ACTIVITY REPORT

October 2001

Natural Gas & Oil Technology Partnership

bringing department of energy national laboratories capabilities to the petroleum industry

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Note: Natural Gas and Oil Technology Partnership projects are reported according to the following schedule:

January, March, May, July, September, November	**February, April, June, August, October, December**
Oil and Gas Recovery Technology Drilling, Completion, and Stimulation Technology Diagnostic and Imaging Technology	Upstream Environmental Technology Downstream Environmental Technology Ultra-Clean Fuels Technology

Natural Gas and Oil Technology Partnership on the World Wide Web: http://www.sandia.gov/ngotp/

Upstream Environmental Technology

Continuous Monitoring of Particulate Matter and Particulate Matter Precursor Emissions from Stationary Sources (Chevron and SNL)

Project is in close-out phase.

Development of an In-Well Oil/Water Separator for *In Situ* Recycling of Produced Water (Baker Hughes, Chevron, CINC, Oak Ridge Tool & Engineering, Phillips, REDA Pump, Texaco, Unocal, and ORNL)

Report in preparation.

Reducing Chemical Use and Toxicity in Produced-Water Systems (BP Amoco, Rhorback Casasco Systems, and ANL)

Report unavailable.

Sulfide Removal in Produced Brines by Microbial Oxidation (Phillips, U of Tulsa, and INEEL)

Report unavailable.

Characterization of Soluble Organics in Petroleum Waste Water (Chevron, Marathon, Phillips, Shell, Statoil, and ORNL)

Report unavailable.

Ecological Framework to Evaluate the Effect of Size and Distribution of Releases at Upstream Petroleum Sites (American Petroleum Institute, BP Amoco, Chevron, Exxon, Gas Technology Institute, Texaco, Unocal, LBNL, ORNL, and LLNL)

Highlights:

- Completed the calculation of preliminary site statistics for the TPP.
- Completed the conceptual modeling plan for the TPP.
- Presentation given at the NGOTP review meeting.

Project researchers completed the calculation of the preliminary site statistics using the Geographic Information System (GIS) developed for the Tall Grass Prairie Preserve (TPP) in Oklahoma. The site statistics calculated include total reserve area, pasture data (numbers, sizes, fencing lengths), length and area covered by roads, length of streams, number and area of wells, burn rate, brine spill number and area, and vegetation coverage (woodland, savanna, prairie, pasture, crop and other). These data will be used to support the modeling effort, described below.

LBNL completed the conceptual plan for creating a simulation model to evaluate the effect of size and distribution of spills and habitat patches on ecological populations at the TPP. The goal is to develop a generic, or template, model that can be used to determine a frequency, size and distribution of spills that would lead to a density of herbivores and/or predators that can't persist. We also plan to use the model to quantify how the effects of petroleum-related habitat loss differs for species with different life history attributes, mobility, and spatial habitat requirements.

The template model will be a spatially-explicit individual-based model (IBM). This type of model was selected as it lends itself best to representing realistic ecological interactions between animals and petroleum activities

because it facilitates simulation of animal movements that bring populations in contact with human activities. The template model will be implemented using an object-oriented programming structure using the C++ programming language. The structure of the template model consists of an hierarchical set of classes, each class containing a number of objects. Within the ecosystem class (which keeps track of the initial conditions and climatic variables) is a species class and a landscape class. The landscape class contains a basic landscape disturbance grid upon which the species class will interact. Simulations will occur on the background of a dynamic landscape that includes prescribed burning and grazing. Map layers in the GIS will be input landscapes for the model.

The template model will be developed with two specific species in mind to begin with, the prairie vole, and its predator, the short-eared owl. The vole is territorial, and has high site and mate fidelity, with the high-site fidelity including storage of food and long-term burrow occupancy. In addition, the vole comprises the majority of the short-eared owl's diet.

Research status was presented at the NGOTP meeting October 24, 2001 in Houston, TX. During this meeting we presented our research plans for FY02 and our requested budget.

Estimation and Reduction of Air Quality Modeling Uncertainties (Envair, EPRI, and LBNL)

Highlights:

- Presentations given at NGOTP review meeting and the American Association of Aerosol Research meeting.

Temporal and spatial resolution requirements for measuring atmospheric optical properties for reducing uncertainties in the determination of key photolysis reaction rates were investigated. These rates are required for modeling ozone (smog) formation using air quality models. This is especially important since an error of 50% on the nitrogen dioxide (NO_2) photolysis rate results in a 20% error in ozone. LBNL continues to estimate the rate of change and spatial variability of the:

- total ozone column,
- aerosol optical depth, and
- ground surface albedo.

We are using TOMS (Total Ozone Mapping Spectrometer) satellite data as a means of characterizing the variability of tropospheric ozone, which is important for computing the NO_2 photolysis rate. A necessary part of this effort is to determine how frequently the optical depth at a given spatial location must be updated. This is being determined using time series analysis and examining the autocorrelation as a function of lag rate (the time between two values of a quantity).

In other work, LBNL continued:

- work on an application to evaluate uncertainties in modeled hydrocarbon concentrations as a function of space and time. This is important because hydrocarbons are ozone and secondary organic aerosol precursors.
- comprehensive review of the application of elements of our uncertainty framework.
- writing a review paper describing approaches to evaluating model uncertainty.

LBNL staff attended and gave a talk at the NGOTP review meeting in Houston, TX. They also prepared and gave a presentation of their research results at the annual meeting of the American Association of Aerosol Research in October.

Remote Sensing for Environmental Baselining and Monitoring

(Chevron, UC-Davis, and ORNL)

Highlights:

- Identified data sources and established collaborations.
- Progress made on mixed-pixel problem.

In June 2000, a road grader cut an oil pipeline at the Jornada Experimental Range (Jornada) near Las Cruces, NM, causing oil to spray over a five acre region. Jornada has been operated as a research park by the Agricultural Research Service since 1912, and was selected as a Long-Term Ecological Research (LTER) site by the National Science Foundation in 1981. In 1995 (five years before the oil-spray event), the Jornada Experiment (JORNEX) was begun. This experiment involved the systematic collection of remotely sensed data from ground, airborne, and satellite platforms. The AVIRIS airborne hyperspectral sensor was used to collected data at Jornada in May 1997, and has been used to obtain additional data at least twice a year since 1998. One of the AVIRIS flights occurred on June 10, 2000, just ten days after the oil-spray accident, The rich set of "before and after" airborne hyperspectral data for Jornada provides an exceptional dataset for evaluating the effects of oil on desert vegetation. ORNL will collaborate with Dr. G. P. Asner (Stanford University) and Professor S. Ustin (UC-Davis) on the data processing and analysis of the massive AVIRIS data sets for Jornada.

ORNL has established collaborations with three groups that have collected hyperspectral field data at Jornada. The collaborators include Dr. Asner, Dr. A. Rango (Jornada), and the Army Corps of Engineers Topographic Engineering Center (TEC) Dr. Asner has compiled a field spectroscopic database containing more than 98,000 spectrum measurements of bare soils, green canopies, and litter canopies for 17 arid and semiarid sites in North and South America, representing a wide array of plant growth forms and species, vegetation conditions, and soil mineralogical-hydrological properties. Dr. Rango has collected hyperspectral plant canopy and soil reflectance measurements at Jornada at 5 m intervals along a series of 150 m transects twice a year since May 1997. Separate radiometric measurements were made for the dominant plant species, litter, and bare soil (10 measurements of each selected type) at each site. Our third collaborator (TEC) has provided a well documented library of 583 measurements of grasses, herbs, shrubs, trees, rocks and soils. Many of the measurements were made at Jornada. In addition, the TEC has provided hyperspectral measurements for oil on sand.

The presence of chlorophyll in green vegetation leads to the characteristic red-edge signature, which is strong absorption in the red band followed by strong reflectance in the near infrared. At Jornada, woody stems, dead plants, and oil-sprayed soil have a characteristic brown spectrum, that increases steadily from 400 to 1050 nm. This brown spectrum does not have the red-edge signature. During stress (autumn color is a familiar example), green plants become more brown with more reflectance in the red (and become yellow, orange, or red) and less reflectance in the near infrared. The length of each side of a hyperspectral pixel can range from less than 1 m for a field spectrometer to 17 m for the AVIRIS data. At Jornada, green leaves and plants are small and each pixel (both field and AVIRIS) will contain a mixture of green plants, brown stems, and brown soil and will have a spectrum that is between the extremes of green and brown. Since a stressed leaf moves from green toward brown, a "mixed pixel" will appear stressed. A key challenge for stress differentiation is to "un-mix" the pixels. ORNL has developed a method for subtracting the brown signature from the mixed data to reveal the red-edge signature of the green plants. This method can also be used to distinguish between brown stems and brown soil.

Downstream Environmental Technology

Bioprocessing of High-Sulfur Crudes via Application of Critical Fluid Biocatalysts

(Texaco, UOP, and INEEL)

Highlights:

- Enzymatic oxidation of DBT demonstrated in SCF emulsion.
- Maximum product yield of 4.1% obtained in 50/50 buffer/SCF emulsion.

Work exploring biocatalysis in organized media, including reverse micelles, microemulsions and emulsions continued into October. Previous efforts focused on surfactant-protein interactions.

By exploring surfactant - protein interactions, it was determined that the use of soybean peroxidase (SBP) with the perfluoropolyetherammonium carboxylate (PFPE) surfactant offered a potential route for the oxidation of dibenzothiophene (DBT). SBP demonstrated conversion in glycine buffer, pH = 2.4 at 47°C in 14 hours. Product was also detected in the presence of PFPE. SBP is active at low pH and at temperatures to 65°C, thus, oxidation in organized media using supercritical (SC) carbon dioxide (CO_2) with PFPE surfactant appeared promising. Reactions in SC CO_2 emulsions with PFPE were carried out at 45 and 65°C at 3,000 psi. Optimum performance was obtained at the higher temperature resulting in a 4.1% conversion of DBT to product. Due to the significantly higher solubility of DBT in the SCF emulsion, this conversion represented a 17-fold increase in product yield over aqueous phase biocatalysis.

Buffer/SCF mixtures from 20/80 to 50/50 wt% were explored. Reaction conditions were 3,000 psi, 65°C, pH=2.4, with conversion measured at reaction times of 15 hours. At the lower buffer level of 20 wt%, a two-phase mixture was formed. Buffer levels of 30 wt% and higher resulted in stable emulsions. By increasing buffer concentrations from 30–50 wt%, DBT conversion to product increased from 2.8–4.1%. The reaction carried out in a microemulsion (Wn = 19) produced a low conversion of 0.5%. The effect of pressure was briefly explored with 50/50 buffer/SC CO_2 mixtures at 2,000, 3,000 and 3,500 psi. At the lowest pressure only a trace quantity of DBT oxide products were formed, compared to a 4.1% conversion at 3,000 psi. At the highest pressure of 3,500 psi, the reactor cell leaked and no data was obtained. This experiment will be repeated in the near future.

Biological Upgrading of Heavy Oils for Viscosity Reduction

(BP Amoco, Chevron, EPRI Chemicals, Natural Gas Center, Texaco, and LBNL)

The objective of this project is to develop novel biocatalytic agents for the terminal oxidation of alkanes in mixtures. Biocatalytic agents that will oxidize longer chain (C_8 and greater) alkanes without oxidizing gasoline range alkanes (less than C_8) are particularly desirable. These biocatalytic agents are being evaluated for use in the biological up-grading of crude oil. LBNL currently has over 50 pure bacterial cultures capable of oxidizing alkanes. These cultures are being systematically evaluated for their phylogenic, genetic and physiological diversity.

A meeting was held in October with the Eidgenossische Technische Hochschule (ETH) Zurich Institute for Biotechnology. At this meeting we discussed the formation of a collaborative research effort and their interest in commercialization of chemical bioprocessing. ETH Zurich Institute for Biotechnology is the leading research organization for investigating fundamental and applied aspects of alkane oxidation by bacteria in the world. They have over 200 strains of alkane oxidizing bacteria that they have characterized to some degree. They have not evaluated the application of their organisms to oil bioprocessing and were interested in collaborating on this project, if the proper licensing agreements can be reached.

Kinetics of Biochemical Upgrading of Petroleum (Biocat, Chevrontexaco, Shell, and BNL)

Highlight:

- Biochemical upgrading process tested with three heavy crude oils and reservoir water.

Three heavy crude oils and reservoir water were used to test the biochemical upgrading process. The reservoir water was cultured with nutrient broth at 30°C under aerobic conditions. Several strains were isolated and tested for biochemical upgrading activities. A routine screening test consists of culturing 2% oil in mineral medium with strains isolated from an agar plate at 30°C for four days. The treated oils were dissolved in methylene chloride and analyzed by gas chromatography-mass spectrometry (GC-MS). The control samples were prepared in the same way except for the inoculation of the isolated strain. The results showed one unidentified bacilli was able to increase the gasoline and diesel fractions by 30% without the corresponding increment of organic sulfur content. The biochemical process may break down the heavy fractions into lighter gasoline and diesel fractions. The process is difficult to study because the heavy fraction is not analyzable by conventional GC. The results also show the need to develop an analytic method for analyzing the heavy macromolecular species in heavy crude oils.

Enzymatic Upgrading of Heavy Crudes via Partial Oxidation or Conversion of PAHs (Chevron, Phillips, Texaco, ORNL, and INEEL)

Previous efforts to improve expression of lignin peroxidase (*lip*) enzyme in *Pichia pastoris* have resulted in marginal improvements. To investigate the bottlenecks in the enzyme expression, following two experiments are being conducted. Firstly, a gene known to express in the host, *P. pastoris* (*lacZ*) is being introduced into the plasmid used for lignin peroxidase enzyme expression, downstream of the methanol-inducible promoter. The *lacZ* will also contain its own (IPTG-inducible) promoter and the expression will be induced using both, methanol and IPTG. If the expression of *lacZ* is successful, it would confirm that the methanol promoter is operational. Following this, the *lip* gene would be fused in-frame (1) at the 5-ft end of the *lacZ* gene next to the *lacZ* promoter, and (2) at the 3-ft end of the *lacZ*. The expression of the fusion protein will be verified by testing activity of beta-galactosidase (expressed by *lacZ*), and the *lip*.

In the second experiment, we will change the composition of the *lip* gene by changing codon make-up to enhance its expression in *P. pastoris* (codon usage in *P.pastoris* differs from that in *P. chrysosporium*). This is being done by decreasing the gas chromatography (GC) content of the *lip* gene. Three primers were designed to lower GC content of the *lip* gene and are being used to reconstruct the gene. The three modified gene fragments will be recombined together into a gene and expressed in *P. pastoris* to determine the effect of GC content on the expression efficacy. This experiment would reduce the GC content by approximately 2%.

The gene for the enzyme yeast cytochrome c peroxidase (ccp) obtained from Dr. Grant Mauk was restricted and transformed into *E. coli*. This is a third approach to improve activity of the peroxidase enzyme. In the system engineered by Dr. Mauk, the enzyme is produced intracellularly as an apoprotein in *E. coli*, which limits the ability to improve its expression. This ccp gene will be introduced into *P. pastoris* through subcloning into the pPICZC vector, which would facilitate gene shuffling with lignin peroxidase and generation of chimeric gene libraries. The goal is to produce a gene, which would carry the peroxidase active site while allowing higher levels of expression. By this time, we have completed a preliminary analysis of the secondary structures of the enzymes and generated predictions for rational shuffling.

A Predictive Model of Indoor Concentrations of Outdoor $PM_{2.5}$ in Homes

(Aerosol Dynamics, Envair, Western States Petroleum Association, and LBNL)

Highlights:

- Completed level 1 data analyses from Sampling Intensives.
- Progress made on model development.
- Presentations made at NGOTP review meeting and American Association of Aerosol Research meeting.

LBNL has completed the analysis of the filter samples and the Level 1 quality assurance and calibration of the real-time, size-resolved data. We are now working at bringing all data analysis to Level II. Level I analysis is concerned with ascertaining the precision of the various particle measurements, and Level II is concerned with inter-comparing measurements to ascertain accuracy. In the current period special emphasis was placed on examining the various measurements of carbonaceous aerosols. We also analyzed the Indoor/Outdoor concentration ratio of ammonia nitrate particles as a function of ventilation rate (expressed in air changes per hour) and as a function of the temperature difference between the indoor and outdoor environments. Under most conditions, the particulate ammonia nitrate indoors decreased markedly over outdoor values. Conditions that favored particulate ammonia nitrate indoors were very high ventilation rates and very cool interior temperatures.

Progress was made on all three aspects of model development: representation of the physical losses due to deposition and penetration, losses due to phase changes, and modeling of infiltration.

LBNL staff attended and gave a talk at the NGOTP review meeting in Houston. They also prepared and gave four presentations of their research results at the annual meeting of the American Association of Aerosol Research in October.

A Predictive Model of Indoor Concentrations of Outdoor Volatile Organic Compounds in Homes

(Aerosol Dynamics, Western States Petroleum Association, and LBNL)

LBNL is developing a model to estimate indoor VOC concentrations from outdoor VOC concentrations determined from outdoor measurements. The approach will be to integrate existing models (e.g., LBL infiltration model and gas phase reaction models) with information derived from experiments to provide essential data required for risk assessment. Ambient air concentrations of VOCs vary temporally, and often exhibit a diurnal pattern in response to changes in source terms and atmospheric processes. When these compounds enter buildings from outdoors by infiltration, they may interact with indoor surfaces acting as “sinks” (i.e., through sorption processes). Thus, when ambient concentrations are high, indoor concentrations may be reduced relative to outdoor values due to sorption. Conversely, re-emission from surfaces can result in higher indoor concentrations after outdoor concentrations have decreased. To capture this, LBNL designed and has nearly completed coding for a coupled outdoor/indoor air model. For the outdoor portion of the model, LBNL used a Lagrangian photochemical box model. The researchers implemented the chemical mechanism, SAPRC-99, into the model because it is the best representation of atmospheric VOC chemistry. A set of diurnally varying emissions representative of an urban area is used as model input. Dilution and entrainment of air aloft due to cell height variations induced by a temporally varying mixing height are included in the model to generate representative VOC concentrations. A state-of-the-art radiation model will be included to calculate actinic flux that will drive the photochemistry. Outdoor air concentrations of VOCs will be computed for several days for different seasons and for different spatial locations. The model then has the outdoor air flow into an indoor space of specific volume at a fixed infiltration rate. Infiltration rates in the range 0.1–5.0 ACH (air changes per hour) will be considered. Calculations have revealed that the photochemistry ceases indoors because there is insufficient actinic flux to support photochemistry. However, the VOC reactions continue to occur

because they are driven by reactions with ozone. Physical loss via sorption and gain via desorption are also represented in the model, and appropriate rate parameters required to evaluate them will be evaluated experimentally or taken from the literature.

Developing Enzyme and Biomimetic Catalysts for Upgrading Heavy Crudes via Biological Hydrogenation and Hydrodesulfurization (ORNL and ANL)

This project is aimed at investigating the potential of enzymatic and biomimetic catalysts for hydrogenation of oil compounds with the goal of upgrading crudes via sulfur removal and potentially molecular weight reduction.

A thermophilic hydrogenase from *Pyrococcus furiosus* and a mesophilic hydrogenase from *Desulfovibrio gigas* were selected as candidates for modification. Both of these are nitrogen-iron (Ni-Fe) hydrogenases. The structure for hydrogenase from *D. gigas* has been elucidated by Volbeda et.al., 1995. The primary and secondary structural details of the peptide harboring the Ni active site in the enzyme are known. The location of the Ni active site was used to select sites and amino acid residues for modification. The regions encompassing amino acids 48–52, 83–85, 122–126, 138–143, 318–322, 342–345, and 478–482 were found to be near the site where the activated hydrogen was released into the outside environment. Alkyl and aromatic groups will be attached at these sites to enhance substrate binding. The mesophilic enzyme will be produced and isolated in house. The laboratory set-up required to handle and process anaerobic organisms and enzymes including an anaerobic chamber and a Hungate station is being assembled. The thermophilic enzyme will be produced and purified by Dr. Michael Adams, University of Georgia, Athens. A statement of work has been written for this purpose and will include investigations into the activity and specificity of the unmodified enzyme.

Ultra-Clean Fuels Technology

Application of Barrier Membrane Technology to Catalytic Cracker Recycle Gas Hydrogen Separations (Chevron, Phillips, and ORNL)

Highlight:

- Separation tests completed on Knudsen membrane using binary gas mixtures.

Inorganic membrane gas separation testing has continued at ORNL. This initial testing utilized a Knudsen membrane provided by Inorganic Membrane Technology Laboratory (IMTL). The experimental phase of separation testing on this membrane was completed using five binary gas mixtures (helium-carbon dioxide, helium argon, hydrogen-methane and hydrogen-propane, and hydrogen-ethane); data analysis is proceeding. Separation efficiency of the membrane is being examined over a range of temperatures (23–200°C) and pressures (1–8 atm). For this membrane, when corrected for known operational effects (i.e., back pressure, cut, and mixing effects), separation has shown the expected behavior, with efficiencies trending toward ideal separation at low pressures and weakening at higher pressure (where viscous flow will increase). At lower temperatures, separation of hydrogen or helium from the higher boiling components (carbon dioxide and propane) is distinctly poorer than at higher temperatures, possibly due to enhanced surface transport of the heavier species.

Work on the Knudsen membrane has been carried out largely to develop experimental methods and to demonstrate that the apparatus and approach is capable of determining separation efficiency with reasonable accuracy in the range of physical conditions of interest. For the relatively low separation factors that a Knudsen membrane affords, this has been accomplished and thus there should be no difficulty studying membranes with higher separation factors.

Smaller pore-size membranes, some designed to enhance surface flow of heavier components and others to take advantage of molecular sieving, are being developed by IMTL and will be made available for testing in the near future.

Development of a Solid Catalyst Alkylation Process Using Supercritical Fluid Regeneration

(Marathon-Ashland and INEEL)

Highlights:

- Experimental system modified to increase catalyst loading.
- Product yields increased by 30%.
- Results presented at the American Institute of Chemical Engineers meeting.

Experimental efforts in September and October focused on increasing alkylate product yields. Earlier work exploring a commercial grade alkylation feed showed that the automated reaction/regeneration system did not reach steady state before the catalyst deactivated, resulting in modest overall product yields. By increasing the reactor catalyst loading from 2–10 grams and maintaining the feed flow rate to catalyst ratio constant product yields increase by approximately 30%.

The reactor/regenerator, heating and control systems were modified to accommodate the larger catalyst loading. In the modified system, total alkylate yields of 1.85 grams product/gram butene were obtained with selectivities of 79.5 wt% for C_8 and 63.6 wt% for trimethylpentanes (TMP). Catalyst regeneration at the higher catalyst loading was somewhat poorer compared to the lower catalyst loading experiments.

The flow rate of the supercritical fluid (SCF) was explored as a possible variable to improve regeneration. By changing the SCF flow rate to match the reaction feed rate, regeneration was improved by about 6%. Future efforts will focus on improving the regeneration process at the higher catalyst loading.

Results from the reaction/regeneration work using the model feed and the commercial feed were presented at the American Institute of Chemical Engineers annual meeting on November 5 in Reno, NV.