

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

TECHNICAL PROGRESS REPORT NO. 32

For The Period

1 April – 30 June 2002

Prepared by

**Air Products and Chemicals, Inc.
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 100% availability during this quarter. (Availability is defined as the percentage of time that the LPMEOH™ Demonstration Unit was available to operate, with the exclusion of schedule outages.) There were three syngas interruptions that were experienced on 23 May 2002 (37.9 hours duration), 27 May 2002 (7.6 hours duration), and 04 June 2002 (16.2 hours duration). A major activity during the quarter involved the preparation for and execution of the steps to perform the in-situ activation of methanol synthesis catalyst within the 29C-01 LPMEOH™ Reactor.

At the conclusion of the catalyst withdrawal/addition catalyst campaign completed on 01 April 2002, the LPMEOH™ Demonstration Unit was operated at a constant reactor temperature of 235°C and a reactor pressure of 700 psig. The flowrate of the primary syngas feed (Balanced Gas) was controlled at an average value of 590 KSCFH during this time.

A catalyst deactivation rate of 0.69% per day was calculated for the period 01 April 2002 to 18 May 2002 (48 days). This is similar to the results that have been generally calculated over the past 2 years (averaging between 0.6% and 0.7% per day). As a basis of comparison, the calculated deactivation rate from the 4-month proof-of-concept run at the LaPorte Alternative Fuels Development Unit (AFDU) in 1988/89 was 0.4% per day. (This run was performed on a syngas rich in carbon monoxide (CO) derived from natural gas at a reactor temperature of 250°C.)

Analyses of catalyst samples to determine changes in physical characteristics and levels of poisons have continued. Chemical analysis of catalyst samples has indicated the presence of all expected crystal phases. This analysis has indicated a significant increase in iron on the catalyst since the start of this current catalyst campaign.

Several activities were conducted during the quarter in an effort to determine the source of the iron contamination on the catalyst. Gas sampling and analysis that was conducted during the week of 22 April 2002 indicated that the concentration of iron in the Balanced Gas matched the historical levels of 10 parts per billion by volume (ppbv) or less.

To further investigate the potential sources of iron, the LPMEOH™ Demonstration Unit was shutdown on 05 June 2002. The slurry contents of the reactor were cooled and transferred in batches to the 29C-30 catalyst reduction vessel for final cooling and draining into drums. A total of 317 drums or 90,484 pounds of slurry were removed from the reactor. The reactor

was then inspected during the week of 10 June 2002, just prior to the start of the second in-situ catalyst activation procedure. Carbon steel components were found to have been installed during the biennial inspection of pressure vessels in April of 2001; these were replaced with the correct stainless steel items. A visual inspection of the reactor did not indicate damage to the internal heat exchanger or vessel wall, which could have been additional sources of iron.

Additionally, a hot function test with Balanced Gas and mineral oil (in the absence of catalyst) was performed during the week of 17 June 2002. During this procedure, sampling of the reactor feed gas stream was performed; during the three-day period, the measured levels of iron carbonyl into the LPMEOH™ Reactor dropped gradually from over 200 ppbv to approximately 30 ppbv. Based upon this decline in concentration, preparations were made to initiate the procedure for the in-situ activation of methanol synthesis catalyst within the LPMEOH™ Reactor.

As noted in Technical Progress Report No. 31, a fresh quantity of adsorbent (activated carbon impregnated with copper oxide) was charged to the 29C-40 catalyst guard bed on 21 February 2002. This material was chemically reduced using dilute syngas in nitrogen. (In this case, reduction refers to the reaction of the copper oxide with a reductant such as CO or hydrogen (H₂) to copper metal and either carbon dioxide (CO₂) or water (H₂O) in a temperature controlled manner). The catalyst guard bed was placed into service on 08 March 2002.

During the period of 26 March 2002 to 24 April 2002, gas sampling was performed to assess the performance of the adsorbent. The analytical techniques involved analyzing the catalyst guard bed inlet and outlet streams for arsenic (reported as arsine) using standard techniques. Initial performance was acceptable, as the average concentration of arsine across the guard bed was reduced from an inlet value of approximately 50 ppbv to the detection limit of 2 ppbv. However, towards the end of the sampling period, the average concentration of arsine at the outlet of the catalyst guard bed was determined to be approximately 10 ppbv, which indicated that the performance of the adsorbent was beginning to degrade.

Prior to the start of the second in-situ catalyst activation procedure, the adsorbent in the catalyst guard bed was again replaced with fresh material. Fresh adsorbent in the catalyst guard bed would limit the amount of contaminants (in particular arsine, sulfur, and iron) that could enter the LPMEOH™ Reactor with the fresh feed syngas.

The catalyst guard bed reduction procedure was started at 1700 hrs on 20 June 2002. During this reduction procedure, a dilute stream of Balanced Gas in nitrogen was preheated and used as the reductant. The temperature control during the procedure was excellent, and the reduction was completed at 1700 hrs on 22 June 2002.

After cooling with nitrogen, a pressure check to full supply pressure with Balanced Gas was conducted. The adsorbent temperature increased by about 28°C and stabilized during this examination. This observation was consistent with the temperature increase which would be expected from the adsorption of CO on the adsorbent surface.

As reported in Technical Progress Report No. 31, an assessment of the performance of the methanol synthesis catalyst following the execution of the in-situ activation procedure in August of 2001 was completed. The in-situ activation procedure used the LPMEOH™ Reactor (instead of the catalyst reduction vessel) to prepare fresh methanol synthesis catalyst for production of methanol by reacting metal oxides (for example, copper oxide) with a reductant such as CO or H₂ to produce the base metal plus either CO₂ or H₂O. It was found that storage of the fresh catalyst in the presence of mineral oil at elevated temperatures prior to reduction was the cause of the lower than expected catalyst activity. The operating steps in the procedure were modified so that the fresh methanol catalyst would not be exposed to temperatures in excess of 100°C prior to the introduction of dilute syngas.

Beginning on 21 June 2002, batches of fresh catalyst were mixed with mineral oil in the catalyst reduction vessel, heated to 100°C, and transferred, without being reduced or activated, to the reactor. Since the 29D-02 slurry tank was not used to collect the batches during the three days of catalyst loading, the process was greatly simplified compared with the first in-situ activation procedure in August of 2001. A total of eleven batches of fresh catalyst slurry, each containing about 3,700 pounds of fresh catalyst, was prepared in this manner. After the final transfer, the total amount of fresh catalyst in the reactor was 40,040 pounds, which is approximately the design catalyst inventory for the LPMEOH™ Demonstration Unit. Nitrogen was introduced in the bottom of the reactor during the entire loading procedure, and the unreduced catalyst in the reactor was maintained at 100°C by adding steam as necessary to the 29C-02 steam drum and internal heat exchanger. The catalyst loading and transfer operation was completed at 1725 hours on 24 June 2002.

The reactor was cooled overnight to the desired initial temperature for catalyst activation. The 29K-01 recycle compressor was started to assist in cooling the reactor contents. By 0800 hours on 25 June 2002, the reactor inlet flow was set at approximately 300 KSCFH, and 52 KSCFH of make-up nitrogen was introduced at the compressor suction. Reactor pressure was set at 85 psig. Cooling was completed after the reactor temperature reached 86°C.

After reactor cooling was completed, the in-situ catalyst activation procedure was started at 1200 hrs on 25 June 2002. In addition to the 52 KSCFH of nitrogen, a small quantity of Balanced Gas was introduced into the discharge of the recycle compressor to ensure that sufficient reductant was present at all times in the reactor inlet. The in-situ catalyst activation procedure ended at 0200 hrs on 27 June 2002.

The progress of the in-situ catalyst activation procedure was tracked by evaluating the uptake of reductant by the catalyst. A preliminary uptake of 98% of the theoretical value was calculated based upon analytical data and measured flowrates, which is an indication that the methanol synthesis catalyst was properly activated during the in-situ catalyst activation procedure.

The LPMEOH™ Demonstration Unit was then re-started at 1500 hrs on 27 June 2002. Preliminary heat and material balances indicate that the activity of the catalyst was excellent and similar to the results that have been achieved with catalyst that has been properly activated in the catalyst reduction vessel. These initial results also show an improvement

when compared with the performance of the methanol synthesis catalyst following the first in-situ catalyst activation procedure in August of 2001 (when the uptake of reductant was 70% of the theoretical value and the initial activity was 60 to 70% of the value for properly activated catalyst).

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. During the preparations for the second in-situ activation of methanol synthesis catalyst, the sparger was inspected and cleaned. The performance of the sparger will continue to be monitored closely for any changes.

During the reporting period, a total of 3,328,693 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, about 95.2 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.

Activities associated with Design Verification Testing (DVT) of the Liquid Phase Dimethyl Ether (LPDME) Process have been completed. A Topical Report, which provides the status of the current market for DME and an outlook on potential market developments through 2006, was approved by DOE and issued.

A formal recommendation to DOE to perform a second test of the in-situ catalyst activation procedure was issued on 15 May 2002. DOE accepted the recommendation in a letter to Air Products dated 24 May 2002.

Comments were received from DOE on a draft report which provides publicly available technical data on the Eastman chemicals-from-coal complex in Kingsport. This report provides operational performance of the chemicals-from-coal complex in Kingsport as well as specific data on the major feed and effluent streams for the coal gasification and syngas cleanup systems.

One hundred percent (100%) of the \$38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 30 June 2002. Ninety-four percent (94%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 June 2002.

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ACRONYMS AND DEFINITIONS

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

ACRONYMS AND DEFINITIONS (cont'd)

NEPA	-	National Environmental Policy Act
OSHA	-	Occupational Safety and Health Administration
ρ	-	density, pounds per cubic foot
Partnership	-	Air Products Liquid Phase Conversion Company, L.P.
PDU	-	Process Development Unit
PFD	-	Process Flow Diagram(s)
ppbv	-	parts per billion (volume basis)
ppmw	-	parts per million (weight basis)
Project	-	Production of Methanol/DME Using the LPMEOH™ Process at an Integrated Coal Gasification Facility
psi	-	pounds per square inch
psia	-	pounds per square inch (absolute)
psig	-	pounds per square inch (gauge)
P&ID	-	Piping and Instrumentation Diagram(s)
Raw Methanol	-	sum of Refined Grade Methanol and Crude Grade Methanol; represents total methanol which is produced after stabilization
Reactor Feed	-	sum of Fresh Feed and Recycle Gas
Reactor O-T-M Conversion	-	percentage of energy (on a lower heating value basis) in the Reactor Feed converted to methanol (Once-Through-Methanol basis)
Reactor Volumetric Productivity	-	the quantity of Raw Methanol produced (tons per day) per cubic foot of reactor volume up to the Gassed Slurry Level
Recycle Gas	-	the portion of unreacted syngas effluent from the reactor “recycled” as a feed gas
Refined Grade Methanol	-	Distilled methanol, defined as 99.8 wt% minimum purity; used directly in downstream Eastman processes
SCF	-	Standard Cubic Feet
SCFH	-	Standard Cubic Feet per Hour
Slurry Concentration	-	percentage of weight of slurry (solid plus liquid) which is catalyst (on an oxide basis)
Sl/hr-kg	-	Standard Liter(s) per Hour per Kilogram of Catalyst
Syngas	-	Abbreviation for Synthesis Gas
Syngas Utilization	-	defined as the number of standard cubic feet of Balanced Gas plus CO Gas to the LPMEOH™ Demonstration Unit required to produce one pound of Raw Methanol
Synthesis Gas	-	A gas containing primarily hydrogen (H ₂) and carbon monoxide (CO), or mixtures of H ₂ and CO; intended for "synthesis" in a reactor to form methanol and/or other hydrocarbons (synthesis gas may also contain CO ₂ , water, and other gases)
Tie-in(s)	-	the interconnection(s) between the LPMEOH™ Process Demonstration Unit and the Eastman Facility
TPD	-	Ton(s) per Day
V	-	volumetric flowrate, thousand standard cubic feet per hour
VOC	-	volatile organic compound
vol%	-	volume percent
WBS	-	Work Breakdown Structure
wt	-	weight

Executive Summary

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

The LPMEOH™ Demonstration Unit operated at 100% availability during this quarter. (Availability is defined as the percentage of time that the LPMEOH™ Demonstration Unit was available to operate, with the exclusion of schedule outages.) There were three syngas interruptions that were experienced on 23 May 2002 (37.9 hours duration), 27 May 2002 (7.6 hours duration), and 04 June 2002 (16.2 hours duration). A major activity during the quarter involved the preparation for and execution of the steps to perform the in-situ activation of methanol synthesis catalyst within the 29C-01 LPMEOH™ Reactor.

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After reactor cooling was completed, the in-situ catalyst activation procedure was started at 1200 hrs on 25 June 2002. In addition to the 52 KSCFH of nitrogen, a small quantity of Balanced Gas was introduced into the discharge of the recycle compressor to ensure that sufficient reductant was present at all times in the reactor inlet. The in-situ catalyst activation procedure ended at 0200 hrs on 27 June 2002.

The progress of the in-situ catalyst activation procedure was tracked by evaluating the uptake of reductant by the catalyst. A preliminary uptake of 98% of the theoretical value was calculated based upon analytical data and measured flowrates, which is an indication that the methanol synthesis catalyst was properly activated during the in-situ catalyst activation procedure.

The LPMEOH™ Demonstration Unit was then re-started at 1500 hrs on 27 June 2002. Preliminary heat and material balances indicate that the activity of the catalyst was excellent and similar to the results that have been achieved with catalyst that has been properly activated in the catalyst reduction vessel. These initial results also show an improvement when compared with the performance of the methanol synthesis catalyst following the first in-situ catalyst activation procedure in August of 2001 (when the uptake of reductant was 70% of the theoretical value and the initial activity was 60 to 70% of the value for properly activated catalyst).

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. During the preparations for the second in-situ activation of methanol synthesis catalyst, the sparger was inspected and cleaned. The performance of the sparger will continue to be monitored closely for any changes.

During the reporting period, a total of 3,328,693 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, about 95.2 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.

Activities associated with Design Verification Testing (DVT) of the Liquid Phase Dimethyl Ether (LPDME) Process have been completed. A Topical Report, which provides the status

of the current market for DME and an outlook on potential market developments through 2006, was approved by DOE and issued.

A formal recommendation to DOE to perform a second test of the in-situ catalyst activation procedure was issued on 15 May 2002. DOE accepted the recommendation in a letter to Air Products dated 24 May 2002.

Comments were received from DOE on a draft report which provides publicly available technical data on the Eastman chemicals-from-coal complex in Kingsport. This report provides operational performance of the chemicals-from-coal complex in Kingsport as well as specific data on the major feed and effluent streams for the coal gasification and syngas cleanup systems.

One hundred percent (100%) of the \$38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 30 June 2002. Ninety-four percent (94%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 June 2002.

A. Introduction

The Liquid Phase Methanol (LPMEOH™) demonstration project at Kingsport, Tennessee, is a \$213.7 million cooperative agreement between the U.S. Department of Energy (DOE) and Air Products Liquid Phase Conversion Company, L. P. (the Partnership). Air Products and Chemicals, Inc. (Air Products) and Eastman Chemical Company (Eastman) formed the Partnership to execute the Demonstration Project. A demonstration unit producing 80,000 gallons per day (260 TPD) of methanol was designed, constructed, and is operating at a site located at the Eastman chemicals-from-coal complex in Kingsport. The Partnership will own and operate the facility for the demonstration period.

This project is sponsored under the DOE's Clean Coal Technology Program, and its primary objective is to “demonstrate the production of methanol using the LPMEOH™ Process in conjunction with an integrated coal gasification facility.” The project has been demonstrating the suitability of the methanol produced for use as a chemical feedstock or as a low-sulfur dioxide, low-nitrogen oxides alternative fuel in stationary and transportation applications. The project has also evaluated the demonstration of the production of dimethyl ether (DME) as a mixed coproduct with methanol.

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

B. Project Description

The demonstration unit, which occupies an area of 0.6 acre, is integrated into the existing 4,000-acre Eastman complex located in Kingsport, Tennessee. The Eastman complex

employs approximately 8,600 people. In 1983, Eastman constructed a coal gasification facility utilizing Texaco technology. The synthesis gas (syngas) generated by this gasification facility is used to produce carbon monoxide and methanol. Both of these products are used to produce methyl acetate and ultimately cellulose acetate and acetic acid. The availability of this highly reliable coal gasification facility was the major factor in selecting this location for the LPMEOH™ Process Demonstration. Three different feed gas streams (hydrogen gas or H₂ Gas, carbon monoxide gas or CO Gas, and the primary syngas feed known as Balanced Gas) are diverted from existing operations to the LPMEOH™ Demonstration Unit, thus providing the range of coal-derived syngas ratios (hydrogen to carbon monoxide) needed to meet the technical objectives of the demonstration project.

For descriptive purposes and for design and construction scheduling, the project has been divided into four major process areas with their associated equipment:

- *Reaction Area* - Syngas preparation and methanol synthesis reaction equipment.
- *Purification Area* - Product separation and purification equipment.
- *Catalyst Preparation Area* - Catalyst and slurry preparation and disposal equipment.
- *Storage/Utility Area* - Methanol product, slurry, and oil storage equipment.

The physical appearance of this facility closely resembles the adjacent Eastman process plants, including process equipment in steel structures.

- *Reaction Area*

The reaction area includes feed gas compressors, catalyst guard beds, the reactor, a steam drum, separators, heat exchangers, and pumps. The equipment is supported by a matrix of structural steel. The most salient feature is the reactor, since with supports, it is approximately 84-feet tall.

- *Purification Area*

The purification area features two distillation columns with supports; one is approximately 82-feet tall, and the other 97-feet tall. These vessels resemble the columns of the surrounding process areas. In addition to the columns, this area includes the associated reboilers, condensers, air coolers, separators, and pumps.

- *Catalyst Preparation Area*

The catalyst preparation area consists of a building with a roof and partial walls, in which the catalyst preparation vessels, slurry handling equipment, and spent slurry disposal equipment are housed. In addition, a hot oil utility system is included in the area.

- *Storage/Utility Area*

The storage/utility area includes two diked lot-tanks for methanol, two tanks for oil storage, a slurry holdup tank, a trailer loading/unloading area, and an underground oil/water separator. A vent stack for safety relief devices is located in this area.

C. Process Description

The LPMEOH™ Demonstration Unit is integrated with Eastman's coal gasification facility. A simplified process flow diagram is included in Appendix A. Syngas is introduced into the slurry reactor, which contains a slurry of liquid mineral oil with suspended solid particles of catalyst. The syngas dissolves through the mineral oil, contacts the catalyst, and reacts to form methanol. The heat of reaction is absorbed by the slurry and is removed from the slurry by steam coils. The methanol vapor leaves the reactor, is condensed to a liquid, sent to the distillation columns for removal of higher alcohols, water, and other impurities, and is then stored in the day tanks for sampling before being sent to Eastman's methanol storage. Most of the unreacted syngas is recycled back to the reactor with the syngas recycle compressor, improving cycle efficiency. The methanol will be used for downstream feedstocks and has been used in off-site, product-use testing to determine its suitability as a transportation fuel and as a fuel for stationary applications in the power industry.

D. Results and Discussion

The project status is reported by task, covering those areas in which activity took place during the reporting period. Major accomplishments during this period are as follows:

D.1 Off-Site Testing (Product-Use Demonstration)

The LPMEOH™ Demonstration Project has completed the testing of stabilized methanol from both the LaPorte AFDU and the Kingsport LPMEOH™ Demonstration Unit in various off-site mobile and stationary applications. The product-use test program was developed to enhance the early commercial acceptance of central clean coal technology processing facilities, coproducing electricity and methanol to meet the needs of the local community. One of the advantages of the LPMEOH™ Process for coproduction from coal-derived syngas is that the as-produced, stabilized (degassed) methanol product is of unusually high quality (e.g. less than 1 wt% water) which may be suitable for the premium fuel applications. When compared to conventional methanol synthesis processes, cost savings (10 to 15%) of several cents per gallon of methanol can be achieved in coproduction facilities, if the suitability of the stabilized product can be demonstrated.

Product-use tests commenced during the first year of demonstration operations. An inventory of approximately 12,000 gallons of stabilized methanol was produced at LPMEOH™ Demonstration Unit in February 1998 to supply the needs of the product-use test program; due to the pre-1998 timing for certain tests, methanol was shipped from the inventory produced and held at the LaPorte AFDU. The stabilized methanol from the February 1998 production campaign has been stored in an offsite facility; during the reporting period, the unused stabilized methanol was returned to Eastman for further distillation prior to use within the chemicals-from-coal complex.

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

D.2 DME Design Verification Testing

The LPMEOHTM 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 LPMEOHTM 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 LPMEOHTM Process during the remaining time within the operating program; any improvement in the catalyst performance for the methanol synthesis catalyst will also yield benefits for the LPDME catalyst system.

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

A Topical Report, which provides the status of the current market for DME and an outlook on potential market developments through 2006, was approved by DOE and issued.

D.3 LPMEOHTM Process Demonstration Unit - Methanol Operation

Table D.3-1 contains the summary table of performance data for the LPMEOHTM 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 LPMEOHTM Demonstration Unit during the reporting period.

During the reporting period, a total of 3,328,693 gallons of methanol was produced at the LPMEOHTM 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 LPMEOHTM Demonstration Unit operated at 100% availability during this quarter. Appendix C, Table 1 contains the summary of outages for the LPMEOHTM Demonstration Unit during this quarter. There were three syngas interruptions that were experienced on 23 May 2002 (37.9 hours duration), 27 May 2002 (7.6 hours duration), and 04 June 2002 (16.2 hours duration). A major activity during the quarter involved the preparation for and execution of the steps to perform the in-situ activation of methanol synthesis catalyst within the 29C-01 LPMEOHTM Reactor.

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

Case	Date	Stream	Days On	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 (1/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)
2000-8	1-Apr-02	37347	Balanced	235	700	658	2,209	2.32	60.1	0.66	4018	39.0	23.8	50.0	40,904	0.305	24.4	19.2	40.8	193.5	12.33	0.092	120	7.07	6.50
2000-8	2-Apr-02	37348	Balanced	235	700	651	2,192	2.32	57.7	0.66	3970	37.2	19.9	52.0	40,904	0.295	24.0	18.9	40.9	190.9	11.97	0.087	115	7.20	6.53
2000-8	3-Apr-02	37349	Balanced	235	700	659	2,239	2.58	60.8	0.67	4121	39.4	25.9	50.5	40,904	0.308	26.6	19.4	40.5	195.4	12.45	0.092	116	7.74	6.79
2000-8	4-Apr-02	37350	Balanced	235	700	657	2,244	2.72	61.7	0.67	4098	40.3	24.6	48.0	40,904	0.312	27.7	19.4	41.3	191.2	12.19	0.095	112	7.44	6.51
2000-8	5-Apr-02	37351	Balanced	235	700	655	2,242	2.72	64.3	0.67	4098	38.9	20.9	48.5	40,904	0.308	27.4	19.2	41.5	189.7	12.10	0.093	106	7.47	6.58
2000-8	6-Apr-02	37352	Balanced	235	700	643	2,252	2.72	63.2	0.67	4098	38.9	20.9	48.5	40,904	0.301	26.9	18.9	41.4	186.3	11.88	0.092	103	7.47	6.58
2000-8	7-Apr-02	37353	Balanced	235	700	654	2,234	2.71	69.6	0.67	4075	40.1	23.2	47.5	40,904	0.306	27.2	19.1	41.7	188.4	12.02	0.095	108	7.28	6.48
2000-8	8-Apr-02	37354	Balanced	235	700	654	2,220	2.67	76.8	0.66	4047	39.7	20.3	46.5	40,904	0.300	26.8	18.9	42.1	186.2	11.88	0.095	104	7.17	6.43
2000-8	9-Apr-02	37355	Balanced	235	700	654	2,216	2.69	74.6	0.66	4047	41.5	26.4	47.0	40,904	0.305	27.4	19.3	41.8	187.8	11.98	0.095	111	7.44	6.61
2000-8	10-Apr-02	37356	Balanced	235	700	655	2,233	2.71	77.2	0.66	4051	40.7	24.8	47.5	40,904	0.299	27.0	19.0	42.3	185.7	11.85	0.093	107	7.45	6.66
2000-8	11-Apr-02	37357	Balanced	235	700	653	2,220	2.70	88.7	0.66	4029	40.6	22.9	46.5	40,904	0.291	26.4	18.5	43.2	181.4	11.58	0.093	104	7.68	6.93
2000-8	12-Apr-02	37358	Balanced	235	700	650	2,209	2.66	89.1	0.66	4012	40.9	22.2	45.5	40,904	0.291	26.3	18.6	43.1	180.8	11.54	0.095	105	7.71	6.93
2000-8	13-Apr-02	37359	Balanced	234	700	651	2,213	2.57	99.3	0.66	4014	39.5	17.0	45.0	40,904	0.289	25.3	18.4	43.6	179.2	11.43	0.095	103	7.36	6.50
2000-8	14-Apr-02	37360	Balanced	235	700	659	2,195	2.57	100.4	0.65	4002	42.7	21.2	42.0	40,904	0.293	25.7	18.7	43.7	180.9	11.54	0.103	115	7.39	6.51
2000-8	15-Apr-02	37361	Balanced	235	700	655	2,205	2.52	87.6	0.65	4007	40.7	18.9	44.0	40,904	0.303	26.0	19.1	42.6	184.7	11.79	0.100	114	7.53	6.52
2000-8	16-Apr-02	37362	Balanced	234	700	589	2,245	2.57	66.3	0.65	3972	41.0	17.8	43.0	40,904	0.279	24.7	17.8	41.6	169.9	10.84	0.094	108	7.55	6.61
2000-8	17-Apr-02	37363	Balanced	234	700	576	2,238	2.65	68.9	0.65	3955	41.2	16.6	42.0	40,904	0.265	24.2	17.2	42.2	163.6	10.44	0.093	105	7.12	6.44
2000-8	18-Apr-02	37364	Balanced	235	700	574	2,214	2.89	63.4	0.64	3913	40.1	16.8	44.0	40,904	0.268	26.2	17.4	42.1	163.6	10.44	0.089	107	6.83	6.45
2000-8	19-Apr-02	37365	Balanced	235	700	578	2,213	2.88	61.2	0.64	3914	39.6	20.0	46.5	40,904	0.279	27.1	18.0	41.1	168.7	10.76	0.087	109	6.85	6.44
2000-8	20-Apr-02	37366	Balanced	235	700	577	2,213	2.85	62.1	0.64	3915	39.3	16.3	45.0	40,904	0.272	26.4	17.7	41.6	166.7	10.63	0.088	106	6.77	6.40
2000-8	21-Apr-02	37367	Balanced	235	700	577	2,210	2.76	65.0	0.64	3923	40.6	17.4	43.5	40,904	0.270	25.6	17.6	41.7	165.9	10.59	0.091	108	6.86	6.37
2000-8	22-Apr-02	37368	Balanced	235	700	581	2,252	2.72	69.2	0.64	3982	42.0	20.1	43.5	40,904	0.263	24.5	20.1	43.5	164.8	10.52	0.090	106	7.04	6.51
2000-8	23-Apr-02	37369	Balanced	234	700	578	2,250	2.79	73.0	0.65	3974	40.5	16.3	43.0	40,904	0.258	24.5	16.9	42.7	162.5	10.37	0.090	106	6.75	6.44
2000-8	24-Apr-02	37370	Balanced	234	700	569	2,237	2.46	69.5	0.64	3935	40.0	14.7	43.0	40,904	0.255	24.8	16.8	42.8	159.5	10.17	0.090	106	6.57	6.39
2000-8	8-May-02	37384	Balanced	234	700	556	2,186	2.80	78.6	0.63	3858	40.3	14.6	42.5	40,904	0.243	24.0	16.5	43.4	153.7	9.81	0.086	110	6.46	6.44
2000-8	9-May-02	37385	Balanced	235	700	545	2,218	2.69	65.0	0.64	3895	41.4	19.1	43.0	40,904	0.241	23.2	16.4	42.4	154.2	9.84	0.086	109	6.68	6.40
2000-8	10-May-02	37386	Balanced	235	700	550	2,249	2.59	69.8	0.64	3935	39.7	15.5	44.0	40,904	0.239	22.2	16.2	42.9	154.1	9.83	0.084	103	6.76	6.33
2000-8	11-May-02	37387	Balanced	235	700	550	2,232	2.59	71.9	0.64	3910	41.1	16.3	42.0	40,904	0.241	22.5	16.3	42.6	154.9	9.89	0.088	112	6.76	6.39
2000-8	12-May-02	37388	Balanced	234	700	552	2,214	2.58	74.4	0.63	3882	40.0	14.5	43.0	40,904	0.242	22.4	16.3	42.8	154.6	9.87	0.086	107	6.58	6.37
2000-8	13-May-02	37389	Balanced	234	700	556	2,277	2.64	74.7	0.65	3982	41.6	17.8	42.0	40,904	0.235	21.7	15.6	43.7	152.9	9.75	0.087	105	6.73	6.54
2000-8	14-May-02	37390	Balanced	234	700	538	2,300	2.60	77.0	0.66	4026	40.7	13.9	41.5	40,904	0.224	20.3	14.8	43.7	147.5	9.41	0.085	97	6.70	6.46
2000-8	15-May-02	37391	Balanced	234	700	553	2,292	2.62	87.6	0.65	4010	40.0	10.5	41.0	40,904	0.228	20.6	14.9	44.5	148.9	9.51	0.087	101	6.57	6.46
2000-8	16-May-02	37392	Balanced	235	700	537	2,283	2.72	62.3	0.65	3992	39.4	14.6	44.0	40,904	0.235	22.1	14.6	42.3	152.5	9.73	0.083	101	6.59	6.49
2000-8	17-May-02	37393	Balanced	235	700	541	2,258	2.68	65.5	0.65	3953	42.0	18.0	41.5	40,904	0.235	22.2	15.7	42.6	152.2	9.71	0.088	110	6.67	6.48
2000-8	18-May-02	37394	Balanced	235	700	543	2,286	2.75	59.0	0.66	4012	40.1	21.4	46.5	40,904	0.237	22.6	15.8	41.9	155.5	9.91	0.080	101	6.69	6.60
2000-8	19-May-02	37395	Balanced	235	700	543	2,296	2.71	63.9	0.66	4032	39.2	16.0	45.0	40,904	0.233	21.8	16.0	42.3	153.8	9.81	0.082	100	6.57	6.48
2000-8	28-May-02	37404	Balanced	235	700	497	2,291	2.53	96.6	0.65	3964	42.1	20.3	42.5	40,904	0.188	17.5	13.0	46.5	128.2	8.19	0.072	81	7.03	6.86
2000-8	29-May-02	37405	Balanced	240	700	569	2,214	2.78	60.8	0.65	3917	42.7	22.8	43.0	40,904	0.229	24.5	17.2	41.7	163.8	10.45	0.091	127	7.12	6.82
2000-8	30-May-02	37406	Balanced	240	700	536	2,239	2.74	64.7	0.65	3921	40.7	20.4	45.0	40,904	0.205	22.6	16.6	42.7	150.7	9.62	0.080	102	7.04	6.86
2000-8	31-May-02	37407	Balanced	240	700	541	2,225	2.64	72.6	0.64	3895	41.7	19.8	43.0	40,904	0.205	22.0	15.7	43.4	149.5	9.55	0.083	111	6.90	6.64
2000-8	1-Jun-02	37408	Balanced	240	700	555	2,232	2.65	83.0	0.64	3911	41.9	19.5	42.5	40,904	0.198	21.3	15.2	45.4	146.5	9.36	0.082	99	7.00	6.83
2000-8	2-Jun-02	37409	Balanced	240	700	540	2,233	2.67	74.6	0.65	3932	41.8	20.1	43.0	40,904	0.200	21.5	15.3	43.5	148.9	9.51	0.083	100	7.02	6.97
2000-8	3-Jun-02	37410	Balanced	242	700	510	2,255	2.68	66.6	0.65	3928	42.3	19.5	42.0	40,904	0.177	20.6	14.6	43.4	141.0	9.01	0.080	88	7.12	6.95

Catalyst Life (η) – April - June 2002

The “age” of the methanol synthesis catalyst can be expressed in terms of a dimensionless variable η , which is defined as the ratio of the rate constant at any time to the rate constant for freshly reduced catalyst (as determined in the laboratory autoclave). Appendix C, Figure 1 plots $\log \eta$ versus days onstream for the third catalyst campaign (which began in August 2001, following the first 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.

At the conclusion of the catalyst withdrawal/addition catalyst campaign completed on 01 April 2002, the LPMEOH™ Demonstration Unit was operated at a constant reactor temperature of 235°C and a reactor pressure of 700 psig. The flowrate of Balanced Gas was controlled at an average value of 590 KSCFH during this time.

A catalyst deactivation rate of 0.69% per day was calculated for the period 01 April 2002 to 18 May 2002 (48 days). This is similar to the results that have been generally calculated over the past 2 years (averaging between 0.6% and 0.7% per day). As a basis of comparison, the calculated deactivation rate from the 4-month proof-of-concept run at the LaPorte AFDU in 1988/89 was 0.4% per day. (This run was performed on CO-rich syngas derived from natural gas at a reactor temperature of 250°C.)

Analyses of catalyst samples to determine changes in physical characteristics and levels of poisons have continued. Appendix C, Table 3 summarizes the results from August 2001, corresponding to the third catalyst campaign. Chemical analysis of catalyst samples has indicated the presence of all expected crystal phases. This analysis has indicated a significant increase in iron on the catalyst since the start of this current catalyst campaign.

Several activities were conducted during the quarter in an effort to determine the source of the iron contamination on the catalyst. Gas sampling and analysis that was conducted during the week of 22 April 2002 indicated that the concentration of iron in the Balanced Gas matched the historical levels of 10 ppbv or less.

To further investigate the potential sources of iron, the LPMEOH™ Demonstration Unit was shutdown on 05 June 2002. The slurry contents of the reactor were cooled and transferred in batches to the 29C-30 catalyst reduction vessel for final cooling and draining into drums. A total of 317 drums or 90,484 pounds of slurry were removed from the reactor. The reactor was then inspected during the week of 10 June 2002, just prior to the start of the second in-situ catalyst activation procedure. Carbon steel components were found to have been installed during the biennial inspection of pressure vessels in April of 2001; these were replaced with the correct stainless steel items. A visual inspection of the reactor did not indicate damage to the internal heat exchanger or vessel wall, which could have been additional sources of iron.

Additionally, a hot function test with Balanced Gas and mineral oil (in the absence of catalyst) was performed during the week of 17 June 2002. During this procedure, sampling

of the reactor feed gas stream was performed; during the three-day period, the measured levels of iron carbonyl into the LPMEOH™ Reactor dropped gradually from over 200 ppbv to approximately 30 ppbv. Based upon this decline in concentration, preparations were made to initiate the procedure for the in-situ activation of methanol synthesis catalyst within the LPMEOH™ Reactor.

Second In-situ Catalyst Activation – June 2002

As reported in Technical Progress Report No. 31, an assessment of the performance of the methanol synthesis catalyst following the execution of the in-situ activation procedure in August of 2001 was completed. The in-situ activation procedure used the LPMEOH™ Reactor (instead of the catalyst reduction vessel) to prepare fresh methanol synthesis catalyst for production of methanol by reacting metal oxides (for example, copper oxide) with a reductant such as CO or H₂ to produce the base metal plus either CO₂ or H₂O. It was found that storage of the fresh catalyst in the presence of mineral oil at elevated temperatures prior to reduction was the cause of the lower than expected catalyst activity. The operating steps in the procedure were modified so that the fresh methanol catalyst would not be exposed to temperatures in excess of 100°C prior to the introduction of dilute syngas.

Beginning on 21 June 2002, batches of fresh catalyst were mixed with mineral oil in the catalyst reduction vessel, heated to 100°C, and transferred, without being reduced or activated, to the reactor. Since the 29D-02 slurry tank was not used to collect the batches during the three days of catalyst loading, the process was greatly simplified compared with the first in-situ activation procedure in August of 2001. A total of eleven batches of fresh catalyst slurry, each containing about 3,700 pounds of fresh catalyst, was prepared in this manner. After the final transfer, the total amount of fresh catalyst in the reactor was 40,040 pounds, which is approximately the design catalyst inventory for the LPMEOH™ Demonstration Unit. Nitrogen was introduced in the bottom of the reactor during the entire loading procedure, and the unreduced catalyst in the reactor was maintained at 100°C by adding steam as necessary to the 29C-02 steam drum and internal heat exchanger. The catalyst loading and transfer operation was completed at 1725 hours on 24 June 2002.

The reactor was cooled overnight to the desired initial temperature for catalyst activation. The 29K-01 recycle compressor was started to assist in cooling the reactor contents. By 0800 hours on 25 June 2002, the reactor inlet flow was set at approximately 300 KSCFH, and 52 KSCFH of make-up nitrogen was introduced at the compressor suction. Reactor pressure was set at 85 psig. Cooling was completed after the reactor temperature reached 86°C.

After reactor cooling was completed, the in-situ catalyst activation procedure was started at 1200 hrs on 25 June 2002. In addition to the 52 KSCFH of nitrogen, a small quantity of Balanced Gas was introduced into the discharge of the recycle compressor to ensure that sufficient reductant was present at all times in the reactor inlet. The in-situ catalyst activation procedure ended at 0200 hrs on 27 June 2002.

The progress of the in-situ catalyst activation procedure was tracked by evaluating the uptake of reductant by the catalyst. A preliminary uptake of 98% of the theoretical value was

calculated based upon analytical data and measured flowrates, which is an indication that the methanol synthesis catalyst was properly activated during the in-situ catalyst activation procedure.

The LPMEOH™ Demonstration Unit was then re-started at 1500 hrs on 27 June 2002. Conditions were chosen to compensate for the hyperactivity that typically occurs when fresh methanol synthesis catalyst is initially exposed to syngas. During the first few days of operation, conditions were fairly constant with a reactor pressure of 450 psig, a reactor temperature of 214°C, and an average flowrate of Balanced Gas of 603 KSCFH. Preliminary heat and material balances indicate that the activity of the catalyst was excellent and similar to the results that have been achieved with catalyst that has been properly activated in the catalyst reduction vessel.

These initial results also compare favorably with the performance of the methanol synthesis catalyst following the first in-situ catalyst activation procedure in August of 2001. After this first attempt, the uptake of reductant was calculated to be 70% of the theoretical value and the initial activity was 60 to 70% of the value for properly activated catalyst. Also, a reactor pressure of between 550 and 600 psig was necessary following the first in-situ activation to convert 500-525 KSCFH of Balanced Gas at a reactor temperature of between 212 and 220°C. This comparison provides an indication that, since conditions of lower severity are now required to process a higher quantity of Balanced Gas, the activity of the catalyst is greater following the most recent in-situ catalyst activation procedure.

29C-40 Catalyst Guard Bed Performance

As noted in Technical Progress Report No. 31, a fresh quantity of adsorbent (activated carbon impregnated with copper oxide) was charged to the 29C-40 catalyst guard bed on 21 February 2002. This material was chemically reduced using dilute syngas in nitrogen. (In this case, reduction refers to the reaction of the copper oxide with a reductant such as CO or H₂ to copper metal and either CO₂ or H₂O). The catalyst guard bed was placed into service on 08 March 2002.

During the period of 26 March 2002 to 24 April 2002, gas sampling was performed to assess the performance of the adsorbent. The analytical techniques involved analyzing the catalyst guard bed inlet and outlet streams for arsine using standard techniques. Initial performance was acceptable, as the average concentration of arsine across the guard bed was reduced from an inlet value of approximately 50 ppbv to the detection limit of 2 ppbv. However, towards the end of the sampling period, the average concentration of arsine at the outlet of the catalyst guard bed was determined to be approximately 10 ppbv, which indicated that the performance of the adsorbent was beginning to degrade.

Prior to the start of the second in-situ catalyst activation procedure, the adsorbent in the catalyst guard bed was again replaced with fresh material. Fresh adsorbent in the catalyst guard bed would limit the amount of contaminants (in particular arsine, sulfur, and iron) that could enter the LPMEOH™ Reactor with the fresh feed syngas.

The catalyst guard bed reduction procedure was started at 1700 hrs on 20 June 2002. During this reduction procedure, a dilute stream of Balanced Gas in nitrogen was preheated and used as the reductant. The temperature control during the procedure was excellent, and the reduction was completed at 1700 hrs on 22 June 2002.

After cooling with nitrogen, a pressure check to full supply pressure with Balanced Gas was conducted. The adsorbent temperature increased by about 28°C and stabilized during this examination. This observation was consistent with the temperature increase which would be expected from the adsorption of CO on the adsorbent surface.

Sparger Resistance

The performance of the gas sparger, which was designed by Air Products and first installed into the LPMEOH™ Reactor prior to the restart of the LPMEOH™ Demonstration Unit in March of 1999, was monitored. The device had been inspected and cleaned during the March 2001 biennial outage. Appendix C, Figure 2 plots the average daily sparger resistance coefficient for the third catalyst campaign (which began in August 2001) until the end of the campaign in early June of 2002. 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.

During the preparations for the second in-situ activation of methanol synthesis catalyst, the sparger was inspected and cleaned. The performance of the sparger will be monitored closely for any changes following the restart on 27 June 2002.

D.4 Planning and Administration

A formal recommendation to DOE to perform a second test of the in-situ catalyst activation procedure was issued on 15 May 2002. DOE accepted the recommendation in a letter to Air Products dated 24 May 2002.

Comments were received from DOE on a draft report which provides publicly available technical data on the Eastman chemicals-from-coal complex in Kingsport. This report provides operational performance of the chemicals-from-coal complex in Kingsport as well as specific data on the major feed and effluent streams for the coal gasification and syngas cleanup systems.

The Milestone Schedule Status Report and the Cost Management Report, through the period ending 30 June 2002, 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 30 June 2002. Ninety-four percent (94%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 June 2002.

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

E. Planned Activities for the Next Quarter

- Continue executing Phase 3, Task 2.1 Methanol Operation per the Demonstration Test Plan. Focus activities on temperature programming to maintain the required methanol productivity, monitoring catalyst activity, assessing the performance of the catalyst guard bed, and monitoring the performance of the gas sparger in the reactor.
- Schedule a Project Review Meeting with DOE.

F. Conclusion

The LPMEOHTM Demonstration Unit operated at 100% availability during this quarter. There were three syngas interruptions that were experienced on 23 May 2002 (37.9 hours duration), 27 May 2002 (7.6 hours duration), and 04 June 2002 (16.2 hours duration). A major activity during the quarter involved the preparation for and execution of the steps to perform the in-situ activation of methanol synthesis catalyst within the LPMEOHTM Reactor.

At the conclusion of the catalyst withdrawal/addition catalyst campaign completed on 01 April 2002, the LPMEOHTM Demonstration Unit was operated at a constant reactor temperature of 235°C and a reactor pressure of 700 psig. The flowrate of Balanced Gas was controlled at an average value of 590 KSCFH during this time.

A catalyst deactivation rate of 0.69% per day was calculated for the period 01 April 2002 to 18 May 2002 (48 days). This is similar to the results that have been generally calculated over the past 2 years (averaging between 0.6% and 0.7% per day). As a basis of comparison, the calculated deactivation rate from the 4-month proof-of-concept run at the LaPorte AFDU in 1988/89 was 0.4% per day. (This run was performed on CO-rich syngas derived from natural gas at a reactor temperature of 250°C.)

Analyses of catalyst samples to determine changes in physical characteristics and levels of poisons have continued. Chemical analysis of catalyst samples has indicated the presence of all expected crystal phases. This analysis has indicated a significant increase in iron on the catalyst since the start of this current catalyst campaign.

Several activities were conducted during the quarter in an effort to determine the source of the iron contamination on the catalyst. Gas sampling and analysis that was conducted during the week of 22 April 2002 indicated that the concentration of iron in the Balanced Gas matched the historical levels of 10 ppbv or less.

To further investigate the potential sources of iron, the LPMEOH™ Demonstration Unit was shutdown on 05 June 2002. The slurry contents of the reactor were cooled and transferred in batches to the catalyst reduction vessel for final cooling and draining into drums. A total of 317 drums or 90,484 pounds of slurry were removed from the reactor. The reactor was then inspected during the week of 10 June 2002, just prior to the start of the second in-situ catalyst activation procedure. Carbon steel components were found to have been installed during the biennial inspection of pressure vessels in April of 2001; these were replaced with the correct stainless steel items. A visual inspection of the reactor did not indicate damage to the internal heat exchanger or vessel wall, which could have been additional sources of iron.

Additionally, a hot function test with Balanced Gas and mineral oil (in the absence of catalyst) was performed during the week of 17 June 2002. During this procedure, sampling of the reactor feed gas stream was performed; during the three-day period, the measured levels of iron carbonyl into the LPMEOH™ Reactor dropped gradually from over 200 ppbv to approximately 30 ppbv. Based upon this decline in concentration, preparations were made to initiate the procedure for the in-situ activation of methanol synthesis catalyst within the LPMEOH™ Reactor.

As noted in Technical Progress Report No. 31, a fresh quantity of adsorbent (activated carbon impregnated with copper oxide) was charged to the 29C-40 catalyst guard bed on 21 February 2002. This material was chemically reduced using dilute syngas in nitrogen. (In this case, reduction refers to the reaction of the copper oxide with a reductant such as CO or H₂ to copper metal and either CO₂ or H₂O). The catalyst guard bed was placed into service on 08 March 2002.

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Prior to the start of the second in-situ catalyst activation procedure, the adsorbent in the catalyst guard bed was again replaced with fresh material. Fresh adsorbent in the catalyst guard bed would limit the amount of contaminants (in particular arsine, sulfur, and iron) that could enter the LPMEOH™ Reactor with the fresh feed syngas.

The catalyst guard bed reduction procedure was started at 1700 hrs on 20 June 2002. During this reduction procedure, a dilute stream of Balanced Gas in nitrogen was preheated and used as the reductant. The temperature control during the procedure was excellent, and the reduction was completed at 1700 hrs on 22 June 2002.

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examination. This observation was consistent with the temperature increase which would be expected from the adsorption of CO on the adsorbent surface.

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Beginning on 21 June 2002, batches of fresh catalyst were mixed with mineral oil in the catalyst reduction vessel, heated to 100°C, and transferred, without being reduced or activated, to the reactor. Since the slurry tank was not used to collect the batches during the three days of catalyst loading, the process was greatly simplified compared with the first in-situ activation procedure in August of 2001. A total of eleven batches of fresh catalyst slurry, each containing about 3,700 pounds of fresh catalyst, was prepared in this manner. After the final transfer, the total amount of fresh catalyst in the reactor was 40,040 pounds, which is approximately the design catalyst inventory for the LPMEOH™ Demonstration Unit. Nitrogen was introduced in the bottom of the reactor during the entire loading procedure, and the unreduced catalyst in the reactor was maintained at 100°C by adding steam as necessary to the steam drum and internal heat exchanger. The catalyst loading and transfer operation was completed at 1725 hours on 24 June 2002.

The reactor was cooled overnight to the desired initial temperature for catalyst activation. The recycle compressor was started to assist in cooling the reactor contents. By 0800 hours on 25 June 2002, the reactor inlet flow was set at approximately 300 KSCFH, and 52 KSCFH of make-up nitrogen was introduced at the compressor suction. Reactor pressure was set at 85 psig. Cooling was completed after the reactor temperature reached 86°C.

After reactor cooling was completed, the in-situ catalyst activation procedure was started at 1200 hrs on 25 June 2002. In addition to the 52 KSCFH of nitrogen, a small quantity of Balanced Gas was introduced into the discharge of the recycle compressor to ensure that sufficient reductant was present at all times in the reactor inlet. The in-situ catalyst activation procedure ended at 0200 hrs on 27 June 2002.

The progress of the in-situ catalyst activation procedure was tracked by evaluating the uptake of reductant by the catalyst. A preliminary uptake of 98% of the theoretical value was calculated based upon analytical data and measured flowrates, which is an indication that the methanol synthesis catalyst was properly activated during the in-situ catalyst activation procedure.

The LPMEOH™ Demonstration Unit was then re-started at 1500 hrs on 27 June 2002. Preliminary heat and material balances indicate that the activity of the catalyst was excellent

and similar to the results that have been achieved with catalyst that has been properly activated in the catalyst reduction vessel. These initial results also show an improvement when compared with the performance of the methanol synthesis catalyst following the first in-situ catalyst activation procedure in August of 2001 (when the uptake of reductant was 70% of the theoretical value and the initial activity was 60 to 70% of the value for properly activated catalyst).

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. During the preparations for the second in-situ activation of methanol synthesis catalyst, the sparger was inspected and cleaned. The performance of the sparger will continue to be monitored closely for any changes.

During the reporting period, a total of 3,328,693 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, about 95.2 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.

Activities associated with Design Verification Testing (DVT) of the LPDME Process have been completed. A Topical Report, which provides the status of the current market for DME and an outlook on potential market developments through 2006, was approved by DOE and issued.

A formal recommendation to DOE to perform a second test of the in-situ catalyst activation procedure was issued on 15 May 2002. DOE accepted the recommendation in a letter to Air Products dated 24 May 2002.

Comments were received from DOE on a draft report which provides publicly available technical data on the Eastman chemicals-from-coal complex in Kingsport. This report provides operational performance of the chemicals-from-coal complex in Kingsport as well as specific data on the major feed and effluent streams for the coal gasification and syngas cleanup systems.

One hundred percent (100%) of the \$38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 30 June 2002. Ninety-four percent (94%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 June 2002.

APPENDICES

APPENDIX A - SIMPLIFIED PROCESS FLOW DIAGRAM

APPENDIX B - SAMPLES OF DETAILED MATERIAL BALANCE REPORTS

APPENDIX C - RESULTS OF DEMONSTRATION UNIT OPERATION

**Table 1 - Summary of LPMEOH™ Demonstration Unit Outages -
April/June 2002**

Table 2 - Summary of Catalyst Samples - Third Catalyst Batch

Figure 1 - Catalyst Age (η): September 2001 - June 2002

**Figure 2 - Sparger Resistance Coefficient vs. Days Onstream
(September 2001 - June 2002)**

Table 1
Summary of LPMEOH™ Demonstration Unit Outages - April/June 2002

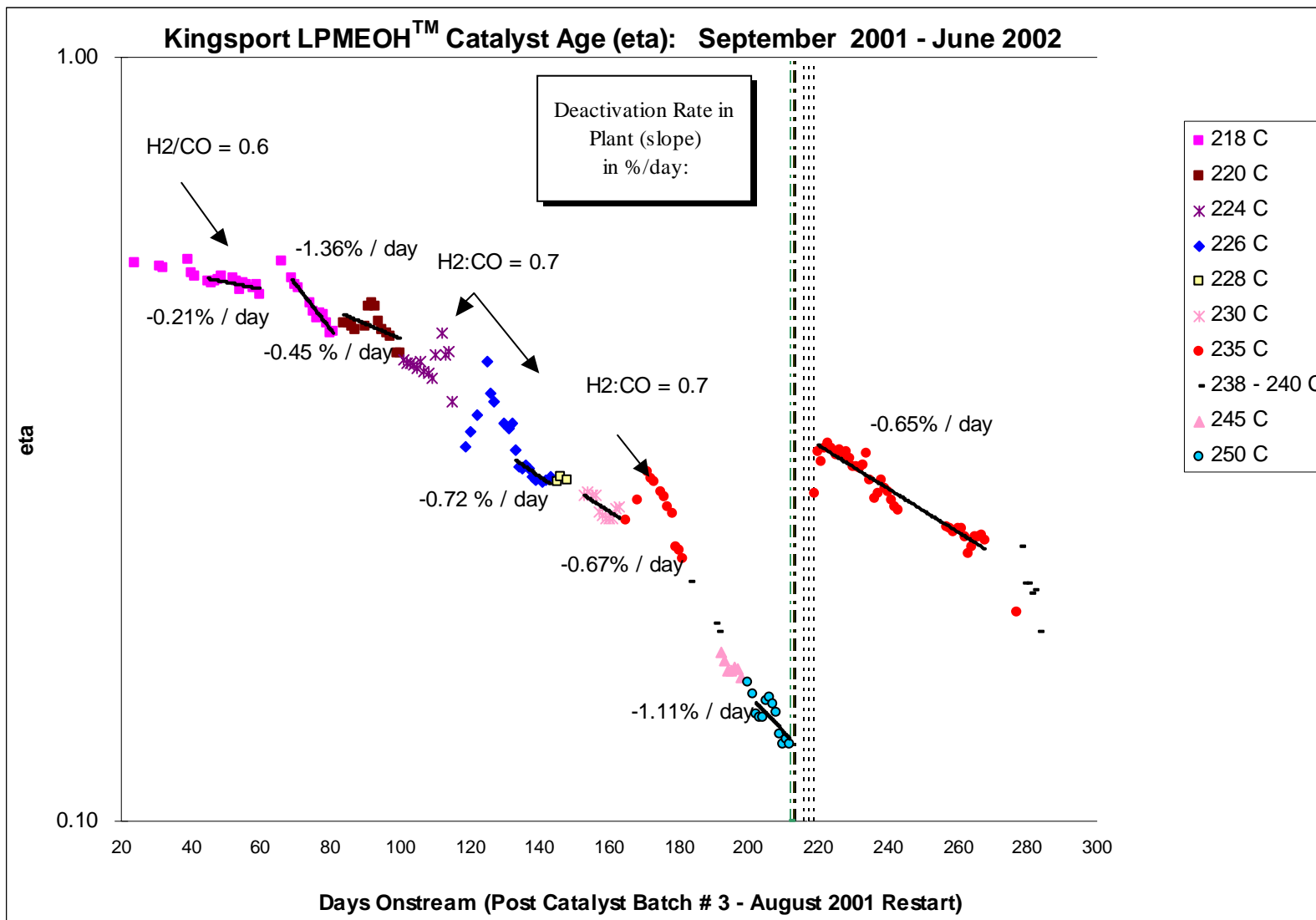
Operation Start	Operation End	Operating Hours	Shutdown Hours	Reason for Shutdown
4/1/02 00:00	5/23/02 15:00	1263.0	37.9	Syngas Outage
5/25/02 04:52	5/27/02 13:06	56.2	7.6	Syngas Outage
5/27/02 20:42	6/4/02 17:00	188.3	16.2	Syngas Outage
6/5/02 09:15	6/5/02 09:15	0.0	532.8	In-situ Activation
6/27/02 14:00	6/30/02 23:59	82.0		End of Reporting Period
Total Operating Hours			1589.5	
Syngas Available Hours			1589.5	
Plant Availability, %			100.00	

Table 2
Summary of Catalyst Samples - Third Catalyst Batch

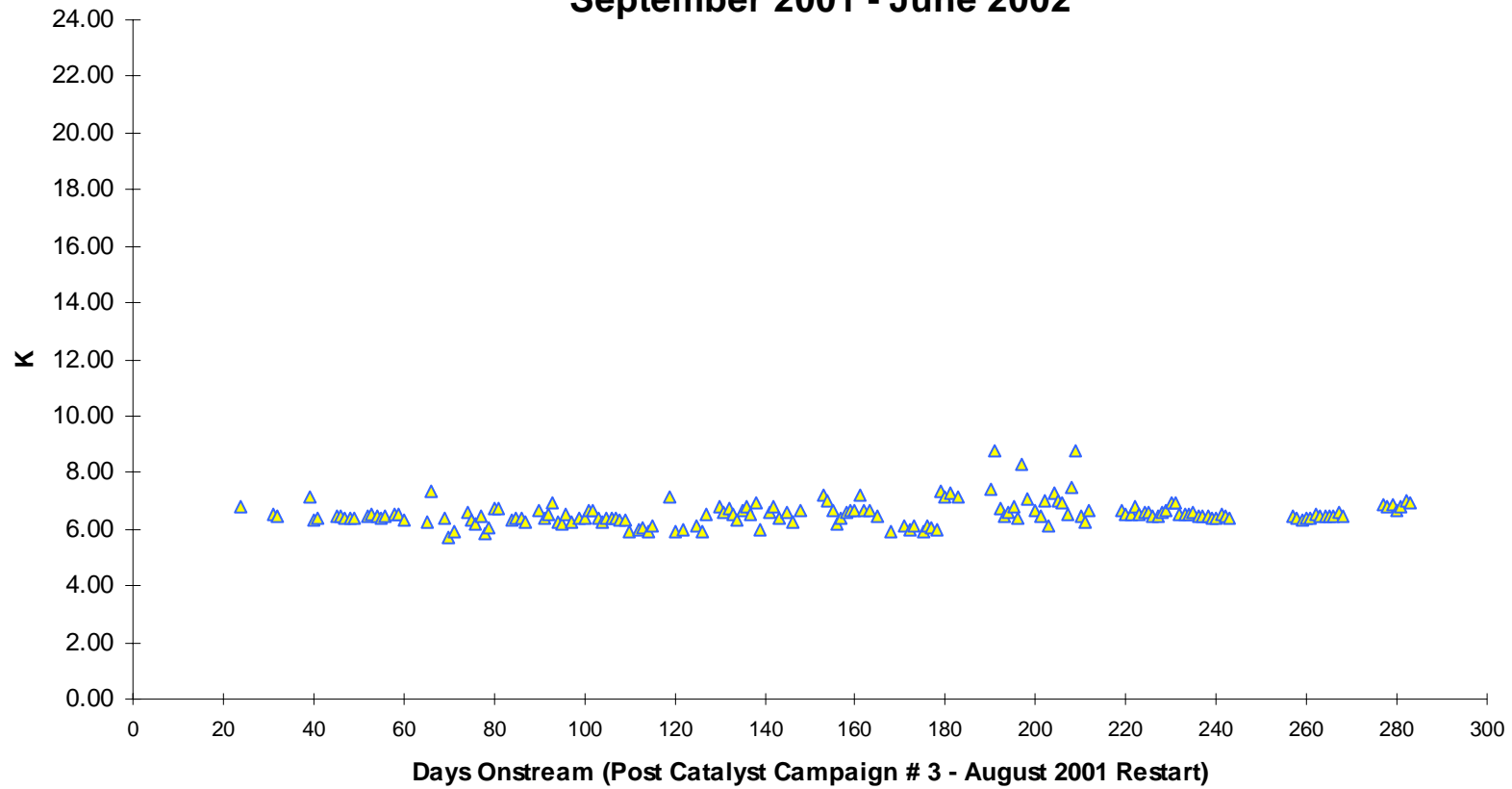
Sample	Identity	XRD		BET	Analytical (ppmw)				
		Cu	ZnO	m ² /g	Fe	Ni	S	As	Cl
K0109-1	Reactor Sample 9/5/01	178	90	78	48	≤19	< 140	≤73	nd
K0109-2	Reactor Sample 9/12/01	188	88						
K0109-3	Reactor Sample 9/19/01	185	159	85	52	< 10	< 140	≤70	nd
K0110-1	Reactor Sample 10/3/01	181	78	82	71	< 10	≤85	65	nd
K0110-2	Reactor Sample 10/17/01	203	98	82	97	< 10	≤100	83	40
K0110-3	Reactor Sample 10/31/01	197	45	78	147	< 10	≤120	139	nd
K0111-1	Reactor Sample 11/14/01	200	80	80	191	< 10	≤130	138	50
K0111-3	Reactor Sample 11/30/01	204	79	82	241	< 10	≤140	150	30
K0112-1	Reactor Sample 12/12/01	256	77	74	293	≤20	≤80	260	nd
K0201-1	Reactor Sample 1/09/02	212	78	50	355	<10	≤95	192	30
K0201-2	Reactor Sample 1/23/02	207	121		363	≤10	≤57	152	nd
K0202-1	Reactor Sample 2/6/02	211	84		390	<10	≤91	138	nd
K0203-1	Reactor Sample 3/6/02	220	71		428	<10	<40	210	nd
K0203-2	Reactor Sample 3/14/02	216	102		477	<10	<160	318	nd
K0203-3	Reactor Sample 3/20/02	215	143	24	508	≤ 12	<160	314	nd
K0203-4	Reactor Sample 3/25/02	218	101	51	534	<10	<160	203	40
K0204-1	Reactor Sample 4/4/02	213	87						
K0204-2	Reactor Sample 4/11/02	206	117						
K0204-3	Reactor Sample 4/25/02	211	98						

Notes:

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



**Kingsport LPMEOH™
Sparger Resistance Coefficient
September 2001 - June 2002**



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