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

TECHNICAL PROGRESS REPORT NO. 25

For The Period

1 July - 30 September 2000

Prepared by

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and

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for the
Air Products Liquid Phase Conversion Company, L.P.

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

Patents cleared by Chicago on 14 December 2000.

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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 94.45% availability throughout the quarter. There were four short syngas interruptions experienced which totaled 76 hours. A planned maintenance turnaround was accomplished on 30 July 2000 to 04 August 2000 for a total of 119 hours. During this outage, maintenance was performed on reactor pressure transmitters, the economizer heat exchanger was inspected and cleaned, and the adsorbent in the catalyst guard bed was changed (although the catalyst guard bed was bypassed for most of the quarter).

Two catalyst withdrawal and addition campaigns were undertaken during the quarter. A catalyst withdrawal (2 batches) and addition (3 batches) campaign was undertaken in July of 2000 to raise catalyst activity on an interim basis prior to the changeout of the adsorbent in the catalyst guard bed within the LPMEOH™ Demonstration Unit. A second campaign was started at the end of the reporting period, and will be completed at the beginning of October of 2000.

During most of the quarter, the flowrate of the primary syngas feed (Balanced Gas) was controlled at an average flowrate of 697 KSCFH. During these operating periods, the reactor pressure was set at between 705 and 710 psig and temperature was maintained at 235ºC.

There were two extended periods of operation (minimum of about 2 weeks) at a reactor temperature of 235°C during which catalyst activity was measured to track catalyst deactivation during the quarter. When the catalyst guard bed was in service with aged adsorbents (manganese dioxide and activated carbon), an overall deactivation rate of 0.85% per day was calculated over a 12-day period. When the catalyst guard bed was bypassed over a 28-day period, an overall deactivation rate of 0.76% per day was calculated. These results are statistically the same and also indicate that the aged adsorbents in the catalyst guard bed were no longer effective in removing trace contaminants. These deactivation results are greater than the baseline deactivation rate of 0.4% per day from the 4-month proof-of-concept run at the LaPorte AFDU in 1988/89 (this run was performed at 250°C), and may reflect the impact of poisons on catalyst aging.

Analyses of catalyst samples for changes in physical characteristics and levels of poisons have continued. Samples have continued to show an increase in arsenic, which has been demonstrated in the laboratory to act as a poison to methanol synthesis catalyst. Sulfur,

another known catalyst poison, continues to be measured above the analytical detection limit. Copper crystallite size measurements have decreased over the last few samples. Levels of nickel (a known catalyst poison) have remained low and steady since the restart in December of 1997. The concentration of iron (another poison) is low (less than 200 ppmw), and has stabilized in the most recent samples.

Eastman had previously accepted a recommendation by Air Products to remove trace amounts of arsine in the Balanced Gas by using a commercially available copper-impregnated activated carbon in the catalyst guard bed within the LPMEOH™ Demonstration Unit. This adsorbent was selected after investigative work in the laboratory and at Kingsport (via a slip-stream apparatus) was performed.

After the loading operation was completed, the catalyst guard bed was operationally tested on 03 August 2000. Within 30 minutes of the introduction of Balanced Gas, temperatures in the catalyst guard bed began to rise beyond the expected values based upon the results of the laboratory testing. The Eastman operations team moved quickly to depressurize the catalyst guard bed by venting the syngas to the Eastman purge gas header. No environmental issues were experienced due to this incident. The catalyst guard bed was then isolated from the feed syngas streams to allow the LPMEOH™ Demonstration Unit to be returned to operation.

The results of additional laboratory testing have indicated that the probable cause of this temperature excursion was the reduction of copper oxide to copper metal. A meeting was held between Air Products and Eastman on 21 September 2000 to discuss the results of this testing. A plan to bring the catalyst guard bed back into service, including modifications which will be necessary to complete the reduction of copper oxide within the catalyst guard bed, is being developed; the finalized version will be reviewed with DOE prior to implementation.

Discussions were held between Air Products and Eastman to initiate the design engineering activities in support of the proposed in-situ activation of methanol synthesis catalyst at the LPMEOH™ Demonstration Facility. Piping and instrumentation are needed to allow for the introduction of the Balanced Gas and nitrogen streams which will be required to reduce the catalyst.

The performance of the 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. After the restart of the LPMEOH™ Demonstration Facility on 04 August 2000, the sparger flow resistance increased approximately 50%. Subsequent flushing of the sparger with clean oil resulted in a recovery of about ½ of the earlier increase. In addition, the resistance does not appear to be increasing over time.

During the reporting period, a total of 5,363,012 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, about 64.2 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 safety or environmental incidents were reported during this quarter.

During this quarter, planning, procurement, and test operations continued on one of the project sites selected for the off-site, product-use test program. A draft of the paper entitled "Lubricity Problems and Solutions for a Methanol Fueled Gas Turbine" was sent to DOE; this paper was accepted for presentation at the International Mechanical Engineering Conference and Exposition in Orlando, FL (05-10 November 2000). Results of experiments in the reformer test apparatus have provided convincing evidence that the performance of the low temperature catalyst is adversely affected by the presence of the trace mineral oil in the stabilized methanol. The performance of stabilized methanol in a higher-temperature steam reforming process may yield increased conversion and selectivity which are required for the fuel cell application.

Activities associated with Design Verification Testing of the LPDME Process have been completed. A draft Topical Report which presents the results of the design verification test at the LaPorte AFDU was sent to DOE. Comments were received, and a revision will be issued for final review and approval by January of 2001.

During this reporting period, an update to the Demonstration Test Plan which defined the tests which are planned for the remainder of the operating program was sent to DOE for review and comment.

The paper entitled "Liquid Phase Methanol (LPMEOH™) Process Development" was presented at Energex 2000 - The 8th International Energy Forum (23-28 July 2000).
A paper entitled "Catalyst and Process Development for Liquid Phase DME Synthesis" was presented at 17th Annual International Pittsburgh Coal Conference (11-15 September 2000).

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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 2000. Sixty-four percent (64%) of the $158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 September 2000.

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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
Btu	-	British Thermal Unit
Carbon Monoxide Gas	-	A syngas containing primarily carbon monoxide (CO); also called CO Gas
Catalyst Activity	-	the rate at which the catalyst promotes the desired chemical reaction to proceed within the limitations of chemical equilibrium
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 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)
MW	-	molecular weight, pound per pound mole

ACRONYMS AND DEFINITIONS (cont'd)

NEPA - National Environmental Policy Act
OSHA - Occupational Safety and Health Administration
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
SCF - Standard Cubic Feet
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 Unit and the Eastman Facility
TPD - Ton(s) per Day
V - volumetric flowrate, thousand standard cubic feet per hour
VOC - volatile organic compound
vol% - volume %
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 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 has also completed design verification testing (DVT), including laboratory- and pilot-scale research and market verification studies, to evaluate whether to include a demonstration of the production of dimethyl ether (DME) as a mixed coproduct with methanol. 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 94.45% availability throughout the quarter. There were four short syngas interruptions experienced which totaled 76 hours. A planned maintenance turnaround was accomplished on 30 July 2000 to 04 August 2000 for a total of 119 hours. During this outage, maintenance was performed on reactor pressure transmitters, the economizer heat exchanger was inspected and cleaned, and the adsorbent in the catalyst guard bed was changed (although the catalyst guard bed was bypassed for most of the quarter).

Two catalyst withdrawal and addition campaigns were undertaken during the quarter. A catalyst withdrawal (2 batches) and addition (3 batches) campaign was undertaken between

17 and 25 July 2000 to raise catalyst activity on an interim basis prior to the changeout of the adsorbent in the catalyst guard bed within the LPMEOH™ Demonstration Unit. In the second campaign, a series of four withdrawals and three activations and additions were conducted between 18 and 24 September 2000. Plans for the next quarter called for the activation and addition of two more batches of fresh catalyst.

During most of the quarter, the flowrate of the primary syngas feed (Balanced Gas) was controlled at an average flowrate of 697 KSCFH. During these operating periods, the reactor pressure was set at between 705 and 710 psig and temperature was maintained at 235ºC.

There were two extended periods of operation (minimum of about 2 weeks) at a reactor temperature of 235°C during which catalyst activity was measured to track catalyst deactivation during the quarter. An overall deactivation rate of 0.85% per day was calculated for the period 24 June to 05 July 2000 (12 days). This data was based on operation with the catalyst guard bed in service with aged adsorbents (manganese dioxide and activated carbon). An overall deactivation rate of 0.76% per day was calculated for the period 15 August 2000 to 11 September 2000 (28 days). This data was based on operation with the catalyst guard bed bypassed and out of service. These deactivation results are greater than the baseline deactivation rate of 0.4% per day from the 4-month proof-of-concept run at the LaPorte AFDU in 1988/89 (this run was performed at 250°C), and may reflect the impact of poisons on catalyst aging. These results are statistically the same and also indicate that the aged adsorbents in the catalyst guard bed were no longer effective in removing trace contaminants.

Analyses of catalyst samples for changes in physical characteristics and levels of poisons have continued. Samples have continued to show an increase in arsenic, which has been demonstrated in the laboratory to act as a poison to methanol synthesis catalyst. Sulfur, another known catalyst poison, continues to be measured above the analytical detection limit. Copper crystallite size measurements have decreased over the last few samples. Levels of nickel (a known catalyst poison) have remained low and steady since the restart in December of 1997. The concentration of iron (another poison) is low (less than 200 ppmw), and has stabilized in the most recent samples.

Eastman had previously accepted a recommendation by Air Products to use a commercially available copper-impregnated activated carbon in the catalyst guard bed within the LPMEOH™ Demonstration Unit. This material is chemically treated with copper oxide to enhance its arsine removal capabilities. This adsorbent was selected after investigative work in the laboratory and at Kingsport (via a slip-stream apparatus) was performed to evaluate the effectiveness of the material and to address questions on potential interaction with the components of the syngas stream.

After the loading operation was completed, the catalyst guard bed was operationally tested on 03 August 2000. After the catalyst guard bed was purged with nitrogen, Balanced Gas was introduced to the closed vessel in order to leak-check the flanges which had been opened during the maintenance activities. Within 30 minutes of the introduction of syngas, temperatures in the catalyst guard bed began to rise beyond the expected values based upon the results of the laboratory testing. The Eastman operations team moved quickly to depressurize the catalyst guard bed by venting the syngas to the Eastman purge gas header. No environmental issues were experienced due to this incident. The catalyst guard bed was then isolated from the feed syngas streams to allow the LPMEOH™ Demonstration Unit to be returned to operation.

The results of additional laboratory testing have indicated that the probable cause of this temperature excursion was the reduction of copper oxide to copper metal. Preliminary engineering calculations and subsequent laboratory experiments have indicated that this is the only likely source of the magnitude of the heat rise which was experienced. A meeting was held between Air Products and Eastman on 21 September 2000 to discuss the results of this testing. A plan to bring the catalyst guard bed back into service, including modifications which will be necessary to complete the reduction of copper oxide within the catalyst guard bed, is being developed; the finalized version will be reviewed with DOE prior to implementation.

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During the reporting period, a total of 5,363,012 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, about 64.2 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 safety or environmental incidents were reported during this quarter.

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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 has been demonstrating 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 has also evaluated the demonstration of the production of dimethyl ether (DME) as a mixed coproduct with methanol.

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 8,600 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, 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

One of the product-use test projects has ongoing activities; Appendix B contains the status report for this work. The other six projects have completed testing of stabilized methanol, and are at stages of development of their respective final reports. Status and highlights include:

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

Stationary Turbine for Nitrogen Oxide (NO_x) Control - The test results on the low-NO_x gas turbine combustor fueled with stabilized methanol from the LPMEOH™ Demonstration Unit have been prepared. Air Products is awaiting the submittal of the draft final report from ARCADIS Geraghty & Miller.

West Virginia University (WVU) Stationary Gas Turbine - Testing of stabilized methanol in the gas turbine system has been completed, and preparation of the final report is underway. A draft of the paper entitled “Lubricity Problems and Solutions for a Methanol Fueled Gas Turbine” was sent to DOE; this paper was accepted for presentation at the International Mechanical Engineering Conference and Exposition in Orlando, FL (05-10 November 2000).

Aircraft Ground Equipment Emulsion - A final report on the use of a methanol emulsion as the fuel for a flight line generator at Tyndall Air Force Base was approved by Air Products.

University of Florida Fuel Cell - Results of experiments in the reformer test apparatus have provided convincing evidence that the performance of the low temperature catalyst is adversely affected by the presence of the trace mineral oil in the stabilized methanol. The investigators have hypothesized that some of the oil is present in liquid form at reaction conditions; this phase presumably coats the surface of the catalyst. The performance of stabilized methanol in a higher-temperature steam reforming process may yield increased conversion and selectivity which are required in the fuel cell application.

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 Unit 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

The LPMEOH™ Demonstration Project has completed Design Verification Testing (DVT) to coproduce dimethyl ether (DME) with methanol via the Liquid Phase Dimethyl Ether (LPDME) Process. DVT was required to provide additional data for engineering design and evaluation of the potential for demonstration at the LPMEOH™ Demonstration Unit. The essential steps required for decision-making were: 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.

Execution of the LPDME design verification test at the LaPorte AFDU was completed during October and November of 1999, and preliminary results from the operation were presented in Technical Progress Report No. 22. Results from a cost estimate for a commercial-scale LPDME plant were presented in Technical Progress Report No. 23. After discussing the results from the LPDME Design Verification Testing activities and the ongoing performance results from Kingsport, the project participants agreed that the available resources should be directed toward improving the catalyst performance for the LPMEOH™ Process during the remaining time within the operating program; any improvement in the catalyst performance for the methanol synthesis catalyst will also yield benefits for the LPDME catalyst system.

A draft Topical Report which presents the results of the design verification test at the LaPorte AFDU was sent to DOE. Comments were received, and a revision will be issued for final review and approval by January of 2001.

A separate Topical Report on the market analysis for DME and review of the economics of the LPDME™ Process will be prepared by the LPMEOH™ Demonstration Project following the release of the DVT Topical Report.

D.3 LPMEOH™ Process Demonstration Unit - 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 C 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 5,363,012 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 or injuries were reported during this quarter.

The LPMEOH™ Demonstration Unit operated at 94.45% availability throughout the quarter. There were four short syngas interruptions experienced on 06 August 2000 (33.5 hours), 08 August 2000 (23.5 hours), 21 September 2000 (1.5 hours), and 25 September 2000 (17.5 hours). A planned maintenance turnaround was accomplished on 30 July 2000 to 04 August 2000 for a total of 119 hours. During this outage, maintenance was performed on reactor pressure transmitters, the economizer heat exchanger (29E-02) was inspected and cleaned, and the adsorbent in the catalyst guard bed (29C-40) was changed (although the catalyst guard bed was bypassed for most of the quarter). Appendix D, Table 1 contains the summary of outages for the LPMEOH™ Demonstration Unit during this quarter.

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

Case	Date	Days On Stream	Gas Type	Temp (Deg C)	Pres. (psig)	Fresh Feed (KSCFH)	Recycle Gas (KSCFH)	Reactor Feed (H_2: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 Prod. (TPD)	Catalyst MeOH Prod. (gmol/hr-kg)	Reactor Vol. Prod. (TPD/ft3)	U Overall (Btu hr ft2 F)	Sparger dP (psi)	Sparger Resist. ("K")
11	1-Jul-00	924	Balanced	234	710	684	2,158	3.11	90.5	0.66	3699	42.3	23.7	48.5	45,207	0.290	30.8	19.4	42.6	192.5	11.13	0.095	132	4.36	4.22
11	2-Jul-00	925	Balanced	234	710	685	2,135	3.10	88.3	0.65	3680	42.5	24.2	48.5	45,207	0.291	30.7	19.5	42.6	192.7	11.14	0.095	130	4.25	4.19
11	3-Jul-00	926	Balanced	234	710	687	2,127	3.06	94.5	0.65	3656	42.6	25.3	49.0	45,207	0.289	30.5	19.5	43.1	191.3	11.06	0.093	119	4.14	4.07
11	4-Jul-00	927	Balanced	234	710	663	2,124	3.05	76.5	0.65	3637	42.9	23.9	47.5	45,207	0.290	30.7	19.5	41.7	190.8	11.02	0.096	142	4.35	4.29
11	5-Jul-00	928	Balanced	234	710	667	2,116	3.31	84.8	0.65	3641	40.8	14.0	45.5	45,207	0.285	31.7	18.9	42.6	187.8	10.85	0.098	148	4.3	4.51
11	8-Jul-00	931	Balanced	234	710	670	2,174	3.01	82.8	0.66	3731	44.4	26.4	46.5	45,207	0.279	29.1	18.6	42.0	191.4	11.06	0.098	140	4.52	4.55
11	9-Jul-00	932	Balanced	234	710	683	2,156	3.08	89.5	0.66	3691	42.7	26.3	49.5	45,207	0.284	30.1	18.9	42.6	192.3	11.12	0.093	133	4.15	4.25
11	10-Jul-00	933	Balanced	234	710	677	2,143	3.04	89.5	0.66	3695	42.1	28.9	50.0	45,207	0.286	30.0	19.0	42.1	192.9	11.15	0.092	134	4.25	4.25
11	11-Jul-00	934	Balanced	234	710	682	2,174	2.82	89.3	0.66	3725	40.5	18.3	48.5	45,207	0.279	28.0	18.9	42.6	192.0	11.09	0.094	133	4.82	4.64
11	12-Jul-00	935	Balanced	234	710	675	2,169	2.85	88.1	0.66	3706	42.2	25.7	50.0	45,207	0.277	28.1	18.8	42.6	190.4	11.00	0.091	130	4.18	4.08
11	13-Jul-00	936	Balanced	234	710	668	2,164	2.88	84.8	0.66	3712	43.7	25.2	47.0	45,207	0.274	28.1	18.6	42.5	188.9	10.92	0.096	141	4.55	4.41
11	14-Jul-00	937	Balanced	234	710	679	2,057	3.24	69.9	0.64	3581	41.4	16.6	46.0	45,207	0.279	31.3	19.1	43.4	187.7	10.84	0.097	165	4.35	4.88
11	16-Jul-00	939	Balanced	234	710	681	2,158	2.81	90.3	0.66	3716	42.8	25.9	49.0	45,207	0.276	27.6	18.7	42.9	190.5	11.01	0.093	135	4.36	4.24
11	17-Jul-00	940	Balanced	234	710	669	2,150	2.94	88.0	0.66	3686	42.9	25.5	48.5	45,207	0.273	28.4	18.5	42.6	188.3	10.89	0.093	136	3.8	3.88
11	18-Jul-00	941	Balanced	233	710	643	2,214	2.69	105.0	0.66	3984	41.2	21.5	46.0	42,278	0.265	24.1	16.9	44.2	174.6	10.80	0.091	133	4.55	4.49
11	19-Jul-00	942	Balanced	233	710	614	2,220	2.82	95.3	0.65	4237	43.7	39.7	50.5	39,349	0.276	24.3	16.4	44.0	167.5	11.13	0.079	114	4.4	4.44
11	20-Jul-00	943	Balanced	234	710	655	2,215	3.07	63.6	0.67	4094	42.1	29.4	48.5	41,549	0.316	29.8	18.8	40.8	192.6	12.10	0.095	123	4.41	4.27
11	22-Jul-00	945	Balanced	235	710	676	2,169	3.53	40.7	0.66	3848	42.3	26.9	49.0	43,749	0.361	37.3	20.6	39.3	206.5	12.41	0.100	150	4.4	4.35
11	23-Jul-00	946	Balanced	234	710	676	2,171	3.46	42.6	0.67	3876	42.4	24.7	47.5	43,749	0.350	36.0	20.5	39.3	206.6	12.32	0.104	151	4.53	4.43
11	24-Jul-00	947	Balanced	234	710	684	2,172	3.34	47.8	0.67	3880	42.2	25.1	48.0	43,749	0.342	34.8	20.5	39.6	207.2	12.36	0.103	142	4.46	4.33
11	25-Jul-00	948	Balanced	234	710	713	2,165	3.28	64.6	0.67	3897	43.2	27.8	48.0	43,749	0.342	34.4	20.6	40.7	210.4	12.55	0.104	139	3.98	3.87
11	26-Jul-00	949	Balanced	234	710	787	2,122	3.34	57.2	0.68	3766	43.0	22.0	47.0	45,949	0.392	39.0	23.0	39.7	237.8	13.50	0.121	146	4.8	4.51
11	27-Jul-00	950	Balanced	234	705	808	2,091	3.24	66.3	0.68	3737	43.8	25.3	47.5	45,949	0.392	38.4	23.3	40.3	240.7	13.67	0.121	144	4.72	4.53
11	28-Jul-00	951	Balanced	234	705	812	2,096	3.16	70.6	0.68	3741	44.3	30.9	50.5	45,949	0.394	37.9	23.5	40.3	241.6	13.73	0.114	134	4.53	4.28
11	29-Jul-00	952	Balanced	234	705	816	2,098	3.14	79.5	0.68	3742	44.3	27.5	48.0	45,949	0.392	37.5	23.3	40.7	240.5	13.67	0.119	137	4.46	4.18
11	10-Aug-00	964	Balanced	234	706	622	2,147	4.69	48.3	0.64	3539	42.9	24.2	48.5	45,949	0.322	42.5	19.1	40.3	185.3	10.53	0.083	121	5.88	6.94
11	11-Aug-00	965	Balanced	234	709	618	2,177	4.63	46.3	0.65	3570	43.4	26.5	49.0	45,949	0.317	41.6	18.8	40.4	183.5	10.51	0.082	119	5.87	6.86
11	12-Aug-00	966	Balanced	234	701	641	2,147	4.32	47.1	0.65	3569	44.6	31.1	50.0	45,949	0.332	40.5	19.5	40.5	190.1	10.80	0.084	115	5.9	6.61
11	13-Aug-00	967	Balanced	235	709	668	2,177	4.34	54.2	0.66	3649	44.5	28.7	48.5	45,949	0.331	41.0	19.8	40.7	197.1	11.19	0.087	123	6.05	6.66
11	14-Aug-00	968	Balanced	235	705	708	2,229	3.62	52.9	0.69	3767	44.3	28.1	48.5	45,949	0.342	37.0	20.7	40.2	211.0	11.98	0.090	123	6.78	6.46
11	15-Aug-00	969	Balanced	235	705	750	2,171	3.41	67.4	0.69	3772	44.6	27.5	47.5	45,949	0.354	36.5	21.5	40.6	221.6	12.58	0.096	121	6.78	6.43
11	16-Aug-00	970	Balanced	234	705	760	2,176	3.34	74.0	0.68	3758	44.8	27.2	47.0	45,949	0.365	36.9	22.1	40.4	225.5	12.81	0.098	125	6.9	6.38
11	17-Aug-00	971	Balanced	234	705	760	2,138	3.49	78.9	0.68	3730	44.7	26.8	46.5	45,949	0.364	37.9	22.0	40.9	223.0	12.67	0.098	124	6.64	6.35
11	18-Aug-00	972	Balanced	234	705	735	2,137	3.50	78.4	0.67	3667	44.9	26.5	46.5	45,949	0.348	37.2	21.5	41.1	214.4	12.19	0.094	121	6.56	6.47
11	20-Aug-00	974	Balanced	234	705	743	2,194	3.14	64.5	0.69	3767	44.4	29.1	49.0	45,949	0.354	34.7	21.6	40.0	223.0	12.66	0.098	117	6.98	6.36
11	21-Aug-00	975	Balanced	234	705	739	2,190	3.29	76.9	0.68	3757	44.2	31.5	51.0	45,949	0.342	34.8	21.0	41.1	216.1	12.27	0.095	110	6.85	6.38
11	22-Aug-00	976	Balanced	234	705	735	2,168	3.33	79.6	0.68	3736	44.6	33.2	51.5	45,949	0.340	35.0	20.9	41.3	213.8	12.19	0.094	109	6.73	6.39
11	23-Aug-00	977	Balanced	234	708	727	2,172	3.09	76.7	0.68	3734	45.7	29.7	47.0	45,949	0.321	32.6	20.6	41.0	212.9	12.10	0.094	107	6.77	6.46
11	24-Aug-00	978	Balanced	234	710	670	2,168	3.68	44.1	0.66	3635	44.9	25.8	46.0	45,949	0.332	37.4	20.4	39.4	203.8	11.57	0.091	121	6.28	6.52
11	25-Aug-00	979	Balanced	234	710	656	2,188	3.70	46.6	0.66	3639	42.5	23.7	49.0	45,949	0.319	36.5	19.9	39.6	198.7	11.28	0.088	115	6.24	6.48
11	26-Aug-00	980	Balanced	234	710	655	2,173	3.55	46.1	0.66	3638	43.0	23.8	48.0	45,949	0.315	35.3	19.8	39.8	197.3	11.21	0.098	126	6.32	6.42
11	27-Aug-00	981	Balanced	234	710	655	2,168	3.45	49.0	0.66	3637	43.8	29.7	50.5	45,949	0.302	33.7	19.7	40.0	196.6	10.93	0.087	105	6.41	6.40
11	28-Aug-00	982	Balanced	234	707	692	2,166	3.12	63.4	0.66	3661	44.0	34.9	54.0	45,949	0.314	32.6	20.3	40.5	205.0	11.65	0.090	107	6.72	6.52
11	29-Aug-00	983	Balanced	234	706	732	2,131	3.10	82.8	0.66	3659	44.0	30.2	50.5	45,949	0.321	32.9	20.6	41.7	210.5	11.97	0.099	114	6.55	6.54
11	30-Aug-00	984	Balanced	234	706	738	2,116	3.16	84.0	0.66	3648	42.7	26.0	50.0	45,949	0.326	33.8	21.0	41.6	212.9	12.10	0.101	123	6.46	6.51
11	31-Aug-00	985	Balanced	235	706	730	2,111	3.20	77.6	0.66	3622	42.0	23.2	49.5	45,949	0.326	34.5	21.2	41.2	212.5	12.08	0.102	128	6.39	6.49
11	1-Sep-00	986	Balanced	235	706	726	2,123	3.19	76.5	0.66	3634	42.1	22.7	49.0	45,949	0.326	34.3	21.1	41.0	212.4	12.07	0.094	118	6.45	6.45
11	2-Sep-00	987	Balanced	235	706	725	2,123	3.09	75.7	0.66	3647	42.4	23.6	49.0	45,949	0.327	33.7	21.1	41.1	211.6	12.13	0.092	116	6.64	6.39
11	3-Sep-00	988	Balanced	234	706	726	2,138	3.13	85.2	0.66	3654	42.9	25.7	49.5	45,949	0.316	33.1	20.8	41.8	208.4	11.85	0.100	124	6.58	6.37
11	4-Sep-00	989	Balanced	234	706	726	2,127	3.17	96.6	0.66	3651	43.5	28.1	50.0	45,949	0.308	32.7	20.4	42.5	204.7	11.64	0.098	118	6.49	6.43
11	5-Sep-00	990	Balanced	234	706	735	2,147	3.12	104.6	0.67	3694	43.7	29.5	50.5	45,949	0.303	31.8	20.1	43.1	204.4	11.63	0.096	114	6.58	6.41
11	6-Sep-00	991	Balanced	235	709	705	2,169	3.27	77.0	0.67	3708	41.1	19.6	49.0	45,949	0.304	33.1	20.1	41.4	204.2	11.60	0.099	137	7.58	6.45

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

Case	Date	Days On Stream	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 Prod. (TPD)	Catalyst MeOH Prod. (gmol/hr-kg)	Reactor Vol. Prod. (TPD/ft3)	U Overall (Btu hr ft2 F)	Sparger dP (psi)	Sparger Resist. ("K")
11	7-Sep-00	992	Balanced	235	707	705	2,156	3.17	81.0	0.67	3696	41.4	25.9	52.5	45,949	0.304	32.6	20.2	41.6	203.1	11.55	0.092	128	6.68	6.43
11	8-Sep-00	993	Balanced	235	707	697	2,156	3.15	79.4	0.67	3670	41.4	25.9	52.5	45,949	0.298	31.9	19.9	41.7	200.6	11.41	0.091	127	6.68	6.45
11	9-Sep-00	994	Balanced	235	707	696	2,140	3.13	84.5	0.66	3642	42.1	24.1	50.0	45,949	0.292	31.5	19.8	42.2	198.0	11.19	0.094	125	6.63	6.42
11	10-Sep-00	995	Balanced	235	707	692	2,151	3.11	88.1	0.66	3637	42.5	24.7	49.5	45,949	0.290	31.2	19.6	42.4	195.6	11.14	0.094	123	6.64	6.44
11	11-Sep-00	996	Balanced	235	707	688	2,156	3.06	89.7	0.66	3646	42.8	25.5	49.5	45,949	0.287	30.5	19.4	42.6	194.0	11.04	0.093	121	6.67	6.36
11	12-Sep-00	997	Balanced	234	708	684	2,157	3.01	91.7	0.66	3641	43.4	27.9	50.0	45,949	0.283	29.8	19.2	42.8	191.7	10.91	0.091	116	6.72	6.37
11	13-Sep-00	998	Balanced	234	708	687	2,149	2.98	91.7	0.66	3640	43.4	27.8	50.0	45,949	0.284	29.8	19.3	42.8	192.7	10.97	0.092	118	6.71	6.33
11	14-Sep-00	999	Balanced	234	708	683	2,148	3.03	97.1	0.66	3632	43.9	29.9	50.5	45,949	0.281	29.9	19.2	43.1	190.3	10.84	0.090	115	6.62	6.31
11	15-Sep-00	1000	Balanced	234	710	666	2,198	3.08	90.0	0.66	3675	44.5	32.1	51.0	45,949	0.274	29.4	18.7	42.6	187.5	10.67	0.088	113	6.65	6.34
11	16-Sep-00	1001	Balanced	234	710	661	2,229	3.08	91.7	0.67	3697	44.8	33.0	51.0	45,949	0.266	28.7	18.3	42.9	184.8	10.51	0.086	111	6.61	6.35
11	17-Sep-00	1002	Balanced	235	710	675	2,180	3.02	85.7	0.66	3669	42.4	25.8	50.5	45,949	0.271	29.2	18.9	42.6	190.1	10.81	0.090	124	6.60	6.39
11	19-Sep-00	1004	Balanced	234	710	599	2,240	2.87	111.9	0.66	4614	39.3	24.3	44.0	36,163	0.273	23.3	15.7	45.6	157.7	11.41	0.086	142	6.54	6.45
11	20-Sep-00	1005	Balanced	234	711	579	2,186	2.94	108.8	0.64	4504	39.4	26.3	45.0	36,163	0.263	23.7	15.7	45.3	153.4	11.03	0.081	126	6.34	6.39
11	22-Sep-00	1007	Balanced	235	710	634	2,140	3.69	86.4	0.64	4248	39.9	31.0	50.0	38,363	0.317	33.0	18.4	42.8	177.6	12.11	0.085	149	5.94	6.53
11	27-Sep-00	1012	Balanced	235	706	817	2,126	3.89	62.0	0.69	3859	42.2	32.8	55.0	44,963	0.458	45.7	24.3	40.4	242.8	14.10	0.105	174	5.89	5.34
11	28-Sep-00	1013	Balanced	235	706	851	2,095	3.52	65.7	0.69	3866	41.4	30.1	54.5	44,963	0.458	43.6	24.9	40.0	255.5	14.84	0.112	160	5.81	5.34
11	30-Sep-00	1015	Balanced	230	706	676	2,122	4.57	33.1	0.64	3636	41.8	26.8	51.0	44,963	0.451	47.8	21.8	39.0	208.2	12.30	0.097	167	4.86	5.64

Catalyst Life (eta) – July – September 2000

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 D, Figure 1 plots log η versus days onstream from September of 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.

Two catalyst withdrawal and addition campaigns were undertaken during the quarter. A catalyst withdrawal and addition campaign was undertaken during the month of July 2000 to raise catalyst activity on an interim basis prior to the changeout of the adsorbent in the catalyst guard bed within the LPMEOH™ Demonstration Unit. Two batches of aged catalyst were removed on 17 and 18 July 2000. Three batches of fresh catalyst were activated and added to the LPMEOH™ Reactor between 20 and 25 July 2000.

A major catalyst withdrawal and addition campaign was undertaken during the month of September 2000 to raise catalyst activity. A series of four withdrawals were conducted on 18 and 19 September 2000. At the end of the reporting period, three batches of fresh catalyst were activated and added to the LPMEOH™ Reactor between 21 and 24 September 2000. Plans for the next quarter called for the activation and addition of two more batches of fresh catalyst.

During most of the quarter, the flowrate of Balanced Gas was controlled at an average flowrate of 697 KSCFH. During these operating periods, the reactor pressure was set at between 705 and 710 psig and temperature was maintained at 235ºC.

There were two extended periods of operation (minimum of about 2 weeks) at a reactor temperature of 235°C during which catalyst activity was measured to track catalyst deactivation during the quarter. An overall deactivation rate of 0.85% per day was calculated for the period 24 June to 05 July 2000 (12 days). This data was based on operation with the catalyst guard bed in service with aged adsorbents (manganese dioxide and activated carbon). An overall deactivation rate of 0.76% per day was calculated for the period 15 August 2000 to 11 September 2000 (28 days). This data was based on operation with the catalyst guard bed bypassed and out of service. The fact that the rate of deactivation is statistically the same for these two cases supports the hypothesis that the adsorbent materials in the catalyst guard bed had become ineffective at removing trace contaminants from the syngas streams supplying the LPMEOH™ Demonstration Unit. These deactivation results are greater than the baseline deactivation rate of 0.4% per day from the 4-month proof-of-concept run at the LaPorte AFDU in 1988/89 (this run was performed at 250°C), and may reflect the impact of poisons on catalyst aging.

Analyses of catalyst samples for changes in physical characteristics and levels of poisons have continued. Appendix D, Table 2 summarizes the results to date. Samples have continued to show an increase in arsenic (to around 1,250 ppmw), which has been demonstrated in the laboratory to act as a poison to methanol synthesis catalyst. Sulfur, another known catalyst poison, continues to increase (present levels are at about 350 ppmw). Copper crystallite size measurements have decreased in the most recent samples, and may reflect a higher concentration of less aged catalyst in the sample. Levels of nickel (a known catalyst poison) have remained low and steady since the restart in December of 1997. The concentration of iron (another poison) is low (less than 200 ppmw), and has stabilized in the most recent samples.

Eastman had previously accepted a recommendation by Air Products to use a commercially available copper-impregnated activated carbon to replace the manganese oxide which is currently used in the catalyst guard bed within the LPMEOH™ Demonstration Unit. The catalyst guard bed was filled with a commercially available activated carbon adsorbent on 02 August 2000. This material is chemically treated with copper oxide to enhance its arsine removal capabilities. This adsorbent was selected after investigative work in the laboratory and at Kingsport (via a slip-stream apparatus) was performed to evaluate the effectiveness of the material and to address questions on potential interaction with the components of the syngas stream.

The catalyst guard bed was operationally tested on 03 August 2000. After the catalyst guard bed was purged with nitrogen, Balanced Gas was introduced to the closed vessel in order to leak-check the flanges which had been opened during the maintenance activities. Within 30 minutes of the introduction of syngas, temperatures in the catalyst guard bed began to rise beyond the expected values based upon the results of the laboratory testing. The Eastman operations team moved quickly to depressurize the catalyst guard bed by venting the syngas to the Eastman purge gas header, and the guard bed internal pressure did not reach the set pressure for the safety relief valve on the vessel. No environmental issues were experienced due to this incident. The catalyst guard bed was then isolated from the feed syngas streams to allow the LPMEOH™ Demonstration Unit to be returned to operation.

The results of additional laboratory testing have indicated that the probable cause of this temperature excursion was the reduction of copper oxide to copper metal. Preliminary engineering calculations and subsequent laboratory experiments have indicated that this is the only likely source of the magnitude of the heat rise which was experienced. Laboratory tests have shown the reduction of the copper oxide on the activated carbon substrate can occur rapidly and at low temperatures. A meeting was held between Air Products and Eastman on 21 September 2000 to discuss the results of this testing. A plan to bring the catalyst guard bed back into service, including modifications which will be necessary to complete the reduction of copper oxide within the catalyst guard bed, is being developed; the finalized version will be reviewed with DOE prior to implementation.

In-situ Catalyst Activation

Discussions were held between Air Products and Eastman to initiate the design engineering activities in support of the proposed in-situ activation of methanol synthesis catalyst at the LPMEOH™ Demonstration Facility. Piping and instrumentation are needed to allow for the introduction of the Balanced Gas and nitrogen streams which will be required to reduce the catalyst.

Sparger Resistance

After the restart of the LPMEOH™ Demonstration Facility on 04 August 2000, the sparger flow resistance increased approximately 50%. Subsequent flushing of the sparger with clean oil resulted in a sizable reduction of the increased flow resistance (from an average resistance coefficient of 6.6 to an average of 5.3). In addition, the resistance does not appear to be increasing over time; longer-term performance of the sparger will be closely monitored to verify these observations. Appendix D, Figure 2 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.

D.4 Planning and Administration

A 15-month, no-cost time extension (from 31 December 2001 to 31 March 2003) to the Cooperative Agreement, was approved by the DOE on 24 April 2000, and was accepted by Air Products on behalf of the Partnership on 08 May 2000. This extension is necessary to complete some of the key tests which were originally defined in the September 1996 Demonstration Test Plan, and to allow the opportunity to perform new tests of significant commercial interest. During this reporting period, an update to the Demonstration Test Plan which defined the tests which are planned for the remainder of the operating program was sent to DOE for review and comment.

The Milestone Schedule Status Report and the Cost Management Report, through the period ending 30 September 2000, are included in Appendix E. 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 2000. Sixty-four percent (64%) of the $158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 September 2000.

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 paper entitled “Liquid Phase Methanol (LPMEOH™) Process Development” was presented at Energex 2000 - The 8th International Energy Forum (23-28 July 2000).
A paper entitled “Catalyst and Process Development for Liquid Phase DME Synthesis” was presented at 17th Annual International Pittsburgh Coal Conference (11-15 September 2000).

Eastman has recently identified a change to the requirements for compliance monitoring of the purge gas stream from the LPMEOH™ Demonstration Unit. The Environmental Monitoring Plan (EMP) was modified to reflect this change; DOE subsequently suggested that supplemental monitoring should be performed on this stream and the results should be included in a future Environmental Monitoring Report. The Partnership accepted this recommendation, and those pages which were impacted by this change were reissued for inclusion into the EMP.

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, monitoring the performance of the gas sparger in the reactor, and completing the design for changes for in-situ catalyst activation and pre-treatment of the copper oxide-impregnated activated carbon in the 29C-40 catalyst guard bed.
- Reply to comments from DOE on the update to the Demonstration Test Plan.
- Continue execution of the Off-Site, Product-Use Test Program.
- Conduct a Project Review Meeting with DOE.

F. Conclusion

The LPMEOH^{TM} Demonstration Unit operated at 94.45% availability throughout the quarter. There were four short syngas interruptions experienced which totaled 76 hours. A planned maintenance turnaround was accomplished on 30 July 2000 to 04 August 2000 for a total of 119 hours. During this outage, maintenance was performed on reactor pressure transmitters, the economizer heat exchanger was inspected and cleaned, and the adsorbent in the catalyst guard bed was changed (although the catalyst guard bed was bypassed for most of the quarter).

Two catalyst withdrawal and addition campaigns were undertaken during the quarter. A catalyst withdrawal (2 batches) and addition (3 batches) campaign was undertaken between 17 and 25 July 2000 to raise catalyst activity on an interim basis prior to the changeout of the adsorbent in the catalyst guard bed within the LPMEOH™ Demonstration Unit. In the second campaign, a series of four withdrawals and three activations and additions were conducted between 18 and 24 September 2000. Plans for the next quarter called for the activation and addition of two more batches of fresh catalyst.

During most of the quarter, the flowrate of Balanced Gas was controlled at an average flowrate of 697 KSCFH. During these operating periods, the reactor pressure was set at between 705 and 710 psig and temperature was maintained at 235ºC.

There were two extended periods of operation (minimum of about 2 weeks) at a reactor temperature of 235°C during which catalyst activity was measured to track catalyst deactivation during the quarter. An overall deactivation rate of 0.85% per day was calculated for the period 24 June to 05 July 2000 (12 days). This data was based on operation with the catalyst guard bed in service with aged adsorbents (manganese dioxide and activated carbon). An overall deactivation rate of 0.76% per day was calculated for the period 15 August 2000 to 11 September 2000 (28 days). This data was based on operation with the catalyst guard bed bypassed and out of service. These deactivation results are greater than the baseline deactivation rate of 0.4% per day from the 4-month proof-of-concept run at the LaPorte AFDU in 1988/89 (this run was performed at 250°C), and may reflect the impact of poisons on catalyst aging. These results are statistically the same and also indicate that the aged adsorbents in the catalyst guard bed were no longer effective in removing trace contaminants.

Analyses of catalyst samples for changes in physical characteristics and levels of poisons have continued. Samples have continued to show an increase in arsenic, which has been demonstrated in the laboratory to act as a poison to methanol synthesis catalyst. Sulfur, another known catalyst poison, continues to be measured above the analytical detection limit. Copper crystallite size measurements have decreased over the last few samples. Levels of nickel (a known catalyst poison) have remained low and steady since the restart in December of 1997. The concentration of iron (another poison) is low (less than 200 ppmw), and has stabilized in the most recent samples.

Eastman had previously accepted a recommendation by Air Products to use a commercially available copper-impregnated activated carbon in the catalyst guard bed within the LPMEOH™ Demonstration Unit. This material is chemically treated with copper oxide to enhance its arsine removal capabilities. This adsorbent was selected after investigative work in the laboratory and at Kingsport (via a slip-stream apparatus) was performed to evaluate the effectiveness of the material and to address questions on potential interaction with the components of the syngas stream.

After the loading operation was completed, the catalyst guard bed was operationally tested on 03 August 2000. After the catalyst guard bed was purged with nitrogen, Balanced Gas was introduced to the closed vessel in order to leak-check the flanges which had been opened during the maintenance activities. Within 30 minutes of the introduction of syngas, temperatures in the catalyst guard bed began to rise beyond the expected values based upon the results of the laboratory testing. The Eastman operations team moved quickly to depressurize the catalyst guard bed by venting the syngas to the Eastman purge gas header. No environmental issues were experienced due to this incident. The catalyst guard bed was then isolated from the feed syngas streams to allow the LPMEOH™ Demonstration Unit to be returned to operation.

The results of additional laboratory testing have indicated that the probable cause of this temperature excursion was the reduction of copper oxide to copper metal. Preliminary engineering calculations and subsequent laboratory experiments have indicated that this is the only likely source of the magnitude of the heat rise which was experienced. A meeting was held between Air Products and Eastman on 21 September 2000 to discuss the results of this testing. A plan to bring the catalyst guard bed back into service, including modifications which will be necessary to complete the reduction of copper oxide within the catalyst guard bed, is being developed; the finalized version will be reviewed with DOE prior to implementation.

Discussions were held between Air Products and Eastman to initiate the design engineering activities in support of the proposed in-situ activation of methanol synthesis catalyst at the LPMEOH™ Demonstration Facility. Piping and instrumentation are needed to allow for the introduction of the Balanced Gas and nitrogen streams which will be required to reduce the catalyst.

The performance of the 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. After the restart of the LPMEOH™ Demonstration Facility on 04 August 2000, the sparger flow resistance increased approximately 50%. Subsequent flushing of the sparger with clean oil resulted in a recovery of about ½ of the earlier increase. In addition, the resistance does not appear to be increasing over time; longer-term performance of the sparger will be closely monitored to verify these observations.

During the reporting period, a total of 5,363,012 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, about 64.2 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 safety or environmental incidents were reported during this quarter.

During this quarter, planning, procurement, and test operations continued on one of the project sites selected for the off-site, product-use test program. A draft of the paper entitled “Lubricity Problems and Solutions for a Methanol Fueled Gas Turbine” was sent to DOE; this paper was accepted for presentation at the International Mechanical Engineering Conference and Exposition in Orlando, FL (05-10 November 2000). Results of experiments in the reformer test apparatus have provided convincing evidence that the performance of the low temperature catalyst is adversely affected by the presence of the trace mineral oil in the stabilized methanol. The performance of stabilized methanol in a higher-temperature steam reforming process may yield increased conversion and selectivity which are required for the fuel cell application.

Activities associated with Design Verification Testing of the LPDME Process have been completed. A draft Topical Report which presents the results of the design verification test at the LaPorte AFDU was sent to DOE. Comments were received, and a revision will be issued for final review and approval by January of 2001. A separate Topical Report on the market analysis for DME and review of the economics of the LPDME™ Process will be prepared by the LPMEOH™ Demonstration Project following the release of the DVT Topical Report.

During this reporting period, an update to the Demonstration Test Plan which defined the tests which are planned for the remainder of the operating program was sent to DOE for review and comment.

The paper entitled “Liquid Phase Methanol (LPMEOH™) Process Development” was presented at Energex 2000 - The 8th International Energy Forum (23-28 July 2000).
A paper entitled “Catalyst and Process Development for Liquid Phase DME Synthesis” was presented at 17th Annual International Pittsburgh Coal Conference (11-15 September 2000).

Eastman has recently identified a change to the requirements for compliance monitoring of the purge gas stream from the LPMEOH™ Demonstration Unit. The Environmental Monitoring Plan (EMP) was modified to reflect this change; DOE subsequently suggested that supplemental monitoring should be performed on this stream and the results should be included in a future Environmental Monitoring Report. The Partnership accepted this recommendation, and those pages which were impacted by this change were reissued for inclusion into the EMP.

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 2000. Sixty-four percent (64%) of the $158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 September 2000.

APPENDICES

APPENDIX A - SIMPLIFIED PROCESS FLOW DIAGRAM

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

Quarterly Report:

University of Florida Fuel Cell (six pages)

APPENDIX C - SAMPLES OF DETAILED MATERIAL BALANCE REPORTS

APPENDIX D - RESULTS OF DEMONSTRATION UNIT OPERATION

Table 1 - Summary of LPMEOH™ Demonstration Unit Outages - July/September 2000
Table 2 - Summary of Catalyst Samples - Second Catalyst Batch

Figure 1 - Catalyst Age (η): July - September 2000
Figure 2 - Sparger Resistance Coefficient vs. Days Onstream (August 1999 - September 2000)

Table 1
Summary of LPMEOH™ Demonstration Unit Outages - July/September 2000

Operation Start	Operation End	Operating Hours	Shutdown Hours	Reason for Shutdown
7/1/00 00:00	7/30/00 16:03	712.0	118.9	Planned Outage - Maintenance
8/4/00 14:57	8/6/00 07:20	40.4	33.5	Syngas Outage
8/7/00 16:50	8/8/00 16:28	23.6	23.2	Syngas Outage
8/9/00 15:38	9/21/00 11:11	1027.5	1.7	Syngas Outage
9/21/00 12:52	9/25/00 02:40	85.8	5.5	Syngas Outage
9/25/00 08:10	9/30/00 23:59	135.8		End of Reporting Period
	Total Operating Hours		2025.2	
	Syngas Available Hours		2144.1	
	Plant Availability, %		**94.45**	

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	56	161	15.1	406	1470	50
K9909-2	Reactor Sample 9/21/99	357	109	64	132	11.2	253	1050	nd
K9910-2	Reactor Sample 10/19/99	135	94	55	157	15.4	343	1270	30
K9911-1	Reactor Sample 11/4/99				184	12.8	335	1580	na
K9912-1	Reactor Sample 12/8/99	797	121	60	167	13.9	248	1400	40
K0001-1	Reactor Sample 1/5/00	613	105	63	199	10.8	292	1190	nd
	Reactor Sample 1/19/00				205	10.0	432	1250	na
	Reactor Sample 3/2/00	187	88.7	67	137	8.2	226	1010	30
	Reactor Sample 4/23/00	175	114.5	59	164	6.6	248	1240	20
K0001-2	Reactor Sample 7/18/00	174	107.5	69	166	na	349	1270	30

Notes:

1) nd = none detected
2) * - these values represent re-analysis of the sample as compared to Technical Progress Report no. 17
3) na = data not available

Figure 1 - Kingsport LPMEOH^{TM} Catalyst Age (eta): September 1999 - September 2000

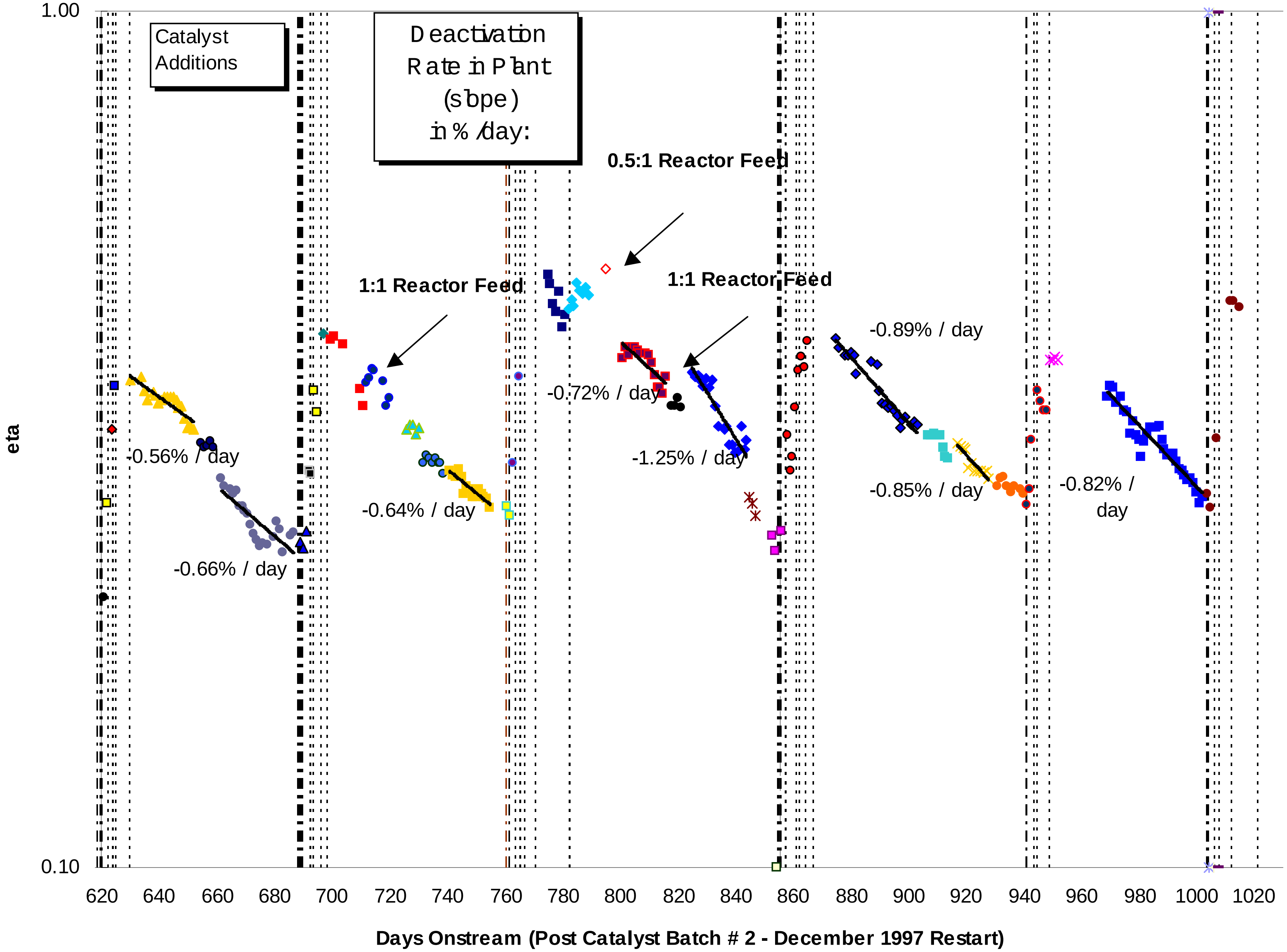
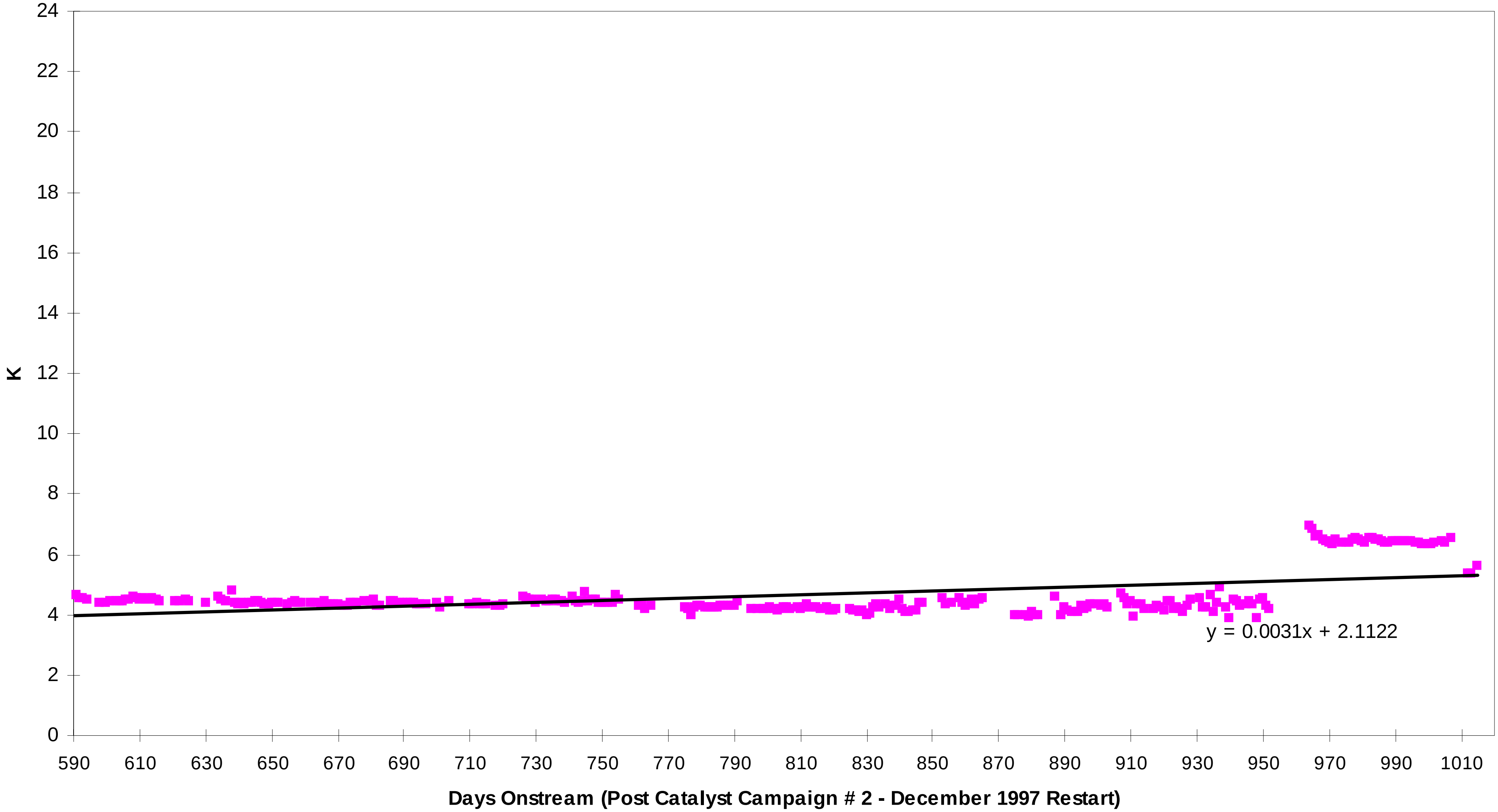


Figure 2 - Kingsport LPMEOH^{TM}
Sparger Resistance Coefficient



APPENDIX E - MILESTONE SCHEDULE STATUS AND COST MANAGEMENT REPORTS