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

TECHNICAL PROGRESS REPORT NO. 20

For The Period

1 April - 30 June 1999

Prepared by

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and

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for the
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DISCLAIMER

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 throughout the quarter. During the reporting period, the flowrate of the primary syngas feed (Balanced Gas) averaged 577 KSCFH, and reactor temperature was held at 235°C. Several withdrawals of aged catalyst and additions of fresh activated catalyst were conducted during the quarter to maintain reactor productivity. As of 30 June 1999, the catalyst inventory in the reactor was calculated to be 55,274 pounds.

Catalyst activity, as defined by the ratio of the rate constant at any point in time to the rate constant for freshly reduced catalyst (as determined in the laboratory autoclave), was monitored throughout the reporting period. During a 27-day continuous operating period which ended on 15 June 1999, a stable catalyst deactivation rate of 0.551 %/day was achieved in the LPMEOH™ Reactor at a reactor temperature of 235°C. This deactivation result is slightly greater than the baseline deactivation rate from the 4-month proof-of-concept run at the LaPorte AFDU in 1988/89 (this run was performed at 250°C).

Analyses of catalyst samples have continued to show an increase in the concentration of arsenic. Beginning on 05 June 1999, the Eastman operations team began the preparations for changing the adsorbents in the two catalyst guard beds which treat the Balanced Gas stream; one vessel (upstream of the LPMEOH™ Demonstration Unit) was charged with arsine-removal adsorbent, and the guard bed within the battery limits of the LPMEOH™ Demonstration Unit was split between arsine and carbonyl removal materials. Both guard beds were back in service on 13 June 1999. After the guard beds were brought back online, the operating pressure for the LPMEOH™ Reactor had to be lowered by 10 psi to account primarily for higher pressure drop through both beds.

Sulfur continues to be measured on the catalyst above the analytical detection limit. Copper crystallite size measurements have shown an increase in the most recent samples. Levels of iron and nickel have remained low and steady since the restart in December of 1997.

The performance of the alternative 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, has met the expectations for pressure drop and reactor operation.

During the reporting period, a total of 3,787,589 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, about 38.4 million gallons of methanol has been produced. Eastman accepted all of this methanol for use in the production of methyl acetate, and ultimately cellulose acetate and acetic acid. No safety or environmental incidents were reported during this quarter.

During this quarter, planning, procurement, and test operations continued on the seven project sites selected for the off-site, product-use test program. Air Products received a recommendation from ARCADIS Geraghty & Miller that a gas turbine project should be redirected from VOC control to NO_x control. Testing of stabilized methanol as an emulsion fuel in a flight line generator was suspended due to loss of funding from other sources. Shakedown was completed on the small-scale reformer test apparatus which will test the viability of using fuel-grade methanol from the LPMEOH™ Demonstration Project as feedstock to a fuel cell.

During the reporting period, planning for a design verification test run of the Liquid Phase Dimethyl Ether (LPDME) Process at the LaPorte AFDU continued. Results of two autoclave experiments have provided an indication that an artifact exists within the autoclave apparatus when the LPDME catalyst system is operated at elevated slurry concentrations. At a review meeting for the DOE's Liquid Fuels Program on 09 June 1999, members of the LPMEOH™ Project Team from Air Products and DOE were given an update on the activities regarding the status of catalyst development and the economics for the LPDME Process. The participants agreed that the next test for the LPDME Process at the LaPorte AFDU should be treated as an interim campaign, with the primary objective being the determination of a tie-point between catalyst performance in the autoclave and the pilot plant scale. Shakedown activities at the LaPorte AFDU are scheduled to commence in September of 1999.

The LPMEOH™ Demonstration Unit served as the site for the plant tour which is associated with the Seventh Clean Coal Technology Conference (Knoxville, TN - 21-24 June 1999). Secretary of Energy Bill Richardson was the guest speaker at a reception which followed the tour. The paper entitled "Commercial-Scale Demonstration of the Liquid Phase Methanol (LPMEOH™) Process: Operating Experience Update" was presented at the Conference.

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An abstract for the paper entitled "Direct Applications of Stabilized Methanol from the Liquid Phase Methanol (LPMEOH™) Process" was submitted. This paper will be presented at the 16th Annual International Pittsburgh Coal Conference in Pittsburgh, PA (11-15 October 1999).

Ninety-nine percent (99%) 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 1999. Forty-one percent (41%) of the $158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 June 1999.

Table of Contents

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
Carbon Monoxide Gas	-	A syngas containing primarily carbon monoxide (CO); also called CO Gas
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-FETC	-	The DOE's Federal Energy Technology Center (Project Team)
DOE-HQ	-	The DOE's Headquarters - Coal Fuels and Industrial Systems (Project Team)
DTP	-	Demonstration Test Plan - The four-year 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)
MTBE	-	methyl tertiary butyl ether
MW	-	molecular weight, pound per pound mole
NEPA	-	National Environmental Policy Act
OSHA	-	Occupational Safety and Health Administration
ρ	-	density, pounds per cubic foot

ACRONYMS AND DEFINITIONS (cont'd)

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
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 Facility and the Eastman Facility
TPD	-	Ton(s) per Day
V	-	volumetric flowrate, thousand standard cubic feet per hour
VOC	-	volatile organic compound
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 will be 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 four-year 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 may also demonstrate the production of dimethyl ether (DME) as a mixed coproduct with methanol if laboratory- and pilot-scale research and market verification studies show promising results. If implemented, the DME would be produced during the last six months of the four-year demonstration period. 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 throughout the quarter. An extended complex-wide outage at Eastman's facility and two short duration syngas outages were the only operating interruptions experienced. During the reporting period, the flowrate of the primary syngas feed (Balanced Gas) averaged 577 KSCFH, and reactor temperature was held at 235°C.

Several withdrawals of aged catalyst and additions of fresh activated catalyst were conducted during this quarter to maintain reactor productivity. A fresh batch of catalyst, without withdrawal, was activated and added to the reactor on 03 April 1999. Two steps of catalyst withdrawal and addition were performed during the reporting period (17-19 May 1999, and 16-19 June 1999); as of 30 June 1999, the catalyst inventory in the reactor was calculated to be 55,274 pounds.

Catalyst activity, as defined by the ratio of the rate constant at any point in time to the rate constant for freshly reduced catalyst (as determined in the laboratory autoclave), was monitored throughout the reporting period. During a 27-day continuous operating period which ended on 15 June 1999, a stable catalyst deactivation rate of 0.551 %/day was achieved in the LPMEOH™ Reactor at a reactor temperature of 235°C. This deactivation result is slightly greater than the baseline deactivation rate from the 4-month proof-of-concept run at the LaPorte AFDU in 1988/89 (this run was performed at 250°C). During the quarter, there were no other stable operating periods of sufficient length (2 to 3 weeks) which are needed to quantify changes in catalyst activity with time.

Analyses of catalyst samples for changes in physical characteristics and levels of poisons have continued. As noted in Technical Progress Report No. 19, materials were ordered to increase the arsine removal capacity of the guard bed system. Beginning on 05 June 1999, the Eastman operations team began the preparations for changing the adsorbents in the two catalyst guard beds which treat the Balanced Gas stream; one vessel (upstream of the LPMEOH™ Demonstration Unit) was charged with arsine-removal adsorbent, and the guard bed within the battery limits of the LPMEOH™ Demonstration Unit was split between arsine and carbonyl removal materials. Both guard beds were back in service on 13 June 1999. After the guard beds were brought back online, the operating pressure for the LPMEOH™ Reactor had to be lowered by 10 psi to account primarily for higher pressure drop through both beds.

Sulfur continues to be measured on the catalyst above the analytical detection limit. Copper crystallite size measurements have shown an increase in the most recent samples. Levels of iron and nickel have remained low and steady since the restart in December of 1997.

The performance of the alternative 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 of the new sparger has met the design expectations for pressure drop and reactor operation.

During the reporting period, a total of 3,787,589 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, about 38.4 million gallons of methanol has been produced. Eastman accepted all of this methanol for use in the production of methyl acetate, and ultimately cellulose acetate and acetic acid. No safety or environmental incidents were reported during this quarter.

During this quarter, planning, procurement, and test operations continued on the seven project sites selected for the off-site, product-use test program. Air Products received a recommendation from ARCADIS Geraghty & Miller that a gas turbine project should be redirected from VOC control to NO_x control. Testing of stabilized methanol as an emulsion fuel in a flight line generator at Tyndall Air Force Base, Florida, was suspended due to the loss of funding from other sources. Shakedown was completed on the small-scale reformer test apparatus which will test the viability of using fuel-grade methanol from the LPMEOH™ Demonstration Project as feedstock to a fuel cell.

During the reporting period, planning for a design verification test run of the Liquid Phase Dimethyl Ether (LPDME) Process at the LaPorte AFDU continued. In one autoclave test with a commercially available dehydration catalyst, the LPDME catalyst system operated at the desired catalyst life at a total of 30 grams methanol and dehydration catalyst. A second experiment yielded a different aging pattern for the LPDME catalyst system. These results provide an indication that an artifact exists within the autoclave apparatus. At a review meeting for the DOE's Liquid Fuels Program on 09 June 1999, members of the LPMEOH™ Project Team from Air Products and DOE were given an update on the activities regarding the status of catalyst development and the economics for the LPDME Process. The participants agreed that the next test for the LPDME Process at the LaPorte AFDU should be treated as an interim campaign, with the primary objective being the determination of a tie-point between catalyst performance in the autoclave and the pilot plant scale. A draft letter which summarizes this change in objectives for the test at the LaPorte AFDU was sent to DOE for review. Shakedown activities at the LaPorte AFDU are scheduled to commence in September of 1999.

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A. Introduction

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This project is sponsored under the DOE's Clean Coal Technology Program, and its primary objective is to "demonstrate the production of methanol using the LPMEOH™ Process in conjunction with an integrated coal gasification facility." The project will also demonstrate 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 may also demonstrate the production of dimethyl ether (DME) as a mixed coproduct with methanol, if laboratory- and pilot-scale research and market verification studies show promising results. If implemented, the DME would be produced during the last six months of the four-year demonstration period.

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 10,000 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 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 is being made available for product-use tests. Product-use tests are targeted for an approximate 18 to 30-month period, and 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 (formerly Acurex Environmental Corporation), 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

During an evaluation period, eight sites involving a variety of product-use tests were selected to participate in this task. In a letter to the DOE dated 31 July 1997, Air Products formally recommended that seven of the eight projects had been defined in sufficient detail so that final planning and implementation should begin. DOE accepted Air Products' recommendation to proceed with the seven projects in August of 1997. The sites and project titles are listed in Appendix B-1. The eighth project, which involved the testing of a water/naphtha/methanol emulsion as a transportation fuel, was removed from the Product-Use Test Program during a review meeting between DOE, Air Products, and ARCADIS Geraghty & Miller.

All of the remaining product-use test projects are at varying phases of project planning, equipment procurement, and execution; two projects have been completed. Methanol produced from carbon monoxide (CO)-rich syngas at the LaPorte AFDU has been shipped to three of the project sites. Appendix B-2 through B-5 contain summary reports from the approved active projects. Highlights from these reports include:

Acurex Flexible-Fuel Vehicle (FFV) - The draft final report for this project was submitted to Air Products (no update in this reporting period).

Stationary Turbine for Volatile Organic Carbon (VOC) Control - ARCADIS Geraghty & Miller has indicated that it is unlikely that the California Energy Commission will provide the necessary support for a project to demonstrate VOC control from small gas turbines. However, the California Energy Commission is supporting the demonstration of a low-NO_x gas turbine for distributed power generation applications. Air Products received a recommendation from ARCADIS Geraghty & Miller that this project should be redirected from VOC control to NO_x control.

West Virginia University (WVU) Stationary Gas Turbine - Pratt and Whitney Aircraft has agreed to install four axially spaced thermocouples inside the combustion chamber of the gas turbine. This will be used in future computer modeling work on the turbine. Additional testing of fuel-grade methanol from the LPMEOH™ Demonstration Project is expected to commence during the next reporting period.

Aircraft Ground Equipment Emulsion - Testing of stabilized methanol as an emulsion fuel in a 110 horsepower flight line generator at Tyndall Air Force Base, Florida was suspended due to the loss of funding from other sources. A final report on the initial test results will be prepared.

University of Florida Fuel Cell - The small-scale reformer test apparatus is fully operational. The tests which will compare the behavior of chemical-grade methanol with fuel-grade methanol from the LPMEOH™ Demonstration Project are scheduled to be performed during the next reporting period.

West Virginia University Tri-Boro Bus - The draft final report for this project was submitted to Air Products (no update in this reporting period).

Florida Institute of Technology Bus & Light Vehicle - A final report on the results of this project is scheduled to be submitted to Air Products on 31 August 1999.

D.2 Commercialization Studies

Discussion

Several areas have been identified for development to support specific commercial design studies. These include: a) product purification options; b) feed gas impurity removal options; c) catalyst addition/withdrawal options; and d) plant design configuration options. Plant sizes in the range of 300 TPD to 1,800 TPD and plant design configurations for the range from 20% up to 70% syngas conversion will be considered. The Kingsport demonstration unit design and costs will be the basis for value engineering work to focus on specific cost reduction targets in developing the initial commercial plant designs.

The Process Economics Study - Outline has been prepared to provide guidance for the overall study work. The four part outline is included in Appendix C. This Outline addresses several needs for this Task 1.5.2 Commercialization Study:

a) to provide process design guidance for commercial plant designs.
b) to meet the Cooperative Agreement's technical objectives requirement for comparison with gas phase methanol technology. This preliminary assessment will help set demonstration operating goals, and identify the important market opportunities for the liquid phase technology.
c) to provide input to the Demonstration Test Plan (Task 2.3).
d) to provide input to the Off-Site Testing (Task 1.4) product-use test program.

Results

- Part One of the Outline - "Coproduction of Methanol" has been written for release as a Topical Report. The Topical Report entitled “Economic Analysis - LPMEOH™ Process as an Add-on to IGCC for Coproduction” was approved and issued for release on 18 February 1999.

- Part Two of the Outline - "Baseload Power and Methanol Coproduction", has been incorporated into the paper, "Fuel and Power Coproduction - The Liquid Phase Methanol (LPMEOH™) Process Demonstration at Kingsport", that was presented at the DOE's Fifth Annual Clean Coal Technology Conference in January of 1997.

- Part Three of the Outline - "Coproduction for Intermediate Electric Load Following", has been incorporated into the paper, "Dispatchable IGCC Facilities: Flexibility through Coproduction", that was presented at POWER-GEN EUROPE ’97 in June of 1997.

- Part Four of the Outline - "Methanol Fuel Applications", was used as the basis to update the product-use test program.

D.3 DME Design Verification Testing

Discussion

The first decision milestone, on whether to continue with dimethyl ether (DME) Design Verification Testing (DVT), was targeted for 01 December 1996. This milestone was relaxed to July of 1997 to allow time for further development of the Liquid Phase Dimethyl Ether (LPDME) catalyst system. DVT is required to provide additional data for engineering design and demonstration decision-making. The essential steps required for decision-making are: 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.

Prior work in this task included a recommendation to continue with DME DVT and Market Economic Studies. Ongoing activity is focusing on Laboratory R&D and preparations for the design verification test run at the LaPorte AFDU.

1997 DME DVT Recommendation

DOE issued a letter dated 31 July 1997 accepting Air Products' recommendation to continue with the design verification testing to coproduce DME with methanol, and to proceed with planning a design verification test run at the LaPorte AFDU. A copy of the recommendation (dated 30 June 1997) is included in Appendix D. The recommendation was based on the results of the Market Economic Studies and on the LPDME catalyst system R&D work, and is summarized in the following.

The Market Economic Studies show that the LPDME Process should have a significant economic advantage for the coproduction of DME with methanol for local markets. The studies show that the market applications for DME are large. DME is an ultra clean diesel fuel; and an 80% DME mixture with methanol and water is now being developed and tested by others. DME is a key intermediate in a commercial syngas-to-gasoline process, and is being developed as an intermediate for other chemicals and fuels. An LPDME catalyst system with reasonable long-term activity and stability has been developed from the laboratory R&D work.

Based upon the potential size of the markets and the promise of the LPDME catalyst system, design verification planning for the LaPorte AFDU was recommended. A summary of the DME DVT recommendation is:

- Planning for a DME DVT run at the LaPorte AFDU, in conjunction with other DOE Liquid Fuels Programs, should be initiated. Test plans, budgets, and a schedule for these LaPorte AFDU tests are under development. Up to $875,000 of Clean Coal

Technology Program budget support from the LPMEOH™ Project budget could be made available to support a suitable LPDME test run at LaPorte.

- An implementation decision, made mutually by the DOE's Clean Coal Technology Program (DE-FC22-92PC90543) LPMEOH™ project participants, and by the DOE's Liquid Fuels Program (DE-FC22-95PC93052) project participants, will be made in order to finalize the schedule for testing at LaPorte.

LPDME is not applicable to hydrogen (H_2)-rich syngas; and it is unlikely that a substantive LPDME demonstration will be recommended for Kingsport. Therefore, a convincing case that the test-run on CO-rich syngas at LaPorte will lead to successful commercialization must be made, prior to approving the final test-run plan. The strategy for commercialization must present the technical logic to combine the results of the following two areas:

1) catalyst performance (productivity, selectivity, and life) for the LPDME catalyst system under CO-rich syngas from the design verification testing at the LaPorte AFDU; and

2) reactor performance (methanol catalyst activity and life, hydrodynamics, and heat transfer) from the LPMEOH™ Process Demonstration Unit at Kingsport.

The DME DVT recommendation summarizes the catalyst targets, experimental results, and the corresponding economics for a commercially successful LPDME catalyst.

Market Economic Studies

Work on the feasibility study for the coproduction of DME and methanol with electric power has been completed. The product DME would be used as a domestic liquid cooking fuel, to replace imported Liquid Petroleum Gas, for China and the Pacific Rim regions. The results are included in the DME recommendation in Appendix D.

Laboratory R&D

Initially, synthesis of DME concurrently with methanol in the same reactor was viewed as a way of overcoming the syngas conversion limitations imposed by equilibrium in the LPMEOH™ Process. Higher syngas conversion would provide improved design flexibility for the coproduction of power and liquid fuels from an IGCC facility. The LPDME Process concept seemed ideally suited for the slurry-based liquid phase technology, since the second reaction (methanol to DME) could be accomplished by adding a second catalyst with dehydration activity to the methanol-producing reactor. Initial research work determined that two catalysts, a methanol catalyst and an alumina-based dehydration catalyst, could be physically mixed in different proportions to control the yield of DME and of methanol in the mixed product. These two commercially available catalysts comprise the LPDME catalyst system. Previously, proof-of-concept runs, in the laboratory and at the AFDU, confirmed that a higher syngas conversion could be obtained when a mixture of DME and methanol is produced in the liquid phase reactor.

Subsequent catalyst activity-maintenance experiments have shown the catalyst system utilized in the proof-of-concept runs experienced relatively fast deactivation compared to the LPMEOH™ process catalyst system. Further studies of the LPDME catalyst deactivation phenomenon, initially undertaken under the DOE's Liquid Fuels Program (Contract No. DE-FC22-95PC93052), was continued under this Task 1.5.3 through Fiscal Year 1996, and is now again being continued under the DOE Liquid Fuels Program. This LPDME catalyst deactivation research has determined that an interaction between the methanol catalyst and the dehydration catalyst is the cause of the loss of activity. Parallel research efforts--a) to determine the nature of the interaction; and b) to test new dehydration catalysts--was undertaken. In late 1995, the stability of the LPDME catalyst system was greatly improved, to near that of an LPMEOH™ catalyst system, when a new aluminum-based (AB) dehydration catalyst was developed. This new AB catalyst development showed that modification of the LPDME catalyst system could lead to long life.

Summary of Laboratory Activity and Results during Reporting Period

- Air Products performed laboratory autoclave tests of samples of the AB dehydration catalyst from the commercial catalyst manufacturer (Engelhard). The results to date have not been consistent, indicating that all issues related to catalyst scale-up have not been resolved. As a result, the decision was made within the DOE’s Liquid Fuels Program to delay the start of the AFDU design verification test. Changes to the commercial production procedure were made, and additional batches of dehydration catalyst were made and tested. These tests did not yield the desired catalyst aging characteristics.

- During an earlier reporting period, a set of experiments was performed on a commercially available dehydration catalyst to compare this material with the AB dehydration catalyst. These results showed that the desired catalyst life could be achieved with a commercially available dehydration catalyst at a 10-20% reduction in system productivity (primarily a reduction in the selectivity to DME).

- In one test with a commercially available dehydration catalyst, the LPDME catalyst system operated at the desired catalyst life at a total of 30 grams methanol and dehydration catalyst in the autoclave; this corresponds to a slurry concentration of 18 wt%. A second experiment yielded a different aging pattern for the LPDME catalyst system. These results provide an indication that an artifact exists within the autoclave apparatus. For example, loss of methanol synthesis and/or dehydration catalyst from the autoclave is interpreted as loss of activity of the LPDME catalyst system, as these activity calculations are performed assuming the presence of the initial charge of both catalysts. The loss of process oil may change the mass transfer resistance within the autoclave by increasing the viscosity of the slurry.

- Members of the LPMEOH™ Project Team from Air Products and DOE participated in a portion of the review meeting for the DOE’s Liquid Fuels Program on 09 June 1999. At this meeting, an update on the activities regarding the status of catalyst development and the economics for the LPDME Process were presented. The participants agreed that the next test for the LPDME Process at the LaPorte AFDU should be treated as an interim campaign, with the primary objective being the determination of a tie-point between catalyst performance in the autoclave and the pilot plant scale. Run time will also be provided to perform process variable scans on other operating conditions which are of potential commercial interest. A draft letter which summarizes this change in objectives for the test at the LaPorte AFDU was sent to DOE for review.

- Work is continuing in the laboratory autoclaves to finalize the test conditions for the run plan for the design verification test at the LaPorte AFDU. These experiments are expected to be concluded by 01 August 1999. Shakedown activities at the LaPorte AFDU are scheduled to commence in September of 1999. Operation of the LaPorte AFDU will then be completed by 30 November 1999, and a decision to include testing of the LPDME Process at the LPMEOH™ Demonstration Unit is scheduled to be made by 14 January 2000.

D.4 LPMEOH™ Process Demonstration Facility - Methanol Operation

Table D.4-1 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 E contains samples of the detailed material balance reports which are representative of the operation of the LPMEOH™ Demonstration Unit during the reporting period.

During the reporting period, a total of 3,787,589 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 safety or environmental incidents were reported during this quarter.

The LPMEOH™ Demonstration Unit operated at 100% availability throughout the quarter. An extended complex-wide outage at Eastman's facility and two short duration syngas outages were the only operating interruptions experienced. During the complex-wide outage, the catalyst inventory was held in the reactor until syngas was available. No issues were experienced in conjunction with the standby operation, and no major maintenance items were required for the LPMEOH™ Demonstration Unit during this complex-wide outage. Appendix F, Table 1 contains the summary of outages for the LPMEOH™ Demonstration Unit during this reporting period.

<u>Catalyst Life (eta) - April - June 1999</u>

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

Table D.4-1. Data Summary for LPMEOH™ Demonstration Unit

Case	Date	Days Onstream	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 Production (TPD)	Catalyst MeOH Prod. (gmol/hr-kg)	Reactor Vol. Prod. (TPD/Cu ft)	U Overall (BTU/hr ft2 F)	Sparger dP (psi)	Sparger Resistance ("K")
10	1-Apr-99	467	Balanced	235	709	632	2,282	3.67	131.4	0.68	3148	45.3	26.0	54.0	54,388	0.19	27.8	15.6	47.0	161.5	7.77	0.071	175	3.90	4.18
10	2-Apr-99	468	Balanced	235	710	650	2,261	3.57	137.8	0.67	3147	45.3	26.1	54.0	54,388	0.19	27.5	15.8	47.9	162.9	7.84	0.072	175	3.97	4.19
10	3-Apr-99	469	Balanced	235	710	606	2,236	4.38	84.5	0.66	2953	44.0	27.3	61.0	56,588	0.21	34.7	16.9	43.3	167.8	7.74	0.065	161	3.67	4.22
10	4-Apr-99	470	Balanced	235	710	653	2,201	4.24	108.1	0.66	2960	44.1	26.6	59.0	56,588	0.21	35.2	17.6	44.6	175.8	8.11	0.071	176	3.73	4.23
10	5-Apr-99	471	Balanced	235	710	653	2,217	4.09	100.5	0.66	3101	45.3	25.3	53.5	56,588	0.22	34.0	17.5	44.5	176.1	8.47	0.078	191	3.80	4.22
10	6-Apr-99	472	Balanced	235	710	651	2,218	4.09	103.9	0.66	2985	44.8	26.5	56.5	56,588	0.21	33.6	17.3	44.7	174.9	8.06	0.074	181	3.75	4.20
10	7-Apr-99	473	Balanced	235	710	654	2,225	4.07	109.3	0.67	2990	45.6	29.2	58.0	56,588	0.21	33.3	17.2	45.0	174.2	8.04	0.071	177	3.93	4.38
10	8-Apr-99	474	Balanced	235	710	652	2,188	4.07	120.8	0.66	2940	45.5	29.0	58.0	56,588	0.20	33.0	17.1	46.2	169.6	7.82	0.069	171	3.88	4.41
10	9-Apr-99	475	Balanced	235	710	645	2,149	3.93	122.2	0.65	2902	45.9	30.1	58.0	56,588	0.20	32.6	17.4	45.6	169.9	7.84	0.070	181	3.90	4.44
10	10-Apr-99	476	Balanced	235	710	641	2,181	3.98	117.8	0.65	2914	47.0	29.8	55.5	56,588	0.20	32.4	17.0	45.7	168.5	7.82	0.072	176	3.75	4.41
10	11-Apr-99	477	Balanced	235	709	653	2,199	3.72	104.0	0.66	2954	47.5	31.0	55.5	56,588	0.21	32.1	17.8	43.9	178.6	8.24	0.077	185	3.92	4.37
10	12-Apr-99	478	Balanced	235	709	651	2,249	3.80	116.0	0.67	3001	47.8	31.9	55.5	56,588	0.20	30.7	16.8	45.5	171.7	7.92	0.074	179	3.91	4.38
10	13-Apr-99	479	Balanced	234	709	651	2,263	3.48	108.6	0.67	3025	47.6	30.7	55.0	56,588	0.20	29.1	17.0	44.7	174.9	8.06	0.076	184	4.24	4.43
10	14-Apr-99	480	Balanced	235	710	535	2,304	3.89	45.5	0.66	2954	47.1	27.4	53.5	56,588	0.19	30.3	16.0	40.4	158.8	7.32	0.071	191	4.13	4.51
10	15-Apr-99	481	Balanced	235	711	535	2,303	3.80	44.2	0.65	2943	46.7	27.7	54.5	56,588	0.19	28.4	16.2	39.9	160.8	7.41	0.070	191	4.21	4.53
10	16-Apr-99	482	Balanced	235	710	510	2,313	3.90	48.4	0.65	2942	47.1	28.2	54.0	56,588	0.18	28.2	15.0	41.0	149.4	6.88	0.066	178	3.83	4.48
10	3-May-99	499	Balanced	235	711	554	2,234	3.39	69.7	0.65	2898	45.7	29.4	58.0	56,588	0.17	26.7	15.7	42.7	155.8	7.19	0.064	184	4.38	4.83
10	4-May-99	500	Balanced	235	711	601	2,156	3.21	106.7	0.64	2874	46.2	30.9	58.0	56,588	0.18	26.2	16.2	45.2	159.5	7.36	0.065	188	4.43	4.89
10	5-May-99	501	Balanced	235	710	607	2,122	3.34	110.1	0.63	2844	45.1	27.9	58.0	56,588	0.18	27.2	16.4	45.3	160.6	7.41	0.066	191	4.16	4.90
10	6-May-99	502	Balanced	235	710	610	2,308	3.20	102.7	0.68	3041	46.0	25.1	54.0	56,588	0.19	25.2	15.6	44.7	163.9	7.56	0.072	201	4.23	4.29
10	8-May-99	504	Balanced	235	710	587	2,219	2.87	88.1	0.65	2934	46.3	29.3	56.5	56,588	0.18	23.7	15.8	43.7	161.0	7.43	0.068	185	4.78	4.53
10	9-May-99	505	Balanced	235	710	587	2,198	2.97	87.6	0.65	2916	46.7	28.3	55.0	56,588	0.18	24.7	16.0	43.4	162.2	7.48	0.070	194	4.25	4.53
10	10-May-99	506	Balanced	235	710	593	2,176	3.13	103.1	0.64	2884	47.0	27.0	53.5	56,588	0.18	25.3	15.8	44.8	158.9	7.34	0.071	192	4.32	4.56
10	11-May-99	507	Balanced	235	710	596	2,121	3.09	99.6	0.63	2846	46.2	27.7	55.5	56,588	0.18	25.6	16.2	44.8	159.5	7.37	0.068	193	4.06	4.59
10	12-May-99	508	Balanced	235	709	592	2,143	3.01	99.2	0.64	2858	46.6	27.4	54.5	56,588	0.18	24.9	16.1	44.8	158.9	7.33	0.069	193	4.07	4.52
10	13-May-99	509	Balanced	235	709	581	2,177	2.92	99.2	0.64	2873	46.6	27.4	54.5	56,588	0.17	23.8	15.8	44.2	157.6	7.27	0.069	187	4.07	4.52
10	14-May-99	510	Balanced	235	710	585	2,161	2.82	104.7	0.64	2891	47.4	27.6	53.0	56,588	0.17	23.3	15.7	44.5	157.7	7.32	0.071	193	4.15	4.47
10	15-May-99	511	Balanced	235	709	588	2,179	2.73	107.1	0.64	2889	47.5	27.7	53.0	56,588	0.171	22.5	15.7	45.0	157.0	7.25	0.071	184	4.21	4.46
10	16-May-99	512	Balanced	235	709	588	2,177	2.74	111.0	0.65	2890	47.8	27.2	52.0	56,588	0.169	22.4	15.6	45.2	155.9	4.20	0.071	183	4.20	4.46
11	18-May-99	514	Balanced	235	709	546	2,191	2.69	107.0	0.64	3048	47.9	26.3	48.5	53,494	0.170	21.1	14.9	44.5	149.3	7.30	0.073	188	4.16	4.44
11	19-May-99	515	Balanced	235	710	566	2,193	3.72	63.8	0.64	2907	46.9	27.4	53.0	55,694	0.206	30.7	16.9	41.1	165.5	7.75	0.074	186	3.75	4.57
11	21-May-99	517	Balanced	235	710	575	2,173	3.65	57.4	0.63	2896	47.2	27.4	52.5	55,694	0.210	30.9	17.0	41.1	167.7	7.86	0.076	194	3.72	4.43
11	22-May-99	518	Balanced	235	710	554	2,228	3.17	63.8	0.64	2940	47.2	27.1	52.0	55,694	0.198	27.3	16.6	40.3	164.8	7.72	0.075	186	4.15	4.37
11	23-May-99	519	Balanced	234	710	558	2,207	3.10	48.6	0.64	2921	47.5	28.0	52.0	55,694	0.198	26.9	16.7	40.8	164.1	7.69	0.075	190	4.20	4.38
11	24-May-99	520	Balanced	235	709	560	2,208	3.06	52.0	0.64	2927	46.8	28.8	54.0	55,694	0.197	26.9	16.8	40.5	165.8	7.77	0.073	194	4.26	4.44
11	25-May-99	521	Balanced	235	710	564	2,182	3.24	63.5	0.63	2896	46.8	28.2	53.5	55,694	0.193	27.4	16.6	41.8	162.3	7.60	0.072	190	4.01	4.45
11	26-May-99	522	Balanced	235	710	566	2,187	3.21	68.3	0.64	2909	47.3	28.0	52.5	55,694	0.192	26.8	16.4	42.1	161.3	7.56	0.073	188	3.98	4.45
11	27-May-99	523	Balanced	235	710	569	2,195	3.24	51.0	0.64	2907	46.5	29.3	55.0	55,694	0.202	28.0	16.9	40.9	166.9	7.82	0.072	191	3.96	4.44
11	28-May-99	524	Balanced	235	710	561	2,204	3.19	50.3	0.64	2922	46.3	28.1	54.5	55,694	0.198	27.3	16.7	40.7	165.4	7.75	0.072	190	3.97	4.40
11	29-May-99	525	Balanced	235	710	565	2,155	3.32	55.5	0.63	2878	46.2	27.3	54.0	55,694	0.203	28.3	16.8	41.2	164.5	7.73	0.072	192	3.78	4.43
11	30-May-99	526	Balanced	234	710	566	2,178	3.25	55.9	0.63	2883	46.5	27.3	53.5	55,694	0.205	27.3	16.7	41.3	164.4	7.72	0.073	195	3.84	4.43
11	31-May-99	527	Balanced	234	710	562	2,172	3.11	54.4	0.63	2888	46.9	27.1	52.5	55,694	0.202	26.9	16.7	41.2	163.9	7.68	0.074	198	3.95	4.40
11	1-Jun-99	528	Balanced	234	710	566	2,142	3.41	68.0	0.63	2854	46.9	26.4	52.0	55,694	0.202	28.4	16.6	42.1	161.2	7.55	0.074	193	3.65	4.41
11	2-Jun-99	529	Balanced	235	710	562	2,129	3.43	72.4	0.62	2839	47.0	27.3	52.5	55,694	0.195	28.0	16.3	42.8	157.5	7.38	0.071	181	3.65	4.45
11	3-Jun-99	530	Balanced	235	710	565	2,141	3.26	65.9	0.63	2858	46.9	27.6	53.0	55,694	0.196	27.5	16.6	42.1	160.9	7.54	0.072	187	3.79	4.41
11	4-Jun-99	531	Balanced	235	710	567	2,153	3.12	67.8	0.63	2877	46.8	26.1	51.0	55,694	0.194	26.4	16.5	42.3	161.0	7.55	0.075	191	3.90	4.41
11	5-Jun-99	532	Balanced	235	710	565	2,162	2.93	65.2	0.63	2885	47.5	26.6	51.0	55,694	0.190	25.2	16.4	42.1	161.1	7.55	0.075	186	4.02	4.37
11	6-Jun-99	533	Balanced	235	711	560	2,147	3.03	67.3	0.63	2870	47.5	26.5	51.0	55,694	0.189	25.6	16.3	42.1	159.7	7.49	0.075	189	3.93	4.42
11	7-Jun-99	534	Balanced	235	711	560	2,146	2.97	69.8	0.63	2866	47.8	26.6	50.5	55,694	0.187	25.4	16.4	42.1	159.5	7.48	0.075	197	3.99	4.40
11	8-Jun-99	535	Balanced	235	710	561	2,154	2.89	69.4	0.63	2868	48.2	27.9	51.5	55,694	0.185	24.9	16.4	42.2	159.4	7.47	0.074	189	4.06	4.36
11	9-Jun-99	536	Balanced	235	710	562	2,141	2.75	67.0	0.63	2865	46.2	25.2	52.5	55,694	0.185	24.2	16.4	42.1	160.1	7.51	0.073	196	4.17	4.38
11	10-Jun-99	537	Balanced	235	710	550	2,151	2.70	66.9	0.63	2863	46.4	26.5	53.0	55,694	0.180	23.2	16.0	42.4	155.6	7.30	0.070	191	4.22	4.37
11	11-Jun-99	538	Balanced	235	705	559	2,112	2.91	77.9	0.62	2823	47.2	27.1	52.0	55,694	0.181	24.5	16.1	43.5	154.2	7.23	0.071	186	3.94	4.38
11	12-Jun-99	539	Balanced	235	704	554	2,093	3.51	86.8	0.62	2799	47.0	26.7	52.0	55,694	0.181	25.3	15.8	44.1	150.7	7.07	0.069	181	3.72	4.43
11	13-Jun-99	540	Balanced	235	706	559	2,140	2.75	82.0	0.63	2847	47.2	27.1	52.0	55,694	0.178	23.2	15.9	43.6	154.1	7.23	0.071	189	4.07	4.39
11	14-Jun-99	541	Balanced	235	705	554	2,109	3.01	87.5	0.62	2816	47.3	27.3	52.0	55,694	0.177	24.3	15.7	44.2	150.5	7.07	0.069	189	3.77	4.40
11	15-Jun-99	542	Balanced	235	705	507	2,130	3.21	56.6	0.62	2789	47.3	26.8	51.5	55,694	0.177	25.4	15.2	41.7	145.9	6.84	0.067	196	3.71	4.46
11	16-Jun-99	543	Balanced	235	705	497	2,160	3.11	68.0	0.62	2953	48.0	27.8	48.5	53,074	0.169	23.0	14.4	43.2	138.1	6.80	0.068	192	3.72	4.41

Table D.4-1. Data Summary for LPMEOH™ Demonstration Unit (continued)

Case	Date	Days Onstream	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 Production (TPD)	Catalyst MeOH Prod. (gmol/hr-kg)	Reactor Vol. Prod. (TPD/Cu ft)	U Overall (BTU/hr ft2 F)	Sparger dP (psi)	Sparger Resistance ("K")
11	17-Jun-99	544	Balanced	235	705	478	2,172	3.28	69.7	0.62	2943	48.3	25.3	46.5	53,074	0.162	22.5	13.6	44.1	130.1	6.40	0.067	177	3.58	4.44
11	19-Jun-99	546	Balanced	235	700	499	2,109	4.17	32.1	0.60	2715	45.7	27.2	54.5	55,274	0.210	32.2	16.0	41.0	145.8	6.89	0.064	190	3.16	4.52
11	20-Jun-99	547	Balanced	235	700	497	2,109	4.13	31.4	0.60	2716	45.7	27.2	54.5	55,274	0.215	32.5	16.3	40.2	148.4	7.01	0.065	200	3.16	4.54
11	23-Jun-99	550	Balanced	235	697	569	2,091	3.17	70.0	0.63	2828	46.5	27.3	53.0	55,274	0.197	27.7	17.1	42.5	160.7	7.59	0.072	185	3.94	4.41
11	24-Jun-99	551	Balanced	234	695	565	2,128	3.11	64.3	0.64	2871	47.0	27.3	52.0	55,274	0.203	27.6	16.1	42.0	161.6	7.63	0.074	184	4.07	4.43
11	25-Jun-99	552	Balanced	235	693	577	2,061	3.07	73.3	0.63	2816	46.9	27.0	52.0	55,274	0.193	26.9	17.1	43.0	161.0	7.61	0.074	191	3.90	4.49
11	26-Jun-99	553	Balanced	234	692	575	2,045	3.11	76.5	0.62	2788	46.6	26.7	52.5	55,274	0.198	27.6	17.4	42.7	161.6	7.64	0.073	205	3.86	4.45
11	28-Jun-99	555	Balanced	235	705	569	2,081	3.47	81.5	0.62	2817	47.7	27.0	50.5	55,274	0.196	28.2	16.4	43.6	156.7	7.41	0.074	196	3.45	4.47
11	29-Jun-99	556	Balanced	235	702	547	2,108	3.01	62.0	0.62	2835	47.2	27.6	52.0	55,274	0.185	25.5	16.3	42.0	156.2	7.38	0.072	189	3.92	4.51
11	30-Jun-99	557	Balanced	235	696	540	2,074	3.05	66.5	0.62	2797	46.8	25.8	51.5	55,274	0.182	25.8	16.4	42.0	154.4	7.30	0.071	194	3.84	4.50

constant for freshly reduced catalyst (as determined in the laboratory autoclave). Appendix F, Figure 1 plots log η versus days onstream from the restart in March 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.

Several withdrawals of aged catalyst and additions of fresh activated catalyst were conducted during this quarter to maintain reactor productivity. A fresh batch of catalyst, without withdrawal, was activated and added to the reactor on 03 April 1999. In anticipation of adding fresh catalyst, a catalyst withdrawal from the reactor was conducted on 17 May 1999. A fresh batch of catalyst was then activated and added to the reactor on 19 May 1999. A second catalyst withdrawal was conducted on 16 June 1999. A fresh batch of catalyst was then activated and added on 19 June 1999. As of 30 June 1999, the catalyst inventory in the reactor was calculated to be 55,274 pounds.

During a 27-day continuous operating period which ended on 15 June 1999, a stable catalyst deactivation rate of 0.551%/day was achieved in the LPMEOH™ Reactor at a reactor temperature of 235°C. This deactivation result is slightly greater than the baseline deactivation rate from the 4-month proof-of-concept run at the LaPorte AFDU in 1988/89 (this run was performed at 250°C). During the quarter, there were no other stable operating periods of sufficient length (2 to 3 weeks) which are needed to quantify changes in catalyst activity with time.

During the quarter, Balanced Gas flow averaged 577 KSCFH, and reactor temperature was held at 235°C. As noted in Technical Progress Report No. 19, there appears to be a weak correlation between this increased arsenic concentration and the rate of deactivation of the catalyst. Materials were ordered to increase the arsine removal capacity of the guard bed system. Beginning on 05 June 1999, the Eastman operations team began the preparations for changing the adsorbents in the two catalyst guard beds (the 10C-30 vessel, upstream of both the LPMEOH™ Demonstration Plant and the fixed-bed methanol plant, and the 29C-40 carbonyl guard bed) which treat the Balanced Gas stream; the 10C-30 was charged with arsine-removal adsorbent, and the 29C-40 was split between arsine and carbonyl removal materials. During this changeout, the LPMEOH™ Reactor was able to be maintained in service by way of bypass lines around each guard bed. The 10C-30 guard bed was returned to service on 11 June 1999 and the 29C-40 guard bed was placed in service on 13 June 1999. After the guard beds were brought back online, the operating pressure for the LPMEOH™ Reactor had to be lowered by 10 psi to account primarily for higher pressure drop through both beds.

Analyses of catalyst samples for changes in physical characteristics and levels of poisons have continued. Appendix F, Table 2 summarizes the results to date. The most recent concentration of arsenic on the methanol synthesis catalyst is 1,680 ppmw; the arsenic loading continues to slowly increase with time. Sulfur is present at about 300 ppmw, which is above the analytical detection limit. Copper crystallite size measurements have shown an increase in the most recent samples. Levels of iron and nickel have remained low and steady since the restart in December of 1997.

Sparger Resistance

During the March 1999 scheduled outage for the LPMEOH™ Demonstration Unit, the reactor gas sparger was replaced with a design which was developed by Air Products. Appendix F, Figure 4 plots the average daily sparger resistance coefficient for the period following this outage. The data for this plot, along with the corresponding average pressure drop, are also included in Table D.4-1. The performance to date of the new sparger has met the design expectations for pressure drop and reactor operation. The flow resistance will be monitored in order to determine the changes in performance with operating time.

D.5 Planning and Administration

The Seventh Clean Coal Technology Conference was held in Knoxville, TN (21-24 June 1999). The LPMEOH™ Demonstration Unit served as the site for the plant tour which was associated with the Conference. Secretary of Energy Bill Richardson was the guest speaker at a reception which followed the tour. The paper entitled "Commercial-Scale Demonstration of the Liquid Phase Methanol (LPMEOH™) Process: Operating Experience Update" was presented at the Conference. Both the site tour and the presentation were well received.

A Project Review Meeting was held in Knoxville, TN, on 25 June 1999. The results of the unit operation were reviewed, and a draft update of the Demonstration Test Plan was presented. The meeting agenda, extracts from the meeting handouts, and the meeting notes are included in Appendix G.

The Milestone Schedule Status Report and the Cost Management Report, through the period ending 30 June 1999, are included in Appendix H. These two reports show the current schedule, the percentage completion and the latest cost forecast for each of the Work Breakdown Structure (WBS) tasks. Ninety-nine percent (99%) 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 1999. Forty-one percent (41%) of the $158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 June 1999.

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.

An abstract for the paper entitled "Direct Applications of Stabilized Methanol from the Liquid Phase Methanol (LPMEOH™) Process" was submitted. This paper will be presented at the 16th Annual International Pittsburgh Coal Conference in Pittsburgh, PA (11-15 October 1999).

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 and monitoring the performance of the gas sparger in the reactor.
- Inspect the 29C-40 guard bed within the LPMEOH™ Demonstration Unit to determine the cause of the increased pressure drop across the vessel since the bed was split between arsine and carbonyl removal materials.
- Complete laboratory testing of the LPDME catalyst system and begin shakedown activities in preparation for a LPDME design verification test run at the LaPorte AFDU. Issue an updated DME DVT Recommendation to DOE.
- Continue execution of the Off-Site, Product-Use Test Program (Phase 1, Task 1.4).
- Conduct a Project Review Meeting with DOE.

F. Conclusion

The LPMEOH™ Demonstration Unit operated at 100% availability throughout the quarter. An extended complex-wide outage at Eastman's facility and two short duration syngas outages were the only operating interruptions experienced. During the reporting period, Balanced Gas flow averaged 577 KSCFH, and reactor temperature was held at 235°C.

Several withdrawals of aged catalyst and additions of fresh activated catalyst were conducted during this quarter to maintain reactor productivity. A fresh batch of catalyst, without withdrawal, was activated and added to the reactor on 03 April 1999. Two steps of catalyst withdrawal and addition were performed during the reporting period (17-19 May 1999, and 16-19 June 1999); as of 30 June 1999, the catalyst inventory in the reactor was calculated to be 55,274 pounds.

Catalyst activity, as defined by the ratio of the rate constant at any point in time to the rate constant for freshly reduced catalyst (as determined in the laboratory autoclave), was monitored throughout the reporting period. During a 27-day continuous operating period which ended on 15 June 1999, a stable catalyst deactivation rate of 0.551 %/day was achieved in the LPMEOH™ Reactor at a reactor temperature of 235°C. This deactivation result is slightly greater than the baseline deactivation rate from the 4-month proof-of-concept run at the LaPorte AFDU in 1988/89 (this run was performed at 250°C). During the quarter, there were no other stable operating periods of sufficient length (2 to 3 weeks) which are needed to quantify changes in catalyst activity with time.

Analyses of catalyst samples for changes in physical characteristics and levels of poisons have continued. As noted in Technical Progress Report No. 19, materials were ordered to increase the arsine removal capacity of the guard bed system. Beginning on 05 June 1999, the Eastman operations team began the preparations for changing the adsorbents in the two catalyst guard beds which treat the Balanced Gas stream; one vessel (upstream of the LPMEOH™ Demonstration Unit) was charged with arsine-removal adsorbent, and the guard bed within the battery limits of the LPMEOH™ Demonstration Unit was split between arsine and carbonyl removal materials. Both guard beds were back in service on 13 June 1999. After the guard beds were brought back online, the operating pressure for the LPMEOH™ Reactor had to be lowered by 10 psi to account primarily for higher pressure drop through both beds.

Sulfur continues to be measured on the catalyst above the analytical detection limit. Copper crystallite size measurements have shown an increase in the most recent samples. Levels of iron and nickel have remained low and steady since the restart in December of 1997.

The performance of the alternative 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 of the new sparger has met the design expectations for pressure drop and reactor operation.

During the reporting period, a total of 3,787,589 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, about 38.4 million gallons of methanol has been produced. Eastman accepted all of this methanol for use in the production of methyl acetate, and ultimately cellulose acetate and acetic acid. No safety or environmental incidents were reported during this quarter.

During this quarter, planning, procurement, and test operations continued on the seven project sites selected for the off-site, product-use test program. Air Products received a recommendation from ARCADIS Geraghty & Miller that a gas turbine project should be redirected from VOC control to NO_x control. Testing of stabilized methanol as an emulsion fuel in a flight line generator at Tyndall Air Force Base, Florida, was suspended due to the loss of funding from other sources. Shakedown was completed on the small-scale reformer test apparatus which will test the viability of using fuel-grade methanol from the LPMEOH™ Demonstration Project as feedstock to a fuel cell.

During the reporting period, planning for a design verification test run of the Liquid Phase Dimethyl Ether (LPDME) Process at the LaPorte AFDU continued. In one autoclave test with a commercially available dehydration catalyst, the LPDME catalyst system operated at the desired catalyst life at a total of 30 grams methanol and dehydration catalyst. A second experiment yielded a different aging pattern for the LPDME catalyst system. These results provide an indication that an artifact exists within the autoclave apparatus. At a review meeting for the DOE’s Liquid Fuels Program on 09 June 1999, members of the LPMEOH™ Project Team from Air Products and DOE were given an update on the activities regarding the status of catalyst development and the economics for the LPDME Process. The participants agreed that the next test for the LPDME Process at the LaPorte AFDU should be treated as an interim campaign, with the primary objective being the determination of a tie-point between catalyst performance in the autoclave and the pilot plant scale. A draft letter which summarizes this change in objectives for the test at the LaPorte AFDU was sent to

DOE for review. Shakedown activities at the LaPorte AFDU are scheduled to commence in September of 1999.

The LPMEOH™ Demonstration Unit served as the site for the plant tour which is associated with the Seventh Clean Coal Technology Conference (Knoxville, TN - 21-24 June 1999). Secretary of Energy Bill Richardson was the guest speaker at a reception which followed the tour. The paper entitled "Commercial-Scale Demonstration of the Liquid Phase Methanol (LPMEOH™) Process: Operating Experience Update" was presented at the Conference.

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An abstract for the paper entitled "Direct Applications of Stabilized Methanol from the Liquid Phase Methanol (LPMEOH™) Process" was submitted. This paper will be presented at the 16^{th} Annual International Pittsburgh Coal Conference in Pittsburgh, PA (11-15 October 1999).

Ninety-nine percent (99%) 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 1999. Forty-one percent (41%) of the $158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 June 1999.

APPENDICES

APPENDIX A - SIMPLIFIED PROCESS FLOW DIAGRAM

APPENDIX B - OFF-SITE TESTING (DEFINITION AND DESIGN)

Quarterly Reports:

Appendix B-2 - ARCADIS Projects (two pages):

- Stationary Turbine for VOC Control
Appendix B-3 - West Virginia University Stationary Gas Turbine (three pages)

Appendix B-5 - Florida Institute of Technology Bus & Light Vehicle (nine pages)

APPENDIX C - PROCESS ECONOMIC STUDY

Process Economics Study - Outline
(Draft - 3/31/97 - four pages)

and

LPMEOH™ Process Economics - for IGCC Coproduction
(Memo - 31 March 1997 - two pages)

APPENDIX D - DME DESIGN VERIFICATION TESTING

APPENDIX E - SAMPLES OF DETAILED MATERIAL BALANCE REPORTS

APPENDIX F - RESULTS OF DEMONSTRATION PLANT OPERATION

Table 1 - Summary of LPMEOH™ Demonstration Unit Outages - April/June 1999
Table 2 - Summary of Catalyst Samples - Second Catalyst Batch

Figure 1 - Catalyst Age (η): March - June 1999
Figure 2 - Sparger Resistance Coefficient vs. Days Onstream (March 1999 to June 1999)

Table 1
Summary of LPMEOH™ Demonstration Unit Outages - April/June 1999

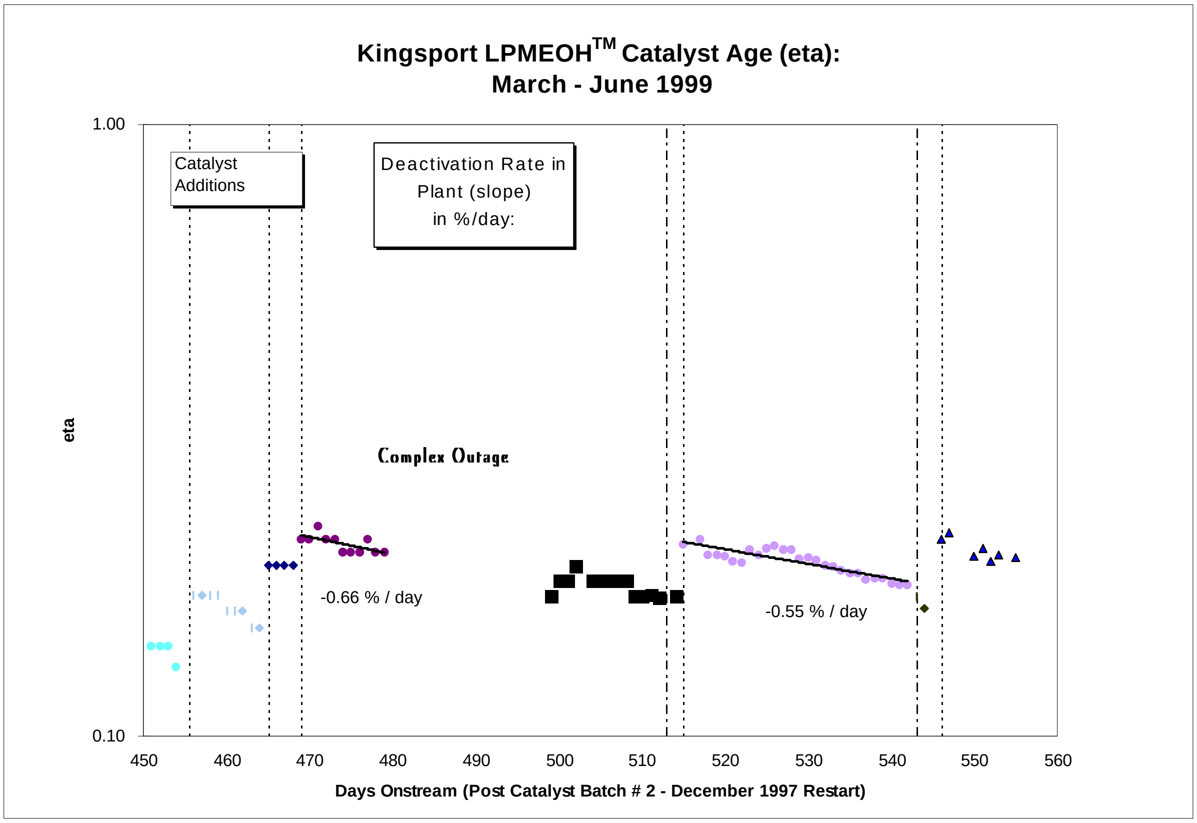
Operation Start	Operation End	Operating Hours	Shutdown Hours	Reason for Shutdown
7/1/99 00:00	7/30/99 01:40	697.7	1.0	Syngas Outage
7/30/99 02:40	8/7/99 17:15	206.6	22.7	Syngas Outage
8/8/99 16:00	8/9/99 11:00	19.0	12.5	Syngas Outage
8/9/99 23:30	9/30/99 23:59	1248.5		
	Total Operating Hours		2171.7	
	Syngas Available Hours		2171.7	
	Plant Availability, %		**100.00**	

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 - Alternative Catalyst	72.5	84.9	105	23	11	<=110	<=12	
K9712-1	Transfer sample from 29D-02 to Reactor	95.3	74		362	47.2	66.7	10.2	nd
K9712-2	Reactor Sample Day 1	100	123.8	75	92.1	<=18	<=167	<50	nd
K9712-3	Reactor Sample Day 4	130.9	64						
K9712-4	Reactor Sample Day 10	126.8	73.3	73	126	<=22	<=127	<50	nd
K9801-2	Reactor Sample 1/26/98	132.05	98.3		63.5	39.5	42.7	29.2	<100
K9802-1	Reactor Sample 2/3/98	141.1	91.5						
K9802-2	Reactor Sample 2/9/98	158.1	113						
K9802-3	Reactor Sample 2/15/98	145.7	91		67.1	36	<=97	209	
K9802-4	Reactor Sample 2/23/98	176.8	114.5						
K9803-2	Reactor Sample 3/10/1998	154.3	95.8	44	61.4	35.8	<=94	408	
K9803-4	Reactor Sample 3/29/98	169.6	87.9						
K9804-2	Reactor Sample 4/14/98	152.4	89.3		81.7	30.8	<=170	615	
K9805-2	Reactor Sample 5/11/98	219.2	109.6		73.15	35.85	163	538	
K9606-2	Reactor Sample 6/16/98	272.3	117.2		86.4	31.1	220	1110	
K9807-2	Reactor Sample 7/8/98	263.2	108.6		88.7	27.6	277	1045	
K9807-3	Reactor Sample 7/29/98	412*	112		93.25	30.95	209	1620	
K9807-4	Reactor Sample 8/14/98	353.9*	124		121.5	37.1	213.5	1215	
K9809-1	Reactor Sample 9/24/98	347.4	129.8		69.6	29.8	326	1149	
K9810-1	Reactor Sample 10/5/98	331.1	130.4						
K9811-2	Reactor Sample 11/25/98	293.9			57.3	23.4	264	1400	<100
K9812-1	Reactor Sample 12/29/98	283.1			72.3	20.4	260	1300	<100
K9901-1	Reactor Sample 1/15/99	252.5	61.4						
	Reactor Sample 2/17/99	474.7	133.6		82.6	22.2	385	1490	<300
	Reactor Sample 4/27/99	417.8	110.4	15	131	18.2	348	1460	<30
	Reactor Sample 6/1/99	517	105		109	19.7	316	1680	40

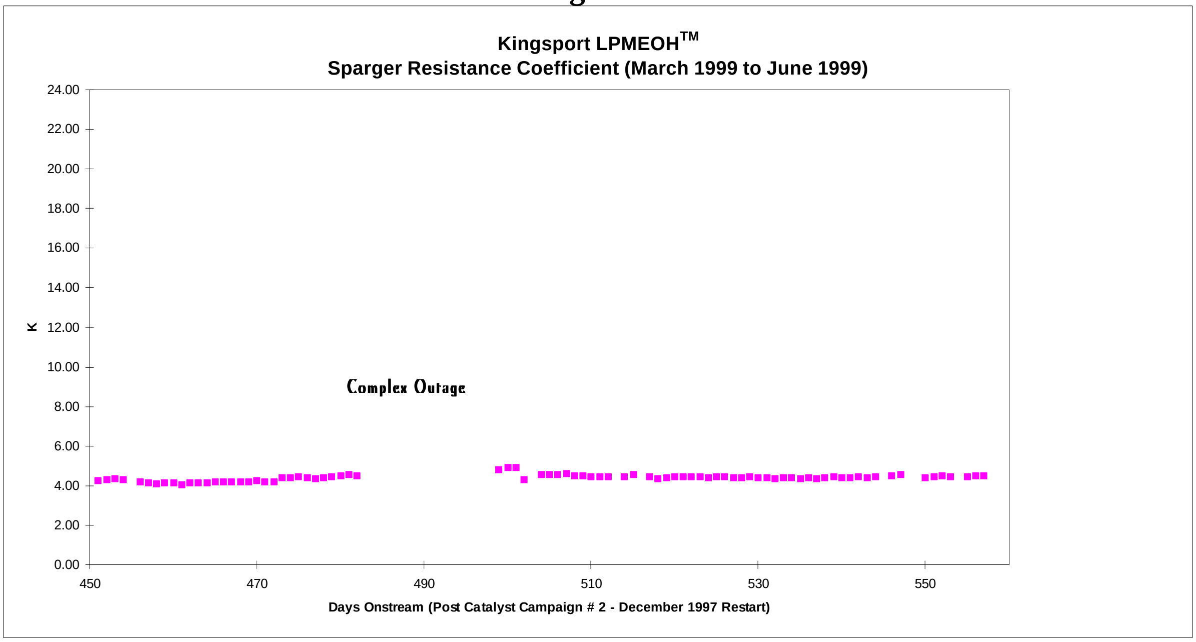
Notes:
1) nd = none detected
2) * - these values represent re-analysis of the sample as compared to Technical Progress Report no. 17

Figure 1



Kingsport LPMEOH™ Catalyst Age (eta):
March - June 1999
1.00
0.10
eta
Catalyst Additions
Deactivation Rate in Plant (slope) in %/day:
Complex Outage
-0.66 % / day
-0.55 % / day
450
460
470
480
490
500
510
520
530
540
550
560
Days Onstream (Post Catalyst Batch # 2 - December 1997 Restart)

Figure 2


Kingsport LPMEOH™
Sparger Resistance Coefficient (March 1999 to June 1999)
24.00
22.00
20.00
18.00
16.00
14.00
12.00
10.00
8.00
6.00
4.00
2.00
0.00
K
Complex Outage
450
470
490
510
530
550
Days Onstream (Post Catalyst Campaign # 2 - December 1997 Restart)

APPENDIX G - PROJECT REVIEW MEETING (25 JUNE 1999)

APPENDIX H - MILESTONE SCHEDULE STATUS AND COST MANAGEMENT REPORTS