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The Effect of Water on the
Thermal Solution of Stuart
Deposit Oil Shale

by

Ralph A. Briley

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A thesis submitted to the Faculty and the Board of Trustees of the Colorado School of Mines in partial fulfillment of the requirements for the degree of Master of Science (Chemical and Petroleum-Refining Engineering).

Golden, Colorado

Date January 20, 1984

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T-2806

DEDICATION

To my wife Susan.

ABSTRACT

Research on the effect of water on production of shale oil by the thermal solution of oil shale from the Stuart A deposit near Gladstone, Queensland, Australia has been carried out in a batch stirred autoclave reactor. The objective of the work was to determine the effect of water on organic carbon conversion and the resulting yield structure of oil to gas. Yields of oil and gas have been quantified for the simultaneous extraction and hydrogenation of shale oil under a wide range of operating conditions, including both hydrogen donor and non-donor solvents, hydrogen and inert gas atmospheres, and when water was and was not added to the system.

This study discusses the effect of water and type of solvents on hydrogen consumption, organic carbon conversion, and selectivity for oil over gas formation. The mechanics by which the water effects the thermal solution are also discussed.

TABLE OF CONTENTS

| <u>SECTION</u> | <u>PAGE</u> |
|--|-------------|
| ABSTRACT | iv |
| LIST OF FIGURES | vii |
| LIST OF TABLES | viii |
| ACKNOWLEDGEMENTS | ix |
| LITERATURE SURVEY | 1 |
| Introduction | 1 |
| Kerogen Recovery | 4 |
| Hydrotorting of Shale | 7 |
| Conclusions | 8 |
| EXPERIMENTAL DESIGN | 10 |
| System Design Considerations | 11 |
| Reaction Conditions | 11 |
| EXPERIMENTAL APPARATUS | 15 |
| EXPERIMENTAL AND ANALYTICAL PROCEDURES | 21 |
| Oil Shale Selection | 21 |
| Oil Shale Preparation | 21 |
| Solvents | 22 |
| Experimental Run Procedure | 23 |
| Gas Analysis | 27 |
| Liquid Analysis | 32 |
| Solid Analysis | 34 |

| | |
|--|-----|
| DATA ANALYSIS | 36 |
| Ash Balances | 36 |
| Organic Carbon Conversion | 37 |
| Hydrogen Consumption and Yield Structure | 38 |
| Statistical Analysis | 39 |
| DISCUSSION OF RESULTS | 42 |
| Introduction | 42 |
| Excluded Data | 42 |
| Processing Errors | 43 |
| Effect of Drying on Conversion | 45 |
| Hydrogen Consumption Results | 46 |
| Water Addition Results | 48 |
| Non-donor Solvent Results | 49 |
| Water Effect Mechanism | 53 |
| CONCLUSIONS | 59 |
| RECOMMENDATIONS | 61 |
| LITERATURE CITED | 63 |
| APPENDIX A - Raw Data | 67 |
| APPENDIX B - Oxygen Analysis | 122 |
| APPENDIX C - Water Content | 124 |
| APPENDIX D - Sample Calculations | 126 |

LIST OF FIGURES

| <u>FIGURES</u> | | <u>PAGE</u> |
|----------------|--|-------------|
| 1 | Location of Stuart Deposit Oil Shale | 3 |
| 2 | 300 cc Batch Autoclave Reactor | 16 |
| 3 | Time/Temperature Profile | 20 |
| 4 | Gas Chromatograph Integrator Print-out | 29 |
| 5 | Gas Chromatograph System | 31 |
| 6 | Liquid Gas Chromatogram | 33 |

LIST OF TABLES

| <u>TABLE</u> | | <u>PAGE</u> |
|--------------|--|-------------|
| 1 | Fixed Operating Conditions | 14 |
| 2 | Modified Fischer Assay of Vacuum Dried Beneficiated Stuart-A Oil Shale | 22 |
| 3 | Initial Pressures When Water Added | 27 |
| 4 | Gas Chromatograph Calibration Results for RB-63 | 30 |
| 5 | Repetitive Experimental Run Results | 40 |
| 6 | Repetitive Experimental Run Results for Pure Hydrogen | 40 |
| 7 | 1-Methylnaphthalene Results | 50 |
| 8 | Heavy Gas Oil Results | 52 |
| 9 | Carbon Oxide Calculation Results | 58 |
| 10 | Oxygen Concentration | 123 |
| 11 | Water Content | 125 |

ACKNOWLEDGEMENTS

The author wishes to express his gratitude to Dr. Robert Baldwin and Dr. Gary Baughman for their helpful advice and guidance during the work. In addition, the financial support provided by Southern Pacific Petroleum, N. L., Sydney, Australia, and the Colorado School of Mines, Office of Research Development, is gratefully acknowledged.

LITERATURE SURVEY

Introduction

As the 1980's were ushered in, the demand for crude oil in the United States amounted to 18 million barrels a day, or about one barrel for every twelve citizens. Of that amount, about 40 percent was oil imported from other countries -- much of it from countries in the politically unstable Middle East.(1) This ever increasing energy demand in the United States and the need for foreign sources of petroleum have highlighted the necessity of developing alternative sources of liquid fuels. One of the major sources that can be tapped for this product is oil shale. Due to the economic considerations and the present "oil glut", interest in synthetic fuels in general has diminished significantly in the past few years. However, there are those in the industry who realize the potential oil shale has in this country's and the world's future. Even though technology has existed for two centuries to process oil shale, many consider the present methods in the infant stage in relation to optimum liquid fuel recovery.

Oil shale is a common earth resource found on all of the inhabited continents. There are an estimated 3,000 billion(2) to 2,000,000 billion(3) barrels of shale oil reserves world wide. Of special interest in this thesis are

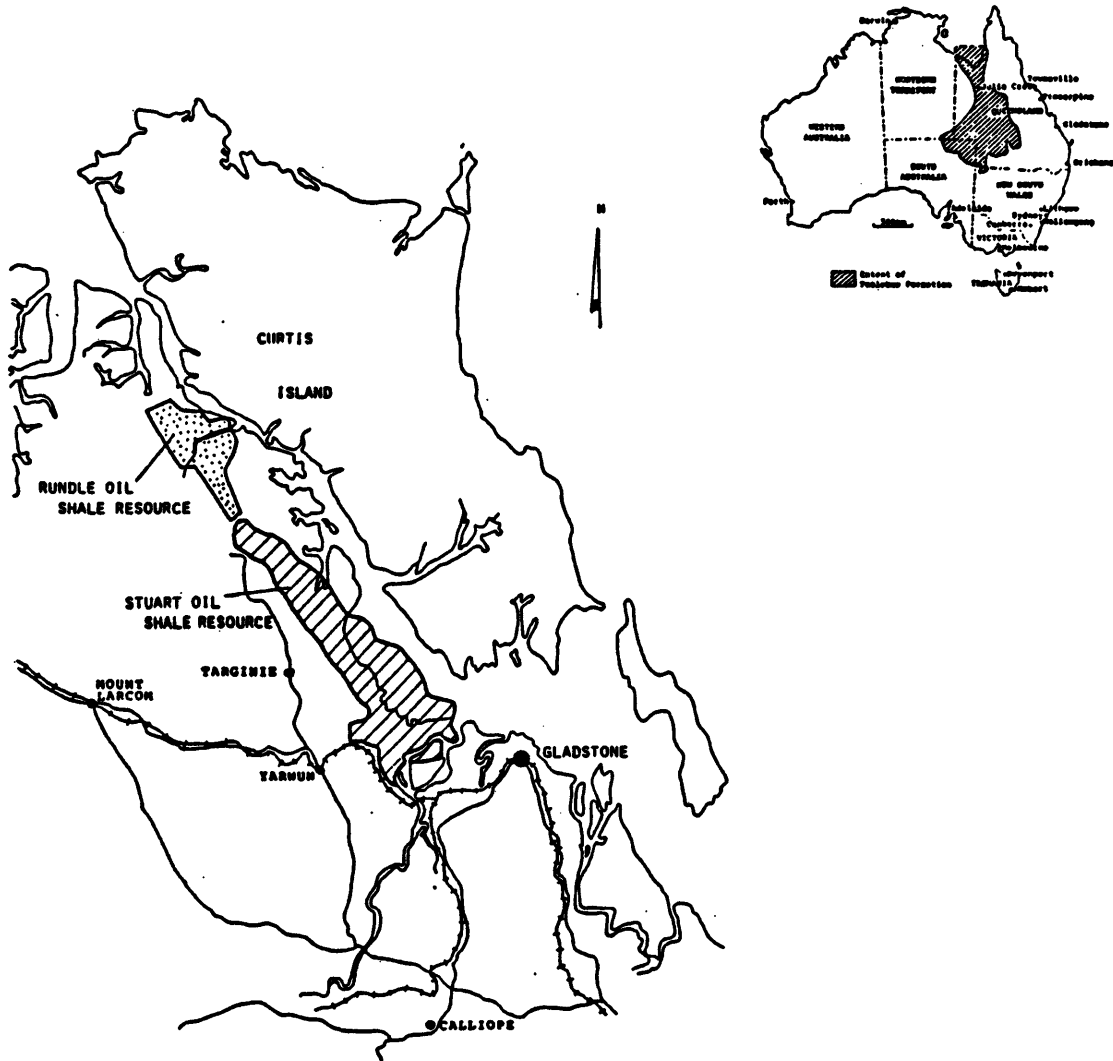
the 1.5×10^9 to 3.1×10^{12} barrels of reserves located in three states of Australia: New South Wales, Queensland, and Tasmania.(4) The State of Queensland is where the bulk of the oil shale reserves in Australia exist. The oil shale used in this study was from the Stuart deposit in Queensland, Australia. The location of this deposit is shown in Figure 1.

Shale oil is derived from the organic portion of the oil shale which may be thermally degraded and separated from the inorganic portion of the oil shale. The organic portion is known as kerogen. Kerogen is thought to be a three-dimensional polymer, is insoluble in conventional organic solvents, and is associated with small amounts of a benzene-soluble organic material, bitumen.(5) From elemental analyses, Robinson(6) developed the following formulae for kerogen from the Green River formation oil shale, based upon heteroatoms: $C_{18}H_{28}O$, $C_{39}H_{60}N$, or $C_{215}H_{330}O_{12}N_5S$. The last formula indicates that kerogen has a molecular weight of approximately 3200.

Baker(7) describes the Stuart and adjacent Rundle deposits as being of Tertiary age and occurring in a narrow graben about 5 kilometers wide. The kerogen is of algal origin deposited in a lacustrine environment giving rise to fine grained mudstone with a specific gravity of 1.75.

FIGURE 1

Location of Stuart Deposit Oil Shale



Hutton et al.(8) claim that both Stuart and Green River kerogen are of "Alginite B" type. This type of organic matter was derived from finely-banded, layered blue-green algae which grew in relatively warm, shallow lakes. The decomposing algae was deposited in an incoherent ooze on the lake-bed. The Stuart deposit was laid down in seven layers of varying thickness.

Kerogen Recovery

Several different methods have been explored to recover oil shale kerogen to provide a petroleum type product or feed stock. Of the several procedures available, only pyrolysis has received the attention required to develop a commercial processing technology(9). Retorting involves heating oil shale in the absence of oxygen resulting in the thermal decomposition of the organic matter in the shale.

It is technically possible to upgrade oil shale kerogen to a petroleum-like product by hydrogen addition. Although direct hydrogenation, or hydrogenation in vehicle oils, is well developed technology in coal liquefaction processes, applying this technique to oil shale is far less developed.

Kerogen is characterized by its insolubility in such organic solvents as benzene, phenol and acetone at their normal boiling points. At higher temperatures kerogen decomposes to gas, oil, and carbonaceous residue. When

heated to temperatures above 600°K, the organic matter in shale may be solvent extracted in high yield^(10, 11). This extraction of organic matter in oil shale by means of a solvent at elevated temperatures is known as thermal solution. When the reaction temperature and pressure exceed the critical properties of the solvent, the process is then known as supercritical extraction.

Temperatures employed in thermal solution processes are lower than retorting temperatures. The added presence of the solvent allows this to happen, while still maintaining acceptable levels of carbon conversion. Lower temperatures decrease the conversion of the kerogen material into gas, thereby, yielding a higher selectivity to oil. Thermal solution of oil shale involves the partial fragmentation of the organic material into compounds of comparatively low molecular weight, which are soluble in organic solvents. The solvent improves heat transfer characteristics in the solution process, adds its own ability to detach organic material from its original matrix, and may act as a hydrogen transferring vehicle⁽¹²⁾.

Several early patents describe solvent processing of torbanite and other shale-like materials at elevated temperatures both with and without hydrogen gas atmospheres^(13, 14, 15, 16). These early studies used

natural solvents such as crude shale oil, shale oil distillate, kerosene fraction, crude petroleum, and distillation residue.

Dulhunty(17, 18) in his research, found that aromatic compounds were more effective solvents than those of an aliphatic nature. Ethyl alcohol, ethyl ether, and light petroleum were totally unsuitable as they dissolved only a portion of the extract. The aromatic solvents and torbanite crude oil distillate, completely extracted the soluble products, and were capable of holding all the extracted material in solution when cold. Dulhunty determined that the extraction temperature would control both the amount of hydrocracking and rate of extraction. In addition, he concluded that there was a limiting temperature of operation above which the oil yield structure will decrease. The influence of pressure, in relation to the extraction temperature, directly determines the removal of certain products from the reaction almost as soon as they are formed.

Since 1940, numerous articles on thermal solution of solid fuels including oil shale have been published. D'yakova(19, 20, 21) reported yields of 72 to 96 percent of the organic matter for seven different U.S.S.R. shales. The extraction temperatures were 385 to 420°C. The various

solvents used were: tetralin, anthracene oil, petroleum fuel oil, diesel fuel, hydrogenated shale tars, and shale oil distillate (220 to 370°C).

In the past 30 years there have been many studies done on oil shale. In an extensive study, Jensen et al.(22) reported on hydroprocessing of Green River oil shale in batch, semi-continuous, and continuous mode reactors. This work reaffirmed the applicability of the thermal solution process to oil recovery employing a wide range of solvents under widely ranging times, temperature, and pressure conditions. Concurrent hydrogenation and thermal extraction was also studied. A cold initial hydrogen pressure of 2005 psi proved adequate to extract 100% of the shale organic matter for a reaction at or above 650°F for 110 minutes. An increased quality of oil was not seen by Jensen in the hydrogenation experiments.

Recently, patents on thermal solution processes in vehicle oils have been granted to Gregoli(23), Patzer(24), and Greene(25). Thermal solution applied to Stuart deposit oil shale using tetralin and other solvents have also been published by Baldwin et al.(26, 27, 28), and Frank(29).

Hydrotorting of Shale

Schlinger et a.(30) researched the recovery of oil from oil shale, as it relates to the hydrotorting of raw shale

with hydrogen-rich gas and H₂O. The shale oil produced had substantially reduced nitrogen and sulfur content, and yields of greater than 110 percent of the Fischer Assay. They found the addition of H₂ reduced the hydrogen consumption and heat load required for a given yield of shale oil. The hydrogen-rich gas used contained at least 45 volume percent H₂, and included pure hydrogen and synthesis gas. The reaction conditions for their system were a temperature range of 750 to 1500°F and a pressure range of 300 to 1000 psig, and .01 to .6 ton of H₂O per ton of oil shale.

Conclusion

The preceding discussion and literature cited illustrates the feasibility of the extraction of organic carbon from oil shale, by employing the thermal solution technique. This system allows the extraction and hydrogenation process to be operated at less severe conditions than those required for retorting. In addition, there is a high organic carbon conversion, high oil yield, and low gas make.

There have been many suggestions as to which solvents to use in this process, without identifying the optimum one. The suggested solvents have been hydrogen donor (e.g. tetralin), non-donor (e.g. toluene), and natural solvents

(e.g. distillate fractions of the product shale oil).

Another area which has not been fully explored is the amount of hydrogen consumed for the various solvents. Along the same line, studies have been very limited on how to minimize the hydrogen consumption, and still maintain high conversions and oil yields. The thermal solution technique works, yet it needs to be optimized to make it a viable alternative to crude oil.

EXPERIMENTAL DESIGN

The overall objective of this study was to determine the effect of water on the simultaneous extraction and hydrogenation of kerogen from an Australian oil shale. Both hydrogen-rich donor and non-donor solvents were used. The scope of the research program encompassed total hydrogen consumption, organic carbon conversion, and selectivity for oil over gas formation. This section discusses the basic experimental design, along with the specification of the conditions used to accomplish this objective.

Experimental Design

To accurately determine any effect due to water on the thermal solution of oil shale, all confounding factors had to be minimized. This required the operating conditions to be set so as to allow any effect to be observed and recognized. In addition, all of the fixed experimental variables could not vary throughout the study.

This study was designed around four general principles. The first principle was the need to quantify the hydrogen consumption, the organic carbon conversion, and the oil yield of the thermal solution of oil from the subject shale using tetralin as the solvent. Once this reference was established, the second principle was to find out if the addition of water to the system changed the total hydrogen

consumption, the organic carbon conversion, or oil yield. The third principle was to determine how the water effects the system; if the water addition showed positive results. The final principle was to compare the results in the donor solvent system to that of a non-donor solvent system.

Additive composition and type of solvents used were the only variables in this study. Time and temperature were fixed for each run throughout this study. The order of the runs was randomized in order to make any errors in the study independent.

System Design Consideration

The system used in this study was designed to allow batch reactor data to be obtained. From previous studies, heat-up to reaction temperature did not introduce significant experimental error into organic carbon conversion data.⁽³¹⁾ Coupling this with the fact that kinetic data was not being obtained, slurry injection was not employed.

Reaction Conditions

Specification of reaction conditions was based primarily on typical conditions used in similar coal and shale research.

There were several factors used to set the reaction temperature. The reaction temperature range commonly used

in donor solvent reactions is 350° to 460°C. It has been observed that minimum kerogen conversion using tetralin is around 285°C, with the optimum conversion occurring at 425°C.⁽³²⁾ Tetralin has a critical temperature of 446°C.⁽³³⁾ It was assumed that when tetralin was mixed, its critical temperature would not vary significantly from the 446°C. This assumption could be verified by conducting enthalpy measurements on the tetralin/shale mixture at the temperatures and pressures being studied. Solvents in their supercritical range exhibit properties markedly different from those of subcritical solvents.⁽³⁴⁾ Setting the reaction temperature at this point provided the optimum thermal effect on the rate of thermal solution and allowed any effect by the water to be recognized as such.

A cold reactor pressure of 800 psi was chosen to establish the base case data. When water was added to the various systems, the initial gas pressure was varied so that the reaction pressure (at temperature) was between 1200 and 1500 psig.

There was only one reaction time analyzed in this study. The reaction time was set at 60 minutes, once the reaction temperature was reached. It has been shown that the overall conversion does not change significantly past 60 minutes of reaction time.⁽³⁵⁾ This long reaction time also

has the advantage of eliminating any effects on conversion that might be due to different heating rates during the non-isothermal reaction period.

Pure tetralin was used to prevent donor solvent starvation. As in other related coal and shale studies, donor solvent starvation occurs at low donor solvent concentrations and ultimately effects the rate of hydrogenation. For this study, tetralin in excess of the minimum amount required for complete conversion of the kerogen was chosen as the donor solvent. A total of 50 grams of tetralin was used per 25 grams of shale. This amount of tetralin insured adequate mixing. Using 25 grams of shale allowed a number of different analyses to be run on one product sample.

For those runs where a non-donor solvent was used, the solvent to shale ratio was kept at 2:1. As in the tetralin cases, 50 grams of solvent and 25 grams of shale were present. The four non-donor solvents used were: 1-methylnaphthalene, toluene, heavy and light gas oils.

A summary of the fixed operating conditions is given in Table 1.

TABLE 1

FIXED OPERATING CONDITIONS

| Fixed Operating Variables | Explanation |
|----------------------------------|---|
| Temperature = 425°C | <ol style="list-style-type: none"> 1. Optimum thermal Solution. (31) 2. Tetralin's critical temperature at 446°C 3. Allows the effect of water to be recognized. |
| Initial Cold Pressure = 800 psig | Obtains a reaction pressure of approximately 1500 psig. |
| Solvent (Tetralin) = 50 grams | <ol style="list-style-type: none"> 1. In excess of theoretical amount required for complete conversion of kerogen. 2. Insures adequate mixing. |
| Solvent (Non-donor) = 50 grams | Insures adequate mixing. |
| Shale = 20 grams | Allows multiple analyses on one product sample. |
| Shale : minus 200 mesh | Minimize mass transfer effects. |

EXPERIMENTAL APPARATUS

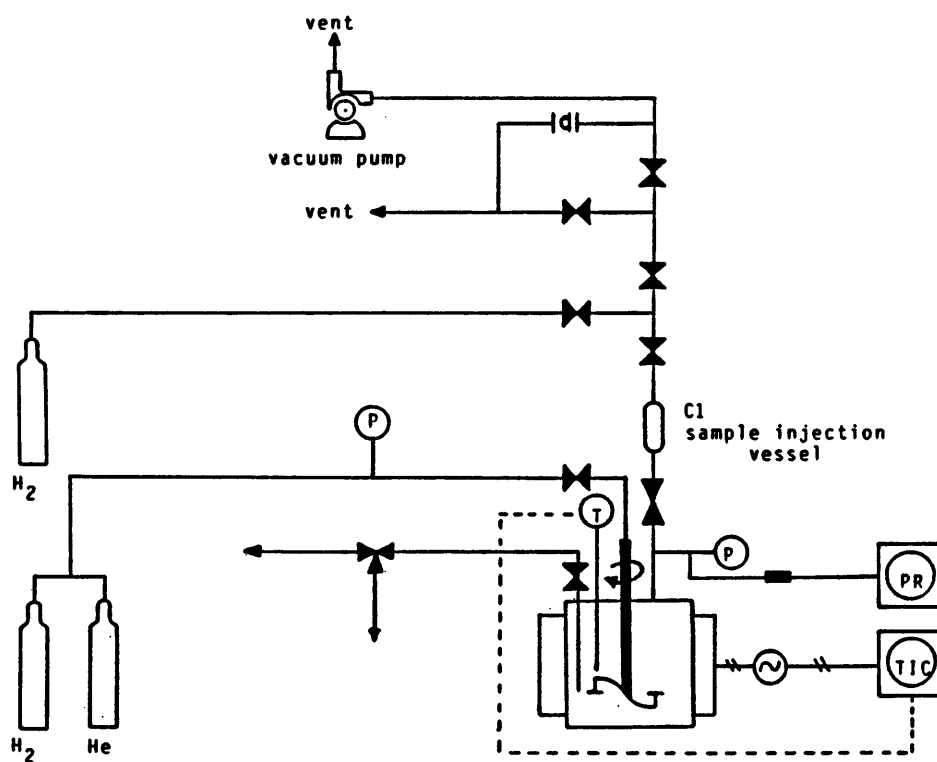
All experiments for this study were conducted in a 300 cc batch stirred autoclave reactor, manufactured by Autoclave Engineers, Inc. A schematic of the reactor and associated piping are shown in Figure 2. The batch autoclave system was designed to fulfill the following requirements:

- (1) rapid heating of the oil shale slurry to reaction temperature (30 minutes or less);
- (2) rapid quenching of the reaction mixture temperature upon reaction completion;
- (3) isothermal operation at desired reaction temperature;
- (4) safe operation at pressures in excess of 2000 psig.

The reactor contained a variable speed stirrer, internal cooling coils, and a thermowell to permit monitoring of reaction temperature, and was constructed of 316 stainless steel. The stirring assembly consisted of a 1.25 inch diameter impeller and attached baffle. The impeller shaft was connected to the stirring head and driven by a variable speed motor. Impeller rotation speed was monitored via magnetic detection ring and a tachometer. The stirrer operated at 750 rpm during the reaction to insure a

FIGURE 2

300 cc Batch Autoclave Reactor



uniform liquid composition and high liquid-gas interfacial area.

The heat source was a jacket-type heater manufactured by Autoclave Engineers. The heater delivered 1.2 KW from a 115 AC electrical circuit and had a maximum operating temperature of 1400°F. A standard scissors-type laboratory jack was used to raise and lower the heater.

A type K (nickel-chromium vs. nickel-aluminum) thermocouple extended into the heater thermowell to monitor the heater temperature. It was connected initially to a Leeds and Northrup Electromax III temperature controller which was used to maintain isothermal operation. This controller was replaced with a Leeds and Northrup Electromax V microprocessor digital industrial controller.

A type J (iron vs. constantan) thermocouple extended into the reactor thermowell to monitor the reaction temperature. This thermocouple was connected to a Thermo Electric ELPH 3 digital temperature indicator. Calibration of this system was periodically done, based upon the National Bureau of Standards Monograph "Thermocouple Reference Tables Based on the IPTS 68" correlating emf and temperature.

A gas delivery system was used which was designed to allow the reactor to be purged with helium for pressure

testing prior to each run. It was also designed to allow high pressure hydrogen/argon to be used to pressurize the system to the initial (cold) reaction pressure. The system allowed a gas sample to be taken upon reaction completion and cool down. All remaining gas in the reactor system was vented to the atmosphere through a knock-out pot.

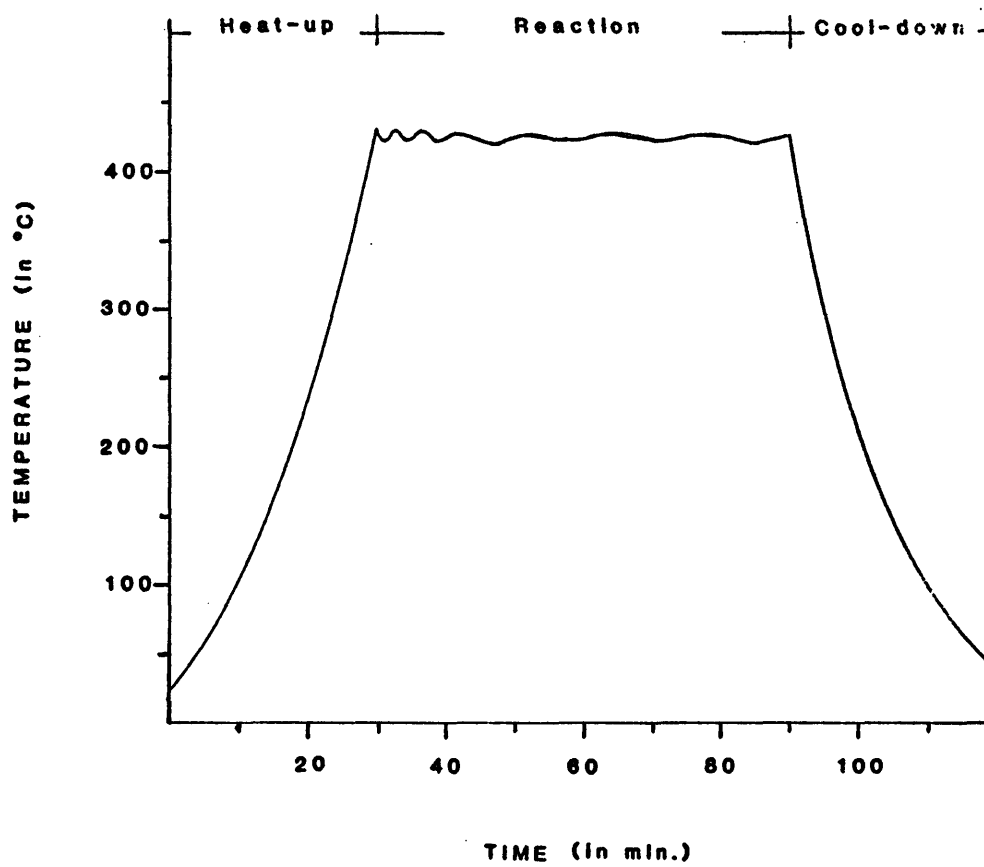
The gas delivery system consisted of two gas cylinders, two pressure regulators, two pressure gauges, a rupture disk, and associated valves, fittings and tubing. The pressure regulators were connected directly to the gas cylinders and were rated at 5000 psig. System pressure was monitored externally with a Autoclave Engineers, model TSW-6-05 transducer and a Autoclave Engineers, model AECI-5000 digital pressure indicator. The pressure indicator, with a range of 0 to 5000 psig, was connected between the reactor and valve no. 1. The rupture disk was designed to fail at 5400 psig at 650°F. All valves, tubing, and fittings were 316 stainless steel.

Rapid heating of the reactor and its contents was accomplished by allowing maximum power input into the heater for a short period of time. Quenching of the reaction mixture temperature was accomplished by cutting off power to the heater, lowering the heater support jack, activating a high speed fan, and allowing cooling water to flow through

the reactor cooling coils. This rapid heating and temperature quenching minimized errors in determining reaction time. A typical time/temperature profile is shown in Figure 3.

FIGURE 3

Time/Temperature Profile



EXPERIMENTAL AND ANALYTICAL PROCEDURES

This section contains the experimental and analytical procedures used in the selection and preparation of the oil shale. The various solvents used in this research are also included. Concluding this section are the experimental run procedures, and the liquid and gaseous product analysis methods used.

Oil Shale Selection

The oil shale used was from the Stuart A deposit near New Gladstone, Queensland, Australia. This sapropelic shale was supplied by Southern Pacific Petroleum N.L., of Sydney, Australia. This research did not concern itself with other types of oil shales from different parts of the world.

Oil Shale Preparation

The Stuart shale was beneficiated by de-sliming prior to grinding. It was then wet ground in a rod mill, sieved to 100% minus 74 micron (minus 200 mesh), and vacuum dried at 40°C prior to use. Once the shale was dried, it was stored in a Labconco vacuum desiccator until used. The Modified Fischer Assay analysis of the oil shale is shown in Table 2. Guin et al.(36), have shown that with coal there are no mass or heat transfer limitations for hydrogenation of particles smaller than 147 micron diameter, thus use of this particle size should eliminate these effects from the results obtained.

TABLE 2
Modified Fischer Assay of Vacuum Dried
Beneficated Stuart-A Oil Shale

| Testing Facility | Spent Shale (wt%) | Oil (wt%) | Water (wt%) | Gas + Loss (gal/ton) | Oil (gal/ton) | Water (gal/ton) | Oil Density (gr/ml) |
|------------------|-------------------|-----------|-------------|----------------------|---------------|-----------------|---------------------|
| CT & E(1) | 72.4 | 16.3 | 5.3 | 6.0 | 44.0 | 12.6 | .886 |
| CT & E(1) | 73.4 | 15.5 | 5.1 | 6.0 | 42.4 | 12.3 | .877 |
| CSMRI(2) | | | | 6.2 | 40.4 | 11.3 | .88 |

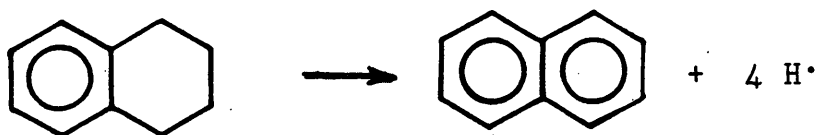
(1) Commerical Testing and Engineering Company, Golden, Colorado.

(2) Colorado School of Mines Research Institute, Golden, Colorado.

Solvents

For the majority of this research there were two different types of solvents used. The hydrogen donor solvent was 1,2,3,4-tetrahydronaphthalene (tetralin). The two non-donor solvents employed were toluene, and 1-methyl naphthalene.

Tetralin is a hydrogen rich solvent that has been widely used in coal liquefaction research as a hydrogen donor. It serves as a hydrogen donor via the following reaction:



The tetralin used was commercial grade.

For a few experiments heavy (610°F to 710°F) gas oil was used as a natural solvent. This fraction was obtained from a pyrolysis oil. The oil was received from Geokinetics of Vernal, Utah. It was distilled using a 36 inch spinning band adiabatic distillation column, with a monel band, manufactured by Perkin-Elmer.

Experimental Run Procedure

The following is a description of the experimental procedure developed for making the base case runs (i.e. with no water addition, tetralin solvent used, H₂ with a 5% argon tracer) in this research:

- (1) 25 grams of oil shale were weighed and placed into the reactor.
- (2) 50 grams of tetralin were weighed and placed into the reactor.
- (3) The reactor was connected to the system.
- (4) The system was purged and tested for any gas leakage using 1800 psig of helium.
- (5) The system was twice purged with 1000 psig of hydrogen gas (with a 5% argon tracer.) Then the

reactor was pressurized to the desired initial reactor pressure of 800 psig.

- (6) The stirrer was set at 750 rpm, the reactor head cooling water turned on, main gas cylinders isolated, and temperature controller set at 700°C.
- (7) While the tetralin in the reactor was heating, a vacuum was drawn on the gas sample line to remove any gases. After the vacuum was drawn, the evacuated gas sample vessel was attached to the gas sample line by quick-disconnect.
- (8) When the slurry temperature reached 400°C, the temperature controller setting on the heater was decreased to 580°C.
- (9) Once the reaction temperature reached 425°C, the experimental run was monitored for temperature and pressure fluctuations for 60 minutes.
- (10) When the reaction time had elapsed, the heater was immediately lowered and the reactor cooled using a high speed fan and cooling water.
- (11) After the reactor had cooled to ambient temperature and pressure were recorded. Then a gas sample was drawn into an evacuated sample bottle. The final temperature and pressure were recorded for the entire system, including the sample bottle.

- (12) Excess gas was then vented.
- (13) The reactor slurry was washed with acetone into centrifuge tubes, sonicated, then centrifuged for 15 minutes at 3000 rpm.
- (14) The product oil and solvent mixture were decanted and stored.
- (15) The spent oil shale was twice re-suspended with acetone, sonicated for 6 minutes, spun, and the oil and acetone mixture decanted and saved.
- (16) The shale oil and solvent mixture was Roto-Vapped at 60 °C and ambient pressure until all recoverable acetone was removed. The oil and solvent mixture was then recovered.
- (17) The spent oil shale was dried for 18 hours at 100°C and ambient pressure. A Soxhlet extraction was then done on the residue with a solution of 50% benzene/50% methanol.
- (18) The residue was again dried for 18 hours at 100°C and ambient pressure. It was analyzed for total carbon and inorganic carbon using a Coulometrics system, and then ashed in muffle furnace.
- (19) The liquids were analyzed by chromatographic simulated distillation on an HP model 5840 gas chromatograph to determine the boiling range, and .

by elemental analysis on a Carlo-Erba elemental analyzer.

- (20) Reaction product gases were determined on a Carle model 111-H gas chromatograph, with hydrogen, hydrocarbon gases (through C₅), and carbon oxide gases quantified.

Modifications to the experimental procedure were made when testing the addition of water, other solvents, and when no shale and/or solvent was used. When distilled water was added to the system, the initial gas pressure was decreased to compensate for the vapor pressure of the water during the run. This allowed the experimental runs to have a run pressure of 1100 to 1500 psig, using the tetralin solvent. The initial starting pressures for when water was added are in the Table 3. In the runs where water was added and tetralin was not the solvent used, the initial charging pressures contained in Table 3 were used. For all of the other modifications the changes were straight-forward.

TABLE 3

Initial Pressures When Water Added

| Amount of Water Added | Percent Water in Shale | Initial Charging Pressure |
|-----------------------|------------------------|---------------------------|
| 3.78 grams | 17.7 | 330 psig |
| 7.60 grams | 27.3 | 280 psig |
| 11.40 grams | 34.9 | 100 psig |

Gas Analysis

Reaction product gases were determined using a Carle Analytical Gas Chromatograph, model 111-H, in conjunction with a Hewlett-Packard, model 3390A integrator. The system was programmed to quantitatively determine the following components:

- | | |
|----------------------|----------------------|
| (1) hydrogen | (7) carbon dioxide |
| (2) propane | (8) ethylene |
| (3) propylene | (9) ethane |
| (4) isobutane | (10) argon |
| (5) hydrogen sulfide | (11) methane |
| (6) normal butane | (12) carbon monoxide |

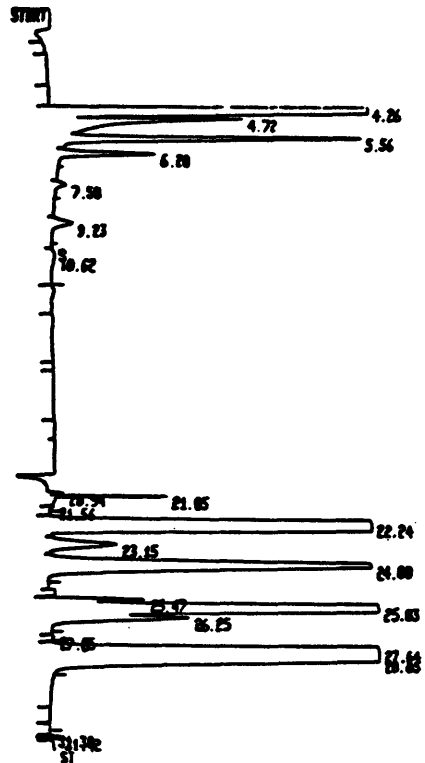
The integrator output identified the above components with corresponding peak areas and mole percentages. Figure 4

shows a typical print-out from the integrator. The integrator was calibrated each time with a multi-component calibration gas and response factors for each component determined. An example of a chromatograph calibration result is given in Table 4.

To analyze a gas sample, the sample cylinder was attached to the evacuated gas chromatograph system(37) shown in Figure 5. A gas sample was taken with an evacuated 500 cc stainless steel cylinder. The cylinder was then attached to the gas chromatograph system and the system purged with helium and the sample gas. The system pressure was increased to 40 inches of mercury and the sample vessel valve closed. The sample gas was then released into the system and analysis begun by simultaneously starting the chromatograph and integrator.

FIGURE 4

Gas Chromatograph Integrator Print-out



RUN 8 12 NOV/01/83 17:49:25

| RT | AREA | TYPE | CALC | AMOUNT |
|-------|------------|------|------|--------|
| 4.26 | 1.6187E+07 | SB | | 0.000 |
| 4.72 | 363998 | TBY | | 0.000 |
| 5.56 | 361600 | TV | 1R | 2.036 |
| 6.20 | 193550 | TP | 2R | 0.730 |
| 7.50 | 29411 | TB | 3R | 0.093 |
| 9.23 | 67754 | TB | 4R | 0.211 |
| 10.62 | 10063 | B | 5R | 0.050 |
| 20.94 | 233320 | BV | | 0.000 |
| 21.05 | 47043 | VB | | 0.000 |
| 21.56 | 1404 | BB | | 0.000 |
| 22.24 | 1.2519E+07 | BV | | 0.000 |
| 23.15 | 235300 | VV | 7R | 1.124 |
| 24.00 | 1703100 | VB | 8R | 7.655 |
| 25.47 | 145300 | PV | | 0.000 |
| 25.83 | 9114000 | VV | 9R | 57.100 |
| 26.25 | 392910 | VB | | 0.000 |
| 27.85 | 2755 | BB | | 0.000 |
| 27.64 | 4700600 | BV | 10R | 30.975 |
| 28.05 | 1374200 | VB | | 0.000 |
| 31.20 | 826 | D BB | | 0.000 |
| 31.42 | 3200 | PB | | 0.000 |

TOTAL AREA= 4.7904E+07
 MUL FACTOR= 1.0000E+00

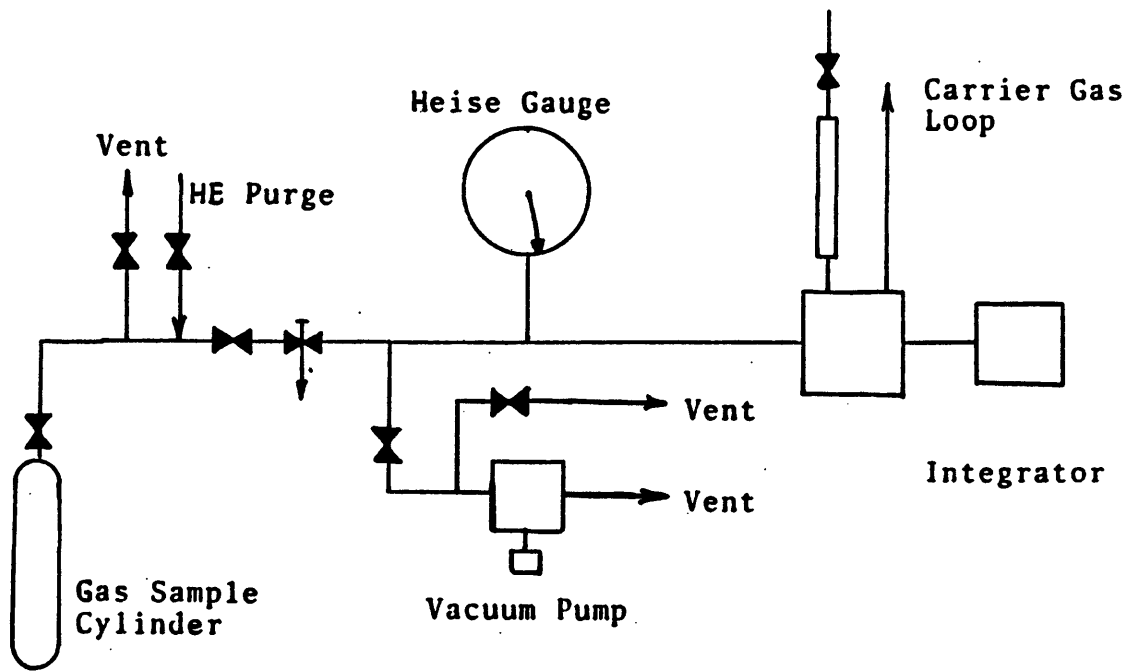
TABLE 4

Gas Chromatograph Calibration Results for RB-63

| Component | Area | Response Factor (x 10 ⁻⁷) | Mole Percent | Normalized Mole Percent |
|-------------------------------|----------|---|-----------------|-------------------------------|
| H ₂ | 16187000 | 61.4130 | 99.41 | 84.70 |
| C ₃ H ₈ | 561680 | 3.4141 | .19 | .16 |
| C ₃ H ₆ | 193550 | 3.5900 | .07 | .06 |
| i-C ₄ | 29411 | 2.9690 | .01 | .01 |
| H ₂ S | ----- | ----- | ----- | ----- |
| n-C ₄ | 67754 | 2.9390 | .02 | .02 |
| C ₄ ⁺ | 18063 | 2.6911 | .01 | .01 |
| CO ₂ | 12519000 | 6.1304 | 7.67 | 6.54 |
| C ₂ H ₄ | 235580 | 4.4925 | .11 | .09 |
| C ₂ H ₆ | 1703100 | 4.2329 | .72 | .61 |
| Ar | 9040147 | 5.8998 | 5.33 | 4.54 |
| CH ₄ | 4708600 | 6.1954 | 2.92 | 2.49 |
| CO | 1374200 | 6.5754 | .90 | .77 |

FIGURE 5

Gas Chromatograph System



Liquid Analysis

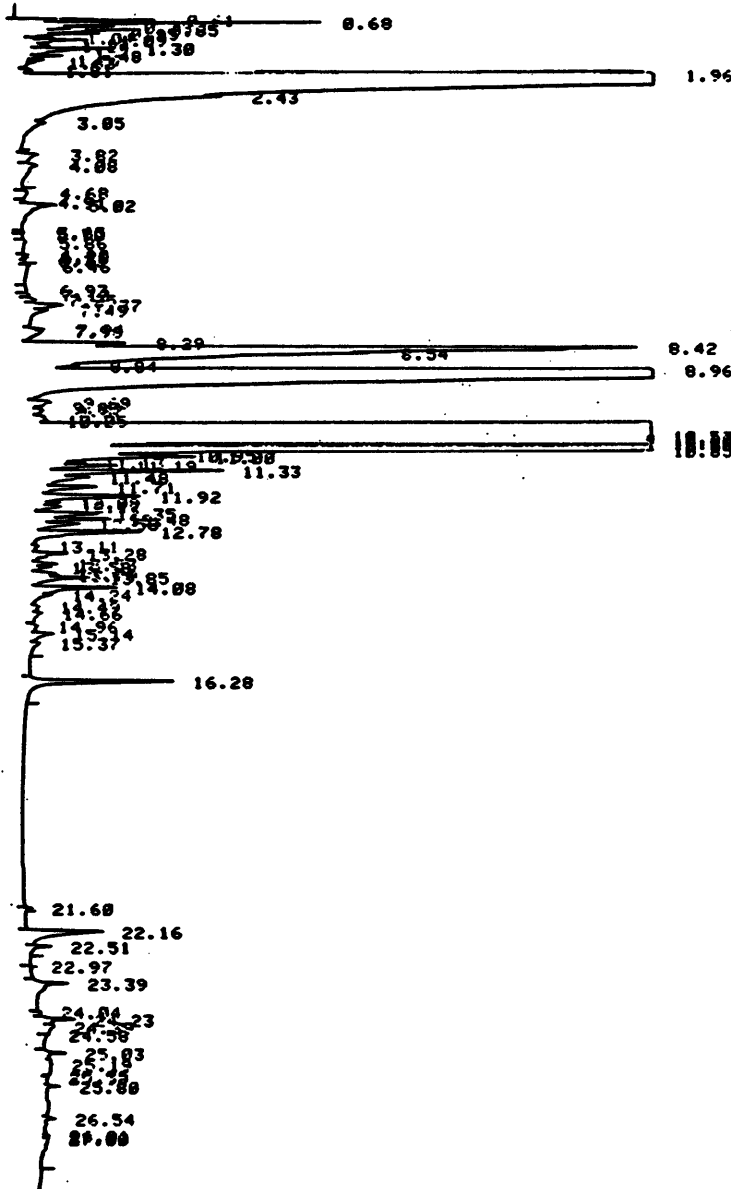
A Hewlett-Packard Model 5840A Gas Chromatograph was used for capillary column chromatographic analysis of the liquid products of reaction. This apparatus was equipped with a 15 m x 0.256 mm J & W fused silica capillary column with DB-5 methyl silicone liquid phase. The pressure drop across the column was 75 kPa and samples were run at a split ratio of 100:1. The gas chromatograph's output included a tabular summation of retention times and area percents. Being able to identify the retention times which identified tetralin and naphthalene, their respective area percents could be accurately determined. It was assumed that the area percents were equivalent to the relative volume percents, and that the total moles of tetralin and naphthalene did not change during the reaction. This allowed the increase in the mole fraction of naphthalene to be calculated. Figure 6 shows a typical chromatogram.

A number of product fractions were subjected to elemental analysis. A Carlo-Erba, model 1104 elemental analyzer was used to analyze for carbon, hydrogen, and nitrogen.

Samples of product oils from RB-45 through RB-51 runs were sent to the IRT Corporation, San Diego, California, for direct oxygen analysis. These particular samples were not

FIGURE 6

Liquid Gas Chromatogram



MP RUN 0 147
13:49
MPCR 1

| RT | AREA | AREA % |
|-------|---------|--------|
| 0.41 | 882 | 0.032 |
| 0.60 | 2493 | 0.097 |
| 0.70 | 328 | 0.012 |
| 0.82 | 1342 | 0.049 |
| 0.93 | 696 | 0.026 |
| 1.04 | 237 | 0.009 |
| 1.11 | 811 | 0.030 |
| 1.19 | 242 | 0.009 |
| 1.24 | 283 | 0.010 |
| 1.38 | 1324 | 0.049 |
| 1.48 | 544 | 0.020 |
| 1.57 | 241 | 0.009 |
| 1.63 | 344 | 0.013 |
| 1.81 | 246 | 0.009 |
| 1.94 | 170708 | 0.279 |
| 2.43 | 8492 | 0.320 |
| 3.03 | 1142 | 0.043 |
| 3.82 | 369 | 0.014 |
| 4.08 | 642 | 0.024 |
| 4.68 | 97 | 0.004 |
| 4.91 | 29 | 0.001 |
| 5.62 | 895 | 0.033 |
| 5.63 | 46 | 0.002 |
| 5.70 | 7 | 0.000 |
| 5.86 | 238 | 0.009 |
| 6.20 | 8 | 0.001 |
| 6.26 | 46 | 0.002 |
| 6.32 | 8 | 0.000 |
| 6.32 | 133 | 0.005 |
| 6.93 | 83 | 0.003 |
| 7.12 | 43 | 0.002 |
| 7.23 | 213 | 0.008 |
| 7.37 | 739 | 0.027 |
| 7.49 | 784 | 0.029 |
| 7.94 | 164 | 0.006 |
| 7.98 | 539 | 0.020 |
| 8.29 | 1498 | 0.055 |
| 8.42 | 13708 | 0.504 |
| 8.54 | 844 | 0.319 |
| 8.84 | 76 | 0.003 |
| 8.96 | 61048 | 2.246 |
| 9.49 | 442 | 0.016 |
| 9.83 | 84 | 0.003 |
| 9.87 | 468 | 0.017 |
| 10.05 | 383 | 0.014 |
| 10.53 | 1021000 | 37.559 |
| 10.60 | 504388 | 18.339 |
| 10.60 | 71808 | 2.621 |
| 10.85 | 154088 | 5.643 |
| 10.95 | 1833 | 0.007 |
| 11.00 | 3374 | 0.012 |
| 11.13 | 566 | 0.002 |
| 11.19 | 1438 | 0.005 |
| 11.33 | 2623 | 0.009 |
| 11.49 | 1681 | 0.006 |
| 11.71 | 1575 | 0.006 |
| 11.92 | 2388 | 0.009 |
| 12.00 | 492 | 0.002 |
| 12.17 | 738 | 0.003 |
| 12.33 | 1446 | 0.005 |
| 12.40 | 1844 | 0.007 |
| 12.50 | 1134 | 0.004 |
| 12.70 | 2493 | 0.009 |
| 13.11 | 328 | 0.012 |
| 13.20 | 1881 | 0.007 |
| 13.33 | 465 | 0.017 |
| 13.39 | 426 | 0.016 |
| 13.74 | 299 | 0.011 |
| 13.79 | 318 | 0.012 |
| 13.85 | 1189 | 0.044 |
| 14.00 | 1582 | 0.058 |
| 14.24 | 668 | 0.024 |
| 14.49 | 249 | 0.009 |
| 14.66 | 671 | 0.025 |
| 14.96 | 191 | 0.007 |
| 15.14 | 677 | 0.025 |
| 15.37 | 383 | 0.014 |
| 16.20 | 1981 | 0.073 |
| 21.68 | 26 | 0.001 |
| 22.16 | 1944 | 0.070 |
| 22.51 | 288 | 0.011 |
| 23.29 | 882 | 0.036 |
| 24.04 | 211 | 0.008 |
| 24.23 | 642 | 0.024 |
| 24.36 | 918 | 0.034 |
| 24.38 | 381 | 0.014 |
| 25.03 | 783 | 0.029 |
| 25.19 | 1348 | 0.049 |
| 25.35 | 69 | 0.003 |
| 25.50 | 278 | 0.010 |
| 25.88 | 842 | 0.031 |
| 26.54 | 1542 | 0.057 |
| 26.91 | 375 | 0.014 |
| 27.00 | 842 | 0.031 |

DIL FACTOR: 1.0000 E+0

washed with acetone, but were separated from the spent shale by centrifuging alone. The non-destructive oxygen analysis was done by neutron activation and these results are contained in Appendix B.

Additional analytical work was done by Huffman Laboratories, Wheat Ridge, Colorado. Samples RB-45 through RB-48 were analyzed for water content by the Karl-Fischer procedure. These particular results are contained in Appendix C. In addition, Huffman Laboratories did elemental analysis on the product oils from RB-59, RB-61, RB-62, and RB-63. Before these samples were analyzed, they were distilled in a Aldrich Kugelrohr Distillation Apparatus. The distillation was done after the acetone washing and Roto-vaping. This was in an attempt to remove all the acetone from the product oil. The technique was very effective.

Solid Analysis

From previous studies it was found that to maintain a 100% homogeneous feed stock was almost impossible. Consequently, both the feed and spent shale were analyzed for all elements and species of interest for each run.

The feed and spent shale were analyzed for total and inorganic carbon using a Coulometrics system. A total carbon analyzer and carbon dioxide coulometer, models 5020

and 5030, respectively, were used to determine the total carbon and inorganic carbon in a sample. The total carbon was determined by combusting the sample in oxygen and automatically titrating the evolved carbon dioxide. Inorganic carbon was determined in parallel by mildly heating a sample in the presence of a dilute perchloric acid solution to evolve all carbonates as carbon dioxide. The evolved carbon dioxide was then automatically titrated in the coulometer.⁽³⁸⁾ Knowing the total and inorganic carbon values, the organic carbon content of the oil shale sample was calculated by difference.

All shales were analyzed for ash content. The dried shale samples were ashed for 3 hours, at 750°C, in a muffle furnace. Ash content was calculated in accordance with ASTM procedure D3180.

As with the product oils of RB-45 through RB-51, the corresponding spent shale samples were analyzed for oxygen. The analysis was done by IRT Corporation in the same manner that the product oils were. Prior to shipping the samples, the spent shale was soxhlet extracted in hexane. The results of this analysis are contained in Appendix B.

DATA ANALYSIS

Analyzing the experimental data to reduce it to a form which would allow accurate interpretation of the results was an integral part of this study. This section gives a detailed description of the ash balances made, the development of the methods used to calculate the product yields, and the statistical method used for analyzing the duplicated experimental runs.

Ash Balances

Being able to account for all material entering and leaving the reaction system is always important in this type of research. However, due to experimental constraints, there were difficulties in recovering all of the spent shale. Even though the reactor was thoroughly washed with acetone after the reaction, residue still remained on the impeller, baffle, and in the reactor headset bolt holes. The residue problem was compounded in those experimental runs when there was no washing with acetone after the reaction.

As a result, a technique was needed to determine the weight of the spent shale. It has been shown during solvent extraction of oil from oil shale, that the ash content of the shale did not significantly change.⁽³⁹⁾ Therefore, ash was used as a tie element. Knowing the exact weight of the

feed stock, and the percent ash in both the feed and spent shale; the spent shale weight was calculated as follows:

$$\text{Wt. of S.S.} = ((\% \text{Ash in Feed}) \times (\text{Wt. Feed})) / (\% \text{Ash S.S.})$$

where,

S.S. = Spent Shale.

Organic Carbon Conversion

The fraction of kerogen extracted from oil shale which went to oil and gas is referred to as organic carbon conversion. As indicated in the ash balance analysis section, recovery difficulties precluded direct spent shale weight determination. However, employing the ash balance to calculate final spent shale weight allowed accurate calculation of organic carbon remaining in the shale. The organic carbon conversion (OCC) calculation method was as follows:

$$\text{OCC} = M_F \times (\% \text{OC}_F - (\% \text{OC}_{SS} \times (\% \text{Ash}_F / \% \text{Ash}_{SS}))) / ((\% \text{OC}_F) \times M_F)$$

where,

M_F = mass of the feed,

$\% \text{OC}_F$ = % organic carbon in the feed,

$\% \text{OC}_{SS}$ = % organic carbon in the spent shale,

$\% \text{Ash}_F$ = % ash in the feed,

$\% \text{Ash}_{SS}$ = % ash in the spent shale.

Hydrogen Consumption and Yield Calculations

To determine the amount of hydrogen consumed in the batch reactor system, it was necessary to measure the number of moles of hydrogen present in the system before and after the hydrogenation reactions occur. This measurement was made by determining the composition of the gaseous atmosphere before and after the reaction through the use of a gas chromatograph, and then applying this information to the total number of moles present in the reactor system. This latter value was directly determined by the Ideal Gas Law from information about reactor temperatures and pressures. Argon, a gas inert to the reactions occurring in the system, was used as a tie element between the starting and ending gas compositions. By knowing the relative amounts of hydrogen and argon in the initial gas mixture and then comparing this to the relative amounts in the exiting gas mixture, it was possible to accurately calculate the number of moles of hydrogen consumed in the process. Example 1 in Appendix D illustrates the calculational procedures used. In addition, example 4 shows how the percent hydrogen consumption and consumption rate (SCF/BBL) were calculated.

This calculated hydrogen consumption reflects the total hydrogen consumed, including hydrogen to hydrocarbon gases,

hydrogen added to the oil, and hydrogen left on the spent shale. This value does not reflect a maximum or minimum consumption, but rather an average value for hydroprocessing under the particular conditions of temperature, pressure, and residence time.

An excellent gauge to the success of the thermal solution is the organic carbon conversion, coupled with the percent carbon converted to oil. The percent carbon converted to oil in this study is called "percent oil yield." Example 2 in Appendix D illustrates how the percent oil yield was calculated.

Appendix D contains the various examples of the calculations needed for this research. These mathematical calculations were programmed on an Apple II-plus personal computer to do the large number of repetitive calculations needed.

Statistical Analysis

The experimental and analytical procedures established for this research were designed to yield reproducible values for oil yield, organic carbon conversion, and hydrogen consumption. In order to ascertain that the results presented were accurately developed, a check on the reproducibility of the data was required. Since reaction temperature and residence time were constant, the analysis

had to be based on groups of data with the same solvent and additives. It was felt, for this particular study, that the mean was the "best" value to quote for the various quantities desired - accompanied with the respective standard deviation.

TABLE 5
Repetitive Experimental Run Results

| Solvent Added to Shale | Amount of H ₂ O Added (gr.) | Oil Yield (%) | Organic C Conversion (%) | Hydrogen Consumption (SCF/BBL) | No. of Runs |
|------------------------|--|---------------|--------------------------|--------------------------------|-------------|
| Tetralin | None | 93.52± .53 | 87.01±2.80 | 1422±175 | 7 |
| Tetralin | 3.78 | 91.30±1.54 | 87.11± .40 | 599±224 | 4 |
| Toluene | None | 94.22± .77 | 90.40±4.44 | 930± 56 | 3 |
| Toluene | 7.60 | 89.89±3.18 | 74.71± .91 | 201± 50 | 3 |

TABLE 6
Repetitive Experimental Run Results
for Pure Hydrogen

| How the Oil Shale was Prepared | Organic Carbon Conversion (%) | Number of Runs |
|--------------------------------|-------------------------------|----------------|
| Oven Dried | 87.52 ± 1.34 | 4 |
| Vacuum Dried | 89.04 ± 2.61 | 4 |

As can be deduced from Table 5 and Table 6, the errors associated with the oil yield and organic carbon conversion were very small. The error associated with the hydrogen consumption was relatively small, except for the tetralin runs with water addition. It was therefore concluded that the experimental runs produced reproducible results.

DISCUSSION OF RESULTS

Introduction

This section presents a critical discussion of the results obtained in this study. A brief discussion of the experimental data excluded from the statistical analysis is given initially, followed by a discussion of the processing errors. The final portion of this section presents the results of this research and critically evaluates them.

Excluded Data

The initial emphasis of this study was concerned with developing an experimental procedure which gave reproducible results. In developing this procedure, experimental data were generated which for various reasons could not be included. Those experiments which had a problem during the run were stopped at that point. The following were the only reasons why an experimental run would be stopped before completion:

- (1) greater than $+5^{\circ}\text{C}$ overshoot of the desired reaction temperature when heating the system up;
- (2) pressure leak during the run;
- (3) unable to maintain the reaction temperature, during the run, to within $\pm 5^{\circ}\text{C}$ of the designated 425°C ;

- (4) equipment failure during any portion of the experiment; and
- (5) discrepancies in the calculated product yields which could not be logically explained.

If a run could not be completed for one of the above reasons, it was repeated in its entirety. It should be pointed out, that runs were not repeated just because their results did not agree with the expected results. There had to exist a significant discrepancy or equipment failure to disregard the experimental run.

Those experimental results reported in Appendix A of this document met the run criteria set, and did not exhibit any unusual discrepancies in the calculated results.

Processing Errors

There were a large number of sources of error which are common to research programs of this type. The process variables contributing to most of the experimental errors were temperature, pressure, and reaction time.

The most important effect was fluctuations in the reaction temperature. When water was not added to the various systems, heat-up to reaction temperature took between 20 to 30 minutes using the tetralin solvent. For the other solvents the heat-up time was 30 to 40 minutes. When the internal system temperature approached 400°C, the

controller set point was decreased to allow the reaction temperature to plateau at 425°C. Approximately two to five minutes were required to establish the desired reaction temperature. Subsequently, the temperature oscillated around the set point with an amplitude of $\pm 5^{\circ}\text{C}$, for a period of 10 minutes using the first controller (see apparatus section.) The oscillation was due to the mass of the reactor which produced a lag in the control system with respect to the surface temperature of the heater and temperature sensed by the thermocouple in the reactor. With the modifications made of the new controller and internal cooling via cooling coils, the maximum oscillation was kept to $\pm 2^{\circ}\text{C}$. This oscillation decreased to $\pm 1^{\circ}\text{C}$ within five minutes.

Whenever water was added to the reaction mass, the reaction temperature was much harder to control. In the vicinity of 400°C, the reaction would increase very rapidly to 425°C and overshoots in temperature of greater than 10°C were commonly experienced. To insure the maximum $+5^{\circ}\text{C}$ overshoot, tremendous care had to be exercised in the latter portions of the heat-up period. The same magnitude of oscillation was observed as in the non-water addition cases, when the reaction temperature was approached correctly. Another problem was that the reaction temperature could be

quenched by more than 5°C within 30 seconds when using the internal cooling system.

The pressure set for the tetralin solvent runs was 1500 psig. For the base case runs without water addition, the pressure deviated less than 5%. However, when water was added the pressure deviated as much as 20%. This was due to the variations in the amount of water added, and the technique used to approximate the initial cold reactor gas pressure. The effect of the pressure variation on this study is unknown; however, it is felt to have had minimal effect since all experimental runs had similar variations and the 60 minute base case runs were reproducible to within +1.54% oil yield.

The residence time of the shale was defined as the interval between reaching the reaction temperature and the cooling of the reactor to quench the reaction. This interval was timed fairly accurately (± 10 seconds.) The major cause of any variation was the time required to manually lower the heater, turn on the cooling water, turn off the heater, and turn on the cooling fan.

Effect of Drying on Conversion

Samples of beneficiated Stuart A shale were subjected to drying by two different means in order to assess the effect of drying mechanisms on shale reactivity. One batch of

shale was atmospherically dried in an oven at 95°C, and the dry shale was then stored in the oven until used. A second batch was vacuum dried at 40°C, and greater than 3 mm Hg total pressure. The vacuum dried shale was stored at room temperature in a vacuum dessicator until used. Both samples were hydrogenated at 420°C, and 1500 psig total pressure (H₂ atmosphere with a tetralin solvent.) Results of these experiments are shown in Table 6. As may be seen, the shale activity was essentially unchanged by drying technique when hydrogenation was used to extract shale oil.

Hydrogen Consumption Results for Tetralin

Results of hydrogen consumption and yield structure calculations for base case conditions (tetralin, indigenous water content of 5%, 1500 psig total pressure, 425°C, one hour reaction time, 2/1 solvent-to-shale) are shown in Table 5. Test results showed that for the base case, the hydrogen consumption corresponded to approximately 1422 ± 174 SCF/BBL of oil product. The reproducibility of the determination was excellent, reflecting the overall reliability of the run procedure and chromatography techniques. It must be stressed that this level of hydrogen consumption reflects the total hydrogen consumed, including hydrogen to hydrocarbon gases, hydrogen added to the oil, and hydrogen left on the spent shale. This value reflects an average

consumption for hydroprocessing under these conditions of temperature, pressure, and residence time. Therefore, it would be logical that extraction at lower residence times would tend to lower this value. Conversely, increased residence time favors cracking, increased gas make, and higher hydrogen consumptions. Frank⁽⁴⁰⁾, has demonstrated that lower residence times can achieve the same organic carbon conversion, and hydrogen utilization at shorter times (e.g. 20 minutes) should be markedly better.

As discussed in the experimental design section, it was assumed that the tetralin would not exhibit those properties associated with supercritical solvents. As a pure solvent tetralin has a critical temperature of 446°C and a critical pressure of 35 atm (510 psi).⁽³³⁾ It is obvious that our reaction temperature is very close to the critical temperature. In addition, the critical pressure and temperature of the hydrogen gas used to pressurize the system is much lower than that for tetralin. This combination would cause a net decline in the critical properties of the total system. However, it is possible that this effect was counterbalanced by the shale oil in the tetralin/hydrogen solution. To clarify the question, the appropriate phase-equilibrium research is needed to study this particular set of conditions and determine whether or

not the system is supercritical.

The desire to use a subcritical solvent has some very positive aspects. If the thermal solution of oil shale was found to be economical; the subcritical solvent would probably demonstrate the minimal effects. Supercritical solvents are very useful. In fact, they have certain physical properties which make them very attractive to use in thermal solution.

Water Addition Results for Tetralin

There were seven experimental runs with water added to the vacuum dried shale. The indigenous water content of the "dried" shale (dried at a total pressure of 3 mm Hg, 40°C) is 5.2% water by weight. This water content reflects the water associated with the minimal matrix as hydrates, and represents water which can not be removed by conventional drying procedures. Runs RB-12 through RB-17 were performed to determine the change in conversion, yield structure, and hydrogen consumption as a function of water addition to the tetralin/oil slurry. For runs RB-12 through RB-15, the water content of the shale was increased to 17.7% by weight. For run RB-16, the water content of the shale was increased to 27.3% by weight. RB-17's water content was increased to 34.9%. Although the results were somewhat variable for the 17.7 % runs, as illustrated in Table 5, the average hydrogen

consumption for these runs was approximately 50% lower than the "base case" hydrogen consumption with no added water. This decrease in net consumption was realized without substantially changing the total conversion of organic carbon, or the yield structure of the process. There was only one run each, with 27.3% and 34.9% water by weight. For the 27.3 % run, the oil yield was 93.1% at an organic carbon conversion of 86.7% and a hydrogen consumption of 315 SCF/BBL. For the 34.9% run, the oil yield was 91.5% at an organic carbon conversion of 85.7% and a hydrogen consumption of 213 SCF/BBL. Even though only one run was done for each condition, the hydrogen consumption indicated by this data point is decreased by approximately 80% from the base case runs.

The results from these particular runs indicate that significant savings in operating cost for hydrogen manufacture may be realized by simply processing a wet shale.

Non-donor Solvent Results

Table 5 illustrates that the organic carbon conversion, oil yield, and hydrogen consumption of toluene supercritical extraction parallel the results for the tetralin runs. A notable exception was the 15% reduction in organic carbon conversion when water was added to the toluene system. This

relatively lower conversion may be due to water addition, or may simple be an artifact as some difficulty in temperature control was found during the water addition to toluene runs.

The results of the other non-donor solvent (1-methylnaphthalene) generated similar results as tetralin and toluene. As with the toluene runs, there was a significant decrease in the organic carbon conversion and hydrogen consumption when water was added. In addition, there was more hydrogen produced than consumed. There was only one run with water and one without for this solvent, so the value for each respective hydrogen consumption must be scrutinized very carefully. However, it can be concluded the same phenomenon of decreased hydrogen consumption with water addition was present in the non-donor solvent systems as in the donor solvent system.

TABLE 7

1-Methylnaphthalene Results

| Amount of Water Added (grams) | Oil Yield (%) | Organic Carbon Conversion (%) | Hydrogen Consumption (SCF/BBL) |
|-------------------------------------|------------------|-------------------------------------|--------------------------------------|
| None | 85.95 | 89.10 | 1545 |
| 7.60 | 89.95 | 77.15 | -463 |

It is interesting to note in the tetralin, toluene, and 1-methylnaphthalene runs the selectivity for oil formation was high (approximately 90%) when hydrogen was present in either donatable form from the solvent, or from the reaction gas atmosphere. This was as expected. Baldwin et al.(41), have postulated that the oil forming reactions most probably proceed via free radical chemistry, involving homolysis of bonds in the kerogen matrix resulting in the formation of high molecular weight free radical species. Increasing reaction temperature accelerates the rate of the bond scission reactions, promoting oil formation. Once formed, the free radical species can react in a number of ways; cracking, condensation/polmerization, or by hydrogen abstraction and "capping" in the presence of molecular hydrogen or donatable hydrogen from a hydroaromatic in the shale oil or solvent. The extent to which each of these reactions proceed dictates the ultimate product distribution of oil, gas, and unreacted carbonaceous residue.

Table 8 contains the summary of the heavy gas solvent runs. Again, the trends parallel those runs using toluene and tetralin. The organic carbon conversion and oil yield were not as great as with the other solvents, however these results are still better than the standard retorting process. There was a dramatic decrease in hydrogen

consumption when using this natural solvent and water addition.

TABLE 8
Heavy Gas Oil Results

| Amount of Water Added (grams) | Oil Yield (%) | Organic Carbon Conversion (%) | Hydrogen Consumption (SCF/BBL) |
|-------------------------------|---------------|-------------------------------|--------------------------------|
| None | 82.71 | 72.98 | 2575 |
| 7.60 | 83.69 | 74.80 | 628 |

The tetralin, toluene, and 1-methylnaphthalene solvent runs showed a greater selectivity for oil production versus gas generation, then the natural solvent runs.

Again, Baldwin et al.⁽⁴¹⁾ have postulated that when non-donor solvents are used as the reaction vehicle, hydrogen transfer to reactive species could only be accomplished by hydrogen shuttling rather than direct hydrogen transfer from the solvent. Due to the relative bond strengths of the C-H bond in tetralin and the H-H bond in molecular hydrogen, the rate of hydrogen abstraction and thus the rate of the free radical "capping" reactions will be slower in a non-donor solvent. Hence, free radical

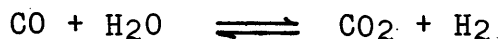
cracking can proceed to a greater extent in the absence of hydrogen donors, leading to increased hydrocarbon gas make and poorer hydrogen management efficiency.

Water Effect Mechanism

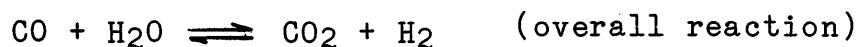
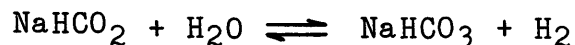
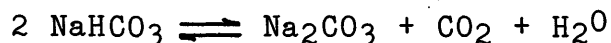
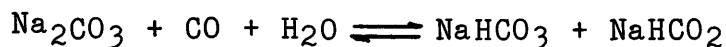
Experimental runs were conducted to determine the hydrogen production mechanism and the role each constituent in the reacting system.

Possible mechanisms for the observed phenomenon include:

- (1) the water/gas shift reaction



- (2) reactions of carbonate minerals in the shale
(apparent shift reaction)



- (3) hydrogen production due to reactions of the solvent and/or shale oil with water.

The homogeneous water gas shift reaction was investigated in runs RB-26 and 28. Run RB-26 involved merely reacting CO and water at typical reaction conditions in the batch reactor. As indicated by the run summary in Appendix A, no significant hydrogen production was noted.

Run RB-28 was designed to determine whether the shift reaction could be catalyzed by inorganics present in spent shale. The results showed that no significant reaction took place under the set reaction conditions. These two observations, coupled with the fact that the CO₂ concentration in the product gas was not sufficient to explain the increased gaseous hydrogen produced in runs with added water seemed to rule out the shift reaction. However, if either the organics (shale oil or solvent) or the inorganics are acting as CO₂ acceptors, then the shift reaction could still be the source of increased hydrogen from the system.

It was concluded from the results of the non-donor solvents and runs RB-24 and 25, that raw shale plays a role in the hydrogen phenomenon in this particular reaction system. RB-24 and 25 were conducted with 1-methylnaphthalene, without and with water addition respectively, helium atmosphere, and no shale. The result from these two runs was that no hydrogen was produced with the addition of water. Since no hydrogen could be consumed in this system; there should have been a significant production of hydrogen if the hydrogen phenomenon was independent of the shale.

Tests were also made in a helium atmosphere. Runs RB-

22, 23, and 27 were made to determine if there was a significant increase in hydrogen production from tetralin with water addition in the absence of shale and hydrogen. The results showed that no significant production occurred, again indicating that the shale plays an important role, not only as a hydrogen acceptor but as a participant in the hydrogen production mechanism.

This was confirmed in runs RB-30 and 31, in which the system was run with helium and shale, both with and without water addition. Without water addition (RB-30), hydrogen consumption from the solvent was substantial but there was no hydrogen gas make. In the other run with water, hydrogen consumption from the solvent remained high but hydrogen gas make increased significantly -- indicating the presence of the hydrogen production reaction. The hydrogen gas make for RB-31 was considerably lower than in RB-16, in which there was an initial hydrogen atmosphere. This would seem to indicate that rather than inhibiting the production of hydrogen, a high hydrogen partial pressure actually may enhance production.

Two runs were designed to determine the effect of shale in the hydrogen formation reactions of the donor solvent system (tetralin). One of the runs RB-29 (no shale present and 27.3% water added) produced no significant amounts of

hydrogen, even though there were no hydrogen acceptors present. This reconfirmed the results of RB-22, 23, and 27; and paralleled similar non-donor solvent runs. When spent shale was the feed for RB-20, the hydrogen gas make increased substantially as did the hydrogen consumption from the solvent. Compared to other tetralin runs, the gas make was greater than previously noted. This can be explained by the fact that even though the hydrogen forming reactions were still taking place, far fewer hydrogen acceptors were present in the form of shale organics.

To investigate the possibility the hydrogen phenomenon was caused by the carbonates present in the raw shale, two "decarbonated" shale experiments were done. The runs were with water (RB-32) and without water (RB-34) addition on decarbonated shale that had been extracted with perchloric acid. The lack of carbonates did cause the hydrogen production phenomenon to occur when water was added. Therefore, this ruled out the apparent shift reaction, involving carbonates in the shale, as the source of hydrogen generation. A major effort was taken to investigate the fate of the oxygen liberated by water during the reaction. The analytical results for water and oxygen concentration of RB-47 (tetralin solvent and no water addition) and RB-48 (tetralin solvent and water addition) are contained in

Appendices B and C. As shown, the oxygen contents of the respective spent shales were approximately equal. The magnitude of the oxygen content in the oils where the hydrogen production occurred was insufficient to account for the magnitude of excess hydrogen found. Taking into account the amount of water present in each sample, there was not an increase in the amount of oxygen present in the oil. In fact, there was a slight decrease. These results rule out the spent shale and product oil as oxygen acceptors.

Another possible fate for the liberated oxygen was in the additional production of carbon oxide gases. Material balances were made on the gaseous products from runs made with and without water addition. These results are shown in Table 9. Carbon monoxide decreases slightly, whereas carbon dioxide make increases slightly with the addition of water. However, the changes were insufficient to account for the oxygen liberated from the added water.

It appears that the mechanism for the hydrogen production phenomenon is much more complex than simply the water shift reaction or solvent dehydrogenation enhanced by water addition. In all probability, the mechanism involves many of the system components either directly or as a catalyst.

TABLE 9
Carbon Oxide Calculation Results

| Run Number | Amount of Water Added (grams) | CO (g-moles) | CO ₂ (g-moles) |
|---------------|-------------------------------------|-----------------|------------------------------|
| GB-2 thru 7 | None | .0045 | .0155 |
| RB-12 thru 15 | 3.78 | .0032 | .0191 |
| RB-16 | 7.60 | .0031 | .0184 |
| RB-17 | 11.40 | .0030 | .0218 |

CONCLUSIONS

The conclusions which follow pertain exclusively to the experimental system employed for this research. These conclusions were drawn from the results of this study.

- (1) This research has reconfirmed some of the major advantages of the thermal solution of oil shale vs. conventional pyrolysis. First, organic carbon conversions of 85% (plus) are readily attained at temperatures substantially below retorting temperatures. Secondly, the process selectivity to oil is much greater, with approximately 90% of the total carbon converted going to the formation of an oil product.
- (2) When the hydrogen donor solvent (tetralin) was employed, there was a decrease of 50% in the average total hydrogen consumption when water was added to the system. Organic carbon conversion and oil yield remained, within the accuracy of the analytical methods employed, approximately equal whether or not water was added.
- (3) When the non-donor solvent (toluene) was used, the average total hydrogen consumption decreased 78% when water was added. Unlike when the tetralin was used, there was a decrease of 15% in

the organic carbon conversion when water was added. The oil yield was about equal with or without water.

- (4) The organic carbon conversion for the base case runs of tetralin and toluene, were approximately equal and greater than 90%. Comparing the oil yields; the toluene runs were slightly higher than the tetralin runs (90% and 87% respectively.) The hydrogen consumption for the toluene runs was less than the tetralin runs.
- (5) Shale activity was essentially unchanged by drying technique when hydrogenation was used to extract shale oil.
- (6) When a natural solvent, heavy gas oil, was used as the reaction solvent; the conversion, yield structure, and hydrogen consumption paralleled the results from the hydrogen donor and non-donor solvent runs.

RECOMMENDATIONS

(1) Verify whether or not tetralin is supercritical at the conditions that the experimental runs were made at. This should be done by conducting enthalpy measurements on the tetralin/shale mixture, pressurized with hydrogen.

(2) Use deuterium oxide (D_2O) in place of the water additive, and study its effect. If the deuterium atom goes into the liquid phase, this will prove that some mechanism similar to the shift reaction is occurring and it is having a positive effect. Should the majority of the deuterium atom go into the gas phase, then the effect of adding water would be counterproductive. This recommendation is predicated on the assumption that deuterium oxide will act like the water and will not complicate the situation by additional reactions.

(3) Should the second recommendation prove positive, then further experimentation should be investigated using a continuous flow reactor system. The experimentation should concentrate on oil shales with different water content, operation at lower hydrogen partial pressures and lower residence times. The goal would be to determine the lowest possible operating conditions, while maintaining high organic carbon conversions and oil yields, and lowering total hydrogen consumption in the system.

(4) The research performed for this study should be expanded to include other types of oil shales (i.e. humic shale) from various parts of the world.

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APPENDIX A

RAW DATA

Run Number: RB-2

Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Oven Dried) |
| Atmosphere: | H ₂ | |
| Initial Gas Pressure: | 800 psig | Solvent: Tetralin |
| Initial Temperature: | 28 C | |
| Reaction Time: | 60 min. | Additive: None |
| Reaction Temperature: | 420 C | |
| Maximum Pressure : | 1410 psig | |
| Final Temp. & Press.: | 33 C - 770 psig | |
| Gas Temp. & Press.: | 31 C - 220 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 60.69 | 24.94 | .31 | 24.64 |
| Spent | 88.39 | 5.08 | .05 | 5.03 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|-----------------------------|-----------|---------------------------|---------------|
| Total H Consumed: | ----- gr. | CO:CO ₂ Ratio: | 30.21 % |
| H Consumed in Gas : | ----- gr. | Oil Yield: | ----- % |
| H Consumed in Sol.: | ----- gr. | Org. C Conv.: | 85.99 % |
| C in CO _x Gases: | ----- gr. | H Consumed: | ----- % |
| C in Total Gases: | ----- gr. | H Consumed: | ----- SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|-------------------------------|---------------|-------------------------------|---------------|
| H ₂ | 95.136 | C ₃ H ₈ | .098 |
| C ₃ H ₆ | .056 | i-C ₄ | .006 |
| n-C ₄ | .010 | C ₄ + | ----- |
| CO ₂ | 2.824 | C ₂ H ₄ | ----- |
| C ₂ H ₆ | .238 | Ar | ----- |
| CH ₄ | .781 | CO | .853 |

Additional Information: Mole Frac. Naphthalene in: -----
Mole Frac. Naphthalene out: -----

Run Number: RB-3

Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Vacuum Dried) |
| Atmosphere: | H ₂ | |
| Initial Gas Pressure: | 800 psig | Solvent: Tetralin |
| Initial Temperature: | 27 C | |
| Reaction Time: | 60 min. | Additive: None |
| Reaction Temperature: | 420 C | |
| Maximum Pressure : | 1445 psig | |
| Final Temp. & Press.: | 31 C - 750 psig | |
| Gas Temp. & Press.: | 30 C - 225 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 58.86 | 25.08 | .31 | 24.77 |
| Spent | 88.37 | 5.57 | .06 | 5.51 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|-----------------------------|-----------|---------------------------|---------------|
| Total H Consumed: | ----- gr. | CO:CO ₂ Ratio: | 21.98 % |
| H Consumed in Gas : | ----- gr. | Oil Yield: | ----- % |
| H Consumed in Sol.: | ----- gr. | Org. C Conv.: | 85.20 % |
| C in CO _x Gases: | ----- gr. | H Consumed: | ----- % |
| C in Total Gases: | ----- gr. | H Consumed: | ----- SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|-------------------------------|---------------|-------------------------------|---------------|
| H ₂ | 95.390 | C ₃ H ₈ | .070 |
| C ₃ H ₆ | .028 | i-C ₄ | .004 |
| n-C ₄ | .008 | C ₄ + | ----- |
| CO ₂ | 2.835 | C ₂ H ₄ | .024 |
| C ₂ H ₆ | .234 | Ar | ----- |
| CH ₄ | .784 | CO | .623 |

Additional Information: Mole Frac. Naphthalene in: -----
Mole Frac. Naphthalene out: -----

Run Number: RB-4

Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Oven Dried) |
| Atmosphere: | H2 | |
| Initial Gas Pressure: | 800 psig | Solvent: Tetralin |
| Initial Temperature: | 30 C | |
| Reaction Time: | 60 min. | Additive: None |
| Reaction Temperature: | 420 C | |
| Maximum Pressure : | 1425 psig | |
| Final Temp. & Press.: | 37 C - 775 psig | |
| Gas Temp. & Press.: | 35 C - 220 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 60.69 | 24.94 | .31 | 24.64 |
| Spent | 90.62 | 4.36 | .05 | 4.31 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|---------------------|-----------|---------------|---------------|
| Total H Consumed: | ----- gr. | CO:CO2 Ratio: | 30.50 % |
| H Consumed in Gas : | ----- gr. | Oil Yield: | ----- % |
| H Consumed in Sol.: | ----- gr. | Org. C Conv.: | 88.27 % |
| C in COx Gases: | ----- gr. | H Consumed: | ----- % |
| C in Total Gases: | ----- gr. | H Consumed: | ----- SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 94.822 | C3H8 | .086 |
| C3H6 | .045 | i-C4 | .006 |
| n-C4 | .013 | C4+ | ----- |
| CO2 | 3.095 | C2H4 | .027 |
| C2H6 | .237 | Ar | ----- |
| CH4 | .781 | CO | .853 |

Additional Information: Mole Frac. Naphthalene in: -----
Mole Frac. Naphthalene out: -----

Run Number: RB-5Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Vacuum Dried) |
| Atmosphere: | H ₂ | |
| Initial Gas Pressure: | 800 psig | Solvent: Tetralin |
| Initial Temperature: | 19 C | |
| Reaction Time: | 60 min. | Additive: None |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1455 psig | |
| Final Temp. & Press.: | 34 C - 735 psig | |
| Gas Temp. & Press.: | 31 C - 204 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 58.86 | 25.08 | .31 | 24.77 |
| Spent | 91.76 | 3.58 | .08 | 3.50 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|-----------------------------|-----------|---------------------------|---------------|
| Total H Consumed: | ----- gr. | CO:CO ₂ Ratio: | 34.74 % |
| H Consumed in Gas : | ----- gr. | Oil Yield: | ----- % |
| H Consumed in Sol.: | ----- gr. | Org. C Conv.: | 90.94 % |
| C in CO _x Gases: | ----- gr. | H Consumed: | ----- % |
| C in Total Gases: | ----- gr. | H Consumed: | ----- SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|-------------------------------|---------------|-------------------------------|---------------|
| H ₂ | 94.404 | C ₃ H ₈ | .203 |
| C ₃ H ₆ | .109 | i-C ₄ | .014 |
| n-C ₄ | .020 | C ₄ + | ----- |
| CO ₂ | 2.861 | C ₂ H ₄ | .033 |
| C ₂ H ₆ | .350 | Ar | ----- |
| CH ₄ | 1.012 | CO | .994 |

Additional Information: Mole Frac. Naphthalene in: -----
Mole Frac. Naphthalene out: -----

Run Number: RB-6

Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Vacuum Dried) |
| Atmosphere: | H ₂ | |
| Initial Gas Pressure: | 800 psig | Solvent: Tetralin |
| Initial Temperature: | 23 C | |
| Reaction Time: | 60 min. | Additive: None |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1445 psig | |
| Final Temp. & Press.: | 29 C - 710 psig | |
| Gas Temp. & Press.: | 27 C - 210 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 58.86 | 25.08 | .31 | 24.77 |
| Spent | 90.69 | 4.02 | .10 | 3.92 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | e§ | | | | |

Calculated Yields

| | | | |
|-----------------------------|-----|---------------------------|---------|
| Total H Consumed: | gr. | CO:CO ₂ Ratio: | % |
| H Consumed in Gas : | gr. | Oil Yield: | % |
| H Consumed in Sol.: | gr. | Org. C Conv.: | % |
| C in CO _x Gases: | gr. | H Consumed: | % |
| C in Total Gases: | gr. | H Consumed: | SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|-------------------------------|---------------|-------------------------------|---------------|
| H ₂ | | C ₃ H ₈ | |
| C ₃ H ₆ | | i-C ₄ | |
| n-C ₄ | | C ₄ + | |
| CO ₂ | | C ₂ H ₄ | |
| C ₂ H ₆ | | Ar | |
| CH ₄ | | CO | |

Additional Information: Mole Frac. Naphthalene in:
Mole Frac. Naphthalene out:

Run Number: RB-7

Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Vacuum Dried) |
| Atmosphere: | H ₂ | |
| Initial Gas Pressure: | 800 psig | Solvent: Tetralin |
| Initial Temperature: | 20 C | |
| Reaction Time: | 60 min. | Additive: None |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1505 psig | |
| Final Temp. & Press.: | 43 C - 815 psig | |
| Gas Temp. & Press.: | 41 C - 225 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 58.86 | 25.08 | .31 | 24.77 |
| Spent | 90.90 | 3.81 | .10 | 3.71 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|-----------------------------|-----------|---------------------------|---------------|
| Total H Consumed: | ----- gr. | CO:CO ₂ Ratio: | 33.23 % |
| H Consumed in Gas : | ----- gr. | Oil Yield: | ----- % |
| H Consumed in Sol.: | ----- gr. | Org. C Conv.: | 90.29 % |
| C in CO _x Gases: | ----- gr. | H Consumed: | ----- % |
| C in Total Gases: | ----- gr. | H Consumed: | ----- SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|-------------------------------|---------------|-------------------------------|---------------|
| H ₂ | 94.800 | C ₃ H ₈ | .136 |
| C ₃ H ₆ | .046 | i-C ₄ | .010 |
| n-C ₄ | .023 | C ₄ + | ----- |
| CO ₂ | 2.820 | C ₂ H ₄ | ----- |
| C ₂ H ₆ | .320 | Ar | ----- |
| CH ₄ | .908 | CO | .937 |

Additional Information: Mole Frac. Naphthalene in: -----
Mole Frac. Naphthalene out: -----

Run Number: RB-8

Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Oven Dried) |
| Atmosphere: | H2 | |
| Initial Gas Pressure: | 800 psig | Solvent: Tetralin |
| Initial Temperature: | 23 C | |
| Reaction Time: | 60 min. | Additive: None |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1500 psig | |
| Final Temp. & Press.: | 22 C - 735 psig | |
| Gas Temp. & Press.: | 22 C - 220 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 60.69 | 24.94 | .31 | 24.64 |
| Spent | 90.58 | 4.11 | .05 | 4.06 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|---------------------|-----------|---------------|---------------|
| Total H Consumed: | ----- gr. | CO:CO2 Ratio: | 33.54 % |
| H Consumed in Gas : | ----- gr. | Oil Yield: | ----- % |
| H Consumed in Sol.: | ----- gr. | Org. C Conv.: | 88.96 % |
| C in COx Gases: | ----- gr. | H Consumed: | ----- % |
| C in Total Gases: | ----- gr. | H Consumed: | ----- SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 93.959 | C3H8 | .083 |
| C3H6 | .041 | i-C4 | .004 |
| n-C4 | .011 | C4+ | ----- |
| CO2 | 3.551 | C2H4 | .027 |
| C2H6 | .284 | Ar | ----- |
| CH4 | .851 | CO | 1.191 |

Additional Information: Mole Frac. Naphthalene in: -----
Mole Frac. Naphthalene out: -----

Run Number: RB-9

Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Oven Dried) |
| Atmosphere: | H ₂ | |
| Initial Gas Pressure: | 800 psig | Solvent: Tetralin |
| Initial Temperature: | 23 C | |
| Reaction Time: | 60 min. | Additive: None |
| Reaction Temperature: | 420 C | |
| Maximum Pressure : | 1345 psig | |
| Final Temp. & Press.: | 22 C - 600 psig | |
| Gas Temp. & Press.: | 22 C - 184 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 60.69 | 24.94 | .31 | 24.64 |
| Spent | 89.40 | 4.81 | .04 | 4.77 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|-----------------------------|-----------|---------------------------|---------------|
| Total H Consumed: | ----- gr. | CO:CO ₂ Ratio: | 28.07 % |
| H Consumed in Gas : | ----- gr. | Oil Yield: | ----- % |
| H Consumed in Sol.: | ----- gr. | Org. C Conv.: | 86.86 % |
| C in CO _x Gases: | ----- gr. | H Consumed: | ----- % |
| C in Total Gases: | ----- gr. | H Consumed: | ----- SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|-------------------------------|---------------|-------------------------------|---------------|
| H ₂ | 93.478 | C ₃ H ₈ | .084 |
| C ₃ H ₆ | .046 | i-C ₄ | ----- |
| n-C ₄ | .012 | C ₄ + | ----- |
| CO ₂ | 4.118 | C ₂ H ₄ | ----- |
| C ₂ H ₆ | .278 | Ar | ----- |
| CH ₄ | .829 | CO | 1.156 |

Additional Information: Mole Frac. Naphthalene in: -----
Mole Frac. Naphthalene out: -----

Run Number: RB-10

Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 0 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Not Used) |
| Atmosphere: | Helium | |
| Initial Gas Pressure: | 800 psig | Solvent: Tetralin |
| Initial Temperature: | 24 C | |
| Reaction Time: | 60 min. | Additive: None |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1390 psig | |
| Final Temp. & Press.: | 28 C - 675 psig | |
| Gas Temp. & Press.: | 25 C - 208 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | ----- | ----- | ----- | ----- |
| Spent | ----- | ----- | ----- | ----- |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|---------------------|-----------|---------------|---------------|
| Total H Consumed: | ----- gr. | CO:CO2 Ratio: | ----- % |
| H Consumed in Gas : | ----- gr. | Oil Yield: | ----- % |
| H Consumed in Sol.: | ----- gr. | Org. C Conv.: | ----- % |
| C in COx Gases: | ----- gr. | H Consumed: | ----- % |
| C in Total Gases: | ----- gr. | H Consumed: | ----- SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 100.0 | C3H8 | ----- |
| C3H6 | ----- | i-C4 | ----- |
| n-C4 | ----- | C4+ | ----- |
| CO2 | ----- | C2H4 | ----- |
| C2H6 | ----- | Ar | ----- |
| CH4 | ----- | CO | ----- |

Additional Information: Mole Frac. Naphthalene in: -----
Mole Frac. Naphthalene out: -----

Run Number: RB-11

Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Vacuum Dried) |
| Atmosphere: | Helium | |
| Initial Gas Pressure: | 800 psig | Solvent: Tetralin |
| Initial Temperature: | 23 C | |
| Reaction Time: | 60 min. | Additive: None |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1500 psig | |
| Final Temp. & Press.: | 28 C - 745 psig | |
| Gas Temp. & Press.: | 24 C - 220 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 58.86 | 25.08 | .31 | 24.77 |
| Spent | 89.50 | 4.93 | .06 | 4.87 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|---------------------|-----------|---------------|---------------|
| Total H Consumed: | ----- gr. | CO:CO2 Ratio: | 20.14 % |
| H Consumed in Gas : | ----- gr. | Oil Yield: | ----- % |
| H Consumed in Sol.: | ----- gr. | Org. C Conv.: | 87.05 % |
| C in COx Gases: | ----- gr. | H Consumed: | ----- % |
| C in Total Gases: | ----- gr. | H Consumed: | ----- SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 78.890 | C3H8 | .488 |
| C3H6 | .114 | i-C4 | .037 |
| n-C4 | .078 | C4+ | ----- |
| CO2 | 12.610 | C2H4 | .068 |
| C2H6 | 1.269 | Ar | ----- |
| CH4 | 3.906 | CO | 2.540 |

Additional Information: Mole Frac. Naphthalene in: -----
Mole Frac. Naphthalene out: -----

Run Number: RB-12

Conditions:

| | | |
|-----------------------|---------------------|-------------------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Vacuum Dried) |
| Atmosphere: | H ₂ + Ar | |
| Initial Gas Pressure: | 330 psig | Solvent: Tetralin |
| Initial Temperature: | 22 C | |
| Reaction Time: | 60 min. | Additive: 3.78 gr. H ₂ O |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1100 psig | |
| Final Temp. & Press.: | 27 C - 330 psig | |
| Gas Temp. & Press.: | 25 C - 100 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 58.86 | 25.08 | .31 | 24.77 |
| Spent | 89.13 | 4.80 | .01 | 4.79 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|-----------------------------|-----------|---------------------------|-------------|
| Total H Consumed: | .041 gr. | CO:CO ₂ Ratio: | 18.38 % |
| H Consumed in Gas : | -.153 gr. | Oil Yield: | 89.28 % |
| H Consumed in Sol.: | .194 gr. | Org. C Conv.: | 87.39 % |
| C in CO _x Gases: | .391 gr. | H Consumed: | .70 % |
| C in Total Gases: | .579 gr. | H Consumed: | 395 SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|-------------------------------|---------------|-------------------------------|---------------|
| H ₂ | 82.99 | C ₃ H ₈ | .21 |
| C ₃ H ₆ | .10 | i-C ₄ | .01 |
| n-C ₄ | .04 | C ₄ + | ---- |
| CO ₂ | 7.89 | C ₂ H ₄ | .10 |
| C ₂ H ₆ | .66 | Ar | 4.45 |
| CH ₄ | 2.11 | CO | 1.45 |

Additional Information: Mole Frac. Naphthalene in: .00468
Mole Frac. Naphthalene out: .13260

Run Number: RB-13

Conditions:

| | | |
|-----------------------|---------------------|-------------------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Vacuum Dried) |
| Atmosphere: | H ₂ + Ar | |
| Initial Gas Pressure: | 330 psig | Solvent: Tetralin |
| Initial Temperature: | 21 C | |
| Reaction Time: | 60 min. | Additive: 3.78 gr. H ₂ O |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1195 psig | |
| Final Temp. & Press.: | 29 C - 395 psig | |
| Gas Temp. & Press.: | 28 C - 88 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 58.86 | 25.08 | .31 | 24.77 |
| Spent | 89.77 | 4.80 | .02 | 4.78 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|-----------------------------|-----------|---------------------------|-------------|
| Total H Consumed: | .125 gr. | CO:CO ₂ Ratio: | 17.42 % |
| H Consumed in Gas : | -.071 gr. | Oil Yield: | 90.92 % |
| H Consumed in Sol.: | .196 gr. | Org. C Conv.: | 87.50 % |
| C in CO _x Gases: | .368 gr. | H Consumed: | 2.11 % |
| C in Total Gases: | .491 gr. | H Consumed: | 874 SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|-------------------------------|---------------|-------------------------------|---------------|
| H ₂ | 84.61 | C ₃ H ₈ | .15 |
| C ₃ H ₆ | .07 | i-C ₄ | .01 |
| n-C ₄ | .02 | C ₄ + | ---- |
| CO ₂ | 6.60 | C ₂ H ₄ | .07 |
| C ₂ H ₆ | .47 | Ar | 5.31 |
| CH ₄ | 1.54 | CO | 1.15 |

Additional Information: Mole Frac. Naphthalene in: .00468
Mole Frac. Naphthalene out: .13420

Run Number: RB-14

Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Vacuum Dried) |
| Atmosphere: | H2 + Ar | |
| Initial Gas Pressure: | 330 psig | Solvent: Tetralin |
| Initial Temperature: | 22 C | |
| Reaction Time: | 60 min. | Additive: 3.78 gr. H2O |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1145 psig | |
| Final Temp. & Press.: | 28 C - 380 psig | |
| Gas Temp. & Press.: | 28 C - 86 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 58.86 | 25.08 | .31 | 24.77 |
| Spent | 89.45 | 4.96 | .00 | 4.96 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | 8.97 | 90.32 | .13 | .06 | .22 |

Calculated Yields

| | | | |
|---------------------|-----------|---------------|-------------|
| Total H Consumed: | .074 gr. | CO:CO2 Ratio: | 16.99 % |
| H Consumed in Gas : | -.098 gr. | Oil Yield: | 92.61 % |
| H Consumed in Sol.: | .172 gr. | Org. C Conv.: | 86.82 % |
| C in COx Gases: | .272 gr. | H Consumed: | 1.21 % |
| C in Total Gases: | .403 gr. | H Consumed: | 685 SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 85.46 | C3H8 | .15 |
| C3H6 | .07 | i-C4 | .01 |
| n-C4 | .02 | C4+ | ---- |
| CO2 | 6.12 | C2H4 | .06 |
| C2H6 | .45 | Ar | 5.06 |
| CH4 | 1.56 | CO | 1.04 |

Additional Information: Mole Frac. Naphthalene in: .00468
Mole Frac. Naphthalene out: .11790

Run Number: RB-15

Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Vacuum Dried) |
| Atmosphere: | H2 + Ar | |
| Initial Gas Pressure: | 330 psig | Solvent: Tetralin |
| Initial Temperature: | 21 C | |
| Reaction Time: | 60 min. | Additive: 3.78 gr. H2O |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1190 psig | |
| Final Temp. & Press.: | 28 C - 405 psig | |
| Gas Temp. & Press.: | 28 C - 90 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 58.86 | 25.08 | .31 | 24.77 |
| Spent | 89.77 | 5.02 | .00 | 5.02 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|---------------------|-----------|---------------|-------------|
| Total H Consumed: | .084 gr. | CO:CO2 Ratio: | 16.72 % |
| H Consumed in Gas : | -.084 gr. | Oil Yield: | 92.37 % |
| H Consumed in Sol.: | .168 gr. | Org. C Conv.: | 86.71 % |
| C in COx Gases: | .280 gr. | H Consumed: | 1.38 % |
| C in Total Gases: | .416 gr. | H Consumed: | 440 SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 85.18 | C3H8 | .18 |
| C3H6 | .08 | i-C4 | .03 |
| n-C4 | .03 | C4+ | ----- |
| CO2 | 6.38 | C2H4 | .06 |
| C2H6 | .47 | Ar | 5.02 |
| CH4 | 1.50 | CO | 1.07 |

Additional Information: Mole Frac. Naphthalene in: .00468
Mole Frac. Naphthalene out: .11570

Run Number: RB-16Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Vacuum Dried) |
| Atmosphere: | H2 + Ar | |
| Initial Gas Pressure: | 280 psig | Solvent: Tetralin |
| Initial Temperature: | 19 C | |
| Reaction Time: | 60 min. | Additive: 7.60 gr. H2O |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1400 psig | |
| Final Temp. & Press.: | 28 C - 495 psig | |
| Gas Temp. & Press.: | 26 C - 85 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 60.46 | 25.85 | .32 | 25.53 |
| Spent | 89.15 | 4.92 | .02 | 4.90 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | 8.82 | 89.94 | .07 | .07 | 1.03 |

Calculated Yields

| | | | |
|---------------------|-----------|---------------|-------------|
| Total H Consumed: | .065 gr. | CO:CO2 Ratio: | 16.77 % |
| H Consumed in Gas : | -.103 gr. | Oil Yield: | 93.12 % |
| H Consumed in Sol.: | .168 gr. | Org. C Conv.: | 86.71 % |
| C in COx Gases: | .258 gr. | H Consumed: | 1.08 % |
| C in Total Gases: | .375 gr. | H Consumed: | 315 SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 85.27 | C3H8 | .15 |
| C3H6 | .08 | i-C4 | .01 |
| n-C4 | .03 | C4+ | ---- |
| CO2 | 6.68 | C2H4 | .08 |
| C2H6 | .43 | Ar | 4.78 |
| CH4 | 1.37 | CO | 1.12 |

Additional Information: Mole Frac. Naphthalene in: .00468
Mole Frac. Naphthalene out: .11570

Run Number: RB-17

Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Vacuum Dried) |
| Atmosphere: | H2 + Ar | |
| Initial Gas Pressure: | 100 psig | Solvent: Tetralin |
| Initial Temperature: | 19 C | |
| Reaction Time: | 60 min. | Additive: 11.4 gr. H2O |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1355 psig | |
| Final Temp. & Press.: | 33 C - 209 psig | |
| Gas Temp. & Press.: | 32 C - 35 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 60.45 | 25.88 | .36 | 25.52 |
| Spent | 88.47 | 5.39 | .03 | 5.36 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | 8.99 | 89.80 | .11 | .08 | .92 |

Calculated Yields

| | | | |
|---------------------|-----------|---------------|-------------|
| Total H Consumed: | .023 gr. | CO:CO2 Ratio: | 13.95 % |
| H Consumed in Gas : | -.160 gr. | Oil Yield: | 91.49 % |
| H Consumed in Sol.: | .183 gr. | Org. C Conv.: | 85.65 % |
| C in COx Gases: | .320 gr. | H Consumed: | .38 % |
| C in Total Gases: | .473 gr. | H Consumed: | 213 SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 79.22 | C3H8 | .26 |
| C3H6 | .17 | i-C4 | .04 |
| n-C4 | .04 | C4+ | .03 |
| CO2 | 12.19 | C2H4 | .14 |
| C2H6 | .74 | Ar | 3.04 |
| CH4 | 2.43 | CO | 1.70 |

Additional Information: Mole Frac. Naphthalene in: .00468
Mole Frac. Naphthalene out: .12550

Run Number: RB-18

Conditions:

| | | |
|-----------------------|---------------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Vacuum Dried) |
| Atmosphere: | H ₂ + Ar | |
| Initial Gas Pressure: | 800 psig | Solvent: 1-Methylnaph. |
| Initial Temperature: | 29 C | |
| Reaction Time: | 60 min. | Additive: None |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1132 psig | |
| Final Temp. & Press.: | 38 C - 440 psig | |
| Gas Temp. & Press.: | 37 C - 112 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 60.21 | 25.51 | .38 | 25.13 |
| Spent | 90.24 | 4.16 | .05 | 4.11 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|-----------------------------|----------|---------------------------|--------------|
| Total H Consumed: | .161 gr. | CO:CO ₂ Ratio: | 27.89 % |
| H Consumed in Gas : | .161 gr. | Oil Yield: | 85.95 % |
| H Consumed in Sol.: | 0 gr. | Org. C Conv.: | 89.10 % |
| C in CO _x Gases: | .260 gr. | H Consumed: | 2.73 % |
| C in Total Gases: | .800 gr. | H Consumed: | 1545 SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|-------------------------------|---------------|-------------------------------|---------------|
| H ₂ | 83.58 | C ₃ H ₈ | .22 |
| C ₃ H ₆ | .06 | i-C ₄ | .02 |
| n-C ₄ | .05 | C ₄ + | .03 |
| CO ₂ | 3.37 | C ₂ H ₄ | .04 |
| C ₂ H ₆ | .44 | Ar | 5.20 |
| CH ₄ | 6.05 | CO | .94 |

Additional Information: Mole Frac. Naphthalene in: 0
Mole Frac. Naphthalene out: 0

Run Number: RB-19

Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 0 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Not Used) |
| Atmosphere: | H2 + Ar | |
| Initial Gas Pressure: | 280 psig | Solvent: Tetralin |
| Initial Temperature: | 26 C | |
| Reaction Time: | 60 min. | Additive: 8.90 gr. H2O |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1272 psig | |
| Final Temp. & Press.: | 36 C - 264 psig | |
| Gas Temp. & Press.: | 39 C - 78 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | ----- | ----- | ----- | ----- |
| Spent | ----- | ----- | ----- | ----- |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|---------------------|-----------|---------------|---------------|
| Total H Consumed: | .147 gr. | CO:CO2 Ratio: | ----- % |
| H Consumed in Gas : | -.014 gr. | Oil Yield: | ----- % |
| H Consumed in Sol.: | .161 gr. | Org. C Conv.: | ----- % |
| C in COx Gases: | .001 gr. | H Consumed: | ----- % |
| C in Total Gases: | .005 gr. | H Consumed: | ----- SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 95.17 | C3H8 | ----- |
| C3H6 | ----- | i-C4 | ----- |
| n-C4 | ----- | C4+ | ----- |
| CO2 | .06 | C2H4 | .01 |
| C2H6 | ----- | Ar | 4.69 |
| CH4 | .07 | CO | ----- |

Additional Information: Mole Frac. Naphthalene in: .00468
Mole Frac. Naphthalene out: .11060

Run Number: RB-20

Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Spent Shale) |
| Atmosphere: | H2 + Ar | |
| Initial Gas Pressure: | 280 psig | Solvent: Tetralin |
| Initial Temperature: | 24 C | |
| Reaction Time: | 60 min. | Additive: 8.90 gr. H2O |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1560 psig | |
| Final Temp. & Press.: | 36 C - 429 psig | |
| Gas Temp. & Press.: | 37 C - 123 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 87.86 | 5.23 | .03 | 5.20 |
| Spent | 91.88 | 2.85 | .01 | 2.84 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|---------------------|-----------|---------------|--------------|
| Total H Consumed: | .022 gr. | CO:CO2 Ratio: | 37.78 % |
| H Consumed in Gas : | -.183 gr. | Oil Yield: | 58.38 % |
| H Consumed in Sol.: | .210 gr. | Org. C Conv.: | 47.77 % |
| C in COx Gases: | .022 gr. | H Consumed: | 6.09 % |
| C in Total Gases: | .261 gr. | H Consumed: | 3445 SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 93.98 | C3H8 | .26 |
| C3H6 | .09 | i-C4 | .04 |
| n-C4 | .06 | C4+ | .02 |
| CO2 | .45 | C2H4 | .12 |
| C2H6 | .66 | Ar | 3.25 |
| CH4 | .90 | CO | .17 |

Additional Information: Mole Frac. Naphthalene in: .00468
Mole Frac. Naphthalene out: .14340

Run Number: RB-21Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Vacuum Dried) |
| Atmosphere: | H2 + Ar | |
| Initial Gas Pressure: | 280 psig | Solvent: 1-Methylnaph. |
| Initial Temperature: | 23 C | |
| Reaction Time: | 60 min. | Additive: 7.60 gr. H2O |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1245 psig | |
| Final Temp. & Press.: | 27 C - 342 psig | |
| Gas Temp. & Press.: | 25 C - 101 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 59.39 | 25.75 | .34 | 25.41 |
| Spent | 84.17 | 8.24 | .01 | 8.23 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|---------------------|-----------|---------------|--------------|
| Total H Consumed: | -.044 gr. | CO:CO2 Ratio: | 21.31 % |
| H Consumed in Gas : | -.044 gr. | Oil Yield: | 89.95 % |
| H Consumed in Sol.: | 0 gr. | Org. C Conv.: | 77.15 % |
| C in COx Gases: | .293 gr. | H Consumed: | -.82 % |
| C in Total Gases: | .501 gr. | H Consumed: | -463 SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 80.68 | C3H8 | .22 |
| C3H6 | .11 | i-C4 | .01 |
| n-C4 | .04 | C4+ | .03 |
| CO2 | 8.12 | C2H4 | .13 |
| C2H6 | .58 | Ar | 3.74 |
| CH4 | 4.61 | CO | 1.73 |

Additional Information: Mole Frac. Naphthalene in: -----
Mole Frac. Naphthalene out: -----

Run Number: RB-22

Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 0 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Not Used) |
| Atmosphere: | Helium | |
| Initial Gas Pressure: | 280 psig | Solvent: Tetralin |
| Initial Temperature: | 20 C | |
| Reaction Time: | 60 min. | Additive: 8.90 gr. H2O |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1494 psig | |
| Final Temp. & Press.: | 31 C - 297 psig | |
| Gas Temp. & Press.: | 31 C - 88 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | ----- | ----- | ----- | ----- |
| Spent | ----- | ----- | ----- | ----- |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|---------------------|-----------|---------------|---------------|
| Total H Consumed: | ----- gr. | CO:CO2 Ratio: | ----- % |
| H Consumed in Gas : | ----- gr. | Oil Yield: | ----- % |
| H Consumed in Sol.: | ----- gr. | Org. C Conv.: | ----- % |
| C in COx Gases: | ----- gr. | H Consumed: | ----- % |
| C in Total Gases: | ----- gr. | H Consumed: | ----- SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 92.16 | C3H8 | .19 |
| C3H6 | .13 | i-C4 | .10 |
| n-C4 | .19 | C4+ | .31 |
| CO2 | 1.02 | C2H4 | .21 |
| C2H6 | .14 | Ar | 4.54 |
| CH4 | 1.01 | CO | ---- |

Additional Information: Mole Frac. Naphthalene in: -----
Mole Frac. Naphthalene out: -----

Run Number: RB-23

Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 0 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Not Used) |
| Atmosphere: | Helium | |
| Initial Gas Pressure: | 800 psig | Solvent: Tetralin |
| Initial Temperature: | 28 C | |
| Reaction Time: | 60 min. | Additive: None |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1520 psig | |
| Final Temp. & Press.: | 28 C - 811 psig | |
| Gas Temp. & Press.: | 27 C - 255 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | ----- | ----- | ----- | ----- |
| Spent | ----- | ----- | ----- | ----- |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|---------------------|-----------|---------------|---------------|
| Total H Consumed: | ----- gr. | CO:CO2 Ratio: | 58.74 % |
| H Consumed in Gas : | ----- gr. | Oil Yield: | ----- % |
| H Consumed in Sol.: | ----- gr. | Org. C Conv.: | ----- % |
| C in COx Gases: | ----- gr. | H Consumed: | ----- % |
| C in Total Gases: | ----- gr. | H Consumed: | ----- SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 96.07 | C3H8 | ----- |
| C3H6 | ----- | i-C4 | ----- |
| n-C4 | ----- | C4+ | ----- |
| CO2 | 2.06 | C2H4 | ----- |
| C2H6 | ----- | Ar | ----- |
| CH4 | .66 | CO | 1.21 |

Additional Information: Mole Frac. Naphthalene in: -----
Mole Frac. Naphthalene out: -----

Run Number: RB-24

Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 0 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Not Used) |
| Atmosphere: | Helium | |
| Initial Gas Pressure: | 800 psig | Solvent: 1-Methylnaph. |
| Initial Temperature: | 29 C | |
| Reaction Time: | 60 min. | Additive: None |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1422 psig | |
| Final Temp. & Press.: | 30 C - 809 psig | |
| Gas Temp. & Press.: | 29 C - 262 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | ----- | ----- | ----- | ----- |
| Spent | ----- | ----- | ----- | ----- |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|---------------------|-----------|---------------|---------------|
| Total H Consumed: | ----- gr. | CO:CO2 Ratio: | ----- % |
| H Consumed in Gas : | ----- gr. | Oil Yield: | ----- % |
| H Consumed in Sol.: | ----- gr. | Org. C Conv.: | ----- % |
| C in COx Gases: | ----- gr. | H Consumed: | ----- % |
| C in Total Gases: | ----- gr. | H Consumed: | ----- SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 65.70 | C3H8 | ----- |
| C3H6 | ----- | i-C4 | ----- |
| n-C4 | ----- | C4+ | ----- |
| CO2 | .18 | C2H4 | ----- |
| C2H6 | ----- | Ar | ----- |
| CH4 | 34.12 | CO | ----- |

Additional Information: Mole Frac. Naphthalene in: -----
Mole Frac. Naphthalene out: -----

Run Number: RB-25

Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 0 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Not Used) |
| Atmosphere: | Helium | |
| Initial Gas Pressure: | 280 psig | Solvent: 1-Methylnaph. |
| Initial Temperature: | 23 C | |
| Reaction Time: | 60 min. | Additive: 8.90 gr. H2O |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1237 psig | |
| Final Temp. & Press.: | 27 C - 286 psig | |
| Gas Temp. & Press.: | 26 C - 92 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | ----- | ----- | ----- | ----- |
| Spent | ----- | ----- | ----- | ----- |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|---------------------|-----------|---------------|---------------|
| Total H Consumed: | ----- gr. | CO:CO2 Ratio: | ----- % |
| H Consumed in Gas : | ----- gr. | Oil Yield: | ----- % |
| H Consumed in Sol.: | ----- gr. | Org. C Conv.: | ----- % |
| C in COx Gases: | ----- gr. | H Consumed: | ----- % |
| C in Total Gases: | ----- gr. | H Consumed: | ----- SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 70.09 | C3H8 | ----- |
| C3H6 | ----- | i-C4 | ----- |
| n-C4 | ----- | C4+ | ----- |
| CO2 | 5.53 | C2H4 | ----- |
| C2H6 | ----- | Ar | ----- |
| CH4 | 24.38 | CO | ----- |

Additional Information: Mole Frac. Naphthalene in: -----
Mole Frac. Naphthalene out: -----

Run Number: RB-26Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 0 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Not Used) |
| Atmosphere: | CO | |
| Initial Gas Pressure: | 100 psig | Solvent: None |
| Initial Temperature: | 12 C | |
| Reaction Time: | 60 min. | Additive: 15.0 gr. H2O |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1068 psig | |
| Final Temp. & Press.: | 37 C - 112 psig | |
| Gas Temp. & Press.: | 37 C - 35 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | ----- | ----- | ----- | ----- |
| Spent | ----- | ----- | ----- | ----- |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|---------------------|-----------|---------------|---------------|
| Total H Consumed: | ----- gr. | CO:CO2 Ratio: | ----- % |
| H Consumed in Gas : | ----- gr. | Oil Yield: | ----- % |
| H Consumed in Sol.: | ----- gr. | Org. C Conv.: | ----- % |
| C in COx Gases: | ----- gr. | H Consumed: | ----- % |
| C in Total Gases: | ----- gr. | H Consumed: | ----- SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 2.07 | C3H8 | ----- |
| C3H6 | ----- | i-C4 | ----- |
| n-C4 | ----- | C4+ | ----- |
| CO2 | 3.05 | C2H4 | ----- |
| C2H6 | ----- | Ar | ----- |
| CH4 | ----- | CO | 94.9 |

Additional Information: Mole Frac. Naphthalene in: -----
Mole Frac. Naphthalene out: -----

Run Number: RB-27Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 0 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Not Used) |
| Atmosphere: | Helium | |
| Initial Gas Pressure: | 280 psig | Solvent: Tetralin |
| Initial Temperature: | 18 C | |
| Reaction Time: | 60 min. | Additive: 8.90 gr. H2O |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1432 psig | |
| Final Temp. & Press.: | 27 C - 288 psig | |
| Gas Temp. & Press.: | 27 C - 86 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | ----- | ----- | ----- | ----- |
| Spent | ----- | ----- | ----- | ----- |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|---------------------|-----------|---------------|---------------|
| Total H Consumed: | ----- gr. | CO:CO2 Ratio: | 26.22 % |
| H Consumed in Gas : | ----- gr. | Oil Yield: | ----- % |
| H Consumed in Sol.: | ----- gr. | Org. C Conv.: | ----- % |
| C in COx Gases: | ----- gr. | H Consumed: | ----- % |
| C in Total Gases: | ----- gr. | H Consumed: | ----- SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 97.35 | C3H8 | ---- |
| C3H6 | .06 | i-C4 | ---- |
| n-C4 | ----- | C4+ | ---- |
| CO2 | 1.64 | C2H4 | .18 |
| C2H6 | ----- | Ar | ---- |
| CH4 | .34 | CO | .43 |

Additional Information: Mole Frac. Naphthalene in: -----
Mole Frac. Naphthalene out: -----

Run Number: RB-28

Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 10 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 20 gr. | Shale (Spent Shale) |
| Atmosphere: | CO | |
| Initial Gas Pressure: | 100 psig | Solvent: H2O |
| Initial Temperature: | 19 C | |
| Reaction Time: | 60 min. | Additive: None |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1201 psig | |
| Final Temp. & Press.: | 29 C - 115 psig | |
| Gas Temp. & Press.: | 28 C - 36 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 88.48 | 5.08 | .05 | 5.03 |
| Spent | 90.92 | 3.65 | .03 | 3.62 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|---------------------|-----------|---------------|---------------|
| Total H Consumed: | ----- gr. | CO:CO2 Ratio: | ----- % |
| H Consumed in Gas : | ----- gr. | Oil Yield: | ----- % |
| H Consumed in Sol.: | ----- gr. | Org. C Conv.: | ----- % |
| C in COx Gases: | ----- gr. | H Consumed: | ----- % |
| C in Total Gases: | ----- gr. | H Consumed: | ----- SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 8.18 | C3H8 | .17 |
| C3H6 | .14 | i-C4 | .03 |
| n-C4 | .05 | C4+ | .13 |
| CO2 | 4.94 | C2H4 | .07 |
| C2H6 | .26 | Ar | ----- |
| CH4 | ----- | CO | 86.03 |

Additional Information: Mole Frac. Naphthalene in: -----
Mole Frac. Naphthalene out: -----

Run Number: RB-29

Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 0 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Not Used) |
| Atmosphere: | H2 + Ar | |
| Initial Gas Pressure: | 280 psig | Solvent: Tetralin |
| Initial Temperature: | 20 C | |
| Reaction Time: | 60 min. | Additive: 8.90 gr. H2O |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1480 psig | |
| Final Temp. & Press.: | 22 C - 286 psig | |
| Gas Temp. & Press.: | 21 C - 86 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | ----- | ----- | ----- | ----- |
| Spent | ----- | ----- | ----- | ----- |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|---------------------|-----------|---------------|---------------|
| Total H Consumed: | 1.204 gr. | CO:CO2 Ratio: | 50.00 % |
| H Consumed in Gas : | .002 gr. | Oil Yield: | ----- % |
| H Consumed in Sol.: | 1.202 gr. | Org. C Conv.: | ----- % |
| C in COx Gases: | ----- gr. | H Consumed: | ----- % |
| C in Total Gases: | ----- gr. | H Consumed: | ----- SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 95.13 | C3H8 | ---- |
| C3H6 | ----- | i-C4 | ---- |
| n-C4 | ----- | C4+ | ---- |
| CO2 | .02 | C2H4 | ---- |
| C2H6 | ----- | Ar | 4.83 |
| CH4 | .01 | CO | .01 |

Additional Information: Mole Frac. Naphthalene in: .00468
Mole Frac. Naphthalene out: .79800

Run Number: RB-30Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Vacuum Dried) |
| Atmosphere: | Helium | |
| Initial Gas Pressure: | 800 psig | Solvent: Tetralin |
| Initial Temperature: | 21 C | |
| Reaction Time: | 60 min. | Additive: None |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1633 psig | |
| Final Temp. & Press.: | 29 C - 830 psig | |
| Gas Temp. & Press.: | 26 C - 254 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 59.42 | 25.88 | .37 | 25.51 |
| Spent | 87.38 | 6.14 | .05 | 6.09 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|---------------------|-----------|---------------|---------------|
| Total H Consumed: | ----- gr. | CO:CO2 Ratio: | ----- % |
| H Consumed in Gas : | ----- gr. | Oil Yield: | ----- % |
| H Consumed in Sol.: | ----- gr. | Org. C Conv.: | ----- % |
| C in COx Gases: | ----- gr. | H Consumed: | ----- % |
| C in Total Gases: | ----- gr. | H Consumed: | ----- SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 58.06 | C3H8 | 1.68 |
| C3H6 | .60 | i-C4 | .15 |
| n-C4 | .13 | C4+ | 25.42 |
| CO2 | .11 | C2H4 | 2.02 |
| C2H6 | 6.88 | Ar | ----- |
| CH4 | ----- | CO | 4.95 |

Additional Information: Mole Frac. Naphthalene in: -----
Mole Frac. Naphthalene out: -----

Run Number: RB-31

Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Vacuum Dried) |
| Atmosphere: | Helium | |
| Initial Gas Pressure: | 280 psig | Solvent: Tetralin |
| Initial Temperature: | 35 C | |
| Reaction Time: | 60 min. | Additive: 7.60 gr. H2O |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1355 psig | |
| Final Temp. & Press.: | 31 C - 362 psig | |
| Gas Temp. & Press.: | 29 C - 108 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 59.48 | 25.98 | .37 | 25.61 |
| Spent | 86.97 | 6.38 | .01 | 6.37 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|---------------------|-----------|---------------|---------------|
| Total H Consumed: | ----- gr. | CO:CO2 Ratio: | 18.29 % |
| H Consumed in Gas : | ----- gr. | Oil Yield: | ----- % |
| H Consumed in Sol.: | ----- gr. | Org. C Conv.: | ----- % |
| C in COx Gases: | ----- gr. | H Consumed: | ----- % |
| C in Total Gases: | ----- gr. | H Consumed: | ----- SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 61.18 | C3H8 | .39 |
| C3H6 | .27 | i-C4 | ----- |
| n-C4 | .04 | C4+ | .01 |
| CO2 | 26.13 | C2H4 | .33 |
| C2H6 | 1.52 | Ar | ----- |
| CH4 | 5.34 | CO | 4.78 |

Additional Information: Mole Frac. Naphthalene in: -----
Mole Frac. Naphthalene out: -----

Run Number: RB-32Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Decarbonated) |
| Atmosphere: | H2 + Ar | |
| Initial Gas Pressure: | 800 psig | Solvent: Tetralin |
| Initial Temperature: | 23 C | |
| Reaction Time: | 60 min. | Additive: None |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1510 psig | |
| Final Temp. & Press.: | 26 C - 792 psig | |
| Gas Temp. & Press.: | 24 C - 244 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 57.62 | 27.84 | 0 | 27.84 |
| Spent | 89.78 | 4.49 | 0 | 4.49 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|---------------------|----------|---------------|--------------|
| Total H Consumed: | .167 gr. | CO:CO2 Ratio: | 67.11 % |
| H Consumed in Gas : | .036 gr. | Oil Yield: | 95.00 % |
| H Consumed in Sol.: | .131 gr. | Org. C Conv.: | 89.65 % |
| C in COx Gases: | .162 gr. | H Consumed: | 2.34 % |
| C in Total Gases: | .312 gr. | H Consumed: | 1324 SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 91.39 | C3H8 | .09 |
| C3H6 | .04 | i-C4 | .01 |
| n-C4 | .01 | C4+ | ---- |
| CO2 | 1.49 | C2H4 | .02 |
| C2H6 | .23 | Ar | 4.94 |
| CH4 | .79 | CO | 1.00 |

Additional Information: Mole Frac. Naphthalene in: .00468
Mole Frac. Naphthalene out: .09100

Run Number: RB-33Conditions:

Weight of Shale: 0 gr. Stuart "A" Beneficiated
 Weight of Solvent: 50 gr. Shale (Not Used)
 Atmosphere: Ar
 Initial Gas Pressure: 280 psig Solvent: Tetralin
 Initial Temperature: 29 C
 Reaction Time: 60 min. Additive: 8.90 gr. H₂O
 Reaction Temperature: 425 C
 Maximum Pressure : 1438 psig
 Final Temp. & Press.: 36 C - 297 psig
 Gas Temp. & Press.: 32 C - 94 psig

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | ----- | ----- | ----- | ----- |
| Spent | ----- | ----- | ----- | ----- |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|-----------------------------|-----------|---------------------------|---------------|
| Total H Consumed: | .140 gr. | CO:CO ₂ Ratio: | ----- % |
| H Consumed in Gas : | -.021 gr. | Oil Yield: | ----- % |
| H Consumed in Sol.: | .161 gr. | Org. C Conv.: | ----- % |
| C in CO _x Gases: | .002 gr. | H Consumed: | ----- % |
| C in Total Gases: | .002 gr. | H Consumed: | ----- SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|-------------------------------|---------------|-------------------------------|---------------|
| H ₂ | 6.28 | C ₃ H ₈ | ----- |
| C ₃ H ₆ | .01 | i-C ₄ | ----- |
| n-C ₄ | ----- | C ₄ + | ----- |
| CO ₂ | .08 | C ₂ H ₄ | .01 |
| C ₂ H ₆ | ----- | Ar | 93.62 |
| CH ₄ | ----- | CO | ----- |

Additional Information: Mole Frac. Naphthalene in: .00468
 Mole Frac. Naphthalene out: .11060

Run Number: RB-34

Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Decarbonated) |
| Atmosphere: | H2 + Ar | |
| Initial Gas Pressure: | 280 psig | Solvent: Tetralin |
| Initial Temperature: | 32 C | |
| Reaction Time: | 60 min. | Additive: 7.60 gr. H2O |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1350 psig | |
| Final Temp. & Press.: | 35 C - 341 psig | |
| Gas Temp. & Press.: | 34 C - 100 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 56.65 | 26.87 | 0 | 26.87 |
| Spent | 89.91 | 4.93 | 0 | 4.93 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|---------------------|-----------|---------------|--------------|
| Total H Consumed: | .127 gr. | CO:CO2 Ratio: | 24.50 % |
| H Consumed in Gas : | -.053 gr. | Oil Yield: | 93.90 % |
| H Consumed in Sol.: | .180 gr. | Org. C Conv.: | 88.44 % |
| C in COx Gases: | .206 gr. | H Consumed: | 1.89 % |
| C in Total Gases: | .362 gr. | H Consumed: | 1067 SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 85.87 | C3H8 | .17 |
| C3H6 | .09 | i-C4 | .01 |
| n-C4 | .03 | C4+ | .01 |
| CO2 | 6.00 | C2H4 | .11 |
| C2H6 | .54 | Ar | 3.88 |
| CH4 | 1.82 | CO | 1.47 |

Additional Information: Mole Frac. Naphthalene in: .00468
Mole Frac. Naphthalene out: .12330

Run Number: RB-35

Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Vacuum Dried) |
| Atmosphere: | H2 + Ar | |
| Initial Gas Pressure: | 280 psig | Solvent: Heavy Gas Oil |
| Initial Temperature: | 26 C | |
| Reaction Time: | 60 min. | Additive: 7.60 gr. H2O |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1445 psig | |
| Final Temp. & Press.: | 27 C - 320 psig | |
| Gas Temp. & Press.: | 27 C - 94 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 59.70 | 24.81 | .37 | 24.44 |
| Spent | 85.41 | 8.86 | .05 | 8.81 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | 1.13 |

Calculated Yields

| | | | |
|---------------------|----------|---------------|-------------|
| Total H Consumed: | .052 gr. | CO:CO2 Ratio: | 23.76 % |
| H Consumed in Gas : | .052 gr. | Oil Yield: | 83.69 % |
| H Consumed in Sol.: | 0 gr. | Org. C Conv.: | 74.80 % |
| C in COx Gases: | .276 gr. | H Consumed: | 1.11 % |
| C in Total Gases: | .759 gr. | H Consumed: | 628 SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 73.19 | C3H8 | .97 |
| C3H6 | .28 | i-C4 | .04 |
| n-C4 | .17 | C4+ | .10 |
| CO2 | 9.30 | C2H4 | .16 |
| C2H6 | 2.60 | Ar | 4.46 |
| CH4 | 6.52 | CO | 2.21 |

Additional Information: Mole Frac. Naphthalene in: 0
Mole Frac. Naphthalene out: 0

Run Number: RB-36

Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Vacuum Dried) |
| Atmosphere: | H2 + Ar | |
| Initial Gas Pressure: | 800 psig | Solvent: Heavy Gas Oil |
| Initial Temperature: | 27 C | |
| Reaction Time: | 60 min. | Additive: None |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1385 psig | |
| Final Temp. & Press.: | 28 C - 585 psig | |
| Gas Temp. & Press.: | 28 C - 181 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 59.36 | 25.10 | .36 | 24.74 |
| Spent | 83.66 | 9.52 | .10 | 9.42 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | .76 |

Calculated Yields

| | | | |
|---------------------|----------|---------------|--------------|
| Total H Consumed: | .206 gr. | CO:CO2 Ratio: | 25.65 % |
| H Consumed in Gas : | .206 gr. | Oil Yield: | 81.71 % |
| H Consumed in Sol.: | 0 gr. | Org. C Conv.: | 72.98 % |
| C in COx Gases: | .278 gr. | H Consumed: | 4.55 % |
| C in Total Gases: | .839 gr. | H Consumed: | 2575 SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 84.99 | C3H8 | .58 |
| C3H6 | .14 | i-C4 | .03 |
| n-C4 | .11 | C4+ | .05 |
| CO2 | 4.21 | C2H4 | .07 |
| C2H6 | 1.30 | Ar | 4.35 |
| CH4 | 3.11 | CO | 1.08 |

Additional Information: Mole Frac. Naphthalene in: 0
Mole Frac. Naphthalene out: 0

Run Number: RB-37

Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 0 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Not Used) |
| Atmosphere: | Ar | |
| Initial Gas Pressure: | 800 psig | Solvent: Heavy Gas Oil |
| Initial Temperature: | 19 C | |
| Reaction Time: | 60 min. | Additive: None |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1664 psig | |
| Final Temp. & Press.: | 31 C - 841 psig | |
| Gas Temp. & Press.: | 25 C - 276 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | ----- | ----- | ----- | ----- |
| Spent | ----- | ----- | ----- | ----- |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | .51 |

Calculated Yields

| | | | |
|---------------------|-----------|---------------|---------------|
| Total H Consumed: | ----- gr. | CO:CO2 Ratio: | ----- % |
| H Consumed in Gas : | ----- gr. | Oil Yield: | ----- % |
| H Consumed in Sol.: | ----- gr. | Org. C Conv.: | ----- % |
| C in COx Gases: | .056 gr. | H Consumed: | ----- % |
| C in Total Gases: | .767 gr. | H Consumed: | ----- SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | .97 | C3H8 | .83 |
| C3H6 | .22 | i-C4 | .07 |
| n-C4 | .37 | C4+ | 2.35 |
| CO2 | .20 | C2H4 | .09 |
| C2H6 | 1.64 | Ar | 90.04 |
| CH4 | 2.68 | CO | .55 |

Additional Information: Mole Frac. Naphthalene in: 0
Mole Frac. Naphthalene out: 0

Run Number: RB-38

Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 0 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Not Used) |
| Atmosphere: | Ar | |
| Initial Gas Pressure: | 280 psig | Solvent: Heavy Gas Oil |
| Initial Temperature: | 21 C | |
| Reaction Time: | 60 min. | Additive: 8.90 gr. H2O |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1354 psig | |
| Final Temp. & Press.: | 32 C - 323 psig | |
| Gas Temp. & Press.: | 29 C - 101 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | ----- | ----- | ----- | ----- |
| Spent | ----- | ----- | ----- | ----- |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | .60 |

Calculated Yields

| | | | |
|---------------------|-----------|---------------|---------------|
| Total H Consumed: | -.017 gr. | CO:CO2 Ratio: | ----- % |
| H Consumed in Gas : | -.017 gr. | Oil Yield: | ----- % |
| H Consumed in Sol.: | 0 gr. | Org. C Conv.: | ----- % |
| C in COx Gases: | .056 gr. | H Consumed: | ----- % |
| C in Total Gases: | .697 gr. | H Consumed: | ----- SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 4.74 | C3H8 | 1.60 |
| C3H6 | .72 | i-C4 | .06 |
| n-C4 | .45 | C4+ | .42 |
| CO2 | .85 | C2H4 | .28 |
| C2H6 | 3.50 | Ar | 79.64 |
| CH4 | 6.55 | CO | 1.18 |

Additional Information: Mole Frac. Naphthalene in: 0
Mole Frac. Naphthalene out: 0

Run Number: RB-39Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 0 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Not Used) |
| Atmosphere: | Ar | |
| Initial Gas Pressure: | 280 psig | Solvent: Tetralin |
| Initial Temperature: | 17 C | |
| Reaction Time: | 60 min. | Additive: 8.90 gr. H2O |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1483 psig | |
| Final Temp. & Press.: | 32 C - 301 psig | |
| Gas Temp. & Press.: | 26 C - 92 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | ----- | ----- | ----- | ----- |
| Spent | ----- | ----- | ----- | ----- |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|---------------------|-----------|---------------|---------------|
| Total H Consumed: | -.001 gr. | CO:CO2 Ratio: | 25.00 % |
| H Consumed in Gas : | -.011 gr. | Oil Yield: | ----- % |
| H Consumed in Sol.: | .010 gr. | Org. C Conv.: | ----- % |
| C in COx Gases: | .001 gr. | H Consumed: | ----- % |
| C in Total Gases: | .004 gr. | H Consumed: | ----- SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 2.64 | C3H8 | .01 |
| C3H6 | .01 | i-C4 | ----- |
| n-C4 | ----- | C4+ | .01 |
| CO2 | .04 | C2H4 | .01 |
| C2H6 | ----- | Ar | 97.25 |
| CH4 | .02 | CO | .01 |

Additional Information: Mole Frac. Naphthalene in: .00468
Mole Frac. Naphthalene out: .01120

Run Number: RB-40

Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 0 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Not Used) |
| Atmosphere: | Ar | |
| Initial Gas Pressure: | 800 psig | Solvent: 1-Methylnaph. |
| Initial Temperature: | 28 C | |
| Reaction Time: | 60 min. | Additive: None |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1528 psig | |
| Final Temp. & Press.: | 27 C - 797 psig | |
| Gas Temp. & Press.: | 22 C - 267 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | ----- | ----- | ----- | ----- |
| Spent | ----- | ----- | ----- | ----- |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|---------------------|-----------|---------------|---------------|
| Total H Consumed: | -.002 gr. | CO:CO2 Ratio: | ----- % |
| H Consumed in Gas : | -.002 gr. | Oil Yield: | ----- % |
| H Consumed in Sol.: | 0 gr. | Org. C Conv.: | ----- % |
| C in COx Gases: | 0 gr. | H Consumed: | ----- % |
| C in Total Gases: | .008 gr. | H Consumed: | ----- SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | .21 | C3H8 | ----- |
| C3H6 | ----- | i-C4 | ----- |
| n-C4 | ----- | C4+ | ----- |
| CO2 | .01 | C2H4 | ----- |
| C2H6 | ----- | Ar | 99.68 |
| CH4 | .10 | CO | ----- |

Additional Information: Mole Frac. Naphthalene in: 0
Mole Frac. Naphthalene out: 0

Run Number: RB-41Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 0 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Not Used) |
| Atmosphere: | Ar | |
| Initial Gas Pressure: | 280 psig | Solvent: 1-Methylnaph. |
| Initial Temperature: | 23 C | |
| Reaction Time: | 60 min. | Additive: 8.90 gr. H2O |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1149 psig | |
| Final Temp. & Press.: | 43 C - 293 psig | |
| Gas Temp. & Press.: | 29 C - 85 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | ----- | ----- | ----- | ----- |
| Spent | ----- | ----- | ----- | ----- |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|---------------------|-----------|---------------|---------------|
| Total H Consumed: | -.002 gr. | CO:CO2 Ratio: | ----- % |
| H Consumed in Gas : | -.002 gr. | Oil Yield: | ----- % |
| H Consumed in Sol.: | 0 gr. | Org. C Conv.: | ----- % |
| C in COx Gases: | .001 gr. | H Consumed: | ----- % |
| C in Total Gases: | .010 gr. | H Consumed: | ----- SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | .61 | C3H8 | .01 |
| C3H6 | ----- | i-C4 | ----- |
| n-C4 | ----- | C4+ | ----- |
| CO2 | .02 | C2H4 | ----- |
| C2H6 | .03 | Ar | 99.07 |
| CH4 | .26 | CO | ----- |

Additional Information: Mole Frac. Naphthalene in: 0
Mole Frac. Naphthalene out: 0

Run Number: RB-47Conditions:

| | | |
|-----------------------|---------------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Vacuum Dried) |
| Atmosphere: | H ₂ + Ar | |
| Initial Gas Pressure: | 800 psig | Solvent: Tetralin |
| Initial Temperature: | 26 C | |
| Reaction Time: | 60 min. | Additive: None |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1493 psig | |
| Final Temp. & Press.: | 40 C - 803 psig | |
| Gas Temp. & Press.: | 38 C - 242 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 60.25 | 25.77 | .35 | 25.42 |
| Spent | 88.40 | 6.01 | .06 | 5.95 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|-----------------------------|----------|---------------------------|--------------|
| Total H Consumed: | .123 gr. | CO:CO ₂ Ratio: | 24.61 % |
| H Consumed in Gas : | .025 gr. | Oil Yield: | 94.09 % |
| H Consumed in Sol.: | .098 gr. | Org. C Conv.: | 84.05 % |
| C in CO _x Gases: | .204 gr. | H Consumed: | 2.00 % |
| C in Total Gases: | .320 gr. | H Consumed: | 1132 SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|-------------------------------|---------------|-------------------------------|---------------|
| H ₂ | 91.23 | C ₃ H ₈ | .09 |
| C ₃ H ₆ | .04 | i-C ₄ | .01 |
| n-C ₄ | .01 | C ₄ + | .01 |
| CO ₂ | 2.56 | C ₂ H ₄ | ---- |
| C ₂ H ₆ | .21 | Ar | 4.60 |
| CH ₄ | .61 | CO | .63 |

Additional Information: Mole Frac. Naphthalene in: .00468
Mole Frac. Naphthalene out: .06920

Run Number: RB-48

Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Vacuum Dried) |
| Atmosphere: | H2 + Ar | |
| Initial Gas Pressure: | 280 psig | Solvent: Tetralin |
| Initial Temperature: | 24 C | |
| Reaction Time: | 60 min. | Additive: 7.60 gr. H2O |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1389 psig | |
| Final Temp. & Press.: | 30 C - 359 psig | |
| Gas Temp. & Press.: | 30 C - 108 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 60.45 | 25.60 | .35 | 25.25 |
| Spent | 89.93 | 4.41 | 0 | 4.41 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|---------------------|-----------|---------------|-------------|
| Total H Consumed: | .099 gr. | CO:CO2 Ratio: | 18.54 % |
| H Consumed in Gas : | -.079 gr. | Oil Yield: | 93.69 % |
| H Consumed in Sol.: | .178 gr. | Org. C Conv.: | 88.26 % |
| C in COx Gases: | .237 gr. | H Consumed: | 1.54 % |
| C in Total Gases: | .357 gr. | H Consumed: | 873 SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 86.42 | C3H8 | .14 |
| C3H6 | .07 | i-C4 | .01 |
| n-C4 | .02 | C4+ | .01 |
| CO2 | 6.58 | C2H4 | .07 |
| C2H6 | .46 | Ar | 3.51 |
| CH4 | 1.50 | CO | 1.22 |

Additional Information: Mole Frac. Naphthalene in: .00468
Mole Frac. Naphthalene out: .12200

Run Number: RB-49Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Vacuum Dried) |
| Atmosphere: | H2 + Ar | |
| Initial Gas Pressure: | 800 psig | Solvent: Toluene |
| Initial Temperature: | 27 C | |
| Reaction Time: | 60 min. | Additive: None |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 2177 psig | |
| Final Temp. & Press.: | 28 C - 559 psig | |
| Gas Temp. & Press.: | 27 C - 174 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 59.60 | 25.33 | .35 | 24.98 |
| Spent | 90.85 | 5.09 | .10 | 4.99 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|---------------------|----------|---------------|-------------|
| Total H Consumed: | .108 gr. | CO:CO2 Ratio: | 35.06 % |
| H Consumed in Gas : | .108 gr. | Oil Yield: | 93.39 % |
| H Consumed in Sol.: | 0 gr. | Org. C Conv.: | 86.87 % |
| C in COx Gases: | .192 gr. | H Consumed: | 1.75 % |
| C in Total Gases: | .364 gr. | H Consumed: | 990 SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 89.60 | C3H8 | .12 |
| C3H6 | .05 | i-C4 | .01 |
| n-C4 | .02 | C4+ | .01 |
| CO2 | 2.51 | C2H4 | .04 |
| C2H6 | .34 | Ar | 5.24 |
| CH4 | 1.19 | CO | .88 |

Additional Information: Mole Frac. Naphthalene in: 0
Mole Frac. Naphthalene out: 0

Run Number: RB-50Conditions:

| | | |
|-----------------------|---------------------|-------------------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Vacuum Dried) |
| Atmosphere: | H ₂ + Ar | |
| Initial Gas Pressure: | 300 psig | Solvent: Toluene |
| Initial Temperature: | 27 C | |
| Reaction Time: | 60 min. | Additive: 7.60 gr. H ₂ O |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 2194 psig | |
| Final Temp. & Press.: | 27 C - 312 psig | |
| Gas Temp. & Press.: | 26 C - 95 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 59.24 | 25.48 | .36 | 25.12 |
| Spent | 84.14 | 9.41 | .03 | 9.38 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | 10.30 | 84.22 | 1.81 | .41 | 3.63 |

Calculated Yields

| | | | |
|-----------------------------|----------|---------------------------|-------------|
| Total H Consumed: | .016 gr. | CO:CO ₂ Ratio: | 14.85 % |
| H Consumed in Gas : | .016 gr. | Oil Yield: | 89.57 % |
| H Consumed in Sol.: | 0 gr. | Org. C Conv.: | 73.71 % |
| C in CO _x Gases: | .237 gr. | H Consumed: | .32 % |
| C in Total Gases: | .492 gr. | H Consumed: | 179 SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|-------------------------------|---------------|-------------------------------|---------------|
| H ₂ | 80.04 | C ₃ H ₈ | .39 |
| C ₃ H ₆ | .10 | i-C ₄ | .02 |
| n-C ₄ | .05 | C ₄ + | .02 |
| CO ₂ | 7.81 | C ₂ H ₄ | .12 |
| C ₂ H ₆ | 1.28 | Ar | 4.34 |
| CH ₄ | 4.67 | CO | 1.15 |

Additional Information: Mole Frac. Naphthalene in: 0
Mole Frac. Naphthalene out: 0

Run Number: RB-51

Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Vacuum Dried) |
| Atmosphere: | H2 + Ar | |
| Initial Gas Pressure: | 800 psig | Solvent: Tetralin |
| Initial Temperature: | 28 C | |
| Reaction Time: | 60 min. | Additive: None |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1516 psig | |
| Final Temp. & Press.: | 40 C - 816 psig | |
| Gas Temp. & Press.: | 43 C - 252 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 58.96 | 26.31 | .17 | 26.14 |
| Spent | 82.54 | 6.61 | .04 | 6.57 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|---------------------|----------|---------------|--------------|
| Total H Consumed: | .134 gr. | CO:CO2 Ratio: | 28.51 % |
| H Consumed in Gas : | .044 gr. | Oil Yield: | 94.24 % |
| H Consumed in Sol.: | .090 gr. | Org. C Conv.: | 82.05 % |
| C in COx Gases: | .187 gr. | H Consumed: | 2.19 % |
| C in Total Gases: | .31 gr. | H Consumed: | 1236 SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 90.90 | C3H8 | .10 |
| C3H6 | .04 | i-C4 | .01 |
| n-C4 | .02 | C4+ | ---- |
| CO2 | 2.35 | C2H4 | .02 |
| C2H6 | .25 | Ar | 4.95 |
| CH4 | .69 | CO | .67 |

Additional Information: Mole Frac. Naphthalene in: .00468
Mole Frac. Naphthalene out: .06378

Run Number: RB-59

Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Vacuum Dried) |
| Atmosphere: | H2 + Ar | |
| Initial Gas Pressure: | 800 psig | Solvent: Toluene |
| Initial Temperature: | 25 C | |
| Reaction Time: | 60 min. | Additive: None |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 2195 psig | |
| Final Temp. & Press.: | 25 C - 697 psig | |
| Gas Temp. & Press.: | 24 C - 220 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 58.72 | 26.25 | .17 | 26.08 |
| Spent | 90.15 | 4.28 | .08 | 4.20 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | 11.20 | 84.50 | 1.50 | .21 | 2.40 |

Calculated Yields

| | | | |
|---------------------|----------|---------------|-------------|
| Total H Consumed: | .105 gr. | CO:CO2 Ratio: | 35.32 % |
| H Consumed in Gas : | .105 gr. | Oil Yield: | 95.38 % |
| H Consumed in Sol.: | 0 gr. | Org. C Conv.: | 89.51 % |
| C in COx Gases: | .158 gr. | H Consumed: | 1.56 % |
| C in Total Gases: | .271 gr. | H Consumed: | 880 SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 90.78 | C3H8 | .07 |
| C3H6 | .05 | i-C4 | .01 |
| n-C4 | .01 | C4+ | ---- |
| CO2 | 2.01 | C2H4 | .02 |
| C2H6 | .19 | Ar | 5.28 |
| CH4 | .87 | CO | .71 |

Additional Information: Mole Frac. Naphthalene in: 0
Mole Frac. Naphthalene out: 0

Run Number: RB-61

Conditions:

| | | |
|-----------------------|---------------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Vacuum Dried) |
| Atmosphere: | H ₂ + Ar | |
| Initial Gas Pressure: | 800 psig | Solvent: Toluene |
| Initial Temperature: | 23 C | |
| Reaction Time: | 60 min. | Additive: None |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 2249 psig | |
| Final Temp. & Press.: | 32 C - 733 psig | |
| Gas Temp. & Press.: | 31 C - 237 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 61.77 | 24.25 | .24 | 24.01 |
| Spent | 91.21 | 4.09 | .17 | 3.92 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | 11.22 | 84.52 | 1.43 | .19 | 2.10 |

Calculated Yields

| | | | |
|-----------------------------|----------|---------------------------|-------------|
| Total H Consumed: | .100 gr. | CO:CO ₂ Ratio: | 35.53 % |
| H Consumed in Gas : | .100 gr. | Oil Yield: | 94.90 % |
| H Consumed in Sol.: | 0 gr. | Org. C Conv.: | 88.94 % |
| C in CO _x Gases: | .156 gr. | H Consumed: | 1.63 % |
| C in Total Gases: | .274 gr. | H Consumed: | 921 SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|-------------------------------|---------------|-------------------------------|---------------|
| H ₂ | 90.78 | C ₃ H ₈ | .08 |
| C ₃ H ₆ | .05 | i-C ₄ | ---- |
| n-C ₄ | .01 | C ₄ + | ---- |
| CO ₂ | 1.97 | C ₂ H ₄ | .03 |
| C ₂ H ₆ | .21 | Ar | 5.30 |
| CH ₄ | .87 | CO | .70 |

Additional Information: Mole Frac. Naphthalene in: 0
Mole Frac. Naphthalene out: 0

Run Number: RB-62

Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Vacuum Dried) |
| Atmosphere: | H2 + Ar | |
| Initial Gas Pressure: | 300 psig | Solvent: Toluene |
| Initial Temperature: | 22 C | |
| Reaction Time: | 60 min. | Additive: 7.60 gr. H2O |
| Reaction Temperature: | 460 C | |
| Maximum Pressure : | 2337 psig | |
| Final Temp. & Press.: | 23 C - 304 psig | |
| Gas Temp. & Press.: | 23 C - 93 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 61.75 | 24.48 | .21 | 24.27 |
| Spent | 85.48 | 8.67 | .25 | 8.42 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | 9.31 | 85.06 | 1.60 | .93 | 2.78 |

Calculated Yields

| | | | |
|---------------------|----------|---------------|-------------|
| Total H Consumed: | .014 gr. | CO:CO2 Ratio: | 6.60 % |
| H Consumed in Gas : | .014 gr. | Oil Yield: | 86.89 % |
| H Consumed in Sol.: | 0 gr. | Org. C Conv.: | 74.94 % |
| C in COx Gases: | .214 gr. | H Consumed: | .30 % |
| C in Total Gases: | .597 gr. | H Consumed: | 166 SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 79.07 | C3H8 | .49 |
| C3H6 | .10 | i-C4 | .02 |
| n-C4 | .05 | C4+ | .02 |
| CO2 | 7.42 | C2H4 | .10 |
| C2H6 | 1.71 | Ar | 4.27 |
| CH4 | 6.26 | CO | .49 |

Additional Information: Mole Frac. Naphthalene in: 0
Mole Frac. Naphthalene out: 0

Run Number: RB-63Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Vacuum Dried) |
| Atmosphere: | H2 + Ar | |
| Initial Gas Pressure: | 300 psig | Solvent: Toluene |
| Initial Temperature: | 26 C | |
| Reaction Time: | 60 min. | Additive: 7.60 gr. H2O |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 2102 psig | |
| Final Temp. & Press.: | 29 C - 305 psig | |
| Gas Temp. & Press.: | 30 C - 89 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 61.59 | 24.56 | .23 | 24.33 |
| Spent | 85.08 | 8.39 | .15 | 8.24 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | 10.75 | 83.54 | 1.37 | .38 | 3.59 |

Calculated Yields

| | | | |
|---------------------|----------|---------------|-------------|
| Total H Consumed: | .024 gr. | CO:CO2 Ratio: | 11.77 % |
| H Consumed in Gas : | .024 gr. | Oil Yield: | 93.22 % |
| H Consumed in Sol.: | 0 gr. | Org. C Conv.: | 75.48 % |
| C in COx Gases: | .179 gr. | H Consumed: | .46 % |
| C in Total Gases: | .314 gr. | H Consumed: | 259 SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 84.70 | C3H8 | .16 |
| C3H6 | .06 | i-C4 | .01 |
| n-C4 | .02 | C4+ | .01 |
| CO2 | 6.54 | C2H4 | .09 |
| C2H6 | .61 | Ar | 4.54 |
| CH4 | 2.49 | CO | .77 |

Additional Information: Mole Frac. Naphthalene in: 0
Mole Frac. Naphthalene out: 0

Run Number: GB-2

Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Vacuum Dried) |
| Atmosphere: | H2 + Ar | |
| Initial Gas Pressure: | 800 psig | Solvent: Tetralin |
| Initial Temperature: | 22 C | |
| Reaction Time: | 60 min. | Additive: None |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1400 psig | |
| Final Temp. & Press.: | 27 C - 660 psig | |
| Gas Temp. & Press.: | 26 C - 170 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 58.86 | 25.40 | .32 | 25.08 |
| Spent | 90.21 | 4.37 | .08 | 4.29 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|---------------------|----------|---------------|--------------|
| Total H Consumed: | .178 gr. | CO:CO2 Ratio: | 30.82 % |
| H Consumed in Gas : | .088 gr. | Oil Yield: | 93.57 % |
| H Consumed in Sol.: | .090 gr. | Org. C Conv.: | 88.84 % |
| C in COx Gases: | .232 gr. | H Consumed: | 2.81 % |
| C in Total Gases: | .363 gr. | H Consumed: | 1587 SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 87.99 | C3H8 | .10 |
| C3H6 | .06 | i-C4 | .01 |
| n-C4 | .01 | C4+ | ---- |
| CO2 | 2.92 | C2H4 | .02 |
| C2H6 | .28 | Ar | 6.91 |
| CH4 | .80 | CO | .90 |

Additional Information: Mole Frac. Naphthalene in: .00239
Mole Frac. Naphthalene out: .06180

Run Number: GB-3

Conditions:

| | | |
|-----------------------|---------------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Vacuum Dried) |
| Atmosphere: | H ₂ + Ar | |
| Initial Gas Pressure: | 800 psig | Solvent: Tetralin |
| Initial Temperature: | 20 C | |
| Reaction Time: | 60 min. | Additive: None |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1425 psig | |
| Final Temp. & Press.: | 26 C - 685 psig | |
| Gas Temp. & Press.: | 23 C - 203 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 58.86 | 25.40 | .32 | 25.08 |
| Spent | 90.11 | 4.77 | .06 | 4.71 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|-----------------------------|----------|---------------------------|--------------|
| Total H Consumed: | .167 gr. | CO:CO ₂ Ratio: | 24.07 % |
| H Consumed in Gas : | .088 gr. | Oil Yield: | 93.30 % |
| H Consumed in Sol.: | .079 gr. | Org. C Conv.: | 87.73 % |
| C in CO _x Gases: | .264 gr. | H Consumed: | 2.67 % |
| C in Total Gases: | .373 gr. | H Consumed: | 1509 SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|-------------------------------|---------------|-------------------------------|---------------|
| H ₂ | 87.59 | C ₃ H ₈ | .11 |
| C ₃ H ₆ | .05 | i-C ₄ | .01 |
| n-C ₄ | .02 | C ₄ + | ----- |
| CO ₂ | 3.49 | C ₂ H ₄ | ---- |
| C ₂ H ₆ | .07 | Ar | 6.98 |
| CH ₄ | .86 | CO | .84 |

Additional Information: Mole Frac. Naphthalene in: .00468
Mole Frac. Naphthalene out: .05683

Run Number: GB-4Conditions:

| | | |
|-----------------------|---------------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Vacuum Dried) |
| Atmosphere: | H ₂ + Ar | |
| Initial Gas Pressure: | 800 psig | Solvent: Tetralin |
| Initial Temperature: | 23 C | |
| Reaction Time: | 60 min. | Additive: None |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1460 psig | |
| Final Temp. & Press.: | 31 C - 725 psig | |
| Gas Temp. & Press.: | 30 C - 185 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 58.86 | 25.40 | .32 | 25.08 |
| Spent | 90.80 | 4.32 | .07 | 4.25 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | 9.14 | 90.39 | .13 | .05 | .30 |

Calculated Yields

| | | | |
|-----------------------------|----------|---------------------------|---------------|
| Total H Consumed: | .165 gr. | CO:CO ₂ Ratio: | 26.69 % |
| H Consumed in Gas : | .093 gr. | Oil Yield: | 66.30 % |
| H Consumed in Sol.: | .072 gr. | Org. C Conv.: | 88.99 % |
| C in CO _x Gases: | .249 gr. | H Consumed: | 18.25 % |
| C in Total Gases: | .381 gr. | H Consumed: | 10315 SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|-------------------------------|---------------|-------------------------------|---------------|
| H ₂ | 87.68 | C ₃ H ₈ | .10 |
| C ₃ H ₆ | .04 | i-C ₄ | .01 |
| n-C ₄ | .02 | C ₄ + | ---- |
| CO ₂ | 3.26 | C ₂ H ₄ | .02 |
| C ₂ H ₆ | .28 | Ar | 6.93 |
| CH ₄ | .78 | CO | .87 |

Additional Information: Mole Frac. Naphthalene in: .00468
Mole Frac. Naphthalene out: .05206

Run Number: GB-5

Conditions:

| | | |
|-----------------------|---------------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Vacuum Dried) |
| Atmosphere: | H ₂ + Ar | |
| Initial Gas Pressure: | 800 psig | Solvent: Tetralin |
| Initial Temperature: | 29 C | |
| Reaction Time: | 60 min. | Additive: None |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1420 psig | |
| Final Temp. & Press.: | 27 C - 680 psig | |
| Gas Temp. & Press.: | 25 C - 174 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 60.74 | 25.40 | .32 | 25.08 |
| Spent | 90.88 | 4.41 | .05 | 4.36 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|-----------------------------|----------|---------------------------|--------------|
| Total H Consumed: | .175 gr. | CO:CO ₂ Ratio: | 5.19 % |
| H Consumed in Gas : | .085 gr. | Oil Yield: | 92.65 % |
| H Consumed in Sol.: | .090 gr. | Org. C Conv.: | 88.35 % |
| C in CO _x Gases: | .258 gr. | H Consumed: | 2.82 % |
| C in Total Gases: | .410 gr. | H Consumed: | 1593 SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|-------------------------------|---------------|-------------------------------|---------------|
| H ₂ | 88.92 | C ₃ H ₈ | .15 |
| C ₃ H ₆ | .05 | i-C ₄ | .02 |
| n-C ₄ | .02 | C ₄ + | ---- |
| CO ₂ | 3.47 | C ₂ H ₄ | .02 |
| C ₂ H ₆ | .32 | Ar | 6.77 |
| CH ₄ | .08 | CO | .18 |

Additional Information: Mole Frac. Naphthalene in: .00468
Mole Frac. Naphthalene out: .06450

Run Number: GB-6Conditions:

| | | |
|-----------------------|-----------------|-------------------------|
| Weight of Shale: | 25 gr. | Stuart "A" Beneficiated |
| Weight of Solvent: | 50 gr. | Shale (Vacuum Dried) |
| Atmosphere: | H2 + Ar | |
| Initial Gas Pressure: | 800 psig | Solvent: Tetralin |
| Initial Temperature: | 23 C | |
| Reaction Time: | 60 min. | Additive: None |
| Reaction Temperature: | 425 C | |
| Maximum Pressure : | 1460 psig | |
| Final Temp. & Press.: | 28 C - 750 psig | |
| Gas Temp. & Press.: | 28 C - 186 psig | |

Chemical Analysis:

| <u>Shale</u> | <u>% Ash</u> | <u>% Total C</u> | <u>% Inorg. C</u> | <u>% Org. C</u> |
|--------------|--------------|------------------|-------------------|-----------------|
| Feed | 60.74 | 25.40 | .32 | 25.08 |
| Spent | 90.47 | 4.17 | .04 | 4.13 |

Elemental Analysis

| <u>Fraction</u> | <u>% Hydrogen</u> | <u>% Carbon</u> | <u>% Nitrogen</u> | <u>% Sulfur</u> | <u>% Oxy.</u> |
|-----------------|-------------------|-----------------|-------------------|-----------------|---------------|
| Oil | ----- | ----- | ----- | ----- | ----- |

Calculated Yields

| | | | |
|---------------------|----------|---------------|--------------|
| Total H Consumed: | .162 gr. | CO:CO2 Ratio: | 29.39 % |
| H Consumed in Gas : | .077 gr. | Oil Yield: | 93.58 % |
| H Consumed in Sol.: | .085 gr. | Org. C Conv.: | 89.02 % |
| C in COx Gases: | .234 gr. | H Consumed: | 2.53 % |
| C in Total Gases: | .366 gr. | H Consumed: | 1428 SCF/BBL |

Gas Analysis

| <u>Component</u> | <u>Mole %</u> | <u>Component</u> | <u>Mole %</u> |
|------------------|---------------|------------------|---------------|
| H2 | 87.98 | C3H8 | .10 |
| C3H6 | .04 | i-C4 | .01 |
| n-C4 | .02 | C4+ | ---- |
| CO2 | 2.96 | C2H4 | .02 |
| C2H6 | .27 | Ar | 6.96 |
| CH4 | .77 | CO | .87 |

Additional Information: Mole Frac. Naphthalene in: .00468
Mole Frac. Naphthalene out: .06077

APPENDIX B

OXYGEN ANALYSIS

The following analytical work was done by Oxygen Analysis Service, IRT Corporation, San Diego, California. Each sample was irradiated, along with an oxygen standard, with 14 MeV neutrons and counted for nitrogen-16 induced activity on a single-channel pulse-height analyzer using a pair of 5 inch by 5 inch NaI(Tl) scintillation crystals. The oxygen concentration was determined by comparing the intensity of the 6.13 MeV gamma-ray photopeak of nitrogen-16 from the sample with that from the oxygen standard. Oxygen forms nitrogen-16 by interaction with 14 MeV neutrons via the $O-16(n,p)N-16$ reaction.

TABLE 10
Oxygen Concentration

| Sample No. | Oxygen Concentration | |
|------------|----------------------|-------------------|
| | Shale | Oil |
| RB-47 (1) | 39.2% \pm .49% | .307% \pm .008% |
| (2) | 40.6% \pm .52% | .305% \pm .008% |
| RB-48 (1) | 40.7% \pm .52% | .406% \pm .009% |
| (2) | 41.7% \pm .53% | .400% \pm .009% |
| RB-49 | 40.3% \pm .49% | .186% \pm .006% |
| RB-50 | 39.2% \pm .50% | .280% \pm .007% |
| RB-51 | 40.3% \pm .49% | .349% \pm .008% |

APPENDIX C

WATER CONTENT

The following Karl Fischer analysis for water was performed by Huffman Laboratories, Inc., Wheat Ridge, Colorado.

TABLE 11
Karl Fischer Analysis for Water

| Sample Number | Water % |
|---------------|---------|
| RB-14 | .06 |
| RB-16 | .25 |
| RB-17 | .35 |
| RB-35 | .16 |
| RB-36 | .07 |
| RB-37 | .19 |
| RB-38 | .08 |
| RB-47 | .06 |
| RB-48 | .27 |
| GB- 4 | .04 |

APPENDIX D

SAMPLE CALCULATIONS

1. Calculation of Hydrogen Consumption.

A. From chromatogram of initial gas sample:

$$\text{H}_2 \text{ area} = 1.5130 \times 10^7$$

$$\text{Ar} + \text{O}_2 \text{ area} = 1.1257 \times 10^7$$

$$\text{N}_2 \text{ area} = 541790$$

$$\text{O}_2 \text{ area} = (0.19) \cdot (541790) = 102940$$

Corrected

$$\text{Ar area} = 1.1257 \times 10^7 - 102940 = 1.1154 \times 10^7$$

$$\text{H}_2/\text{Ar area} = 1.365$$

B. From chromatogram of the final gas sample:

$$\text{H}_2 \text{ area} = 1.4161 \times 10^7$$

$$\text{Ar} + \text{O}_2 \text{ area} = 1.1707 \times 10^7$$

$$\text{N}_2 \text{ area} = 374840$$

$$\text{O}_2 \text{ area} = (0.19) (374840) = 71220$$

Corrected

$$\text{Ar area} = 1.1707 \times 10^7 - 71220 = 1.1636 \times 10^7$$

$$\text{H}_2/\text{Ar area} = 1.217$$

C. Calculation of number of initial gmoles of Argon

$$n_T = PV/RT \quad P = 55.42 \text{ atm}$$

$$V = 0.23 \text{ ml}$$

$$R = 0.0821$$

$$T = 296^\circ\text{K}$$

$$n_T = 0.525 \text{ gmoles}$$

$$\% \text{ Ar in initial sample} = 6.752$$

$$\% \text{ H}_2 \text{ in initial sample} = 93.231$$

$$\text{gmoles H}_2 \text{ in} = (0.525) (.93231) = 0.490$$

$$\text{gmoles Ar in} = (0.525) (.06752) = 0.035$$

$$\text{gmoles H}_2 \text{ out} = (\text{gmoles Ar in}) \left(\frac{\text{H}_2/\text{Ar area out}}{\text{relative response factor}} \right)$$

$$\text{gmoles Ar in} = 0.035$$

$$\text{H}_2/\text{Ar area} = 1.217$$

$$\text{RRF} = 10.331 \text{ (from calibration gas data)}$$

$$\text{gmoles H}_2 \text{ out} = 0.440$$

$$\text{gmoles H}_2 \text{ in} = 0.490$$

$$\text{difference} = 0.05 \text{ gmoles H}_2 \text{ consumed from gas}$$

Naphthalene make from chromatogram of solvent =

$$(0.379) (.05206 - .00468) = 0.01795 \text{ gmoles}$$

$$\text{Therefore H}_2 \text{ make} = (.01795) (2) = .036 \text{ gmoles}$$

$$\text{Total H}_2 \text{ consumption} = (.036) + (.050) = .086 \text{ gmoles}$$

2. Calculation of Carbon Balance and Yield

| Component | Area From Chromatogram* | $\frac{\text{Area of Comp. } i}{\text{Area of Ar}} = A'$ | RRF |
|-------------------------------|--------------------------|--|--------|
| C ₃ H ₈ | 309740 | 0.0262 | 0.6496 |
| C ₃ H ₆ | 129140 | 0.0109 | 0.6866 |
| i-C ₄ | 31176 | 0.0026 | 0.5171 |
| n-C ₄ | 64976 | 0.0055 | 0.5421 |
| CO ₂ | 5692900 | 0.4811 | 0.9766 |
| C ₂ H ₄ | - | - | 0.8685 |
| C ₂ H ₆ | 701270 | 0.0593 | 0.8119 |
| CH ₄ | 1382300 | 0.1168 | 1.2019 |
| CO | 1284200 | 0.1085 | 1.3253 |
| Ar + O ₂ | 1.1942 x 10 ⁷ | | |
| N ₂ | 578430 | | |
| Corrected Ar | 1.1832 x 10 ⁷ | | |

* Based on average of two samples.

| Component | $\frac{\text{gmoles of carbon}}{\text{gmoles of comp. } i} = B'$ | $\text{gmoles carbon out} = (N_{Ar})(A')(RRF)(B')$ |
|-------------------------------|--|--|
| C ₃ H ₈ | 3 | .00166 |
| C ₃ H ₆ | 3 | .00061 |
| i-C ₄ | 4 | .00017 |
| n-C ₄ | 4 | .00032 |
| CO ₂ | 1 | .01390 |
| C ₂ H ₄ | 2 | .00023 |
| C ₂ H ₆ | 2 | .00345 |
| CH ₄ | 1 | .00466 |
| CO | 1 | .00539 |

g. carbon in CO_x gases = 0.23148

g. carbon in total gases = 0.36468

% total carbon in feed shale = 25.075

% total carbon in spent shale = 4.37

% ash in feed shale = 58.86

% ash in spent shale = 90.21

g. shale out = (g. shale in) (ash in/ash out)

= (25) (.5886/.9021) = 16.312 g.

g. carbon converted = (g. shale in) (%C) -
(g. shale out) (%C)

= (25) (.25075) - (16.31) (.0437)

= 5.56 g

$$\begin{aligned}\% \text{ oil yield} &= (5.56 - 0.3647) / 5.56 \\ &= 93.44\%\end{aligned}$$

3. Calculation of Organic Carbon Conversion

$$\% \text{ organic C in feed} = 24.74$$

$$\% \text{ organic C in spent shale} = 4.32$$

$$\begin{aligned}\% \text{ converted} &= \frac{((\text{g shale in})(\text{org. C in feed}) - (\text{g shale in})(\text{ash in/ash out})(\text{organic carbon in spent shale}))}{(\text{g shale in})(\text{organic C in feed})} \times 100 \\ &= \frac{(25)(.2475) - (25)(.5886/.9021)(.0432)}{(25)(.2475)} \times 100 \\ &= 88.61\%\end{aligned}$$

4. Calculation of Percent Hydrogen Consumption and SCF/BBL

Assumptions: 1) carbon content of shale oil = 83% (wt)

2) specific gravity of oil product = 0.9

$$\begin{aligned}\text{g oil produced} &= \frac{((\text{g shale in})(\text{organic C in shale})(\text{organic C conversion})(\text{oil yield}))}{(\text{carbon content of shale oil})} \\ &= \frac{(25)(.25075)(.8861)(.9344)}{.83} \\ &= 6.25 \text{ g}\end{aligned}$$

$$\begin{aligned}\% \text{ hydrogen consumed} &= \text{g H}_2 \text{ consumed/g oil prod.} \times 100 \\ &= (.086)(2)(100) / 6.25 \\ &= 2.75\%\end{aligned}$$

$$\begin{aligned} \text{SCF/BBL} &= (350 \text{ lb/BBL})(0.9 \text{ sp gr})(359 \text{ SCF/lb mole}) \\ &\quad (\text{lb mole} / 2 \text{ lbs})(\% \text{ consumed}) \\ &= \text{SCF/BBL @ 1 atmosphere, } 32^{\circ}\text{F} \\ &= (350)(.9)(359)(.0275) / 2 = 1555 \text{ SCF/BBL} \end{aligned}$$