

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

TECHNICAL PROGRESS REPORT NO. 27

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for the

Air Products Liquid Phase Conversion Company, L.P.

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Abstract

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

The LPMEOH™ Demonstration Unit operated at 99.76% availability during this quarter. There was a single syngas outage experienced on 03 February 2001 (63 hours). There were also six short duration forced trips during the month for a total of 4.7 hours. The trips were caused by leakage of boiler feed water through the flow control valve which feeds the steam drum. This valve will be repaired during the biennial outage which began on 28 March 2001. During this outage, a scheduled inspection of all pressure vessels as required by Tennessee state code will be performed, and the engineering modifications required for the reduction of the adsorbent in the catalyst guard bed and for the in-situ activation of methanol synthesis catalyst in the LPMEOH™ Reactor will be completed.

A major catalyst withdrawal and addition campaign was undertaken during January and February of 2001 to raise catalyst activity. A series of three withdrawals were conducted on 25 and 26 January 2001. This was followed by four catalyst additions which were activated and added between 28 January and 07 February 2001. After the addition of the fourth batch of catalyst, the total catalyst inventory was calculated to be 47,160 pounds.

The syngas outage that occurred on 03 February 2001 also interrupted the completion of the activation of the final catalyst batch, as the syngas stream which contains primarily carbon monoxide (Carbon Monoxide Gas, or CO Gas), which is used in the activation procedure, was not available to the 29C-30 catalyst reduction vessel. As soon as CO Gas became available on 05 February 2001, the program for activation was restarted at the point of the interruption. Analysis of the data showed that the uptake of CO, which indicates the extent of reduction, was acceptable for this catalyst batch.

During most of the quarter, the flowrate of the primary syngas feed (Balanced Gas) was controlled at an average flowrate of 775 KSCFH. During these operating periods, the reactor pressure was set an average of 710 psig and temperature was maintained at 235°C.

There were two extended periods of operation (minimum of about 2 weeks) at a reactor temperature of 235°C during which catalyst activity was measured to track catalyst deactivation during the quarter. During both periods, the deactivation rate was achieved with the 29C-40 catalyst guard bed bypassed. A catalyst deactivation rate of 0.39% per day was calculated for the period 27 November 2000 to 16 January 2001 (51 days). A catalyst deactivation rate of 0.47% per day was calculated for the period 08 February 2001 to 02 March 2001 (23 days). These deactivation rates compare favorably with results over the past 12 months where the

calculated deactivation rate has varied from 0.6 to 0.8% per day. These deactivation results are also similar to the baseline deactivation rate of 0.4% per day from the 4-month proof-of-concept run at the LaPorte AFDU in 1988/89 (this run was performed at 250°C).

Analyses of catalyst samples for changes in physical characteristics and levels of poisons have continued. The concentration of arsenic, which has been demonstrated in the laboratory to act as a poison to methanol synthesis catalyst, appeared to have stabilized during most of the periods of improved deactivation noted above. During the same period, iron concentration was also low and steady. However, sulfur, another known catalyst poison, was still increasing. The reduction in the rate of increase of arsenic and iron on the catalyst may have contributed to the improvement in the rate of catalyst deactivation for those time periods. Copper crystallite size measurements have not continued to increase in the most recent samples. Levels of nickel (a known catalyst poison) have remained low and steady since the restart in December of 1997. Any of these measurements will stabilize when the net rate of accumulation of these species on the catalyst is constant, indicating no large changes in either the concentration in the gas phase or the catalyst withdrawal rate from the LPMEOH™ Reactor.

The performance of the gas sparger, which was designed by Air Products and installed into the LPMEOH™ Reactor prior to the restart of the LPMEOH™ Demonstration Unit in March of 1999, was monitored. The performance to date has met the design expectations for pressure drop and reactor operation.

The first two tests of the ability of the LPMEOH™ Reactor to operate in a load-following environment were performed during the week of 15 January 2001. For two consecutive days, the reactor was taken offline for 12 hours, and then Balanced Gas was introduced to the process as quickly as possible (within the limitations of other systems at the Eastman chemicals-from-coal complex). The attempts to perform these ramping tests were hampered by the standby conditions which were selected, including the lack of flow to the reactor (which lowered the reactor heat transfer coefficient) and the reactor temperature (180°C). Once Balanced Gas flowrate was reestablished and reactor temperature and pressure were placed into control, the performance of the LPMEOH™ Demonstration Unit, including the gas sparger in the reactor, was determined to have returned to the condition prior to the shutdown.

Three additional ramping tests were performed between 04 and 13 March 2001. For the first two tests (on 04 and 06 March), the reactor standby temperature was 216°C and 212°C, respectively; from this starting point, a reasonable ramping rate could not be reached. During the third test on 13 March 2001, a standby reactor temperature of 227°C was used; for all of the tests performed in March of 2001, gas flow from the recycle compressor was maintained to the LPMEOH™ Reactor. Balanced Gas was then introduced to the process as quickly as possible (within the limitations of other systems at the Eastman chemicals-from-coal complex). A maximum ramp rate of 5.7% full load per minute and an average ramp rate of 4.2% full load per minute were achieved during this testing. This ramp performance approached the minimum target ramp rate of 5% full load per minute. For each test, once Balanced Gas flowrate was reestablished and reactor temperature and pressure were placed

into control, the performance of the LPMEOH™ Demonstration Unit was determined to have returned to the condition prior to the shutdown.

During the reporting period, a total of 5,951,274 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, about 76.4 million gallons of methanol has been produced. Eastman accepted all of this methanol for use in the production of methyl acetate, and ultimately cellulose acetate and acetic acid. No safety or environmental incidents were reported during this quarter.

During this quarter, planning, procurement, and test operations were concluded on the project sites selected for the off-site, product-use test program. During the reporting period, Air Products approved final reports for two of the seven projects which tested stabilized methanol from the LPMEOH™ Demonstration Project. All of the final reports have now been approved by Air Products.

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.

During this reporting period, DOE approved an update to the Demonstration Test Plan which defines the tests which are planned for the remainder of the operating program, and those pages which were impacted by this change were reissued.

A DOE quarterly review meeting was held during the week of 22 January 2001 at the Kingsport site. The performance of the LPMEOH™ Demonstration Facility since the last meeting (June 2000) was the primary topic of discussion.

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 March 2001. Seventy-two percent (72%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 March 2001.

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ACRONYMS AND DEFINITIONS

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

This project 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.

The LPMEOH™ Demonstration Unit operated at 99.76% availability during this quarter. There was a single syngas outage experienced on 03 February 2001 (63 hours). There were also six short duration forced trips during the month for a total of 4.7 hours. The trips were caused by leakage of boiler feed water through the flow control valve which feeds the steam drum. This resulted in a high steam drum level which initiated a shutdown of the recycle compressor which tripped the plant. The boiler feed water valve will be repaired during the biennial outage which began on 28 March 2001. During this outage, a scheduled inspection of all pressure vessels as required by Tennessee state code will be performed. Some of the key activities include the inspection of reactor and economizer heat exchanger and completion of the engineering modifications required for the reduction of the adsorbent in

the catalyst guard bed and for the in-situ activation of methanol synthesis catalyst in the LPMEOH™ Reactor.

A major catalyst withdrawal and addition campaign was undertaken during January and February of 2001 to raise catalyst activity. A series of three withdrawals were conducted on 25 and 26 January 2001. This was followed by four catalyst additions which were activated and added between 28 January and 07 February 2001. After the addition of the fourth batch of catalyst, the total catalyst inventory was calculated to be 47,160 pounds.

The syngas outage that occurred on 03 February 2001 also interrupted the completion of the activation of the final catalyst batch, as the syngas stream which contains primarily carbon monoxide (Carbon Monoxide Gas, or CO Gas), which is used in the activation procedure, was not available to the 29C-30 catalyst reduction vessel. As soon as CO Gas became available on 05 February 2001, the program for activation was restarted at the point of the interruption. Analysis of the data showed that the uptake of CO, which indicates the extent of reduction, was acceptable for this catalyst batch.

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There were two extended periods of operation (minimum of about 2 weeks) at a reactor temperature of 235°C during which catalyst activity was measured to track catalyst deactivation during the quarter. During both periods, the deactivation rate was achieved with the 29C-40 catalyst guard bed bypassed. A catalyst deactivation rate of 0.39% per day was calculated for the period 27 November 2000 to 16 January 2001 (51 days). A catalyst deactivation rate of 0.47% per day was calculated for the period 08 February 2001 to 02 March 2001 (23 days). These deactivation rates compare favorably with results over the past 12 months where the calculated deactivation rate has varied from 0.6 to 0.8% per day. These deactivation results are also similar to the baseline deactivation rate of 0.4% per day from the 4-month proof-of-concept run at the LaPorte AFDU in 1988/89 (this run was performed at 250°C), and may reflect the impact of poisons on catalyst aging.

Analyses of catalyst samples for changes in physical characteristics and levels of poisons have continued. The concentration of arsenic, which has been demonstrated in the laboratory to act as a poison to methanol synthesis catalyst, appeared to have stabilized during most of the periods of improved deactivation noted above. During the same periods, iron concentration was also low and steady. However, sulfur, another known catalyst poison, was still increasing. The reduction in the rate of increase of arsenic and iron on the catalyst may have contributed to the improvement in the rate of catalyst deactivation for those time periods. Copper crystallite size measurements have not continued to increase in the most recent samples. Levels of nickel (a known catalyst poison) have remained low and steady since the restart in December of 1997. Any of these measurements will stabilize when the net rate of accumulation of these species on the catalyst is constant, indicating no large changes in either the concentration in the gas phase or the catalyst withdrawal rate from the LPMEOH™ Reactor.

The performance of the gas sparger, which was designed by Air Products and installed into the LPMEOH™ Reactor prior to the restart of the LPMEOH™ Demonstration Unit in March of 1999, was monitored. The performance to date has met the design expectations for pressure drop and reactor operation.

The first two tests of the ability of the LPMEOH™ Reactor to operate in a load-following environment were performed during the week of 15 January 2001. For two consecutive days, the reactor was taken offline for 12 hours, and then Balanced Gas was introduced to the process as quickly as possible (within the limitations of other systems at the Eastman chemicals-from-coal complex) from a standby condition. The attempts to perform these ramping tests were hampered by the standby conditions which were selected, including the lack of flow to the reactor (which lowered the reactor heat transfer coefficient) and the reactor temperature (180°C). Once Balanced Gas flowrate was reestablished and reactor temperature and pressure were placed into control, the performance of the LPMEOH™ Demonstration Facility was determined to have returned to the condition prior to the shutdown. In particular, the flowrate of syngas to the gas sparger in the LPMEOH™ Reactor was stopped during most of the downtime; although there was evidence of settling of catalyst, the sparger was able to remix the slurry, and the sparger resistance coefficient returned to its pre-outage level.

Three additional ramping tests were performed on 04 March 2001, 06 March 2001, and 13 March 2001, respectively. For the first two tests, the reactor was taken offline for 7-12 hours, and then the plant was restarted to obtain additional on-off performance data. For these tests, the reactor standby temperature was 216°C and 212°C, respectively; from this starting point, a reasonable ramping rate could not be reached. During the third test on 13 March 2001, the reactor was taken offline for 9.6 hours. A standby reactor temperature of 227°C was selected for this test; for all of the tests performed in March of 2001, gas flow from the recycle compressor was maintained to the LPMEOH™ Reactor. Balanced Gas was then introduced to the process as quickly as possible (within the limitations of other systems at the Eastman chemicals-from-coal complex) from a standby condition. A maximum ramp rate of 5.7% full load per minute and an average ramp rate of 4.2% full load per minute were achieved during this testing. This ramp performance approached the minimum target ramp rate of 5% full load per minute. For each test, once Balanced Gas flowrate was reestablished and reactor temperature and pressure were placed into control, the performance of the LPMEOH™ Demonstration Facility was determined to have returned to the condition prior to the shutdown.

During the reporting period, a total of 5,951,274 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, about 76.4 million gallons of methanol has been produced. Eastman accepted all of this methanol for use in the production of methyl acetate, and ultimately cellulose acetate and acetic acid. No safety or environmental incidents were reported during this quarter.

During this quarter, planning, procurement, and test operations were concluded on the project sites selected for the off-site, product-use test program. During the reporting period, Air Products approved final reports for two of the seven projects which tested stabilized methanol from the LPMEOH™ Demonstration Project. One project (at West Virginia

University) measured the emissions from a low NO_x microturbine in a distributed power generation application; the second project (at the University of Florida) studied the acceptability of using stabilized methanol as a fuel to phosphoric acid fuel cell power systems. All of the final reports have now been approved by Air Products. Sponsorship (in the form of supply of stabilized methanol) has been provided to the Florida Institute of Technology as part of their contract with the Florida Energy Office to test methanol as a transportation fuel; this sponsorship was concluded during this quarter.

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

During this reporting period, DOE approved an update to the Demonstration Test Plan which defines the tests which are planned for the remainder of the operating program, and those pages which were impacted by this change were reissued.

A DOE quarterly review meeting was held during the week of 22 January 2001 at the Kingsport site. The performance of the LPMEOH™ Demonstration Facility since the last meeting (June 2000) was the primary topic of discussion.

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 March 2001. Seventy-two percent (72%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 March 2001.

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 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)

Discussion

The product-use test program, developed in 1992 to support the demonstration at the original Cool Water Gasification Facility site, became outdated due in large part to changes within

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

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

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

Activity during this quarter

All of the projects have completed testing of stabilized methanol, and are at stages of development of their respective final reports. Status and highlights include:

ARCADIS Geraghty & Miller Flexible-Fuel Vehicle (FFV) - The final report for this project was approved by Air Products (no update in this reporting period).

Stationary Turbine for Nitrogen Oxide (NO_x) Control - Air Products approved a final report on the measurement of emissions from a low NO_x microturbine in a distributed power generation application which was fueled with stabilized methanol from the LPMEOH™ Demonstration Project. As noted in a prior report, this methanol yields similar or slightly better emissions results for NO_x and CO when compared with natural gas.

West Virginia University (WVU) Stationary Gas Turbine - Testing of stabilized methanol in the gas turbine system has been completed, the final report has been approved by Air Products.

Aircraft Ground Equipment Emulsion - A final report on the use of a methanol emulsion as the fuel for a flight line generator at Tyndall Air Force Base was approved by Air Products.

University of Florida Fuel Cell - Air Products approved a final report on the acceptability of using stabilized methanol from the LPMEOH™ Demonstration Project as a fuel to phosphoric acid fuel cell power systems. As noted in prior reports, the cost advantage of avoiding distillation of higher alcohols and trace mineral oil in the stabilized methanol is offset by the need for higher operating temperature to process these species in the reformer which is upstream of the fuel cell stack.

West Virginia University Tri-Boro Bus - The final report for this project was approved by Air Products.

Florida Institute of Technology Bus & Light Vehicle - The final report on testing of stabilized methanol as a transportation fuel at the Florida Institute of Technology was approved by Air Products. Since September 1999, stabilized methanol from the LPMEOH™ Process Demonstration Unit has been provided to Florida Institute of Technology for use as part a new contract between the Institute and the Florida Energy Office. Air Products has received copies of the reports which are submitted to the State of Florida. This sponsorship was concluded during the reporting period.

A Topical Report will be developed which summarizes the objectives and results of the off-site, product-use test program.

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, was approved by DOE and issued.

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

D.3 LPMEOH™ Process Demonstration Unit - Methanol Operation

Table D.3-1 contains the summary table of performance data for the LPMEOH™ Demonstration Unit during the reporting period. These data represent daily averages, typically from a 24-hour material balance period, and those days with less than 12 hours of stable operation are omitted. Appendix B contains samples of the detailed material balance reports which are representative of the operation of the LPMEOH™ Demonstration Unit during the reporting period.

During the reporting period, a total of 5,951,274 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Eastman accepted all of this methanol for use in the production of methyl acetate, and ultimately cellulose acetate and acetic acid. No environmental incidents or injuries were reported during this quarter.

The LPMEOH™ Demonstration Unit operated at 99.76% availability during this quarter. Appendix C, Table 1 contains the summary of outages for the LPMEOH™ Demonstration Unit during this quarter. There was a single syngas outage experienced on 03 February 2001 (63 hours). There were also six short duration forced trips during the month for a total of 4.7 hours. The trips were caused by leakage of boiler feed water through the flow control valve which feeds the steam drum. This resulted in a high steam drum level which initiated a shutdown of the recycle compressor which tripped the plant. The boiler feed water valve will be repaired during the biennial outage which began on 28 March 2001. During this outage, a scheduled inspection of all pressure vessels as required by Tennessee state code will be performed. Some of the key activities include the inspection of reactor and economizer heat exchanger and completion of the engineering modifications required for the reduction of the adsorbent in the catalyst guard bed and for the in-situ activation of methanol synthesis catalyst in the LPMEOH™ Reactor.

Catalyst Life (η) – January - March 2001

The “age” of the methanol synthesis catalyst can be expressed in terms of a dimensionless variable η , which is defined as the ratio of the rate constant at any time to the rate constant for freshly reduced catalyst (as determined in the laboratory autoclave). Appendix C, Figure 1 plots $\log \eta$ versus days onstream from June of 2000 to the end of the reporting period. Since catalyst activity typically follows a pattern of exponential decay, the plot of $\log \eta$ is fit to a series of straight lines, with step-changes whenever fresh catalyst was added to the reactor.

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		Syngas Util. (SCF/lb)	Raw MeOH Prod. (TPD)	Catalyst MeOH Prod. (gmol/hr-kg)	Reactor Vol. Prod. (TPD/l3)	U		
																		O-TM Conv. (%)	U Util. (%)					Overall (Btu/hr ft2 F)	Sparger dP (psi)	Sparger Resist. ("K")
11	11-Jan-01	1118	Balanced	235	710	731	2,146	3.33	75.0	0.67	3613	44.9	34.2	53.0	47,163	0.299	31.5	20.9	41.1	213.7	11.82	0.096	135	5.44	5.28	
11	12-Jan-01	1119	Balanced	236	710	720	2,143	3.14	73.6	0.67	3584	46.8	35.5	50.5	47,163	0.291	32.8	20.8	41.0	210.8	11.66	0.099	139	5.19	5.35	
11	13-Jan-01	1120	Balanced	234	710	720	2,168	3.05	83.5	0.67	3623	44.7	29.6	50.0	47,163	0.294	31.4	20.3	41.4	208.7	11.54	0.099	135	5.33	5.31	
11	14-Jan-01	1121	Balanced	234	710	721	2,158	3.28	76.6	0.67	3607	46.2	33.5	50.0	47,163	0.298	30.7	20.6	41.1	210.5	11.69	0.100	141	5.42	5.28	
11	15-Jan-01	1122	Balanced	234	710	714	2,156	3.02	80.6	0.67	3592	44.7	30.2	50.5	47,163	0.290	31.1	20.3	41.5	206.5	11.42	0.097	128	5.23	5.29	
11	16-Jan-01	1123	Balanced	235	710	679	2,218	3.36	79.0	0.67	3624	42.3	23.5	50.5	47,163	0.265	28.6	19.0	41.8	195.1	10.79	0.092	117	5.41	5.31	
11	19-Jan-01	1126	Balanced	235	710	723	2,117	2.96	103.2	0.66	3582	42.7	24.1	50.0	47,163	0.281	30.5	20.2	42.7	203.1	11.24	0.097	118	5.41	5.25	
11	22-Jan-01	1129	Balanced	235	715	735	2,196	2.75	119.3	0.68	3682	45.2	31.6	50.5	47,163	0.266	27.4	19.2	44.1	200.3	11.08	0.094	124	5.57	5.16	
11	24-Jan-01	1131	Balanced	235	715	726	2,232	2.91	107.0	0.68	3703	45.1	31.2	50.5	47,163	0.267	28.3	19.1	43.4	200.8	11.11	0.095	129	5.37	5.12	
11	26-Jan-01	1133	Balanced	234	715	589	2,141	3.54	92.3	0.63	4196	42.6	29.4	44.0	38,360	0.272	28.3	16.5	43.7	161.6	10.99	0.088	154	4.55	5.80	
11	27-Jan-01	1134	Balanced	234	715	590	2,226	3.32	90.7	0.65	4309	41.8	28.6	45.0	38,360	0.262	26.2	16.1	44.1	160.6	10.93	0.085	142	4.79	5.54	
11	28-Jan-01	1135	Balanced	234	714	606	2,218	3.15	96.6	0.65	4326	42.3	30.1	45.0	38,360	0.270	26.0	16.6	43.7	166.5	11.33	0.088	151	4.87	5.41	
11	2-Feb-01	1140	Balanced	235	710	847	2,088	3.72	100.0	0.68	3834	39.2	21.6	53.0	44,960	0.401	42.4	23.8	41.5	244.8	14.20	0.110	129	4.99	5.33	
11	8-Feb-01	1146	Balanced	235	710	859	2,120	3.33	61.1	0.69	3721	38.2	23.4	59.0	47,160	0.433	42.3	25.5	39.3	262.3	14.50	0.106	136	5.97	5.40	
11	9-Feb-01	1147	Balanced	235	710	811	2,113	3.51	57.3	0.67	3632	41.3	30.3	57.5	47,160	0.412	42.7	24.8	39.4	247.2	13.67	0.102	131	6.62	5.36	
11	10-Feb-01	1148	Balanced	235	710	833	2,129	3.26	60.2	0.69	3714	41.2	30.1	57.5	47,160	0.403	40.0	24.6	40.0	253.1	13.67	0.105	128	6.14	5.65	
11	11-Feb-01	1149	Balanced	235	710	838	2,110	3.40	65.9	0.68	3684	41.2	27.6	55.5	47,160	0.404	41.4	24.7	39.7	253.7	14.03	0.109	135	5.38	5.23	
11	12-Feb-01	1150	Balanced	235	710	809	2,130	3.36	49.0	0.69	3693	41.6	26.7	54.0	47,160	0.405	40.9	24.5	38.6	251.5	13.90	0.111	137	5.51	5.25	
11	14-Feb-01	1152	Balanced	235	710	867	2,067	3.26	67.2	0.68	3666	41.8	25.1	52.5	47,160	0.426	41.7	25.7	39.7	262.2	14.50	0.119	137	5.53	5.25	
11	15-Feb-01	1153	Balanced	234	710	869	2,048	3.34	89.8	0.67	3634	44.3	30.7	51.5	47,160	0.421	42.0	25.2	40.5	257.4	14.27	0.119	131	5.32	5.21	
11	16-Feb-01	1154	Balanced	234	710	884	2,024	3.51	103.0	0.67	3625	42.0	23.3	51.0	47,160	0.415	43.0	25.0	41.4	256.5	14.20	0.120	130	5.45	5.75	
11	17-Feb-01	1155	Balanced	234	710	884	2,019	3.52	102.9	0.67	3627	40.3	18.4	51.0	47,160	0.405	42.5	24.8	41.5	255.4	14.13	0.119	131	5.93	6.41	
11	18-Feb-01	1156	Balanced	236	710	883	2,089	3.25	94.3	0.69	3717	40.1	16.7	50.5	47,160	0.387	40.1	24.9	40.8	259.8	14.36	0.123	121	5.56	5.39	
11	19-Feb-01	1157	Balanced	236	710	883	2,068	3.15	96.4	0.69	3707	45.1	29.1	49.0	47,160	0.384	41.0	39.1	41.0	258.2	14.28	0.126	125	5.64	5.35	
11	20-Feb-01	1158	Balanced	236	710	891	2,067	3.11	103.6	0.69	3719	45.1	30.6	50.0	47,160	0.383	38.7	24.8	41.3	259.1	14.33	0.123	121	5.52	5.16	
11	21-Feb-01	1159	Balanced	235	710	886	2,083	3.02	90.6	0.68	3652	41.4	24.5	53.0	47,160	0.397	38.8	25.4	41.1	258.8	14.31	0.116	124	5.8	5.23	
11	22-Feb-01	1160	Balanced	234	710	891	2,052	2.98	94.8	0.68	3641	42.9	27.5	52.0	47,160	0.405	41.2	25.6	41.2	259.7	14.36	0.119	129	5.95	5.38	
11	23-Feb-01	1161	Balanced	234	710	886	2,061	3.08	100.1	0.68	3642	43.0	26.3	51.0	47,160	0.391	38.4	24.8	41.9	253.7	14.03	0.118	126	5.7	5.33	
11	28-Feb-01	1166	Balanced	234	710	823	2,077	2.98	72.7	0.67	3597	45.2	28.8	48.5	47,160	0.386	37.4	24.7	40.5	246.5	13.63	0.121	131	5.94	5.42	
11	1-Mar-01	1167	Balanced	234	710	832	2,069	3.05	80.5	0.66	3558	44.2	25.2	48.0	47,160	0.382	38.0	24.6	40.9	243.8	13.48	0.121	130	5.57	5.25	
11	2-Mar-01	1168	Balanced	234	710	827	2,058	3.08	88.3	0.66	3555	46.3	29.4	47.0	47,160	0.372	37.4	24.1	41.9	236.6	13.09	0.120	128	5.59	5.18	
11	6-Mar-01	1172	Balanced	234	710	736	2,227	3.13	78.2	0.67	3633	44.4	22.6	46.0	47,160	0.304	32.3	22.6	42.0	210.2	11.62	0.109	112	5.48	5.28	
11	7-Mar-01	1173	Balanced	233	710	781	2,183	3.06	89.0	0.67	3641	44.7	18.0	43.0	47,160	0.329	33.6	21.8	42.0	222.8	12.32	0.124	126	5.5	5.19	
11	8-Mar-01	1174	Balanced	234	710	792	2,118	3.26	74.8	0.66	3559	42.3	22.7	50.0	47,160	0.356	37.7	23.3	41.3	230.2	12.73	0.110	128	5.48	5.31	
11	13-Mar-01	1179	Balanced	235	709	662	2,226	3.36	73.7	0.66	3533	41.0	23.4	53.0	47,160	0.271	32.0	19.3	42.7	185.8	10.29	0.083	107	5.92	5.50	
11	15-Mar-01	1181	Balanced	235	710	733	2,186	3.23	86.0	0.66	3578	41.8	24.2	52.0	47,160	0.311	34.2	21.3	42.3	207.7	11.49	0.095	130	5.88	5.27	
11	17-Mar-01	1183	Balanced	235	710	742	2,182	3.24	101.8	0.67	3586	43.0	25.6	50.5	47,160	0.292	32.9	20.5	43.4	204.9	11.34	0.097	128	5.68	5.47	
11	18-Mar-01	1184	Balanced	235	710	749	2,182	3.22	99.4	0.67	3584	42.8	21.7	48.5	47,160	0.294	33.0	20.6	43.4	207.1	11.46	0.102	133	5.55	5.42	
11	19-Mar-01	1185	Balanced	235	710	755	2,164	3.32	115.0	0.66	3573	41.1	18.4	49.5	47,160	0.296	33.8	20.7	43.9	206.4	11.42	0.099	132	5.48	5.40	
11	20-Mar-01	1186	Balanced	235	710	736	2,152	3.33	101.7	0.66	3561	44.5	26.0	48.0	47,160	0.292	33.5	20.5	44.2	203.7	11.27	0.101	134	5.49	5.43	
11	21-Mar-01	1187	Balanced	234	710	737	2,169	3.13	115.9	0.66	3576	41.8	17.8	48.0	47,160	0.284	31.5	17.8	44.4	199.0	11.01	0.099	131	5.7	5.28	
11	22-Mar-01	1188	Balanced	235	710	742	2,156	3.29	137.3	0.66	3562	41.0	16.1	48.5	47,160	0.272	31.6	19.6	45.9	193.8	10.72	0.095	127	5.74	5.59	
11	23-Mar-01	1189	Balanced	235	708	716	2,186	3.28	95.0	0.67	3589	40.1	14.0	49.0	47,160	0.277	29.3	19.8	42.9	200.5	11.09	0.097	122	6.02	5.60	
11	24-Mar-01	1190	Balanced	235	708	744	2,171	2.86	97.2	0.67	3585	43.5	24.9	49.0	47,160	0.297	30.7	20.9	42.4	210.5	11.65	0.102	132	6.02	5.46	
11	25-Mar-01	1191	Balanced	234	710	739	2,212	2.88	91.6	0.68	3648	43.0	23.3	49.0	47,160	0.290	29.9	20.3	42.7	207.5	11.48	0.101	124	6.43	5.79	
11	26-Mar-01	1192	Balanced	234	710	746	2,201	2.90	90.6	0.67	3630	42.8	24.3	48.0	47,160	0.293	30.4	20.5	42.8	208.8	11.55	0.104	130	5.81	5.33	
11	27-Mar-01	1193	Balanced	234	710	732	2,226	2.87	100.1	0.68	3657	43.2	18.9	46.0	47,160	0.280	28.9	19.6	43.5	202.0	11.17	0.105	124	6.05	5.50	

A major catalyst withdrawal and addition campaign was undertaken during January and February of 2001 to raise catalyst activity. A series of three withdrawals were conducted on 25 and 26 January 2001. This was followed by four catalyst additions which were activated and added between 28 January and 07 February 2001. After the addition of the fourth batch of catalyst, the total catalyst inventory was calculated to be 47,160 pounds.

The syngas outage that occurred on 03 February 2001 also interrupted the completion of the activation of the final catalyst batch, as CO Gas, which is used in the activation procedure, was not available to the 29C-30 catalyst reduction vessel. As soon as CO Gas became available on 05 February 2001, the program for activation was restarted at the point of the interruption. Analysis of the data showed that the uptake of CO, which indicates the extent of reduction, was acceptable for this catalyst batch.

During most of the quarter, the flowrate of Balanced Gas was controlled at an average flowrate of 775 KSCFH. During these operating periods, the reactor pressure was set an average of 710 psig and temperature was maintained at 235°C.

There were two extended periods of operation (minimum of about 2 weeks) at a reactor temperature of 235°C during which catalyst activity was measured to track catalyst deactivation during the quarter. During both periods, the deactivation rate was achieved with the 29C-40 catalyst guard bed bypassed. A catalyst deactivation rate of 0.39% per day was calculated for the period 27 November 2000 to 16 January 2001 (51 days). A catalyst deactivation rate of 0.47% per day was calculated for the period 08 February 2001 to 02 March 2001 (23 days). These deactivation rates compare favorably with results over the past 12 months where the calculated deactivation rate has varied from 0.6 to 0.8% per day as plotted in Appendix C, Figure 1. These deactivation results are also similar to the baseline deactivation rate of 0.4% per day from the 4-month proof-of-concept run at the LaPorte AFDU in 1988/89 (this run was performed at 250°C), and may reflect the impact of poisons on catalyst aging.

Analyses of catalyst samples for changes in physical characteristics and levels of poisons have continued. Appendix C, Table 2 summarizes the results to date. The concentration of arsenic, which has been demonstrated in the laboratory to act as a poison to methanol synthesis catalyst, appeared to have stabilized during most of the periods of improved deactivation noted above. During the same periods, iron concentration was also low and steady (about 100 ppmw). However, sulfur, another known catalyst poison, was still increasing (to about 400 ppmw). The reduction in the rate of increase of arsenic and iron on the catalyst may have contributed to the improvement in the rate of catalyst deactivation for those time periods. Copper crystallite size measurements have not continued to increase in the most recent samples. Levels of nickel (a known catalyst poison) have remained low and steady since the restart in December of 1997. Any of these measurements will stabilize when the net rate of accumulation of these species on the catalyst is constant, indicating no large changes in either the concentration in the gas phase or the catalyst withdrawal rate from the LPMEOH™ Reactor.

Sparger Resistance

The performance of the new sparger continues to exhibit excellent performance during this quarter. The sparger resistance has not increased significantly over this past quarter. Appendix C, Figure 2 plots the average daily sparger resistance coefficient for the reporting period. The data for this plot, along with the corresponding average pressure drop, are also included in Table D.3-1.

Ramping Studies

The first two tests of the ability of the LPMEOH™ Reactor to operate in a load-following environment were performed during the week of 15 January 2001. For two consecutive days, the reactor was taken offline for 12 hours, and then Balanced Gas was introduced to the process as quickly as possible (within the limitations of other systems at the Eastman chemicals-from-coal complex) from a standby condition. The attempts to perform these ramping tests were hampered by the standby conditions which were selected, including the lack of flow to the reactor (which lowered the reactor heat transfer coefficient) and the reactor temperature (180°C). Once Balanced Gas flowrate was reestablished and reactor temperature and pressure were placed into control, the performance of the LPMEOH™ Demonstration Unit was determined to have returned to the condition prior to the shutdown. In particular, the flowrate of syngas to the gas sparger in the LPMEOH™ Reactor was stopped during most of the downtime; although there was evidence of settling of catalyst, the sparger was able to remix the slurry, and the sparger resistance coefficient returned to its pre-outage level.

Three additional ramping tests were performed on 04 March 2001, 06 March 2001, and 13 March 2001, respectively. For the first two tests, the reactor was taken offline for 7-12 hours, and then the plant was restarted to obtain additional on/off performance data. For these tests, the reactor standby temperature was 216°C and 212°C, respectively; from this starting point, a reasonable ramping rate could not be reached. During the third test on 13 March 2001, the reactor was taken offline for 9.6 hours. A standby reactor temperature of 227°C was selected for this test; for all of the tests performed in March of 2001, gas flow from the recycle compressor was maintained to the LPMEOH™ Reactor. Balanced Gas was then introduced to the process as quickly as possible (within the limitations of other systems at the Eastman chemicals-from-coal complex) from a standby condition. A maximum ramp rate of 5.7% full load per minute and an average ramp rate of 4.2% full load per minute were achieved during this testing. This ramp performance approached the minimum target ramp rate of 5% full load per minute. For each test, once Balanced Gas flowrate was reestablished and reactor temperature and pressure were placed into control, the performance of the LPMEOH™ Demonstration Unit was determined to have returned to the condition prior to the shutdown.

D.4 Planning and Administration

A 15-month, no-cost time extension (from 31 December 2001 to 31 March 2003) to the Cooperative Agreement, was approved by the DOE on 24 April 2000, and was accepted by Air Products on behalf of the Partnership on 08 May 2000. This extension is necessary to complete some of the key tests which were originally defined in the September 1996 Demonstration Test Plan, and to allow the opportunity to perform new tests of significant commercial interest. DOE approved an update to the Demonstration Test Plan which reflects these changes, and those pages which were impacted by this change were reissued.

A DOE quarterly review meeting was held during the week of 22 January 2001 at the Kingsport site. The performance of the LPMEOH™ Demonstration Facility since the last meeting (June 2000) was the primary topic of discussion.

The Milestone Schedule Status Report and the Cost Management Report, through the period ending 31 March 2001, are included in Appendix D. 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 March 2001. Seventy-two percent (72%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 March 2001.

The monthly reports for January, February, and March 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.
- Complete the biennial state code inspection of equipment at the LPMEOH™ Demonstration Unit, and install the piping and instrumentation systems required to perform the pre-treatment of the adsorbent in the 29C-40 catalyst guard bed and the in-situ activation of methanol synthesis catalyst in the LPMEOH™ Reactor.
- After the outage, continue executing Phase 3, Task 2.1 Methanol Operation per the Demonstration Test Plan. Focus activities on increasing catalyst activity, monitoring the performance of the gas sparger in the reactor, and execute the procedure to pre-treat the adsorbent in the 29C-40 catalyst guard bed.
- Develop the procedure and schedule for the in-situ activation of methanol synthesis catalyst in the LPMEOH™ Reactor.
- Schedule a Project Review Meeting with DOE.

F. Conclusion

The LPMEOH™ Demonstration Unit operated at 99.76% availability during this quarter. There was a single syngas outage experienced on 03 February 2001 (63 hours). There were also six short duration forced trips during the month for a total of 4.7 hours. The trips were caused by leakage of boiler feed water through the flow control valve which feeds the steam drum. This resulted in a high steam drum level which initiated a shutdown of the recycle compressor which tripped the plant. The boiler feed water valve will be repaired during the biennial outage which began on 28 March 2001. During this outage, a scheduled inspection of all pressure vessels as required by Tennessee state code will be performed. Some of the key activities include the inspection of reactor and economizer heat exchanger and completion of the engineering modifications required for the reduction of the adsorbent in the catalyst guard bed and for the in-situ activation of methanol synthesis catalyst in the LPMEOH™ Reactor.

A major catalyst withdrawal and addition campaign was undertaken during January and February of 2001 to raise catalyst activity. A series of three withdrawals were conducted on 25 and 26 January 2001. This was followed by four catalyst additions which were activated and added between 28 January and 07 February 2001. After the addition of the fourth batch of catalyst, the total catalyst inventory was calculated to be 47,160 pounds.

The syngas outage that occurred on 03 February 2001 also interrupted the completion of the activation of the final catalyst batch, as CO Gas, which is used in the activation procedure, was not available to the 29C-30 catalyst reduction vessel. As soon as CO Gas became available on 05 February 2001, the program for activation was restarted at the point of the interruption. Analysis of the data showed that the uptake of CO, which indicates the extent of reduction, was acceptable for this catalyst batch.

During most of the quarter, the flowrate of Balanced Gas was controlled at an average flowrate of 775 KSCFH. During these operating periods, the reactor pressure was set an average of 710 psig and temperature was maintained at 235°C.

There were two extended periods of operation (minimum of about 2 weeks) at a reactor temperature of 235°C during which catalyst activity was measured to track catalyst deactivation during the quarter. During both periods, the deactivation rate was achieved with the 29C-40 catalyst guard bed bypassed. A catalyst deactivation rate of 0.39% per day was calculated for the period 27 November 2000 to 16 January 2001 (51 days). A catalyst deactivation rate of 0.47% per day was calculated for the period 08 February 2001 to 02 March 2001 (23 days). These deactivation rates compare favorably with results over the past 12 months where the calculated deactivation rate has varied from 0.6 to 0.8% per day. These deactivation results are also similar to the baseline deactivation rate of 0.4% per day from the 4-month proof-of-concept run at the LaPorte AFDU in 1988/89 (this run was performed at 250°C), and may reflect the impact of poisons on catalyst aging.

Analyses of catalyst samples for changes in physical characteristics and levels of poisons have continued. The concentration of arsenic, which has been demonstrated in the laboratory to act as a poison to methanol synthesis catalyst, appeared to have stabilized during most of the periods of improved deactivation noted above. During the same periods,

iron concentration was also low and steady. However, sulfur, another known catalyst poison, was still increasing. The reduction in the rate of increase of arsenic and iron on the catalyst may have contributed to the improvement in the rate of catalyst deactivation for those time periods. Copper crystallite size measurements have not continued to increase in the most recent samples. Levels of nickel (a known catalyst poison) have remained low and steady since the restart in December of 1997. Any of these measurements will stabilize when the net rate of accumulation of these species on the catalyst is constant, indicating no large changes in either the concentration in the gas phase or the catalyst withdrawal rate from the LPMEOH™ Reactor.

The performance of the gas sparger, which was designed by Air Products and installed into the LPMEOH™ Reactor prior to the restart of the LPMEOH™ Demonstration Unit in March of 1999, was monitored. The performance to date has met the design expectations for pressure drop and reactor operation.

The first two tests of the ability of the LPMEOH™ Reactor to operate in a load-following environment were performed during the week of 15 January 2001. For two consecutive days, the reactor was taken offline for 12 hours, and then Balanced Gas was introduced to the process as quickly as possible (within the limitations of other systems at the Eastman chemicals-from-coal complex) from a standby condition. The attempts to perform these ramping tests were hampered by the standby conditions which were selected, including the lack of flow to the reactor (which lowered the reactor heat transfer coefficient) and the reactor temperature (180°C). Once Balanced Gas flowrate was reestablished and reactor temperature and pressure were placed into control, the performance of the LPMEOH™ Demonstration Facility was determined to have returned to the condition prior to the shutdown. In particular, the flowrate of syngas to the gas sparger in the LPMEOH™ Reactor was stopped during most of the downtime; although there was evidence of settling of catalyst, the sparger was able to remix the slurry, and the sparger resistance coefficient returned to its pre-outage level.

Three additional ramping tests were performed on 04 March 2001, 06 March 2001, and 13 March 2001, respectively. For the first two tests, the reactor was taken offline for 7-12 hours, and then the plant was restarted to obtain additional on/off performance data. For these tests, the reactor standby temperature was 216°C and 212°C, respectively; from this starting point, a reasonable ramping rate could not be reached. During the third test on 13 March 2001, the reactor was taken offline for 9.6 hours. A standby reactor temperature of 227°C was selected for this test; for all of the tests performed in March of 2001, gas flow from the recycle compressor was maintained to the LPMEOH™ Reactor. Balanced Gas was then introduced to the process as quickly as possible (within the limitations of other systems at the Eastman chemicals-from-coal complex) from a standby condition. A maximum ramp rate of 5.7% full load per minute and an average ramp rate of 4.2% full load per minute were achieved during this testing. This ramp performance approached the minimum target ramp rate of 5% full load per minute. For each test, once Balanced Gas flowrate was reestablished and reactor temperature and pressure were placed into control, the performance of the LPMEOH™ Demonstration Facility was determined to have returned to the condition prior to the shutdown.

During the reporting period, a total of 5,951,274 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, about 76.4 million gallons of methanol has been produced. Eastman accepted all of this methanol for use in the production of methyl acetate, and ultimately cellulose acetate and acetic acid. No safety or environmental incidents were reported during this quarter.

During this quarter, planning, procurement, and test operations were concluded on the project sites selected for the off-site, product-use test program. During the reporting period, Air Products approved final reports for two of the seven projects which tested stabilized methanol from the LPMEOH™ Demonstration Project. One project (at West Virginia University) measured the emissions from a low NO_x microturbine in a distributed power generation application; the second project (at the University of Florida) studied the acceptability of using stabilized methanol as a fuel to phosphoric acid fuel cell power systems. All of the final reports have now been approved by Air Products. Sponsorship (in the form of supply of stabilized methanol) has been provided to the Florida Institute of Technology as part of their contract with the Florida Energy Office to test methanol as a transportation fuel; this sponsorship was concluded during this quarter.

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

During this reporting period, DOE approved an update to the Demonstration Test Plan which defines the tests which are planned for the remainder of the operating program, and those pages which were impacted by this change were reissued.

A DOE quarterly review meeting was held during the week of 22 January 2001 at the Kingsport site. The performance of the LPMEOH™ Demonstration Facility since the last meeting (June 2000) was the primary topic of discussion.

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 March 2001. Seventy-two percent (72%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 March 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 -
January/March 2001**

Table 2 - Summary of Catalyst Samples - Second Catalyst Batch

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

Figure 2 - Sparger Resistance Coefficient vs. Days Onstream

Table 1
Summary of LPMEOH™ Demonstration Unit Outages - January/March 2001

Operation Start	Operation End	Operating Hours	Shutdown Hours	Reason for Shutdown
1/1/01 00:00	2/3/01 17:30	809.5	63.0	Syngas Outage
2/6/01 08:31	3/10/01 12:07	771.6	0.5	ESD Steam Drum Level
3/10/01 12:37	3/10/01 14:51	2.2	0.1	ESD Steam Drum Level
3/10/01 14:59	3/14/01 05:05	86.1	0.3	ESD Steam Drum Level
3/14/01 05:23	3/14/01 12:17	6.9	0.5	ESD Steam Drum Level
3/14/01 12:50	3/14/01 21:10	8.3	1.1	ESD Steam Drum Level
3/14/01 22:14	3/25/01 13:40	255.4	2.2	ESD Steam Drum Level
3/25/01 15:52	3/28/01 07:30	63.6	88.5	Shutdown for Scheduled Outage
3/31/01 23:59	3/31/01 23:59	0.0		End of Reporting Period
Total Operating Hours			2003.7	
Syngas Available Hours			2008.5	
Plant Availability, %			99.76	

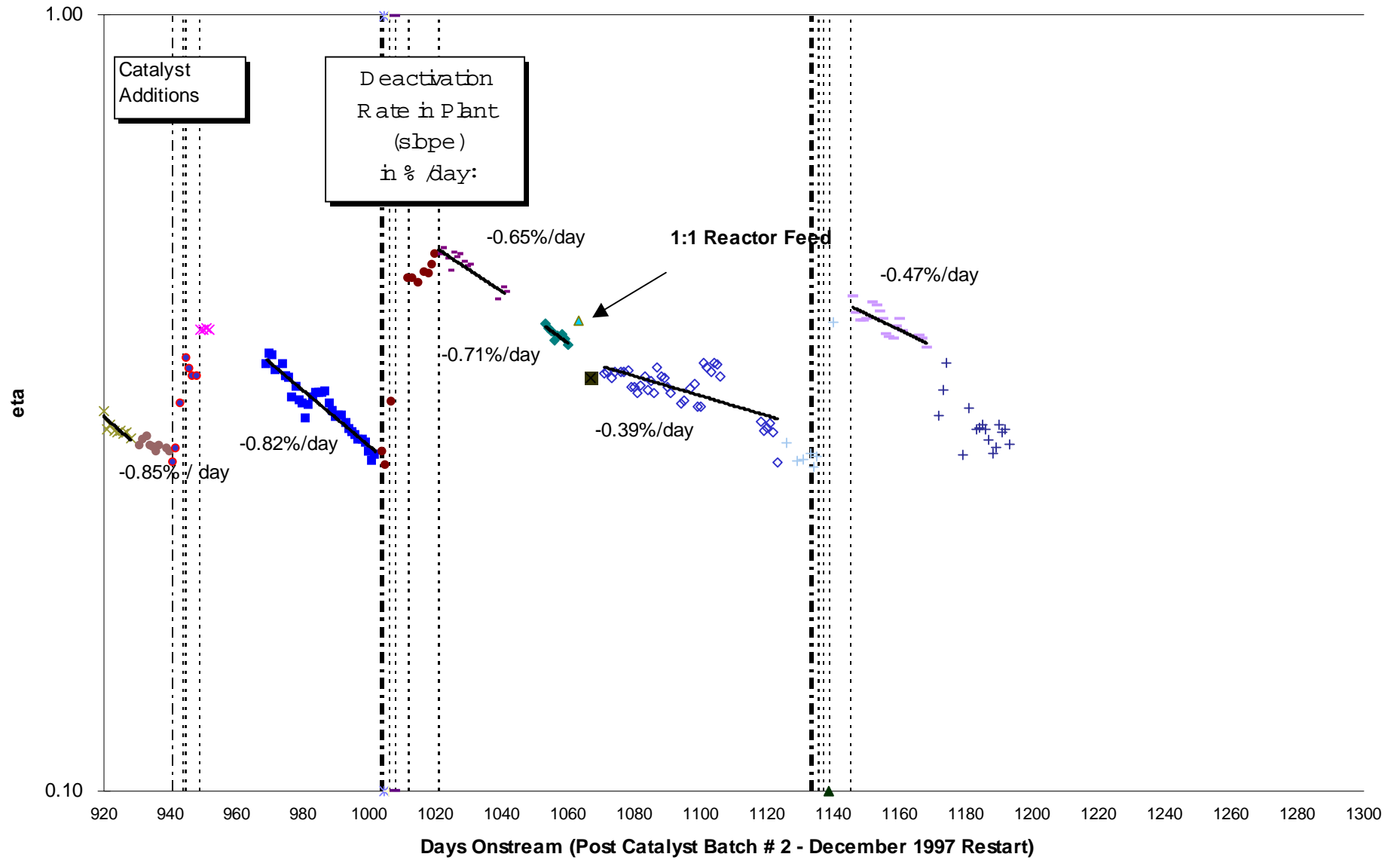
Table 2
Summary of Catalyst Samples - Second Catalyst Batch

Sample	Identity	XRD		BET	Analytical (ppmw)				
		Cu	ZnO	m ² /g	Fe	Ni	S	As	Cl
K9804-1	Reduction Sample 4/2/98 - Alternative Catalyst	72.5	84.9	105	23	11	<=110	<=12	
K9712-1	Transfer sample from 29D-02 to Reactor	95.3	74		362	47.2	66.7	10.2	nd
K9712-2	Reactor Sample Day 1	100	123.8	75	92.1	<=18	<=167	<50	nd
K9712-3	Reactor Sample Day 4	130.9	64						
K9712-4	Reactor Sample Day 10	126.8	73.3	73	126	<=22	<=127	<50	nd
K9801-2	Reactor Sample 1/26/98	132.05	98.3		63.5	39.5	42.7	29.2	<100
K9802-1	Reactor Sample 2/3/98	141.1	91.5						
K9802-2	Reactor Sample 2/9/98	158.1	113						
K9802-3	Reactor Sample 2/15/98	145.7	91		67.1	36	<=97	209	
K9802-4	Reactor Sample 2/23/98	176.8	114.5						
K9803-2	Reactor Sample 3/10/1998	154.3	95.8	44	61.4	35.8	<=94	408	
K9803-4	Reactor Sample 3/29/98	169.6	87.9						
K9804-2	Reactor Sample 4/14/98	152.4	89.3		81.7	30.8	<=170	615	
K9805-2	Reactor Sample 5/11/98	219.2	109.6		73.15	35.85	163	538	
K9606-2	Reactor Sample 6/16/98	272.3	117.2		86.4	31.1	220	1110	
K9807-2	Reactor Sample 7/8/98	263.2	108.6		88.7	27.6	277	1045	
K9807-3	Reactor Sample 7/29/98	412*	112		93.25	30.95	209	1620	
K9807-4	Reactor Sample 8/14/98	353.9*	124		121.5	37.1	213.5	1215	
K9809-1	Reactor Sample 9/24/98	347.4	129.8		69.6	29.8	326	1149	
K9810-1	Reactor Sample 10/5/98	331.1	130.4						
K9811-2	Reactor Sample 11/25/98	293.9			57.3	23.4	264	1400	<100
K9812-1	Reactor Sample 12/29/98	283.1			72.3	20.4	260	1300	<100
K9901-1	Reactor Sample 1/15/99	252.5	61.4						
K9902-1	Reactor Sample 2/17/99	474.7	133.6		82.6	22.2	385	1490	<300
K9904-3	Reactor Sample 4/27/99	417.8	110.4	15	131	18.2	348	1460	<30
K9906-1	Reactor Sample 6/1/99	517	105	43	109	19.7	316	1680	40
K9907-1	Reactor Sample 7/13/99	446	116	59	175	19.7	488	1810	30
K9908-2	Reactor Sample 8/31/99	632	117	56	161	15.1	406	1470	50
K9909-2	Reactor Sample 9/21/99	357	109	64	132	11.2	253	1050	nd
K9910-2	Reactor Sample 10/19/99	135	94	55	157	15.4	343	1270	30
K9911-1	Reactor Sample 11/4/99				184	12.8	335	1580	na
K9912-1	Reactor Sample 12/8/99	797	121	60	167	13.9	248	1400	40
K0001-1	Reactor Sample 1/5/00	613	105	63	199	10.8	292	1190	nd
K0001-2	Reactor Sample 1/19/00				205	10.0	432	1250	na
K0003-1	Reactor Sample 3/2/00	187	88.7	67	137	8.2	226	1010	30
K004-1	Reactor Sample 4/23/00	175	114.5	59	164	6.6	248	1240	20
K0007-1	Reactor Sample 7/18/00	174	107.5	69	166	< 9.6	349	1270	30
K0008-1	Reactor Sample 8/31/00	385	90.4	66	186	< 10	379	1080	50
K0009-3	Reactor Sample 9/19/00	157	96	67	145	< 10	273	1390	nd
K0011-1	Reactor Sample 11/7/00	248	79.6	70	120	< 10	237	1490	nd
K0011-2	Reactor Sample 11/27/00	263	109.2		128	< 10	258	1470	20
K0012-2	Reactor Sample 12/19/01				100	< 10	410	1480	
K0101-1	Reactor Sample 1/3/01	280	110	66	166	< 10	355	1980	30
K0102-1	Reactor Sample 2/7/01	172	104	73	121	< 10	375	1750	40
K0103-1	Reactor Sample 3/7/01	164	138		90	< 10	416	1830	20

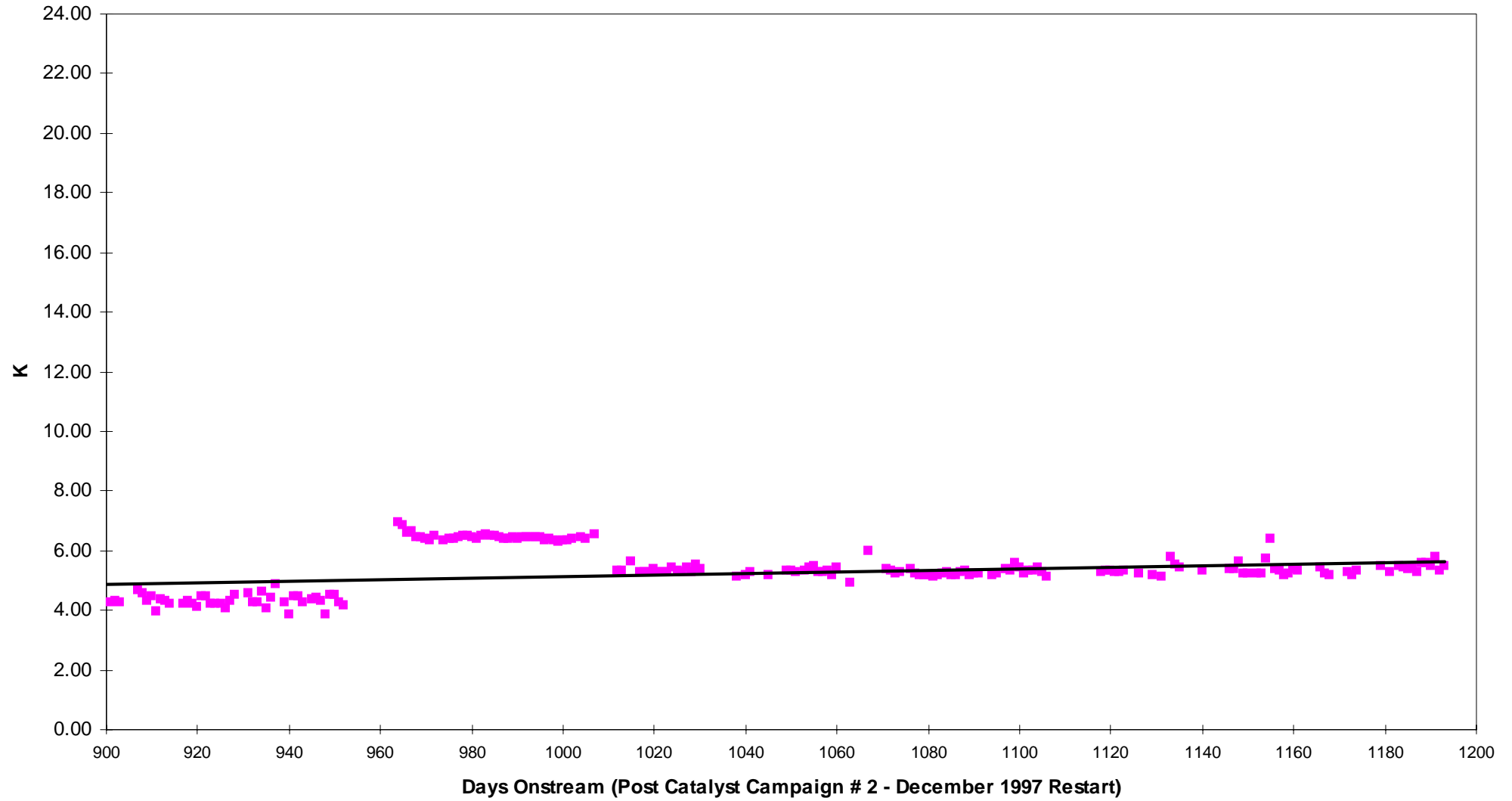
Notes:

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

**Figure 3 - Kingsport LPMEOH™ Catalyst Age (eta):
June 2000 - March 2001**



**Figure 2 - Kingsport LPMEOH™
Sparger Resistance Coefficient**



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