

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

TECHNICAL PROGRESS REPORT NO. 29

For The Period

1 July – 30 September 2001

Prepared by

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Allentown, Pennsylvania**

and

**Eastman Chemical Company
Kingsport, Tennessee**

for the

Air Products Liquid Phase Conversion Company, L.P.

**Prepared for the United States Department of Energy
National Energy Technology Laboratory
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Abstract

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

The LPMEOH™ Demonstration Unit operated at 96.2% availability during this quarter. The forced downtime experienced this month (66.6 hours) was associated with the changeout and reduction of the adsorbent in the 29C-40 catalyst guard bed (described below). There were also two short syngas interruptions that were experienced on 17 September 2001 (8 hours duration) and 29 September 2001 (21 hours duration).

At the start of the reporting period, a catalyst addition was undertaken during the quarter to raise the catalyst activity. A single batch of fresh catalyst was activated and added to the reactor on 18 July 2001. After this batch of freshly activated catalyst was transferred into the reactor, the catalyst inventory was calculated to be 47,820 pounds.

A catalyst deactivation rate of 0.60% per day was calculated for the period 19 June 2001 to 15 July 2001 (26 days). This deactivation rate was achieved with the catalyst guard bed bypassed and out of service. This is a slightly higher deactivation rate than the results that have been calculated over the past 8 months.

For the month of July 2001, the flowrate of the primary syngas feed (Balanced Gas) was controlled at an average value of 560 KSCFH, the reactor pressure was set an average of 710 psig, and reactor temperature was maintained at 235°C.

During the reporting period, Eastman agreed with a recommendation by Air Products that the adsorbent (activated carbon impregnated with copper oxide) in the catalyst guard bed should be replaced. On 24 July 2001, the catalyst guard bed was emptied and refilled with the same commercially available adsorbent. The catalyst guard bed was then prepared for reduction (in this case, the reaction of the copper oxide with a reductant such as carbon monoxide [CO] or hydrogen [H₂] to copper metal and either carbon dioxide [CO₂] or water [H₂O]) of the adsorbent.

The guard bed adsorbent reduction procedure was started at 1700 hrs on 26 July 2001. During this reduction procedure, a dilute stream of Balanced Gas in nitrogen was preheated and used to reduce the copper oxide to copper metal in a temperature controlled manner. The temperature control during the procedure was excellent, and the reduction was completed at 0200 hrs on 28 July 2001. After cooling with nitrogen, a pressure check to full supply pressure with Balanced Gas was conducted. The adsorbent temperatures increased by about 25°C and stabilized during this examination. This observation was consistent with the

temperature increase which would be expected from the adsorption of CO on the guard bed adsorbent surface. The catalyst guard bed was bypassed after the pressure check, and was placed in service on 24 August 2001, after completion of in-situ catalyst activation of fresh methanol synthesis catalyst (which is described below).

In coordination with the timing for the in-situ activation of the methanol synthesis catalyst in the 29C-01 LPMEOH™ Reactor, an inspection of the 29K-01 recycle compressor was performed by personnel from Eastman, Air Products, and the equipment supplier. The only maintenance that was performed was the cleaning of accumulated solids on the impellor and the replacement of the dry gas seals. All other mechanical components were in excellent shape.

The LPMEOH™ Demonstration Unit was shutdown on 06 August 2001 to prepare for the in-situ catalyst activation procedure (the procedure used the LPMEOH™ Reactor [instead of the 29C-30 catalyst reduction vessel] to prepare fresh methanol synthesis catalyst for production of methanol by reacting metal oxides [for example, copper oxide] with a reductant such as CO or H₂ to produce the base metal plus either CO₂ or H₂O). The slurry contents of the reactor were cooled and transferred to the 29D-02 slurry tank. Batches of slurry were then transferred from the slurry tank to the catalyst reduction vessel for final cooling and draining into drums. A total of 305 drums or 88,150 pounds of slurry was removed from the reactor.

Beginning on 13 August 2001, batches of fresh catalyst were mixed with mineral oil in the catalyst reduction vessel, heated to 150°C, and transferred, without being reduced (or activated), to the slurry tank. A total of ten batches of fresh catalyst slurry, each containing about 4,000 pounds of fresh catalyst, was prepared in this manner. After the final transfer, the total catalyst inventory in the slurry tank was 41,580 pounds of fresh catalyst, which is approximately the design catalyst inventory for the LPMEOH™ Reactor. The catalyst loading and transfer operation was completed on 21 August 2001.

The common inlet and outlet line of the slurry tank (2" diameter) developed a plug during the transfer of the tenth batch of fresh catalyst slurry from the catalyst reduction vessel. After modifications to the piping system were made, Eastman operations personnel were able to clear this obstruction in the early morning hours of 22 August 2001. The contents of the slurry tank were then successfully transferred into the LPMEOH™ Reactor. The transfer of fresh catalyst slurry was completed at 0847 hrs on 22 August 2001.

Immediately upon completion of the slurry transfer, the recycle compressor was started to begin to cool the reactor contents to less than 90°C, which was the desired initial temperature for catalyst activation. After this step was completed, the in-situ catalyst activation procedure was started at 2300 hrs on 22 August 2001. Make-up nitrogen flow was controlled at 52 KSCFH, and a small quantity of Balanced Gas was introduced into the discharge of the recycle compressor to ensure that sufficient reductant was present at all times in the reactor inlet. The in-situ catalyst activation procedure ended at 0100 hrs on 24 August 2001.

The progress of the in-situ catalyst activation procedure was tracked by evaluating the uptake of reductant by the catalyst. A preliminary uptake of 70% of the theoretical value was calculated based upon analytical data and measured flowrates. The LPMEOH™ Demonstration Unit was then restarted at 1200 hrs on 24 August 2001.

A reactor catalyst sample from 05 September 2001 was submitted for chemical analysis and for a check of catalyst activity in the autoclave to determine the effectiveness of the in-situ catalyst activation procedure. Results from the activity determination from this sample were not comparable to similar tests using fresh catalyst. Reactor performance, however, appears to indicate adequate catalyst activity. Subsequent reactor samples from later in September 2001 have indicated better activity results in the autoclave. Additional tests are being developed to resolve these discrepancies and to develop the means to compare data from different operating periods.

Following the completion of the in-situ catalyst activation procedure, conditions were selected to deal with the hyperactivity that typically occurs when fresh methanol synthesis catalyst is initially exposed to syngas. During the first few weeks of operation, conditions were varying between a reactor pressure of 550-600 psig, reactor temperature of 212-220°C, and flowrate of Balanced Gas of 500-525 KSCFH. Once the hyperactivity period had passed, operating conditions were set at 218°C reactor temperature, 685 psig reactor pressure, and 600-650 KSCFH of Balanced Gas for the remainder of the quarter.

Analyses of catalyst samples to determine changes in physical characteristics and levels of poisons have continued. Prior to the in-situ activation of the fresh charge of catalyst, analysis of all pertinent poisons showed an increase in the levels of arsenic, sulfur, and iron (all of which are known poisons to methanol synthesis catalysts). Chemical analysis of catalyst samples that have been taken following the completion of the in-situ catalyst activation procedure indicated the presence of all expected crystal phases, and no accumulation of trace contaminants was detected.

The performance of the gas sparger, which was designed by Air Products and first installed into the LPMEOH™ Reactor prior to the restart of the LPMEOH™ Demonstration Unit in March of 1999, was monitored. The device had been inspected and cleaned during the March 2001 biennial outage. The sparger resistance continues to show no significant increase over time, which is consistent with the operating history with this device. The performance of the sparger will continue to be monitored closely for any changes.

During the reporting period, a total of 3,942,198 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, about 83.0 million gallons of methanol have 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.

A draft Topical Report entitled “Off-Site Testing of Stabilized Methanol from the Liquid Phase Methanol (LPMEOH™) Process” was sent to DOE for review. Comments were received on the main body of the report; this section provides the perspective of the

LPMEOH™ Demonstration Project (the reports from the seven participants will be included in volumes for Transportation and Power Generation Systems).

The paper entitled “Liquid Phase Methanol (LPMEOH™) Process Development Demonstration Plant Availability” was released for presentation at the Gasification Technologies Conference in San Francisco, CA (07-10 October 2001). Work began on developing the poster which will be displayed at the Clean Coal and Power Conference (formerly the Clean Coal Technology Conference) in Washington, DC (19-20 November 2001). The draft paper entitled “Liquid Phase Methanol (LPMEOH™) Process Development” was prepared; this paper is scheduled to be included for the proceeding of the 18th Annual International Pittsburgh Coal Conference in Newcastle, Australia (04-07 December 2001).

One hundred percent (100%) of the \$38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 30 September 2001. Eighty-two percent (82%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 September 2001.

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ACRONYMS AND DEFINITIONS

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

ACRONYMS AND DEFINITIONS (cont'd)

NEPA	-	National Environmental Policy Act
OSHA	-	Occupational Safety and Health Administration
ρ	-	density, pounds per cubic foot
Partnership	-	Air Products Liquid Phase Conversion Company, L.P.
PDU	-	Process Development Unit
PFD	-	Process Flow Diagram(s)
ppbv	-	parts per billion (volume basis)
ppmw	-	parts per million (weight basis)
Project	-	Production of Methanol/DME Using the LPMEOH™ Process at an Integrated Coal Gasification Facility
psi	-	pounds per square inch
psia	-	pounds per square inch (absolute)
psig	-	pounds per square inch (gauge)
P&ID	-	Piping and Instrumentation Diagram(s)
Raw Methanol	-	sum of Refined Grade Methanol and Crude Grade Methanol; represents total methanol which is produced after stabilization
Reactor Feed	-	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
SCF	-	Standard Cubic Feet
SCFH	-	Standard Cubic Feet per Hour
Slurry Concentration	-	percentage of weight of slurry (solid plus liquid) which is catalyst (on an oxide basis)
Sl/hr-kg	-	Standard Liter(s) per Hour per Kilogram of Catalyst
Syngas	-	Abbreviation for Synthesis Gas
Syngas Utilization	-	defined as the number of standard cubic feet of Balanced Gas plus CO Gas to the LPMEOH™ Demonstration Unit required to produce one pound of Raw Methanol
Synthesis Gas	-	A gas containing primarily hydrogen (H ₂) and carbon monoxide (CO), or mixtures of H ₂ and CO; intended for "synthesis" in a reactor to form methanol and/or other hydrocarbons (synthesis gas may also contain CO ₂ , water, and other gases)
Tie-in(s)	-	the interconnection(s) between the LPMEOH™ Process Demonstration Unit and the Eastman Facility
TPD	-	Ton(s) per Day
V	-	volumetric flowrate, thousand standard cubic feet per hour
VOC	-	volatile organic compound
vol%	-	volume %
WBS	-	Work Breakdown Structure
wt	-	weight

Executive Summary

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

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

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

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

At the Eastman chemicals-from-coal complex, the technology is integrated with existing coal gasifiers. A carefully developed test plan will allow operations at Eastman to simulate electricity demand load-following in coal-based IGCC facilities. The operations will also demonstrate the enhanced stability and heat dissipation of the conversion process, its reliable

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

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

This project has also completed design verification testing (DVT), including laboratory- and pilot-scale research and market verification studies, to evaluate whether to include a demonstration of the production of dimethyl ether (DME) as a mixed coproduct with methanol. DME has several commercial uses. In a storable blend with methanol, the mixture can be used as a peaking fuel in gasification-based electric power generating facilities, or as a diesel engine fuel. Blends of methanol and DME can be used as chemical feedstocks for synthesizing chemicals, including new oxygenated fuel additives.

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

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In coordination with the timing for the in-situ activation of the methanol synthesis catalyst in the 29C-01 LPMEOH™ Reactor, an inspection of the 29K-01 recycle compressor was performed by personnel from Eastman, Air Products, and the equipment supplier. The only maintenance that was performed was the cleaning of accumulated solids on the impellor and the replacement of the dry gas seals. All other mechanical components were in excellent shape. Work began on 13 August 2001, and the compressor was reassembled by 16 August 2001. On 17 August 2001, the compressor was successfully tested to check vibration and bearing temperatures.

The LPMEOH™ Demonstration Unit was shutdown on 06 August 2001 to prepare for the in-situ catalyst activation procedure (the procedure used the LPMEOH™ Reactor [instead of the 29C-30 catalyst reduction vessel] to prepare fresh methanol synthesis catalyst for production of methanol by reacting metal oxides [for example, copper oxide] with a reductant such as CO or H₂ to produce the base metal plus either CO₂ or H₂O). The slurry contents of the reactor were cooled and transferred to the slurry tank. Batches of slurry were then transferred from the slurry tank to the 29C-30 catalyst reduction vessel for final cooling

and draining into drums. A total of 305 drums or 88,150 pounds of slurry was removed from the reactor.

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Immediately upon completion of the slurry transfer, the recycle compressor was started to begin to cool the reactor contents to less than 90°C, which was the desired initial temperature for catalyst activation. Cooling was completed at 2250 hrs on 22 August 2001 after the reactor temperature reached 88°C.

After reactor cooling was completed, the in-situ catalyst activation procedure was started at 2300 hrs on 22 August 2001. Make-up nitrogen flowrate was controlled at 52 KSCFH, and a small quantity of Balanced Gas was introduced into the discharge of the recycle compressor to ensure that sufficient reductant was present at all times in the reactor inlet. The in-situ catalyst activation procedure ended at 0100 hrs on 24 August 2001.

The progress of the in-situ catalyst activation procedure was tracked by evaluating the uptake of reductant by the catalyst. A preliminary uptake of 70% of the theoretical value was calculated based upon analytical data and measured flowrates. The LPMEOH™ Demonstration Unit was then restarted at 1200 hrs on 24 August 2001.

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Activities associated with Design Verification Testing (DVT) of the LPDME Process have been completed. A Topical Report, which presents the results of the DVT at the LaPorte AFDU, was approved by DOE and issued (March 2001). The LPMEOH™ Demonstration Project will prepare a separate Topical Report on the market analysis for DME and review of the economics of the LPDME Process.

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One hundred percent (100%) of the \$38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 30 September 2001. Eighty-two percent (82%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 September 2001.

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 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 has been demonstrating the suitability of the methanol produced for use as a chemical feedstock or as a low-sulfur dioxide, low-nitrogen oxides alternative fuel in stationary and transportation applications. The project has also evaluated the demonstration of the production of dimethyl ether (DME) as a mixed coproduct with methanol.

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

B. Project Description

The demonstration unit, which occupies an area of 0.6 acre, is integrated into the existing 4,000-acre Eastman complex located in Kingsport, Tennessee. The Eastman complex employs approximately 8,600 people. In 1983, Eastman constructed a coal gasification facility utilizing Texaco technology. The synthesis gas (syngas) generated by this gasification facility is used to produce carbon monoxide and methanol. Both of these products are used to produce methyl acetate and ultimately cellulose acetate and acetic acid. The availability of this highly reliable coal gasification facility was the major factor in selecting this location for the LPMEOH™ Process Demonstration. Three different feed gas streams (hydrogen gas or 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 has been used 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)

The LPMEOH™ Demonstration Project has completed the testing of stabilized methanol from both the LaPorte AFDU and the Kingsport LPMEOH™ Demonstration Unit in various off-site mobile and stationary applications. The product-use test program was 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 can be demonstrated.

Product-use tests commenced during the first year of demonstration operations. An 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 produced and held at the LaPorte AFDU.

A draft Topical Report entitled “Off-Site Testing of Stabilized Methanol from the Liquid Phase Methanol (LPMEOH™) Process” was sent to DOE for review. Comments were received on the main body of the report; this section provides the perspective of the LPMEOH™ Demonstration Project (the reports from the seven participants will be included in volumes for Transportation and Power Generation Systems).

D.2 DME Design Verification Testing

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

essential steps required for decision-making were: a) confirm catalyst activity and stability in the laboratory, b) develop engineering data in the laboratory, and c) confirm market(s), including fuels and chemical feedstocks.

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

A Topical Report, which presents the results of the DVT at the LaPorte AFDU, has been issued (March 2001).

The LPMEOH™ Demonstration Project will prepare a separate Topical Report on the market analysis for DME and review of the economics of the LPDME Process.

D.3 LPMEOH™ Process Demonstration Unit - Methanol Operation

Table D.3-1 contains the summary table of performance data for the LPMEOH™ Demonstration Unit during the reporting period. These data represent daily averages, typically from a 24-hour material balance period, and those days with less than 12 hours of stable operation are omitted. Appendix B contains samples of the detailed material balance reports, which are representative of the operation of the LPMEOH™ Demonstration Unit during the reporting period. On-stream time for the third campaign began with the restart after the in-situ activation of methanol synthesis catalyst (described below).

During the reporting period, a total of 3,942,198 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Eastman accepted this entire methanol for use in the production of methyl acetate, and ultimately cellulose acetate and acetic acid. No environmental incidents or injuries were reported during this quarter.

The LPMEOH™ Demonstration Unit operated at 96.2% availability during this quarter. Appendix C, Table 1 contains the summary of outages for the LPMEOH™ Demonstration Unit during this quarter. It should be noted that the time associated with the in-situ catalyst activation was not included in this availability calculation as it was implemented as part of the demonstration test plan. The forced downtime experienced this month (66.6 hours) was associated with the changeout and reduction of the adsorbent in the 29C-40 catalyst guard bed (described below). There were also two short syngas interruptions that were experienced on 17 September 2001 (8 hours duration) and 29 September 2001 (21 hours duration). The major activities during the quarter involved the preparation for and execution of the steps to perform the in-situ activation of methanol synthesis catalyst within the 29C-01 LPMEOH™ Reactor.

Catalyst Life (η) – July - August 2001

The “age” of the methanol synthesis catalyst can be expressed in terms of a dimensionless variable 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 C, Figure 1 plots $\log \eta$ versus days onstream from September of 2000 to the end of the second operating campaign (prior to in-situ catalyst activation). 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.

A catalyst addition was undertaken during the quarter to raise catalyst activity. A single batch of fresh catalyst (totaling 2,200 pounds) was activated and added to the reactor on 18 July 2001. After this batch of freshly activated catalyst was transferred into the reactor, the catalyst inventory was calculated to be 47,820 pounds.

A catalyst deactivation rate of 0.75% per day was calculated for the period 20 June 2001 to 17 July 2001 (28 days). This deactivation rate was achieved with the catalyst guard bed bypassed and out of service. As noted in Appendix C, Figure 1, this is a slightly higher deactivation rate than the results that have been calculated over the past 8 months.

During this period, the flowrate of Balanced Gas was controlled at an average value of 560 KSCFH, the reactor pressure was set an average of 710 psig, and reactor temperature was maintained at 235°C.

29C-40 Catalyst Guard Bed Adsorbent Replacement and Reduction – July 2001

As reported in Technical Progress Report No. 28, initial performance results for the adsorbent (activated carbon impregnated with copper oxide) in the catalyst guard bed indicated that breakthrough of arsenic may have occurred within the first two weeks of operation. It is possible that damage to the adsorbent may have occurred during the August 2000 temperature excursion when this material was first placed into service. Eastman agreed with a recommendation by Air Products that the adsorbent should be replaced. On 24 July 2001, the catalyst guard bed was emptied and refilled with the same commercially available adsorbent. The catalyst guard bed was then prepared for reduction (in this case, the reaction of the copper oxide with a reductant such as carbon monoxide [CO] or hydrogen [H₂] to copper metal and either carbon dioxide [CO₂] or water [H₂O]) of the adsorbent.

The guard bed reduction procedure was started at 1700 hrs on 26 July 2001. During this reduction procedure, a dilute stream of Balanced Gas in nitrogen was preheated and used to reduce the copper oxide to copper metal in a temperature controlled manner. The temperature control during the procedure was excellent, and the reduction was completed at 0200 hrs on 28 July 2001. After cooling with nitrogen, a pressure check to full supply pressure with Balanced Gas was conducted. The adsorbent temperatures increased by about 25°C and stabilized during this examination. This observation was consistent with the temperature increase which would be expected from the adsorption of CO on the guard bed adsorbent surface. The catalyst guard bed was bypassed after the pressure check, and was placed in service on 24 August 2001, after completion of the in-situ catalyst activation of

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

Case	Date	Days On Stream	Gas Type	Temp (Deg C)	Pres. (psig)	Fresh Feed (KSCFH)	Recycle Gas (KSCFH)	Reactor Feed (H ₂ -CO)	Purge Gas (KSCFH)	Inlet Sup. Velocity (ft/sec)	Space Velocity (L/hr-kg)	Slurry Conc. (wt% ox)	Gas Holdup (vol%)	Gassed Slurry Hgt (ft)	Catalyst Inventory (lb)	Catalyst Age (eta)	CO Conv. (%)	Reactor			Raw		U		
																		O-T-M Conv. (%)	Syngas Util. (SCF/lb)	MeOH Prod. (TPD)	Catalyst MeOH Prod. (gmol/hr-kg)	Reactor Vol. Prod. (TPD/ft ³)	Overall (Btu hr ft ² F)	Sparger dP (psi)	Sparger Resist. ("K")
11	1-Jul-01	1289	Balanced	235	710	570	2,152	2.58	60.8	0.62	3469	41.7	21	48.5	45,620	0.241	23.7	16.9	41.8	163.5	9.36	0.08	121	6.8	6.84
11	2-Jul-01	1290	Balanced	235	710	573	2,163	2.81	64.3	0.63	3480	43	25	48.5	45,620	0.242	23.6	16.7	42.1	163.2	9.35	0.08	117	6.7	6.82
11	3-Jul-01	1291	Balanced	235	710	578	2,150	2.58	67.1	0.62	3477	44.4	25.6	46.5	45,620	0.238	23.5	16.8	42.4	163.4	9.36	0.08	118	6.7	6.74
11	4-Jul-01	1292	Balanced	235	710	572	2,168	2.57	71.9	0.63	3484	44.7	24.8	45.5	45,620	0.230	22.9	16.4	42.9	159.9	9.16	0.079	115	6.91	6.92
11	5-Jul-01	1293	Balanced	235	710	566	2,146	2.61	58.2	0.62	3458	45.2	29.5	47.5	45,620	0.234	24.0	16.8	42	161.9	9.28	0.073	122	6.68	6.67
11	6-Jul-01	1294	Balanced	235	710	560	2,171	2.61	62.1	0.63	3485	44.3	26.4	47.0	45,620	0.228	23.4	16.5	41.8	160.5	9.19	0.073	152	6.8	6.74
11	8-Jul-01	1296	Balanced	235	710	564	2,155	2.52	69.0	0.62	3461	45.1	26.8	46.0	45,620	0.223	22.6	16.4	42.8	158.1	9.06	0.072	118	7.17	7.12
11	9-Jul-01	1297	Balanced	235	710	549	2,145	2.80	62.0	0.63	3489	43.6	21.9	45.5	45,620	0.222	24.0	16.0	42.4	155.5	8.91	0.071	185	6.82	6.96
11	10-Jul-01	1298	Balanced	235	710	542	2,100	3.05	60	0.65	3593	42.8	20.2	46.0	45,620	0.218	24.2	15.2	42.3	153.5	8.80	0.070	195	6.49	6.64
11	11-Jul-01	1299	Balanced	235	710	524	2,181	2.55	73.2	0.62	3433	45.3	22.4	43.0	45,620	0.206	20.9	15.1	43.6	144.4	8.27	0.068	160	6.99	7.13
11	12-Jul-01	1300	Balanced	235	710	526	2,158	2.91	66.7	0.63	3530	45.8	25.6	44.0	45,620	0.206	22.6	14.7	43.1	146.4	8.40	0.070	203	7.01	7.44
11	13-Jul-01	1301	Balanced	235	710	524	2,181	2.54	72.7	0.62	3440	45.5	25.5	44.5	45,620	0.199	20.7	15.0	43.8	143.6	8.23	0.068	159	6.94	7.03
11	14-Jul-01	1302	Balanced	235	710	533	2,118	2.92	80.7	0.65	3589	44	20.3	44.0	45,620	0.202	21.6	14.2	44.5	143.6	8.23	0.068	185	6.54	6.90
11	15-Jul-01	1303	Balanced	235	710	529	2,154	2.71	80.2	0.64	3543	44	18.6	43.5	45,620	0.196	20.6	14.2	44.6	142.4	8.17	0.068	187	6.27	6.49
11	16-Jul-01	1304	Balanced	237	710	510	2,189	2.89	55.7	0.63	3477	43.7	18.2	43.5	45,620	0.193	22.5	14.6	42.5	142.3	8.16	0.069	190	6.32	6.66
11	17-Jul-01	1305	Balanced	237	710	495	2,162	2.86	59.7	0.63	3494	46	21.3	41.5	45,620	0.184	21.4	14.0	43.1	138.0	7.91	0.066	185	6.75	7.07
11	19-Jul-01	1307	Balanced	240	710	658	2,089	2.86	60.2	0.67	3516	42.4	23.3	51.0	47,820	0.240	28.0	18.4	41.4	190.8	10.42	0.087	146	7.1	6.64
11	22-Jul-01	1310	Balanced	240	710	655	2,102	2.72	89.6	0.63	3312	46.5	26.2	45.5	47,820	0.232	27.8	19.1	42.8	183.9	10.06	0.096	161	6.52	6.46
11	23-Jul-01	1311	Balanced	235	710	471	2,196	2.62	27.2	0.61	3219	46.4	25.4	45.0	47,820	0.217	26.3	15.3	40.1	141.0	7.70	0.075	147	6.71	7.13
11	28-Jul-01	1316	Balanced	250	710	566	2,179	2.67	47.3	0.65	3329	47.0	27.6	46.0	47,820	0.172	24.9	17.2	41.3	164.8	8.99	0.085	141	7.86	7.30
11	29-Jul-01	1317	Balanced	250	710	577	2,221	2.68	48.3	0.66	3393	48.7	33.5	47.0	47,820	0.174	24.9	17.2	41.4	167.4	9.14	0.085	148	8.36	7.41
11	31-Jul-01	1319	Balanced	251	710	619	2,144	2.47	70.6	0.65	3349	47.2	30.4	47.5	47,820	0.174	24.7	17.9	42.7	173.9	9.51	0.087	154	7.85	6.88
11	1-Aug-01	1320	Balanced	250	710	606	2,176	3.27	71.3	0.65	3356	46.6	26.6	46.0	47,820	0.172	24.2	17.4	42.4	171.2	9.46	0.089	160	6.69	6.98
11	2-Aug-01	1321	Balanced	250	710	600	2,156	2.48	75.4	0.65	3347	46	23.3	45.0	47,820	0.166	23.7	17.2	42.8	168.4	9.21	0.089	155	7.56	6.81
11	3-Aug-01	1322	Balanced	250	710	560	2,170	2.52	62.8	0.64	3322	46.4	22.7	44.0	47,820	0.158	23.2	16.6	42.1	159.5	8.72	0.087	156	8.47	8.72
11	4-Aug-01	1323	Balanced	250	710	556	2,169	2.53	61.7	0.64	3311	47.7	24.4	43.0	47,820	0.156	23.0	16.4	42.4	157.3	8.6	0.087	157	7.61	6.92
11	5-Aug-01	1324	Balanced	250	710	544	2,177	2.50	59.2	0.64	3299	46.6	18.8	42.5	47,820	0.154	22.5	16.1	42.3	154.5	8.45	0.087	152	6.94	6.28
2000-8	17-Sep-01	24	Balanced	216	573	580	1,798	3.33	71.8	0.65	3320	40.7	20.8	45	41580	0.537	1.159	32.5	19.9	43	161.8	10.17	0.086	105	5.38
2000-8	24-Sep-01	31	Balanced	218	666	649	2,080	3.53	54.9	0.64	3804	43	33.5	49	41580	0.532	1.095	35.4	20.6	41.2	189	11.87	0.092	108	6.05
2000-8	25-Sep-01	32	Balanced	218	675	663	2,100	3.64	58.5	0.64	3841	43	33.3	49	41580	0.530	1.09	36.2	20.6	41.3	192.4	12.08	0.094	107	5.79

fresh methanol synthesis catalyst (which is described below).

29K-01 Recycle Compressor Inspection

In coordination with the timing for the in-situ activation of the methanol synthesis catalyst in the LPMEOH™ Reactor, an inspection of the 29K-01 recycle compressor was performed by personnel from Eastman, Air Products, and the equipment supplier. The recommended time for this initial inspection is 3 years after start-up; since the machine was operating well, the inspection was deferred until the 5th year of service. The only maintenance that was performed was the cleaning of accumulated solids on the impellor and the replacement of the dry gas seals. All other mechanical components were in excellent shape. Work began on 13 August 2001, and the compressor was reassembled by 16 August 2001. On 17 August 2001, the compressor was successfully tested to check vibration and bearing temperatures.

In-situ Catalyst Activation – August 2001

The LPMEOH™ Demonstration Unit was shutdown on 06 August 2001 to prepare for the in-situ catalyst activation procedure (the procedure used the LPMEOH™ Reactor [instead of the 29C-30 catalyst reduction vessel] to prepare fresh methanol synthesis catalyst for production of methanol by reacting metal oxides [for example, copper oxide] with a reductant such as CO or H₂ to produce the base metal plus either CO₂ or H₂O). The slurry contents of the reactor were cooled and transferred to the 29D-02 slurry tank. Batches of slurry were then transferred from the slurry tank to the 29C-30 catalyst reduction vessel for final cooling and draining into drums. A total of 305 drums or 88,150 pounds of slurry was removed from the reactor.

Beginning on 13 August 2001, batches of fresh catalyst were mixed with mineral oil in the catalyst reduction vessel, heated to 150°C, and transferred, without being reduced or activated, to the slurry tank. A total of ten batches of fresh catalyst slurry, each containing about 4,000 pounds of fresh catalyst, was prepared in this manner. After the final transfer, the total catalyst inventory in the slurry tank was 41,580 pounds of fresh catalyst, which is approximately the design catalyst inventory for the LPMEOH™ Reactor. The catalyst loading and transfer operation was completed on 21 August 2001.

The common inlet and outlet line of the slurry tank (2" diameter) developed a plug during the transfer of the tenth batch of fresh catalyst from the catalyst reduction vessel. After modifications to the piping system were made, Eastman operations personnel were able to clear this obstruction in the early morning hours of 22 August 2001. The contents of the slurry tank were then successfully transferred into the LPMEOH™ Reactor. The transfer of fresh catalyst slurry was completed at 0847 hrs on 22 August 2001.

Immediately upon completion of the slurry transfer, the recycle compressor was started to begin to cool the reactor contents to the desired initial temperature for catalyst reduction. The reactor inlet flow was set at approximately 300 KSCFH, and 52 KSCFH of make-up nitrogen was introduced at the compressor suction. Reactor pressure was set at 85 psig. Cooling was completed at 2250 hrs on 22 August 2001 after the reactor temperature reached 88°C.

After reactor cooling was completed, the in-situ catalyst activation procedure was started at 2300 hrs on 22 August 2001. In addition to the 52 KSCFH of nitrogen, a small quantity of Balanced Gas was introduced into the discharge of the recycle compressor to ensure that sufficient reductant was present at all times in the reactor inlet. The in-situ catalyst activation procedure ended at 0100 hrs on 24 August 2001.

The progress of the in-situ catalyst activation procedure was tracked by evaluating the uptake of reductant by the catalyst. A preliminary uptake of 70% of the theoretical value was calculated based upon analytical data and measured flowrates. The LPMEOH™ Demonstration Unit was then restarted at 1200 hrs on 24 August 2001.

Catalyst Life (η) – August - September 2001

Appendix C, Figure 2 plots $\log \eta$ versus days onstream from August 2001 to the end of the reporting period (following the completion of the in-situ catalyst activation procedure).

A reactor catalyst sample from 05 September 2001 was submitted for chemical analysis and for a check of catalyst activity in the autoclave to determine the effectiveness of the in-situ catalyst activation procedure. Results from the activity determination from this sample were significantly lower than the results of similar tests using fresh catalyst. Reactor performance, however, appears to indicate adequate catalyst activity. Subsequent reactor samples from later in September 2001 have indicated higher activity results in the autoclave when compared with both the 05 September 2001 sample and with fresh catalyst. Additional tests are being developed to resolve these discrepancies and to develop the means to compare data from different operating periods.

Following the completion of the in-situ catalyst activation procedure, conditions were selected to deal with the hyperactivity that typically occurs when fresh methanol synthesis catalyst is initially exposed to syngas. During the first few weeks of operation, conditions were varying between a reactor pressure of 550-600 psig, reactor temperature of 212-220°C, and flowrate of Balanced Gas of 500-525 KSCFH. Coincident with this catalyst run-in period was a period of variability in the composition of Balanced Gas, the combination of which resulted in the limited number of material balance cases that are provided in Table D.3-1. Once this 3-week period had passed, operating conditions were set at 218°C reactor temperature, 685 psig reactor pressure, and 600-650 KSCFH of Balanced Gas for the remainder of the quarter.

Analyses of catalyst samples to determine changes in physical characteristics and levels of poisons have continued. Appendix C, Table 2 summarizes the results for the second catalyst campaign (prior to in-situ catalyst activation). Prior to the in-situ activation of the fresh charge of catalyst, analysis of catalyst samples showed an increase in the levels of arsenic, sulfur, and iron (all of which are known poisons to methanol synthesis catalysts).

Appendix C, Table 3 summarizes the results for the third catalyst campaign (following the completion of the in-situ catalyst activation procedure). Chemical analysis of catalyst samples that have been taken following the completion of the in-situ catalyst activation

procedure indicated the presence of all expected crystal phases, and no accumulation of trace contaminants was detected.

Sparger Resistance

The performance of the gas sparger, which was designed by Air Products and first installed into the LPMEOH™ Reactor prior to the restart of the LPMEOH™ Demonstration Unit in March of 1999, was monitored. The device had been inspected and cleaned during the March 2001 biennial outage. Appendix C, Figure 3 plots the average daily sparger resistance coefficient for the second catalyst campaign (prior to in-situ catalyst activation). Appendix C, Figure 4 plots the average daily sparger resistance coefficient for the third catalyst campaign (which began with the completion of the in-situ catalyst activation procedure) until the end of the reporting period. The data for this plot, along with the corresponding average pressure drop, are also included in Table D.3-1. Data for the third catalyst campaign were limited due to the number of material balance points following the completion of the in-situ catalyst activation procedure (described above). The sparger resistance continues to show no significant increase over time, which is consistent with the operating history with this device. The measured pressure drop is slightly greater than earlier performance, which may be related to maintenance which was performed on the differential pressure transmitters during the March 2001 biennial outage. The performance of the sparger will continue to be monitored closely for any changes.

D.4 Planning and Administration

The paper entitled “Liquid Phase Methanol (LPMEOH™) Process Development Demonstration Plant Availability” was released for presentation at the Gasification Technologies Conference in San Francisco, CA (07-10 October 2001).

Work began on developing the poster which will be displayed at the Clean Coal and Power Conference (formerly the Clean Coal Technology Conference) in Washington, DC (19-20 November 2001).

The draft paper entitled “Liquid Phase Methanol (LPMEOH™) Process Development” was prepared; this paper is scheduled to be included for the proceeding of the 18th Annual International Pittsburgh Coal Conference in Newcastle, Australia (04-07 December 2001).

The Milestone Schedule Status Report and the Cost Management Report, through the period ending 30 September 2001, are included in Appendix E. These two reports show the current schedule, the percentage completion and the latest cost forecast for each of the Work Breakdown Structure (WBS) tasks. One hundred percent (100%) of the \$38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 30 September 2001. Eighty-two percent (82%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 September 2001.

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

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.
- Assess the performance of the methanol synthesis catalyst following the completion of the in-situ catalyst activation procedure.
- Continue executing Phase 3, Task 2.1 Methanol Operation per the Demonstration Test Plan. Focus activities on temperature programming to maintain the required methanol productivity, monitoring catalyst activity, assessing the performance of the catalyst guard bed, and monitoring the performance of the gas sparger in the reactor.
- Issue a revision to the draft Topical Report on the objectives and results of the off-site, product-use test program for stabilized methanol from the LPMEOH™ Process.
- Issue the fiscal year 2002 cost plan to DOE.
- Schedule a Project Review Meeting with DOE.

F. Conclusion

The LPMEOH™ Demonstration Unit operated at 96.2% availability during this quarter. The forced downtime experienced this month (66.6 hours) was associated with the changeout and reduction of the adsorbent in the catalyst guard bed (described below). There were also two short syngas interruptions that were experienced on 17 September 2001 (8 hours duration) and 29 September 2001 (21 hours duration).

At the start of the reporting period, a catalyst addition was undertaken during the quarter to raise the catalyst activity. A single batch of fresh catalyst was activated and added to the reactor on 18 July 2001. After this batch of freshly activated catalyst was transferred into the reactor, the catalyst inventory was calculated to be 47,820 pounds.

A catalyst deactivation rate of 0.60% per day was calculated for the period 19 June 2001 to 15 July 2001 (26 days). This deactivation rate was achieved with the catalyst guard bed bypassed and out of service. This is a slightly higher deactivation rate than the results that have been calculated over the past 8 months.

For the month of July 2001, the flowrate of Balanced Gas was controlled at an average value of 560 KSCFH, the reactor pressure was set an average of 710 psig, and reactor temperature was maintained at 235°C.

During the reporting period, Eastman agreed with a recommendation by Air Products that the adsorbent (activated carbon impregnated with copper oxide) in the catalyst guard bed should be replaced. On 24 July 2001, the catalyst guard bed was emptied and refilled with the same commercially available adsorbent. The catalyst guard bed was then prepared for reduction of the copper oxide on the adsorbent.

The guard bed adsorbent reduction procedure was started at 1700 hrs on 26 July 2001. During this reduction procedure, a dilute stream of Balanced Gas in nitrogen was preheated and used to reduce the copper oxide to copper metal in a temperature controlled manner. The temperature control during the procedure was excellent, and the reduction was completed at 0200 hrs on 28 July 2001. After cooling with nitrogen, a pressure check to full supply pressure with Balanced Gas was conducted. The adsorbent temperatures increased by about 25°C and stabilized during this examination. This observation was consistent with the temperature increase which would be expected from the adsorption of carbon monoxide on the guard bed adsorbent surface. The catalyst guard bed was bypassed after the pressure check, and was placed in service on 24 August 2001, after completion of in-situ catalyst activation of fresh methanol synthesis catalyst (which is described below).

In coordination with the timing for the in-situ catalyst activation of the methanol synthesis catalyst in the LPMEOH™ Reactor, an inspection of the recycle compressor was performed by personnel from Eastman, Air Products, and the equipment supplier. The only maintenance that was performed was the cleaning of accumulated solids on the impellor and the replacement of the dry gas seals. All other mechanical components were in excellent shape. Work began on 13 August 2001, and the compressor was reassembled by 16 August 2001. On 17 August 2001, the compressor was successfully tested to check vibration and bearing temperatures.

The LPMEOH™ Demonstration Unit was shutdown on 06 August 2001 to prepare for the in-situ catalyst activation procedure. The slurry contents of the reactor were cooled and transferred to the slurry tank. Batches of slurry were then transferred from the slurry tank to the catalyst reduction vessel for final cooling and draining into drums. A total of 305 drums or 88,150 pounds of slurry was removed from the reactor.

Beginning on 13 August 2001, batches of fresh catalyst were mixed with mineral oil in the catalyst reduction vessel, heated to 150°C, and transferred, without being reduced (or activated), to the slurry tank. A total of ten batches of fresh catalyst slurry, each containing about 4,000 pounds of fresh catalyst, was prepared in this manner. After the final transfer, the total catalyst inventory in the slurry tank was 41,580 pounds of fresh catalyst, which is approximately the design catalyst inventory for the LPMEOH™ Reactor. The catalyst loading and transfer operation was completed on 21 August 2001.

The common inlet and outlet line of the slurry tank (2" diameter) developed a plug during the transfer of the tenth batch of fresh catalyst slurry from the catalyst reduction vessel. After modifications to the piping system were made, Eastman operations personnel were able to clear this obstruction in the early morning hours of 22 August 2001. The contents of the slurry tank were then successfully transferred into the LPMEOH™ Reactor. The transfer of fresh catalyst slurry was completed at 0847 hrs on 22 August 2001.

Immediately upon completion of the slurry transfer, the recycle compressor was started to begin to cool the reactor contents to the desired initial temperature for catalyst reduction. Cooling was completed at 2250 hrs on 22 August 2001 after the reactor temperature reached 88°C.

After reactor cooling was completed, the in-situ catalyst activation procedure was started at 2300 hrs on 22 August 2001. Make-up nitrogen flow was controlled at 52 KSCFH, and a small quantity of Balanced Gas was introduced into the discharge of the recycle compressor to ensure that sufficient reductant was present at all times in the reactor inlet. The in-situ catalyst activation procedure ended at 0100 hrs on 24 August 2001.

The progress of the in-situ catalyst activation procedure was tracked by evaluating the uptake of reductant by the catalyst. A preliminary uptake of 70% of the theoretical value was calculated based upon analytical data and measured flowrates. The LPMEOH™ Demonstration Unit was then restarted at 1200 hrs on 24 August 2001.

A reactor catalyst sample from 05 September 2001 was submitted for chemical analysis and for a check of catalyst activity in the autoclave to determine the effectiveness of the in-situ catalyst activation procedure. Results from the activity determination from this sample were not comparable to similar tests using fresh catalyst. Reactor performance, however, appears to indicate adequate catalyst activity. Subsequent reactor samples from later in September 2001 have indicated better activity results in the autoclave. Additional tests are being developed to resolve these discrepancies and to develop the means to compare data from different operating periods.

Following the completion of the in-situ catalyst activation procedure, conditions were selected to deal with the hyperactivity that typically occurs when fresh methanol synthesis catalyst is initially exposed to syngas. During the first few weeks of operation, conditions were varying between a reactor pressure of 550-600 psig, reactor temperature of 212-220°C, and flowrate of Balanced Gas of 500-525 KSCFH. Once the hyperactivity period had passed, operating conditions were set at 218°C reactor temperature, 685 psig reactor pressure, and 600-650 KSCFH of Balanced Gas for the remainder of the quarter.

Analyses of catalyst samples to determine changes in physical characteristics and levels of poisons have continued. Prior to the in-situ activation of the fresh charge of catalyst, analysis of all pertinent poisons showed an increase in the levels of arsenic, sulfur, and iron (all of which are known poisons to methanol synthesis catalysts). Chemical analysis of catalyst samples that have been taken following the completion of the in-situ catalyst activation procedure indicated the presence of all expected crystal phases, and no accumulation of trace contaminants was detected.

The performance of the gas sparger, which was designed by Air Products and first installed into the LPMEOH™ Reactor prior to the restart of the LPMEOH™ Demonstration Unit in March of 1999, was monitored. The device had been inspected and cleaned during the March 2001 biennial outage. The sparger resistance continues to show no significant

increase over time, which is consistent with the operating history with this device. The performance of the sparger will continue to be monitored closely for any changes.

During the reporting period, a total of 3,942,198 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, about 83.0 million gallons of methanol have 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.

A draft Topical Report entitled “Off-Site Testing of Stabilized Methanol from the Liquid Phase Methanol (LPMEOH™) Process” was sent to DOE for review. Comments were received on the main body of the report; this section provides the perspective of the LPMEOH™ Demonstration Project (the reports from the seven participants will be included in volumes for Transportation and Power Generation Systems).

Activities associated with Design Verification Testing (DVT) of the LPDME Process have been completed. A Topical Report, which presents the results of the DVT at the LaPorte AFDU, was approved by DOE and issued (March 2001). The LPMEOH™ Demonstration Project will prepare a separate Topical Report on the market analysis for DME and review of the economics of the LPDME Process.

The paper entitled “Liquid Phase Methanol (LPMEOH™) Process Development Demonstration Plant Availability” was released for presentation at the Gasification Technologies Conference in San Francisco, CA (07-10 October 2001). Work began on developing the poster which will be displayed at the Clean Coal and Power Conference (formerly the Clean Coal Technology Conference) in Washington, DC (19-20 November 2001). The draft paper entitled “Liquid Phase Methanol (LPMEOH™) Process Development” was prepared; this paper is scheduled to be included for the proceeding of the 18th Annual International Pittsburgh Coal Conference in Newcastle, Australia (04-07 December 2001).

One hundred percent (100%) of the \$38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 30 September 2001. Eighty-two percent (82%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 September 2001.

APPENDICES

APPENDIX A - SIMPLIFIED PROCESS FLOW DIAGRAM

APPENDIX B - SAMPLES OF DETAILED MATERIAL BALANCE REPORTS

APPENDIX C - RESULTS OF DEMONSTRATION UNIT OPERATION

**Table 1 - Summary of LPMEOH™ Demonstration Unit Outages -
July/September 2001**

Table 2 - Summary of Catalyst Samples - Second Catalyst Batch

Table 3 - Summary of Catalyst Samples - Third Catalyst Batch

Figure 1 - Catalyst Age (η): March 2000 - August 2001

Figure 2 - Catalyst Age (η): August - September 2001

**Figure 3 - Sparger Resistance Coefficient vs. Days Onstream
(December 1999 - August 2001)**

**Figure 4 - Sparger Resistance Coefficient vs. Days Onstream
(August - September 2001)**

Table 1
Summary of LPMEOH™ Demonstration Unit Outages - July/September 2001

Operation Start	Operation End	Operating Hours	Shutdown Hours	Reason for Shutdown
7/1/01 00:00	7/25/01 15:08	591.1	66.6	Guard Bed Changeout & Reduction
7/28/01 09:47	8/6/01 07:45	214.0	424.7	In-situ Activation
8/24/01 00:25	9/17/01 18:00	593.6	8.2	Syngas Outage
9/18/01 02:14	9/29/01 13:00	274.8	20.8	Syngas Outage
9/30/01 09:50	9/30/01 23:59	14.2		End of Reporting Period
Total Operating Hours			1687.6	
Syngas Available Hours			1754.2	
Plant Availability, %			96.20	

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	
K9806-2	Reactor Sample 6/16/98	272.3	117.2		86.4	31.1	220	1110	
K9807-2	Reactor Sample 7/8/98	263.2	108.6		88.7	27.6	277	1045	
K9807-3	Reactor Sample 7/29/98	412*	112		93.25	30.95	209	1620	
K9807-4	Reactor Sample 8/14/98	353.9*	124		121.5	37.1	213.5	1215	
K9809-1	Reactor Sample 9/24/98	347.4	129.8		69.6	29.8	326	1149	
K9810-1	Reactor Sample 10/5/98	331.1	130.4						
K9811-2	Reactor Sample 11/25/98	293.9			57.3	23.4	264	1400	<100
K9812-1	Reactor Sample 12/29/98	283.1			72.3	20.4	260	1300	<100
K9901-1	Reactor Sample 1/15/99	252.5	61.4						
K9902-1	Reactor Sample 2/17/99	474.7	133.6		82.6	22.2	385	1490	<300
K9904-3	Reactor Sample 4/27/99	417.8	110.4	15	131	18.2	348	1460	<30
K9906-1	Reactor Sample 6/1/99	517	105	43	109	19.7	316	1680	40
K9907-1	Reactor Sample 7/13/99	446	116	59	175	19.7	488	1810	30
K9908-2	Reactor Sample 8/31/99	632	117	56	161	15.1	406	1470	50
K9909-2	Reactor Sample 9/21/99	357	109	64	132	11.2	253	1050	nd
K9910-2	Reactor Sample 10/19/99	135	94	55	157	15.4	343	1270	30
K9911-1	Reactor Sample 11/4/99				184	12.8	335	1580	na
K9912-1	Reactor Sample 12/8/99	797	121	60	167	13.9	248	1400	40
K0001-1	Reactor Sample 1/5/00	613	105	63	199	10.8	292	1190	nd
K0001-2	Reactor Sample 1/19/00				205	10.0	432	1250	na
K0003-1	Reactor Sample 3/2/00	187	88.7	67	137	8.2	226	1010	30
K004-1	Reactor Sample 4/23/00	175	114.5	59	164	6.6	248	1240	20
K0007-1	Reactor Sample 7/18/00	174	107.5	69	166	< 9.6	349	1270	30
K0008-1	Reactor Sample 8/31/00	385	90.4	66	186	< 10	379	1080	50
K0009-3	Reactor Sample 9/19/00	157	96	67	145	< 10	273	1390	nd
K0011-1	Reactor Sample 11/7/00	248	79.6	70	120	< 10	237	1490	nd
K0011-2	Reactor Sample 11/27/00	263	109.2		128	< 10	258	1470	20
K0012-2	Reactor Sample 12/19/00				100	< 10	410	1480	
K0101-1	Reactor Sample 1/3/01	280	110	66	166	< 10	355	1980	30
K0102-1	Reactor Sample 2/7/01	172	104	73	121	< 10	375	1750	40
K0103-1	Reactor Sample 3/7/01	164	138	69	90	< 10	416	1830	20
K0106-1	Reactor Sample 6/5/01	243	103	62	213	< 10	527	1510	nd
K0107-1	Reactor Sample 7/11/01	186	159	65	254	< 30	640	1789	40
K0108-1	Reactor Sample 8/6/01	208	128	64	283	< 30	774	1849	40

Notes:

- 1) nd = none detected
- 3) na = data not available

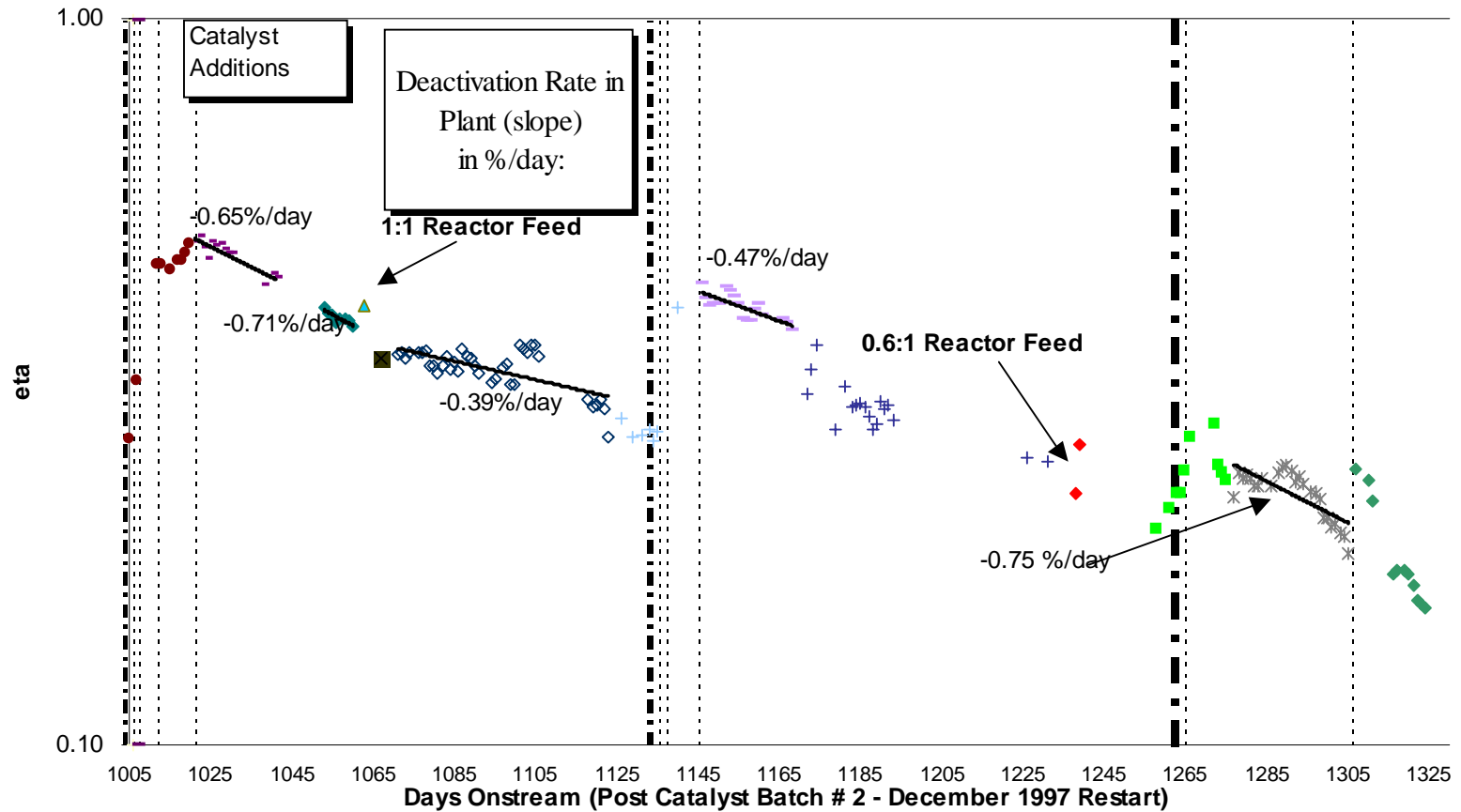
Table 2
Summary of Catalyst Samples - Third Catalyst Batch

Sample	Identity	XRD		BET m ² /g	Analytical (ppmw)				
		Cu	ZnO		Fe	Ni	S	As	Cl
K0109-1	Reactor Sample 9/5/01	178	90		48	≤19	< 140	≤73	nd
K0109-2	Reactor Sample 9/12/01	188	88						
K0109-3	Reactor Sample 9/19/01	185			52	< 10	< 140	≤70	nd
K0110-1	Reactor Sample 10/3/01	181	78						
K0110-2	Reactor Sample 10/17/01	203	98						

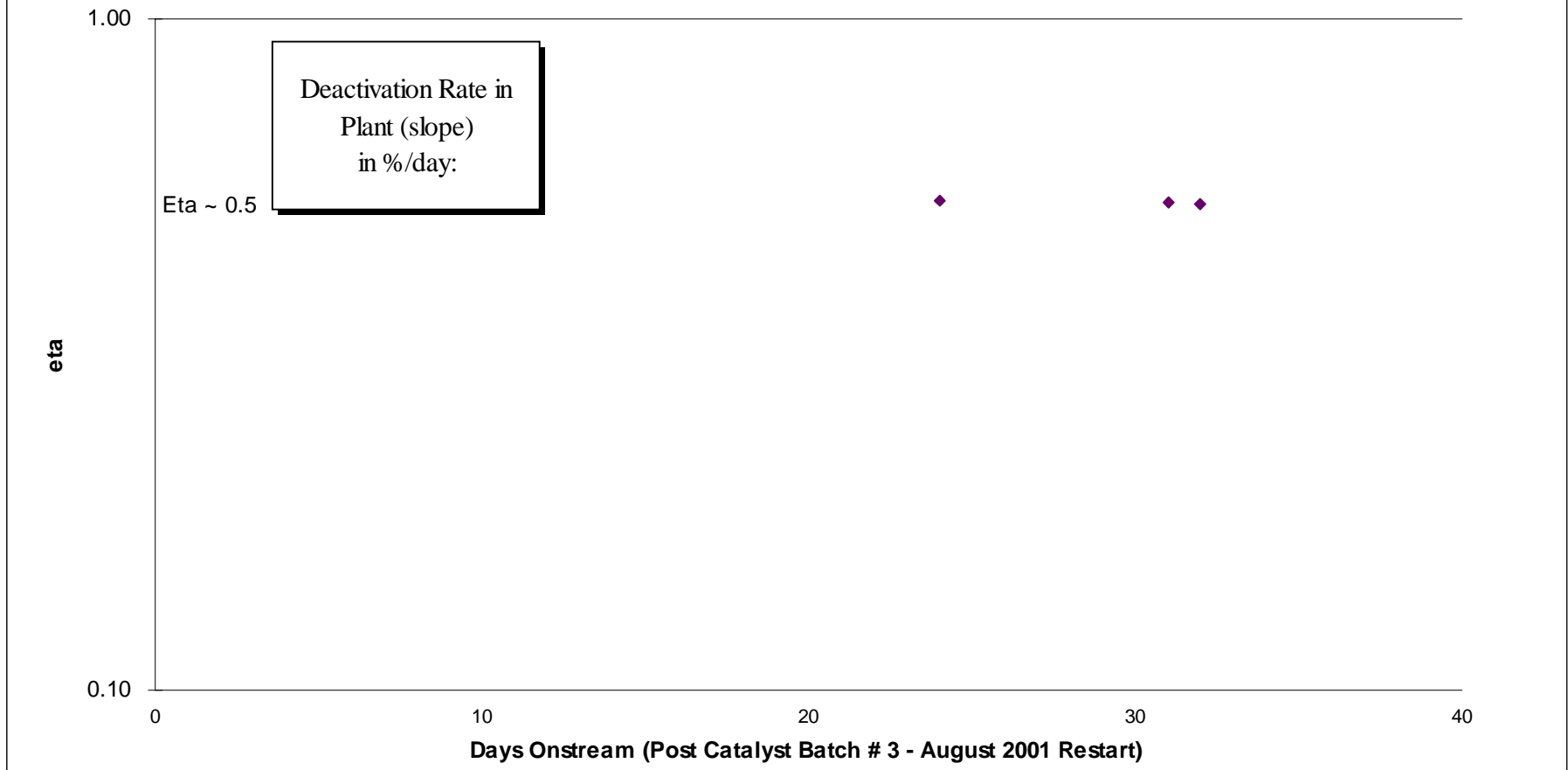
Notes:

- 1) nd = none detected
- 3) na = data not available

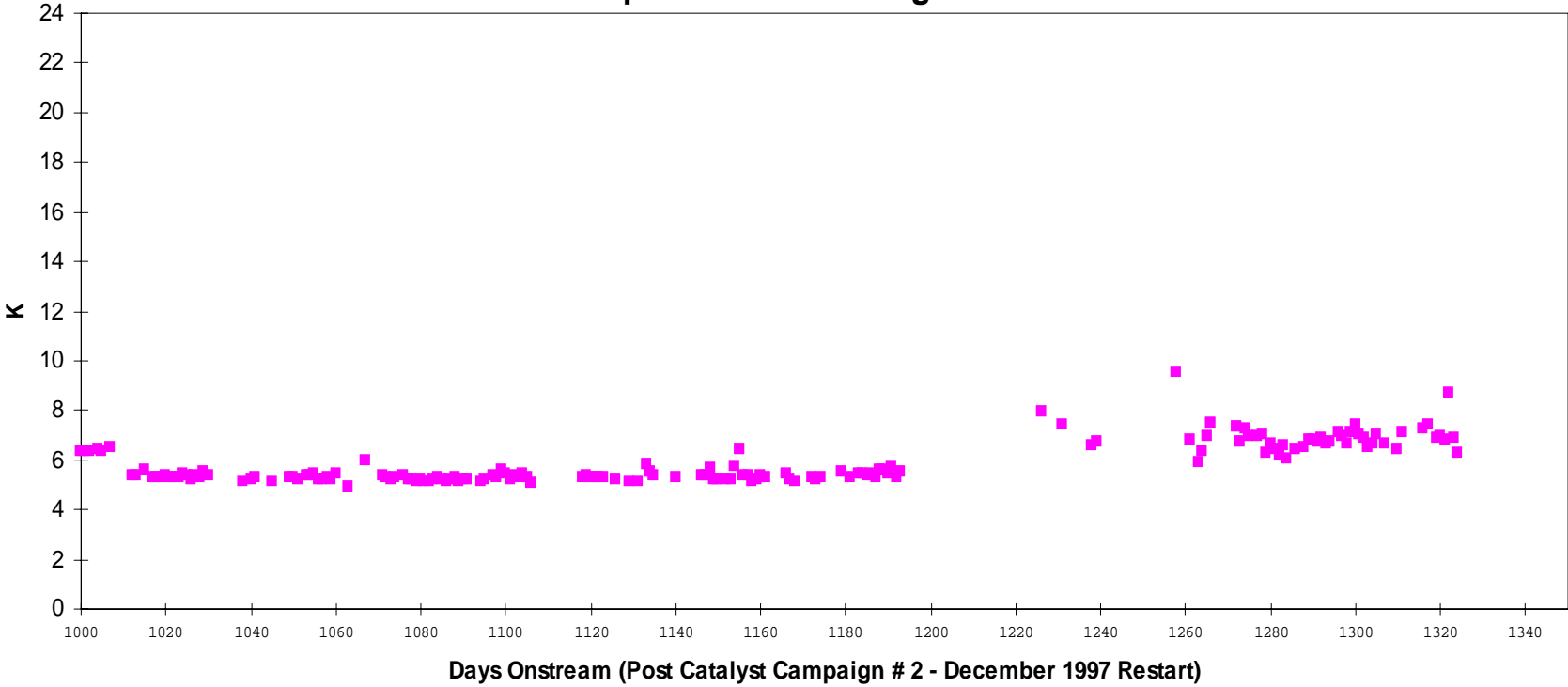
**Figure 1 - Kingsport LPMEOH™ Catalyst Age (eta):
September 2000 - August 2001**



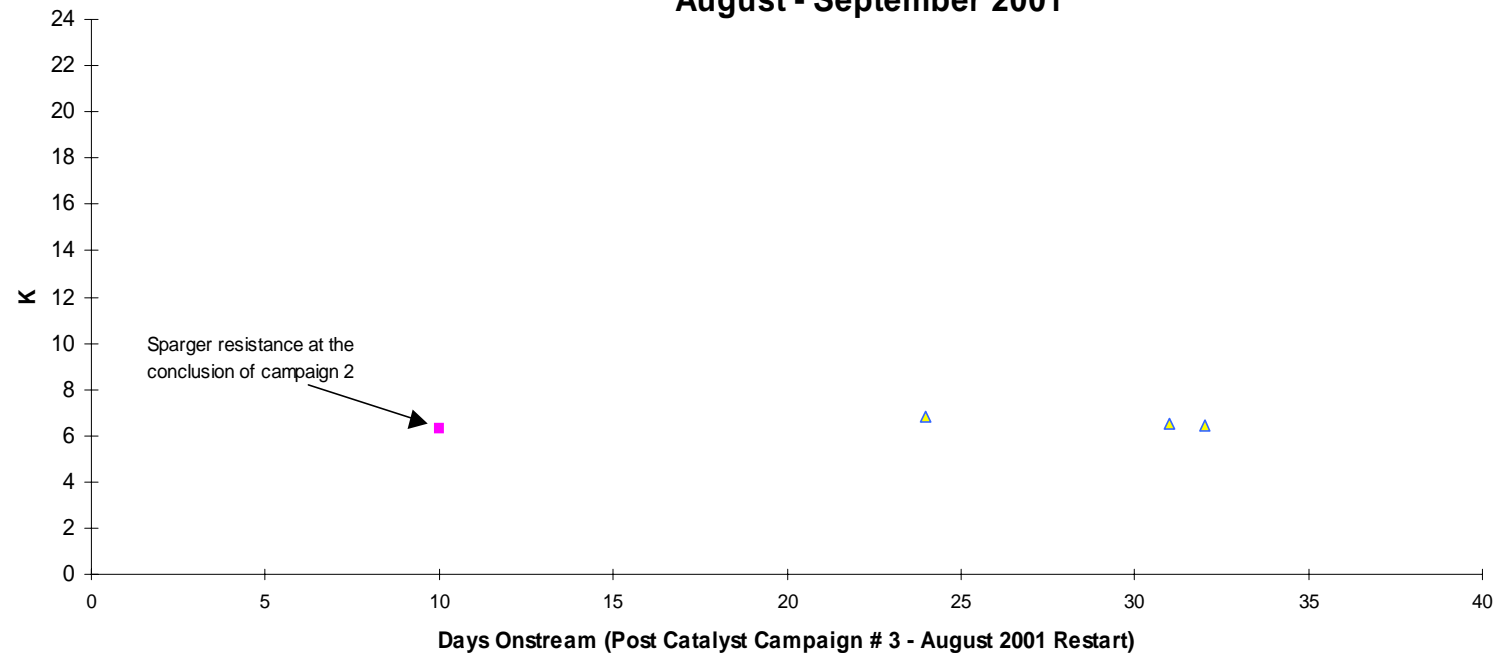
**Figure 2 - Kingsport LPMEOH™ Catalyst Age (eta):
September 2001 - October 2001**



**Figure 3 - Kingsport LPMEOH™
Sparger Resistance Coefficient
September 2000 - August 2001**



**Figure 4 - Kingsport LPMEOH™
Sparger Resistance Coefficient
August - September 2001**



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