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

TECHNICAL PROGRESS REPORT NO. 26

For The Period

1 October - 31 December 2000

Prepared by

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and

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for the
Air Products Liquid Phase Conversion Company, L.P.

Prepared for the United States Department of Energy
National Energy Technology Laboratory
Under Cooperative Agreement No. DE-FC22-92PC90543

Patents cleared by Chicago on 21 February 2001.

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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.

During the month of October 2000, a new monthly production record of 2,500,000 gallons of methanol was set at the LPMEOH™ Demonstration Unit and nameplate production of 80,000 gallons per day (260 tons per day (TPD)) or higher was maintained for 23 consecutive days. The October monthly average production was 101% of nameplate. Production rates as high as 93,800 gallons per day (305 TPD), which corresponds to 117% of nameplate capacity, were also achieved for shorter periods during this month of record-breaking operation.

The LPMEOH™ Demonstration Unit operated at 98.11% availability during this quarter. A forced outage was taken on 18 November 2000 to repair a leak in a tube of the air-cooled condenser (29E-21) on the methanol rectifier column. The leaking tube was plugged and the exchanger was placed back in service without an environmental or safety incident. Total time for this forced outage was 41 hours. There were also three short syngas outages experienced which totaled 41.7 hours.

The major catalyst withdrawal and addition campaign begun in the previous quarter was completed on 05 October 2000. After the addition of the final batch of catalyst, the total catalyst inventory in the LPMEOH™ Reactor was calculated to be 47,163 pounds.

During most of the quarter, the flowrate of the primary syngas feed (Balanced Gas) was controlled at an average flowrate of 775 KSCFH. The maximum instantaneous flowrate of Balanced Gas was over 1,000 KSCFH during the high production period in October 2000. During these operating periods, the reactor pressure was set an average of 710 psig and temperature was maintained at 235ºC.

On 15 November 2000, a period of operation on carbon monoxide- (CO)-rich syngas was performed. An average of 50 KSCFH of a syngas stream which contains primarily CO (CO Gas) was introduced with sufficient Balanced Gas to achieve a ratio of hydrogen (H_2) to CO at the reactor inlet of 1.0. This test ended with the forced outage that occurred on 18 November 2000.

After the restart on 20 November 2000, over 1,000 KSCFH of Balanced Gas was fed to the LPMEOH™ Demonstration Unit. Reactor temperature was increased from 235°C to 248°C during this period to allow for greater catalyst productivity and to lower the purge rate. This particular test was concluded on 22 November 2000, coincident with a syngas outage.

There was one extended period 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. An overall deactivation rate of 0.65% per day was calculated for the period 06 October to 26 October 2000 (21 days). This data was based on operation with the 29C-40 catalyst guard bed bypassed. The results of this data set is statistically similar to other recent periods of operation. This deactivation rate is greater than 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. Samples have continued to show an increase in arsenic, which has been demonstrated in the laboratory to act as a poison to methanol synthesis catalyst. Sulfur, another known catalyst poison, continues to be measured above the analytical detection limit. Copper crystallite size measurements have stabilized in the most recent samples. Levels of nickel (a known catalyst poison) have remained low and steady since the restart in December of 1997. The concentration of iron (another poison), is low (less than 200 ppmw), and does not seem to be increasing in the most recent samples. 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.

Eastman has developed a design package which defines the necessary modifications to the piping and instrument systems to allow for both the in-situ activation of methanol synthesis catalyst in the LPMEOH™ Reactor and a proposed reuse of the copper oxide-impregnated activated carbon in the 29C-40 catalyst guard bed (background information on the catalyst guard bed can be found in Technical Progress Report No. 25). The timing for implementation of these changes must still be determined.

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.

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During this quarter, planning, procurement, and test operations were concluded on the project sites selected for the off-site, product-use test program. Final reports have been approved by Air Products for five of the seven projects.

Activities associated with Design Verification Testing of the Liquid Phase Dimethyl Ether (LPDME) Process have been completed. During the last quarter, comments were received from DOE on a draft Topical Report which presents the results of the design verification test

at the LaPorte AFDU. A revision will be issued for final review and approval by January of 2001.

During this reporting period, comments were received from DOE regarding an update to the Demonstration Test Plan. In this update, the tests which are planned for the remainder of the operating program were defined. Based upon this feedback, a second revision was prepared and sent to DOE for final review and approval.

One hundred percent (100%) of the $38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 31 December 2000. Sixty-eight percent (68%) of the $158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 December 2000.

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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	-	sun 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.

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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 H_2 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 submitted to Air Products (no update in this reporting period).

Stationary Turbine for Nitrogen Oxide (NO_x) Control - The test results on the low-NO_x gas turbine combustor fueled with stabilized methanol from the LPMEOH™ Demonstration Unit have been prepared. Air Products is awaiting the submittal of the draft final report from ARCADIS Geraghty & Miller.

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 - Results of experiments in the reformer test apparatus have provided convincing evidence that the performance of the catalyst is adversely affected by the presence of the trace mineral oil in the stabilized methanol. Preparation of the final report is underway.

West Virginia University Tri-Boro Bus - The final report for this project was submitted to 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 received. 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.

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 draft Topical Report which presents the results of the DVT at the LaPorte AFDU was sent to DOE on 18 July 2000. Comments were received on 23 August 2000, and a revision will be issued for final review and approval by January of 2001.

A separate Topical Report on the market analysis for DME and review of the economics of the LPDME Process will be prepared by the LPMEOH™ Demonstration Project following the release of the DVT Topical Report.

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 month of October 2000, a new monthly production record of 2,500,000 gallons of methanol was set and nameplate production of 80,000 gallons per day (260 TPD) or higher was maintained for 23 consecutive days. The October monthly average production was 101% of nameplate. Production rates as high as 93,800 gallons per day (305 TPD), which corresponds to 117% of nameplate capacity, were also achieved for shorter periods during this month of record-breaking operation.

During the reporting period, a total of 6,236,149 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 98.11% availability during this quarter. A forced outage was taken on 18 November 2000 to repair a leak in a tube of the air-cooled condenser (29E-21) on the methanol rectifier column. The leaking tube was plugged and the exchanger was placed back in service without an environmental or safety incident. Total

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

Case	Date	Days On Stream	Gas Type	Temp (Deg C)	Pres. (psig)	Fresh Feed (KSCFH)	Recycle Gas (KSCFH)	Reactor Feed (H2:CO)	Purge Gas (KSCFH)	Inlet Sup. Velocity (ft/sec)	Space Velocity (l/hr-kg)	Slurry Conc. (wt% ox)	Gas Holdup (vol%)	Gassed Slurry Hgt (ft)	Catalyst Inventory (lb)	Catalyst Age (eta)	CO Conv. (%)	Reactor O-T-M Conv. (%)	Syngas Util. (SCF/lb)	Raw MeOH Prod. (TPD)	Catalyst MeOH Prod. (gmol/hr-kg)	Reactor Vol. Prod. (TPD/ft3)	U Overall (Btu hr ft2 F)	Sparger dP (psi)	Sparger Resist. ("K")
11	2-Oct-00	1017	Balanced	235	706	811	2,422	3.51	46.8	0.68	3838	40.2	27.4	55.0	44,963	0.465	43.7	25.0	38.9	250.4	14.53	0.108	148	5.87	5.27
11	3-Oct-00	1018	Balanced	235	706	832	2,091	3.39	53.1	0.68	3845	42.2	27.5	51.0	44,963	0.464	42.9	25.1	39.2	254.6	14.77	0.119	155	5.90	5.30
11	4-Oct-00	1019	Balanced	235	706	856	2,088	3.48	59.7	0.69	3849	42.1	27.9	51.5	44,963	0.476	44.4	25.6	39.5	260.1	15.09	0.120	158	5.76	5.27
11	5-Oct-00	1020	Balanced	235	705	867	2,027	3.65	67.5	0.67	3780	39.4	29.0	54.0	44,963	0.491	46.9	26.2	39.7	262.5	15.23	0.116	156	5.52	5.40
11	6-Oct-00	1021	Balanced	235	705	911	2,071	3.49	67.8	0.70	3715	45.4	33.9	52.0	47,163	0.492	46.3	26.8	39.6	275.7	15.25	0.126	167	5.86	5.29
11	7-Oct-00	1022	Balanced	235	705	934	2,068	3.60	73.9	0.70	3726	44.7	31.0	51.0	47,163	0.501	47.7	27.0	39.9	281.3	15.55	0.131	172	5.63	5.30
11	8-Oct-00	1023	Balanced	235	705	925	2,057	3.55	80.2	0.69	3710	43.3	32.0	54.5	47,163	0.485	46.6	26.6	40.2	275.8	15.27	0.120	144	5.63	5.30
11	9-Oct-00	1024	Balanced	235	709	900	2,057	3.73	79.0	0.68	3680	43.9	33.5	54.5	47,163	0.468	47.1	26.0	40.1	269.0	14.88	0.117	137	5.37	5.42
11	10-Oct-00	1025	Balanced	235	709	916	2,038	3.63	67.5	0.69	3682	44.5	33.8	53.5	47,163	0.494	47.8	26.8	39.6	277.6	15.36	0.123	151	5.42	5.35
11	11-Oct-00	1026	Balanced	235	707	911	2,053	3.46	64.9	0.69	3709	44.4	27.9	49.5	47,163	0.487	45.9	26.7	39.5	276.9	15.32	0.133	157	5.63	5.26
11	12-Oct-00	1027	Balanced	235	709	928	2,074	3.41	66.3	0.70	3742	44.8	31.8	51.5	47,163	0.491	45.6	26.8	39.6	281.1	15.55	0.130	155	5.85	5.41
11	13-Oct-00	1028	Balanced	235	707	918	2,085	3.31	68.1	0.70	3764	44.3	33.3	53.5	47,163	0.480	44.1	26.5	39.4	279.4	15.46	0.124	140	5.87	5.29
11	14-Oct-00	1029	Balanced	234	706	922	2,083	3.30	73.8	0.70	3745	44.3	33.2	53.5	47,163	0.474	43.9	26.6	39.6	279.2	15.44	0.124	136	6.05	5.51
11	15-Oct-00	1030	Balanced	234	708	920	2,070	3.43	81.3	0.69	3729	44.1	27.5	50.0	47,163	0.476	45.1	26.6	39.9	276.5	15.31	0.132	145	5.83	5.40
11	23-Oct-00	1038	Balanced	234	706	865	2,103	3.27	76.8	0.69	3717	44.1	30.1	51.5	47,163	0.430	41.0	24.9	40.1	259.0	14.33	0.120	124	5.69	5.14
11	25-Oct-00	1040	Balanced	238	711	993	1,906	3.35	140.8	0.68	3622	45.2	36.9	55.0	47,163	0.445	45.2	27.0	43.1	276.6	15.34	0.120	117	5.3	5.19
11	26-Oct-00	1041	Balanced	234	709	888	2,098	3.29	79.3	0.70	3742	44.4	26.5	48.5	47,163	0.439	41.9	25.4	40.2	265.1	14.68	0.130	148	5.92	5.28
11	30-Oct-00	1045	Balanced	234	710	792	2,248	3.31	54.9	0.71	3824	45.5	34.4	52.0	47,163	0.393	38.2	23.0	39.3	241.9	13.37	0.111	123	6.18	5.18
11	3-Nov-00	1049	Balanced	234	710	786	2,178	3.57	59.0	0.69	3703	42.1	24.4	51.5	47,163	0.385	40.4	23.1	39.9	236.3	13.08	0.109	127	5.67	5.32
11	4-Nov-00	1050	Balanced	234	710	788	2,189	3.49	59.8	0.69	3705	44.6	30.6	51.0	47,163	0.395	40.5	23.5	39.5	239.3	13.25	0.112	131	5.84	5.32
11	5-Nov-00	1051	Balanced	234	710	791	2,191	3.45	63.1	0.69	3712	45.6	33.3	51.0	47,163	0.393	39.9	23.4	39.7	238.9	13.22	0.112	127	5.81	5.26
11	7-Nov-00	1053	Balanced	234	710	786	2,146	3.56	60.1	0.68	3658	44.1	27.9	50.0	47,163	0.400	41.6	23.7	39.4	239.4	13.25	0.114	139	5.68	5.34
11	8-Nov-00	1054	Balanced	234	705	775	2,095	3.41	55.9	0.67	3605	44.2	28.8	50.5	47,163	0.394	40.3	23.7	39.5	235.2	13.02	0.111	134	5.80	5.41
11	9-Nov-00	1055	Balanced	234	705	782	2,118	3.61	65.0	0.68	3616	44.9	28.6	49.0	47,163	0.391	41.6	23.5	39.8	235.5	13.03	0.115	136	5.61	5.48
11	10-Nov-00	1056	Balanced	236	705	800	2,162	3.23	62.3	0.70	3726	45.6	33.2	51.0	47,163	0.381	38.2	23.4	39.7	241.6	13.37	0.113	126	5.98	5.26
11	11-Nov-00	1057	Balanced	234	705	792	2,148	3.21	61.8	0.69	3708	45.0	30.3	50.0	47,163	0.385	37.7	23.1	39.9	238.4	13.20	0.114	130	5.94	5.26
11	12-Nov-00	1058	Balanced	234	705	792	2,143	3.11	59.0	0.69	3698	44.6	28.5	49.5	47,163	0.387	37.1	23.3	39.6	240.0	13.28	0.116	129	6.02	5.31
11	13-Nov-00	1059	Balanced	234	705	790	2,152	3.06	63.6	0.69	3702	46.7	36.1	51.0	47,163	0.383	36.3	23.1	39.9	237.7	13.16	0.111	117	5.92	5.19
11	14-Nov-00	1060	Balanced	234	705	800	2,145	3.49	78.0	0.69	3683	43.3	27.2	51.0	47,163	0.376	39.3	23.0	40.7	236.2	13.06	0.110	123	5.53	5.44
21	17-Nov-00	1063	Balanced	235	704	726	2,350	1.08	62.4	0.74	3958	46.3	35.7	51.5	47,163	0.403	16.8	20.3	39.6	220.2	12.19	0.102	125	9.94	4.91
11	21-Nov-00	1067	Balanced	248	730	1019	1,902	4.23	232.7	0.66	3569	45.4	29.9	49.5	47,163	0.340	49.1	25.2	47.9	255.5	14.14	0.123	133	4.76	5.97
11	25-Nov-00	1071	Balanced	234	705	650	2,149	4.12	36.3	0.65	3471	44.5	31.0	51.5	47,163	0.345	41.3	21.0	38.8	200.9	11.11	0.093	134	4.85	5.39
11	26-Nov-00	1072	Balanced	235	705	650	2,169	3.88	31.6	0.65	3499	45.1	30.5	50.0	47,163	0.347	40.0	21.3	38.3	203.4	11.24	0.097	138	5.09	5.33
11	27-Nov-00	1073	Balanced	234	701	693	2,155	3.33	39.4	0.68	3599	43.3	25.7	50.0	47,163	0.340	35.9	21.5	38.9	213.9	11.83	0.102	129	5.45	5.21
11	28-Nov-00	1074	Balanced	234	701	704	2,141	3.52	47.8	0.67	3553	43.6	28.6	51.5	47,163	0.347	37.9	21.8	39.2	215.6	11.92	0.100	125	5.17	5.28
11	30-Nov-00	1076	Balanced	236	709	699	2,114	3.22	47.6	0.66	3556	41.4	21.6	51.0	47,163	0.346	41.1	21.7	38.9	215.7	11.93	0.101	123	5.07	5.39
11	1-Dec-00	1077	Balanced	236	710	698	2,182	3.68	37.7	0.67	3618	44.2	28.7	50.5	47,163	0.346	39.4	21.5	38.6	217.1	12.01	0.102	122	5.27	5.22
11	2-Dec-00	1078	Balanced	236	710	700	2,194	3.41	34.2	0.68	3642	44.3	28.3	50.0	47,163	0.348	37.6	21.7	38.3	219.3	12.12	0.104	124	5.6	5.19
11	3-Dec-00	1079	Balanced	236	710	701	2,232	3.37	39.3	0.69	3684	44.5	30.2	51.0	47,163	0.332	36.2	21.2	38.9	215.9	11.94	0.101	116	5.66	5.17
11	4-Dec-00	1080	Balanced	236	710	711	2,217	3.31	44.4	0.68	3671	44.9	29.9	50.0	47,163	0.332	35.9	21.4	39.1	218.1	12.07	0.104	118	5.63	5.19
11	5-Dec-00	1081	Balanced	234	710	686	2,231	3.28	43.8	0.68	3666	43.9	28.9	51.0	47,163	0.325	34.5	20.8	39.0	211.2	11.68	0.099	119	5.56	5.14
11	6-Dec-00	1082	Balanced	235	710	701	2,190	3.51	49.3	0.67	3629	43.1	30.1	53.5	47,163	0.333	37.0	21.2	39.3	214.0	11.84	0.095	122	5.3	5.17
11	7-Dec-00	1083	Balanced	235	710	714	2,121	3.59	51.0	0.66	3561	42.4	26.7	52.5	47,163	0.342	38.6	21.7	39.9	215.0	11.90	0.097	131	5.18	5.24
11	8-Dec-00	1084	Balanced	235	710	701	2,200	3.54	49.7	0.67	3631	42.8	27.3	52.0	47,163	0.328	36.8	21.1	39.3	213.8	11.82	0.098	125	5.27	5.28
11	9-Dec-00	1085	Balanced	234	710	701	2,139	3.43	49.3	0.66	3576	43.1	28.7	52.5	47,163	0.337	37.0	21.6	39.2	214.3	11.86	0.097	126	5.3	5.20
11	10-Dec-00	1086	Balanced	234	710	700	2,191	3.36	48.3	0.67	3616	43.1	27.3	51.5	47,163	0.326	35.6	21.0	39.6	212.4	11.75	0.098	123	5.36	5.18
11	11-Dec-00	1087	Balanced	235	710	741	2,108	3.37	60.7	0.66	3559	43.9	31.5	53.0	47,163	0.351	37.9	22.4	39.8	223.3	12.36	0.100	138	5.29	5.26
11	12-Dec-00	1088	Balanced	235	710	758	2,167	3.25	62.4	0.68	3657	44.3	33.7	54.0	47,163	0.342	36.2	22.1	40.0	227.5	12.58	0.100	133	5.47	5.32
11	13-Dec-00	1089	Balanced	235	710	750	2,147	3.10	62.4	0.67	3623	44.8	31.2	51.0	47,163	0.340	35.1	22.1	40.1	224.6	12.43	0.105	137	5.49	5.17
11	14-Dec-00	1090	Balanced	235	710	749	2,921	3.01	62.3	0.68	3659	45.5	31.7	50.0	47,163	0.332	33.6	21.7	40.4	222.7	12.32	0.106	135	5.66	5.22
11	15-Dec-00	1091	Balanced	235	710	735	2,195	3.06	60.6	0.68	3663	45.5	31.6	50.0	47,163	0.325	33.5	21.4	40.0	220.5	12.20	0.105	131	5.59	5.24
11	18-Dec-00	1094	Balanced	234	710	743	2,148	3.20	82.7	0.67	3620	44.7	31.5	51.5	47,163	0.316	33.8	21.1	41.3	215.8	11.94	0.100	115	5.12	5.17
11	19-Dec-00	1095	Balanced	234	710	743	2,158	3.04	79.0	0.67	3626	43.8	31.2	53.0	47,163	0.319	33.0	21.3	41.0	217.4	12.03	0.098	116	5.42	5.25

time for this forced outage was 41 hours. There were also three syngas outages experienced on 27 October 2000 (30 hours), 20 November 2000 (7 hours), and 22 November 2000 (4.7 hours). Appendix C, Table 1 contains the summary of outages for the LPMEOH™ Demonstration Unit during this quarter.

Catalyst Life (eta) – October - December 2000

The "age" of the methanol synthesis catalyst can be expressed in terms of a dimensionless variable eta (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 η versus days onstream from November of 1999 to the end of the reporting period. Since catalyst activity typically follows a pattern of exponential decay, the plot of log η is fit to a series of straight lines, with step-changes whenever fresh catalyst was added to the reactor.

The major catalyst withdrawal and addition campaign begun in September 2000 was completed during the month of October in order to increase catalyst activity. One more addition of fresh catalyst was activated and added on 05 October 2000. After the final addition of catalyst, the total catalyst inventory was calculated to be 47,163 pounds.

During most of the quarter, the flowrate of Balanced Gas was controlled at an average flowrate of 775 KSCFH. The maximum instantaneous flowrate of Balanced Gas was over 1,000 KSCFH during the high production period in October 2000. During these operating periods, the reactor pressure was set an average of 710 psig and temperature was maintained at 235ºC.

There was one extended period of operation (minimum 2 weeks) at a reactor temperature of 235°C during which catalyst activity was measured to track catalyst deactivation during the quarter. An overall deactivation rate of 0.65% per day was calculated for the period 06 October to 26 October 2000 (21 days). This data was based on operation with the 29C-40 catalyst guard bed bypassed. The results of this data set is statistically similar to other periods of operation as plotted in Appendix C, Figure 1, given the scatter in the calculated values for the catalyst rate constant. This deactivation result is greater than 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.

On 15 November 2000, a period of operation on CO-rich syngas was performed. An average of 50 KSCFH of CO Gas was introduced to the LPMEOH™ Demonstration Unit. Balanced Gas was adjusted to achieve a H_2:CO ratio at the reactor inlet of 1.0. This test ended with the forced outage that occurred on 18 November 2000.

After the restart on 20 November 2000, over 1,000 KSCFH of Balanced Gas was fed to the LPMEOH™ Demonstration Unit. Reactor temperature was increased from 235°C to 248°C during this period to allow for greater catalyst productivity and to lower the purge rate. When compared to a similar test in October of 2000, the methanol production rates were lower (78,800 gallons per day, or 256 TPD) due to the increased age of the catalyst within the LPMEOH™ Reactor. This particular test was concluded on 22 November 2000, coincident with a syngas outage.

Analyses of catalyst samples for changes in physical characteristics and levels of poisons have continued. Appendix C, Table 2 summarizes the results to date. Samples have continued to show an increase in arsenic (to around 1,500 ppmw), which has been demonstrated in the laboratory to act as a poison to methanol synthesis catalyst. Sulfur, another known catalyst poison, has stabilized at a level of 200-300 ppmw). 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. The concentration of iron (another poison) has continued to remain low (less than 200 ppmw) and does not seem to be increasing in the most recent samples. 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.

In-situ Catalyst Activation

Eastman has been reviewing design options for both the in-situ activation of methanol synthesis catalyst in the LPMEOH™ Reactor and a proposed reuse of the copper oxide-impregnated activated carbon in the 29C-40 catalyst guard bed (background information on the catalyst guard bed can be found in Technical Progress Report No. 25). A design package has been developed by Eastman which defines the necessary modifications to the piping and instrument systems. The timing for implementation of these changes must still be determined.

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. During this reporting period, comments were received from DOE regarding an update to the Demonstration Test Plan. In this revision, a timetable to complete some of the key tests from the original Plan was presented, and new tests of significant commercial interest were defined. Based upon this feedback, a second revision was prepared and sent to DOE for final review and approval.

The Milestone Schedule Status Report and the Cost Management Report, through the period ending 31 December 2000, 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 December 2000. Sixty-eight percent (68%) of the $158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 December 2000.

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

E. Planned Activities for the Next Quarter

- Continue to analyze catalyst slurry samples and reactor performance data to determine causes for deactivation of methanol synthesis catalyst.
- 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, performing on-off and ramping tests on the LPMEOH™ Reactor, and scheduling the timing to implement the changes for in-situ catalyst activation and pre-treatment of the copper oxide-impregnated activated carbon in the 29C-40 catalyst guard bed.
- Reply to any comments from DOE on the revised update to the Demonstration Test Plan.
- Issue an update to the Topical Report on the Fall 1999 LPDME design verification test at the LaPorte AFDU.
- Conduct a Project Review Meeting with DOE.

F. Conclusion

During the month of October 2000, a new monthly production record of 2,500,000 gallons of methanol was set at the LPMEOH™ Demonstration Unit and nameplate production of 80,000 gallons per day (260 TPD) or higher was maintained for 23 consecutive days. The October monthly average production was 101% of nameplate. Production rates as high as 93,800 gallons per day (305 TPD), which corresponds to 117% of nameplate capacity, were also achieved for shorter periods during this month of record-breaking operation.

The LPMEOH™ Demonstration Unit operated at 98.11% availability during this quarter. A forced outage was taken on 18 November 2000 to repair a leak in a tube of the air-cooled condenser (29E-21) on the methanol rectifier column. The leaking tube was plugged and the exchanger was placed back in service without an environmental or safety incident. Total time for this forced outage was 41 hours. There were also three short syngas outages experienced which totaled 41.7 hours.

The major catalyst withdrawal and addition campaign begun in the previous quarter was completed during the month of October. The final batch of fresh catalyst was activated and added to the LPMEOH™ Reactor on 05 October 2000. After the final addition of catalyst, the total catalyst inventory was calculated to be 47,163 pounds.

During most of the quarter, the flowrate of Balanced Gas was controlled at an average flowrate of 775 KSCFH. The maximum instantaneous flowrate of Balanced Gas was over 1,000 KSCFH during the high production period in October 2000. During these operating periods, the reactor pressure was set an average of 710 psig and temperature was maintained at 235ºC.

On 15 November 2000, a period of operation on CO-rich syngas was performed. An average of 50 KSCFH of CO Gas was introduced with sufficient Balanced Gas to achieve a H_2:CO ratio at the reactor inlet of 1.0. This test ended with the forced outage that occurred on 18 November 2000.

After the restart on 20 November 2000, over 1,000 KSCFH of Balanced Gas was fed to the LPMEOH™ Demonstration Unit. Reactor temperature was increased from 235°C to 248°C during this period to allow for greater catalyst productivity and to lower the purge rate. This particular test was concluded on 22 November 2000, coincident with a syngas outage.

There was one extended period 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. An overall deactivation rate of 0.65% per day was calculated for the period 06 October to 26 October 2000 (21 days). This data was based on operation with the 29C-40 catalyst guard bed bypassed. The results of this data set is statistically similar to other recent periods of operation. This deactivation rate is greater than 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. Samples have continued to show an increase in arsenic, which has been demonstrated in the laboratory to act as a poison to methanol synthesis catalyst. Sulfur, another known catalyst poison, continues to be measured above the analytical detection limit. 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. The concentration of iron (another poison), is low (less than 200 ppmw), and does not seem to be increasing in the most recent samples. 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.

Eastman has been reviewing design options for both the in-situ activation of methanol synthesis catalyst in the LPMEOH™ Reactor and a proposed reuse of the copper oxide-impregnated activated carbon in the 29C-40 catalyst guard bed (background information on the catalyst guard bed can be found in Technical Progress Report No. 25). A design package has been developed by Eastman which defines the necessary modifications to the piping and

instrument systems. The timing for implementation of these changes must still be determined.

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.

During the reporting period, a total of 6,236,149 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, about 70.5 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. Final reports have been approved by Air Products for five of the seven projects.

Activities associated with Design Verification Testing of the LPDME Process have been completed. During the last quarter, comments were received from DOE on a draft Topical Report which presents the results of the design verification test at the LaPorte AFDU. A revision will be issued for final review and approval by January of 2001. A separate Topical Report on the market analysis for DME and review of the economics of the LPDME Process will be prepared by the LPMEOH™ Demonstration Project following the release of the DVT Topical Report.

During this reporting period, comments were received from DOE regarding an update to the Demonstration Test Plan. In this update, the tests which are planned for the remainder of the operating program were defined. Based upon this feedback, a second revision was prepared and sent to DOE for final review and approval.

One hundred percent (100%) of the $38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 31 December 2000. Sixty-eight percent (68%) of the $158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 December 2000.

APPENDICES

APPENDIX A - SIMPLIFIED PROCESS FLOW DIAGRAM

APPENDIX B - SAMPLES OF DETAILED MATERIAL BALANCE REPORTS

APPENDIX C - RESULTS OF DEMONSTRATION UNIT OPERATION

Table 1
Summary of LPMEOH™ Demonstration Unit Outages - October/December 2000

Operation Start	Operation End	Operating Hours	Shutdown Hours	Reason for Shutdown
10/1/00 00:00	10/27/00 20:30	644.5	30.0	Syngas Outage
10/29/00 02:30	11/18/00 09:30	487.0	41.0	29E-21 Tube Leak
11/20/00 02:30	11/20/00 02:30	0.0	7.0	Syngas Outage
11/20/00 09:30	11/22/00 19:00	57.5	4.7	Syngas Outage
11/22/00 23:44	12/31/00 23:59	936.2		End of Reporting Period
	Total Operating Hours		2125.2	
	Syngas Available Hours		2166.2	
	Plant Availability, %		**98.11**	

Table 2
Summary of Catalyst Samples - Second Catalyst Batch

Sample	Identity	XRD		BET	Analytical (ppmw)				
		Cu	ZnO	m2/g	Fe	Ni	S	As	Cl
K9804-1	Reduction Sample 4/2/98	72.5	84.9	105	23	11	<=110	<=12	
K9712-1	Transfer sample - 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
	Reactor Sample 1/19/00				205	10.0	432	1250	na
	Reactor Sample 3/2/00	187	88.7	67	137	8.2	226	1010	30
	Reactor Sample 4/23/00	175	114.5	59	164	6.6	248	1240	20
	Reactor Sample 7/18/00	174	107.5	69	166	na	349	1270	30
	Reactor Sample 8/31/00	385	90.4	66	167	na	379	1080	50
	Reactor Sample 11/7/00	248	79.6	70	169	na	237	1490	nd
	Reactor Sample 11/27/00	263	109.2		170	na	258	1470	20

Notes:

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

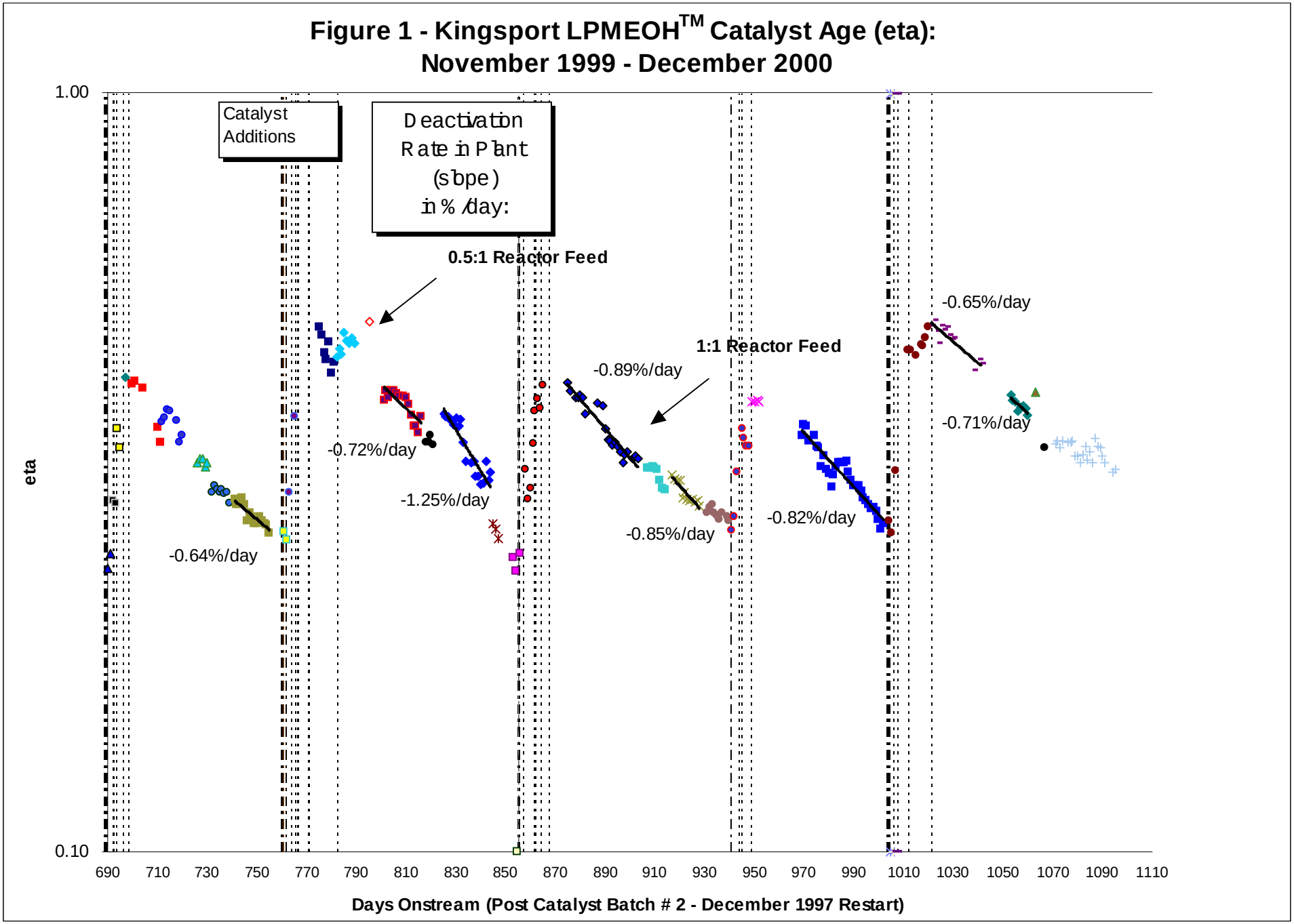
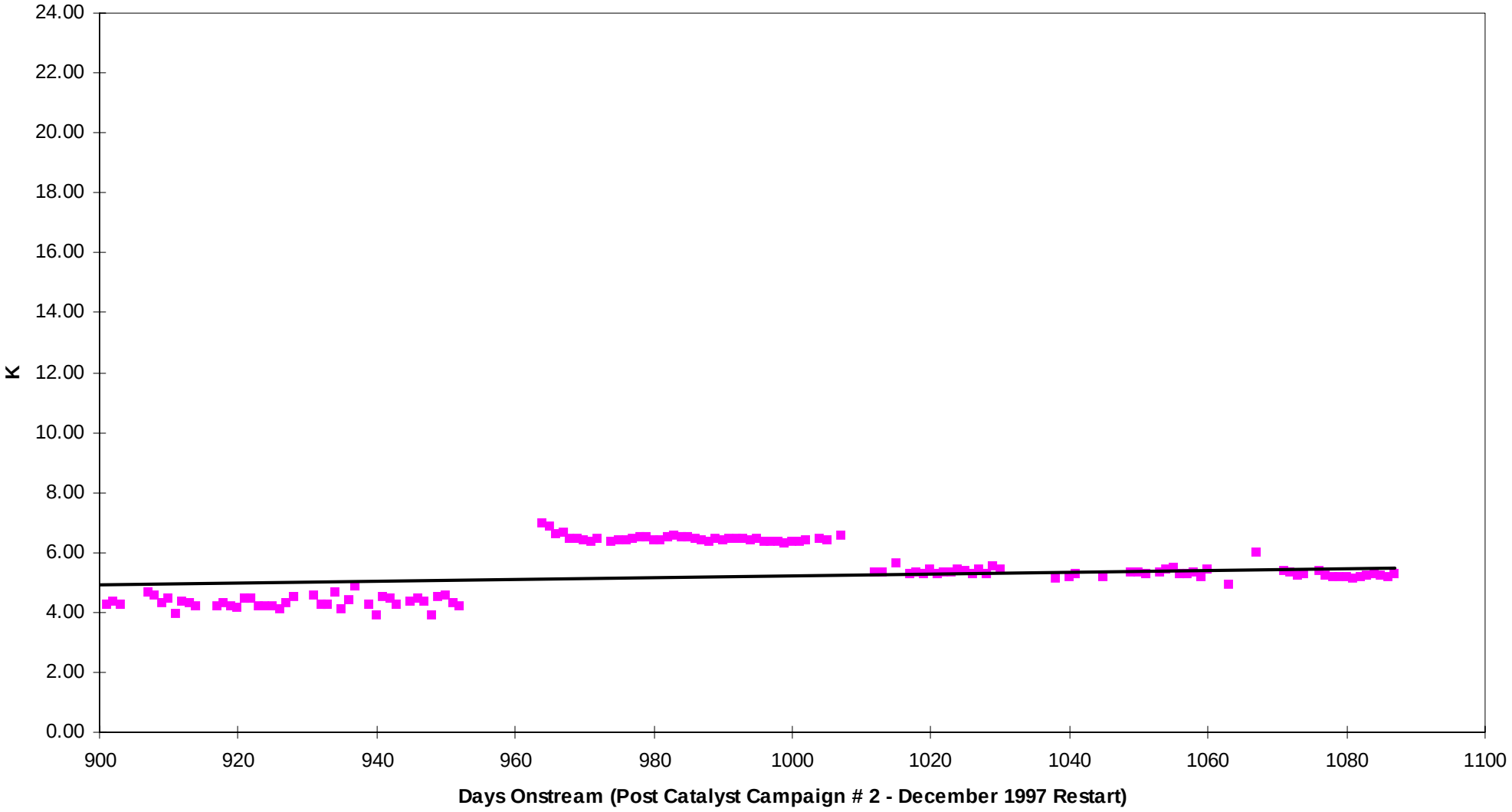


Figure 1 - Kingsport LPMEOH™ Catalyst Age (eta):
November 1999 - December 2000
1.00
0.10
eta
Catalyst Additions
Deactivation Rate in Plant (slope) in %/day:
0.5:1 Reactor Feed
1:1 Reactor Feed
-0.64%/day
-0.72%/day
-1.25%/day
-0.89%/day
-0.85%/day
-0.82%/day
-0.65%/day
-0.71%/day
690
710
730
750
770
790
810
830
850
870
890
910
930
950
970
990
1010
1030
1050
1070
1090
1110
Days Onstream (Post Catalyst Batch # 2 - December 1997 Restart)

Figure 2 - Kingsport LPMEOH™
Sparger Resistance Coefficient



APPENDIX D - MILESTONE SCHEDULE STATUS AND COST MANAGEMENT REPORTS