COMMERCIAL-SCALE DEMONSTRATION OF THE LIQUID PHASE METHANOL (LPMEOH™) PROCESS

TECHNICAL PROGRESS REPORT NO. 19

For The Period

1 January - 31 March 1999

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and

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for the
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Prepared for the United States Department of Energy
Federal Energy Technology Center
Under Cooperative Agreement No. DE-FC22-92PC90543

Patents cleared by Chicago on 18 May 1999.

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Abstract

The Liquid Phase Methanol (LPMEOH™) Demonstration Project at Kingsport, Tennessee, is a $213.7 million cooperative agreement between the U.S. Department of Energy (DOE) and Air Products Liquid Phase Conversion Company, L.P. (the Partnership) to produce methanol from coal-derived synthesis gas (syngas). Air Products and Chemicals, Inc. (Air Products) and Eastman Chemical Company (Eastman) formed the Partnership to execute the Demonstration Project. The LPMEOH™ Process Demonstration Unit was built at a site located at the Eastman chemicals-from-coal complex in Kingsport.

The LPMEOH™ Demonstration Unit operated at 99% availability throughout the reporting period. The LPMEOH™ Demonstration Unit was shutdown on 02 March 1999 to begin a scheduled biannual inspection of all pressure vessels as required by Tennessee state code. No issues were observed with any of the equipment items evaluated as part of the code inspection. The walls and internal heat exchanger within the LPMEOH™ Reactor showed no evidence of erosion, pitting, or fouling by catalyst slurry. The LPMEOH™ Demonstration Unit was re-started on 14 March 1999.

Catalyst activity, as defined by the ratio of the rate constant at any point in time to the rate constant for freshly reduced catalyst (as determined in the laboratory autoclave), was monitored throughout the reporting period. A ten-day period of stable operation ended on 04 January 1999. During this stable operating period, the rate of catalyst deactivation was 0.45% per day at a reactor temperature of 235°C. This result is comparable to the baseline deactivation rate from the 4-month proof-of-concept run at the LaPorte Alternative Fuels Development Unit (AFDU) in 1988/89 (this run was performed at 250°C).

During the quarter, the LPMEOH™ Demonstration Unit has continued to perform with high slurry concentrations. Slurry concentrations greater than 48 wt% were achieved during this quarter with no apparent hydrodynamic instability. Three batches of catalyst were activated and added during the reporting period, bringing catalyst loading up to 151% of design. During late February 1999, three separate transfers of catalyst slurry from the reactor were conducted in order to dilute the slurry concentration in anticipation of the outage activities in March of 1999. These steps reduced the calculated slurry concentration in the reactor from over 48 wt% to approximately 40 wt%. Reactor conditions were held at 235°C except for a brief production test in late February of 1999 when reactor temperature was increased to 240°C.

Catalyst slurry samples from the LPMEOH™ Reactor have been taken on a regular basis to correlate any change in unit performance with changes in the physical properties of the catalyst. Samples have continued to show an increase in arsenic loading; there appears to be a weak correlation between this increased arsenic concentration and the rate of deactivation of the catalyst. A plan to add arsenic removal capacity to the guard bed system is expected to be executed in the May/June 1999 time-frame.

The performance of the alternative gas sparger, which was designed by Air Products and installed into the LPMEOH™ Reactor prior to the restart of the LPMEOH™ Demonstration Unit in December of 1997, was monitored. During January and February of 1999, the sparger resistance coefficient across the LPMEOH™ Reactor continued to increase beyond the step-change experienced in October of 1999. It was necessary to reduce the flowrate of the primary syngas feed (Balanced Gas) from greater than 700 KSCFH to less than 600 KSCFH to compensate for higher sparger pressure drop. During the March 1999 scheduled outage, a modified gas sparger was installed in the reactor; Air Products developed this design using the same principles as the original device. Upon restarting on 14 March 1999, the performance of the new sparger met the design expectations for pressure drop and reactor operation.

During the reporting period, a total of 3,889,278 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, over 34.6 million gallons of methanol has been produced. Eastman accepted all of this methanol for use in the production of methyl acetate, and ultimately cellulose acetate and acetic acid. No environmental incidents were reported during this quarter.

During this quarter, planning, procurement, and test operations continued on the seven project sites selected for the off-site, product-use test program. DOE accepted a recommendation by Air Products to perform additional experiments and emissions testing on the stationary gas turbine apparatus at West Virginia University. Construction of a reformer test apparatus to determine the operating characteristics of fuel-grade methanol as a feed to a fuel cell is nearing completion.

During the reporting period, planning for a design verification test run of the Liquid Phase Dimethyl Ether (LPDME™) Process at the LaPorte AFDU continued. The results of a set of experiments on a commercially available dehydration catalyst showed that the desired catalyst life could be achieved at a 10-20% reduction in system productivity. In continued tests during the reporting period, the LPDME™ dual catalyst system exhibited higher catalyst deactivation when the slurry concentration in the autoclave was increased; this result was not observed when LPMEOH™ catalyst was tested alone at the higher concentration. Additional laboratory experiments are expected to be completed by 01 July 1999, so that shakedown activities at the LaPorte AFDU can commence in September of 1999.

The Topical Report entitled “Economic Analysis - LPMEOH™ Process as an Add-on to IGCC for Coproduction” was approved and issued for release.

Ninety-nine percent (99%) of the $38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 31 March 1999. Thirty-seven percent (37%) of the $158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 March 1999.

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ACRONYMS AND DEFINITIONS

Acurex	-	Acurex Environmental Corporation
Air Products	-	Air Products and Chemicals, Inc.
AFDU	-	Alternative Fuels Development Unit - The “LaPorte PDU”
AFFTU	-	Alternative Fuels Field Trailer Unit
Balanced Gas	-	A syngas with a composition of hydrogen (H_2), carbon monoxide (CO), and carbon dioxide (CO_2) in stoichiometric balance for the production of methanol
Carbon Monoxide Gas	-	A syngas containing primarily carbon monoxide (CO); also called CO Gas
Catalyst Age (η -eta)	-	the ratio of the rate constant at any point in time to the rate constant for a freshly reduced catalyst (as determined in the laboratory autoclave)
Catalyst Concentration	-	Synonym for Slurry Concentration
Catalyst Loading	-	Synonym for Slurry Concentration
CO Conversion	-	the percentage of CO consumed across the reactor
Crude Grade Methanol	-	Underflow from rectifier column (29C-20), defined as 80 wt% minimum purity; requires further distillation in existing Eastman equipment prior to use
DME	-	dimethyl ether
DOE	-	United States Department of Energy
DOE-FETC	-	The DOE's Federal Energy Technology Center (Project Team)
DOE-HQ	-	The DOE's Headquarters - Coal Fuels and Industrial Systems (Project Team)
DTP	-	Demonstration Test Plan - The four-year Operating Plan for Phase 3, Task 2 Operation
DVT	-	Design Verification Testing
Eastman	-	Eastman Chemical Company
EIV	-	Environmental Information Volume
EMP	-	Environmental Monitoring Plan
EPRI	-	Electric Power Research Institute
FFV	-	flexible-fuel vehicle
Fresh Feed	-	sum of Balanced Gas, H_2 Gas, and CO Gas
Gas Holdup	-	the percentage of reactor volume up to the Gassed Slurry Height which is gas
Gassed Slurry Height	-	height of gassed slurry in the reactor
HAPs	-	Hazardous Air Pollutants
Hydrogen Gas	-	A syngas containing an excess of hydrogen (H_2) over the stoichiometric balance for the production of methanol; also called H_2 Gas
IGCC	-	Integrated Gasification Combined Cycle, a type of electric power generation plant
IGCC/OTM	-	An IGCC plant with a "Once-Thru Methanol" plant (the LPMEOH™ Process) added-on
Inlet Superficial Velocity	-	the ratio of the actual cubic feet of gas at the reactor inlet (calculated at the reactor temperature and pressure) to the reactor cross-sectional area (excluding the area contribution by the internal heat exchanger); typical units are feet per second
K	-	Sparger resistance coefficient (term used in calculation of pressure drop)
KSCFH	-	Thousand Standard Cubic Feet per Hour
LaPorte PDU	-	The DOE-owned experimental unit (PDU) located adjacent to Air Products’ industrial gas facility at LaPorte, Texas, where the LPMEOH™ process was successfully piloted
LPDME™	-	Liquid Phase DME process, for the production of DME as a mixed coproduct with methanol
LPMEOH™	-	Liquid Phase Methanol (the technology to be demonstrated)
M85	-	a fuel blend of 85 volume percent methanol and 15 volume percent unleaded gasoline
MeOH	-	methanol
Methanol Productivity	-	the gram-moles of methanol produced per hour per kilogram catalyst (on an oxide basis)
MTBE	-	methyl tertiary butyl ether
MW	-	molecular weight, pound per pound mole
NEPA	-	National Environmental Policy Act
OSHA	-	Occupational Safety and Health Administration

ACRONYMS AND DEFINITIONS (cont'd)

ρ	density, pounds per cubic foot
Partnership	Air Products Liquid Phase Conversion Company, L.P.
PDU	Process Development Unit
PFD	Process Flow Diagram(s)
ppbv	parts per billion (volume basis)
ppmw	parts per million (weight basis)
Project	Production of Methanol/DME Using the LPMEOH™ Process at an Integrated Coal Gasification Facility
psi	Pounds per Square Inch
psia	Pounds per Square Inch (Absolute)
psig	Pounds per Square Inch (gauge)
P&ID	Piping and Instrumentation Diagram(s)
Raw Methanol	sum of Refined Grade Methanol and Crude Grade Methanol; represents total methanol which is produced after stabilization
Reactor Feed	sun of Fresh Feed and Recycle Gas
Reactor O-T-M Conversion	percentage of energy (on a lower heating value basis) in the Reactor Feed converted to methanol (Once-Through-Methanol basis)
Reactor Volumetric Productivity	the quantity of Raw Methanol produced (tons per day) per cubic foot of reactor volume up to the Gassed Slurry Level
Recycle Gas	the portion of unreacted syngas effluent from the reactor "recycled" as a feed gas
Refined Grade Methanol	Distilled methanol, defined as 99.8 wt% minimum purity; used directly in downstream Eastman processes
SCFH	Standard Cubic Feet per Hour
Slurry Concentration	percentage of weight of slurry (solid plus liquid) which is catalyst (on an oxide basis)
Sl/hr-kg	Standard Liter(s) per Hour per Kilogram of Catalyst
Syngas	Abbreviation for Synthesis Gas
Syngas Utilization	defined as the number of standard cubic feet of Balanced Gas plus CO Gas to the LPMEOH™ Demonstration Unit required to produce one pound of Raw Methanol
Synthesis Gas	A gas containing primarily hydrogen (H_2) and carbon monoxide (CO), or mixtures of H_2 and CO; intended for "synthesis" in a reactor to form methanol and/or other hydrocarbons (synthesis gas may also contain CO_2, water, and other gases)
Tie-in(s)	the interconnection(s) between the LPMEOH™ Process Demonstration Facility and the Eastman Facility
TPD	Ton(s) per Day
V	volumetric flowrate, thousand standard cubic feet per hour
VOC	volatile organic compound
WBS	Work Breakdown Structure
wt	weight

Executive Summary

The Liquid Phase Methanol (LPMEOH™) Demonstration Project at Kingsport, Tennessee, is a $213.7 million cooperative agreement between the U.S. Department of Energy (DOE) and Air Products Liquid Phase Conversion Company, L.P. (the Partnership) to produce methanol from coal-derived synthesis gas (syngas). Air Products and Chemicals, Inc. (Air Products) and Eastman Chemical Company (Eastman) formed the Partnership to execute the Demonstration Project. The LPMEOH™ Process Demonstration Unit was designed, constructed, and is in operation at a site located at the Eastman chemicals-from-coal complex in Kingsport.

On 04 October 1994, Air Products and Eastman signed the agreements that would form the Partnership, secure the demonstration site, and provide the financial commitment and overall project management for the project. These partnership agreements became effective on 15 March 1995, when DOE authorized the commencement of Budget Period No. 2 (Modification No. A008 to the Cooperative Agreement). The Partnership has subcontracted with Air Products to provide the overall management of the project, and to act as the primary interface with DOE. As subcontractor to the Partnership, Air Products provided the engineering design, procurement, construction, and commissioning of the LPMEOH™ Process Demonstration Unit, and is providing the technical and engineering supervision needed to conduct the operational testing program required as part of the project. As subcontractor to Air Products, Eastman is responsible for operation of the LPMEOH™ Process Demonstration Unit, and for the interconnection and supply of syngas, utilities, product storage, and other needed services.

The project involves the operation of an 80,000 gallons per day (260 tons per day (TPD)) methanol unit utilizing coal-derived syngas from Eastman's integrated coal gasification facility. The new equipment consists of syngas feed preparation and compression facilities, the liquid phase reactor and auxiliaries, product distillation facilities, and utilities.

The technology to be demonstrated is the product of a cooperative development effort by Air Products and DOE in a program that started in 1981. Developed to enhance electric power generation using integrated gasification combined cycle (IGCC) technology, the LPMEOH™ process is ideally suited for directly processing gases produced by modern day coal gasifiers. Originally tested at the Alternative Fuels Development Unit (AFDU), a small, DOE-owned experimental unit in LaPorte, Texas, the technology provides several improvements essential for the economic coproduction of methanol and electricity directly from gasified coal. This liquid phase process suspends fine catalyst particles in an inert liquid, forming a slurry. The slurry dissipates the heat of the chemical reaction away from the catalyst surface, protecting the catalyst and allowing the methanol synthesis reaction to proceed at higher rates.

At the Eastman chemicals-from-coal complex, the technology is integrated with existing coal gasifiers. A carefully developed test plan will allow operations at Eastman to simulate electricity demand load-following in coal-based IGCC facilities. The operations will also demonstrate the enhanced stability and heat dissipation of the conversion process, its reliable on/off operation, and its ability to produce methanol as a clean liquid fuel without additional upgrading. An off-site, product-use test program will be conducted to demonstrate the suitability of the methanol product as a transportation fuel and as a fuel for stationary applications for small modular electric power generators for distributed power.

The four-year operating test phase and off-site product-use test program will demonstrate the commercial viability of the LPMEOH™ process and allow utilities to evaluate the application of this technology in the coproduction of methanol with electricity. A typical commercial-scale IGCC coproduction facility, for example, could be expected to generate 200 to 350 MW of electricity, and to also manufacture 45,000 to 300,000 gallons per day of methanol (150 to 1,000 TPD). A successful demonstration at Kingsport will show the ability of a local resource (coal) to be converted in a reliable (storable) and environmentally preferable way to provide the clean energy needs of local communities for electric power and transportation.

This project may also demonstrate the production of dimethyl ether (DME) as a mixed coproduct with methanol if laboratory- and pilot-scale research and market verification studies show promising results. If implemented, the DME would be produced during the last six months of the four-year demonstration period. DME has several commercial uses. In a storable blend with methanol, the mixture can be used as a peaking fuel in gasification-based electric power generating facilities, or as a diesel engine fuel. Blends of methanol and DME can be used as chemical feedstocks for synthesizing chemicals, including new oxygenated fuel additives.

The project was reinitiated in October of 1993, when DOE approved a site change to the Kingsport location. DOE conditionally approved the Continuation Application to Budget Period No. 2 (Design and Construction) in March of 1995 and formally approved it on 01 June 1995 (Modification No. M009). After approval, the project initiated Phase 1 - Design - activities. Phase 2 - Construction - activities were initiated in October of 1995. The project required review under the National Environmental Policy Act (NEPA) to move to the construction phase. DOE prepared an Environmental Assessment (DOE/EA-1029), and subsequently a Finding of No Significant Impact (FONSI) was issued on 30 June 1995. The Cooperative Agreement was modified (Modification No. A011) on 08 October 1996, authorizing the transition from Budget Period No. 2 (Design and Construction) to the final Budget Period (Commissioning, Start-up, and Operation). This modification provides the full $213,700,000 of authorized funding, with 56.7% participant cost share and 43.3% DOE cost share.

The LPMEOH™ Demonstration Unit operated at 99% availability throughout the reporting period. Two short unit shutdowns were experienced in early January and February of 1999; both were due to electrical transients.

The LPMEOH™ Demonstration Unit was shutdown on 02 March 1999 to begin a scheduled biannual inspection of all pressure vessels as required by Tennessee state code. Catalyst slurry was transferred to the 29D-02 slurry tank during this outage. No issues were observed with any of the equipment items evaluated as part of the code inspection. The walls and internal heat exchanger within the LPMEOH™ Reactor showed no evidence of erosion, pitting, or fouling by catalyst slurry. Inspection of the 29E-02 economizer heat exchanger, which condenses process oil from the reactor effluent stream, revealed a significant blockage of the heat exchanger tubes with catalyst slurry. This observation was expected based upon the loss of heat transfer performance of the economizer heat exchanger during the last few months of operation.

Maintenance activities were also completed during this outage. This work included the addition of an oil flush at the economizer heat exchanger, cleaning of all heat exchanger tubes, and re-routing of the piping between the 29C-06 cyclone and the reactor.

Catalyst was pressure-transferred from the slurry tank to the reactor, and the LPMEOH™ Demonstration Unit was re-started on 14 March 1999. The economizer heat exchanger returned to performance levels expected for clean service. Condensed oil was gravity-drained from the 29C-05 secondary oil knock-out drum to the reactor without problems. However, the new piping from the cyclone to the reactor was not placed into service due to an obstruction that could not be cleared by flushing with clean oil. This entrained slurry is presently returned to the reactor via the original routing along with condensed oil from the secondary oil knock-out drum. During an upcoming complex-wide outage in April of 1999, this new section of piping from the cyclone will be cleared.

Catalyst activity, as defined by the ratio of the rate constant at any point in time to the rate constant for freshly reduced catalyst (as determined in the laboratory autoclave), was monitored throughout the reporting period. A ten-day period of stable operation ended on 04 January 1999. During this stable operating period, the rate of catalyst deactivation was 0.45% per day at a reactor temperature of 235°C. This result is comparable to the baseline deactivation rate from the 4-month proof-of-concept run at the LaPorte AFDU in 1988/89 (this run was performed at 250°C). During the quarter, there were other no stable operating periods of sufficient length which are needed to quantify changes in catalyst activity with time.

During the quarter, the LPMEOH™ Demonstration Unit has continued to perform with high slurry concentrations. Slurry concentrations greater than 48 wt% were achieved during this quarter with no apparent hydrodynamic instability. Three batches of catalyst were activated and added during the reporting period, bringing catalyst loading up to 151% of design. During late February 1999, three separate transfers of catalyst slurry from the reactor to the 29C-30 catalyst reduction vessel were conducted in order to dilute the slurry concentration in anticipation of the outage activities in March of 1999. These steps reduced the calculated slurry concentration in the reactor from over 48 wt% to approximately 40 wt%. No apparent negative impacts were experienced based on the catalyst removal from the reactor. The withdrawal process itself proceeded easily and without incident. Reactor conditions were held at 235°C except for a brief production test in late February of 1999 when reactor temperature was increased to 240°C.

Catalyst slurry samples from the LPMEOH™ Reactor have been taken on a regular basis to correlate any change in unit performance with changes in the physical properties of the catalyst. Samples have continued to show an increase in arsenic loading, with levels in excess of the concentrations measured on samples of the initial charge of methanol synthesis catalyst taken from the reactor at the end of the 1997 operating campaign. There appears to be a weak correlation between this increased arsenic loading and the rate of deactivation of the catalyst. A meeting was held at Kingsport on 5 January 1999 between Air Products, Eastman, and United Catalysts, Inc. to review the plant configuration and analytical results. A plan to add arsenic removal capacity to the guard bed system is expected to be executed in the May/June 1999 time-frame.

Sulfur continues to be measured above the analytical detection limit. Copper crystallite size measurements have shown an increase in the most recent samples, however, the size increase has stabilized over the last few samples. Other methods of crystallite size determination have corroborated the increased size measurements. Levels of iron and nickel have remained low and steady since the restart in December of 1997.

The performance of the alternative gas sparger, which was designed by Air Products and installed into the LPMEOH™ Reactor prior to the restart of the LPMEOH™ Demonstration Unit in December of 1997, was monitored. During January and February of 1999, the sparger resistance coefficient across the LPMEOH™ Reactor continued to increase beyond the step-change experienced in October of 1999 (as reported in Technical Progress Report No. 18). As a result of the greater sparger pressure drop, it was necessary to reduce the Balanced Gas flow from greater than 700 KSCFH to less than 600 KSCFH. Also, condensed oil and entrained slurry could not be gravity-drained to the flush connection at the gas inlet line to the reactor. In order to overcome the pressure drop, the streams were batch-transferred to the catalyst reduction vessel and returned to the reactor via the 29G-30 slurry transfer pump.

During the March 1999 scheduled outage, the reactor gas sparger was removed and inspected. It was estimated that the blockage in the sparger was consistent with the resistance coefficient which was calculated prior to shutdown. During this outage, a modified gas sparger was installed in the reactor; Air Products developed this design using the same principles as the original device. Upon restarting on 14 March 1999, the performance of the new sparger met the design expectations for pressure drop and reactor operation.

During the reporting period, a total of 3,889,278 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, over 34.6 million gallons of methanol has been produced. Eastman accepted all of this methanol for use in the production of methyl acetate, and ultimately cellulose acetate and acetic acid. No environmental incidents were reported during this quarter.

During this quarter, planning, procurement, and test operations continued on the seven project sites selected for the off-site, product-use test program. Air Products recommended to DOE that a proposal which was submitted on 01 December 1998 to perform additional experiments and emissions testing on the stationary gas turbine apparatus at West Virginia University should be accepted. DOE accepted this recommendation during the reporting period. A final report was issued on the results of operation of a flexible-fuel vehicle on fuel-grade methanol from the LPMEOH™ Demonstration Project. Construction of a reformer test apparatus to determine the operating characteristics of fuel-grade methanol as a feed to a fuel cell is near completion. Additional equipment which is used to activate the reformer catalyst is being assembled.

During the reporting period, planning for a design verification test run of the Liquid Phase Dimethyl Ether (LPDME™) Process at the LaPorte AFDU continued. Air Products has been performing laboratory autoclave tests of samples of the dehydration catalyst from the commercial catalyst manufacturer (Engelhard). The results to date have not been consistent, indicating that all issues related to catalyst scale-up have not been resolved. A set of experiments was performed on a commercially available dehydration catalyst. These results showed that the desired catalyst life could be achieved with the commercially available dehydration catalyst at a 10-20% reduction in system productivity (primarily a reduction in the selectivity to DME). In continued tests with the commercially available dehydration catalyst, the LPDME™ catalyst system operated at the desired catalyst life at a total of 10 grams methanol and dehydration catalyst in the autoclave; this corresponds to a slurry concentration of 7 wt%. Experiments were performed at the 30 gram catalyst loading (18 wt%), which approaches the system limitation for the autoclave, in order to study the effects of slurry concentration (note that the trial at the LaPorte AFDU is expected to be performed at 40 to 45 wt% slurry concentration). The rate of decline in the dual LPDME™ catalyst activity at this higher loading was greater than results at the 10 gram condition. An experiment was performed with the LPMEOH™ catalyst alone at the 30 gram loading in order to determine a point for comparison; this catalyst performed at the laboratory baseline for catalyst life. Work is continuing in the laboratory autoclaves to identify the causes of catalyst deactivation at the higher slurry concentration. Activities in the laboratory are expected to be concluded by 01 July 1999, so that shakedown activities at the LaPorte AFDU can commence in September of 1999.

A Project Review Meeting was held in Pittsburgh, PA on 24-25 March 1999. The results of the unit operation and equipment inspection during the recent outage were reviewed, and an update on the status of the catalyst development for the LPDME™ design verification test at the LaPorte AFDU was provided.

The Topical Report entitled “Economic Analysis - LPMEOH™ Process as an Add-on to IGCC for Coproduction” was approved and issued for release on 18 February 1999.

An abstract for the paper entitled “Commercial-Scale Demonstration of the Liquid Phase Methanol (LPMEOH™) Process: Operating Experience Update” was submitted. This paper will be presented at the Seventh Clean Coal Technology Conference in Knoxville, TN (21-24 June 1999). The LPMEOH™ Demonstration Unit will serve as the site for the plant tour which is associated with the Conference. Secretary of Energy Bill Richardson is expected to attend a reception following the tour. A presentation entitled “Liquid Phase Methanol (LPMEOH™) Process Development” was given at the Spring Meeting of the American Chemical Society on 24 March 1999.

Ninety-nine percent (99%) of the $38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 31 March 1999. Thirty-seven percent (37%) of the $158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 March 1999.

A. Introduction

The Liquid Phase Methanol (LPMEOH™) demonstration project at Kingsport, Tennessee, is a $213.7 million cooperative agreement between the U.S. Department of Energy (DOE) and Air Products Liquid Phase Conversion Company, L. P. (the Partnership). Air Products and Chemicals, Inc. (Air Products) and Eastman Chemical Company (Eastman) formed the Partnership to execute the Demonstration Project. A demonstration unit producing 80,000 gallons per day (260 TPD) of methanol was designed, constructed, and is operating at a site located at the Eastman chemicals-from-coal complex in Kingsport. The Partnership will own and operate the facility for the four-year demonstration period.

This project is sponsored under the DOE's Clean Coal Technology Program, and its primary objective is to "demonstrate the production of methanol using the LPMEOH™ Process in conjunction with an integrated coal gasification facility." The project will also demonstrate the suitability of the methanol produced for use as a chemical feedstock or as a low-sulfur dioxide, low-nitrogen oxides alternative fuel in stationary and transportation applications. The project may also demonstrate the production of dimethyl ether (DME) as a mixed coproduct with methanol, if laboratory- and pilot-scale research and market verification studies show promising results. If implemented, the DME would be produced during the last six months of the four-year demonstration period.

The LPMEOH™ process is the product of a cooperative development effort by Air Products and the DOE in a program that started in 1981. It was successfully piloted at a 10-TPD rate in the DOE-owned experimental unit at Air Products' LaPorte, Texas, site. This demonstration project is the culmination of that extensive cooperative development effort.

B. Project Description

The demonstration unit, which occupies an area of 0.6 acre, is integrated into the existing 4,000-acre Eastman complex located in Kingsport, Tennessee. The Eastman complex employs approximately 12,000 people. In 1983, Eastman constructed a coal gasification facility utilizing Texaco technology. The synthesis gas (syngas) generated by this gasification facility is used to produce carbon monoxide and methanol. Both of these products are used to produce methyl acetate and ultimately cellulose acetate and acetic acid. The availability of this highly reliable coal gasification facility was the major factor in selecting this location for the LPMEOH™ Process Demonstration. Three different feed gas streams (hydrogen gas or H_2 Gas, carbon monoxide gas or CO Gas, and the primary syngas feed known as Balanced Gas) are diverted from existing operations to the LPMEOH™ Demonstration Unit, thus providing the range of coal-derived syngas ratios (hydrogen to carbon monoxide) needed to meet the technical objectives of the demonstration project.

For descriptive purposes and for design and construction scheduling, the project has been divided into four major process areas with their associated equipment:

- *Reaction Area* - Syngas preparation and methanol synthesis reaction equipment.

- *Purification Area* - Product separation and purification equipment.
- *Catalyst Preparation Area* - Catalyst and slurry preparation and disposal equipment.
- *Storage/Utility Area* - Methanol product, slurry, and oil storage equipment.

The physical appearance of this facility closely resembles the adjacent Eastman process plants, including process equipment in steel structures.

- *Reaction Area*

The reaction area includes feed gas compressors, catalyst guard beds, the reactor, a steam drum, separators, heat exchangers, and pumps. The equipment is supported by a matrix of structural steel. The most salient feature is the reactor, since with supports, it is approximately 84-feet tall.

- *Purification Area*

The purification area features two distillation columns with supports; one is approximately 82-feet tall, and the other 97-feet tall. These vessels resemble the columns of the surrounding process areas. In addition to the columns, this area includes the associated reboilers, condensers, air coolers, separators, and pumps.

- *Catalyst Preparation Area*

The catalyst preparation area consists of a building with a roof and partial walls, in which the catalyst preparation vessels, slurry handling equipment, and spent slurry disposal equipment are housed. In addition, a hot oil utility system is included in the area.

- *Storage/Utility Area*

The storage/utility area includes two diked lot-tanks for methanol, two tanks for oil storage, a slurry holdup tank, a trailer loading/unloading area, and an underground oil/water separator. A vent stack for safety relief devices is located in this area.

C. Process Description

The LPMEOH™ Demonstration Unit is integrated with Eastman's coal gasification facility. A simplified process flow diagram is included in Appendix A. Syngas is introduced into the slurry reactor, which contains a slurry of liquid mineral oil with suspended solid particles of catalyst. The syngas dissolves through the mineral oil, contacts the catalyst, and reacts to form methanol. The heat of reaction is absorbed by the slurry and is removed from the slurry by steam coils. The methanol vapor leaves the reactor, is condensed to a liquid, sent to the distillation columns for removal of higher alcohols, water, and other impurities, and is then stored in the day tanks for sampling before being sent to Eastman's methanol storage. Most of the unreacted syngas is recycled back to the reactor with the syngas recycle compressor, improving cycle efficiency. The methanol will be used for downstream feedstocks and in off-site, product-use testing to determine its suitability as a transportation fuel and as a fuel for stationary applications in the power industry.

D. Results and Discussion

The project status is reported by task, covering those areas in which activity took place during the reporting period. Major accomplishments during this period are as follows:

D.1 Off-Site Testing (Product-Use Demonstration)

Discussion

The product-use test program, developed in 1992 to support the demonstration at the original Cool Water Gasification Facility site, became outdated due in large part to changes within the power and chemical industries. This original product test program under-represented new utility dispersed electric power developments, and possibly new mobile transport engine developments. The updated product-use test program attempts for broader market applications and for commercial fuels comparisons. The objective of the product-use test program is to demonstrate commercial market applications for the “as produced” methanol as a replacement fuel and as a fuel supplement. Fuel economics will be evaluated for the “as produced” methanol for use in municipal, industrial, and utility applications and as fuel supplements for gasoline, diesel, and natural gas. These fuel evaluations will be based on the U.S. energy market needs projected during the 1998 to 2018 time period when the LPMEOH™ technology is expected to be commercialized.

The product-use test program has been developed to enhance the early commercial acceptance of central clean coal technology processing facilities, coproducing electricity and methanol to meet the needs of the local community. One of the advantages of the LPMEOH™ Process for coproduction from coal-derived syngas is that the as-produced, stabilized (degassed) methanol product is of unusually high quality (e.g. less than 1 wt% water) which may be suitable for the premium fuel applications. When compared to conventional methanol synthesis processes, cost savings (10 to 15%) of several cents per gallon of methanol can be achieved in coproduction facilities, if the suitability of the stabilized product as a fuel can be demonstrated. The applications (for example, as a hydrogen source for fuel cells, and as a clean transportable, storable fuel for dispersed power) will require testing of the product to confirm its suitability. Chemical feedstock applications will also be tested as warranted.

A limited quantity (up to 400,000 gallons) of the methanol product as produced from the demonstration unit is being made available for product-use tests. Product-use tests are targeted for an approximate 18 to 30-month period, and commenced during the first year of demonstration operations. An initial inventory of approximately 12,000 gallons of stabilized methanol was produced at LPMEOH™ Demonstration Unit in February of 1998 to supply the needs of the product-use test program; due to the pre-1998 timing for certain tests, methanol was shipped from the inventory held at the LaPorte AFDU. Air Products, ARCADIS, Geraghty & Miller (formerly Acurex Environmental Corporation), and the DOE have worked together to select the projects to be included in the off-site, product-use test program.

Activity during this quarter

Eight sites involving a variety of product-use tests have been selected to participate in this task. In a letter to the DOE dated 31 July 1997, Air Products formally recommended that seven of the eight projects had been defined in sufficient detail so that final planning and implementation should begin. DOE accepted Air Products' recommendation to proceed with the seven projects in August of 1997. The sites and project titles are listed in Appendix B-1. The eighth project, involving the testing of a water/naphtha/methanol emulsion as a transportation fuel, was removed from the Product-Use Test Program during a review meeting between DOE, Air Products, and ARCADIS, Geraghty & Miller.

All of the remaining product-use test projects are at varying phases of project planning, equipment procurement, and execution; two projects have been completed. Methanol produced from carbon monoxide (CO)-rich syngas at the LaPorte AFDU has been shipped to three of the project sites. Appendix B-3 through B-5 contain summary reports from the approved active projects. Highlights from these reports include:

Acurex Flexible-Fuel Vehicle (FFV) - A final project report was submitted to Air Products. This update of the October 1998 draft final report includes a discussion of formaldehyde emissions from M85-fueled FFV's. The final report is included in Appendix B-2.

Stationary Turbine for Volatile Organic Carbon (VOC) Control - Additional cost share (potentially from the California Energy Commission or the Electric Power Research Institute) is required before the project can be initiated.

West Virginia University (WVU) Stationary Gas Turbine - Air Products recommended to DOE that a proposal which was submitted on 01 December 1998 to perform additional experiments and emissions testing on the gas turbine apparatus should be accepted. DOE accepted this recommendation during the reporting period. A project kick-off meeting between Air Products and members of the WVU staff is scheduled for 08 April 1999.

Aircraft Ground Equipment Emulsion - During the next reporting period, two of the generators will be placed in service using the methanol emulsion as the fuel. After this test, the equipment will be disassembled and inspected.

University of Florida Fuel Cell - Activities focused on the construction of a small-scale reformer test to compare the behavior of chemical-grade methanol with fuel-grade methanol from the LPMEOH™ Demonstration Project. Additional equipment which is used to activate the reformer catalyst is being assembled.

West Virginia University Tri-Boro Bus - The draft final report for this project was submitted to Air Products (no update in this reporting period).

Florida Institute of Technology Bus & Light Vehicle - The two light vehicles were operated a total of 2,982 miles during the reporting period. Problems with the fuel pumps on both vehicles were identified. Fuel-grade methanol from the LPMEOH™ Demonstration Project was used to operate the vehicles.

D.2 Commercialization Studies

Discussion

Several areas have been identified for development to support specific commercial design studies. These include: a) product purification options; b) feed gas impurity removal options; c) catalyst addition/withdrawal options; and d) plant design configuration options. Plant sizes in the range of 300 TPD to 1,800 TPD and plant design configurations for the range from 20% up to 70% syngas conversion will be considered. The Kingsport demonstration unit design and costs will be the basis for value engineering work to focus on specific cost reduction targets in developing the initial commercial plant designs.

The Process Economics Study - Outline has been prepared to provide guidance for the overall study work. The four part outline is included in Appendix C. This Outline addresses several needs for this Task 1.5.2 Commercialization Study:

a) to provide process design guidance for commercial plant designs.
b) to meet the Cooperative Agreement's technical objectives requirement for comparison with gas phase methanol technology. This preliminary assessment will help set demonstration operating goals, and identify the important market opportunities for the liquid phase technology.
c) to provide input to the Demonstration Test Plan (Task 2.3).
d) to provide input to the Off-Site Testing (Task 1.4) product-use test program.

Recent Activities

- Part One of the Outline - "Coproduction of Methanol" has been written for release as a Topical Report. The Topical Report entitled “Economic Analysis - LPMEOH™ Process as an Add-on to IGCC for Coproduction” was approved and issued for release on 18 February 1999.

- Part Two of the Outline - "Baseload Power and Methanol Coproduction", has been incorporated into the paper, "Fuel and Power Coproduction - The Liquid Phase Methanol (LPMEOH™) Process Demonstration at Kingsport", that was presented at the DOE's Fifth Annual Clean Coal Technology Conference in January of 1997.

- Part Three of the Outline - "Coproduction for Intermediate Electric Load Following", has been incorporated into the paper, "Dispatchable IGCC Facilities: Flexibility through Coproduction", that was presented at POWER-GEN EUROPE ’97 in June of 1997.

- Part Four of the Outline - "Methanol Fuel Applications", was used as the basis to update the product-use test program (Task 1.4).

D.3 DME Design Verification Testing

Discussion

The first decision milestone, on whether to continue with dimethyl ether (DME) Design Verification Testing (DVT), was targeted for 01 December 1996. This milestone was relaxed to July of 1997 to allow time for further development of the Liquid Phase Dimethyl Ether (LPDME™) catalyst system. DVT is required to provide additional data for engineering design and demonstration decision-making. The essential steps required for decision-making are: a) confirm catalyst activity and stability in the laboratory, b) develop engineering data in the laboratory, and c) confirm market(s), including fuels and chemical feedstocks. The DME Milestone Plan, showing the DVT work and the decision and implementation timing, is included in Appendix D.

Prior work in this task included a recommendation to continue with DME DVT and Market Economic Studies. Ongoing activity is focusing on Laboratory R&D.

DME DVT Recommendation

DOE issued a letter dated 31 July 1997 accepting Air Products' recommendation to continue with the design verification testing to coproduce DME with methanol, and to proceed with planning a design verification test run at the LaPorte AFDU. A copy of the recommendation (dated 30 June 1997) is included in Appendix D. The recommendation was based on the results of the Market Economic Studies and on the LPDME™ catalyst system R&D work, and is summarized in the following.

The Market Economic Studies show that the LPDME™ process should have a significant economic advantage for the coproduction of DME with methanol for local markets. The studies show that the market applications for DME are large. DME is an ultra clean diesel fuel; and an 80% DME mixture with methanol and water is now being developed and tested by others. DME is a key intermediate in a commercial syngas-to-gasoline process, and is being developed as an intermediate for other chemicals and fuels. An LPDME™ catalyst system with reasonable long-term activity and stability has been developed from the laboratory R&D work.

Based upon the potential size of the markets and the promise of the LPDME™ catalyst system, design verification planning for the LaPorte AFDU was recommended. A summary of the DME DVT recommendation (dated 30 June 1997) is:

- Planning for a DME DVT run at the LaPorte AFDU, in conjunction with other DOE Liquid Fuels Programs, should be initiated. Test plans, budgets, and a schedule for these LaPorte AFDU tests are under development. Up to $875,000 of Clean Coal Technology Program budget support from the LPMEOH™ Project budget could be made available to support a suitable LPDME™ test run at LaPorte.

- An implementation decision, made mutually by the DOE's Clean Coal Technology Program (DE-FC22-92PC90543) LPMEOH™ project participants, and by the DOE's Liquid Fuels Program (DE-FC22-95PC93052) project participants, will be made in order to finalize the schedule for testing at LaPorte.

LPDME™ is not applicable to hydrogen (H_2)-rich syngas; and it is unlikely that a substantive LPDME™ demonstration will be recommended for Kingsport. Therefore, a convincing case that the test-run on CO-rich syngas at LaPorte will lead to successful commercialization must be made, prior to approving the final test-run plan. The strategy for commercialization must present the technical logic to combine the results of the following two areas:

1) catalyst performance (productivity, selectivity, and life) for the LPDME™ catalyst system under CO-rich syngas from the design verification testing at the LaPorte AFDU; and

2) reactor performance (methanol catalyst activity and life, hydrodynamics, and heat transfer) from the LPMEOH™ Process Demonstration Unit at Kingsport.

The DME DVT recommendation summarizes the catalyst targets, experimental results, and the corresponding economics for a commercially successful LPDME™ catalyst.

Market Economic Studies

Work on the feasibility study for the coproduction of DME and methanol with electric power has been completed. The product DME would be used as a domestic liquid cooking fuel, to replace imported Liquid Petroleum Gas, for China and the Pacific Rim regions. The results are included in the DME recommendation in Appendix D.

Laboratory R&D

Initially, synthesis of DME concurrently with methanol in the same reactor was viewed as a way of overcoming the syngas conversion limitations imposed by equilibrium in the LPMEOH™ process. Higher syngas conversion would provide improved design flexibility for the coproduction of power and liquid fuels from an IGCC facility. The LPDME™ process concept seemed ideally suited for the slurry-based liquid phase technology, since the second reaction (methanol to DME) could be accomplished by adding a second catalyst with dehydration activity to the methanol-producing reactor. Initial research work determined that two catalysts, a methanol catalyst and an alumina-based dehydration catalyst, could be physically mixed in different proportions to control the yield of DME and of methanol in the mixed product. These two commercially available catalysts comprise the LPDME™ catalyst system. Previously, proof-of-concept runs, in the laboratory and at the AFDU, confirmed that a higher syngas conversion could be obtained when a mixture of DME and methanol is produced in the liquid phase reactor.

Subsequent catalyst activity-maintenance experiments have shown the catalyst system utilized in the proof-of-concept runs experienced relatively fast deactivation compared to the LPMEOH™ process catalyst system. Further studies of the LPDME™ catalyst deactivation phenomenon, initially undertaken under the DOE's Liquid Fuels Program (Contract No. DE-FC22-95PC93052), was continued under this Task 1.5.3 through Fiscal Year 1996, and is now again being continued under the DOE Liquid Fuels Program. This LPDME™ catalyst deactivation research has determined that an interaction between the methanol catalyst and the dehydration catalyst is the cause of the loss of activity. Parallel research efforts--a) to determine the nature of the interaction; and b) to test new dehydration catalysts--was undertaken. In late 1995, the stability of the LPDME™ catalyst system was greatly improved, to near that of an LPMEOH™ catalyst system, when a new aluminum-based (AB) dehydration catalyst was developed. This new AB catalyst development showed that modification of the LPDME™ catalyst system could lead to long life.

Summary of Laboratory Activity and Results

- Air Products performed laboratory autoclave tests of samples of the AB dehydration catalyst from the commercial catalyst manufacturer (Engelhard). The results to date have not been consistent, indicating that all issues related to catalyst scale-up have not been resolved. As a result, the decision was made within the DOE’s Liquid Fuels Program to delay the start of the AFDU design verification test. Changes to the commercial production procedure were made, and additional batches of dehydration catalyst were made and tested. These tests did not yield the desired catalyst aging characteristics.

- A set of experiments was performed on a commercially available dehydration catalyst to compare this material with the AB dehydration catalyst. These results showed that the desired catalyst life could be achieved with the commercially available dehydration catalyst at a 10-20% reduction in system productivity (primarily a reduction in the selectivity to DME).

- In continued tests with the commercially available dehydration catalyst, the LPDME™ catalyst system operated at the desired catalyst life at a total of 10 grams methanol and dehydration catalyst in the autoclave; this corresponds to a slurry concentration of 7 wt%. Experiments were performed at the 30 gram catalyst loading (18 wt%), which approaches the system limitation for the autoclave, in order to study the effects of slurry concentration (note that the trial at the LaPorte AFDU is expected to be performed at 40 to 45 wt% slurry concentration). The rate of decline in the LPDME™ catalyst activity at this higher loading was greater than results at the 10 gram condition. A test with the AB dehydration catalyst also exhibited higher deactivation at the higher slurry concentration than earlier results at the 10 gram catalyst loading.

- Two other sets of experiments were performed. Catalyst from the 30 gram experiment was recovered and operated in follow-up tests at the 10 gram loading; the catalyst life met the expectation for the LPDME™ catalyst system. A second test was performed with the LPMEOH™ catalyst alone at the 30 gram loading in order to determine a point for comparison; this catalyst also performed at the laboratory baseline for catalyst life. In all experiments at the 30 gram catalyst loading (for both the LPMEOH™ catalyst alone and the LPDME™ catalyst system), the performance appeared to be unsteady, possibly indicating that the test conditions were close to the physical limitation for the autoclave.

- A test of the LPDME™ catalyst system at a catalyst loading of 20 grams was performed; the calculated catalyst deactivation was approximately the same as the results at the 30 gram catalyst loading.

- Work is continuing in the laboratory autoclaves to identify the causes of catalyst deactivation at the higher slurry concentration. Activities in the laboratory are expected to be concluded by 01 July 1999, so that shakedown activities at the LaPorte AFDU can commence in September of 1999.

D.4 LPMEOH™ Process Demonstration Facility - Methanol Operation

Table D.4-1 contains the summary table of performance data for the LPMEOH™ Demonstration Unit during the reporting period. These data represent daily averages, typically from a 24-hour material balance period, and those days with less than 12 hours of stable operation are omitted. Appendix E contains samples of the detailed material balance reports which are representative of the operation of the LPMEOH™ Demonstration Unit during the reporting period.

During the reporting period, a total of 3,889,278 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Eastman accepted all of this methanol for use in the production of methyl acetate, and ultimately cellulose acetate and acetic acid. No environmental incidents were reported during this quarter.

The LPMEOH™ Demonstration Unit operated at 99% availability throughout the quarter. Two short unit shutdowns were experienced in early January and February of 1999; both were due to electrical transients. Appendix F, Table 1 contains the summary of outages for the LPMEOH™ Demonstration Unit during this quarter.

Table D.4-1. Data Summary for LPMEOH™ Demonstration Unit

Case	Date	Days Onstream	Gas Type	Temp (Deg C)	Pres. (psig)	Fresh Feed (KSCFH)	Recycle Gas (KSCFH)	Reactor Feed (H2:CO)	Purge Gas (KSCFH)	Inlet Sup. Velocity (ft/sec)	Space Velocity (l/hr-kg)	Slurry Conc. (wt% ox)	Gas Holdup (vol%)	Gassed Slurry Hgt (ft)	Catalyst Inventory (lb)	Catalyst Age (eta)	CO Conv. (%)	Reactor O-T-M Conv. (%)	Syngas Util. (SCF/lb)	Raw MeOH Production (TPD)	Catalyst MeOH Prod. (gmol/hr-kg)	Reactor Vol. Prod. (TPD/Cu ft)	U Overall (BTU/hr ft2 F)	Sparger dP (psi)	Sparger Resistance ("K")
10	1-Jan-99	377	Balanced	235	710	756	1,964	3.64	111.1	0.63	2842	45.6	29.9	58.0	56,200	0.27	39.1	22.0	43.4	209.1	9.82	0.086	176	15.36	19.56
10	2-Jan-99	378	Balanced	235	710	766	1,924	3.48	102.3	0.62	2819	46.1	31.2	58.0	56,200	0.28	39.1	22.6	42.7	215.2	10.10	0.088	179	15.50	19.88
10	3-Jan-99	379	Balanced	235	710	778	1,915	3.52	107.5	0.63	2835	47.6	31.8	55.5	56,200	0.28	39.5	22.8	42.8	218.4	10.23	0.094	184	15.16	19.77
10	4-Jan-99	380	Balanced	235	710	770	1,952	3.47	107.5	0.64	2867	48.6	34.3	55.5	56,200	0.28	38.8	22.3	42.9	215.7	10.24	0.092	180	15.53	19.68
10	5-Jan-99	381	Balanced	235	710	735	1,909	3.97	132.1	0.61	2767	49.0	35.4	55.5	56,200	0.25	39.4	21.0	44.7	197.5	9.18	0.085	165	14.01	20.78
10	6-Jan-99	382	Balanced	235	710	773	1,848	3.88	137.5	0.61	2745	47.5	31.6	55.5	56,200	0.27	41.1	22.2	44.7	207.4	9.67	0.089	186	13.97	20.97
10	7-Jan-99	383	Balanced	235	710	752	1,949	3.43	116.1	0.63	2837	47.2	30.9	55.5	56,200	0.25	36.6	21.6	43.6	207.1	9.66	0.089	172	16.52	21.02
10	8-Jan-99	384	Balanced	235	710	749	1,949	3.38	124.2	0.62	2822	45.9	29.4	57.0	56,200	0.25	35.9	21.3	44.1	203.7	9.51	0.085	167	16.30	20.67
10	9-Jan-99	385	Balanced	235	710	741	1,972	3.47	123.9	0.63	2851	45.6	27.3	56.0	56,200	0.24	35.5	20.9	44.0	202.0	9.40	0.086	170	16.06	20.79
10	10-Jan-99	386	Balanced	235	710	741	1,983	3.32	131.6	0.63	2865	46.7	30.8	56.5	56,200	0.24	33.9	20.7	44.4	200.3	9.31	0.084	165	16.61	20.71
10	11-Jan-99	387	Balanced	235	709	663	1,970	3.88	98.1	0.61	2766	47.0	27.1	53.0	56,200	0.24	37.3	19.8	43.0	184.8	8.71	0.083	190	15.50	21.00
10	12-Jan-99	388	Balanced	235	710	632	1,982	3.81	91.8	0.61	2743	46.5	24.9	52.5	56,200	0.22	35.6	19.2	42.5	178.3	8.37	0.081	175	14.95	21.21
10	13-Jan-99	389	Balanced	235	710	642	1,994	3.83	103.2	0.60	2722	46.4	28.2	55.0	56,200	0.22	35.6	19.1	43.8	175.8	8.23	0.076	169	15.29	21.86
10	15-Jan-99	391	Balanced	235	710	626	1,950	4.42	71.7	0.60	2605	45.8	28.4	58.5	58,400	0.23	41.5	20.0	41.9	179.4	8.10	0.073	168	14.27	21.19
10	16-Jan-99	392	Balanced	235	710	639	1,940	4.43	78.6	0.60	2604	44.8	29.3	61.5	58,400	0.24	43.7	20.5	43.3	184.8	8.53	0.073	182	13.58	20.37
10	18-Jan-99	394	Balanced	235	709	691	1,948	3.77	100.4	0.61	2659	44.5	28.5	61.5	58,400	0.23	37.7	20.5	43.7	189.9	8.56	0.073	164	15.22	20.49
10	19-Jan-99	395	Balanced	235	710	685	1,935	3.86	103.5	0.61	2636	44.8	29.2	61.5	58,400	0.23	38.3	20.5	43.6	188.8	8.50	0.073	161	14.74	20.60
10	20-Jan-99	396	Balanced	235	709	694	1,923	3.77	113.5	0.61	2635	44.5	26.7	60.0	58,400	0.23	37.6	20.5	44.2	188.5	8.49	0.075	166	14.92	20.57
10	29-Jan-99	405	Balanced	235	711	539	1,931	4.64	30.5	0.57	2397	42.9	24.7	64.5	60,600	0.22	42.0	19.3	39.7	163.2	7.02	0.060	171	14.27	22.25
10	30-Jan-99	406	Balanced	235	710	538	1,802	4.60	33.6	0.54	2275	45.1	23.7	58.5	60,600	0.22	43.7	20.2	39.7	162.7	7.00	0.066	171	14.21	24.50
10	1-Feb-99	408	Balanced	235	710	529	1,648	3.78	33.0	0.51	2118	45.4	25.0	59.0	60,600	0.21	38.8	21.1	38.9	163.4	7.02	0.066	171	12.18	25.25
10	3-Feb-99	410	Balanced	235	709	515	1,926	4.02	24.5	0.56	2356	46.1	25.8	58.0	60,600	0.21	36.4	18.6	38.7	159.9	6.87	0.066	160	13.84	23.37
10	5-Feb-99	412	Balanced	235	710	521	1,542	4.82	34.8	0.48	2013	46.4	21.7	54.5	60,600	0.22	46.8	20.9	40.9	152.8	6.58	0.067	179	9.93	24.54
10	6-Feb-99	413	Balanced	235	711	504	1,663	4.63	29.3	0.50	2111	46.0	23.3	56.5	60,600	0.21	44.7	20.5	39.5	153.1	6.59	0.064	182	11.26	22.69
10	8-Feb-99	415	Balanced	235	709	561	2,079	4.30	82.1	0.58	2439	47.1	22.2	53.5	60,600	0.18	36.3	17.7	43.4	155.2	6.68	0.069	148	14.59	21.86
10	9-Feb-99	416	Balanced	235	710	565	1,940	3.75	51.5	0.58	2447	47.3	24.2	54.5	60,600	0.20	35.4	19.1	40.3	168.4	7.24	0.073	164	15.65	21.89
10	10-Feb-99	417	Balanced	235	710	542	1,985	4.20	52.7	0.59	2456	47.4	22.6	53.0	60,600	0.19	36.1	17.7	41.2	158.1	6.80	0.071	172	14.59	21.86
10	11-Feb-99	418	Balanced	235	710	532	1,994	4.02	51.0	0.59	2455	48.6	21.4	50.0	60,600	0.18	34.4	17.4	41.2	154.8	6.67	0.074	172	15.12	21.72
10	12-Feb-99	419	Balanced	235	711	533	2,014	4.05	51.0	0.59	2488	48.7	22.5	50.5	60,600	0.19	33.8	17.1	41.1	155.4	6.69	0.073	180	14.87	21.70
10	13-Feb-99	420	Balanced	235	710	558	2,062	3.75	64.0	0.61	2566	48.4	24.4	52.5	60,600	0.18	30.9	16.8	42.3	159.2	6.85	0.072	164	15.19	21.28
10	14-Feb-99	421	Balanced	235	709	581	2,039	3.76	71.2	0.61	2560	48.7	24.6	52.0	60,600	0.18	31.5	17.0	42.9	162.3	6.98	0.074	173	15.06	21.74
10	15-Feb-99	422	Balanced	235	710	541	1,976	4.01	58.1	0.58	2451	48.3	24.2	52.5	60,600	0.18	33.4	17.0	41.9	154.9	6.67	0.070	173	14.99	23.65
10	16-Feb-99	423	Balanced	235	710	581	1,904	3.87	80.5	0.58	2415	47.8	22.2	52.0	60,600	0.18	33.9	18.0	43.5	160.3	6.90	0.073	172	14.81	23.46
11	17-Feb-99	424	Balanced	235	710	585	1,926	3.72	103.2	0.58	2579	48.1	25.7	51.0	57,303	0.18	31.3	17.2	45.6	153.8	7.01	0.072	165	15.43	23.16
11	18-Feb-99	425	Balanced	235	710	594	1,939	3.58	113.6	0.58	2744	47.5	24.7	48.5	53,951	0.19	30.1	17.0	46.3	153.7	7.43	0.076	179	15.43	23.26
11	19-Feb-99	426	Balanced	235	709	582	1,956	3.50	109.8	0.59	2773	48.5	25.2	47.0	53,951	0.19	28.7	16.6	46.4	151.3	7.32	0.077	174	15.76	23.36
11	20-Feb-99	427	Balanced	237	709	557	1,942	3.88	105.6	0.58	2904	46.6	25.8	48.0	50,676	0.19	30.6	16.3	45.7	146.9	7.56	0.073	175	14.49	23.40
11	21-Feb-99	428	Balanced	237	709	546	1,923	4.01	112.6	0.58	2870	46.7	25.5	47.5	50,676	0.18	30.3	15.8	46.9	140.6	7.24	0.071	168	14.23	24.21
11	23-Feb-99	430	Balanced	235	710	572	1,783	4.43	156.3	0.55	2775	45.8	25.5	49.0	50,676	0.19	31.6	15.3	52.0	132.6	6.83	0.064	177	12.04	23.71
11	28-Feb-99	435	Balanced	240	709	583	2,010	3.35	100.0	0.61	3008	44.9	26.4	51.5	50,676	0.18	28.8	17.2	44.4	157.4	8.10	0.073	168	16.20	21.26
10	16-Mar-99	451	Balanced	235	710	488	2,066	3.02	123.0	0.59	3019	39.8	31.1	65.5	49,988	0.14	19.3	12.6	50.3	116.5	6.09	0.042	190	3.17	4.23
10	17-Mar-99	452	Balanced	235	710	487	2,029	2.99	122.9	0.59	2985	40.6	30.5	63.0	49,988	0.14	19.6	12.9	49.5	118.1	6.17	0.044	193	3.15	4.29
10	18-Mar-99	453	Balanced	235	710	495	2,061	3.04	131.3	0.59	3008	41.1	31.4	62.5	49,988	0.14	19.2	12.4	51.7	115.0	6.01	0.044	193	3.20	4.33
10	19-Mar-99	454	Balanced	235	710	493	2,005	3.02	146.0	0.58	2955	41.0	29.8	61.5	49,988	0.13	19.1	12.4	53.0	111.7	5.91	0.043	185	3.07	4.29
10	21-Mar-99	456	Balanced	235	711	521	2,291	3.63	84.9	0.65	3163	42.0	30.6	62.5	52,188	0.17	24.5	13.9	44.9	139.2	6.96	0.053	176	3.56	4.19
10	22-Mar-99	457	Balanced	235	710	530	2,401	3.36	93.5	0.68	3296	43.1	30.1	59.5	52,188	0.17	22.4	13.5	45.1	141.0	7.05	0.056	180	3.96	4.14
10	23-Mar-99	458	Balanced	235	710	623	2,370	2.92	173.7	0.70	3390	44.6	30.5	56.5	52,188	0.17	20.2	13.5	51.3	145.9	7.30	0.061	175	4.33	4.07
10	24-Mar-99	459	Balanced	235	710	630	2,370	2.94	175.2	0.69	3381	44.6	30.4	56.5	52,188	0.17	20.5	13.7	51.3	147.3	7.37	0.062	187	4.39	4.15
10	25-Mar-99	460	Balanced	235	710	614	2,379	2.94	172.7	0.70	3387	45.6	31.9	55.5	52,188	0.16	20.0	13.3	51.3	143.7	7.19	0.062	171	4.34	4.12
10	26-Mar-99	461	Balanced	235	710	621	2,385	2.78	170.6	0.70	3397	43.5	31.8	60.0	52,188	0.16	19.5	13.5	50.9	146.3	7.32	0.058	182	4.39	4.02
10	27-Mar-99	462	Balanced	235	710	618	2,393	2.76	174.9	0.40	3421	43.5	31.9	60.0	52,188	0.16	18.9	13.1	51.7	143.6	7.19	0.057	184	4.56	4.13
10	28-Mar-99	463	Balanced	235	711	628	2,331	2.96	199.0	0.69	3348	44.7	30.2	56.0	52,188	0.15	19.5	12.9	54.5	138.1	6.92	0.059	178	4.26	4.14
10	29-Mar-99	464	Balanced	235	711	625	2,310	3.10	205.3	0.68	3323	44.9	27.5	53.5	52,188	0.15	19.9	12.8	55.4	135.3	6.78	0.060	181	4.09	4.15
10	30-Mar-99	465	Balanced	235	711	615	2,322	3.60	105.2	0.68	3187	44.6	28.7	57.5	54,388	0.19	27.4	15.5	45.6	161.8	7.81	0.067	160	4.04	4.18
10	31-Mar-99	466	Balanced	235	711	620	2,302	3.56	119.7	0.67	3153	45.3	26.2	54.0	54,388	0.19	27.3	15.6	46.0	161.7	7.79	0.071	174	3.98	4.19

LPMEOH™ Demonstration Unit Inspection - March 1999

The LPMEOH™ Demonstration Unit was shutdown on 02 March 1999 to begin a scheduled biannual inspection of all pressure vessels as required by Tennessee state code. Catalyst slurry was transferred to the slurry tank during this outage. No issues were observed with any of the equipment items evaluated as part of the code inspection. The walls and internal heat exchanger within the LPMEOH™ Reactor showed no evidence of erosion, pitting, or fouling by catalyst slurry. Inspection of the economizer heat exchanger, which condenses process oil from the reactor effluent stream, revealed a significant blockage of the heat exchanger tubes with catalyst slurry. This observation was expected based upon the loss of heat transfer performance of the economizer heat exchanger during the last few months of operation.

Maintenance activities were also completed during this outage. This work included the addition of an oil flush at the economizer heat exchanger, cleaning of all heat exchanger tubes, and re-routing of the piping between the cyclone and the reactor. This final maintenance item was designed to provide a second separate pathway to return the stream of entrained slurry from the cyclone to the reactor from the condensed oil stream (in the original design, this slurry was combined with the condensed oil from the secondary oil knock-out drum, and the combined stream is returned to the reactor).

Catalyst was pressure-transferred from the slurry tank to the reactor, and the LPMEOH™ Demonstration Unit was re-started on 14 March 1999. The economizer heat exchanger returned to performance levels expected for clean service. Condensed oil was gravity-drained from the secondary oil knock-out drum to the reactor without problems. However, the new piping from the cyclone to the reactor was not placed into service due to an obstruction that could not be cleared by flushing with clean oil. This entrained slurry is presently returned to the reactor via the original routing along with condensed oil from the secondary oil knock-out drum. During an upcoming complex-wide outage in April of 1999, this new section of piping from the cyclone will be cleared.

Catalyst Life (eta) - January - March 1999

The “age” of the methanol synthesis catalyst can be expressed in terms of a dimensionless variable eta (η), which is defined as the ratio of the rate constant at any time to the rate constant for freshly reduced catalyst (as determined in the laboratory autoclave). Appendix F, Figure 1 plots log η versus days onstream from the restart in December of 1997 through February 1999. Appendix F, Figure 2 plots log η versus days onstream from the restart in March 1999 to the end of the reporting period. Since catalyst activity typically follows a pattern of exponential decay, the plot of log η is fit to a series of straight lines, with step-changes whenever fresh catalyst was added to the reactor.

A ten-day period of stable operation ended on 04 January 1999. During this stable operating period, the rate of catalyst deactivation was 0.45% per day at a reactor temperature of 235°C. This result is comparable to the baseline deactivation rate from the 4-month proof-of-concept run at the LaPorte AFDU in 1988/89 (this run was performed at 250°C). During the quarter, there were other no stable operating periods of sufficient length (2 to 3 weeks) which are needed to quantify changes in catalyst activity with time.

During the quarter, the LPMEOH^{TM} Demonstration Unit has continued to perform with high slurry concentrations. Slurry concentrations greater than 48 wt% were achieved during this quarter with no apparent hydrodynamic instability. Three batches of catalyst were activated and added during the reporting period, bringing catalyst loading up to 151% of design. During late February 1999, three separate transfers of catalyst slurry from the reactor to the catalyst reduction vessel were conducted in order to dilute the slurry concentration in anticipation of the outage activities in March of 1999. These steps reduced the calculated slurry concentration in the reactor from over 48 wt% to approximately 40 wt%. No apparent negative impacts were experienced based on the catalyst removal from the reactor. The withdrawal process itself proceeded easily and without incident.

During the early part of the quarter, it was necessary to reduce the Balanced Gas flow from greater than 700 KSCFH to less than 600 KSCFH to compensate for higher sparger pressure drop. Reactor conditions were held at 235°C except for a brief production test in late February of 1999 when reactor temperature was increased to 240°C.

Analyses of catalyst samples for changes in physical characteristics and levels of poisons have continued. Appendix F, Table 2 summarizes the results to date. Samples have continued to show an increase in arsenic loading, with levels in excess of the concentrations measured on samples of the initial charge of methanol synthesis catalyst taken from the reactor at the end of the 1997 operating campaign. There appears to be a weak correlation between this increased arsenic loading and the rate of deactivation of the catalyst. A meeting was held at Kingsport on 5 January 1999 between Air Products, Eastman, and United Catalysts, Inc. to review the plant configuration and analytical results. A plan to add arsenic removal capacity to the guard bed system is expected to be executed in the May/June 1999 time-frame.

Sulfur continues to be measured above the analytical detection limit. Copper crystallite size measurements have shown an increase in the most recent samples, however, the size increase has stabilized over the last few samples. Other methods of crystallite size determination have corroborated the increased size measurements. Levels of iron and nickel have remained low and steady since the restart in December of 1997.

Sparger Resistance

During January and February of 1999, the sparger resistance coefficient across the LPMEOH™ Reactor continued to increase beyond the step-change experienced in October of 1999 (as reported in Technical Progress Report No. 18). This increased flow resistance had two consequences on operation of the LPMEOH™ Demonstration Unit during the reporting period. First, the Balanced Gas flowrate into the LPMEOH™ Demonstration Unit was limited due to higher pressure drop within the reactor synthesis loop. Second, condensed oil and entrained slurry could not be gravity-drained to the flush connection at the gas inlet line to the reactor. In order to overcome the pressure drop, the streams were batch-transferred to the catalyst reduction vessel and returned to the reactor via the slurry transfer pump. Appendix F, Figure 3 plots the average daily sparger resistance coefficient for the period of January and February 1999. The data for this plot, along with the corresponding average pressure drop, are included in Table D.4-1.

During the March 1999 scheduled outage, the reactor gas sparger was removed and inspected. It was estimated that the blockage in the sparger was consistent with the resistance coefficient which was calculated prior to shutdown. During this outage, a modified gas sparger was installed in the reactor; Air Products developed this design using the same principles as the original device. Upon restarting on 14 March 1999, the performance of the new sparger met the design expectations for pressure drop and reactor operation. Appendix F, Figure 4 plots the average daily sparger resistance coefficient for the period following the March 1999 outage. The data for this plot, along with the corresponding average pressure drop, are also included in Table D.4-1.

D.5 Planning and Administration

A Project Review Meeting was held in Pittsburgh, PA on 24-25 March 1999. The results of the unit operation and equipment inspection during the recent outage were reviewed, and an update on the status of the catalyst development for the LPDME™ proof-of-concept test at the LaPorte AFDU was provided. The meeting agenda, extracts from the meeting handouts, and the meeting notes are included in Appendix G.

The Milestone Schedule Status Report and the Cost Management Report, through the period ending 31 March 1999, are included in Appendix H. These two reports show the current schedule, the percentage completion and the latest cost forecast for each of the Work Breakdown Structure (WBS) tasks. Ninety-nine percent (99%) of the $38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 31 March 1999. Thirty-seven percent (37%) of the $158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 March 1999.

The monthly reports for January, February, and March were submitted. These reports include the Milestone Schedule Status Report, the Project Summary Report, and the Cost Management Report.

An abstract for the paper entitled “Commercial-Scale Demonstration of the Liquid Phase Methanol (LPMEOH™) Process: Operating Experience Update” was submitted. This paper will be presented at the Seventh Clean Coal Technology Conference in Knoxville, TN (21-24 June 1999). The LPMEOH™ Demonstration Unit will serve as the site for the plant tour which is associated with the Conference. Secretary of Energy Bill Richardson is expected to attend a reception following the tour.

A presentation entitled “Liquid Phase Methanol (LPMEOH™) Process Development” was given at the Spring Meeting of the American Chemical Society on 24 March 1999.

E. Planned Activities for the Next Quarter

- Continue to analyze catalyst slurry samples and reactor performance data to determine causes for deactivation of methanol synthesis catalyst.
- Continue executing Phase 3, Task 2.1 Methanol Operation per the Demonstration Test Plan. Focus activities on increasing catalyst activity and monitoring the performance of the gas sparger in the reactor.
- Prepare for and complete the replacement of the materials in the guard bed system.
- Resume preparations for a LPDME™ design verification test run at the LaPorte AFDU pending the completion of the testing of the dehydration catalyst. Conduct a review meeting on the status of catalyst development for the LPDME™ Process with the DOE's Liquid Fuels Program on 09 June 1999.
- Continue execution of the Off-Site, Product-Use Test Program (Phase 1, Task 1.4).
- Serve as host site for the tour in conjunction with the Clean Coal Technology Conference. Present the paper on the status of the operations at the LPMEOH™ Demonstration Unit at the Conference.

F. Conclusion

The LPMEOH™ Demonstration Unit operated at 99% availability throughout the reporting period. Two short unit shutdowns were experienced in early January and February of 1999; both were due to electrical transients.

The LPMEOH™ Demonstration Unit was shutdown on 02 March 1999 to begin a scheduled biannual inspection of all pressure vessels as required by Tennessee state code. Catalyst slurry was transferred to the slurry tank during this outage. No issues were observed with any of the equipment items evaluated as part of the code inspection. The walls and internal heat exchanger within the LPMEOH™ Reactor showed no evidence of erosion, pitting, or fouling by catalyst slurry. Inspection of the economizer heat exchanger, which condenses process oil from the reactor effluent stream, revealed a significant blockage of the heat exchanger tubes with catalyst slurry. This observation was expected based upon the loss of heat transfer performance of the economizer heat exchanger during the last few months of operation.

Maintenance activities were also completed during this outage. This work included the addition of an oil flush at the economizer heat exchanger, cleaning of all heat exchanger tubes, and re-routing of the piping between the cyclone and the reactor.

Catalyst was pressure-transferred from the slurry tank to the reactor, and the LPMEOH™ Demonstration Unit was re-started on 14 March 1999. The economizer heat exchanger returned to performance levels expected for clean service. Condensed oil was gravity-drained from the secondary oil knock-out drum to the reactor without problems. However, the new piping from the cyclone to the reactor was not placed into service due to an obstruction that could not be cleared by flushing with clean oil. This entrained slurry is presently returned to the reactor via the original routing along with condensed oil from the secondary oil knock-out drum. During an upcoming complex-wide outage in April of 1999, this new section of piping from the cyclone will be cleared.

Catalyst activity, as defined by the ratio of the rate constant at any point in time to the rate constant for freshly reduced catalyst (as determined in the laboratory autoclave), was monitored throughout the reporting period. A ten day period of stable operation ended on 04 January 1999. During this stable operating period, the rate of catalyst deactivation was 0.45% per day at a reactor temperature of 235°C. This result is comparable to the baseline deactivation rate from the 4-month proof-of-concept run at the LaPorte AFDU in 1988/89 (this run was performed at 250°C). During the quarter, there were other no other stable operating periods of sufficient length which are needed to quantify changes in catalyst activity with time.

During the quarter, the LPMEOH^{TM} Demonstration Unit has continued to perform with high slurry concentrations. Slurry concentrations greater than 48 wt% were achieved during this quarter with no apparent hydrodynamic instability. Three batches of catalyst were activated and added during the reporting period, bringing catalyst loading up to 151% of design. During late February 1999, three separate transfers of catalyst slurry from the reactor to the catalyst reduction vessel were conducted in order to dilute the slurry concentration in anticipation of the outage activities in March of 1999. These steps reduced the calculated slurry concentration in the reactor from over 48 wt% to approximately 40 wt%. No apparent negative impacts were experienced based on the catalyst removal from the reactor. The withdrawal process itself proceeded easily and without incident. Reactor conditions were held at 235°C except for a brief production test in late February of 1999 when reactor temperature was increased to 240°C.

Catalyst slurry samples from the LPMEOH™ Reactor have been taken on a regular basis to correlate any change in unit performance with changes in the physical properties of the catalyst. Samples have continued to show an increase in arsenic loading, with levels in excess of the concentrations measured on samples of the initial charge of methanol synthesis catalyst taken from the reactor at the end of the 1997 operating campaign. There appears to be a weak correlation between this increased arsenic loading and the rate of deactivation of the catalyst. A meeting was held at Kingsport on 5 January 1999 between Air Products, Eastman, and United Catalysts, Inc. to review the plant configuration and analytical results. A plan to add arsenic removal capacity to the guard bed system is expected to be executed in the May/June 1999 time-frame.

Sulfur continues to be measured above the analytical detection limit. Copper crystallite size measurements have shown an increase in the most recent samples, however, the size increase has stabilized over the last few samples. Other methods of crystallite size determination have corroborated the increased size measurements. Levels of iron and nickel have remained low and steady since the restart in December of 1997.

The performance of the alternative gas sparger, which was designed by Air Products and installed into the LPMEOH™ Reactor prior to the restart of the LPMEOH™ Demonstration Unit in December of 1997, was monitored. During January and February of 1999, the sparger resistance coefficient across the LPMEOH™ Reactor continued to increase beyond the step-change experienced in October of 1999 (as reported in Technical Progress Report No. 18). As a result of the greater sparger pressure drop, it was necessary to reduce the Balanced Gas flow from greater than 700 KSCFH to less than 600 KSCFH. Also, condensed oil and entrained slurry could not be gravity-drained to the flush connection at the gas inlet line to the reactor. In order to overcome the pressure drop, the streams were batch-transferred to the catalyst reduction vessel and returned to the reactor via the 29G-30 slurry transfer pump.

During the March 1999 scheduled outage, the reactor gas sparger was removed and inspected. It was estimated that the blockage in the sparger was consistent with the resistance coefficient which was calculated prior to shutdown. During this outage, a modified gas sparger was installed in the reactor; Air Products developed this design using the same principles as the original device. Upon restarting on 14 March 1999, the performance of the new sparger met the design expectations for pressure drop and reactor operation.

During the reporting period, a total of 3,889,278 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, over 34.6 million gallons of methanol has been produced. Eastman accepted all of this methanol for use in the production of methyl acetate, and ultimately cellulose acetate and acetic acid. No environmental incidents were reported during this quarter.

During this quarter, planning, procurement, and test operations continued on the seven project sites selected for the off-site, product-use test program. Air Products recommended to DOE that a proposal which was submitted on 01 December 1998 to perform additional experiments and emissions testing on the stationary gas turbine apparatus at West Virginia University should be accepted. DOE accepted this recommendation during the reporting period. A final report was issued on the results of operation of a flexible-fuel vehicle on fuel-grade methanol from the LPMEOH™ Demonstration Project. Construction of a reformer test apparatus to determine the operating characteristics of fuel-grade methanol as a feed to a fuel cell is near completion. Additional equipment which is used to activate the reformer catalyst is being assembled.

During the reporting period, planning for a design verification test run of the LPDME™ Process at the LaPorte AFDU continued. Air Products has been performing laboratory autoclave tests of samples of the dehydration catalyst from the commercial catalyst manufacturer (Engelhard). The results to date have not been consistent, indicating that all issues related to catalyst scale-up have not been resolved. A set of experiments was performed on a commercially available dehydration catalyst. These results showed that the desired catalyst life could be achieved at a 10-20% reduction in system productivity (primarily a reduction in the selectivity to DME). In continued tests with the commercially available dehydration catalyst, the LPDME™ catalyst system operated at the desired catalyst life at a total of 10 grams methanol and dehydration catalyst in the autoclave; this corresponds to a slurry concentration of 7 wt%. Experiments were performed at the 30 gram catalyst loading (18 wt%), which approaches the system limitation for the autoclave, in order to study the effects of slurry concentration (note that the trial at the LaPorte AFDU is expected to be performed at 40 to 45 wt% slurry concentration). The rate of decline in the LPDME™ catalyst activity at this higher loading was greater than results at the 10 gram condition. An experiment was performed with the LPMEOH™ catalyst alone at the 30 gram loading in order to determine a point for comparison; this catalyst performed at the laboratory baseline for catalyst life. Work is continuing in the laboratory autoclaves to identify the causes of catalyst deactivation at the higher slurry concentration. Activities in the laboratory are expected to be concluded by 01 July 1999, so that shakedown activities at the LaPorte AFDU can commence in September of 1999.

A Project Review Meeting was held in Pittsburgh, PA on 24-25 March 1999. The results of the unit operation and equipment inspection during the recent outage were reviewed, and an update on the status of the catalyst development for the LPDME™ design verification test at the LaPorte AFDU was provided.

The Topical Report entitled “Economic Analysis - LPMEOH™ Process as an Add-on to IGCC for Coproduction” was approved and issued for release on 18 February 1999.

An abstract for the paper entitled “Commercial-Scale Demonstration of the Liquid Phase Methanol (LPMEOH™) Process: Operating Experience Update” was submitted. This paper will be presented at the Seventh Clean Coal Technology Conference in Knoxville, TN (21-24 June 1999). The LPMEOH™ Demonstration Unit will serve as the site for the plant tour which is associated with the Conference. Secretary of Energy Bill Richardson is expected to attend a reception following the tour. A presentation entitled “Liquid Phase Methanol (LPMEOH™) Process Development” was given at the Spring Meeting of the American Chemical Society on 24 March 1999.

Ninety-nine percent (99%) of the $38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 31 March 1999. Thirty-seven percent (37%) of the $158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 March 1999.

APPENDICES

APPENDIX A - SIMPLIFIED PROCESS FLOW DIAGRAM

APPENDIX B - OFF-SITE TESTING (DEFINITION AND DESIGN)

Appendix B-1 - Summary Table of Seven Test Sites
Appendix B-2 - ARCADIS Geraghty & Miller FFV Final Report (thirty-one pages)

Quarterly Reports:

Appendix B-3 - ARCADIS Projects (two pages):
- **Acurex FFV**
- **Stationary Turbine for VOC Control**
- **Aircraft Ground Equipment Emulsion**

Appendix B-4 - University of Florida Fuel Cell (four pages)
Appendix B-5 - Florida Institute of Technology Bus & Light Vehicle (thirty pages)

APPENDIX C - PROCESS ECONOMIC STUDY

Process Economics Study - Outline
(Draft - 3/31/97 - four pages)

and

LPMEOH™ Process Economics - for IGCC Coproduction
(Memo - 31 March 1997 - two pages)

APPENDIX D - DME DESIGN VERIFICATION TESTING

APPENDIX E - SAMPLES OF DETAILED MATERIAL BALANCE REPORTS

APPENDIX F - RESULTS OF DEMONSTRATION PLANT OPERATION

Table 1
Summary of LPMEOH™ Demonstration Unit Outages - January/March 1999

Operation Start	Operation End	Operating Hours	Shutdown Hours	Reason for Shutdown
1/1/99 00:00	1/5/99 15:10	111.2	2.8	Electrical Transient
1/5/99 17:55	1/21/99 02:00	368.1	17.5	Syngas Outage
1/21/99 19:30	2/22/99 10:20	758.8	16.8	Electrical Transient
2/23/99 03:10	3/2/99 07:00	171.8	296.6	Shutdown for Outage
3/14/99 15:35	3/31/99 23:59	416.4		

Total Operating Hours	1826.3
Syngas Available Hours	1845.9
Plant Availability, %	**98.94**

Table 2
Summary of Catalyst Samples - Second Catalyst Batch

Sample	Identity	XRD		BET	Analytical (ppmw)				
		Cu	ZnO	m2/g	Fe	Ni	S	As	Cl
K9804-1	Reduction Sample 4/2/98 - Alternative Catalyst	72.5	84.9	105	23	11	<=110	<=12	
K9712-1	Transfer sample from 29D-02 to Reactor	95.3	74		362	47.2	66.7	10.2	nd
K9712-2	Reactor Sample Day 1	100	123.8	75	92.1	<=18	<=167	<50	nd
K9712-3	Reactor Sample Day 4	130.9	64						
K9712-4	Reactor Sample Day 10	126.8	73.3	73	126	<=22	<=127	<50	nd
K9801-2	Reactor Sample 1/26/98	132.05	98.3		63.5	39.5	42.7	29.2	<100
K9802-1	Reactor Sample 2/3/98	141.1	91.5						
K9802-2	Reactor Sample 2/9/98	158.1	113						
K9802-3	Reactor Sample 2/15/98	145.7	91		67.1	36	<=97	209	
K9802-4	Reactor Sample 2/23/98	176.8	114.5						
K9803-2	Reactor Sample 3/10/1998	154.3	95.8	44	61.4	35.8	<=94	408	
K9803-4	Reactor Sample 3/29/98	169.6	87.9						
K9804-2	Reactor Sample 4/14/98	152.4	89.3		81.7	30.8	<=170	615	
K9805-2	Reactor Sample 5/11/98	219.2	109.6		73.15	35.85	163	538	
K9606-2	Reactor Sample 6/16/98	272.3	117.2		86.4	31.1	220	1110	
K9807-2	Reactor Sample 7/8/98	263.2	108.6		88.7	27.6	277	1045	
K9807-3	Reactor Sample 7/29/98	412*	112		93.25	30.95	209	1620	
K9807-4	Reactor Sample 8/14/98	353.9*	124		121.5	37.1	213.5	1215	
K9809-1	Reactor Sample 9/24/98	347.4	129.8		69.6	29.8	326	1149	
K9810-1	Reactor Sample 10/5/98	331.1	130.4						
K9811-2	Reactor Sample 11/25/98	293.9			57.3	23.4	264	1400	<100
K9812-1	Reactor Sample 12/29/98	283.1			72.3	20.4	260	1300	<100
K9901-1	Reactor Sample 1/15/99	252.5		61.4					
K9902-1	Reactor Sample 2/17/99	474.7		133.6	82.6	22.2	385	1490	<300

Figure 1

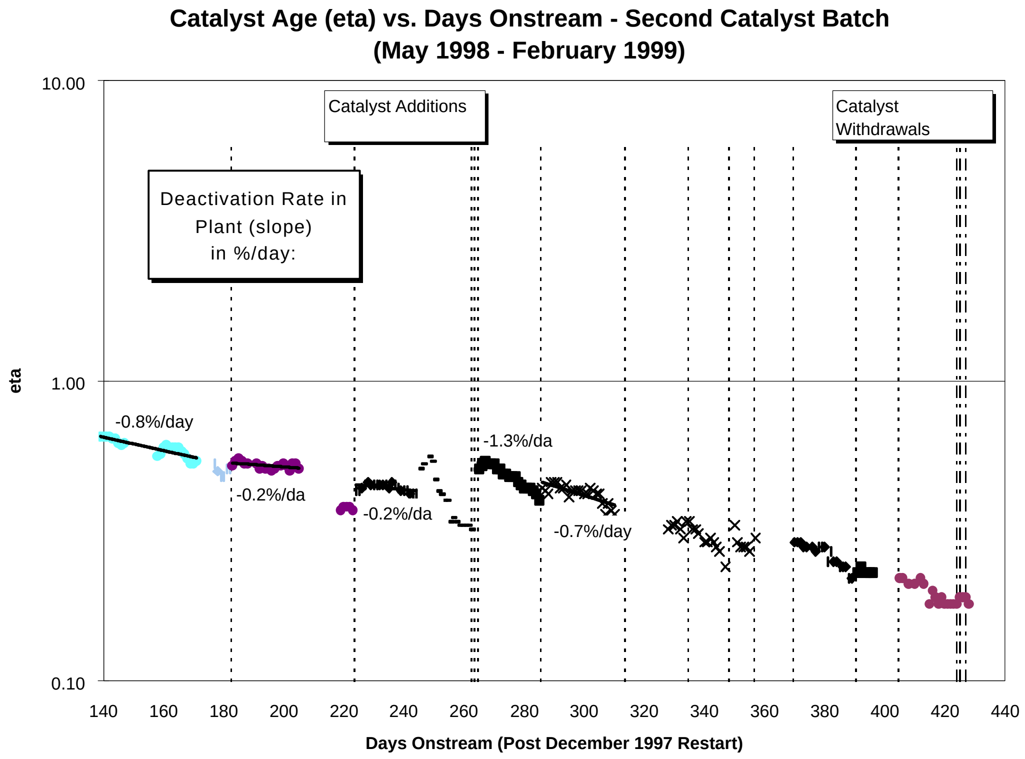

Catalyst Age (eta) vs. Days Onstream - Second Catalyst Batch
(May 1998 - February 1999)
10.00
1.00
0.10
eta
140
160
180
200
220
240
260
280
300
320
340
360
380
400
420
440
Days Onstream (Post December 1997 Restart)
Catalyst Additions
Catalyst Withdrawals
Deactivation Rate in Plant (slope) in %/day:
-0.8%/day
-0.2%/da
-0.2%/da
-1.3%/da
-0.7%/day

Figure 2

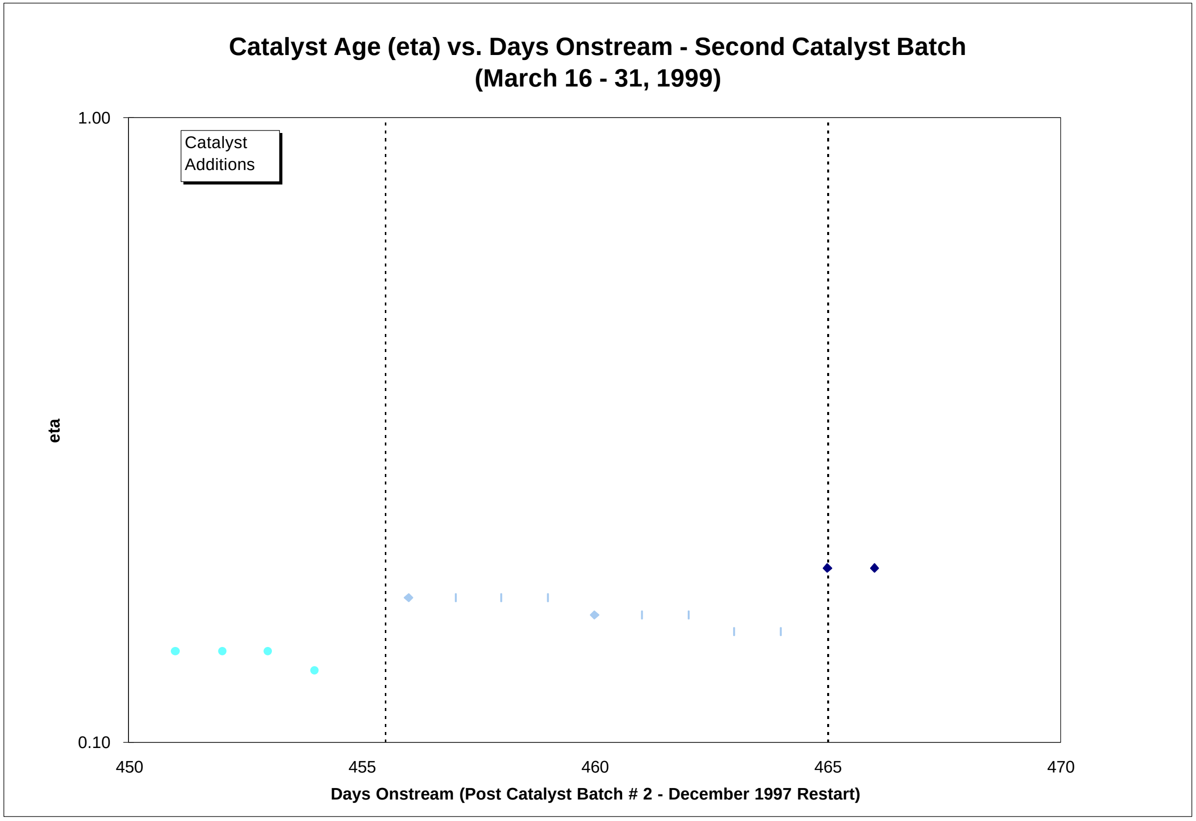
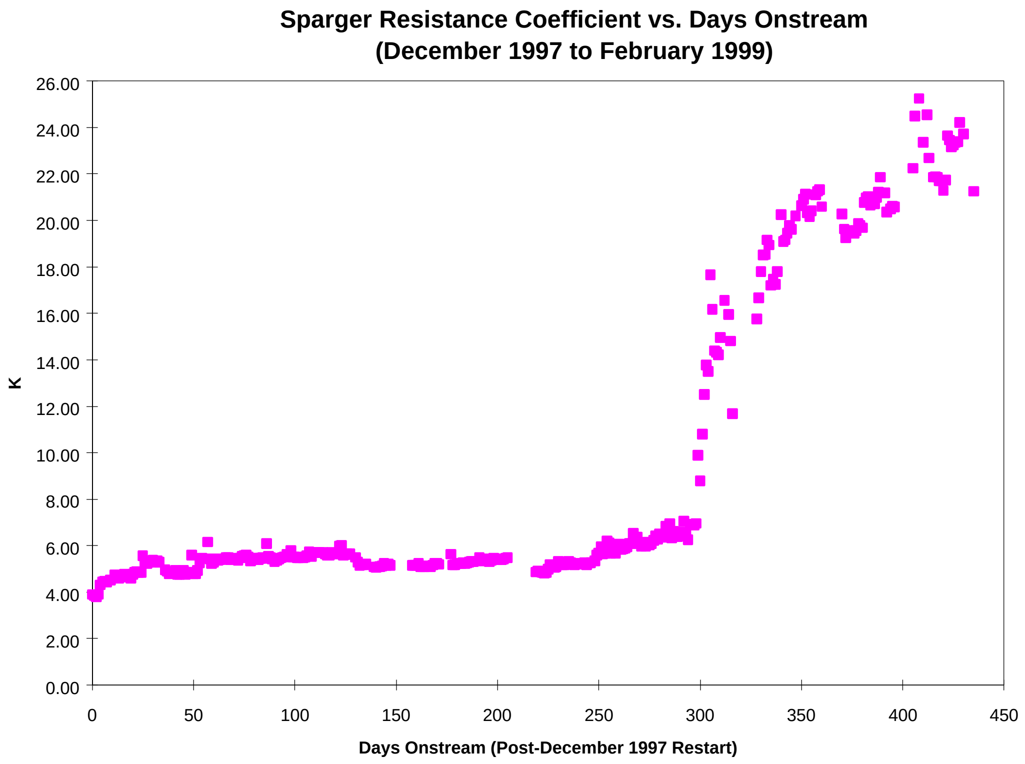
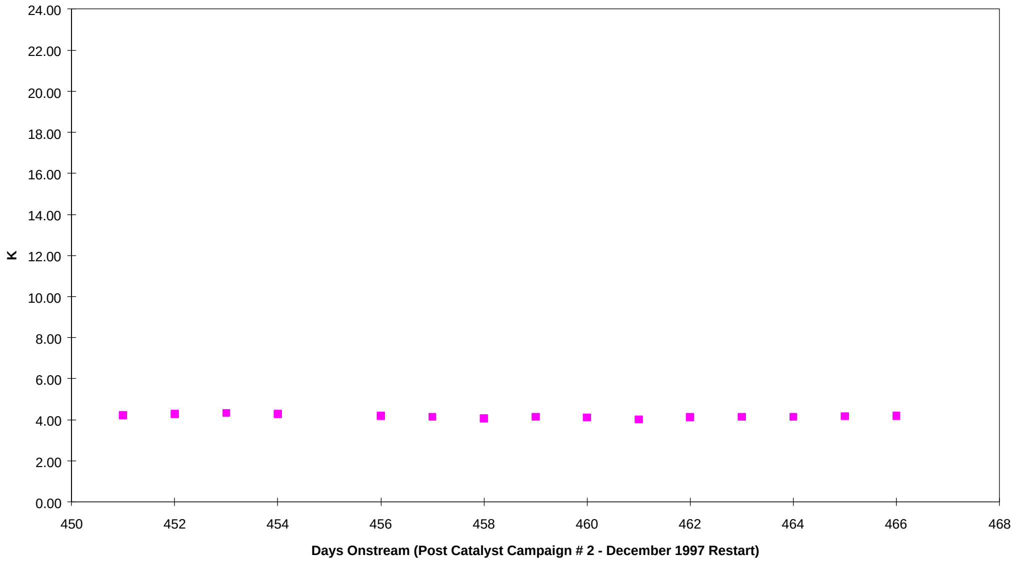

Catalyst Age (eta) vs. Days Onstream - Second Catalyst Batch
(March 16 - 31, 1999)
1.00
0.10
eta
Catalyst Additions
450
455
460
465
470
Days Onstream (Post Catalyst Batch # 2 - December 1997 Restart)

Figure 3



Sparger Resistance Coefficient vs. Days Onstream
(December 1997 to February 1999)

Figure 4



Sparger Resistance Coefficient vs. Days Onstream (March 1999)

APPENDIX G - PROJECT REVIEW MEETING (24-25 MARCH 1999)

APPENDIX H - MILESTONE SCHEDULE STATUS AND COST MANAGEMENT REPORTS