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DOE/MC/11089-3212

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DE93 008639

PRESSURIZED FLUIDIZED-BED HYDRORETORTING OF EASTERN OIL SHALES

Progress Report for the Period June-August 1992

By
M. J. Roberts
M. C. Mensinger
D. M. Rue
F. S. Lau

September 1992

Work Performed Under Contract No.: DE-AC21-87MC11089

For
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
Morgantown, West Virginia 26505

By
Institute of Gas Technology
Chicago, Illinois 60616

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EXECUTIVE SUMMARY

This report presents the work performed during the program quarter from June 1, 1992 through August 31, 1992, under U.S. Department of Energy (DOE) Contract No. DE-AC21-87MC11089 for the project "Pressurized Fluidized-Bed Hydroretorting (PFH) of Eastern Oil Shales." The Institute of Gas Technology (IGT) is the prime contractor for the program extension to develop the PFH II system technology. Four institutions are working with IGT as subcontractors; their responsibilities and achievements in the program are discussed below in the appropriate tasks.

Some of the tasks in the original program were not continued in the current program extension. The task achievements discussed below represent the active tasks of the program.

In Subtask 3.7 (Innovative Reactor Concept Testing), IGT prepared detailed design and construction drawings for modifications to the laboratory-scale cracking unit. Also, a new reactor was fabricated from a length of 1.25-inch schedule 40 type 309 stainless steel pipe. Cracking tests will be initiated next quarter. A detailed operating procedure is being developed.

In Subtask 3.9 (Catalytic Hydroretorting), IGT attempted to impregnate beneficiated Alabama shale with throw-away catalysts. However, the catalysts formed colloidal suspensions and were not uniformly dispersed throughout the solids. Next quarter, IGT will prepare samples of shale impregnated with catalysts that are soluble in the selected solvent.

Catalyzed shale hydroretorting tests will be conducted in the existing laboratory-scale PFH batch reactor at a specific set of test conditions (925°F and 1000 psig for 20 minutes) to provide a statistically valid basis for comparing oil yields and properties.

Also during the quarter, IGT conducted five replicate PFH tests at the selected conditions with uncatalyzed shale. The results of these tests will provide a statistically valid bench mark for comparing the effects of various catalysts on oil quantity and quality.

In Subtask 3.10 (Autocatalysis in Hydroretorting), the University of Alabama (UA) prepared the bench-scale batch autoclave unit for hydroretorting experiments and developed procedures for carrying out the experiments. UA has also prepared a standard sample of oil shale concentrate for the autocatalysis experiments.

In Subtask 3.11 (Shale Oil Upgrading and Evaluation), IGT identified three catalysts as possible conventional shale oil upgrading catalysts, including two commercial oil hydrotreating catalysts and a catalyst to be prepared at IGT. During the quarter, IGT readied the commercial catalysts for oil upgrading tests. IGT also prepared two catalysts for unconventional oil upgrading tests. IGT modified an existing laboratory-scale oil upgrading unit. Oil upgrading tests will begin next quarter.

The University of Alabama Mineral Resources Institute (MRI) is responsible for the work being conducted in Task 4 (Beneficiation Research).

In Subtask 4.1.3 (Stirred Ball Mill Grinding), MRI designed and fabricated a 1.8-liter volume polyurethane lined continuous attrition grinder. The grinder was built to avoid the mill wear by steel media and to minimize the quantity of material required for test runs.

In Subtask 4.1.5 (Alternative Technology Evaluation), MRI began their evaluation of four comminution technologies that could be used in the beneficiation process. These technologies are roller mills, roller presses, vibratory mills, and semi-autogenous grinding. During the quarter, MRI personnel visited roller mill and roller press installations in Alabama and Michigan. Their evaluation showed that the roller mill had an energy consumption of about 15 to 20 kWh/ton for a chalky limestone. Because this material is substantially softer than a typical shale, MRI concluded that there would be no cost savings in applying this technology to shale beneficiation. Therefore, no further evaluation of roller mills will be made.

In the roller press installation, about 10 percent of the power is consumed in the hammer mills with the balance approximately equally divided between the roller press and the ball mill (i.e., 6.75 to 8.7 kWh/ton each). Thus, the power consumption in a roller press is slightly higher than that in a tertiary crusher (such as a ball mill), which it might replace. However, because a roller press generates a considerable amount of fines, the quantity of material that must be ground in the ball mill would be reduced. This would reduce the power consumption in the ball mill. The power consumption in the ball mill at the plant was substantially lower than what MRI personnel had estimated for the primary ball mill in a shale grinding circuit.

In Subtask 4.1.6 (Ultrafine Size Separation), MRI ground about 765 pounds of Alabama shale in a ball mill to -150 mesh to produce feedstock for the ultrafine size separation studies. The results of these grinding operations showed that about 65 percent of the -150 mesh material is finer than 24 μm, the desired size for flotation studies.

In Subtask 4.2.1 (Column Flotation Tests), MRI concentrated on developing a computer model of column flotation for ultrafine ground shale. The model requires three parameters: the mean residence time (t), the vessel dispersion number (Nd), and the first-order flotation kinetic coefficient (K). The first two parameters can be estimated from experimental data; the last one can be determined with additional mathematical models related to flotation hydrodynamics.

To test the applicability of the model, MRI conducted a series of tests to measure the mixing behavior of liquid and solid particles in the deep froth column. The experimental mean residence time and the vessel dispersion number were initially determined to be 12.79 min and 0.14, respectively. The mean residence time estimated by the model was 12.76 min. Based on these preliminary results, the axial dispersion model appears suitable for the deep froth flotation column.

MRI also developed a correlation to evaluate x-ray backscattering intensity as a means of predicting the oil yield from shale oil. Backscattering intensity values were calculated for different wavelengths and correlations were obtained between scattering intensity and oil yield. Next quarter, MRI will continue to refine the parameters in the equation to improve the correlation.

In Subtask 4.4 (Integrated Grinding and Flotation), the University of Kentucky Center for Applied Energy Research (UK-CAER) conducted column flotation and dewatering studies with Alabama Chattanooga shale. A baseline flotation test was conducted on shale ground to 90 percent passing 10-μm size. The froth product was cleaned in six stages using a Denver flotation machine. The purpose of this test was to establish a guideline for grade and recovery of kerogen that could be obtained using conventional flotation. The results show that recovery and grade have a linear relationship. With conventional flotation, a concentrate containing 30 percent organic carbon was obtained at a recovery of about 60 percent.

Initial pilot-scale column flotation studies were conducted to evaluate the effect of feed rate and retention time of slurry on grade and recovery of kerogen. The results showed that feed rate did not significantly affect carbon recovery. However, a high-grade (≈33 percent carbon) product was obtained at a feed rate of 1 gallon per minute.

In other tests conducted in the pilot-scale column with an air flow rate of 0.3 SCFM, recoveries of over 50 percent were not achieved even when the retention time was extended beyond 20 minutes. Tests conducted previously in the 2-inch column achieved about 80 percent recovery of about 40 percent carbon grade kerogen at a 20-minute retention time. Increasing the air flow to 0.5 SCFM, at a lower retention time of 10 to 15 minutes yielded even lower kerogen recovery. UK-CAER can not explain why the data obtained from the pilot-scale column does not agree with those obtained previously in the 2-inch column. UK-CAER personnel are conducting additional tests in an attempt to explain the discrepancy.

UK-CAER conducted vacuum and pressure filtration tests with froth from the column flotation tests. After 2 minutes of filtration with a vacuum of 22.5 inches of Hg, a filter cake containing 45 percent moisture was obtained. Increasing filtration time did not improve moisture reduction. Pressure filtration conducted using 40 psig pressure also yielded a filter cake with a moisture content of 45 percent moisture. Increasing the filtration time to 5 minutes yielded a cake with a moisture content of about 40 percent. This reduction could be due to air drying, which occurs in pressure filtration after cracks develop in the cake. Increasing the filtration pressure to 60 psig yielded a 35 percent moisture filter cake after 2 minutes. After 5 minutes of filtration at this pressure, a 20 percent moisture was obtained in the filter cake, some of which may be due to air drying.

Next quarter, UK-CAER plans to conduct additional pilot-scale column tests to improve carbon recovery. Tests will also be conducted using the modified SYMUSEP separator column.

In Subtask 4.7 (Economic Analysis), MRI began to update the estimate of costs to beneficiate Alabama shale to reflect inflation. Cost indices in Chemical Engineering magazine were selected as the basis for updating the capital cost estimate. The preliminary updated capital cost estimate represents an increase of 4.9 percent from June 1988 to June 1992 dollars.

In Subtask 6.2.2 (Wastewater Treatability), Tennessee Technological University (TTU) prepared samples of PFH product water for treatability studies. The ammonia and hydrogen sulfide were removed by air stripping the product water. TTU also initiated acclimation of microbial seed to dilutions of these stripped product water samples. Biological growth was observed in solutions containing up to 50 volume percent PFH product water. TTU continued attempts to acclimate microorganisms to a pure compound, 4-methyl pyridine.

In Subtask 6.2.3 (Waste Management Facility Conceptual Design), TTU estimated the total quantity of water required for a commercial-scale PFH plant to be 10.7 million gallons per day. Three major areas of water consumption are boiler feed water, quench water, and cooling. TTU is also compiling water quality criteria for initial import water and for reuse and recycling.

In Task 8 (Project Management and Reporting), IGT personnel visited MRI, UK-CAER, UA, and TTU during the quarter to review work plans and tour their facilities. IGT continued to monitor the progress and activities of the subcontractors working in the program via telephone and written reports.

In Task 9 (Information Required for the National Environmental Policy Act), IGT prepared and submitted the NEPA document to DOE for review and comments. The NEPA document contains input from IGT as well as each of the subcontractors participating in the program.

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INTRODUCTION

The Devonian oil shales of the Eastern United States are a significant domestic energy resource. The overall objective of the multi-year program, initiated in October 1987 by the U.S. Department of Energy (Contract No. DE-AC21-87MC11089), is to perform the research necessary to develop the pressurized fluidized-bed hydroretorting (PFH) process for producing oil from Eastern oil shales. The program also incorporates research on technologies in areas such as raw shale preparation, beneficiation, product separation and upgrading, and waste disposal that have the potential of improving the economics and/or environmental acceptability of recovering oil from oil shales using the PFH process. The program is divided into the following active* tasks:

- Task 3. Testing of Process Improvement Concepts
- Task 4. Beneficiation Research
- Task 6. Environmental Data and Mitigation Analyses
- Task 8. Project Management and Reporting
- Task 9. Information Required for the National Environmental Policy Act

In order to accomplish all of the program objectives, the Institute of Gas Technology (IGT), the prime contractor, is working with four other institutions: The University of Alabama/Mineral Resources Institute (MRI), the University of Alabama College of Engineering (UA), University of Kentucky Center for Applied Energy Research (UK-CAER), and Tennessee Technological University (TTU).

This report presents the work performed during the program quarter from June 1, 1992 through August 31, 1992.

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* Some of the tasks in the original program were not continued in the current program extension. The task achievements discussed below represent the active tasks of the program.

ACHIEVEMENTS

Task 3. Testing of Process Improvement Concepts

The objective of this task is to obtain data on novel process concepts that have the potential to improve the economics for PFH processing of beneficiated Eastern shales. This task is divided into four active subtasks that correspond to the novel concepts being tested: 3.7. Innovative PFH Reactor Concept Testing (Methane Cracking and Cracking-Combustion Tests), 3.9. Catalytic Hydroretorting, 3.10. Autocatalysis in Hydroretorting, and 3.11. Shale Oil Upgrading and Evaluation. Subtask 3.10 is being conducted by the University of Alabama College of Engineering (UA).

Subtask 3.7. Innovative Reactor Concept Testing

The objective of this subtask is to demonstrate the technical feasibility of generating hydrogen by cracking by-product methane gas and combusting the carbon produced by the cracking reactions. Work is divided into two subtasks: 3.7.1. PFB Methane Cracking and 3.7.2. PFBC of Carbon From Cracking.

Subtask 3.7.1. PFB Methane Cracking

The objective of this subtask is to determine operating conditions needed to crack the hydrocarbon gases from the PFH process to generate hydrogen and carbon by conducting laboratory-scale tests. An existing laboratory-scale reactor will be modified to include continuous gas flow through a batch fluidized bed. On-line gas detectors will be used to collect data during the cracking and combustion tests.

Discussion

During the quarter, IGT prepared detailed design and construction drawings for modifications to the laboratory-scale cracking unit. A new reactor was fabricated from a length of 1.25-inch schedule 40 type 309 stainless steel pipe and prepared for installation in the pressure shell. The reactor system was modified to allow continuous feeding of gas to the bottom of the reactor, which passes through a batch of hydroretorted shale supported by a bed of alumina balls (4- to 6-mm diameter). A schematic diagram of the modified cracking-combustion unit is shown in Figure 3-1.

The reactor, external thermocouple, and differential pressure controller/gauge will be installed early next quarter to complete the unit assembly. After the unit has been assembled, pressure and heating tests will be conducted prior to initiating the cracking tests. A detailed operating procedure is being developed.

Subtask 3.7.2. PFBC of Carbon From Cracking

The objectives of this subtask are to demonstrate the removal of deposited carbon from the surface of the spent beneficiated shale by combustion and to evaluate shale for cracking effectiveness and particle stability

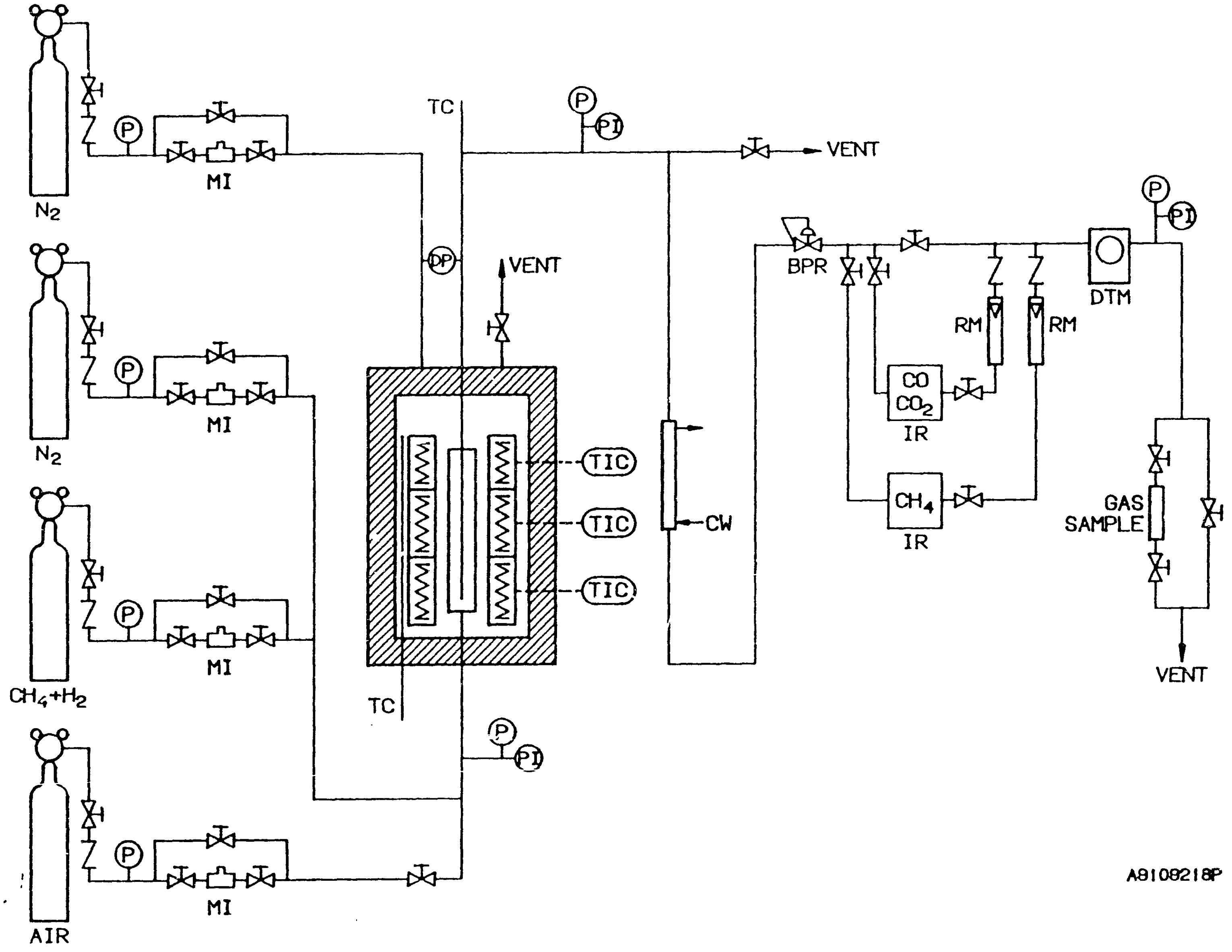

Figure 3-1. SCHEMATIC DIAGRAM OF THE MODIFIED CRACKING AND COMBUSTION APPARATUS

through multiple cycles of cracking and combustion. The apparatus being built for tests in Subtask 3.7.1 will also be used for these tests.

Discussion

This subtask was inactive during the quarter pending completion of modifications to the cracking unit.

Subtask 3.9. Catalytic Hydroretorting

The objective of this subtask is to improve the quality of PFH-produced shale oil in-situ by utilizing catalysts. Tests will be conducted in a small laboratory-scale hydroretorting unit using hydrotreating catalysts. These catalysts will be used in the retort itself and will be of two types, regenerable and throw-away.

Discussion

During the quarter, IGT attempted to impregnate beneficiated Alabama shale with throw-away catalysts. In these attempts, however, the catalysts formed colloidal suspensions and were not uniformly dispersed throughout the solids. In the preferred procedure for catalyst impregnation to a surface, the catalyst should be solubilized in the solvent to ensure uniform catalyst dispersion throughout the solid.

Based on the results of the catalyst impregnation attempts, IGT prepared a list of eleven potential catalysts (Table 3-1). Materials needed to prepare these catalysts are either available at IGT or have been ordered.

Table 3-1. THROW-AWAY AND REGENERABLE CATALYSTS

Throw-Away Catalysts	Regenerable Catalysts
$(NH_4)_6Mo_7O_{24}$	MoS_{2+X} on Al_2O_3
MoS_{2+X} (as colloid)	Ni + MoS_{2+X} on Al_2O_3
$Mo(CO)_6$ (as colloid)	Co + MoS_{2+X} on Al_2O_3
$Ni(CH_3CO_2)_2$	Fe + MoS_{2+X} on Al_2O_3
$Co(CH_3CO_2)_2$	MoS_{2+X} on $ZrO_2 \cdot CaO$
$FeOH(CH_3CO_2)_2$	

These catalysts selected can be dispersed evenly on either the shale or a desired support. The colloidal suspensions can also be evenly applied. The MoS_{2+X} catalyst will be prepared by adding ammonium tetrathiomolybdate (ATM) to the shale or support and then decomposing the ATM at elevated temperature to produce MoS_{2+X}. The ammonium molybdenum oxide catalyst [$(NH_4)_6Mo_7O_{24}$] will likely decompose at elevated temperature to form MoO_X.

The catalyzed shale hydroretorting tests will be conducted in the existing laboratory-scale PFH batch reactor. A specific set of test conditions (925°F and 1000 psig for 20 minutes) will be used in the tests to ensure a valid basis for comparing individual tests for oil yield and properties. This set of operating conditions was found to provide the highest oil yield from uncatalyzed Alabama shale in previous work.

During the quarter, IGT conducted five replicate PFH tests at the selected conditions with uncatalyzed shale. The results of these tests will provide a statistically valid bench mark for comparing the effects of various catalysts on oil quantity and quality. After the baseline tests were completed, the reactor was prepared for the first test with catalyzed shale. Material balances will be made when laboratory analyses of the baseline test samples are completed. The baseline test data will be compiled next quarter and testing will begin with the catalyzed shale.

Subtask 3.10. Autocatalysis in Hydroretorting

The objective of this subtask is to investigate the effects of potentially catalytic minerals or alkali metal oxides naturally present in shale on hydroretorting characteristics and product yields. Tests will be conducted in an existing laboratory-scale batch reactor using as-received shale and shale impregnated with potentially catalytic metals.

Discussion

During the quarter, UA prepared the bench-scale unit for hydroretorting experiments and developed procedures for carrying out the experiments. The reactor is an Autoclave Engineers Model 79-07117 vessel capable of operating at 1000°F and 3000 psig. It is a bolted closure reactor, consisting of a body, cover, closure gasket, draft tube, cap screws, and two retainer screens, one of which is welded to the thermocouple well of the reactor. All gas-contacted parts are type 316 stainless steel. The reactor has a volume of 5.9 cubic inches. The reaction gas-vapor mixture is stirred by a magnetically activated packless impeller system that consists of a 1/4-HP DC motor, coupled with a rheostat, switch and RPM meter.

Reactor heating is provided by an electric jacket-type furnace with the resistance winding and insulation enclosed in a sleeve-type jacket. The furnace slides over the body of the reactor. It is divided into three heating zones (top, middle and bottom), which are wired into three separate electric circuits. The furnace and reactor temperatures are maintained by separate controllers. The furnace controller input signal is from a type K thermocouple. A 5-channel digital thermometer with type K thermocouples indicates gas temperatures at the reactor top and bottom, gas preheater outlet, reactor inlet line, and the product gas leaving the liquid collector.

Nitrogen is used to purge the system. Hydrogen is fed through a 1/8-inch capillary flowmeter. The flowmeter is calibrated to supply a nominal 0.5 standard liters per minute (SLPM) at 400 psig. The hydrogen flows through a preheater consisting of a coil of tubing cast in heat-transfer cement. Electrical strip heaters attached to the outside of the preheater are controlled manually by a rheostat to give the desired gas outlet temperature. The gas then passes through a short line to the reactor. This line is wrapped with a heating tape also on a rheostat. Inlet gas pressure is measured by a precision Heise pressure gauge. The reactor product gas passes through an in-line filter to collect entrained particles from the reactor. It then flows through a tee that can be used to introduce solvent to wash the exit line. The product gas then goes through a heat exchanger and into the liquid collector. The liquid collector vessel is constructed of Monel and has a volume of 1 liter. The product gas exits the liquid collector, passes through a back-pressure regulator, through an in-line filter, and to a gas receiving tank. The gas receiving tank has a volume of about 1.6-cubic feet and is used as to collect the total quantity of gas produced. A thermocouple and a precision pressure gauge measure the temperature and pressure of this tank.

A Hewlett-Packard 5890-A gas chromatograph (GC) is used to determine product gas and liquid compositions. It has a keyboard and alphanumeric display to define instrument set points with two channels of electronic flow sensing. Thermal conductivity and flame ionization detectors are available. The gas column is 3.2 mm by 6.1 m long and is packed with -60+80 mesh VZ-7. The carrier gas is helium, the oven temperature is maintained at 40°C, and the thermal conductivity detector is used. A capillary column is used to determine the equivalent boiling point curve of the liquid product. The column has a 0.32 mm ID (inside diameter), is 30 m long, and is coated with dimethyl polysiloxane.

A standard sample of oil shale concentrate has been prepared for the autocatalysis experiments. The sample is stored moist in an air-free environment. Samples will be dried under argon using standard procedures for drying coal samples. Prior to each test, about 50 grams of shale will be charged to the reactor. After sealing, the reactor will be purged with nitrogen for 15 minutes to eliminate air. The pressure regulator will be set at 400 psig and hydrogen flow will be initiated at about 0.5 SLPM. The reactor will be heated at 8°F/min until the reactor temperature reaches 900°F. Liquid and gas products will be collected continuously during operation.

The gas receiving tank will also be evacuated before a test. The total quantity of gas produced will be determined by measuring the volume, temperature, and pressure of the gas receiving tank. The gas composition will be determined by GC.

After a test, the system is depressurized and the liquid product is drained. The product line from the reactor through the collector is washed out with a known amount of solvent (hexane). This procedure will be used to determine if product liquid condenses on the walls of the tubing and if so, to recover those liquids quantitatively. The liquid density will be measured and the carbon and hydrogen contents determined by a Leco analyzer. Residue solids collected from the reactor will be weighed and the composition determined by a Leco analyzer.

Subtask 3.11. Shale Oil Upgrading and Evaluation

The objective of this subtask is to investigate conventional and novel methods for upgrading the oil produced by shale hydroretorting. This subtask is divided into two subtasks: 3.11.1. Conventional Upgrading, and 3.11.2. Novel Approach to Upgrading.

Subtask 3.11.1. Conventional Upgrading

The objective of this subtask is to evaluate conventional hydrotreating technology for upgrading shale oil produced from the PFH process to transportation fuel.

Discussion

Three catalysts have been identified as possible conventional shale oil upgrading catalysts. These catalysts include two commercial oil hydrotreating catalysts and a catalyst to be prepared at IGT. The IGT catalyst will be MoS_{2+X} on $ZrO_2 \cdot CaO$. During the quarter, the two commercial catalysts were readied for testing. The first consists of a mixture of nickel, molybdenum, and phosphorous oxides on alumina; the second consists of a mixture of cobalt, molybdenum, and phosphorous oxides on alumina. The IGT catalyst will be prepared when needed.

These three catalysts will be tested at 752°F (400°C) and 2000 psig in a hydrogen atmosphere. Oil elemental analyses, specific gravities, and simulated distillation data will be used to evaluate the degree of upgrading achieved.

An existing laboratory-scale oil upgrading unit at IGT was modified this quarter for tests in this subtask. A schematic diagram of the modified apparatus is presented in Figure 3-2. A catalyst sample of about 20 grams is charged to the reactor before each test. The catalyst is sulfided if necessary and then maintained at the desired temperature and pressure while oil at 19 mL per hour and feed gases are passed downward through the bed. Oil and product gas samples are collected periodically during each test.

The oil feed pump and mass flow controllers were calibrated, and the furnace operation and control were confirmed. A pressure test with hydrogen was successfully completed at 2000 psig. After all equipment checks were completed, the first commercial catalyst was loaded in the reactor, and raw shale oil was charged to the pump. The first oil upgrading test will be conducted next quarter. Each oil upgrading test will have a steady-state period of at least 4 hours. Steady state will begin after a uniform rate of oil collection is established. Gas and oil samples will be taken every hour to determine the effects of each catalyst on oil properties and to provide samples for material balance calculations.

Oil upgrading tests will be conducted next quarter under usually acceptable hydrotreater design conditions with the two commercial hydrotreating catalysts and the MoS_{2+X} on $ZrO_2 \cdot CaO$ catalyst prepared at IGT. The effects of these catalysts on oil quality and composition will be determined. Results will be compared with novel oil upgrading test results from Subtask 3.11.2.

Subtask 3.11.2. Novel Upgrading

The objective of this subtask is to determine if the whole product oil can be hydrotreated in a novel one-step catalytic process without the use of hydrogen or severe operating conditions.

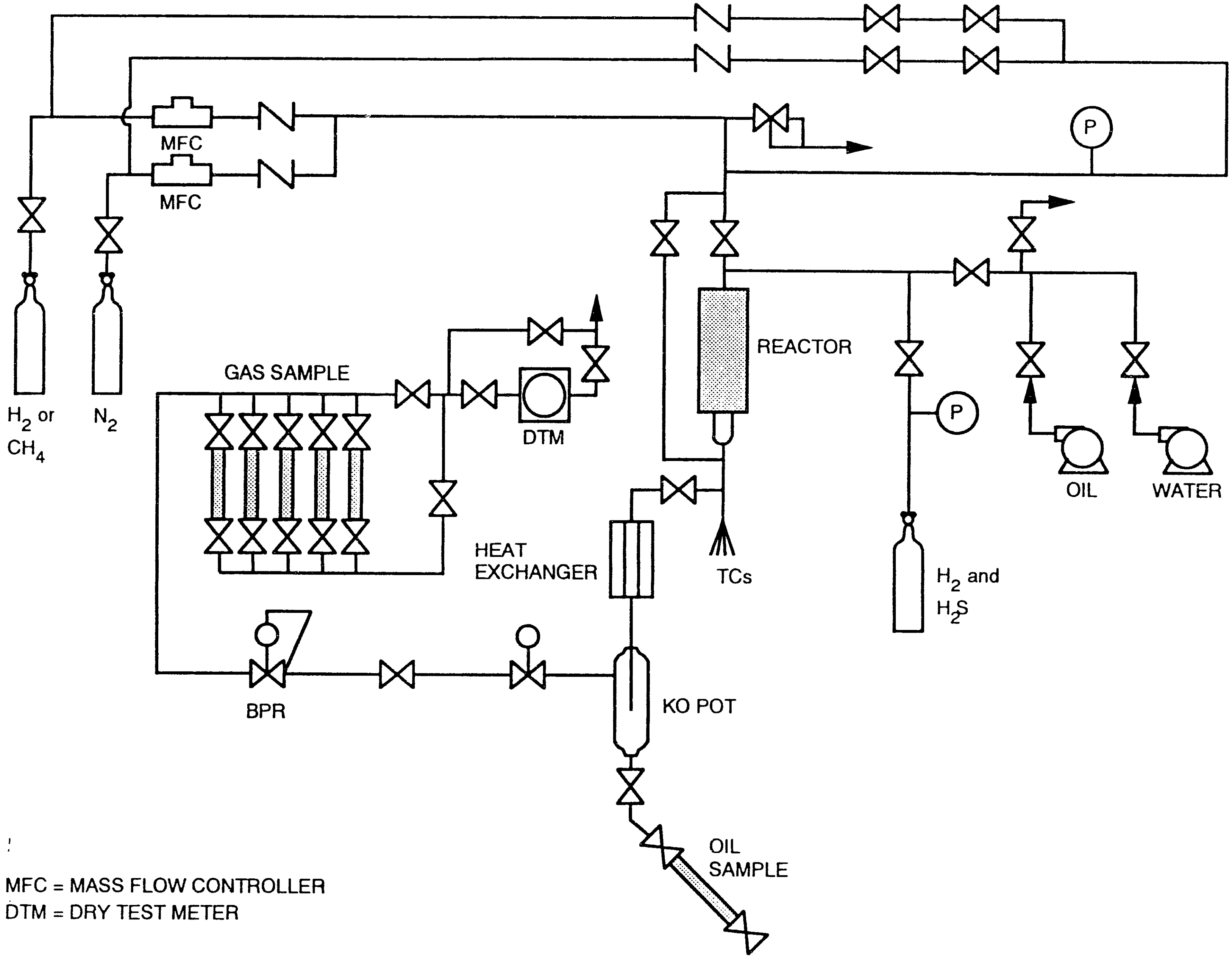

Figure 3-2. SCHEMATIC DIAGRAM OF THE OIL UPGRADING UNIT

Discussion

Novel shale oil upgrading tests will be conducted in the same reactor modified and used for conventional oil upgrading tests in Subtask 3.11.1. Six potential catalysts for shale oil upgrading in a methane atmosphere have been identified and are presented in Table 3-2. The first two catalysts listed in Table 3-2 were prepared this quarter and are on hand for oil upgrading tests. The other four catalysts will be prepared as needed for upgrading tests. The catalysts were prepared by combining ammonium tetrathiomolybdate (ATM) with the support and then decomposing the ATM at elevated temperature to produce MoS_{2+X}. MoO_X catalysts will be prepared using ammonium molybdenum oxide - $(NH_4)_6Mo_7O_{24}$.

Table 3-2. NOVEL SHALE OIL UPGRADING CATALYSTS

MoS_{2+X}	on	CaO
MoS_{2+X}	on	$TiO_2 \cdot CaO$
MoS_{2+X}	on	$ZrO_2 \cdot CaO$
MoS_{2+X}	on	$ZrO_2 \cdot Y_2O_3$
MoO_X	on	CaO
MoO_X	on	$ZrO_2 \cdot CaO$

During the quarter, IGT modified the shale oil upgrading unit. Tests will be conducted after completion of the conventional oil upgrading tests in Subtask 3.11.1.

Task 4. Beneficiation Research

The objective of this task is to test several novel and advanced grinding and beneficiation concepts for processing Eastern oil shales. The ultimate goal is to develop a minimum-cost system for delivering shale concentrates of suitable form to the PFH process. The Mineral Resources Institute (MRI) at the University of Alabama has primary responsibility for this part of the program.

The active work in this task is divided into four subtasks: 4.1. Grinding Studies, 4.2.1. Column Flotation Tests, 4.4. Integrated Grinding and Flotation, and 4.7. Economic Analysis. Subtask 4.4 is being conducted by the University of Kentucky Center for Applied Energy Research (UK-CAER).

Subtask 4.1. Grinding Studies

This subtask is divided into three subtasks: 4.1.3. Stirred Ball Mill Grinding, 4.1.5. Alternative Technology Evaluation, and 4.1.6. Ultrafine Size Separation.

Subtask 4.1.3. Stirred Ball Mill Grinding

During the quarter, MRI designed and built a small-scale polyurethane-lined continuous attrition grinder, which is dimensionally similar to the LME-4 Netzsch mill. The major difference between these two mills is that the attrition mill is in a vertical configuration whereas the Netzsch mill is in a horizontal configuration. The reasons for fabricating this grinder were to avoid mill wear by the steel media and to minimize the quantity of material required for test runs.

The working volume of the attrition mill is 1.8 liters. Presently, efforts are underway to modify the mechanical and electrical circuitry to overcome the resistance offered by media loading in achieving the rotor speed range of 2500 to 3000 rpm.

Subtask 4.1.5. Alternative Technology Evaluation

The objective of Subtask 4.1.5 is to evaluate several alternative comminution technologies to determine which could replace conventional technologies in the oil shale beneficiation scheme.

Discussion

Four alternative comminution technologies were selected for evaluation in this program: roller mills, roller presses, vibratory mills, and semi-autogenous grinding (SAG). During the quarter, MRI personnel visited roller mill and roller press installations in Alabama and Michigan.

The Lafarge Corporation operates a cement plant at Demopolis, Alabama. The plant produces cement from a local "chalk" formation. The so-called "chalk" is actually a weakly consolidated, siliceous limestone that approximates, in composition, a natural cement rock. A small amount (about 3 wt %) of iron ore and clay are added to the chalk to adjust the composition to that desired for cement making.

The raw natural mix is ground to 90 percent passing 200 mesh [74 micrometers (μm)] in a single roller mill. The roller mill consists of 3 steel-faced rollers rotating in an annular track about a central cone. The roller mill is enclosed and is constantly swept by heated air. The air acts both to dry the feed and to convey the product through a series of air classifiers (cyclones).

The apparent energy consumption attributable to comminution is quite low. The roller mill consumes about 500 kW in grinding an average of 220 tons of raw material per hour. The fans, however, which provide the air sweep through the mill and power the air classifiers, consume 2500 kW. In addition, a substantial amount of energy is consumed in heating the air to dry the raw material.

When the power to the fan and thermal energy are included, the total power consumption in the system is approximately 15 to 20 kWh/ton. When one considers that the raw material at the Demopolis plant is significantly softer than a typical shale, there is no apparent opportunity to reduce the cost of grinding shale by the application of roller mill technology. Therefore, no further evaluation of roller mills will be made.

Roller presses are a relatively recent adaptation of the long established roll crusher. A roll crusher consists of two counter-rotating horizontal cylinders (rolls) that nip and break rock fragments between them. The size of the roll crusher product is fixed by adjusting the gap between the rolls. Fragments smaller than the gap pass through untouched. An important attribute of roll crushers is the tendency to produce a minimum amount of fines.

Roller presses, in contrast to roll crushers, are designed and operated to maximize fines production. Roller presses differ from roll crushers in that there is no fixed gap between the rolls, instead hydraulic cylinders maintain a high constant pressure on the rock between the roll faces. During typical operation, the roller presses are "choke fed" to maintain multiple layers of rock fragments between the rolls. The high applied pressure causes a high degree of rock-upon-rock breakage and a resulting high rate of fines production. The high pressures between the rolls also force the broken rock to re-consolidate as rather friable flakes.

The Lafarge Corporation cement plant at Alpena, Michigan uses roller presses in its raw-grind circuit. The raw material mix, consisting of 82 percent limestone and 18 percent Antrim shale, enters the plant at a nominal -3-inch size. It is blended and fed to the roller presses. Each roller press is driven by two 1200 HP motors. Material from the roller press discharges into a 500 HP hammer mill where it is "deagglomerated" and dried. The deagglomerated product from the hammer mills is classified and the finished product is sent directly to the homogenizing silos. Classifier oversize is further ground in a 12 x 24 foot ball mill driven by a 2250 HP motor. A portion of the oversize is normally returned to the roller presses to provide bridging material, which enhances the rock-upon-rock crushing action.

The raw grinding circuit used in the Alpena plant consists of two parallel circuits each having a roller press, hammer mill, and ball mill operating in series. During normal operation, the capacity of each circuit is about 200 t/h with power consumption, exclusive of thermal energy, in the range of 15 to 19 kWh/ton.

About 10 percent of the power is consumed in the hammer mills with the balance approximately equally divided between the roller press and the ball mill (i.e., 6.75 to 8.7 kWh/ton each). Thus, the power consumption in a roller press is slightly higher than that in a tertiary crusher, which it might replace. However, because a roller press generates a considerable amount of fines, the quantity of material that must be ground in the ball mill would be reduced. This would reduce the power consumption in the ball mill. The power consumption in the ball mill at the plant was substantially lower than what MRI personnel had estimated for the primary ball mill in a shale grinding circuit.

Therefore, in spite of certain negative aspects, roller presses may have applications in oil shale beneficiation. Further evaluation will be performed next quarter. The evaluation of SAG milling will also be initiated.

Subtask 4.1.6. Ultrafine Size Separation

About 765 pounds of Alabama shale were ball mill ground to produce feed stock for the ultrafine size separation studies. The grinding circuit consisted of a constant weight feeder, a Denver 18 x 30 inch ball mill, and a 150 mesh Sweco vibrating screen to close the grinding circuit.

While producing the feedstock for the ultrafine size separation studies, the dry solids feed rate to the ball mill circuit was varied from two to three pounds per minute at 50 percent solids to determine the effect of increased circulating load on the size consist of the ball mill discharge and the quantity of -24 μm material produced. The data in Table 4-1 shows that increasing the feed rate from 1.84 to 2.97 lb/min, significantly increased the size of the mill discharge from a d_{90} of 172.9 to 257.8 μm.

Table 4-1. EFFECT OF INCREASED CIRCULATING LOAD
ON SIZE CONSIST OF BALL MILL DISCHARGE

Feed Rate lb/min	Circulating Load, %	Mill Discharge Size, μm		-24 μm	
		d_{90}	d_{50}	%	lb/min
1.84	16	172.9	37.0	43.9	0.81
2.97	77	257.8	71.7	31.3	0.93

Size analysis of the -150 mesh shale; i.e. feedstock for the ultrafine sizing studies, at the two different ball mill feed rates gave the results shown in Table 4-2. While this data indicate some minor differences in size, based on feed rate and/or circulating load, the data also shows that about 65 percent of the -150 mesh material is finer than 24 μm, the desired size for flotation studies.

Table 4-2. EFFECT OF INCREASED CIRCULATING LOAD
ON SIZE CONSIST OF SCREEN UNDERSIZE

Feed Rate lb/min	Circulating Load, %	Screen Undersize, μm				μm	
		+64	-64+24	-24+12	-12	d_{90}	d_{50}
1.84	16	8.8	24.3	21.3	45.6	58.9	13.7
2.97	77	10.0	24.8	21.0	44.2	63.6	14.2

Preliminary hydrocyclone studies of Alabama oil shale, which had been ball mill ground to pass 150 mesh, were completed during the reporting period. Because hydrocycloning studies conducted previously had indicated that the most effective separations were obtained using relatively high feed solids and low feed pressures, the present studies were conducted at 30 percent solids and feed pressures of 10, 15 and 20 psig.

The hydrocyclone studies were conducted using a 2-inch Mozley cyclone with variable vortex finders (8, 11 and 14 mm) and variable apex spigot sizes (3.2, 4.5, 6.4, and 9.4 mm). Data collected during the hydrocycloning studies included cyclone overflow and underflow rates and solids contents, size analysis and size distribution.

MRI will conduct column cell flotation of the shale having a size consist of about 24 μm. Determination of the response of the -24 μm fraction of the hydrocyclone feed to the various operating conditions was the primary objective of this test series. The data given Table 4-3 shows the effect of increasing the size of the cyclone vortex finder at constant apex spigot size and feed solids. These results represent the most effective conditions found in each series of tests.

Table 4-3. EFFECT OF VORTEX FINDER SIZE ON REMOVAL OF -24 μm MATERIAL FROM CYCLONE FEED (30% solids, 6.4-mm Apex Spigot)

Vortex Finder, mm	Feed* psig	Cyclone Overflow Size, μm	Cyclone Overflow % -24 μm	Solids Recovery, lb/min
8	10	23.2	31.8	3.3
11	15	23.4	45.8	5.2
14	10	23.4	56.3	11.4

* The feed consisted of 33.1% +24 μm, 21.3% -24+12 μm, and 45.6% -12μm material.

Data in Table 4-3 shows that at a constant d_{90} of about 23.3 μm, increasing the size of the vortex finder at a constant feed solids and apex spigot size increased the recovery of the -24 μm material from 31 to 56 percent. The increase in vortex finder size also gave an increase in production rate at constant size, which in effect, would cause a decrease in the number of cyclones required.

Figure 4-1 shows the relationship between the cyclone feed rate at constant solids and cyclone overflow d_{90} (23 μm) when the vortex finder to apex spigot is changed. In this case the apex spigot size was held constant at 6.4 mm and the vortex finder sizes were 8, 11, and 14 mm. Although there is a difference in the cyclone feed pressure required when using the 11 mm vortex finder, the data is based on the production rate at a given d_{90}.

Figure 4-2 shows the effect of vortex finder size on the cumulative size distribution of the cyclone underflow product. The tests were conducted with the 6.4 mm apex spigot. For an overflow product size of about 23 μm (d_{90}), increasing the vortex finder from 8 to 14 mm increased the d_{50} of the underflow product from about 19 to 30 μm.

Figure 4-3 shows the cumulative size analysis of the cyclone underflow products at constant vortex finder and apex spigot size when varying the feed pressure. The data indicates only minimal changes in the size of the cyclone underflow product. Hence, the lower feed pressure is indicative of lower pumping costs. Data in Figure 4-4 shows the effect of increased feed pressure on the cyclone overflow product at constant feed solids and cyclone vortex and apex sizes. The results show little advantage in increasing the cyclone feed pressure above 10 psi.

Table 4-1 shows that only 56 percent of the -24 μm material in the cyclone feed was removed in the cyclone overflow under the best test conditions. One additional test was made to investigate the potential for

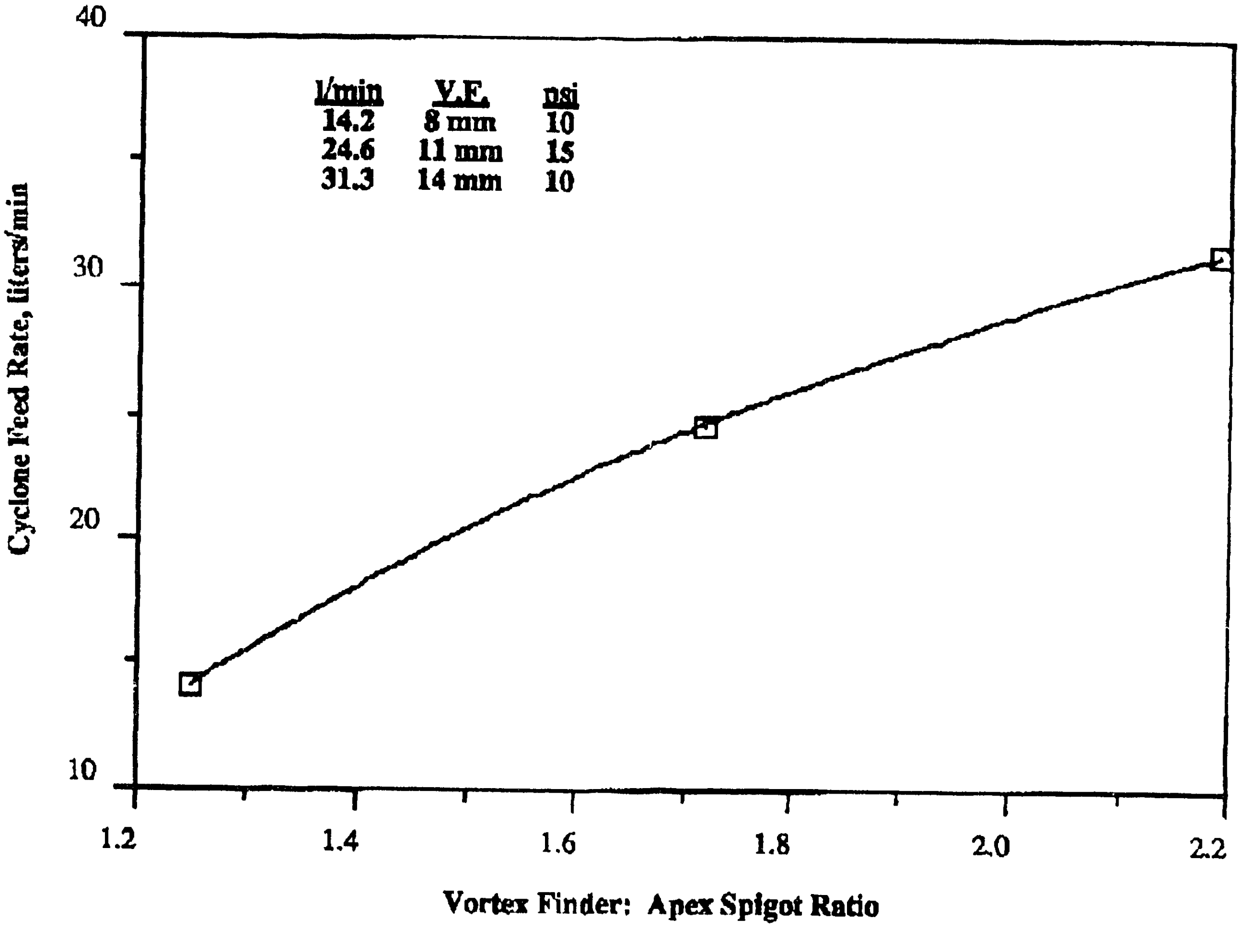

Figure 4-1. EFFECT OF VORTEX FINDER AND APEX SPIGOT RATIO ON CYCLONE FEED RATE

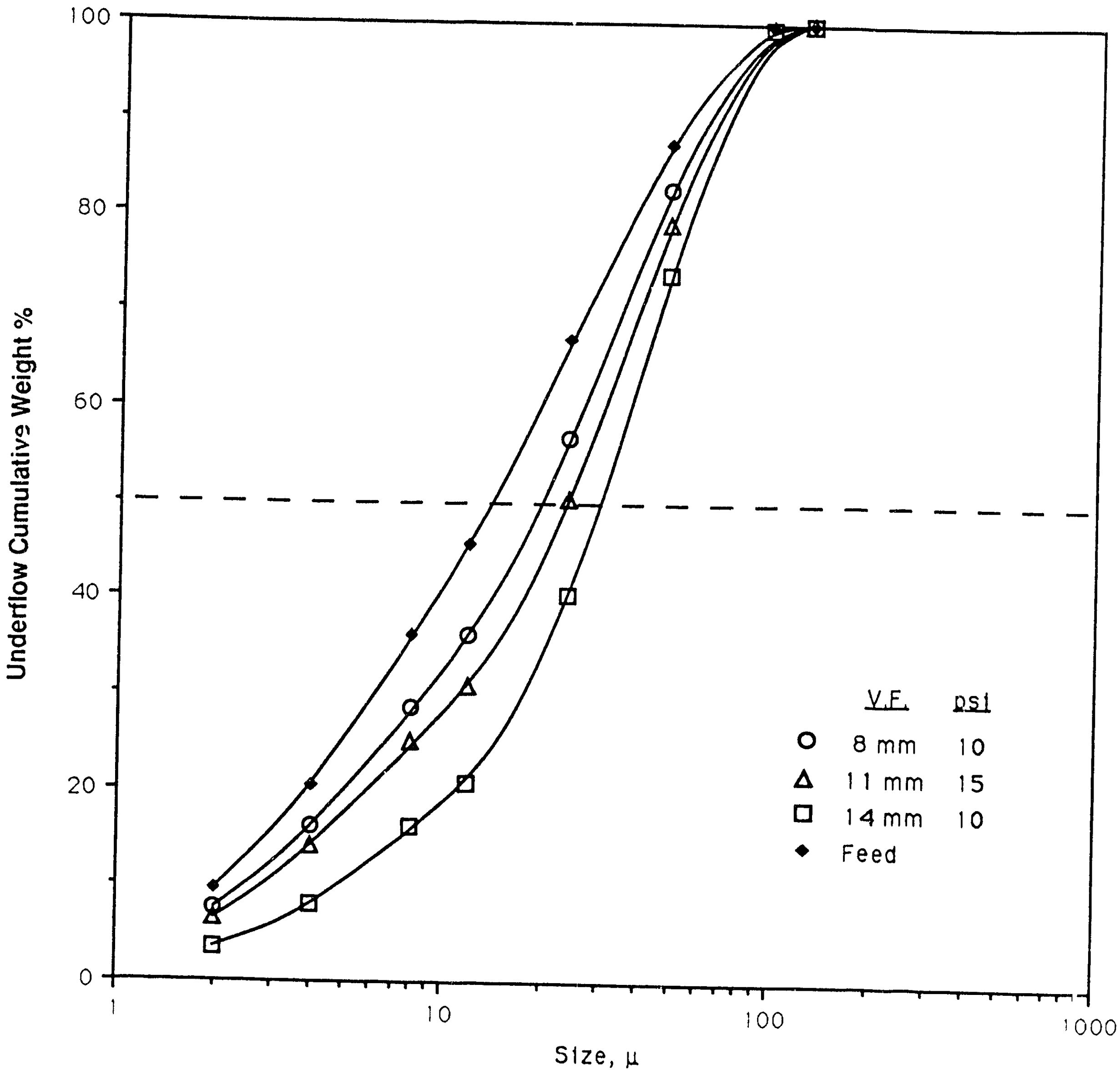

Figure 4-2. SELECTED CYCLONE OPERATING CURVES AT CYCLONE OVERFLOW d_{90} OF 23 μm

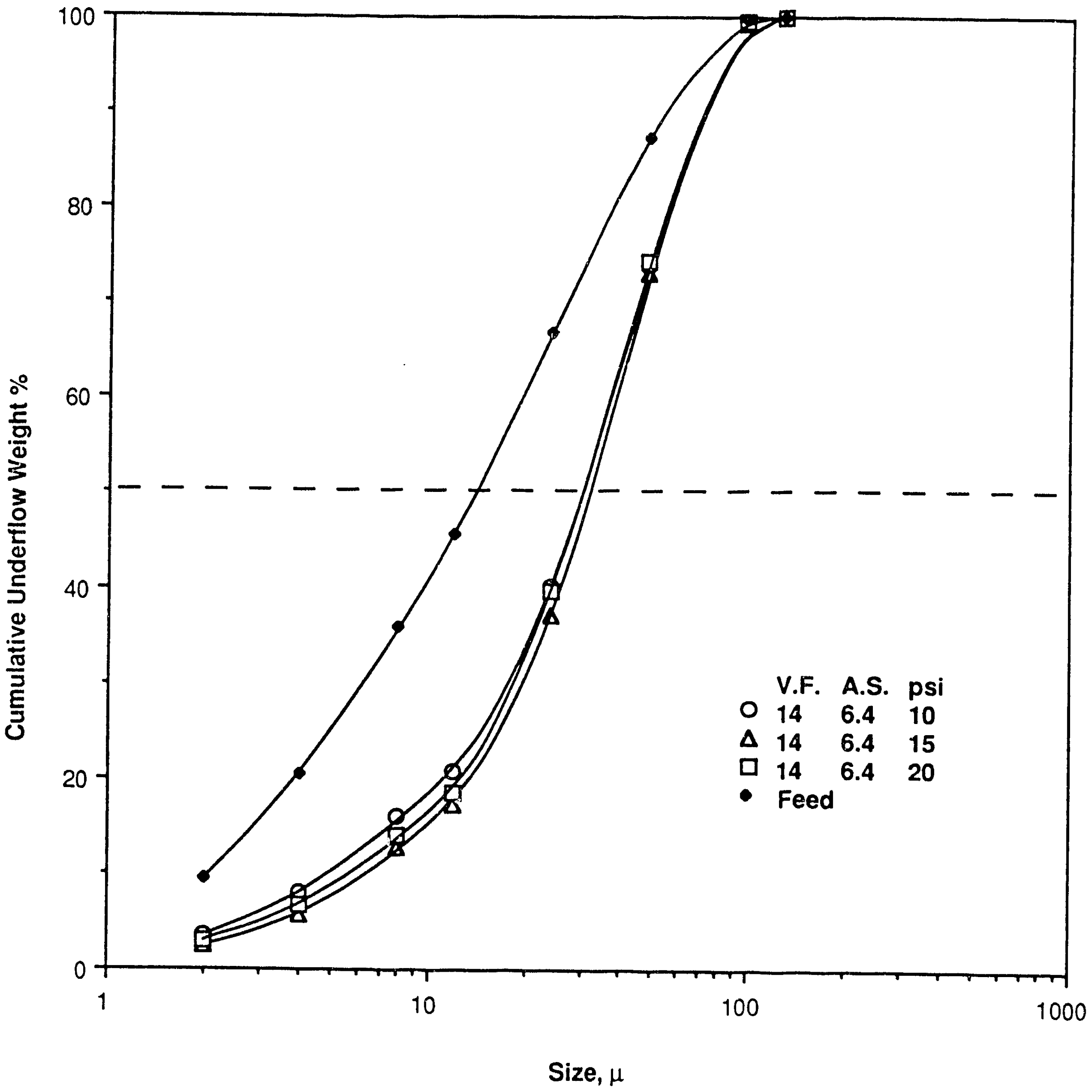

Figure 4-3. EFFECT OF INCREASING THE FEED PRESSURE ON THE CUMULATIVE UNDERFLOW WITH A CONSTANT SIZE VORTEX FINDER AND APEX SPIGOT CAP

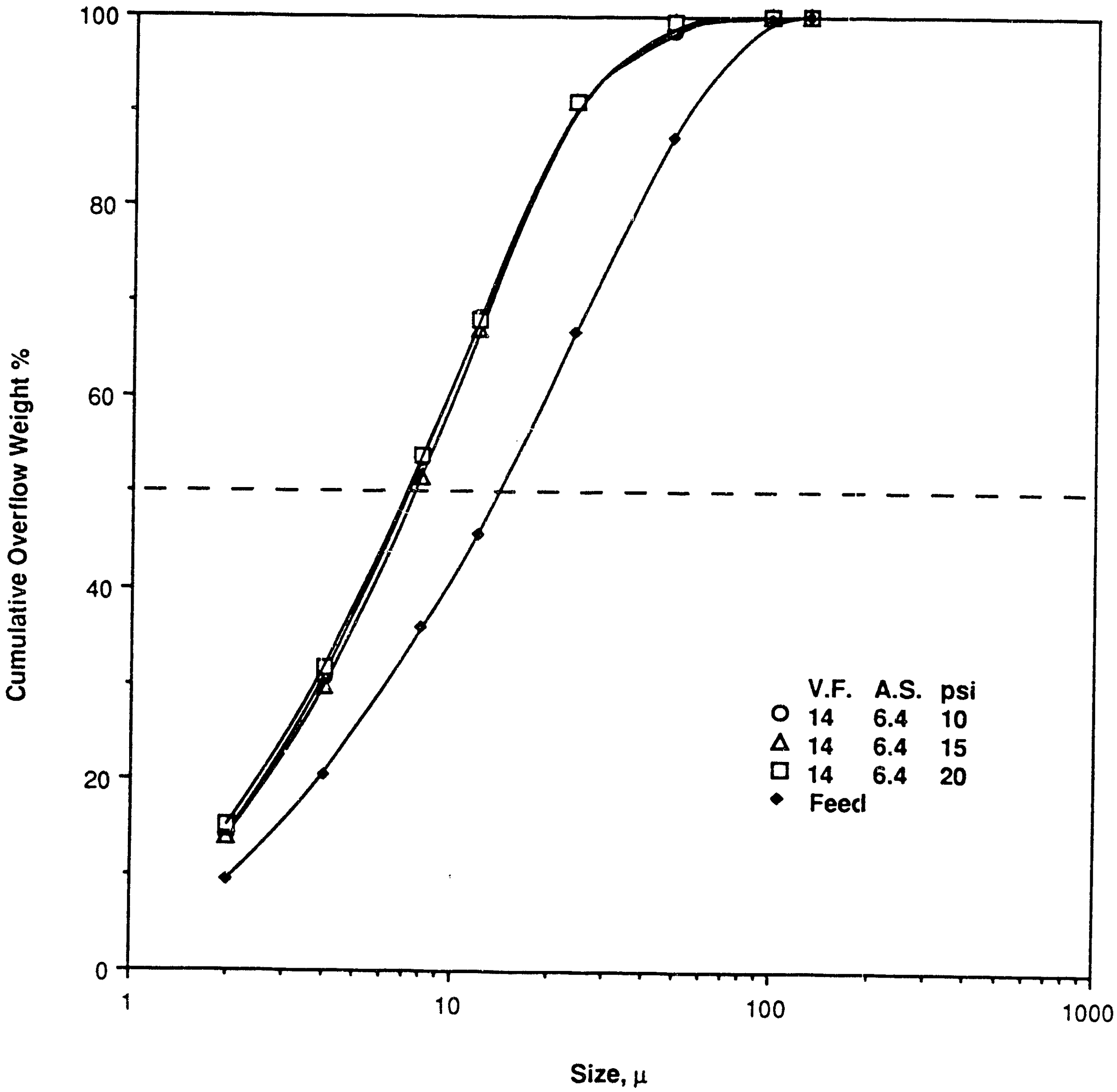

Figure 4-4. EFFECT OF INCREASING THE FEED PRESSURE ON THE CUMULATIVE OVERFLOW WITH A CONSTANT SIZE VORTEX FINDER AND APEX SPIGOT CAP

increasing the recovery by adding an additional cyclone in series, to treat the primary cyclone underflow. Results of this test showed that the primary cyclone underflow solids content was too high to yield an effective separation, hence, future testing will be conducted to determine the optimum dilution of the primary cyclone underflow prior to recycloning to increase the recovery of the -24 μm fraction.

Subtask 4.2.1. Column Flotation Tests

This subtask is divided into two subtasks: 4.2.1.1. Column Flotation Modeling, and 4.2.1.2. Circuit Evaluation - Indiana Shale. The primary objective of Subtask 4.2.1.1 is to develop a steady-state phenomenological model of column froth flotation for ultrafine ground shale, which relates the behavior of various classes of particles to column operating parameters. Another objective is to develop an on-line procedure for measuring the oil content of a shale sample on the basis of x-ray back-scattering intensity.

Discussion

During the quarter, MRI concentrated on developing the model of column flotation for ultrafine ground shale. The transport phenomena in a flotation column is described by the axial dispersion and convection equation as follows:

$$\frac{\delta c}{\delta \theta} = Nd\,\frac{\delta^2 c}{\delta\,\lambda^2} - \frac{\delta c}{\delta \lambda}$$

where: C = concentration of solid particle component
Nd = dispersion number
θ = dimensionless time (time/average retention time)
λ = dimensionless distance (length/total column length)

The first-order kinetic equation for conversion to the flotation phase is:

$$\frac{d\ c}{d\ \Theta} = -.\ \ c$$

These are the principal component equations used in the modeling of column flotation. Based on this model, a computer simulation of column flotation was developed to predict the recoveries of different shale components in a flotation column under various operating conditions. With this simulation, the optimum conditions for deep froth column flotation and the optimum column design parameters can be determined. The simulation can also be used to study the hydrodynamic conditions in the flotation column.

The model requires three parameters: the mean residence time (t), the vessel dispersion number (Nd), and the first-order flotation kinetic coefficient (K). The first two parameters can be estimated with experimental data; the last one can be determined with additional mathematical models related to flotation hydrodynamics.

To estimate the first two parameters, a computer parameter estimation program was also developed. This program can optimize the two parameters simultaneously using the experimental data.

A series of experiments were conducted to evaluate the mixing behavior of liquid and solid particles in the deep froth column. Potassium chloride was the soluble tracer; fine silica sand was the solid particle tracer. In the tests, the liquid and solid particle residence time distributions (RTD) can be measured with the KCl or sand concentration distribution at the outlet of the column versus time. The experimental data are then used to estimate the parameters by a nonlinear least-square optimization subroutine. The mean residence time and the vessel dispersion number with KCl were initially determined to be 12.79 min and 0.14, respectively. The estimated mean residence time was in reasonable agreement with the measured mean residence time of 12.76 min. Furthermore, the chloride ion concentration data obtained from different test conditions show that the liquid mixing behavior follows the same rules under different conditions (Figure 4-5). These curves can be successfully fitted with the model-generated curve (Figure 4-6). Therefore, the axial dispersion model is applicable to the deep froth flotation column.

The particle dispersion number for fine particles and liquids should be the same. If this assumption can be verified, then liquid dispersion numbers can be used and the difficult task of measuring fine particle dispersion numbers can be eliminated. RTD measurements with fine silica sand tracer were used to test the assumption. In the experiment, 5 grams of silica sand tracer were injected into the column, the tracer concentration at the column outlet was determined by a sampling and weighing procedure. The experimental results for Nd show similar trends. However, the data is not sufficient to draw a conclusion. This is probably because the amount of the solid tracer used in the experiment was not large enough to show the optimum features of the solid particle RTDs. Further tests are in progress.

In addition, the flotation kinetic coefficient, K, can be determined with additional equations as well as experimental measurements of the induction time of a species, which is related to the hydrophobicity and then floatability of that species. The induction time of oil shale samples (feed, concentrate and tailings) have been obtained and the results indicate that there is a great difference between the hydrophobicities of oil shale concentrate and tailings, e.g. the induction time for oil shale concentrate and tailings are 10 and 23 ms, respectively.

After all the parameters have been determined, the computer simulation based on this model will be used to optimize the experimental conditions, flotation column design parameters, and flotation column control strategies. The initial simulation coded in FORTRAN will be refined in the next step. Experimental verification of the model is also needed.

One objective of this subtask was to identify and develop an on-line procedure for measuring the oil and ash content of oil shale samples through the measurement of x-ray backscatter intensity for quality control purposes.

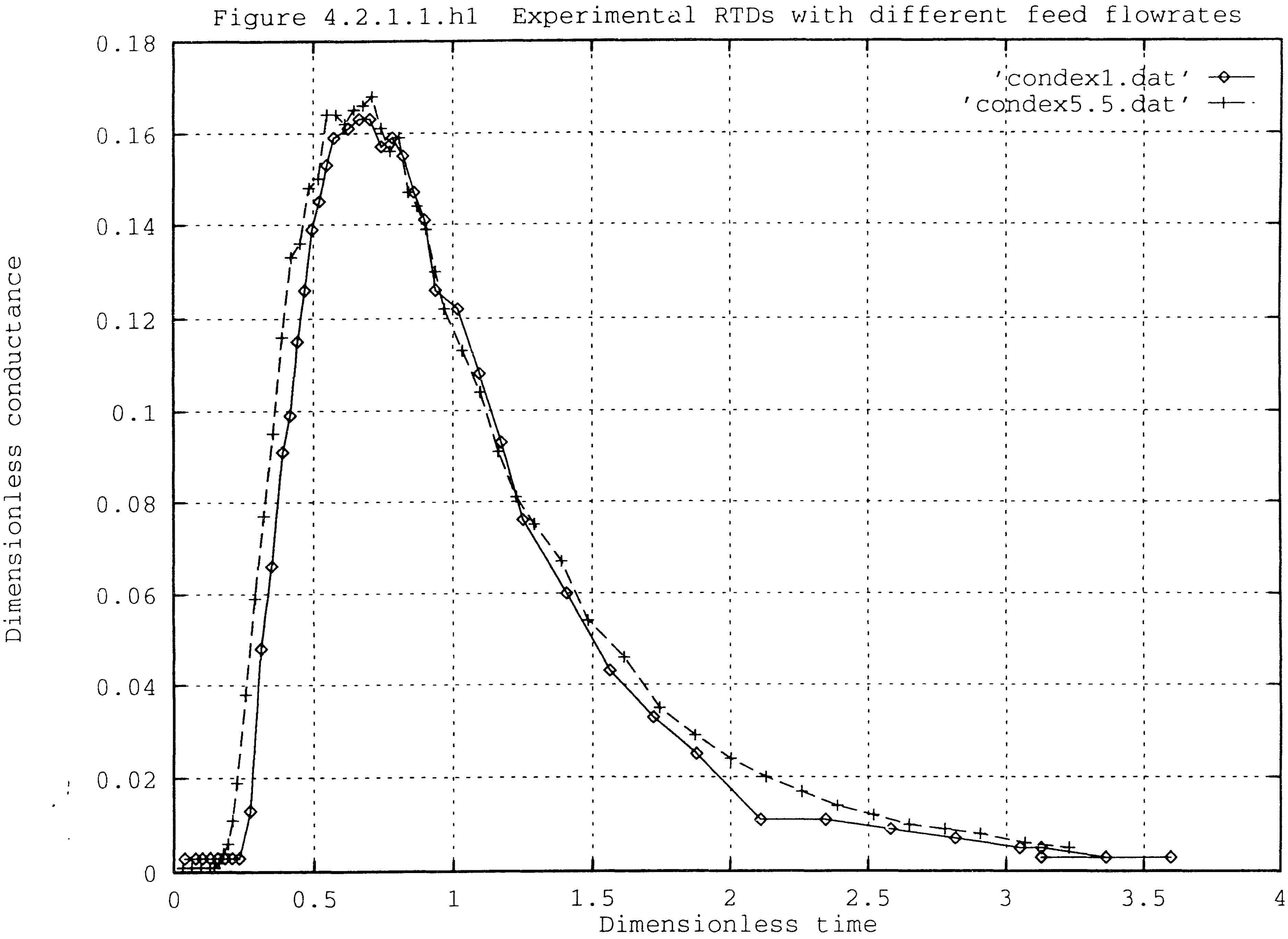

Figure 4-5. EXPERIMENTAL RTDs WITH DIFFERENT FEED FLOWRATES

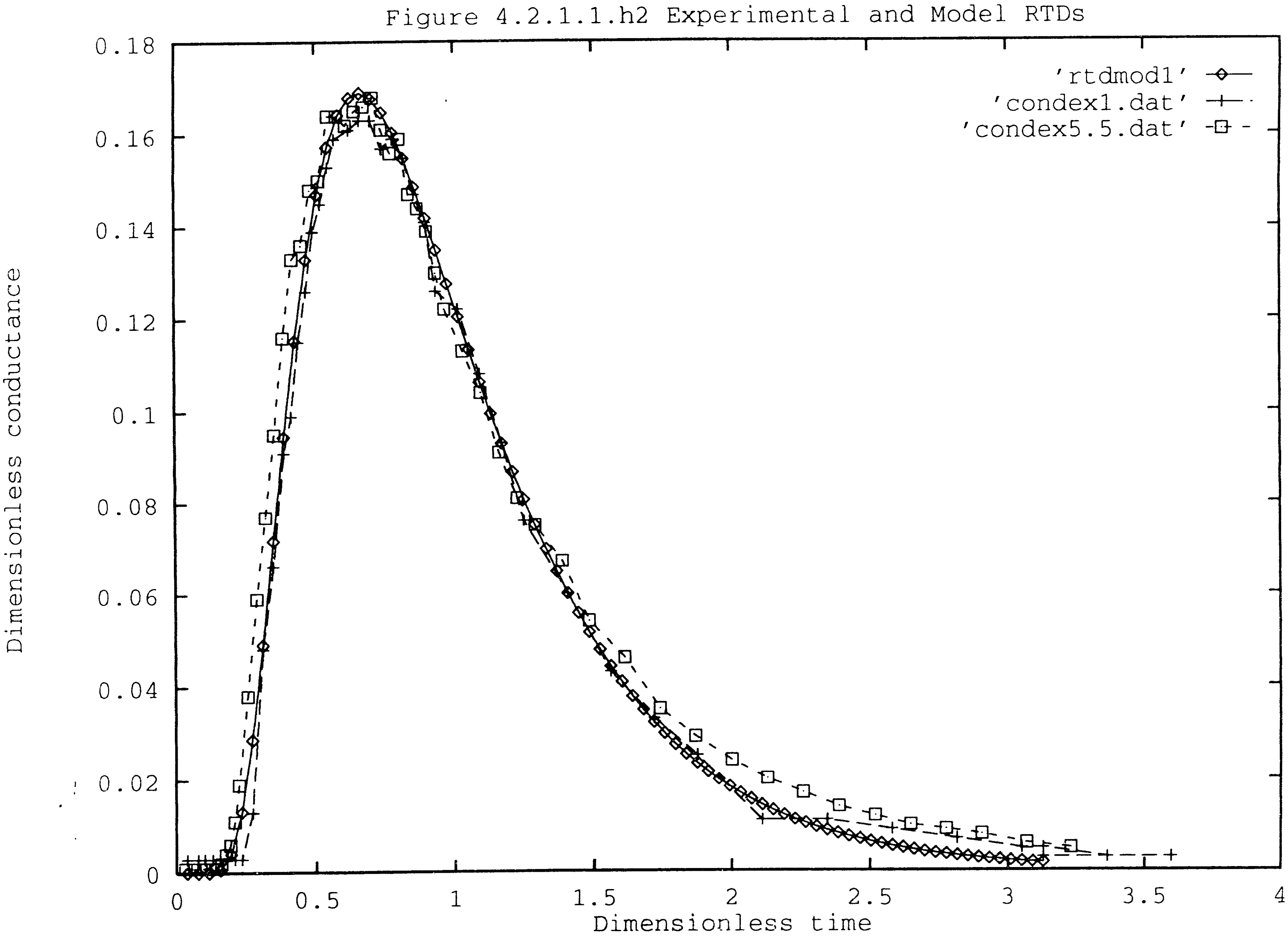

Figure 4-6. COMPARISON OF EXPERIMENTAL AND MODEL RTDs

A correlation was developed to evaluate the scattering intensity of oil shale and predict the oil yield. The kerogen in shale was configured into carbon, hydrogen and nitrogen. The x-ray backscatter intensity values were calculated for different wavelengths and correlations were obtained between scattering intensity, elemental composition, and oil yield. The scattering intensity equation developed is

$$n = k\ (\delta C^{*}\ rC + \delta H^{*}\ rH + \delta N^{*}\ rN)/(\mu C^{*}\ rC + \mu H^{*}\ rH + \mu N^{*}\ rN)$$

* Where δC, δH, and δN refer to scattering coefficients for carbon, hydrogen, and nitrogen; μC, μH, μN refer to mass absorption coefficients; and rC, rH, rN refer to weight concentrations for C, H, and N. The wavelengths used were 0.154, 0.5609, 0.7107, 1.5418, 1.6591, 1.9373, and 2.2909 Å.

Initial testing was done on samples from Michigan, Indiana and Tennessee. The data exhibited an exponential relationship of order 2. Both second and third order fits were evaluated, but there was no significant difference between the r^2 values for the third and second order except for Tennessee. The present analysis was done keeping the scattering intensity equation as a function of carbon, hydrogen and nitrogen.

The work was extended to Alabama shale so that a uniform sample size involving heads, concentrates, and tails could be included. The r^2 values were around 0.94. The variation explained by the model was quite satisfactory.

The next step was to characterize the scattering intensity equation including sulfur. The correlations obtained with Indiana shale had r^2 values of 0.996 when fitted with a second-order model.

When samples from Tennessee and Michigan were tested in the model equation involving sulfur, r^2 values for Michigan shale ranged from 0.75 to 0.83 for different wavelengths. The correlations improved slightly with a second-order fit and with the removal of heads taking into consideration only the concentrates and tails in the sample size. This moderate correlation is attributed to the lack of one-to-one relation between carbon content and Fischer Assay finding at this point of research. The geochemistry and maturity of the shales must be taken into account.

The model worked well with Tennessee shale and the r^2 value was 0.99 with a second-order fit. It would be of interest to record relative intensities with moisture content and increased temperature.

MRI is attempting to make this an economical and nondestructive laboratory procedure. Based on a literature review, it was decided that an X-ray spectrometer would be a suitable means for evaluating relative intensities. Pellet samples will be prepared from shales of different oil contents. Chromium has been chosen as the target material since it is expected to yield optimum results. The samples will be mounted in LiF crystal.

Since variations in compositions of organic matter, water, volatile species, and temperature are factors that could affect intensity, a thorough qualitative analysis is planned. A procedure for determining scattering intensity of wet samples with hopes of reducing sample preparation time will be investigated. The overall goal is to develop correlations to predict oil yield from the backscatter intensity of wet oil shale.

The objectives of Subtask 4.2.1.2 are to test the applicability of grinding circuits developed for Alabama shale to Indiana shale and to modify the circuits for the specific grinding and kerogen liberation characteristics of Indiana shale.

Arrangements were completed for shipping a one-ton sample of Indiana shale from the Laramie, WY Project Office to MRI for circuit evaluation studies. Treatment of the Indiana shale will follow that of the Alabama shale under Subtask 4.1.6.

Subtask 4.4. Integrated Grinding and Flotation

The objective of this subtask is to continue the development of the integrated grinding and flotation SYMUSEP process and to investigate methods for dewatering the resultant kerogen/water slurry. Subtask 4.4 is divided into three subtasks: 4.4.1. Acquisition and Characterization of Samples, 4.4.2. SYMUSEP Separator Testing, 4.4.3. Pilot Plant Studies, and 4.4.4. Dewatering Studies.

Discussion

During the quarter, UK-CAER conducted column flotation and dewatering studies with Alabama Chattanooga shale. For the column flotation work, the shale was ground using the Drais-Werke Mill, which is a continuous grinding attritor mill. About 40 pounds of shale were ground for each test. The particle size distribution of a representative sample of the ground material was determined using a Granulometer, which utilizes the laser beam scattering principle.

Conventional batch flotation tests were conducted using a Denver (Model D-12) flotation machine. The pilot-scale Ken-Flote column consists of a 6-inch ID (inside diameter) glass tube 20-feet in height. A slurry containing 5-percent solids was prepared in an 80-gallon tank. Flotation reagent (Frother M250) was added to the slurry and the conditioned slurry was fed at the 14-foot elevation into the column. Bubbles were generated using a sintered metal sparger. The froth-pulp interface was controlled with a differential pressure cell. A gentle spray of wash water was introduced in the froth layer to remove entrained and entrapped impurities. The operating conditions for the column flotation tests are listed in Table 4-4. Samples of the froth and tailings were collected after 30 minutes of starting the test. The froth generated in each test was stored in a plastic 55-gallon drum for use in dewatering studies.

Dewatering studies were conducted using standard laboratory vacuum and pressure filtration equipment (Figure 4-7). For the dewatering study, a fixed amount of slurry was used and the amount of filtrate collected was recorded with time. At the end of dewatering tests, the moisture content of the filter

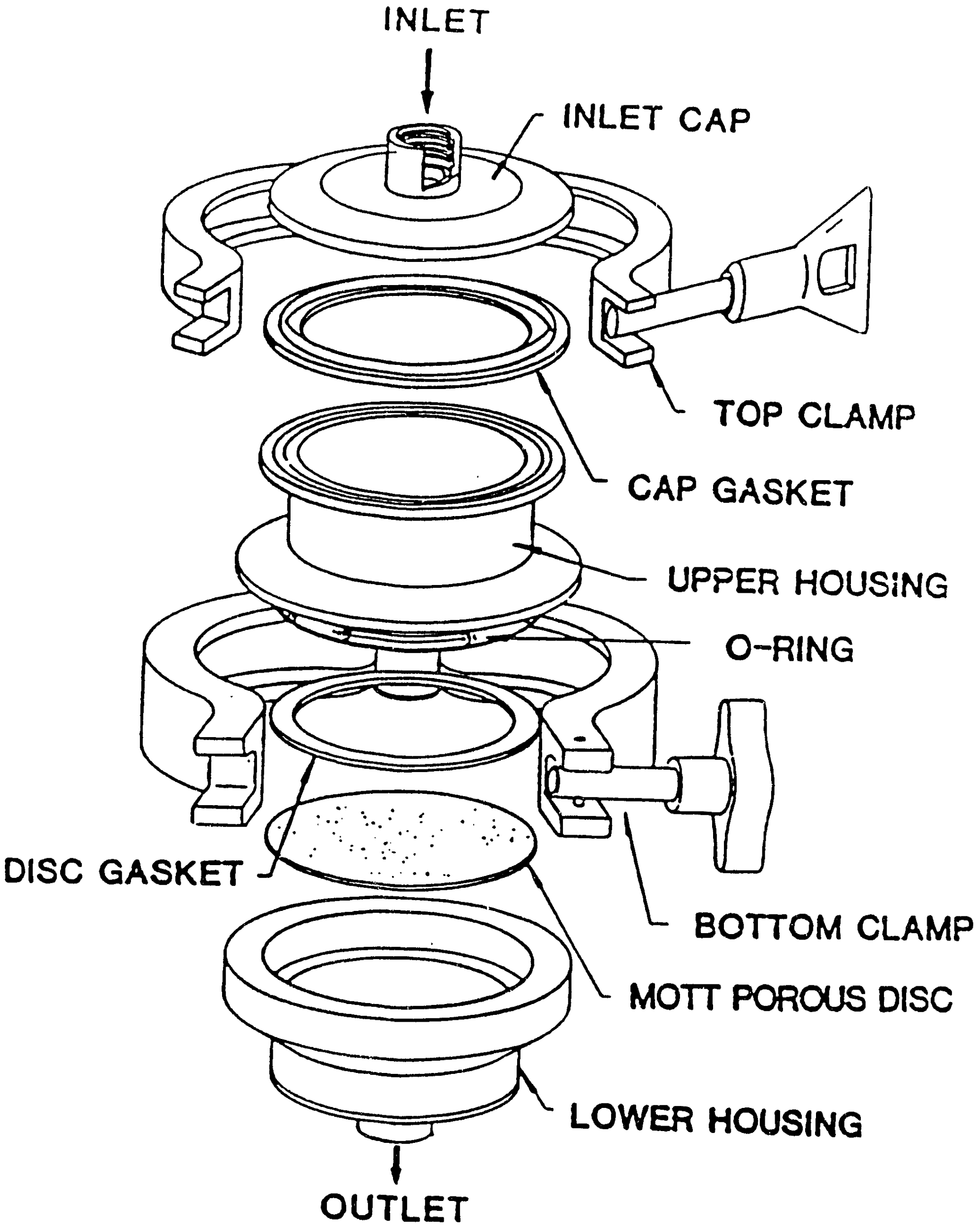

Figure 4-7. BENCH-SCALE PRESSURE DEWATERING APPARATUS

cake formed was determined. Surface charge (electrophoretic mobility) of kerogen particles were determined using the Zeta Meter (Model 3.0).

About 1 ton of Alabama shale received from MRI was crushed to -4 mesh and stored in 55-gallon drums. The chemical analysis and Fischer Assay of a representative sample of the shale are presented in Table 4-5.

Table 4-4. COLUMN FLOTATION TEST CONDITIONS

Column Dimensions	
Column Diameter, in.	6
Total Height, ft	20
Recovery Zone Height, ft	16
Froth Depth, ft	4
Froth Wash Zone, ft	2-1/2
Froth Drain Zone, ft	1-1/2
Feed Level, ft	14
Slurry Feed Rate (5% Solids), gal/min	0.5-1.6
Frother, lb/ton	3.5
Wash Water Rate, gal/min	0.26-0.9
Air Flow Rate, SCFM	0.3-0.5

Table 4-5. CHEMICAL ANALYSIS OF OIL SHALE

Component	Weight %
Moisture	1.20
Ash	73.20
Organic Carbon	16.39
Volatile Matter	15.80
Sulfur	
Pyritic	6.35
Sulfate	0.31
Organic	1.73
Total	8.39
Fischer Assay, GPT	11.3

A baseline flotation study was conducted on shale ground to 90 percent passing 10-μm size. In this series of tests, the froth product was cleaned in six stages using a Denver flotation machine. The purpose of this test was to determine a guideline for grade and recovery of kerogen that could be obtained using conventional flotation. The grade and recovery curve of the kerogen obtained with the conventional flotation technique are shown in Figure 4-8. The results show that recovery and grade have a linear relationship. With conventional flotation, a concentrate containing 30 percent organic carbon is obtained at a recovery of about 60 percent.

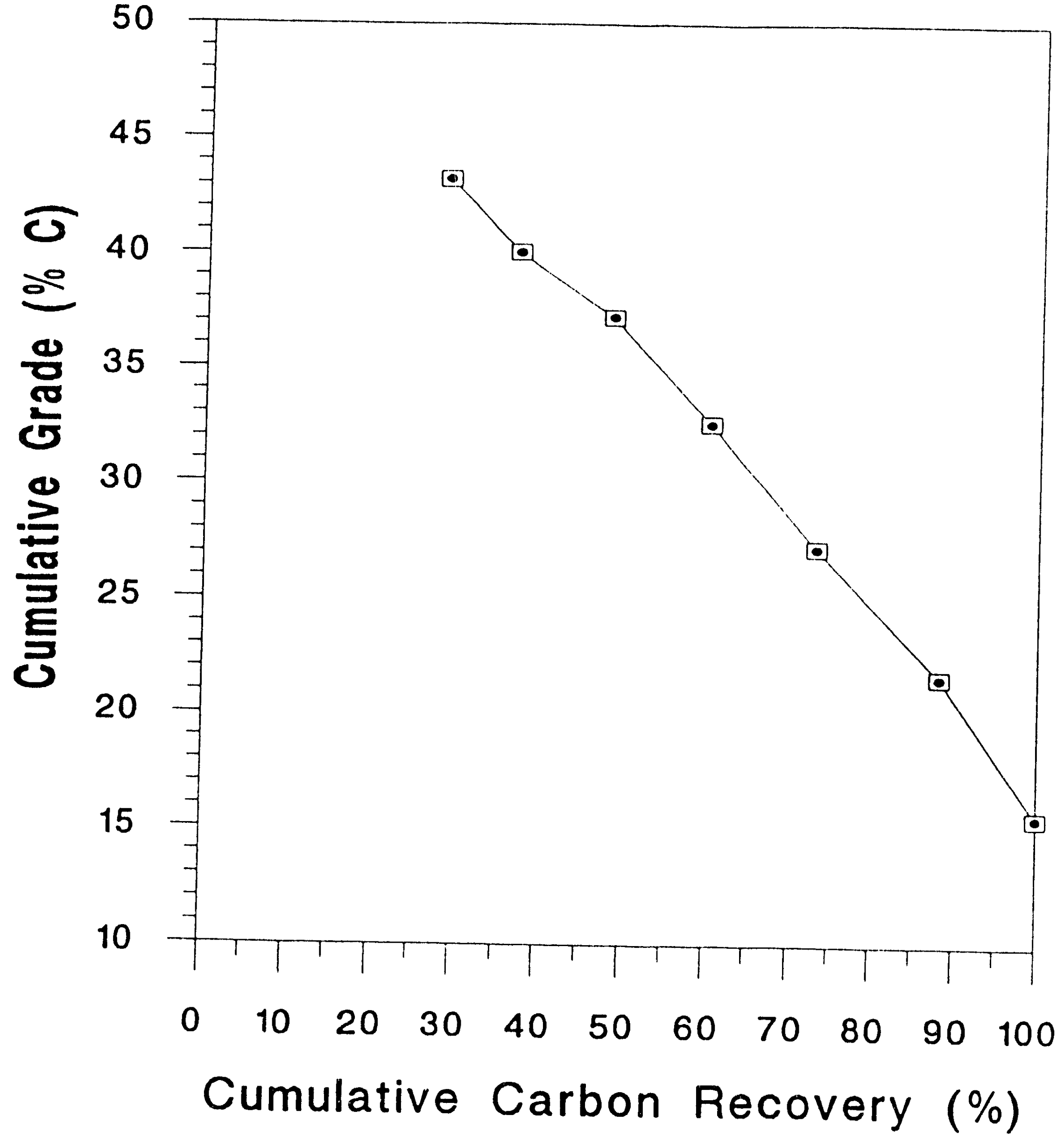

Figure 4-8. GRADE AND RECOVERY OF KEROGEN USING CONVENTIONAL FLOTATION

Column Flotation Studies

Initial pilot scale column flotation studies were conducted to study the effect of feed rate and retention time of slurry on grade and recovery of kerogen. Figure 4-9 shows that feed rate has very little impact on carbon recovery, however, a high (≈33 percent) carbon grade product was obtained at 1 gal/min feed rate.

Figure 4-10 shows the effect of slurry retention time in the column using two different air flow rates. For an air flow rate of 0.3 SCFM, recovery does not show any significant improvement over 50 percent even using more than 20 minutes of retention time. This result was unexpected. In previous tests conducted in the 2-inch column, about 80 percent recovery of about 40 percent carbon grade kerogen was obtained at 20 minutes retention time. Increasing air flow to 0.5 scfm, at lower retention time of 10 to 15 minutes even provided lower recovery of kerogen. At present, UK-CAER does not have any explanation for this behavior, however, they are conducting additional tests to obtain more information.

Dewatering Studies

Dewatering studies were conducted on the froth product collected from the pilot scale column. The composite froth sample contained 30 percent carbon and 10 percent solids by weight. Particle size distribution data shows an average particle size of 7.7 μm.

The electrophoretic mobility data of the froth is shown in Figure 4-11. It shows a zero point-of-charge at pH 3.5; this means that the particles are positively charged below pH 3.5 and are negatively charged above pH 3.5. This information is necessary in modifying the surface of kerogen for dewatering studies to be conducted using metal ions and surfactants.

Figure 4-12 shows filtration data of the froth using vacuum and pressure filtration equipment. With a vacuum of 22.5 inches of Hg, after 2 minutes of filtration time a filter cake containing 45 percent moisture was obtained. Increasing filtration time did not improve moisture reduction. Pressure filtration conducted using 40 psig pressure also gave 45 percent moisture filter cake at 2 minutes filtration time, however, after 5 minutes moisture was reduced to about 40 percent. This lowering could be due to air drying, which happens in pressure filtration after cracks develop in the cake. At 60 psig, a 35 percent moisture filter cake was obtained at 2 minutes filtration time and at 5 minutes a 20 percent moisture was obtained in the filter cake, the low moisture content again may be due to air drying.

Based on the above results, it can be concluded that:

- Flotation characteristics of the oil shale are similar to those of tests conducted previously, however, the pilot-scale column provided only 50 percent recovery of a 33-percent carbon containing kerogen using up to 20 minutes of retention time. These results do not confirm the 2-inch ID column results obtained last year.

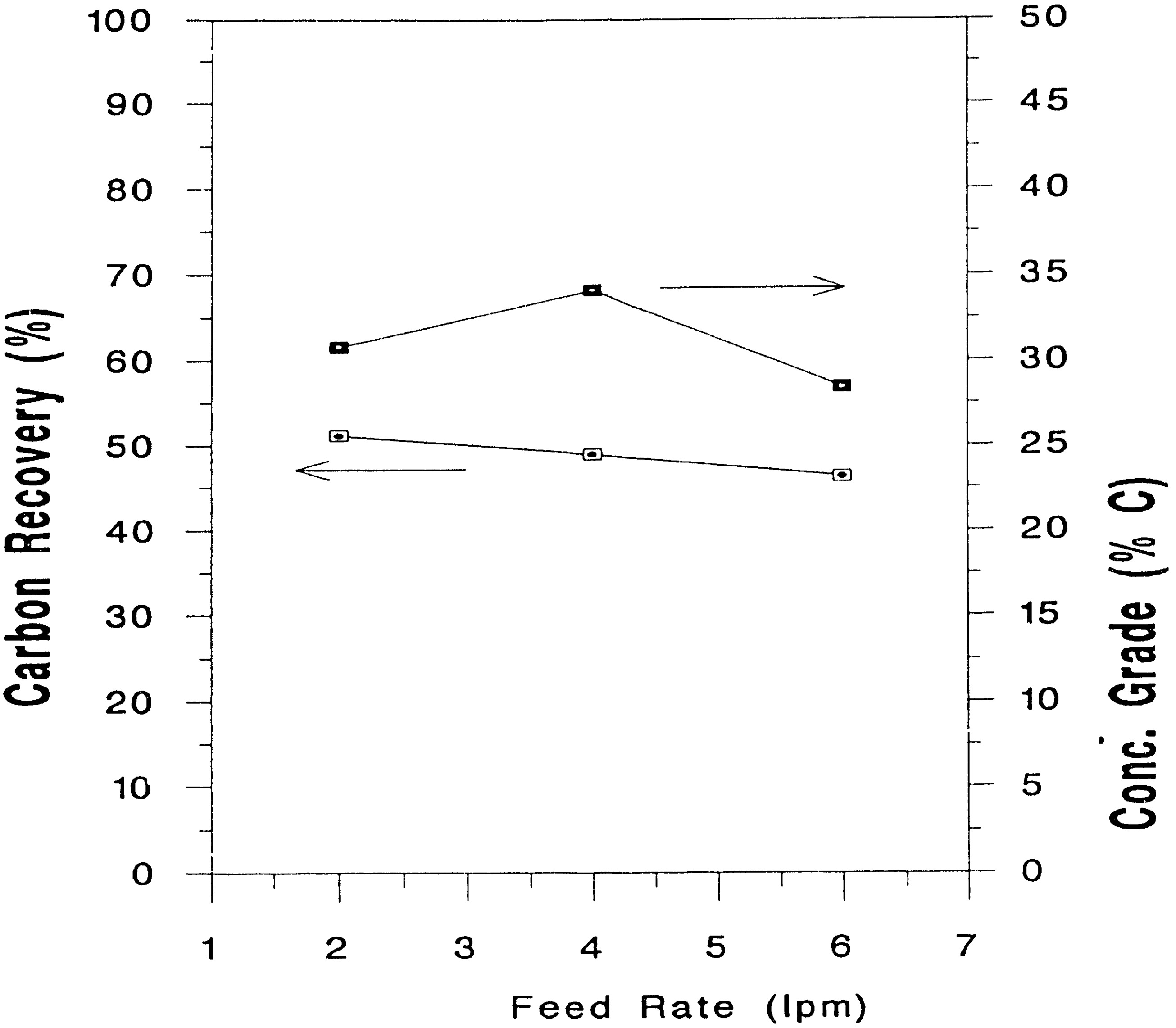

Figure 4-9. EFFECT OF FEED RATE ON GRADE AND RECOVERY OF KEROGEN

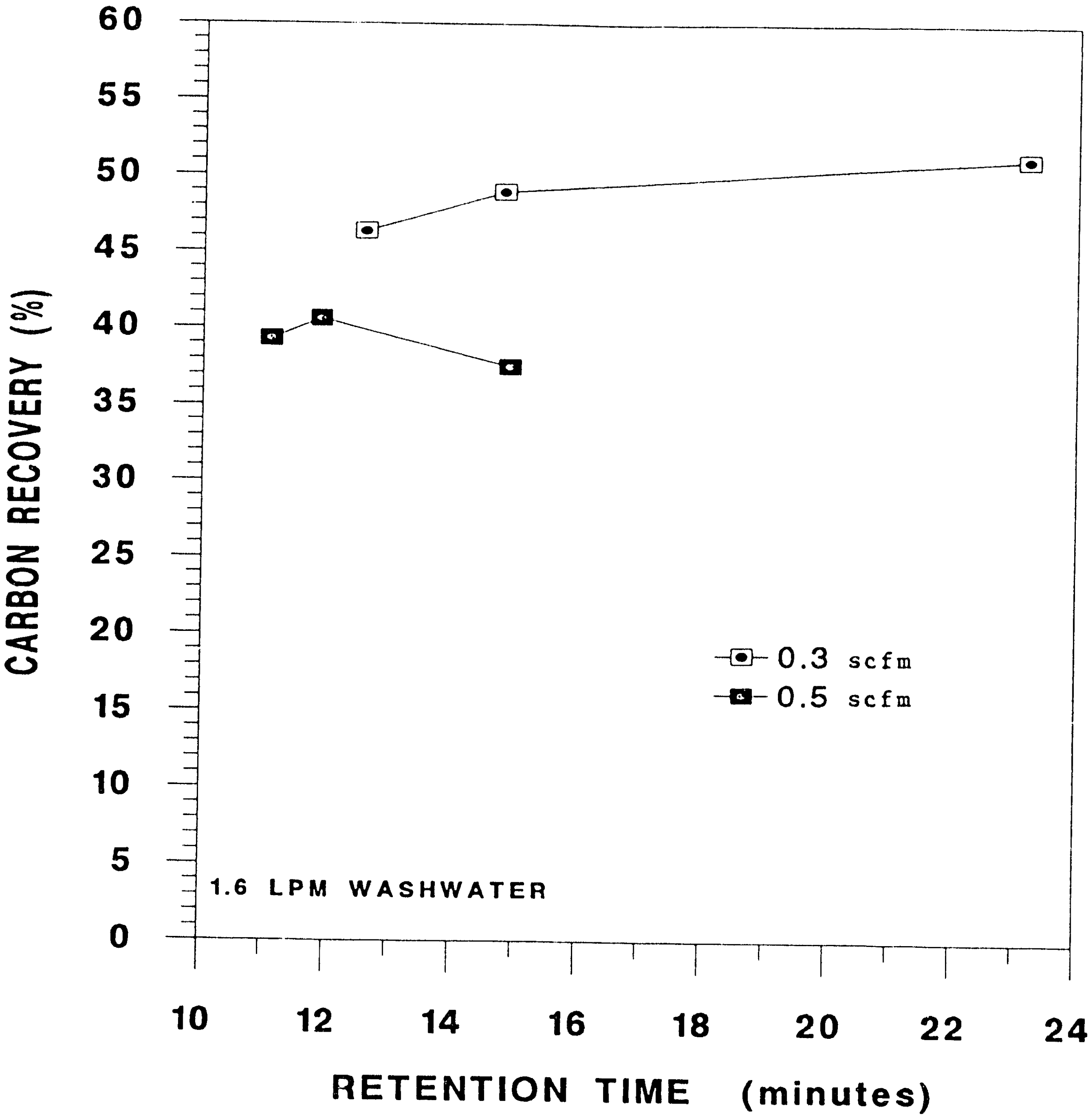

Figure 4-10. EFFECT OF RETENTION TIME ON GRADE AND RECOVERY OF KEROGEN

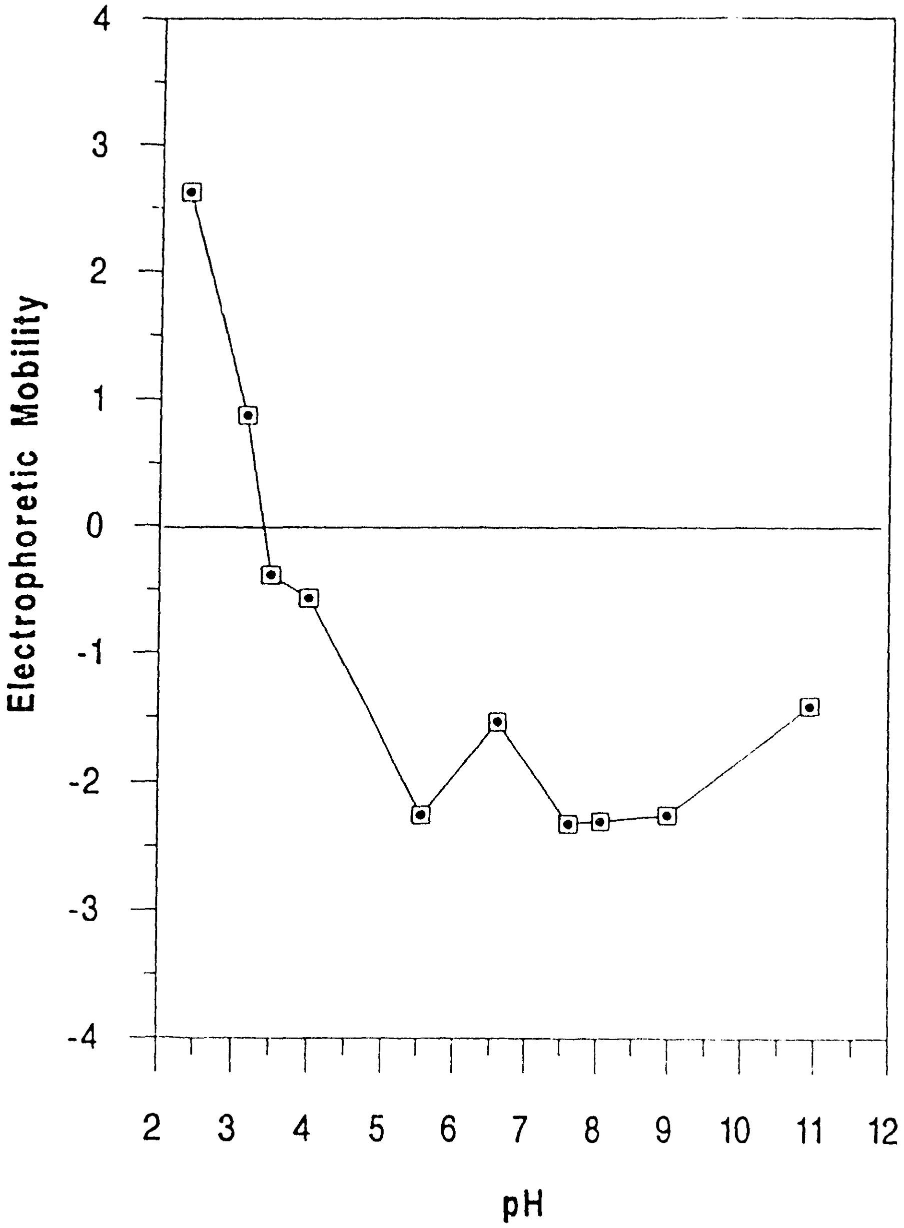

Figure 4-11. EFFECT OF pH ON ELECTROPHORETIC MOBILITY OF KEROGEN CONCENTRATE

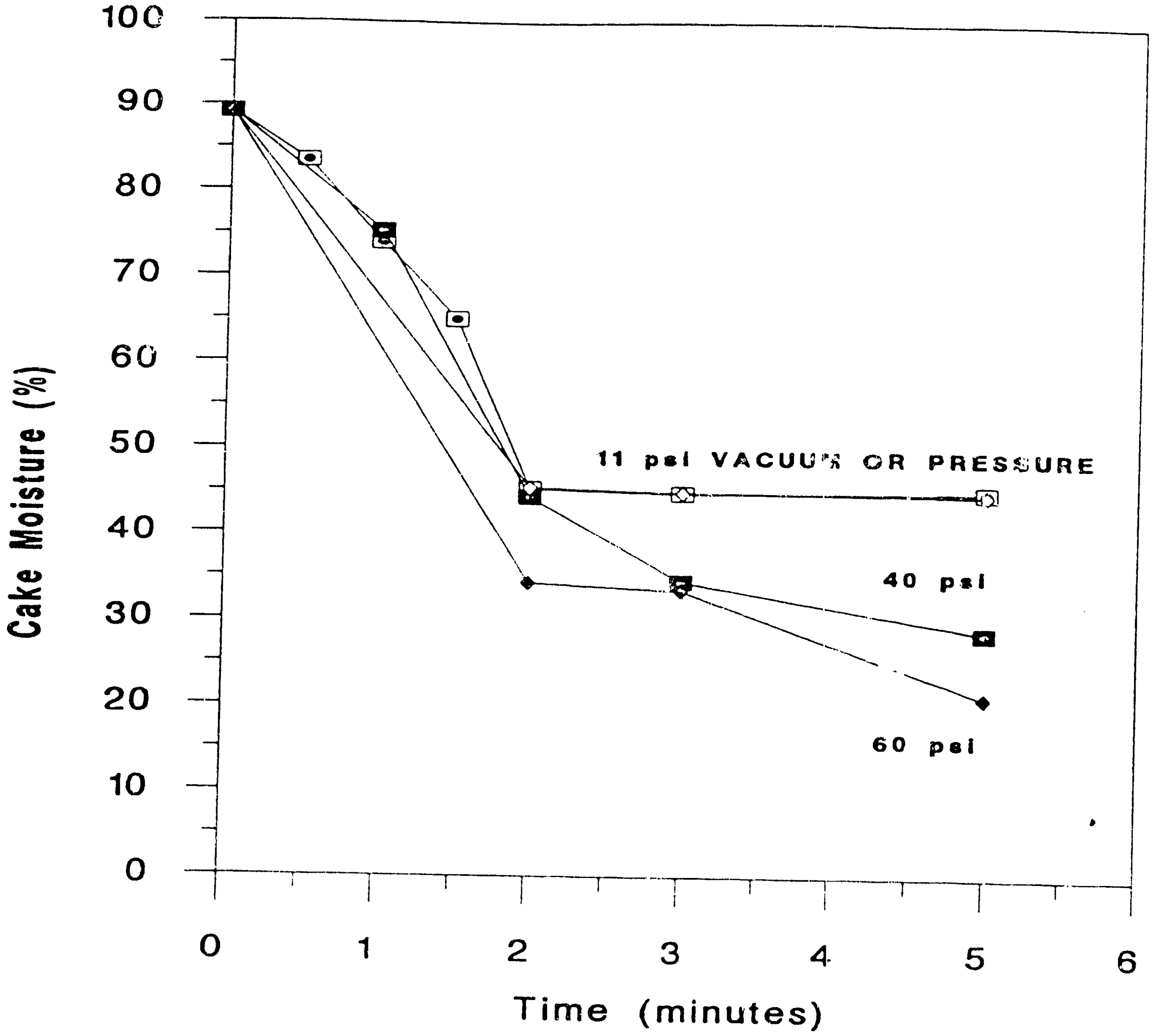

Figure 4-12. DEWATERING OF KEROGEN CONCENTRATE USING VACUUM AND PRESSURE FILTRATION

- The cumulative froth product obtained from the column containing about 30 percent carbon and of 7.7 μm size could be dewatered to 45 percent moisture level using vacuum filtration and pressure filtration (40 psig) at 2 minutes of filtration time. A lower moisture of 35 percent was obtained using 60 psig pressure.

Next quarter, UK-CAER plans to conduct additional pilot-scale column tests to improve carbon recovery. Tests will also be conducted using the modified SYMUSEP separator column. Dewatering studies will be conducted using the novel surface modification approach developed at the UK-CAER.

Subtask 4.7. Economic Analysis

The objective of this subtask is to update the estimate of the cost of beneficiating Alabama shale to reflect the effects of current technological development.

Discussion

The most recent (June 1988) cost estimate for beneficiating Alabama shale was made by the Roberts and Schaefer Company as part of a prior DOE contract with the University of Alabama. A new cost analysis is being performed that will update the former cost estimate to reflect the effects of inflation and other changes in the economic climate.

The cost indices published monthly in Chemical Engineering magazine were selected as the basis for updating the capital cost estimate. Several indices were used so as to provide a more accurate update rather than a simple single index inflation factor. Indices used in the cost update are presented in Table 4-6.

Table 4-6. CHEMICAL ENGINEERING PLANT COST INDICES

Index	June 1988	June 1992	Ratio
Process Machinery	342.0	377.6	1.1008
Construction Labor	262.8	271.3	1.032
Structural Supports	373.0	329.2	0.8826
Engineering & Supervision	343.8	351.8	1.024

A detailed breakdown of the estimated 1988 costs by area, material class and labor is presented in Appendix A-1. The estimated costs are summarized in Table A-2. The individual costs in Table A-2 were multiplied by the appropriate index ratio from Table 4-6. All labor classes were adjusted with the general factor for construction labor (1.032). The process machinery index ratio (1.1008) was used to update the equipment costs. The structural materials index ratio (0.8826) was used to adjust all other materials except electrical materials. A review of copper prices indicates that the current price of copper (100.1 ¢/lb) is slightly below the 1988 prices. Because electrical materials include other materials than copper wire, it was decided to use a zero inflation factor for electrical materials.

The original cost estimate used a factored cost estimating procedure to establish such costs as engineering labor, construction management, etc. Therefore, those factors were retained in the update rather than using index ratios such as the Engineering and Supervision index value.

The preliminary updated capital cost estimate (see Table A-3 in the appendix) represents an increase of 4.9 percent from June 1988 to June 1992 dollars, or $240,205,000 to $251,960,000.

Task 6. Environmental Data and Mitigation Analyses

The overall objective of this task is to obtain environmental data relating to PFH and to shale beneficiation and to analyze the potential environmental impact of the integrated PFH process. This task consists of four subtasks: 6.2.2. Wastewater Treatability, 6.2.3. Waste Management Facility Conceptual Design, 6.4.1 PFH Process Analyses, and 6.4.4. Economics. Subtasks 6.2.2 and 6.2.3 are being conducted by the Tennessee Technological University (TTU).

Subtask 6.2. Water Availability and Treatment Studies

Subtask 6.2.2. Wastewater Treatability

The objective of this subtask is to determine suitable techniques for treating wastewater generated during shale processing.

Discussion

During the quarter, TTU prepared samples of liquid wastes for biological treatability studies. The samples of liquid waste were generated during bench-scale PFH tests with beneficiated shale. Before attempting biological acclimation of the liquid wastes, it was necessary to reduce the hydrogen sulfide (H_2S) and ammonia (NH_3) concentrations to below toxic levels. This was achieved by raising the temperature of the waste to about 80°C (just below boiling), adjusting the pH, and bubbling air through it. To get the nitrogen into strippable gaseous form (NH_3), the pH was raised to about 10.5. The free ammonia was then stripped by contacting the waste with a large volume of air. To prevent the loss of volume due to evaporation, the air bubbling through the waste was passed through a condenser and the condensate returned to the flask holding the liquid waste.

The concentration of NH_3 in the liquid waste was monitored using a Fisher specific-ion probe. When the concentration fell to below the toxic level of 500 mg/L, the pH was reduced slowly to 6.5. At this low pH, the sulfides present tend to be converted into gaseous H_2S, which can likewise be removed via air stripping. If the pH was reduced too rapidly, solids formed in the liquid phase. This was attributed to the formation of H_2S at a rate faster than it could be stripped out, possibly forcing the formation of insoluble metal sulfides and/or elemental sulfur. To avoid this, the pH was reduced slowly allowing the H_2S to be liberated at a slower rate. To determine if the sulfides were reduced to acceptable levels, a qualitative evaluation was made using lead acetate strips. When the strips showed no H_2S in the off gas, the procedure was stopped.

To determine the quantity of volatile organics in the waste water, a 300-mL sample (Alabama shale) was brought to a boil and the condensate was collected. Figure 6-1 shows the apparatus used to trap the volatile components. The pH of the sample was adjusted to 9.5. The waste began to boil at 88°C. The temperature in the liquid was stabilized at about 90°C, which resulted in a temperature of about 80°C at the base of the condenser. After 1 hour and twenty minutes, about 50 mL of distillate were collected. Since this was a sufficient quantity for analysis, the procedure was terminated. The results of this analysis will be reported next quarter. The characteristics of the raw PFH liquid waste are presented in Table 6-1.

Table 6-1. CHARACTERISTICS OF RAW PFH LIQUID WASTES

Characteristic	Raw Alabama	Raw Indiana
pH	8.7	9.3
	------------ mg/L	------------
COD	70,400	67,000
NH_3-N		
Free	15,200	22,800
Bound	3,770	7,840
Total	19,000	30,700

The buffering capacity of each waste was determined by preparing pH titration curves using 6N NaOH and 6N HCl. As shown in Figure 6-2, both samples are strongly buffered in the pH range of 7 to 11. The Indiana shale liquid waste has the greater buffering capacity of the two. The quantity of NaOH (100 percent) needed to increase the pH of the Alabama shale sample to 10.5 was 33.4 g/L; the quantity was 39.1 g/L for the Indiana shale sample.

Prior to attempting acclimation of microorganisms to the stripped PFH liquid waste, certain chemical characteristics (Table 6-2) were needed to evaluate the compatibility of the waste to microorganisms.

The liquid waste from the Alabama shale was selected as the initial feedstock for the acclimation experiments. As indicated in Table 6-2, the COD of the stripped Alabama waste was 31,300 mg/L. This was used as a general determination of the availability of biodegradable organics. The high concentration of sodium was a result of the use of NaOH to adjust the pH during the stripping process.

A titration curve was developed with the stripped Alabama waste and, as indicated in Figure 6-3, the stripped waste had a very low buffering capacity.

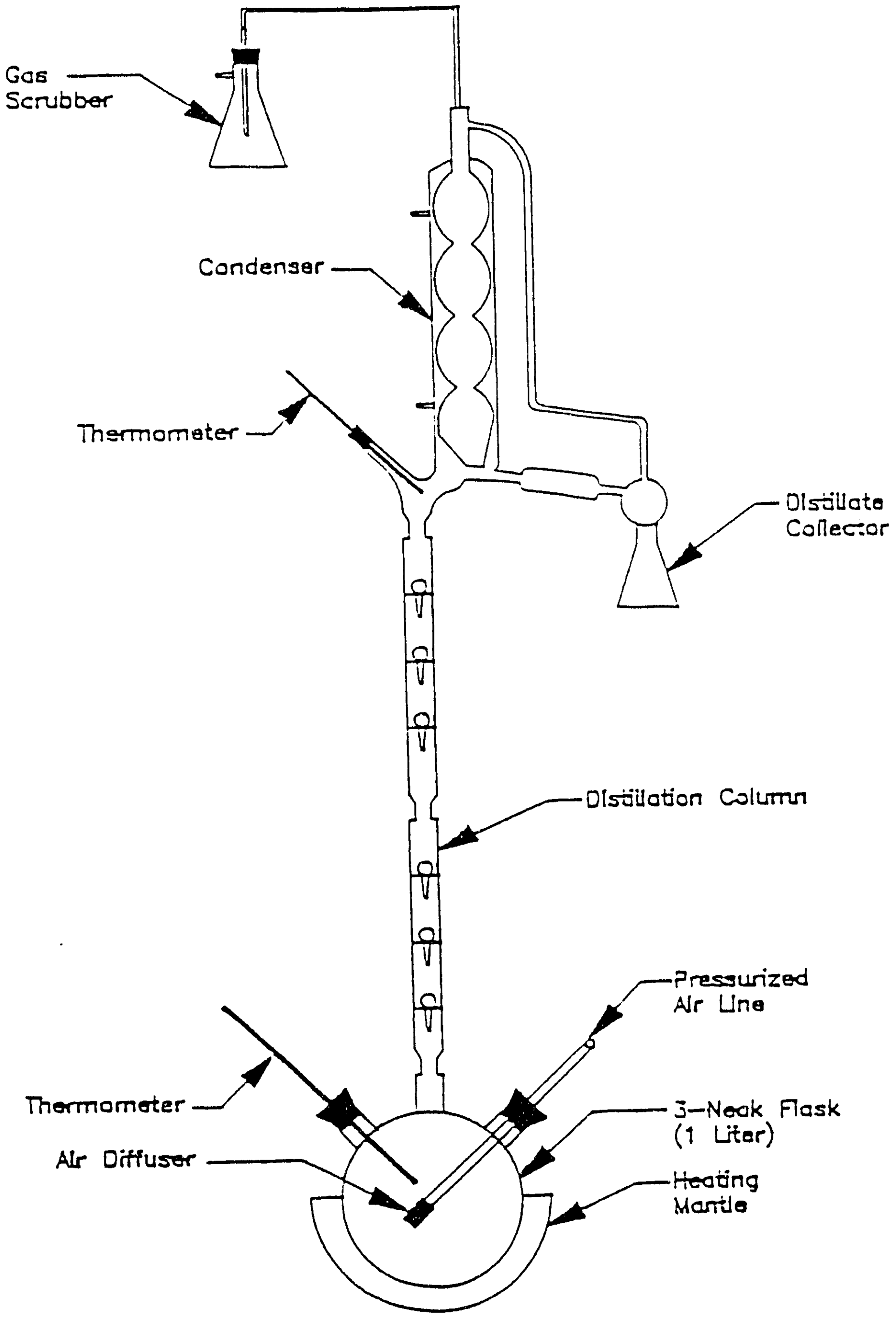

Figure 6-1. SCHEMATIC DIAGRAM OF THE APPARATUS USED FOR VOLATILES COLLECTION AND AMMONIA AND SULFIDE REMOVAL

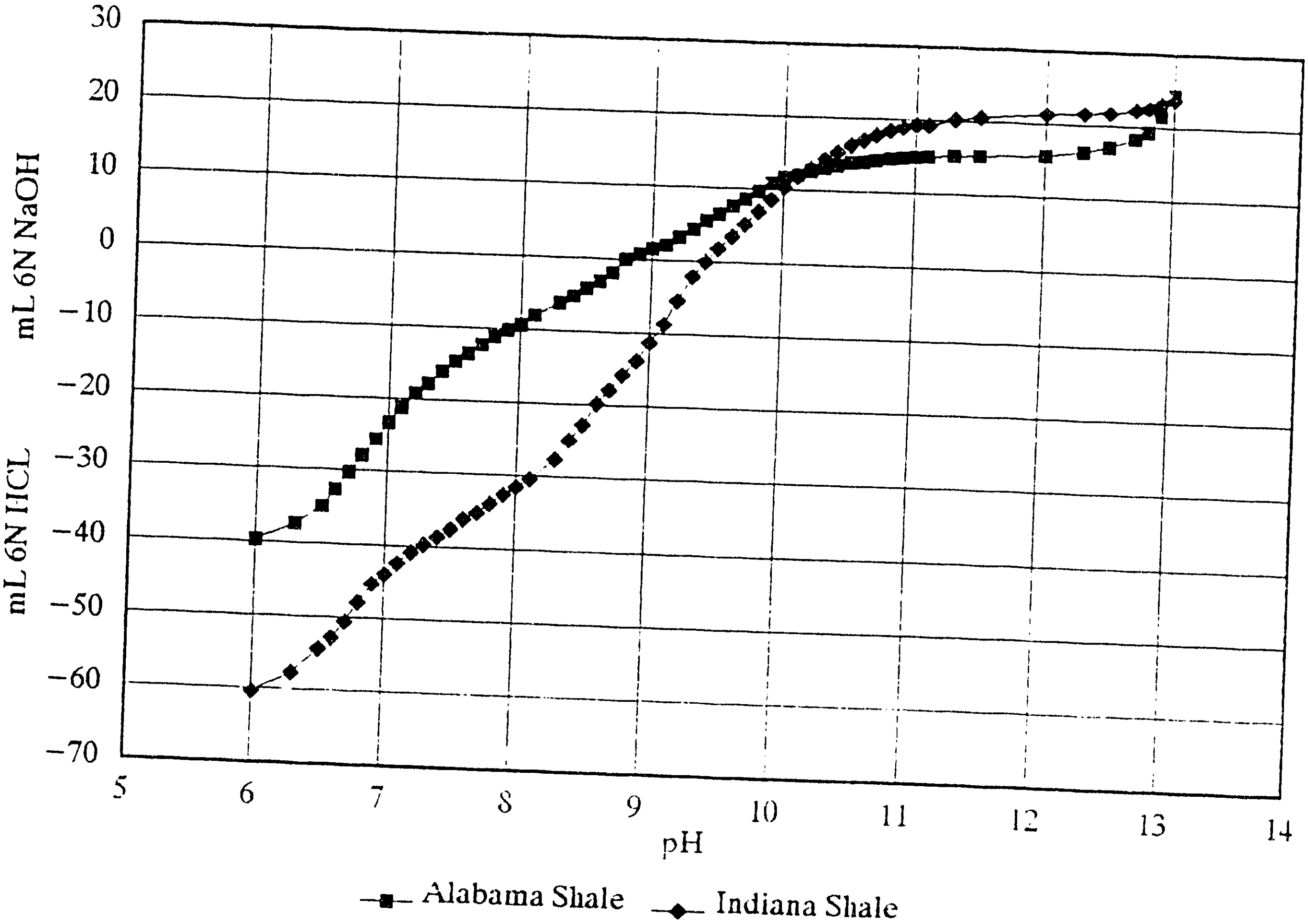

Figure 6-2. COMPARISON OF THE TITRATION CURVES FOR WASTEWATERS FROM PFH PROCESSING OF ALABAMA AND INDIANA SHALE

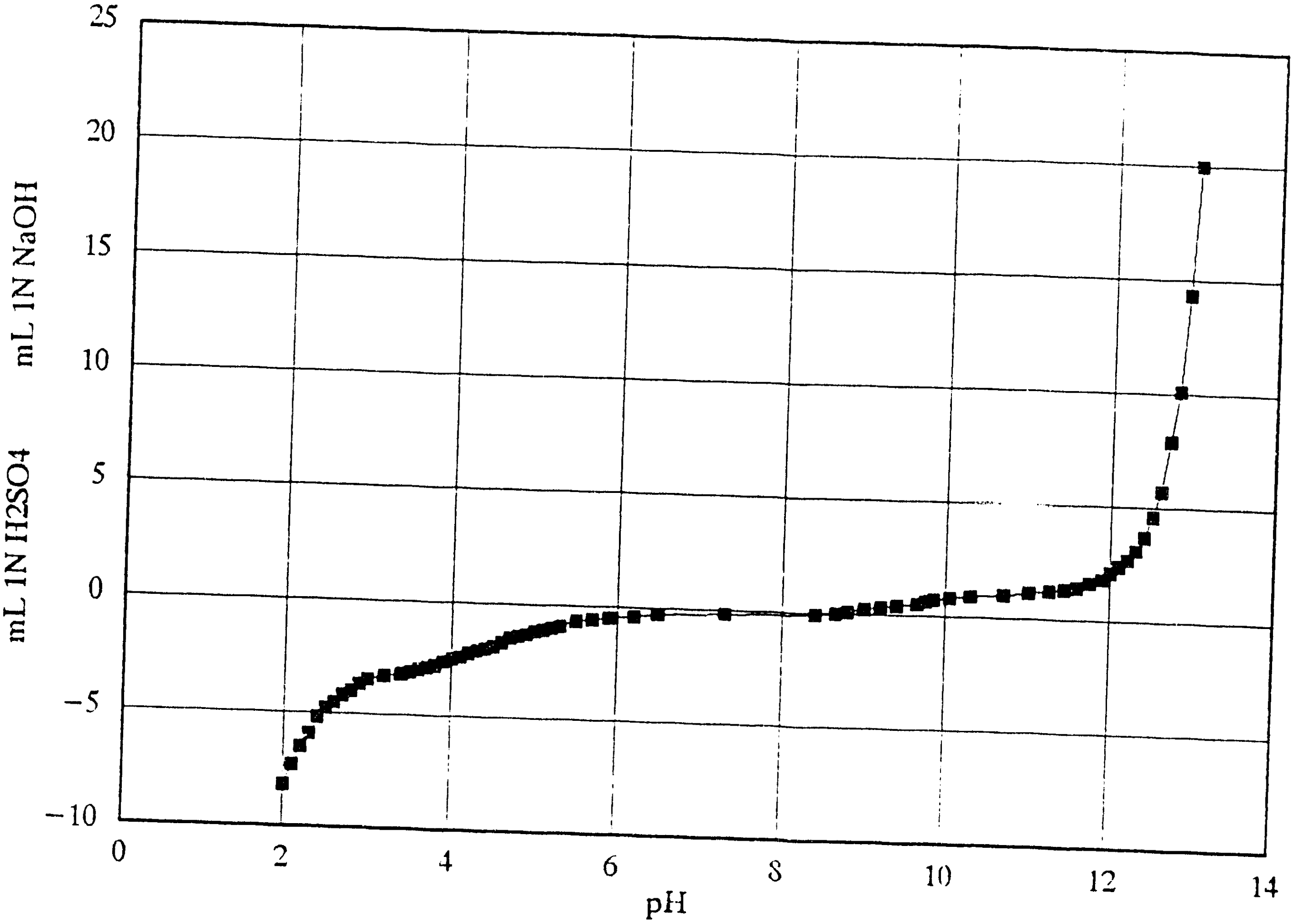

Figure 6-3. TITRATION CURVE FOR STRIPPED ALABAMA SHALE WASTEWATER

Table 6-2. STRIPPED PFH WASTEWATER CHARACTERISTICS

Component	Alabama (mg/L)	Indiana (mg/L)
COD	31,300	32,200
Ca	0.16	0.28
Fe	0.45	0.70
Mn	0.93	0.33
Mg	0.090	0.080
Na	19,200	34,000
SCN^-	1130	1320
Buffer Capacity	Low	Low
NH_3-N (Total)	550	1060
NH_3-N (Bound)	410	950
NH_3-N (Free)	130	110
pH	8.3	7.1

Determination of Nutrient Additions

Because the stripped waste had a total nitrogen concentration of 550 mg/L, it was decided to attempt acclimation without adding supplemental nitrogen. A phosphate buffer was used both as a phosphorous source and to buffer the waste at an approximately neutral pH. The needed concentrations of other required elements were also added. All of the additions made to the waste prior to attempting acclimation are tabulated in Table 6-3.

Table 6-3. ADDITIVES TO PFH WASTE

Nutrients and Buffer	Desired Concentration, mg/L
$CaCl_2$	7.5
$FeCl_3 \bullet 6H_2O$	0.5
$MnSO_4 \bullet H_2O$	10.0
$MgSO_4$	50.0
KH_2PO_4	530
K_2HPO_4	1070
Nitrogen	None Added

A number of dilutions were prepared for the initial acclimation attempt to the PFH Alabama liquid waste. These dilutions are presented in Table 6-4 with the calculated COD and the thiocyanate concentrations.

The dilutions, including the nutrient and buffer additions, were placed in 250-mL Erlenmeyer shaker flasks. A seed was taken from reactors with microorganisms that had been previously acclimated to synthetic waste containing some of the major organic constituents in the PFH waste.

Microbial growth was established in the dilutions containing up to 50 percent by volume of the PFH waste. Growth was confirmed by preparing spread plates of the dilutions. A tabulation of the steps taken with the shaker flasks is given in Table 6-5. As indicated in the table, no microbial growth was evident in the 100 percent sample in three attempts.

Table 6-4. SHAKER DILUTIONS

% Solution	COD	SCN$^-$
	mg/L	mg/L
100	31,300	1,130
50	15,650	565
25	7,725	280
10	3,130	110
5	1,560	57
3.33	1,040	38
2.5	780	28
2	630	23
1.67	520	19
1.4	450	16
1.25	390	14
1.11	350	13
1	310	11

Table 6-5. SHAKER FLASK HISTORY

Date	Event
8-12-92	Started shakers of all dilutions except 25 and 50%
8-13-92	Growth in all except 100%
8-14-92	Started 25 and 50% shakers
8-15-92	Prepared plates on all dilutions
8-16-92	Plates positive except 100%
8-17-92	Prepared second series of dilutions at 50, 25, 10, 5, 2, and 1%. Reseeded 100% dilution out of 50%. Repeated plates on 100, 50, and 25% dilutions from first series.
8-18-92	Growth in second series. Repeat of plates 25 and 50% dilutions from first series positive. Negative on 100%.
8-20-92	Prepared third series of dilutions 50, 25, 10, 5, 2, 1. Plated 100% reseed of 8-17-92. Continued all three series.
8-21-92	Growth in third series. 100% plate negative.
8-24-92	Prepared fourth series of 25 & 50%. Continued 2nd & 3rd series of 50 & 25% and 1st of 100%.

The compounds being evaluated for treatability in combination are phenol, aniline, MEK, and 4-methyl pyridine. The concentrations chosen for the study are 250 mg/L for phenol, and MEK, and 300 mg/L for aniline and 4-methyl pyridine.

To evaluate the treatability of these compounds in combination, a method of determining the concentrations simultaneously was needed. TTU installed a new non-polar capillary column on the gas chromatograph. After a series of adjustments of the testing parameters were made, definable peaks were established for each compound in a combination solution. The parameters of the test are given in Table 6-6.

Table 6-6. GAS CHROMATOGRAPH OPERATING PARAMETERS

Carrier Gas	H_2 @ 8 mL/min
Detector Gases	H_2 @ 17 mL/min Air @ 400 mL/min
Injection Port Temperature, °C	240
Detection Port Temperature, °C	280
Initial Temperature, °C	35
Initial Time, min	1
Temperature Rate, °C/min	10
Final Temperature, °C	140
Final Time, min	1

The approximate elution times of the compounds are MEK, 1.48 minutes; 4-methyl pyridine, 6.8 minutes; aniline, 11.48 minutes; and phenol, 13.08 minutes. Standard curves were established for each compound in a combination solution.

Prior to proceeding with the treatability studies of the combined compounds, a microbial growth acclimated to the specific synthetic waste combination was needed. Microorganisms were acclimated to waste with the compounds in combinations of two compounds. Three 1.5-liter reactors were prepared with the essential nutrients and the combination of 250 mg/L of phenol and MEK, 250 mg/L of phenol and 300 mg/L of aniline, and 250 mg/L of MEK and 300 mg/L of aniline. A seed was taken from the reactors that had been previously acclimated to the individual compounds, and was added to each reactor that had the compound present in combination. Within 24 hours, a visible growth of microorganisms was present in each of the three combination reactors.

In continued efforts to acclimate microorganisms to 4-methyl pyridine, TTU collected a sample of sludge known to contain polyaromatic hydrocarbons from Chattanooga Creek in Chattanooga, TN. This creek has been polluted with both industrial and municipal wastes for many years. Seed taken from the sludge was placed in shaker flasks with 4-methyl pyridine (concentration of 250 mg/L) and essential nutrients and buffer required for biological growth. After two days in a shaking apparatus, a spread plate of the contents of the flasks indicated the absence of viable microorganisms. TTU will continue attempts to acclimate microorganisms to 4-methyl pyridine.

Subtask 6.2.3. Waste Management Facility Conceptual Design

The objective of this subtask is to design a conceptual wastewater and process water treatment facility for a commercial-scale PFH plant.

Discussion

During the quarter, TTU estimated water quantity requirements for a PFH plant liquid waste management facility. The requirements are based on estimated demands that the combined PFH plant and steam power plant will place on the treatment facility during daily plant operation. The three major water needs identified - boiler feed water, quench water, and cooling water - are described below.

The water quantity requirement for boiler feed water is estimated to be 2.02 MGD (million gallons per day) and the requirement for quench water within the hydrogen plant is estimated to be 0.53 MGD. These quantities are based on mass flow rate data for the "Combined PFH Plant and Steam Power Plant Original Design."[1]

The water quantity requirement for cooling purposes is based on the following assumptions. Primary cooling will be accomplished by a closed recirculating system. A closed recirculating system is one in which water is circulated through desired heat exchange locations in the PFH plant in a closed loop with negligible evaporation or exposure to the atmosphere. Closed systems generally require little or no makeup water except for leaks, expansion tank overflows, and surface evaporation from system vents.[2] Water losses for the closed cooling water loop are considered negligible in this study.

Heat will be removed from the closed system loop by a secondary cooling water cycle. The secondary cooling water cycle will be an open recirculating system utilizing one or more cooling towers. Makeup water will be provided for this system to balance the water lost by evaporation, blowdown, tower drift, and other miscellaneous losses.

Based on a cooling system as described above, the required daily makeup water is estimated to be 7.55 MGD.[3] This value depends to a large extent on plant location and cooling tower design. The value chosen is closer to the high end of quantity demands.

Additional water needs for the plant, such as dust control and shale moistening, will be addressed as they are quantified. It is assumed that potable and fire protection demands will be supplied by local municipal water services. Preliminary water quantity demands are summarized in Table 6-7.

Table 6-7. WATER QUANTITY REQUIREMENTS

	10^6 GPD
Boiler Feed Water	2.02
Quench Water	0.53
Cooling Water	7.55
Total	10.10

Water quantity requirements will be satisfied by liquid waste from the PFH facility and by import water. The quantity of water to be generated by the PFH facility is estimated to be 0.74 MGD, which is based on mass flow rate data for the "Combined PFH Plant and Steam Power Plant Original Design."[1]

The total import water requirement is estimated to be 9.36 MGD. This consists of water imported from sources such as free-flowing streams and impoundments. The import requirement is less than the lowest import quantity (11.3 MGD) used in the "Water Availability Study" conducted by TTU to evaluate water availability for potential Eastern Oil shale processing facilities in six eastern states.[4] Therefore, all sites recommended in the study will adequately satisfy the current import requirement.

Before the design of the liquid waste management facility can proceed, various water quality criteria must be obtained. The two categories of water quality are 1) initial quality of water requiring treatment, and 2) water quality criteria for recycle and discharge. Since two water sources have been identified, the initial water qualities are needed for the liquid waste generated from the PFH plant and for import water. Based on water quantity demands, water quality criteria for boiler feed water, quench water and cooling water are needed. Water quality standards for discharge are also required.

Boiler feed water should be of such quality as to avoid the three major water-related boiler system problems - deposits, corrosion, and carryover[2] - which are described below.

Deposits, particularly scale, are a serious problem at the high temperatures found in boilers. Scale is formed when contaminants in the water precipitate onto equipment surfaces. Precipitates form when water comes in contact with hot metal surfaces such as boiler heat-exchange tubes. Silica and aluminum have a strong tendency to form scale.[5] In addition to scale, damaging deposits may be in the form of sludge. These deposits are carried by water to low-velocity areas where they then drop out, compacting to form a dense agglomerate similar to scale. The most common components of boiler deposits are calcium phosphate, calcium carbonate (in low pressure boilers), magnesium hydroxide, magnesium silicate, various forms of iron oxide, silica absorbed on the previously mentioned precipitates, and alumina.[2] Deposits insulate boiler tubes, reducing the heat transfer rate. This leads to overheating and eventual boiler tube failure.

The second major water-related boiler problem is corrosion. The most common form of corrosion is the attack of steel by oxygen. This form of corrosion occurs in any portion of the steam cycle where oxygen is present. It is accelerated by high temperature and low pH. Corrosion may also occur due to alkali attack, improper application of feed water treatment chemicals, and other less common forms. The main step that can be made to prevent corrosion is the elimination of oxygen from boiler feed water.

The third major water-related boiler problem is carryover, defined as entrainment of boiler water into the steam system. A major cause of carryover is the volatilization of boiler water salts, such as silica and sodium compounds. Volatilized salts are carried as a mist into the steam system and may subsequently form turbine blade deposits or deposits in the superheater, causing tube failures. Foaming is an additional problem which results in carryover. Excessive concentrations of sodium or potassium can result in foaming.[5]

Quality criteria for boiler feed water becomes increasingly more stringent as boiler pressure increases. Therefore, a boiler pressure must be determined to insure adequate quality criteria. Based on the "Combined PFH Plant and Steam Power Plant Original Design", the maximum boiler pressure is estimated to be 2860 psi.[1] Based on this pressure, the boiler feed water quality criteria located during literature review is summarized in Table 6-8. TTU is confirming this criteria through industrial contacts.

Table 6-8. SUMMARY OF WATER QUALITY CRITERIA FOR BOILER FEED WATER[5]

Component	Conc. mg/L	Component	Conc. mg/L
Aluminum	0.01	Bicarbonate[c]	0.5
Calcium[c]	0.01	Alkalinity	0
Copper	0.01	Hardness ($CaCO_3$)	0.07
Iron	0.01	Dissolved Solids	0.5
Magnesium[c]	0.01	Suspended Solids	0
Manganese[c]	0.01	pH	8.8-9.2
Silica	0.01	Dissolved Oxygen	<0.005
Zinc[c]	0.01	COD	0
Ammonia[c]	0.07	Organics[c]	
Hydrogen Sulfide[c]	a	MBAS	0.1
Sulfate[c]	a	CTE	0
Chloride[c]	a,b	Turbidity (JTU)	0.05

a Accepted as-received (if meeting other limiting values); has never been a problem at concentrations encountered.
b Controlled by treatment of other constituents.
c Reference 6.

Subtask 6.4.1. PFH Process Analyses

The objective of this subtask is to analyze the data obtained from Task 3 and develop correlations describing the processes being developed as data become available.

Discussion

This task is scheduled to begin during the seventh month of the program.

Subtask 6.4.4. Economics

The objective of this subtask is to determine the cost to produce oil from beneficiated Eastern shale by evaluating the effects of operating conditions in the PFH retort, including the methane/hydrocarbon gas cracking and cracked-carbon combustion processing steps.

Discussion

This task is scheduled to begin during the seventh month of the program.

Task 8. Project Management and Reporting

The objective of this task is to coordinate all aspects of the program among DOE, IGT, and the various subcontractors to insure that a cohesive program is maintained.

During the quarter, IGT personnel visited MRI, UK-CAER, UA, and TTU to review program plans. IGT continued to monitor the progress and activities of the subcontractors working in the program via telephone and written reports.

Task 9. Information Required for the NEPA

The requirements of the National Environmental Policy Act (NEPA) are defined by statute. The objective of this task is to provide information to DOE relevant to the potential environmental impacts of the program tasks so that DOE can determine if an environmental assessment or environmental impact statement is required.

Discussion

During the quarter, IGT prepared and submitted the NEPA document to DOE for review and comments. The NEPA document contains input from IGT as well as each of the subcontractors participating in the program.

FUTURE PLANS

Work on the scheduled tasks will continue as planned.

ACKNOWLEDGMENTS

IGT acknowledges the significant work being conducted on this project by the individual subcontractors, their principal investigators, and colleagues -

MRI:	Dr. C. W. Schultz
UA:	Professor W. E. Hatcher
UK-CAER:	Dr. B. K. Parekh
TTU:	Professor W. P. Bonner

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Appendix A.

Summary of Estimated Capital Costs to Prepare Beneficiated Shale

Table A-1. 1988 CAPITAL COST ESTIMATE BREAKDOWN

Area Cost	Area Description	Mech/Equp Mat'l	Mech/Equ Labor	Piping Mat'l	Piping Labor	Mat'l	Struct Labor	Platewrk Mat'l	Platewrk Labor	Concrete Mat'l	Concrete Labor	Earthwk Mat'l	Earthwk Labor	Elect Mat'l	Elect Labor	Total
00	General	534,800	47,800	3,443,937	2,816,118	628,490	0							4,757,268	142,477	12,370,890
01	Crushg./Ore Storage	6,624,557	929,871			2,108,550	1,938,999	307,378	284,745	974,500	1,441,900			493,060	298,664	15,402,224
03	Ball Mill Grinding	5,699,499	1,429,513			127,379	129,092	166,648	143,995					739,959	412,317	8,848,402
04	Stir. Ball Mill Grndg.	64,337,500	1,320,855			584,978	601,225	33	30,607					675,918	366,173	67,950,453
05	Flotation	8,310,414	422,365			236,111	217,489	186,659	202,166					311,087	274,495	10,151,786
06	Thickening	12,004,200	89,102			263,997	193,359			81,420	52,370			788,381	684,550	15,219,511
07	Filtering	17,870,286	726,330			215,641	217,489	62,487	64,741					447,275	396,782	20,001,031
08	Drying	4,895,286	499,142			192,474	193,359	12,331	12,493					214,175	140,532	6,159,792
09	Briquetting	8,548,379	815,031			358,470	193,359	61,828	62,018					485,169	373,603	10,897,857
11	Tailings Disposal	567,000	39,483											42,037	14,786	663,306
12	Mill Buildings					2,376,231	3,514,316			2,080,313	2,935,192			482,464	273,114	11,661,630
13	Ancillary Buildings					3,057,800				311,610	455,430			106,079	106,079	4,096,097
14	Site Prep/Fence						0					0	3,710,209			3,710,209
15	Mobile Equipment	1,745,000														1,745,000
	SUBTOTALS	132,127,921	6,319,492	3,443,937	2,816,118	10,150,121	7,260,819	830,528	800,765	3,447,843	4,884,892	0	3,710,209	9,542,872	3,542,671	188,878,188
	Engineering Labor	(4% Mat'l Labor)														7,555,128
	Eng. Direct Costs	(2.5% Eng.)														188,878
	Eng. Consultants															200,000
	Construction Mgmt.															6,659,250
	Startup	(1% Mat'l & Labor)														1,888,782
	Field Changes	(2% Labor)														586,699
	Sales & Use Tax	(4% Mat'l)														6,381,729
	Ins. (Gen. Liability)	(.65% Mat'l & Labor)														1,227,708
	Contractor Profit	(5% Mat'l & Labor)														9,443,909
	Construction Svcs.															1,495,000
	TOTAL															224,505,271
	Contingency															15,669,654
	GRAND TOTAL															240,204,925

Table A-2. 1988 CAPITAL COST ESTIMATE SUMMARY

COST	DESCRIPTION	MATERIAL	LABOR	TOTAL
00	General	$9,364,495	$3,006,395	$12,370,890
01	Crushing & Ore Storage	$10,508,045	$4,894,179	$15,402,224
02	Ball Mill Grinding	$6,733,485	$2,114,917	$8,848,402
03	Stirred Ball Mill Grinding	$65,631,593	$2,318,860	$67,950,453
04	Flotation	$9,035,271	$1,116,515	$10,151,786
05	Thickening	$14,137,998	$1,081,513	$15,219,511
06	Filtering	$18,595,689	$1,405,342	$20,001,031
07	Drying	$5,314,266	$845,526	$6,159,792
08	Briquetting	$9,453,846	$1,444,011	$10,897,857
09	Tailings Disposal	$609,037	$54,269	$663,306
11	Mill Buildings	$4,939,008	$6,722,622	$11,661,630
12	Ancillary Buildings	$3,475,489	$620,608	$4,096,097
13	Site Preparation & Fence	$0	$3,710,209	$3,710,209
14	Mobile Equipment	$1,745,000	$0	$1,745,000
	SUBTOTALS	**$159,543,222**	**$29,334,966**	**$188,878,188**
	Engineering Labor			$7,555,128
	Engineering Direct Costs			$188,878
	Engineering Consultants			$200,000
	Construction Mgmt.			$6,659,250
	Startup			$1,888,782
	Field Changes			$586,699
	Sales and Use Tax			$6,381,729
	Insurance (Gen. Liability)			$1,227,708
	Contractor Profit			$9,443,900
	Construction Services			$1,495,000
	SUBTOTAL			**$224,505,271**
	Contingency			$15,699,654
	GRAND TOTAL			**$240,204,925**

Table A-3. 1992 CAPITAL COST ESTIMATE SUMMARY

COST	DESCRIPTION	MATERIAL	LABOR	TOTAL
00	General	$8,940,295	$3,102,600	$12,042,895
01	Crushing & Ore Storage	$9,917,670	$5,050,793	$14,968,463
03	Ball Mill Grinding	$7,273,476	$2,182,594	$9,456,070
04	Stirred Ball Mill Grinding	$72,044,240	$2,393,064	$74,437,304
05	Flotation	$9,822,421	$1,152,243	$10,974,664
06	Thickening	$15,408,319	$1,116,121	$16,524,440
07	Filtering	$20,364,362	$1,450,313	$21,814,675
08	Drying	$5,783,667	$872,583	$6,656,250
09	Briquetting	$10,266,180	$1,490,219	$11,756,399
11	Tailings Disposal	$666,191	$56,006	$722,197
12	Mill Buildings	$4,415,810	$6,937,746	$11,353,556
13	Ancillary Buildings	$3,079,920	$640,467	$3,720,387
14	Site Preparation & Fence	0	$3,828,936	$3,828,936
15	Mobile Equipment	$1,920,896	0	$1,920,896
	SUBTOTALS	**$169,903,447**	**$30,273,685**	**$200,177,132**
	Engineering Labor			$8,007,085
	Engineering Direct Costs			$200,177
	Engineering Consultants			$200,000
	Construction Mgmt.			$6,819,072
	Startup			$2,001,771
	Field Changes			$605,474
	Sales and Use Tax			$6,796,138
	Insuranace (Gen. Liability)			$1,301,151
	Contractor Profit			$10,008,857
	Construction Services			$1,581,373
	SUBTOTAL			**$37,521,098**
	TOTAL			**$237,698,230**
	Contingency (6%)			$14,261,894
	GRAND TOTAL			**$251,960,124**

END

DATE FILMED

3/26/93