BASE PROGRAM ON ENERGY RELATED RESEARCH

Quarterly Technical Progress Report

For Reporting Period: April–July 1998

Under Cooperative Agreement
DE-FC26-98FT40322

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

By
Western Research Institute
Laramie, Wyoming
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EXECUTIVE SUMMARY

Introduction

The Base Research Program at Western Research Institute (WRI) is planned to develop technologies to a level that will attract industrial sponsors for continued development under the Jointly Sponsored Research (JSR) Program. In many instances, a potential JSR cosponsor has been identified but additional laboratory or bench-scale data are necessary to assess the utility of the technology prior to cosponsor investment. Both peer and management review are employed prior to proposing Base projects to the U.S. Department of Energy (DOE).

The goals of the Base Research Program are in support of those of the JSR Program, which are designed to:

- Increase the production of United States and western energy resources, particularly low-sulfur coal, natural gas, oil, and renewable energy resources.
- Enhance the competitiveness of United States and western energy technologies in international markets and assist in technology transfer.
- Reduce the nation's dependence on foreign energy supplies and strengthen both the United States and regional economies.
- Minimize environmental impacts of energy production and utilization.

The goals of the JSR and Base Programs are accomplished by focusing research, development, demonstration, and commercialization in three major technology areas:

- THE ENERGY PROGRAM emphasize the increased production and utilization of domestic energy resources and include enhanced oil recovery, coal bonification and upgrading, coalbed methane recovery, and renewable energy resources.
- THE ENVIRONMENTAL PROGRAM minimize the impact of energy production and utilization by providing technology to clean underground oily wastes, mitigate acid mine drainage, and demonstrate uses for clean coal technology (CCT) and pressurized fluidized bed combustion (PFBC) waste solids.
- THE TECHNOLOGY ENHANCEMENT PROGRAM encompass resource characterization studies, the development of improved environmental monitors and sensors, and improved techniques and models for predicting the dispersion of hazardous gas releases.
Interactions between the Base and JSR Programs are dynamic and continuous, as commercialization of promising new technologies is the driving force for both. The Base Program explores innovative concepts that will attract industrial cosponsors for continued development under the JSR Program. In many instances, a potential JSR cosponsor has been identified, but additional laboratory or bench-scale data are necessary to verify the process or product concept. Once done, the project moves to the JSR stage where the objective is to develop and demonstrate the technology sufficiently for sound and prudent commercialization decisions to be made.

Not infrequently the reverse occurs. A project will demonstrate considerable promise at the JSR stage but require a supplemental Base Program activity to complete the applied research required for process optimization and a thorough technical understanding of the phenomena being exploited. As a result, the two programs are tightly integrated and complementary components of the overall development process.

In a high-technology global economy, technology development is crucial to the preservation and growth of the American economy and standard of living. Much of this technology and growth will come from companies that were small or nonexistent just a decade ago. Unlike the large multinational companies with captive research and development facilities, these companies must rely on creative and innovative contractors such as Western Research Institute to assist with their process and product development activities. It is a tribute to the wisdom and the foresight of the United States Department of Energy and the Federal Energy Technology Center (FETC) that activities such as the Cooperative Agreement Programs exist to meet this crucial national need.

The Base Cooperative Agreement (DE-FC26-98FT40322) was initiated on April 10, with funding of $500,000. A summary of funding and the number of projects is shown in Table 1.

Table 1. Summary of Base Funding for FY 1998–2003

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<th>Number of Projects</th>
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<td>TOTAL</td>
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Overview of Accomplishments During April 10 Through July 9, 1998

Task 1.0 Energy Program

Research was initiated on two of the four tasks in this program. Under Task 1.1 (CROW™ Process Application for Sites Contaminated With Trichloroethylene), equipment for physical
simulations has been constructed and testing to remediate soils contaminated with trichloroethylene has begun.

Under Task 1.2 (Petroleum Residua Solubility Parameter/Polarity Map), an analytical size exclusion chromatography unit was set up to evaluate changes in residua during processing.

Task 2.0 Environmental Program

One task was initiated this quarter. The experimental apparatus to evaluate remediation of metals-contaminated soils was assembled and tested under Task 2.1 (Evaluation of a Method Using Colloidal Aphrons to Remediate Metals-Contaminated Mine Drainage Water).

Task 3.0 Technology Enhancement

The only current task in this program was initiated and nearly completed this quarter. Experiments to evaluate two initiators for thermal degradation of plastics to usable products were completed. Plastics conversions of about 90% were achieved with one of the initiators. The final report on this task was drafted and is in review.

Table 2. Tasks Approved for Funding, FY 98

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<tr>
<th>Task</th>
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<td>CROW™ Process Application for Sites Contaminated With LNAPL and Chlorinated Solvents</td>
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<td>1.2</td>
<td>Petroleum Residual Solubility Parameter/Polarity Map</td>
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Federal Assistance Management Summary Reports (Substitute DOE Form F 4600.5) are included on pages 13–19.
## CONTRACT STATUS REPORT

**Contract No. DE-FC26-98FT40322**  
**Contract Start: April 10, 1998**  
**Contract Completion: April 9, 2003**  
**Reporting Period: Through 1st Quarter, 1st Year**

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* WRI fiscal reporting periods are on a 4/4/5 week per quarter basis. The accounting period ends on the fourth or fifth Saturday of the fiscal month. In this case, it was June 27, 1998.


TASK 1.0 ENERGY PROGRAM

BACKGROUND

Conventional petroleum resources in the United States continue to diminish, resulting in our continued dependence on foreign sources. The development and application of methods to increase and support production from domestic resources is important to the nation’s independence and well-being. A related area of environmental concern is the cleanup of oil waste materials. Numerous sites exist throughout the country where oily materials have been deposited and pose the threat of contamination to the environment. Effective methods for cleanup of these materials need to be developed for remediation of such sites.

LONG-TERM OBJECTIVES

The long-term objective of this task is to develop efficient processes that can be economically applied. The present market for technologies developed by Western Research Institute (WRI) appears to be with the major oil and gas companies, as well as with independent operators.

Areas currently being targeted are:

1. CROW™ Process Application for Sites Contaminated With LNAPL and Chlorinated Solvents (Task 1.1), which has the objective to extend WRI technology to remediation of sites contaminated with light nonaqueous phase liquids and chlorinated solvents.

2. Petroleum Residual Solubility Parameter/Polarity Map (Task 1.2), which has the objective to develop a mapping tool to enhance the understanding of changes occurring in resida during upgrading processes.

3. Laboratory and Bench-Scale Testing for Treating Used Motor Oil (Task 1.3), which has the objective to extend WRI TaBoRR® technology to processing of used motor oil. This task was not started this quarter.

4. Development and Testing of a Coal-Fired Gas Turbine System (Task 1.4), which has the objective to design and test a bench-scale fuel preparation system for coal use in a solid fuel-fired gas turbine unit. This task was not started this quarter.
TASK 1.1

CROW™ PROCESSES APPLICATION FOR SITES CONTAMINATED WITH LNAPL AND CHLORINATED SOLVENTS

Lyle A. Johnson

April – July 1998

TASK OBJECTIVES

The objectives of this task are:

- Demonstrate the effectiveness of applying the Contained Recovery of Oily Wastes (CROW™) process to sites contaminated with light non-aqueous phase liquids (LNAPL) and chlorinated solvents

- Conduct experiments and determine a range of operating conditions necessary to optimize LNAPL and chlorinated solvent recovery from contaminated soils

- Conduct numerical simulations to match the laboratory experiments and determine field-scale recoveries.

QUARTER OBJECTIVES

- Set up equipment for the physical simulation testing

- Perform shakedown testing to identify any problems with testing of the LNAPL or chlorinated solvents

- Conduct a literature survey to identify previous and present work by others in these areas.
ACCOMPLISHMENTS

- This task is a three-year study with this first year being a low-funded preparatory phase.

- The equipment for the physical simulations has been constructed and preliminary testing with trichloroethylene (TCE) has been completed. Four tests were conducted at ambient temperature, 150, 180, and 200°F (66, 82, and 93°C). Removal rates for the four tests were 47, 50, 96, and 98%, respectively.

- A literature survey has been conducted to assist in the design of the physical simulations and to identify operating parameters.

ACTIVITIES NEXT QUARTER

- Continue the physical simulation to validate equipment and operating protocol.
**TASK 1.2**

**PETROLEUM RESIDUA SOLUBILITY PARAMETER / POLARITY MAP**

John F. Schabron

April - July 1998

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**TASK OBJECTIVE**

The objective of this task is to develop a mapping tool that will enhance the understanding of the changes that occur in residua during upgrading and thus support the proprietary industry-sponsored work in which WRI is engaged.

**QUARTER OBJECTIVES**

- Begin the project by ordering materials, including solvents, analytical size exclusion column, and size exclusion chromatography software.

- Identify heavy oil sample materials and begin sequential solvent extraction separations.

- Begin analysis of fractions from sequential solvent extractions.

**ACCOMPLISHMENTS**

- The project was initiated and supplies were ordered. An analytical size exclusion chromatography instrument was set up using a new column and software for interpretation.

- Three heavy oil sample materials were obtained from another project at WRI. These were three stripper bottoms from a bench-scale study related to the TaBoRR (Tank Bottoms Recovery and Remediation) process. These are ideal materials for the mapping work since they represent the same heavy oil material undergoing three increasing severities of thermal treatment: 525, 650, and 700 °F, (273, 343, and 371 °C), respectively. The last material is producing some coke. The first two materials are fully soluble in toluene. Heithaus stability titrations were performed on the 525 and 650 °F (273 and 343 °C) treated materials.
and the toluene soluble oils from the 700 °F (371 °C) treated material. There were significant differences.

- Sequential solvent extractions were performed on the materials, and the fractions were weighed. The toluene soluble materials were analyzed for number average molecular weight by vapor pressure osmometry in toluene. The results were plotted on the new map.

**ACTIVITIES FOR NEXT QUARTER**

- Prepare abstracts for presentations of results at the spring 1999 American Chemical Society national meeting. Begin to prepare manuscripts.

- Perform additional solubility studies with cyclohexane on the whole oil samples and the heptane asphaltenes.

- Begin analytical scale size exclusion chromatography on the toluene soluble fractions.
TASK 2.0 ENVIRONMENTAL PROGRAM

BACKGROUND

Environmental issues in the United States have focused on ensuring clean air and water. The implementation of the Clean Air Act (CAA) and its amendments has indirectly resulted in an additional emphasis on solid waste management. The volume of solid waste is increasing from the fossil fuel production and consumption sectors, and the character of these wastes is also changing. The remediation of contaminated sites in the United States still remains a high priority. However, efforts to speed the cleanup of these sites are being hampered by the lack of effective high-volume treatment processes. Effective means of mitigating the environmental consequences related to energy development and utilization and the remediation of contaminated sites are needed today.

LONG-TERM OBJECTIVES

The Environmental Technologies task is designed to provide a range of environmental waste management services and research and development activities. The scope of the program includes developing waste management options for fossil fuel related industries and regulatory agencies. The long-term objectives of this task are to develop technologies that improve solid waste management practices, remediate hazardous waste and contaminated soils, and provide predictive models for environmental technologies.

Current areas of work include:

Evaluation of a Method Using Colloidal Gas Aphrons to Remediate Metals-Contaminated Mine Drainage Waters (Task 2.1), which has the objective to clean such waters by a unique method.

Task 2.2 (Development of a Procedure for Production of a Protective Covering for PEAC™ Units), which was not started this quarter.
TASK 2.1

EVALUATION OF A METHOD USING COLLOIDAL GAS APHRONS TO REMEDIATE METALS CONTAMINATED MINE DRAINAGE WATERS

R. William Grimes

April - July 1998

TASK OBJECTIVES

The objectives of this task are to provide proof of concept for the use of colloidal gas aphron enhanced precipitate flotation as a method for cleaning up metals contaminated waters and to provide preliminary data with which to assess the technical feasibility of this method as a remediation alternative for drainage from the Ferris-Haggarty Mine.

QUARTER OBJECTIVES

The objectives for this quarter were to assemble the experimental apparatus and colloidal gas aphron generator, select appropriate reagents, to establish procedures for sample digestion and analysis, and to begin the experimental work.

ACCOMPLISHMENTS

The experimental apparatus was assembled and tested. The apparatus consists of a colloidal gas aphron (CGA) generator similar to that described by Seba (Scamehorn, 1989), a chemical metering pump, a loop of tubing with a tee and valve, and a glass column 2.54 cm inside diameter and 40 cm tall. The metering pump is connected to circulate the CGA from the generator through the loop and back to the reservoir. The tee and valve provide a small flow of the circulating CGA to an orifice fitted to the bottom of the glass column by means of a rubber stopper. A second hole in the rubber stopper is fitted with a valve through which the column can be drained and sampled. The experimental apparatus is shown schematically in figure 1.
The proposed separation is based on precipitation of the metallic contaminant followed by flotation of the precipitate using CGA. Sodium hydroxide and sodium sulfide were selected for use as precipitating reagents following common industrial practice. Ferric hydroxide was used as a coprecipitant for some experiments. Sodium dodecyl sulfate (NaDDS) and hexadecyltrimethylammonium bromide (HTA) were selected as surfactants for the generation of CGA.

An EPA-approved mild digestion procedure (*Hach Water Analysis Handbook*, 1992) was found to be sufficiently vigorous to digest the precipitates formed in these experiments. A Hach
pocket colorimeter was purchased to analyze samples for copper content. The acceptability of the digestion and analysis were verified using standard samples.

Preliminary experiments in which the CGA was generated directly in the contaminated water were conducted. These tests were abandoned when it became evident that excessive surfactant consumption would make the method prohibitively expensive for application at remote sites.

Several series of experiments have been conducted in which the contaminated water sample was treated with a precipitating reagent and then transferred to the column (figure 1) and treated with a small volume of CGA. Experiments have been conducted using sodium hydroxide with and without ferric hydroxide coprecipitation followed by treatment with CGA. Experiments have been conducted to demonstrate the effect of pH and ferric ion concentration on the separation efficiency with respect to dissolved copper (the contaminant of concern at the Ferris-Haggerty Mine site). A series of sulfide precipitation experiments has been started but is not completed at the time of this writing.

**ACTIVITIES FOR NEXT QUARTER**

In the next quarter the sulfide precipitation experiments will be completed, data will be analyzed and a final report will be prepared.

**RESULTS**

Preliminary experiments in which the CGA was generated directly in the contaminated water required surfactant concentrations on the order of 300 ppm to generate a stable CGA. Upon standing, much of this surfactant drains back into the treated liquid. Contamination of the treated liquid with surfactant and consequent excessive consumption of surfactant would make this method undesirable for applications at remote sites.

The experiments in which a small flow of CGA is allowed to rise through the contaminated liquid have not yet been completed.

**REFERENCES**


TASK 3.0 TECHNOLOGY ENHANCEMENT

BACKGROUND

The refining of crude oils to transportation fuels is becoming more costly as a result of the gradually increasing amounts of heavy crude oils in the feedstock that must be refined. The common characteristics of these resources are a high heteroatom and metals content and, in some cases, a large amount of the material distilling in the vacuum gas oil boiling range. Preliminary work on a different type of resource has recently been conducted that demonstrates that a light distillate can be produced that contains very low concentrations of heteroatomic species. In addition, it is estimated that the cost of recovering this resource is not as great as producing a syncrude from oil or oil shale. The feedstocks for this process are the waste plastics that are discarded into landfills and waste motor oil. The conversion of these waste streams to useful products would go a long way toward improving our balance of payments with the oil-producing countries of the world and decreasing our dependence on foreign oil.

The above discussion indicates that the resources that can potentially be investigated under this task include not only the more conventional resources, such as heavy crude oils, coal and coal-derived liquids, but also waste plastics and other streams that may potentially be converted into useful products. The end uses of the former three resources are fairly well established. However, the successful conversion of the latter resources to useful products also has the potential of making a significant impact on our demand for foreign oil. In addition, the utilization of waste streams also helps resolve an impending problem with the status of landfills in the United States. Legislation is being enacted in many states that will severely limit the types of wastes that can be disposed of in a landfill.

LONG-TERM OBJECTIVE

The long-term objective of this task is to conduct basic science research to better understand and evaluate various applied energy technologies as they pertain to different feedstocks. This can be accomplished either through a better understanding of the nature and impact of the composition of different fossil fuels during the application of current processes or through the development of new processes. For the task proposed here, the objective for the fourth year is to access the technical feasibility of a process to desulfurize heavy oils using plasma reactions.

The current area of work is:

Heavy Oil/plastics Co-processing (Task 3.1), which has the objective to evaluate initiators for lower temperature processing of plastics in various heavy oils.
TASK 3.1

HEAVY OIL/PLASTICS CO-PROCESSING

Frank D. Guffey

April - July 1998

OBJECTIVES

The objective of the task is to begin optimization of the plastics/heavy oil process with the new initiators identified in earlier research. In particular, the activity of each of the two initiators, the concentration effects of the initiator added, and the frequency of addition of the initiator will be studied to maximize plastics conversion.

QUARTER OBJECTIVES

The objectives for this quarter were to evaluate the activity of the two initiators identified as potential candidates in earlier research. The initiators are designated as I1 and I2.

ACCOMPLISHMENTS

The activity of the two initiators has been completed, and the experimental phase of the research is complete. The topical report has been drafted and is undergoing review.

ACTIVITIES FOR NEXT QUARTER

• Complete and submit a topical report of the research effort
• Screen the four initiators identified from the literature search
• Begin evaluation of additional processing oils.
RESULTS

The results generated during this research effort can be summarized as follows:

2. Using initiator I2 as the free radical initiator, a plastics conversion of 90% can be achieved, which is higher than the 80% plastics conversion achieved with similar concentrations of initiator I1, and higher than observed using PVC as the initiator.

3. Maximum plastics conversion with initiator I2 can be achieved with the addition of 15% or less of the initiator, while the maximum conversion achieved with initiator I1 requires the addition of between 15 and 20% of I1 to the system.

4. The results suggest that there is a minimum concentration of plastics in the oil below which the initiator has no effect on plastics conversion. For initiator I2, the minimum concentration appears to be approximately 1% plastics in the oil.

5. In the concentration range investigated for initiator I2, the addition frequency does not appear to have a significant effect on plastics conversion.

6. The thermal decomposition products from initiator I1 collect in the condensers used for distillate collection, plugging them and causing a halt to operation of the process. For this reason alone, initiator I1 does not appear suitable as an initiator for this process.

7. The results generated from experiments conducted in the bench-scale reactor system indicate that initiator I2 is the free radical initiator of choice for the heavy oil/plastics coprocessing concept.

REFERENCE

### FEDERAL ASSISTANCE MANAGEMENT SUMMARY REPORT

**Contract No:** DE-FC26-98FT40322  
**Client:** U.S. Department of Energy  
**Contract Start:** April 10, 1998  
**Contract Complete:** April 9, 2003  
**Subtask Manager:** Johnson, L.  
**Project Title:** Task 1.1 - Contained Recovery of Oily Waste  
**Reporting Period:** April 10, 1998 - July 9, 1998

### EXPENDITURES ($)

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### EXPENDITURES VERSUS APPROVED BUDGET

![Graph showing expenditures versus approved budget]

### Milestones

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### Signature of Recipient and Date

**Signature of DOE Reviewing Representative and Date**

Substitute DOE Form F 4600.5 replacing Form EPA-499E

13
EXPENDITURES ($) 

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EXPENDITURES VERSUS APPROVED BUDGET

Milestones

- 98-1.2 Petroleum Resid Solubility Parameter
- 98-1.2A Technical Report
- 98-1.2B Quarterly Reports C, D

Signature of Recipient and Date

Signature of USDOE Reviewing Representative and Date

Substitute DOE Form F 8605.5 replacing Form EPA-499E
FEDERAL ASSISTANCE MANAGEMENT SUMMARY REPORT

Contract No: DE-FC26-98FT40322
Client: U.S. Department of Energy
Contract Start: April 10, 1998
Contract Complete: April 9, 2003
Subtask Manager: Satchwell, R.
Project Title: Task 1.3 - Treatment of Used Motor Oils
Reporting Period: April 10, 1998 - July 9, 1998

EXPENDITURES ($)

<table>
<thead>
<tr>
<th>Quarter 1</th>
<th>Quarter 2</th>
<th>Quarter 3</th>
<th>Quarter 4</th>
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EXPENDITURES VERSUS APPROVED BUDGET

Milestones

- 98-1.2 Tank Bottom Remediation of Used Motor Oils
- 98-1.2A Topical Report
- 98-1.3B, Quarterly Reports C, D

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Substitute DOE Form F 4600.5 replacing Form EPA-499E
FEDERAL ASSISTANCE MANAGEMENT SUMMARY REPORT

Contract No: DE-FC26-98FT40322
Client: U.S. Department of Energy
Contract Start: April 10, 1998
Contract Complete: April 9, 2003
Subtask Manager: Sethi, V.
Project Title: Task 1.4 - Coal-Fired Gas Turbine
Reporting Period: April 10, 1998 - July 9, 1998

EXPENDITURES ($)

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EXPENDITURES VERSUS APPROVED BUDGET

Milestones

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FEDERAL ASSISTANCE MANAGEMENT SUMMARY REPORT

Contract No: DE-FC26-98FT40322
Client: U.S. Department of Energy
Contract Start: April 10, 1998
Contract Complete: April 9, 2003
Subtask Manager: Grimes, W.
Project Title: Task 2.1 - Use of Gas Aphrons to Remediate Mine Drainage
Reporting Period: April 10, 1998 - July 9, 1998

**EXPENDITURES ($)**

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**EXPENDITURES VERSUS APPROVED BUDGET**

![Graph showing expenditures versus approved budget]

**Milestones**

- Milestone: Gas Aphrons Remediation
  - Planned: April 10, 1998
  - Progress: July 10, 1998
  - Scheduled: October 10, 1998
  - Delivered: January 10, 1999

- Milestone: Topical Report
  - July 10, 1998

- Milestone: Quarterly Report
  - July 10, 1998

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FEDERAL ASSISTANCE MANAGEMENT SUMMARY REPORT

Contract No: DE-FC26-98FT40322
Client: U.S. Department of Energy
Contract Start: April 10, 1998
Contract Complete: April 9, 2003
Subtask Manager: Sheesley, D.
Project Title: Task 2.2 - Develop Protective Covering for PEAC™ Units
Reporting Period: April 10, 1998 - July 9, 1998

EXPENDITURES ($)  

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EXPENDITURES VERSUS APPROVED BUDGET

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Signature of Recipient and Date

Signature of DOE Reviewing Representative and Date

Substitute DOE Form F 4400.5 replacing Form EPM-499E

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**FEDERAL ASSISTANCE MANAGEMENT SUMMARY REPORT**

Contract No: DE-FC26-98FT40322  
Client: U.S. Department of Energy  
Contract Start: April 10, 1998  
Contract Complete: April 9, 2003  
Subtask Manager: Guffey, F.  
Project Title: Task 3.1 - Heavy Oil/Plastics Coprocessing  
Reporting Period: April 10, 1998 - July 9, 1998

### EXPENDITURES ($)

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### EXPENDITURES VERSUS APPROVED BUDGET

![Chart showing expenditures versus approved budget]

### Milestones

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![Milestone chart with dates]

Signature of Recipient and Date:  
Signature of USDOE Reviewing Representative and Date:  

Substitute DOE Form F 4600.5 replacing Form EPM-459E

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