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

TECHNICAL PROGRESS REPORT NO. 14

For The Period

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and

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for the
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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 complex in Kingsport.

During this quarter, initial planning and procurement work began on the seven project sites which have been accepted for participation in the off-site, methanol product-use test plan. Two of the projects have begun pre-testing of equipment, and three other projects have commenced with equipment procurement. Methanol produced from carbon monoxide (CO)-rich syngas at the Alternative Fuels Development Unit (AFDU) in LaPorte, TX has been shipped to four of the project sites in anticipation of the start of testing during the first quarter of calendar year 1998.

During the reporting period, planning for a proof-of-concept test run of the Liquid Phase Dimethyl Ether (LPDME) process at the LaPorte AFDU continued. A manufacturer for the dehydration catalyst was selected by the DOE's Liquid Fuels Program. This manufacturer has made acceptable dehydration catalyst in small-scale equipment.

Catalyst activity, as defined by 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), continued to decline more rapidly than expected. In response to concentrations of arsenic and sulfur detected on catalyst samples from the LPMEOH™ Reactor, Eastman replaced both the arsine- and sulfur-removal material in the Eastman guard bed which treats the primary syngas feed stream (Balanced Gas) prior to its introduction into both the Eastman fixed-bed methanol plant and the LPMEOH™ Demonstration Unit. After restarting the demonstration unit, the catalyst deactivation rate remained essentially unchanged. Parallel testing in the laboratory using arsine-doped, and subsequently arsine- and sulfur-doped syngas, also failed to prove that arsine was responsible for the higher-than-expected rate of catalyst deactivation in the demonstration unit.

Based on the results of plant operation and catalyst sampling, DOE accepted a recommendation by Air Products and Eastman to drain the initial charge of catalyst from the reactor and replace the charge with fresh catalyst. Prior to this catalyst turnaround, a final test was performed to determine the impact of raising the operating temperature of the LPMEOH™ Reactor from 250°C to 260°C.

A total of 90,800 pounds of catalyst slurry was drained from the LPMEOH™ Reactor in November of 1997. At the same time, the carbonyl guard bed within the battery limits of the LPMEOH™ Demonstration Unit was recharged with 6,300 pounds of fresh activated carbon. Activation of the new fresh charge of catalyst began on 13 November 1997. Just as

in the original start-up in April of 1997, only a partial charge of catalyst (20,700 pounds) was activated to limit the amount of material exposed to poisons at the outset. An attempted restart of the LPMEOH™ Demonstration Unit on 26 November 1997 was unsuccessful; settling of the fresh catalyst appeared to have occurred in the LPMEOH™ Reactor and gas inlet piping, which resulted in the plugging of the gas sparger at the bottom of the vessel.

During the reactor inspection in early December, Air Products developed an alternative gas sparger design based upon the operating experience to date. Eastman fabricated and installed the alternative gas sparger into the LPMEOH™ Reactor on 16 December 1997. The restart proceeded smoothly, and syngas was re-introduced to the LPMEOH™ Demonstration Unit at 22:50 on 19 December 1997. The initial operating temperature of 235°C was determined so that the nameplate capacity of 80,000 gallons-per-day (260 tons-per-day) of methanol could be achieved at a low syngas utilization (i.e. high overall conversion of syngas to methanol). This result confirmed the excellent initial activity of the catalyst. Because of the brief time onstream within this reporting period, no definitive conclusions can be made yet about the catalyst deactivation rate. However, the catalyst certainly did not exhibit the rapid decline in activity seen during an equivalent period onstream in the April 1997 operation.

The Alternative Fuels Field Test Unit (AFFTU), a transportable laboratory equipped with an autoclave and analytical equipment, was shipped from the Air Products' Iron Run laboratory in Allentown, PA to Kingsport to perform additional testing on the reactor feed gas at the site. Analytical results from the AFFTU showed less than 10 ppbv concentrations of metal carbonyls, hydrogen sulfide, and carbonyl sulfide at the gas inlet to the LPMEOH™ Reactor. Performance results of the catalyst in the autoclave were inconclusive during the brief span of operation in this reporting period.

Pressure drop and resistance coefficient across the gas sparger at the bottom of the reactor continued at a manageable level by flushing with entrained slurry collected at the cyclone and secondary oil knock-out drum. However, a new test was initiated to gravity-drain the condensed oil and entrained slurry continuously to the flush connection at the average rate of liquid traffic in the reactor loop (1 to 2 gallons per minute), thus eliminating the batch-transfer operating steps used in prior operation. Additional operating time should establish the long-term stability of the flow resistance coefficient in this operating mode.

During the reporting period, a total of 2,417,290 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, a total of 9,678,237 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 the October/November operation, slurry concentration in the reactor exceeded the design 40 wt% level for the first time, and the LPMEOH™ Demonstration Unit achieved its longest continuous operating campaign to date (756 hours).

A project review meeting was held at Kingsport on 16 and 17 December 1997. The meeting focused on reviewing the performance of the demonstration unit, the results of the analysis of the attempted restart of the unit in November of 1997, and discussion on the selection of the initial operating conditions for the restart in December of 1997.

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 31 December 1997. Fourteen percent (14%) of the $158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 December 1997.

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ACRONYMS AND DEFINITIONS

Acurex	Acurex Environmental Corporation
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 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 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 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.

During this quarter, initial planning and procurement work began on the seven project sites which have been accepted for participation in the off-site, product-use test plan. Two of the projects have begun pre-testing of equipment, and three other projects have commenced with equipment procurement. Methanol produced from carbon monoxide (CO)-rich syngas at the LaPorte AFDU has been shipped to four of the project sites in anticipation of the start of testing during the first quarter of calendar year 1998.

During the reporting period, planning for a proof-of-concept test run of the LPDME process at the Alternative Fuels Development Unit (AFDU) in LaPorte, TX continued. A manufacturer for the dehydration catalyst (Calsicat) was selected by the DOE's Liquid Fuels

Program. Three initial milestones in the scale-up of the dehydration catalyst have been achieved:

(1) the use of commercial-grade reagents in the manufacturing process are acceptable;
(2) Air Products has successfully transferred the catalyst manufacturing procedure to Calsicat; and
(3) Calsicat has made acceptable product in small-scale equipment.

Catalyst activity, as defined by 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), continued to decline more rapidly than expected. Catalyst slurry samples taken from the LPMEOH™ Reactor have continued to show an increase in the levels of iron, arsenic, and sulfur as compared with fresh catalyst. On 01 October, Eastman replaced both the arsine- and sulfur-removal material in the Eastman guard bed which treats the 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) prior to its introduction into both the Eastman fixed-bed methanol plant and the LPMEOH™ Demonstration Unit. After restarting the demonstration unit, the catalyst deactivation rate remained essentially unchanged. Parallel testing in the laboratory using arsine-doped, and subsequently arsine- and sulfur-doped syngas, also failed to prove that arsine was responsible for the catalyst deactivation in the plant.

Based on the results of plant operation and catalyst sampling, DOE accepted a recommendation by Air Products and Eastman to drain the initial charge of catalyst from the reactor and replace the charge with fresh catalyst. By starting with fresh catalyst, any effects of the presence of iron on the catalyst, which was detected shortly after start-up and had not increased appreciably since that time, would be eliminated. Prior to this catalyst turnaround, a final test was performed to determine the impact of raising the operating temperature of the LPMEOH™ Reactor from 250°C to 260°C.

A total of 90,800 pounds of catalyst slurry was drained from the LPMEOH™ Reactor in November of 1997. At the same time, the carbonyl guard bed within the battery limits of the LPMEOH™ Demonstration Unit was recharged with 6,300 pounds of fresh activated carbon. Activation of the new fresh charge of catalyst began on 13 November 1997. Just as in the original start-up in April of 1997, only a partial charge of catalyst (20,700 pounds) was activated to limit the amount of material exposed to poisons at the outset, while still producing the nameplate capacity of 80,000 gallons-per-day (260 tons-per-day) of methanol. An attempted restart of the LPMEOH™ Demonstration Unit on 26 November 1997 was unsuccessful; settling of the fresh catalyst appeared to have occurred in the LPMEOH™ Reactor and gas inlet piping, which resulted in the plugging of the gas sparger at the bottom of the vessel. In general, catalyst settling is greatest with fresh catalyst (largest particle size), and the rate of catalyst settling decreases rapidly with time in the reactor.

During the reactor inspection in early December, Air Products developed an alternative gas sparger design based upon the operating experience to date. Eastman fabricated and installed the alternative gas sparger into the LPMEOH™ Reactor on 16 December 1997. The restart proceeded smoothly, and Balanced Gas was re-introduced to the LPMEOH™ Demonstration Unit at 22:50 on 19 December 1997. A discussion on the philosophy for selecting the initial operating conditions for the LPMEOH™ Reactor was included in the agenda for a scheduled project review meeting between DOE, Air Products, and Eastman on 16 and 17 December 1997. Following the restart, the final operating temperature of 235°C maintained through the end of the reporting period was determined jointly by Air Products and Eastman so that the nameplate capacity of 80,000 gallons-per-day (260 tons-per-day) of methanol could be achieved at a low syngas utilization (i.e. high overall conversion of syngas to methanol). This result confirmed the excellent initial activity of the catalyst. Because of the brief time onstream within this reporting period, no definitive conclusions can be made yet about the catalyst deactivation rate. However, the catalyst certainly did not exhibit the rapid decline in activity seen during an equivalent period onstream in the April 1997 operation.

The Alternative Fuels Field Test Unit (AFFTU), a transportable laboratory equipped with an autoclave and analytical equipment, was shipped from the Air Products' Iron Run laboratory in Allentown, PA to Kingsport to perform additional testing on the reactor feed gas at the site. Analytical results from the AFFTU showed less than 10 ppbv concentrations of metal carbonyls and hydrogen sulfide at the gas inlet to the LPMEOH™ Reactor. Carbonyl sulfide was typically less than 10 ppbv at the same sampling location. Performance results of the catalyst in the autoclave were inconclusive during the brief span of operation in this reporting period.

Pressure drop and resistance coefficient across the gas sparger at the bottom of the reactor continued at a manageable level by flushing with entrained slurry collected at the cyclone and secondary oil knock-out drum. However, during this reporting period, a new test began to gravity-drain the condensed oil and entrained slurry continuously to the flush connection at the average rate of liquid traffic in the reactor loop (1 to 2 gallons per minute), thus eliminating the batch-transfer operating steps used in prior operation. After the restart of the LPMEOH™ Demonstration Unit on 19 December 1997, the resistance coefficient across the gas sparger operated at the design level using the gravity-drain concept. Additional operating time should establish the long-term stability of the flow resistance coefficient in this operating mode.

During the reporting period, a total of 2,417,290 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. During the October/November operation, slurry concentration in the reactor exceeded the design 40 wt% level for the first time; the LPMEOH™ Reactor operated in a stable hydrodynamic regime during this period, and the LPMEOH™ Demonstration Unit achieved its longest continuous operating campaign to date (756 hours).

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A. Introduction

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

This project is sponsored under the DOE's Clean Coal Technology Program, and its primary objective is to “demonstrate the production of methanol using the LPMEOH™ Process in conjunction with an integrated coal gasification facility.” The project 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 12,000 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, carbon monoxide gas, and balanced gas) will be 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 plan, developed in 1992 to support the demonstration at the original Cool Water Gasification Facility site, has become outdated. Since the site change to Eastman, the original product test plan under-represents new utility dispersed electric power developments, and possibly new mobile transport engine developments. The updated product-use test plan will attempt for broader market applications and for commercial fuels comparisons. The objective of the fuel-use test plan update will be 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 plan will be 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. Cost savings (10 to 15%) of several cents per gallon of methanol can be achieved, if the suitability of the stabilized product as a fuel can be demonstrated. The applications: 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.

A limited quantity (up to 400,000 gallons) of the methanol product as produced from the demonstration unit will be made available for product-use tests. Product-use tests will be targeted for an approximate 18 to 30-month period, commencing in the first year of demonstration operations. The methanol product will generally be available for shipment from the demonstration unit in Kingsport, Tennessee; methanol for some off-site tests may be shipped from the inventory held at the Alternative Fuels Development Unit (AFDU) in LaPorte, TX. Air Products, ARCADIS, Geraghty & Miller (formerly Acurex), and the DOE will develop the final off-site, product-use test plan.

Activity during this quarter

Eight sites involving a variety of product-use tests have been selected to participate in this task. The sites and project titles are listed in Appendix B-1. 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 eighth project, involving the testing of a water/naphtha/methanol emulsion as a transportation fuel, is awaiting final project definition.

All of the remaining product-use test projects have begun planning and equipment procurement. Methanol produced from carbon monoxide (CO)-rich syngas at the LaPorte AFDU has been shipped to four of the project sites. Appendix B-2 through B-8 contain summary reports from the approved projects. Highlights from these reports include:

Acurex Flexible Fuel Vehicle (FFV) - About 2,800 miles have been logged on the flexible fuel vehicle since end of September of 1997. During this initial period, M85 has been used to operate the automobile. Baseline emissions tests on M85 will commence after another 1,200 miles have been logged. At that point, the vehicle will operate on methanol supplied by the LPMEOH™ Demonstration Project.

Stationary Turbine for Volatile Organic Carbon (VOC) Control - ARCADIS, Geraghty & Miller has commenced activities to select the mini gas turbine technology that will be used in the demonstration, locate a host site, and secure co-funding for the project.

West Virginia University (WVU) Stationary Gas Turbine - The dual-fluid (methanol and jet fuel) selection and pressurization equipment has been purchased and installed. Fuel metering equipment has been ordered.

Aircraft Ground Equipment Emulsion - Planning activities have focused on identifying commercial vendors with water-fuel emulsion technologies previously tested in diesel engines. Initial discussions were held with representatives from Tyndall Air Force Base.

University of Florida Fuel Cell - Site preparations include the design of the gas analysis system and the upgrading of the fuel cell system.

West Virginia University Tri-Boro Bus - The analytical equipment on the WVU Heavy Duty Vehicle Emissions Laboratory is being improved to provide a more accurate measurement of carbon monoxide. The test program is planned for April of 1998.

Florida Institute of Technology Bus & Light Vehicle - The automobile is now operational after some refurbishment and installation of a new starting system. New fuel injectors have been ordered for the bus. Both units should be operational during the next reporting period.

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) front-end 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 plan update.

Activities during this quarter

- Part One of the Outline - "Coproduction of Methanol" has been written for release as a Topical Report. Comments from DOE on the 31 March 1997 draft of the Topical Report “Economic Analysis - LPMEOH™ Process as an Add-on to IGCC for Coproduction” are the current basis for discussion. As part of reviewing this report, Air Products has submitted a recommendation that the cost breakdown by plant area matches the format to be used in the Final Report - Volume 1 - Public Design. Once the area breakdown has been reviewed and accepted by DOE, the Topical Report on the Economic Analysis of LPMEOH™ will be updated and sent to DOE for further comment.

- 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 Four of the Outline - "Methanol Fuel Applications", is being used as the basis to update the product-use test plan (Task 1.4).

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. The DME Milestone Plan, showing the DVT work and the decision and implementation timing, is included in Appendix D.

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.

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 proof-of-concept test run at the DOE's AFDU in LaPorte, Texas. 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, proof-of-concept planning for the LaPorte AFDU was recommended. A summary of the DME DVT recommendation is:

- Planning for a DME test 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 should now be developed. 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 time to meet 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 proof-of-concept testing at the LaPorte AFDU; and

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

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 continued. 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 to date, 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 liquid phase DME (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. Previously, proof-of-concept runs, in the laboratory and at the Alternative Fuels Development Unit (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. During this quarter, laboratory work continued on developing an LPDME catalyst system based on the AB series of catalysts.

Summary of Laboratory Activity and Results

- A manufacturer for the dehydration catalyst (Calsicat) was selected by the Liquid Fuels Program. The initial schedule (contained in the DME Milestone Plan in Appendix D) showed a catalyst delivery date to the LaPorte AFDU of 01 March 1998. This date could be met assuming that the dehydration catalyst would be produced in a series of campaigns in a pilot plant. The Liquid Fuels Program has determined that it is important to complete the scale-up of the dehydration catalyst as part of the proposed LaPorte run. This will increase the time requirement, as a production test in the pilot plant is still required before operating the commercial catalyst production unit. The new estimated delivery date of dehydration catalyst to LaPorte is 01 June 1998. The DME DVT Recommendation will be updated to reflect the change in schedule and the impact (if any) on the implementation of the coproduction of DME with methanol at the LPMEOH™ Demonstration Unit.

- Three initial milestones in the scale-up of the dehydration catalyst have been achieved:

  (1) the use of commercial-grade reagents in the manufacturing process are acceptable;
  (2) Air Products has successfully transferred the catalyst manufacturing procedure to Calsicat; and
  (3) Calsicat has made acceptable product in small-scale equipment.

- LPDME laboratory tests using different feed gas compositions were continued for understanding the correlation between gas phase composition and catalyst deactivation.

D.4 LPMEOH™ Process Demonstration Facility - Methanol Operation

The summary table of performance data for the period 03 October through 02 November 1997 for the LPMEOH™ Demonstration Unit is included in Table D.4-1. Performance data from 20 December through 31 December 1997 are summarized in Table D.4-2. 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.

Appendix F, Table 1 contains the summary of outages for the LPMEOH™ Demonstration Unit. This table also calculates the availability of the LPMEOH™ Demonstration Unit for the reporting period.

During the reporting period, a total of 2,417,290 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. During the October/November 1997 operation, slurry concentration in the reactor exceeded the design 40 wt% level for the first time; the LPMEOH™ Reactor operated in a stable hydrodynamic regime during this period, and the LPMEOH™ Demonstration Unit achieved its longest continuous operating campaign to date (756 hours).

Operations focused on resolution of key issues identified during operations in the prior quarter.

Catalyst Life (eta) - October/November 1997

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 F, Figure 1 plots log η versus days onstream from the start-up in April of 1997 through 02 November 1997. 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.

During the April/May 1997 operating period, catalyst activity showed a much faster decline than prior experience at the LaPorte AFDU. Performance since the restart in late June, after the sparger inspection and cleaning during Eastman's complex-wide outage, confirmed that this decline was not induced by poor hydrodynamics related to the sparger performance. After that restart, the activity decrease slowed but remained faster than predicted. With some slight variations, the deactivation rate has remained relatively constant from June through November of 1997. At the beginning of October, an additional batch of fresh catalyst was activated in the catalyst reduction equipment and added to the LPMEOH™ Reactor. The increase in reactor performance roughly matched model predictions, and that last step-change is noted in Appendix F, Figure 1.

Table D.4-1

Case	Date	Gas Type	Temp (Deg C)	Pres. (psig)	Balanced Gas (KSCFH)	CO Gas (KSCFH)	H2 Gas (KSCFH)	Recycle 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")
6	3-Oct-97	Balanced	249	676	552	0	0	2,161	0.69	4847	41.0	40.7	49.0	33,300	0.28	34.1	17.6	39.8	166.4	13.02	0.081	143	15.23	17.11
6	4-Oct-97	Balanced	249	675	497	0	0	2,125	0.66	4671	39.2	39.5	51.5	33,300	0.28	35.1	17.2	38.4	155.2	12.15	0.072	146	15.08	17.88
6	5-Oct-97	Balanced	249	675	548	0	0	2,017	0.65	4584	40.1	38.0	48.5	33,300	0.28	34.4	18.2	40.2	163.6	12.80	0.080	141	17.92	22.81
6	6-Oct-97	Balanced	249	675	578	0	0	2,098	0.67	4749	40.0	38.2	49.0	33,300	0.27	31.5	18.2	40.8	170.0	13.31	0.083	139	15.37	17.23
6	7-Oct-97	Balanced	249	675	577	0	0	2,098	0.68	4766	40.2	38.0	48.5	33,300	0.27	29.9	18.2	40.6	170.5	13.35	0.084	140	16.12	17.47
6	8-Oct-97	Balanced	249	675	578	0	0	2,093	0.67	4763	40.9	37.1	46.5	33,300	0.27	30.0	18.0	40.9	169.3	13.25	0.087	142	14.49	16.14
6	9-Oct-97	Balanced	250	675	577	0	0	2,092	0.67	4753	37.0	40.9	57.5	33,300	0.27	30.2	18.3	40.6	170.6	13.36	0.071	135	13.97	15.19
6	10-Oct-97	Balanced	250	674	577	0	0	2,098	0.68	4768	37.0	40.9	57.5	33,300	0.26	29.7	18.1	40.9	169.3	13.25	0.070	136	13.37	14.35
6	11-Oct-97	Balanced	250	675	578	0	0	2,082	0.67	4743	38.1	39.8	54.0	33,300	0.26	29.7	18.0	41.1	168.6	13.20	0.074	140	13.01	14.31
6	12-Oct-97	Balanced	249	674	578	0	0	2,089	0.67	4750	38.2	40.5	54.5	33,300	0.26	28.9	18.0	41.2	168.1	13.16	0.073	143	13.28	14.25
6	13-Oct-97	Balanced	249	674	577	0	0	2,067	0.67	4698	38.2	39.4	53.5	33,300	0.25	29.3	17.5	42.8	161.7	12.66	0.072	142	12.83	14.66
6	14-Oct-97	Balanced	249	672	522	0	0	2,145	0.68	4760	40.5	42.0	51.0	33,300	0.24	27.2	16.7	40.4	154.8	12.12	0.072	154	14.08	14.85
6	15-Oct-97	Balanced	250	674	578	0	0	2,107	0.68	4785	39.0	39.6	52.0	33,300	0.24	26.7	17.1	43.2	160.7	12.58	0.074	144	14.45	15.03
6	16-Oct-97	Balanced	250	675	577	0	0	2,084	0.67	4747	38.7	39.4	52.5	33,300	0.24	27.6	17.3	42.7	162.1	12.69	0.073	147	13.34	14.44
6	17-Oct-97	Balanced	249	675	578	0	0	2,075	0.67	4717	39.5	39.0	50.5	33,300	0.24	28.1	17.2	43.1	160.9	12.59	0.076	146	12.54	14.36
6	18-Oct-97	Balanced	249	674	577	0	0	2,081	0.67	4742	39.5	38.9	50.5	33,300	0.24	27.1	17.4	42.5	163.2	12.78	0.077	146	12.72	13.68
6	19-Oct-97	Balanced	249	673	578	0	0	2,071	0.67	4733	39.9	39.3	50.0	33,300	0.24	26.6	17.3	42.8	161.8	12.67	0.077	149	12.79	13.66
6	21-Oct-97	Balanced	250	675	560	0	0	2,070	0.67	4692	40.7	38.6	48.0	33,300	0.21	26.5	15.9	45.6	147.2	11.53	0.073	149	12.73	14.49
6	22-Oct-97	Balanced	250	675	515	0	0	2,115	0.66	4687	41.5	39.8	47.5	33,300	0.21	25.4	15.8	42.7	144.7	11.33	0.073	152	12.88	14.09
6	23-Oct-97	Balanced	249	675	508	0	0	2,104	0.66	4662	41.6	38.9	46.5	33,300	0.20	24.9	15.5	43.1	141.6	11.09	0.073	152	12.61	13.85
6	24-Oct-97	Balanced	250	675	513	0	0	2,062	0.65	4596	40.2	39.4	49.5	33,300	0.21	24.8	15.9	43.0	143.2	11.22	0.069	150	12.98	14.58
28	25-Oct-97	Balanced	259	675	499	0	0	2,043	0.66	4540	40.3	37.2	48.0	33,300	0.21	29.6	16.7	40.2	149.0	11.67	0.074	145	12.12	14.74
28	26-Oct-97	Balanced	259	676	499	0	0	2,064	0.66	4583	39.7	38.3	50.0	33,300	0.21	28.7	16.8	39.7	150.9	11.81	0.072	150	12.77	14.70
28	27-Oct-97	Balanced	258	674	513	0	0	2,075	0.66	4598	41.7	42.4	49.5	33,300	0.20	28.0	16.5	41.6	148.0	11.59	0.071	159	13.15	15.03
28	28-Oct-97	Balanced	259	674	513	0	0	2,041	0.66	4550	40.8	39.1	48.5	33,300	0.19	28.0	16.2	42.5	144.8	11.34	0.071	157	12.31	14.42
28	29-Oct-97	Balanced	259	674	501	0	0	2,069	0.66	4568	41.6	38.4	46.5	33,300	0.19	26.9	16.1	41.3	145.4	11.38	0.075	156	13.12	14.93
28	30-Oct-97	Balanced	259	675	508	0	0	2,017	0.65	4501	40.2	38.2	49.0	33,300	0.19	28.0	16.0	42.8	142.7	11.17	0.069	153	11.66	14.24
28	31-Oct-97	Balanced	259	675	508	0	0	2,042	0.66	4534	40.4	36.8	47.5	33,300	0.18	27.2	15.6	43.5	140.2	10.97	0.070	152	12.16	14.70
28	1-Nov-97	Balanced	259	674	508	0	0	2,030	0.65	4512	41.4	38.4	47.0	33,300	0.18	27.1	15.7	43.5	140.4	10.99	0.071	155	12.27	14.99
28	2-Nov-97	Balanced	259	673	508	0	0	2,059	0.66	4566	41.5	38.2	46.5	33,300	0.18	26.4	15.5	43.4	140.4	11.00	0.072	159	12.59	15.08

Table D.4-2

Case	Date	Gas Type	Temp (Deg C)	Pres. (psig)	Balanced Gas (KSCFH)	CO Gas (KSCFH)	H2 Gas (KSCFH)	Recycle Gas (KSCFH)	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")
6	20-Dec-97	Balanced	225	680	772	0	0	2,193	66.1	0.71	8,994	27.3	49.3	59.0	19,500	1.24	44.4	23.3	39.3	236.0	31.53	0.095	171	3.98	3.90
6	21-Dec-97	Balanced	233	680	907	0	0	2,276	74.4	0.77	9,688	26.2	44.6	57.5	19,500	1.27	48.2	25.1	39.9	272.8	36.45	0.113	144	4.67	3.84
6	22-Dec-97	Balanced	235	680	908	0	0	2,250	71.6	0.77	9,587	29.0	51.6	57.6	19,500	1.34	50.9	25.9	39.5	275.8	36.85	0.114	146	4.67	3.79
6	23-Dec-97	Balanced	235	680	920	0	0	2,259	78.0	0.77	9,657	27.7	49.8	59.0	19,500	1.32	50.7	25.7	39.8	277.5	37.08	0.112	162	4.76	3.90
6	24-Dec-97	Balanced	235	680	921	0	0	2,288	82.0	0.77	9,558	27.5	49.8	59.5	19,500	1.31	48.6	25.6	39.8	277.7	37.10	0.111	166	5.21	4.31
6	25-Dec-97	Balanced	235	680	918	0	0	2,255	72.9	0.77	9,625	28.1	49.8	58.0	19,500	1.29	48.5	25.5	39.4	279.5	37.35	0.115	164	5.27	4.45
6	26-Dec-97	Balanced	235	680	929	0	0	2,276	84.4	0.77	9,664	28.9	50.0	56.0	19,500	1.27	48.2	25.4	39.9	279.0	37.28	0.118	163	5.32	4.46
6	27-Dec-97	Balanced	235	679	914	0	0	2,280	81.7	0.78	9,714	30.0	50.8	54.0	19,500	1.21	45.4	25.4	39.3	279.3	37.32	0.123	163	5.42	4.44
6	29-Dec-97	Balanced	235	680	919	0	0	2,199	94.6	0.76	9,462	30.7	51.9	53.5	19,500	1.23	48.7	24.9	40.8	270.7	36.18	0.120	159	4.93	4.51
6	31-Dec-97	Balanced	234	681	733	0	0	2,303	46.8	0.73	9,111	32.1	51.2	49.0	19,500	1.22	54.2	21.7	38.8	226.8	30.35	0.110	154	4.17	4.75

Earlier catalyst sampling from the LPMEOH™ Reactor revealed the presence of arsenic and sulfur on the catalyst. As reported in Technical Progress Report No. 13, preparations were made to replace both the arsine- and sulfur-removal material in the Eastman guard bed, which treats the Balanced Gas prior to its introduction into both the Eastman fixed-bed methanol plant and the LPMEOH™ Demonstration Unit. A total of 75 cubic feet of arsine-removal catalyst (manganese oxide) and 155 cubic feet of sulfur-removal catalyst (zinc oxide) were removed from the Eastman guard bed and replaced with fresh material.

Following the guard bed changeout, Balanced Gas was introduced to the LPMEOH™ Reactor on 03 October 1997. A catalyst sample from early November showed negligible changes in arsenic or sulfur loading since late September, confirming the effectiveness of the guard bed changeout. All catalyst samples analyzed between April and November of 1997 are summarized in Appendix F, Table 2. During October, however, the catalyst deactivation rate remained essentially unchanged. Parallel testing in the laboratory using arsine-doped, and subsequently arsine- and sulfur-doped syngas, also failed to prove that arsine was responsible for the catalyst deactivation in the plant.

Based on the results of demonstration unit operation (including the value of the flow resistance coefficient for the gas sparger in the LPMEOH™ Reactor) and catalyst sampling, DOE accepted a recommendation from Air Products and Eastman to drain the initial charge of catalyst from the reactor and replace the charge with fresh catalyst. By starting with fresh catalyst, any effects of the presence of iron on the catalyst (as reported in Technical Progress Report No.13), which was detected shortly after start-up and had not increased appreciably since that time, would be eliminated. Also, the full benefits of the changeout of the guard bed materials could be determined. Prior to this catalyst turnaround, a final test was performed to determine the impact of raising the operating temperature of the LPMEOH™ Reactor from 250°C to 260°C (Test 28 of the Demonstration Test Plan). As noted in Table D.4-1, the methanol productivity of the catalyst increased, indicating that the improvement in the rate of reaction was greater than the less favorable chemical equilibrium at the higher temperature.

After completion of Test 28, the LPMEOH™ Demonstration Unit was shut down in order to drain the spent catalyst from the process and begin preparations for activating a new charge of catalyst.

Plant Inspection and Catalyst Activation - November 1997

After the syngas feed was secured, catalyst slurry was pressure-transferred from the LPMEOH™ Reactor to the 29C-30 catalyst reduction vessel in a several batches. The utility oil system on the external jacket of the catalyst reduction vessel cooled each slurry batch below 120°F, the threshold at which operators would be required to wear burn-resistant personnel protective equipment while draining the slurry to drums. A total of 90,800 pounds of slurry was removed from the process for shipment to a metals reclaimer early in calendar year 1998.

After the system was drained, the LPMEOH™ Reactor was inspected by video camera. Dried catalyst slurry was observed in the bottom head and around the gas sparger. An attempt to flush and clean the bottom of the vessel with the gas sparger in place proved unsuccessful. The gas sparger was then removed, inspected, cleaned, and reinstalled. Apart from the catalyst, no other solid material was found in the bottom of the LPMEOH™ Reactor.

The demister pads were inspected at the vapor outlets of the 29C-05 secondary oil knock-out drum and the 29C-03 high-pressure methanol separator. Both devices were still positioned properly, and the demister for the secondary oil knock-out showed little evidence of catalyst accumulation on the screen.

On 11 November 1997, the 29C-40 carbonyl guard bed within the battery limits of the LPMEOH™ Demonstration Unit was recharged with 6,300 pounds of fresh activated carbon. Samples taken on 05 September 1997 showed a significant arsenic loading at the top of this bed, and it was decided to replace the material outright rather than risk stripping the arsenic off with clean syngas. Also, the inlet screen to the carbonyl guard bed has plugged routinely with debris since start-up. A new inlet screen was installed to improve accessibility for maintenance.

Activation of the new fresh charge of catalyst began on 13 November 1997. Just as in the original start-up in April of 1997, only a partial charge of catalyst (20,700 pounds) was activated to limit the amount of material exposed to poisons at the outset, while still producing the nameplate capacity of 80,000 gallons-per-day (260 tons-per-day) of methanol. This new charge of catalyst was prepared in eight separate batches instead of nine, as was the case in April; the first two batches were activated using the same quantity of catalyst (2,300 pounds) as in all prior reductions, while the final six batches were prepared using an additional 385 pounds. No other conditions within the catalyst activation procedure changed. Activation of all eight batches proceeded without incident and met the success criteria for properly activated catalyst. Representative results of the reductant gas uptake vs. temperature for batch # 6 are included in Appendix F, Figure 2.

Plant Restart - November 1997

On 26 November 1997, the first seven batches of freshly reduced catalyst slurry were pressure-transferred from the 29D-02 slurry storage tank to the LPMEOH™ Reactor. Nitrogen flow was introduced through the gas sparger prior to beginning the transfer operation, and the 29K-01 recycle compressor was started under nitrogen at the completion of the transfer. While this was occurring, the activation of the eighth batch of catalyst was proceeding in the catalyst reduction vessel. Heat-up of the reactor using 600 psig steam on the internal heat exchanger proceeded smoothly; however, the pressure drop at the bottom of the reactor increased slowly, from 1.4 psi to 2.1 psi over a four-hour period. When the gas sparger was flushed at the gas inlet piping connection using fresh oil from the oil storage tank, the pressure drop across the sparger rose to about 7 psi. Then, when the eighth batch of slurry was transferred to the reactor using the 29G-30 slurry transfer pump, the pressure drop rose to 14 psi. After this transfer, the recycle compressor began to approach surge, indicating significant blockage at the sparger. Attempts to clean the flow path were unsuccessful, and the contents of the reactor were pressure-transferred back to the slurry tank.

After completing the slurry transfer and securing the steam and syngas supplies, the manway on the bottom of the reactor was opened. The gas sparger was found to be plugged with fresh catalyst. In addition, a review of the data during the slurry transfer indicates that the nitrogen flow to the reactor was quite low, and that there was evidence that the gas inlet piping to the reactor had filled with slurry during the transfer operation. A sample of the catalyst found in the bottom of the reactor was analyzed for water, and less than 100 ppm by weight was detected (water at high concentration could cause the catalyst particles to agglomerate). Settling of the fresh catalyst appears to have occurred in the reactor and gas inlet piping, which resulted in the plugging of the gas sparger. In general, catalyst settling is greatest with fresh catalyst (largest particle size), and the rate of catalyst settling decreases rapidly with time in the reactor.

During the reactor inspection in early December, Air Products and Eastman discussed the operating experience with the gas sparger and reviewed some of the design changes under consideration by Air Products. Air Products then developed an alternative gas sparger design and provided a sketch to Eastman. Eastman fabricated and installed the new gas sparger on 16 December 1997.

Catalyst Life (eta) - December 1997

The fresh catalyst slurry was pressure-transferred from the slurry storage tank back to the LPMEOH™ Reactor on 19 December 1997. Pressure drop across the gas sparger remained less than 3 psi during the transfer and heat-up steps, indicating that catalyst was not settling. Balanced Gas was re-introduced to the LPMEOH™ Demonstration Unit at 22:50 on 19 December 1997.

One important change in the startup condition compared to the initial operation in April of 1997 was the operating temperature of the LPMEOH™ Reactor. Operating temperature was determined jointly by Air Products and Eastman so that the nameplate capacity of 80,000 gallons-per-day (260 tons-per-day) of methanol could be achieved at a low syngas utilization (i.e. high overall conversion of syngas to methanol). During the first 24 hours, a temperature of 230°C was required to meet this criteria. Shortly thereafter, the reactor temperature was raised to 235°C, where it remained for the balance of the reporting period. This result further confirmed the excellent initial activity of the catalyst, in that the design production capacity of methanol could be achieved at a lower reactor temperature and pressure than during the April 1997 operating period (refer to Technical Progress Report No. 12 for these results).

Results from the operating period encompassing 20 December through 31 December 1997 are included in Table D.4-2. Because of the brief time onstream within this reporting period, no definitive conclusions can be made yet about the catalyst deactivation rate. However, the catalyst certainly did not exhibit the rapid decline in activity seen during an equivalent period onstream in the April 1997 operation.

Alternative Fuels Field Trailer Unit (AFFTU) Results - December 1997

The Alternative Fuels Field Test Unit (AFFTU), a transportable laboratory equipped with an autoclave and analytical equipment, was shipped from the Air Products' Iron Run laboratory in Allentown, PA to Kingsport to perform additional testing on the reactor feed gas at the site. The autoclave operated in parallel with the demonstration unit on the same reactor feed gas, although at 250°C, the typical laboratory baseline condition. Analytical results from the AFFTU showed less than 10 ppbv concentrations of metal carbonyls and hydrogen sulfide at the gas inlet to the LPMEOH™ Reactor. Carbonyl sulfide was typically less than 10 ppbv at the same sampling location, but occasionally drifted higher; these excursions could not be correlated with any changes in the feed gas cleanup operations upstream of the LPMEOH™ Demonstration Unit. As with the demonstration unit, performance results of the catalyst in the autoclave were inconclusive during the brief span of operation in this reporting period. Analyses of catalyst samples collected during and after the run will be compared to results from the plant samples, and more definitive conclusions will be included in the next Technical Progress Report.

Sparger Resistance

As reported in Technical Progress Report No. 13, flow resistance through the gas sparger of the LPMEOH™ Reactor had been stabilized using a batch flush of condensed oil and entrained slurry from the 29C-05 secondary oil knock-out drum and 29C-06 cyclone. These streams were batch-transferred to the catalyst reduction vessel and returned to the reactor via the slurry transfer pump through a flush connection at the gas inlet line to the reactor. The flow rate of the flush was 30 gallons per minute, performed 2 to 3 times per day for approximately 15 minutes.

On 09 October 1997, a new test began to gravity-drain the condensed oil and entrained slurry continuously to the flush connection at the average rate of liquid traffic in the reactor loop (1 to 2 gallons per minute), thus eliminating the batch-transfer operating steps. An inventory of condensed oil was collected in the secondary oil knock-out drum, and the gravity-drain line to the flush connection was then placed in service. Level in the secondary oil knock-out drum dropped and the temperature at the bottom of the reactor fell slightly, confirming the flowing path of oil to the reactor despite the already high pressure drop through the gas sparger. During subsequent operation, the gravity-drain line became obstructed about twice per day. This blockage, either resulting from accumulation of catalyst or from vapor-locking within the piping system, cleared easily with a brief flush of clean oil from the 29D-30 oil storage tank via the 29G-03 oil makeup pump. At this frequency of operation, the amount of fresh oil added to the process during line flushing was less than the average oil loss rate with the methanol product (0.1 - 0.2 gallons per minute).

Operation in this mode continued until the shutdown of the LPMEOH™ Demonstration Unit on 02 November 1997. Appendix F, Figure 3 plots the average daily sparger resistance coefficient, K, since the restart of the LPMEOH™ Demonstration Unit after the complex-wide shutdown in June. (Note that K, as reported, contains an arbitrary factor to make the value more manageable, and therefore has meaning only in a relative sense.) The data for this plot, along with the corresponding average pressure drop, are included in Table D.4-1. As described in Technical Progress Report No. 13, the flow resistance coefficient had experienced a significant step-change increase during a week-long shutdown in early

September of 1997. During the gravity-drain operation, the flow resistance coefficient across the gas sparger showed some modest improvement, compared to the period in September when the condensed oil and entrained slurry were pumped batch-wise via the slurry transfer pump.

Part of the reasoning for draining the initial charge of catalyst slurry from the process in November of 1997 was to demonstrate that the operation of the gas sparger could be maintained at the design flow resistance coefficient by gravity-draining the condensed oil and entrained slurry to the flush connection at the gas inlet piping to the LPMEOH™ Reactor. Within 24 hours of restarting the LPMEOH™ Demonstration Unit in December of 1997, the gravity-draining flow path for this material was placed in service. Appendix F, Figure 4 plots the average daily sparger resistance coefficient since the restart. The data for this plot, along with the corresponding average pressure drop, are included in Table D.4-2. These early results are encouraging because the value of K has remained very stable, compared to the days following the plant restart in June of 1997 when K increased steadily with time onstream. Also, the frequency of obstruction in the gravity-drain line has decreased to once every 2 to 3 days, as opposed to about twice per day during the October 1997 trial. During interruptions in the supply of condensed oil and entrained slurry to the flush connection, the new gas sparger showed the same tendency for increasing pressure drop as the original design. However, once obstructions in the gravity-drain line are cleared with fresh oil from the oil makeup pump, the flow resistance coefficient for the gas sparger returns to the baseline value. Additional operating time should establish the long-term stability of the flow resistance coefficient in this operating mode.

D.5 On-Site Testing (Product-Use Demonstration)

Since the startup of the LPMEOH™ Demonstration Unit, Eastman has been monitoring the quality of the refined methanol as determined by a set of in-house fitness-for-use criteria. These criteria, as well as a summary of the results of the analysis of over 7.19 million gallons of refined methanol, are provided in Appendix G. Through 31 December 1997, a total of 24 samples of refined methanol have failed to meet one of the analytical tests. refined methanol is pumped from the methanol lot tanks within the battery limits of the LPMEOH™ Demonstration Unit to a primary methanol storage tank elsewhere within the Eastman complex. When results of a sample of refined methanol fall outside the fitness-for-use criteria, Eastman evaluates the impact of this material on the contents of this primary storage tank. As long as the properties of the methanol in this tank stay within the methanol specification, the lot of refined methanol can be accepted. All 24 lots of refined methanol which have failed to meet all of the fitness-for-use criteria have been accepted in this manner.

D.6 Planning and Administration

The third update of the Partnership Annual Operating Plan was prepared and submitted in November of 1997 (see Appendix H). The main goal and objective for this third annual plan is that the LPMEOH™ demonstration unit will have been successfully restarted after the catalyst changeout of November 1997 and have completed all Task 2.1.1 operation, and will have resolved those technical issues encountered during FY'97 operation (catalyst life, gas sparger resistance coefficient). Other objectives include completion of the operational proof-of-concept testing of the LPDME process at the LaPorte AFDU, and initial execution of the off-site product-use test program.

A project review meeting was held at Kingsport on 16 and 17 December 1997. The meeting focused on reviewing the performance of the demonstration unit, the results of the analysis of the attempted restart of the unit in November of 1997, and discussion on the selection of the initial operating conditions for the restart in December of 1997. A brief review of the catalyst development work for the upcoming LPDME proof-of-concept test at the LaPorte AFDU, the work concerning the on-site product-use testing, and the status of the off-site product-use test plan. The meeting agenda, extracts from the meeting handouts, and the meeting notes are included in Appendix I.

Work has continued on the Final Report - Volume 1 - Public Design. Air Products received the latest comments from DOE (letter dated 12 October 1997). In response to these comments, Air Products has submitted a cost breakdown by plant area for the equipment within the LPMEOH™ Demonstration Unit. In addition, as a follow-up to the December project review meeting, Eastman is preparing comments on the disclosure of operating costs from the Kingsport complex, and in particular the costs for the LPMEOH™ Demonstration Unit. Once these changes have been reviewed and approved by DOE, an updated version of this report will be issued for comment.

The Milestone Schedule Status Report and the Cost Management Report, through the period ending 31 December 1997, are included in Appendix J. 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 31 December 1997. Fourteen percent (14%) of the $158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 December 1997.

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

- Write and submit the Demonstration Technology Start-up Report to DOE.
- Continue to analyze catalyst slurry samples and gas samples to determine causes for deactivation of methanol synthesis catalyst.
- Continue executing Phase 3, Task 2.1 Methanol Operation per the Demonstration Test Plan. Update the Demonstration Test Plan based upon the most recent operating results.
- Reissue the DVT Recommendation for a DME proof-of-concept test run at the LaPorte AFDU to reflect the change in delivery date for the dehydration catalyst.
- Continue execution of the Off-Site, Product-Use Test Plan (Phase 1, Task 1.4).
- Continue to incorporate DOE comments into the Topical Report on Process Economic Studies.
- Reach agreement with DOE on the equipment breakdown and operating cost summary for use in the Final Technical Report, Volume 1, Public Design Report.
- Issue the Topical Report on Liquid Phase Reactor Design to DOE for review and comment.

F. Conclusion

During this quarter, initial planning and procurement work began on the seven project sites which have been accepted for participation in the off-site, product-use test plan. Two of the projects have begun pre-testing of equipment, and three other projects have commenced with equipment procurement. Methanol produced from carbon monoxide (CO)-rich syngas at the LaPorte AFDU has been shipped to four of the project sites in anticipation of the start of testing during the first quarter of calendar year 1998.

During the reporting period, planning for a proof-of-concept test run of the LPDME process at the Alternative Fuels Development Unit (AFDU) in LaPorte, TX continued. A manufacturer for the dehydration catalyst (Calsicat) was selected by the DOE's Liquid Fuels Program. Three initial milestones in the scale-up of the dehydration catalyst have been achieved:

(1) the use of commercial-grade reagents in the manufacturing process are acceptable;
(2) Air Products has successfully transferred the catalyst manufacturing procedure to Calsicat; and
(3) Calsicat has made acceptable product in small-scale equipment.

Catalyst activity, as defined by 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), continued to decline more rapidly than expected. Catalyst slurry samples taken from the LPMEOH™ Reactor have continued to show an increase in the levels of iron, arsenic, and sulfur as compared with fresh catalyst. On 01

October, Eastman replaced both the arsine- and sulfur-removal material in the Eastman guard bed which treats the 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) prior to its introduction into both the Eastman fixed-bed methanol plant and the LPMEOH™ Demonstration Unit. After restarting the demonstration unit, the catalyst deactivation rate remained essentially unchanged. Parallel testing in the laboratory using arsine-doped, and subsequently arsine- and sulfur-doped syngas, also failed to prove that arsine was responsible for the catalyst deactivation in the plant.

Based on the results of plant operation and catalyst sampling, DOE accepted a recommendation by Air Products and Eastman to drain the initial charge of catalyst from the reactor and replace the charge with fresh catalyst. By starting with fresh catalyst, any effects of the presence of iron on the catalyst, which was detected shortly after start-up and had not increased appreciably since that time, would be eliminated. Prior to this catalyst turnaround, a final test was performed to determine the impact of raising the operating temperature of the LPMEOH™ Reactor from 250°C to 260°C.

A total of 90,800 pounds of catalyst slurry was drained from the LPMEOH™ Reactor in November of 1997. At the same time, the carbonyl guard bed within the battery limits of the LPMEOH™ Demonstration Unit was recharged with 6,300 pounds of fresh activated carbon. Activation of the new fresh charge of catalyst began on 13 November 1997. Just as in the original start-up in April of 1997, only a partial charge of catalyst (20,700 pounds) was activated to limit the amount of material exposed to poisons at the outset, while still producing the nameplate capacity of 80,000 gallons-per-day (260 tons-per-day) of methanol. An attempted restart of the LPMEOH™ Demonstration Unit on 26 November 1997 was unsuccessful; settling of the fresh catalyst appeared to have occurred in the LPMEOH™ Reactor and gas inlet piping, which resulted in the plugging of the gas sparger at the bottom of the vessel. In general, catalyst settling is greatest with fresh catalyst (largest particle size), and the rate of catalyst settling decreases rapidly with time in the reactor.

During the reactor inspection in early December, Air Products developed an alternative gas sparger design based upon the operating experience to date. Eastman fabricated and installed the alternative gas sparger into the LPMEOH™ Reactor on 16 December 1997. The restart proceeded smoothly, and Balanced Gas was re-introduced to the LPMEOH™ Demonstration Unit at 22:50 on 19 December 1997. A discussion on the philosophy for selecting the initial operating conditions for the LPMEOH™ Reactor was included in the agenda for a scheduled project review meeting between DOE, Air Products, and Eastman on 16 and 17 December 1997. Following the restart, the final operating temperature of 235°C maintained through the end of the reporting period was determined jointly by Air Products and Eastman so that the nameplate capacity of 80,000 gallons-per-day (260 tons-per-day) of methanol could be achieved at a low syngas utilization (i.e. high overall conversion of syngas to methanol). This result confirmed the excellent initial activity of the catalyst. Because of the brief time onstream within this reporting period, no definitive conclusions can be made yet about the catalyst deactivation rate. However, the catalyst certainly did not exhibit the rapid decline in activity seen during an equivalent period onstream in the April 1997 operation.

The Alternative Fuels Field Test Unit (AFFTU), a transportable laboratory equipped with an autoclave and analytical equipment, was shipped from the Air Products' Iron Run laboratory in Allentown, PA to Kingsport to perform additional testing on the reactor feed gas at the site. Analytical results from the AFFTU showed less than 10 ppbv concentrations of metal carbonyls and hydrogen sulfide at the gas inlet to the LPMEOH™ Reactor. Carbonyl sulfide was typically less than 10 ppbv at the same sampling location. Performance results of the catalyst in the autoclave were inconclusive during the brief span of operation in this reporting period.

Pressure drop and resistance coefficient across the gas sparger at the bottom of the reactor continued at a manageable level by flushing with entrained slurry collected at the cyclone and secondary oil knock-out drum. However, during this reporting period, a new test began to gravity-drain the condensed oil and entrained slurry continuously to the flush connection at the average rate of liquid traffic in the reactor loop (1 to 2 gallons per minute), thus eliminating the batch-transfer operating steps used in prior operation. After the restart of the LPMEOH™ Demonstration Unit on 19 December 1997, the resistance coefficient across the gas sparger operated at the design level using the gravity-drain concept. Additional operating time should establish the long-term stability of the flow resistance coefficient in this operating mode.

During the reporting period, a total of 2,417,290 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. During the October/November operation, slurry concentration in the reactor exceeded the design 40 wt% level for the first time; the LPMEOH™ Reactor operated in a stable hydrodynamic regime during this period, and the LPMEOH™ Demonstration Unit achieved its longest continuous operating campaign to date (756 hours).

A project review meeting was held at Kingsport on 16 and 17 December 1997. The meeting focused on reviewing the performance of the demonstration unit, the results of the analysis of the attempted restart of the unit in November of 1997, and discussion on the selection of the initial operating conditions for the restart in December of 1997.

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 31 December 1997. Fourteen percent (14%) of the $158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 December 1997.

APPENDICES

APPENDIX A - SIMPLIFIED PROCESS FLOW DIAGRAM

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

Appendix B-1 - Summary Table of Eight Candidates (one page)

Quarterly Reports:

Appendix B-2 - Acurex FFV (one page)
Appendix B-3 - Stationary Turbine for VOC Control (two pages)
Appendix B-4 - West Virginia University Stationary Gas Turbine (two pages)
Appendix B-5 - Aircraft Ground Equipment Emulsion (four pages)
Appendix B-6 - University of Florida Fuel Cell (one page)
Appendix B-7 - West Virginia University Tri-Boro Bus (one page)
Appendix B-8 - Florida Institute of Technology Bus & Light Vehicle (twelve 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 Catalyst Samples

Sample	Identity	XRD		BET	Analytical (ppm)				
		Cu	ZnO	m2/g	Fe	Ni	S	As	Cl
14987-54	Lab run using 383-4119 (450 hours)	175	74						
Trailer Run	AFFTU run in Kingsport (672 hours)	179	101		172	58	<=660	184	5570
Reduction #3	Reduction Batch sample from Kingsport	73	55	57	49	32	<=110	<25	
Reduction #4	Reduction Batch sample from Kingsport			73	83	28	<100	<25	
Reduction #6	Reduction Batch sample from Kingsport			90	29	18	<=150	<25	
Reduction #8	Reduction Batch sample from Kingsport			81	26	23	<=110	<25	
K0597-2	Reactor Sample 6/15/97 (30 days)	274	89	40	281	61	<=190	446	<200
K0897-1	Reactor Sample 8/19/97	283	87	43	169	<20	235	601	
K0997-1	Reactor Sample 9/5/97	281	118	42	261	37	575	779	
K1097-1	Reactor Sample 9/29/97	289	187	45	189	28	330	711	
K1197-1b	Reactor Sample 11/7/97	292	111	40	194	37	340	699	

APPENDIX G - ON-SITE PRODUCT-USE TESTING

APPENDIX H - PARTNERSHIP ANNUAL PLAN
(For FY'98)

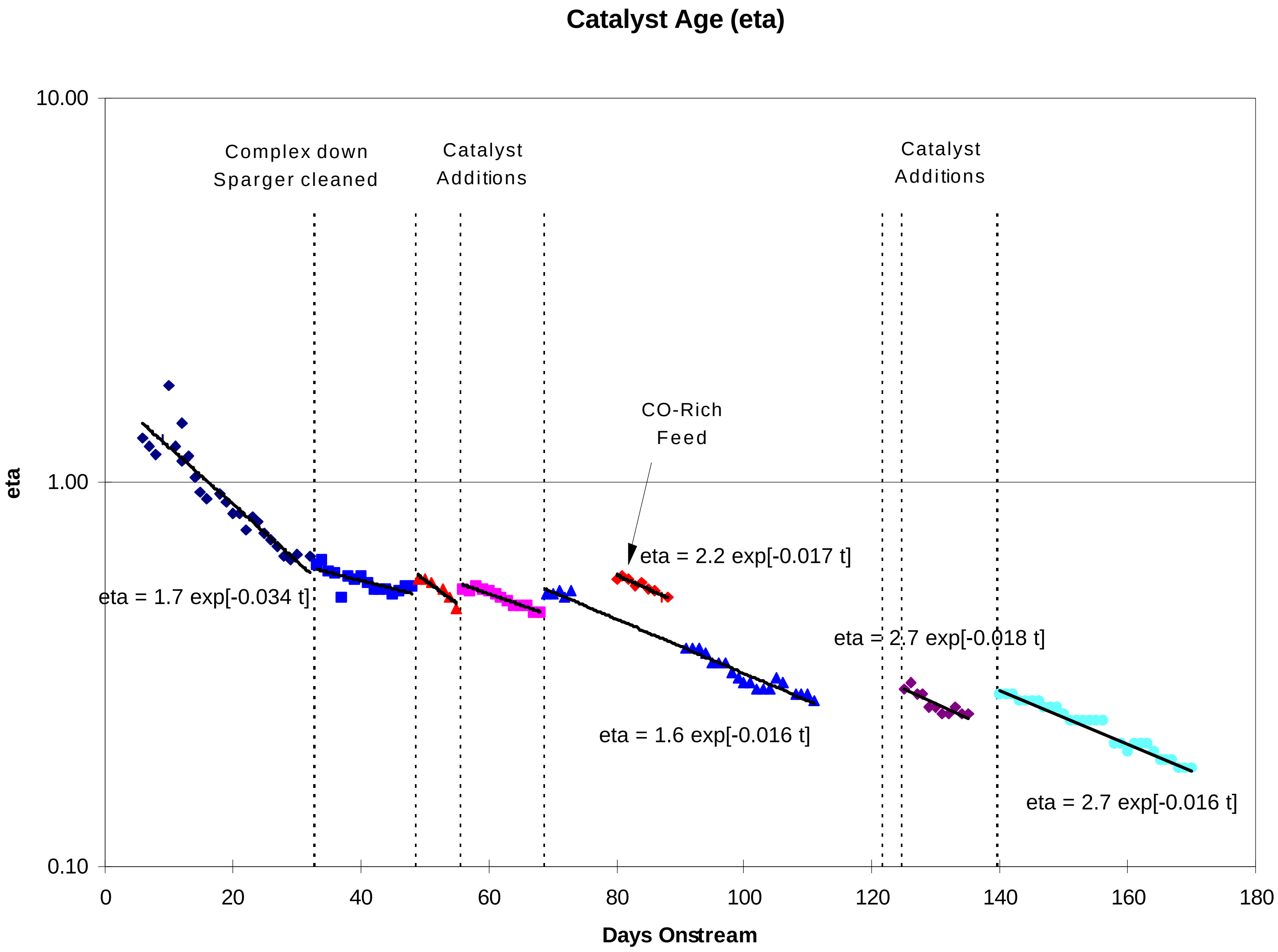
APPENDIX I - PROJECT REVIEW MEETING (16-17 DECEMBER 1997)

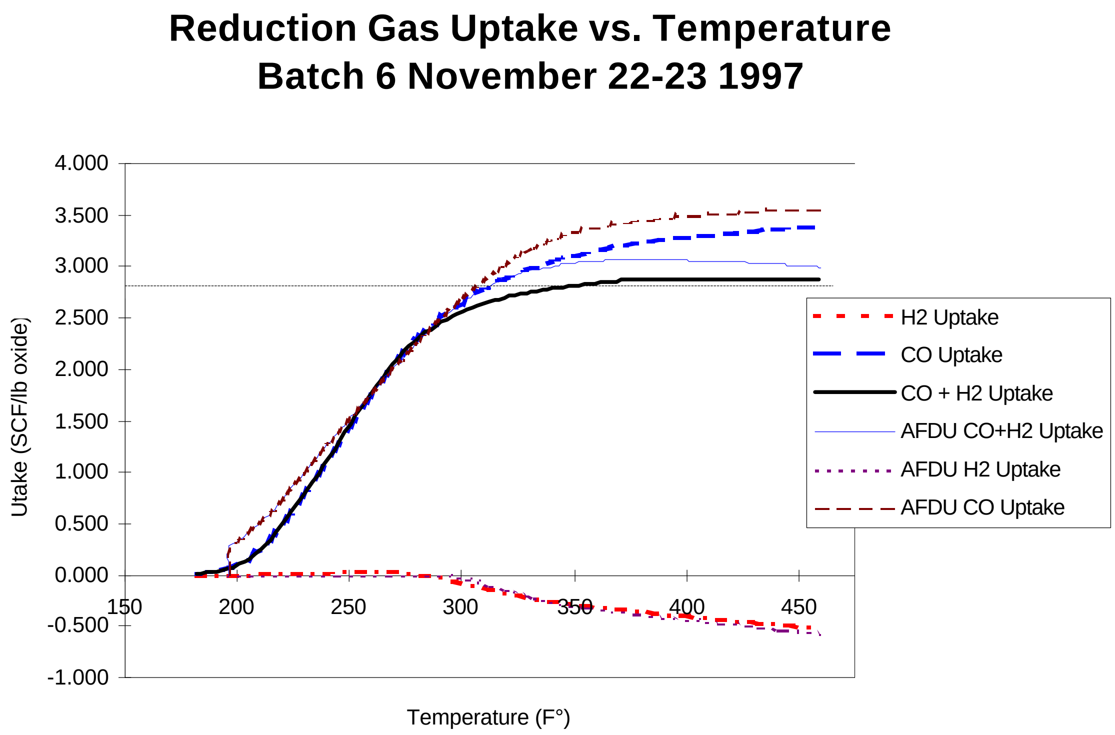
APPENDIX J - MILESTONE SCHEDULE STATUS AND COST MANAGEMENT REPORTS

Operation Start	Operation End	Operating Hours	Shutdown Hours	Reason for Shutdown
10/1/97 00:01	10/1/97 00:01	0.0	51.5	G-03 Electrical Tie-in and Eastman Guard Bed Change
10/3/97 03:30	11/3/97 14:50	756.3	1112.0	End of Catalyst Run
12/19/97 22:50	12/31/97 23:59	289.1		End of Reporting Period

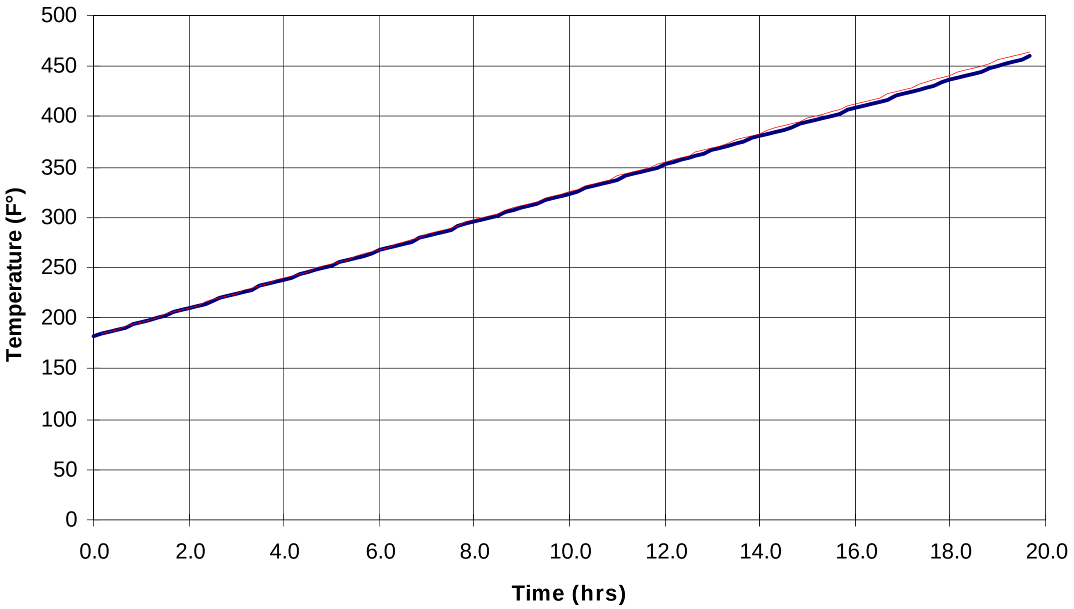
Total Operating Hours	1045.5
Syngas Available Hours	1097.0 *
Plant Availability, %	95.3 *

* Excluding catalyst changeout to restart test program.


Catalyst Age (eta)
10.00
1.00
0.10
eta
0
20
40
60
80
100
120
140
160
180
Days Onstream
Complex down
Sparger cleaned
Catalyst
Additions
Catalyst
Additions
CO-Rich
Feed
eta = 1.7 exp[-0.034 t]
eta = 2.2 exp[-0.017 t]
eta = 1.6 exp[-0.016 t]
eta = 2.7 exp[-0.018 t]
eta = 2.7 exp[-0.016 t]


Reduction Gas Uptake vs. Temperature
Batch 6 November 22-23 1997
Utake (SCF/lb oxide)
4.000
3.500
3.000
2.500
2.000
1.500
1.000
0.500
0.000
-0.500
-1.000
150
200
250
300
350
400
450
Temperature (F°)
H2 Uptake
CO Uptake
CO + H2 Uptake
AFDU CO+H2 Uptake
AFDU H2 Uptake
AFDU CO Uptake

Reduction Temperature Profile
Batch 6 November 22-23 1997



Sparger Resistance Coefficient (Post-December 1997 Restart)