

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

**TECHNICAL PROGRESS REPORT NO. 16**

**For The Period**

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**for the**

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## Abstract

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

The LPMEOH™ Demonstration Facility completed its first year of operation on 02 April 1998. The LPMEOH™ Demonstration Facility also completed the longest continuous operating run (65 days) on 21 April 1998.

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. During a six-week test at a reactor temperature of 225°C and Balanced Gas flowrate of 700 KSCFH, the rate of decline in catalyst activity was steady at 0.29-0.36% per day. During a second one-month test at a reactor temperature of 220°C and a Balanced Gas flowrate of 550 - 600 KSCFH, the rate of decline in catalyst activity was 0.4% per day, which matched the performance at 225°C, as well as the 4-month proof-of-concept run at the LaPorte AFDU in 1988/89.

Beginning on 08 May 1998, the LPMEOH™ Reactor temperature was increased to 235°C, which was the operating temperature after the December 1997 restart with the fresh charge of catalyst (50% of design loading). The flowrate of the primary syngas feed stream (Balanced Gas) was also increased to 700 - 750 KSCFH. During two stable operating periods between 08 May and 09 June 1998, the average catalyst deactivation rate was 0.8% per day. Due to the scatter of the statistical analysis of the results, this test was extended to better quantify the catalyst aging behavior. During the reporting period, two batches of fresh catalyst were activated and transferred to the reactor (on 02 April and 20 June 1998). The weight of catalyst in the LPMEOH™ Reactor has reached 80% of the design value.

At the end of the reporting period, a step-change in the pressure-drop profile within the LPMEOH™ Reactor and an increase in the pressure of the steam system which provides cooling to the LPMEOH™ Reactor were observed. No change in the calculated activity of the catalyst was detected during either of these transients. These parameters will be monitored closely for any additional changes.

Catalyst slurry samples from the LPMEOH™ Reactor have been taken on a regular basis to correlate any change in plant performance with changes in the physical properties of the catalyst. Samples have continued to show an increase in arsenic loading, continuing the trend from the prior reporting period. Copper crystallite size measurements have shown a continuing slow growth, consistent with expectations given the length of time on-stream. Levels of iron and nickel have remained steady since the restart in December of 1997.

The performance of the alternative gas sparger, which was designed by Air Products and installed into the LPMEOH™ Reactor prior to the restart of the LPMEOH™ Demonstration Unit in December of 1997, was monitored throughout the reporting period. Pressure drop through the gas sparger of the LPMEOH™ Reactor remained steady by maintaining a continuous flush of condensed oil and entrained slurry which was gravity-drained from the 29C-05 secondary oil knock-out drum and 29C-06 cyclone. These results provide a confirmation of the encouraging data collected during the prior reporting period. This parameter will continue to be closely monitored for any change in flow resistance.

During the reporting period, a total of 4,645,166 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, over 20.3 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. Availability has exceeded 99% since the restart of the LPMEOH™ Demonstration Unit on 19 December 1997.

During this quarter, initial planning, procurement, and test operations continued on the seven project sites which have been accepted for participation in the off-site, product-use test program. At the three projects which are testing transportation vehicles, over 4,000 miles of operation have been completed on chemical-grade methanol and on fuel-grade methanol provided by the Demonstration Project. In a stationary turbine test, a glow plug ignition system was added to eliminate the flame-out which occurred when the turbine was switched from jet fuel to methanol at idle speed. The start of testing of fuel-grade methanol in a fuel cell is pending the completion of a system component analysis.

During the reporting period, planning for a proof-of-concept test run of the Liquid Phase Dimethyl Ether (LPDME™) Process at the Alternative Fuels Development Unit (AFDU) in LaPorte, TX continued. Production of the remaining dehydration catalyst by the commercial catalyst manufacturer (Engelhard, formerly Calsicat) is awaiting the completion of testing of a sample of the first production batch in the laboratory autoclave. The resulting delay in the scheduled delivery of the catalyst has not impacted the timing for the fall 1998 AFDU proof-of-concept test.

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 June 1998. Twenty-four percent (24%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 June 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 <sub>2</sub> ), carbon monoxide (CO), and carbon dioxide (CO <sub>2</sub> ) in stoichiometric balance for the production of methanol
Carbon Monoxide Gas	-	A syngas containing primarily carbon monoxide (CO); also called CO Gas
Catalyst Age ( $\eta$ -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 <sub>2</sub> 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 <sub>2</sub> ) over the stoichiometric balance for the production of methanol; also called H <sub>2</sub> 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

$\rho$  - density, pounds per cubic foot

ACRONYMS AND DEFINITIONS (cont'd)

Partnership	-	Air Products Liquid Phase Conversion Company, L.P.
PDU	-	Process Development Unit
PFD	-	Process Flow Diagram(s)
ppbv	-	parts per billion (volume basis)
ppmw	-	parts per million (weight basis)
Project	-	Production of Methanol/DME Using the LPMEOH™ Process at an Integrated Coal Gasification Facility
psi	-	Pounds per Square Inch
psia	-	Pounds per Square Inch (Absolute)
psig	-	Pounds per Square Inch (gauge)
P&ID	-	Piping and Instrumentation Diagram(s)
Raw Methanol	-	sum of Refined Grade Methanol and Crude Grade Methanol; represents total methanol which is produced after stabilization
Reactor Feed	-	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 <sub>2</sub> ) and carbon monoxide (CO), or mixtures of H <sub>2</sub> and CO; intended for "synthesis" in a reactor to form methanol and/or other hydrocarbons (synthesis gas may also contain CO <sub>2</sub> , 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 coal-to-chemicals 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 Facility completed its first year of operation on 02 April 1998. The LPMEOH™ Demonstration Facility also completed the longest continuous operating run (65 days) on 21 April 1998; an outage was taken as the result of a failure in a reactor temperature measurement device which is tied into a plant emergency shutdown.

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. During a six-week test at a reactor temperature of 225°C and flowrate of the primary syngas feed (Balanced Gas) of 700 KSCFH, the rate of decline in catalyst activity was steady at 0.29-0.36% per day. On 02 April 1998, an additional catalyst batch of the alternate methanol

synthesis catalyst was added to the LPMEOH™ Reactor. At the same time, reactor temperature was lowered to 220°C and Balanced Gas flowrate was reduced to 550 - 600 KSCFH. Over the next month, the rate of decline in catalyst activity was 0.4% per day, which matched the performance at 225°C, as well as the 4-month proof-of-concept run at the LaPorte AFDU in 1988/89.

Beginning on 08 May 1998, the LPMEOH™ Reactor temperature was increased to 235°C, which was the operating temperature after the December 1997 restart with the fresh charge of catalyst (50% of design loading). The Balanced Gas flowrate was also increased to 700 - 750 KSCFH. During two stable operating periods between 08 May and 09 June 1998, the average catalyst deactivation rate was 0.8% per day. In addition, the absolute value of the calculated rate constant in the kinetic model increased by 15% (relative), confirming earlier observations that the model tends to underpredict the rate constant at lower operating temperature. Due to the scatter of the statistical analysis of the results, the test was extended to better quantify the catalyst aging behavior at this condition. A fresh batch of catalyst was activated and transferred to the reactor on 20 June 1998 to maintain process viability for a minimum three-week test. The weight of catalyst in the LPMEOH™ Reactor has reached 80% of the design value.

At the end of the reporting period, a step-change in the pressure-drop profile within the LPMEOH™ Reactor and an increase in the pressure of the steam system which provides cooling to the LPMEOH™ Reactor were observed. No change in the calculated activity of the catalyst was detected during either of these transients. These parameters will be monitored closely for any additional changes.

Catalyst slurry samples from the LPMEOH™ Reactor have been taken on a regular basis to correlate any change in plant performance with changes in the physical properties of the catalyst. Samples have continued to show an increase in arsenic loading, continuing the trend from the prior reporting period. Copper crystallite size measurements have shown a continuing slow growth, consistent with expectations given the length of time on-stream. Levels of iron and nickel have remained steady since the restart in December of 1997.

The performance of the alternative gas sparger, which was designed by Air Products and installed into the LPMEOH™ Reactor prior to the restart of the LPMEOH™ Demonstration Unit in December of 1997, was monitored throughout the reporting period. Pressure drop through the gas sparger of the LPMEOH™ Reactor remained steady by maintaining a continuous flush of condensed oil and entrained slurry which was gravity-drained from the 29C-05 secondary oil knock-out drum and 29C-06 cyclone. These results provide a confirmation of the encouraging data collected during the prior reporting period. This parameter will continue to be closely monitored for any change in flow resistance.

During the reporting period, a total of 4,645,166 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, over 20.3 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. Availability has exceeded 99% since the restart of the LPMEOH™ Demonstration Unit on 19 December 1997.

During this quarter, initial planning, procurement, and test operations continued on the seven project sites which have been accepted for participation in the off-site, product-use test program. At the three projects which are testing transportation vehicles, over 4,000 miles of operation have been completed on chemical-grade methanol and on fuel-grade methanol from either the LPMEOH™ Demonstration Unit or from inventory at the LaPorte AFDU. In a stationary turbine test, a glow plug ignition system was added to eliminate the flame-out which occurred when the turbine was switched from jet fuel to methanol at idle speed. The start of testing of fuel-grade methanol in a fuel cell is pending the completion of the analysis of the effect of trace components in the methanol on components in the fuel cell system.

During the reporting period, planning for a proof-of-concept test run of the Liquid Phase Dimethyl Ether (LPDME™) Process at the LaPorte AFDU continued. The commercial catalyst manufacturer (Engelhard, formerly Calsicat) has prepared the first batch of dehydration catalyst in large-scale equipment. Production of the remaining catalyst is awaiting the completion of testing of a sample of this material in the laboratory autoclave. The resulting delay in the scheduled delivery of the catalyst has not impacted the timing for the AFDU proof-of-concept test, which is scheduled for the fall of 1998.

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 June 1998. Twenty-four percent (24%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 June 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 complex in Kingsport. The Partnership will own and operate the facility for the four-year demonstration period.

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

The LPMEOH™ process is the product of a cooperative development effort by Air Products and the DOE in a program that started in 1981. It was successfully piloted at a 10-TPD rate

in the DOE-owned experimental unit at Air Products' LaPorte, Texas, site. This demonstration project is the culmination of that extensive cooperative development effort.

## **B. Project Description**

The demonstration unit, which occupies an area of 0.6 acre, is integrated into the existing 4,000-acre Eastman complex located in Kingsport, Tennessee. The Eastman complex employs approximately 12,000 people. In 1983, Eastman constructed a coal gasification facility utilizing Texaco technology. The synthesis gas (syngas) generated by this gasification facility is used to produce carbon monoxide and methanol. Both of these products are used to produce methyl acetate and ultimately cellulose acetate and acetic acid. The availability of this highly reliable coal gasification facility was the major factor in selecting this location for the LPMEOH™ Process Demonstration. Three different feed gas streams (hydrogen gas or H<sub>2</sub> 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 will be made available for product-use tests. Product-use tests were 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 AFDU in LaPorte, TX. 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. The sites and project titles are listed in Appendix B-1. 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 eighth project, involving the testing of a water/naphtha/methanol emulsion as a transportation fuel, is awaiting final project definition.

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-2 through B-6 contain summary reports from the approved projects. Highlights from these reports include:

Acurex Flexible Fuel Vehicle (FFV) - The FFV has begun emission testing on both M85 made from chemical-grade methanol and on M85 made from methanol supplied from the inventory at the LaPorte AFDU. The FFV has accumulated 1,500 miles on the LPMEOH™ M85 fuel.

Stationary Turbine for Volatile Organic Carbon (VOC) Control - AlliedSignal has committed to serve as host site for this demonstration, and an outline of the demonstration tests was prepared.

West Virginia University (WVU) Stationary Gas Turbine - A glow plug ignition system was added to the gas turbine to eliminate the flame-out which occurred when the turbine was switched from jet fuel to methanol fuel at idle speed. Methanol from inventory at the LaPorte AFDU is being used in this program.

Aircraft Ground Equipment Emulsion - Scoping tests were delayed until August of 1998 pending the results of studies to determine the best emulsion composition.

University of Florida Fuel Cell - Based upon the results of analysis of the fuel-grade methanol from the LPMEOH™ Demonstration Project, an investigation is underway to determine the potential (if any) for degradation of the reformer or the stack components due to trace components in the methanol.

West Virginia University Tri-Boro Bus - Testing has been completed, and a draft final report was prepared. Results indicate that fuel-grade methanol is well suited to use in alcohol fuel compression ignition engines from the standpoint of emissions benefits (lower emissions of nitrogen oxides and particulate matter than chemical-grade methanol, but higher emissions of hydrocarbons for fuel-grade methanol).

Florida Institute of Technology Bus & Light Vehicle - Fuel-grade methanol from the LPMEOH™ Demonstration Project was used to operate both vehicles. The car has been operating for 6 months and over 2,000 miles, and the bus has completed 500 miles of testing. A preliminary car exhaust sample was submitted for analysis (methanol, nitrogen oxides, formaldehyde).

## ***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) front-end 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 31 March 1997 draft of the Topical Report "Economic Analysis - LPMEOH™ Process as an Add-on to IGCC for Coproduction" are the current basis for discussion. As part of reviewing this report, Air Products has submitted a recommendation that the cost breakdown by plant area matches the format to be used in the Final Report - Volume 1 - Public Design. A letter from DOE dated 07 April 1998 indicated that the Topical Report could be issued using a different cost breakdown than the Final Report - Volume 1 - Public Design. Air Products began incorporating this and other comments from DOE in anticipation of providing an updated Topical Report to DOE for further comment.
- 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 Four of the Outline - "Methanol Fuel Applications", was used as the basis to update the product-use test program (Task 1.4).

### ***D.3 DME Design Verification Testing***

#### Discussion

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

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



### *DME DVT Recommendation*

DOE issued a letter dated 31 July 1997 accepting Air Products' recommendation to continue with the design verification testing to coproduce DME with methanol, and to proceed with planning a proof-of-concept test run at the DOE's AFDU in LaPorte, Texas. 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, proof-of-concept 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 (H<sub>2</sub>)-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 proof-of-concept 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 continued. 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 to date, 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. During this quarter, laboratory work continued on developing an LPDME™ catalyst system based on the AB series of catalysts.

### ***Summary of Laboratory Activity and Results***

- The commercial catalyst manufacturer (Engelhard) completed the preparation of the first batch of dehydration catalyst in larger-scale (500 gallon) equipment. Air Products began testing a sample of this material in the laboratory autoclave. This testing continued through the end of the reporting period, causing a delay in the production and shipment

of the dehydration catalyst to the LaPorte AFDU (from the June of 1998 scheduled date). To date, this delay has not impacted the timing for the AFDU proof-of-concept test, which is scheduled for the fall of 1998.

#### ***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 4,645,166 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 completed its first year of operation on 02 April 1998, and the longest continuous operating run without a shutdown of any kind lasted until 21 April 1998 (65 days). That campaign ended when a reactor temperature transmitter failed, causing a false emergency shutdown on high temperature. Eastman operating personnel quickly identified the problem, and the plant was back onstream within 30 minutes. A second fault occurred in this same circuit two days later, prompting a review by Eastman to determine if a system of 2-out-of-3 voting can be applied to temperature measurements in the LPMEOH™ Reactor to limit the upsets resulting from instrumentation faults. On 27 April, a tubing leak on the syngas recycle compressor required a 10-hour shutdown for repair; a similar leak on 18 May required a 9-hour shutdown for repair. No other shutdowns during the reporting period were related to operation of the LPMEOH™ Demonstration Unit.

Despite this series of trips, the LPMEOH™ Demonstration Unit continues to operate at greater than 99% availability since being brought back onstream on 19 December 1997. The resulting extended operating periods provide an indication of the flexibility of the LPMEOH™ Process and the opportunity to collect sufficient steady-state data on the performance of the catalyst and the various components within the LPMEOH™

**TABLE D.4-1  
DATA SUMMARY FOR LPMEOH™ DEMONSTRATION UNIT**

Case	Date	Days Onstream	Gas Type	Temp (Deg C)	Pres. (psig)	Fresh Feed (KSCFH)	Recycle Gas (KSCFH)	Reactor Feed (H2:CO)	Purge Gas (KSCFH)	Inlet Sup. Velocity (ft/sec)	Space Velocity (l/hr-kg)	Slurry Conc. (wt% ox)	Gas Holdup (vol%)	Gassed Slurry Hgt (ft)	Catalyst Inventory (lb)	Catalyst Age (eta)	CO Conv. (%)	Reactor O-T-M Conv. (%)	Syngas Util. (SCF/lb)	Raw MeOH Production (TPD)	Catalyst MeOH Prod. (gmol/hr-kg)	Reactor Vol. Prod. (TPD/Cu ft)	U Overall (BTU/hr ft2 F)	Sparger dP (psi)	Sparger Resistance (°K)
6	1-Apr-98	102	Balanced	224	709	682	2,247	3.46	71.9	0.66	6,268	39.2	42.9	44.0	27,450	0.66	33.9	19.7	40.5	202.1	19.20	0.110	152	5.44	5.46
6	2-Apr-98	103	Balanced	221	710	684	2,234	3.51	91.6	0.66	5,696	40.7	43.1	45.5	30,050	0.61	32.8	19.0	42.2	194.6	16.90	0.102	152	5.28	5.49
6	3-Apr-98	104	Balanced	220	710	641	2,240	3.51	82.1	0.65	5,615	39.2	41.6	47.0	30,050	0.60	31.9	18.4	41.6	185.0	16.07	0.094	145	5.19	5.48
6	4-Apr-98	105	Balanced	219	710	630	2,267	3.39	79.4	0.65	5,646	40.8	42.1	44.5	30,050	0.60	30.2	17.9	41.6	181.8	15.79	0.098	149	5.24	5.51
6	5-Apr-98	106	Balanced	219	709	630	2,261	3.33	85.8	0.64	5,620	41.2	40.9	43.0	30,050	0.59	29.5	17.8	42.0	179.9	15.63	0.100	148	5.25	5.57
6	6-Apr-98	107	Balanced	218	709	605	2,269	3.47	75.6	0.64	5,605	41.9	39.0	40.5	30,050	0.59	30.0	17.4	41.2	176.2	15.31	0.104	150	5.28	5.73
6	7-Apr-98	108	Balanced	218	710	593	2,252	3.44	65.3	0.64	5,578	42.2	37.3	39.0	30,050	0.58	29.8	17.3	40.9	173.8	15.10	0.107	147	5.19	5.53
6	8-Apr-98	109	Balanced	218	710	587	2,264	3.30	66.2	0.64	5,604	43.0	36.8	37.5	30,050	0.58	28.6	17.2	40.6	173.6	15.09	0.111	143	5.43	5.70
6	9-Apr-98	110	Balanced	219	709	583	2,227	3.61	50.2	0.63	5,519	37.6	42.1	50.5	30,050	0.61	32.1	17.9	39.7	176.4	15.31	0.083	143	5.26	5.71
6	10-Apr-98	111	Balanced	220	710	578	2,195	4.03	45.0	0.63	5,462	37.5	44.8	53.0	30,050	0.61	35.4	18.0	39.2	177.1	15.40	0.080	152	4.83	5.70
6	11-Apr-98	112	Balanced	220	710	582	2,185	4.05	50.0	0.63	5,447	37.9	43.9	51.5	30,050	0.60	35.2	17.9	39.7	176.0	15.28	0.081	151	4.75	5.70
6	12-Apr-98	113	Balanced	220	711	582	2,192	3.80	52.0	0.63	5,451	38.1	42.7	50.0	30,050	0.59	33.5	17.9	39.7	176.0	15.28	0.084	150	4.91	5.69
6	13-Apr-98	114	Balanced	219	710	576	2,208	3.61	49.3	0.63	5,468	37.4	41.8	50.5	30,050	0.58	31.8	17.8	39.6	174.4	15.14	0.082	146	5.07	5.71
6	14-Apr-98	115	Balanced	220	710	580	2,189	3.68	46.4	0.62	5,436	36.9	43.9	53.5	30,050	0.60	33.1	18.3	39.4	176.9	15.36	0.079	152	5.02	5.64
6	15-Apr-98	116	Balanced	220	710	578	2,183	3.65	46.5	0.62	5,426	36.3	44.5	55.5	30,050	0.59	32.7	18.2	39.5	175.5	15.24	0.075	146	5.01	5.60
6	16-Apr-98	117	Balanced	219	709	578	2,227	3.57	48.1	0.63	5,506	37.1	44.8	54.0	30,050	0.59	31.7	18.0	39.5	175.5	15.24	0.077	151	5.15	5.59
6	17-Apr-98	118	Balanced	220	710	573	2,258	3.58	46.5	0.64	5,570	37.2	45.0	54.0	30,050	0.59	31.4	17.7	39.2	175.7	15.25	0.077	151	5.30	5.70
6	18-Apr-98	119	Balanced	220	710	567	2,267	3.54	39.3	0.64	5,550	36.5	44.9	55.5	30,050	0.59	31.3	17.8	39.0	174.5	15.17	0.075	149	5.33	5.63
6	19-Apr-98	120	Balanced	220	710	577	2,229	3.57	50.4	0.63	5,505	37.6	44.4	52.5	30,050	0.57	31.3	17.7	40.0	173.0	15.06	0.078	148	5.19	5.67
6	20-Apr-98	121	Balanced	220	710	583	2,198	3.52	61.0	0.63	5,473	37.5	43.5	52.0	30,050	0.56	30.8	17.7	40.6	172.5	14.98	0.079	142	5.11	5.63
6	21-Apr-98	122	Balanced	219	710	583	2,240	3.35	65.0	0.64	5,543	38.4	41.0	48.0	30,050	0.55	28.7	17.2	41.0	170.9	14.84	0.085	144	5.60	5.98
6	22-Apr-98	123	Balanced	219	711	583	2,229	3.34	70.2	0.63	5,535	39.2	42.3	47.5	30,050	0.55	28.5	17.1	41.2	169.9	14.76	0.085	141	5.54	6.01
6	23-Apr-98	124	Balanced	220	710	583	2,230	3.38	63.3	0.63	5,517	36.7	43.5	53.5	30,050	0.55	29.4	17.5	40.7	172.1	14.94	0.077	146	5.23	5.60
6	24-Apr-98	125	Balanced	220	710	583	2,201	3.55	67.3	0.63	5,473	37.1	42.0	51.5	30,050	0.55	30.3	17.4	41.2	170.0	14.76	0.079	145	5.00	5.62
6	25-Apr-98	126	Balanced	219	709	583	2,193	3.53	69.7	0.63	5,459	37.4	41.7	50.5	30,050	0.55	30.0	17.3	41.4	168.9	14.68	0.080	144	4.96	5.64
6	26-Apr-98	127	Balanced	219	708	583	2,182	3.47	70.2	0.63	5,440	37.7	40.7	49.0	30,050	0.55	29.7	17.3	41.3	169.4	14.72	0.082	149	4.94	5.66
6	29-Apr-98	130	Balanced	220	709	554	2,292	3.80	63.3	0.64	5,555	37.2	43.5	52.5	30,050	0.53	30.0	16.0	41.0	162.0	14.08	0.073	145	4.87	5.49
6	30-Apr-98	131	Balanced	220	709	552	2,259	3.98	63.1	0.63	5,487	36.7	41.1	51.5	30,050	0.54	31.1	16.0	41.4	159.9	13.89	0.074	147	4.53	5.29
6	1-May-98	132	Balanced	219	710	549	2,249	3.96	62.7	0.63	5,481	37.4	41.2	50.0	30,050	0.54	30.8	15.9	41.6	158.2	13.74	0.075	149	4.46	5.15
6	2-May-98	133	Balanced	219	710	554	2,233	3.93	67.3	0.63	5,455	38.4	39.7	47.0	30,050	0.54	30.5	15.9	42.2	157.7	13.70	0.080	150	4.40	5.17
6	3-May-98	134	Balanced	219	709	554	2,206	4.15	67.9	0.62	5,413	37.1	40.5	50.0	30,050	0.55	32.2	16.1	42.1	158.0	13.73	0.075	151	4.25	5.18
6	4-May-98	135	Balanced	220	710	555	2,289	4.16	59.8	0.64	5,554	37.7	41.2	49.5	30,050	0.55	31.8	15.9	41.7	159.9	13.89	0.077	150	4.41	5.20
6	8-May-98	139	Balanced	234	709	735	2,145	4.45	54.8	0.67	5,619	37.3	41.7	51.5	30,050	0.65	47.3	22.4	40.2	219.5	19.05	0.101	147	5.01	5.08
6	9-May-98	140	Balanced	235	707	738	2,176	4.20	49.5	0.67	5,683	36.9	42.0	52.5	30,050	0.65	45.7	22.6	39.5	224.1	19.45	0.102	148	5.24	5.07
6	10-May-98	141	Balanced	234	707	738	2,130	4.41	55.2	0.66	5,601	37.6	41.4	50.5	30,050	0.65	47.1	22.6	40.1	221.1	19.19	0.104	149	4.90	5.08
6	11-May-98	142	Balanced	235	710	729	2,139	4.48	52.0	0.66	5,615	36.8	42.4	53.0	30,050	0.65	47.6	22.5	39.7	220.0	19.09	0.099	148	4.94	5.09
6	12-May-98	143	Balanced	235	710	738	2,139	4.55	60.1	0.67	5,625	36.5	42.1	53.5	30,050	0.64	47.7	22.5	40.4	219.4	19.05	0.098	147	4.89	5.12
6	13-May-98	144	Balanced	234	710	733	2,096	4.73	67.2	0.65	5,537	37.1	40.2	50.5	30,050	0.64	48.9	22.3	40.5	216.8	18.83	0.102	148	4.64	5.24
6	14-May-98	145	Balanced	234	710	713	2,138	4.40	53.2	0.66	5,584	37.4	40.2	50.0	30,050	0.62	46.2	22.1	39.6	215.9	18.75	0.103	145	4.97	5.14
6	15-May-98	146	Balanced	235	710	732	2,107	4.13	54.6	0.66	5,577	35.9	41.4	54.0	30,050	0.61	44.6	22.4	40.1	218.8	19.00	0.096	143	5.19	5.21
6	16-May-98	147	Balanced	235	710	732	2,087	4.34	59.3	0.65	5,516	36.5	40.5	52.0	30,050	0.62	46.5	22.6	40.0	219.6	19.07	0.101	144	4.78	5.16
6	27-May-98	158	Balanced	234	710	733	2,115	3.99	80.4	0.66	5,584	37.2	40.4	50.5	30,050	0.56	41.7	21.6	41.2	213.5	18.54	0.101	144	5.07	5.16
6	28-May-98	159	Balanced	235	710	731	2,078	4.01	71.5	0.65	5,507	37.1	40.3	50.5	30,050	0.57	42.5	21.9	40.6	216.1	18.77	0.102	142	4.76	5.16
6	29-May-98	160	Balanced	234	710	731	2,050	4.08	63.6	0.65	5,456	36.5	40.4	52.0	30,050	0.60	44.3	22.6	39.9	219.5	19.06	0.100	144	4.63	5.16
6	30-May-98	161	Balanced	234	710	732	2,023	4.25	68.8	0.64	5,412	37.1	39.5	50.0	30,050	0.61	45.6	22.7	40.3	217.8	18.91	0.104	144	4.53	5.23
6	31-May-98	162	Balanced	234	710	726	2,054	3.88	57.3	0.65	5,469	37.7	39.8	49.0	30,050	0.60	42.9	22.9	39.6	219.9	19.09	0.107	144	4.83	5.10

**TABLE D.4-1  
DATA SUMMARY FOR LPMEOH™ DEMONSTRATION UNIT**

Case	Date	Days Onstream	Gas Type	Temp (Deg C)	Pres. (psig)	Fresh Feed (KSCFH)	Recycle Gas (KSCFH)	Reactor Feed (H2:CO)	Purge Gas (KSCFH)	Inlet Sup. Velocity (ft/sec)	Space Velocity (l/hr-kg)	Slurry Conc. (wt% ox)	Gas Holdup (vol%)	Gassed Slurry Hgt (ft)	Catalyst Inventory (lb)	Catalyst Age (eta)	CO Conv. (%)	Reactor O-T-M Conv. (%)	Syngas Util. (SCF/lb)	Raw MeOH Production (TPD)	Catalyst MeOH Prod. (gmol/hr-kg)	Reactor Vol. Prod. (TPD/Cu ft)	U Overall (BTU/hr ft2 F)	Sparger dP (psi)	Sparger Resistance ("K")
6	1-Jun-98	163	Balanced	235	710	721	2,085	3.88	56.1	0.65	5,498	38.3	39.9	48.0	30,050	0.60	42.8	22.7	39.4	219.6	19.06	0.109	144	4.88	5.08
6	2-Jun-98	164	Balanced	234	710	705	2,064	3.94	49.3	0.64	5,442	37.4	37.9	48.0	30,050	0.60	43.3	22.6	39.2	215.8	18.73	0.107	148	4.82	5.08
6	3-Jun-98	165	Balanced	235	710	730	2,013	4.13	66.4	0.64	5,388	37.2	39.7	50.0	30,050	0.60	45.2	22.9	40.1	218.6	18.98	0.104	144	4.51	5.12
6	4-Jun-98	166	Balanced	234	710	725	2,029	4.28	75.2	0.64	5,412	37.8	39.4	48.5	30,050	0.58	45.1	22.4	40.8	213.4	18.52	0.105	145	4.51	5.13
6	5-Jun-98	167	Balanced	234	709	736	2,050	3.97	79.9	0.65	5,460	37.5	39.9	49.5	30,050	0.57	42.5	22.4	41.0	215.4	18.70	0.104	145	4.75	5.10
6	6-Jun-98	168	Balanced	235	710	735	2,072	3.98	81.3	0.65	5,504	37.5	40.4	50.0	30,050	0.55	41.9	22.0	41.1	214.4	18.61	0.102	146	4.72	5.17
6	7-Jun-98	169	Balanced	234	710	719	2,064	4.05	79.8	0.65	5,461	38.0	39.9	48.5	30,050	0.53	41.4	21.4	41.5	207.8	18.04	0.102	144	4.61	5.25
6	8-Jun-98	170	Balanced	234	710	734	2,047	3.94	89.1	0.64	5,447	37.4	40.3	50.0	30,050	0.53	41.0	21.7	41.8	210.9	18.32	0.100	143	4.61	5.24
6	9-Jun-98	171	Balanced	234	709	733	2,058	3.76	80.5	0.64	5,444	36.3	41.2	53.0	30,050	0.54	40.1	21.9	41.4	212.4	18.44	0.095	144	4.78	5.19
6	15-Jun-98	177	Balanced	234	709	732	2,032	4.58	112.2	0.64	5,420	38.7	42.7	49.5	30,050	0.53	44.3	20.8	43.6	201.4	17.51	0.097	146	4.70	5.63
6	16-Jun-98	178	Balanced	230	711	497	2,212	5.24	27.6	0.62	5,304	37.4	41.1	50.5	30,050	0.50	40.4	16.7	39.3	151.7	13.18	0.072	142	4.32	5.17
6	17-Jun-98	179	Balanced	230	710	497	2,246	4.94	26.2	0.63	5,359	36.7	39.0	50.0	30,050	0.48	38.5	16.7	39.1	152.5	13.24	0.073	140	4.56	5.18
6	18-Jun-98	180	Balanced	230	707	487	2,178	5.08	26.6	0.62	5,229	37.6	38.6	48.0	30,050	0.48	39.6	16.7	39.1	149.7	13.01	0.074	143	4.38	5.22
6	19-Jun-98	181	Balanced	235	709	733	2,084	3.74	96.2	0.65	5,482	38.1	40.2	48.5	30,050	0.51	38.4	21.1	42.4	207.2	18.01	0.102	141	4.81	5.23
6	20-Jun-98	182	Balanced	235	709	732	2,080	3.83	100.5	0.65	5,470	38.2	38.5	47.0	30,050	0.51	39.2	21.2	42.5	206.7	17.98	0.105	142	4.81	5.23
6	21-Jun-98	183	Balanced	235	709	732	2,062	4.00	69.5	0.65	5,003	38.4	41.2	53.0	32,700	0.52	43.3	22.5	40.8	215.4	17.20	0.097	137	4.91	5.26
6	22-Jun-98	184	Balanced	235	709	734	2,061	4.05	59.8	0.65	5,013	37.9	41.2	54.0	32,700	0.54	44.3	22.8	40.2	219.0	17.48	0.096	135	4.85	5.24
6	23-Jun-98	185	Balanced	235	709	733	2,035	4.17	62.4	0.64	4,953	37.9	39.4	52.5	32,700	0.55	45.6	22.9	40.2	218.5	17.44	0.099	137	4.63	5.25
6	24-Jun-98	186	Balanced	235	709	721	2,031	4.29	64.8	0.64	4,942	39.4	40.7	50.5	32,700	0.54	45.7	22.5	40.5	213.6	17.04	0.101	137	4.59	5.28
6	25-Jun-98	187	Balanced	235	709	739	1,982	4.30	84.0	0.63	4,882	39.3	38.6	49.0	32,700	0.53	45.6	22.5	41.8	212.3	16.95	0.103	134	4.44	5.32
6	26-Jun-98	188	Balanced	234	709	740	1,949	4.36	87.2	0.62	4,826	38.0	38.0	51.0	32,700	0.53	46.4	22.6	42.2	210.7	16.84	0.098	133	4.34	5.31
6	29-Jun-98	191	Balanced	234	710	722	1,927	4.43	77.8	0.62	4,780	36.0	33.9	52.0	32,700	0.53	46.9	22.6	41.3	210.1	16.77	0.096	179	4.20	5.47
6	30-Jun-98	192	Balanced	235	710	631	1,978	4.72	41.5	0.61	4,707	34.7	28.7	51.0	32,700	0.51	47.2	21.3	39.6	191.6	15.28	0.089	190	4.18	5.34

Demonstration Unit. Appendix F, Table 1 contains the summary of outages for the LPMEOH™ Demonstration Unit during this quarter.

At the very end of the reporting period, rapid changes occurred in the pressure-drop profile within the LPMEOH™ Reactor, as well as in the pressure of the steam system which provides cooling to the LPMEOH™ Reactor. Over a 12-hour period, the liquid level in the LPMEOH™ Reactor dropped about six feet with little appreciable change in overall pressure drop, indicating a decrease in the gas holdup. Shortly thereafter, the steam pressure (as measured by two independent transmitters and confirmed by a temperature measurement device) ramped up over a 4-hour period. Since the productivity of the catalyst did not change during either of these transients, the increased steam pressure caused the calculated heat transfer coefficient for the internal heat exchanger to increase. However, the new value of the heat transfer coefficient at the end of the event exceeded even the original startup value for the clean system. The pressure drop across the gas sparger remained steady during the changes in the other measurements. Since these events are as yet unexplained, these parameters will be monitored closely for any additional changes.

Operations focused on resolution of key issues identified during prior operating periods.

#### Catalyst Life (eta) - December of 1997 - June 1998

The “age” of the methanol synthesis catalyst can be expressed in terms of a dimensionless variable  $\eta$ , 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 from the restart in December of 1997 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.

An extended operating test at a reactor temperature of 225°C and Balanced Gas flowrate of 700 KSCFH was completed on 02 April 1998. During this six-week test, the rate of decline in catalyst activity was steady at 0.29-0.36% per day, exclusive of a small negative step change apparently related to a gasifier switch. This activity decline was a measurable improvement over the 1% per day rate seen at 235°C in January and met the original target from the 4-month proof-of-concept run at the LaPorte AFDU in 1988/89. On 02 April 1998, a batch of an alternate methanol synthesis catalyst was activated and transferred to the LPMEOH™ Reactor. At the same time, reactor temperature was reduced again to 220°C and Balanced Gas flowrate was reduced to 550 - 600 KSCFH to maintain overall efficiency. Over the next month, the average catalyst deactivation rate was 0.4% per day, matching the performance at 225°C.

Beginning on 08 May 1998, the LPMEOH™ Reactor temperature was increased back to 235°C, which was the original operating temperature after the restart in December of 1997 with the fresh charge of catalyst (50% of design loading). The Balanced Gas flowrate was also increased to 700 - 750 KSCFH. Notably, the calculated rate constant from the kinetic model increased by 18% (relative) immediately after the transition, confirming earlier

observations that the model tends to underpredict the rate constant at lower operating temperature. During the first nine days at this condition, the average catalyst deactivation rate was 0.8% per day. This result approaches the 1% per day rate seen in January of 1998, although the confidence limits on the data were still rather broad. Unfortunately, a one-week curtailment in syngas availability interrupted the test after ten days, necessitating an additional two to three weeks to better quantify the catalyst aging behavior at this condition.

During a second stable operating period, the rate of decline in catalyst activity was again 0.8% per day at this condition; however, on 09 June 1998 another one-week interruption in syngas supply cut short the test after two weeks, while the confidence limits on the data were still rather broad. The plant restarted on June 15, but remained at reduced rates until June 19. A fresh batch of catalyst was activated and transferred to the reactor on 20 June 1998 to maintain process viability for a minimum three-week test to better quantify the catalyst aging behavior at this condition.

Analyses of catalyst samples for changes in physical characteristics and levels of poisons have begun. Appendix F, Table 2 summarizes the results to date. Samples have continued to show an increase in arsenic loading, although not nearly to the levels seen in the summer of 1997. Copper crystallite size measurements have shown a continuing slow growth, consistent with expectations given the length of time on-stream. Levels of iron and nickel have remained steady since the restart in December of 1997.

### Sparger Resistance

As reported in earlier Technical Progress Reports, flow resistance through the gas sparger of the LPMEOH™ Reactor had been stabilized using a continuous flush of condensed oil and entrained slurry from the 29C-05 secondary oil knock-out drum and 29C-06 cyclone. These streams are gravity-drained back to the reactor through a flush connection at the gas inlet line to the reactor, thus eliminating a batch-transfer operation which had been used during prior operation. The flow rate of the flush is equivalent to the average rate of liquid traffic in the reactor loop (1 to 2 gallons per minute).

This technique was first applied to a clean sparger at the restart of operations on 19 December 1997. Appendix F, Figure 2 plots the average daily sparger resistance coefficient since then, and provides continued confirmation of the earlier encouraging results. The various shutdowns caused no negative effects. The data for this plot, along with the corresponding average pressure drop, are included in Table D.4-1. This parameter will continue to be closely monitored for any change in flow resistance.

## ***D.5 Planning and Administration***

The Milestone Schedule Status Report and the Cost Management Report, through the period ending 30 June 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 30 June 1998. Twenty-four percent (24%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 June 1998.

The monthly reports for April, May, and June were submitted. These reports include the Milestone Schedule Status Report, the Project Summary Report, and the Cost Management Report.

A paper entitled "Commercial-Scale Demonstration of the Liquid Phase Methanol (LPMEOH™) Process: Initial Operating Experience" was presented at the Clean Coal Technology Conference in Reno, Nevada on April 29, 1998.

A draft topical report entitled "Design and Fabrication of the First Commercial-Scale LPMEOH™ Reactor" was submitted to DOE for review.

A draft of the Demonstration Technology Start-up Report was issued internally for review.

#### **E. Planned Activities for the Next Quarter**

- Write and submit the Demonstration Technology Start-up Report to DOE.
- Continue to analyze catalyst slurry samples and gas samples 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).
- Continue preparations for a DME proof-of-concept test run at the LaPorte AFDU pending the completion of the production of the dehydration catalyst.
- Continue execution of the Off-Site, Product-Use Test Program (Phase 1, Task 1.4).
- Continue to incorporate DOE comments into the Topical Report on Process Economic Studies.
- Reach agreement with DOE on the equipment breakdown and operating cost summary for use in the Final Technical Report, Volume 1, Public Design Report.
- Reissue the Topical Report on Liquid Phase Reactor Design to DOE for review and comment.

#### **F. Conclusion**

The LPMEOH™ Demonstration Facility completed its first year of operation on 02 April 1998. The LPMEOH™ Demonstration Facility also completed the longest continuous operating run (65 days) on 21 April 1998; an outage was taken as the result of a failure in a reactor temperature measurement device which is tied into a plant emergency shutdown.



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. During a six-week test at a reactor temperature of 225°C and Balanced Gas flowrate of 700 KSCFH, the rate of decline in catalyst activity was steady at 0.29-0.36% per day. On 02 April 1998, an additional catalyst batch of the alternate methanol synthesis catalyst was added to the LPMEOH™ Reactor. At the same time, reactor temperature was lowered to 220°C and Balanced Gas flowrate was reduced to 550 - 600 KSCFH. Over the next month, the rate of decline in catalyst activity was 0.4% per day, which matched the performance at 225°C, as well as the 4-month proof-of-concept run at the LaPorte AFDU in 1988/89.

Beginning on 08 May 1998, the LPMEOH™ Reactor temperature was increased to 235°C, which was the operating temperature after the December 1997 restart with the fresh charge of catalyst (50% of design loading). The Balanced Gas flowrate was also increased to 700 - 750 KSCFH. During two stable operating periods between 08 May and 09 June 1998, the average catalyst deactivation rate was 0.8% per day. In addition, the absolute value of the calculated rate constant in the kinetic model increased by 15% (relative), confirming earlier observations that the model tends to underpredict the rate constant at lower operating temperature. Due to the scatter of the statistical analysis of the results, the test was extended to better quantify the catalyst aging behavior at this condition. A fresh batch of catalyst was activated and transferred to the reactor on 20 June 1998 to maintain process viability for a minimum three-week test. The weight of catalyst in the LPMEOH™ Reactor has reached 80% of the design value.

At the end of the reporting period, a step-change in the pressure-drop profile within the LPMEOH™ Reactor and an increase in the pressure of the steam system which provides cooling to the LPMEOH™ Reactor were observed. No change in the calculated activity of the catalyst was detected during either of these transients. These parameters will be monitored closely for any additional changes.

Catalyst slurry samples from the LPMEOH™ Reactor have been taken on a regular basis to correlate any change in plant performance with changes in the physical properties of the catalyst. Samples have continued to show an increase in arsenic loading, continuing the trend from the prior reporting period. Copper crystallite size measurements have shown a continuing slow growth, consistent with expectations given the length of time on-stream. Levels of iron and nickel have remained steady since the restart in December of 1997.

The performance of the alternative gas sparger, which was designed by Air Products and installed into the LPMEOH™ Reactor prior to the restart of the LPMEOH™ Demonstration Unit in December of 1997, was monitored throughout the reporting period. Pressure drop through the gas sparger of the LPMEOH™ Reactor remained steady by maintaining a continuous flush of condensed oil and entrained slurry which was gravity-drained from the 29C-05 secondary oil knock-out drum and 29C-06 cyclone. These results provide a confirmation of the encouraging data collected during the prior reporting period. This parameter will continue to be closely monitored for any change in flow resistance.

During the reporting period, a total of 4,645,166 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, over 20.3 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. Availability has exceeded 99% since the restart of the LPMEOH™ Demonstration Unit on 19 December 1997.

During this quarter, initial planning, procurement, and test operations continued on the seven project sites which have been accepted for participation in the off-site, product-use test program. At the three projects which are testing transportation vehicles, over 4,000 miles of operation have been completed on chemical-grade methanol and on fuel-grade methanol from either the LPMEOH™ Demonstration Unit or from inventory at the LaPorte AFDU. In a stationary turbine test, a glow plug ignition system was added to a eliminate the flame-out which occurred when the turbine was switched from jet fuel to methanol at idle speed. The start of testing of fuel-grade methanol in a fuel cell is pending the completion of the analysis of the effect of trace components in the methanol on components in the fuel cell system.

During the reporting period, planning for a proof-of-concept test run of the Liquid Phase Dimethyl Ether (LPDME™) Process at the LaPorte AFDU continued. The commercial catalyst manufacturer (Engelhard) has prepared the first batch of dehydration catalyst in large-scale equipment. Production of the remaining catalyst is awaiting the completion of testing of a sample of this material in the laboratory autoclave. The resulting delay in the scheduled delivery of the catalyst has not impacted the timing for the AFDU proof-of-concept test, which is scheduled for the fall of 1998.

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 June 1998. Twenty-four percent (24%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 June 1998.

**APPENDICES**

**APPENDIX A - SIMPLIFIED PROCESS FLOW DIAGRAM**

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

**Appendix B-1 - Summary Table of Eight Candidates (one page)**

**Quarterly Reports:**

**Appendix B-2 - ARCADIS Projects (two pages):**

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

**Appendix B-3 - West Virginia University Stationary Gas Turbine (five pages)**

**Appendix B-4 - University of Florida Fuel Cell (three pages)**

**Appendix B-5 - West Virginia University Tri-Boro Bus (twenty-six pages)**

**Appendix B-6 - Florida Institute of Technology Bus & Light Vehicle (twenty-six pages)**

**Off-Site Product-Use Testing  
Proposals Under Consideration**

<b><u>Demonstration Project</u></b>	<b><u>Site</u></b>
Acurex FFV	California
Stationary Turbine for VOC Control	Site to be determined in cooperation with EPRI
West Virginia Univ. Stationary Gas Turbine	West Virginia
Water/Naphtha/MeOH Bus,	California
Aircraft Ground Equipment Emulsion	Tyndall AFB, Florida Brooks AFB, Texas
University of Florida Fuel Cell Fuel Cell, Florida	Florida
West Virginia Univ. Tri-Boro Bus	New York
Florida Inst. of Tech. Bus & Light Vehicle	Florida

**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 -  
April/June 1998**

**Table 2 - Summary of Catalyst Samples - Second Catalyst Batch**

**Figure 1 - Catalyst Age ( $\eta$ ) 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 Plant Outages - April/June 1998**

Operation Start	Operation End	Operating Hours	Shutdown Hours	Reason for Shutdown
4/1/98 00:01	4/21/98 01:40	480.6	0.5	ESD on Bad Reactor TT
4/21/98 02:10	4/22/98 20:10	42.0	0.8	ESD on Bad Reactor TT
4/22/98 21:00	4/27/98 12:42	111.7	10.2	Tubing Leak on K-01
4/27/98 22:52	5/18/98 19:50	501.0	9.0	Fitting Leak on K-01
5/19/98 04:50	5/19/98 04:50	0.0	154.8	Syngas Outage
5/25/98 15:40	6/9/98 19:40	364.0	43.4	Syngas Outage
6/11/98 15:05	6/11/98 21:35	6.5	15.5	Syngas Outage
6/12/98 13:05	6/12/98 13:55	0.8	66.3	Syngas Outage
6/15/98 08:10	6/30/98 23:59	375.8		End of Reporting Period
Total Operating Hours			1882.5	
Syngas Available Hours			1903.0	
Plant Availability, %			98.92	

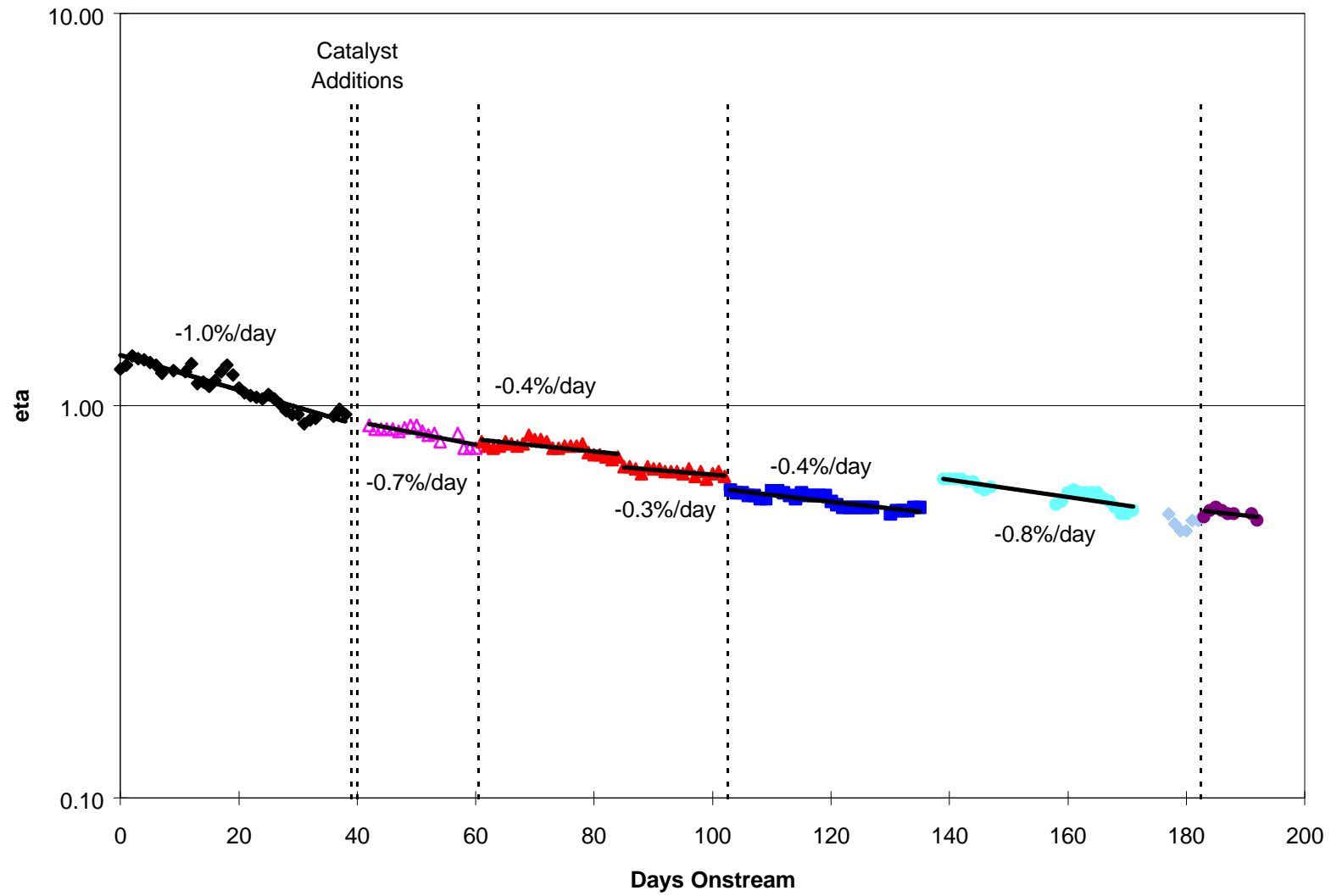
**Table 2 - Summary of Catalyst Analyses - Second Catalyst Batch**

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

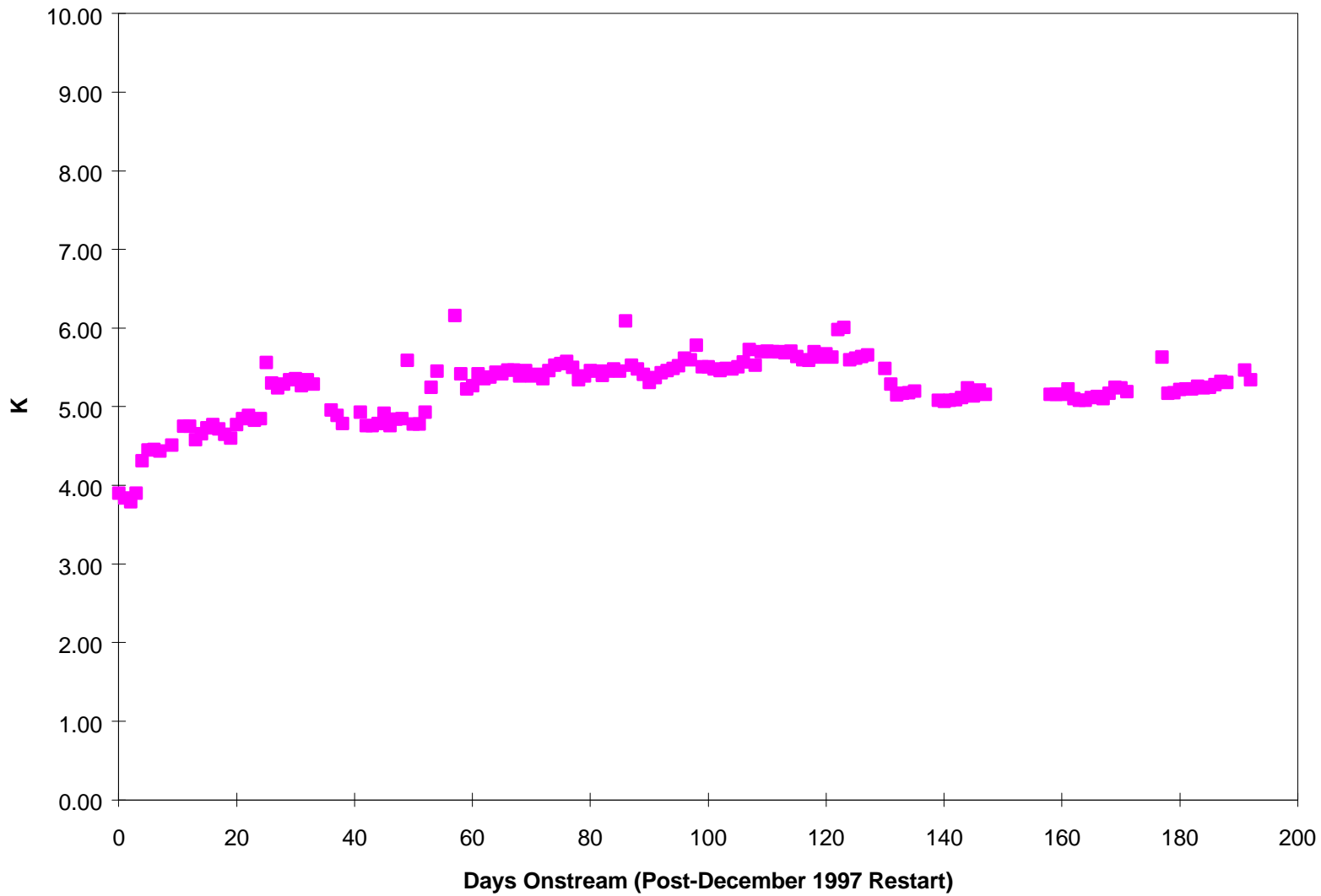
Notes:

1) nd = none detected

**Figure 1**  
**Catalyst Age (eta)**



**Figure 2**  
**Sparger Resistance Coefficient (Post-December 1997 Restart)**



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