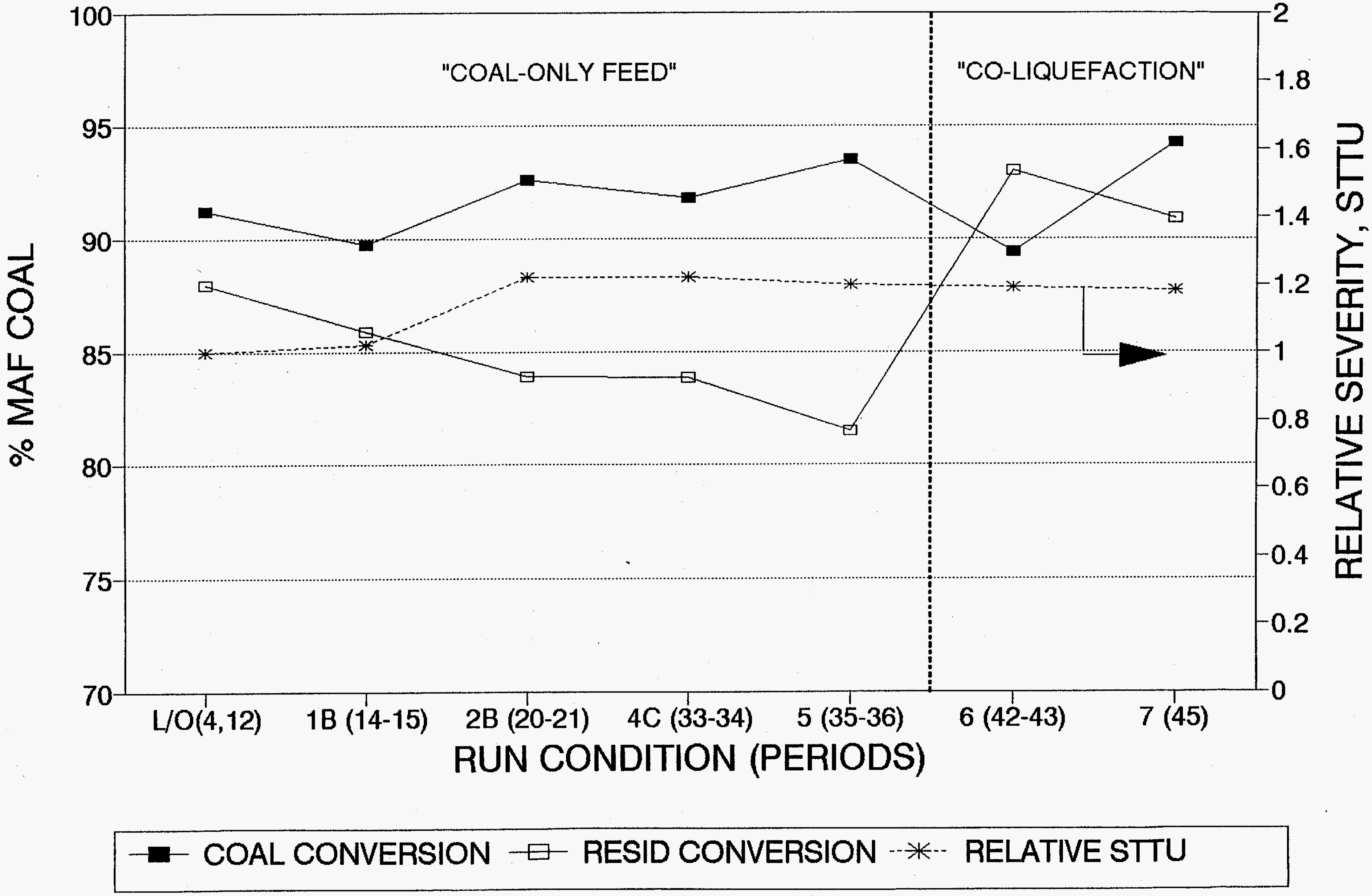


FIGURE 3.2
Process Performance During POC-02 Coal Light Distillate Yields

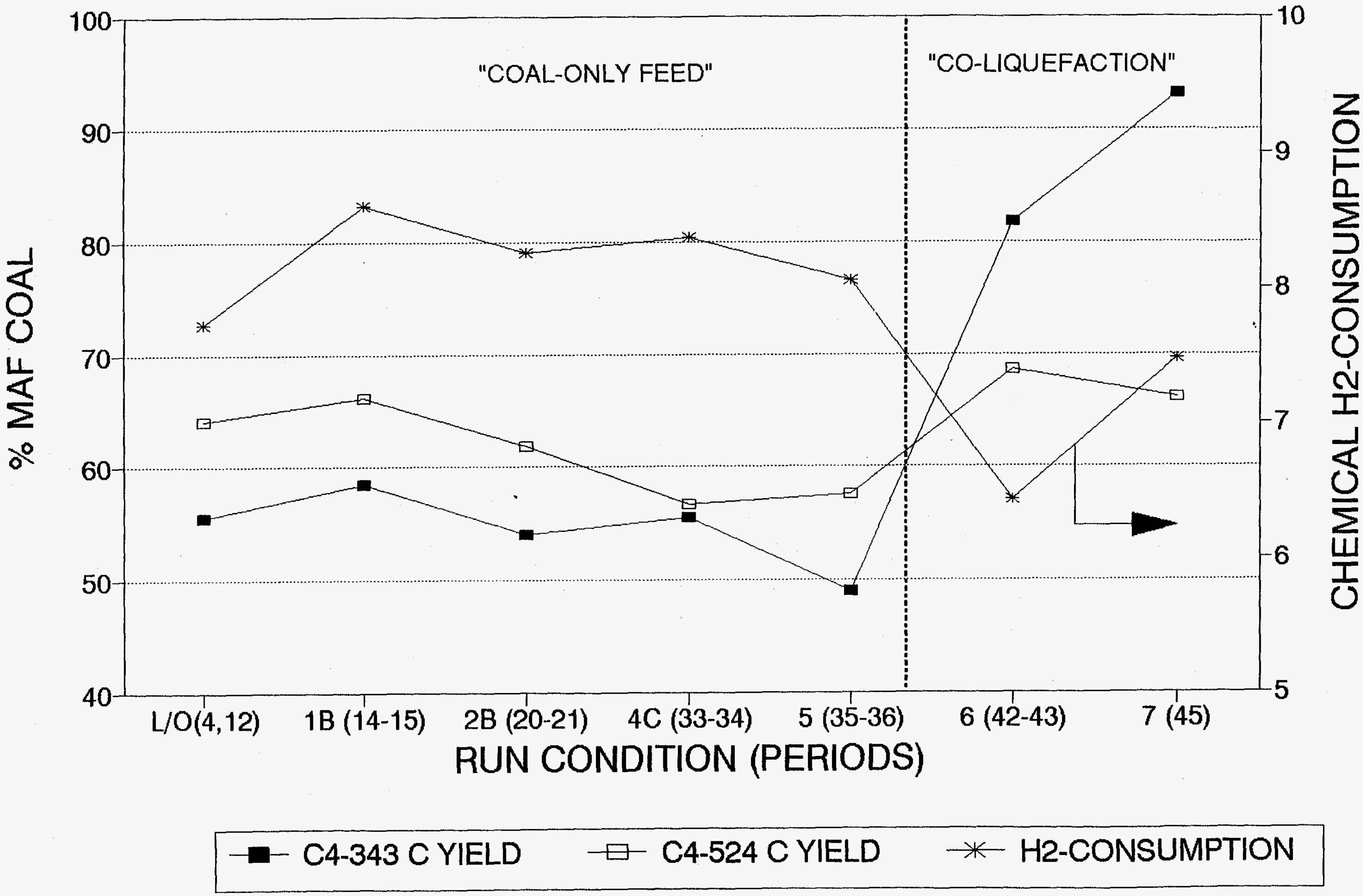


FIGURE 3.3
Process Performance During POC-02 Deasher Performance

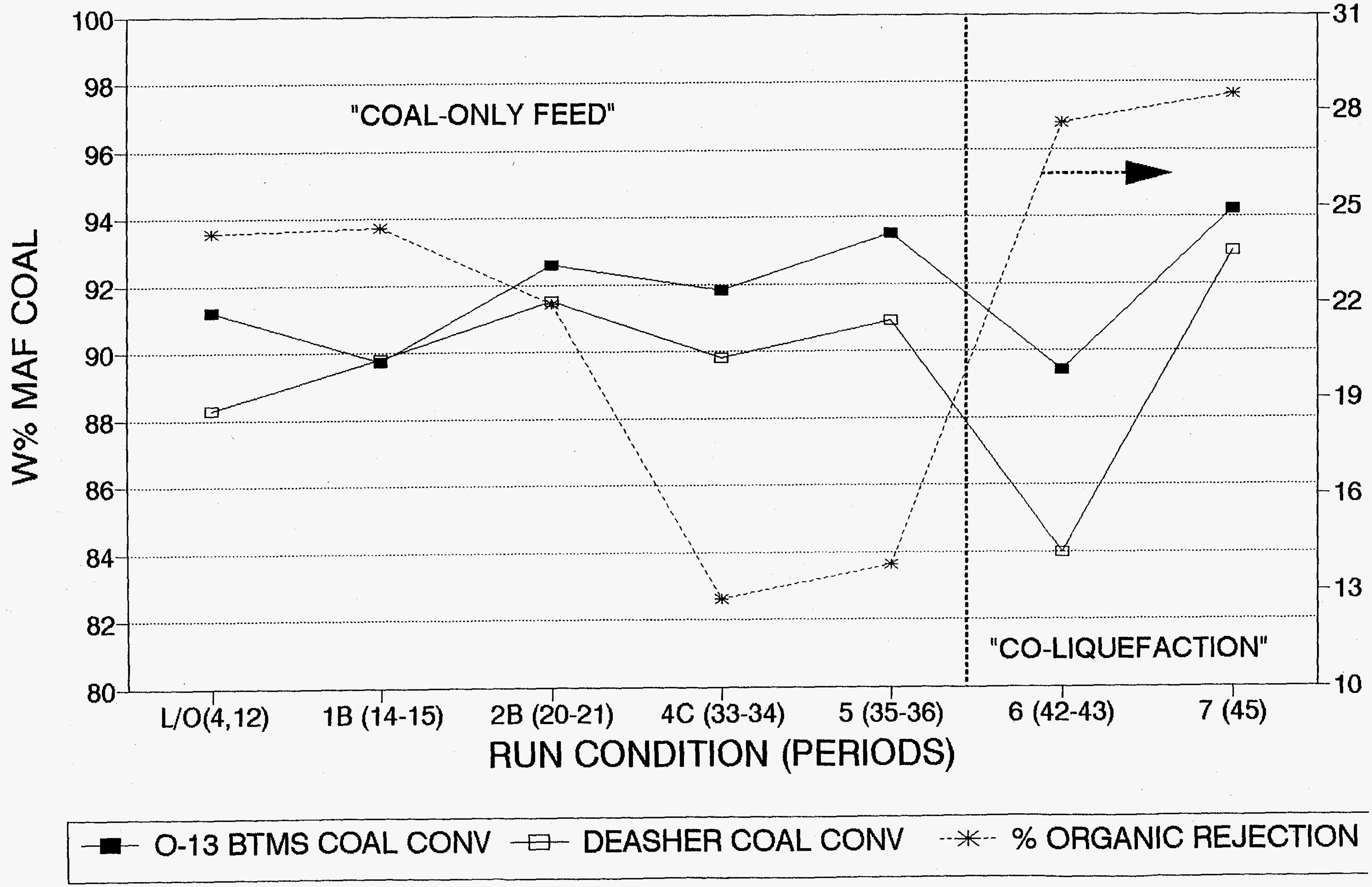


FIGURE 3.4
Daily Feed Composition During 'Co-Liquefaction' Conditions

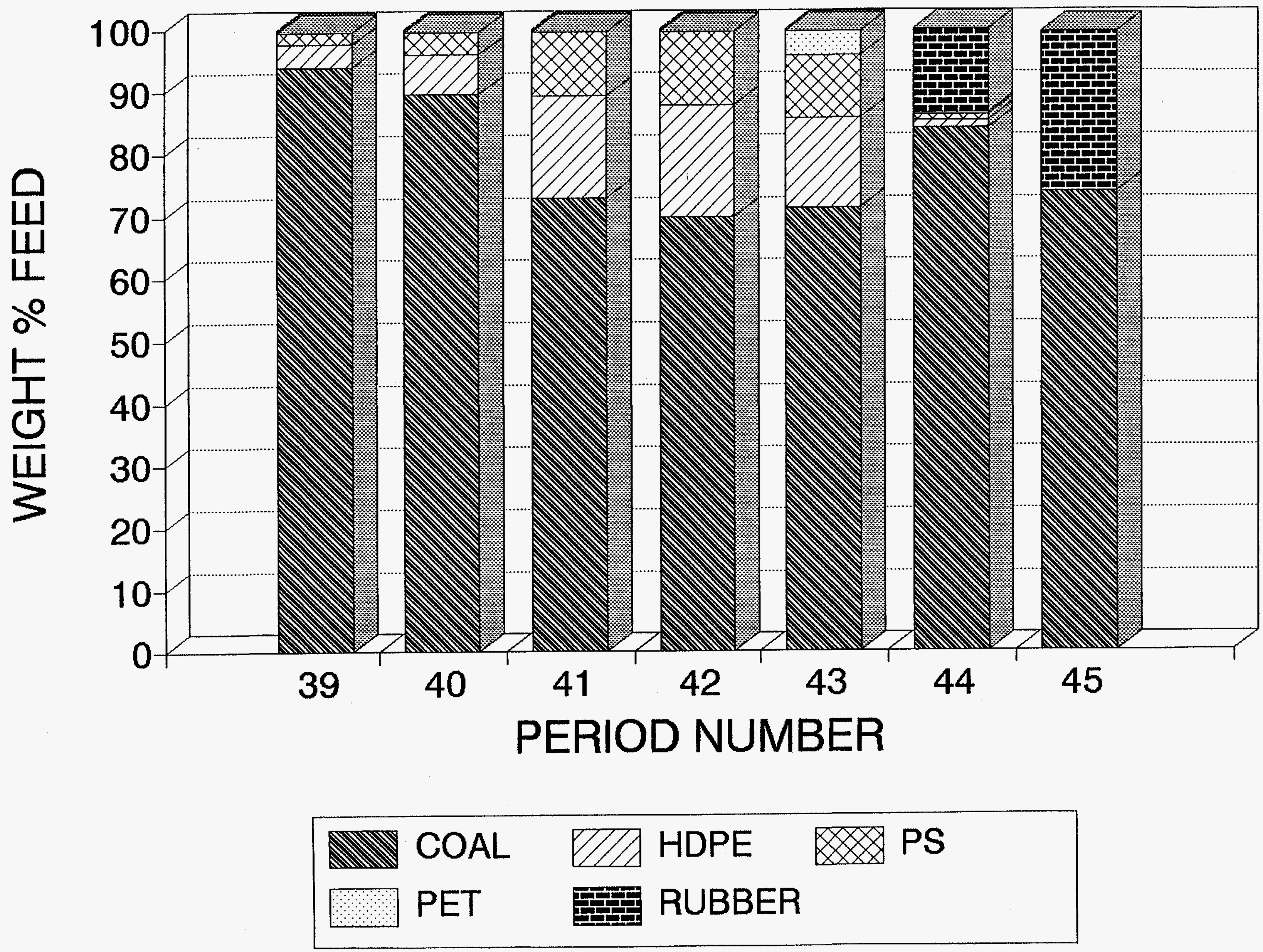


FIGURE 3.5
Mass Balance/Yield Process Flow Diagrams: Period 15

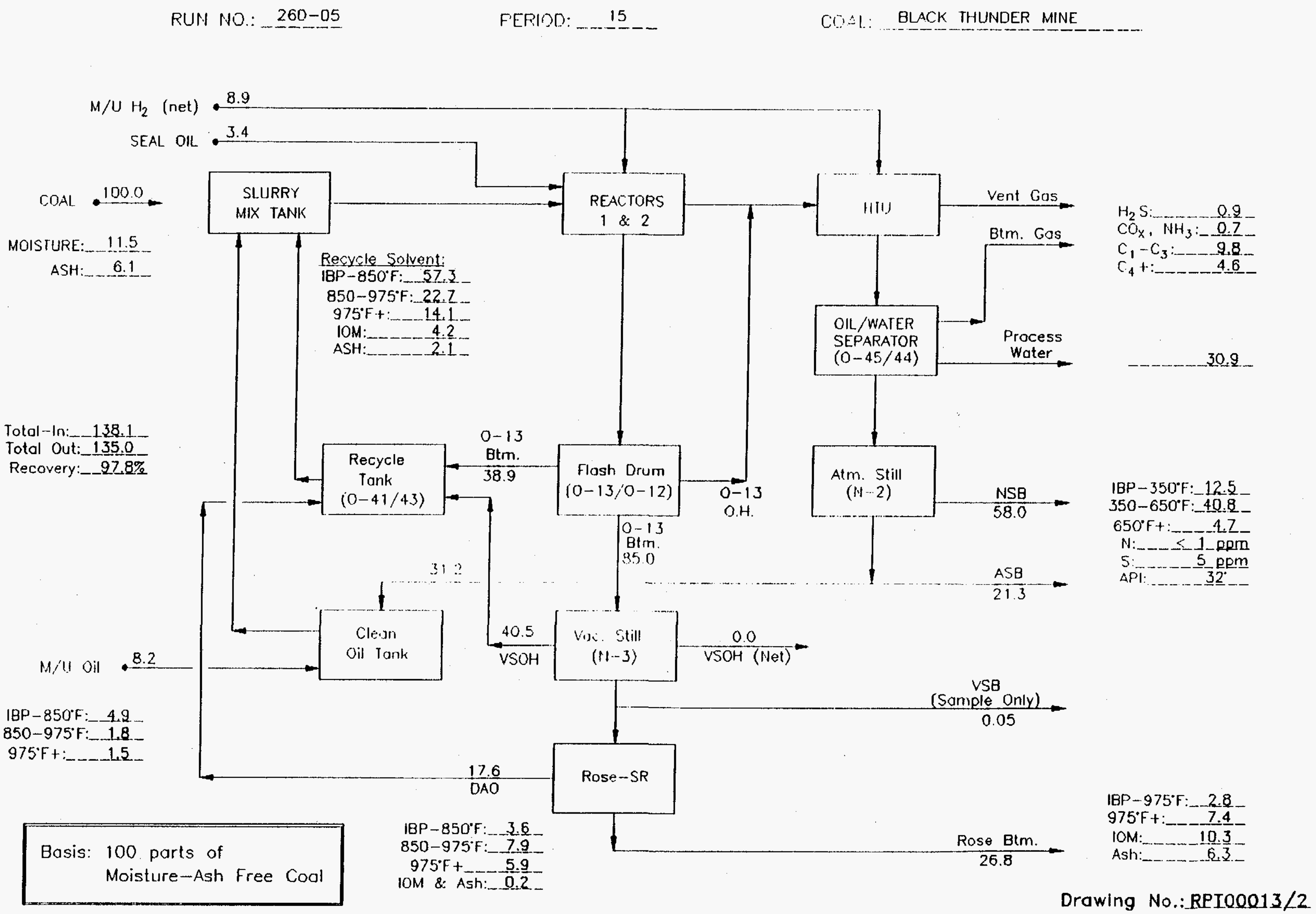


FIGURE 3.6
Mass Balance/Yield Process Flow Diagrams: Period 20

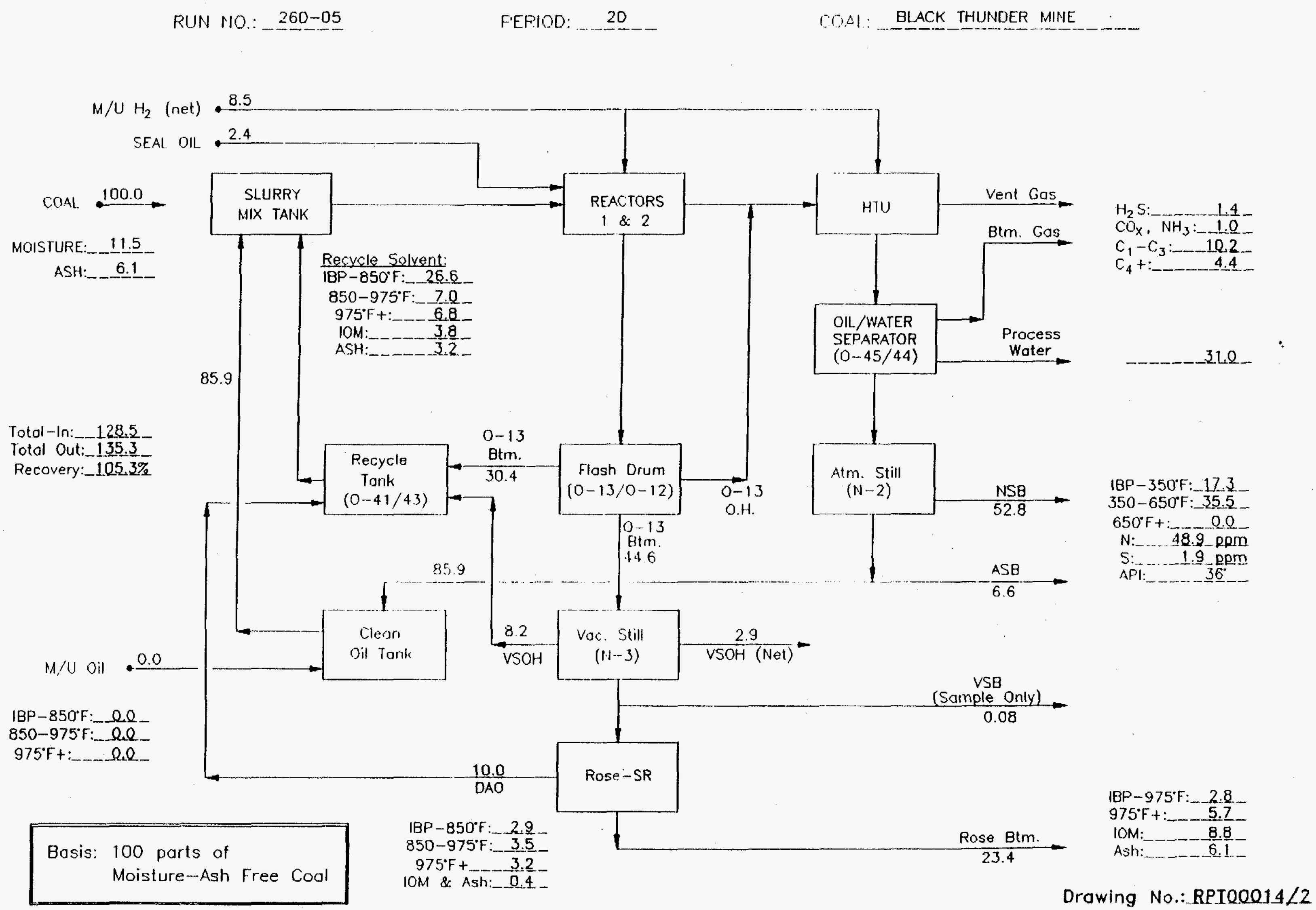


FIGURE 3.7
Mass Balance/Yield Process Flow Diagrams: Period 34

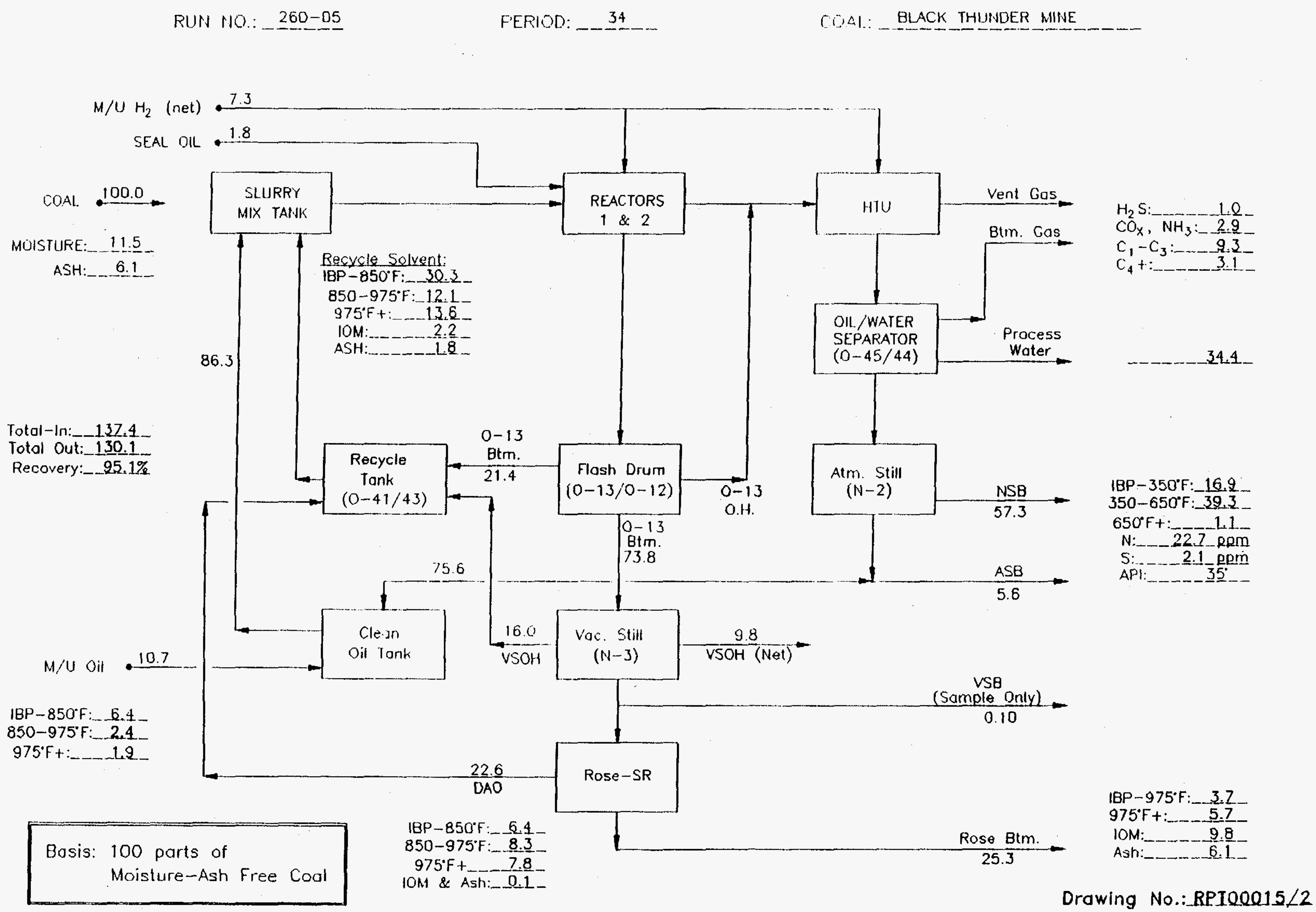


FIGURE 3.8
Mass Balance/Yield Process Flow Diagrams: Period 36

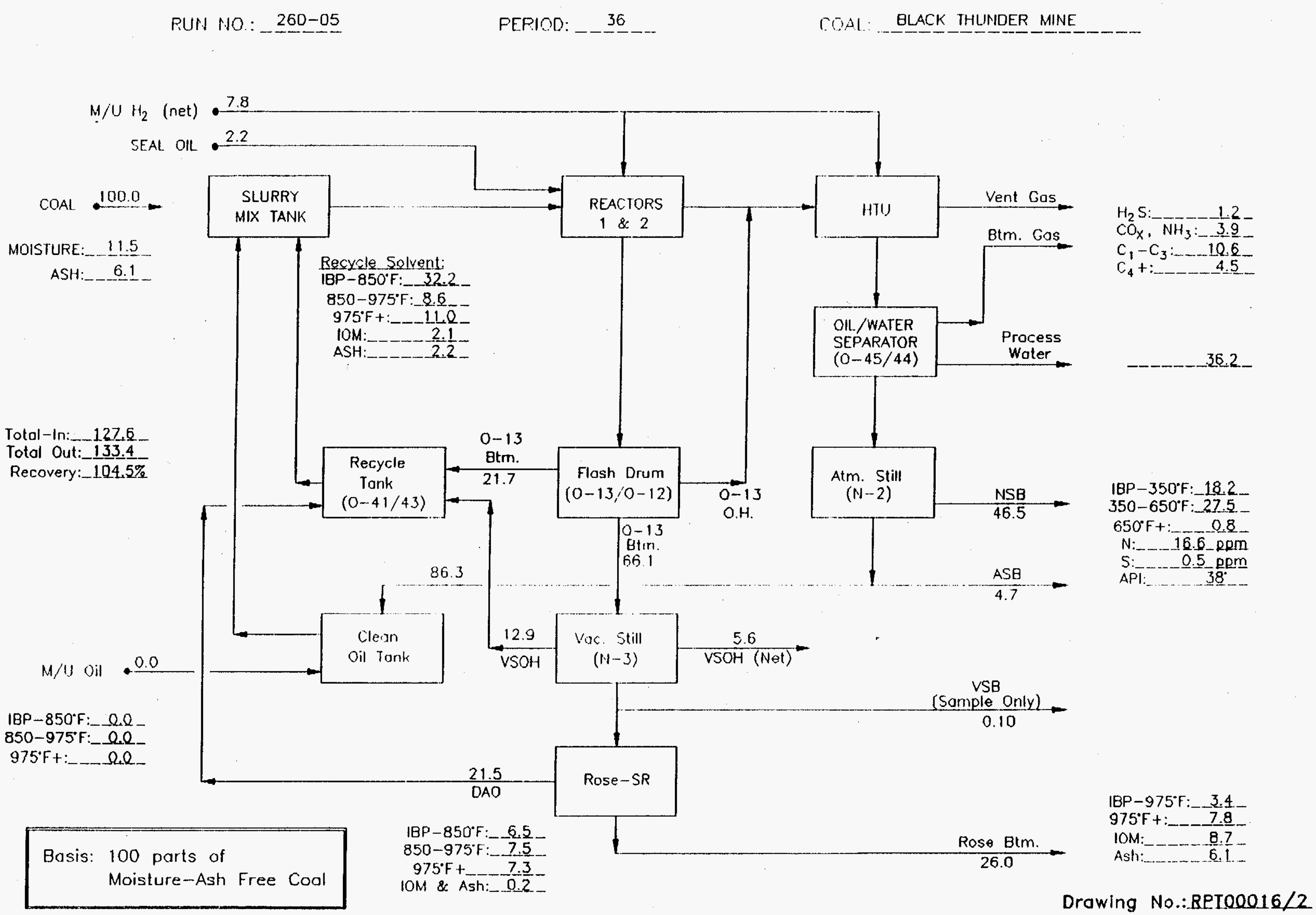


FIGURE 3.9
Process Performance Comparisons: POC-02 PDU vs CC-01 Bench Run

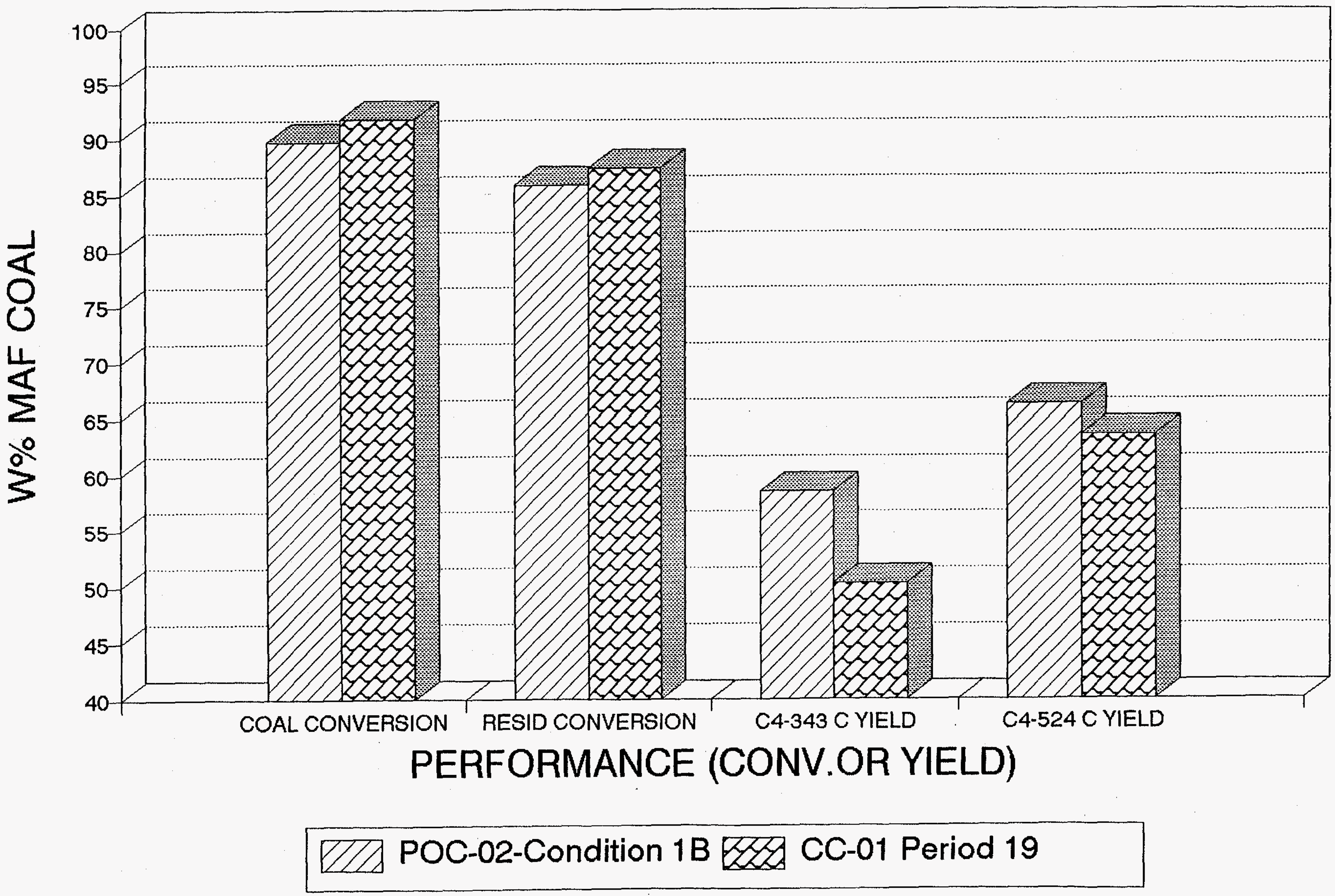
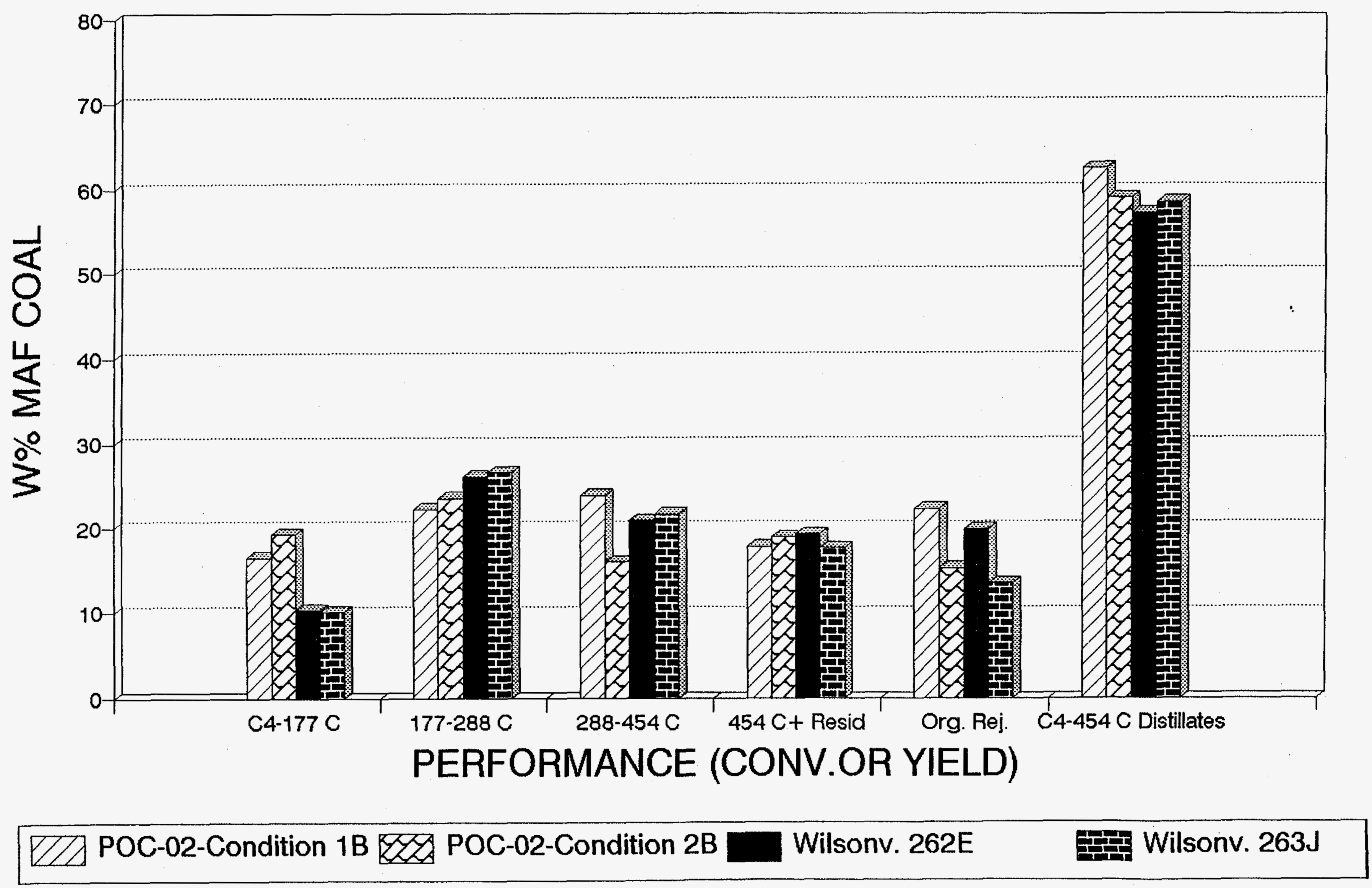


FIGURE 3.10
Process Performance Comparisons: POC-02 PDU vs Wilsonville Runs 262 E & 263 J



SECTION IV

DETAILED RUN ANALYSIS

A. CATALYST : ACTIVITY AND INVENTORY

A.1. Startup Catalyst

In order to provide an accelerated approach line-out, an equilibrated catalyst, recovered from POC-01 operations, was used to start up POC-02. This catalyst was oil-free +20 mesh extrudates from the daily reactor withdrawals of POC-01. The estimated average ages of the spent catalysts loaded into reactors K-1 and K-2 were 587 and 500 kg coal/kg catalyst. Properties, are shown in Figures 4.1 and 4.2. Catalyst activities were determined using conversion of 524°C+ residual oil in microautoclave tests. The activity results, shown in Figure 4.3, indicate that the POC-01 recovered catalysts had activities between 45-60 % of freshly presulfided AO-60 catalyst. It was also found that, typically, first stage spent catalysts were 10-15 % more active than second stage catalysts, despite the lower catalyst replacement rates to stage 1. It was estimated that the catalyst deactivation factor for the first stage catalyst was only 55 % of that of the second stage catalyst.

A.2. Catalyst Aging

It is desirable to carry out PDU operations under "equilibrated catalyst activity" conditions. To maintain uniform catalyst activity in both reactors, periodic catalyst replacement is implemented. Equilibrium catalyst activity is a function of catalyst replacement rate and several other factors. Catalyst replacement rates of 0.5 and 1 kg/ton coal were selected for the first and the second stages, respectively. The higher replacement rate for the second stage is essential to maintain performance, as the catalyst is expected to age at a much faster rate in Stage 2 than in the lower temperature first stage. The effect of catalyst aging on the resid concentration in the recycle solvent was simulated (based upon resid conversion kinetics) as an indicator of catalyst/process equilibration. As shown in Figure 4.4, with the exception of Condition 1, most of the resid levels are very close to the estimated equilibrium values.

In CTSL operations, it is customary to define catalyst age as the amount of dry coal the catalyst has been exposed to per unit weight of dry catalyst (fresh basis). The average catalyst age profile (kg of coal/kg of catalyst) during POC-02 is shown in *Figure 4.5*. The irregularities and humps in the age profile are due to shut-downs and startups during POC-02. Equilibrium catalyst age on stages one and two was attained around Periods 20 and 10, respectively.

A.3 Catalyst Daily Inventory and Mass Balance

Catalyst inventory calculations were made daily, using catalyst addition and withdrawal rates and the toluene soluble oil-content of the spent catalyst charge, its weight loss upon ignition, and its metal (Mo and Ni) contents. Catalyst fines lost in the solid products (O-13 bottoms solids) were accounted for by the following equation that was used to estimate catalyst inventory in the reactors on a fresh basis:

> Catalyst Inventory @ Day N = Catalyst Inventory @ Day (N-1) + Fresh Catalyst Added - Catalyst withdrawn (fresh basis) - Catalyst Fines lost in Solid Products (Fresh Basis)

The fresh catalyst contained 2.75 Wt% moisture. Attrition of the fresh catalyst to -20 mesh size fines was found to be, very significant during POC-02 operations. High reactor temperatures, high gas velocities, and the high moisture content of the feed coalprobably account for the abnormally high attrition rate observed during POC-02 and especially during the coprocessing part of the run. The -20 mesh fines in the catalyst withdrawals are shown in Figure 4.6.

The daily catalyst inventory relative to the targeted catalyst inventory in both reactors is shown in *Figure 4.7.* Reactor K-1 on average had 95 Wt% or lower of the targeted catalyst inventory, while reactor K-2 had close to the targeted inventory except for a few periods around Period 30. The catalyst fines lost through O-13 bottoms relative to feed space velocity are plotted in Figure 4.8.

A.4 Spent Catalyst Properties

Spent catalyst obtained from periodic withdrawals during POC-02 was typically washed thoroughly with toluene and then with acetone to remove residual oil. The catalyst was then dried and screened to separate +20 mesh solids from the fines. Detailed analyses of the properties of the catalysts are listed in *Table 4.1.* Catalyst withdrawals that were only washed with toluene and acetone were found to retain as much as 30 Wt% oil. Due to this, all of the work-up periods' catalyst withdrawals were soxhlet extracted with toluene to remove all the soluble oil from the catalyst pores, and the detailed characterization was carried out on these catalysts. In Table 5.3, spent catalysts (withdrawals during POC-02) are compared with freshly sulfided Akzo AO-60 extrudates and the spent catalyst from POC-1, which was used as the start-up catalyst for POC-02.

The carbon loading on the first and second stage withdrawn catalysts, soxhlet extracted with toluene, ranged from 12 to 19 W%. As expected, the second stage catalysts were noticeably higher in carbon than the first stage catalysts. Although the first stage catalysts contained less carbonaceous materials, metal contaminants (Fe, Ca, Ti) were higher than for second stage catalysts. Analyses are shown in catalysts withdrawn during Periods 33 and 36. During Period 36, when a molybdenum precursor was added to the feed, the moly-contents of the withdrawn catalysts (especially reactor K-1) were noticeably higher. This suggests that the added molybdenum precursor, after thermal decomposition, may deposit on the surface of the extrudate catalyst, thus improving its activity for hydrogenation reactions. The activities of withdrawn catalysts from several periods, determined by microautoclave testing, are presented in Section VII Laboratory Support.

B. Recycle Solvent Composition

Under normal operation the slurrying oil was taken from the Recycle Weigh Drum (O-43). However, vacuum still overheads and makeup oil were also introduced through the Clean Oil Tank during startup periods or when solvent imbalance occurred. As mentioned in the process description section, material fed to O-43 included atmospheric still bottoms(ASB), deasphalted oil(DAO) from ROSE-SR^{SM} operations or topped filtrate from filter operations, and reactor liquid flash vessel bottoms under ashy recycle mode. Ashy recycle mode was implemented during all line-out periods and in Condition 1 in conjunction with ROSE-SR^{SM} operation. Composition of the slurrying oil is presented in *Figures 4.9 and 4.10.*

During ashy-recycle operations, the major fractions of the recycle solvent were O-13 bottoms, VSOH, DAO, and ASB being the remainder of the recycle oil. The percentage of ROSE-SR derived deasphalted oil (DAO) in the recycle solvent was relatively low (between 10-20 w%). During some periods, especially those in the co-liquefaction mode, significant amounts of make-up oil had to be used. The overall make-up of the recycle solvent stream to the unit, along with the analyses of the individual components of this stream, are shown in Table 4.2.

TABLE 4.1
Analyses of Catalyst Withdrawals

Period	33	33	36	36
Stage	1	2	1	2
Particle Density, gm/cc	1.073	1.154	1.098	1.140
Ignition Loss, W%	18.66	24.13	18.39	27.39
Carbon, W%	11.84	18.96	11.72	18.34
Hydrogen, W%	0.81	0.85	0.77	0.91
Nitrogen,W%	0.13	0.15	0.11	0.21
Sulfur, W%			6.52	6.06
Molybdenum, W%	7.86	7.76	8.13	7.77
Nickel, W%	1.95	1.86	1.90	1.81
Iron, W%	1.23	0.11	1.07	0.10
Sodium, W%	1.01	1.48	0.90	1.21
Calcium, W%	0.09	0.04	0.06	0.06
Molybdenum, gm/cc	0.0843	0.0896	0.0893	0.0885
Nickel, gm/cc	0.0209	0.0215	0.0209	0.0206
Fresh Catalyst Content, W%				
Basis Particle Density	81.3	75.6	79.4	76.5
Basis Ignition Loss	77.9	73.2	81.9	73.4
Basis Contaminants	82.7	76.3	80.2	74.3
Basis Molybdenum Content	69.5	68.6	71.9	68.7
Basis Nickel Content	78.4	75.0	76.6	73.0

TABLE 4.2
Detailed Recycle Stream Composition

	Flowrates, kg/hr						Composition, W%					
Period	O-13	DAO	VSOH	VSB	ASB	m/u	O-13	DAO	VSOH	VSB	ASB	m/u
1	60.2	0	4.2	57.3	0	0	49.47	0.00	3.45	47.08	0.00	0.00
2	23.9	0	0	15.8	27.8	17.4	28.15	0.00	0.00	18.61	32.74	20.49
3	25.8	0	8	0	5.4	23.6	41.08	0.00	12.74	0.00	8.60	37.58
4	0	39.7	8	0	44.4	0	0.00	43.11	8.69	0.00	48.21	0.00
5	0	20.2	23.6	0	13	30	0.00	23.27	27.19	0.00	14.98	34.56
6	0	0	22.4	0	20	0	0.00	0.00	52.83	0.00	47.17	0.00
7	66.3	0	43.3	0	4.5	0	58.11	0.00	37.95	0.00	3.94	0.00
8	43.5	0	28.2	0	18.5	0	48.23	0.00	31.26	0.00	20.51	0.00
9	29.3	0	16.5	0	34	22.3	28.70	0.00	16.16	0.00	33.30	21.84
10	26.5	13.7	10.7	0	29.4	15.5	27.66	14.30	11.17	0.00	30.69	16.18
11	18.5	11.1	35.3	0	27.6	0	20.00	12.00	38.16	0.00	29.84	0.00
12	30.5	12	12	0	19.5	0	41.22	16.22	16.22	0.00	26.35	0.00
13	24.6	14.8	2.8	0	47.2	2.4	26.80	16.12	3.05	0.00	51.42	2.61
14	24.4	6.7	15.9	0	36.8	10.5	25.87	7.10	16.86	0.00	39.02	11.13
15	23.3	9.7	26.8	0	18.7	5.1	27.87	11.60	32.06	0.00	22.37	6.10
16	18.3	8.3	12.2	0	35.7	3.3	23.52	10.67	15.68	0.00	45.89	4.24
17	18.4	7.3	12.1	0	45.2	0	22.17	8.80	14.58	0.00	54.46	0.00
18	27.1	10.7	10.1	0	64.5	0	24.11	9.52	8.99	0.00	57.38	0.00
19	28.3	10.9	8.5	0	86.8	0	21.04	8.10	6.32	0.00	64.54	0.00
20	29.9	6.9	8.1	0	84.6	0	23.09	5.33	6.25	0.00	65.33	0.00
21	28.4	3.2	9.9	0	93.6	0	21.02	2.37	7.33	0.00	69.28	0.00
22	43.3	0	2.6	0	89.1	0	32.07	0.00	1.93	0.00	66.00	0.00
23	17.4	0	0	0	33.3	13	27.32	0.00	0.00	0.00	52.28	20.41
24	69.3	0	13.9	3	9.8	12	64.17	0.00	12.87	2.78	9.07	11.11
25	40.1	12	19.5	0	9.1	0	49.69	14.87	24.16	0.00	11.28	0.00
26	46.5	15	24.5	0	38.8	21.7	31.74	10.24	16.72	0.00	26.48	14.81
27	48.8	17.4	23	0	25.2	0	42.66	15.21	20.10	0.00	22.03	0.00
28	29	16.2	20.2	0	58	0	23.50	13.13	16.37	0.00	47.00	0.00
29	26.2	14.4	16.3	0	83.6	0	18.65	10.25	11.60	0.00	59.50	0.00
30	19.5	9.3	20.4	0	82.6	22.8	12.61	6.02	13.20	0.00	53.43	14.75
31	30.3	14.9	23.1	0	34.5	20.2	24.63	12.11	18.78	0.00	28.05	16.42
32	23.8	21.2	23.1	0	113.1	10.9	12.39	11.04	12.02	0.00	58.88	5.67
33	27.5	18.6	18.9	0	94.4	26.7	14.78	9.99	10.16	0.00	50.73	14.35
34	27.7	20.8	20.7	0	97.8	13.9	15.31	11.50	11.44	0.00	54.06	7.68
35	28.1	22	20.2	0	107.2	0	15.83	12.39	11.38	0.00	60.39	0.00
36	27.9	21.5	16.6	0	110.9	0	15.77	12.15	9.38	0.00	62.69	0.00
37	11.7	21.7	16.3	0	144	31.6	5.19	9.63	7.23	0.00	63.91	14.03
38	18.3	21.6	22.7	0	99.7	0	11.28	13.31	13.99	0.00	61.43	0.00
39	2.5	23.3	51.3	0	109.4	23.7	1.19	11.08	24.41	0.00	52.05	11.27
40	0	26.6	47	0	60	55	0.00	14.10	24.92	0.00	31.81	29.16
41	0	22.4	95.1	0	70.3	35	0.00	10.05	42.68	0.00	31.55	15.71
42	0	23.3	83.2	0	55.8	40	0.00	11.52	41.13	0.00	27.58	19.77
43	0	29.3	34.8	0	96.4	58.9	0.00	13.35	15.86	0.00	43.94	26.85
44	0	20.6	61.9	0	77.7	54	0.00	9.62	28.90	0.00	36.27	25.21
45	0	40.9	29.5	0	54.6	93.7	0.00	18.70	13.49	0.00	24.97	42.84

FIGURE 4.1
Carbon Contents of Spent Catalysts from POC-1 Withdrawals

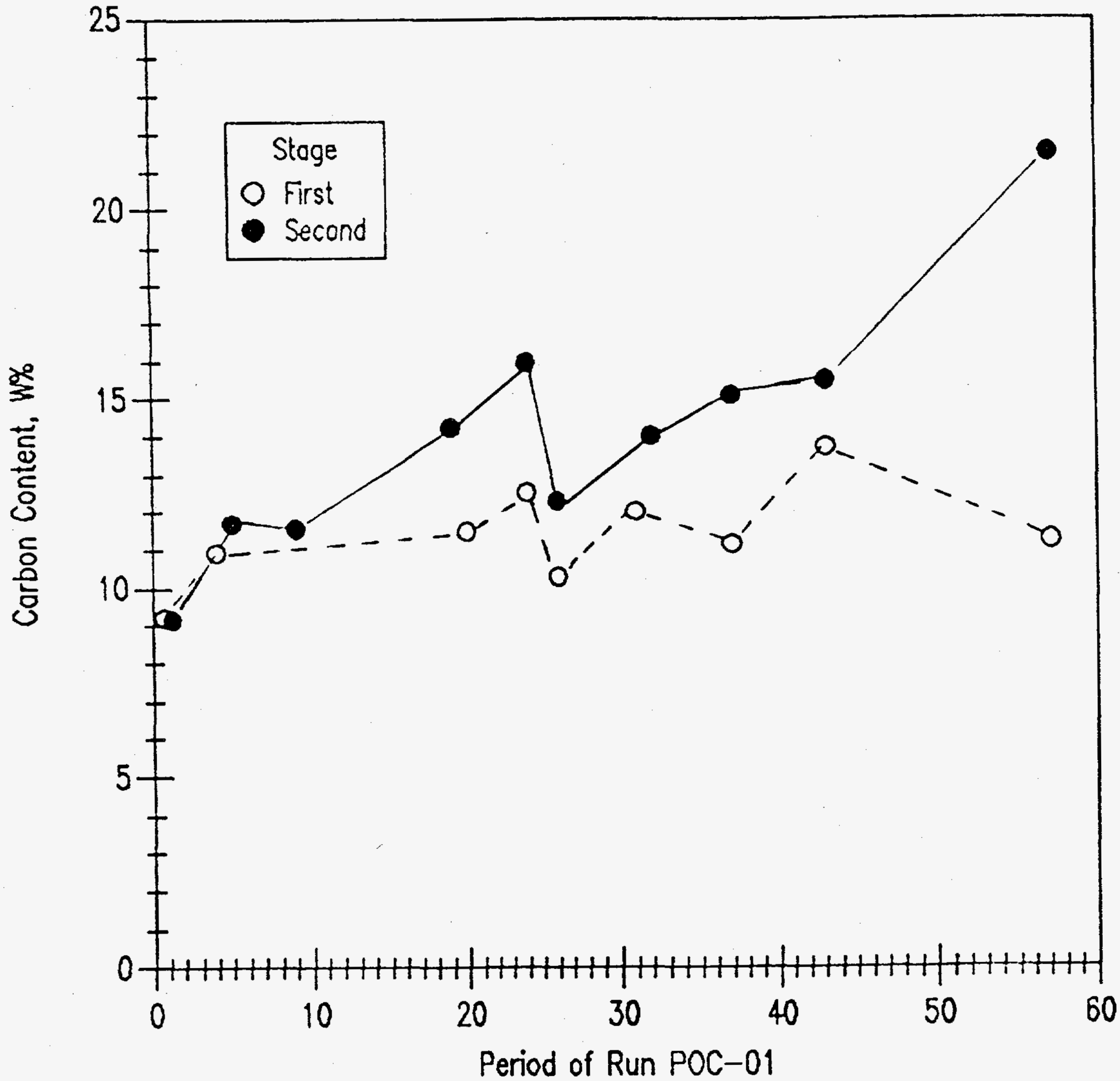


FIGURE 4.2
Contamination of Spent Catalysts from POC-1 Withdrawals

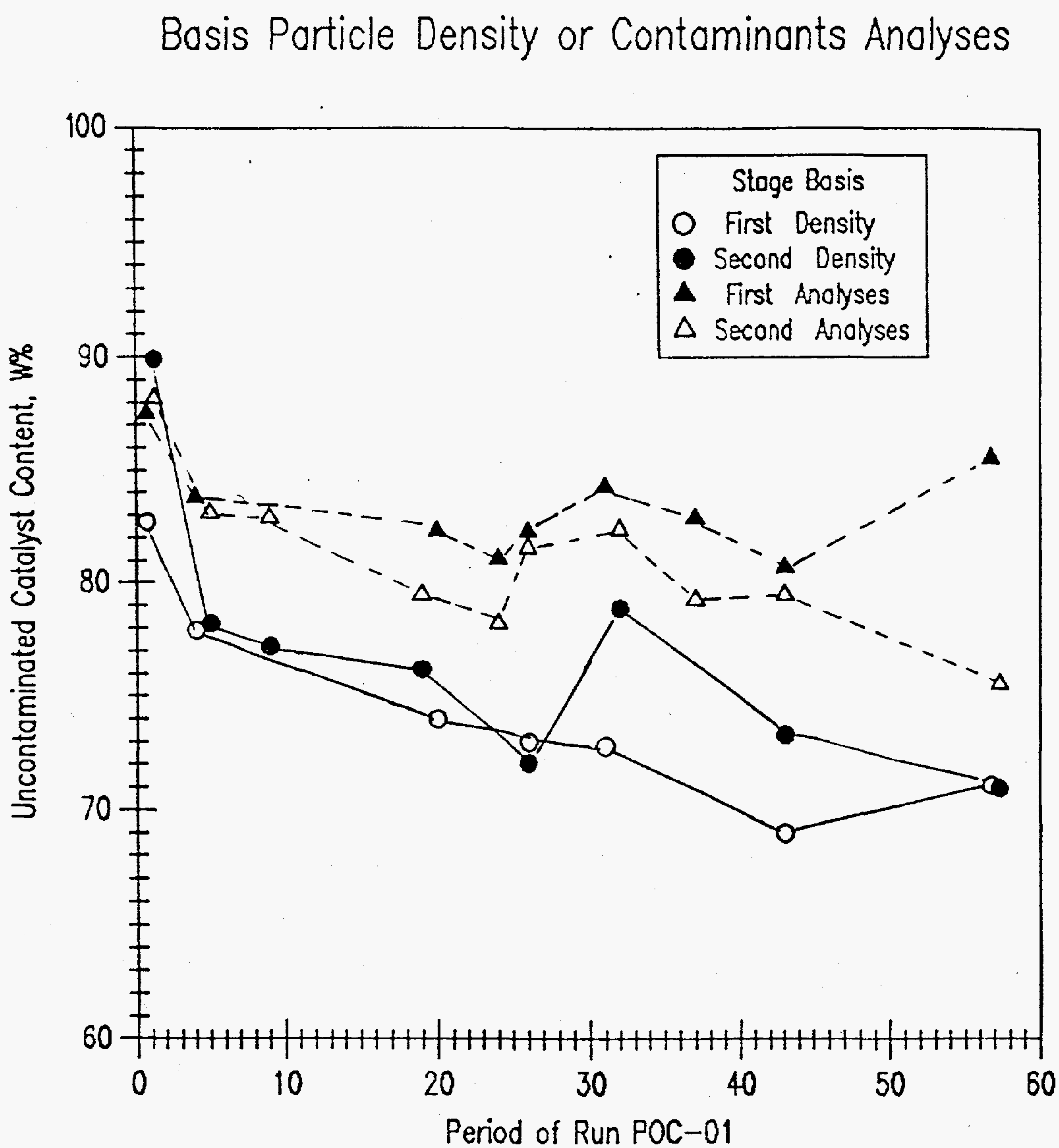


FIGURE 4.3
Relative Activity of Spent Catalysts from POC-1 Withdrawals

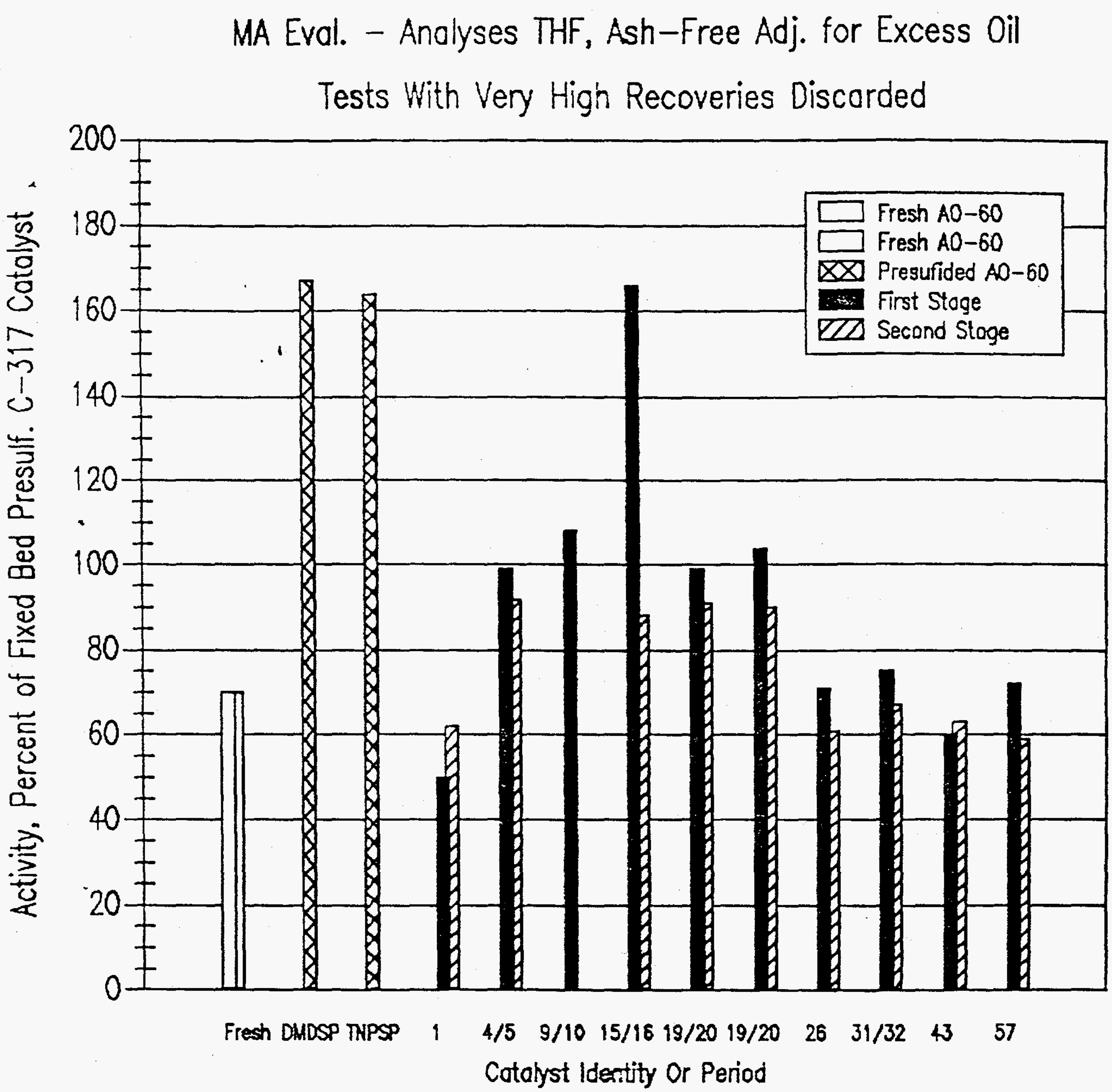


FIGURE 4.4
Target vs. Actual Catalyst/Process Equilibration

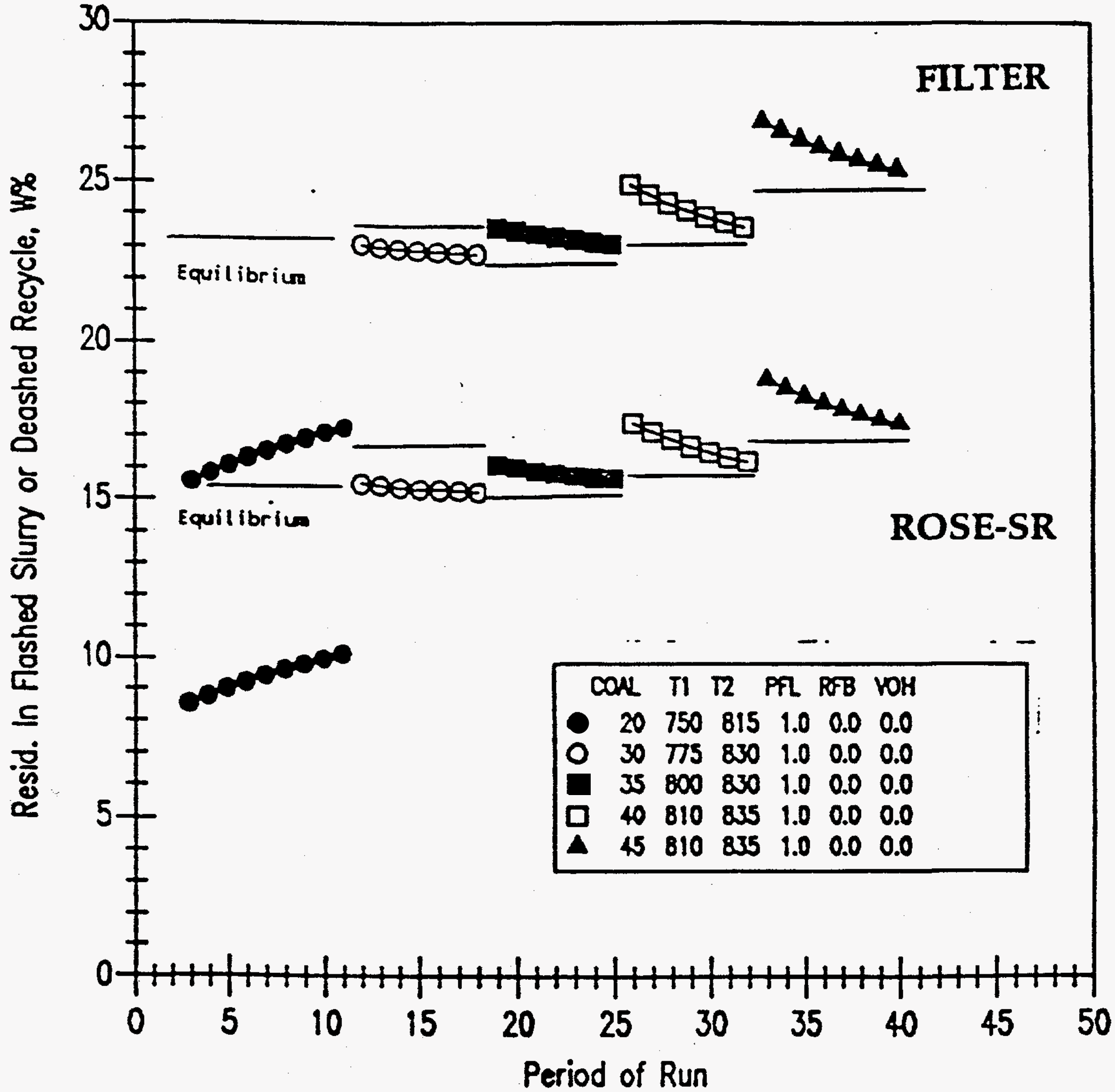


FIGURE 4.5
Catalyst Age Profiles

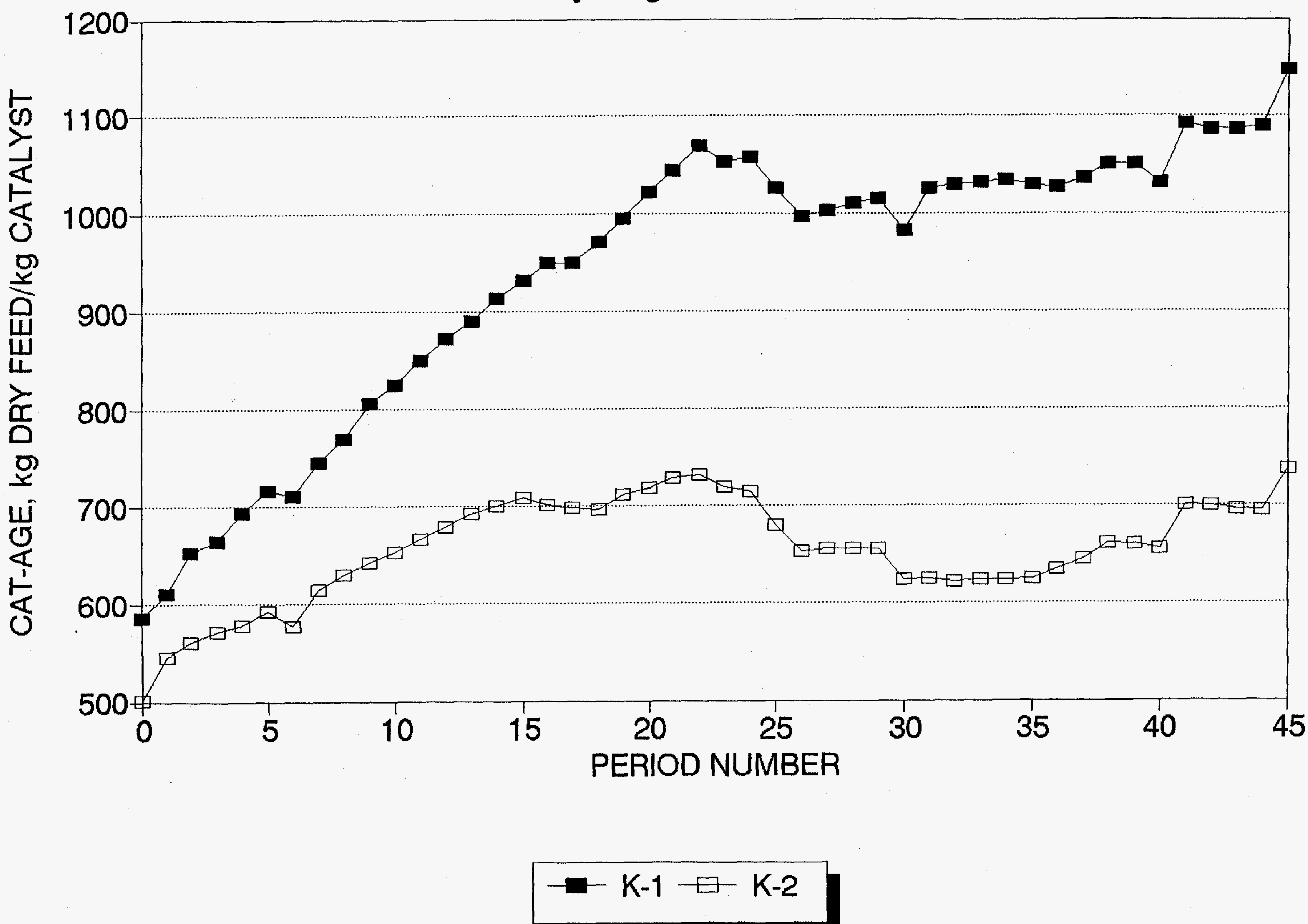


FIGURE 4.6
Weight % of -20 Mesh Fines in Daily Catalyst Withdrawals

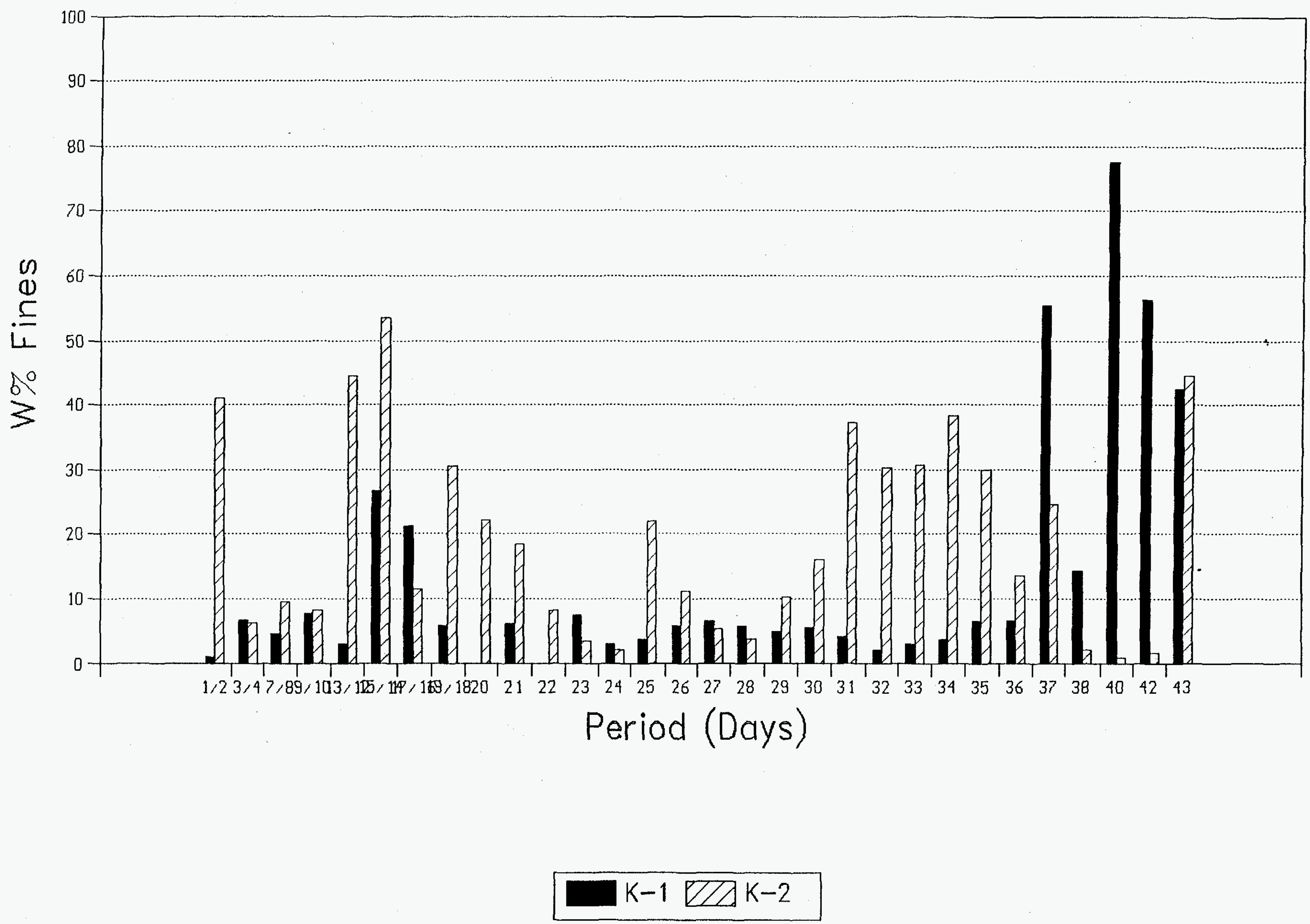


FIGURE 4.7
Target vs. Actual Catalyst Inventories (End of Run)

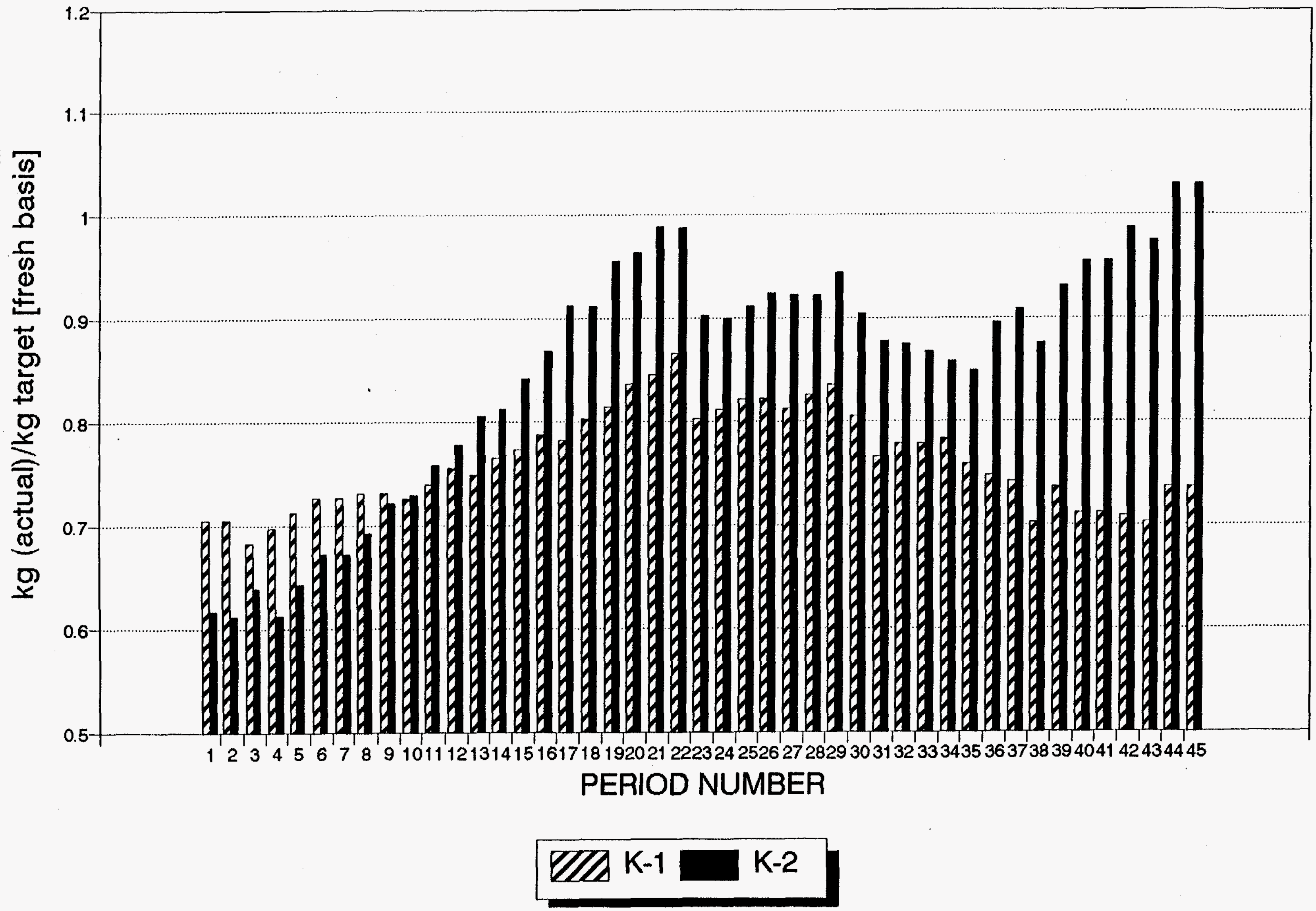


FIGURE 4.8
Catalyst Fines Lost through O-13 Bottoms

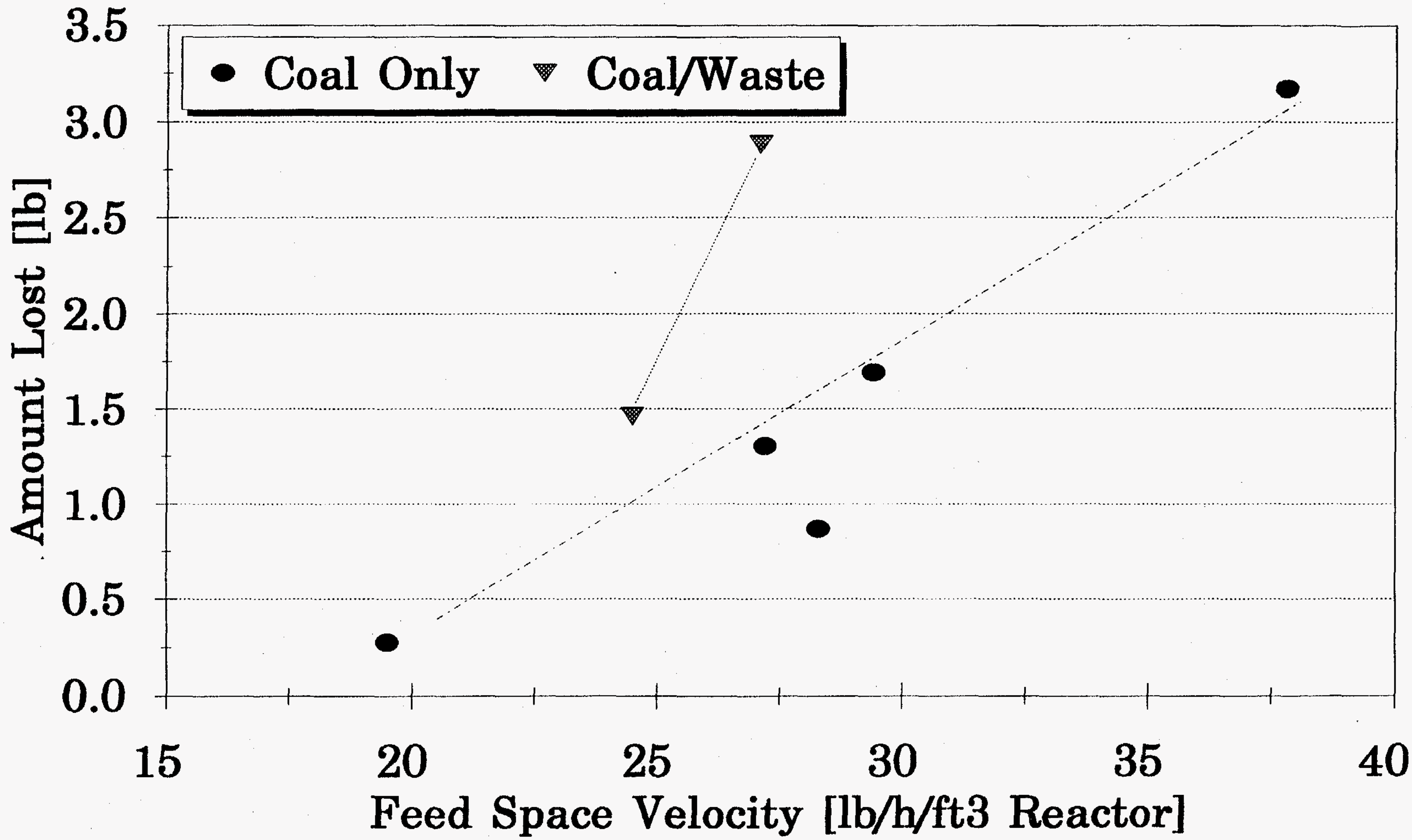


FIGURE 4.9
Periods 1 through 23 Recycle Stream Composition

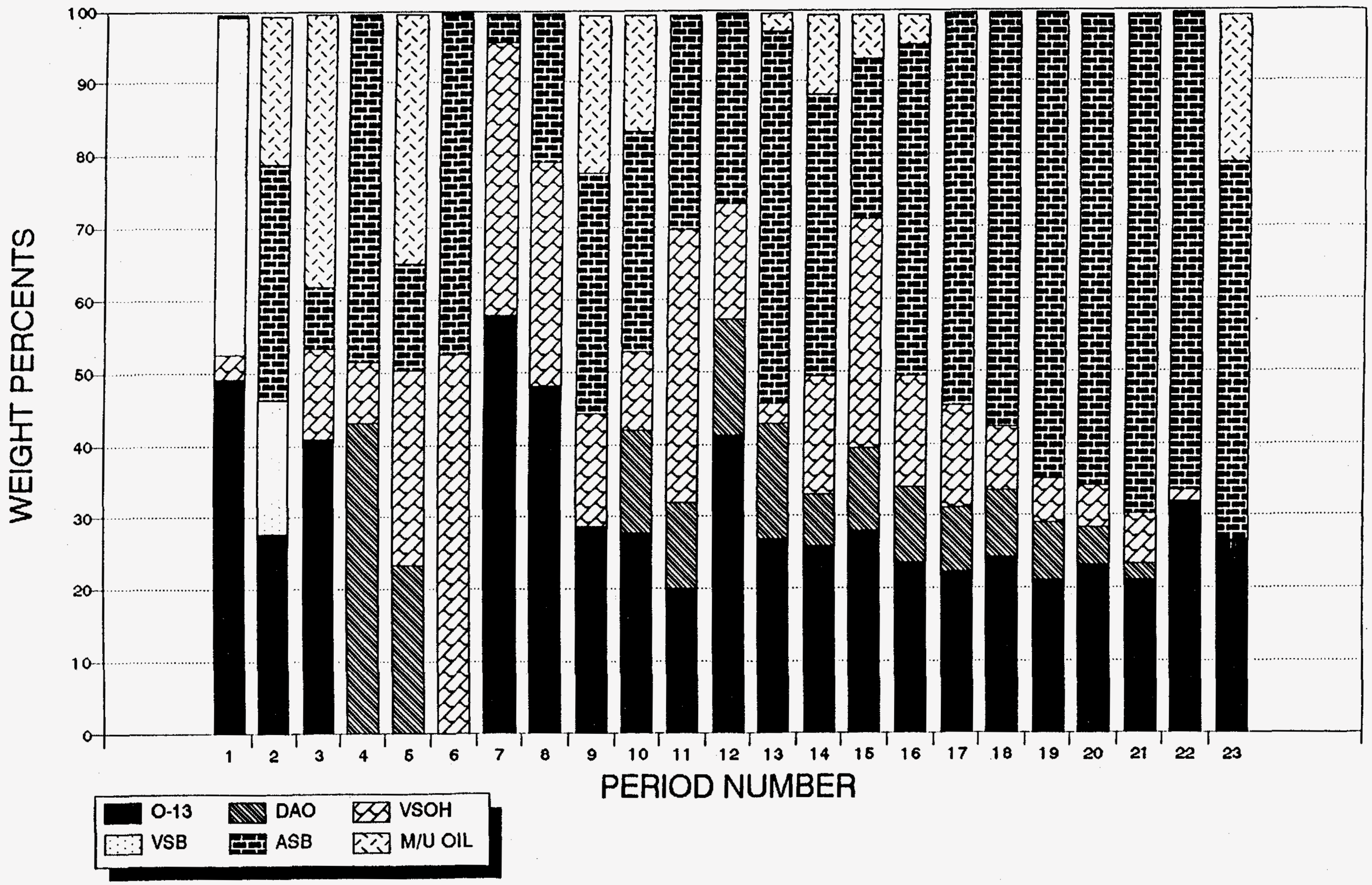
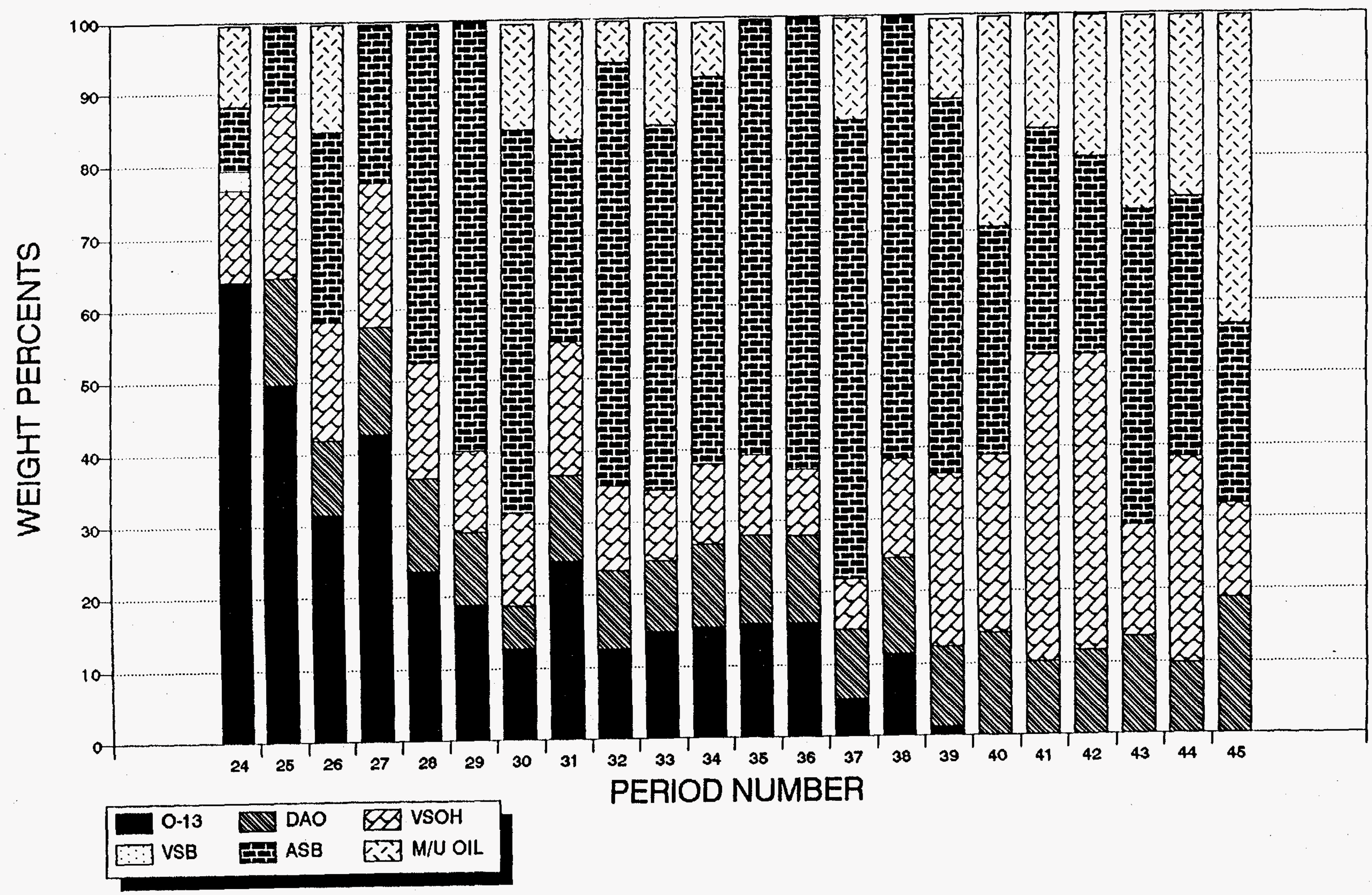


FIGURE 4.10
Periods 24 through 45 Recycle Stream Composition



C. PRODUCT QUALITY

The product streams from PDU operations are vent gas, bottoms gas, sour water, naphtha stabilizer bottoms and ash reject (ROSE-SR^{SM} bottoms). Analyses of these streams and several internal streams (atmospheric still bottoms, vacuum still overheads, feed slurry, recycle oils and reactor liquid flash drum bottoms) are given in Appendix C.

The qualities of selected product and internal streams are discussed in this section.

C.1 Naphtha Stabilizer Bottoms

Naphtha stabilizer bottoms (NSB), the overhead stream from the Atmospheric Still Column, was slated to be the sole liquid product stream under resid extinction mode in POC-02. In an ideal extinction recycle mode, all the material boiling above 343°C gets recycled. This means most of the vacuum still overheads and atmospheric still bottoms are recycled with the other heavy internal streams, such as O-13 bottoms and deasphalted oil. As seen from Figures 3.5 through 3.8 in Section V, this was not really the case during POC-02; and, indeed, net liquid products (7.8-8.7 W% MAF) in the boiling range of 343-524°C were produced due to contributions from the unrecycled portions of ASB and VSOH. After POC-01, the improved capacity of the oil/water separator allowed NSB product to be obtained during Run POC-02 with negligible amounts of water. In POC-02 the product recovery section was configured so that the feed to the Atmospheric Still was composed of products from the Hydrotreater.

NSB quality was, to some extent, positively influenced by changes in process severity, although the H/C ratios and heteroatom contents did not vary much with process severity, primarily because most of the NSB is composed of hydrotreated material. This product was very clean in general, with less than 50 ppm nitrogen and 10 ppm sulfur; API gravities were generally high (>35°), and the H/C atomic ratio was around 1.8. The increase in process severity after Period 21 improved the quality of the NSB product. The lightest naphtha fraction (IBP-177°C) increased from about 22 w% to over 35 w% at the expense of the heavier boiling fractions during conditions with slightly higher process severity. Period 36, which employed a molybdenum-additive, also indicated an improved API gravity and H/C ratio for the NSB product. The properties of the NSB did not change significantly during the co-liquefaction periods.

The NSB target cut point was 343°C (650°F). However, in most cases the end point was in the range of 350-380°C (660-716°F), reflecting the inefficiency of the fractionation operation. The main product was separated into four boiling point fractions by ASTM D-86 distillation. The weight distribution of these fractions:

Light Naphtha (IBP-177°C)	18.0-39.2 Wt%
Heavy Naphtha (177-288°C)	28.9-54.01 Wt%
Light Distillate (288-343°C)	5.15-35.4 Wt%
Heavy Distillate (343°C+)	0.6-17.3 Wt%

as shown in *Table 4.3*

NSB from Conditions 2B to 4C contained over 80 Wt% of material boiling below 288°C (550°F). This material was rich in hydrogen, ranging from 13.4 to 13.7 Wt%, and contained less than 5 and 40 ppm of sulfur and nitrogen, respectively. (Sulfur determinations were performed on caustic washed samples.) The in-line hydrotreater was by-passed during Periods 34B and 44B of the operation. The by-passed overhead stream, O-5, was, for both the periods in question, found to be much lower in quality (H-content, heteroatoms, and API gravity) than the regular NSB product that had gone through the in-line hydrotreater. The O-5 oil, obtained during Periods 34B and 44B, contained only 11.3-11.5 w% hydrogen, as high as 1400 ppm nitrogen and 370-680 ppm sulfur; the API gravities were also lower (23-26) compared the hydrotreated samples. Detailed analyses of un-hydrotreated O-5 oil and its comparison with the regular NSB products is shown in Table 4.3. Inspections of NSB products, obtained during 'co-liquefaction' conditions, are shown in Figure 4.11

C.2 Sour Water

In addition to water in the feed coal and generated from hydrogenation of oxygen in coal, water was injected downstream of the hydrotreater to avoid salt build-up. As part of the elemental balance, nitrogen and sulfur in the sour water were determined. The sour water stream contained dissolved ammonia and hydrogen sulfide. Typical nitrogen and sulfur contents were 0.3-1.1 Wt% and 0.04-0.33 Wt%, respectively, as given in *Table 4.4*.

A special sample of sour water was collected in Period 36 and analyzed by Environmental Science & Engineering, Inc. of Plymouth, PA. The results of these analyses are shown in *Table 4.5* along with the allowable limit for each category. Sour water will require special treatment as per the inspection indicated in this report. The average sour water flowrate was about 123.1 kg/h which was almost three times as high as that during POC-01 operations. This was because the water/coal ratio was higher for POC-02 than POC-01 (3.1 vs. 1.9). Also, the moisture content of POC-02 Wyoming Black Thunder coal was about 11 % as compared to only 4 % for Illinois No. 6 coal used during POC-01 operations. Most POC-02 sour water samples showed only 0.23 mg/l for phenolics. This means a phenol recovery unit would not be required for a commercial plant operating under conditions similar to POC-02 Period 36. This water sample was slightly basic with a pH of 8.67.

C.3 Reactor Liquid Flash Drum Bottoms (O-46)

Slurry product from the O-1 Hot Separator was flashed in the Reactor Liquid Flash Drum (O-13). The bottoms from O-13 became the feed to the recycle liquid/solids separation system. In POC-02 the O-13 bottoms stream (also called O-46 slurry) served as a tie between the coal liquefaction and the solids separation sections. For this reason O-46 slurry was characterized in detail. Based on the analysis of O-46 slurry, the performance of the coal liquefaction and the solids separation systems were determined.

The composition of O-46 slurry is summarized in *Table 4.6*. In general, there was 9.2 to 12.7Wt% unreacted coal in O-46 slurry. Ash levels were on the high side, between 6.7 to 12.1 Wt%, as the ashy recycle mode was practised for most of the 'coal-only' operations of POC-02. The ash level in O-46 slurry dropped approximately 50% to about 3.5 Wt% during 'co-liquefaction' Periods 43 and 45, when solid-free recycle was implemented.

The overall quality of O-46 slurry followed a trend similar to that for the naphtha stabilizer bottoms. The H/C ratios were in the same range as the deasphalted oil throughout the run. The H/C ratio was high, around 1.31 to 1.33 in Conditions 1B and

2B and declined thereafter to a low level of 1.08-1.13. The decline in quality was generally due to an increase in space velocity. The H/C ratio bounced back to around 1.33 during the 'co-liquefaction' periods. The 524°C (975°F) resid content followed a similar trend. The O-46 slurry contained 18.0 to 25.5 Wt% solids-free resid at the beginning of the run (Conditions 1B and 2B) and increased to 24.9-30.7 Wt% in the second half of the run; the resid content decreased to 19-24 Wt% during 'co-liquefaction' operations. As mentioned earlier, a significant amount of catalyst fines were lost in the O-13 bottoms stream. The molybdenum analysis of the O-13 bottoms is listed in Table 4.7.

C.4 Atmospheric Still Bottoms and Vacuum Still Overheads

Atmospheric still bottoms (ASB) was a major component of the recycle solvent, especially during solids-free recycle mode, as discussed earlier in this section (Solvent Composition). The relative proportion of ASB in the recycle solvent is compared with the concentration of vacuum still overheads (VSOH) in the table below:

Wt% ASB and VSOH in Recycle Solvent		
Periods	**ASB**	**VSOH**
9-15	22.3-51.4	3.1-38.1
18-22	57.3-69.3	1.9-9.1
30-33	28.1-53.4	10.1-13.2
34-36	54.1-62.7	10.1-11.4
41-43	27.6-43.9	15.9-42.7
44-45	25.0-36.3	13.5-28.9

The API gravity of ASB ranged from 15.6 to 22.9°. The amount of 343°C- (650°F-) distillate varied considerably from one condition to another, during POC-02: from a range of between 14-24 V% for Conditions 1B and 2B, to as high as 60 V% for Period 36, and between 33-44 V% during the 'co-liquefaction' periods. The initial boiling point and end point were in the range of 247-303°C (477-577°F) and 432-485°C (810-905°F), respectively. Detailed boiling point distributions (determined by ASTM D1160 distillation), API gravities and elemental analyses of selected periods are given in *Table 4.8 and Appendix C.* This stream was very rich in hydrogen and low in heteroatoms.

Vacuum still overheads were recycled through the Clean Oil Tank. The VSOH IBP varied slightly between 257-309°C (494-588°F) and contained from 5.36-27.0 Wt% boiling below 343°C (650°F). *Table 4.9* presents the boiling point distributions and elemental analyses of VSOH collected from the workup periods of each condition. As anticipated, the hydrogen content of VSOH was generally lower than that of ASB.

D. ROSE-SR^{SM} Solids Separation Unit

Prior to the start of POC-01, HRI installed a ROSE-SR^{SM} solids-separation unit, licensed by Kerr-McGee Corporation, as part of the Proof-of-Concept direct coal liquefaction facility. Major equipment for this unit was obtained from the Wilsonville Advanced Liquefaction facility; however, extensive new equipment was added, and the flow scheme was modified. The POC ROSE-SR^{SM} unit, as shown in Figure 2.3 was designed to use a pentane solvent in place of the toluene and mixed solvents employed at Wilsonville. The lighter pentane solvent is preferred due to the improved quality (lower preasphaltene content) of the resid with the latest Catalyst Two-Stage Liquefaction (CTSL) Technology.

Also, the third settler stage used at Wilsonville was eliminated, providing a single liquid deasphalted oil (DAO) product. The ROSE-SR^{SM} unit feed is the vacuum tower bottoms which is nominally a 454°C+ (850°F+) slurry stream. The purpose of the ROSE-SR^{SM} unit is to separate solids (ash and unconverted coal) from liquefaction bottoms to recover a solids free recycle oil for coal liquefaction and to reject a solids-containing product. When operating properly, a fine powder solid product is produced. This ash concentrate can be used as feed for gasification (for hydrogen or fuel gas production) or for combustion (for steam or power generation).

D.1 Specific Objectives and Performance Projections

The specific goals for the operation of the ROSE-SR solid separation system were set out at the beginning of the POC-02 Run. These included a minimum 14 continuous days of integrated operations, operability with a mixed n-pentane/toluene solvent, evaluation of the effects of solvent type, achieving less than 1 Wt% solids in the deasphalted oil (DAO), energy rejection of less than 15% and a QI content of more than 65 Wt% in the ROSE bottoms product. Some projections were also made to predict the performance of ROSE-SR during POC-02. The CAS bottoms product from one of the CMSL Bench Runs, CMSL-04, 227-81, was used to study the solubility of the 343°C+ heavy slurry product in solvents, such as n-pentane, toluene, and quinoline. Based upon the results from these CAS bottoms samples obtained from the direct liquefaction of Wyoming Black Thunder mine coal during Run 227-81, performance in terms of the compositions of the ROSE-SR feeds and the recycled streams was predicted. This is shown in Table 4.10. The projections indicate a steady increase in the n-pentane insoluble ROSE bottoms product in going from Condition 1 to Condition 4.

D.2 Integrated Operations

Starting from Period 1 of POC-02, the Residuum Oil Supercritical Extraction - Solids Rejection (ROSE-SR^{SM}) unit was brought on-line as an integral part of the liquefaction operations. Deasphalted oil (DAO) from the ROSE-SR^{SM} was recycled, while the unreacted coal and mineral matter were rejected as a solids-rich bottoms stream.

Detailed analyses of the ROSE DAO (deasphalted oil) and bottoms is listed in Appendix C. The QI of the ROSE bottoms ranged between 46 and 69 Wt%, while the pentane insolubles varied between 62-95 Wt%. Coal conversion, based upon the QI/ash ratios in the ROSE bottoms, varied between 86-93 Wt% maf. Coal conversion based upon O-13 bottoms and ROSE bottoms were similar for the 'coal-only' periods; however, during 'co-liquefaction' operations, coal conversion, based upon ROSE bottoms analyses were significantly lower (7-13 Wt% maf) than those based upon analyses of O-13 bottoms. This difference may be due to 'retrograde reactions' occuring in the solid separation system with the highly reactive plastics/rubber derived products during co-liquefaction operation. The ROSE bottoms contained between 10-24 Wt% of IBP-524°C distillable material. The bottoms products were, in general, dry powdery to lumpy (solids). The overall performance of ROSE-SR, in terms of the relative contents of asphaltenes and solids in the feed and the residual streams, is shown in Figure 4.12. The ratio of asphaltenes/solids in the O-63 bottoms residual product was similar to that in the feed, O-61. The variation in the solubility properties of these two streams (O-61 and O-63) is displayed in Figures 4.13 and 4.14. The solubilities of the residual O-63 products are much lower (by as much as 35-40%) than those of the feed. This indicates the success and effectiveness of ROSE-SR as a deashing operation.

ROSE performance improved through the course of the run. During the initial phase of integrated operations, a considerable portion (29-53 Wt%) of the solids remained in the DAO. Following this solid content of the DAO was mostly below 2 Wt%, (with the exception of Periods 17 and 24) during 'coal-only' operations. The solids content went up (3-20 Wt%) during 'co-liquefaction' operations. The daily ash-balance of the PDU is shown in Table 4.12.

D.2a Organic and Energy Rejections

Organic and estimated energy rejections are listed in *Table 3.1* in Section V, Process Performance. The lowest organic (12.7 Wt% maf coal) and energy (12.0 Wt% coal) rejections were attained in Condition 4C. Organic and energy rejections were in the lower 20s during the first three conditions of POC-02; they decreased to below 15 Wt% of maf coal feed during Conditions 4C and 5, while rejections increased during 'co-liquefaction' operations. This could be due to the sudden change in the chemical composition and solubility properties of the feed to the ROSE unit when rubber and/or plastics were co-fed to the PDU.

D.2b Summary of ROSE-SR^{SM} Operations and Performance

The ROSE-SR^{SM} unit was successfully commissioned and operated during POC-02. Both the off-line tests and on-line service resulted in effective rejection of unreacted coal and mineral matter from the two-stage liquefaction of Wyoming Black Thunder Mine coal. Within a relatively short time, performance of the ROSE-SR^{SM} unit at HTI, was comparable to that achieved at Wilsonville.

As shown in Table 3.1, ROSE-SR as a deasher performed exceedingly well, resulting in as low as 11-12 Wt% organic and energy rejections, less than 2 Wt% rejection of distillable 524°C- material, without any noticeable degradation of coal conversion. The overall composition of the recycle stream, containing the ROSE-SR deasphalted oil (DAO), is listed in Table 4.11. The resid and solids content in the actual recycle stream are depicted in Figures 4.15 through 4.18.

E. On-line Hydrotreater

After the by-passing problems experienced in the in-line fixed-bed hydrotreater during POC-01, the reactor was redesigned by removing the baskets which held the catalyst beds. The new hydrotreater design two extrudate catalyst beds, separated to provide a quenching zone, gave an excellent performance in terms of achieving high levels of hydrogen addition and heteroatoms removal levels for the net process distillates. The typical nitrogen contents of the NSB product during POC-02 varied between 1-52 ppm, while the sulfur contents varied between 1-25 ppm. High H/C ratios (1.7-1.8) were also obtained. Figures 4.19 and 4.20 depict the nitrogen and sulfur contents of NSB distillates and the HTU temperatures.

The hydrotreated distillates collected for the end-use and upgrading studies for the other DOE contractors were much cleaner than those obtained during POC-01. These process distillates had API gravities between 32-36°, hydrogen contents of about 13 Wt%, and nitrogen and sulfur levels below 50 and 10 ppm respectively. To determine hydrotreater performance during POC-02, it was by-passed during Periods 34 and 44. Table 4.13 shows the analyses of O-5 cold separator oil and O-12 reactor flash vessel overhead receiver, both with hydrotreater on-line and off-line. When the hydrotreater was taken off-line, the heteroatom contents of the distillate liquids streams, went up significantly. The nitrogen content increased to about 2433-3323 ppm, while the sulfur content was up to 373-793 ppm. The hydrogen contents went down from about 12.2 to only 11.0 Wt%. Thus, the overall quality of distillates degraded significantly when the in-line hydrotreater was by-passed.

TABLE 4.3
Inspection of Naphtha Stabilizer Bottoms

Period	1	2	4	8	9	10	12	14	15	17
API	30.4	30.4	29.1	32.3	31.6	31.5	32.2	31.8	32.0	32.9
IBP	86	79	82	79	83	83	81	85	83	77
5, V%	109	105	112	103	107	107	107	108	106	97
10, V%	124	121	131	118	-4	123	123	123	122	111
20, V%	171	157	172	151	157	157	156	157	157	146
30, V%	216	206	217	192	199	202	194	196	196	198
40, V%	248	244	258	229	234	238	229	230	231	228
50, V%	276	273	286	257	262	263	253	256	257	256
60, V%	291	292	304	279	283	285	278	279	279	281
70, V%	307	308	321	296	301	302	297	296	296	297
80, V%	322	324	336	314	316	317	315	313	313	316
90, V%	339	341	353	333	334	334	334	332	333	336
95, V%	-18	356	368	346	348	346	349	343	346	351
EP	376	378	383	367	364	367	368	362	367	365
Distribution, W%										
IBP-177C		20.7	18.0	23.0		21.2	22.3	21.3	21.6	
177-288C		33.6	28.9	38.3		37.4	40.0	39.3	39.7	
288-343C		34.1	35.4	30.8		32.8	29.7	30.9	30.6	
343C+		11.0	17.3	7.4		7.9	7.8	8.3	7.6	
Loss		0.6	0.4	0.5		0.7	0.2	0.2	0.5	
Distribution, V%										
IBP-177C		24	21	26		24	25	25	25	
177-288C		34	30	39		38	40	40	40	
288-343C		33	34	29		31	28	30	29	
343C+		8.5	14.5	5.5		6.5	7	4	5.5	
Whole										
API										
Carbon, W%							86.76		86.82	
Hydrogen, W%							12.72		12.83	
Nitrogen, ppm	135.8	<1.0	51.3	29.4	42.9	20.0	143.3	89.8	0.12	9.0
N ppm (Washed)		<1.0	46.5	26.6	40.0	16.1	137.0	80.0	0.2	7.5
Sulfur, ppm	544.4	26.5	51.9	4.6	18.1	21.5	7.3	7.2	5.5	5.0
S, ppm (Washed)		17.2	24.3	0.8	6.1	4.0	7.1	1.5	0.5	1.8
IBP-177C Fraction										
API									50.8	
Carbon, W%									85.47	
Hydrogen, W%									14.17	
Nitrogen, ppm									<0.0	
Sulfur, ppm									<0.0	
177-288C Fraction										
API									31.9	
Carbon, W%									87.73	
Hydrogen, W%									12.99	
Nitrogen, ppm									<0.1	
Sulfur, ppm									<0.0	
288-343C Fraction										
API									23.2	
Carbon, W%									88.01	
Hydrogen, W%									12.39	
Nitrogen, W%									2.0	
Sulfur, W%									<0.1	
343C+ Fraction										
API									19.7	
Carbon, W%									88.22	
Hydrogen, W%									12.11	
Nitrogen, ppm									2.3	
Sulfur, ppm									3.2	

Table 4.3 Contd.

Period	19	21	24	28	29	31	33	34	36
API	35.0	35.8	33.4	35.2	34.2	32.8	35.2	34.9	38.0
IBP	77	75	84	77	78	79	72	78	75
5, V%	95	97	115	102	101	108	99	102	98
10, V%	108	108	132	118	118	123	113	116	109
20, V%	133	131	174	144	145	153	139	139	125
30, V%	166	159	219	172	175	186	164	167	146
40, V%	196	189	246	199	207	218	197	195	168
50, V%	224	216	264	226	232	243	226	220	194
60, V%	246	239	277	249	256	268	246	242	214
70, V%	269	258	289	269	276	288	265	261	234
80, V%	289	278	302	291	294	305	281	278	254
90, V%	311	304	318	313	314	326	301	298	275
95, V%	326	320	331	332	332	341	314	312	287
EP	352	338	349	349	349	360	330	326	309
Distribution, W%									
IBP-177C		32.51					29.47		39.16
177-288C		50.59					52.07		54.01
288-343C		16.08					16.57		5.15
343C+		0.59					1.42		1.20
Loss		0.23					0.47		0.48
Distribution, V%									
IBP-177C		36	21	32	31	27	33	33	43
177-288C		50	48	47	46	43	51	52	52
288-343C		13.5	29	18	20	26	15	14	5
343C+		0.5	1.5	2	2	3			
Whole									
API		35.8	33.4	35.2	34.2	32.8	35.2	34.9	38.0
Carbon, W%		86.55					86.94		86.91
Hydrogen, W%		13.1					12.78		12.99
Nitrogen, ppm	46.4	51	53	42	45.4	28.6	24.5	51.0	16.6
N ppm (Washed)	44.5	48.9	52.2	39.2	44.9	25.9	22.7	48.0	44.0
Sulfur, ppm	11.6	5	36	7.7	13.5	12.4	13.8	12.0	17.4
S, ppm (Washed)	4.4	1.9	20.2	4.5	1.4	3.1	2.14	6.0	0.5
IBP-177C Fraction									
API									
Carbon, W%							85.82		85.87
Hydrogen, W%							15.06		14.04
Nitrogen, ppm							1.7		9.0
Sulfur, ppm							<0.5		<0.5
177-288C Fraction									
API									
Carbon, W%							87.86		87.84
Hydrogen, W%							12.51		12.54
Nitrogen, ppm							30.7		56.7
Sulfur, ppm							0.1		0.8
288-343C Fraction									
API									
Carbon, W%							88.03		88.33
Hydrogen, W%							11.81		11.83
Nitrogen, W%							35.2		62.1
Sulfur, W%							3.2		4.6
343C+ Fraction									
API									
Carbon, W%							88.2		88.29
Hydrogen, W%							11.46		11.63
Nitrogen, ppm							52.1		66.5
Sulfur, ppm							16.7		8.9

Table 4.3 Contd.

							End Use Product (Trailer)		
Period	37	39	41	42	43	45	Front	Middle	Rear
API	36.2	29.3	30.6	31.6	30.8	32.8	32.1	36.2	34.9
IBP	76	84	79	84	78	69	81	73	83
5, V%	102	116	113	117	113	103	103	101	105
10, V%	115	135	128	130	127	121	119	113	117
20, V%	139	171	159	153	148	152	153	136	139
30, V%	166	217	189	181	176	188	196	161	172
40, V%	193	254	232	219	213	224	231	189	204
50, V%	217	277	259	253	251	248	257	213	227
60, V%	236	293	281	277	274	269	279	237	252
70, V%	251	304	296	294	292	288	296	256	271
80, V%	264	317	312	312	306	303	312	275	293
90, V%	280	331	327	329	322	326	332	295	312
95, V%	291	342	340	343	334	341	345	308	332
EP	307	357	357	363	355	353	363	333	348
Distribution, W%									
IBP-177C			22.91	25.80		24.04	22.43	32.82	28.12
177-288C			37.46	37.79		43.32	38.84	51.30	48.59
288-343C			32.99	29.26		27.53	30.29	13.86	18.94
343C+			6.07	6.34		4.88	7.86	1.66	3.65
Loss			0.57	0.81		0.23	0.58	0.36	0.7
Distribution, V%									
IBP-177C	340	21	26	29	31	27	25	36	31
177-288C	60	36	39	38	37	43	40	51	48
288-343C	5	39	32	28	29	26	30	13	18
343C+		4	3	5	3	4	5		2
Whole									
API	36.2	29.3	30.6	31.6	30.8	32.8	32.1	36.2	34.9
Carbon, W%			86.98	86.81	87.07	86.75	86.52	87.06	86.58
Hydrogen, W%			11.99	12.31	12.28	12.50	12.90	12.93	13.03
Nitrogen, ppm	18.0	65.6	47.0	57.2	29.5	39.0	53.5	21.2	48.4
N ppm (Washed)	16.0	64.1	46.0	51.4	26.1	35.5	52.0	19.7	44.8
Sulfur, ppm	11.0	65.3	48.5	43.7	29.9	32.8	8.0	10.0	<1.0
S, ppm (Washed)	2.4	40.9	25.4	13.9	9.4	15.4	7.3	2.0	<1.0
IBP-177C Fraction									
API									
Carbon, W%							85.43	85.82	85.56
Hydrogen, W%							14.21	13.87	14.06
Nitrogen, ppm							13.0	5.1	<1.0
Sulfur, ppm							<1.0	<0.5	<1.0
177-288C Fraction									
API									
Carbon, W%							86.93	87.40	87.53
Hydrogen, W%							13.02	12.49	12.95
Nitrogen, ppm							50.7	29.5	48.0
Sulfur, ppm							<1.0	<0.5	<1.0
288-343C Fraction									
API									
Carbon, W%							87.77	87.71	88.06
Hydrogen, W%							12.51	11.74	12.34
Nitrogen, W%							58.9	38.3	63.0
Sulfur, W%							5.0	9.0	0.9
343C+ Fraction									
API									
Carbon, W%							87.94	88.07	87.89
Hydrogen, W%							12.28	11.46	12.02
Nitrogen, ppm							63.0	81.4	67.6
Sulfur, ppm							17.0	39.9	7.8

TABLE 4.4
Nitrogen and Sulfur Content of Sour water

Period No.	Concentration, wt%	
	Nitrogen	Sulfur
4	0.61	0.33
8	1.11	0.14
12	0.65	0.06
15	0.74	0.09
21	0.73	0.08
33	0.52	0.04
36	0.48	0.04
42	0.33	0.28

TABLE 4.5
Sour Water Sample Analysis (POC-02 Period 36)

Inspection	Units	260-05-36	Limits
pH		8.67	6-10
Chloride	MG/L	5	
Nitrogen, Ammonia	MG/L	9,120	100
Sulfide, Total	MG/L	6,890	
Total Organic Carbon (TOC)	MG/L	1,110	
Phenols	MG/L	0.23	1
Total Suspended Solids (TSS)	MG/L	3	150
Biological Oxygen Demand (BOD)	MG/L	314	150
Chemical Oxygen Demand (COD)	MG/L	12,000	150

Note: Analyses performed by Environmental Science and Engineering, Inc. Plymouth, PA

TABLE 4.6
Inspection of the Reactor Liquid Flash Drum Bottoms (0-46)

Period		2	4	8	10	12	14	15	17	19
PFL, W%		54.44	63.16	52.96	69.10	60.47	68.54	72.28	59.84	54.25
PFS, W%		45.56	36.84	47.04	30.90	39.53	31.46	27.72	40.16	45.75
Pressure Filter Liquids										
API			5.2			7.3		8.0	11.0	8.1
Weight Distribution, W% PFL										
IBP-343C								7.20	3.73	5.92
343-454C			52.75			56.08		54.24	57.86	53.25
454-524C			23.48			20.20		20.22	19.46	18.05
524C+	25.28		23.19	21.87	19.20	23.33	19.68	18.05	18.45	22.29
loss			0.58			0.39		0.29	0.50	0.49
Toluene Insol. W% PFL										
Elemental Analysis, W% PFL										
Carbon						89.72		89.62		
Hydrogen						9.68		9.83		
Nitrogen						0.28		0.27		
Sulfur						0.69		0.02		
Pressure Filter Solids										
Quinoline Insol., W% PFS	49.84		56.69	59.93	60.13	55.60	58.33	60.74	59.77	58.97
QI Ash, W% PFS	17.06		23.47	21.86	25.18	23.55	20.7	23.16	23.94	26.32
S in Ash, W% Ash			3.03	3.59	3.17	2.52	2.35	2.68		2.2
ASTM Ash, W% PFS			23.36	21.03		24.34		23.62	23.91	
S in Ash, W% Ash										
Mo in Ash, ppm Ash						88				
TGA Data, W% PFS										
IBP-524C	40.05		35.23	34.49	32.47	34.01		32.77		33.41
524C+	59.95		64.77	65.51	67.53	65.99		67.24		66.59
Ash	17.12		23.74	21.29	25.67	25.77	21.72	24.38		27.32
Elemental Analysis, W% PFS										
Carbon						64.91		64.86		
Hydrogen						5.32		5.09		
Nitrogen						0.47		0.55		
Sulfur								0.63		

Table 4.6 Contd.

Period	21	22	24	28	31	33	34	36
PFL, W%	52.96		51.29	58.17	59.77	64.15	65.22	64.78
PFS, W%	47.04		48.71	41.83	40.23	35.85	34.78	35.22
Pressure Filter Liquids								
API	9.0					2.7		2.1
Weight Distribution, W% PFL								
IBP-343C	8.44					8.35		8.5
343-454C	52.04					46.30		44.19
454-524C	15.99					18.79		16.62
524C+	22.84		26.59	23.7	25.27	26.09	23.9	30.31
loss	0.69					0.47		0.38
Toluene Insol. W% PFL								
Elemental Analysis, W% PFL								
Carbon	89.96					90.17		90.37
Hydrogen	9.55					8.62		8.57
Nitrogen	0.28					0.37		0.44
Sulfur	0.154					0.042		0.02
Pressure Filter Solids								
Quinoline Insol., W% PFS	58.94		52.48	54.93	55.98	56.92	55.56	56.30
QI Ash, W% PFS	27.43		14.40	22.98	22.37	24.88	25.52	25.92
S in Ash, W% Ash	2.09		5.26	3.22	2.88	2.18	2.54	
ASTM Ash, W% PFS							26.58	26.56
S in Ash, W% Ash							2.4	2.63
Mo in Ash, ppm Ash	195							
TGA Data, W% PFS								
IBP-524C	33.92		37.64	35.79	32.41	32.27	34.92	31.51
524C+	66.08		62.36	64.21	67.59	67.73	65.08	68.49
Ash	27.42		13.79	23.22	24.63	25.99	27.00	27.80
Elemental Analysis, W% PFS								
Carbon	61.59					63.57		62.3
Hydrogen	4.83					4.57		4.26
Nitrogen	0.43					0.46		0.56
Sulfur						0.718		0.815

Table 4.6 Contd.

Period	37	39	41	42	43	44	45
PFL, W%	75.47	82.62	84.06	80.44	85.43	73.13	71.94
PFS, W%	24.53	17.38	15.94	19.56	14.57	26.87	28.06
Pressure Filter Liquids							
API			6.0	7.3	8.2	4.3	5.8
Weight Distribution, W% PFL							
IBP-343C			5.05	4.80	7.40	4.41	6.4
343-454C			48.88	56.08	55.08	63.34	60.14
454-524C			15.74	16.63	12.34	14.59	15.81
524C+	21.69	19.62	29.84	25.10	24.68	17.18	16.97
loss			0.49	0.39	0.50	0.48	0.68
Toluene Insol. W% PFL							
Elemental Analysis, W% PFL							
Carbon			89.49	89.29		90.12	89.84
Hydrogen			9.49	9.82		8389	9.2
Nitrogen			0.17	0.15		0.22	0.21
Sulfur			0.15	0.1		0.16	0.31
Pressure Filter Solids							
Cyclohexane Insol., W% PFS	64.01	68.41	68.81	68.99	68.54		48.47
CI Ash, W% PFS	26.26	21.53	25.37	17.72	24.24		14.45
Quinoline Insol., W% PFS	60.7	62.53	64.8	64.56	64.75		49.47
QI Ash, W% PFS	25.47	20.53	25.86	17.5	24.28		15.55
S in Ash, W% Ash	2.82	2.94		3.13	3.16		
ASTM Ash, W% PFS			17.86			15.8	
S in Ash, W% Ash			3.79	2.63	3.21	5.76	
Mo in Ash, ppm Ash							
TGA Data, W% PFS							
IBP-524C	32.67	33.54	29.47	32.65	31.83	44.95	46.49
524C+	67.33	66.46	70.53	67.35	68.17	55.05	53.51
Ash	28.2	21.34	26.57	18.4	25.43	16.07	15.99
Elemental Analysis, W% PFS							
Carbon			63.4	70.37	64.41	76.67	76.02
Hydrogen			4.67	5.3	5.06	5.22	5.33
Nitrogen			0.48	0.62	0.45	0.32	0.32
Sulfur			1.1	0.64	0.8	1.23	0.99

TABLE 4.7
Molybdenum Content in O-13 Bottoms

Period	Feed Type	Space Velocity [Kg/h/m^3]	Catalyst Addition* K1 [Kg]	K-2 [Kg]	----- O-13 Bottoms ----- Total [Kg]	Recycle [Kg]	To N-3 [Kg]
12	Coal	312	0.87	1.75	76.73	30.5	46.23
15	Coal	296	0.87	1.75	78.18	23.37	54.91
21	Coal	453	1.31	2.62	80.45	28.41	52.05
31	Coal	436	2.29	3.82	93.64	30.27	63.36
33	Coal	606	2.62	4.36	122.36	27.45	94.91
36	Coal	604	2.62	4.36	116.5	27.86	88.64
37	Coal/Plastic	471	2.62	4.36	106.32	11.68	94.64
41	Coal/Plastic	391	1.96	3.27	131.18	0	131.18
42	Coal/Plastic	393	0	0	135.55	0	135.55
43	Coal/Plastic	434	1.96	3.27	135.41	0	135.41
45	Coal/Plastic	399	1.96	3.27	152.32	0	152.32

*Catalyst Added a day before.

Period	Ash in O-13 [W%]	Mo Added [Kg]	Mo Content [ppm] [per Ash]	[per O-13]	Mo in O-13 Bottoms [Kg] Total	Recycle	To N-3	Eq. Fresh Cat. [Kg]
12	9.62		88	8	0.017	0.007	0.011	0.14
15	6.55		148					
21	12.90		195	25	0.054	0.019	0.035	0.436
31	9.00		360	32	0.080	0.026	0.054	0.653
33	8.92		675	60	0.195	0.044	0.151	1.587
36	9.35	0.55	3180					
37	6.25		2160	135	0.379	0.042	0.337	0.847
41	2.85		2300					
42	3.43		732	25	0.090	0.000	0.090	0.732
43	3.54		1400	50	0.177	0.000	0.177	1.444
45	4.36		3340					

Period 37: Assumed 50% of the 0.5Kg of Mo [equivalent to 4.48 Kg of fresh AO60] added in Period 36 was totally discharged in Period 37.

TABLE 4.8
Inspection of the Atmospheric Still Bottoms

Period	2A	2	4	8	10	12	14	15	17
API	19.0	22.9	18.7	19.8	19.4	19.2	20.8	21.0	22.3
IBP	284	291	254	276	288	278	274	288	291
5, V%	319	317	303	314	346	320	320	318	323
10, V%	331	329	336	326	328	335	328	329	335
20, V%	341	339	353	342	338	346	346	342	343
30, V%	348	354	364	350	349	360	354	349	352
40, V%	359	360	374	362	358	371	361	359	361
50, V%	368	367	382	375	368	379	373	372	372
60, V%	378	381	392	383	378	389	381	381	381
70, V%	388	392	401	397	387	398	392	394	391
80, V%	399	405	413	413	401	413	403	403	403
90, V%	419	427	429	432	418	431	424	427	421
95, V%	434	443	443	453	435	448	438	438	433
EP	456	464	473	485	464	471	469	468	463
Distribution, W%									
IBP-343C	22.3	21.7	13.5	25.7	22.2	14.7	19.3	23.3	19.7
343-454C	76.0	76.6	85.2	73.2	76.6	83.6	78.8	74.9	78.8
454C+	1.4	1.3	1.3	0.9	1.2	1.4	1.4	1.4	1.1
Loss	0.3	0.4	0.0	0.3	0.1	0.3	0.5	0.4	0.4
Distribution, V%	23.0	22.0	14.0	25.0	22.0	14.0	19.0	24.0	20.0
IBP-343C	76.0	77.0	85.0	74.0	77.0	85.0	80.0	75.0	79.0
343-454C									
454C+									
Whole									
Carbon, W%						87.8		87.2	
Hydrogen, W%						11.6		12.1	
Nitrogen, ppm						318.0		21.0	
Sulfur, ppm						54.1		15.0	

Table 4.8 Contd.

Period	19	21	24	28	31	33	34	36
API	21.2	21.5	21.5	20.2	19.0	19.3	15.6	20.3
IBP	266	252	303	253	264	263	278	267
5, V%	298	283	311	299	309	289	301	273
10, V%	310	295	316	314	322	305	314	279
20, V%	328	314	324	332	341	319	327	296
30, V%	342	327	335	342	348	331	339	308
40, V%	351	341	341	352	357	340	346	321
50, V%	361	349	349	362	368	352	357	333
60, V%	373	364	358	376	380	362	372	343
70, V%	383	374	371	388	388	374	382	359
80, V%	397	391	381	401	404	388	396	374
90, V%	417	411	400	421	426	404	412	401
95, V%	434	427	414	438	441	421	437	428
EP	464	454	443	474	474	458	468	454
Distribution, W%								
IBP-343C	33.66	44.87	42.05	30.87	26.28	41.17	34.82	59.35
343-454C	64.08	52.87	56.43	67.20	71.38	57.77	62.68	38.63
454C+	1.83	1.62	1.19	1.50	1.70	1.06	1.98	1.29
Loss	0.43	0.64	0.33	0.43	0.64		0.52	0.53
Distribution, V%								
IBP-343C	34	46	43	32	27	43	36	60
343-454C	65	53	56	67	72	56	63	39
454C+								
Whole								
Carbon, W%		87.33				88.11		87.82
Hydrogen, W%		12.01				11.35		11.39
Nitrogen, ppm		76.7				75.6		70.6
Sulfur, ppm		14.2				35.4		11.6

Table 4.8 Contd.

Period	37	39	41	42	43	45
API	21.8	19.0	17.8	18.6	19.1	17.9
IBP	285	268	272	245	274	247
5, V%	288	302	307	299	306	298
10, V%	292	312	318	311	311	309
20, V%	298	323	333	326	327	323
30, V%	305	333	341	341	337	336
40, V%	316	341	354	348	344	342
50, V%	327	348	362	355	353	352
60, V%	338	358	371	366	360	361
70, V%	347	372	378	378	373	373
80, V%	366	383	392	390	383	383
90, V%	387	402	412	407	403	403
95, V%	406	424	429	421	421	414
EP	432	450	447	453	444	441
Distribution, W%						
IBP-343C	65.87	43.40	31.11	36.06	37.87	39.92
343-454C	32.40	54.81	67.30	62.25	59.79	58.08
454C+	1.08	1.06	1.27	1.06	1.81	1.48
Loss	0.65	0.73	0.32	0.63	0.53	0.52
Distribution, V%						
IBP-343C	67	44	33	37	39	41
343-454C	32	55	66	62	60	58
454C+						
Whole						
Carbon, W%			88.33	87.99	87.94	88.09
Hydrogen, W%			11.30	11.32	11.30	11.18
Nitrogen, ppm						
Sulfur, ppm						

TABLE 4.9
Inspection of the Vacuum Still Overheads

Period	2	4	8	10	12	14	15	17
API	10.0	11.7	13.6				14.5	
IBP	558	588	563				534	
5, V%	650	650	667					
10, V%	664	667	686				656	
20, V%	700	698	713				679	
30, V%	721	717	736				711	
40, V%	741	734	754				729	
50, V%	761	747	772				746	
60, V%	780	763	795				762	
70, V%	802	782	812				789	
80, V%	821	810	838				813	
90, V%	856	832	865				841	
95, V%	873	854	884				871	
Last Point	915	908	930				921	
Distribution, W%								
IBP-650F	6.10	5.36					9.08	
650-LP	90.40	92.81	96.72				88.34	
LP+	3.40	1.12	2.97				1.86	
Loss	0.10	0.71	0.31				0.72	
Distribution, V%								
IBP-650F	6	6	<2.0				9	
650-LP	92	92	86				89	
LP+	2							
Whole								
Carbon, W%								
Hydrogen, W%								
Nitrogen, ppm								
Sulfur, ppm								

Table 4.9 Contd.

Period	19	21	24	28	31	33	34	36
API		16.0	13.7	14.2	12.2	12.2	11.5	13.0
IBP			518	494	547	538	518	526
5, V%		621	620	605	615	604	600	608
10, V%		644	642	635	646	637	628	629
20, V%		675	670	661	680	655	652	655
30, V%		699	695	695	700	680	679	687
40, V%		718	710	718	710	704	695	700
50, V%		736	740	735	738	723	716	718
60, V%		751	760	750	755	738	732	733
70, V%		772	790	772	778	758	748	747
80, V%		803	820	805	805	779	771	772
90, V%		840	883	835	832	805	805	812
95, V%		871	908	864	883	830	830	840
Last Point		910	908	900	905	888	886	885
Distribution, W%								
IBP-650F		11.47	13.03	13.18	10.56	15.13	18.18	17.16
650-LP		86.03	78.26	84.25	86.90	83.35	79.70	80.59
LP+		1.88	7.69	1.75	1.62	1.52	1.41	1.23
Loss		0.62	1.02	0.82	0.92		0.71	1.02
Distribution, V%								
IBP-650F		12	13	13	12	16	28	18
650-LP		87	81	86	87	83	71	80
LP+								
Whole								
Carbon, W%						89.40		89.48
Hydrogen, W%						10.16		10.16
Nitrogen, ppm						141.9		141.1
Sulfur, ppm						199.8		128.8

Table 4.9 Contd.

Period	37	39	41	42	43	45
API	14.3	13.0	11.7	10.9	11.2	11.1
IBP	526	572	517	537	519	531
5, V%	582	618	609	624	620	631
10, V%	605	637	635	650	648	648
20, V%	631	657	668	678	680	670
30, V%	655	687	697	700	700	692
40, V%	670	708	710	718	711	708
50, V%	702	732	738	735	730	720
60, V%	720	750	745	748	745	733
70, V%	738	768	760	770	760	750
80, V%	757	785	785	795	785	770
90, V%	795	807	820	830	822	808
95, V%	816	840	860	860	858	840
Last Point	876	884	885	893	882	862
Distribution, W%						
IBP-650F	27.60	16.96	12.65	9.56	10.18	10.93
650-LP	70.44	81.00	85.12	87.42	87.30	86.38
LP+	1.24	1.23	1.22	1.81	1.41	1.30
Loss	0.72	0.81	1.01	1.21	1.11	1.30
Distribution, V%						
IBP-650F	28	17	13	10	11	11
650-LP	71	84	85	90	88	88
LP+						
Whole						
Carbon, W%						
Hydrogen, W%						
Nitrogen, ppm						
Sulfur, ppm						

TABLE 4.10
Projected ROSE-SR^{SM} Unit Performance

Run No.	227-81	227-81	260-5	260-5	260-5	260-5	260-5
Condition	1	1	1	2	3	4	4
Coal SV, Kg/h/m3	312.4	312.4	320.5	480.7	560.8	640.9	721
Coal (dry), Kg/h	282.8	45.4	72.6	108.9	127	145.2	163.3
IOM, Kg/h	264.1	42.4	67.8	101.7	118.7	135.6	152.6
Ash, Kg/h	18.7	3	4.8	7.2	8.4	9.6	10.8
Sol/Coal*	1.2	1.2	1.0	1.0	1.0	1.0	1.0
CASB, Kg/h	438.1	70.3	112.5	168.7	196.8	224.9	253.1
PFL [W%]	0.795	0.795	0.795	0.795	0.795	0.795	0.795
PFS [W%]	0.205	0.205	0.205	0.205	0.205	0.205	0.205
CASB, Kg							
IBP-454C	231.4	37.1	59.4	89.1	104.0	118.8	133.7
454C+	157.5	25.3	40.4	60.7	70.8	80.9	91.0
QI	49.2	7.9	12.6	18.9	22.1	25.3	28.4
Sub-total	438.1	70.3	112.5	168.7	196.8	225.0	253.1
Recycle/Topped CASB	0	0	0	0	0	0	0
ROSE Feed, Kg/h							
DAO	142.0	22.8	36.4	54.7	63.8	72.9	82.0
Bottom (C5 Insol)	64.7	10.4	16.6	24.9	29.1	33.2	37.4
Sub-total	206.7	33.2	53.1	79.6	92.9	106.1	119.4
Recycled, Kg/h							
IBP-454C	231.4	37.1	59.4	89.1	104.0	118.8	133.7
454C+	0.0	0.0	0.0	0.0	0.0	0.0	0.0
QI	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Sub-total	231.4	37.1	59.4	89.1	104.0	118.8	133.7

TABLE 4.11
Overall Recycle Stream Composition

Period	4	12	14	15	19	21	33	34	36
Composition, W%									
O-13 Btm	0	41.29	25.82	27.83	21.05	21.04	14.77	15.3	15.75
M/U Oil	0	0	11.18	6.14	0	0	14.34	7.66	0
ASB	48.2	26.34	39.02	22.34	64.53	69.3	50.75	54.07	62.68
VSOH	8.68	16.18	16.86	32.07	6.35	7.3	10.14	11.46	9.41
DAO	43.12	16.18	7.13	11.63	8.07	2.36	10	11.51	12.16
Total	100	99.99	100.01	100.01	100	100	100	100	100
O-13 Btm, W%									
Resid	18.17	18.21	15.9	15.25	15.58	15.45	20.61	18.9	23.96
IOM	12.11	12.67	11.84	10.42	14.94	14.89	11.49	10.09	10.7
Ash	8.74	9.31	6.51	6.42	12.04	12.89	8.92	9.24	9.13
DAO, W%									
Resid	30.23	31.43	31.17	32.47	27.19	27.19	35.56	33.65	33
IOM	18.54	1.04	1.42	0.95	2.99	5.03	0.66	0.58	0.93
Ash	11.23	0.18	0.16	0.01	0.99	3.05	0	0.14	0.03
M/U Oil, W%									
Resid	18.36	18.36	18.36	18.36	18.36	18.36	18.36	18.36	18.36
Recycle Stream, W%									
Resid	13.04	12.60	8.38	9.15	5.47	3.89	9.23	8.17	7.79
IOM	7.99	5.40	3.16	3.01	3.39	3.25	1.76	1.61	1.80
Ash	4.84	3.87	1.69	1.79	2.61	2.78	1.32	1.43	1.44
IOM + Ash	12.84	9.27	4.85	4.80	6.00	6.04	3.08	3.04	3.24

Table 4.11 Contd.

Period	39	40	41	42	43	44	45
Composition, W%							
O-13 Btm	1.19	0	0	0	0	0	0
M/U Oil	11.27	29.14	15.73	19.79	26.84	25.21	42.84
ASB	52.14	31.81	31.53	27.56	43.94	36.28	24.96
VSOH	24.3	24.95	42.68	41.13	15.85	28.9	13.51
DAO	11.1	14.1	10.06	11.52	13.37	9.61	18.69
Total	100	100	100	100	100	100	100
O-13 Btm, W%							
Resid	16.89						
IOM	7.3						
Ash	3.57						
DAO, W%							
Resid	28.98	28.98	23.12	24.81	23.35	23.35	25.66
IOM	1.18	1.18	3.31	5.83	10.37	10.37	14.7
Ash	0.12	0.12	0.05	0.01	0.2	0.2	4.93
M/U Oil, W%							
Resid	18.36	18.36	18.36	18.36	18.36	18.36	18.36
Recycle Stream, W%							
Resid	5.49	9.44	5.21	6.49	8.05	6.87	12.66
IOM	0.22	0.17	0.33	0.67	1.39	1.00	2.75
Ash	0.06	0.02	0.01	0.00	0.03	0.02	0.92
IOM + Ash	0.27	0.18	0.34	0.67	1.41	1.02	3.67

Table 4.11 Contd.

Period	39	40	41	42	43	44	45
O-13 Btm	1.19	0	0	0	0	0	0
M/U Oil	11.27	29.14	15.73	19.79	26.84	25.21	42.84
ASB	52.14	31.81	31.53	27.56	43.94	36.28	24.96
VSOH	24.3	24.95	42.68	41.13	15.85	28.9	13.51
DAO	11.1	14.1	10.06	11.52	13.37	9.61	18.69
	100	100	100	100	100	100	100
O-13 Btm							
Resid	16.89						
IOM	7.3						
Ash	3.57						
DAO							
Resid	28.98	28.98	23.12	24.81	23.35	23.35	25.66
IOM	1.18	1.18	3.31	5.83	10.37	10.37	14.7
Ash	0.12	0.12	0.05	0.01	0.2	0.2	4.93
M/U Oil							
Resid	18.36	18.36	18.36	18.36	18.36	18.36	18.36
Recycle Stream							
Resid	5.49	9.44	5.21	6.49	8.05	6.87	12.66
IOM	0.22	0.17	0.33	0.67	1.39	1.00	2.75
Ash	0.06	0.02	0.01	0.00	0.03	0.02	0.92
	0.27	0.18	0.34	0.67	1.41	1.02	3.67

TABLE 4.12
Unit Ash Balance

Period	Dry Coal Kg/h	Ash W% mf coal	Ash Kg/day	O-61 Kg/day	O-61 Ash W%	O-61Ash Kg/day	Adj. O-61 Kg/day	Adj/Actual	O-63 Kg/day	O-63 Ash W%	O-63 Ash Kg/day	Adj. O-63 Kg/day	Adj/Actual	O-65 Kg/day	O-65 Ash W%	O-65 Ash Kg/day	O-61 Adj/Actual	O-63 Adj/Actual
1	63.7	5.75	87.9	0					0					0				
2	69.5	5.75	95.9	0					0					0				
3	56.1	5.75	77.4	0					0					0				
4	69.4	5.75	95.8	973.2	11.72	114.0	817.5	0.84	396.6	18.38	72.9	521.2	1.31	982.8			0.840	1.314
5	61.5	5.75	84.9	1250.5					45.8					339.8				
6	28.8	5.75	39.7	0					0					0				
7	58.3	5.75	80.5	0					0					0				
8	69.4	5.75	95.8	0	7.19				0	23.49				0				
9	62.1	5.75	85.7	0					0	17.76				0				
10	70.6	5.75	97.4	809.9	11.54	93.4	844.5	1.04	566.7	21.97	124.5	426.0	0.75	289.9	1.77	5.1	1.043	0.752
11	69.8	5.75	96.3	635.2					738.2					270.4				
12	70.6	5.75	97.4	547.2	14.57	79.7	668.5	1.22	556.7	20.58	114.6	471.2	0.85	293.1	0.18	0.5	1.222	0.846
13	72	5.75	99.4	685.1					501.8					351.2				
14	71.2	5.75	98.3	673.3	13.74	92.5	715.1	1.06	547.2	20.75	113.6	473.4	0.87	299.5			1.062	0.865
15	67.1	5.75	92.6	728.2	13.47	98.1	687.4	0.94	436.9	23.16	101.2	399.7	0.91	264.1	0.01	0.0	0.944	0.915
16	63.4	5.75	87.5	416.1					559.9					223.7				
17	64.4	5.75	88.9	384.8	18.08	69.6	491.7	1.28	376.6	28.10	105.8	298.4	0.79	187.4	3.38	6.3	1.278	0.792
18	87.7	5.75	121.0	611.2					382					244.6				
19	102.5	5.75	141.5	875.7	17.48	153.1	809.1	0.92	590.3	26.45	156.2	525.5	0.89	276.8	0.99	2.7	0.924	0.890
20	104.6	5.75	144.3	699.2					352.5					176.5				
21	102.6	5.75	141.6	1231.9	19.58	241.2	723.1	0.59	172.4	21.34	36.8	636.7	3.69	50.8	3.05	1.5	0.587	3.693
22	103.8	5.75	143.2	0					0					0				
23	11	5.75	15.2	0					0					0				
24	67.6	5.75	93.3	0	5.84				0	27.11				0	14.5			
25	73.9	5.75	102.0	428.3					247.3					111.2				
26	42.8	5.75	59.1	852.1					481.9					368				
27	77.7	5.75	107.2	693.7					363.4					403.4				
28	105.6	5.75	145.7	767.2	11.40	87.5	1278.3	1.67	440.1	21.16	93.1	681.0	1.55	412.9	0.26	1.1	1.666	1.547
29	115.5	5.75	159.4	966					431					324.4				
30	72.4	5.75	99.9	755.4	15.09	114.0	662.1	0.88	457.4	21.11	96.6	473.2	1.03	236.8			0.876	1.035
31	98.7	5.75	136.2	835.3	11.98	100.1	1136.9	1.36	409.7	23.04	94.4	581.5	1.42	372.1	0.42	1.6	1.361	1.419
32	132.2	5.75	182.4	1052.6					435.1					446				
33	137.4	5.75	189.6	1730	12.44	215.2	1524.6	0.88	551.3	24.09	132.8	787.0	1.43	477.3			0.881	1.428
34	137.5	5.75	189.8	1603.4	12.37	198.3	1534.4	0.96	485.9	23.58	114.6	799.8	1.65	505.9	0.14	0.7	0.957	1.646
35	140.4	5.75	193.8	990.9					555.4					525.9				
36	136.7	5.75	188.6	1565.8	13.53	211.9	1393.9	0.89	573	24.14	138.3	780.5	1.36	520.4	0.03	0.2	0.890	1.362
37	106.9	5.75	147.5	1188.7	12.61	149.9	1169.9	0.98	618.4	24.34	150.5	606.0	0.98	496.4			0.984	0.980
38	32.8	5.75	45.3	1256.4					568.5					535.8				
39	71.8	5.75	99.1	960.1	7.66	73.6	1293.0	1.35	412.4	24.42	100.7	402.9	0.98	607.5	0.12	0.7	1.347	0.977
40	78.2	5.75	107.9	1606.2					429.7					684.2				
41	88.7	5.75	122.4	1695.6	7.19	121.9	1703.2	1.00	692.4	1.08	7.5			476.4	0.05	0.2		
42	88.9	5.75	122.7	1688.7	6.35	107.2	1932.0	1.14	703.3	5.90	41.5	2075.6	2.95	512.3	0.01	0.1	1.144	2.951
43	94.8	5.75	130.8	1843.9	7.38	136.1	1772.7	0.96	707.8	12.13	85.8	1062.2	1.50	667	0.2	1.3	0.961	1.501
44	78.7	5.75	108.6	1962.8	5.31	104.2	2045.3	1.04	768.6	11.75	90.3	924.6	1.20	632.9			1.042	1.203
45	90.3	5.75	124.6	1902	6.89	131.1	1807.7	0.95	485.9	19.89	96.6	424.7	0.87	931.5	4.93	45.9	0.950	0.874

TABLE 4.13
Performance of the In-Line Hydrotreater

Period No.	Hydrotreater	O-12 Liquid,ppm			O-5 Liquid, ppm		
		H,%	N	S	H,%	N	S
33B	Yes	11.2	137	122	12.2	58	59
34B	**No**	**11.0**	**3323**	**399**	**11.2**	**2433**	**373**
36B	Yes	11.2	150	68	12.1	69	30
44B	**No**	**10.9**	**1661**	**793**	**11.5**	**1416**	**680**

FIGURE 4.11
Boiling Point Ditribution of Naphtha Stalizer Bottoms Fraction from 'Co-Liquefaction' Conditions

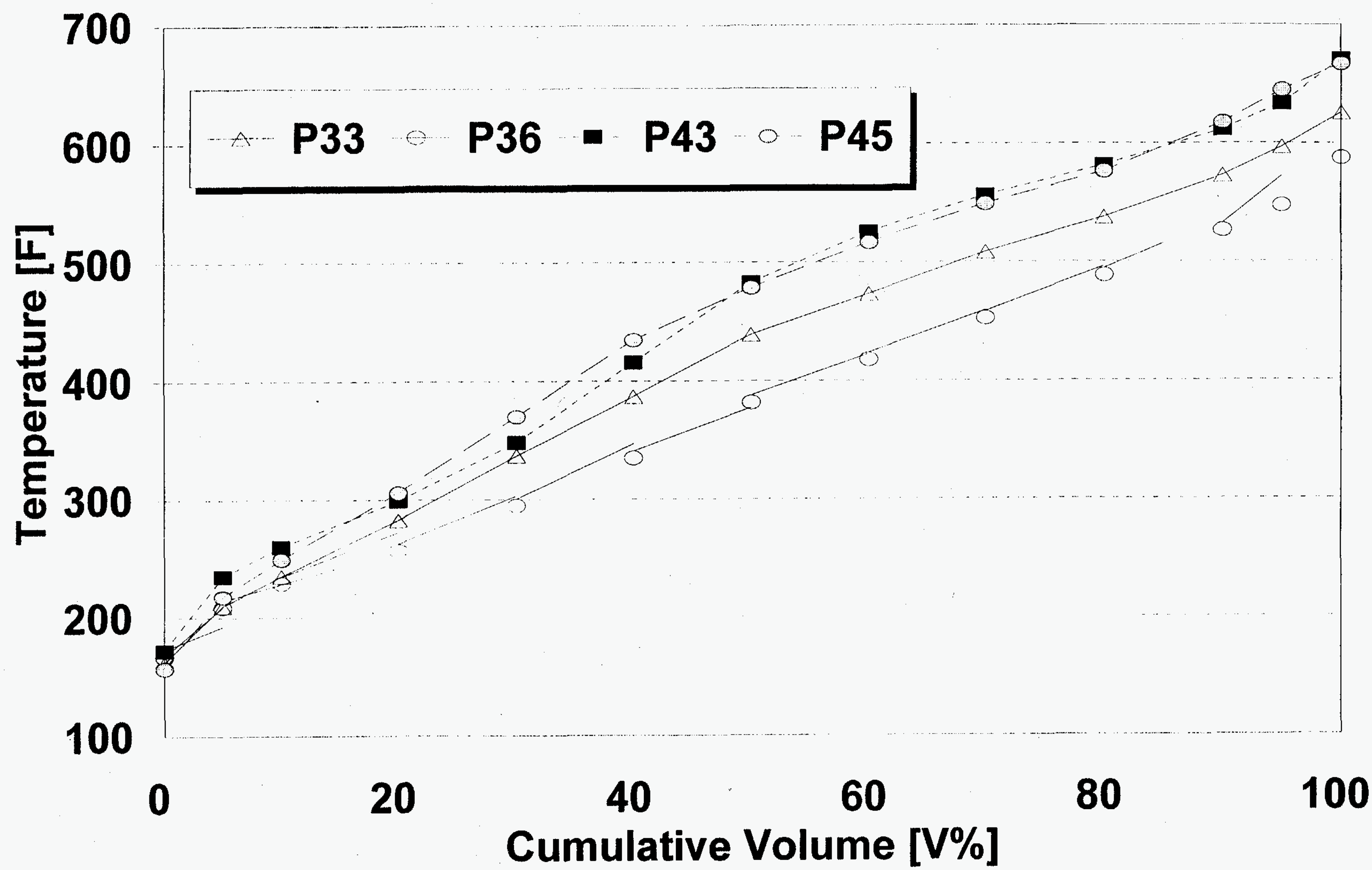


FIGURE 4.12
Ratio of Asphaltenes to Solids in ROSE-SR℠ Feed and Bottoms

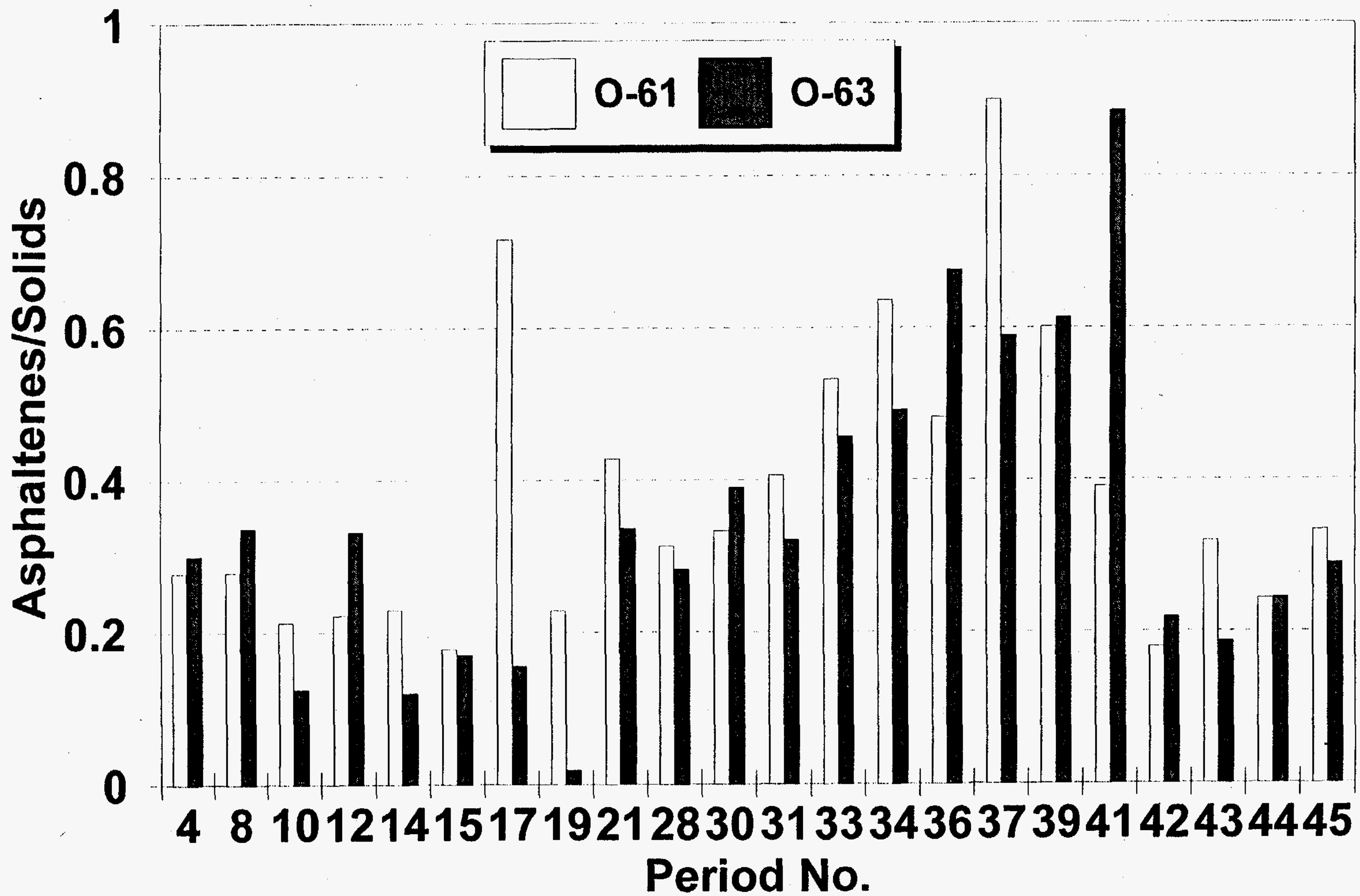


FIGURE 4.13
Insolubles in ROSE-SR^{SM} Feed

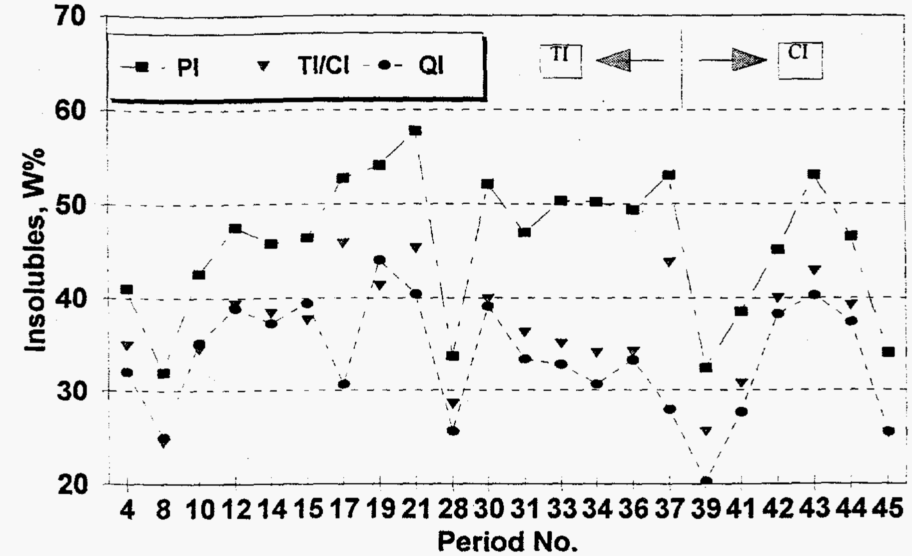


FIGURE 4.14
Insolubles in ROSE-SR^{SM} Residuals

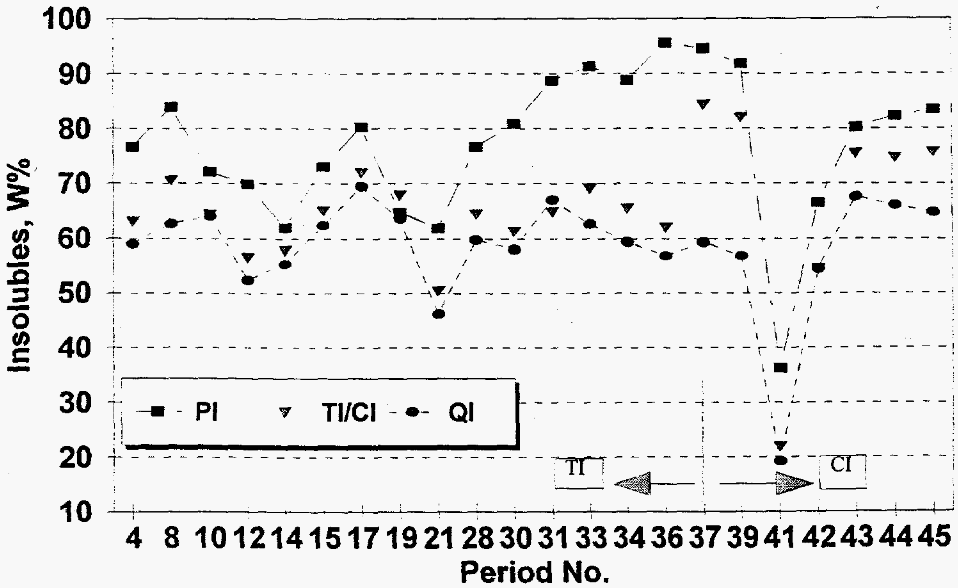


FIGURE 4.15
Periods 4, 12, 14, 15, 19, 21, 33, 34 and 36 Recycle Stream Resid Content

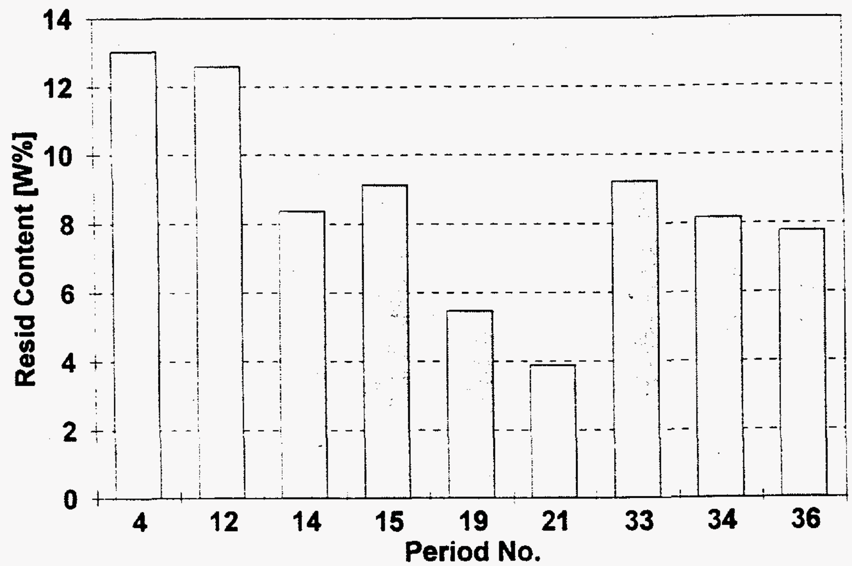


FIGURE 4.16
Periods 4, 12, 14, 15, 19, 21, 33, 34 and 36 Recycle Stream Solids Content

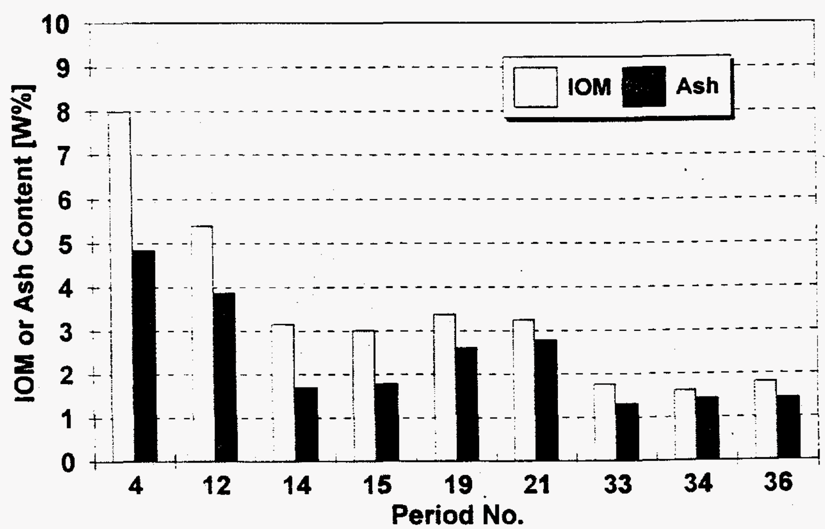


FIGURE 4.17
Periods 39 through 45 Recycle Stream Solids Content

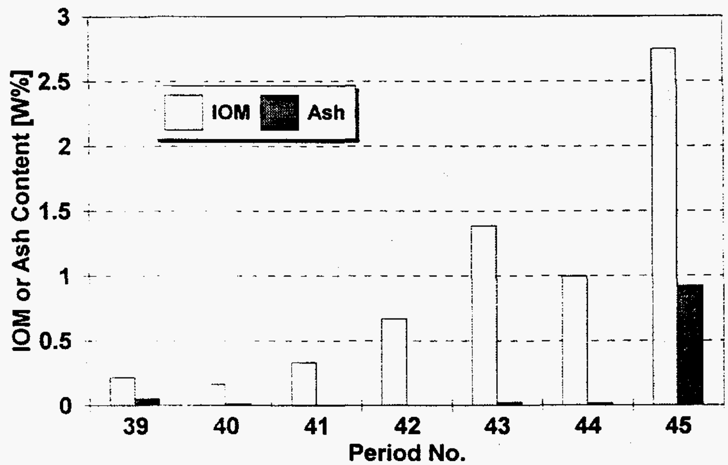


FIGURE 4.18
Periods 39 through 45 Recycle Stream Resid Content

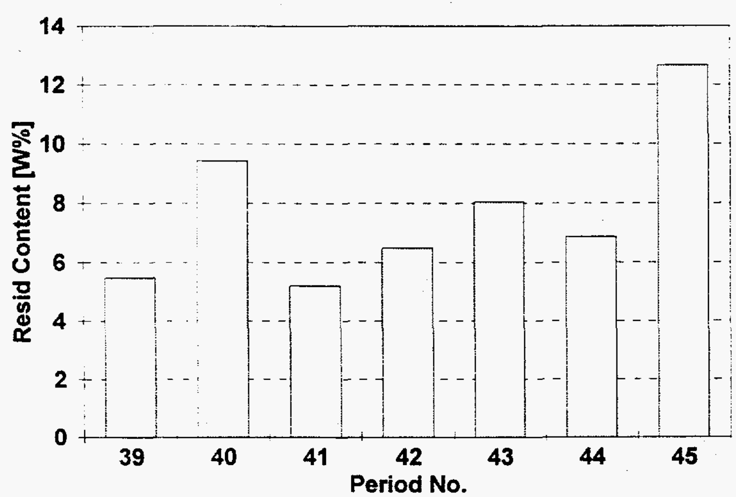


FIGURE 4.19
Nitrogen and Sulfur Contents of Naphtha Stabilizer Bottoms Distillates

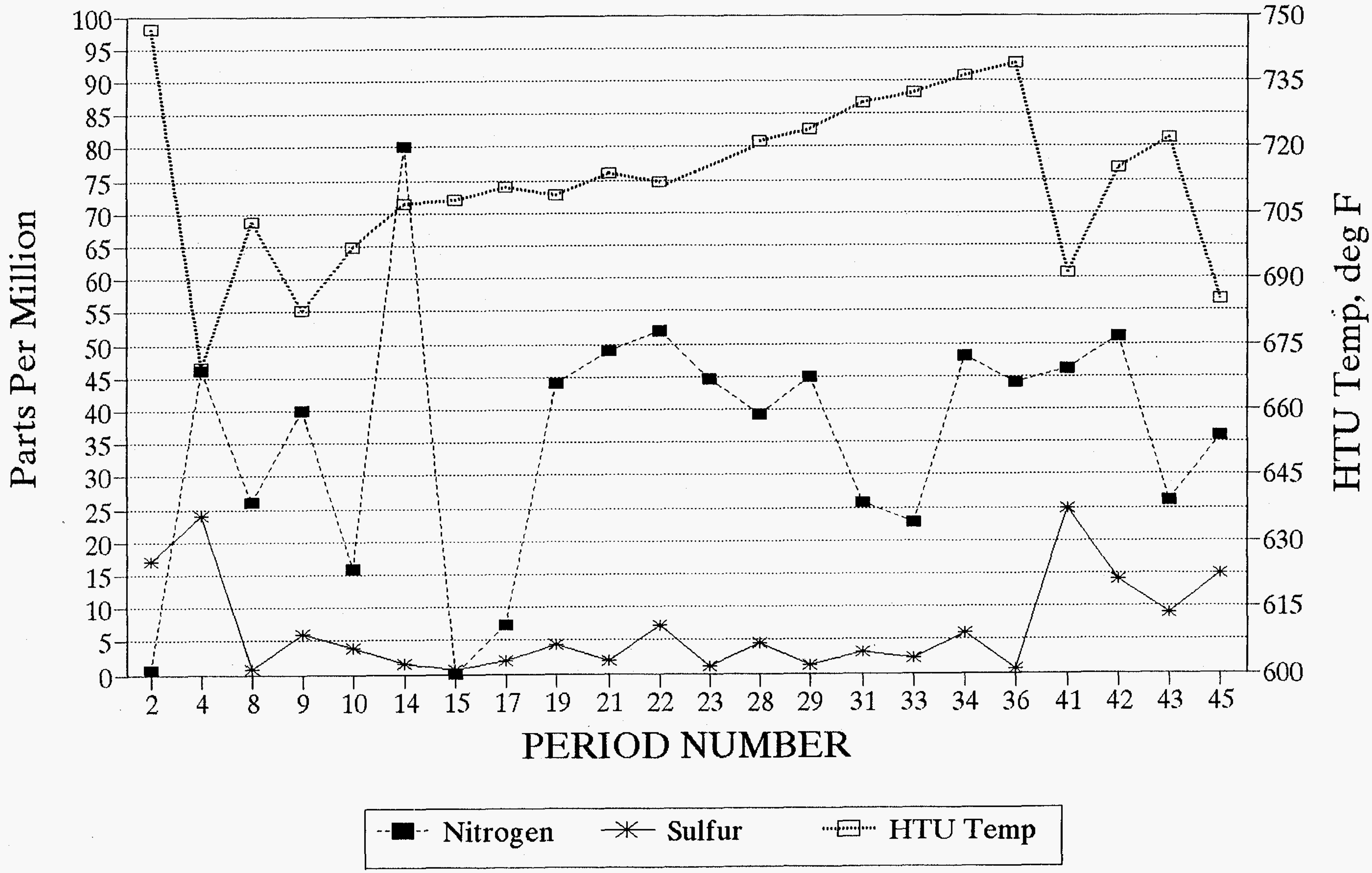
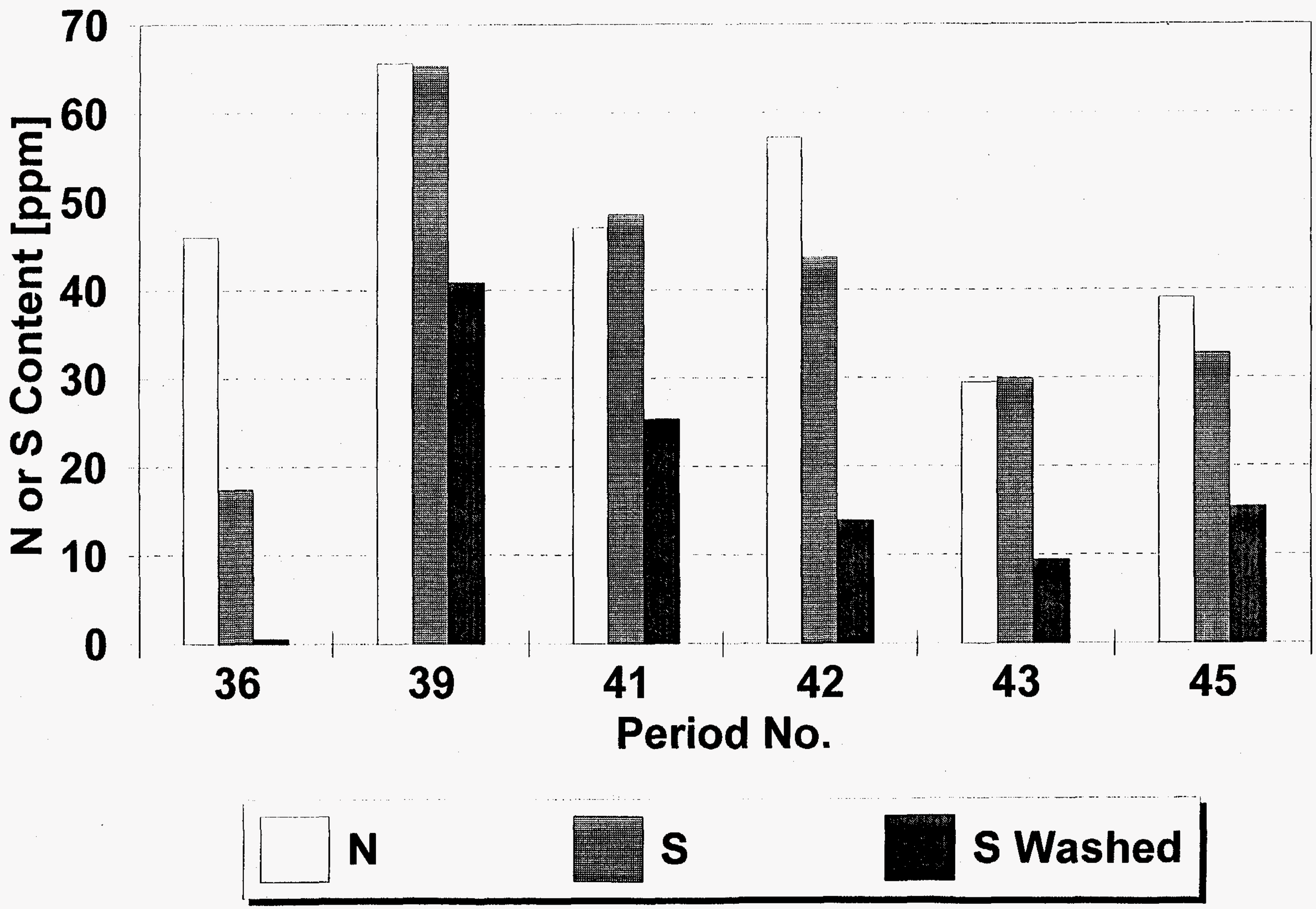


FIGURE 4.20
Nitrogen and Sulfur Content of Naphtha Stabilizer Bottoms Distillates from 'Co-Liquefaction' Conditions



SECTION V

LABORATORY SUPPORT

A. COAL/FEED QUALIFICATION TESTS

A.1 Microautoclave Tests Series I - Feed Coal Qualifications

A total of four microautoclave tests were conducted initially for the qualification of the feed Black Thunder Mine coal (two samples were obtained from the Empire Coke Company). The coal qualification tests were conducted using HTI's 20 cc microautoclaves at 427°C and 2000 psig (hot) hydrogen pressure. A total of 1 hour was allowed for the reaction time (using L-809 as the reaction solvent and presulfided Akzo AO-60 extrudate catalyst); the following results were obtained:

Table 5.1 Results from Coal Qualification Tests

Coal	Coal Conversion, % MAF	524°C+ Resid Conversion, % MAF
HRI-6208	92.5, 93.1	63.6, 63.2
HRI-6209	93.0, 92.2	65.1, 54.6

These coal and resid conversion values indicate high reactivity of Wyoming Black Thunder Mine coal under catalytic conversion conditions.

A.2 Microautoclave Tests Series II - Dissolution, Mixing Behavior, and Reactivity of Co-mingled Plastics with Coal/Solvent Slurries

As part of POC-02 PDU operations, the combined processing of organic wastes, such as mixed plastics and used rubber tires was carried out during the last nine days of operations. Since the HTI staff had no prior experience with high temperature, and high pressure reactions of waste plastics, some laboratory scale experiments were conducted to assess the dissolution behavior and uniformity of slurry formation by waste plastics in coal/recycle oil mixtures. The reactivity of combined coal/plastics feed under coal liquefaction conditions was also studied using microautoclaves.

It was found in the dissolution/mixing experiments that at a 2:1 weight ratio of coal-derived recycle oil to solids (coal+plastics at 50/50) a homogeneous slurry was obtained after heating the mixture with continuous agitation to around 230°C. Even after cooling this mixture to room temperature, no evidence of unmixed solid lumps was observed. The presence of coal with the co-mingled plastics (HDPE+PS+PET) appeared to help the formation of a homogeneous and lump-free slurry. The reactivity of this coal/plastics feed mixture was determined using microautoclave catalytic tests. These tests were conducted using a 4:1 (by weight) recycle solvent-to-solid (coal+plastics) ratio, 427°C, 30 minutes, 1:1 (to feed) of spent AO-60 extrudate catalyst. An acidic catalyst, Engelhard SiO_2-Al_2O_3, to provide a cracking/depolymerization function for co-mingled plastics was also employed at 5 w% of the solid feed during these tests. The results of these reactivity tests, are listed in Table 5. Coal and mixed plastics combined feed resulted in high levels of conversion; also, the acidic catalyst (6% silica-alumina) was detrimental to the conversion/depolymerization of coal/plastics feed mixtures. This suggests that a very strongly acidic catalyst may not be required for the hydrocracking of plastics; rather the initial depolymerization of plastics is primarily thermal under coal liquefaction conditions, and the radicals formed may subsequently react in the presence of a suitable acid catalyst to yield high selectivity of liquids.

B. SPENT CATALYST ACTIVITY

POC-02 PDU operations started using an already equilibrated extrudate catalyst in both reactor stages. During the operations, fresh catalyst was added to both reactors periodically to replace spent/deactivated catalyst. Detailed chemical analyses were done on the spent catalyst withdrawn during Periods 33 and 36. These periods were selected to determine if the added molybdenum catalyst (during Period 36) made any difference in the molybdenum-contents of the spent extrudate catalysts and, if so, to see how catalyst activity was affected.

The analytical data on the spent catalysts from Periods 33 and 36, fresh Akzo Ao-60 catalyst, and POC 01 shut-down catalysts are shown in Table 5.3. The molybdenum-content of spent catalysts from Period 36 (especially the first stage reactor) is higher (by as much as 5%) than the spent catalysts from Period 33. This higher molybdenum content did not seem to improve the activity of Period 36 spent catalysts (Figure 5.1) compared to Period 33 spent catalysts.

TABLE 5.2
Microautoclave Tests to Evaluate Reactivity of Mixed Plastics

Feed(g)	SiO_2 Al_2O_3	% THF Sol. of Products	% 975F+ Conv.	H/C of THF Solubles
Coal	Yes	88.5	64.6	1.22
Coal	No	90.5	67.8	1.24
Plastics*	Yes	75.4	73.6	1.29
Plastics	No	82.8	79.5	1.32
Coal+ Plastics	Yes	85.0	77.6	1.28
Coal+ Plastics	No	88.8	79.1	1.31
Coal+ PET	Yes	88.9	76.6	1.22
Coal+ PET	No	90.8	77.7	1.25

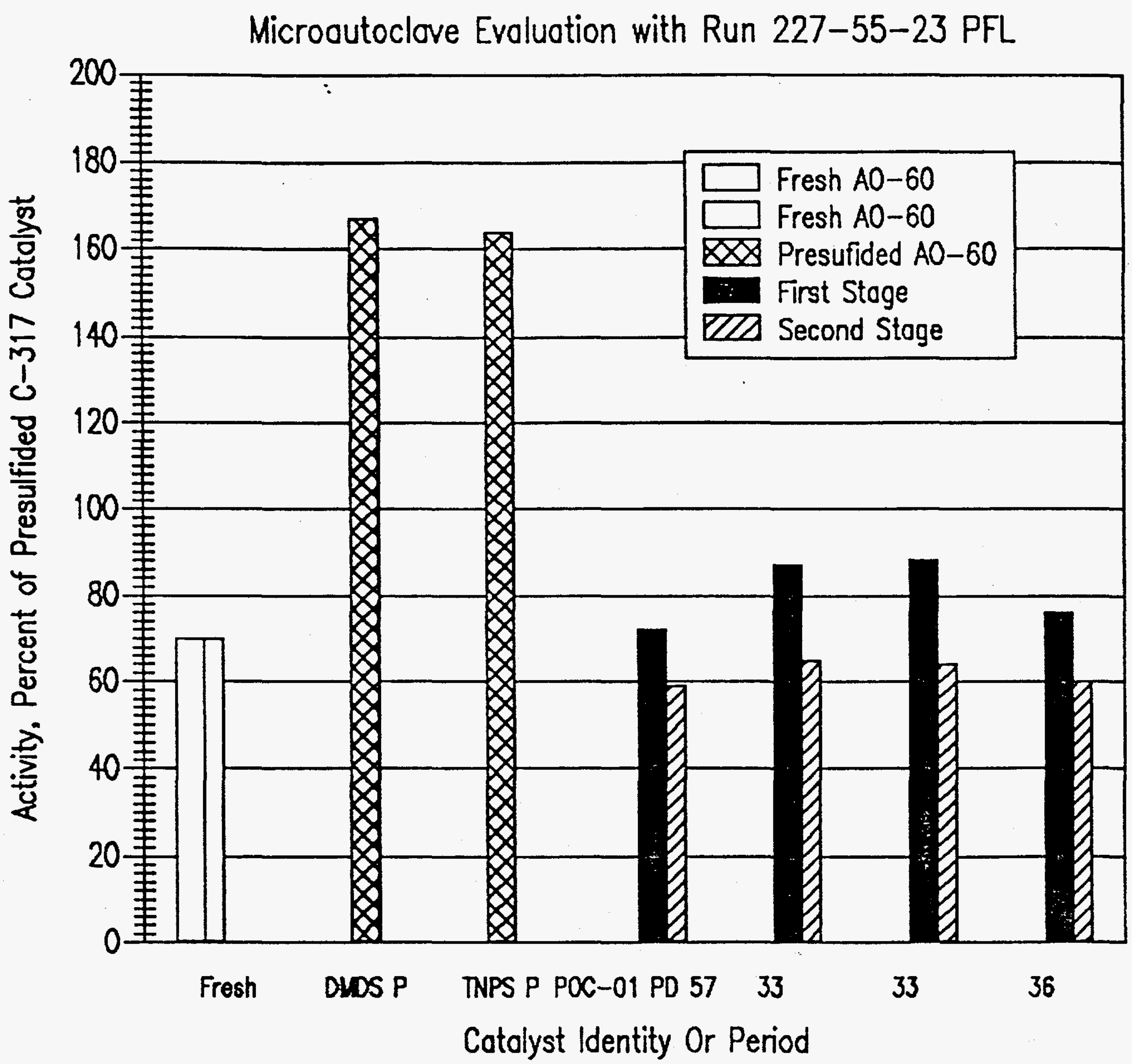
*Plastics: 0.5 g of HDPE + 0.35 g of PS + 0.15 g of PET

TABLE 5.3.
Analyses of Catalyst Withdrawals

	------- HRI 6043 --------								
	Fresh	DMDS Pres.	TNPS Pres.						
Period				POC-01 57*	POC-01 57*	33	33	36	36
Stage				1	2	1	2	1	2
Particle Density, gm/cc	0.872			1.226	1.228	1.073	1.154	1.098	1.140
Ignition Loss, W%	3.08			17.08	24.92	18.66	24.13	18.39	27.39
Carbon, W%		2.8	2.32	11.3	21.5	11.84	18.96	11.72	18.34
Hydrogen, W%		0.46	0.43	0.6	0.57	0.81	0.85	0.77	0.91
Nitrogen, W%		0	0	0.14	0.15	0.13	0.15	0.11	0.21
Sulfur, W%		6.07	7.54					6.52	6.06
Molybdenum, W%	11.31			6.52	7.72	7.86	7.76	8.13	7.77
Nickel, W%	2.48			1.38	1.56	1.95	1.86	1.90	1.81
Iron, W%	0.01			0.66	0.16	1.23	0.11	1.07	0.10
Sodium, W%	0.07			0.64	1.04	1.01	1.48	0.90	1.21
Calcium, W%	0.00			0.10	0.05	0.09	0.04	0.06	0.06
Titanium, W%									
Mo, Gms/cc	0.0986			0.0799	0.0948	0.0843	0.0896	0.0893	0.0885
Ni, Gms/cc	0.0216			0.0169	0.0192	0.0209	0.0215	0.0209	0.0206
Total Metals, W%				2.75	2.83	4.52	3.79	3.95	3.16
Fresh Catalyst Content, W%									
Basis Particle Density				71.1	71.0	81.3	75.6	79.4	76.5
Basis Ignition Loss				80.8	73.0	77.9	73.2	81.9	73.4
Basis Contaminants		93.7	93.5	85.2	74.9	82.7	76.3	80.2	74.3
Basis Moly. Content				57.6	68.3	69.5	68.6	71.9	68.7
Basis Nickel Content				55.6	62.9	78.4	75.0	76.6	73.0
Sulfiding to MoS2, NiS, %								100.8	98.1
Sulfur, gms/cc particle								0.0716	0.0691

* Blended as charge at start of Run POC-02.

FIGURE 5.1
Relative Activity of (Run 260-05) POC-02 Spent Catalysts from Daily Withdrawals



SECTION VI

TECHNICAL ASSESSMENT

A. Introduction

Under a contract awarded in 1992 by the U.S. Department of Energy, Hydrocarbon Technologies, Inc (HTI) has conducted a Proof-of-Concept (POC) program to demonstrate the direct coal liquefaction process. The objective of the POC program is to develop coal liquefaction technology for producing premium coal-derived liquid fuels, which is economically competitive with petroleum-based refining technology. As part of the POC program, HTI is performing technical assessments to provide technical, engineering and economic guidance for the process development unit (PDU) operations.

A technical assessment of the first PDU run (bituminous coal) in the POC contract was presented as part of the final Technical Progress Report DE-92148-TOP-01, in August, 1995. This report contains the technical assessment of the second PDU run, with subbituminous coal, which was conducted in June-July of 1994. In particular, it focuses on the evaluation of the economic impact predicted by the results of the run.

As with the bituminous coal study, the economic evaluation studies were based on construction of a fully-integrated grass-roots commercial facility to manufacture finished gasoline and diesel fuel. The liquefaction plant is a multi-train facility, and the capacity has been selected assuming construction of maximum-sized heavy-walled pressure vessels to carry out the liquefaction reactions. Liquefaction plant bottoms are gasified to meet part of the hydrogen requirements of the complex. All utility needs in the complex are internally produced. Natural gas is imported to meet fuel requirements and for hydrogen manufacture. Equipment sizes and costs for the liquefaction plant are factored from detailed engineering studies. Costs and operating requirements of the other process facilities and the off-sites estimated from the Baseline Design Study, developed by Bechtel for DOE. The Bechtel Baseline Design Study also provides the economic criteria and financing basis used in this evaluation.

B. Objectives and Scope of Work

The objective of POC-2 (Run 260-05) was to demonstrate the liquefaction of sub-bituminous coal in a two-stage reaction system with steady-state catalyst addition and withdrawal. Solids-separation by the ROSE process was demonstrated, as was the use of an in-line hydrotreater for heteroatom removal from the liquefaction products. Figures 6.1 shows a simplified schematic of the CTSL Process while, Figure 6.2 is a block diagram of major processing areas, under consideration for the present Technical Assessment.

The coal used for POC-2 was a Wyoming coal from the Black Thunder Mine, and the properties are summarized in Table 6.1. Three conditions explored during the run were assessed: Condition 2B (Period 20/21) was operated at a low space velocity with relatively low first and second stage reactor temperatures. For Condition 4C, (Periods 33/34) both space velocity and reactor operating temperature were increased. Finally, Condition 5 (Periods 35/36) was operated at almost the same reactor space velocity and temperatures as condition 4C, but with the addition of dispersed Molyvan 822 catalyst to the first-stage reactor feed. Table 6.2 summarizes the operating conditions for the three periods, as compared to those used in Run CMSL-6 of the bench program, which utilized the same coal.

Supported catalyst addition rates were lower for the first condition than for the subsequent two conditions. For this reason, process performance and yields were brought to a common catalyst addition rate by means of the CTSL Process Simulator; the projected results are shown in Table 6.2. Since CMSL-6 used an iron dispersed catalyst in addition to Molyvan, a slightly lower catalyst addition rate was used in comparison to the PDU results.

Comparison of projected results for the three run conditions shows almost constant coal conversion. Performance in the first condition was nearly identical with the bench-scale results. In the latter part of the run, however, residuum conversion decreased and gas yields increased. While hydrogen consumption remained almost constant, the combination of reduced residuum conversion and higher gas yield resulted in lower distillate yields for Conditions 4C and 5, when compared to Condition 2B and to CMSL-6.

Overall material balances are shown in Table 6.3. While liquids production in Condition 2B is almost equal to CMSL-6, a marked decrease is noted for the other two periods. Propane and butane by-products increase, but not nearly enough to offset the decreased liquids make.

Table 6.4 presents net hydrogen balances, showing a steady increase in the amount of hydrogen required per barrel of product as the run progressed. The percentage of hydrogen supplied by partial oxidation was initially 38 percent of the total requirement, but increased to 58 percent of the total in the final run condition, because of the increased residuum yield. While reducing the natural gas need, partial oxidation is a relatively more expensive source of hydrogen compared to steam reforming, both in terms of cost and in utility requirements.

Table 6.5 summarizes the thermal efficiency for the cases and shows no substantial differences. Table 6.6 is a summary of the utilities demand, pointing out the increased usage when partial oxidation becomes the major hydrogen source. Table 6.7 shows the capacities of the various process units and off sites.

Liquefaction plant investment details are presented in Table 6.8. The advantage of a higher space velocity in conditions two and three is seen in terms of lower reactor cost, but there is very little difference in total liquefaction plant costs between the cases. Liquefaction plant investment costs for all PDU results are slightly lower than the CMSL-6 projection, primarily owing to reactor savings, due to lower gas yields.

Table 6.9 is a summary of the total plant investment for all the cases. The investment costs are within ±2 percent, costs on a per daily barrel of liquids basis show a significant increase as liquids production falls off.

Overall economics are summarized in Table 6.10. In addition to detailing annual operating costs and by-product credits, this table presents the net product cost and the equivalent crude oil price (ECP). The equivalent crude oil price is a price of crude at which the refiner is indifferent whether he buys crude or coal liquids. The ECP for the first condition is $32.29/B, compared to $32.72/B for the CMSL-6 projection. Increased space velocity in Condition 4C lowers the net annual product cost, but because of the decreased liquid production, the ECP increases to $33.76/B. Dispersed catalyst cost has a very slight effect on the economics, but the decreased liquid production increases the ECP to $36.34/B in the third condition.

Table 6.11 provides a breakdown of the ECP. As with bituminous coal, the capital-related charges are by far the largest contributor to the ECP. Of the variable costs, the largest is natural gas, then the cost of the catalyst and chemicals. Since the cost of sub-bituminous coal is roughly one-third that of bituminous coal, coal price is a small contributor to the overall ECP.

Sensitivity analyses were performed on the largest contributors to the equivalent crude oil price. Figure 6.3 illustrates the effect of total plant investment, showing roughly a ± 12% change in ECP for a ± 15% change in the total plant investment.

Natural gas price sensitivity is shown in Figure 6.4, disclosing roughly a ± 2% change in ECP for a ± 15% change in natural gas price. Figure 6.5 presents the sensitivity of the cost of catalysts and chemicals, showing less than a ± 2% change in ECP for a ± 15% change in cost of catalyst and chemicals. Finally coal price sensitivity is presented in Figure 6.6. The effect of a ± 15% change in coal cost is less than ± 1% change in ECP.

C. Summary and Conclusions

The results of the technical assessment of POC-2 show an equivalent crude oil price of $32.29/B, which is lower than the $32.72/B calculated from the results of bench run CMSL-6 with the same coal, at almost the same reactor operating conditions. Because of lower liquid product yield, an increased space velocity reduces the total plant investment, but increases the equivalent crude price to $33.76/B. Use of dispersed catalyst does not contribute to overall product cost, but no economic benefit was observed, again because of lower liquid product yield. As with bituminous coal, the equivalent crude oil price is most significantly affected by a change in the total capital investment.

TABLE 6.1
Feed Coal Analysis

Proximate Analysis, W% Dry Coal	
Volatile Matter	43.48
Fixed Carbon	50.52
Ash	6.00
Total	100.00
Ultimate Analysis, W% Dry Coal	
Carbon	69.95
Hydrogen	4.50
Nitrogen	0.89
Sulfur	0.39
Oxygen (by difference)	18.27
Ash (SO3-free)	6.00
Total	100.00
SO3-free ash	5.13
H/C Atomic Ratio	0.77
O/C Atomic Ratio	0.20
Heating Value, BTU/lb dry coal calculated	11,664
Coal Moisture, W%	11.47

TABLE 6.2
Design Basis Comparison

	Period 20/21	Period 33/34	Period 35/36	CMSL - 6 Period 13
Operating Conditions				
Inline hydrotreater	yes	yes	yes	yes
Space Velocity, lb/hr/ft3	28.6	38.0	38.4	28.4
Supported Catalyst Rate, lb/Ton feed	3.5	3.5	3.5	2.7
Dispersed catalyst, ppm				
Iron	0	0	0	615
Molyvan	0	0	150	100
Recycle Solvent				
Solvent/feed, lb/lb	1.3	1.3	1.30	1.3
Resid, W%	8.0	9.0	7.0	37.5
Temperatures, F				
First Stage	775	808	810	818
Second Stage	831	837	830	810
Process Performance				
Feed Conversion, W% MAF	92.6	91.8	93.5	93.6
Resid Conversion, W% MAF	84.9	83.0	80.7	87.9
Denitrogenation, W%	82.0	98.6	88.9	91.2
Process Yields, W% Dry Coal				
H2S	0.44	0.70	0.53	0.33
NH3	0.89	1.07	0.96	0.99
H2O	18.10	20.36	18.27	11.76
COx	0.44	1.02	1.04	4.74
C1-C3	8.75	9.20	9.61	12.34
C4-390 F	18.67	17.21	21.30	16.56
390-500 F	22.77	24.98	23.30	14.91
500-650 F	10.69	9.68	1.23	19.12
650-850 F	5.21	0.89	3.85	8.07
850-975 F	2.37	0.12	4.02	2.05
975 + F	7.28	8.31	12.13	5.41
Unconverted Feed	7.02	7.80	6.18	6.07
Ash	5.13	5.13	5.13	5.13
Total:	107.76	106.47	107.56	107.48
C4-975 F, W% dry feed	59.72	52.88	53.71	60.71
C4-850 F, W% dry feed	57.35	52.76	49.68	58.66

TABLE 6.3
Overall Process Material Balance

	Period 20/21	Period 33/34	Period 35/36	CMSL - 6 Period 13
Coal, TPSD	10,500	10,500	10,500	10,500
Natural Gas, MMSCFD	99	89	68	124
Liquid Products, BPSD				
Gasoline	11,816	11,177	10,158	12,139
Diesel	28,639	27,090	24,620	29,422
TOTAL:	40,455	38,267	34,778	41,561
Barrels/Ton total feed	3.85	3.64	3.31	3.96
Byproducts				
Propane, BPSD	3,258	3,033	3,749	4,616
Butane, BPSD	1,896	1,477	2,025	1,815
Sulfur, TPSD	66	68	67	60
Ammonia, TPSD	93	112	101	104
Ash to disposal, TPSD	773	555	726	995

TABLE 6.4
Plant Net Hydrogen Balance

	Period 20/21	Period 33/34	Period 35/36	CMSL - 6 Period 13
Hydrogen Consumption, MMSCFD				
Liquefaction	307	256	299	296
Product Upgrading	19	64	15	25
TOTAL:	327	320	314	321
Hydrogen Consumption, SCF/B	8,076	8,368	9,035	7,724
Hydrogen Production, MMSCFD				
Partial Oxidation	125	140	182	76
Steam Reforming	202	180	132	245
TOTAL:	327	320	314	321

TABLE 6.5
Thermal Efficiency

	Period 20/21	Period 33/34	Period 35/36	CMSL - 6 Period 13
Inputs, GBTU/Day				
Coal	244.5	244.5	244.5	244.5
Natural Gas	89.5	81.2	61.7	112.5
Total	334.0	325.7	306.2	357.0
Outputs, GBTU/Day				
Gasoline	65.4	61.9	56.2	67.2
Diesel	165.0	156.0	141.7	169.5
Propane	12.5	11.7	14.4	17.8
Butane	8.3	6.4	8.8	7.9
Sulfur	0.6	0.6	0.6	0.5
Ammonia	1.8	2.2	2.0	2.0
Total	253.6	238.8	223.7	264.9
Thermal Efficiency, %	75.9	73.3	73.1	74.2

TABLE 6.6
Utilities Summary

	Period 20/21	Period 33/34	Period 35/36	CMSL - 6 Period 13
Electric Power, MW	203	200	223	205
600 Psig Steam, Mlbs/hr	18	33	76	292
Cooling Water, Mgpm	122	127	132	170
Process Fuel, GBTU/Day	90	81	62	113
Raw Water, gpm	3,066	3,475	4,176	4,656

TABLE 6.7
Capacities of Process Units and Offsites

Area No.	Description	Sizing basis, Units	Period 20/21	Period 33/34	Period 35/36	CMSL - 6 Period 13
100	Coal Preparation	TPSD Dry coal	10,500	10,500	10,500	10,500
200	Liquefaction	TPSD Coal feed	10,500	10,500	10,500	10,500
300	Hydrogen Manufacture					
	Partial Oxidation	MMSCFD H2	125	140	182	76
	Steam Reforming	MMSCFD H2	202	180	132	245
400	Oxygen Plant	TPSD Oxygen	1,565	1,760	2,284	952
500	Product Treating					
	Sour Water treating	gpm sour water	1,628	1,774	1,655	1,693
	Sulfur recovery	TPSD Sulfur	66	68	67	60
	Gas plant	lb/hr C1-C3	76,563	80,526	84,044	107,949
600	Upgrading & Solids Sep'n					
	Hydrotreating	BPSD Coal liquids	40,455	38,267	34,778	41,561
	Catalytic reforming	BPSD 180-350	11,816	11,177	10,158	12,139
700	Utilities					
	Steam generation	Mlbs/hr	18	33	76	292
	Power distribution	MW	203	200	223	205
	Cooling water	MGPM	122	127	132	170
800	Tankage					
	Coal liquids	BPSD liquids	40,455	38,267	34,778	41,561
	LPG	BPSD LPG	3,258	3,033	3,749	4,616
	Waste solids handling	TPSD solids	773	555	726	995
900	General Off-Sites	TPSD Coal Feed	10,500	10,500	10,500	10,500

TABLE 6.8
Liquefaction Plant Investment Details

	Period 20/21	Period 33/34	Period 35/36	CMSL - 6 Period 13
Major Equipment Costs, $M				
Pumps	25,698	25,807	25,829	24,790
Reactors	44,814	39,323	37,345	50,246
Fired Heaters	15,154	14,916	14,680	15,416
Exchangers	17,608	17,874	17,587	18,755
Drums, Hydrotreaters & Towers	36,100	36,819	36,394	36,146
Compressors	35,043	32,395	34,639	34,462
HPU	12,684	13,234	12,589	14,427
TOTAL	187,101	180,368	179,063	194,242
Plant Investment, $MM				
Materials & Equipment	339.0	326.9	324.4	351.9
Labor & Subcontracts	146.7	141.4	140.4	152.3
Indirects	122.0	117.6	116.8	126.7
Contingency	0.0	0.0	0.0	0.0
Total Liquefaction Plant Investment	607.7	585.9	581.6	630.9

TABLE 6.9
Total Plant Investment Summary

Area	Name	Period 20/21	Period 33/34	Period 35/36	CMSL - 6 Period 13
100	Coal Preparation	200.0	200.0	200.0	200.0
200	Liquefaction	607.7	585.9	581.6	630.9
300	Hydrogen Manufacture	268.5	264.7	258.7	261.0
400	Oxygen Plant	59.7	64.8	77.7	42.1
500	Product Treating	243.7	252.7	258.1	300.7
600	Upgrading & Solids Sep'n	84.1	82.5	78.7	100.7
700	Utilities	245.3	244.1	261.1	274.1
800	Tankage	113.6	108.3	99.8	116.2
900	General Off-Sites	189.8	187.5	189.3	192.0
	Subtotal	2012.4	1990.5	2005.0	2117.7
	Fee, Contingency	401.9	397.6	400.5	423.1
	Total Plant Investment	2,414	2,388	2,406	2,541
	$/BPD of Product	59,679	62,406	69,167	61,134

TABLE 6.10
Product Cost Calculation

	Period 20/21	Period 33/34	Period 35/36	CMSL - 6 Period 13
Operating Costs, $MM/year				
Coal ($7.00/Ton)	24.14	24.14	24.14	24.14
Natural Gas ($2.00/MMBTU)	58.78	53.33	40.55	73.88
Water ($2.50/Mgal)	3.63	4.11	4.94	5.51
Catalysts and Chemicals	44.33	43.31	40.99	23.20
Dispersed Catalyst	0.00	0.00	0.83	10.93
Ash Disposal ($5.00/Ton)	1.27	0.91	1.19	1.63
Labor	24.43	24.43	24.43	24.43
Maintenance	21.89	21.89	21.89	21.89
Capital-Related	360.80	356.01	356.70	379.04
TOTAL:	539.27	528.13	515.66	564.65
Byproduct Credits, $MM/year				
Propane ($12.29/Bbl)	13.15	12.24	15.14	18.64
Butanes ($14.50/B)	9.03	7.04	9.65	8.65
Sulfur ($52.00/Ton)	1.14	1.16	1.14	1.03
Ammonia ($200.00/Ton)	6.12	7.36	6.64	6.83
TOTAL:	29.44	27.80	32.57	35.15
Net Product Cost, $MM/year	509.83	500.33	483.09	529.50
Product Cost, $/B	38.36	39.80	42.29	38.78
Crude Oil Equivalent Factor	0.842	0.848	0.859	0.844
Equivalent Crude Oil Price, $/B	32.29	33.76	36.34	32.72

TABLE 6.11
Break-down of Equivalent Crude Price

	Period 20/21	Period 33/34	Period 35/36	CMSL - 6 Period 13
Contribution to Price, $/B				
Coal	1.53	1.63	1.82	1.49
Natural Gas	3.72	3.60	3.05	4.57
Water	0.23	0.28	0.37	0.34
Supported Catalyst	1.82	0.00	0.00	0.00
Dispersed Catalyst- Mo	0.00	0.00	0.00	0.05
Dispersed Catalyst - Iron	0.00	0.00	0.06	0.63
Other Catalysts & Chemicals	0.99	2.92	3.08	1.43
Ash Disposal	0.08	0.06	0.09	0.10
Labor	1.55	1.65	1.84	1.51
Maintenance	1.39	1.48	1.65	1.35
Capital-Related	22.85	24.02	26.83	23.42
Byproduct credit	-1.86	-1.88	-2.45	-2.17
Equivalent Crude Oil Price, $/B	32.29	33.76	36.34	32.72

FIGURE 6.1
Simplified Flow Plan of Catalytic Two-Stage Coal Liquefaction Process

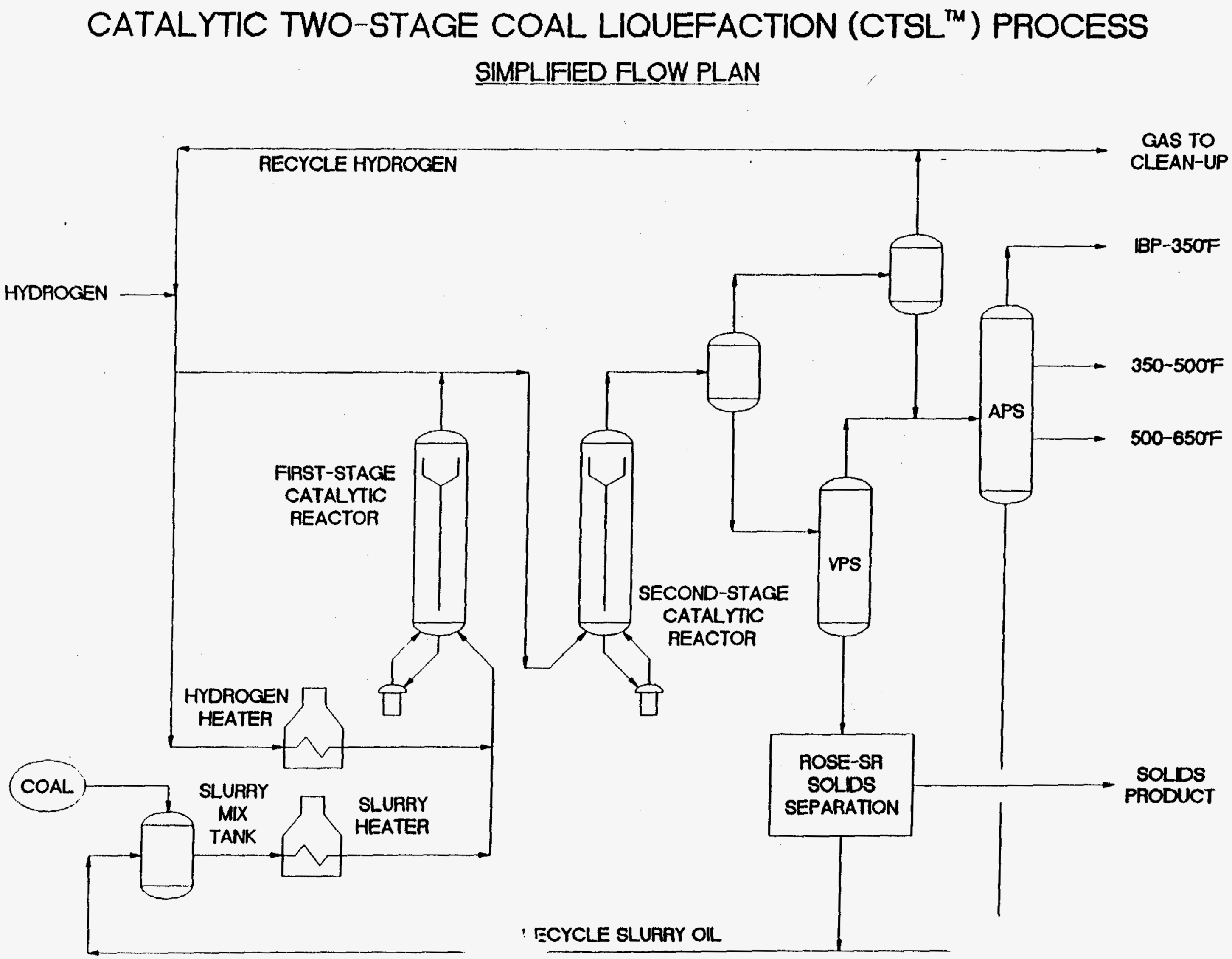


FIGURE 6.2
Block Flow Diagram Of Major Processing Areas

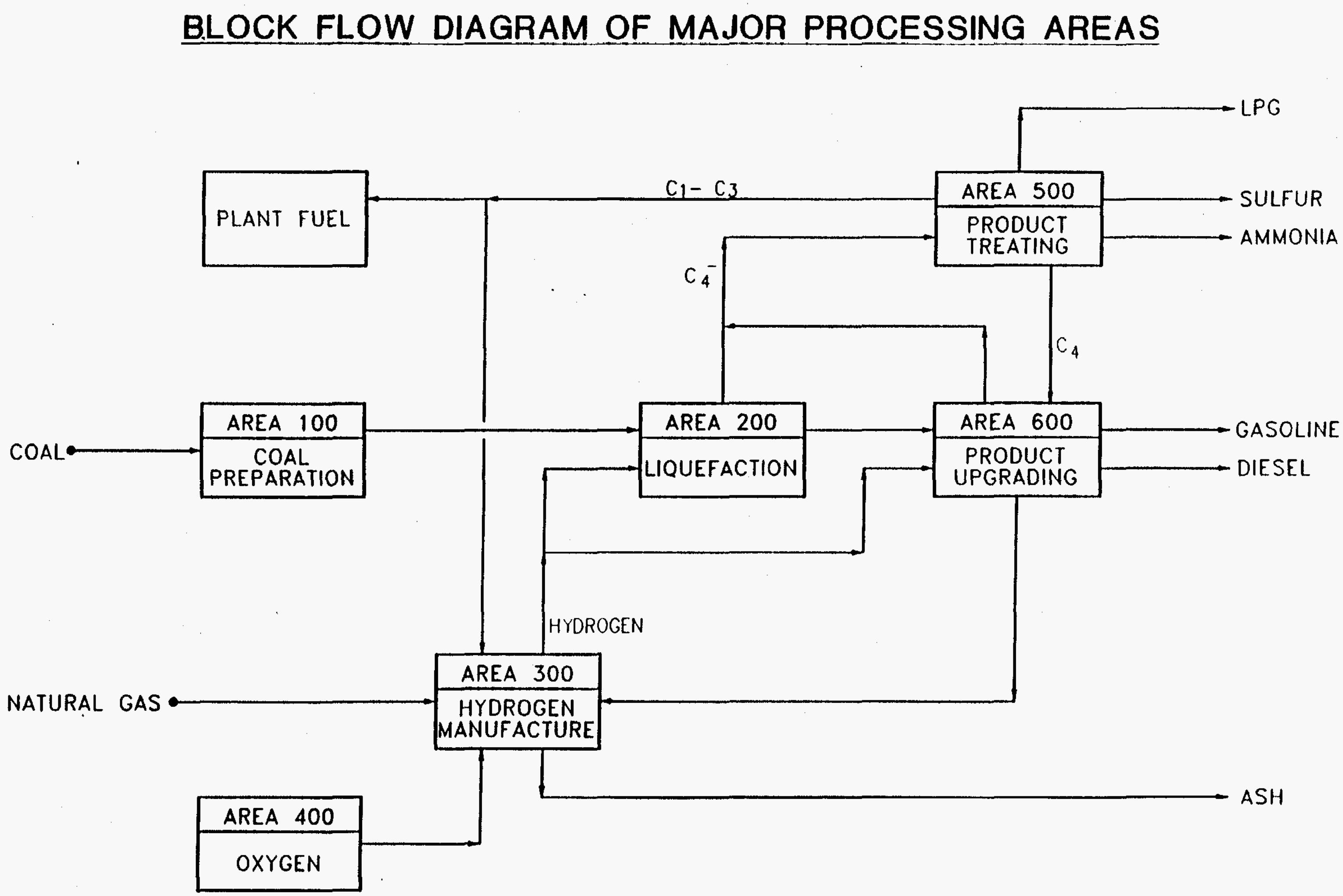


FIGURE 6.3
Sensitivity Analysis - Effect of Total Plant Investment

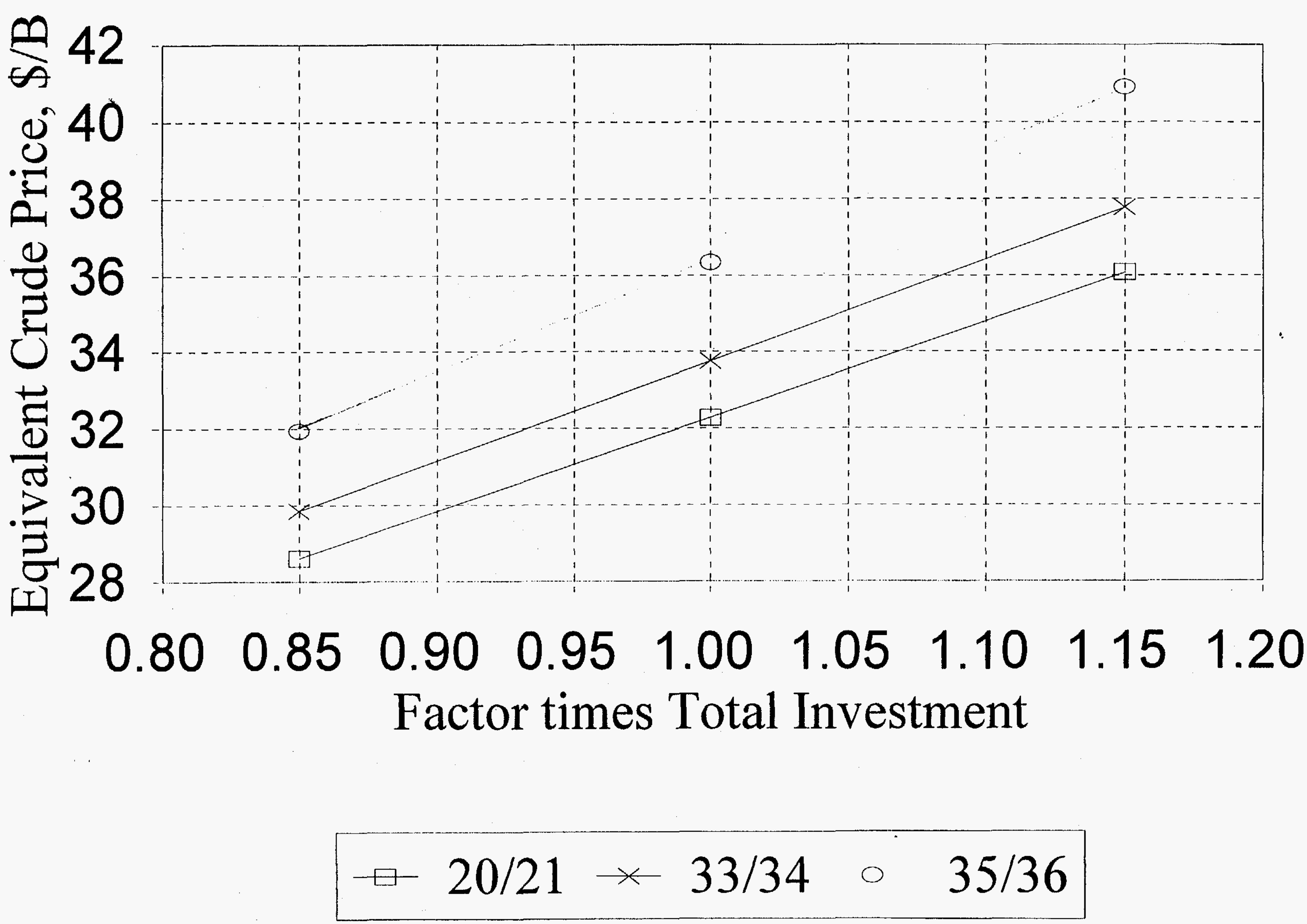


FIGURE 6.4
Sensitivity Analysis - Effect of Natural Gas Cost

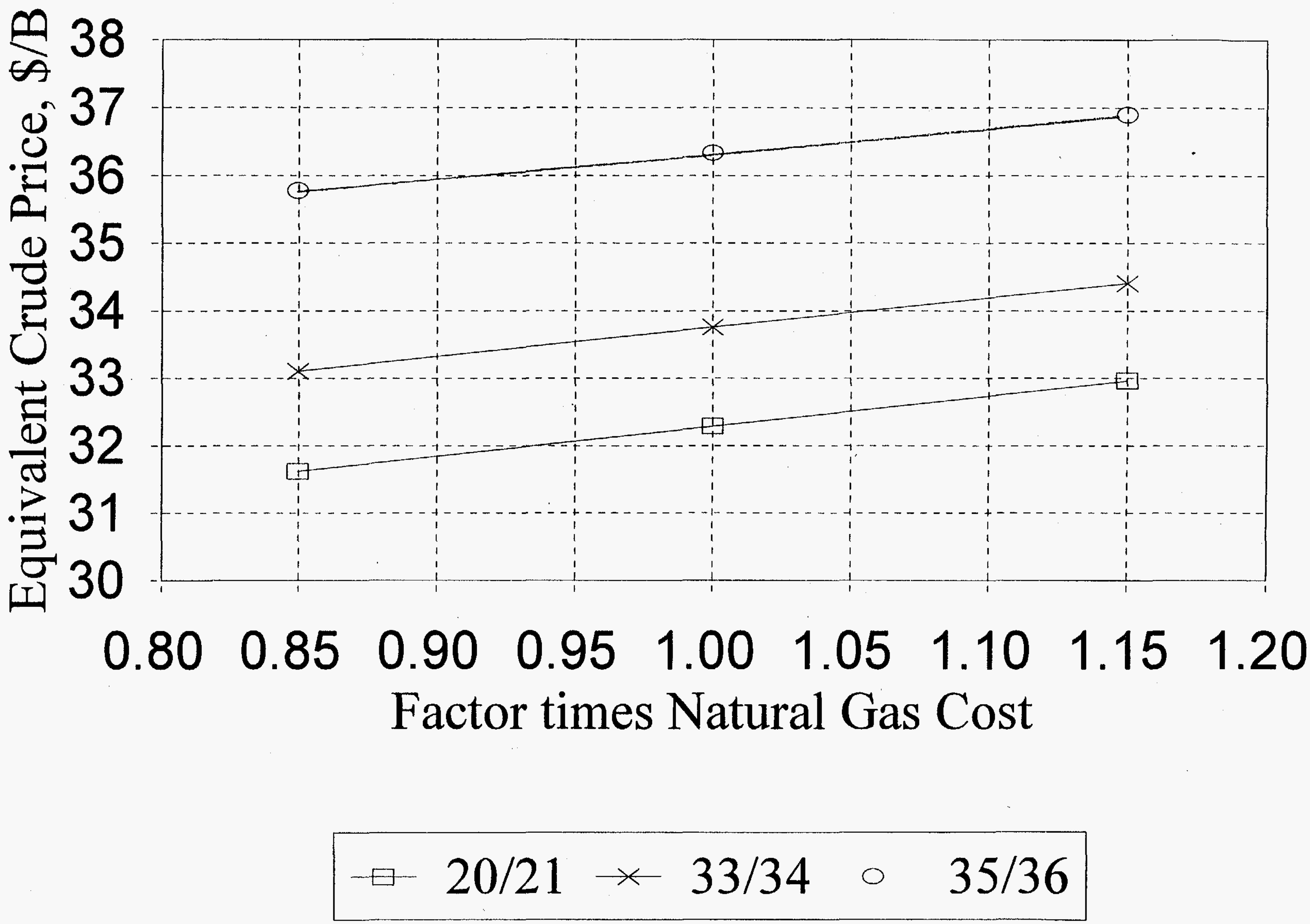


FIGURE 6.5
Sensitivity Analysis - Effect of Catalyst/Chemical Cost

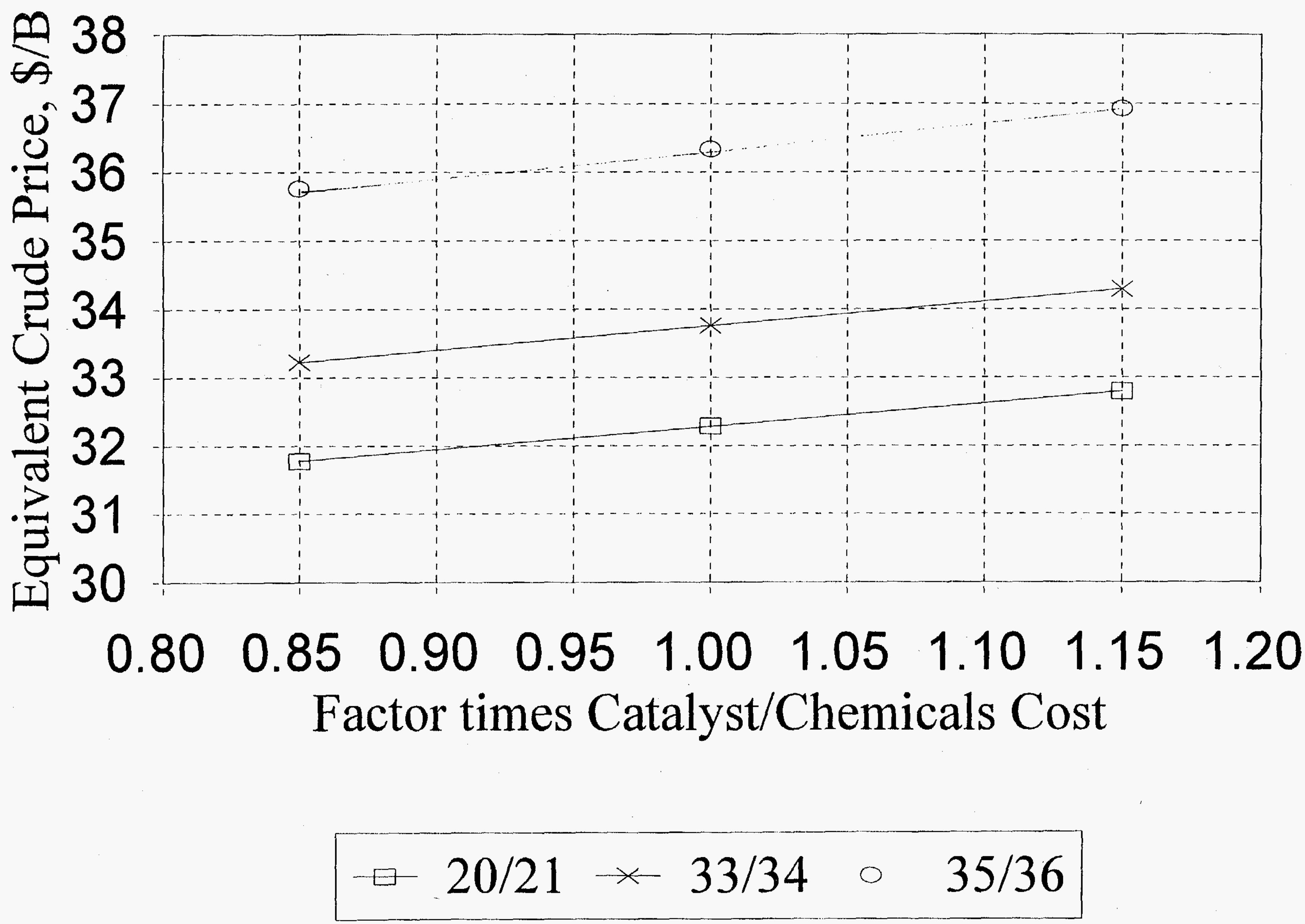
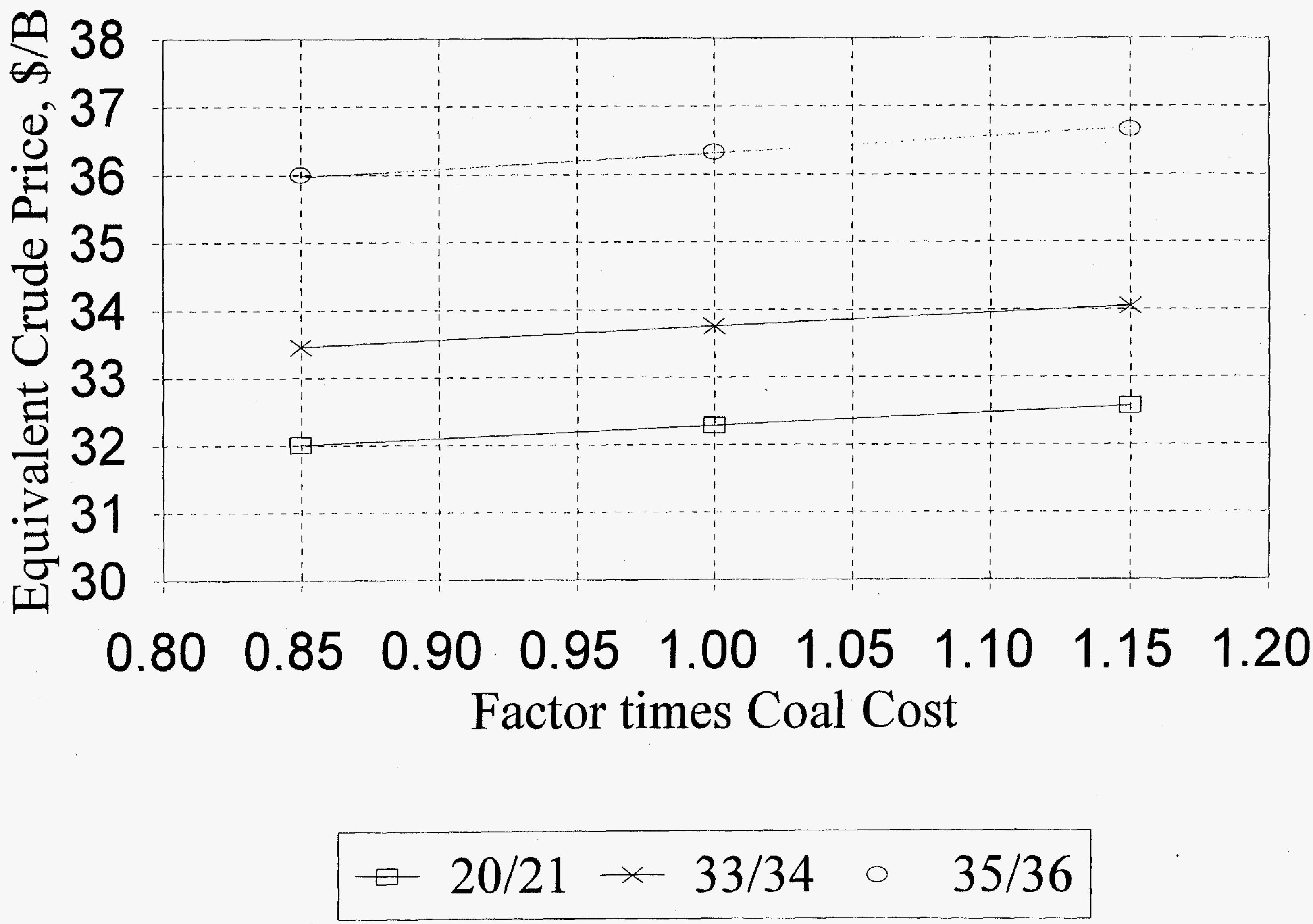


FIGURE 6.6
Sensitivity Analysis - Effect of Coal Cost



SECTION VII

SAMPLES/MATERIAL TESTING

A. EXTERNAL SAMPLES

Among the several objectives of PDU run POC-02, an important one was to collect samples of various internal end product streams for analyses. Several requests for these POC-02 samples were made by other U.S. DOE contractors; Consol, Inc. and Bechtel, Inc. were among the sample recipients. Table 7.1 lists all the daily and special samples collected during POC-02. The daily samples of gaseous, liquid, and solid products and various internal streams were analyzed in house at HTI to determine process yields and product quality. Figure 7.1 shows the locations of the sampling points. A list of all external samples and their recipients is given in Table 7.2. External samples mainly consisted of recycle oil (O-43 oil), naphtha stabilizer bottoms (N-5), O-13 reactor flash vessel bottoms, and make-up oil (Tank 4 oil). The special samples for Consol, Inc. consisted of a number of different process streams and were collected only during the full work-up periods of POC-01. These include Periods 4, 15, 20, 34, 36, 43, and 45 (Table 7.3). The list of special samples, collected during the 'Co-Liquefaction' conditions of POC-02 is given in Table 7.4. These special samples were primarily distributed to Consol, Inc., PETC, and the member universities of the Consortium for Fossil Fuels Liquefaction Science (CFFLS).

The special sample for Bechtel, Inc. was about a 3000 gallon distillate sample collected in three different compartments of a tank truck during the equilibrated periods of the coal-only part of POC-02. This sample was collected for end-use characterization and upgrading to be conducted for Bechtel by Southwest Research Institute in Texas. The collection history and detailed analyses of this distillate sample are given in Table 7.5.

TABLE 7.1
Sampling Points and Sample Description

SP-3	N-5 BOTTOMS
SP-3	N-5 BOTTOMS
SP-4	N-2 BOTTOMS
SP-4	N-2 BOTTOMS
SP-4	N-2 BOTTOMS
SP-5	N-3 OVERHEADS
SP-5	N-3 OVERHEADS
SP-6	N-3 BOTTOMS
SP-9	O-46
SP-11	O-43
SP-11	O-43
SP-12	K-1 LIQUID
SP-16	P-2 COAL
SP-17	K-1 CATALYST
SP-18	K-2 CATALYST
SP-19	O-5 LIQUID
SP-19	O-5 LIQUID
SP-19	O-5 LIQUID
SP-24	P-3 COT
SP-24	P-3 COT
SP-25	DAO, O-65
SP-25	DAO, O-65
SP-27A/B	ROSE SOLIDS
SP-27A/B	ROSE SOLIDS
SP-28	TANK 4

TABLE 7.2
Summary of External Samples

Sample Recepient	Sample ID (type)	Amount	Special Instructions
Bechtel [for SwRI]	C_5-399°C Net Distillates	<=3500 Gallons	Net Distillates* (NSB+ASB)
Bechtel [for SwRI]	O-5 Liquid	Two Gallons	Periods 34 and 44 (HTU Offline)
Bechtel [for SwRI]	C_5-399°C Net Distillates	One Gallon	Net Distillates
PETC	*****See TABLE 7.4*****		
CFFLS	*****See TABLE 7.4*****		
Consol, Inc.	*****See TABLE 7.3*****		

TABLE 7.3
Special Stream Samples for the Consol, Inc.

Period No. _______________ **Date:** _______________

Sample Type	Quantity (I)	Sub-Period (B)
Liquids		
Naphtha Stabilizer (N-5) Bottoms	100 Grams	______
Atmospheric Still (N-2) Bottoms	100 Grams	______
Vacuum Still (N-3) Overheads	100 Grams	______
Clean Oil (P-3) Tank (II)	One Gallon	______
Rose Recycle Oil Receiver (O-65)	500 Grams	______
Filtrate Holding Tank (O-48)	500 Grams (III)	______
Cold Separator (O-5) Liquid	Two Quarts (IV)	______
Slurries		
O-13 Bottoms (O-46) Material (V)	750 Grams	______
Recycle Holding (O-43) Tank	One Gallon (VI)	______
Vacuum Still (N-3) Bottoms	100 Grams	______
Reactor 1, K-1 Slurry (VII)	750 Grams	______
Solids		
Rose Solids Receiver (O-63 A/B)	100 Grams	______
Filter Cake (D-8)	100 Grams (III)	______
Feed Coal (P-2)	0.5 Lbs	______
K-1 Catalyst (Drained)	250 Grams (VIII)	______
K-2 Catalyst (Drained)	250 Grams (VIII)	______
Misc. Oils		
Tank 4 Material (M/U Oil)	One Quart (IX)	______

Notes:
I. Five Gallons of each: N-5 Bottoms, N-2 Bottoms, and N-3 Overheads are to be collected during Period 37.
II. Ten Gallons of P-3 Oil are to be collected Daily during Sub-Periods B of Periods 51 through 60.
III. Only during Period 53.
IV. To be collected during Periods 5, 10 ,13, 18, 21, 26, 29, 32, 34, 37, 42, 45, 50, 53
V. To be collected during ALL Run Periods, 1 through 60.
VI. Ten Gallons of O-43 material is to be collected Daily during Periods 41 through 60.
VII. To be Collected during Sub-Periods A of Periods 12, 20, 30, 36, 44, 52
VIII. 2.5 Lbs Composite from Periods 1 through 10
IX. At the end of Period 1

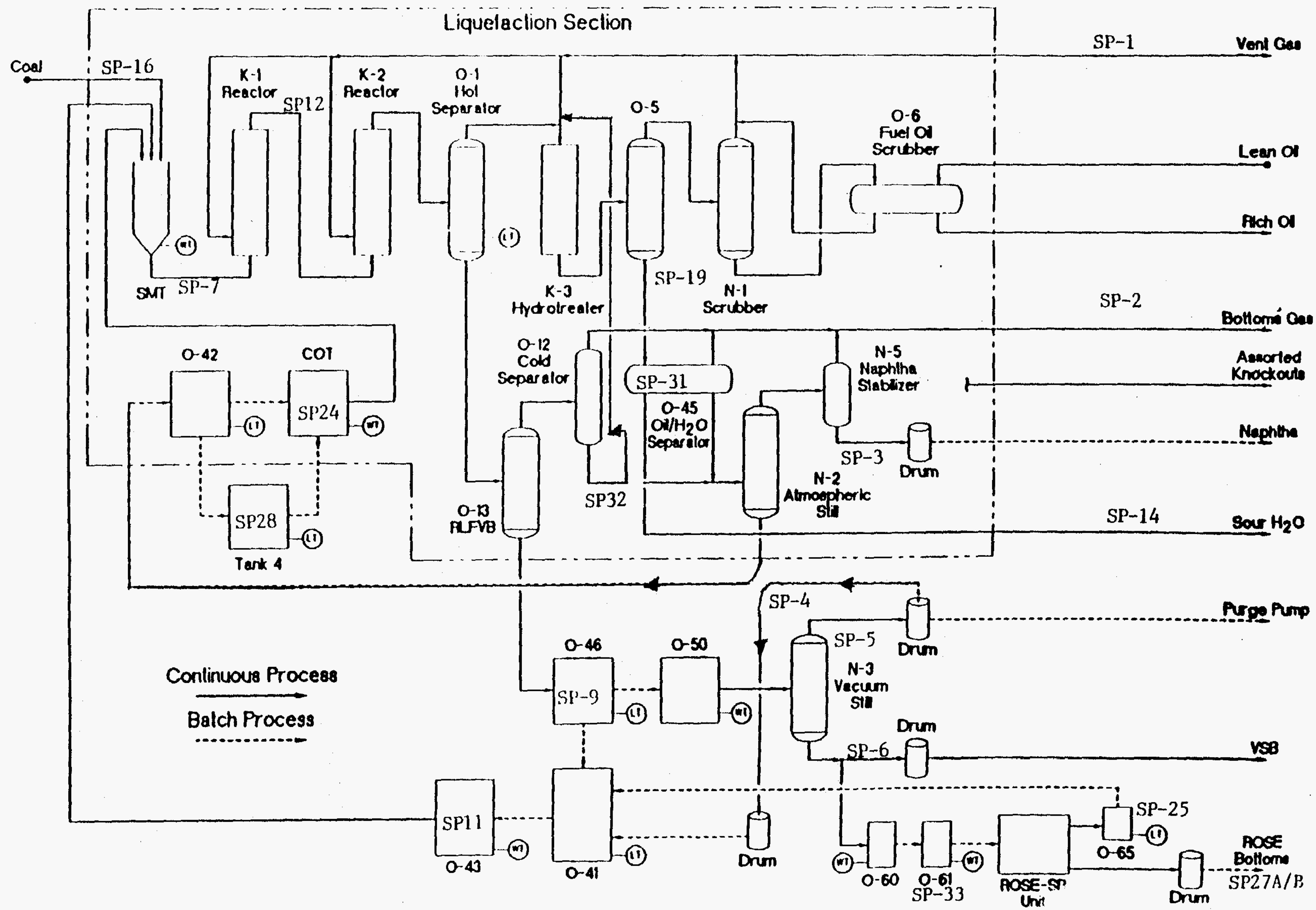
TABLE 7.4
Special Sampling Plan During 'Co-Liquefaction' Conditions

SAMPLE NO.	SAMPLE POINT	DESCRIPTION	QUANTITY	FREQUENCY
1	SP-12	Interstage, K-1 Slurry	1/3 of Total	Periods 39,41, and 43
2	SP-09	O-13 Bottoms [O-46 material]	1 gal	Periods 39,41, and 43
3	SP-19	O-5 oil fraction (HTU feed)	1 gal	Period 41B
4	SP-25	O-65 DAO	1 gal	Periods 39,41, and 43
5	SP-05	N-3 ovhds: VSOH	1 gal	Periods 39, 41, and 43
6	SP-04	N-2 Btms: ASB	1 gal	Periods 39, 41, and 43
7	SP-27	Rose Bottoms	1 gal	Periods 39, 41, and 43
8	SP-07	SMT Feed	1 gal	Periods 39, 41, 42 and 43
9	SP-11	Recycle Oil (O-43)	1 gal	Periods 39, 41, 42, and 43
10	SP-17	K-1 Spent Catalyst	1 lb	Periods 36, 39, 41, and 43
11	SP-18	K-2 Spent Catalyst	1 lb	Periods 36, 39, 41, and 43
12	SP-19	HTU outlet: O-5 oil	1 gal	Periods 39, 41, and 43
13	SP-03	N-5 Btms: NSB	5 gal	Periods 39, 41, and 43
14	SP-16	P-2 Feed Coal	5 lb	Period 41
15	Tank 4	M/U Oil	5 gal	Period 41
16	HDPE PET PS	Plastic Feedstocks	3 lb each	Shut-down

TABLE 7.5
Inspection of Naphtha Stabilizer Bottoms Distillate Sample for End-Use

	Compartments		
	Front	Middle	Rear
Collection Periods	22-31	31-37	14-22
API	32.1	36.2	34.9
IBP, C	81	73.3	83
5 V%	103	101	105
10	119	113	117
20	153	136	139
30	196	161	172
40	231	189	204
50	257	213	227
60	279	237	252
70	296	256	271
80	312	275	293
90	332	295	312
95	345	308	332
EP, C	363	333	348
Distillation, W%			
IBP-177 C	22.43	32.82	28.12
177-288 C	38.84	51.3	48.59
288-343 C	30.29	13.86	18.94
343 C+	7.86	1.66	3.65
LOSS	0.58	0.36	0.7
Elemental, W%			
C	86.52	87.12	86.58
H	12.9	13.06	13.03
N, ppm	53.5	21.2	44.8
S, ppm	8	10	<1

FIGURE 7.1
Material Balance Flow Diagram - Location of Sampling Points



B. MATERIAL TESTING

MATERIAL TESTING

During POC-02, like in POC-01, a cooperative material testing program between Oak Ridge National Laboratory (ORNL) and HTI was implemented. Six sets of corrosion coupons were supplied by ORNL and installed at selected locations in the PDU. These locations included reactors, hydrotreater, hot separator, atmospheric and vacuum stills.

Each set consisted of 5 to 7 individual coupons of different materials. The coupon material and location for each set are given in Table 7.6. The corresponding exposure times are summarized in Table 7.7. The information on corrosion rates, as relayed by the ORNL, is listed in Tables 7.8 and 7.9.

TABLE 7.6

POC-02 Corrosion Coupon Materials

Coupon Location	Coupon Materials
Reactors (1)	a. 2 1/4 Cr - 1 Mo Steel b. 9 Cr - 1 Mo Steel c. 321 Stainless Steel d. Al_2O_3 Plasma Spray Coating - A e. Al_2O_3 Plasma Spray Coating - B
Hydrotreater & Hot Separator	a. 2 1/4 Cr - 1 Mo Steel b. Modified 9 Cr - 1 Mo Steel c. 304L Stainless Steel d. 316L Stainless Steel e. 347 Stainless Steel f. Incoloy 825 g. Fe_3Al
Atmospheric Still & Vacuum Still	a. Carbon Steel b. 1 1/4 Cr - ½ Mo Steel c. 2 1/4 Cr - 1 Mo Steel d. 5 Cr - ½ Mo Steel e. 7 Cr - 1 Mo Steel f. Modified 9 Cr - 1 Mo Steel

(1) Reactor corrosion coupons provided by Mitsui SRC Development Co.

TABLE 7.7
Corrosion Coupon Status-I

	Days of Exposure			
	Run 260-04		Run 260-05	
	Coal	Oil	Coal	Oil
First Stage Reactor	20	5	45	23
Second Stage Reactor	48	18	45	23
Hydrotreater	58	28	42	4
Hot Separator (Vapour)	0	0	45	23
Vacuum Still (Condenser)	22	9	45	23

TABLE 7.8
Corrosion Coupon Status-II

Coupon	Hydrotreater 260-04	Hydrotreater 260-05	Hot Sep. 260-05
316L	0.19	-0.091	0.004
347 SS	<0.03	0.024	-0.027
304L SS	<0.03	0.053	-0.017
321 SS	0.03	0.094	0.028
Incoloy 825	0.07	0.013	0.021
2.25Cr- 1Mo	1.58	2.012	1.925
Fe3Al	<0.03	0.127	0.328
9Cr-1Mo	1.14	1.083	0.685

Coupon	Vac. Still 260-05
CS	0.009
1.25C-0.5Mo	0.008
2Cr-1.0Mo	0.000
5Cr-0.5Mo	-0.001
7Cr-1.0Mo	-0.001
9Cr-1.0Mo	0.000

TABLE 7.9
Relative Corrosion Rates

	Hydrotreater 260-04	Hydrotreater 260-05	Hot Sep. 260-05
Low Rate	Fe3L, 347SS,304L	(316L)	(347SS,304L)
		Incoloy 825,347L	316L
	321SS	304L	Incoloy 825
	Incoloy 825	321SS	321SS
	316L	Fe3Al	Fe3Al
	9Cr-1Mo	9Cr-1Mo	9Cr-1Mo
High Rate	2.25Cr-1Mo	2.25Cr-1Mo	2.25Cr-1Mo

SECTION VIII

REFERENCES

1. A.G. Comolli et al., "Hydrocarbon Research, Inc., Technical Progress Report - POC Run 01 (260-04)" DE-92148-TOP-01, August 1995.

2. A.G. Comolli et al., "Catalytic Two-Stage Liquefaction (CTSL) Process Bench Studies with Bituminous coal", DE-88818-TOP-02, May 1993, Hydrocarbon Research, Inc. for Department of Energy, Contract No. DE-AC22-88PC88818.

3. A.G. Comolli et al., "Catalytic Two-Stage Coal Liquefaction, CTSL-Proof-of-Concept and Developments", a Paper Presented at The NEDO 1994 Coal Liquefaction and Materials for Coal Liquefaction Joint Technical Meeting in Tokyo, Japan, January 1994.

4. "Southern Electric International, Inc., Technical Progress Report", Run 262 with Black Thunder Subbituminous Coal", DOE/PC/90033-22, September 1992.

5. "Southern Electric International, Inc., Technical Progress Report", Run 263 with Black Thunder Subbituminous Coal and Dispersed Molybdenum Catalysts", DOE/PC/90033-23, December 1992.

6. "Final Report on Baseline and Improved Baseline Designs", Bechtel Corporation, under Contract No. DE-AC22 90PC89857, March 1993.

APPENDIX A

DEFINITION AND NOMENCLATURE

Terminologies, that are used in this report, are defined in the following section:

A. Major Process Equipment

Symbol	Description
L-1	Fresh Feed Heater
L-2	Recycle Gas Heater
K-1	First Stage Reactor
K-2	Second Stage Reactor
K-3	In-line Hydrotreater
N-1	Scrubber
N-2	Atmospheric Tower
N-3	Vacuum Still
N-5	Naphtha Stabilizer Column
O-1	Hot Separator
O-5	Reactor Overheads Separator
O-12	Reactor Liquid Flash Drum
O-13	Reactor Liquid Flash Drum
O-40	Purge Oil Tank
O-41	Recycle Holding Tank
O-42	Flush/Purge Oil Storage
O-43	Recycle Weigh Drum
O-46	O-13 Liquid Surge Drum
O-47	Filter Feed Drum
O-48	Filtrate Receiver
O-50	N-3 Feed Accumulator
O-60	VSB Holding Tank
O-61	Settler Feed Tank
O-63	ROSE Residues Receiver
O-65	Recycle Oil Receiver
P-1	Coal Day Hopper
P-2	Coal Feed Hopper
P-3	Clean Oil Tank
P-4	Slurry Oil Tank

NOMENCLATURE:

Normalization Factor — A factor used to normalized the raw material balance data and is defined as:

$$\frac{100}{Wt\%\ Mass\ Recovery}$$

Normalized yields is equal to the product of net yield multiplied by the normalization factor.

Coal Conversion — The conversion of coal into gases, water and quinoline soluble liquid products.

$$100\ x \left[1 - \frac{w\%\ Quinoline\ Insoluble\ Organic\ in\ \ Product}{w\%\ M.A.F.\ coal\ in\ \ Feed}\right]$$

524°C+ Resid Conversion: — The Conversion of coal and 524°C+ resid into gases and 524°C- distillates

$$100 \left[1 - \frac{W\%\ 524°\ C+\ in\ \ Product}{W\%\ 524°C+\ in\ \ Feed}\right]$$

Hydrodesulfurization: — The removal of organic sulfur from the net liquid products.

$$100 \left[1 - \frac{W\%\ Sulfur\ in\ \ Liquid\ Product}{W\%\ Sulfur\ in\ \ Total\ Feed}\right]$$

Hydrodenitrogenation: — The removal of nitrogen from the net liquid products. .

$$\text{Hydrodenitrogenation} = 100 \times \left[- \frac{W\%\ Nitrogen\ in\ Liquid\ and\ Solid}{W\%\ Nitrogen\ in\ Total\ Feed} \right]$$

Organic Rejection: Rejection of organic matter in ROSE Bottoms or Filter Cake.

$$100 \times \left[\frac{W\%\ Organic\ in\ Ash\ Reject \times Wt.\ of\ Adjusted\ Ash\ Reject}{Wt.\ of\ Moisture\text{-}Ash\text{-}Free\ Coal} \right]$$

Energy Rejection: Rejection of heating value of feed coal through Ash Rejects

$$100 \times \left[W\%\ Ash\ Reject,\ MF\ Coal \times \frac{Heating\ Value\ of\ Ash\ Reject}{Heating\ Value\ of\ MF\ Coal} \right]$$

QI	Quinoline Insoluble Material in Bottoms Product
DAO	Deashed oil from ROSE-SR unit
CTSL	Catalytic Two-Stage Liquefaction
CMSL	Catalytic Multi-Stage Liquefaction
COT	Clean Oil Tank
VSO	Vacuum Still Overheads
NSB	Naphtha Stabilizer Bottoms
ASB	Atmospheric Still Bottoms
RLFVB	Reactor Liquid Flash Vessel Bottoms
SMT	Slurry Mix Tank
LCV	Level Control Valve
STTU	Standard Time-Temperature Unit
Hydrogen Efficiency	The ratio of amount of C_4-524°C distillate products formed to the total chemical hydrogen consumption

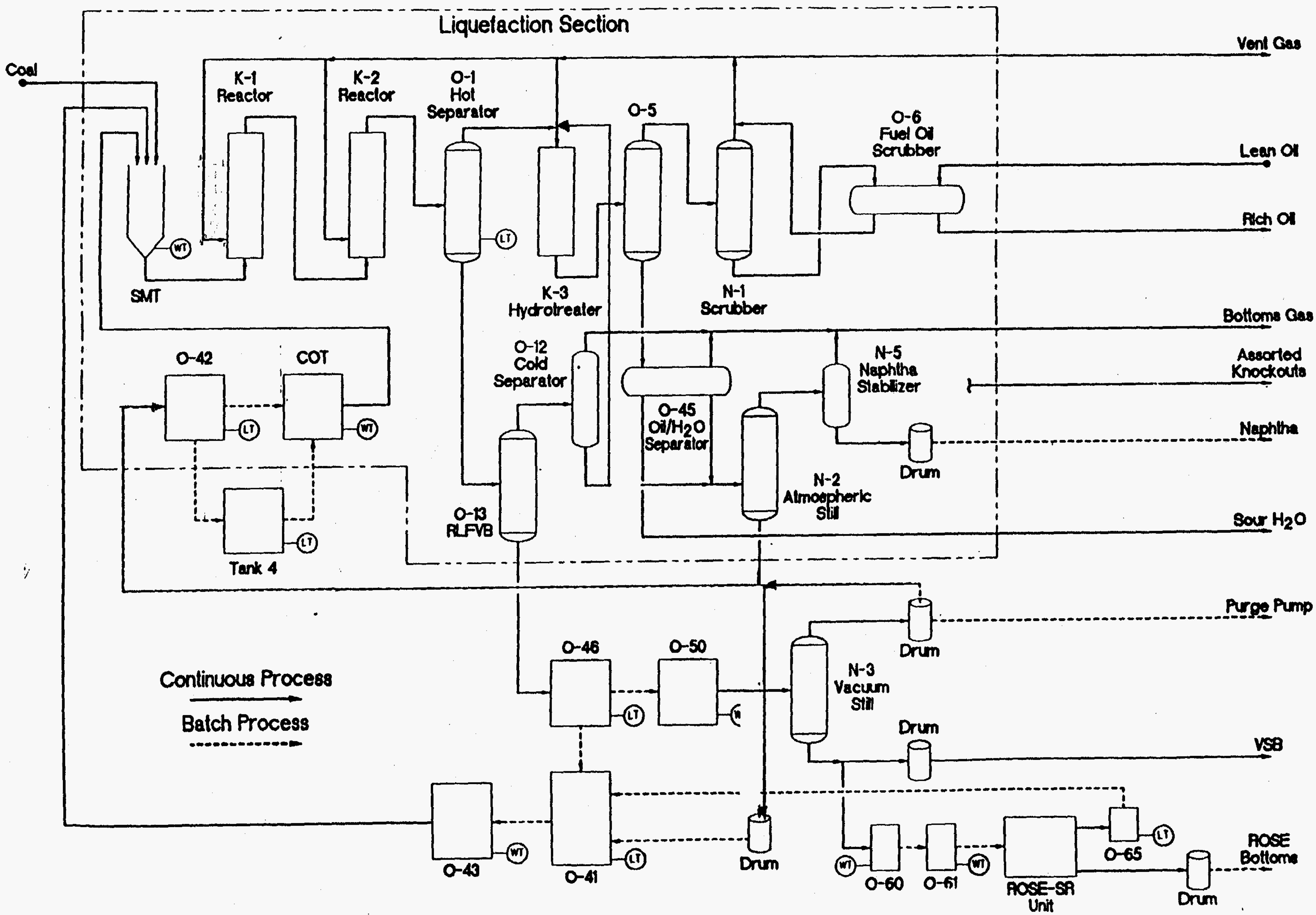
APPENDIX B

MATERIAL BALANCE METHODOLOGY

The material balance for this run was calculated in two different ways, as an overall unit balance around the entire process and as a liquefaction section balance which excludes the solid separation equipment. As can be seen in Figure B-1, the overall balance includes the various feeds and all the final products, which are taken only after the internal recycle is accounted for. This gives a total balance around all the equipment including whichever solid separation system is being used. This includes a number of batch transfer operations (ie. from O-46 to O-50, from O-41 to O-43) which makes the inventory changes in these vessels critical to a tight material balance closure.

The liquefaction balance is performed exclusive of the solid separation equipment. This balance stops at the O-46 vessel which is the Reactor Liquid Flash Vessel Bottoms Receiver. From this point, there are several possible flow paths. During ashy-recycle mode, a portion of this stream is fed to the O-41 vessel which is part of the recycle oil system. This stream can also be feed directly to the filter system or the Vacuum Still Feed Tank, through the Vacuum Still, and the bottoms then routed to the ROSE-SR^{SM} unit. Due to all the various ways that material in the O-46 vessel can be routed, this was chosen as the cut-off point for the liquefaction balance. Results determined at this point would be independent of which solid separation system is actually being used (except as the quality of the recycle solvent from the various solid separation systems would vary and affect performance). The liquefaction section material balance is the one used to determine all the process performance and the normalized yield calculations.

260-05 MATERIAL BALANCE FLOW DIAGRAM



POC-02 (RUN 260-05) MATERIAL BALANCE

COAL: Wyoming Black Thunder Mine (HRI-6209)
CATALYST: Reactors ==> Akzo AO-60 1/16" (HRI-6043)
Hydrotreater ==> Criterion 411 (HRI-6135)

OVERALL MATERIAL BALANCE

Period	01T	02T	03T	04T	05T	06T	07T	08T
Period Start Date	06/01/94	06/02/94	06/03/94	06/04/94	06/05/94	06/06/94	06/11/94	06/12/94
Period Start Time	04:00	04:00	04:00	04:00	04:00	04:00	04:00	04:00
Period Duration Hours	24	24	24	24	24	12	24	24
Solids Separation Type	VAC STIL	VAC STIL	VAC STIL	ROSE-SR	ROSE-SR	VAC STIL	VAC STIL	VAC STIL
STREAMS IN, KGS								
Coal Feed Wet (Less Sample)	1705.4	1908.6	1508.5	1884.2	1633.1	401.4	1614.2	1892.6
Make-Up Oil to Mix Tank	0.0	422.7	565.2	0.0	719.8	0.0	0.0	0.0
Mix Tank Inventory Loss	-18.6	-55.3	21.8	-21.8	70.8	-27.7	-78.9	-24.0
Seal Oil to Ebullating Pumps	43.1	43.5	41.9	41.4	37.6	11.4	43.2	38.6
Make-Up Oil to Purge Pumps	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Water Injected to O-1	785.1	952.6	1073.1	1245.2	1431.7	343.6	870.2	943.9
Fresh Hydrogen Feed	161.9	198.7	112.1	179.2	182.8	82.3	125.4	162.9
DMDS (TNPS)	54.7	37.0	34.2	32.2	32.8	5.4	28.8	22.0
TOTAL FEED:	2731.6	3507.8	3356.8	3360.4	4108.6	816.6	2602.9	3036.0
STREAMS OUT, KGS								
Vent Gas (Dry & N2-Free)	31.6	61.3	38.4	53.3	58.6	6.2	39.3	50.5
Bottoms Flash Gas (Dry & N2-Free)	118.6	269.1	220.9	280.4	255.0	70.7	163.4	247.8
Mix Tank Vent Drain	1.0	1.1	0.8	1.2	1.2	0.4	1.7	0.1
Unit Knockouts	76.5	134.2	33.4	165.6	48.3	0.0	2.7	5.7
Naphtha Stabilizer Bottoms	740.0	1046.2	1062.9	1088.8	977.4	296.2	163.8	767.7
Atmospheric Still Bottoms Product	320.6	-224.0	476.5	-369.1	343.7	117.0	127.3	476.0
Separated Water (Plus Water in Gases)	1061.0	1574.5	1873.1	1854.1	2051.2	525.2	1685.2	1494.6
Vacuum Still Overhead Product	238.9	221.8	-83.4	81.2	-154.1	-41.8	127.7	-359.2
Vacuum Still Bottoms Product	0.0	674.1	220.3	2.0	1.9	168.7	283.3	663.1
Pressure Filter Cake	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Filter Section Net Inv. Change	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ROSE Unit DAO Product	0.0	0.0	0.0	2.7	-55.3	0.0	0.0	0.0
ROSE Unit Bottoms	0.0	0.0	0.0	396.4	45.8	0.0	0.0	0.0
ROSE Section Net Inv. Change	0.0	0.0	0.0	-277.6	456.3	0.0	0.0	0.0
Recycle Oil Net Inv. Change	15.0	-277.6	-107.5	265.8	-219.1	-100.7	-61.7	-13.2
Vacuum Still Feed Tank Inv. Change	125.2	-390.1	109.3	-174.6	440.4	-49.4	-122.9	-135.6
RLFV Bottoms Holding Tank Inv. Change	2.3	-38.6	-108.9	48.5	230.9	57.2	-56.7	-54.4
TOTAL PRODUCTS:	2730.6	3052.0	3735.9	3418.6	4482.2	1049.7	2353.1	3142.9
OVERALL UNIT MATERIAL RECOVERY, W%	100.0	87.0	111.3	101.7	109.1	128.5	90.4	103.5

LIQUEFACTION SECTION MATERIAL BALANCE (Reactor Section)

Period	01T	02T	03T	04T	05T	06T	07T	08T
STREAMS IN, KGS								
Coal Feed Wet (Less Sample)	1705.4	1908.6	1508.5	1884.2	1633.1	401.4	1614.2	1892.6
Oil Streams to the SMT								
Recycle to SMT	2852.3	1129.9	915.8	878.2	1268.2	368.3	2686.2	1731.4
Make-Up Oil to SMT	0.0	422.7	565.2	0.0	719.8	0.0	0.0	0.0
VSOH recycled to SMT	104.5	0.0	88.5	197.8	461.8	249.5	1004.6	685.4
Mix Tank Inventory Loss	-18.6	-55.3	21.8	-21.8	70.8	-27.7	-78.9	-24.0
Seal Oil to Ebullating Pumps	43.1	43.5	41.9	41.4	37.6	11.4	43.2	38.6
VSO to Purge Pumps	221.5	225.8	182.7	172.0	157.7	41.8	243.2	193.3
Make Up Oil to Purge Pumps	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Water Injected to O-1	785.1	952.6	1073.1	1245.2	1431.7	343.6	870.2	943.9
Fresh Hydrogen Feed	161.9	198.7	112.1	179.2	182.8	82.3	125.4	162.9
DMDS (TNPS)	54.7	37.0	34.2	32.2	32.8	5.4	28.8	22.0
TOTAL FEED:	5805.5	5528.4	4585.0	5473.7	5844.8	1466.6	5641.5	5402.5
STREAMS OUT, KGS								
Vent Gas (Dry & N2-Free)	31.6	61.3	38.4	53.3	58.6	6.2	39.3	50.5
Bottoms Flash Gas (Dry & N2-Free)	118.6	269.1	220.9	280.4	255.0	70.7	163.4	247.8
Mix Tank Vent Drain	1.0	1.1	0.8	1.2	1.2	0.4	1.7	0.1
Unit Knockouts	76.5	134.2	33.4	165.6	48.3	0.0	2.7	5.7
Naphtha Stabilizer Bottoms	740.0	1046.2	1062.9	1088.8	977.4	296.2	163.8	767.7
Atmospheric Still Bottoms	320.6	440.9	606.2	694.1	653.9	357.0	236.6	917.8
Separated Water (Plus Water in Gases)	1061.0	1574.5	1873.1	1854.1	2051.2	525.2	1685.2	1494.6
Reactor Liquid Flash Vessel Bottoms	3557.5	1803.9	1160.3	1593.9	2069.7	347.9	3143.8	2024.4
TOTAL PRODUCTS:	5906.8	5331.3	4996.0	5731.3	6115.4	1603.6	5436.6	5508.4
LIQUEFACTION SECTION RECOVERY, W%	101.7	96.4	109.0	104.7	104.6	109.3	96.4	102.0
SOLVENT TO COAL (MF) RATIO	1.86	1.31	1.21	1.16	1.59	1.71	1.96	1.30

Period	01T	02T	03T	04T	05T	06T	07T	08T
VACUUM STILL SECTION MATERIAL BALANCE (Includes Inventory Changes)								
STREAMS IN, KGS								
Feed to Vacuum Still	1935.9	1458.3	875.0			340.2	1787.1	1156.2
STREAMS OUT, KGS								
Vacuum Still Overhead Product	564.9	447.5	187.7	450.9	465.4	249.5	1375.4	519.5
Vacuum Still Bottoms Product	0.0	674.1	220.3	2.0	1.9	168.7	283.3	663.1
VAC STILL SECTION MATERIAL RECOVERY, W%	100.0	102.8	46.6			122.9	92.8	102.2
FEED RATES, KGS/HR								
Feed to Vacuum Still	80.7	60.8	36.5			28.3	74.5	48.2
PRODUCT RATES, KGS/HR								
Vacuum Still Overhead Product	23.5	18.6	7.8	18.8	19.4	20.8	57.3	21.6
Vacuum Still Bottoms Product	57.2	43.8	9.1	0.0	0.0	14.1	11.7	27.6
ROSE UNIT MATERIAL BALANCE (Includes Inventory Changes)								
STREAMS IN, KGS								
Feed to ROSE Unit	0.0	0.0	0.0	1404.3	402.3	0.0	0.0	0.0
Makeup Solvent to Rose Unit	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
STREAMS OUT, KGS								
ROSE Unit DAO Product	0.0	0.0	0.0	982.5	339.7	0.0	0.0	0.0
ROSE Unit Residuals	0.0	0.0	0.0	396.4	45.8	0.0	0.0	0.0
ROSE SECTION MATERIAL RECOVERY, W%				98.2	95.8			
FEED RATES, KGS/HR								
Feed to ROSE Section	0.0	0.0	0.0	58.5	16.8	0.0	0.0	0.0
Makeup Solvent to Rose Unit	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
PRODUCT RATES, KGS/HR								
ROSE Unit DAO Product	0.0	0.0	0.0	40.9	14.2	0.0	0.0	0.0
ROSE Unit Residuals	0.0	0.0	0.0	16.5	1.9	0.0	0.0	0.0

Period	01T	02T	03T	04T	05T	06T	07T	08T
RECYCLE RATES TO SMT, KGS/HR								
Reactor Liq Flash Vessel Bottoms	60.1	23.9	25.7	0.0	0.0	0.0	66.1	43.5
Atmospheric Still Bottoms	0.0	27.7	5.4	44.3	12.9	20.0	4.6	18.4
ROSE Unit DAO	0.0	0.0	0.0	39.7	20.2	0.0	0.0	0.0
Vacuum Still Overheads	4.2	0.0	8.0	8.0	23.6	22.3	43.2	28.1
Vacuum Still Bottoms	57.2	15.7	0.0	0.0	0.0	0.0	0.0	0.0
COMPOSITION OF RECYCLE TO SMT (100% RECYCLE BASIS), W%								
Reactor Liq Flash Vessel Bottoms	49.5	35.5	65.8	0.0	0.0	0.0	58.0	48.3
Atmospheric Still Bottoms	0.0	41.2	13.8	48.2	22.8	47.3	4.0	20.5
ROSE Unit DAO	0.0	0.0	0.0	43.1	35.6	0.0	0.0	0.0
Vacuum Still Overheads	3.5	0.0	20.4	8.7	41.6	52.7	38.0	31.3
Vacuum Still Bottoms	47.1	23.4	0.0	0.0	0.0	0.0	0.0	0.0
NET COLLECTED PRODUCTS, KGS/HR (Includes Samples)								
GASES: C1-C3	2.44	6.76	5.93	7.02	6.45	3.09	4.47	6.93
C4-C7	1.16	2.93	2.48	2.77	2.90	1.60	1.54	3.48
CO & CO2	0.01	0.24	0.23	0.42	0.46	0.22	0.47	0.21
H2S	1.50	3.00	1.26	2.94	2.22	1.21	1.10	1.02
Net Water	4.4	16.8	26.1	16.4	18.0	11.3	26.2	13.9
Mix Tank Vent Drain	0.0	0.0	0.0	0.0	0.1	0.0	0.1	0.0
Unit Knockouts	3.2	5.6	1.4	6.9	2.0	0.0	0.1	0.2
Naphtha Stabilizer Bottoms	30.8	43.6	44.3	45.4	40.7	24.7	6.8	32.0
Atmospheric Still Bottoms	13.4	-9.3	19.9	-15.4	14.3	9.8	5.3	19.8
Vacuum Still Overhead (Net)	10.0	9.2	-3.5	3.4	-6.4	-3.5	5.3	-15.0
Vacuum Still Bottoms (Net)	0.0	28.1	9.2	0.1	0.1	14.1	11.8	27.6
Filter Cake	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ROSE Unit DAO	0.0	0.0	0.0	0.1	-2.3	0.0	0.0	0.0
ROSE Unit Residuals	0.0	0.0	0.0	16.5	1.9	0.0	0.0	0.0
TOTAL:	66.9	106.7	107.1	86.1	80.0	62.2	62.8	90.0

POC-02 (RUN 260-05) MATERIAL BALANCE

COAL: Wyoming Black Thunder Mine (HRI-6209)
CATALYST: Reactors ==> Akzo AO-60 1/16" (HRI-6043)
Hydrotreater ==> Criterion 411 (HRI-6135)

OVERALL MATERIAL BALANCE

Period	09T	10T	11T	12T	13T	14T	15T	16T
Period Start Date	06/13/94	06/14/94	06/15/94	06/16/94	06/17/94	06/18/94	06/19/94	06/20/94
Period Start Time	04:00	04:00	04:00	04:00	04:00	04:00	04:00	04:00
Period Duration Hours	24	24	24	24	24	24	24	24
Solids Separation Type	VAC STIL	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR
STREAMS IN, KGS								
Coal Feed Wet (Less Sample)	1689.4	1899.9	1899.3	1904.4	1943.0	1931.6	1798.9	1723.0
Make-Up Oil to Mix Tank	533.9	370.1	0.0	0.0	58.1	253.6	122.9	79.4
Mix Tank Inventory Loss	-17.7	34.9	-23.1	17.2	34.9	-5.9	43.1	-10.4
Seal Oil to Ebullating Pumps	42.6	41.7	41.8	52.2	51.7	50.2	50.9	59.3
Make-Up Oil to Purge Pumps	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Water Injected to O-1	942.6	945.0	961.9	610.8	621.4	797.8	920.9	950.5
Fresh Hydrogen Feed	174.2	173.0	158.9	157.2	175.3	170.9	91.5	153.3
DMDS (TNPS)	19.6	23.6	5.9	44.0	51.3	43.1	40.4	43.1
TOTAL FEED:	3384.5	3488.2	3044.7	2785.8	2935.6	3241.3	3068.6	2998.1
STREAMS OUT, KGS								
Vent Gas (Dry & N2-Free)	59.3	43.8	67.6	51.3	60.7	51.3	27.7	43.8
Bottoms Flash Gas (Dry & N2-Free)	248.9	282.6	200.8	269.3	219.1	223.3	222.0	230.6
Mix Tank Vent Drain	0.5	0.3	0.0	0.3	0.3	0.3	0.5	0.2
Unit Knockouts	14.2	7.8	6.5	5.3	3.2	4.3	4.0	3.9
Naphtha Stabilizer Bottoms	798.2	927.5	928.2	938.2	1009.5	966.4	947.5	903.6
Atmospheric Still Bottoms Product	17.6	73.9	200.1	319.6	-292.9	-3.0	348.0	52.1
Separated Water (Plus Water in Gases)	1529.6	1526.4	1589.9	1152.4	1126.3	1362.5	1421.2	1415.8
Vacuum Still Overhead Product	-217.8	296.0	-502.3	-39.0	309.8	150.2	-294.7	26.9
Vacuum Still Bottoms Product	626.2	1.8	1.5	2.0	1.7	2.1	1.9	2.3
Pressure Filter Cake	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Filter Section Net Inv. Change	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ROSE Unit DAO Product	0.0	-38.6	-2.3	5.9	-3.6	138.3	-1.8	-0.9
ROSE Unit Bottoms	0.0	566.5	738.0	556.7	501.8	547.2	436.8	559.5
ROSE Section Net Inv. Change	0.0	-23.1	-206.4	-277.6	-36.3	-68.5	-14.5	-141.5
Recycle Oil Net Inv. Change	-56.2	71.2	204.1	-283.9	68.9	47.2	-76.2	-57.2
Vacuum Still Feed Tank Inv. Change	304.8	-222.7	-19.5	195.0	47.6	-147.4	-31.8	-101.2
RLFV Bottoms Holding Tank Inv. Change	-12.2	5.4	-40.8	-53.5	-32.7	75.7	10.4	1.8
TOTAL PRODUCTS:	3313.1	3518.7	3165.5	2842.0	2983.3	3350.1	3000.9	2939.9
OVERALL UNIT MATERIAL RECOVERY, W%	97.9	100.9	104.0	102.0	101.6	103.4	97.8	98.1

LIQUEFACTION SECTION MATERIAL BALANCE (Reactor Section)

Period	09T	10T	11T	12T	13T	14T	15T	16T
STREAMS IN, KGS								
Coal Feed Wet (Less Sample)	1689.4	1899.9	1899.3	1904.4	1943.0	1931.6	1798.9	1723.0
Oil Streams to the SMT								
Recycle to SMT	1152.1	1147.6	1352.2	1587.1	941.7	1078.6	1507.7	985.7
Make-Up Oil to SMT	533.9	370.1	0.0	0.0	58.1	253.6	122.9	79.4
VSOH recycled to SMT	408.8	256.9	862.1	287.4	67.0	381.4	732.4	332.5
Mix Tank Inventory Loss	-17.7	34.9	-23.1	17.2	34.9	-5.9	43.1	-10.4
Seal Oil to Ebullating Pumps	42.6	41.7	41.8	52.2	51.7	50.2	50.9	59.3
VSO to Purge Pumps	182.9	203.0	197.4	224.7	225.6	181.1	168.5	217.3
Make Up Oil to Purge Pumps	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Water Injected to O-1	942.6	945.0	961.9	610.8	621.4	797.8	920.9	950.5
Fresh Hydrogen Feed	174.2	173.0	158.9	157.2	175.3	170.9	91.5	153.3
DMDS (TNPS)	19.6	23.6	5.9	44.0	51.3	43.1	40.4	43.1
TOTAL FEED:	5533.3	5542.3	5254.6	5063.8	5232.2	5382.8	5192.5	5055.6
STREAMS OUT, KGS								
Vent Gas (Dry & N2-Free)	59.3	43.8	67.6	51.3	60.7	51.3	27.7	43.8
Bottoms Flash Gas (Dry & N2-Free)	248.9	282.6	200.8	269.3	219.1	223.3	222.0	230.6
Mix Tank Vent Drain	0.5	0.3	0.0	0.3	0.3	0.3	0.5	0.2
Unit Knockouts	14.2	7.8	6.5	5.3	3.2	4.3	4.0	3.9
Naphtha Stabilizer Bottoms	798.2	927.5	928.2	938.2	1009.5	966.4	947.5	903.6
Atmospheric Still Bottoms	831.4	777.4	860.5	785.9	836.5	878.8	795.7	906.7
Separated Water (Plus Water in Gases)	1529.6	1526.4	1589.9	1152.4	1126.3	1362.5	1421.2	1415.8
Reactor Liquid Flash Vessel Bottoms	2170.9	1951.3	1510.0	1837.5	1936.8	1962.7	1872.9	1572.6
TOTAL PRODUCTS:	5653.0	5517.1	5163.6	5040.3	5192.4	5449.6	5291.3	5077.1
LIQUEFACTION SECTION RECOVERY, W%	102.2	99.5	98.3	99.5	99.2	101.2	101.9	100.4
SOLVENT TO COAL (MF) RATIO	1.67	1.32	1.20	1.22	1.24	1.29	1.31	1.26

Period	09T	10T	11T	12T	13T	14T	15T	16T
VACUUM STILL SECTION MATERIAL BALANCE (Includes Inventory Changes)								
STREAMS IN, KGS								
Feed to Vacuum Still	1148.9							
STREAMS OUT, KGS								
Vacuum Still Overhead Product	373.9	755.9	557.2	473.1	602.3	712.6	606.2	576.7
Vacuum Still Bottoms Product	626.2	1.8	1.5	2.0	1.7	2.1	1.9	2.3
VAC STILL SECTION MATERIAL RECOVERY, W%	87.0							
FEED RATES, KGS/HR								
Feed to Vacuum Still	47.9							
PRODUCT RATES, KGS/HR								
Vacuum Still Overhead Product	15.6	31.5	23.2	19.7	25.1	29.7	25.3	24.0
Vacuum Still Bottoms Product	26.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ROSE UNIT MATERIAL BALANCE (Includes Inventory Changes)								
STREAMS IN, KGS								
Feed to ROSE Unit	0.0	853.2	933.5	983.8	823.7	843.7	666.3	729.4
Makeup Solvent to Rose Unit	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
STREAMS OUT, KGS								
ROSE Unit DAO Product	0.0	289.8	270.3	293.0	351.1	299.4	264.0	223.6
ROSE Unit Residuals	0.0	566.5	738.0	556.7	501.8	547.2	436.8	559.5
ROSE SECTION MATERIAL RECOVERY, W%		100.4	108.0	86.4	103.5	100.3	105.2	107.4
FEED RATES, KGS/HR								
Feed to ROSE Section	0.0	35.6	38.9	41.0	34.3	35.2	27.8	30.4
Makeup Solvent to Rose Unit	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
PRODUCT RATES, KGS/HR								
ROSE Unit DAO Product	0.0	12.1	11.3	12.2	14.6	12.5	11.0	9.3
ROSE Unit Residuals	0.0	23.6	30.7	23.2	20.9	22.8	18.2	23.3

Period	09T	10T	11T	12T	13T	14T	15T	16T
RECYCLE RATES TO SMT, KGS/HR								
Reactor Liq Flash Vessel Bottoms	29.3	26.5	18.5	30.4	24.5	24.3	23.2	18.2
Atmospheric Still Bottoms	33.9	29.3	27.5	19.4	47.1	36.7	18.7	35.6
ROSE Unit DAO	0.0	13.6	11.1	11.9	14.8	6.7	9.7	8.3
Vacuum Still Overheads	16.4	10.7	35.2	11.9	2.8	15.9	26.7	12.2
Vacuum Still Bottoms	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
COMPOSITION OF RECYCLE TO SMT (100% RECYCLE BASIS), W%								
Reactor Liq Flash Vessel Bottoms	36.8	33.1	20.0	41.3	27.5	29.1	29.6	24.5
Atmospheric Still Bottoms	42.6	36.6	29.8	26.4	52.8	43.9	23.8	47.9
ROSE Unit DAO	0.0	17.0	12.1	16.2	16.6	8.0	12.4	11.1
Vacuum Still Overheads	20.6	13.3	38.1	16.2	3.1	19.0	34.2	16.4
Vacuum Still Bottoms	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
NET COLLECTED PRODUCTS, KGS/HR (Includes Samples)								
GASES: C1-C3	6.79	6.20	6.41	6.88	6.55	6.56	6.21	6.55
C4-C7	3.58	4.65	3.15	3.21	3.09	2.94	2.92	3.25
CO & CO2	0.19	0.18	0.22	1.28	0.22	0.20	0.12	0.15
H2S	0.97	1.58	0.21	1.23	0.69	0.77	0.59	0.70
Net Water	16.4	15.1	17.1	13.5	11.8	14.3	12.2	11.2
Mix Tank Vent Drain	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Unit Knockouts	0.6	0.3	0.3	0.2	0.1	0.2	0.2	0.2
Naphtha Stabilizer Bottoms	33.3	38.6	38.7	39.1	42.1	40.3	39.5	37.6
Atmospheric Still Bottoms	0.7	3.1	8.3	13.3	-12.2	-0.1	14.5	2.2
Vacuum Still Overhead (Net)	-9.1	12.3	-20.9	-1.6	12.9	6.3	-12.3	1.1
Vacuum Still Bottoms (Net)	26.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Filter Cake	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ROSE Unit DAO	0.0	-1.6	-0.1	0.2	-0.2	5.8	-0.1	0.0
ROSE Unit Residuals	0.0	23.6	30.7	23.2	20.9	22.8	18.2	23.3
TOTAL:	79.4	104.0	83.9	99.3	85.8	99.8	82.1	86.1

POC-02 (RUN 260-04) MATERIAL BALANCE

COAL: Wyoming Black Thunder Mine (HRI-6209)
CATALYST: Reactors ==> Akzo AO-60 1/16" (HRI-6043)
Hydrotreater ==> Criterion 411 (HRI-6135)

OVERALL MATERIAL BALANCE

Period	17T	18T	19T	20T	21T	22T	23T	24T
Period Start Date	06/21/94	06/22/94	06/23/94	06/24/94	06/25/94	06/26/94	07/03/94	07/04/94
Period Start Time	04:00	04:00	04:00	04:00	04:00	04:00	16:00	04:00
Period Duration Hours	24	24	24	24	24	24	24	24
Solids Separation Type	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR	VAC STIL	VAC STIL	VAC STIL
STREAMS IN, KGS								
Coal Feed Wet (Less Sample)	1743.4	2389.0	2781.3	2834.7	2780.3	2821.5	286.1	1863.2
Make-Up Oil to Mix Tank	0.0	0.0	0.0	0.0	0.0	0.0	311.2	287.6
Mix Tank Inventory Loss	6.4	-45.4	-18.1	6.4	13.2	-63.5	-13.2	-70.3
Seal Oil to Ebullating Pumps	55.9	55.2	55.7	55.8	55.6	55.3	23.8	46.5
Make-Up Oil to Purge Pumps	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Water Injected to O-1	898.7	1156.2	1437.7	1562.8	1548.6	1550.8	329.6	898.1
Fresh Hydrogen Feed	153.5	189.6	221.3	231.3	239.8	239.4	90.0	116.3
DMDS (TNPS)	43.5	53.5	37.2	42.6	40.4	44.5	15.4	41.3
TOTAL FEED:	2901.4	3798.1	4515.1	4733.6	4677.8	4648.0	1042.8	3182.6
STREAMS OUT, KGS								
Vent Gas (Dry & N2-Free)	43.7	49.2	49.5	48.0	59.2	62.6	29.1	51.5
Bottoms Flash Gas (Dry & N2-Free)	169.0	296.8	386.8	360.3	357.9	367.1	68.5	158.1
Mix Tank Vent Drain	0.9	0.8	1.4	1.8	5.1	3.0	1.7	1.0
Unit Knockouts	4.3	6.5	4.6	4.4	4.4	8.7	14.0	156.3
Naphtha Stabilizer Bottoms	894.8	1112.1	1127.3	1126.5	1071.2	1099.3	68.5	449.6
Atmospheric Still Bottoms Product	-46.1	-219.7	-13.2	140.5	106.8	258.7	-584.3	459.6
Separated Water (Plus Water in Gases)	1459.3	1933.4	2229.0	2346.9	2398.2	2399.6	437.6	1413.5
Vacuum Still Overhead Product	-40.0	71.4	158.9	60.5	-32.3	29.8	-89.6	25.1
Vacuum Still Bottoms Product	1.5	1.8	1.2	2.4	0.2	470.6	0.5	538.2
Pressure Filter Cake	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Filter Section Net Inv. Change	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ROSE Unit DAO Product	5.0	-4.1	-1.2	6.4	-25.4	0.0	0.0	0.0
ROSE Unit Bottoms	376.7	381.9	590.1	352.5	172.5	0.0	0.0	0.0
ROSE Section Net Inv. Change	-110.2	4.1	24.0	202.8	467.2	0.0	0.0	0.0
Recycle Oil Net Inv. Change	26.3	15.0	37.2	-42.2	-21.8	150.1	120.2	73.5
Vacuum Still Feed Tank Inv. Change	152.4	48.5	-127.5	31.8	-5.4	-68.5	800.1	-137.0
RLFV Bottoms Holding Tank Inv. Change	-5.0	16.8	-5.9	-1.8	9.1	-39.9	140.2	86.6
TOTAL PRODUCTS:	2932.6	3714.4	4462.2	4640.8	4567.0	4741.2	1006.3	3276.0
OVERALL UNIT MATERIAL RECOVERY, W%	101.1	97.8	98.8	98.0	97.6	102.0	96.5	102.9

LIQUEFACTION SECTION MATERIAL BALANCE (Reactor Section)

Period	17T	18T	19T	20T	21T	22T	23T	24T
STREAMS IN, KGS								
Coal Feed Wet (Less Sample)	1743.4	2389.0	2781.3	2834.7	2780.3	2821.5	286.1	1863.2
Oil Streams to the SMT								
Recycle to SMT	880.0	1133.1	1105.9	1117.2	1015.1	948.9	297.1	1993.1
Make-Up Oil to SMT	0.0	0.0	0.0	0.0	0.0	0.0	311.2	287.6
VSOH recycled to SMT	302.0	236.0	218.3	199.8	235.2	57.7	0.0	319.9
Mix Tank Inventory Loss	6.4	-45.4	-18.1	6.4	13.2	-63.5	-13.2	-70.3
Seal Oil to Ebullating Pumps	55.9	55.2	55.7	55.8	55.6	55.3	23.8	46.5
VSO to Purge Pumps	232.8	225.6	222.2	224.3	210.6	212.6	92.2	211.3
Make Up Oil to Purge Pumps	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Water Injected to O-1	898.7	1156.2	1437.7	1562.8	1548.6	1550.8	329.6	898.1
Fresh Hydrogen Feed	153.5	189.6	221.3	231.3	239.8	239.4	90.0	116.3
DMDS (TNPS)	43.5	53.5	37.2	42.6	40.4	44.5	15.4	41.3
TOTAL FEED:	5095.9	6700.8	7921.9	8101.3	8144.8	7944.5	2230.4	5622.4
STREAMS OUT, KGS								
Vent Gas (Dry & N2-Free)	43.7	49.2	49.5	48.0	59.2	62.6	29.1	51.5
Bottoms Flash Gas (Dry & N2-Free)	169.0	296.8	386.8	360.3	357.9	367.1	68.5	158.1
Mix Tank Vent Drain	0.9	0.8	1.4	1.8	5.1	3.0	1.7	1.0
Unit Knockouts	4.3	6.5	4.6	4.4	4.4	8.7	14.0	156.3
Naphtha Stabilizer Bottoms	894.8	1112.1	1127.3	1126.5	1071.2	1099.3	68.5	449.6
Atmospheric Still Bottoms	1035.7	1324.4	2065.6	2166.7	2348.0	2393.7	214.1	695.0
Separated Water (Plus Water in Gases)	1459.3	1933.4	2229.0	2346.9	2398.2	2399.6	437.6	1413.5
Reactor Liquid Flash Vessel Bottoms	1558.5	2049.8	2104.2	2071.5	1926.4	1641.5	1358.5	2727.4
TOTAL PRODUCTS:	5166.2	6772.9	7968.4	8126.2	8170.5	7975.7	2191.9	5652.4
LIQUEFACTION SECTION RECOVERY, W%	101.4	101.1	100.6	100.3	100.3	100.4	98.3	100.5
SOLVENT TO COAL (MF) RATIO	1.27	1.27	1.29	1.25	1.32	1.23	5.55	1.53

Period	17T	18T	19T	20T	21T	22T	23T	24T
VACUUM STILL SECTION MATERIAL BALANCE (Includes Inventory Changes)								
STREAMS IN, KGS								
Feed to Vacuum Still						794.2	0.9	1180.2
STREAMS OUT, KGS								
Vacuum Still Overhead Product	494.8	533.0	599.4	484.6	413.6	300.0	2.5	556.3
Vacuum Still Bottoms Product	1.5	1.8	1.2	2.4	0.2	470.6	0.5	538.2
VAC STILL SECTION MATERIAL RECOVERY, W%						97.0	284.1	98.8
FEED RATES, KGS/HR								
Feed to Vacuum Still						33.1	0.0	49.2
PRODUCT RATES, KGS/HR								
Vacuum Still Overhead Product	20.6	22.2	25.0	20.2	17.2	12.5	0.1	23.2
Vacuum Still Bottoms Product	0.0	0.0	0.0	0.0	0.0	19.6	0.0	25.4
ROSE UNIT MATERIAL BALANCE (Includes Inventory Changes)								
STREAMS IN, KGS								
Feed to ROSE Unit	547.9	589.7	847.3	528.4	227.2	0.0	0.0	0.0
Makeup Solvent to Rose Unit	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
STREAMS OUT, KGS								
ROSE Unit DAO Product	187.3	244.5	276.5	176.4	50.8	0.0	0.0	0.0
ROSE Unit Residuals	376.7	381.9	590.1	352.5	172.5	0.0	0.0	0.0
ROSE SECTION MATERIAL RECOVERY, W%	102.9	106.2	102.3	100.1	98.3			
FEED RATES, KGS/HR								
Feed to ROSE Section	22.8	24.6	35.3	22.0	9.5	0.0	0.0	0.0
Makeup Solvent to Rose Unit	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
PRODUCT RATES, KGS/HR								
ROSE Unit DAO Product	7.8	10.2	11.5	7.4	2.1	0.0	0.0	0.0
ROSE Unit Residuals	15.7	15.9	24.6	14.7	7.2	0.0	0.0	0.0

Period	17T	18T	19T	20T	21T	22T	23T	24T
RECYCLE RATES TO SMT, KGS/HR								
Reactor Liq Flash Vessel Bottoms	18.4	27.1	28.3	29.8	28.4	43.2	17.4	69.1
Atmospheric Still Bottoms	45.1	64.3	86.6	84.4	93.4	89.0	33.3	9.8
ROSE Unit DAO	7.3	10.7	10.8	6.9	3.2	0.0	0.0	0.0
Vacuum Still Overheads	12.1	10.1	8.5	8.1	9.8	2.6	0.0	13.8
Vacuum Still Bottoms	0.0	0.0	0.0	0.0	0.0	0.0	0.0	3.0
COMPOSITION OF RECYCLE TO SMT (100% RECYCLE BASIS), W%								
Reactor Liq Flash Vessel Bottoms	22.2	24.1	21.1	23.1	21.0	32.0	34.3	72.2
Atmospheric Still Bottoms	54.4	57.4	64.5	65.3	69.3	66.0	65.7	10.2
ROSE Unit DAO	8.8	9.5	8.1	5.3	2.4	0.0	0.0	0.0
Vacuum Still Overheads	14.6	9.0	6.3	6.3	7.3	1.9	0.0	14.5
Vacuum Still Bottoms	0.0	0.0	0.0	0.0	0.0	0.0	0.0	3.1
NET COLLECTED PRODUCTS, KGS/HR (Includes Samples)								
GASES: C1-C3	5.23	8.95	10.28	10.04	10.06	10.24	2.07	4.39
C4-C7	2.15	3.59	5.16	4.28	4.55	4.85	0.64	1.47
CO & CO2	0.11	0.36	0.44	0.44	0.44	0.47	0.16	0.33
H2S	0.43	0.79	1.53	1.42	1.31	1.39	0.74	1.69
Net Water	15.0	21.0	19.7	19.1	22.1	21.9	3.1	12.6
Mix Tank Vent Drain	0.0	0.0	0.1	0.1	0.2	0.1	0.1	0.0
Unit Knockouts	0.2	0.3	0.2	0.2	0.2	0.4	0.6	6.5
Naphtha Stabilizer Bottoms	37.3	46.3	47.0	46.9	44.6	45.8	2.9	18.7
Atmospheric Still Bottoms	-1.9	-9.2	-0.6	5.9	4.4	10.8	-24.3	19.1
Vacuum Still Overhead (Net)	-1.7	3.0	6.6	2.5	-1.3	1.2	-3.7	1.0
Vacuum Still Bottoms (Net)	0.1	0.1	0.1	0.1	0.0	19.6	0.0	22.4
Filter Cake	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ROSE Unit DAO	0.2	-0.2	-0.1	0.3	-1.1	0.0	0.0	0.0
ROSE Unit Residuals	15.7	15.9	24.6	14.7	7.2	0.0	0.0	0.0
TOTAL:	72.7	90.6	114.5	105.5	92.3	116.3	-18.0	88.0

POC-02 (RUN 260-05) MATERIAL BALANCE

COAL: Wyoming Black Thunder Mine (HRI-6209)
CATALYST: Reactors ==> Akzo AO-60 1/16" (HRI-6043)
Hydrotreater ==> Criterion 411 (HRI-6135)

OVERALL MATERIAL BALANCE

Period	25T	26T	27T	28T	29T	30T	31T	32T
Period Start Date	07/05/94	07/06/94	07/09/94	07/10/94	07/11/94	07/12/94	07/13/94	07/14/94
Period Start Time	04:00	04:00	04:00	04:00	04:00	04:00	04:00	04:00
Period Duration Hours	12	24	24	24	24	24	24	24
Solids Separation Type	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR
STREAMS IN, KGS								
Coal Feed Wet (Less Sample)	992.9	1158.4	2127.5	2860.5	3128.0	1961.8	2722.2	3582.7
Make-Up Oil to Mix Tank	0.0	520.7	0.0	0.0	0.0	547.0	483.1	262.2
Mix Tank Inventory Loss	16.3	30.8	-51.7	-0.9	5.9	34.9	-106.1	6.4
Seal Oil to Ebullating Pumps	22.3	48.1	49.8	51.0	52.6	50.9	49.4	54.7
Make-Up Oil to Purge Pumps	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Water Injected to O-1	455.0	802.1	996.9	1509.1	1636.1	1692.8	1715.1	1635.5
Fresh Hydrogen Feed	95.9	115.6	175.2	231.0	261.5	212.1	232.9	290.5
DMDS (TNPS)	15.9	18.6	24.5	37.6	43.5	23.6	29.5	44.5
TOTAL FEED:	1598.2	2694.3	3322.2	4688.3	5127.6	4523.1	5126.1	5876.4
STREAMS OUT, KGS								
Vent Gas (Dry & N2-Free)	53.6	71.6	61.6	58.3	61.6	92.6	50.1	49.5
Bottoms Flash Gas (Dry & N2-Free)	105.9	191.1	255.0	328.9	348.9	292.6	303.3	441.5
Mix Tank Vent Drain	0.3	7.6	0.5	0.3	0.1	0.5	0.4	4.8
Unit Knockouts	7.5	18.7	11.4	15.9	26.2	16.7	21.8	96.8
Naphtha Stabilizer Bottoms	236.7	251.6	822.1	1271.3	1268.1	1057.0	1346.2	1452.1
Atmospheric Still Bottoms Product	404.6	-408.2	572.7	302.0	-126.3	-480.7	463.3	-532.5
Separated Water (Plus Water in Gases)	829.5	1062.3	1592.5	2361.5	2937.3	2290.6	2577.8	3072.6
Vacuum Still Overhead Product	-53.1	259.3	-105.3	-209.1	118.5	175.1	227.0	166.9
Vacuum Still Bottoms Product	0.0	2.0	2.3	3.0	2.7	2.3	2.5	1.9
Pressure Filter Cake	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Filter Section Net Inv. Change	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ROSE Unit DAO Product	-35.8	9.5	-10.4	11.8	-2.3	15.0	15.9	-44.0
ROSE Unit Bottoms	247.2	481.8	363.5	439.8	430.8	457.2	409.4	434.9
ROSE Section Net Inv. Change	24.5	129.3	-98.4	-26.8	137.4	94.8	21.3	174.2
Recycle Oil Net Inv. Change	-81.6	136.5	25.4	-100.2	7.3	-6.8	-99.3	1.4
Vacuum Still Feed Tank Inv. Change	-146.5	215.5	-99.3	223.6	95.7	161.9	-200.5	157.4
RLFV Bottoms Holding Tank Inv. Change	-57.2	315.7	-85.3	13.2	2.3	291.7	-159.7	152.4
TOTAL PRODUCTS:	1535.5	2744.3	3308.2	4693.3	5308.2	4460.6	4979.4	5629.6
OVERALL UNIT MATERIAL RECOVERY, W%	96.1	101.9	99.6	100.1	103.5	98.6	97.1	95.8

LIQUEFACTION SECTION MATERIAL BALANCE (Reactor Section)

Period	25T	26T	27T	28T	29T	30T	31T	32T
STREAMS IN, KGS								
Coal Feed Wet (Less Sample)	992.9	1158.4	2127.5	2860.5	3128.0	1961.8	2722.2	3582.7
Oil Streams to the SMT								
Recycle to SMT	938.9	1925.5	2109.2	1664.2	1355.8	1187.5	1734.1	1626.6
Make-Up Oil to SMT	0.0	520.7	0.0	0.0	0.0	547.0	483.1	262.2
VSOH recycled to SMT	239.2	585.0	545.4	501.0	368.0	485.3	552.9	536.2
Mix Tank Inventory Loss	16.3	30.8	-51.7	-0.9	5.9	34.9	-106.1	6.4
Seal Oil to Ebullating Pumps	22.3	48.1	49.8	51.0	52.6	50.9	49.4	54.7
VSO to Purge Pumps	106.9	197.9	192.1	182.1	176.3	171.9	176.4	154.6
Make Up Oil to Purge Pumps	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Water Injected to O-1	455.0	802.1	996.9	1509.1	1636.1	1692.8	1715.1	1635.5
Fresh Hydrogen Feed	95.9	115.6	175.2	231.0	261.5	212.1	232.9	290.5
DMDS (TNPS)	15.9	18.6	24.5	37.6	43.5	23.6	29.5	44.5
TOTAL FEED:	2752.9	5747.1	6228.1	7922.6	8662.3	7861.5	7861.7	10367.3
STREAMS OUT, KGS								
Vent Gas (Dry & N2-Free)	53.6	71.6	61.6	58.3	61.6	92.6	50.1	49.5
Bottoms Flash Gas (Dry & N2-Free)	105.9	191.1	255.0	328.9	348.9	292.6	303.3	441.5
Mix Tank Vent Drain	0.3	7.6	0.5	0.3	0.1	0.5	0.4	4.8
Unit Knockouts	7.5	18.7	11.4	15.9	26.2	16.7	21.8	96.8
Naphtha Stabilizer Bottoms	236.7	251.6	822.1	1271.3	1268.1	1057.0	1346.2	1452.1
Atmospheric Still Bottoms	513.5	521.2	1177.3	1690.0	1876.3	1498.3	1288.4	2177.3
Separated Water (Plus Water in Gases)	829.5	1062.3	1592.5	2361.5	2937.3	2290.6	2577.8	3072.6
Reactor Liquid Flash Vessel Bottoms	924.0	3539.8	2314.7	2322.4	2392.7	2688.0	2243.9	3035.4
TOTAL PRODUCTS:	2670.9	5664.0	6235.2	8048.5	8911.1	7936.4	7831.8	10329.9
LIQUEFACTION SECTION RECOVERY, W%	97.0	98.6	100.1	101.6	102.9	101.0	99.6	99.6
SOLVENT TO COAL (MF) RATIO	1.19	3.29	1.44	1.21	1.21	2.14	1.26	1.45

Period	25T	26T	27T	28T	29T	30T	31T	32T
VACUUM STILL SECTION MATERIAL BALANCE (Includes Inventory Changes)								
STREAMS IN, KGS								
Feed to Vacuum Still								
STREAMS OUT, KGS								
Vacuum Still Overhead Product	293.0	1042.2	632.2	474.0	662.8	832.3	956.3	857.7
Vacuum Still Bottoms Product	0.0	2.0	2.3	3.0	2.7	2.3	2.5	1.9
VAC STILL SECTION MATERIAL RECOVERY, W%								
FEED RATES, KGS/HR								
Feed to Vacuum Still								
PRODUCT RATES, KGS/HR								
Vacuum Still Overhead Product	24.4	43.4	26.3	19.7	27.6	34.7	39.8	35.7
Vacuum Still Bottoms Product	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ROSE UNIT MATERIAL BALANCE (Includes Inventory Changes)								
STREAMS IN, KGS								
Feed to ROSE Unit	376.0	810.1	738.0	801.0	774.7	660.9	838.7	878.6
Makeup Solvent to Rose Unit	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
STREAMS OUT, KGS								
ROSE Unit DAO Product	111.1	367.9	403.2	412.8	324.3	236.8	371.9	445.9
ROSE Unit Residuals	247.2	481.8	363.5	439.8	430.8	457.2	409.4	434.9
ROSE SECTION MATERIAL RECOVERY, W%	95.3	104.9	103.9	106.4	97.5	105.0	93.2	100.2
FEED RATES, KGS/HR								
Feed to ROSE Section	31.3	33.8	30.7	33.4	32.3	27.5	34.9	36.6
Makeup Solvent to Rose Unit	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
PRODUCT RATES, KGS/HR								
ROSE Unit DAO Product	9.3	15.3	16.8	17.2	13.5	9.9	15.5	18.6
ROSE Unit Residuals	20.6	20.1	15.1	18.3	17.9	19.1	17.1	18.1

Period	25T	26T	27T	28T	29T	30T	31T	32T
RECYCLE RATES TO SMT, KGS/HR								
Reactor Liq Flash Vessel Bottoms	40.0	46.4	48.7	28.9	26.2	19.5	30.2	23.7
Atmospheric Still Bottoms	9.1	38.7	25.2	57.8	83.4	82.5	34.4	112.9
ROSE Unit DAO	12.0	15.0	17.4	16.1	14.4	9.3	14.8	21.1
Vacuum Still Overheads	19.5	24.5	22.9	20.2	16.2	20.4	23.0	23.1
Vacuum Still Bottoms	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
COMPOSITION OF RECYCLE TO SMT (100% RECYCLE BASIS), W%								
Reactor Liq Flash Vessel Bottoms	49.7	37.2	42.6	23.5	18.7	14.8	29.5	13.1
Atmospheric Still Bottoms	11.3	31.1	22.1	47.0	59.5	62.6	33.5	62.5
ROSE Unit DAO	14.9	12.0	15.2	13.1	10.3	7.1	14.5	11.7
Vacuum Still Overheads	24.2	19.7	20.1	16.4	11.6	15.5	22.5	12.8
Vacuum Still Bottoms	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
NET COLLECTED PRODUCTS, KGS/HR (Includes Samples)								
GASES: C1-C3	7.69	5.92	8.14	9.23	11.19	9.43	8.42	10.49
C4-C7	3.44	2.54	3.39	4.21	3.16	3.58	3.74	4.61
CO & CO2	0.26	0.25	0.21	0.47	0.64	0.69	0.44	1.38
H2S	1.15	1.42	0.73	1.13	1.07	1.17	1.17	3.00
Net Water	21.7	5.3	14.7	21.8	39.3	15.5	22.9	42.8
Mix Tank Vent Drain	0.0	0.3	0.0	0.0	0.0	0.0	0.0	0.2
Unit Knockouts	0.6	0.8	0.5	0.7	1.1	0.7	0.9	4.0
Naphtha Stabilizer Bottoms	19.7	10.5	34.3	53.0	52.8	44.0	56.1	60.5
Atmospheric Still Bottoms	33.7	-17.0	23.9	12.6	-5.3	-20.0	19.3	-22.2
Vacuum Still Overhead (Net)	-4.4	10.8	-4.4	-8.7	4.9	7.3	9.5	7.0
Vacuum Still Bottoms (Net)	0.0	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Filter Cake	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ROSE Unit DAO	-3.0	0.4	-0.4	0.5	-0.1	0.6	0.7	-1.8
ROSE Unit Residuals	20.6	20.1	15.1	18.3	17.9	19.1	17.1	18.1
TOTAL:	101.3	41.1	95.9	112.9	126.3	81.5	139.9	126.7

POC-02 (RUN 260-05) MATERIAL BALANCE

COAL: Wyoming Black Thunder Mine (HRI-6209)
CATALYST: Reactors ==> Akzo AO-60 1/16" (HRI-6043)
Hydrotreater ==> Criterion 411 (HRI-6135)

OVERALL MATERIAL BALANCE

Period	33T	34T	35T	36T	37T	38T
Period Start Date	07/15/94	07/16/94	07/17/94	07/18/94	07/19/94	07/20/94
Period Start Time	04:00	04:00	04:00	04:00	04:00	04:00
Period Duration Hours	24	24	24	24	24	24
Solids Separation Type	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR
STREAMS IN, KGS						
Coal Feed Wet (Less Sample)	3705.2	3729.3	3800.4	3702.6	2888.7	1204.3
Make-Up Oil to Mix Tank	638.7	332.0	0.0	0.0	756.6	0.0
Mix Tank Inventory Loss	57.6	-13.6	11.8	18.6	-30.4	461.3
Seal Oil to Ebullating Pumps	55.9	55.9	55.2	56.8	52.3	53.0
Make-Up Oil to Purge Pumps	0.0	0.0	0.0	0.0	0.0	0.0
Water Injected to O-1	1743.5	1674.8	1475.5	1767.2	1625.7	1547.2
Fresh Hydrogen Feed	296.8	296.3	302.7	303.2	282.9	205.5
DMDS (TNPS)	49.4	47.6	49.9	51.7	39.0	35.2
TOTAL FEED:	6547.1	6122.3	5695.4	5900.1	5614.8	3506.4
STREAMS OUT, KGS						
Vent Gas (Dry & N2-Free)	58.3	79.2	57.8	64.1	83.6	70.3
Bottoms Flash Gas (Dry & N2-Free)	377.4	431.2	519.4	559.6	303.1	237.6
Mix Tank Vent Drain	4.3	2.0	5.8	4.4	9.4	5.5
Unit Knockouts	10.4	55.8	17.2	21.9	23.6	14.0
Naphtha Stabilizer Bottoms	1280.3	1557.1	1555.6	1318.5	1284.3	1018.1
Atmospheric Still Bottoms Product	159.6	-152.1	-90.1	131.2	-261.0	-200.5
Separated Water (Plus Water in Gases)	3338.8	2812.4	2481.8	2953.4	2520.6	2143.6
Vacuum Still Overhead Product	202.6	265.1	127.7	156.5	206.3	231.0
Vacuum Still Bottoms Product	1.7	2.1	2.3	3.1	3.4	2.8
Pressure Filter Cake	0.0	0.0	0.0	0.0	0.0	0.0
Filter Section Net Inv. Change	0.0	0.0	0.0	0.0	0.0	0.0
ROSE Unit DAO Product	32.7	7.3	0.0	5.0	-23.6	19.1
ROSE Unit Bottoms	550.9	485.9	555.0	572.9	618.2	568.3
ROSE Section Net Inv. Change	494.0	496.7	256.7	308.4	45.8	137.0
Recycle Oil Net Inv. Change	33.6	19.1	20.4	-14.1	310.3	281.7
Vacuum Still Feed Tank Inv. Change	5.4	-171.9	54.0	-98.9	232.7	-222.7
RLFV Bottoms Holding Tank Inv. Change	-106.6	-37.2	-22.2	-5.0	5.4	-63.0
TOTAL PRODUCTS:	6443.3	5852.6	5541.3	5981.0	5362.1	4242.6
OVERALL UNIT MATERIAL RECOVERY, W%	98.4	95.6	97.3	101.4	95.5	121.0

LIQUEFACTION SECTION MATERIAL BALANCE (Reactor Section)

Period	33T	34T	35T	36T	37T	38T
STREAMS IN, KGS						
Coal Feed Wet (Less Sample)	3705.2	3729.3	3800.4	3702.6	2888.7	1204.3
Oil Streams to the SMT						
Recycle to SMT	1521.3	1638.8	1661.0	1594.4	879.5	1217.9
Make-Up Oil to SMT	638.7	332.0	0.0	0.0	756.6	0.0
VSOH recycled to SMT	451.3	496.2	483.1	398.3	390.1	544.3
Mix Tank Inventory Loss	57.6	-13.6	11.8	18.6	-30.4	461.3
Seal Oil to Ebullating Pumps	55.9	55.9	55.2	56.8	52.3	53.0
VSO to Purge Pumps	171.1	198.4	220.0	226.2	210.5	196.9
Make Up Oil to Purge Pumps	0.0	0.0	0.0	0.0	0.0	0.0
Water Injected to O-1	1743.5	1674.8	1475.5	1767.2	1625.7	1547.2
Fresh Hydrogen Feed	296.8	296.3	302.7	303.2	282.9	205.5
DMDS (TNPS)	49.4	47.6	49.9	51.7	39.0	35.2
TOTAL FEED:	10499.3	10302.3	10144.7	10375.9	10154.3	7309.3
STREAMS OUT, KGS						
Vent Gas (Dry & N2-Free)	58.3	79.2	57.8	64.1	83.6	70.3
Bottoms Flash Gas (Dry & N2-Free)	377.4	431.2	519.4	559.6	303.1	237.6
Mix Tank Vent Drain	4.3	2.0	5.8	4.4	9.4	5.5
Unit Knockouts	10.4	55.8	17.2	21.9	23.6	14.0
Naphtha Stabilizer Bottoms	1280.3	1557.1	1555.6	1318.5	1284.3	1018.1
Atmospheric Still Bottoms	2419.4	2190.7	2478.1	2786.5	3188.6	2187.6
Separated Water (Plus Water in Gases)	3338.8	2812.4	2481.8	2953.4	2520.6	2143.6
Reactor Liquid Flash Vessel Bottoms	2931.1	3060.8	2926.1	2790.0	2546.5	2488.4
TOTAL PRODUCTS:	10420.0	10189.2	10041.8	10498.3	9959.6	8165.1
LIQUEFACTION SECTION RECOVERY, W%	99.2	98.9	99.0	101.2	98.1	111.7
SOLVENT TO COAL (MF) RATIO	1.35	1.31	1.26	1.30	1.99	3.38

Period	33T	34T	35T	36T	37T	38T
VACUUM STILL SECTION MATERIAL BALANCE (Includes Inventory Changes)						
STREAMS IN, KGS						
Feed to Vacuum Still						
STREAMS OUT, KGS						
Vacuum Still Overhead Product	825.0	959.8	830.7	781.0	806.8	972.2
Vacuum Still Bottoms Product	1.7	2.1	2.3	3.1	3.4	2.8
VAC STILL SECTION MATERIAL RECOVERY, W%						
FEED RATES, KGS/HR						
Feed to Vacuum Still						
PRODUCT RATES, KGS/HR						
Vacuum Still Overhead Product	34.4	40.0	34.6	32.5	33.6	40.5
Vacuum Still Bottoms Product	0.0	0.0	0.0	0.0	0.0	0.0
ROSE UNIT MATERIAL BALANCE (Includes Inventory Changes)						
STREAMS IN, KGS						
Feed to ROSE Unit	1059.6	914.4	1085.9	1103.1	1108.1	1047.3
Makeup Solvent to Rose Unit	0.0	0.0	0.0	0.0	0.0	0.0
STREAMS OUT, KGS						
ROSE Unit DAO Product	477.2	505.8	525.7	520.3	496.2	535.7
ROSE Unit Residuals	550.9	485.9	555.0	572.9	618.2	568.3
ROSE SECTION MATERIAL RECOVERY, W%	97.0	108.4	99.5	99.1	100.6	105.4
FEED RATES, KGS/HR						
Feed to ROSE Section	44.1	38.1	45.2	46.0	46.2	43.6
Makeup Solvent to Rose Unit	0.0	0.0	0.0	0.0	0.0	0.0
PRODUCT RATES, KGS/HR						
ROSE Unit DAO Product	19.9	21.1	21.9	21.7	20.7	22.3
ROSE Unit Residuals	23.0	20.2	23.1	23.9	25.8	23.7

Period	33T	34T	35T	36T	37T	38T
RECYCLE RATES TO SMT, KGS/HR						
Reactor Liq Flash Vessel Bottoms	27.4	27.6	28.0	27.8	11.7	18.3
Atmospheric Still Bottoms	94.2	97.6	107.0	110.6	143.7	99.5
ROSE Unit DAO	18.5	20.8	21.9	21.5	21.7	21.5
Vacuum Still Overheads	18.8	20.7	20.1	16.6	16.3	22.7
Vacuum Still Bottoms	0.0	0.0	0.0	0.0	0.0	0.0
COMPOSITION OF RECYCLE TO SMT (100% RECYCLE BASIS), W%						
Reactor Liq Flash Vessel Bottoms	17.2	16.6	15.8	15.7	6.0	11.3
Atmospheric Still Bottoms	59.2	58.6	60.4	62.7	74.4	61.4
ROSE Unit DAO	11.7	12.5	12.4	12.2	11.2	13.3
Vacuum Still Overheads	11.8	12.4	11.4	9.4	8.4	14.0
Vacuum Still Bottoms	0.0	0.0	0.0	0.0	0.0	0.0
NET COLLECTED PRODUCTS, KGS/HR (Includes Samples)						
GASES: C1-C3	11.62	11.95	14.04	13.62	8.84	6.77
C4-C7	2.98	4.04	5.87	5.79	3.65	3.41
CO & CO2	1.29	3.08	1.52	4.20	0.68	0.43
H2S	1.32	1.30	1.89	1.54	1.30	1.03
Net Water	48.8	29.6	23.8	31.7	23.5	19.1
Mix Tank Vent Drain	0.2	0.1	0.2	0.2	0.4	0.2
Unit Knockouts	0.4	2.3	0.7	0.9	1.0	0.6
Naphtha Stabilizer Bottoms	53.3	64.9	64.8	54.9	53.5	42.4
Atmospheric Still Bottoms	6.7	-6.3	-3.8	5.5	-10.9	-8.4
Vacuum Still Overhead (Net)	8.4	11.0	5.3	6.5	8.6	9.6
Vacuum Still Bottoms (Net)	0.1	0.1	0.1	0.1	0.1	0.1
Filter Cake	0.0	0.0	0.0	0.0	0.0	0.0
ROSE Unit DAO	1.4	0.3	0.0	0.2	-1.0	0.8
ROSE Unit Residuals	23.0	20.2	23.1	23.9	25.8	23.7
TOTAL:	158.1	139.5	136.1	144.9	114.8	99.4

POC-02 (RUN 260-05) MATERIAL BALANCE

COAL: Wyoming Black Thunder Mine (HRI-6209)
CATALYST: Reactors ==> Akzo AO-60 1/16" (HRI-6043)
Hydrotreater ==> Criterion 411 (HRI-6135)

OVERALL MATERIAL BALANCE

Period	39T	40T	41T	42T	43T	44T	45T
Period Start Date	07/22/94	07/22/94	07/23/94	07/24/94	07/25/94	07/26/94	07/27/94
Period Start Time	04:00	04:00	04:00	04:00	04:00	04:00	04:00
Period Duration Hours	24	24	24	24	24	24	24
Solids Separation Type	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR	ROSE-SR
STREAMS IN, KGS							
Coal Feed Wet & Waste Feed (Less Sample)	1947.0	2113.4	2416.6	2392.1	2663.3	2135.0	2432.4
Make-Up Oil to Mix Tank	567.0	1315.9	839.1	958.9	1409.8	1293.6	2243.5
Mix Tank Inventory Loss	7.3	54.4	-110.7	88.0	10.4	-28.1	81.2
Seal Oil to Ebullating Pumps	54.8	53.6	55.6	58.0	58.2	59.6	57.4
Make-Up Oil to Purge Pumps	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Water Injected to O-1	1604.2	1520.0	1713.2	1745.5	1697.3	1714.6	1710.5
Fresh Hydrogen Feed	232.1	233.7	243.1	239.6	244.8	224.5	246.0
DMDS (TNPS)	36.3	35.8	38.1	37.6	38.1	37.6	35.4
TOTAL FEED:	4448.7	5326.9	5195.1	5519.7	6121.9	5436.8	6806.3
STREAMS OUT, KGS							
Vent Gas (Dry & N2-Free)	61.9	79.4	81.8	70.9	52.7	78.9	70.1
Bottoms Flash Gas (Dry & N2-Free)	223.6	421.9	378.8	467.0	357.3	435.5	384.9
Mix Tank Vent Drain	1.3	2.5	7.0	6.0	2.9	1.2	0.2
Unit Knockouts	15.5	28.1	20.8	48.0	29.8	60.1	4.1
Naphtha Stabilizer Bottoms	1434.0	1778.7	1862.9	1821.1	1646.0	1530.9	1697.1
Atmospheric Still Bottoms Product	-819.3	-171.6	-16.4	-66.0	-89.0	-49.9	297.5
Separated Water (Plus Water in Gases)	2140.4	2084.7	2384.6	2351.3	2411.8	2290.5	2652.7
Vacuum Still Overhead Product	-515.5	448.0	-922.9	-769.9	471.3	-32.8	274.8
Vacuum Still Bottoms Product	2.3	2.8	2.0	4.4	1.7	2.1	1.7
Pressure Filter Cake	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Filter Section Net Inv. Change	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ROSE Unit DAO Product	49.0	47.6	-61.2	-46.3	-34.9	139.7	-47.6
ROSE Unit Bottoms	412.4	429.7	692.0	703.0	707.7	768.3	485.7
ROSE Section Net Inv. Change	81.2	574.2	598.3	400.1	613.7	520.3	591.9
Recycle Oil Net Inv. Change	137.0	49.4	162.4	68.5	-23.6	-153.3	-30.4
Vacuum Still Feed Tank Inv. Change	386.9	-32.7	-554.3	445.4	87.1	-203.7	143.8
RLFV Bottoms Holding Tank Inv. Change	650.4	-819.2	547.0	-104.3	-92.1	-159.7	198.7
TOTAL PRODUCTS:	4261.1	4923.7	5182.9	5399.1	6142.4	5228.1	6725.3
OVERALL UNIT MATERIAL RECOVERY, W%	95.8	92.4	99.8	97.8	100.3	96.2	98.8

LIQUEFACTION SECTION MATERIAL BALANCE (Reactor Section)

Period	39T	40T	41T	42T	43T	44T	45T
STREAMS IN, KGS							
Coal Feed Wet & Waste Feed (Less Sample)	1947.0	2113.4	2416.6	2392.1	2663.3	2135.0	2432.4
Oil Streams to the SMT							
Recycle to SMT	1709.1	1713.2	2652.6	2483.0	1557.6	2129.2	1716.4
Make-Up Oil to SMT	567.0	1315.9	839.1	958.9	1409.8	1293.6	2243.5
VSOH recycled to SMT	1228.8	1126.3	2281.6	1993.1	832.3	1482.8	707.1
Mix Tank Inventory Loss	7.3	54.4	-110.7	88.0	10.4	-28.1	81.2
Seal Oil to Ebullating Pumps	54.8	53.6	55.6	58.0	58.2	59.6	57.4
VSO to Purge Pumps	223.9	200.5	223.8	223.2	204.8	265.2	264.7
Make Up Oil to Purge Pumps	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Water Injected to O-1	1604.2	1520.0	1713.2	1745.5	1697.3	1714.6	1710.5
Fresh Hydrogen Feed	232.1	233.7	243.1	239.6	244.8	224.5	246.0
DMDS (TNPS)	36.3	35.8	38.1	37.6	38.1	37.6	35.4
TOTAL FEED:	9000.7	8678.0	9754.3	9561.2	10192.7	9692.7	10095.1
STREAMS OUT, KGS							
Vent Gas (Dry & N2-Free)	61.9	79.4	81.8	70.9	52.7	78.9	70.1
Bottoms Flash Gas (Dry & N2-Free)	223.6	421.9	378.8	467.0	357.3	435.5	384.9
Mix Tank Vent Drain	1.3	2.5	7.0	6.0	2.9	1.2	0.2
Unit Knockouts	15.5	28.1	20.8	48.0	29.8	60.1	4.1
Naphtha Stabilizer Bottoms	1434.0	1778.7	1862.9	1821.1	1646.0	1530.9	1697.1
Atmospheric Still Bottoms	1799.7	1265.8	1666.5	1269.3	2219.3	1811.6	1605.2
Separated Water (Plus Water in Gases)	2140.4	2084.7	2384.6	2351.3	2411.8	2290.5	2652.7
Reactor Liquid Flash Vessel Bottoms	3066.7	2702.5	3141.6	3246.3	3243.2	3373.3	3648.2
TOTAL PRODUCTS:	8743.2	8363.7	9543.9	9279.9	9963.0	9582.1	10062.6
LIQUEFACTION SECTION RECOVERY, W%	97.1	96.4	97.8	97.1	97.7	98.9	99.7
SOLVENT TO COAL (MF) RATIO	2.84	2.39	2.42	2.26	2.24	2.80	2.45

Period	39T	40T	41T	42T	43T	44T	45T
VACUUM STILL SECTION MATERIAL BALANCE (Includes Inventory Changes)							
STREAMS IN, KGS							
Feed to Vacuum Still							
STREAMS OUT, KGS							
Vacuum Still Overhead Product	937.2	1774.7	1582.4	1446.4	1508.4	1715.2	1246.6
Vacuum Still Bottoms Product	2.3	2.8	2.0	4.4	1.7	2.1	1.7
VAC STILL SECTION MATERIAL RECOVERY, W%							
FEED RATES, KGS/HR							
Feed to Vacuum Still							
PRODUCT RATES, KGS/HR							
Vacuum Still Overhead Product	39.1	73.9	65.9	60.3	62.9	71.5	51.9
Vacuum Still Bottoms Product	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ROSE UNIT MATERIAL BALANCE (Includes Inventory Changes)							
STREAMS IN, KGS							
Feed to ROSE Unit	983.8	1020.6	1078.6	1144.9	1302.3	1354.9	1360.3
Makeup Solvent to Rose Unit	0.0	0.0	0.0	0.0	0.0	0.0	0.0
STREAMS OUT, KGS							
ROSE Unit DAO Product	607.4	684.0	476.3	512.1	666.8	632.8	931.2
ROSE Unit Residuals	412.4	429.7	692.0	703.0	707.7	768.3	485.7
ROSE SECTION MATERIAL RECOVERY, W%	103.6	109.1	108.3	106.1	105.5	103.4	104.2
FEED RATES, KGS/HR							
Feed to ROSE Section	41.0	42.5	44.9	47.7	54.3	56.5	56.7
Makeup Solvent to Rose Unit	0.0	0.0	0.0	0.0	0.0	0.0	0.0
PRODUCT RATES, KGS/HR							
ROSE Unit DAO Product	25.3	28.5	19.8	21.3	27.8	26.4	38.8
ROSE Unit Residuals	17.2	17.9	28.8	29.3	29.5	32.0	20.2

Period	39T	40T	41T	42T	43T	44T	45T
RECYCLE RATES TO SMT, KGS/HR							
Reactor Liq Flash Vessel Bottoms	2.5	0.0	0.0	0.0	0.0	0.0	0.0
Atmospheric Still Bottoms	109.1	59.9	70.1	55.6	96.2	77.6	54.5
ROSE Unit DAO	23.3	26.5	22.4	23.3	29.2	20.5	40.8
Vacuum Still Overheads	51.2	46.9	94.9	83.0	34.7	61.8	29.5
Vacuum Still Bottoms	0.0	0.0	0.0	0.0	0.0	0.0	0.0
COMPOSITION OF RECYCLE TO SMT (100% RECYCLE BASIS), W%							
Reactor Liq Flash Vessel Bottoms	1.3	0.0	0.0	0.0	0.0	0.0	0.0
Atmospheric Still Bottoms	58.7	44.9	37.4	34.4	60.1	48.5	43.7
ROSE Unit DAO	12.5	19.9	11.9	14.4	18.3	12.8	32.7
Vacuum Still Overheads	27.5	35.2	50.7	51.3	21.7	38.6	23.6
Vacuum Still Bottoms	0.0	0.0	0.0	0.0	0.0	0.0	0.0
NET COLLECTED PRODUCTS, KGS/HR (Includes Samples)							
GASES: C1-C3	5.81	10.79	9.24	10.97	9.04	10.21	9.79
C4-C7	2.54	5.15	4.44	5.64	3.45	6.41	5.14
CO & CO2	0.47	1.01	0.38	0.36	0.82	0.45	0.66
H2S	1.58	2.86	3.39	4.02	2.36	3.21	2.12
Net Water	13.0	13.4	16.4	13.8	17.0	13.8	27.6
Mix Tank Vent Drain	0.1	0.1	0.3	0.2	0.1	0.1	0.0
Unit Knockouts	0.6	1.2	0.9	2.0	1.2	2.5	0.2
Naphtha Stabilizer Bottoms	59.7	74.1	77.6	75.9	68.6	63.8	70.7
Atmospheric Still Bottoms	-34.1	-7.2	-0.7	-2.8	-3.7	-2.1	12.4
Vacuum Still Overhead (Net)	-21.5	18.7	-38.5	-32.1	19.6	-1.4	11.4
Vacuum Still Bottoms (Net)	0.1	0.1	0.1	0.2	0.1	0.1	0.1
Filter Cake	0.0	0.0	0.0	0.0	0.0	0.0	0.0
ROSE Unit DAO	2.0	2.0	-2.6	-1.9	-1.5	5.8	-2.0
ROSE Unit Residuals	17.2	17.9	28.8	29.3	29.5	32.0	20.2
TOTAL:	47.1	139.1	99.5	105.3	145.9	134.4	157.8

RUN 260-05 PRODUCT ANALYSES

GAS ANALYSIS

Period	01T	02T	03T	04T	05T	06T	07T	08T
Period Start Date	06/01/94	06/02/94	06/03/94	06/04/94	06/05/94	06/06/94	06/11/94	06/12/94
VENT GAS, VOL% (N2, O2 Free Basis)								
H2	93.19	78.31	87.53	77.70	81.88	81.88	85.85	81.59
CH4	5.45	18.94	10.78	19.54	15.91	15.91	12.34	16.08
C2H4	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C2H6	0.70	1.13	0.82	1.14	0.88	0.88	0.81	1.33
C3H6	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C3H8	0.19	0.34	0.28	0.33	0.28	0.28	0.27	0.45
C4H8	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
N-C4H10	0.05	0.06	0.06	0.04	0.05	0.05	0.05	0.08
I-C4H10	0.02	0.02	0.01	0.01	0.01	0.01	0.01	0.02
C5H10	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00
N-C5H12	0.04	0.02	0.04	0.00	0.02	0.02	0.02	0.02
I-C5H12	0.00	0.01	0.00	0.00	0.00	0.00	0.00	0.00
Methylcyclopentanes	0.00	0.03	0.00	0.01	0.00	0.00	0.00	0.00
Cyclohexane	0.01	0.00	0.00	0.00	0.01	0.01	0.01	0.01
N-Hexane	0.00	0.02	0.00	0.00	0.00	0.00	0.00	0.00
2-3-Methyl Pentane	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00
Other C6's and C7's	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CO	0.00	0.17	0.13	0.14	0.14	0.14	0.15	0.11
CO2	0.00	0.19	0.13	0.41	0.40	0.40	0.24	0.14
H2S	0.35	0.75	0.21	0.68	0.43	0.43	0.22	0.16
BOTTOMS FLASH GAS, VOL% (N2, O2 Free Basis)								
H2	58.75	13.93	28.01	15.44	25.30	25.30	33.31	15.54
CH4	10.84	33.67	29.83	35.03	32.93	32.93	29.56	33.77
C2H4	0.01	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C2H6	6.51	10.46	11.76	10.21	8.54	8.54	9.08	14.23
C3H6	0.03	0.01	0.01	0.01	0.02	0.02	0.01	0.00
C3H8	5.30	9.49	10.70	9.26	7.82	7.82	8.34	13.23
C4H8	0.00	0.00	0.00	0.00	0.13	0.13	0.06	0.01
N-C4H10	2.86	4.24	4.16	3.75	3.27	3.27	3.05	6.02
I-C4H10	0.86	1.09	1.04	1.06	0.82	0.82	0.76	1.36
C5H10	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
N-C5H12	0.48	1.85	1.37	1.48	1.65	1.65	1.05	2.22
I-C5H12	0.49	1.49	1.09	0.98	0.93	0.93	0.69	1.36
Methylcyclopentanes	0.08	0.22	0.20	0.23	0.29	0.29	0.15	0.37
Cyclohexane	0.33	0.74	0.71	0.81	1.00	1.00	0.58	1.24
N-Hexane	0.21	0.57	0.49	0.59	0.83	0.83	0.40	0.94
2-3-Methyl Pentane	0.10	0.22	0.17	0.22	0.23	0.23	0.14	0.33
Other C6's and C7's	0.00	0.12	0.15	0.15	0.17	0.17	0.11	0.24
CO	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CO2	0.08	1.03	1.10	1.72	1.84	1.84	2.91	1.08
H2S	13.09	20.88	9.19	19.04	14.22	14.22	9.78	8.07

RUN 260-05 PRODUCT ANALYSES

GAS ANALYSIS

Period	09T	10T	11T	12T	13T	14T	15T	16T
Period Start Date	06/13/94	06/14/94	06/15/94	06/16/94	06/17/94	06/18/94	06/19/94	06/20/94
VENT GAS, VOL% (N2, O2 Free Basis)								
H2	88.59	88.59	84.17	80.34	83.47	83.37	78.09	82.95
CH4	9.57	9.57	13.10	16.71	14.18	14.50	18.98	15.07
C2H4	0.00	0.00	0.01	0.00	0.00	0.00	0.00	0.00
C2H6	0.95	0.95	1.50	1.49	1.19	1.22	1.42	1.20
C3H6	0.00	0.00	0.02	0.00	0.00	0.00	0.00	0.00
C3H8	0.34	0.34	0.55	0.54	0.37	0.40	0.45	0.38
C4H8	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
N-C4H10	0.06	0.06	0.10	0.12	0.06	0.08	0.08	0.07
I-C4H10	0.02	0.02	0.02	0.03	0.02	0.02	0.02	0.02
C5H10	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
N-C5H12	0.02	0.02	0.03	0.04	0.02	0.02	0.02	0.01
I-C5H12	0.00	0.00	0.01	0.02	0.00	0.00	0.01	0.00
Methylcyclopentanes	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Cyclohexane	0.06	0.06	0.04	0.02	0.22	0.03	0.35	0.01
N-Hexane	0.00	0.00	0.01	0.00	0.00	0.01	0.01	0.01
2-3-Methyl Pentane	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Other C6's and C7's	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CO	0.11	0.11	0.24	0.13	0.14	0.07	0.14	0.09
CO2	0.09	0.09	0.16	0.26	0.20	0.15	0.26	0.11
H2S	0.19	0.19	0.03	0.28	0.11	0.12	0.17	0.08
BOTTOMS FLASH GAS, VOL% (N2, O2 Free Basis)								
H2	24.18	22.46	28.53	15.01	30.90	28.15	29.76	20.30
CH4	31.16	23.91	27.14	30.25	30.40	31.15	35.02	33.02
C2H4	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C2H6	12.57	12.31	13.92	13.77	11.51	12.00	11.01	13.68
C3H6	0.01	0.00	0.35	0.01	0.04	0.03	0.03	0.01
C3H8	11.60	12.82	13.23	12.43	10.40	11.10	9.58	13.13
C4H8	0.05	0.01	0.35	0.05	0.06	0.09	0.07	0.07
N-C4H10	5.29	6.05	6.51	5.20	4.47	4.85	4.14	6.09
I-C4H10	1.17	1.37	1.23	1.16	0.94	1.02	0.83	1.20
C5H10	0.00	0.00	0.01	0.00	0.00	0.00	0.00	0.00
N-C5H12	1.89	2.41	1.95	2.02	1.75	1.82	1.61	2.10
I-C5H12	1.18	1.44	1.14	1.10	0.90	1.02	0.79	1.17
Methylcyclopentanes	0.38	0.43	0.29	0.32	0.30	0.26	0.32	0.34
Cyclohexane	1.36	1.45	0.93	0.97	0.99	0.76	1.05	1.05
N-Hexane	0.92	1.09	0.99	0.86	0.79	0.86	0.77	0.92
2-3-Methyl Pentane	0.31	0.38	0.31	0.28	0.25	0.26	0.24	0.29
Other C6's and C7's	0.26	1.75	0.32	0.20	0.49	0.09	0.18	0.20
CO	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CO2	0.85	0.82	0.98	7.23	0.85	0.95	0.49	0.75
H2S	6.81	11.29	1.81	9.14	4.97	5.61	4.10	5.68

RUN 260-05 PRODUCT ANALYSES

GAS ANALYSIS

Period	17T	18T	19T	20T	21T	22T	23T	24T
Period Start Date	06/21/94	06/22/94	06/23/94	06/24/94	06/25/94	06/26/94	07/03/94	07/04/94
VENT GAS, VOL% (N2, O2 Free Basis)								
H2	83.71	77.97	78.15	78.50	79.85	79.12	81.32	81.32
CH4	14.19	19.23	18.89	18.61	17.47	18.03	16.16	16.16
C2H4	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C2H6	1.18	1.56	1.55	1.47	1.44	1.52	0.94	0.94
C3H6	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C3H8	0.40	0.52	0.52	0.48	0.47	0.49	0.29	0.29
C4H8	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
N-C4H10	0.08	0.09	0.10	0.09	0.08	0.08	0.05	0.05
I-C4H10	0.02	0.02	0.02	0.02	0.02	0.02	0.01	0.01
C5H10	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
N-C5H12	0.01	0.02	0.01	0.02	0.01	0.01	0.01	0.01
I-C5H12	0.00	0.01	0.01	0.01	0.00	0.00	0.00	0.00
Methylcyclopentanes	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Cyclohexane	0.14	0.01	0.01	0.05	0.02	0.01	0.04	0.04
N-Hexane	0.00	0.01	0.01	0.01	0.01	0.01	0.00	0.00
2-3-Methyl Pentane	0.00	0.00	0.00	0.00	0.01	0.00	0.00	0.00
Other C6's and C7's	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CO	0.10	0.16	0.19	0.20	0.14	0.20	0.23	0.23
CO2	0.08	0.29	0.36	0.33	0.34	0.34	0.40	0.40
H2S	0.08	0.11	0.17	0.20	0.14	0.16	0.56	0.56
BOTTOMS FLASH GAS, VOL% (N2, O2 Free Basis)								
H2	39.88	17.01	15.92	21.75	23.76	17.35	26.97	26.97
CH4	29.22	38.25	35.90	36.39	33.95	34.64	31.92	31.92
C2H4	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C2H6	9.84	14.64	13.71	12.67	12.68	14.17	8.36	8.36
C3H6	0.03	0.01	0.00	0.02	0.02	0.00	0.01	0.01
C3H8	8.68	12.61	12.52	10.87	11.07	12.60	7.30	7.30
C4H8	0.08	0.05	0.04	0.06	0.02	0.04	0.04	0.04
N-C4H10	3.58	4.92	5.26	4.07	4.35	4.99	2.60	2.60
I-C4H10	0.73	1.02	1.07	0.86	0.88	0.99	0.70	0.70
C5H10	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
N-C5H12	1.16	1.73	2.12	1.58	1.63	1.93	1.33	1.33
I-C5H12	0.65	1.03	1.05	0.75	0.79	0.95	0.97	0.97
Methylcyclopentanes	0.26	0.30	0.44	0.30	0.39	0.48	0.17	0.17
Cyclohexane	0.92	0.96	1.32	1.06	1.32	1.48	0.70	0.70
N-Hexane	0.69	0.75	1.06	0.84	0.92	1.08	0.45	0.45
2-3-Methyl Pentane	0.18	0.23	0.31	0.24	0.25	0.31	0.15	0.15
Other C6's and C7's	0.17	0.18	0.22	0.39	0.31	0.26	0.15	0.15
CO	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CO2	0.53	1.43	1.42	1.40	1.33	1.54	1.84	1.84
H2S	3.40	4.86	7.65	6.75	6.33	7.18	16.34	16.34

RUN 260-05 PRODUCT ANALYSES

GAS ANALYSIS

Period	25T	26T	27T	28T	29T	30T	31T	32T
Period Start Date	07/05/94	07/06/94	07/09/94	07/10/94	07/11/94	07/12/94	07/13/94	07/14/94
VENT GAS, VOL% (N2, O2 Free Basis)								
H2	68.59	76.61	74.79	80.88	77.84	77.29	77.64	81.11
CH4	20.37	15.99	21.85	16.65	19.21	19.35	19.62	15.68
C2H4	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C2H6	3.82	2.58	1.91	1.33	1.48	1.49	1.56	0.96
C3H6	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C3H8	2.85	1.84	0.64	0.41	0.45	0.49	0.50	0.29
C4H8	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
N-C4H10	1.41	0.88	0.12	0.07	0.08	0.08	0.09	0.07
I-C4H10	0.24	0.15	0.03	0.02	0.02	0.02	0.02	0.01
C5H10	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
N-C5H12	0.34	0.21	0.02	0.02	0.02	0.02	0.01	0.02
I-C5H12	0.22	0.14	0.01	0.00	0.00	0.01	0.00	0.01
Methylcyclopentanes	0.00	0.00	0.00	0.00	0.00	0.00	0.02	0.00
Cyclohexane	0.18	0.11	0.23	0.01	0.00	0.01	0.00	0.05
N-Hexane	0.17	0.10	0.00	0.01	0.01	0.01	0.03	0.01
2-3-Methyl Pentane	0.04	0.03	0.00	0.00	0.00	0.00	0.00	0.00
Other C6's and C7's	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CO	0.14	0.13	0.05	0.13	0.24	0.54	0.09	0.66
CO2	0.38	0.29	0.19	0.34	0.50	0.54	0.26	0.76
H2S	1.26	0.94	0.14	0.13	0.15	0.15	0.15	0.36

BOTTOMS FLASH GAS, VOL% (N2, O2 Free Basis)	25T	26T	27T	28T	29T	30T	31T	32T
H2	14.99	26.59	14.54	27.55	25.67	16.56	32.31	22.73
CH4	34.75	29.79	36.11	33.44	37.59	37.10	31.62	34.07
C2H4	0.00	0.00	0.00	0.01	0.01	0.00	0.00	0.00
C2H6	14.59	11.36	16.98	11.50	12.52	13.51	10.27	10.27
C3H6	0.00	0.00	0.01	0.04	0.03	0.00	0.00	0.01
C3H8	13.28	10.22	13.06	9.95	10.44	11.97	8.99	8.85
C4H8	0.00	0.04	0.08	0.10	0.08	0.00	0.00	0.12
N-C4H10	5.55	4.08	6.01	4.18	3.86	4.80	4.88	3.62
I-C4H10	1.27	0.99	1.32	0.93	0.90	1.04	0.86	0.90
C5H10	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
N-C5H12	1.91	1.45	1.99	1.58	1.01	1.78	0.00	1.48
I-C5H12	1.12	0.87	1.16	0.88	0.66	0.92	0.80	0.83
Methylcyclopentanes	0.30	0.21	0.27	0.35	0.09	0.37	0.34	0.26
Cyclohexane	1.07	0.84	0.87	1.12	0.27	1.10	1.52	0.73
N-Hexane	0.77	0.58	0.73	0.82	0.30	0.90	0.83	0.68
2-3-Methyl Pentane	0.25	0.18	0.27	0.27	0.13	0.27	0.23	0.22
Other C6's and C7's	0.21	0.18	0.15	0.21	0.05	0.18	0.00	0.12
CO	0.00	0.00	0.00	0.00	0.00	0.00	0.05	0.00
CO2	1.16	1.23	0.99	1.50	1.75	2.19	1.50	3.57
H2S	8.77	11.39	5.46	5.58	4.64	7.31	5.81	11.52

RUN 260-05 PRODUCT ANALYSES

GAS ANALYSIS

Period	33T	34T	35T	36T	37T	38T
Period Start Date	07/15/94	07/16/94	07/17/94	07/18/94	07/19/94	07/20/94
VENT GAS, VOL% (N2, O2 Free Basis)						
H2	73.60	71.42	73.79	74.87	76.21	80.69
CH4	22.15	24.41	21.90	21.04	20.29	16.01
C2H4	0.00	0.00	0.00	0.00	0.00	0.00
C2H6	1.74	1.70	1.70	1.75	1.64	1.41
C3H6	0.00	0.00	0.00	0.00	0.00	0.00
C3H8	0.48	0.43	0.43	0.00	0.00	0.48
C4H8	0.00	0.00	0.00	0.00	0.00	0.00
N-C4H10	0.07	0.06	0.06	0.06	0.05	0.10
I-C4H10	0.02	0.01	0.02	0.01	0.01	0.02
C5H10	0.00	0.00	0.00	0.00	0.00	0.00
N-C5H12	0.02	0.01	0.01	0.02	0.00	0.01
I-C5H12	0.01	0.00	0.01	0.00	0.00	0.01
Methylcyclopentanes	0.00	0.00	0.00	0.00	0.00	0.00
Cyclohexane	0.03	0.03	0.02	0.01	0.03	0.07
N-Hexane	0.01	0.00	0.01	0.00	0.00	0.00
2-3-Methyl Pentane	0.00	0.00	0.00	0.00	0.00	0.00
Other C6's and C7's	0.00	0.00	0.01	0.00	0.00	0.04
CO	0.68	0.68	0.70	0.81	0.59	0.48
CO2	1.00	1.07	1.13	1.28	0.89	0.42
H2S	0.19	0.18	0.21	0.15	0.29	0.25
BOTTOMS FLASH GAS, VOL% (N2, O2 Free Basis)						
H2	26.58	15.78	12.05	12.21	49.87	34.96
CH4	37.46	37.15	37.76	33.07	22.63	27.72
C2H4	0.01	0.00	0.00	0.00	0.02	0.03
C2H6	12.03	13.19	14.98	15.47	7.48	9.74
C3H6	0.04	0.02	0.00	0.00	0.07	0.04
C3H8	9.19	10.21	12.98	11.81	6.84	8.90
C4H8	0.02	0.03	0.00	0.00	0.04	0.29
N-C4H10	3.15	3.91	5.17	4.59	3.03	3.95
I-C4H10	0.78	0.89	1.11	0.98	0.67	0.88
C5H10	0.00	0.00	0.00	0.00	0.00	0.00
N-C5H12	0.88	1.38	1.86	1.52	1.09	1.61
I-C5H12	0.53	0.71	0.94	0.79	0.63	0.97
Methylcyclopentanes	0.09	0.23	0.30	0.25	0.22	0.36
Cyclohexane	0.22	0.65	0.79	0.64	0.64	1.19
N-Hexane	0.32	0.74	0.80	0.66	0.53	0.90
2-3-Methyl Pentane	0.12	0.21	0.26	0.20	0.17	0.28
Other C6's and C7's	0.09	0.09	0.12	1.05	0.11	0.36
CO	0.00	0.00	0.00	0.00	0.00	0.00
CO2	3.28	9.30	3.76	11.13	1.09	1.30
H2S	5.21	5.52	7.13	5.65	4.88	6.52

RUN 260-05 PRODUCT ANALYSES

GAS ANALYSIS

Period	39T	40T	41T	42T	43T	44T	45T
Period Start Date	07/22/94	07/22/94	07/23/94	07/24/94	07/25/94	07/26/94	07/27/94
VENT GAS, VOL% (N2, O2 Free Basis)							
H2	82.87	77.56	83.21	83.40	78.09	77.88	77.92
CH4	14.27	18.61	14.57	13.96	18.31	18.21	18.39
C2H4	0.00	0.00	0.00	0.00	0.00	0.01	0.00
C2H6	1.10	1.68	1.39	1.24	1.46	1.73	1.58
C3H6	0.00	0.00	0.00	0.00	0.00	0.01	0.00
C3H8	0.33	0.45	0.00	0.38	0.35	0.51	0.46
C4H8	0.00	0.00	0.00	0.00	0.00	0.00	0.00
N-C4H10	0.05	0.08	0.05	0.05	0.06	0.09	0.06
I-C4H10	0.02	0.02	0.01	0.02	0.02	0.05	0.04
C5H10	0.00	0.00	0.00	0.00	0.00	0.00	0.00
N-C5H12	0.00	0.02	0.00	0.00	0.00	0.01	0.00
I-C5H12	0.00	0.01	0.00	0.00	0.00	0.03	0.02
Methylcyclopentanes	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Cyclohexane	0.01	0.32	0.09	0.01	0.02	0.04	0.11
N-Hexane	0.00	0.00	0.00	0.01	0.00	0.00	0.00
2-3-Methyl Pentane	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Other C6's and C7's	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CO	0.41	0.38	0.02	0.32	0.57	0.56	0.45
CO2	0.47	0.40	0.13	0.18	0.70	0.36	0.62
H2S	0.48	0.47	0.52	0.44	0.43	0.50	0.35
BOTTOMS FLASH GAS, VOL% (N2, O2 Free Basis)							
H2	52.04	17.12	36.99	25.92	46.45	20.77	32.32
CH4	23.44	28.67	21.80	25.42	25.70	26.92	28.37
C2H4	0.02	0.00	0.01	0.00	0.01	0.00	0.00
C2H6	5.10	13.14	9.14	11.30	7.46	11.40	10.06
C3H6	0.05	0.02	0.04	0.02	0.04	0.25	0.04
C3H8	4.64	12.06	8.45	10.52	5.34	10.75	8.43
C4H8	0.15	0.06	0.07	0.06	0.07	0.19	0.04
N-C4H10	2.23	5.38	3.84	4.75	2.08	5.45	3.63
I-C4H10	0.58	1.52	1.18	1.31	0.70	2.71	2.01
C5H10	0.00	0.00	0.00	0.00	0.00	0.00	0.00
N-C5H12	0.97	1.92	1.32	1.59	0.76	1.77	1.25
I-C5H12	0.60	1.33	1.02	1.17	0.56	2.74	1.96
Methylcyclopentanes	0.19	0.19	0.12	0.23	0.17	0.26	0.20
Cyclohexane	0.65	0.49	0.47	0.82	0.63	0.63	0.68
N-Hexane	0.47	0.63	0.51	0.62	0.40	0.66	0.52
2-3-Methyl Pentane	0.17	0.29	0.23	0.26	0.14	0.31	0.22
Other C6's and C7's	0.14	0.11	0.26	0.17	0.18	0.10	0.19
CO	0.00	2.95	0.94	0.00	0.00	0.00	0.00
CO2	1.17	1.22	0.44	0.76	1.65	0.95	1.50
H2S	7.39	12.89	13.16	15.07	7.68	14.13	8.58

APPENDIX C - ANALYTICAL INSPECTIONS SUMMARY

Gas Analysis

Period	20	21	33	34	35	36	41	42	43	44	45
Space Velc.	28.8	28.3	37.8	37.9	38.7	37.7	24.4	24.5	27.1	21.7	24.9
Un-normalized											
C1-C3, mf %	9.60	9.80	8.46	8.69	10.00	9.97	10.41	12.34	9.20	12.98	10.84
C4-C7, mf %	4.09	4.43	2.17	2.94	4.18	4.23	5.01	6.35	3.51	8.15	5.70
feed ash	5.75	5.75	5.75	5.75	5.75	5.75	4.18	4.00	4.09	5.50	5.58
Calc. C1-C3, maf %	10.19	10.40	8.98	9.22	10.61	10.58	10.86	12.85	9.59	13.74	11.48
Calc. C4-C7, maf %	4.34	4.70	2.30	3.12	4.44	4.49	5.23	6.61	3.66	8.62	6.04
Normalized values											
C1-C3, maf %	9.13	9.72	9.99	9.35	10.11	10.11		14.39	10.28		11.94
C4-C7, maf %	4.14	4.66	2.73	3.35	4.45	4.54		7.77	4.17		6.61
Coal Conc.	100.00	100.00	100.00	100.00	100.00	100.00	69.30	66.00	67.50	81.89	71.99
Un-normalized C1-C3											
Coal contrib., maf % feed						9.89	6.85	6.53	6.68	8.10	7.12
waste contrib. , maf % feed							4.01	6.33	2.92	5.64	4.36
maf % waste							13.06	18.61	8.97	31.12	15.57
							63.09	50.78	69.59	58.96	62.02
Un-normalized C4-C7											
Coal contrib., maf % feed						3.77	2.61	2.49	2.54	3.09	2.71
waste contrib. , maf % feed							2.62	4.13	1.11	5.54	3.32
maf % waste							8.52	12.14	3.43	30.57	11.86
							49.97	37.62	69.53	35.80	44.96

POC-02 Product Quality

Period	Space Velocity	Temperature F K1	K2	K3 Top	K3 Btm	Naphtha Stabilizer Bottoms "As Is" N	Washed N	"As Is" S	Washed S	O-13 Bottoms QI	Resid Solid-free	O-43 Recy. H. Tank QI	Resid Solid-free
2	19.1	750	794	738	757	1.0	1.0	26.5	17.2	22.71	18.37	21.83	19.80
3	15.5	752	800	702	706								
4	19.1	749	811	676	665	51.3	46.5	51.9	24.3	20.85	18.17	14.93	26.67
5	16.9	750	812	676	666								
6													
7	16.1	745	749	668	675								
8	19.1	751	797	701	705	29.4	26.6	4.6	0.8	28.19	14.20	15.56	4.04
9	17.1	749	808	682	684	42.9	40.0	18.1	6.1				
10	19.4	749	915	692	701	20.0	16.1	21.5	4.0	18.58	15.62	7.34	11.33
11	19.2	749	813	697	707								
12	19.5	753	815	678	694	143.0	137.0	7.3	7.1	21.98	18.21	11.87	15.21
13	19.9	750	814	685	706								
14	19.6	750	815	702	711					18.35		7.51	14.72
15	18.5	751	815	704	712	0.1	0.2	5.5	0.5	16.84	15.25	6.21	11.33
16	17.5	750	813	708	712								
17	17.7	750	813	710	712	9.0	7.5	5.0	1.9	24.00		9.57	
18	24.2	762	823	712	710								
19	28.2	776	830	710	708	46.4	44.5	11.6	9.4	26.98	15.58	14.16	
20	28.8	776	831	712	713								
21	28.3	775	831	713	716	51.0	48.9	5.0	1.9	27.70	12.89	16.74	13.43
22	28.6	776	832	712	713	53.5	52.0	8.0	7.3	32.73	15.08		
23		728	731	601	638	48.4	44.8	<1.0	<1.0				
24	18.6	754	757	659	675	53	52.2	36.0	20.2	25.56	7.00	14.10	16.43
25	20.4	774	798	701	697								
26													
27	21.4	776	812	713	723								
28	29.1	778	830	721	723	42.0	39.2	7.7	4.5	22.98	9.61	8.90	11.75
29	31.8	780	832	724	726	45.4	44.9	13.5	1.4				
30	19.9	765	790	689	683								
31	27.2	777	830	730	727	28.6	25.9	12.4	3.1	22.52	9.91	8.58	
32	36.4	794	837	737	739								
33	37.8	805	837	732	691	24.5	22.7	13.8	2.1			7.55	
34	37.9	812	837	734	712	51.0	48.0	12.6	6.0				
35	38.7	811	836	736	739								
36	37.7	809	836	739	741	46.6	44	17.4	0.5				
37	29.4	810	830	728	735	18	16	11	2.4				
38	9.0	780	812	702	704								
39						65.6	64.1	65.3	40.9				
40													
41						47	46	48.5	25.4				
42						57.2	51.4	43.7	13.9				
43						29.5	26.1	29.9	9.4				
44													
45						39	35.5	32.8	15.4				
46													

POC-02 Analytical Summary

Period	1	2	4	8	9	10	11	12	13	14	15
Coal											
Moisture, W%		10	10.6	10.39		10.71		10.39		10.2	10.52
Ash, W% dry	6	5.87	6	5.99		6		5.56		5.74	5.75
O-13 Bottom											
QI, W%		22.71	20.85	28.19		18.58		21.98		18.35	16.84
QI Ash, W%		7.8	8.74	10.01		7.93		9.31		6.51	6.42
Coal Conv., W%		88.1	91.2	88.4		91.4		92.0		88.9	90.1
TGA Resid, W%		18.37	18.17	14.2		15.62		18.21			15.25
O-43											
QI, W%		21.82	14.93	15.56		7.34		11.87		7.51	6.21
QI Ash, W%		6.29	5.24	4.71		2.6		4.68		2.45	2.03
Coal Conv., W%		84.6	88.2	85.3		88.4		91.0		87.4	87.4
TGA Resid (+ash), W%		41.62	41.6	19.6		18.67		27.08		22.23	17.54
TGA Ash, W%		6.34	5.22	4.23		2.56		4.9		2.71	2.34
Naphtha Stab. Bottoms											
Water	Nil	Nil	Nil	Nil	Nil	Nil		Nil		Nil	Nil
API	30.4	30.4	29.1	32.3	31.6	31.5		32.2		31.8	32
IBP, F	186	174	180	175	181	182		178		185	182
5 V%, F	229	221	234	218	225	225		225		227	223
95 V%, F		672	694	655	658	655		660		650	655
EP, F	709	713	722	692	687	692		695		689	693
Antek N, ppm											
"As is"	135.8	<1.0	51.3	29.4	42.9	20		143			0.12
Caustic Washed	125.9	<1.0	46.5	26.6	40	16.1		137			0.2
Antek S, ppm											
"As is"	596.9	26.5	51.9	4.6	18.1	21.5		7.3			5.5
Caustic Washed	544.4	17.2	24.3	0.8	6.1	4		7.1			0.48
ASB, 650F- W%		22.02	13.48	25.67		22.18		14.7		19.27	23.28
VSB, 850F- W%		35.33	10.59	10.1							
Vent Gas Analysis, V%											
H2		78.31		81.59	60.32	88.59	84.17	80.34	83.47	83.37	78.09
CH4		18.94		16.09	21.19	9.57	13.1	16.71	14.18	14.5	18.98
C1-C3		20.42		17.86	31.73	10.86	15.18	18.74	15.74	16.12	20.85
C4-C7		0.16		0.13	5.4	0.16	0.123	0.234	0.328	0.16	0.49
COx		0.36		0.26	0.5	0.2	0.406	0.396	0.348	0.22	0.4
H2S		0.75		0.16	2.05	0.19	0.03	0.285	0.113	0.122	0.165
Mol WT.		5.61		5.06	12.94	4	4.8	5.39	4.9	4.78	5.8
Bottom Gas Analysis, V%											
H2				15.54	24.18	22.46	28.53	15.01	30.9	28.15	29.76
CH4				33.76	31.16	23.91	27.14	30.25	30.4	31.15	35.02
C1-C3				61.22	55.82	49.04	54.64	56.46	52.35	54.27	55.64
C4-C7				14.1	12.82	16.38	14.03	12.16	10.93	11.02	10
COx				1.08	0.85	0.82	0.98	7.23	0.85	0.95	0.49
H2S				8.07	6.81	11.29	1.81	9.14	4.97	5.61	4.1
Mol WT.				28.6	25.83	29.31	25.5	29.29	23.12	23.81	22.2

POC-02 Analytical Summary

Period	1	2	4	8	9	10	11	12	13	14	15
Feed to Solids Sep. Unit											
PI, W%			41.00	32.00		42.62		47.58		45.83	46.42
PI Ash, W%						10.81		13.91		13.59	13.38
TI, W%			34.92	24.43		34.46		39.33		38.41	37.66
TI Ash, W%			11.70	7.19		11.56		14.80		13.84	13.31
QI, W%			32.09	25.03		35.16		38.94		37.28	39.45
Ash, W%			11.73			12.24		15.01		13.79	13.72
ROSE Bot. or Filter Cake											
PI, W%			76.68	83.92	63.34	72.15		69.93		62.08	73.10
PI Ash, W%				24.24	17.91	18.18		20.21		19.33	22.32
TI, W%			63.12	70.46	51.01	64.43		56.61		57.89	65.15
TI Ash, W%			18.04	24.20	17.61	21.50		20.53		21.42	23.18
QI, W%			59.00	62.78		64.15		52.54		55.46	62.49
Ash, W%			18.22	22.04		22.43		20.49		21.51	23.98
Coal Conversion, W%			85.71	88.22		88.13		90.79		90.39	90.20
TGA Resid (+ash), W%			83.14					83.05			86.86
TGA Ash, W%			18.87					21.1			25.02
Appearance			Powder	Powder	Oily	Powder		Gummy			Lumpy
Deashed Oil											
API			-11.8		-28.8	5.8		7.2		7.4	6.4
IBP, F			713		649			717		732	722
IBP-850F W%			22.5		19.96			23.33		23.14	20.47
H/C ratio			0.09		0.09			0.11			0.11
PI, W%			36.53					2.2			1.59
PI Ash, W%											0
QI, W%			29.17		53.16	4.86		1.22		1.58	0.96
QI Ash, W%			11.23			1.77		0.18		0.16	0.01
975F+ Resid, W%			62.4		78.95	35.21		32.65		32.75	33.43

POC-02 Analytical Summary

Period	16	17	18	19	20	21	22	23	24	25	26
Coal											
Moisture, W%		10.81		10.79		10.99	10.79		10.69		
Ash, W% dry		7.3		5.75		5.68	5.75		5.58		
O-13 Bottom											
QI, W%		24		26.98		27.72	32.73		25.56		
QI Ash, W%		9.61		12.04		12.89	15.08		7.01		
Coal Conv., W%		88.2		92.4		93.1	92.9		84.4		
TGA Resid, W%				15.58		15.45			20.40		
O-43											
QI, W%		9.57		14.16		16.74			14.1		
QI Ash, W%		3.23		5.63		7.27			3.34		
Coal Conv., W%		84.5		90.8		92.2			81.0		
TGA Resid (+ash), W%						30.17			30.52		
TGA Ash, W%						7.25			3.44		
							Front	Rear			
Naphtha Stab. Bottoms											
Water		NIL	NIL	NIL		NIL	NIL	NIL	NIL		
API		32.9	32.1	35.0		35.8	32.1	34.9	33.4		
IBP, F		170	177	170		167	177	181	183		
5 V%, F		207	217	203		206	217	221	239		
95 V%, F		663	653	618		608	653	630	628		
EP, F		689	685	665		640	685	658	660		
Antek N, ppm											
"As Is"		9.00	53.50	46.40		51.00	53.50	48.40	53.00		
Caustic Washed		7.50	52.00	44.50		48.90	52.00	44.80	52.20		
Antek S, ppm											
"As Is"		4.96	8.00	11.60		5.00	8.00	<1.0	36.00		
Caustic Washed		1.92	7.30	4.40		1.90	7.30	<1.0	20.20		
ASB, 650F- W%		19.67		33.66		44.87			42.05		
VSB, 850F- W%											
Vent Gas Analysis, V%											
H2	82.95	83.71	77.97	78.15	78.5	79.85	79.12		81.32		89.61
CH4	15.07	14.19	19.23	18.89	18.61	17.47	18.03		16.16		8.89
C1-C3	16.65	15.77	21.31	20.96	20.56	19.37	20.04		17.38		9.64
C4-C7	0.122	0.257	0.163	0.163	0.204	0.153	0.132		0.113		0.051
COx	0.203	0.185	0.448	0.551	0.53	0.479	0.538		0.627		0.284
H2S	0.081	0.082	0.112	0.173	0.204	0.149	0.163		0.555		0.406
Mol WT.	4.80	4.78	5.67	5.67	5.63	5.39	5.5		5.15		3.76
Bottom Gas Analysis, V%											
H2	20.3	39.87	17.01	15.92	21.75	23.76	17.35		26.97		44.01
CH4	33.02	29.22	38.25	35.9	36.39	33.95	34.64		31.92		22.34
C1-C3	59.83	47.78	65.51	62.12	59.96	57.72	61.41		47.59		34.48
C4-C7	13.44	8.42	11.19	12.9	10.15	10.86	12.52		7.253		4.873
COx	0.746	0.534	1.431	1.417	1.396	1.333	1.539		1.844		1.325
H2S	5.68	3.4	4.86	7.65	6.74	6.33	7.19		16.34		15.32
Mol WT.	26.9	19.43	26.28	27.79	24.79	24.88	27.5		22.75		18.01

POC-02 Analytical Summary

Period	16	17	18	19	20	21	22	23	24	25	26
Feed to Solids Sep. Unit											
PI, W%		52.74		54.07		57.75			33.73		
PI Ash, W%		17.65		18.02		19.62			6.14		
TI, W%		45.80		41.27		45.31			28.55		
TI Ash, W%		18.50		16.62		19.85			6.32		
QI, W%		30.72		44.04		40.43			25.69		
Ash, W%		12.11		17.81		19.27			5.05		
ROSE Bot. or Filter Cake											
PI, W%		80.30		64.86		61.93			76.73		
PI Ash, W%		27.97		21.52		21.35			26.63		
TI, W%		71.92		68.01		50.51			64.50		
TI Ash, W%		28.34		26.36		21.67			27.45		
QI, W%		69.51		63.73		46.35			59.82		
Ash, W%		28.00		26.12		20.93			27.26		
Coal Conversion, W%		88.33		91.22		92.69			92.94		
TGA Resid (+ash), W%				85.13		73.27					
TGA Ash, W%				26.88		21.40					
Appearance		Dry Powder		Lumpy Powder					Solid Lump		
Deashed Oil											
API		0.7		6		-0.2			-10.7		
IBP, F		745		693					708		
IBP-850F W%		20.19		29.13					24.17		
H/C ratio		0.1		0.11		0.1			0.1		
PI, W%											
PI Ash, W%											
QI, W%		11.6		3.98		8.88			26.75		
QI Ash, W%		3.38		0.99		3.05			14.5		
975F+ Resid, W%		42.15		31.17					50.21		

POC-02 Analytical Summary

Period	27	28	29	30	31	32	33	34	35	36	37
Coal											
Moisture, W%	10.81	10.21	10.81	10.81	9.7	10.81	10.81	10.81	10.81	11.3	11.88
Ash, W% dry	5.75	5.62	5.75	5.75	5.58	5.75	5.75	5.75	5.75	5.53	5.6
O-13 Bottom											
QI, W%		22.98			22.52		20.41	19.33		19.83	14.89
QI Ash, W%		9.61			9.91		8.92	9.24		9.13	6.92
Coal Conv., W%		91.7			92.5		92.1	93.3		93.1	93.2
TGA Resid, W%		17.67			19.77		20.61	18.9		23.96	18.2
O-43											
QI, W%		8.90			8.58		7.55	7.41		7.55	5.47
QI Ash, W%		3.34			3.49		3.41	3.49			1.6
Coal Conv., W%		90.1			91.4		92.6				
TGA Resid (+ash), W%		20.65			20.21		27.76	21.95	22.01	25.12	19.82
TGA Ash, W%		3.46			3.98		3.41	3.42	3.36		1.88
Naphtha Stab. Bottoms											
Water		Nil	Nil		Nil		Nil	Nil		Nil	Nil
API		35.2	34.2		32.8		35.2	34.9		38	36.2
IBP, F		171	172		175		162	172		167	169
5 V%, F		216	214		226		210	216		209	216
95 V%, F		629	630		645		597	594		548	556
EP, F		660	661		680		626	619		588	584
Antek N, ppm											
"As Is"		42.00	45.40		28.60		24.50	51.00		46.60	18.00
Caustic Washed		39.20	44.90		25.90		22.70	48.00		44.00	16.00
Antek S, ppm											
"As Is"		7.70	13.50		12.40		13.80	12.00		17.40	11.00
Caustic Washed		4.50	1.40		3.10		2.14	6.00		0.50	2.40
ASB, 650F- W%		30.87			26.28		41.7	34.82		59.35	65.87
VSB, 850F- W%											
Vent Gas Analysis, V%											
H2		80.88	77.84	77.29	77.64	81.11	73.6	71.42	73.79	74.87	76.21
CH4		16.65	19.21	19.35	19.62	15.68	22.15	24.41	21.9	21.04	20.29
C1-C3		18.39	21.14	21.33	21.67	16.94	24.37	26.54	24.03	22.79	21.93
C4-C7		0.132	0.132	0.153	0.173	0.172	0.16	0.111	0.142	0.101	0.09
COx		0.467	0.743	1.079	0.356	1.418	1.685	1.753	1.831	2.093	1.48
H2S		0.132	0.153	0.153	0.153	0.365	0.191	0.182	0.212	0.153	0.286
Mol WT.		5.2	5.71	5.86	5.71	5.33	6.58	6.853	6.56	6.32	6.00
Bottom Gas Analysis, V%											
H2		27.55	25.67	16.56	32.31	22.73	26.58	15.78	12.05	12.21	49.87
CH4		33.44	37.58	37.1	31.62	34.07	37.4	37.15	37.76	33.07	22.63
C1-C3		54.94	60.59	62.58	50.85	53.21	58.73	60.57	65.72	60.35	37.04
C4-C7		10.44	7.35	11.35	9.45	8.97	6.19	8.83	11.35	10.67	7.13
COx		1.5	1.75	2.19	1.56	3.57	3.28	9.303	3.76	11.12	1.087
H2S		5.58	4.64	7.31	5.8	11.52	5.21	5.517	7.13	5.65	4.879
Mol WT.		23.5	22.01	26.82	21.82	24.5	21.82	26.67	28.21	29.52	16.91

POC-02 Analytical Summary

Period	27	28	29	30	31	32	33	34	35	36	37
Feed to Solids Sep. Unit											
PI, W%		40.43		52.17	46.98		50.34	50.24		49.94	53.10
PI Ash, W%		11.28		15.05	12.25		12.45	12.37		13.07	13.42
TI, W%		31.94		39.89	36.29		35.05	34.04		34.18	43.80
TI Ash, W%		11.51		15.15	12.37		12.44	12.09		13.97	19.48
QI, W%		30.71		39.15	33.40		32.85	30.70		33.32	27.95
Ash, W%		11.41		15.07	11.32		12.42	12.64		13.56	11.80
ROSE Bot. or Filter Cake											
PI, W%		71.65		80.93	88.69		91.31	88.81		95.53	94.48
PI Ash, W%		20.43		23.34	23.90		24.31	22.93		23.44	23.68
TI, W%		60.90		61.32	64.90		69.08	65.56		61.99	84.35
TI Ash, W%		21.47		22.82	22.34		23.88	23.63		24.77	25.15
QI, W%		58.89		58.2	67.15		62.69	59.52		56.99	59.43
Ash, W%		21.57		17.18	22.88		24.09	24.18		24.22	24.20
Coal Conversion, W%		89.7		85.4	88.6		90.2	91.1		92.1	91.4
TGA Resid (+ash), W%							89.48			89.96	
TGA Ash, W%							24.86			25.31	
Appearance		Lumpy					Powdery	Powdery		Lumpy	Lumpy
Deashed Oil											
API		8.3			3.1		3	1.4		1.8	
IBP, F		734			705		695	686		698	
IBP-850F W%		30.83			26.26		22.3	28.36		30.42	
H/C ratio		0.11					0.09			0.09	
PI, W%							9.23			6.44	
PI Ash, W%											
QI, W%		0.9			1.47		0.66	0.72		0.99	1.93
QI Ash, W%		0.26			0.42			0.14		0.03	0.17
975F+ Resid, W%		26.68			31.78		36.22	34.37		33.99	

POC-02 Analytical Summary

Period	38	39	40	41	42	43	44	45	46	37
Coal										
Moisture, W%	10.81	11.42	10.81	11.31	11.58	11.31	10.81	10.31	10.81	11.88
Ash, W% dry	5.75	5.03	5.75	3.98	4.29	5.34	5.75	5.93	5.75	5.6
O-13 Bottom										
QI, W%		10.87		10.33	12.62	9.43	12.98	13.88		14.89
QI Ash, W%		3.57		4.23	3.42	3.54	4.13	4.36		6.25
Coal Conv., W%		89.2		94.0	87.9	90.6	86.9	94.2*		91.8
TGA Resid, W%		16.89		26.42	20.43	21.46		26.22		18.2
Cyclohexane Insol.		11.89		10.98	13.49	9.99		13.6		15.7
CI Ash		3.74			3.47	3.53		4.04		6.44
O-43										
QI, W%		14.01				11.84		7.49		5.47
QI Ash, W%		1.00						0.64		1.6
Coal Conv., W%										
TGA Resid (+ash), W%					28.75	29.16		23.03		19.82
TGA Ash, W%		1.19			0.11	0.02		1.31		1.88
Naphtha Stab. Bottoms										
Water		0		0	0	0		0		0
API		29.3		30.6	31.6	30.8		32.8		36.2
IBP, F		183		175	183	172		157		169
5 V%, F		241		235	243	235		218		216
95 V%, F		647		644	650	634		645		556
EP, F		674		674	685	671		667		584
Antek N, ppm										
"As Is"		65.60		47.00	57.20	29.50		39.00		18.00
Caustic Washed		64.10		46.00	51.40	26.10		35.50		16.00
Antek S, ppm										
"As Is"		65.30		48.50	43.70	29.90		32.80		11.00
Caustic Washed		40.90		25.40	13.90	9.40		15.40		2.40
ASB, 650F- W%		43.40		31.11	36.06	37.87		39.92		
VSB, 850F- W%										
Vent Gas Analysis, V%										
H2	80.69		77.56	83.20	83.40	78.09	77.88	77.92		76.21
CH4	16.61		18.61	14.57	13.96	18.31	18.21	18.39		20.29
C1-C3	17.90		20.74	15.97	15.58	20.11	20.47	20.43		21.93
C4-C7	0.26		0.46	0.15	0.09	0.10	0.23	0.23		0.09
COx	0.90		0.77	0.15	0.50	1.27	0.92	1.07		1.48
H2S	0.25		0.47	0.53	0.44	0.43	0.50	0.35		0.29
Mol WT.	5.42		6.04	4.79	4.84	5.78	5.88	5.88		6.00
Bottom Gas Analysis, V%										
H2	34.96		17.12	36.99	25.92	46.45	20.77	32.32		49.87
CH4	27.72		28.67	21.80	25.42	25.70	26.92	28.37		22.63
C1-C3	46.44		53.90	39.44	47.27	38.55	49.33	46.90		37.04
C4-C7	10.78		11.92	9.01	10.98	5.68	14.82	10.71		7.13
COx	1.30		4.17	1.39	0.76	1.65	0.95	1.50		1.09
H2S	6.52		12.89	13.16	15.07	7.67	14.13	8.58		4.88
Mol WT.	22.28		27.83	21.68	25.45	16.91	28.02	22.71		16.91

* Adjusted for ash and carbon black from rubber tire.

POC-02 Analytical Summary

Period	38	39	40	41	42	43	44	45	37	S/D	S/D
Feed to Solids Sep. Unit											
PI, W%		32.47		38.52	45.12	53.15	46.54	34.04	53.10		
PI Ash, W%		7.67		6.98	6.05	8.83	5.21	7.02	13.42		
CI, W%		25.55		30.76	39.95	42.65	39.21	25.40	43.80		
CI Ash, W%		7.62		7.10	6.43	6.68	5.34	6.57	19.48		
QI, W%		20.29		27.68	38.26	40.30	37.45	25.54	27.95		
Ash, W%		7.70		7.48	6.57	6.63	5.38	7.09	11.85		
				7.54							
ROSE Bot. or Filter Cake											
PI, W%		91.78		36.34	66.59	80.31	82.25	83.44	94.48	83.06	78.26
PI Ash, W%		24.69		1.01	6.00	12.09	11.83	20.29	23.68	19.80	19.65
CI, W%		81.97		21.95	54.50	75.53	74.61	75.61	84.35	78.36	72.49
CI Ash, W%		24.61		1.1	5.63	12.02	11.91	19.58	25.15	19.42	20.13
QI, W%		56.90		19.28	54.63	67.71	66.08	64.75	59.43	64.56	60.80
Ash, W%		23.95		1.13	6.08	11.79	11.50	19.80	24.27	17.78	18.70
Coal Conversion, W%		92.71			51.3	71.10			91.20		
TGA Resid (+ash), W%				57.95	78.78	84.36	84.39	82.29			
TGA Ash, W%				1.29	4.6	12.61	12.52	20.46			
Appearance									Lumpy		
Deashed Oil											
API		2.3		3.5	4.1	4.4	5.3	5.8			
IBP, F		7.05		723	7.02	716	690	703			
IBP-850F W%		34.06		36.19	35.92	33.72	36.17	36.06			
H/C ratio		0.1		0.1	0.11			0.09			
PI, W%				5.17	11.30	12.72	15.07	24.73			
PI Ash, W%				0	0.12		0.08	4.5			
QI, W%		1.3		3.36	5.84	10.37	12.8	19.63	1.93		
QI Ash, W%		0.12		0.05	0.01	0.2	0.32	4.93	0.17		
975F+ Resid, W%		30.28		26.48	30.65	33.72	32.78	45.29			

POC-02 Product Quality

Period	Space Velocity	Temperature F K1	K2	K3 Top	K3 Btm	Naphtha Stabilizer Bottoms "As Is" N	Washed N	"As Is" S	Washed S	O-13 Bottoms QI	Resid Solid-free	O-43 Recy. H. Tan QI	Resid Solid-free
2	19.1	750	794	738	757	1.0	1.0	26.5	17.2	22.71	18.37	21.83	19.80
3	15.5	752	800	702	706								
4	19.1	749	811	676	665	51.3	46.5	51.9	24.3	20.85	18.17	14.93	26.67
5	16.9	750	812	676	666								
6													
7	16.1	745	749	668	675								
8	19.1	751	797	701	705	29.4	26.6	4.6	0.8	28.19	14.20	15.56	4.04
9	17.1	749	808	682	684	42.9	40.0	18.1	6.1				
10	19.4	749	915	692	701	20.0	16.1	21.5	4.0	18.58	15.62	7.34	11.33
11	19.2	749	813	697	707								
12	19.5	753	815	678	694	143.0	137.0	7.3	7.1	21.98	18.21	11.87	15.21
13	19.9	750	814	685	706								
14	19.6	750	815	702	711					18.35		7.51	14.72
15	18.5	751	815	704	712	0.1	0.2	5.5	0.5	16.84	15.25	6.21	11.33
16	17.5	750	813	708	712								
17	17.7	750	813	710	712	9.0	7.5	5.0	1.9	24.00		9.57	
18	24.2	762	823	712	710								
19	28.2	776	830	710	708	46.4	44.5	11.6	9.4	26.98	15.58	14.16	
20	28.8	776	831	712	713								
21	28.3	775	831	713	716	51.0	48.9	5.0	1.9	27.70	12.89	16.74	13.43
22	28.6	776	832	712	713	53.5	52.0	8.0	7.3	32.73	15.08		
23		728	731	601	638	48.4	44.8	<1.0	<1.0				
24	18.6	754	757	659	675	53	52.2	36.0	20.2	25.56	7.00	14.10	16.43
25	20.4	774	798	701	697								
26													
27	21.4	776	812	713	723								
28	29.1	778	830	721	723	42.0	39.2	7.7	4.5	22.98	9.61	8.90	11.75
29	31.8	780	832	724	726	45.4	44.9	13.5	1.4				
30	19.9	765	790	689	683								
31	27.2	777	830	730	727	28.6	25.9	12.4	3.1	22.52	9.91	8.58	
32	36.4	794	837	737	739								
33	37.8	805	837	732	691	24.5	22.7	13.8	2.1			7.55	
34	37.9	812	837	734	712	51.0	48.0	12.6	6.0				
35	38.7	811	836	736	739								
36	37.7	809	836	739	741	46.6	44	17.4	0.5				
37	29.4	810	830	728	735	18	16	11	2.4				
38	9.0	780	812	702	704								
39						65.6	64.1	65.3	40.9				
40													
41						47	46	48.5	25.4				
42						57.2	51.4	43.7	13.9				
43						29.5	26.1	29.9	9.4				
44													
45						39	35.5	32.8	15.4				
46													
47													

APPENDIX D

Summary of 260-05 Unit Modifications

Item Description

1 **Brooks purge meter coalescers** were installed before and after the 5 hydrogen purge gas mass flow controllers. These meters frequently (especially during start-up) became fouled with oil, either from the compressor or the process, during run 4. During run 5, these meters were nearly trouble-free, requiring only about 3 to 5 total cleanings over the 45 days of operations compared to an estimated 20 cleanings during run 4.

2 **O-41 scale:** O-41 is used as a recycle oil blending tank. Vacuum still overheads, deashed oil, reactor liquid flash bottoms, atmospheric bottoms and vacuum bottoms all enter this vessel during various operating modes. In run 4, the inventory in this vessel was monitored by level indication only. During run 5, the new scale increased the accuracy of transfers to and from this vessel and reduced the operator effort required for these tasks.

3 **O-46 scale:** O-46 is used as a reactor liquid flash weigh tank. Material from this tank goes the filter, the vacuum tower feed accumulator, the recycle oil blending tank or the ROSE unit depending on the unit's operating mode. During run 4, the inventory in this vessel was monitored by level indication only. During run 5, the new scale increased the accuracy of transfer to and from this vessel and reduced the operator effort required for these tasks.

4 **VSO, SW scales:** During run 4, the vacuum still overhead and the sour water scales were BCD type-equipment which were never connected to the revised Process Control system. These over 15 years old scales were replaced for run 5 with 1-5 volt systems which were connected to the process control system. We were able to easily track these streams through our computer system to supplement or verify log sheet data during run 5. Again, this modification improved the accuracy of our data.

5 **Recycle gas compressor:** The recycle gas compressor vent discharged to the outdoors in run 4. This vent was connected to our flare relief header prior to run 5 thus improving the safety of the working environment.

6 **Using the recycle gas heater to preheat recycle gas to the second stage reactor:** Prior to run 4, the recycle gas heater was used to preheat recycle gas to the first stage reactor and electrical resistance windings were used to preheat recycle gas to the second stage reactor. During run 4, the transfer line between the recycle gas heater became restricted during the Period 6 and 11 restarts forcing us to route this recycle gas to the fresh feed preheater. Later in the run, it became apparent that windings were not going to provide adequate heat to the second stage recycle gas to achieve the desired reactor temperatures. We then rerouted the second stage recycle gas through the recycle gas heater. This arrangement has worked well and provided good reactor temperature control. However, the Autoclave check, which became restricted twice during this run 4, was still in the heater discharge line. This check was replaced with a Mogas block valve prior to run 5. This heater was not lost due to restriction between the heater and the reactor in run 5.

7 **O-62 LCVs:** The transfer of high solids containing material from the ROSE-SR^{sm} first stage settler to either of the two bottoms receivers was a difficult task during POC-1. A slight upset and this line became restricted. We had replaced the upstream gate valves with Mogas block valves during run 4. These valves have worked well providing a tight shutoff between the settler and the receiver. Additional changes were completed prior to run 5 including:

- installing an upstream (before the LCV) gas oil flush. This allowed one to flush through the LCV into the receiver.
- installing a normally open gate valve just downstream of the Mogas block valve. This allowed for higher pressure purging through the LCV into the receiver since the Mogas valve is a unidirectional valve.
- relocating the LCV closer to the receiver
- installing a permanent vso purge system. In run 4, operators hand carried gas oil to a local purge pump. This offered a limited supply, was inconvenient and increased the likelihood of oil spills.

With these modifications, operations was frequently able to flush the lines between O-62 and O-63. The ROSE section was seldom off-line due to a restricted bottoms outlet in run 5. During run 4, this section had to be disassembled 6 to 10 times to clear restrictions.

8 **Larger O-45**, Re-designed to handle approximately 40 kg/hr (88 pph) of water and 54 kg/hr (120 pph) of oil. In run 5, Reactor Liquid Flash Overheads were recycled to the hydrotreater with over 91 kg/hr (200 pph) of water. Very few incidents of water in the atmospheric still were observed in run 5. In run 4 the water injection rate had to be limited to about 45 kg/hr (100 pph) in order to maintain good atmospheric still operation.

9 **Purge oil to K-1 sample system:** The first stage reactor sample system has worked very well in the bench units. Two first stage reactor liquid samples were obtained during POC-1. Additional samples would have been taken if we have been able to purge the associated hot check suction line. This was installed prior to run 5 and the sample system operation was improved with 3 samples taken during POC-2. Taking reactor liquid from the side of the ebullating pump suction line and correcting an issue regarding the removal of samples from the sample bomb will further improve this system.

10 **ROSE trim heater:** The ROSE unit utilizes a hot/cold solvent exchanger. This exchanger works well when the unit is lined out. However, it tends to snowball unit upsets. If the returning solvent is too hot, it will heat the feed solvent too much; if the returning solvent is too cold, it will not heat the feed solvent enough. A Dowtherm heated trim heater downstream of the current exchanger was installed prior to run 5 insuring the feed solvent was at the desired temperature before it mixed with the resid stream. This new heater greatly improved the ROSE section operation, to the point that we now are concentrating more on improving separations and less on maintaining operations.

11 **ASBs to COT, VSO to O-41:** In run 4, atmospheric bottoms went to the recycle oil blending tank and excess vacuum overheads go to the clean oil tank or tank farm. These streams were switched prior to run 5 so that any heavy oil

returned to the tank farm was hydrotreated. There were no issues raised by this piping change.

12 **Flare Shell:** The flare is currently located on an outside gasifier tower. The burner is contained within a thin metal shroud. This shroud gets very hot and glows red at night. A thermocouple is also attached to the shroud and used to monitor the flare flame temperature. This thermocouples currently fail within a week as it in direct contact with the flame, causing the metal sheath to melt. HTI's air permit requires a flame temperature of 927 °C (1700°F) be maintained to insure proper combustion of materials. Prior to run 5, we:

- lined the inside of the shroud with a ceramic liner,
- installed a thermowell to protect the thermocouple and

In run 5, the shroud no longer glowed and we were able to consistently measure the flame temperature.

13 **Reactor Thermocouple repair:** Each of our ebullated bed reactors has 10 internal side-entry thermocouples. These thermocouples are all attached to the reactor via threaded stainless steel fittings. Each time the ebullating oil cup is removed from the reactor these thermocouples have to be removed. With time these fittings become galled, unusable and require repair. We are proposing to design a standard repair which will include a Grayloc-type connection. The design for this modification was approved prior to run 5 and parts for 5 assemblies were purchased. These will be installed on an as-needed basis. With this modification thermocouples can be removed from the reactor without unscrewing them and if future repairs are required these can be done inside the machine shop rather than inside the reactor tower. No thermocouple repairs were required during run 5.

14 **HTU internals:** The hydrotreater process performance during the first POC operation indicated material was bypassing the catalyst baskets. A new configuration of the hydrotreater internals was designed and installed prior to run 5. This does not include catalysts baskets and therefore should not allow

bypassing of the catalyst. Operating data from the hydrotreater indicated the new internal design met its objectives.

15 **P-2 rotary valve:** The new coal handling system pneumatically conveys material from the long term storage bins to our day hopper (P-2). Coal is then transported by a screw conveyor into the slurry mix tank. If the conditions in the day hopper are correct, large quantities of coal could be transported into the slurry mix tank when the day hopper was filled. This occurred during run 4. This phenomena can shut down our PDU if the coal loading in the slurry mix tank is allowed to increase excessively. A rotary valve between the day hopper and the screw conveyor was installed prior to run 5 to stop this phenomena and serve as a pressure barrier, preventing the pneumatic transfer of coal through the screw. There were no pneumatic transfers of coal through this screw during run 5.

16 **Replace O-44**: O-44 is an oil/water separator downstream of our primary oil/water separator. Water from our primary separator is decanted in this glass vessel to improve our oil recovery. This vessel ruptured during first POC run, when our flare header became restricted, and was replaced with a carbon steel vessel, for safety reasons, prior to run 5. However, the instrumentation did not arrive until late in the run, hence this vessel was not used in run 5. The few minor upsets in the atmospheric still, which were observed in run 5, may have been avoided if this vessel was in use.

17 **O-1 Inlet:** The separator obtained from Wilsonville was used in the first POC operation because it was larger in diameter than the previously used separator. Both separators have a smaller diameter liquid section and larger diameter vapor section. However, the inlet to the Wilsonville separator is in the 3-inch bottom section. It should be in the 6-inch vapor section. The inlet was relocated prior to run 5. Entrainment of heavy materials into our cold separator and oil/water separator was not observed in run 5.

18 **O-36 Waterboot:** O-36 is the naphtha stabilizer feed accumulator. It a small 6-inch diameter vessel, primarily used as a sight glass. This was replaced with a metal vessel for safety reasons prior to run 5. The new vessel also has a

water boot so that the entry of water into the naphtha stabilizer can be prevented. Water was not detected in NSB samples during run 5.

19 **Relocate Catalyst Addition Valves:** In the past, catalyst has been added through the top of the reactor. The current PDU reactors also have side-entry catalyst addition nozzles. These nozzles were incorporated into the design of these vessels because the Rockwell plug valves originally used for catalyst handling were very large. Top loading of catalyst to the current ebullated bed reactors with Rockwell valves would have exceeded the local zoning. Cold modeling studies indicate that a portion of these side-entry nozzles are liquid/catalyst filled at all times. We believe this restricts the flow of fresh catalyst into the main portion of the catalyst bed. The side-entry nozzles appeared to have become restricted during the first POC campaign. Because the Valtron dual valves are much smaller than the Rockwell plug valves, we were able to relocate the catalyst addition system on top of the reactors prior to run 5. Catalyst addition proceeded without a problem during run 5.