

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

TECHNICAL PROGRESS REPORT NO. 30

For The Period

1 October – 31 December 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 99.89% availability during this quarter. The forced downtime experienced this quarter (2.5 hours) was associated with an upset in the distillation section, which has not recurred. There were also three short syngas interruptions that were experienced on 23 October 2001 (12.4 hours duration), 29 October 2001 (9.9 hours duration), and 19 December 2001 (19.7 hours duration).

A catalyst deactivation rate of 0.2% per day was calculated for the period 08 October 2001 to 21 October 2001 (14 days). This is a significantly lower deactivation rate than the results that have been generally calculated over the past 2 years (averaging between 0.6% and 0.7% per day), and may be related to the improved performance of the adsorbent in the 29C-40 catalyst guard bed during that period (described below). A catalyst deactivation rate of 1.36% per day was calculated for the period 01 November 2001 to 13 November 2001 (13 days). A catalyst deactivation rate of 0.45% per day was calculated for the period 16 November 2001 to 02 December 2001 (17 days). The changes in the calculated rate of catalyst deactivation may be related to the impact of variations in the syngas composition on the kinetic model, or they may be process-related. As a basis of comparison, the calculated deactivation rate from the 4-month proof-of-concept run at the LaPorte Alternative Fuels Development Unit (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).

Following the restart after the syngas outage on 29 October 2001, the composition of the primary syngas feed (Balanced Gas) to the LPMEOH™ Demonstration Unit became rich in carbon monoxide (CO). During this 14-hour period of operation, the reactor inlet ratio of hydrogen (H₂) to CO was approximately 0.6 to 0.7. Later in the reporting period, quantities of the syngas stream that contains primarily CO (Carbon Monoxide Gas, or CO Gas) became available so that additional tests on CO-rich syngas were performed. Two operating periods with a ratio of H₂ to CO in the reactor feed gas of approximately 0.7 were tested during the periods of 12-18 December 2001 and 24-29 December 2001. During these test dates, heat and material balances were generated for periods of at least 12 hours of steady operation.

As part of the operating protocol following the completion of the in-situ activation of methanol synthesis catalyst in the LPMEOH™ Reactor, temperature programming continued during the quarter; this involved the increase of reactor temperature as necessary to control the reactor purge flowrate and maintain reactor productivity. Over the reporting period, the reactor temperature was increased from 218°C to 226°C. The reactor pressure was increased

from 685 psig to 700 psig on 12 November 2001 and maintained at that level for the balance of the reporting period. The flowrate of Balanced Gas was controlled at an average value of 630 KSCFH during this time.

Work was continued during this period to reconcile the calculated activity of the methanol synthesis catalyst in the LPMEOH™ Reactor. Additional reactor catalyst samples from September of 2001 were evaluated for catalyst activity and chemical analysis. A check of catalyst activity in the autoclave was performed on a reactor catalyst sample from 19 September 2001 (26 days on-stream). This sample exhibited excellent activity, approaching the typical performance for properly activated catalyst. Throughout the reporting period, the calculated value of the “age” of the catalyst, 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), continued to appear to be about 50% of the value for freshly activated catalyst; it is not clear whether this discrepancy is real or possibly related to imperfections in the kinetic model. Additional work is planned to resolve this difference and determine the impact of reactor operating temperature on the results from the model.

After the completion of the in-situ activation of methanol synthesis catalyst in the LPMEOH™ Reactor on 24 August 2001, the catalyst guard bed was brought online. This was the first operation with the fresh charge of adsorbent (copper-impregnated activated carbon), which had been loaded into the vessel and reduced in July of 2001. (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 carbon dioxide [CO₂] or water [H₂O]). Throughout the reporting period, gas sampling was performed to assess the performance of the adsorbent. The analytical techniques involved analyzing the Balanced Gas leaving the catalyst guard bed for arsenic (reported as arsine) using standard techniques (CO Gas was not used during this initial operating period). During the initial testing, the outlet concentration of arsine was determined to be less than the detection limit of the analysis (4 to 6 parts per billion by volume (ppbv)). Based on these results, the adsorbent in the catalyst guard bed was assessed to be performing well during the first 2 months of operation (or through late October of 2001).

After approximately two months of service, the concentration of arsine at the outlet of the catalyst guard bed was determined to be above the detection limit and had reached an average value of 14 ppbv. This result was further corroborated during the period of 26 November 2001 to 04 December 2001 (or after approximately three months of service) when the concentration of arsine at the outlet of the catalyst guard bed was determined to be on the order of 20 ppbv. This analysis provided further indication of breakthrough of arsine from the catalyst guard bed.

On 04 December 2001, the catalyst guard bed was taken out of service and a thermal treatment was performed on the adsorbent in an attempt to provide increased capacity for arsine removal. The catalyst guard bed was then brought back online; no excessive temperature rise was measured. Analytical testing was again conducted following the thermal treatment. Initial results indicated that the concentration of arsine in the catalyst guard bed outlet stream was less than the detection limit of 6 ppbv. Additional testing of the

outlet stream of the catalyst guard bed on a weekly basis is planned and will be used to evaluate changes in the performance of the adsorbent.

Analyses of catalyst samples to determine changes in physical characteristics and levels of poisons have continued. 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. Several known catalyst poisons including iron, arsenic, and sulfur have been detected on the catalyst and appear to be increasing.

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 biennial outage in March of 2001. 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 4,832,009 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, about 87.8 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 Topical Report entitled “Off-Site Testing of Stabilized Methanol from the Liquid Phase Methanol (LPMEOH™) Process” was approved by DOE during the reporting period. This report provides the results from the seven test sites. During the reporting period, the unused stabilized methanol was returned to Eastman for further distillation prior to use within the chemicals-from-coal complex.

Activities associated with Design Verification Testing (DVT) of the Liquid Phase Dimethyl Ether (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 the status of the current market for dimethyl ether (DME) and an outlook on potential market developments through 2006.

A DOE quarterly review meeting was held during the week of 10 December 2001 in Kingsport. The performance of the LPMEOH™ Demonstration Unit since the last meeting (June 2001) was the primary topic of discussion.

The paper entitled “Liquid Phase Methanol (LPMEOH™) Process Development Demonstration Plant Availability” was presented at the Gasification Technologies Conference in San Francisco, CA on 07-10 October 2001. The poster entitled “Liquid Phase Methanol (LPMEOH™) Technology” was displayed at the Clean Coal and Power Conference (formerly the Clean Coal Technology Conference) on 19-20 November 2001. The paper entitled “Liquid Phase Methanol (LPMEOH™) Process Development” was included in the proceedings 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 31 December 2001. Eighty-six percent (86%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 December 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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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 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” was approved by DOE during the reporting period. 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).

The LPMEOH™ Demonstration Project will prepare a separate Topical Report on the status of the current market for DME and an outlook on potential market developments through 2006.

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.

During the reporting period, a total of 4,832,009 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 99.89% availability during this quarter. Appendix C, Table 1 contains the summary of outages for the LPMEOH™ Demonstration Unit during this quarter. The forced downtime experienced this quarter (2.5 hours) was associated with an upset in the distillation section, which has not recurred. There were also three short syngas interruptions that were experienced on 23 October 2001 (12.4 hours duration), 29 October 2001 (9.9 hours duration), and 19 December 2001 (19.7 hours duration).

Catalyst Life (η) – October - December 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 for the third catalyst campaign (following the 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.

A catalyst deactivation rate of 0.2% per day was calculated for the period 08 October 2001 to 21 October 2001 (14 days). This is a significantly lower deactivation rate than the results that have been generally calculated over the past 2 years (averaging between 0.6% and 0.7% per day), and may be related to the improved performance of the adsorbent in the 29C-40 catalyst guard bed during that period (described below). A catalyst deactivation rate of 1.36% per day was calculated for the period 01 November 2001 to 13 November 2001 (13 days). A catalyst deactivation rate of 0.45% per day was calculated for the period 16 November 2001 to 02

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 (l/hr-kg)	Slurry Conc. (wt% ox)	Gas Holdup (vol%)	Gassed Slurry Hgt (ft)	Catalyst Inventory (lb)	Catalyst Age (eta)	CO Conv. (%)	Reactor O-T-M		Raw MeOH Prod. (TPD)	Catalyst MeOH Prod. (gmol/hr-kg)	Reactor Vol. Prod. (TPD/ft3)	Overall (Btu hr ft2 F)	Sparger dP (psi)	Sparger Resist. ("K")
																		Conv. (%)	Conv. (%)						
2000-8	2-Oct-01	39	Balanced	217	685	634	2,096	4.55	60.0	0.62	3756	41.1	26.8	48.0	41,580	0.543	40.7	19.5	41.7	182.6	11.47	0.091	107	4.96	7.13
2000-8	3-Oct-01	40	Balanced	218	685	658	2,244	3.09	38.4	0.66	4053	39.3	26.1	51.0	41,580	0.521	31.7	20.2	40.0	197.2	12.38	0.092	111	6.99	6.34
2000-8	4-Oct-01	41	Balanced	219	685	657	2,217	3.20	39.9	0.66	4037	38.2	22.8	51.0	41,580	0.518	32.5	20.3	39.9	197.5	12.40	0.092	104	6.73	6.36
2000-8	8-Oct-01	45	Balanced	218	685	675	2,213	3.15	52.3	0.66	4021	38.3	21.7	50.0	41,580	0.510	31.7	20.0	41.2	196.8	12.35	0.094	99	6.58	6.45
2000-8	9-Oct-01	46	Balanced	218	685	658	2,201	3.24	50.0	0.66	3997	38.6	21.0	49.0	41,580	0.507	32.7	19.9	40.6	194.8	12.23	0.095	100	6.46	6.48
2000-8	10-Oct-01	47	Balanced	220	685	651	2,215	3.39	38.9	0.66	4005	40.5	21.7	46.0	41,580	0.509	34.3	20.1	40.0	195.4	12.27	0.101	105	6.38	6.41
2000-8	11-Oct-01	48	Balanced	219	683	660	2,159	3.12	46.5	0.65	3947	38.1	20.9	50.0	41,580	0.512	32.8	20.4	40.5	195.3	12.26	0.093	102	6.46	6.37
2000-8	12-Oct-01	49	Balanced	218	685	627	2,191	3.30	37.2	0.64	3930	38.2	19.6	49.0	41,580	0.516	32.8	20.0	39.8	188.7	11.85	0.092	108	6.44	6.38
2000-8	15-Oct-01	52	Balanced	218	685	677	2,154	3.09	57.6	0.65	3951	39.1	22.5	49.0	41,580	0.513	32.4	20.4	41.1	198.0	12.43	0.096	109	6.40	6.47
2000-8	16-Oct-01	53	Balanced	218	685	677	2,177	3.09	62.2	0.65	3990	40.1	23.8	48.0	41,580	0.508	31.2	20.1	41.2	197.3	12.39	0.098	108	6.44	6.51
2000-8	17-Oct-01	54	Balanced	218	685	677	2,198	3.00	67.2	0.66	4010	39.4	22.6	48.5	41,580	0.497	30.0	19.7	41.6	195.6	12.28	0.096	102	6.44	6.46
2000-8	18-Oct-01	55	Balanced	218	685	684	2,166	3.02	59.5	0.65	3989	39.6	23.1	48.5	41,580	0.506	31.1	20.2	41.1	199.7	12.54	0.098	107	6.43	6.41
2000-8	19-Oct-01	56	Balanced	218	685	684	2,159	3.01	64.8	0.65	3960	39.3	23.0	49.0	41,580	0.505	31.0	20.2	41.4	198.0	12.43	0.096	108	6.41	6.43
2000-8	21-Oct-01	58	Balanced	218	685	669	2,000	3.06	65.7	0.65	3955	39.4	21.0	47.5	41,580	0.498	30.8	19.8	41.4	193.9	12.18	0.097	106	6.47	6.53
2000-8	22-Oct-01	59	Balanced	218	685	657	2,140	3.13	55.9	0.64	3920	38.8	21.4	49.0	41,580	0.503	31.8	20.1	41.0	192.2	12.07	0.093	109	6.48	6.50
2000-8	23-Oct-01	60	Balanced	218	685	645	2,129	3.10	52.9	0.64	3890	38.3	19.9	49.0	41,580	0.489	31.4	20.0	40.9	189.2	11.89	0.092	104	6.32	6.29
2000-8	28-Oct-01	65	Balanced	218	685	643	2,234	2.92	61.2	0.66	4025	39.7	20.1	46.5	41,580	0.475	28.3	18.9	40.7	189.7	11.89	0.097	102	6.33	6.23
2000-9	29-Oct-01	66	H2:CO = 0.6	218	684	577	2,261	0.64	99.3	0.69	4220	41.5	25.5	46.5	41,580	0.542	9.3	14.1	44.5	155.5	9.76	0.080	128	14.58	7.35
2000-8	1-Nov-01	69	Balanced	218	685	658	2,240	2.90	50.2	0.66	4026	37.5	25.1	54.0	41,580	0.515	30.4	20.2	40.2	196.4	12.31	0.087	111	7.32	6.37
2000-8	2-Nov-01	70	Balanced	218	685	652	2,229	2.88	48.8	0.66	4016	38.1	20.8	50.0	41,580	0.505	29.9	20.0	40.2	194.4	12.19	0.093	114	6.49	5.68
2000-8	3-Nov-01	71	Balanced	218	685	649	2,244	2.90	50.6	0.66	4030	38.0	22.1	51.0	41,580	0.498	29.6	19.8	40.3	193.3	12.12	0.090	113	6.67	5.89
2000-8	6-Nov-01	74	Balanced	218	685	650	2,260	2.81	61.0	0.67	4067	40.2	27.2	50.0	41,580	0.478	27.7	19.0	40.9	190.8	11.96	0.091	114	7.28	6.60
2000-8	7-Nov-01	75	Balanced	218	685	646	2,279	2.77	66.9	0.67	4061	38.3	21.5	50.0	41,580	0.465	26.7	18.5	41.6	186.4	11.69	0.089	102	6.94	6.33
2000-8	8-Nov-01	76	Balanced	218	685	634	2,281	2.76	69.0	0.66	4055	39.4	21.7	48.0	41,580	0.456	26.0	18.2	41.8	182.2	11.43	0.090	104	6.79	6.19
2000-8	9-Nov-01	77	Balanced	218	685	640	2,266	3.02	75.9	0.66	4036	38.9	24.1	50.5	41,580	0.463	28.2	18.5	41.9	183.0	11.48	0.086	106	6.81	6.46
2000-8	10-Nov-01	78	Balanced	218	685	637	2,255	3.14	74.1	0.66	4024	37.2	20.5	51.5	41,580	0.460	29.2	18.6	42.0	182.1	11.42	0.084	108	6.17	5.86
2000-8	11-Nov-01	79	Balanced	218	685	637	2,251	3.00	78.1	0.66	4016	38.3	20.9	49.5	41,580	0.449	27.7	18.2	42.5	179.8	11.28	0.087	106	6.33	6.02
2000-8	12-Nov-01	80	Balanced	218	696	636	2,317	3.06	76.3	0.66	4108	39.8	24.6	49.0	41,580	0.436	27.1	17.6	42.5	179.5	11.25	0.087	105	7.00	6.73
2000-8	13-Nov-01	81	Balanced	218	700	639	2,350	2.91	76.4	0.67	4151	38.6	24.2	51.0	41,580	0.437	26.2	17.7	42.3	181.5	11.38	0.085	101	6.70	6.70
2000-8	16-Nov-01	84	Balanced	220	700	648	2,262	3.47	66.8	0.65	4044	38.1	25.3	53.0	41,580	0.448	32.0	18.9	41.4	187.8	11.78	0.084	110	6.27	6.34
2000-8	17-Nov-01	85	Balanced	220	700	651	2,246	3.57	72.2	0.65	4011	37.7	24.2	53.0	41,580	0.449	32.7	18.9	41.8	186.7	11.71	0.084	108	6.12	6.42
2000-8	18-Nov-01	86	Balanced	220	700	648	2,286	3.35	69.9	0.65	4053	37.8	24.6	53.0	41,580	0.444	31.1	18.8	41.5	187.3	11.74	0.084	108	6.45	6.37
2000-8	19-Nov-01	87	Balanced	220	700	644	2,287	3.13	65.9	0.65	4043	37.9	24.8	53.0	41,580	0.440	29.8	19.0	41.3	186.9	11.72	0.084	110	6.65	6.24
2000-8	22-Nov-01	90	Balanced	220	700	645	2,108	3.49	73.8	0.61	3813	38.7	27.2	53.0	41,580	0.444	33.3	19.6	42.2	183.3	11.49	0.082	118	5.84	6.69
2000-8	23-Nov-01	91	Balanced	220	696	655	2,122	3.94	81.3	0.62	3849	38.1	24.0	52.0	41,580	0.472	36.5	19.6	42.4	185.5	11.63	0.085	130	5.42	6.41
2000-8	24-Nov-01	92	Balanced	220	695	656	2,102	3.92	82.3	0.62	3830	36.5	25.4	56.5	41,580	0.476	36.8	19.8	42.3	186.1	11.67	0.078	119	5.60	6.55
2000-8	25-Nov-01	93	Balanced	220	700	655	2,141	4.06	83.4	0.63	3880	37.2	26.1	55.5	41,580	0.473	36.6	19.2	42.7	184.2	11.55	0.079	117	5.75	6.93
2000-8	26-Nov-01	94	Balanced	220	700	658	2,219	3.27	72.6	0.65	4009	37.9	24.7	53.0	41,580	0.452	31.3	19.3	41.6	190.0	11.91	0.085	121	6.84	6.23
2000-8	27-Nov-01	95	Balanced	220	700	655	2,253	2.88	72.3	0.65	4039	37.6	24.4	53.5	41,580	0.439	28.3	19.1	41.6	188.9	11.85	0.084	115	6.81	6.20
2000-8	28-Nov-01	96	Balanced	220	700	652	2,259	2.86	73.2	0.65	4049	37.6	26.0	53.5	41,580	0.435	27.8	18.9	41.7	187.7	11.77	0.083	117	7.15	6.52
2000-8	29-Nov-01	97	Balanced	220	700	654	2,368	2.87	75.3	0.65	4000	36.8	24.0	55.0	41,580	0.431	28.1	19.0	42.3	185.4	11.63	0.085	123	6.84	6.24
2000-8	1-Dec-01	99	Balanced	220	700	645	2,284	2.79	83.1	0.66	4081	38.2	25.7	53.0	41,580	0.409	26.0	18.0	42.7	181.3	11.37	0.081	112	7.01	6.40
2000-8	2-Dec-01	100	Balanced	220	700	654	2,265	2.85	90.0	0.66	4067	38.2	25.5	53.0	41,580	0.410	26.5	18.1	43.1	182.0	11.42	0.082	113	6.84	6.41
2000-8	3-Dec-01	101	Balanced	223	700	645	2,274	3.05	68.8	0.66	4084	39.2	26.1	51.5	41,580	0.402	28.8	18.7	41.4	187.1	11.73	0.086	109	7.12	6.65
2000-8	4-Dec-01	102	Balanced	224	700	654	2,296	2.89	71.0	0.67	4113	38.5	23.2	51.0	41,580	0.397	27.8	18.7	41.5	189.2	11.87	0.088	103	7.44	6.67
2000-8	5-Dec-01	103	Balanced	224	700	651	2,277	2.90	69.1	0.66	4083	38.5	23.5	51.0	41,580	0.397	28.2	18.9	41.4	188.7	11.84	0.088	110	7.05	6.36
2000-8	6-Dec-01	104	Balanced	224	700	650	2,258	2.83	73.5	0.66	4050	37.8	21.3	51.0	41,580	0.395	27.8	18.9	41.8	186.7	11.71	0.087	108	7.06	6.27
2000-8	7-Dec-01	105	Balanced	224	700	646	2,250	2.81	74.6	0.66	4038	38.9	23.0	50.0	41,580	0.391	27.5	18.8	41.9	184.8	11.59	0.088	110	7.19	6.36
2000-8	8-Dec-01	106	Balanced	224	700	651	2,240	2.83	71.6	0.65	4028	39.4	25.9	51.0	41,580	0.399	28.2	19.3	41.6	187.6	11.77	0.088	118	7.25	6.39

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

Case	Date	Days On Stream	Gas Type	Temp (Deg C)	Pres. (psig)	Fresh Feed (KSCFH)	Recycle Gas (KSCFH)	Reactor Feed (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")
2000-8	9-Dec-01	107	Balanced	224	700	647	2,285	2.79	71.6	0.66	4082	38.9	26.0	52.0	41,580	0.388	27.1	18.8	41.7	186.2	11.68	0.085	114	7.24	6.41
2000-8	10-Dec-01	108	Balanced	224	700	651	2,273	2.75	77.1	0.66	4080	40.6	30.7	52.0	41,580	0.386	26.8	18.7	42.0	185.9	11.66	0.085	111	7.19	6.34
2000-8	11-Dec-01	109	Balanced	224	700	654	2,272	2.68	79.3	0.66	4084	39.1	25.7	51.5	41,580	0.381	26.1	18.5	42.3	185.4	11.63	0.086	111	7.19	6.32
2000-5	12-Dec-01	110	H ₂ :CO = 0.7	224	700	643	2,413	1.06	107.4	0.69	4220	38.0	29.2	54.0	41,580	0.407	13.3	16.3	44.9	171.6	10.78	0.076	83	10.51	5.93
2000-5	14-Dec-01	112	H ₂ :CO = 0.7	224	690	513	2,449	0.58	91.1	0.67	4084	37.6	27.7	56.0	41,580	0.435	8.7	13.7	44.2	139.4	8.76	0.059	142	12.14	6.01
2000-5	15-Dec-01	113	H ₂ :CO = 0.7	224	690	506	2,456	0.54	99.0	0.67	4072	38.9	25.2	51.5	41,580	0.408	7.9	12.7	45.3	134.0	8.42	0.062	150	11.91	6.05
2000-5	16-Dec-01	114	H ₂ :CO = 0.7	224	690	492	2,402	0.50	98.5	0.66	3999	39.1	29.5	51.5	41,580	0.412	7.5	12.3	45.8	128.9	8.09	0.060	144	11.43	5.90
2000-5	17-Dec-01	115	H ₂ :CO = 0.7	224	700	532	2,515	0.72	108.8	0.69	4218	42.7	30.8	48.0	41,580	0.354	8.5	12.4	47.1	135.6	8.51	0.067	114	11.70	6.09
2000-8	21-Dec-01	119	Balanced	226	700	560	2,455	2.75	48.8	0.68	4167	43.9	30.7	46.0	41,580	0.309	22.8	16.0	41.3	162.8	10.20	0.084	112	7.43	7.12
2000-8	22-Dec-01	120	Balanced	226	700	555	2,298	3.57	51.7	0.65	3995	38.5	19.8	49.0	41,580	0.323	28.3	16.4	41.2	161.7	10.14	0.079	116	5.40	5.92
2000-5	24-Dec-01	122	Texaco	226	700	512	2,519	0.65	106.8	0.68	4183	42.0	28.8	48.0	41,580	0.341	8.2	11.6	47.2	130.2	8.17	0.065	106	13.03	5.96
2000-5	27-Dec-01	125	Texaco	225	700	572	2,469	0.73	125.9	0.68	4195	43.2	26.0	44.0	41,580	0.400	8.9	12.9	47.5	144.3	9.05	0.078	164	11.23	6.11
2000-5	28-Dec-01	126	Texaco	225	700	542	2,455	0.70	116.3	0.68	4164	43.1	25.5	44.0	41,580	0.363	8.6	12.6	46.5	139.9	8.78	0.076	139	10.87	5.89
2000-5	29-Dec-01	127	Texaco	226	700	584	2,423	0.95	100.5	0.68	4147	39.0	27.5	53.0	41,580	0.354	11.3	14.7	44.3	158.1	9.91	0.071	106	11.05	6.55

December 2001 (17 days). The changes in the calculated rate of catalyst deactivation may be related to the impact of variations in the syngas composition on the kinetic model, or they may be process-related (for example, a change in either the concentration of trace contaminants in the reactor feed gas or the performance of the adsorbent in the catalyst guard bed could affect the catalyst performance). 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).

Following the restart after the syngas outage on 29 October 2001, the composition of Balanced Gas to the LPMEOH™ Demonstration Unit became CO-rich. During this 14-hour period of operation, the reactor inlet H₂/CO ratio was approximately 0.6 to 0.7. Later in the reporting period, sufficient CO Gas became available so that additional tests using CO-rich syngas were performed. Two operating periods with a ratio of H₂ to CO in the reactor feed gas of approximately 0.7 were tested during the periods of 12-18 December 2001 and 24-29 December 2001. During these test dates, heat and material balances were generated for periods of at least 12 hours of steady operation.

As part of the operating protocol following the completion of the in-situ activation of methanol synthesis catalyst in the LPMEOH™ Reactor, temperature programming continued during the quarter; this involved the increase of reactor temperature as necessary to control the reactor purge flowrate and maintain reactor productivity. On 15 November 2001, the reactor temperature was increased from 218°C to 220°C; on 04 December 2001, the reactor temperature was increased to 224°C; and, on 21 December 2001, the reactor temperature was further increased to 226°C. The reactor pressure was increased from 685 psig to 700 psig on 12 November 2001 and maintained at that level for the balance of the reporting period. The flowrate of Balanced Gas was controlled at an average value of 630 KSCFH during this time.

Work was continued during this period to reconcile the calculated activity of the methanol synthesis catalyst in the LPMEOH™ Reactor. Additional reactor catalyst samples from September of 2001 were evaluated for catalyst activity and chemical analysis. A check of catalyst activity in the autoclave was performed on a reactor catalyst sample from 19 September 2001 (26 days on-stream). This sample exhibited excellent activity, approaching the typical performance for properly activated catalyst. Throughout the reporting period, the calculated value of eta continued to appear to be about 50% of the value for freshly activated catalyst; it is not clear whether this discrepancy is real or possibly related to imperfections in the kinetic model. Additional work is planned to resolve this difference and determine the impact of reactor operating temperature on the results from the kinetic model.

Analyses of catalyst samples to determine changes in physical characteristics and levels of poisons have continued. 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 has indicated the presence of all expected crystal phases. Several known catalyst poisons including iron, arsenic, and sulfur have been detected on the catalyst and appear to be increasing.

29C-40 Catalyst Guard Bed Performance Monitoring and Assessment

After the completion of the in-situ activation of methanol synthesis catalyst in the LPMEOH™ Reactor on 24 August 2001, the 29C-40 catalyst guard bed was brought online. This was the first operation with the fresh charge of adsorbent (copper-impregnated activated carbon), which had been loaded into the vessel and reduced in July of 2001. (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 carbon dioxide [CO₂] or water [H₂O]). Throughout the reporting period, gas sampling was performed to assess the performance of the adsorbent. The analytical techniques involved analyzing the Balanced Gas leaving the catalyst guard bed for arsine using standard techniques (CO Gas was not used during this initial operating period). During the initial testing, the outlet concentration of arsine was determined to be less than the detection limit of the analysis (4 to 6 parts per billion by volume (ppbv)). Based on these results, the adsorbent in the catalyst guard bed was assessed to be performing well during the first 2 months of operation (or through late October of 2001).

During the period of 23 October 2001 to 06 November 2001 (or after approximately two months of service), the concentration of arsine at the outlet of the catalyst guard bed was determined to be above the detection limit and had reached an average value of 14 ppbv. This result was further corroborated during the period of 26 November 2001 to 04 December 2001 (or after approximately three months of service) when the concentration of arsine at the outlet of the catalyst guard bed was determined to be on the order of 20 ppbv. This analysis provided further indication of breakthrough of arsine from the catalyst guard bed.

On 04 December 2001, the catalyst guard bed was taken out of service and a thermal treatment was performed on the adsorbent in an attempt to provide increased capacity for arsine removal. The catalyst guard bed was then brought back online; no excessive temperature rise was measured. Analytical testing was again conducted following the thermal treatment. Initial results indicated that the concentration of arsine in the catalyst guard bed outlet stream was less than the detection limit of 6 ppbv. Additional testing of the outlet stream of the catalyst guard bed on a weekly basis is planned and will be used to evaluate changes in the performance of the adsorbent.

During the current reporting period, sampling of the inlet syngas to the catalyst guard bed to determine the concentration of arsine was not performed. However, the topical report “Alternative Fuels Field Test Unit Support to Kingsport LPMEOH™ Demonstration Unit – December 1997 to January 1998” had previously reported that the concentration in the inlet syngas was at least 31 ppbv based upon standard measurement techniques. It is planned next quarter to again measure and report the arsine concentration in the catalyst guard bed inlet syngas.

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 biennial outage in March of 2001. Appendix C, Figure 2 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. 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.

D.4 Planning and Administration

A DOE quarterly review meeting was held during the week of 10 December 2001 in Kingsport. The performance of the LPMEOH™ Demonstration Unit since the last meeting (June 2001) was the primary topic of discussion. The agenda, extracts from the handouts, and the notes for the meeting are included in Appendix D.

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

The poster entitled “Liquid Phase Methanol (LPMEOH™) Technology” was displayed at the Clean Coal and Power Conference (formerly the Clean Coal Technology Conference) on 19-20 November 2001.

The paper entitled “Liquid Phase Methanol (LPMEOH™) Process Development” was included in the proceedings 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 31 December 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 31 December 2001. Eighty-six percent (86%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 December 2001.

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

- 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 (including sampling of the inlet syngas stream), and monitoring the performance of the gas sparger in the reactor.
- Publish the Topical Report on the objectives and results of the off-site, product-use test program for stabilized methanol from the LPMEOH™ Process.
- Issue a draft Topical Report on the market analysis for DME.
- Schedule a Project Review Meeting with DOE.

F. Conclusion

The LPMEOH™ Demonstration Unit operated at 99.89% availability during this quarter. The forced downtime experienced this quarter (2.5 hours) was associated with an upset in the distillation section, which has not recurred. There were also three short syngas interruptions that were experienced on 23 October 2001 (12.4 hours duration), 29 October 2001 (9.9 hours duration), and 19 December 2001 (19.7 hours duration).

A catalyst deactivation rate of 0.2% per day was calculated for the period 08 October 2001 to 21 October 2001 (14 days). This is a significantly lower deactivation rate than the results that have been generally calculated over the past 2 years (averaging between 0.6% and 0.7% per day), and may be related to the improved performance of the adsorbent in the 29C-40 catalyst guard bed during that period (described below). A catalyst deactivation rate of 1.36% per day was calculated for the period 01 November 2001 to 13 November 2001 (13 days). A catalyst deactivation rate of 0.45% per day was calculated for the period 16 November 2001 to 02 December 2001 (17 days). The changes in the calculated rate of catalyst deactivation may be related to the impact of variations in the syngas composition on the kinetic model, or they may be process-related (for example, a change in either the concentration of trace contaminants in the reactor feed gas or the performance of the adsorbent in the catalyst guard bed could affect the catalyst performance). 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).

Following the restart after the syngas outage on 29 October 2001, the composition of Balanced Gas to the LPMEOH™ Demonstration Unit became CO-rich. During this 14-hour period of operation, the reactor inlet H₂/CO ratio was approximately 0.6 to 0.7. Later in the reporting period, sufficient CO Gas became available so that additional tests using CO-rich syngas were performed. Two operating periods with a ratio of H₂ to CO in the reactor feed gas of approximately 0.7 were tested during the periods of 12-18 December 2001 and 24-29 December 2001. During these test dates, heat and material balances were generated for periods of at least 12 hours of steady operation.

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continued during the quarter; this involved the increase of reactor temperature as necessary to control the reactor purge flowrate and maintain reactor productivity. On 15 November 2001, the reactor temperature was increased from 218°C to 220°C; on 04 December 2001, the reactor temperature was increased to 224°C; and, on 21 December 2001, the reactor temperature was further increased to 226°C. The reactor pressure was increased from 685 psig to 700 psig on 12 November 2001 and maintained at that level for the balance of the period. The flowrate of Balanced Gas was controlled at an average value of 630 KSCFH during this time.

Work was continued during this period to reconcile the calculated activity of the methanol synthesis catalyst in the LPMEOH™ Reactor. Additional reactor catalyst samples from September 2001 were evaluated for catalyst activity and chemical analysis. A check of catalyst activity in the autoclave was performed on a reactor catalyst sample from 19 September 2001 (26 days on-stream). This sample exhibited excellent activity, approaching the typical performance for properly activated catalyst. Throughout the reporting period, the calculated value of eta continued to appear to be about 50% of the value for freshly activated catalyst; it is not clear whether this discrepancy is real or possibly related to imperfections in the kinetic model. Additional work is planned to resolve this difference and determine the impact of reactor operating temperature on the results from the kinetic model.

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outlet stream of the catalyst guard bed on a weekly basis is planned and will be used to evaluate changes in the performance of the adsorbent.

Analyses of catalyst samples to determine changes in physical characteristics and levels of poisons have continued. 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. Several known catalyst poisons including iron, arsenic, and sulfur have been detected on the catalyst and appear to be increasing.

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 biennial outage in March of 2001. 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 4,832,009 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, about 87.8 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 Topical Report entitled “Off-Site Testing of Stabilized Methanol from the Liquid Phase Methanol (LPMEOH™) Process” was approved by DOE during the reporting period. This report provides the results from the seven test sites. During the reporting period, the unused stabilized methanol was returned to Eastman for further distillation prior to use within the chemicals-from-coal complex.

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 status of the current market for DME and an outlook on potential market developments through 2006.

A DOE quarterly review meeting was held during the week of 10 December 2001 in Kingsport. The performance of the LPMEOH™ Demonstration Unit since the last meeting (June 2001) was the primary topic of discussion.

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

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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 31 December 2001. Eighty-six percent (86%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 December 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 -
October/December 2001**

Table 2 - Summary of Catalyst Samples - Third Catalyst Batch

Figure 1 - Catalyst Age (η): September - December 2001

**Figure 2 - Sparger Resistance Coefficient vs. Days Onstream
(September - December 2001)**

Table 1
Summary of LPMEOH™ Demonstration Unit Outages - October/December 2001

Operation Start	Operation End	Operating Hours	Shutdown Hours	Reason for Shutdown
10/1/01 00:00	10/7/01 02:44	146.7	2.5	ESD Distillation Section
10/7/01 05:13	10/23/01 23:27	402.2	12.4	Syngas Outage
10/24/01 11:52	10/29/01 05:25	113.6	9.9	Syngas Outage
10/29/01 15:19	12/19/01 02:00	1210.7	19.7	Syngas Outage
12/19/01 21:44	12/31/01 23:59	290.3		End of Reporting Period
Total Operating Hours			2163.4	
Syngas Available Hours			2165.9	
Plant Availability, %			99.89	

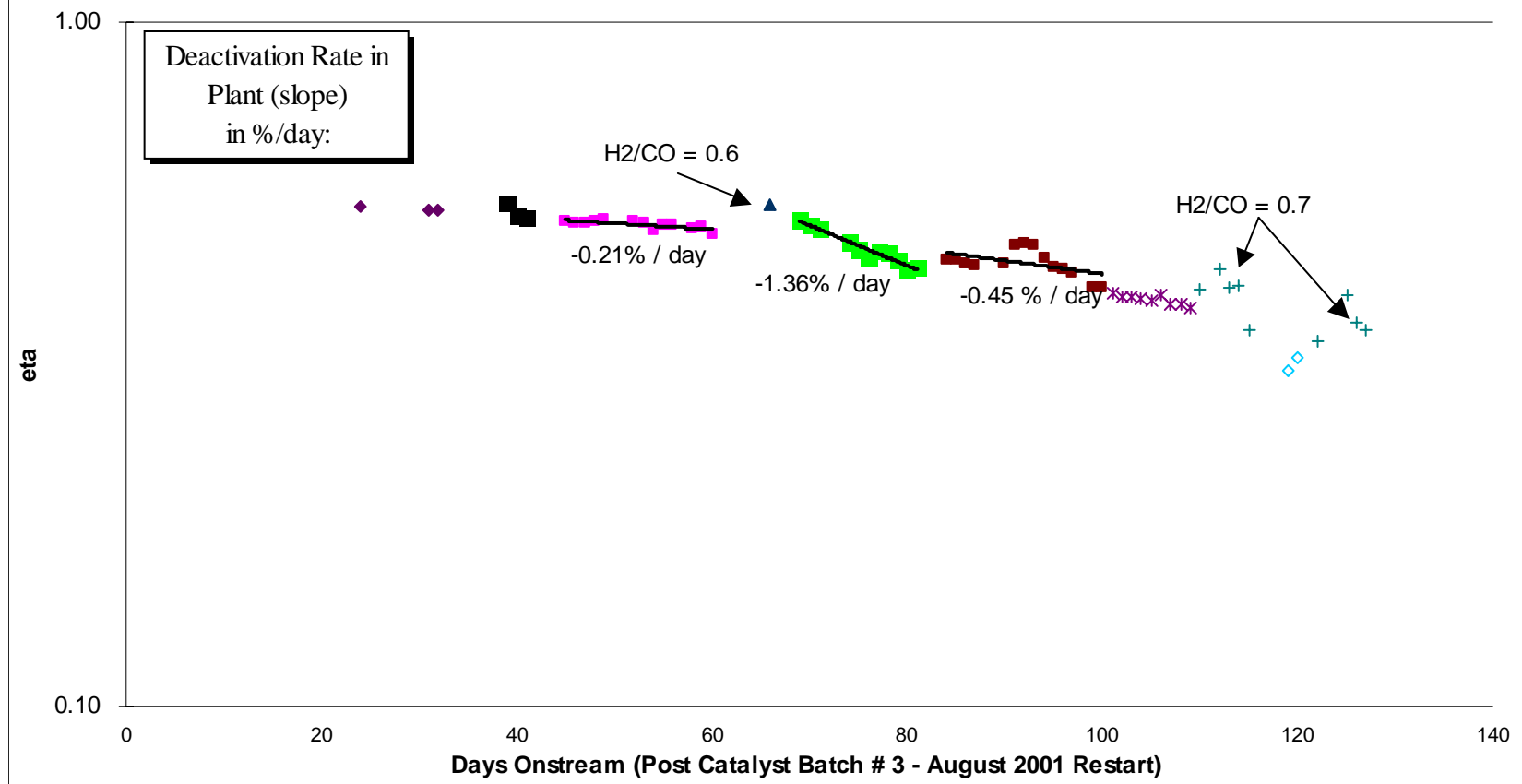
Table 2
Summary of Catalyst Samples - Third Catalyst Batch

Sample	Identity	XRD		BET	Analytical (ppmw)				
		Cu	ZnO	m ² /g	Fe	Ni	S	As	Cl
K0109-1	Reactor Sample 9/5/01	178	90	78	48	≤19	< 140	≤73	nd
K0109-2	Reactor Sample 9/12/01	188	88						
K0109-3	Reactor Sample 9/19/01	185		85	52	< 10	< 140	≤70	nd
K0110-1	Reactor Sample 10/3/01	181	78	82	71	< 10	≤85	65	nd
K0110-2	Reactor Sample 10/17/01	203	98	82	97	< 10	≤100	83	nd
K0110-3	Reactor Sample 10/31/01	197	45	78	147	< 10	≤120	139	40
K0111-1	Reactor Sample 11/14/01	200	80	80	191	< 10	≤130	138	nd
K0111-3	Reactor Sample 11/30/01	204	79	82	241	< 10	≤140	150	50
K0112-1	Reactor Sample 12/12/01	256	77						
K0201-1	Reactor Sample 1/09/02	212	78						

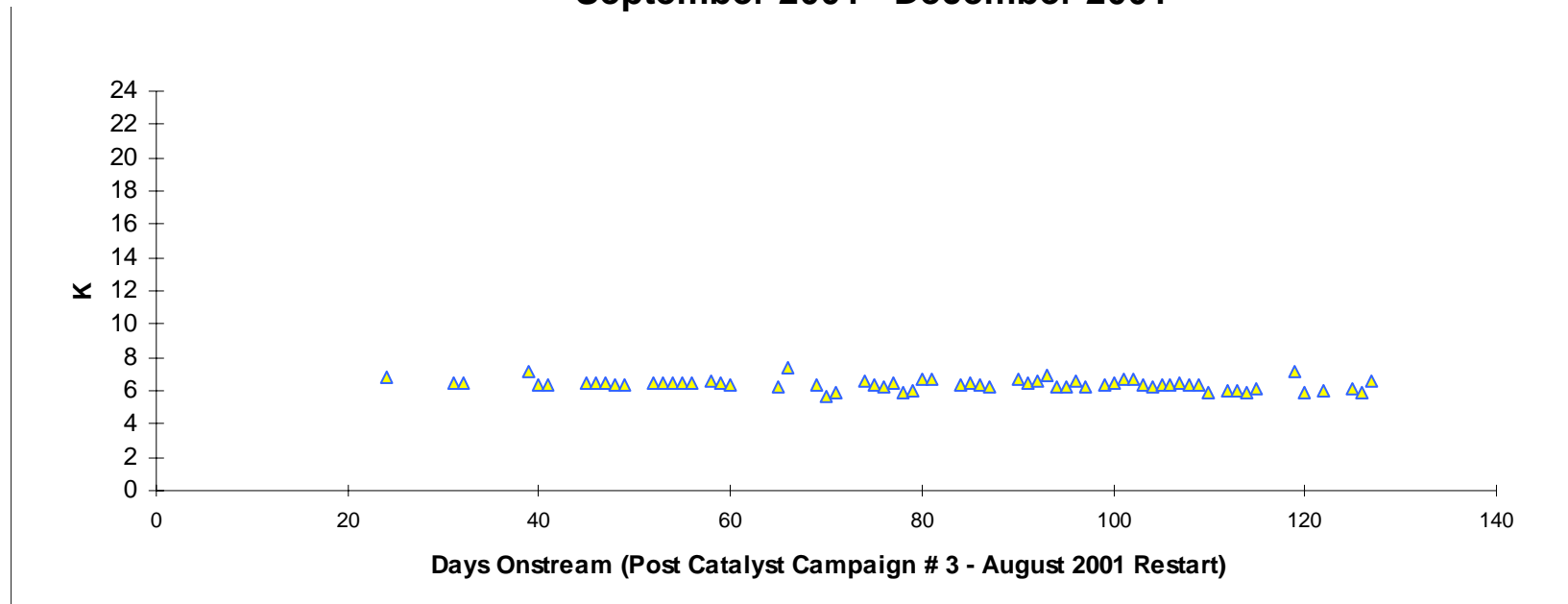
Notes:

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

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



**Figure 2 - Kingsport LPMEOH™ Sparger Resistance Coefficient
September 2001 - December 2001**



APPENDIX D - PROJECT REVIEW MEETING (11-12 DECEMBER 2001)

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