

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

TECHNICAL PROGRESS REPORT NO. 34

For The Period

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Prepared by

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and

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for the

Air Products Liquid Phase Conversion Company, L.P.

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Abstract

The Liquid Phase Methanol (LPMEOH™) Demonstration Project at Kingsport, Tennessee, is a \$213.7 million cooperative agreement between the U.S. Department of Energy (DOE) and Air Products Liquid Phase Conversion Company, L.P. (the Partnership) to produce methanol from coal-derived synthesis gas (syngas). Air Products and Chemicals, Inc. (Air Products) and Eastman Chemical Company (Eastman) formed the Partnership to execute the Demonstration Project. The LPMEOH™ Demonstration Unit completed a successful 69-month demonstration test program on 31 December 2002 at a site located at the Eastman chemicals-from-coal complex in Kingsport.

The LPMEOH™ Demonstration Unit operated at 90.9% availability during this quarter (Availability is defined as the percentage of time that the LPMEOH™ Demonstration Unit was available to operate, with the exclusion of scheduled outages.); the availability during the 69-month demonstration test program was 97.5%. There were two outages (lasting 143 hours and 56 hours) associated with changeout, reduction, and thermal treatment of the adsorbent (copper-impregnated activated carbon) in the 29C-40 catalyst guard bed that accounted for all of the downtime experienced during this reporting period. There were two syngas interruptions that were experienced on 10 October 2002 (12 hours duration) and 31 December 2002 (14.2 hours duration).

During this quarter, the LPMEOH™ Demonstration Unit was operated at a reactor temperature of 214-215°C. The flowrate of the primary syngas feed (Balanced Gas) was controlled at an average value of 551 KSCFH. The reactor pressure was operated at 450 psig until 11 November 2002, at which time the pressure was increased to 500 psig in order to reduce the reactor purge flowrate and to determine if the higher operating pressure would affect the performance of the methanol synthesis catalyst.

A catalyst deactivation rate of 0.1% per day was calculated for the period 25 October 2002 to 09 November 2002 (16 days). This is a significantly lower deactivation rate than the results that have been generally calculated over the past 3 years (averaging between 0.6% and 0.7% per day). As a basis of comparison, the calculated deactivation rate from the 4-month proof-of-concept run at the LaPorte AFDU in 1988/89 was 0.4% per day. (This run was performed on carbon monoxide (CO)-rich syngas derived from natural gas at a reactor temperature of 250°C and pressure of 750 psig.)

For the period 12 November 2002 to 04 December 2002 (23 days), the calculated catalyst deactivation rate was 0.87% per day. This is similar to the historical performance at the LPMEOH™ Demonstration Unit, but a significant increase over the results from the prior 3 weeks of operation. This change could have been caused by a trace contaminant breakthrough of the adsorbent in the catalyst guard bed; as a consequence, a thermal treatment of the adsorbent to provide increased capacity for arsine removal was performed on 06 December 2002.

For the period 16 December 2002 to 30 December 2002 (15 days), the calculated catalyst deactivation rate was 0.13% per day. The return to excellent catalyst deactivation results following the thermal treatment of the adsorbent in the catalyst guard bed provides evidence of the impact of the presence of trace contaminants in coal-derived syngas on the life of methanol synthesis catalyst.

Due to this low catalyst deactivation rate, the practice of temperature programming was not required during the reporting period (temperature programming involves the increase of reactor temperature as necessary to control the reactor purge flowrate and maintain reactor volumetric productivity).

Samples of spent and recently activated fresh catalyst were collected during the quarter to determine changes in levels of trace contaminants (such as iron, nickel, sulfur, and arsenic) that are known poisons to methanol synthesis catalysts. No significant increase in the concentration of any of these poisons was observed.

The LPMEOH™ Demonstration Unit was shut down at 1600 hours on 06 October 2002 to prepare for the replacement of the adsorbent in the catalyst guard bed. (During this outage, Eastman also replaced the adsorbent (230 cubic feet of manganese dioxide) in the 19C-30 catalyst guard bed (located upstream of both the LPMEOH™ Demonstration Unit and the fixed-bed methanol plant) with fresh material; the adsorbent was last changed in June of 1999.)

A total of 5,225 pounds of fresh copper-impregnated activated carbon was charged to the catalyst guard bed on 10 October 2002. This material must be chemically reduced using dilute syngas in nitrogen prior to use. (In this case, reduction refers to the reaction of the copper oxide with a reductant such as CO or hydrogen (H₂) to copper metal and either carbon dioxide (CO₂) or water (H₂O)). The temperature of the adsorbent was first increased to 80°C using heated nitrogen. The 12-hour syngas interruption (mentioned previously) delayed the start of the reduction procedure. A further delay was experienced due to the discovery that the wrong trim was installed in the valve that controls the flowrate of Balanced Gas during the reduction process. The proper trim was installed, and Balanced Gas was introduced at 1418 hours on 11 October 2002. The reduction proceeded without incident and was completed at 1754 hours on 12 October 2002.

Following the completion of the steps to purge and cool the catalyst guard bed with nitrogen, the LPMEOH™ Demonstration Unit was restarted at 0324 hours on 13 October 2002. The catalyst guard bed was brought on-line at 1030 hours on 13 October 2002. The guard bed internal temperatures increased by about 38-40°C and then stabilized following the introduction of the syngas. This was consistent with earlier observations associated with the start-up of the catalyst guard bed.

Based upon results over the past year, an operating schedule for the adsorbent has been developed. Fresh adsorbent can be used for about two months before breakthrough of arsine can be expected. A thermal treatment can then be performed on the adsorbent to provide increased capacity for arsine removal, and the material can be used for another month before the adsorbent needs to be replaced. However, due to the increase in the calculated rate of

catalyst deactivation that was observed in November of 2002, the timing for performing the thermal treatment on the adsorbent was started on 06 December 2002 after 7 weeks of service. The catalyst guard bed was brought back online on 08 December 2002; no excessive temperature rise of the adsorbent was measured.

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 June 2002 outage prior to the second in-situ activation of methanol synthesis catalyst. The sparger resistance continues to show no significant increase over time, which is consistent with the operating history with this device.

During the reporting period, a total of 4,163,251 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, about 103.9 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 DOE project review meeting was held during the week of 04 November 2002 in Pittsburgh. The performance of the LPMEOH™ Demonstration Unit since the last meeting (December 2001) was the primary topic of discussion.

Comments were received from DOE on the report on publicly available technical data on the Eastman chemicals-from-coal complex in Kingsport. This report provides operational performance of the chemicals-from-coal complex in Kingsport as well as specific data on the major feed and effluent streams for the coal gasification and syngas cleanup systems. An updated version was prepared and sent to DOE for review and comment.

A first draft of the topical report entitled “Removal of Trace Contaminants from Coal-Derived Syngas” was submitted to DOE for review and comment.

One hundred percent (100%) of the \$40 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 31 December 2002. One hundred percent (100%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 December 2002.

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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
Availability	-	The percentage of time that the LPMEOH™ Demonstration Unit was able to operate, with the exclusion of scheduled outages
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)
Temperature Programming	-	the increase of reactor temperature as necessary to control the reactor purge flowrate and maintain reactor volumetric productivity
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 percent
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™ Demonstration Unit was designed, constructed, and completed a successful 69-month demonstration test program on 31 December 2002 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 provided the technical and engineering supervision needed to conduct the operational testing program required as part of the project. As subcontractor to Air Products, Eastman was 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 involved 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 consisted of syngas feed preparation and compression facilities, the liquid phase reactor and auxiliaries, product distillation facilities, and utilities.

The technology that was demonstrated was 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 allowed operations at Eastman to simulate electricity demand load-following in coal-based IGCC facilities. The operations also demonstrated 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 off-site product-use test program were 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). Based upon the results from the successful demonstration at Kingsport, future users will be able to utilize a local resource (coal) in a reliable (storable) and environmentally preferable way to provide the clean energy needs of local communities for electric power and transportation.

This project 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 provided the full \$213,700,000 of authorized funding, with 56.7% participant cost share and 43.3% DOE cost share.

The LPMEOH™ Demonstration Unit operated at 90.9% availability during this quarter (Availability is defined as the percentage of time that the LPMEOH™ Demonstration Unit was available to operate, with the exclusion of scheduled outages.); the availability during the 69-month demonstration test program was 97.5%. There were two outages associated with the 29C-40 catalyst guard bed that accounted for all of the downtime experienced during this reporting period. The first outage occurred on 06 October 2002 (143 hours) and was associated with the changeout and reduction of fresh adsorbent (copper-impregnated activated carbon) in the catalyst guard bed. There was also a 12-hour interruption of syngas prior to the start of the reduction of the adsorbent during that outage. The second outage occurred on 06 December 2002 (56 hours) and was associated with a thermal treatment of

the same charge of adsorbent in the catalyst guard bed. In order to provide minimum exposure of the methanol synthesis catalyst to trace contaminants, the LPMEOH™ Demonstration Unit was shutdown while the catalyst guard bed was off-line. A second syngas interruption during the quarter occurred on 31 December 2002 and lasted for 14.2 hours.

During this quarter, the LPMEOH™ Demonstration Unit was operated at a reactor temperature of 214-215°C. Variations in the composition and flowrate of the primary syngas feed (Balanced Gas) that began in August of 2002 continued until 22 October 2002. The supply then stabilized for the remainder of the reporting period; overall, the flowrate of Balanced Gas was controlled at an average value of 551 KSCFH. The reactor pressure was operated at 450 psig until 11 November 2002, at which time the pressure was increased to 500 psig in order to reduce the reactor purge flowrate and to determine if the higher operating pressure would affect the performance of the methanol synthesis catalyst.

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A DOE project review meeting was held during the week of 04 November 2002 in Pittsburgh. The performance of the LPMEOH™ Demonstration Unit since the last meeting (December 2001) was the primary topic of discussion. A follow-up meeting with senior management from DOE-NETL was held on 20 November 2002 to discuss options for extending the operation of the LPMEOH™ Demonstration Unit under sponsorship of the DOE's Clean Coal Technology Program for 6 months beyond the current end date of 31 December 2002. A formal request for this extension was sent by Air Products (on behalf of the Partnership) to DOE on 19 December 2002.

Comments were received from DOE on the report on publicly available technical data on the Eastman chemicals-from-coal complex in Kingsport. This report provides operational performance of the chemicals-from-coal complex in Kingsport as well as specific data on the major feed and effluent streams for the coal gasification and syngas cleanup systems. An updated version was prepared and sent to DOE for review and comment.

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A. Introduction

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This project is sponsored under the DOE's Clean Coal Technology Program, and its primary objective is to "demonstrate the production of methanol using the LPMEOH™ Process in conjunction with an integrated coal gasification facility." The project demonstrated 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 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 was used for downstream feedstocks and was also 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 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. The stabilized methanol from the February 1998 production campaign has been stored in an offsite facility; during the reporting period, the unused stabilized methanol was returned to Eastman for further distillation prior to use within the chemicals-from-coal complex.

A Topical Report entitled “Off-Site Testing of Stabilized Methanol from the Liquid Phase Methanol (LPMEOH™) Process” has been issued (February 2002). This report provides the results from the seven test sites.

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).

A Topical Report, which provides the status of the current market for DME and an outlook on potential market developments through 2006, has been issued (April 2002).

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

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 (H2/CO)	Purge Gas (KSCFH)	Inlet Sup. Velocity (ft/sec)	Space Velocity (1/hr-kg)	Slurry Conc. (wt% ox)	Gas Holdup (vol%)	Gas Slurry Hgt (ft)	Gasged Slurry Hgt (ft)	Catalyst Inventory (lb)	Catalyst Age (eta)	CO Conv. (%)	O-T-M Conv. (%)	Syngas Util. (SCF/lb)	Raw MeOH Prod. (TPD)	Catalyst MeOH Prod. (gmol/hr-kg)	Reactor Vol. Prod. (TPD/H3)	Overall (Rtu hr (2 F))	Sparger dP (ps)	Sparger Resist. (K")
2000-8	1-Oct-02	96	Balanced	215	450	616	1322	3.09	69.2	0.66	2782	38.0	25.2	51.0	40,040	0.996	42.30	27.00	41.90	176.40	11.51	0.082	123	5.12	7.27	
2000-8	3-Oct-02	98	Balanced	216	450	643	1331	2.78	82.4	0.66	2808	37.8	23.9	50.5	40,040	0.989	39.80	27.40	42.40	181.90	11.88	0.086	121	5.32	7.19	
2000-8	4-Oct-02	99	Balanced	215	450	645	1276	2.89	88.8	0.65	2766	38.9	24.8	49.0	40,040	0.983	40.80	27.40	41.78	180.50	11.78	0.088	126	5.01	7.25	
2000-8	5-Oct-02	100	Balanced	215	450	646	1248	3.04	93.0	0.65	2741	38.6	24.0	49.0	40,040	0.986	42.30	27.40	43.00	180.10	11.76	0.088	125	4.78	7.36	
2000-8	25-Oct-02	120	Balanced	215	450	555	1343	3.28	49.8	0.65	2741	38.8	23.1	48.0	40,040	0.937	42.20	25.40	40.70	163.50	10.66	0.081	121	5.64	8.30	
2000-8	26-Oct-02	121	Balanced	215	450	555	1344	3.23	48.1	0.65	2753	40.5	29.4	49.0	40,040	0.945	41.90	25.50	40.70	164.80	10.74	0.080	120	5.51	8.00	
2000-8	30-Oct-02	122	Balanced	215	450	555	1342	3.28	46.3	0.65	2752	39.4	26.4	49.0	40,040	0.958	42.60	26.40	40.20	165.70	10.79	0.081	123	5.55	8.13	
2000-8	28-Oct-02	123	Balanced	215	450	555	1342	3.29	47.9	0.65	2747	39.9	26.3	48.0	40,040	0.949	42.60	26.30	40.40	164.70	10.73	0.084	123	5.54	8.15	
2000-8	29-Oct-02	124	Balanced	215	450	556	1349	3.18	45.4	0.65	2756	40.9	27.7	47.0	40,040	0.955	41.70	25.60	40.40	165.10	10.75	0.082	127	5.72	8.11	
2000-8	30-Oct-02	125	Balanced	215	450	555	1352	3.15	43.0	0.66	2775	40.4	30.8	50.0	40,040	0.957	41.50	25.60	40.00	166.40	10.84	0.079	124	6.16	8.63	
2000-8	31-Oct-02	126	Balanced	215	450	560	1333	3.42	50.7	0.65	2738	39.1	26.3	49.5	40,040	0.936	43.40	25.40	40.90	164.20	10.70	0.079	123	5.12	7.88	
2000-8	1-Nov-02	127	Balanced	215	450	555	1300	3.59	53.2	0.64	2709	40.5	27.3	47.5	40,040	0.919	44.40	25.20	41.30	161.30	10.51	0.081	125	5.03	8.18	
2000-8	4-Nov-02	130	Balanced	215	450	576	1304	3.50	60.5	0.64	2723	41.2	29.3	47.5	40,040	0.948	44.50	25.80	41.70	165.80	10.80	0.083	128	4.98	7.94	
2000-8	5-Nov-02	131	Balanced	215	450	577	1308	3.38	60.2	0.64	2715	41.3	30.2	48.0	40,040	0.970	44.20	26.30	41.50	166.80	10.87	0.083	128	5.28	8.07	
2000-8	6-Nov-02	132	Balanced	215	450	563	1371	3.19	53.6	0.66	2783	41.5	30.8	48.0	40,040	0.929	41.20	25.60	40.60	166.50	10.85	0.083	126	6.06	8.74	
2000-8	7-Nov-02	133	Balanced	215	450	573	1360	3.20	59.0	0.66	2782	39.4	26.9	47.5	40,040	0.907	40.70	25.20	40.70	166.40	10.77	0.083	123	5.77	8.18	
2000-8	8-Nov-02	134	Balanced	215	450	570	1342	3.18	55.1	0.65	2771	41.2	30.0	48.0	40,040	0.932	41.30	25.50	41.10	166.70	10.86	0.083	128	5.69	8.28	
2000-8	9-Nov-02	135	Balanced	215	450	570	1336	3.23	54.0	0.65	2750	37.4	21.1	49.5	40,040	0.949	42.30	25.80	41.10	166.70	10.87	0.080	127	5.06	7.43	
2000-8	10-Nov-02	136	Balanced	215	450	569	1318	3.27	57.8	0.64	2717	40.7	30.1	49.0	40,040	0.967	43.20	26.00	41.10	165.90	10.81	0.081	126	5.45	8.08	
2000-8	12-Nov-02	138	Balanced	215	500	570	1554	3.64	29.3	0.65	3068	40.9	30.6	49.0	40,040	0.942	45.30	24.80	39.10	174.80	11.38	0.083	132	6.34	8.10	
2000-8	13-Nov-02	139	Balanced	215	500	570	1569	3.47	29.0	0.66	3076	40.9	30.5	49.0	40,040	0.912	43.20	24.60	39.20	174.70	11.37	0.085	130	6.43	8.05	
2000-8	14-Nov-02	140	Balanced	215	500	568	1553	3.58	30.9	0.66	3089	40.8	29.6	48.5	40,040	0.902	43.80	24.30	39.40	172.90	11.26	0.085	129	6.15	7.90	
2000-8	15-Nov-02	141	Balanced	215	500	565	1546	3.53	30.9	0.65	3060	39.0	25.3	49.0	40,040	0.929	44.30	24.80	38.90	174.60	11.37	0.085	133	5.89	7.45	
2000-8	16-Nov-02	142	Balanced	215	500	570	1559	3.36	31.2	0.66	3076	39.5	25.1	48.0	40,040	0.902	42.70	24.70	39.40	173.60	11.30	0.086	132	6.72	8.22	
2000-8	17-Nov-02	143	Balanced	215	500	568	1556	3.54	34.8	0.66	3075	39.2	24.1	48.0	40,040	0.872	42.70	24.30	39.70	171.80	11.19	0.085	126	6.08	7.91	
2000-8	19-Nov-02	145	Balanced	215	500	570	1555	3.59	29.4	0.66	3071	38.8	26.3	50.0	40,040	0.900	43.90	24.60	39.30	174.20	11.34	0.083	130	6.08	7.99	
2000-8	20-Nov-02	146	Balanced	215	500	570	1535	3.67	31.0	0.65	3055	38.4	24.9	50.0	40,040	0.919	45.20	24.90	39.10	174.10	11.33	0.084	131	6.00	7.90	
2000-8	21-Nov-02	147	Balanced	215	500	560	1556	3.48	32.6	0.66	3076	40.1	28.4	49.0	40,040	0.897	43.00	24.70	39.10	171.90	11.19	0.083	132	7.03	8.72	
2000-8	22-Nov-02	148	Balanced	215	500	585	1537	3.59	35.3	0.65	3064	38.0	22.1	49.0	40,040	0.939	45.20	25.60	39.40	178.30	11.61	0.087	136	6.55	8.56	
2000-8	24-Nov-02	150	Balanced	215	500	575	1566	3.45	29.3	0.66	3098	40.3	28.3	48.5	40,040	0.904	43.00	24.80	38.90	177.50	11.56	0.087	134	6.97	8.96	
2000-8	25-Nov-02	151	Balanced	215	500	550	1529	3.89	32.5	0.64	3012	40.0	28.1	49.0	40,040	0.836	44.80	23.70	39.60	166.70	10.86	0.081	118	5.66	8.31	
2000-8	26-Nov-02	152	Balanced	215	500	560	1536	3.95	33.9	0.65	3027	38.4	23.3	49.0	40,040	0.852	45.50	23.30	39.60	169.60	11.04	0.082	121	5.94	8.84	
2000-8	27-Nov-02	153	Balanced	215	500	550	1582	3.57	30.6	0.66	3088	38.6	24.1	49.0	40,040	0.813	41.30	23.40	39.40	167.60	10.91	0.081	117	6.21	8.32	
2000-8	28-Nov-02	154	Balanced	215	500	550	1571	3.58	32.3	0.66	3077	40.5	29.5	49.0	40,040	0.809	41.30	23.40	39.40	167.40	10.90	0.081	116	6.32	8.59	
2000-8	29-Nov-02	155	Balanced	215	500	535	1581	3.26	32.0	0.66	3078	39.3	24.6	48.0	40,040	0.781	38.00	22.90	39.20	163.90	10.67	0.081	115	5.58	7.13	
2000-8	30-Nov-02	156	Balanced	215	500	548	1541	3.73	38.4	0.64	3020	42.4	30.1	46.0	40,040	0.794	42.30	23.20	39.90	164.50	10.71	0.085	120	6.30	9.21	
2000-8	1-Dec-02	157	Balanced	215	500	538	1594	3.35	32.7	0.66	3108	38.9	24.1	48.5	40,040	0.778	38.40	24.10	39.10	164.90	10.73	0.081	115	6.35	8.16	
2000-8	14-Dec-02	158	Balanced	215	500	550	1605	3.16	32.9	0.66	3108	38.7	24.1	48.5	40,040	0.791	37.50	23.30	39.30	167.80	10.93	0.082	116	5.82	7.30	
2000-8	3-Dec-02	159	Balanced	215	500	548	1584	3.16	30.6	0.66	3093	38.7	24.4	49.0	40,040	0.785	37.40	24.40	39.50	166.30	10.83	0.081	114	6.48	8.23	
2000-8	4-Dec-02	160	Balanced	215	500	548	1584	3.16	30.6	0.66	3106	38.3	23.2	49.0	40,040	0.799	37.90	23.30	39.00	168.60	10.97	0.082	118	6.31	7.96	
2000-8	9-Dec-02	165	Balanced	215	500	535	1596	3.32	32.8	0.66	3090	40.4	27.1	47.5	40,040	0.753	37.60	22.70	39.20	163.60	10.65	0.082	111	5.74	7.64	
2000-8	10-Dec-02	166	Balanced	215	500	550	1590	3.39	35.7	0.66	3092	37.3	20.0	49.0	40,040	0.782	39.00	23.10	39.40	167.70	10.92	0.082	112	5.08	6.84	
2000-8	11-Dec-02	167	Balanced	215	500	548	1584	3.15	29.6	0.66	3104	37.7	21.9	49.5	40,040	0.789	37.50	23.40	39.10	168.00	10.94	0.081	119	6.59	8.31	
2000-8	13-Dec-02	169	Balanced	215	500	550	1534	3.14	31.6	0.66	3079	37.9	20.3	48.0	40,040	0.807	38.10	23.70	38.90	169.60	11.04	0.084	123	6.36	8.05	
2000-8	14-Dec-02	170	Balanced	215	500	548	1578	3.28	31.7	0.66	3089	39.3	24.5	48.0	40,040	0.783	38.30	23.20	39.10	169.20	10.95	0.084	118	6.17	8.22	
2000-8	15-Dec-02	171	Balanced	215	500	558	1559	3.48	32.7	0.65	3059	38.4	23.3	49.0	40,040	0.814	41.10	23.80	39.30	170.40	11.09	0.083	121	5.80	8.07	
2000-8	16-Dec-02	172	Balanced	215	500	560	1544	3.66	36.2	0.65	3033	36.6	19.4	50.0	40,040	0.827	42.90	23.90	39.30	171.10	11.14	0.082	118	5.34	7.82	
2000-8	23-Dec-02	179	Balanced	215	500	550	1604	3.25	27.0	0.67	3122	37.6	24.8	51.5	40,040	0.808	38.40	23.30	39.20	168.30	10.96	0.078	115	6.43	8.17	
2000-8	25-Dec-02	181	Balanced	215	500	551	1569	3.57	35.3	0.65	3067	39.0	24.5	48.5	40,040	0.782</										

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.

During the reporting period, a total of 4,163,251 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 90.9% availability during this quarter; the availability during the 69-month demonstration test program was 97.5%. Appendix C, Table 1 contains the summary of outages for the LPMEOH™ Demonstration Unit during this quarter. There were two outages associated with the 29C-40 catalyst guard bed that accounted for all of the downtime experienced during this reporting period. The first outage occurred on 06 October 2002 (143 hours) and was associated with the changeout and reduction of fresh adsorbent (copper-impregnated activated carbon) in the catalyst guard bed. This outage also provided the opportunity to perform some minor maintenance work, including the calibration of the inlet guide vane positioner on the 29K-01 recycle compressor. There was also a 12-hour interruption of syngas prior to the start of the reduction of the adsorbent during that outage. The second outage occurred on 06 December 2002 (56 hours) and was associated with a thermal treatment of the same charge of adsorbent in the catalyst guard bed. In order to provide minimum exposure of the methanol synthesis catalyst to trace contaminants, the LPMEOH™ Demonstration Unit was shutdown while the catalyst guard bed was off-line. A second syngas interruption during the quarter occurred on 31 December 2002 and lasted for 14.2 hours.

Catalyst Life (η) – October - December 2002

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 for the fourth catalyst campaign (which began in June 2002 following the second in-situ activation of methanol synthesis catalyst in the LPMEOH™ Reactor). 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 reactor temperature is changed.

During this quarter, the LPMEOH™ Demonstration Unit was operated at a reactor temperature of 214-215°C. Variations in the composition and flowrate of Balanced Gas that began in August of 2002 continued until 22 October 2002. The supply then stabilized for the remainder of the reporting period; overall, the flowrate of Balanced Gas was controlled at an average value of 551 KSCFH. The reactor pressure was operated at 450 psig until 11 November 2002, at which time the pressure was increased to 500 psig in order to reduce the reactor purge flowrate and to determine if the higher operating pressure would affect the performance of the methanol synthesis catalyst.

A catalyst deactivation rate of 0.1% per day was calculated for the period 25 October 2002 to 09 November 2002 (16 days). This is a significantly lower deactivation rate than the results that have been generally calculated over the past 3 years (averaging between 0.6% and 0.7% per day). As a basis of comparison, the calculated deactivation rate from the 4-month proof-of-concept run at the LaPorte AFDU in 1988/89 was 0.4% per day. (This run was performed on CO-rich syngas derived from natural gas at a reactor temperature of 250°C and pressure of 750 psig.)

For the period 12 November 2002 to 04 December 2002 (23 days), the calculated catalyst deactivation rate was 0.87% per day. This is similar to the historical performance at the LPMEOH™ Demonstration Unit, but a significant increase over the results from the prior 3 weeks of operation. This change could have been caused by a trace contaminant breakthrough of the adsorbent in the catalyst guard bed; as a consequence, a thermal treatment of the adsorbent to provide increased capacity for arsine removal was performed on 06 December 2002.

For the period 16 December 2002 to 30 December 2002 (15 days), the calculated catalyst deactivation rate was 0.13% per day. The return to excellent catalyst deactivation results following the thermal treatment of the adsorbent in the catalyst guard bed provides evidence of the impact of the presence of trace contaminants in coal-derived syngas on the life of methanol synthesis catalyst.

Due to this low catalyst deactivation rate, the practice of temperature programming was not required during the reporting period (temperature programming involves the increase of reactor temperature as necessary to control the reactor purge flowrate and maintain reactor volumetric productivity).

Analyses of samples of fresh and spent catalyst to determine changes in levels of poisons were performed. Appendix C, Table 3 summarizes the results for the fourth catalyst campaign (following the completion of the second in-situ catalyst activation procedure). No significant increase in the concentration of any of the known catalyst poisons (including iron, arsenic, and sulfur) was observed.

29C-40 Catalyst Guard Bed Performance

As noted above, the LPMEOH™ Demonstration Unit was shut down at 1600 hours on 06 October 2002 to prepare for the replacement of the adsorbent in the catalyst guard bed. (During this outage, Eastman also replaced the adsorbent (230 cubic feet of manganese dioxide) in the 19C-30 catalyst guard bed (located upstream of both the LPMEOH™ Demonstration Unit and the fixed-bed methanol plant) with fresh material; the adsorbent was last changed in June of 1999.)

A total of 5,225 pounds of fresh copper-impregnated activated carbon was charged to the catalyst guard bed on 10 October 2002. This material must be chemically reduced using dilute syngas in nitrogen prior to use. (In this case, reduction refers to the reaction of the copper oxide with a reductant such as CO or H₂ to copper metal and either CO₂ or H₂O). The temperature of the adsorbent was first increased to 80°C using heated nitrogen. The 12-

hour syngas interruption (mentioned previously) delayed the start of the reduction procedure. A further delay was experienced due to the discovery that the wrong trim was installed in the valve (29FC-2641) that controls the flowrate of Balanced Gas during the reduction process. The proper trim was installed, and Balanced Gas was introduced at 1418 hours on 11 October 2002. The reduction proceeded without incident and was completed at 1754 hours on 12 October 2002.

Following the completion of the steps to purge and cool the catalyst guard bed with nitrogen, the LPMEOH™ Demonstration Unit was restarted at 0324 hours on 13 October 2002. The catalyst guard bed was brought on-line at 1030 hours on 13 October 2002. The guard bed internal temperatures increased by about 38-40°C and then stabilized following the introduction of the syngas. This was consistent with earlier observations associated with the start-up of the catalyst guard bed.

Based upon results over the past year, an operating schedule for the adsorbent was developed. Fresh adsorbent can be used for about two months before breakthrough of arsine can be expected. A thermal treatment can then be performed on the adsorbent to provide increased capacity for arsine removal, and the material can be used for another month before the adsorbent needs to be replaced. However, due to the increase in the calculated rate of catalyst deactivation that was observed in November of 2002, the timing for performing the thermal treatment on the adsorbent was started on 06 December 2002 after 7 weeks of service. The catalyst guard bed was brought back online on 08 December 2002; no excessive temperature rise of the adsorbent was measured.

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 June 2002 outage prior to the second in-situ activation of methanol synthesis catalyst. Appendix C, Figure 2 plots the average daily sparger resistance coefficient for the fourth catalyst campaign. The data for this plot, along with the corresponding average pressure drop, are also included in Table D.3-1. The sparger resistance continues to show no significant increase over time, which is consistent with the operating history with this device.

D.4 Planning and Administration

A DOE project review meeting was held during the week of 04 November 2002 in Pittsburgh. The performance of the LPMEOH™ Demonstration Unit since the last meeting (December 2001) was the primary topic of discussion. The agenda, extracts from the handouts, and the notes for the meeting are included in Appendix D.

Comments were received from DOE on the report on publicly available technical data on the Eastman chemicals-from-coal complex in Kingsport. This report provides operational performance of the chemicals-from-coal complex in Kingsport as well as specific data on the major feed and effluent streams for the coal gasification and syngas cleanup systems. An updated version was prepared and sent to DOE for review and comment.

A first draft of the topical report entitled “Removal of Trace Contaminants from Coal-Derived Syngas” was submitted to DOE for review and comment.

The Milestone Schedule Status Report and the Cost Management Report, through the period ending 31 December 2002, 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 \$40 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 31 December 2002. One hundred percent (100%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 December 2002.

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

E. Planned Activities for the Next Quarter

- Begin close-out activities, including the submittal of the draft version of the Final Report to DOE.

F. Conclusion

The LPMEOH™ Demonstration Unit completed a successful 69-month demonstration test program on 31 December 2002. During the reporting period, the LPMEOH™ Demonstration Unit operated at 90.9% availability during this quarter; the availability during the 69-month demonstration test program was 97.5%. There were two outages associated with the 29C-40 catalyst guard bed that accounted for all of the downtime experienced during this reporting period. The first outage occurred on 06 October 2002 (143 hours) and was associated with the changeout and reduction of fresh adsorbent (copper-impregnated activated carbon) in the catalyst guard bed. There was also a 12-hour interruption of syngas prior to the start of the reduction of the adsorbent during that outage. The second outage occurred on 06 December 2002 (56 hours) and was associated with a thermal treatment of the same charge of adsorbent in the catalyst guard bed. In order to provide minimum exposure of the methanol synthesis catalyst to trace contaminants, the LPMEOH™ Demonstration Unit was shutdown while the catalyst guard bed was off-line. A second syngas interruption during the quarter occurred on 31 December 2002 and lasted for 14.2 hours.

During this quarter, the LPMEOH™ Demonstration Unit was operated at a reactor temperature of 214-215°C. Variations in the composition and flowrate of Balanced Gas that began in August of 2002 continued until 22 October 2002. The supply then stabilized for the remainder of the reporting period; overall, the flowrate of Balanced Gas was controlled at an average value of 551 KSCFH. The reactor pressure was operated at 450 psig until 11 November 2002, at which time the pressure was increased to 500 psig in order to reduce the

reactor purge flowrate and to determine if the higher operating pressure would affect the performance of the methanol synthesis catalyst.

A catalyst deactivation rate of 0.1% per day was calculated for the period 25 October 2002 to 09 November 2002 (16 days). This is a significantly lower deactivation rate than the results that have been generally calculated over the past 3 years (averaging between 0.6% and 0.7% per day). As a basis of comparison, the calculated deactivation rate from the 4-month proof-of-concept run at the LaPorte AFDU in 1988/89 was 0.4% per day. (This run was performed on CO-rich syngas derived from natural gas at a reactor temperature of 250°C and pressure of 750 psig.)

For the period 12 November 2002 to 04 December 2002 (23 days), the calculated catalyst deactivation rate was 0.87% per day. This is similar to the historical performance at the LPMEOH™ Demonstration Unit, but a significant increase over the results from the prior 3 weeks of operation. This change could have been caused by a trace contaminant breakthrough of the adsorbent in the catalyst guard bed; as a consequence, a thermal treatment of the adsorbent to provide increased capacity for arsine removal was performed on 06 December 2002.

For the period 16 December 2002 to 30 December 2002 (15 days), the calculated catalyst deactivation rate was 0.13% per day. The return to excellent catalyst deactivation results following the thermal treatment of the adsorbent in the catalyst guard bed provides evidence of the impact of the presence of trace contaminants in coal-derived syngas on the life of methanol synthesis catalyst.

Due to this low catalyst deactivation rate, the practice of temperature programming was not required during the reporting period (temperature programming involves the increase of reactor temperature as necessary to control the reactor purge flowrate and maintain reactor volumetric productivity).

Samples of spent and recently activated fresh catalyst were collected during the quarter to determine changes in levels of trace contaminants (such as iron, nickel, sulfur, and arsenic) that are known poisons to methanol synthesis catalysts. No significant increase in the concentration of any of these poisons was observed.

As noted above, the LPMEOH™ Demonstration Unit was shut down at 1600 hours on 06 October 2002 to prepare for the replacement of the adsorbent in the catalyst guard bed. (During this outage, Eastman also replaced the adsorbent (230 cubic feet of manganese dioxide) in the 19C-30 catalyst guard bed (located upstream of both the LPMEOH™ Demonstration Unit and the fixed-bed methanol plant) with fresh material; the adsorbent was last changed in June of 1999.)

A total of 5,225 pounds of fresh copper-impregnated activated carbon was charged to the catalyst guard bed on 10 October 2002. This material must be chemically reduced using dilute syngas in nitrogen prior to use. (In this case, reduction refers to the reaction of the copper oxide with a reductant such as CO or H₂ to copper metal and either CO₂ or H₂O). The temperature of the adsorbent was first increased to 80°C using heated nitrogen. The 12-

hour syngas interruption (mentioned previously) delayed the start of the reduction procedure. A further delay was experienced due to the discovery that the wrong trim was installed in the valve that controls the flowrate of Balanced Gas during the reduction process. The proper trim was installed, and Balanced Gas was introduced at 1418 hours on 11 October 2002. The reduction proceeded without incident and was completed at 1754 hours on 12 October 2002.

Following the completion of the steps to purge and cool the catalyst guard bed with nitrogen, the LPMEOH™ Demonstration Unit was restarted at 0324 hours on 13 October 2002. The catalyst guard bed was brought on-line at 1030 hours on 13 October 2002. The guard bed internal temperatures increased by about 38-40°C and then stabilized following the introduction of the syngas. This was consistent with earlier observations associated with the start-up of the catalyst guard bed.

Based upon results over the past year, an operating schedule for the adsorbent has been developed. Fresh adsorbent can be used for about two months before breakthrough of arsine can be expected. A thermal treatment can then be performed on the adsorbent to provide increased capacity for arsine removal, and the material can be used for another month before the adsorbent needs to be replaced. However, due to the increase in the calculated rate of catalyst deactivation that was observed in November of 2002, the timing for performing the thermal treatment on the adsorbent was started on 06 December 2002 after 7 weeks of service. The catalyst guard bed was brought back online on 08 December 2002; no excessive temperature rise of the adsorbent was measured.

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 June 2002 outage prior to the second in-situ activation of methanol synthesis catalyst. The sparger resistance continues to show no significant increase over time, which is consistent with the operating history with this device.

During the reporting period, a total of 4,163,251 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, about 103.9 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 DOE project review meeting was held during the week of 04 November 2002 in Pittsburgh. The performance of the LPMEOH™ Demonstration Unit since the last meeting (December 2001) was the primary topic of discussion.

Comments were received from DOE on the report on publicly available technical data on the Eastman chemicals-from-coal complex in Kingsport. This report provides operational performance of the chemicals-from-coal complex in Kingsport as well as specific data on the major feed and effluent streams for the coal gasification and syngas cleanup systems. An updated version was prepared and sent to DOE for review and comment.

A first draft of the topical report entitled “Removal of Trace Contaminants from Coal-Derived Syngas” was submitted to DOE for review and comment.

One hundred percent (100%) of the \$40 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 31 December 2002. One hundred percent (100%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 December 2002.

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 -
October/December 2002**

Table 2 - Summary of Catalyst Samples - Fourth Catalyst Batch

Figure 1 - Catalyst Age (η): June 2002 - December 2002

**Figure 2 - Sparger Resistance Coefficient vs. Days Onstream
(June 2002 - December 2002)**

Table 1
Summary of LPMEOH™ Demonstration Unit Outages - October/December 2002

Operation Start	Operation End	Operating Hours	Shutdown Hours	Reason for Shutdown
10/1/02 00:00	10/6/02 16:00	136.0	155.4	Guard Bed Adsorbent Change*
10/13/02 03:24	12/6/02 09:30	1302.1	55.5	Guard Bed Thermal Treatment
12/8/02 16:58	12/31/02 09:47	544.8	14.2	Syngas Outage
12/31/02 23:59	12/31/02 23:59	0.0		End of Reporting Period
		Total Operating Hours	1982.9	
		Syngas Available Hours	2181.8	
		Plant Availability, %	90.89	

* - A syngas outage of 12 hours on 10/10/02 prevented the start of the reduction of the adsorbent in the 29C-40 catalyst guard bed.

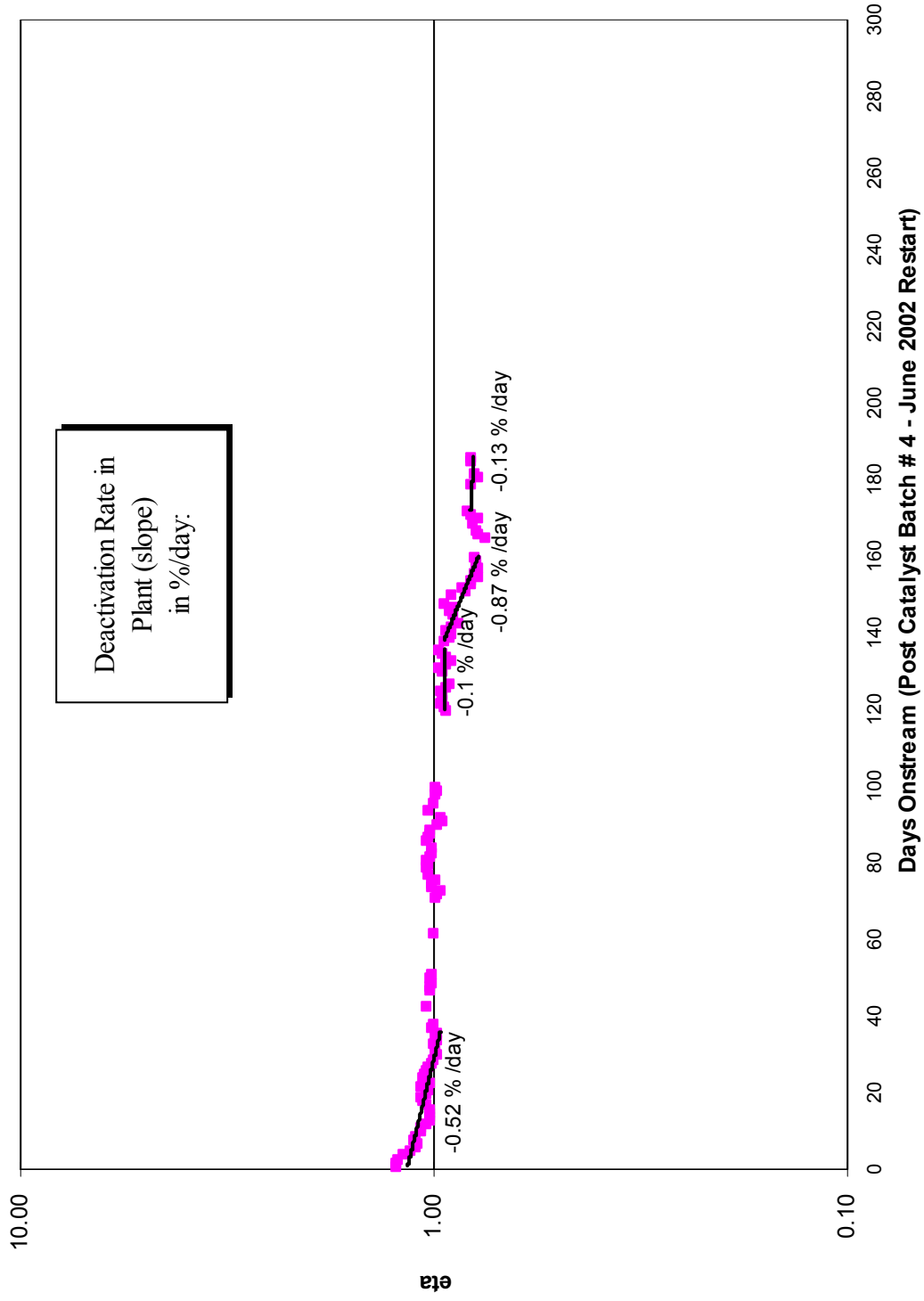
Table 2
Summary of Catalyst Samples - Fourth Catalyst Batch

Sample	Identity	Analytical (ppmw)				
		Fe	Ni	S	As	Cl
K0206-1	Fresh Catalyst (Oxide Powder) 6/24/02	<23	<10	<40	<20	<100
K0206-3	Reactor Sample 6/27/02	28	<7	<19	<2	na
K0207-1	Reactor Sample 7/8/02	44	<7	<25	4.2	na
K0208-2	Reactor Sample 8/15/02	40	<6	<39	5.4	na
K0209-2	Reactor Sample 9/26/02	58	<7	<47	19	na

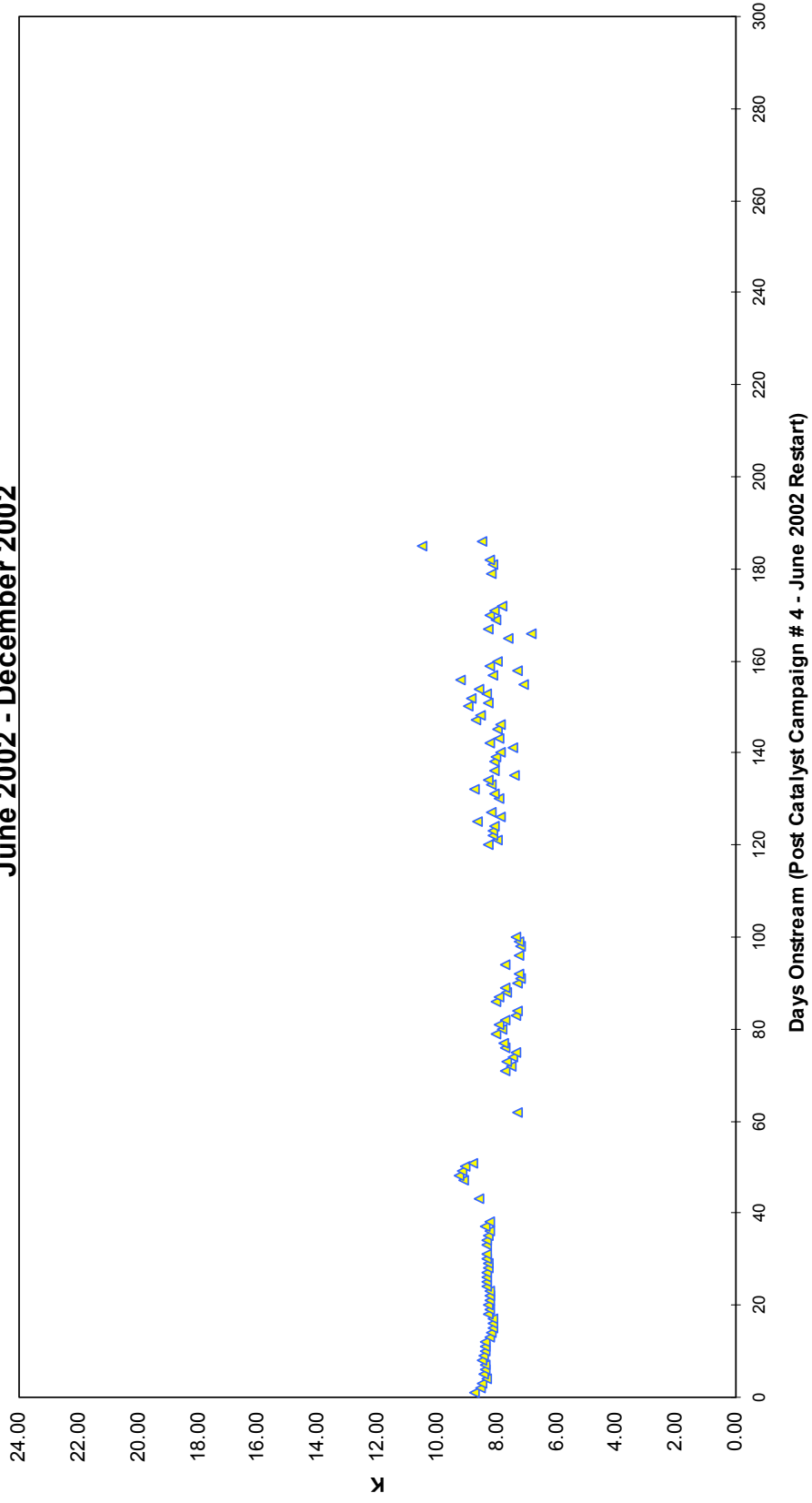
Notes:

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

**Figure 1 - Kingsport LPMEOH™ Catalyst Age (eta):
June 2002 - December 2002**



**Figure 2 - Kingsport LPMEOH™
Sparger Resistance Coefficient
June 2002 - December 2002**



APPENDIX D - PROJECT REVIEW MEETING (06-08 NOVEMBER 2002)

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