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

TECHNICAL PROGRESS REPORT NO. 21

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for the
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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 100% availability throughout the quarter. Three short duration syngas outages were the only operating interruptions experienced.

Several series of catalyst additions and withdrawals were conducted during this quarter to maintain reactor productivity. At the end of the reporting period, the total catalyst inventory was calculated to be 46,488 pounds. Due to the extensive catalyst withdrawal and addition campaigns, there were no extended periods of operation (2 to 3 weeks) which are needed to track catalyst deactivation.

During the quarter, the flowrate of the primary syngas feed (Balanced Gas) averaged 507 KSCFH through 19 September 1999. On 20 September 1999, Balanced Gas flow was increased to 800 KSCFH and held at that rate for the remainder of the reporting period. Reactor temperature was held at 235°C. Reactor pressure was limited to 690-695 psig early in the quarter due to high pressure drop across the 29C-40 carbonyl guard bed (see below).

During the reporting period, the pressure drop across the carbonyl guard bed, which had been recharged with both arsine and carbonyl removal materials in June of 1999, continued to gradually increase. By the end of July of 1999, the pressure drop was measured at over 20 psi; the expected pressure drop was less than 1 psi. During a vessel inspection on 12 August 1999, changes to the bottom support screen and its support were made. When the vessel was returned to service, the pressure drop across the guard bed was 0.5 psi. Reactor pressure was raised to 700 psig after the successful guard bed investigation.

During a routine sampling procedure involving the transfer of catalyst slurry between the reactor and the catalyst activation vessel on 27 July 1999, a leak developed in a gasketed connection on a pressure transmitter in the interconnecting piping circuit. A portion of the contents of the reactor spilled onto the concrete floor within the catalyst reduction building, and was contained within the floor drains and the oil-water separator. There were no injuries as a result of this incident. The cause of the failure was found to be the gasket material for the pressure transmitter connection tap. It was fully corrected by using an alternative gasket material for the service.

Sulfur continues to be measured on the catalyst above the analytical detection limit, and may be adversely impacting catalyst life. Copper crystallite size measurements have shown an

increase in the most recent samples. 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 March of 1999, has continued to meet the expectations for pressure drop and reactor operation.

During the reporting period, a total of 4,391,257 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, about 42.8 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.

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 that a gas turbine project (under development by ARCADIS Geraghty & Miller) should be redirected from VOC control to NO_x control. Results from testing of stabilized methanol in a reformer test apparatus at the University of Florida have shown that the reformer catalyst deactivated quickly. A proposal to perform additional testing of stabilized methanol in the apparatus in order to qualify this material as feedstock to a phosphoric acid fuel cell was recommended to, and accepted by, DOE.

During the reporting period, planning for a design verification test run of the Liquid Phase Dimethyl Ether (LPDME) Process at the LaPorte Alternative Fuels Development Unit (AFDU) continued. DOE accepted Air Products' recommendation to proceed with the interim campaign at the LaPorte AFDU.

A commercially available dehydration material was qualified for the LPDME design verification test. The methanol synthesis and dehydration catalysts were ordered and shipped to LaPorte. An autoclave test of these materials was successfully performed; this verified the suitability of the catalysts for use during the design verification testing.

In anticipation of an October 1999 startup of the LPDME design verification test at the LaPorte AFDU, checkout of mechanical and instrumentation systems was initiated. The date for catalyst loading at the LaPorte AFDU is scheduled for 09 October 1999.

A Project Review Meeting was held in Pittsburgh, PA, on 15-16 September 1999. The results of the unit operation were reviewed, and the plans for the upcoming design verification test of the LPDME Process at the LaPorte AFDU were reviewed and approved.

The topical report entitled "Alternative Fuels Field Test Unit Support to Kingsport LPMEOH™ Demonstration Unit - December 1997 - January 1998" was approved and issued. Comments were received from DOE on the draft of Volume 1 - Public Design, of the Final Report for the LPMEOH™ Demonstration Project, and a revised version was sent to DOE for review.

A presentation entitled “Liquid Phase Methanol (LPMEOH™) Project: Operating Experience Update” was given at the 1999 DOE Fossil Energy Fuels Program Review (21-23 September 1999). This was based upon the paper of the same title, which was submitted for presentation at the 1999 Gasification Technologies Conference (17-20 October 1999).

A paper entitled “Direct Applications of Stabilized Methanol from the Liquid Phase Methanol (LPMEOH™) Process” was submitted for presentation at the 16th Annual International Pittsburgh Coal Conference (11-15 October 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 30 September 1999. Forty-six percent (46%) of the $158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 September 1999.

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ACRONYMS AND DEFINITIONS

Acurex	-	Acurex Environmental Corporation (now ARCADIS Geraghty & Miller)
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-NETL	-	The DOE's National Energy Technology Laboratory (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
ρ	-	density, pounds per cubic foot

ACRONYMS AND DEFINITIONS (cont'd)

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 proof-of-concept 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 100% availability throughout the quarter. Three short duration syngas outages were the only operating interruptions experienced.

Several catalyst additions and withdrawals were conducted during this quarter to maintain reactor productivity. A series of three catalyst batch withdrawals were conducted on 12-13 July 1999. A total of approximately 7,936 pounds of catalyst was removed with these three batches. A fresh batch of catalyst was then added on 19 July 1999.

On 14 August 1999, a batch of catalyst was reduced to approximately 60% of the theoretical maximum uptake of syngas. It was determined that the reduction problems experienced with this batch were related to high catalyst solids concentration of the catalyst slurry prior to the start of reduction. Although not completely activated, this batch of catalyst was pumped to the reactor on 14 August 1999, and the batch appeared to have some activity for the production of methanol. Another batch of catalyst was fully activated and added to the reactor on 20 August 1999.

A major catalyst withdrawal and addition campaign was undertaken during late August and early September of 1999. A series of four withdrawals were conducted between 30 August and 01 September 1999. This was followed by four catalyst additions which were activated and added between 03 September and 11 September 1999. After the addition of the final batch of catalyst, the total catalyst inventory was calculated to be 46,488 pounds. Due to the extensive catalyst withdrawal and addition campaigns, there were no extended periods of operation (2 to 3 weeks) which are needed to track catalyst deactivation.

During the quarter, the flowrate of the primary syngas feed (Balanced Gas) averaged 507 KSCFH through 19 September 1999. On 20 September 1999, Balanced Gas flow was increased to 800 KSCFH and held at that rate for the remainder of the reporting period. Reactor temperature was held at 235°C. Reactor pressure was limited to 690-695 psig early in the quarter due to high pressure drop across the 29C-40 carbonyl guard bed (see below).

During the reporting period, the pressure drop across the carbonyl guard bed, which had been recharged with both arsine and carbonyl removal materials in June of 1999, continued to gradually increase. By the end of July of 1999, the pressure drop was measured at over 20 psi; the expected pressure drop was less than 1 psi. On 12 August 1999, the carbonyl guard bed was emptied and inspected; the bottom support screen was found to be damaged, and some adsorbent had blinded a small portion of the bottom of the screen. After changes to the screen and its support were made, the guard bed was returned to service, and the pressure drop was 0.5 psi. Reactor pressure was raised to 700 psig after the successful guard bed investigation.

During a routine sampling procedure involving the transfer of catalyst slurry between the reactor and the catalyst activation vessel on 27 July 1999, a leak developed in a gasketed connection on a pressure transmitter in the interconnecting piping circuit. A portion of the contents of the reactor spilled onto the concrete floor within the catalyst reduction building, and was contained within the floor drains and the oil-water separator. This corresponds to a loss of approximately 5,558 pounds of catalyst from the system. There were no injuries as a result of this incident. The cause of the failure was found to be the gasket material for the pressure transmitter connection tap. It was fully corrected by using an alternative gasket material for the service.

Analyses of catalyst samples for changes in physical characteristics and levels of poisons have continued. Sulfur continues to be measured on the catalyst above the analytical detection limit, and may be adversely impacting catalyst life. Copper crystallite size measurements have shown an increase in the most recent samples. 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 March of 1999, was monitored. The performance to date of the new sparger has met the design expectations for pressure drop and reactor operation.

During the reporting period, a total of 4,391,257 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, about 42.8 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.

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 that a gas turbine project (under development by ARCADIS Geraghty & Miller) should be redirected from VOC control to NO_x control. Results from testing of stabilized methanol in a reformer test apparatus at the University of Florida have shown that the reformer catalyst deactivated quickly. A proposal to perform additional testing of stabilized methanol in the apparatus in order to qualify this material as feedstock to a phosphoric acid fuel cell was recommended to, and accepted by, DOE. At West Virginia University, four axially spaced thermocouples will be installed inside the combustion chamber of the stationary gas turbine. This will be used in future computer modeling work on the turbine. DOE accepted Air Products' recommendation to provide stabilized methanol from the LPMEOH™ Process Demonstration Facility for use as part a new contract between the Institute and the Florida Energy Office.

During the reporting period, planning for a design verification test run of the Liquid Phase Dimethyl Ether (LPDME) Process at the LaPorte AFDU continued. At a review meeting for the DOE's Liquid Fuels Program on 09 June 1999, the participants agreed that the next test for the LPDME Process at the LaPorte AFDU should be treated as an interim campaign, with the primary objective being the determination of a tie-point between catalyst performance in the autoclave and the proof-of-concept scale. DOE accepted Air Products' recommendation to proceed with the interim campaign at the LaPorte AFDU.

During the reporting period, a commercially available dehydration material was qualified for the LPDME design verification test. The methanol synthesis and dehydration catalysts were ordered and shipped to LaPorte. An autoclave test of these materials was successfully performed; this verified the suitability of the catalysts for use during the design verification testing.

In anticipation of an October 1999 startup of the LPDME design verification test at the LaPorte AFDU, checkout of mechanical and instrumentation systems was initiated. A hot function test on syngas (in the absence of catalyst) is planned for the week of 04 October 1999. The date for catalyst loading at the LaPorte AFDU is scheduled for 09 October 1999.

A Project Review Meeting was held in Pittsburgh, PA, on 15-16 September 1999. The results of the unit operation were reviewed, and the plans for the upcoming design verification test of the LPDME Process at the LaPorte AFDU were reviewed and approved.

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A. Introduction

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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 10,000 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 the 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

During an evaluation period, eight sites involving a variety of product-use tests were 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, which involved 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. Appendices B-2 through B-5 contain summary reports from the approved active projects. Highlights from these reports include:

ARCADIS Geraghty & Miller Flexible-Fuel Vehicle (FFV) - The draft final report for this project was submitted to Air Products (no update in this reporting period).

Stationary Turbine for Volatile Organic Carbon (VOC) Control - In Technical Progress Report No. 20, it was reported that Air Products had received a recommendation from ARCADIS Geraghty & Miller that this project should be redirected from VOC control to NO_x control in order to receive the necessary financial support from the California Energy Commission. Air Products agreed with this assessment, and forwarded this recommendation to DOE. DOE has agreed with this recommendation, and a revised Statement of Work was developed. The test stand at Alzeta Corporation was prepared for use in testing stabilized methanol in this application.

West Virginia University (WVU) Stationary Gas Turbine - Pratt and Whitney Aircraft has agreed to install four axially spaced thermocouples inside the combustion chamber of the gas turbine. This will be used in future computer modeling work on the turbine. Additional testing of fuel-grade methanol from the LPMEOH™ Demonstration Project is expected to commence during the next reporting period.

Aircraft Ground Equipment Emulsion - Testing of stabilized methanol as an emulsion fuel in a 110 horsepower flight line generator at Tyndall Air Force Base, Florida was suspended due to the loss of funding from other sources. A final report on the initial test results will be prepared.

University of Florida Fuel Cell - The test of stabilized methanol from the LPMEOH™ Demonstration Project in a reformer test apparatus was completed. This test was intended to identify if trace components in the stabilized methanol might impact the energy input to the reformer and the quality of the reformate to the fuel cell. Results to date have shown that the reformer catalyst deactivated quickly, likely due to the presence of higher alcohols and mineral oil in the stabilized methanol. A proposal was received from the University of Florida to perform additional testing of stabilized methanol in a reformer test apparatus in order to qualify this material as feedstock to a phosphoric acid fuel cell. DOE accepted Air Products' recommendation to approve this 10-month program extension.

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 final report on testing of stabilized methanol as a transportation fuel at the Florida Institute of Technology was received. DOE accepted Air Products' recommendation to provide stabilized methanol from the LPMEOH™ Process Demonstration Facility for use as part a new contract between the Institute and the Florida Energy Office. Air Products will receive copies of the reports which are submitted to the state of Florida.

D.2 DME Design Verification Testing

<u>Discussion</u>

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.

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 and preparations for the design verification test run at the LaPorte AFDU.

1997 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 C-1. 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 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 1997 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 1997 DME recommendation in Appendix C-1.

Laboratory R&D - Background

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 Activities during Reporting Period

At a review meeting for the DOE's Liquid Fuels Program on 09 June 1999, members of the LPMEOH™ Project Team from Air Products and DOE were given an update on the activities regarding the status of catalyst development and the economics for the LPDME Process. The participants agreed that the next test for the LPDME Process at the LaPorte AFDU should be treated as an interim campaign, with the primary objective being the determination of a tie-point between catalyst performance in the autoclave and the proof-of-concept scale.

Following this meeting, a formal recommendation to proceed with the interim campaign at the LaPorte AFDU was issued by Air Products to DOE. A copy of this letter (dated 06 August 1999) is included in Appendix C-2. The objectives for this campaign, as stated in this recommendation, are:

1) Determine commercial viability of the LPDME Process on a 10 TPD scale, using commercially produced catalysts.
2) Obtain information to correlate scale-up of catalyst aging from the laboratory autoclave to the slurry bubble column.
3) Conduct process variable testing at conditions of potential commercial interest.
4) Perform experiments to better understand the hydrodynamics of the slurry bubble column.

The operating plan for this interim test at the LaPorte AFDU is provided in Table D.2-1. Run time is scheduled to study catalyst life and to perform process variable scans on other operating conditions which are of potential commercial interest. Most of the operating conditions during the plant trial will be performed with Shell-type syngas ($H_2/CO = 0.5$); this composition was used for the majority of the tests in the laboratory autoclaves. DOE issued a letter dated 10 August 1999 accepting accepted Air Products' recommendation to proceed with DME DVT activities at the LaPorte AFDU.

During the reporting period, a commercially available dehydration material was qualified for the LPDME design verification test. The methanol synthesis and dehydration catalysts were ordered and shipped to LaPorte. Samples from the site were shipped to Air Products'

TABLE D.2-1

RUN PLAN
LPDME RUN AT THE LAPORTE AFDU - OCTOBER/NOVEMBER 1999

Run No.	No. of Days	Comment	Gas Type	Reactor Pressure psia	Reactor Temp. deg F	Space Vel. sl/kg-hr	React. Fd. lbmol/hr	Inlet Sup. Vel. ft/sec	Slurry wt% oxide
MEOH + DEHYDRATION CATALYST (% MEOH CAT = 95%)									
	1	Catalyst Loading							
AF-A13	1	Reduction	4% H2 in N2	67		793	32.3	0.62	35
AF-R17.1	18	Life Study + Tracer1	Shell	765	482	6000	247	0.56	35
AF-R17.2	1.5	Low Vel. w/ MeOH Inj.	Shell	765	482	3100	128	0.29	35
AF-R17.3	1.5	Stoch. Feed + MeOH Inj.	1:1 H2/CO	1000	482	3250	134	0.23	35
AF-R17.4	2.5	High Velocity + Tracer2	Shell	765	482	8000	330	0.74	35
AF-R17.5	1.5	High Concentration	Shell	765	482	8000	330	0.74	40
TOTAL	27								

laboratories and were tested in the laboratory autoclave. A simulation of the operating conditions from the run plan (as described in Table D.2-1) was successfully performed; this verified the suitability of the catalysts for use during the design verification testing.

The material which will be used as part of the hydrodynamics tests during the operation at the LaPorte AFDU was prepared. The particle size distribution of the tracer material is almost identical to that of the dehydration catalyst which will be used during the trial.

In anticipation of an October 1999 startup of the LPDME design verification test at the LaPorte AFDU, checkout of mechanical and instrumentation systems was initiated. A review of potential hazards associated with this campaign was performed. Operator training was completed during the week of 22 September 1999, and calibration of the on-site gas chromatographs was initiated. Once a function-test of critical safety circuits is completed, a hot function test on syngas (in the absence of catalyst) is planned for the week of 04 October 1999. The start of catalyst activation and execution of the test plan in Table D.2-1 is scheduled to begin on 09 October 1999.

D.3 LPMEOH™ Process Demonstration Facility - Methanol Operation

Table D.3-1 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 D 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 4,391,257 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.

The LPMEOH™ Demonstration Unit operated at 100% availability throughout the quarter. Three short duration syngas outages were the only operating interruptions experienced. Appendix E, Table 1 contains the summary of outages for the LPMEOH™ Demonstration Unit during this quarter.

<u>Catalyst Life (eta) - July - September 1999</u>

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 E, Figure 1 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.

Table D.3-1. 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")
11	1-Jul-99	558	Balanced	234	694	536	2,059	3.25	74.2	0.61	2763	46.9	24.6	50.5	55,274	0.182	26.6	16.1	42.8	150.3	7.10	0.071	202	3.64	4.50
11	2-Jul-99	559	Balanced	234	692	550	2,050	3.12	86.9	0.62	2771	47.0	27.1	52.0	55,274	0.182	25.5	16.0	44.0	150.1	7.10	0.069	198	3.68	4.51
11	4-Jul-99	561	Balanced	235	692	497	2,070	3.21	57.7	0.61	2729	46.6	24.7	51.0	55,274	0.176	25.5	15.5	41.4	144.2	6.81	0.067	193	3.56	4.55
11	5-Jul-99	562	Balanced	234	690	527	2,064	2.90	72.3	0.62	2754	47.0	25.8	51.0	55,274	0.178	23.9	15.8	42.7	148.1	7.01	0.069	194	3.77	4.54
11	8-Jul-99	565	Balanced	235	689	536	2,036	3.37	93.3	0.61	2744	46.1	26.8	53.5	55,274	0.180	26.1	15.4	44.3	145.1	6.87	0.065	190	3.44	4.60
11	9-Jul-99	566	Balanced	235	689	536	2,037	3.10	95.5	0.62	2747	46.7	26.3	52.0	55,274	0.177	24.2	15.2	45.1	142.7	6.76	0.065	189	3.56	4.56
11	10-Jul-99	567	Balanced	235	690	543	2,066	3.01	99.6	0.62	2782	47.0	27.2	52.0	55,274	0.180	23.4	15.1	45.5	143.2	6.77	0.066	192	3.61	4.49
11	11-Jul-99	568	Balanced	234	691	528	2,068	2.99	92.0	0.62	2771	47.4	26.0	50.5	55,274	0.172	23.2	15.1	44.4	142.7	6.75	0.067	198	3.60	4.53
11	13-Jul-99	570	Balanced	235	691	462	2,145	2.55	85.2	0.62	3256	46.8	29.7	46.5	47,338	0.165	17.7	12.9	44.9	123.5	6.82	0.063	199	3.91	4.48
11	14-Jul-99	571	Balanced	234	690	452	2,123	2.63	90.9	0.62	3223	48.2	32.4	46.0	47,338	0.155	17.9	12.7	45.4	119.5	6.61	0.062	182	3.89	4.51
11	15-Jul-99	572	Balanced	235	690	457	2,125	2.75	99.1	0.62	3237	47.6	30.9	46.0	47,338	0.167	18.3	12.5	46.8	119.5	6.65	0.062	181	3.76	4.52
11	16-Jul-99	573	Balanced	235	690	505	2,027	3.67	43.6	0.61	3020	46.1	29.1	49.5	49,538	0.216	30.0	16.4	40.7	149.0	7.86	0.072	192	3.43	4.58
11	17-Jul-99	574	Balanced	234	691	495	2,042	3.73	46.3	0.60	3020	46.2	28.9	49.0	49,538	0.208	29.5	16.0	40.8	145.4	7.66	0.071	189	3.39	4.58
11	18-Jul-99	575	Balanced	234	690	500	2,020	3.93	53.6	0.60	2988	46.2	29.2	49.5	49,538	0.210	30.7	16.1	41.3	145.2	7.65	0.070	193	3.21	4.61
11	19-Jul-99	576	Balanced	235	690	493	1,981	3.83	53.6	0.59	2935	46.3	29.5	49.5	49,538	0.204	30.8	16.4	41.0	144.2	7.60	0.069	189	3.25	4.60
11	20-Jul-99	577	Balanced	235	690	492	2,057	3.59	47.0	0.61	3041	46.7	29.1	48.5	49,538	0.202	28.3	15.8	40.8	144.7	7.63	0.071	189	3.49	4.57
11	21-Jul-99	578	Balanced	235	690	493	2,085	3.46	47.6	0.61	3064	46.7	29.3	48.5	49,538	0.199	27.3	15.7	40.9	144.7	7.62	0.071	192	3.61	4.56
11	22-Jul-99	579	Balanced	234	690	488	2,070	3.34	47.5	0.61	3050	47.1	28.7	47.5	49,538	0.195	26.2	15.5	41.3	141.8	7.48	0.071	190	3.66	4.55
11	23-Jul-99	580	Balanced	235	689	495	2,026	3.30	61.7	0.60	3002	46.2	28.6	49.0	49,538	0.196	25.6	15.4	42.3	140.3	7.40	0.068	190	3.44	4.57
11	24-Jul-99	581	Balanced	234	690	505	2,054	3.26	70.7	0.61	3056	46.9	29.1	48.0	49,538	0.194	25.0	15.3	42.7	141.8	7.47	0.070	195	3.51	4.57
11	25-Jul-99	582	Balanced	234	690	500	2,072	3.13	64.7	0.61	3059	46.3	28.8	49.0	49,538	0.192	24.4	15.2	42.4	141.5	7.46	0.069	194	3.59	4.53
11	26-Jul-99	583	Balanced	234	690	476	2,090	3.14	50.2	0.61	3041	46.1	29.2	49.5	49,538	0.190	24.1	14.9	41.6	137.3	7.24	0.066	196	3.67	4.51
11	27-Jul-99	584	Balanced	234	689	518	2,069	3.28	115.1	0.62	3468	46.9	27.8	42.0	43,980	0.211	22.3	13.6	48.3	128.7	7.66	0.073	198	3.42	4.54
11	28-Jul-99	585	Balanced	234	685	501	2,026	3.05	106.0	0.60	3362	45.9	29.3	44.5	43,980	0.211	22.1	14.2	46.8	128.6	7.65	0.069	205	3.46	4.47
11	29-Jul-99	586	Balanced	234	681	475	2,011	2.81	80.3	0.60	3327	45.5	29.2	45.5	43,980	0.206	21.1	14.4	44.1	129.3	7.69	0.069	201	3.60	4.58
11	30-Jul-99	587	Balanced	235	684	483	2,024	2.71	81.9	0.60	3353	44.8	29.5	46.5	43,980	0.204	20.8	14.5	44.1	131.4	7.82	0.067	192	3.68	4.52
11	31-Jul-99	588	Balanced	234	684	474	2,019	2.73	85.8	0.60	3340	45.2	29.7	46.0	43,980	0.206	20.5	14.2	44.0	129.1	7.69	0.067	195	3.62	4.57
11	1-Aug-99	589	Balanced	234	685	480	2,040	2.61	81.1	0.60	3366	44.4	27.0	45.5	43,980	0.203	20.0	14.4	43.8	131.5	7.79	0.069	194	3.77	4.59
11	2-Aug-99	590	Balanced	234	685	476	2,067	2.44	81.5	0.61	3418	45.8	31.5	46.0	43,980	0.197	18.6	14.1	43.6	131.0	7.79	0.068	190	3.89	4.57
11	3-Aug-99	591	Balanced	235	685	491	2,049	2.61	98.7	0.61	3396	46.0	27.2	46.0	43,980	0.199	19.4	14.1	45.2	130.3	7.76	0.068	195	3.70	4.63
11	4-Aug-99	592	Balanced	235	687	486	1,987	2.55	99.5	0.59	3314	43.8	29.2	48.0	43,980	0.193	19.4	14.2	45.4	128.4	7.64	0.064	199	3.55	4.56
11	5-Aug-99	593	Balanced	235	685	490	2,015	2.69	96.1	0.60	3360	44.2	30.2	48.0	43,980	0.197	20.1	14.1	45.4	129.5	7.72	0.064	200	3.55	4.52
11	6-Aug-99	594	Balanced	234	686	460	2,031	3.12	106.1	0.60	3350	44.8	29.5	46.5	43,980	0.191	20.9	13.3	46.8	119.7	7.12	0.061	204	3.36	4.47
11	10-Aug-99	598	Balanced	235	705	405	2,196	3.07	41.7	0.61	3472	46.4	33.0	46.0	43,980	0.188	20.3	12.8	41.4	117.3	6.97	0.061	195	3.76	4.38
11	12-Aug-99	600	Balanced	235	705	408	2,158	3.26	56.1	0.59	3413	46.1	32.2	46.0	43,980	0.179	20.8	12.6	43.1	113.5	6.75	0.059	190	3.58	4.41
11	13-Aug-99	601	Balanced	235	704	418	2,149	3.30	59.3	0.60	3429	44.0	29.6	48.0	43,980	0.184	21.1	12.7	43.4	115.6	6.87	0.057	182	3.54	4.43
11	15-Aug-99	603	Balanced	235	696	468	2,094	3.20	68.1	0.60	3260	44.8	29.2	48.5	46,180	0.191	22.6	13.9	44.1	127.1	7.20	0.062	193	3.55	4.42
11	16-Aug-99	604	Balanced	235	700	458	2,109	3.23	60.0	0.60	3259	44.7	28.1	48.0	46,180	0.191	22.9	14.0	43.1	127.6	7.22	0.063	196	3.56	4.44
11	17-Aug-99	605	Balanced	235	702	462	2,082	3.46	69.0	0.59	3228	44.5	28.2	48.5	46,180	0.193	24.0	14.0	43.9	126.2	7.15	0.062	192	3.39	4.46
11	18-Aug-99	606	Balanced	235	702	464	2,059	3.49	65.6	0.59	3224	44.3	27.8	48.5	46,180	0.197	24.7	14.2	43.6	127.7	7.23	0.063	194	3.42	4.47
11	19-Aug-99	607	Balanced	235	700	461	2,054	3.59	70.1	0.59	3199	43.7	28.2	50.0	46,180	0.196	25.0	14.2	43.8	126.4	7.16	0.060	192	3.34	4.51
11	20-Aug-99	608	Balanced	235	700	499	2,031	4.12	49.8	0.59	3072	42.8	28.1	54.0	48,380	0.234	32.7	16.5	40.8	146.6	7.91	0.065	185	3.34	4.59
11	21-Aug-99	609	Balanced	235	700	504	2,007	4.19	50.9	0.59	3046	42.8	28.7	54.5	48,380	0.240	33.8	16.9	40.7	148.4	8.01	0.065	199	3.22	4.53
11	22-Aug-99	610	Balanced	235	700	492	2,033	4.12	46.0	0.59	3049	42.8	28.0	54.0	48,380	0.237	33.1	16.7	40.4	146.4	7.89	0.064	207	3.27	4.47
11	23-Aug-99	611	Balanced	235	700	490	1,985	4.28	52.4	0.58	3012	43.1	27.6	53.0	48,380	0.232	33.5	16.5	41.1	143.2	7.73	0.064	196	3.13	4.53
11	24-Aug-99	612	Balanced	235	710	536	2,065	3.57	63.1	0.60	3156	43.4	27.8	52.5	48,380	0.224	29.8	16.9	41.5	154.9	8.36	0.070	195	3.58	4.48
11	25-Aug-99	613	Balanced	235	710	530	2,112	3.57	62.8	0.61	3217	44.2	28.4	51.5	48,380	0.221	29.0	16.5	41.4	153.7	8.30	0.071	191	3.71	4.47
11	26-Aug-99	614	Balanced	235	710	541	2,058	3.57	69.1	0.60	3160	44.3	28.0	51.0	48,380	0.225	29.6	16.8	42.0	154.7	8.35	0.072	201	3.55	4.53
11	27-Aug-99	615	Balanced	235	710	528	2,071	3.63	60.7	0.60	3159	44.4	27.6	50.5	48,380	0.228	30.0	16.9	41.3	153.5	8.28	0.072	212	3.55	4.50
11	28-Aug-99	616	Balanced	235	710	546	2,063	3.45	75.6	0.61	3180	43.8	28.1	52.0	48,380	0.224	28.4	16.7	42.6	153.8	8.30	0.070	200	3.54	4.45
11	2-Sep-99	621	Balanced	235	710	460	2,173	2.60	95.9	0.62	4151	43.9	36.9	46.0	37,688	0.207	17.9	12.9	45.3	123.6	8.58	0.064	182	3.59	4.43
11	3-Sep-99	622	Balanced	235	711	522	2,133	3.26	57.1	0.62	3923	43.8	33.0	46.0	39,888	0.266	26.7	16.2	41.1	152.3	9.97	0.079	201	3.79	4.44
11	5-Sep-99	624	Balanced	235	710	620	2,083	3.50	54.7	0.63	3772	43.2	30.7	48.0	42,088	0.325	34.3	19.7	40.3	184.7	11.45	0.092	208	4.04	4.48
11	6-Sep-99	625	Balanced	235	710	676	2,051	3.59	49.3	0.63	3617	42.1	26.2	49.5	44,288	0.365	39.4	22.1	39.4	206.2	12.15	0.099	202	4.08	4.43
11	11-Sep-99	630	Balanced	235	710	793	2,042	3.43	85.3	0.66	3587	42.4	32.5	56.0	46,488	0.371	39.8	23.4	41.0	231.9	13.02	0.098	176	4.25	4.40
11	15-Sep-99	634	Balanced	235	695	622	1,948	4.54	44.0	0.60	3237	43.3	28.8	51.5	46,488	0.374	44.4	20.7	39.5	188.9	10.60	0.087	192	3.08	4.62
11	16-Sep-99	635	Balanced	235	695	620	2,066	4.12	32.3	0.63	3374	43.7	30.7	52.0	46,488	0.361	41.6	20.6	38.4	193.8	10.87	0.089	191	3.62	4.48
11	17-Sep-99	636	Balanced	235	695	615	2,023	4.34	37.4	0.62	3340	44.0	30.1	51.0	46,488	0.352	43.1	20.6	38.8	189.9	10.65	0.089	195	3.53	4.46

Table D.3-1. Data Summary for LPMEOH™ Demonstration Unit (continued)

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")
11	19-Sep-99	638	Balanced	235	695	643	1,814	4.64	87.6	0.57	3026	43.9	26.9	49.0	46,488	0.359	46.4	21.6	46.4	186.4	10.47	0.091	209	2.61	4.78
11	20-Sep-99	639	Balanced	235	710	819	1,997	2.98	112.8	0.65	3563	44.6	30.3	50.0	46,488	0.356	35.6	23.2	42.4	231.9	13.03	0.110	187	4.33	4.39
11	21-Sep-99	640	Balanced	235	711	832	2,034	2.73	116.7	0.67	3641	45.1	30.8	49.5	46,488	0.353	33.3	23.2	42.4	235.8	13.24	0.113	189	4.66	4.36
11	22-Sep-99	641	Balanced	234	710	858	1,950	3.18	165.2	0.65	3539	45.3	30.0	48.5	46,488	0.352	36.9	23.2	44.8	230.0	12.92	0.113	195	4.05	4.41
11	23-Sep-99	642	Balanced	234	707	831	1,994	2.94	132.0	0.66	3577	45.2	30.5	49.0	46,488	0.354	35.2	23.3	42.8	232.9	13.08	0.113	189	4.33	4.36
11	24-Sep-99	643	Balanced	235	706	831	1,953	3.11	136.0	0.65	3523	44.0	29.3	50.5	46,488	0.354	36.8	23.4	43.3	230.3	12.94	0.109	191	4.13	4.38
11	25-Sep-99	644	Balanced	235	706	821	1,947	3.21	123.4	0.65	3505	43.0	28.0	51.5	46,488	0.354	37.6	23.3	43.0	228.8	12.86	0.106	190	4.05	4.41
11	26-Sep-99	645	Balanced	235	708	793	1,980	3.28	103.0	0.64	3494	42.6	29.0	53.0	46,488	0.354	38.1	23.2	42.0	226.9	12.74	0.102	190	4.02	4.42
11	27-Sep-99	646	Balanced	235	708	794	1,936	3.13	104.9	0.63	3463	42.6	28.3	52.5	46,488	0.351	37.2	23.5	42.0	227.0	12.75	0.103	192	4.02	4.44
11	28-Sep-99	647	Balanced	235	710	789	2,013	2.89	95.8	0.65	3557	43.0	30.1	53.0	46,488	0.345	34.5	23.0	41.4	228.9	12.85	0.103	188	4.43	4.40
11	29-Sep-99	648	Balanced	235	710	803	2,003	2.81	105.1	0.65	3565	42.9	31.8	54.5	46,488	0.346	34.2	23.3	41.7	230.8	12.96	0.101	186	4.52	4.36
11	30-Sep-99	649	Balanced	235	710	795	2,016	2.89	113.2	0.65	3558	43.3	31.5	53.5	46,488	0.334	34.0	22.8	42.3	225.8	12.68	0.100	185	4.35	4.35

Several catalyst additions and withdrawals were conducted during this quarter to maintain reactor productivity. A series of three catalyst batch withdrawals were conducted on 12-13 July 1999. A total of approximately 7,936 pounds of catalyst was removed with these three batches. A fresh batch of catalyst was then added on 19 July 1999.

On 14 August 1999, a batch of catalyst was reduced to approximately 60% of the theoretical maximum uptake of syngas. It was determined that the reduction problems experienced with this batch were related to high catalyst solids concentration of the catalyst slurry prior to the start of reduction. This was an isolated error in operating procedure that is not expected to re-occur. Although not completely activated, this batch of catalyst was pumped to the reactor on 14 August 1999. The amount of syngas which was being purged from the LPMEOH™ Demonstration Unit was immediately decreased following the addition step; this indicated that the batch did have some activity for the production of methanol. Another batch of catalyst was fully activated and added to the reactor on 20 August 1999.

A major catalyst withdrawal and addition campaign was undertaken during late August and early September of 1999. A series of four withdrawals were conducted: two withdrawals were conducted on 30 August 1999, one withdrawal on 31 August 1999, and the final withdrawal on 01 September 1999. This was followed by four additions of activated catalyst: the first on 03 September 1999, the second on 04 September 1999, the third on 06 September 1999, and the final batch on 11 September 1999. After the addition of the final batch of catalyst, the total catalyst inventory was calculated to be 46,488 pounds. Due to the extensive catalyst withdrawal and addition campaigns, there were no extended periods of operation (2 to 3 weeks) which are needed to track catalyst deactivation.

During the quarter, Balanced Gas flow averaged 507 KSCFH through 19 September 1999. On 20 September 1999, Balanced Gas flow was increased to 800 KSCFH and held at that rate for the remainder of the reporting period. Reactor temperature was held at 235°C. Reactor pressure was limited to 690-695 psig early in the quarter due to high pressure drop across the 29C-40 carbonyl guard bed (see below).

As reported in Technical Progress Report No. 20, the adsorbents in the two catalyst guard beds (the 10C-30 vessel, upstream of both the LPMEOH™ Demonstration Plant and the fixed-bed methanol plant, and the 29C-40 carbonyl guard bed) which treat the Balanced Gas stream entering the LPMEOH™ Demonstration Unit, were changed; the 10C-30 was charged with arsine-removal adsorbent, and the 29C-40 was split between arsine and carbonyl removal materials. After the guard beds were brought back online, the operating pressure for the LPMEOH™ Reactor had to be lowered by 10 psi to account primarily for higher pressure drop through both beds.

During the reporting period, the pressure drop across the 10C-30 stabilized at less than 5 psi pressure drop. However, the pressure drop across the 29C-40 continued to gradually increase during the month of July to over 20 psi; the expected pressure drop was less than 1 psi. On 12 August 1999, the 29C-40 was emptied and inspected so that the reason for the excessive pressure drop could be determined. An internal inspection of the bottom support screen revealed that the screen openings had been compacted, and that some adsorbent had blinded a small portion of the bottom of the screen. To rectify this problem, the internal area of the screen was opened in order to increase the available flow area, and the support for the screen was modified. After this procedure and the subsequent return to service, the guard bed pressure drop was 0.5 psi. Reactor pressure was raised to 700 psig after the successful guard bed investigation.

During a routine sampling procedure involving the transfer of catalyst slurry between the reactor and the catalyst activation vessel (29C-30) on 27 July 1999, a leak developed in a gasketed connection on a pressure transmitter in the interconnecting piping circuit (PT-424). A portion of the contents of the reactor spilled onto the concrete floor within the catalyst reduction building, and was contained within the floor drains and the oil-water separator. The reactor level dropped approximately 4' out of a total of 46' (the level prior to the start of the sampling procedure). This corresponds to a loss of approximately 5,558 pounds of catalyst from the system (this amount includes the quantity retained in the 29C-30 vessel but not transferred). There were no injuries as a result of this incident. The cause of the failure was found to be the gasket material for the pressure transmitter connection tap. It was fully corrected by using an alternative gasket material for the service. No other pressure transmitters were found to affected by this problem.

Analyses of catalyst samples for changes in physical characteristics and levels of poisons have continued. Appendix E, Table 2 summarizes the results to date. The most recent concentration of arsenic on the methanol synthesis catalyst is 1,810 ppmw; the arsenic loading continues to slowly increase with time. Sulfur is present at about 488 ppmw, which is above the analytical detection limit, and may be adversely impacting catalyst life. Copper crystallite size measurements have shown an increase in the most recent samples. Levels of iron and nickel have remained low and steady since the restart in December of 1997.

Sparger Resistance

The performance of the gas sparger continues to meet the design expectations for pressure drop and reactor operation. Appendix E, 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.3-1. The flow resistance will be monitored in order to determine the changes in performance with operating time.

D.4 Planning and Administration

A Project Review Meeting was held in Pittsburgh, PA, on 15-16 September 1999. The results of the unit operation were reviewed, and the plans for the upcoming design verification test of the LPDME Process at the LaPorte AFDU were reviewed and approved. The meeting agenda, extracts from the meeting handouts, and the meeting notes are included in Appendix F.

The Milestone Schedule Status Report and the Cost Management Report, through the period ending 30 September 1999, are included in Appendix G. 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 30 September 1999. Forty-six percent (46%) of the $158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 September 1999.

The monthly reports for July, August, and September were submitted. These reports include the Milestone Schedule Status Report, the Project Summary Report, and the Cost Management Report.

The topical report entitled “Alternative Fuels Field Test Unit Support to Kingsport LPMEOH™ Demonstration Unit - December 1997 - January 1998” was approved and issued. Comments were received from DOE on the draft of Volume 1 - Public Design, of the Final Report for the LPMEOH™ Demonstration Project, and a revised version was sent to DOE for review.

A presentation entitled “Liquid Phase Methanol (LPMEOH™) Project: Operating Experience Update” was given at the 1999 DOE Fossil Energy Fuels Program Review (21-23 September 1999). This was based upon the paper entitled “Liquid Phase Methanol (LPMEOH™) Project: Operating Experience Update” which was submitted for presentation at the 1999 Gasification Technologies Conference (17-20 October 1999).

A paper entitled “Direct Applications of Stabilized Methanol from the Liquid Phase Methanol (LPMEOH™) Process” was submitted for presentation at the 16th Annual International Pittsburgh Coal Conference (11-15 October 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.
- Complete shakedown activities and execute the test plan for the LPDME design verification test run at the LaPorte AFDU.
- Continue execution of the Off-Site, Product-Use Test Program (Phase 1, Task 1.4).
- Conduct a Project Review Meeting with DOE.

F. Conclusion

The LPMEOH^{TM} Demonstration Unit operated at 100% availability throughout the quarter. Three short duration syngas outages were the only operating interruptions experienced.

Several catalyst additions and withdrawals were conducted during this quarter to maintain reactor productivity. A series of three catalyst batch withdrawals were conducted on 12-13 July 1999. A total of approximately 7,936 pounds of catalyst was removed with these three batches. A fresh batch of catalyst was then added on 19 July 1999.

On 14 August 1999, a batch of catalyst was reduced to approximately 60% of the theoretical maximum uptake of syngas. It was determined that the reduction problems experienced with this batch were related to high catalyst solids concentration of the catalyst slurry prior to the start of reduction. Although not completely activated, this batch of catalyst was pumped to the reactor on 14 August 1999, and the batch appeared to have some activity for the production of methanol. Another batch of catalyst was fully activated and added to the reactor on 20 August 1999.

A major catalyst withdrawal and addition campaign was undertaken during late August and early September of 1999. A series of four withdrawals were conducted between 30 August and 01 September 1999. This was followed by four catalyst additions which were activated and added between 03 September and 11 September 1999. After the addition of the final batch of catalyst, the total catalyst inventory was calculated to be 46,488 pounds. Due to the extensive catalyst withdrawal and addition campaigns, there were no extended periods of operation (2 to 3 weeks) which are needed to track catalyst deactivation.

During the quarter, Balanced Gas flow averaged 507 KSCFH through 19 September 1999. On 20 September 1999, Balanced Gas flow was increased to 800 KSCFH and held at that rate for the remainder of the reporting period. Reactor temperature was held at 235°C. Reactor pressure was limited to 690-695 psig early in the quarter due to high pressure drop across the 29C-40 carbonyl guard bed (see below).

During the reporting period, the pressure drop across the carbonyl guard bed, which had been recharged with both arsine and carbonyl removal materials in June of 1999, continued to gradually increase. By the end of July of 1999, the pressure drop was measured at over 20 psi; the expected pressure drop was less than 1 psi. On 12 August 1999, the carbonyl guard bed was emptied and inspected; the bottom support screen was found to be damaged, and some adsorbent had blinded a small portion of the bottom of the screen. After changes to the screen and its support were made, the guard bed was returned to service, and the pressure drop was 0.5 psi. Reactor pressure was raised to 700 psig after the successful guard bed investigation.

During a routine sampling procedure involving the transfer of catalyst slurry between the reactor and the catalyst activation vessel on 27 July 1999, a leak developed in a gasketed connection on a pressure transmitter in the interconnecting piping circuit. A portion of the contents of the reactor spilled onto the concrete floor within the catalyst reduction building, and was contained within the floor drains and the oil-water separator. This corresponds to a loss of approximately 5,558 pounds of catalyst from the system. There were no injuries as a result of this incident. The cause of the failure was found to be the gasket material for the pressure transmitter connection tap. It was fully corrected by using an alternative gasket material for the service.

Analyses of catalyst samples for changes in physical characteristics and levels of poisons have continued. Sulfur continues to be measured on the catalyst above the analytical detection limit, and may be adversely impacting catalyst life. Copper crystallite size measurements have shown an increase in the most recent samples. 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 March of 1999, was monitored. The performance to date of the new sparger has met the design expectations for pressure drop and reactor operation.

During the reporting period, a total of 4,391,257 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, about 42.8 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.

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 that a gas turbine project (under development by ARCADIS Geraghty & Miller) should be redirected from VOC control to NO_x control. Results from testing of stabilized methanol in a reformer test apparatus at the University of Florida have shown that the reformer catalyst deactivated quickly. A proposal to perform additional testing of stabilized methanol in the apparatus in order to qualify this material as feedstock to a phosphoric acid fuel cell was recommended to, and accepted by, DOE. At West Virginia University, four axially spaced thermocouples will be installed inside the combustion chamber of the stationary gas turbine. This will be used in future computer modeling work on the turbine. DOE accepted Air Products' recommendation to provide stabilized methanol from the LPMEOH™ Process Demonstration Facility for use as part a new contract between the Institute and the Florida Energy Office.

During the reporting period, planning for a design verification test run of the LPDME Process at the LaPorte AFDU continued. At a review meeting for the DOE's Liquid Fuels Program on 09 June 1999, the participants agreed that the next test for the LPDME Process at the LaPorte AFDU should be treated as an interim campaign, with the primary objective being the determination of a tie-point between catalyst performance in the autoclave and the pilot plant scale. DOE accepted Air Products' recommendation to proceed with the interim campaign at the LaPorte AFDU.

During the reporting period, a commercially available dehydration material was qualified for the LPDME design verification test. The methanol synthesis and dehydration catalysts were ordered and shipped to LaPorte. An autoclave test of these materials was successfully performed; this verified the suitability of the catalysts for use during the design verification testing.

In anticipation of an October 1999 startup of the LPDME design verification test at the LaPorte AFDU, checkout of mechanical and instrumentation systems was initiated. A hot function test on syngas (in the absence of catalyst) is planned for the week of 04 October 1999. The date for catalyst loading at the LaPorte AFDU is scheduled for 09 October 1999.

A Project Review Meeting was held in Pittsburgh, PA, on 15-16 September 1999. The results of the unit operation were reviewed, and the plans for the upcoming design verification test of the LPDME Process at the LaPorte AFDU were reviewed and approved.

The topical report entitled "Alternative Fuels Field Test Unit Support to Kingsport LPMEOH™ Demonstration Unit - December 1997 - January 1998" was approved and issued. Comments were received from DOE on the draft of Volume 1 - Public Design, of the Final Report for the LPMEOH™ Demonstration Project, and a revised version was sent to DOE for review.

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A paper entitled "Direct Applications of Stabilized Methanol from the Liquid Phase Methanol (LPMEOH™) Process" was submitted for presentation at the 16th Annual International Pittsburgh Coal Conference (11-15 October 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 30 September 1999. Forty-six percent (46%) of the $158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 September 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

Quarterly Reports:

Appendix B-2 - ARCADIS Projects (two pages):
- **Aircraft Ground Equipment Emulsion**
- **Stationary Turbine for NO_x Control**

Appendix B-3 - West Virginia University Stationary Gas Turbine (six pages)
Appendix B-4 - University of Florida Fuel Cell (five pages)
Appendix B-5 - Florida Institute of Technology - Report to Florida Energy Office (thirty-one pages)

APPENDIX C - DME DESIGN VERIFICATION TESTING

Appendix C-1 - 1997 DME Design Verification Testing Recommendation
(fourteen pages)
Appendix C-2 - Recommendation to Proceed with Fall 1999 Test at LaPorte AFDU
(nine pages)

APPENDIX D - SAMPLES OF DETAILED MATERIAL BALANCE REPORTS

APPENDIX E - RESULTS OF DEMONSTRATION PLANT OPERATION

Table 1 - Summary of LPMEOH™ Demonstration Unit Outages - July/September 1999
Table 2 - Summary of Catalyst Samples - Second Catalyst Batch

Figure 1 - Catalyst Age (η): March - September 1999
Figure 2 - Sparger Resistance Coefficient vs. Days Onstream (March 1999 to September 1999)

Table 1
Summary of LPMEOH™ Demonstration Unit Outages - July/September 1999

Operation Start	Operation End	Operating Hours	Shutdown Hours	Reason for Shutdown
7/1/99 00:00	7/30/99 01:40	697.7	1.0	Syngas Outage
7/30/99 02:40	8/7/99 17:15	206.6	22.7	Syngas Outage
8/8/99 16:00	8/9/99 11:00	19.0	12.5	Syngas Outage
8/9/99 23:30	9/30/99 23:59	1248.5		
	Total Operating Hours		2171.7	
	Syngas Available Hours		2171.7	
	Plant Availability, %		**100.00**	

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
K9904-3	Reactor Sample 4/27/99	417.8	110.4	15	131	18.2	348	1460	<30
K9906-1	Reactor Sample 6/1/99	517	105	43	109	19.7	316	1680	40
K9907-1	Reactor Sample 7/13/99	446	116	59	175	19.7	488	1810	30
K9908-2	Reactor Sample 8/31/99	632	117						20

Notes:

1) nd = none detected
2) * - these values represent re-analysis of the sample as compared to Technical Progress Report no. 17

Figure 1

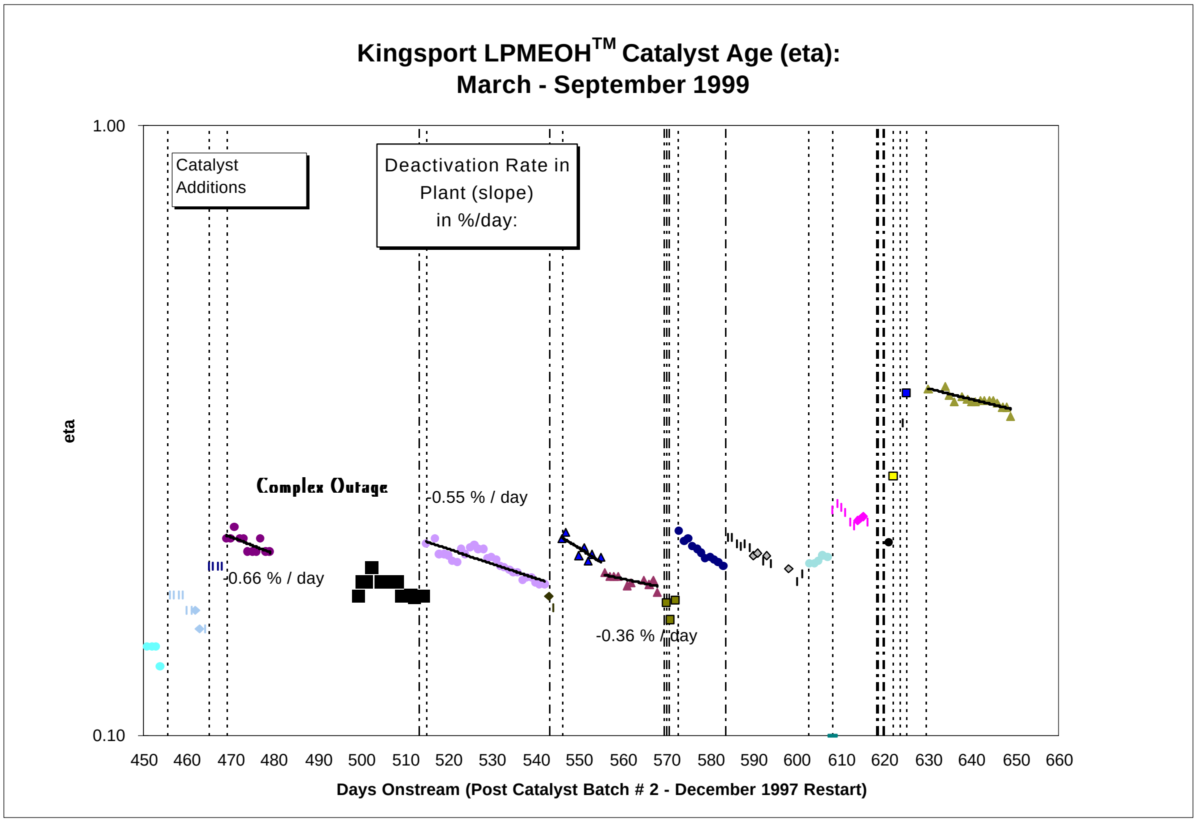
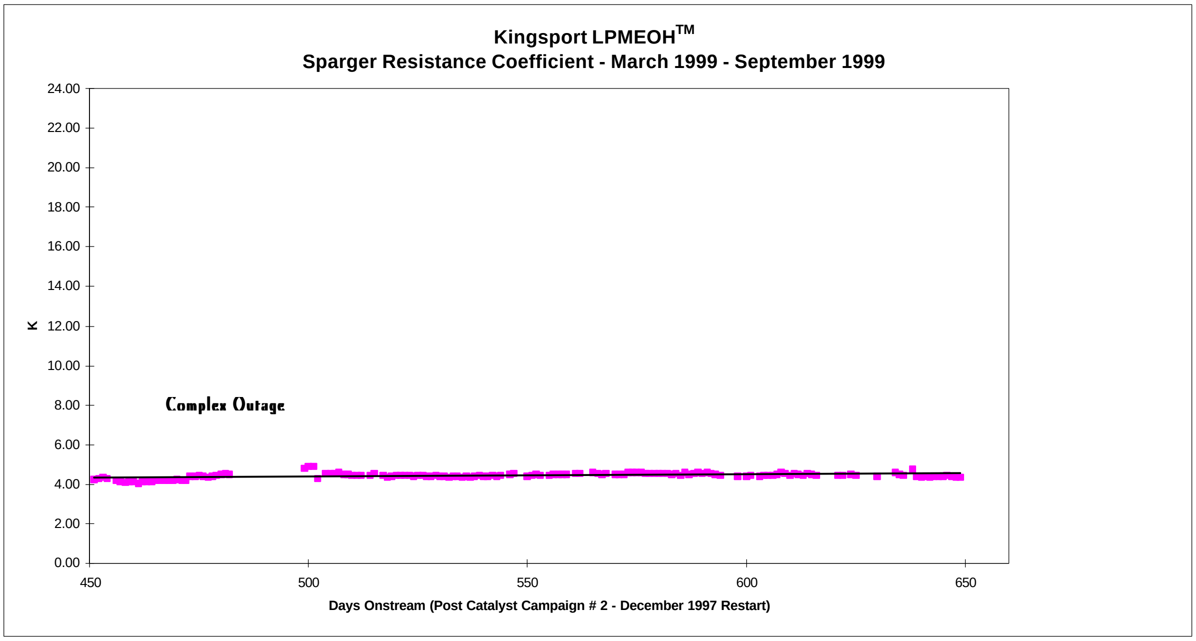

Kingsport LPMEOH™ Catalyst Age (eta):
March - September 1999
eta
1.00
0.10
Catalyst Additions
Deactivation Rate in Plant (slope) in %/day:
Complex Outage
-0.66 % / day
-0.55 % / day
-0.36 % / day
450 460 470 480 490 500 510 520 530 540 550 560 570 580 590 600 610 620 630 640 650 660
Days Onstream (Post Catalyst Batch # 2 - December 1997 Restart)

Figure 2



Kingsport LPMEOH™
Sparger Resistance Coefficient - March 1999 - September 1999
K
24.00
22.00
20.00
18.00
16.00
14.00
12.00
10.00
8.00
6.00
4.00
2.00
0.00
Complex Outage
450
500
550
600
650
Days Onstream (Post Catalyst Campaign # 2 - December 1997 Restart)

APPENDIX F - PROJECT REVIEW MEETING (15-16 SEPTEMBER 1999)

APPENDIX G - MILESTONE SCHEDULE STATUS AND COST MANAGEMENT REPORTS