

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

TECHNICAL PROGRESS REPORT NO. 18

For The Period

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and

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for the

Air Products Liquid Phase Conversion Company, L.P.

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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 achieved a new milestone during the reporting period by completing the longest continuous operating run without interruption of any kind on 27 October 1998 (94 days total). The LPMEOH™ Demonstration Unit operated at 100% availability throughout the quarter and completed a very successful 1998, with a year-end availability of 99.72%.

Six batches of fresh methanol catalyst were activated and added to the reactor during the reporting period. As of 31 December 1998, the catalyst loading in the LPMEOH™ Reactor had been increased to about 140 % of design. No hydrodynamic instability was detected as the catalyst slurry concentration in the reactor ranged from 46 - 48 wt%.

Catalyst activity, as defined by the ratio of the rate constant at any point in time to the rate constant for freshly reduced catalyst (as determined in the laboratory autoclave), was monitored throughout the reporting period. Catalyst deactivation was evaluated over the 25 day period encompassing 02 October 1998 to 26 October 1998. Operating conditions for this campaign were a reactor temperature of 235°C and a flowrate of the primary syngas feed (Balanced Gas) of 700 - 800 KSCFH. During this operating period, the rate of decline in catalyst activity was 0.7% per day. For reference, the original target from the 4-month proof-of-concept run at the LaPorte Alternative Fuels Development Unit (AFDU) in 1988/89 was 0.4% per day. During much of the remainder of the reporting period, there were no extended operating periods which are needed to track changes in catalyst activity.

During several days in November of 1998, unit operation was performed with both Balanced Gas and a carbon-monoxide-rich syngas (CO Gas) as the make-up streams. This resulted in a hydrogen to CO (H₂/CO) ratio at the reactor inlet ranging from 1.88 to 2.3.

Catalyst slurry samples from the LPMEOH™ Reactor have been taken on a regular basis to correlate any change in unit performance with changes in the physical properties of the catalyst. An increase in arsenic loading continues to be the most significant change in levels of known catalyst poisons on catalyst samples which were analyzed during the reporting period. No correlation to date between the change in arsenic readings and catalyst performance has been identified.

The performance of the alternative gas sparger, which was designed by Air Products and installed into the LPMEOH™ Reactor prior to the restart of the LPMEOH™ Demonstration Unit in December of 1997, was monitored. During the reporting period, increases in the resistance coefficient for the gas sparger were detected around the time of maintenance

activities on the flush system for the pressure drop measurement and of an extended syngas outage. As a result of the greater sparger pressure drop, condensed oil and entrained slurry could not be gravity-drained to the flush connection at the gas inlet line to the reactor. In order to overcome the pressure drop, the streams were batch-transferred to the catalyst reduction vessel and returned to the reactor via the slurry transfer pump.

During the reporting period, a total of 4,962,109 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, over 30.7 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 the seven project sites selected for the off-site, product-use test program. Two flexible-fuel vehicles were operated over 3,200 miles on fuel-grade methanol from the LPMEOH™ Demonstration Project. Emissions testing of methanol as an emulsion fuel in a flight line generator showed reductions in nitrogen oxides of 40 - 60% when compared with operation on jet fuel.

During the reporting period, planning for a design verification test run of the Liquid Phase Dimethyl Ether (LPDME™) Process at the LaPorte AFDU continued. Air Products has been performing laboratory autoclave tests of samples of the dehydration catalyst from the commercial catalyst manufacturer (Engelhard). The results to date have not been consistent, indicating that all issues related to catalyst scale-up and commercial-scale production have not been resolved. The results of a set of experiments on a commercially available dehydration catalyst showed that the desired catalyst life could be achieved with the commercially available dehydration catalyst at a 10-20% reduction in system productivity. Additional laboratory experiments must be completed before a new date for the start of the campaign at the LaPorte AFDU will be selected.

Ninety-nine percent (99%) of the \$38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 31 December 1998. Thirty-three percent (33%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 December 1998.

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ACRONYMS AND DEFINITIONS

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

ACRONYMS AND DEFINITIONS (cont'd)

ρ	-	density, pounds per cubic foot
Partnership	-	Air Products Liquid Phase Conversion Company, L.P.
PDU	-	Process Development Unit
PFD	-	Process Flow Diagram(s)
ppbv	-	parts per billion (volume basis)
ppmw	-	parts per million (weight basis)
Project	-	Production of Methanol/DME Using the LPMEOH™ Process at an Integrated Coal Gasification Facility
psi	-	Pounds per Square Inch
psia	-	Pounds per Square Inch (Absolute)
psig	-	Pounds per Square Inch (gauge)
P&ID	-	Piping and Instrumentation Diagram(s)
Raw Methanol	-	sum of Refined Grade Methanol and Crude Grade Methanol; represents total methanol which is produced after stabilization
Reactor Feed	-	sum 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 complex, the technology is integrated with existing coal gasifiers. A carefully developed test plan will allow operations at Eastman to simulate electricity demand load-following in coal-based IGCC facilities. The operations will also demonstrate the enhanced stability and heat dissipation of the conversion process, its reliable on/off operation, and its

ability to produce methanol as a clean liquid fuel without additional upgrading. An off-site, product-use test program will be conducted to demonstrate the suitability of the methanol product as a transportation fuel and as a fuel for stationary applications for small modular electric power generators for distributed power.

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

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

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

The LPMEOH™ Demonstration Unit achieved a new milestone during the reporting period by completing the longest continuous operating run without interruption of any kind on 27 October 1998 (94 days total). The LPMEOH™ Demonstration Unit operated at 100% availability throughout the quarter and completed a very successful 1998, with a year-end availability of 99.72%.

The final batch of fresh baseline methanol catalyst was activated and added to the LPMEOH™ Reactor on 02 October 1998. Five batches of the fresh alternative catalyst batches were activated and added during the reporting period. As of 31 December 1998, the catalyst loading in the reactor had been increased to about 140 % of design. No

hydrodynamic instability was detected as the catalyst slurry concentration in the reactor ranged from 46 - 48 wt%.

Catalyst activity, as defined by the ratio of the rate constant at any point in time to the rate constant for freshly reduced catalyst (as determined in the laboratory autoclave), was monitored throughout the reporting period. Catalyst deactivation was evaluated over the 25 day period encompassing 02 October 1998 to 26 October 1998. Operating conditions for this campaign were a reactor temperature of 235°C and a flowrate of the primary syngas feed (Balanced Gas) of 700 - 800 KSCFH. During this operating period, the rate of decline in catalyst activity was 0.7% per day. For reference, the original target from the 4-month proof-of-concept run at the LaPorte AFDU in 1988/89 was 0.4% per day. During much of the remainder of the reporting period, there were no extended operating periods which are needed to track changes in catalyst activity.

During several days in November of 1998, unit operation was performed with both Balanced Gas and a carbon-monoxide-rich syngas (CO Gas) as the make-up streams. This resulted in a hydrogen to CO (H_2/CO) ratio at the reactor inlet ranging from 1.88 to 2.3.

Catalyst slurry samples from the LPMEOH™ Reactor have been taken on a regular basis to correlate any change in unit performance with changes in the physical properties of the catalyst. Samples have continued to show an increase in arsenic loading, with levels in excess of the concentrations measured on the initial charge of methanol synthesis catalyst from 1997. No correlation to date between the change in arsenic readings and catalyst performance has been identified. Sulfur has been measured at the analytical detection limit. Levels of iron and nickel have remained steady since the restart in December of 1997. Copper crystallite size measurements have shown an increase in the most recent samples.

The performance of the alternative gas sparger, which was designed by Air Products and installed into the LPMEOH™ Reactor prior to the restart of the LPMEOH™ Demonstration Unit in December of 1997, was monitored. During the reporting period, a step-change in the resistance coefficient for the gas sparger was detected around the time when maintenance was performed on the flush system that cleans the pressure taps for the pressure drop measurement. Another increase in resistance occurred around an extended syngas outage in November of 1998. As a result of the greater sparger pressure drop, condensed oil and entrained slurry could not be gravity-drained to the flush connection at the gas inlet line to the reactor. In order to overcome the pressure drop, the streams were batch-transferred to the catalyst reduction vessel and returned to the reactor via the slurry transfer pump. During periods when the catalyst reduction vessel is otherwise in use, the condensed oil and entrained slurry were free-drained directly to a connection on the reactor where the pressure drop was not limiting.

During the reporting period, a total of 4,962,109 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, over 30.7 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 the seven project sites selected for the off-site, product-use test program. Two flexible-fuel vehicles were operated over 3,200 miles on fuel-grade methanol from the LPMEOH™ Demonstration Project. Emissions testing of methanol as an emulsion fuel in a flight line generator showed reductions in nitrogen oxides of 40 - 60% when compared with operation on jet fuel. A proposal was submitted to conduct additional testing of fuel-grade methanol in a stationary gas turbine; goals of this work include the improvement of the lubricity of the methanol and an increase in the power output from the gas turbine. A reformer test apparatus to determine the operating characteristics of fuel-grade methanol as a feed to a fuel cell is under construction.

During the reporting period, planning for a design verification test run of the Liquid Phase Dimethyl Ether (LPDME™) Process at the LaPorte AFDU continued. Air Products has been performing laboratory autoclave tests of samples of the dehydration catalyst from the commercial catalyst manufacturer (Engelhard). The results to date have not been consistent, indicating that all issues related to catalyst scale-up and commercial-scale production have not been resolved. A set of experiments was performed on a commercially available dehydration catalyst. These results showed that the desired catalyst life could be achieved with the commercially available dehydration catalyst at a 10-20% reduction in system productivity (primarily a reduction in the selectivity to DME). Additional laboratory experiments must be completed before a new date for the start of the campaign at the LaPorte AFDU will be selected. The DOE's Liquid Fuels Program has targeted 01 October 1999 as the anticipated start date for the AFDU design verification test.

A paper entitled "Liquid Phase Methanol (LPMEOH™) Project Operational Experience" was presented at the Gasification Technologies Conference on 04-07 October 1998. The topical report entitled "Design and Fabrication of the First Commercial-Scale LPMEOH™ Reactor" was issued to DOE.

Ninety-nine percent (99%) of the \$38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 31 December 1998. Thirty-three percent (33%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 December 1998.

A. Introduction

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

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

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

B. Project Description

The demonstration unit, which occupies an area of 0.6 acre, is integrated into the existing 4,000-acre Eastman complex located in Kingsport, Tennessee. The Eastman complex employs approximately 12,000 people. In 1983, Eastman constructed a coal gasification facility utilizing Texaco technology. The synthesis gas (syngas) generated by this gasification facility is used to produce carbon monoxide and methanol. Both of these products are used to produce methyl acetate and ultimately cellulose acetate and acetic acid. The availability of this highly reliable coal gasification facility was the major factor in selecting this location for the LPMEOH™ Process Demonstration. Three different feed gas streams (hydrogen gas or H₂ Gas, carbon monoxide gas or CO Gas, and the primary syngas feed known as Balanced Gas) are diverted from existing operations to the LPMEOH™ Demonstration Unit, thus providing the range of coal-derived syngas ratios (hydrogen to carbon monoxide) needed to meet the technical objectives of the demonstration project.

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

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

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

- *Reaction Area*

The reaction area includes feed gas compressors, catalyst guard beds, the reactor, a steam drum, separators, heat exchangers, and pumps. The equipment is supported by a matrix of

structural steel. The most salient feature is the reactor, since with supports, it is approximately 84-feet tall.

- *Purification Area*

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

- *Catalyst Preparation Area*

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

- *Storage/Utility Area*

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

C. Process Description

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

D. Results and Discussion

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

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

Discussion

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

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

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

Activity during this quarter

Eight sites involving a variety of product-use tests have been selected to participate in this task. In a letter to the DOE dated 31 July 1997, Air Products formally recommended that seven of the eight projects had been defined in sufficient detail so that final planning and implementation should begin. DOE accepted Air Products' recommendation to proceed with

the seven projects in August of 1997. The sites and project titles are listed in Appendix B-1. The eighth project, involving the testing of a water/naphtha/methanol emulsion as a transportation fuel, was removed from the Product-Use Test Program during a review meeting between DOE, Air Products, and ARCADIS, Geraghty & Miller.

A review meeting on the full product-use program was held on 14 October 1998 in Morgantown, WV. The status of each of the projects was presented, and a tour of the West Virginia University Stationary Gas Turbine Test Site and Transportable Heavy Duty Emissions Testing Laboratory was given. The meeting minutes are included in Appendix B-2.

All of the remaining product-use test projects have begun planning and equipment procurement. Methanol produced from carbon monoxide (CO)-rich syngas at the LaPorte AFDU has been shipped to three of the project sites. Appendix B-3 through B-6 contain summary reports from the approved active projects. Highlights from these reports include:

Acurex Flexible-Fuel Vehicle (FFV) - The FFV has completed the operating phase of the project, having accumulated 3,400 miles on M85 made from methanol supplied from the inventory at the LaPorte AFDU and 3,200 miles on M85 made from chemical-grade methanol. A preliminary draft of the final project report was submitted to Air Products in October of 1998.

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

West Virginia University (WVU) Stationary Gas Turbine - A draft final report on the results of this project was prepared. A proposal was submitted to Air Products on 01 December 1998 to perform additional experiments which will improve the lubricity of the methanol and increase the range of power output from the turbine. Emissions testing on the gas turbine apparatus will also be performed.

Aircraft Ground Equipment Emulsion - Initial testing of methanol as an emulsion fuel in a flight line generator showed reductions in nitrogen oxides compared with operation on jet fuel that were lower than the expected results; additional tests were consistent with project expectations (40% at high output, over 60% at low load). Further evaluation of both tests is underway to explain the difference in these results.

University of Florida Fuel Cell - Air Products accepted a proposal to conduct a small-scale reformer test to compare the behavior of chemical-grade methanol with fuel-grade methanol from the LPMEOH™ Demonstration Project. Equipment procurement is underway.

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

Florida Institute of Technology Bus & Light Vehicle - The two light vehicles were operated a total of 3,279 miles during the reporting period over a variety of methanol and gasoline fuel

formulations (from M-15 to M-100). Fuel-grade methanol from the LPMEOH™ Demonstration Project was used to operate the vehicles.

D.2 Commercialization Studies

Discussion

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

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

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

Recent Activities

- Part One of the Outline - "Coproduct of Methanol" has been written for release as a Topical Report. Comments from DOE on the 24 September 1998 draft of the Topical Report "Economic Analysis - LPMEOH™ Process as an Add-on to IGCC for Coproduction" were received, and a new update was sent to DOE on 22 December 1998.
- Part Two of the Outline - "Baseload Power and Methanol Coproduction", has been incorporated into the paper, "Fuel and Power Coproduction - The Liquid Phase Methanol (LPMEOH™) Process Demonstration at Kingsport ", that was presented at the DOE's Fifth Annual Clean Coal Technology Conference in January of 1997.
- Part Three of the Outline - "Coproduct for Intermediate Electric Load Following", has been incorporated into the paper, "Dispatchable IGCC Facilities: Flexibility through Coproduction", that was presented at POWER-GEN EUROPE '97 in June of 1997.

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

D.3 DME Design Verification Testing

Discussion

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

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

DME DVT Recommendation

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

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

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

- Planning for a DME test 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 should now be developed. 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 time to meet the schedule for testing at LaPorte.

LPDME™ is not applicable to hydrogen (H₂)-rich syngas; and it is unlikely that a substantive LPDME™ demonstration will be recommended for Kingsport. Therefore, a convincing case that the test-run on CO-rich syngas at LaPorte will lead to successful commercialization must be made, prior to approving the final test-run plan. The strategy for commercialization must present the technical logic to combine the results of the following two areas:

- 1) catalyst performance (productivity, selectivity, and life) for the LPDME™ catalyst system under CO-rich syngas from the design verification testing at the LaPorte AFDU; and
- 2) reactor performance (methanol catalyst activity and life, hydrodynamics, and heat transfer) from the LPMEOH™ Process Demonstration Unit at Kingsport.

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

Market Economic Studies

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

Laboratory R&D

Initially, synthesis of DME concurrently with methanol in the same reactor was viewed as a way of overcoming the syngas conversion limitations imposed by equilibrium in the LPMEOH™ process. Higher syngas conversion would provide improved design flexibility for the coproduction of power and liquid fuels from an IGCC facility. The liquid phase DME (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. Previously, proof-of-concept runs, in the laboratory and at the AFDU, confirmed that a higher syngas conversion could be obtained when a mixture of DME and methanol is produced in the liquid phase reactor.

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

Summary of Laboratory Activity and Results

- Air Products has been performing laboratory autoclave tests of samples of the AB dehydration catalyst from the commercial catalyst manufacturer (Engelhard). The results to date have not been consistent, indicating that all issues related to catalyst scale-up have not been resolved. As a result, the decision was made within the DOE's Liquid Fuels Program to delay the start of the AFDU design verification test. Changes to the commercial production procedure were made, and additional batches of dehydration catalyst were made and tested during the reporting period. These tests did not yield the desired catalyst aging characteristics.
- A set of experiments was performed on a commercially available dehydration catalyst to compare this material with the AB dehydration catalyst. These results showed that the desired catalyst life could be achieved with the commercially available dehydration catalyst at a 10-20% reduction in system productivity (primarily a reduction in the selectivity to DME).
- Additional experiments on the commercially available dehydration catalyst will focus on process variable scans associated with the upcoming design verification run at the LaPorte AFDU. The scheduled date for the campaign at the LaPorte AFDU was delayed by the DOE's Liquid Fuels Program in order to complete the additional autoclave work; the current estimate for the start of operations at the LaPorte AFDU is October of 1999.

D.4 LPMEOH™ Process Demonstration Facility - Methanol Operation

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

During the reporting period, a total of 4,962,109 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 safety or environmental incidents were reported during this quarter.

The LPMEOH™ Demonstration Unit achieved a new milestone during the reporting period by completing the longest continuous operating run without interruption of any kind on 27 October 1998 (94 days total). This campaign ended as a result of a syngas outage. Several other extended syngas outages and a power failure outage occurred during the reporting period. However, the LPMEOH™ Demonstration Unit operated at 100% availability throughout the quarter and completed a very successful 1998, with a year-end availability of 99.72%. Appendix F, Table 1 contains the summary of outages for the LPMEOH™ Demonstration Unit during this quarter.

Operations continued to focus on resolution of issues related to catalyst life and sparger resistance.

Catalyst Life (eta) - October - December 1998

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

The final batch of fresh baseline methanol catalyst was activated and added to the reactor on 02 October 1998. Subsequent catalyst additions during the reporting period used batches of the alternative methanol synthesis catalyst. Five batches of fresh alternative catalyst batches were activated and added during the reporting period on 29 October 1998, 19 November 1998, 03 December 1998, 11 December 1998, and 24 December 1998, respectively. The final addition of alternative catalyst during the reporting period brought the catalyst loading to about 140 % of design. No hydrodynamic instability was detected as the catalyst slurry concentration ranged from 46 - 48 wt%.

Catalyst deactivation was evaluated over the 25 day period encompassing 02 October 1998 to 26 October 1998. Operating conditions for this campaign were a reactor temperature of

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

Case	Date	Days Onstream	Temp (Deg C)	Pres. (psig)	Fresh Feed (KSCFH)	Recycle Gas (KSCFH)	Reactor Feed (H2:CO)	Purge Gas (KSCFH)	Inlet Sup. Velocity (ft/sec)	Space Velocity (l/hr-kg)	Slurry Conc. (wt% ox)	Gas Holdup (vol%)	Gassed Slurry Hgt (ft)	Catalyst Inventory (lb)	Catalyst Age (eta)	CO Conv. (%)	Reactor O-T-M Conv. (%)	Syngas Util. (SCF/lb)	Raw MeOH Production (TPD)	Catalyst MeOH Prod. (gmol/hr-kg)	Reactor Vol. Prod. (TPD/Cu ft)	U Overall (BTU/hr ft2 F)	Sparger dP (psi)	Sparger Resistance ("K")	
10	1-Oct-98	285	Balanced	235	710	765	1,913	3.79	84.7	0.62	3663	44.1	42.6	57.0	42,900	0.40	43.6	23.7	41.6	220.4	13.41	0.092	198	5.84	6.94
10	2-Oct-98	286	Balanced	235	710	768	1,873	4.27	73.3	0.61	3451	42.8	40.2	60.5	45,200	0.44	50.2	24.8	40.5	227.3	13.12	0.089	195	5.13	6.33
10	3-Oct-98	287	Balanced	235	709	795	1,880	4.05	89.6	0.62	3485	44.3	40.4	57.5	45,200	0.43	48.0	24.7	41.5	230.2	13.29	0.095	198	5.51	6.62
10	4-Oct-98	288	Balanced	235	710	806	1,875	4.09	92.4	0.62	3491	45.0	39.8	55.5	45,200	0.42	48.3	24.7	41.7	232.0	13.40	0.099	196	5.31	6.56
10	5-Oct-98	289	Balanced	235	710	809	1,837	4.26	81.0	0.61	3447	41.8	37.9	60.5	45,200	0.46	51.7	25.6	40.9	237.1	13.69	0.093	195	4.99	6.38
10	6-Oct-98	290	Balanced	235	709	817	1,859	4.01	76.7	0.62	3480	41.8	39.8	62.5	45,200	0.46	49.9	25.9	40.6	241.3	13.93	0.092	195	5.42	6.58
10	7-Oct-98	291	Balanced	235	710	795	1,879	4.07	69.7	0.62	3470	42.5	40.5	61.5	45,200	0.46	50.1	25.7	40.3	236.8	13.67	0.091	197	5.30	6.40
10	8-Oct-98	292	Balanced	235	709	815	1,888	4.14	88.7	0.63	3518	45.7	43.5	57.5	45,200	0.44	49.4	25.1	41.2	237.4	13.70	0.098	195	5.65	7.05
10	9-Oct-98	293	Balanced	235	710	821	1,910	3.80	81.9	0.63	3563	45.7	40.8	55.0	45,200	0.44	46.9	25.4	40.6	242.5	13.99	0.105	204	5.69	6.55
10	10-Oct-98	294	Balanced	235	711	797	1,897	3.91	74.8	0.62	3505	44.5	40.4	57.0	45,200	0.45	48.2	25.5	40.2	238.1	13.74	0.099	205	5.30	6.25
10	11-Oct-98	295	Balanced	235	710	779	1,873	4.09	81.9	0.62	3464	45.2	39.8	55.0	45,200	0.41	47.8	24.4	41.0	227.6	13.14	0.098	198	5.47	6.90
10	12-Oct-98	296	Balanced	235	710	817	1,879	3.78	92.7	0.62	3503	45.2	41.5	56.5	45,200	0.43	46.5	25.3	41.2	238.2	13.75	0.100	199	5.86	6.93
10	13-Oct-98	297	Balanced	235	709	825	1,858	3.75	93.8	0.62	3492	44.1	41.6	59.0	45,200	0.43	46.6	25.5	41.3	239.9	13.85	0.097	196	5.76	6.89
10	14-Oct-98	298	Balanced	235	710	822	1,836	3.82	93.4	0.62	3466	44.4	41.8	58.5	45,200	0.43	47.5	25.8	41.0	240.5	13.88	0.098	200	5.61	6.96
10	15-Oct-98	299	Balanced	235	710	826	1,853	3.78	96.1	0.62	3490	44.6	41.6	58.0	45,200	0.43	46.9	25.6	41.2	240.7	13.90	0.099	197	8.10	9.88
10	16-Oct-98	300	Balanced	235	710	825	1,855	3.70	95.0	0.62	3483	43.8	40.4	58.5	45,200	0.42	46.0	25.5	41.4	239.0	13.80	0.097	192	7.31	8.79
10	17-Oct-98	301	Balanced	235	712	758	1,808	4.13	72.3	0.59	3348	44.2	40.1	57.5	45,200	0.42	49.3	25.1	40.2	226.3	13.07	0.094	196	7.75	10.80
10	18-Oct-98	302	Balanced	235	710	824	1,931	2.75	53.1	0.64	3591	43.7	40.6	59.0	45,200	0.44	38.8	26.7	38.8	254.6	14.69	0.103	186	12.84	12.50
10	19-Oct-98	303	Balanced	235	709	828	1,889	2.85	66.3	0.63	3541	44.4	39.5	56.5	45,200	0.43	39.4	26.5	39.5	251.5	14.52	0.106	186	13.14	13.77
10	20-Oct-98	304	Balanced	235	711	828	1,841	3.18	79.5	0.62	3488	44.0	38.8	54.5	45,200	0.42	42.1	26.3	40.1	248.1	14.32	0.108	190	11.52	13.49
10	21-Oct-98	305	Balanced	235	709	660	1,649	5.14	52.1	0.54	3006	44.8	36.5	53.0	45,200	0.42	55.6	23.6	41.7	190.2	11.00	0.085	195	9.18	17.66
10	22-Oct-98	306	Balanced	235	707	609	1,867	5.30	38.9	0.58	3222	44.6	37.3	54.0	45,200	0.39	52.9	21.8	39.7	184.2	10.64	0.081	192	10.25	16.18
10	23-Oct-98	307	Balanced	235	710	743	1,864	3.85	91.7	0.60	3371	45.6	39.0	53.5	45,200	0.36	43.5	23.5	42.5	209.9	12.16	0.093	191	11.09	14.39
10	24-Oct-98	308	Balanced	235	710	760	1,820	3.76	88.2	0.60	3356	44.3	39.5	56.5	45,200	0.39	45.0	24.7	41.5	220.1	12.75	0.093	194	12.09	14.32
10	25-Oct-98	309	Balanced	235	710	759	1,804	3.62	91.5	0.60	3343	44.8	38.2	54.5	45,200	0.37	43.3	24.2	42.0	216.8	12.62	0.095	194	11.00	14.21
10	26-Oct-98	310	Balanced	235	710	740	1,800	3.70	94.3	0.59	3309	44.5	37.0	54.0	45,200	0.36	42.9	23.8	42.1	210.6	12.23	0.093	186	11.14	14.97
10	28-Oct-98	312	Balanced	235	710	734	1,777	3.50	93.1	0.58	3226	43.7	38.6	57.0	45,200	0.35	41.7	24.0	42.5	207.2	12.02	0.086	178	12.05	16.56
10	30-Oct-98	314	Balanced	235	710	723	1,760	4.17	62.6	0.58	3085	43.0	36.7	59.5	47,400	0.40	50.2	25.3	39.9	217.3	11.96	0.087	173	11.05	15.95
10	31-Oct-98	315	Balanced	235	710	734	1,723	4.33	68.8	0.57	3056	41.6	36.2	62.5	47,400	0.41	52.0	25.5	40.4	218.1	12.00	0.083	179	9.77	14.80
10	1-Nov-98	316	Balanced	235	710	734	1,988	4.13	65.2	0.63	3369	43.5	37.8	59.5	47,400	0.37	46.0	23.4	40.0	220.0	12.08	0.088	175	9.65	11.67
10	13-Nov-98	328	Balanced	235	701	735	2,028	4.43	101.0	0.65	3443	44.6	35.2	55.0	47,400	0.32	38.5	21.7	41.9	210.4	11.58	0.091	182	14.05	15.75
10	14-Nov-98	329	Balanced	235	695	761	2,134	1.88	91.4	0.68	3590	44.8	40.7	59.5	47,400	0.33	24.7	21.9	41.7	219.1	12.06	0.087	162	22.13	16.68
10	15-Nov-98	330	Balanced	235	694	770	2,075	1.88	100.1	0.68	3540	45.3	40.3	58.0	47,400	0.33	25.4	22.4	41.5	222.5	12.25	0.091	167	22.85	17.79
10	16-Nov-98	331	Balanced	235	692	768	1,980	1.88	101.5	0.65	3393	46.1	38.9	55.0	47,400	0.34	26.2	23.1	41.9	219.9	12.11	0.095	169	21.92	18.51
10	17-Nov-98	332	Balanced	235	693	760	2,031	1.91	110.1	0.66	3466	45.6	37.8	55.0	47,400	0.32	24.9	21.9	42.6	213.9	11.78	0.092	164	22.28	18.53
10	18-Nov-98	333	Balanced	235	692	730	1,967	1.91	98.3	0.65	3396	45.7	36.8	54.0	47,400	0.30	24.8	21.7	42.4	206.5	11.38	0.091	160	22.51	19.16
10	19-Nov-98	334	Balanced	235	695	693	2,111	1.94	52.6	0.66	3326	44.2	37.5	60.5	49,600	0.34	25.4	21.8	39.5	204.5	10.76	0.080	147	24.56	18.94
10	20-Nov-98	335	Balanced	235	695	769	2,066	1.89	77.3	0.67	3363	44.7	37.8	59.5	49,600	0.34	26.8	23.8	40.1	229.9	12.09	0.092	163	25.56	17.21
10	21-Nov-98	336	Balanced	235	699	707	2,135	2.04	56.1	0.67	3370	46.3	35.2	54.0	49,600	0.32	26.8	22.5	39.3	216.1	11.36	0.095	165	21.80	17.46
10	22-Nov-98	337	Balanced	235	708	745	2,060	2.15	66.5	0.65	3311	45.3	36.9	57.5	49,600	0.32	28.8	23.4	40.2	222.2	11.68	0.092	166	20.05	17.25
10	23-Nov-98	338	Balanced	235	705	753	2,055	2.30	87.1	0.66	3327	45.3	34.0	55.0	49,600	0.31	29.0	22.5	41.2	219.4	11.54	0.095	164	19.76	17.79
10	25-Nov-98	340	Balanced	235	709	655	1,895	3.92	63.2	0.59	3035	45.7	33.1	53.5	49,600	0.29	40.5	21.3	41.0	191.8	10.15	0.085	181	14.01	20.25
10	26-Nov-98	341	Balanced	235	709	728	1,956	3.31	95.2	0.62	3185	44.9	32.9	55.0	49,600	0.29	36.3	21.8	42.1	207.3	10.95	0.090	167	15.52	19.10
10	27-Nov-98	342	Balanced	235	695	764	1,997	2.10	109.4	0.65	3268	45.9	34.3	54.0	49,600	0.30	26.7	22.1	42.5	215.6	11.37	0.095	164	20.46	19.16
10	28-Nov-98	343	Balanced	235	695	753	1,990	2.07	111.7	0.65	3248	46.1	34.2	53.5	49,600	0.29	26.0	21.7	42.9	210.5	11.12	0.094	160	20.64	19.46
10	29-Nov-98	344	Balanced	235	695	750	1,969	2.06	118.8	0.64	3223	46.1	33.6	53.0	49,600	0.28	25.5	21.4	43.6	206.6	10.92	0.093	156	20.62	19.79
10	30-Nov-98	345	Balanced	230	696	520	2,117	2.08	27.1	0.61	3116	46.1	31.2	51.0	49,600	0.27	22.7	18.3	38.4	162.3	8.55	0.076	181	15.59	19.61
10	2-Dec-98	347	Balanced	230	695	501	2,027	3.73	51.0	0.58	2959	46.0	27.4	48.5	49,600	0.24	31.2	17.0	40.0	150.4	7.92	0.074	166	13.57	20.20
10	5-Dec-98	350	Balanced	231	700	515	1,850	4.75	28.0	0.55	2702	45.6	32.1	55.0	51,800	0.33	41.8	18.7	39.0	158.6	7.98	0.069	161	10.96	20.63
10	6-Dec-98	351	Balanced	232	700	696	1,883	3.32	86.5	0.60	2921	45.6	32.1	55.0	51,800	0.29	36.4	21.7	42.0	198.7	10.10	0.086	161	15.47	20.91
10																									

Table D.4-1. 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 (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 Production (TPD)	Catalyst MeOH Prod. (gmol/hr-kg)	Reactor Vol. Prod. (TPD/Cu ft)	U Overall (BTU/hr ft ² F)	Sparger dP (psi)	Sparger Resistance ("K")
10	14-Dec-98	359	Balanced	235	709	727	1,900	3.77	84.6	0.61	2843	46.1	30.4	55.0	54,000	0.29	41.4	22.7	42.3	206.4	10.04	0.089	176	15.60	21.32
10	15-Dec-98	360	Balanced	235	710	688	1,899	3.87	84.4	0.60	2815	46.1	28.3	53.5	54,000	0.28	41.2	22.0	41.7	197.7	9.66	0.088	181	14.74	20.59
10	25-Dec-98	370	Balanced	235	705	760	1,995	3.58	89.3	0.64	2890	47.8	29.9	53.5	56,200	0.29	39.8	22.3	42.4	215.0	10.17	0.096	180	17.08	20.28
10	26-Dec-98	371	Balanced	235	705	753	1,962	3.81	95.6	0.63	2844	47.2	28.9	54.0	56,200	0.29	41.6	22.4	42.3	213.5	10.03	0.094	177	15.38	19.63
10	27-Dec-98	372	Balanced	235	709	746	1,937	4.09	92.5	0.62	2817	45.7	29.2	57.2	56,200	0.29	43.8	22.4	42.3	211.5	9.93	0.088	175	15.38	19.25
10	28-Dec-98	373	Balanced	235	710	752	1,926	4.06	104.6	0.62	2802	45.3	25.7	55.5	56,200	0.28	43.2	22.4	42.9	210.2	9.83	0.090	177	14.23	19.56
10	29-Dec-98	374	Balanced	235	709	753	1,901	4.06	113.9	0.62	2791	45.8	26.5	55.0	56,200	0.28	42.7	22.1	43.7	206.8	9.68	0.089	179	14.08	19.52
10	31-Dec-98	376	Balanced	235	710	760	1,940	4.73	108.3	0.63	2828	45.1	29.7	59.0	56,200	0.28	41.6	22.4	43.1	211.8	9.93	0.085	179	14.67	19.45

235°C and a Balanced Gas flowrate of 700 - 800 KSCFH. During this operating period, the rate of decline in catalyst activity was 0.7% per day. For reference, the original target from the 4-month proof-of-concept run at the LaPorte AFDU in 1988/89 was 0.4% per day.

A short syngas outage occurred on 27 October 1998 which ended the extended operating period in October. On 02 November 1998, operation of the LPMEOHTM Demonstration Facility was interrupted by a complex-wide power outage. During this outage, the catalyst slurry was held in the reactor. Syngas supply was reestablished on 10 November 1998. A noticeable negative step-change in catalyst activity occurred following the restart. However, some of the calculated catalyst activity was regained several days following the restart. An analysis of temperature or pressure measurements around the reactor during this time period has not resulted in identifying a cause for this observation.

During most of the reporting period, the flow of Balanced Gas was between 700 and 800 KSCFH. However, a reduction in the Balanced Gas flowrate to 450 KSCFH occurred between 01 and 06 December 1998. On 16 December 1998, the syngas supply was interrupted for 8 days. The calculated value of the catalyst rate constant was unchanged as a result of this outage. There were two other short syngas outages experienced during the reporting period. Due to the lack of extended (2 week minimum) stable operating periods, changes in catalyst activity could not be quantified during the November and December 1998 time frame.

During several days in November of 1998, unit operation was performed with both Balanced Gas and CO Gas as the make-up streams at an average total flow of 740 KSCFH. This resulted in a H₂/CO ratio at the reactor inlet ranging from 1.88 to 2.3. CO Gas was not used during December of 1998 due to the greater sparger pressure drop experienced with the higher molecular weight reactor feed gas.

Analyses of catalyst samples for changes in physical characteristics and levels of poisons have continued. Appendix F, Table 2 summarizes the results to date. Samples have continued to show an increase in arsenic loading, with levels in excess of the concentrations measured on the initial charge of methanol synthesis catalyst from 1997. At this point in time, no correlation between the change in arsenic levels and catalyst performance has been identified. Sulfur has been measured at the analytical detection limit. Copper crystallite size measurements have shown an increase in the most recent samples; however, the size increase has stabilized over the last few samples. Other methods of crystallite size determination have corroborated the increased size measurements. Levels of iron and nickel have remained low and steady since the restart in December of 1997.

Sparger Resistance

In October of 1998, maintenance work was performed on the oil flush system that cleans the taps used to measure the pressure drop across the reactor gas sparger. Upon completion of this work, the measured pressure drop increased by 2.0 psi. An analysis of other pressure readings around the reactor was performed to determine the rate of increase of this pressure drop. It appears that the increase in pressure drop across the sparger occurred around the time of the maintenance work. Another step-change increase in pressure drop occurred after

restarting from the complex-wide power outage in early November of 1998. Prior to those two events, the sparger resistance was very stable with a very small increase observed over time. Appendix F, Figure 2 plots the average daily sparger resistance coefficient. The data for this plot, along with the corresponding average pressure drop, are included in Table D.4-1.

As a result of the greater sparger pressure drop, condensed oil and entrained slurry could not be gravity-drained to the flush connection at the gas inlet line to the reactor. In order to overcome the pressure drop, the streams were batch-transferred to the catalyst reduction vessel and returned to the reactor via the slurry transfer pump. This procedure was last used in October of 1997. During periods when the catalyst reduction vessel is otherwise in use, the condensed oil and entrained slurry were free-drained directly to a connection on the reactor where the pressure drop was not limiting.

The greater sparger resistance limited the introduction of CO Gas during the reporting period. The reactor pressure was reduced from 710 to 695 psig to account for the higher pressure drop resulting from both the increased molecular weight of the reactor feed gas and the added flow resistance when CO Gas was supplied. As the sparger flow resistance increased further, the flow of CO Gas was stopped completely.

D.5 Planning and Administration

The Milestone Schedule Status Report and the Cost Management Report, through the period ending 31 December 1998, 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 31 December 1998. Thirty-three percent (33%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 December 1998.

The monthly reports for October, November, and December were submitted. These reports include the Milestone Schedule Status Report, the Project Summary Report, and the Cost Management Report.

A paper entitled "Liquid Phase Methanol (LPMEOH™) Project Operational Experience" was presented at the Gasification Technologies Conference on 04-07 October 1998. A preprint for a paper which will be presented at the Spring 1999 American Chemical Society meeting was also submitted.

A draft topical report entitled "Alternative Fuels Field Test Unit Support to Kingsport LPMEOH™ Demonstration Unit - December 1997 - January 1998" was submitted to DOE for review.

The topical report entitled "Design and Fabrication of the First Commercial-Scale LPMEOH™ Reactor" was issued to DOE.

Volume 1 - Public Design, of the Final Report was updated and sent to DOE for review.

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 concentration in the LPMEOH™ Reactor to determine the maximum slurry concentration (Test 9 of Test Plan).
- Prepare for and complete biannual state code inspection of equipment at the LPMEOH™ Demonstration Unit.
- Resume preparations for a LPDME™ design verification test run at the LaPorte AFDU pending the completion of the testing of the dehydration catalyst.
- Continue execution of the Off-Site, Product-Use Test Program (Phase 1, Task 1.4).

F. Conclusion

The LPMEOH™ Demonstration Unit achieved a new milestone during the reporting period by completing the longest continuous operating run without interruption of any kind on 27 October 1998 (94 days total). The LPMEOH™ Demonstration Unit operated at 100% availability throughout the quarter and completed a very successful 1998, with a year-end availability of 99.72%.

The final batch of fresh baseline methanol catalyst was activated and added to the LPMEOH™ Reactor on 02 October 1998. Five batches of the fresh alternative catalyst batches were activated and added to the reactor during the reporting period. As of 31 December 1998, the catalyst loading in the reactor had been increased to about 140 % of design. No hydrodynamic instability was detected as the catalyst slurry concentration in the reactor ranged from 46 - 48 wt%.

Catalyst activity, as defined by the ratio of the rate constant at any point in time to the rate constant for freshly reduced catalyst (as determined in the laboratory autoclave), was monitored throughout the reporting period. Catalyst deactivation was evaluated over the 25 day period encompassing 02 October 1998 to 26 October 1998. Operating conditions for this campaign were a reactor temperature of 235°C and a Balanced Gas flowrate of 700 - 800 KSCFH. During this operating period, the rate of decline in catalyst activity was 0.7% per day. For reference, the original target from the 4-month proof-of-concept run at the LaPorte AFDU in 1988/89 was 0.4% per day. During much of the remainder of the reporting period, there were no extended operating periods which are needed to track changes in catalyst activity.

During several days in November of 1998, unit operation was performed with both Balanced Gas and CO Gas as the make-up streams. This resulted in a H₂/CO ratio at the reactor inlet ranging from 1.88 to 2.3.

Catalyst slurry samples from the LPMEOH™ Reactor have been taken on a regular basis to correlate any change in unit performance with changes in the physical properties of the catalyst. Samples have continued to show an increase in arsenic loading, with levels in excess of the concentrations measured on the initial charge of methanol synthesis catalyst from 1997. No correlation to date between the change in arsenic readings and catalyst performance has been identified. Sulfur has been measured at the analytical detection limit. Levels of iron and nickel have remained steady since the restart in December of 1997. Copper crystallite size measurements have shown an increase in the most recent samples.

The performance of the alternative gas sparger, which was designed by Air Products and installed into the LPMEOH™ Reactor prior to the restart of the LPMEOH™ Demonstration Unit in December of 1997, was monitored. During the reporting period, a step-change in the resistance coefficient for the gas sparger was detected around the time when maintenance was performed on the flush system that cleans the pressure taps for the pressure drop measurement. Another increase in resistance occurred around an extended syngas outage in November of 1998. As a result of the greater sparger pressure drop, condensed oil and entrained slurry could not be gravity-drained to the flush connection at the gas inlet line to the reactor. In order to overcome the pressure drop, the streams were batch-transferred to the catalyst reduction vessel and returned to the reactor via the slurry transfer pump. During periods when the catalyst reduction vessel is otherwise in use, the condensed oil and entrained slurry were free-drained directly to a connection on the reactor where the pressure drop was not limiting.

During the reporting period, a total of 4,962,109 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, over 30.7 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 the seven project sites selected for the off-site, product-use test program. Two flexible-fuel vehicles were operated over 3,200 miles on fuel-grade methanol from the LPMEOH™ Demonstration Project. Emissions testing of methanol as an emulsion fuel in a flight line generator showed reductions in nitrogen oxides of 40 - 60% when compared with operation on jet fuel. A proposal was submitted to conduct additional testing of fuel-grade methanol in a stationary gas turbine; goals of this work include the improvement of the lubricity of the methanol and an increase in the power output from the gas turbine. A reformer test apparatus to determine the operating characteristics of fuel-grade methanol as a feed to a fuel cell is under construction.

During the reporting period, planning for a design verification test run of the LPDME™ Process at the LaPorte AFDU continued. Air Products has been performing laboratory autoclave tests of samples of the dehydration catalyst from the commercial catalyst

manufacturer (Engelhard). The results to date have not been consistent, indicating that all issues related to catalyst scale-up and commercial-scale production have not been resolved. A set of experiments was performed on a commercially available dehydration catalyst. These results showed that the desired catalyst life could be achieved with the commercially available dehydration catalyst at a 10-20% reduction in system productivity (primarily a reduction in the selectivity to DME). Additional laboratory experiments must be completed before a new date for the start of the campaign at the LaPorte AFDU will be selected. The DOE's Liquid Fuels Program has targeted 01 October 1999 as the anticipated start date for the AFDU design verification test.

A paper entitled "Liquid Phase Methanol (LPMEOH™) Project Operational Experience" was presented at the Gasification Technologies Conference on 04-07 October 1998. The topical report entitled "Design and Fabrication of the First Commercial-Scale LPMEOH™ Reactor" was issued to DOE.

Ninety-nine percent (99%) of the \$38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 31 December 1998. Thirty-three percent (33%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 December 1998.

G. Acknowledgment

Shortly after the 14 October 1998 review meeting for the Product-Use Test Program, we were informed of the untimely death of Bob Senn, who until October of 1998 had lead the Air Products management efforts for this Program. The excellent results of the Test Program are a tribute to his efforts.

APPENDICES

APPENDIX A - SIMPLIFIED PROCESS FLOW DIAGRAM

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

Appendix B-1 - Summary Table of Seven Test Sites

Appendix B-2 - Review Meeting Notes (14 October 1998)

Quarterly Reports:

Appendix B-3 - ARCADIS Projects (two pages):

- Acurex FFV
- Stationary Turbine for VOC Control
- Aircraft Ground Equipment Emulsion

Appendix B-4 - West Virginia University Stationary Gas Turbine (forty-five pages)

Appendix B-5 - University of Florida Fuel Cell (thirteen pages)

Appendix B-6 - Florida Institute of Technology Bus & Light Vehicle (nineteen pages)

APPENDIX C - PROCESS ECONOMIC STUDY

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

and

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

APPENDIX D - DME DESIGN VERIFICATION TESTING

APPENDIX E - SAMPLES OF DETAILED MATERIAL BALANCE REPORTS

APPENDIX F - RESULTS OF DEMONSTRATION PLANT OPERATION

**Table 1 - Summary of LPMEOH™ Demonstration Unit Outages -
October/December 1998**

Table 2 - Summary of Catalyst Samples - Second Catalyst Batch

Figure 1 - Catalyst Age (η) vs. Days Onstream - Second Catalyst Batch

**Figure 2 - Sparger Resistance Coefficient vs. Days Onstream
(Post-19 December 1997 Restart)**

Table 1
Summary of LPMEOH™ Demonstration Unit Outages - October/December 1998

Operation Start	Operation End	Operating Hours	Shutdown Hours	Reason for Shutdown
10/1/98 00:01	10/27/98 14:30	637.5	15.4	Syngas Outage
10/28/98 05:55	11/1/98 14:10	104.3	1.0	Syngas Outage
11/1/98 15:10	11/3/98 13:15	46.1	163.8	Power / Syngas Outage
11/10/98 09:05	11/24/98 08:40	335.6	9.0	Syngas Outage
11/24/98 17:40	11/24/98 23:05	5.4	1.6	Syngas Outage
11/25/98 00:40	12/16/98 11:35	514.9	190.7	Syngas Outage
12/24/98 10:20	12/31/98 23:59	181.6		End of Reporting Period
Total Operating Hours			1825.4	
Syngas Available Hours			1825.4	
Plant Availability, %			100.00	

Table 2
Summary of Catalyst Samples - Second Catalyst Batch

Sample	Identity	XRD		BET	Analytical (ppmw)				
		Cu	ZnO	m ² /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						

Notes:

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

Figure 1

Catalyst Age (eta) vs. Days Onstream - Second Catalyst Batch

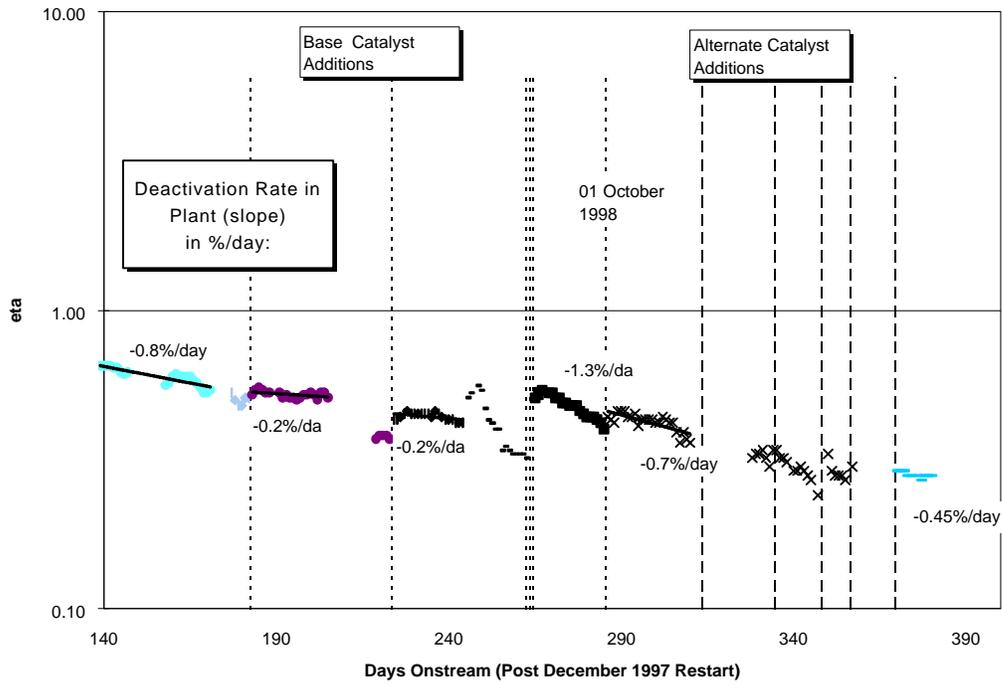
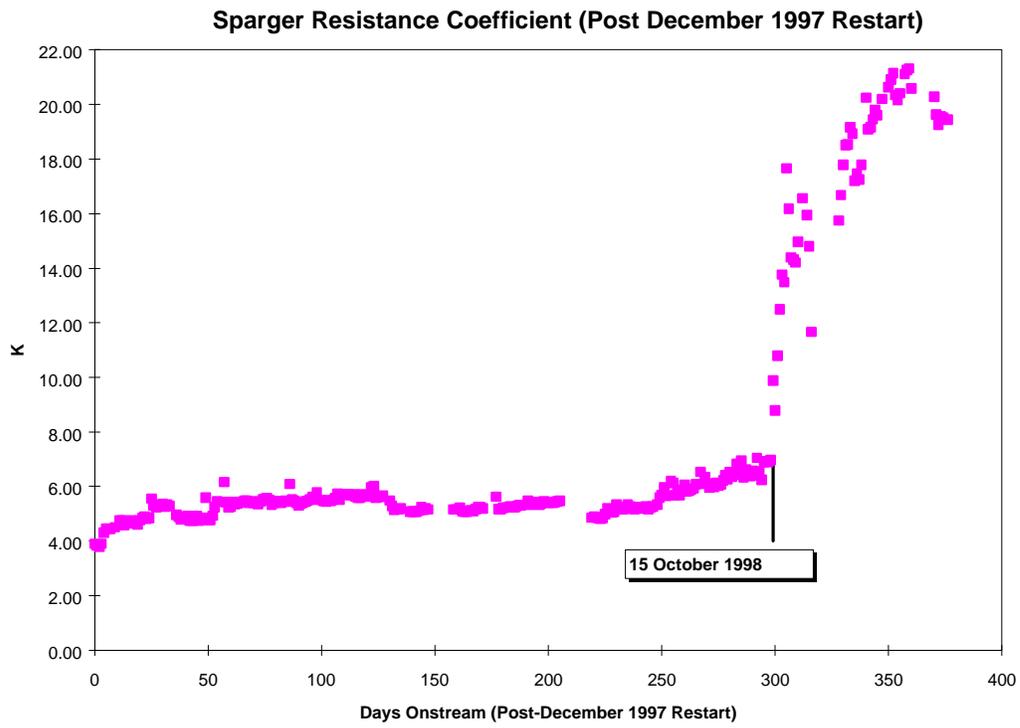


Figure 2



**APPENDIX G - MILESTONE SCHEDULE STATUS AND COST MANAGEMENT
REPORTS**