Commercial-Scale Demonstration of the Liquid Phase Methanol (LPMEOHTM) Process

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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 LPMEOHTM 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.

Table of Contents

Abstract	3
ACRONYMS AND DEFINITIONS.....	7
Executive Summary	9
A. Introduction.....	13
B. Project Description.....	13
C. Process Description.....	14
D. Results and Discussion.....	15
D.1 Off-Site Testing (Product-Use Demonstration)	15
D.2 Commercialization Studies	17
D.3 DME Design Verification Testing.....	18
D.4 LPMEOH™ Process Demonstration Facility - Methanol Operation.....	21
D.5 On-Site Testing (Product-Use Demonstration).....	28
D.6 Planning and Administration	29
E. Planned Activities for the Next Quarter	30
F. Conclusion.....	30
 APPENDICES.....	 33
APPENDIX A - SIMPLIFIED PROCESS FLOW DIAGRAM	33
APPENDIX B - OFF-SITE PRODUCT-USE TESTING	34
APPENDIX C - PROCESS ECONOMIC STUDY	35
APPENDIX D - DME DESIGN VERIFICATION TESTING.....	36
APPENDIX E - SAMPLES OF DETAILED MATERIAL BALANCE REPORTS.....	37
APPENDIX F - RESULTS OF DEMONSTRATION PLANT OPERATION.....	38
APPENDIX G - ON-SITE PRODUCT-USE TESTING	39
APPENDIX H - PARTNERSHIP ANNUAL PLAN	40
APPENDIX I - PROJECT REVIEW MEETING (16-17 DECEMBER 1997)	41
APPENDIX J - MILESTONE SCHEDULE STATUS AND COST MANAGEMENT REPORTS	42

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	-	sum of Fresh Feed and Recycle Gas
Reactor O-T-M Conversion	-	percentage of energy (on a lower heating value basis) in the Reactor Feed converted to methanol (Once-Through-Methanol basis)
Reactor Volumetric Productivity	-	the quantity of Raw Methanol produced (tons per day) per cubic foot of reactor volume up to the Gassed Slurry Level
Recycle Gas	-	the portion of unreacted syngas effluent from the reactor "recycled" as a feed gas
Refined Grade Methanol	-	Distilled methanol, defined as 99.8 wt% minimum purity; used directly in downstream Eastman processes
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 (H₂), carbon monoxide (CO), and carbon dioxide (CO₂) 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.

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 (H₂)-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 AFUD 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 (η) - October/November 1997

The “age” of the methanol synthesis catalyst can be expressed in terms of a dimensionless variable η , which is defined as the ratio of the rate constant at any time to the rate constant for freshly reduced catalyst (as determined in the laboratory autoclave). Appendix F, Figure 1 plots $\log \eta$ 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 $\log \eta$ 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

DATA SUMMARY FOR LPMEOH™ DEMONSTRATION UNIT

Case	Date	Gas Type	Temp (Deg C)	Pres. (psig)	Balanced				Recycle		Inlet Sup.		Space		Slurry		Gas		Gassed		Catalyst		CO		Reactor		Syngas		Raw MeOH		Catalyst		Reactor		U		Sparger	
					Gas (KSCFH)	CO Gas (KSCFH)	H2 Gas (KSCFH)	Gas (KSCFH)	Gas (ft/sec)	Velocity (ft/hr-kg)	Space Velocity	Conc. (wt% ox)	Holdup (vol%)	Gas Hgt (ft)	Slurry Hgt (ft)	Age (elo)	Conv. (%)	O-T-M Conv. (%)	Util. (SCF/lb)	Production (TPD)	MeOH Prod. (gmol/hr-kg)	Vol. Prod. (TPD/Cu ft)	Overall (BTU/hr ft2 F)	dP (psi)	Resistance (K)													
6	3-Oct-97	Balanced	249	676	552	0	0	2161	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	2125	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	2017	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	2098	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	2098	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	2093	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	2092	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	2098	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	2082	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	2089	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	2067	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	2145	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	2107	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	2084	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	2075	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	2081	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	2071	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	20-Oct-97	Balanced	250	675	560	0	0	2070	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	21-Oct-97	Balanced	250	675	515	0	0	2115	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	2104	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	2062	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	2043	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	2064	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	2075	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	2041	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	2069	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	2017	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	2042	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	2030	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	2059	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

DATA SUMMARY FOR LPMEOH™ DEMONSTRATION UNIT

Case	Date	Gas Type	Temp (Deg C)	Pres. (psig)	Balanced			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 (ela)	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 (psd)	Sparger Resistance (K)
					Gas (KSCFH)	CO Gas (KSCFH)	H2 Gas (KSCFH)																	
6	20-Dec-97	Balanced	225	680	772	0	0	2,193	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	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	0.77	9,597	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	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	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	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	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	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	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	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	164	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 (H₂), carbon monoxide (CO), and carbon dioxide (CO₂) 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

SIMPLIFIED PROCESS DIAGRAM KINGSPORT LPMEOH - 260 TPD PLANT LPMEOH™ PROCESS DEMONSTRATION FACILITY

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)

**Off-Site Product-Use Testing
Proposals Under Consideration**

<u>Demonstration Project</u>	<u>Site</u>
Acurex FFV	California
Stationary Turbine for VOC Control	Site to be determined in cooperation with EPRI
West Virginia Univ. Stationary Gas Turbine	West Virginia
Water/Naphtha/MeOH Bus,	California
Aircraft Ground Equipment Emulsion	Tyndall AFB, Florida Brooks AFB, Texas
University of Florida Fuel Cell Fuel Cell, Florida	Florida
West Virginia Univ. Tri-Boro Bus	New York
Florida Inst. of Tech. Bus & Light Vehicle	Florida

Appendix B-2

ARCADIS GERAGHTY & MILLER

ARCADIS Geraghty & Miller, Inc.
555 Clyde Avenue
Mountain View
California 94043
Tel 650 961 6700
Fax 650 254 2487

MEMO

To:
Larry Waterland

Copies:

From:
Doug Coleman

Date:
29 January 1998

Subject:
Progress update report: LPMEOH Testing in a Flexible Fuel Vehicle

This project was initiated in the last week of September 1997. Initial project efforts included securing a 55-gal drum supply of LPMEOH from Air Products and modifying the vehicle sign-out and log form for the ARCADIS Geraghty & Miller (formerly Acurex Environmental) Ford Taurus flexible fuel vehicle to include project-specific information and to note that, for this test period, vehicle fueling will be with M85 only; fueling with regular unleaded gasoline (RUL) during the test period has been forbidden.

During October 1997 through January 1998 the vehicle has been driven in routine ARCADIS Geraghty & Miller business use, fueled by M85 from the network of M85 fueling stations in Northern California. Through December, about 2,800 miles have been logged on the vehicle. Plans are to log about another 1,200 miles (4,000 miles total) on standard M85, perform the initial set of emissions and performance tests on the vehicle, then switch to M85 prepared from LPMEOH. This switch will likely occur in February or March. Vehicle fuel economy will be continually tracked throughout the test program.

ARCADIS GERAGHTY & MILLER

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MEMO

To:
Larry Waterland

Copies:

From:
Carlo Castaldini

Date:
28 January 1998

Subject:
Project update report: LPMEOH Testing in a GasTurbine for Control

The objective of this project is to demonstrate the use of a mini gas turbine for the destruction of VOC from an emission source while burning LPMEOHTM. Mini gas turbines, with a gross generating capacity of 75 kW or less, promise to be one of the most successful technologies for distributed power generation in the new deregulated power industry. There are two main considerations in the use of these mini turbines. The first is the cost of their operation and the second is their emissions. When the mini turbine is also used to destroy VOC from vented process air, the economics improve. The use of methanol for remote applications or for emission control may alleviate other key obstacles to commercial development.

ARCADIS, Geraghty & Miller, Inc., formerly Acurex Environmental Corporation, is leading the effort to procure, modify, install, and test a mini gas turbine (<30 kW) with LPMEOHTM to demonstrate this concept. The work to date has concentrated on the following ongoing activities:

- 1) Select the mini gas turbine technology that will be used for the demonstration,
- 2) Locate a host site for the demonstration
- 3) Secure the co-funding for the demonstration project.

1. Select the mini gas turbine technology:

Mini gas turbines that are commercially available and are adequate for this project include:

- Capstone (30 kW)
- Ellison (75 kW)
- Allied Signal ASE8-100 (525 kW)

ARCADIS

Preliminary discussions have taken place with representatives from Capstone and Allied Signal regarding the availability, cost, performance and fuel flexibility of available machines. Capstone currently makes a 30 kW engine, burning natural gas. The engine requires about 300 scfm of air at standard conditions. The cost of the engine is particularly high, \$50,000 with accessories, because Capstone has only recently initiated commercial sales. Allied Signal is currently developing a 75 kW gas turbine equipped with a precombustor chamber for VOC destruction. ARCADIS Geraghty & Miller is currently exploring the cost and opportunity to test one of these engines at the Allied Signal Research Plant. Discussions with Elliott have not yet taken place. Elliott makes a 75kW gas turbine that can be used for these tests. The ARCADIS Geraghty & Miller engineering team expects to arrive at a recommendation for the selection of the turbine based on cost, test opportunity, and availability of test site with minimal cost to the program.

1. Site Selection

The site selection effort is ongoing and is being conducted in conjunction with the selection of the mini turbine. Potential host sites for this demonstration are at (1) the combustion laboratory for the New Jersey Electric and Gas Company in Newark, NJ; (2) Capstone plant in Tarzana, California; University of Utah, Provo Utah; and (4) Research Laboratory in Torrance, California for Allied Signal. ARCADIS also attempted to contact a commercial bakery for the field demonstration of this technology, but the management at the Weydemyer bakery declined participation. Another commercial site being considered is a medical glove manufacturing facility in Santa Clara, California. A meeting is being planned in the next two weeks to discuss mutual interests in the project and technical details.

2. Securing cofunding for the project

There are several potential sources of cofunding for this project. The applicable source of cofunding is based on the selection of the technology and the host site for the demonstration. ARCADIS has submitted a proposal to the Empire State Electric Energy Research Corporation (ESEERCO) for a research grant to demonstrate the mini turbine application for combined VOC destruction. ESEERCO is currently evaluating this project in connection with Public Service Electric & Gas (PSE&G) of New Jersey. A second source of cofunding is the vendors of current mini turbines (Capstone, Allied Signal or Elliott). The additional source of cofunding and participation in the project will come from a planned investment by the California Energy Commission in the demonstration of distributed power generation technology with an environmental side benefit. The procurement for CEC cofunding will be made available in the next few months. ARCADIS Geraghty & Miller is currently pursuing the participation of the existing equipment vendors as well as the demonstration at PSE&G in connection with ESEERCO co-sponsoring.

Appendix B-4

WVU Emission Studies on a Dual-Fuel, Methanol/Jet Fuel Stationary Gas Turbine (Loth/Clark)

The progress made so far is summarized below:

- a) The Dual-Fuel Change-Over Technique has been developed.
- b) The Dual-Fuel Selection and Pressurization Equipment has been purchased and installed. Compressed shop air was selected to power two pneumatic fuel pumps. Each pump is mounted directly on the methanol and the jet-fuel containing barrel.
- c) A pneumatic tire supported cart was constructed to carry the equipment operating console, with the battery pack required for gas-turbine starting.
- d) The gas turbine pneumatic horsepower output measuring equipment has been designed as is under construction.
- e) The aircraft gas turbine controls have been re-arranged to permit stationary (airport ramp) operation from outside the cockpit. A turbine containment sheet is being installed to protect the operator from flying debris in case of turbine failure.
- f) The fuel metering equipment is on order.

B-4

Proposal Time Line

Category	Month
	1 2 3 4 5 6 7 8 9 10 11 12
Gas Turbine Fuel System Conversion	----->
Fuel Type Selection Control Panel Construction with Pumps Fuel Flow and Turbine Power measurements	----->
Test Runs on Both Fuels	----->
Emission Testing on Both Fuels	----->
Report Preparation	----->

(2)

Appendix B-5

ARCADIS GERAGHTY & MILLER

ARCADIS Geraghty & Miller, Inc.
665 Clyde Avenue
Mountain View
California 94043
Tel 650 951 5700
Fax 650 254 2497

MEMO

To:
Larry Waterland

Copies:

From:
Shyam Venkatesh

Date:
28 January 1998

Subject:
Project update report: LPMEOH testing with -86 generators at Tyndall AFB

September/October 1997

The project was officially started in the last week of September 1997. A search was launched for commercial vendors with water-fuel emulsion technologies previously tested in diesel engines. The search resulted in the identification of three vendors with experience in alternative fuels for diesel engines: Clean Diesel Technologies Inc. (CDTI); Quantum Energy Technologies (QET); and A-55 L.P. Inquiries for participation in the test program were not responded to by CDTI. QET, being a newer company, did not have proven experience with water-diesel emulsion fuels. Only A-55 responded positively and extended an invitation to visit their facility in Reno, NV.

A-55 is the manufacturer of a custom water-naphtha-methanol emulsion fuel that has been tested mainly on some diesel bus engines in the Reno Area. A set of validation tests were also performed with the fuel on a DDC 6V-92TA MUI Engine at Southwest Research Institute in San Antonio, Texas.

Dr. Larry Waterland and Dr. Shyam Venkatesh, both of ARCADIS Geraghty & Miller, visited the A-55 offices on October 13, 1997. The visit commenced with a meeting between the ARCADIS Geraghty & Miller team and A-55 officials Mr. Bruce Anderson, Operations Manager, Mr. Michael Marquess, Project Manager, and Mr. John Hill, Director of Sales and Marketing. A site visit to the A-55 testing facility was arranged after an initial slide-show presentation by Mr. John Hill explaining the capabilities and experience of A-55. During the test-site visit, the ARCADIS Geraghty & Miller team was able to observe demonstrations of the fuel on a stationary diesel engine and a transit bus. A-55 demonstrated significant interest at this point in participating in the subject test program. It was also suggested to A-55 that their participation would involve the supply of fuel and any technical help necessary. A-55 relayed no significant concerns at this time.

B-5

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Following the meeting with A-55, intent of testing with the A-55 fuel was conveyed to Tyndall AFB engineer Mr. Alan Canfield. Mr. Canfield of Applied Research Associates (ARA), Inc., is the key point of contact and the project engineer for this test. ARA is the operating contractor for the Air Force Research Laboratory facility (AFRL/MLQ) at Tyndall AFB. Mr. Canfield assumed responsibility for coordinating the various base personnel that would be involved with the test project. A meeting was scheduled for November 13, 1997, at Tyndall AFB to discuss test program requirements and issues.

November 1997

A draft test plan was prepared for presentation at the meeting in Tyndall AFB. Mr. Canfield indicated that, in addition to the A-55 emulsion fuel, the Tyndall team would like to test a water-diesel emulsion fuel formulated and tested by ARA in another project for the AF. The scope of this additional test was increased by suggesting the addition of methanol to the ARA emulsion-fuel blend to which ARA agreed.

The meeting scheduled for November 13 was held as planned and was attended by the following persons:

Dr. Joe Wander, Project Officer, AFRL/MLQ, Tyndall AFB
Mr. Alan Canfield, Project Engineer, ARA, Tyndall AFB
Lt. Hugh Verhoof, 325 MS, Tyndall AFB
MSgt Steve Price, 325 MS, Tyndall AFB
MSgt Larry Rodebaugh, 83 FWS, Tyndall AFB
Sgt Ralph Pearish, 83FWS, Tyndall AFB
Mr. Mark Manley, Lockheed Martin, Tyndall AFB
Dr. Shyam Venkatesh, ARCADIS Geraghty & Miller, Mountain View, CA
Dr. Larry Waterland, ARCADIS Geraghty & Miller, Mountain View, CA

The following is a copy of the meeting notes.

MEETING NOTES:

Meeting started at 0900 in the AFRL/MLQ EQ Conference Room, Bldg 1120. Mr. Canfield and Dr. Venkatesh provided the group an introduction of the work plan involved in demonstrating the A-55 and water in fuel (WIF) mixtures in A/M32A-86 generators at Tyndall AFB. Three -86 generators will be racked for six months--one running A-55 Clean Fuel, one running Dr. Aly Shaaban's WIF emulsion, and one operating on standard military diesel (control unit). Lockheed Martin operates approximately 14 -86 generators. Previous tests indicate the -86 will achieve a maximum load of 100 amps running these fuel mixtures. Equipment at Tyndall does not require over 100 amps, so the group decided that larger injectors would not need to be installed for this test, pending satisfactory operation. (If greater than 100 amps is required, one of the 12 remaining units running diesel could be used). Mr. Canfield spoke with Mr. Dave Elliot, SA-ALC/LDPG (DSN 945-6537), prior to the meeting to inquire on authorization for deviation from the technical order for operating AGE on diesel. Mr. Elliot has conducted similar tests for the Air Force, and offered to help coordinate authorization for the test at Tyndall through SM-ALC/LIE (POC: Mr. Loren Len) at McClellan AFB CA, the reported owner of Air Force AGE. Mr. Canfield will email a

ARCADIS

request to Mr. Elliot. (ACTION: Mr. Canfield) Mr. Elliot will also be questioned regarding changing injectors, since the Air Force may be going to different injectors (B65) in the -86. The initial maintenance inspection will consist of removing the covers and heads from the test units and performing a visual inspection of the condition of the rings, pistons, etc. Observations will be recorded and photographs will be taken. Hopefully the same technician can perform the inspection at the end of the test period to provide a consistent (subjective) evaluation. Estimated time for inspection of the three units was 4-8 hours. A-55 Clean Fuel and WIF emulsion will be provided to Lockheed Martin in 55-gallon drums as needed for fueling the test vehicles. Estimates of use will determine initial quantities delivered and communication with Mr. Manley will insure stores are not depleted. Mr. Manley will set up the fueling station, including spill retention and absorbent materials. (ACTION: Mr. Manley). The test generators will be marked as such with high-visibility tape, indicating proper fuel and possibly color-coded to the respective fuel containers. The maintenance logs will be checked to find -86 units recently out of 6-month overhaul for the test. (ACTION: MSgt Price) A tentative start-date for the program was set at Wed 3 Dec 97, to include the preliminary maintenance inspection. Mr. Canfield has another commitment and would like to reschedule to Mon 8 Dec, pending approval from the group.

END MEETING NOTES

The following items were attended to in the two-week period following the meeting:

- Written approval from Mr. Dave Elliot was obtained by Mr. Canfield permitting the use of the -86 generators for the test program.
- A-55 was intimated of the target test dates. Mr. John Hill of A-55 inquired about obtaining access to a local -86 generator set for performing some exploratory studies.
- Two drums of methanol were ordered from Air Products through Mr. Robert Senn. One drum was delivered to A-55 and another drum was delivered to ARA.

In the last week of November, Mr. Canfield suggested that testing should be postponed to the week of January 12, 1998 because of the unavailability of himself and other personnel during the holiday season. All parties agreed to the new test date.

Dr. Venkatesh explored for the possibility of obtaining on-site emission measurement capabilities at Tyndall AFB. Mr. Canfield, after enquiring with AF scientists and sampling personnel, extended further support to the test program by arranging on-site emission measurement capabilities. A comprehensive sampling matrix was developed with the help of Mr. Stewart Makgraf the on-site field-sampling scientist.

In response to Mr. John Hill's request for obtaining access to a nearby -86 generator, Dr. Venkatesh, with the help of Mr. Canfield and Mr. Dave Elliot, arranged for A-55 to work with MSgt. John Ternau of the Nevada Air National Guard in Reno. The Air National Guard at Reno operates about eight -86 generator sets. MSgt Ternau indicated that they would not be able to commit any of their existing -86s to A-55 within the time frame of this project; however, A-55 was welcome to study the operation characteristics of the -86 generator sets at their facility.

B-5

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December 1997

While no significant activity occurred during the month of December, A-55 sent a fax-letter on December 24th summarily withdrawing from the test program. The reasons stated by A-55 were as follows:

- No foreseeable commercial benefit from the test program.
- Inability to commit resources to the test program.
- The lack of a -86 genset to perform scoping tests.
- The limited scope of the test program (less than 1,000 gallons of fuel over 6 months)
- The distant location.

The letter also stated that A-55 may be willing to participate if the above issues were resolved.

January 1998

In January negotiations were started with A-55 to see if some of the above issues could be resolved. The following offer of support was made to A-55:

- Purchase of fuel at cost plus shipping. (\$0.60/gallon + shipping).
- Purchase the services of an A-55 engineer as technical support during the test phase of the program. The engineer would also have the chance, if necessary, to perform scoping tests at Tyndall AFB in lieu of the non-availability of a -86 genset at the Air National Guard facility.

However, after nearly two weeks of discussions, A-55 declined the above offer stating that at the current time they are not interested in the program, laying serious doubt to their claims (in their literature and during the visit to their facility) about the fuel and their capabilities.

It was decided to continue the test program with the ARA fuel blend. A change of plan was made by ARA regarding their fuel blend. Instead of diesel, JP-8 would be the fuel. This decision was made by ARA keeping in view the desire of the AF to have a universal fuel for all its engines. ARA has started to formulate a JP8-water-methanol based emulsion fuel. The blend will contain 5 percent methanol. Testing will begin in February as soon as preliminary tests with the new fuel blend are complete.

Appendix B-6

Air Products "Coal-Based Methanol" Contract Progress Report

V.P. Roan, University of Florida, Principal Investigator

January 21, 1998

This report details the progress accomplished towards the completion of the Air Products "Coal-Based Methanol as a Fuel for PAFC Systems" contract.

A testing laboratory has been identified to perform the chemical analysis of the methanol to be delivered by Air Products. A preliminary list of constituents has been compiled and delivered to the laboratory. The list is a combination of the composition listing provided by Air Products and chemicals (such as sulfur) or compounds known to be harmful to the steam reformer and/or the fuel cell stack. Work is continuing to comprehensively identify any harmful constituents through literature searches and personal contacts with fuel cell specialists.

The fuel cell data acquisition system has been reviewed and is the process of being upgraded to suit the contract requirements. Hardware upgrades include the following: Installation of numerous thermocouples and thermocouple cards; installation of a fuel flow meter; and the installation of air flow meter. It is envisioned that more upgrades will be completed in the near future. The existing Labview-based software program has been updated to incorporate the increased hardware capabilities.

The design of the gas analysis system is nearly complete. The gas analysis requirements have been identified and capable gas chromatograph has been sourced. An order has been placed and the gas chromatograph should be available within a month. The necessary gas collection equipment (bottles, valves, piping, etc.) are being identified and should be purchased soon.

Work is underway to upgrade the fuel cell system for the contract. This work includes replacing a faulty CO₂ purge gas regulator and replacing the thermal switch on the fuel cell stack. Space in the fuel storage area has been provided for two 55 gallon containers of the Air Products methanol. Preliminary design work has begun on a separate in-lab fuel tank and associated piping and valving.

(1)

Appendix B-7

FUEL GRADE METHANOL IN TRANSIT BUS APPLICATIONS

PROGRESS REPORT, JANUARY 1998

Submitted by: Nigel N. Clark, Principal Investigator
Dept. of Mechanical & Aerospace Engineering
West Virginia University
Morgantown WV 26506-6106

The objectives of this program are to quantify the emissions and performance of full size transit buses with compression ignition engines on Air Products fuel grade methanol and to compare the data with operation on the present methanol fuel in use. This project centers around an intensive experimental effort planned to take place in New York City in April, 1998. The owners of the subject transit buses, Triboro Coach, have been contacted and have reaffirmed their desire to support this study and that the buses will be available and in service in April. These buses have the Detroit Diesel 6V92 methanol engine. Fuel has been received at West Virginia University and is being stored at the University's airport facility. The Mechanical & Aerospace Engineering Chromatography Laboratory is preparing to perform analysis of the Air Products fuel using a Varian 3400 Chromatograph. Triboro have been contacted to provide samples of the existing fuel for comparative speciation. An improvement is being implemented on the WVU Heavy Duty Vehicle Emissions Laboratory (No. 1) that will be used for the test program: this change in sampling protocols will provide for more accurate carbon monoxide measurement.

Recovery of unburned methanol and formaldehyde as a combustion product in the vehicle exhaust for purposes of reporting emissions has long proven a problem when using a full scale dilution tunnel. This has been examined from a theoretical standpoint and there is concern that there may be hangup of formaldehyde in the tunnel. A sequence of formaldehyde injections has been planned to verify recovery. Since the resulting aldehyde analyses are costly, a surrogate hydrocarbon with similar vapor pressure will be identified for initial testing.

Immediate research plans include making ready the WVU Laboratory (No. 1) for travel to New York. WVU Laboratory No. 2, presently occupying the WVU test facility building, is leaving for the west coast on Feb. 9, after which Laboratory No. 1 will be installed in the test facility building where it will be serviced, calibrated and pre-tested for the Air Products research.

Appendix B-8

Table of Contents

1.	Introduction	1
2.	Activities	1
3.	Results and Discussion	4
4.	Work Scheduled for Next Quarter	4
5.	Appendix	6
a.	Injector modifications	
b.	Starting systems	

B.-8

1. Introduction

The first priority in this project was to return the methanol powered car and bus to normal operational status. Both vehicles were at one time operating normally but they have not been driven in at least five years. The greatest need was acquiring Avocet, the ignition enhancer for methanol in the diesel bus. The only time the bus performed acceptably in service in Jacksonville, was when Avocet was added to the methanol fuel. Avocet also contains a lubricating additive which is also necessary for dependable performance.

Cleaning the vehicles was second priority. The methanol car (1988 Chevrolet Corsica) had to have all its old decals removed. The bus, virtually covered in black algae on the outside, and covered with graffiti on the inside, required intensive cleaning. After removal of all decals from the car, it was obvious that repainting was still necessary. The third priority was to do all the necessary mechanical work to make the vehicles operational. This required, so far, new injectors for both the car and the bus, a starting system for the car (in cold weather), new fuel line fittings for the bus, and a new exhaust system for the car.

2. Activities (as recorded by the staff)

The activities performed in the first quarter of the contract period are reported below in chronological order:

1. J. Thomas contacted many potential sources of Avocet including John Sheehan, ICI America, Anthony Stiff, ICI South Africa; Glyn Short Consultant, Don Dudzinsky SCAQMD; Dr. Carroll Goering, U. of Illinois; Charles Di Ciuccio, DDC (Detroit Diesel Corp.); Larry Roemer DDC.
2. Started taking emblems off of Corsica using heat gun.
3. The bus was washed and crayon marker graffiti was removed from seats.
4. Met with Erik Gordon and he briefed us about Bus: fuel lines, fuel pumps, fuel tanks, fuel line fittings, injectors, methanol conversion manual Corsica Vehicle: computer programmer, fuel pump.

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(2)

B-8

5. Towed Corsica vehicle to campus for repairs.
6. Aeroflex Comp. representatives came to Bio-West looking at the bus fuel line and stainless steel fuel line fittings. They also went to campus to see the Corsica vehicle injectors, took one as a sample to inquire for availability. They would send a price list for possible needed items.
7. Scraped emblems off of Corsica vehicle.
8. Campus mechanic took Corsica's engine plenum off. Took fuel injectors off and checked them for clogging. The car was given an oil change.
9. Continued taking emblems off of Corsica vehicle.
10. Started to clean injectors with carburetor cleaner. Two seem okay.
11. Got new fuel injectors from GM for the Corsica vehicle.
12. Used woodgrain remover to get emblem glue leftover off the Corsica vehicle. The adhesive remover was purchased at Thompson PBE.
13. Campus mechanics installed a new fuel pump in the fuel tank. They tested it for proper pressure. It was OK. They tried to start the car but only one injector was working. Vehicle did not start. Hoses coming out of the fuel tank were replaced.
14. Fuel injectors were found to be short in length so they could not be used.
15. The interior of the car was cleaned. Diagram of the old injectors were faxed back to GM (Frank Ament) to show the actual size we need with the same flow rate.
16. The bus was towed over to campus.
17. Interior of bus was cleaned. Methanol car was taken to the muffler shop. There is a constant drain on the battery which has not been found.
18. Received 25 gallons of Avocet from U. of Illinois.
19. Received 25 gallons of Avocet from SCAQMD Diamond Bar, California.
20. Injectors were pulled out of the bus and cleaned. One out of the six injectors was deemed unusable/ the tip was in poor condition. Car came back from shop and was driven to Bio-

B-8

West for a fill-up (~5 gal.). The car ran decent, but there was a noticeable delay when starting from a stop.

21. Car was put on Analyzer to find out why it is not running right. Checked out alright. A bad connection on one sensor (MAF) was fixed. Ran it for about 30-40 minutes. Hesitation is intermittent, not constant. Otherwise running well.
22. Car was taken to be painted and have new hood put on it. Dr. Thomas went to Jacksonville to attempt to get new injectors made for the bus. The staff familiarized themselves with bus engine and its manual.
23. Picked up car from paint shop and drove it to Bio-West and added 2 gallons of methanol.
24. Car tires (4) were replaced.
25. The staff continued learning about the bus engine components such as, fuel lines. They tightened loose air lines for the air brakes compressor, checked the location of the computer (ELM), checked both halon fire extinguishers on the engine compartment; they were OK. They introduced air pressure in the system to test the front door, door is OK (rear door is not connected to system). They checked the air conditioner compressor and lines, oil in crankcase looks clear but oil filter tube is corroded inside, transmission fluid seems to be OK. Other air lines seem to be OK (no leaks), disconnected batteries, checked throttle plate actuators (Vacuum).
26. Ordered new injectors for bus with a 180cm³/cycle flow rate. They were ordered from Roy's Diesel Injection, 4020 Lenox Av. Jacksonville, FL. A starting system is needed for the car to correct the cold starting characteristics of the engine. Ether as used to get it running for a short time. An ether injection system is probably needed.
27. Installed a cold weather quick start (NAPA 605-3270 kit and 605-3252 cylinder). Received injectors from Roy's Diesel injection with a 195cm.
28. The installed cold start is working in the car.

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3-8

3. Results and Discussion

The custom fabricated fuel injector which GM used in the original methanol car were replaced with standard Cadillac high flow injectors which work well although machining of the fuel rail injector brackets was necessary to insure a proper fit. The original custom made injectors were no longer usable because of intractable clogging of the parts.

The Corsica is now operational and the newly installed starting system allowed instantaneous cold starting.

This starting system may be particularly useful in the southeast where the standard M-85 fuel is not very useful. The M-85 tends to separate in the high humidity conditions encountered most of the year in the southeast. Also, since the number of cold days in this region are much less than up north, the starting system and fluid should last a much longer time.

We received a new set of custom injectors for the bus from Roy's Diesel in Jacksonville. These will be installed in a week after a very thorough flushing of the fuel lines. We expect the bus to be operational after installation of the injectors.

4. Work Schedule for second quarter

1. Install new injectors in the bus after thorough flushing of the fuel lines.
2. Start the bus and determine any problems with brakes and driveability.
3. Repair any problems that interfere with normal bus operation.
4. Put the bus in regular service.
5. Re-commission the car and turn over responsibility to Dr. John J. Thomas for routine operation.
6. When steady state normal operation of the vehicles is achieved, work will intensify in the areas of emissions measurements. This will include installation of the Chassis dynamometer.

4
5

5-8

Listing of Project Personnel

1. Dr. John J. Thomas, Principal Investigator
2. Carlos Terreaux, Research Assistant
3. Frank Aransky, Research Assistant
4. Greg Palubin, Research Assistant
5. Rich Mackenzie, Director, Florida Tech Vehicle Maintenance
6. Bert Austin
7. David Cash
8. Jeff Reilly

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B-8

APPENDIX

6

7

NOV 05 '97 13:41 FR GMPGTG-MILFORD

248 685 5432 TO 814079848461

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B-8

STANDARD CONFIGURATION MODIFIED FUEL RAIL BRACKET NEW INSTALLATION

FUEL RAIL ASSY ~ 0.040-0.050" LOWER

Post-It® Fax Note	7871	Date	11/6/97	Page	1
To: Dr. Ted Thomas		From:	F. Amos		
Company: Biological Services		Co:	EMPT		
Phone: 407 768 8000		Phone:	248 476 1877		
Fax: 407 984 8461		Fax:	248 476 1808		

B-8

INSTRUCTIONS

DIESEL STARTING SYSTEMS

A ZERO START COLD WEATHER AID FOR QUICK STARTS

Starting Procedure

Electric Systems

CAUTION:

Inject Starting Fluid only while cranking engine.
Use only for starting.

1. Start cranking engine.
2. Activate system — press switch.
3. Release — Injecting starting fluid to engine.

If engine fails to start, repeat 2 and 3.

IMPORTANT: Read all instructions and cylinder label before installing and using system
Read "Danger" on page 4.

E-Z MOUNT VALVE INSTALLATION

VALVE LOCATION

The valve should be mounted on the outside of the cab wall, firewall, any main frame member, radiator frame, hood side, or other similar location. A metal plate can be used to mount the system to the frame when a convenient location is not available. The valve should be mounted within 48" of the atomizer location and may be mounted at any angle within 20° of vertical.

After holes have been marked and drilled, mount the valve. Use 5/16" screws and nuts with lock washers to secure the valve to the mounting surface.

DO NOT MOUNT VALVE

1. In the operators cab.
2. Next to the exhaust manifold or any area where temperature may reach 160° or above.
3. Where it is subjected to road dirt, ice, snow, etc.

VALVE MOUNTING

After location is selected, screw cylinder onto valve and hold in place to determine if you will have enough clearance to remove the fuel cylinder after valve is installed so replacement of a new cylinder is possible. Remove fuel cylinder and set aside while mounting valve to avoid accidental discharge of fuel. Put on dust cap to keep foreign matter out of valve.

Hole spacing for mounting purposes is shown below. The E-Z Mount valve (without cylinder installed) can be used as a template to mark the desired hole locations. It is best to mount the valve with four screws. Avoid twisting or distorting the valve when the mounting bolts are tightened.

B-8

WIRING ELECTRICAL SYSTEM

SEE WIRING DIAGRAMS BELOW

WIRING:

For wiring the valve use No. 18 gauge or heavier, flexible braided or stranded wire, with insulation impervious to oil, grease and other engine or road dirt. (If leads are over 6 ft. you must use heavier gauge wire. Follow SAE suggestions.)

DANGER: ALWAYS REMOVE FUEL CYLINDER WHEN WIRING VALVE OR CHECKING WIRING SYSTEM. READ "DANGER" ON PAGE 4.

Install the Push Button switch in a convenient location to the operator.

Using the blue three-way splice furnished, connect a wire between any wire connected to the "run" position of the ignition switch and either terminal of the push button switch.

Use the small terminals furnished to connect the wires to the push button switch and use the splices to connect the wires from the switch and the thermoswitch to the valve leads.

WITH THERMOSWITCH

Connect one wire of the thermoswitch to the other valve wire using the splices provided. The remaining thermoswitch wire should be grounded. This can be done by installing the terminal to the wire and put it under the thermoswitch mounting bolt.

WITHOUT THERMOSWITCH

Install the large terminal to the other valve wire and place under one of the valve mounting bolts or to another convenient grounding location.

TESTING:

Turn ignition switch on, push the Push Button switch -- plunger at top of valve should move up. When the switch released, plunger should go down.

The valve is now ready to operate.

If unit fails to operate, refer to "Troubleshooting Procedure" on page 4.

WITH THERMOSTAT **WITHOUT THERMOSTAT** **TUBING INSTALLATION****LOCATION:**

IMPORTANT: Route the tubing so that it does not contact hot exhaust manifolds or areas where it may be damaged by mechanical action or vibration. Also avoid possible kinking of the tube.

The tubing should be cut to as short a length as possible to accomplish best operation and installation.

INSTALLATION:

When connecting the tubing to the valve and atomizer fittings, be certain to keep tubing fully seated while tightening the nut. Tighten nuts 1 to 1 1/4 turns with wrench, after running down fingertight (20 to 30 in. lbs. of torque).

B-8

TROUBLESHOOTING PROCEDURE

BEFORE PROCEEDING Read "DANGER" at top of page.

DANGER

WHEN MAINTAINING OR TROUBLESHOOTING THESE SYSTEMS, ALWAYS MAKE SURE THAT YOU ARE IN A WELL VENTILATED AREA AWAY FROM HEAT, OPEN FLAMES, OR SPARKS. WEAR GOGGLES WHEN TESTING TO AVOID EYE INJURY. MAKE SURE THAT OPENINGS OF THE VALVE, TUBE, OR ATOMIZER ARE POINTED AWAY FROM YOURSELF WHILE TESTING.

THE ETHER USED IN THIS STARTING FUEL FOR THESE SYSTEMS IS EXTREMELY FLAMMABLE, TOXIC, HARMFUL, OR FATAL IF SWALLOWED. AVOID CONTACT WITH THE SKIN OR EYES AND BREATHING THE FUMES. IF SWALLOWED, DO NOT INDUCE VOMITING. CALL PHYSICIAN IMMEDIATELY.

IF FUEL ENTERS EYES OR FUMES IRRITATE EYES, THEY SHOULD BE WASHED WITH LARGE QUANTITIES OF CLEAN WATER FOR 15 MINUTES. A PHYSICIAN, PREFERABLY AN EYE SPECIALIST, SHOULD BE CONTACTED.

DO NOT STORE CYLINDERS IN TEMPERATURES ABOVE 160° F. CONTENTS ARE UNDER PRESSURE. DO NOT INCINERATE, PUNCTURE, OR ATTEMPT TO REMOVE CENTER CORE VALVE OR SIDE SAFETY VALVE FROM CYLINDER.

CHECK THESE POINTS WHEN THE UNIT DOES NOT OPERATE CORRECTLY.

A. FUEL CYLINDER:

Check to see if cylinder is handtight and screwed all the way down. Check starting fluid supply (empty cylinder weight is approximately 16oz/453.6g; a FULL cylinder is approximately 33oz/935.6g).

If the cylinder is empty - replace. Securely tighten cylinder clamp after replacing cylinder.

IMPORTANT ALWAYS CHECK VALVE GASKET UNDER CYLINDER (it is suggested to replace gasket when changing fuel cylinders). IF GASKET IS DAMAGED OR MISSING, THE VALVE WILL LEAK WHEN OPERATED. REPLACE WITH NEW GASKET. IF TWO GASKETS ARE USED, THE VALVE MAY NOT OPERATE. (Gasket No. 020-3903).

ALWAYS CLEAN DIRT AWAY FROM THE TOP AND EXTERIOR OF THE VALVE BEFORE REMOVING CYLINDERS AND CLEAN OUT VALVE AREA WHERE CYLINDER IS INSTALLED SINCE ANY DIRT OR FOREIGN MATTER CAN CLOG THE VALVE OR ATOMIZER OR YOUR SYSTEM.

After checking all other components of the system in accordance with the listed troubleshooting points, if the cause of the inoperative condition still is not discovered, check the cylinder to determine if it has pressure.

B. ATOMIZERS:

IMPORTANT: Atomizer or Orifice clogging is a common cause for failure of a system and occurs because dirt has been allowed to get into the valve when changing fuel cylinders.

To check atomizer, remove from engine and activate the system. If no fuel sprays out of the atomizer, disconnect it from the tubing. Activate system, if fuel flows out of the tubing, atomizer must be replaced.

C. TUBING:

If fuel has not flowed out of the end of the tubing after removing the atomizer, disconnect the tubing from the valve and reactivate system. If fuel flows out of the valve, the tubing is obstructed and should be replaced.

D. MECHANICAL VALVE:

Activate valve by moving lever or knob up and then down, if no fuel is dispensed the valve is clogged or damaged. Replace valve.

If valve operates, check control cable for damage and wire stop screw for tightness.

E. ELECTRIC VALVE:

NOTE: NEVER hold the Push Button switch in for more than 5 seconds, as this can damage the electric valve solenoid. Activate valve by turning ignition switch on and pressing and releasing the Push Button switch, if no fuel is dispensed, remove fuel cylinder.

1. Activate valve. Plunger should move up and stay up until the Push Button switch is released. Valve is operative if plunger responds correctly. Go on to "F".

2. If plunger does not move up:

A. Jump the Push Button switch. If valve is operative, switch is defective and should be replaced. Go on to "F".

B. If no Thermostat is used, go on to "C". Short out the Thermostat using a jumper wire. If the valve operates the Thermostat is defective and should be replaced.

C. If after jumping the Push Button switch, and the Thermostat if used, the valve is not operative, check wiring for continuity.

D. If the valve is still inoperative, replace it.

F. REASSEMBLY:

After correcting the problem, reinstall tubing and atomizer. Double check to see that all fittings have been replaced properly and are tight.

PREVENTATIVE MAINTENANCE

Take every precaution to avoid getting dirt inside top of valve, since this can get into valve chamber causing a clogged valve or atomizer. Refer to "Troubleshooting" Point "A" before replacing empty cylinders.

Check fittings for leaks.

Check all mounting bolts to make sure that they have not loosened. Periodically test unit for proper operation by following the steps recommended in the "Troubleshooting Procedures".

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)**

Process Economics Study - Outline

LPMEOH™ Process, as an add-on to IGCC for Coproduction

Part One - Coproduction of Methanol Note - 2nd Draft was dated 10/01/96;
comments received 11/25/96, 3d Draft released ~03/31/97.

1. Introduction

1.1. Process Design Options.

- Develop process flow diagram and plant design options for the LPMEOH™ process, for design variables such as: a) feed gas pressure, b) feed gas compositions, and c) % syngas conversion.

2. Liquid Phase (LP) Methanol Advantage versus Gas Phase (GP) Methanol.

2.1. Syngas Conversion Cost for Methanol Production from CO-Rich syngas.

- For the various LPMEOH™ process (LP) design options (from 1.1) develop plant capital and conversion costs derived from the Kingsport Project design and costs. Develop conversion costs for:
 - 500 t/d Plant size, with 500 psi feed gas pressure;
 - 500 t/d Plant size, with 1000 psi feed gas pressure
 - Impact of Plant Size on Conversion Costs
- Summarize in a series of graphs, conversion costs, in cents per gallon over the range of syngas conversion from 18% (LP - Once-through) to 94% (GP), for baseload annual coproduction operation. This will show LP's advantage at higher feed pressures and lower conversions; and will highlight areas for LP design development/demonstration improvements. *(For future: include plant size impact on product distribution (freight) cost, assuming that local markets are served. Freight cost will increase with plant size, as the distribution radius increases.)*

2.2. Methanol Product Purification Cost.

- Develop capital and operating costs for these product purification design alternatives:
 - MTBE Grade;
 - Fuel Grade;
 - Chem. Grade;

Over a range of feed gas compositions, summarize LP's advantage versus the GP process (in cents per gallon), especially for MTBE and Fuel Grade from CO-rich feed gas at low syngas conversions.

2.3. Feedgas (Syngas) Composition Variations; (Impact on LP vs. GP).

- Higher Sulfur content in the feedgas will have a negative cost impact on LP at low syngas conversion, relative to GP at high conversions. Conversely, higher feedgas inert content will have a negative relative cost impact on GP.
 - Sulfur content variation; over the above range of syngas conversion
 - Inert gas content variation; over the above range of syngas conversion

2.4. Syngas Usage (Btu per Gallon) - Impact on IGCC Power Plant.

- Summarize differences in syngas utilization (Btu per gallon of methanol), and in mass flow loss/gain to the combustion turbine (kwh production loss/gain per gallon of methanol); for the cases in 2.1 above.

Process Economics Study - Outline

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2.5. Summary of Cost Advantage(s) - (LP Vs GP).

- Summarize the cost impact (cents per gallon) of the above design variables and syngas utilization differences. Show the impact of methanol plant size on the conversion costs. Also (separately show) the impact of 90% and 70% annual load utilization for use with Section 4. - "Intermediate Load Coproduction and Stored Energy" of this Economics Study.

2.6. Recommendations for Further Study.

- Recommend areas for process design value engineering work; and areas for demonstration at Kingsport.

Part Two - Baseload Power and Methanol Coproduction

Note - Portions of Part Two, Section 3.1; was included in the Tampa CCT Conference's Paper, 1/9/97.

3. Baseload Coproduction with Methanol Sales - Impact on Electric Power Cost -

For baseload coproduction, the gasifier must be sized for both the power and methanol products. The results of Part One indicate the LP technology can make coproduction economic, even at small methanol plant sizes (400 to 1200 TPD) suitable to serve local markets near the power plant. The LP technology's advantage (over GP) is also greatest at the lower (up to 34%) Syngas Conversions which are consistent with these methanol plant sizes. A matrix of power plant and methanol plant sizes of interest, at up to 34% Syngas Conversion to methanol, is shown in the following tables. These examples are based on Advanced Gas Turbine Technology (*reference (G.E.'s) published paper*) with the base gasification plant sized for two gasifiers, of about 1735×10^6 Btu(HHV)/hr. output each (1626×10^6 LHV)

3.1 Gasification Plant Size Fixed

- With a given gasification plant size, the methanol plant and power plant can be sized to accommodate a range of Methanol to Power output ratio's.

<u>Syngas Conversion</u>	<u>Power Plant Size</u>	<u>Methanol Plant Size</u>	<u>Methanol to Power Ratio</u>	<u>Gasification Plant Size</u>
0.0 %	500 MW	0 T/D	0 T/D per MW	Base
13.8%	426 MW	500 T/D	1.2 T/D per MW	Base
20.0%	394 MW	691 T/D	1.8 T/D per MW	Base
30.0%	342 MW	1085 T/D	3.2 T/D per MW	Base

3.2 Power Plant Size Fixed

- With a given power plant size, the gasifier size may be increased to accommodate the coproduction of methanol. For Gasification Plant size increases of up to 50% (to say, three x 1735×10^6 Btu(HHV)/hr. gasifiers), the methanol to power coproduction ratio's could be:

<u>Syngas Conversion</u>	<u>Power Plant Size</u>	<u>Methanol Plant Size</u>	<u>Methanol to Power Ratio</u>	<u>Gasification Plant Size</u>
0.0 %	500 MW	0 T/D	0 T/D per MW	1.00 x Base
16.7 %	500 MW	736 T/D	1.5 T/D per MW	1.20 x Base
25.0 %	500 MW	1227 T/D	2.5 T/D per MW	1.33 x Base
33.3 %	500 MW	1825 T/D	3.7 T/D per MW	1.50 x Base

- The impact of coproduction on electricity generation costs could be shown in graphs of electricity cost Vs. methanol net back price.

End of Part Two.

Process Economics Study - Outline

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Part Three - Coproduction for Intermediate Electric Load Following.

4. Intermediate Load Coproduction

Note - Part Three, Section 4.2: is being developed as a paper for the June 1997 Power-Gen Europe Conference.

4.1. Syngas Value as a function of (time of day) Power Value.

Earlier electric power daily load following studies indicate that LPMEOH™ coproduction optimizes for daily or seasonal power peaks in the 500 to 2500 hr./yr. range. This means the methanol plant operates, during daily or seasonal "off-peak" power periods, in the 8260 to 6260 hr./yr. range, with stop/start operations for these on/off power peaks. This is the "intermediate load" area of a typical power grid system. (8760 hr./yr. = 100%; all exclude gasifier/plant outages)

4.1.2. Syngas value as function of seasonal opportunity fuels/feeds.

- *Natural gas may be available seasonally, for use in the CC power plant, allowing syngas to be used for conversion in an LPM add-on. Other feeds?*

4.2. Intermediate Load Coproduction - for Methanol Sales.

- For intermediate load coproduction cases, redundant investment to utilize syngas is required; so that when the methanol plant shuts down during peak power periods, all of the syngas can be converted to electric power. There are several intermediate load coproduction power plant design choices; a) a CC power plant turned down, or b) a baseload CC power plant with other CC or CT power plant(s) for peak. These may be combined with methanol plant design choices such as size/% syngas conversion. To evaluate the system properly, time of day power values (also called Lambda Curves) are needed. The Lambda Curve examples from published EPRI studies can be used for initial evaluations. The Section 2.(above) Methanol Plant design choices can then be combined with power plant design options, to optimize the system.

4.3. Intermediate Load Coproduction, for Methanol Sales and for Dispersed Power.

- Dispersed power can provide electricity and heat locally, at the use point, eliminating the need for new power distribution lines in congested areas. The world wide package (0.2 MW to 10 MW) power plant market is large, and growing. A variety of technologies (combustion turbine, internal combustion engine, fuel cell) are being packaged. Methanol produced at a nearby IGCC power plant during off-peak power periods could provide clean local (peak) power; bypassing the local electric power distribution system.

4.4. Intermediate Load Stored Energy Production, with Methanol Fuel for Peak Power Production.

- When other peaking fuels are not available, or are too expensive, then methanol may also be used as a peaking fuel. The design optimization for this is quite complex. The IGCC/OTM plant design has an additional variable: the peaking power plant size and hours of operation is an independent variable. A study option would be to compare ourselves (IGCC/OTM) to the various published EPRI (IG-Cash, et. al.) studies, which provide Lambda Curve examples for energy storage. However, selling methanol and using distillate fuel for peaking, is the economic choice at currently forecasted world oil and methanol prices. Therefore, this study should have low priority, until a site specific need is identified.
- *Methanol could be transported to remote existing, or to new peaking power plants, to unload grid systems.*
- When other back up fuels are not available, or are too expensive, then methanol may also be used to enhance power plant availability. Coproduction with multiple gasifier trains may also be used to enhance power plant availability. (e.g. - Three by 50%, where Baseload Power = 2 x 50%; Peaking Power = 1x 50% plus methanol fuel; Methanol Plant = 1 x 50%, but operates only when all three gasifiers are operating and peak power is not required.)

End of Part Three.

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Part Four - Methanol Fuel Applications

5. Premium Methanol Fuel Applications

- At 46 cents per gallon, methanol as a fuel (\$6.90 per mmBtu) will not compete with oil in most applications (\$20/bbl crude = \$3.30/mmBtu; \$27/bbl diesel = \$4.50 /mmBtu). However, methanol coproduced at a central IGCC power station, may be a valuable premium fuel for two evolving developments: as an economical Hydrogen source for small fuel cells, and as an environmentally advantaged fuel for dispersed electric power.
- "Central clean coal technology processing plants, making coproducts of electricity and methanol; to meet the needs of local communities for dispersed power and transportation fuel" - meets the DOE Clean Coal Technology Program's objectives. Serving (initially) small local fuel markets also builds on LP's (the LPMEOH™ process) strengths; good economics at small methanol plant sizes, fuel grade product distillation savings, and a freight advantage in local markets vis-à-vis large off-shore remote gas methanol. Baseload methanol coproduction studies show that 46 cent per gallon methanol can be provided from an abundant, non-inflationary local fuel source.. *We need to arrange fuel tests to confirm the dispersed energy environmental advantage.*

5.1. Hydrogen Source for:

- Hydrogen fuel cells, being developed for transportation applications, can achieve 65% system efficiency, as compared to 45% for diesel IC engines and 32% for gasoline IC engines. Methanol is a storable, transportable liquid fuel which can be reformed under mild conditions to provide H₂. For small H₂ applications, *and at low utilization factors*, methanol reforming is a more economical source of hydrogen than : a) natural gas reforming, b) distillate (oil) reforming; and is cheaper than liquid H₂.

5.1.1. Fuel Cells for Transportation

5.1.2. Fuel Cells for Stationary Power

(See also dispersed power below).

5.1.3. Industrial Applications - Small Hydrogen Plants

Small pressurized methanol reformers for transportation applications may be suitable for adapting to meet the needs of small commercial hydrogen gas requirements.

5.2. Dispersed Power

- Dispersed power can provide power and heat locally, at the use point, eliminating the need for new power distribution lines in congested city areas. The world wide package (0.2 MW to 10 MW) power plant market is large, and growing. A variety of technologies (combustion turbine, internal combustion engine, fuel cell) are being packaged. Methanol produced at a nearby IGCC power plant during off-peak power periods could provide clean local power; bypassing the local electric power distribution system.

5.3. Dimethyl Ether as an Enhancement to Methanol in Premium Fuel Applications

Can coproduced mixtures of methanol and dimethyl ether improve upon methanol, in the above?

End of Part Four.

Memorandum

To: Distribution Dept./Loc.:
From: W. R. Brown Dept./Ext.: PSED, X17584
Date: 31 March 1997
Subject: LPMEOH™ Process Economics - for IGCC Coproduction

Distribution:

c: D. M. Brown - APE (Hersham)
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W. C. Jones - Eastman

The third draft of the DOE Topical Report on LPMEOH™ Process Economics (Part One) is attached for your use (review, comment). This Topical Report develops plant design options for our LPMEOH™ process, as an add-on to IGCC power plants for the coproduction of methanol and power. Part One also compares our LPMEOH™ (LP) methanol process with the gas phase (GP) methanol process.

LP's advantage over GP is about 10 cents per gallon; when the syngas conversion is low (less than 34%), and when the feed gas pressure is high (greater the 750 psig), and when the methanol plant size is relatively small (400 to 1200 TPD). Surprisingly, even at these small plant sizes, the LP technology can coproduce methanol at less than 50 cents per gallon (good). The GP technology is over 50 cents per gallon (not good). Therefore, when baseload IGCC power is viable, the LP Technology makes coproduction viable.

The DOE Topical Report (Part One) looks specifically at:

- Determining and optimizing **conversion costs** for our LP technology as a function of feed gas pressure and % syngas conversion. (See graphs on pages A - 5, 6, 7, 9, 10).
- Determining **purification (distillation) costs** for "Fuel", "MTBE", and "Chemical" grade methanol. (See graph, page A - 15). *Distillation savings are a significant part of LP's advantage.*

- Comparing LP with GP technology. (See the above graphs, plus Summary Table on page 16).
- Listing of **future LP design improvements**, expected from actual operation, or that are recommended for further engineering study (see pages 17,18).

Parts Two, Three and Four of the DOE Topical Report are planned for the future (the outline is attached). **Part Two** will examine the impact of baseload coproduction on electric power costs. **Part Two, Section 3.1** was included in the Tampa CCT Conference's Paper; "Fuel and Power Coproduction" (1/9/97). **Part Three** will look at time-of-day energy values: a) intermediate load coproduction (e.g.- off-peak methanol production), and b) methanol as stored energy for peaking and/or dispersed electric power. **Part Four** of the Topical Report plans to look at Methanol Fuel Applications, where locally produced (non-inflationary) methanol, at less than 50 cents per gallon, could be a viable source of hydrogen for industrial or fuel (cells) power applications. Serving (initially) small local fuel markets builds on LP's strengths; good economics at small plant sizes, fuel grade product distillation savings, and a freight advantage in local markets vis-a-vis large off-shore remote gas methanol.

Your comments on this third draft of the Topical Report(Part One) would be appreciated. After your further comments are received; we will formally release this as the final (draft) of a Topical Report.

Bill

APPENDIX D - DME DESIGN VERIFICATION TESTING

Air Products and Chemicals, Inc.
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Allentown, PA 18195-1501
Telephone (610) 481-4911

30 June 1997

Mr. Robert M. Kornosky
Technical Project Manager
Mail Stop 920-L
U. S. Department of Energy
Federal Energy Technology Center
P. O. Box 10940
Pittsburgh, PA 15236-0940

Subject: Cooperative Agreement DE-FC22-92PC90543
Liquid Phase Methanol Demonstration Project
Liquid Phase Dimethyl Ether Design Verification Testing -
Recommendation

Dear Bob:

The updated version of the Recommendation to proceed with Design Verification Testing of the Liquid Phase Dimethyl Ether Process is attached. This document will be used during the Project Review Meeting on 24-25 July, at which time final approval by DOE and the Partnership will be requested.

Very truly yours,

A handwritten signature in black ink, appearing to read "E. Heydorn", with a long horizontal line extending to the right.

Edward C. Heydorn
Program Manager
LPMEOH™ Demonstration Project

Enclosure

cc: Mr. William C. Jones - Eastman Chemical Co.
Mr. William J. O'Dowd - DOE-FETC
Mr. Edward Schmetz - DOE-FE-HQ
Dr. John Shen - DOE-FE-HQ
Mr. Barry T. Street - Eastman Chemical Co.
Mr. Peter Tijm - Air Products & Chemicals, Inc.

LPDME Recommendation

Summary

From the Statement of Work, "Commercial-Scale Demonstration of the Liquid Phase Methanol (LPMEOH™) Process," selected under Round 3 of the U.S. Department of Energy's (DOE's) Clean Coal Technology (CCT) Program: "Subject to Design Verification Testing (DVT), the Partnership proposes to enhance the Project by including the demonstration of the slurry reactor's capability to produce DME (*dimethyl ether*) as a mixed co-product with methanol." The first DVT step (Phase 1, Task 5), to address issues such as catalyst activity and stability, to provide data for engineering design, and to verify the market through engine tests and through market and economic study, is now complete. The market potential for DME is large, and progress in the laboratory toward developing a catalyst system whose performance meets the economic targets of a methanol equivalent productivity of 14 mol/kg catalyst-hr after 6 months of operation, producing at least 75% (by heating value) DME and 25% methanol.

A test of the Liquid Phase Dimethyl Ether (LPDME) at the LaPorte Alternative Fuels Development Unit (AFDU), in conjunction with the DOE's Liquid Fuels Program, would be appropriate if the catalyst system development can be completed successfully. An implementation decision, made mutually by the DOE's Clean Coal Technology LPMEOH™ project participants, and by the DOE's Liquid Fuels Program participants, should be made (by *July of 1997*) to implement testing at LaPorte in *early 1998*. (*Final dates should be recommended by the DOE's Liquid Fuels Program, based on progress in developing the LPDME catalyst system*).

Liquid Phase Dimethyl Ether (LPDME) Design Verification Testing (DVT)

From the Statement of Work, DOE's CCT LPMEOH™ project (Cooperative Agreement No. DE-FC22-92PC90543): "Subject to Design Verification Testing (DVT), the Partnership proposes to enhance the Project by including the demonstration of the slurry reactor's capability to produce DME as a mixed co-product with methanol. The production of DME from synthesis gas is a natural extension of the LPMEOH™ process in that three reactions occur concurrently in a single liquid phase reactor, methanol synthesis, methanol dehydration and water-gas shift. This process enhancement can significantly improve the overall conversion of coal derived synthesis gas to a storable blend of methanol and DME. -- -- the enhanced (DME production demonstration is complementary to ongoing studies being sponsored by DOE's Liquid Fuels Program --) -- . -- At the conclusion of each of the DVT steps, a joint Partnership/DOE decision will be made regarding continuation of methanol/DME demonstration.."

The first DVT step (Phase 1, Task 5), to address issues such as catalyst activity and stability, to provide data for engineering design, and to verify the market through engine tests and through market and economic study, is now complete.

LPDME Recommendation

The LPDME Process Concept: - Three Concurrent Reactions:

- $2 \text{ CO} + 4 \text{ H}_2 = 2 \text{ CH}_3\text{OH}$ (Methanol Synthesis).
- $2 \text{ CH}_3\text{OH} = 1 \text{ CH}_3\text{-O-CH}_3 + 1 \text{ H}_2\text{O}$ (Methanol Dehydration).
- $1 \text{ CO} + 1 \text{ H}_2\text{O} = 1 \text{ CO}_2 + 1 \text{ H}_2$ (Water-gas Shift).

The overall reaction, with carbon monoxide (CO)-rich synthesis gas (syngas), in a single liquid phase (slurry) reactor:

- $3 \text{ CO} + 3 \text{ H}_2 = 1 \text{ CH}_3\text{-O-CH}_3 + 1 \text{ CO}_2$ (DME from CO-rich syngas)

This is the "once-through" CO-rich syngas concept for the LPDME process utilizing a single slurry reactor.

Conversion per pass, with CO-rich syngas, can be higher than for the LPMEOHTM process. Methanol may also be produced, as a mixed co-product with the DME, and can easily be separated and recovered. The separation of DME from carbon dioxide (CO₂) will be necessary for certain market applications.

Status of the LPDME DVT Work

The status of a) the LPDME process economics/market study work, and of b) the LPDME catalyst system R&D work, follows:

A-1. The market applications for DME are extensive. DME is, or may be, used as:

- Aerosol - Small, but established market. High purity DME is required.
- Cooking Fuel - Potentially a large market, to replace imported liquefied petroleum gas (LPG). There is a lot of interest in China, and DME is on the agenda for DOE's Pittsburgh Coal Conference in China (Sept. of 1997). Purity, of about >95% DME, with <2% methanol, < 3% CO₂ is estimated. An unresolved application issue is CO emissions during cooking. How does DME purity impact this? Use testing is needed. Our contacts with representatives from the Institute of Coal Chemistry of the Chinese Academy of Sciences in Shanxi has provided the following assessment of the potential market for DME as a cooking fuel:
Of the 1.2 billion people in China, 0.3 billion live in cities. Of these, 1/3 currently use natural gas or LPG. Assuming 4 people per family, the 0.2 billion people who do not use gas or LPG converts to 50 million families. If DME captures 20-30% of the market share for these new applications, and the DME consumption is 200 kg per family per year, the demand for DME would be 2.4-3.0 million tons per year.
- Diesel Replacement Fuel. DME is an ultra clean (high Cetane) diesel fuel; and an 80% DME mixture with methanol and water is now being engine-tested by others (Amoco, et. al.). Market development (at least in the U.S.) faces a fuel distribution infrastructure problem. DME might

LPDME Recommendation

more easily replace LPG in countries where LPG is already an engine fuel. Diesel use in the U.S. is projected to increase by 1.5 percent a year, assuming an economic growth of 1.9 percent a year. This will raise consumption from over 4 quadrillion BTU to approaching 6 quadrillion BTU (Reference 1). This corresponds to an annual increase of almost 1.4 million gallons per year of diesel consumption.

- DME Derivatives, as a Diesel Fuel Additive. Quotes from the DOE Liquid Fuels Program (Contract No. DE-FC22-95PC93052) quarterly report for April-June 1996: "Initial Cetane number (CN) testing of a three-component composition of 1,2-dimethoxy ethane, 1,1-dimethoxy methane and methanol blended with diesel fuel showed a 40% increase in the CN of the diesel fuel when the blend was 50/50." "The concept of adding a blend of oxygenated compounds to diesel fuel in order to enhance the Cetane value and cold start properties is being investigated. The blend of oxygenated compounds is derived from dimethyl ether chemistry, and builds on work conducted earlier --." The testing of this DME feedstock chemistry is in its early days, but it is possible that CO₂ may not need to be separated from the DME prior to the production of DME derivatives. The 50/50 blend referenced above would therefore provide a large market opportunity for the projected U.S. market growth (Reference 1), let alone for the present consumption.
- DME Derivatives, as Chemicals/Other Fuels. DME is a key intermediate in a commercial synthesis gas-to-gasoline process, and is being developed as an intermediate for other chemicals and fuels as part of the DOE's Liquid Fuels Program. The fit for DME here is long-term.

A-2. The economics studies, for once-through coproduction (with an integrated gasification combined cycle (IGCC) power plant, for example) on synthesis gas rich in carbon oxides, show that the LPDME process will have an economic advantage greater than the LPMEOHTM process. A once-through LPDME reactor is able to convert greater than 50% of such a syngas, whereas a once-through LPMEOHTM reactor can convert only about 30%. The economics, of course, depend upon the end-use (purity) of the DME and upon the gasification plant's coproduct mix (amount of power, methanol, DME, etc.). The same liquid phase reactor design options to increase syngas conversion (Reference 2); such as feed gas compression and/or CO-rich gas recycle; are also be applicable for LPDME. So, the LPDME technology has the potential to improve on the 5-10 cents per gallon (methanol equivalent) advantage over the LPMEOHTM process for the coproduction of DME to serve local markets.

As with the LPMEOHTM process, gas phase process technology must be considered as the economic competitor. The gas phase DME process (Reference 3) must run with hydrogen (H₂)-rich syngas. In the IGCC coproduction flow sheet (shown in Figure 1), gas phase technology is at an economic disadvantage, since separate shift and CO₂ removal are required. As is the case for methanol, inexpensive remote natural gas would therefore be the economic plant site choice for gas phase technology. A comparison, of IGCC/LPDME coproduction with DME imported from remote gas facilities, shows an advantage of 20-30% for locally produced DME relative to

LPDME Recommendation

imported DME. The transportation cost to import DME is much higher than for methanol, and the LPDME coproduction advantage is even greater than that for LPMEOH™ (vs. methanol import) (Reference 2). Dehydration of imported methanol to make DME is not competitive either. Therefore, for DME in local markets, LPDME coproduction should be a winner!

With H₂-rich syngas, the LPDME process loses its (once-through, high conversion per pass) economic advantage. The overall reaction, with (> 2:1) H₂-rich syngas is:

Since water inhibits the methanol dehydration reaction, the slurry reactor must be staged, with water removal between stages. Staging could be by high ratio gas recycle, and/or with multiple reactors; but the once-through simplicity is lost. Therefore, it is unlikely that the LPDME process would be developed for use in H₂-rich syngas applications.

A cost estimate of commercial-scale LPDME plants has been performed. This work has helped quantify the targets for the laboratory R&D program (summarized in Part B). From these studies, a commercially successful LPDME system is defined for a Texaco-type synthesis gas (35 mol% H₂, 51 mol% CO, 13 mol% CO₂) available at 500 PSIG. At a reactor operating pressure of 950 PSIG and a space velocity of 4,000 liters/hr-kg catalyst, the LPDME catalyst system must have a methanol equivalent productivity of 14 mol/kg catalyst-hr after 6 months of operation, producing at least 75% (by heating value) DME and 25% methanol. Figure 2 shows the effect of plant size on DME cost. These costs are competitive with LPG in China (Section A-1).

B. Laboratory R&D Results

Summary of work through end of funding by CCT LPMEOH™ Project (9/96): An LPDME catalyst system, with reasonable long-term activity (57% of initial activity after 1000 hours), productivity (equivalent methanol productivity of 29 mol/kg catalyst-hr), and selectivity (79% carbon selectivity to DME, CO₂-free basis), was identified and tested. The system exhibits best activity under CO-rich syngas conditions, i.e. those most likely for (IGCC) coproduction. Accelerated aging of the catalyst system is a remaining issue. Water concentrations in the liquid phase reactor are higher with syngases richer in H₂, and its effect needs to be evaluated.

Laboratory work has continued under the DOE's Liquid Fuels Program. The issues, to be addressed in the lab before a decision on a test run at the DOE's AFDU in LaPorte, are:

- 1) Understanding the LPDME catalyst system's accelerated aging; and modifying the catalyst and/or the system operating conditions; and
- 2) Manufacturing scale-up of catalyst for a LaPorte AFDU run.

LPDME Recommendation

Progress has been made in the laboratory effort. Figure 3 shows the performance for the first DME catalyst which was tested; goals from the Liquid Fuels Program are provided for reference. After further study, an improved DME catalyst (AB-05) was tested with two LPMEOH™ catalysts (S3-86 and MK-101); the results of a 700 hour life study are presented in Figure 4. When compared with the program goals (summarized in Figure 5), the catalyst performance of the newer catalyst is approaching the commercial targets defined in Section A. The status of the laboratory program is summarized in the following table:

	Liquid Fuels Program Goals	Commercial Targets	Laboratory Results
Catalyst Productivity, mol/kg catalyst-hr (MeOH-equivalent)	> 28 (Initial Productivity)	> 14 (productivity for aged catalyst)	28 (Initial Productivity)
Catalyst Selectivity	DME Selectivity > 80% (% Carbon, CO ₂ -free)	DME = 75%, Methanol = 25% (heating value basis)	DME Selectivity = 79% (% Carbon, CO ₂ -free)
Catalyst Life	> 50% Remaining Activity after 1000 hours	Target Productivity after 6 months of operation	57% Remaining Activity after 1000 hours

Initial discussions with catalyst manufacturers have been held. Once a manufacturer is selected, a laboratory-scale catalyst batch will be produced and tested in the autoclave to verify the production technique developed at Air Products. An interim 1 lb batch will then be produced and tested. Once the catalyst production techniques have been verified at this scale, the 200 lb LaPorte batch will be produced using the same methodology as for a full commercial batch. An autoclave check of this material will be performed prior to the start of the LaPorte AFDU run.

Recommendations

The catalyst system and the market applications/opportunities are sufficiently promising that proof-of-concept testing at the LaPorte AFDU is recommended. Kingsport is an unlikely site for the commercial size demonstration of LPDME, since there are limited times for CO-rich syngas testing; and H₂-rich syngas would create water buildup. Therefore, the basis for commercializing LPDME must come from:

- 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;

LPDME Recommendation

- 2) continuing work in hydrodynamics of slurry reactors (other ongoing DOE programs); and
- 3) reactor performance (methanol catalyst activity and life, hydrodynamics, and heat transfer) from the LPMEOH™ Process Demonstration Unit.

The tie-in between the laboratory and the LaPorte AFDU is important. Historically, the rate of deactivation of methanol synthesis catalyst has been greater in the autoclave than at the AFDU; this may be a result of loss of catalyst from the autoclave, or due to greater carbonyl poisoning as a result of the higher surface-to-volume ratio at the laboratory scale. Testing at the engineering scale of the LaPorte AFDU can eliminate this variable. Operation of the LPMEOH™ Process Demonstration Unit will provide data on catalyst life under coal-derived syngas and at the larger engineering scale (the tie-in to the LaPorte AFDU for commercialization).

The recommendations for proceeding with DVT of the LPDME catalyst system are:

- An LPDME test run at the LaPorte AFDU, in conjunction with the DOE's Liquid Fuels Program, would be appropriate if the LPDME catalyst system development can be completed successfully. Up to \$875,000 of CCT LPMEOH™ Project budget support, from the Cost Plan (22 October 1996), should be made available to support a suitable LPDME test run at LaPorte.
- An implementation decision, made mutually by the DOE's CCT (DE-FC22-92PC90543) LPMEOH™ Project participants, and by the DOE's Liquid Fuels (DE-FC22-95PC93052) Program participants, should be made (by July of 1997) in time to implement testing at LaPorte in early 1998. (*Final dates should be recommended by the DOE's Liquid Fuels Program, based on progress in developing the LPDME catalyst system*). The CCT LPMEOH™ Project participants shall be kept informed (via review meetings and status reports) by Air Products of the development by the DOE Liquid Fuels Program participants of the LaPorte AFDU LPDME test-run plans, so that a timely final approval can be made
- In the interim, some DME product-use testing may be appropriate for the LPMEOH™ Demonstration Project's off-site product-use testing.

The schedule for the proposed LPDME testing at the LaPorte AFDU and possible implementation at the Kingsport LPMEOH™ Process Demonstration Facility is summarized below:

DME DVT Decision Made	July 1997
Commercial-Scale DME Catalyst Produced/Tested	
in Laboratory Autoclave	January 1998
LaPorte AFDU Test	February/March 1998
Kingsport Decision Made	March/April 1998
Kingsport Implementation (Provisional) Plan	July 1998 - March 2001

LPDME Recommendation

Impact on CCT Project

Technical: The commercialization of the LPDME Process can be successfully achieved by the combination of the activities at the LaPorte AFDU and the LPMEOH™ Process Demonstration Unit described previously.

Cost: Up to \$875,000 of Project funds would be available to support a suitable LPDME run. An update of the CCT Project's Cost Plan (22 October 1996), based upon the DVT Recommendation, will be performed following the joint Partnership/DOE decision.

Schedule: If the DVT Recommendation is approved by the Partnership and DOE, the operating schedule for the LPMEOH™ Process Demonstration Unit will remain unchanged from the current Demonstration Test Plan (September 1996). The DVT would proceed according to the September 1996 DME Milestone Plan (included in the Demonstration Test Plan) and the schedule of the Liquid Fuels Program.

References

1. Transportation energy consumption by fuel, 1975, 1995 and 2015: History: Energy Information Administration, *Short-Term Energy Outlook*, DOE/EIA-0202(96/4Q) (Washington, DC, October 1996), and *State Energy Data Report 1994*, DOE/EIA-0214(93). Projections: Table A2. Internet access at <http://www/eia.doe.gov/oiaf/aeo97/figure.html#fig46>.
2. "Fuel and Power Coproduction - The Liquid Phase Methanol™ Process Demonstration at Kingsport", paper presented at Fifth Annual DOE Clean Coal Technology Conference, Tampa, FL, January 7-9, 1997.
3. Haldor Topsoe AS, "Preparation of Fuel Grade Dimethyl Ether", International Publication Number WO9623755, World International Property Organization, 08 August 1996.

(end).

Figure 1. Once-through Methanol Coproduction with IGCC Electric Power

Figure 2

DME COST VERSUS SIZE

TEXACO-TYPE SYNTHESIS GAS, 1:1 FEED:RECYCLE

GOALS

FIGURE 3

FIGURE 4

MEOH Equiv. Prod. (mol/kg-hr)

- Six catalyst samples (#1 - #6) were developed with good stability and decent activity.

FIGURE 5

APPENDIX E - SAMPLES OF DETAILED MATERIAL BALANCE REPORTS

TITLE: Catalyst Addition and Aging

RUN NO: K6-10/24

Balance Period: 24oct97 07:00:00 25oct97 07:00:00

Time From Start of Run (days)

Start 200.7

End 201.7

Reaction Conditions

Temperature (°F) 482

Pressure (psig) 675

Space Velocity (sL/kg-hr) 4596

Vg (inlet) 0.65

Slurry Data

Catalyst Weight (lb oxide) 33,300

Slurry Concentration (wt %) 40.2

Slurry Level (ft) 49.5

Gas Holdup (vol %) 39.4

Gassed Slurry Volume (ft3) 2078

Performance Results

Raw MeOH Production (ton/day) 143.3

Raw MeOH Production (ton/day) 143.2

Syngas Utilization (SCF/lb MeOH) 43.0

Catalyst Life (eta) 0.21

CO Conversion (total) (%) 22.9

CO Conversion to MeOH (%) 24.8

CO Conversion to H2 (%) -1.92

Syngas Conversion (% LHV) 76.5

Syngas Usage (BTU/gallon MeOH) 66,728

Recycle Ratio 4.02

MeOH Productivity (gmol/kg-hr) 11.22

Rxr Volumetric Productivity (ton/day-ft3) 0.069

Sparger "K"-value 14.58

Energy Balance

(gas measurements) 143.3

(liquid measurements) 143.2

43.0

0.21

22.9

24.8

-1.92

76.5

66,728

4.02

11.22

0.069

14.58

Atom/mass Balance Closure (% of reactor Inlet)

C 100.28

H 102.69

O 100.13

N 101.13

Total Mass 100.48

CRUDE PRODUCT

REFINED PRODUCT

Liquid Product Analysis (wt%)

10/24 10/25

19:00 7:00

99.99 99.96

0.01 0.01

0.01 0.03

0.00 0.00

100.01 100.00

Methanol

Ethanol

Water

Oil

Total

10/25 7:00 79.23 0.25 20.39 0.15 100.02

RUN NO: K6-10/24 TITLE: Catalyst Addition and Aging

	FRESH FEED	CO MAKEUP	H2 MAKEUP	K-01 OUTLET	REACTOR FEED	C-05 OUTLET	MAIN PURGE	DISTILL. PURGE	CRUDE PRODUCT	REFINED PRODUCT
T	85	55	55	105	325	239	84	61	226	78
P	758	871	515	710	704	668	657	7	185	144
Comp	67.06	1.93	77.66	69.85	69.31	64.78	69.85	8.00	0.00	0.00
(mol %)	28.78	97.05	9.12	20.45	22.06	19.02	20.45	7.00	0.00	0.00
N2	0.43	0.97	6.50	2.67	2.24	2.50	2.67	2.00	0.00	0.00
CH4	0.04	0.05	0.92	0.44	0.36	0.40	0.44	0.00	0.00	0.00
CO2	3.68	0.00	3.76	6.60	6.03	6.27	6.60	62.00	0.00	0.00
DME	0.00	0.00	0.01	0.00	0.01	0.01	0.00	4.00	0.00	0.00
MeOH	0.00	0.00	0.06	0.00	0.00	6.12	0.00	10.00	68.49	99.95
EtOH	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.15	0.01
H2O	0.00	0.00	0.00	0.00	0.00	0.48	0.00	0.00	31.35	0.04
others	0.00	0.00	1.97	0.00	0.00	0.41	0.00	7.00	0.01	0.00
TOTAL	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Mole Wt	11.165	27.501	7.770	10.861	10.920	12.288	10.861	39.217	27.709	32.035
LHV	141.7	0.0	0.0	537.5	683.2	669.3	21.1	0.6	19.6	88.8
Enthalpy	-26.87	0.00	0.00	-113.21	-129.78	-149.67	-4.48	-1.15	-10.41	-31.43
Flow	513,074	0	0	2,062,127	2,590,496	2,315,829	80,999	3,369	37,208	115,899
SCFH	1,352.5	0.0	0.0	5,436.0	6,828.9	6,104.8	213.5	8.9	98.1	305.5
lbmol/hr	15,100	0	0	59,040	74,569	75,015	2,319	348	2,718	9,787
lb/hr										

TITLE: Reactor Operation at 260 C

RUN NO: K28-10/27

Balance Period:		Time From Start of Run (days)		
Start Date	27oct97 07:00:00	Start	203.7	
End Date	28oct97 07:00:00	End	204.7	
Reaction Conditions		Slurry Data		
Temperature (°F)	496	Catalyst Weight (lb oxide)	33,300	
Pressure (psig)	674	Slurry Concentration (wt %)	41.7	
Space Velocity (sL/kg-hr)	4598	Slurry Level (ft)	49.5	
Vg (inlet)	0.66	Gas Holdup (vol %)	42.4	
		Gassed Slurry Volume (ft3)	2078	
Performance Results		Energy Balance		
Raw MeOH Production (ton/day)	148.0	(gas measurements)	5436	
Raw MeOH Production (ton/day)	148.0	(liquid measurements)	476	
Syngas Utilization (SCF/lb MeOH)	41.6	Steam Production (lb/hr)	1410	
Catalyst Life (eta)	0.20	Steam Drum Pressure (psig)	16.5	
CO Conversion (total) (%)	25.8	Steam Import/Export (lb/hr)	45.7	
CO Conversion to MeOH (%)	28.0	Reactor O-T-M Conversion (% LHV)	1773	
CO Conversion to H2 (%)	-2.25	Weighted Tube Length (ft)	159	
Syngas Conversion (% LHV)	79.2	Heat Transfer Area (ft2)		
Syngas Usage (BTU/gallon MeOH)	66,542	Reactor Overall U (BTU/hr-ft2-F)		
Recycle Ratio	4.05			
MeOH Productivity (gmol/kg-hr)	11.59			
Rxr Volumetric Productivity (ton/day-ft3)	0.071			
Sparger "K"-value	15.03			
		Atom/Mass Balance Closure (% of reactor inlet)		
		C	100.15	
		H	102.93	
		O	99.55	
		N	102.35	
		Total Mass	100.17	

Liquid Product Analysis (wt%)	REFINED PRODUCT		CRUDE PRODUCT	
	10/27 19:00	10/28 7:00	10/27 19:00	10/28 7:00
Methanol	99.99	99.99	79.33	76.72
Ethanol	0.01	0.01	0.25	0.25
Water	0.00	0.00	19.27	22.79
Oil	0.00	0.00	0.15	0.15
Total	100.00	100.00	99.00	99.91

RUN NO: K28-10/27 TITLE: Reactor Operation at 260 C

	FRESH FEED	CO MAKEUP	H2 MAKEUP	K-01 OUTLET	REACTOR FEED	C-05 OUTLET	MAIN PURGE	DISTILL. PURGE	CRUDE PRODUCT	REFINED PRODUCT
T	76	46	46	99	333	239	78	55	226	74
P	761	872	511	709	704	667	657	7	185	144
Comp	67.12	1.93	77.66	71.20	70.42	65.85	71.20	8.00	0.00	0.00
(mol %)	28.57	97.05	9.12	18.20	20.19	16.83	18.20	7.00	0.00	0.00
N2	0.43	0.97	6.50	3.32	2.76	3.10	3.32	2.00	0.00	0.00
CH4	0.05	0.05	0.92	0.65	0.53	0.60	0.65	0.00	0.00	0.00
CO2	3.83	0.00	3.76	6.63	6.09	6.33	6.63	62.00	0.00	0.00
DME	0.00	0.00	0.01	0.01	0.01	0.02	0.01	4.00	0.00	0.00
MeOH	0.00	0.00	0.06	0.00	0.00	6.34	0.00	10.00	67.48	99.99
EtOH	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.15	0.01
H2O	0.00	0.00	0.00	0.00	0.00	0.52	0.00	0.00	32.35	0.00
others	0.00	0.00	1.97	0.00	0.00	0.41	0.00	7.00	0.01	0.00
TOTAL	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Mole Wt	11.172	27.501	7.770	10.490	10.622	12.003	10.490	39.217	27.569	32.040
LHV	141.3	0.0	0.0	537.7	680.1	665.7	17.2	0.6	20.1	91.8
Enthalpy	-27.15	0.00	0.00	-108.96	-124.49	-145.22	-3.51	-1.19	-10.86	-32.52
Flow	512,857	0	0	2,074,730	2,591,513	2,307,407	66,290	3,457	38,775	119,834
lbmol/hr	1,352.0	0.0	0.0	5,469.2	6,831.5	6,082.6	174.7	9.1	102.2	315.9
lb/hr	15,104	0	0	57,374	72,561	73,008	1,833	357	2,818	10,121

TITLE: Catalyst Addition and Aging

RUN NO: K6-12/25

Balance Period:		Time From Start of Run (days)	
Start Date	25dec97 07:00:00	Start	5.3
End Date	26dec97 07:00:00	End	6.3

Reaction Conditions		Slurry Data	
Temperature (°F)	455	Catalyst Weight (lb oxide)	19,500
Pressure (psig)	680	Slurry Concentration (wt %)	28.1
Space Velocity (sL/kg-hr)	9625	Slurry Level (ft)	58.0
Vg (inlet)	0.77	Gas Holdup (vol %)	49.8
		Gassed Slurry Volume (ft3)	2440

Performance Results		Energy Balance	
Raw MeOH Production (ton/day)	294.7	Steam Production (lb/hr)	22130
Raw MeOH Production (ton/day)	279.5	Steam Drum Pressure (psig)	214
Syngas Utilization (SCF/lb MeOH)	39.4	Steam Import/Export (lb/hr)	1471
Catalyst Life (eta)	1.29	Reactor O-T-M Conversion (% LHV)	25.5
CO Conversion (total) (%)	45.7	Wetted Tube Length (ft)	54.2
CO Conversion to MeOH (%)	48.5	Heat Transfer Area (ft2)	2103
CO Conversion to H2 (%)	-2.96	Reactor Overall U (BTU/hr-ft2-F)	164

Atom/Mass Balance Closure (% of reactor inlet)	
C	100.93
H	100.97
O	98.14
N	101.81
Total Mass	99.53

CRUDE PRODUCT

12/25	12/26
19:00	7:00
93.15	93.40
0.25	0.25
6.67	6.48
0.15	0.15
100.22	100.28

REFINED PRODUCT

12/25	12/26
19:00	7:00
96.92	99.97
0.04	0.01
3.01	0.03
0.00	0.00
99.97	100.00

Liquid Product Analysis (wt%)

Methanol
Ethanol
Water
Oil
Total

RUN NO: K6-12/25 TITLE: Catalyst Addition and Aging

	FRESH FEED	CO MAKEUP	H2 MAKEUP	K-01 OUTLET	REACTOR FEED	C-05 OUTLET	MAIN PURGE	DISTILL. PURGE	CRUDE PRODUCT	REFINED PRODUCT
T	75	50	50	109	299	248	89	56	224	76
P	739	874	523	708	701	670	656	7	185	133
Comp	67.65	1.93	77.66	75.05	72.55	65.97	75.05	8.00	0.00	0.00
(mol %)	28.18	97.05	9.12	12.98	17.92	11.76	12.98	7.00	0.00	0.00
N2	0.43	0.97	6.50	5.35	3.86	4.67	5.35	2.00	0.00	0.00
CH4	0.08	0.05	0.92	0.31	0.22	0.27	0.31	0.00	0.00	0.00
CO2	3.66	0.00	3.76	6.08	4.97	5.37	6.08	62.00	0.00	0.00
DME	0.00	0.00	0.01	0.00	0.00	0.01	0.00	4.00	0.00	0.00
MeOH	0.00	0.00	0.06	0.00	0.47	11.08	0.00	10.00	88.70	97.31
EtOH	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.00	0.17	0.02
H2O	0.00	0.00	0.00	0.00	0.00	0.66	0.00	0.00	11.12	2.67
others	0.00	0.00	1.97	0.23	0.01	0.22	0.23	7.00	0.02	0.00
TOTAL	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Mole Wt	11.003	27.501	7.770	9.445	9.942	12.053	9.445	39.217	30.554	31.668
LHV	253.4	0.0	0.0	563.4	831.0	803.6	18.2	0.9	80.4	131.2
Enthalpy	-47.47	0.00	0.00	-97.21	-132.26	-165.18	-3.17	-1.63	-31.68	-47.98
Flow	917.632	0	0	2,255,040	3,176,705	2,627,892	72,932	4,758	117,935	175,888
SCFH	2,419.0	0.0	0.0	5,944.6	8,374.2	6,927.4	192.3	12.5	310.9	463.7
lbmol/hr	26,616	0	0	56,145	83,258	83,496	1,816	492	9,499	14,683
lb/hr										

APPENDIX F - RESULTS OF DEMONSTRATION PLANT OPERATION

**Table 1 - Summary of LPMEOH™ Demonstration Unit Outages -
October/December 1997**

Table 2 - Summary of Catalyst Samples

Figure 1 - Catalyst Life (η) vs. Days Onstream - First Catalyst Batch

**Figure 2 - Reduction Gas Uptake vs. Temperature - Batch 6,
November 22-23 1997**

**Figure 3 - Sparger Resistance Coefficient vs. Days Onstream
(Post May/June 1997 Outage)**

**Figure 4 - Sparger Resistance Coefficient vs. Days Onstream
(Post-19 December 1997 Restart)**

Table 1 - Summary of LPMEOH™ Demonstration Unit Outages - October/December 1997

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

* Excluding catalyst changeout to restart test program.

Table 2

Summary of Catalyst Samples

Sample	Identity	XRD		BET m ² /g	Analytical (ppm)					
		Cu	ZnO		Fe	Ni	S	As	Cl	
14987-54 Trailer Run	Lab run using 383-4119 (450 hours) AFFTU run in Kingsport (672 hours)	175 179	74 101		172	58	<=660	184	5570	
Reduction #3	Reduction Batch sample from Kingsport				49	32	<=110	<25		
Reduction #4	Reduction Batch sample from Kingsport			57	83	28	<100	<25		
Reduction #6	Reduction Batch sample from Kingsport			73	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		

Figure 1
Catalyst Age (eta)

Figure 2

Reduction Gas Uptake vs. Temperature Batch 6 November 22-23 1997

Figure 3

Sparger Resistance Coefficient (Post-May/June 1997 Outage)

Figure 4

Sparger Resistance Coefficient (Post-December 1997 Restart)

APPENDIX G - ON-SITE PRODUCT-USE TESTING

TM
LPMEOH Refined Methanol Quality and
Fitness for Use
1997

Property	1 LIML	2 UIML	3 # Obs.	4 Ave.	5 Min.	6 Max.	7 # Out IMLs
Acetone, ppm		125	207	11.217	10	30	0
Appearance	pass	pass	207				3
Color by pcs		5	207	5	5	5	0
Decane, ppm		12	207	10.642	10	76	5
Ethanol, ppm		500	207	101.01	100	220	0
Water, % by Coulometric			170	0.0094	0.0023	0.0571	
Water, % by Titration		0.03	96	0.0127	0.0023	0.0581	5
Acetic Acid, %		0.003	207	0.0004	0.0002	0.003	0
Methanol, %	99.9		207	99.985	99.92	100	0
Alkalinity, ppm		0.8	51	0.596	0.1	2.3	11
Specific Gravity @ 20/20	0.792	0.793	96	0.7928	0.792	0.793	0

Eastman uses methanol internally only and has certain requirements that must be met before it can be accepted into the primary methanol storage tank. The chart above describes these requirements and shows the analytical tests and the statistics for all the methanol produced from the LPMEOHTM process during 1997.

Column 1 - Lower Internal Manufacturing Limit

Column 2 - Upper Internal Manufacturing Limit

Column 3 - Number of lot tank samples analyzed for this test

Column 4 - Average value of analytical test for all lot tank samples

Column 5 - Minimum value of analytical test for all lot tank samples

Column 6 - Maximum value of analytical test for all lot tank samples

Column 7 - Number of lot tank samples falling outside Internal Manufacturing Limit

The analytical tests above are run on each methanol lot with the exception of Alkalinity (every 4th lot) and Specific Gravity (every other lot). It should also be noted that the Coulometric water test was replaced by a water titration in August.

The internal manufacturing limits are based on statistic and represent process capabilities. Occasionally as noted in column 7, a lot tank sample will fall outside of that IML. When this occurs, an evaluation is made to determine how the composition of the primary storage tank would be affected. As long as the primary tank composition would not exceed the IML, the lot tank can be accepted.

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

To: Distribution

Dept/Loc:

From: E. C. Heydorn

Dept/Ext: APCI/(610) 481-7099

Date: 21 November 1997

Subject: Partnership Annual Operating Plan for FY'98

Distr: R. J. Jelle'y/EMN
W. C. Jones/EMN
S. J. Kasinecz/APCI
R. B. Moore/APCI
B. T. Street/EMN
V. E. Stein/APCI
P. J. A. Tijm/APCI

c: R. M. Kornosky/DOE-FETC

Background

The Partnership Agreement requires that an Annual Operating Plan be prepared each Fiscal Year for the approval of the Partners. Article 5.2 of the Partnership Agreement sets forth the requirements. This memo constitutes the Partnership's Annual Operating Plan for FY'98.

Goals and Objectives for FY'98

The goals and objectives for FY'98 are to continue with Phase 3 operation of the LPMEOH™ demonstration plant in accordance with the Statement of Work. The Milestone Schedule (Attachment A), the Demonstration Test Plan Schedule (Attachment B), the FY'98 Cost Plan (Attachment C) and the Project Success Factors (Attachment D) are attached for reference. These attachments summarize the Phase 3, Operation activities, and the schedule for their performance. The Partnership's major FY'98 objectives are:

- the LPMEOH™ demonstration plant will have been successfully restarted after the catalyst changeout of November 1997 and have completed all Task 2.1.1 operation (by September '98), and will have resolved those technical issues encountered during FY'97 operation (catalyst life, gas sparger resistance coefficient).

21 November 1997

- operational proof of concept testing of the LPDME process at the LaPorte AFDU will have been completed (late Summer '98).

- implementation of the Off-site Product-use Test Plan will have commenced (in October '97), and the methanol production run on CO-rich gas at the LPMEOH™ demonstration plant will have been completed (Winter/Spring '98) to supply methanol to those sites designated to receive product from Kingsport.

- the project Success Factors will continue to have been achieved during FY'98.

E/C. Heydorn

Approved:

Air Products/E. C. Heydorn

Eastman/W.C. Jones

MILESTONE SCHEDULE STATUS REPORT
LIQUID PHASE METHANOL DEMONSTRATION
DE-FC22-92PC90543

Task Name	Duration In Months	Start Date	End Date	% Com	% Sched	1993	1994	1995	1996	1997	1998	1999	2000	2001
PHASE 1: DESIGN	49.43mon	10/1/93	12/30/97	98	86%									
PROJECT DEFINITION (TASK 1)	16.78mon	10/1/93	9/30/94	100	100%									
CONTINUATION APPLICATION (B.P.#2)	0.41mon	8/2/94	8/10/94	100	100%									
PERMITTING (TASK 2)	47.31mon	11/17/93	9/10/96	100	100%									
NEPA FONSI APPROVAL	6/30/95		6/30/95	100	100%									
DESIGN ENGINEERING (TASK 3)	38.62mon	4/15/94	8/1/96	100	100%									
VENDOR ENGINEERING	33.15mon	8/10/94	7/30/96	100	100%									
OFF-SITE TESTING (TASK 4)	44.78mon	2/25/94	12/30/97	45	45%									
UPDATED FUEL TEST PLAN APPROVAL	8/29/97		8/29/97	100	100%									
DECISION TO CONTINUE DME TESTING	12/4/96		12/4/96	100	100%									
PLANNING, ADMIN & DME DVT (TASK 5)	55.54mon	10/1/93	1/20/97	100	100%									
PHASE 2: CONSTRUCTION	41.24mon	10/17/94	5/1/98	97	90%									
PROCUREMENT (TASK 1)	30.02mon	10/17/94	7/30/96	100	100%									
CONSTRUCTION (TASK 2)	22.44mon	10/2/95	1/31/97	100	100%									
TRAINING & COMMISSIONING (TASK 3)	24.92mon	9/5/95	2/27/97	100	100%									
OFF-SITE TESTING (TASK 4)	8.74mon	8/1/97	5/1/98	0	0%									
PLANNING & ADMINISTRATION (TASK 5)	34.02mon	6/1/95	5/1/98	97	96%									
CONTINUATION APPLICATION (B.P.#3)	4.46mon	5/31/96	9/4/96	100	100%									
PHASE 3: OPERATION	57.66mon	1/20/97	12/28/01	12	12%									
START-UP (TASK 1)	3.22mon	1/23/97	4/2/97	100	100%									
METHANOL OPERATION (TASK 2.1)	46.48mon	4/2/97	3/28/01	15	15%									
DISMANTLE PLANT (TASK 2.3)	6.76mon	6/1/01	12/28/01	0	0%									
ON-SITE PRODUCT USE DEMO (TASK 3)	2.02mon	8/1/97	10/2/97	0	0%									
OFF-SITE PRODUCT USE DEMO (TASK 4)	19.26mon	5/15/98	1/11/00	0	0%									
DATA ANALYSIS/REPORTS (TASK 5)	54.53mon	1/20/97	9/21/01	13	13%									
PLANNING & ADMINISTRATIVE (TASK 6)	57.66mon	1/20/97	12/28/01	14	14%									
PROVISIONAL DME IMPLEMENTATION	49.70mon	4/1/97	7/5/01	0	0%									
DME DVT (PDU TESTS) (TASK 3.6)	16.05mon	4/1/97	8/14/98	0	0%									
DECISION TO IMPLEMENT	6.94mon	3/1/98	10/1/98	0	0%									
DESIGN, MODIFY & OPERATE (TASK 3.2.2)	35.13mon	7/1/98	7/5/01	0	0%									

TABLE 5-1 - DEMONSTRATION TEST PLAN

Phase III, Task 2 - Operation

Test Run #	Test Run Description	Temp (Deg C)	Wt% Cat	H ₂ /CO Ratio at Inlet	Space Velocity (SI/hr-kg)	MeOH (tpd)	Balanced (KSCFH)	CO Gas (KSCFH)	H ₂ Gas (KSCFH)	Recycle Gas (KSCFH)	Inlet Sup. Velocity (ft/sec)	Time Period (weeks)	Elapsed Time (incl. outages) (weeks)	Start of Test
Task 2.1.1 - Process Shakedown and Catalyst Aging:														
1.	Initial Shakedown; and Design Production Tests	250	28	2.42	8,000	260	900	50	40	1,800	0.64	6	6	Nov-97
2.	Gassed Slurry Level	Part of other tests												
3.	Reactor Feed: Texaco-Type Syngas	250	28	0.67	9,240	202	650	95 (*)	0	2,612 (*)	0.77	2	9	Jan-98
4.	Early Testing @ High Superficial Velocity	250	28	2.54	10,300	TBD	1,200 (**)	50	40	2,520 (*)	0.88	2	12	Jan-98
5.	Check @ Test 1 Conditions	250	28	2.42	8,000	< 260	900	50	40	1,800	0.64	2	15	Feb-98
6.	Catalyst Addition and Aging	250 or less	28 - 40	2.51	Dec. from 8,000	237	765	40	45	Max	0.79	10	33	Mar-98 to Jul-98
7.	Free-Drain Entrained/ Condensed Oil to Reactor	250 or less	28 - 40	2.51	Dec. from 8,000	237	765	40	45	Max	0.79	During Test 6		
8.	Operation @ Design Feed Gas Rates	250	40	2.42	4,000	260	900	50	40	1,800	0.64	2	35	Jul-98
9.	Check for Limitation on Catalyst Slurry Concentration	250	> 40	2.51	Varies	TBD	765	40	45	Max (2,700)	0.79	6	42	Jul-98
10.	Catalyst Addition to Reach Max Productivity	250 or less	Target 45	2.49	3,320	256	765	40	45	2,605	0.79	12	60	Sep-98
				2.29	3,500	293	900	50	40	2,520	0.81	2		
				TBD	TBD	TBD	1,110 (***)	50	40	2,520	0.86	2		

U.S. DEPARTMENT OF ENERGY
COST PLAN

DOE F 1327
(12-85)

OMB Control No.
1910-1400

Attachment C

1. TITLE		2. IDENTIFICATION NUMBER																		
Liquid Phase Methanol Demonstration		DE-FC22-92PC90543																		
2. PARTICIPANT NAME AND ADDRESS		5. START DATE																		
Air Products Liquid Phase Conversion Co., L.P.		January 1, 1990																		
7201 Hamilton Boulevard		6. COMPLETION DATE																		
Allentown, PA 18195-4911		December 30, 2001																		
7. Element Code	8. Reporting Element	CURRENT FISCAL YEAR (FY98)												12. FUTURE FISCAL YEARS			13. Subsequent Fiscal Years	14. Total		
		OCT	NOV	DEC	JAN	FEB	MAR	APR	MAY	JUN	JUL	AUG	SEP	Total	99	00	01			
	Prior to Mod 2	0	0	0	0	0	0	0	0	0	0	0	0	0	0					16,304
1.1.1	Project Definition	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1,011
1.1.2	Permitting	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	248
1.1.3	Design Eng.	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	10,895
1.1.4	Off-Site Testing	54	54	53	106	107	106	54	54	54	54	54	53	803	0	0	0	0	0	864
1.1.5	Plan, Admin, DMB Verif Tes	2	2	2	4	4	4	2	2	2	2	2	2	30	0	0	0	0	0	3,030
1.2.1	Procurement	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	10,226
1.2.2	Construction	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	11,728
1.2.3	Training & Commissioning	47	47	46	46	47	46	0	0	0	0	0	0	279	0	0	0	0	0	864
1.2.4	Off-Site Test-Proo.&Constr	66	66	67	66	66	67	66	67	66	66	66	66	795	0	0	0	0	0	795
1.2.5	Planning & Admin	2	2	2	2	2	2	2	2	2	2	2	2	24	0	0	0	0	0	978
1.3.1	Startup	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1,497
1.3.2	Operations	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
1.3.2.1	Methanol Operation	20,321	20,321	2,373	2,374	2,374	2,374	3,154	2,374	2,374	2,374	2,374	2,375	29,266	34,635	36,137	33,470	0	0	146,862
1.3.2.2	DMB Design, Mod., Oper.	0	0	0	0	0	0	0	0	0	0	0	0	0	560	1,207	0	0	0	1,767
1.3.2.3	LEMOBH Disinfectant	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	472
1.3.3	On-Site Product Use Demo	2	2	2	2	2	2	2	2	2	2	2	2	24	1,595	45	0	0	0	1,688
1.3.4	Off-Site Product Use Demo	0	0	0	0	0	0	0	0	0	0	0	0	47	49	93	0	0	0	296
1.3.5	Data Analysis & Reports	390	63	4	4	4	4	4	4	3	4	4	3	44	652	936	0	0	0	4,170
1.3.6	Planning & Admin.	520	461	81	81	81	81	81	81	81	81	81	81	972	1,149	652	936	0	0	4,170
15. TOTAL		78,337	70,387	2,631	2,631	2,629	2,683	2,687	2,685	3,365	2,586	2,584	2,585	32,237	36,415	39,645	35,016	0	0	213,700
16. DOLLARS EXPRESSED IN:		17. SIGNATURE OF PARTICIPANT'S PROJECT MANAGER AND DATE																		
Thousands		18. SIGNATURE OF PARTICIPANTS AUTHORIZED FINANCIAL REPRESENTATIVE AND DATE
		11/4/97

**DOE/Air Products/Eastman
SUCCESS FACTORS
LPMEOH™ DEMONSTRATION PROJECT**

The three participants will judge the project success on the following factors:

- SAFE AND ENVIRONMENTALLY SOUND OPERATION
- DEMONSTRATE THE NEW TECHNOLOGY:
 - RESOLVE ALL TECHNICAL ISSUES
 - ACQUIRE SUFFICIENT ENGINEERING DATA FOR COMMERCIAL DESIGNS
 - OBTAIN INDUSTRY ACCEPTANCE
- FOR EASTMAN OPERATION AT KINGSPORT:
 - NO ADVERSE IMPACT DURING DEMONSTRATION
 - VALUABLE PLANT ASSET AT END
- MEET BUDGET AND SCHEDULE EXPECTATIONS
- POSITIVE WORKING RELATIONSHIPS BETWEEN THE PARTICIPANTS

Bob Kamosky

Bill Brown

Bill Jones

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

NOTES FROM MEETING

DISTRIBUTION (NAME/ORGANIZATION) *Unable to attend. **Chairman				COPIED FOR INFORMATION ONLY	
Bill Jones - EMN Bob Kornosky - DOE-FETC Bob Moore - APCI John Shen - DOE-HQ Barry Street - EMN				Bill O'Dowd - DOE-FETC Ed Schmetz - DOE-HQ Bob Senn - APCI Van Eric Stein - APCI Peter Tijm - APCI Bernie Toseland - APCI	
FROM		ORGANIZATION		EXTENSION	
Edward C. Heydorn SA 1898		APCI - Program Manager		17099	
DATE OF MEETING		WEEKDAY		TODAY'S DATE	
16 & 17 Dec. 1997		Tues. & Wed.		01/09/98	
		TIME		LOCATION	
		STARTED 12:15 PM ENDED 11:30 AM		APLPCC (JV) Trailer, Kingsport, TN	

SUBJECT AND/OR PURPOSE

• Project Review Meeting - DOE, Eastman, and Air Products

ITEM NO.	RESPONSIBLE PERSON (INITIALS)	TARGET DATE	DISCUSSION
1.			<p><u>Operations/Plant Performance (Task 3.2)</u></p> <p>The discussions focused on the attempt at restarting the LPMEOH™ Demonstration Unit on 26 November, and the inspection following the restart attempt.</p> <p>Activation of the 8 batches of catalyst in preparation for restarting the unit proceeded smoothly. The last 6 batches were activated using 7 drums of catalyst instead of the 6 drums used all prior reductions. All 8 batches showed the same curve of uptake of reductant versus temperature.</p> <p>During the activation of the eighth batch of catalyst, the other seven batches were pressure-transferred from the slurry tank to the reactor on 26 November. Heat-up of the reactor using 600 psig steam on the internal heat exchanger was proceeding smoothly; however, the pressure drop at the sparger was increasing with time, from 1.4 psi to 2.1 psi over a 4 hour period. The 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. When the eighth batch of slurry was transferred to the reactor, the pressure drop rose to 14 psi. After this transfer, the recycle compressor began to show signs of approaching 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.</p> <p>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 showed</p>

NOTES FROM MEETING
CONTINUATION

ITEM NO.	RESPONSIBLE PERSON (INITIALS)	TARGET DATE	DISCUSSION
			<p>indications that the nitrogen flow to the reactor was low, and 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; less than 100 ppm by weight was detected. The conclusion is that 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.</p> <p>Air Products has (outside of the Clean Coal Program) begun a program to look at alternative gas sparger designs. During the reactor inspection in early December, Air Products and Eastman discussed the possibility of testing some of the design ideas during the next reactor start-up. Air Products developed an alternative sparger design based upon some of the design changes which have been considered in this proprietary program. A sketch of the revised gas sparger was provided to Eastman; Eastman has a shop at the Kingsport site which was able to perform the fabrication. The work was completed on 16 December, and the sparger was installed in the reactor that evening.</p> <p>A discussion on the possible causes of the loss of catalyst life followed. Air Products indicated that the known causes for the increase in crystal size of the copper (which is a component of catalyst aging) are poisoning and excessive temperature. Autoclave tests on arsine and sulfur compounds have been completed, with no indication of extraordinary deactivation of the catalyst or promotion of crystal growth of the copper. There has been no indication of the presence of hot spots in the reactor. However, the discussion led to a recommendation (<i>being implemented</i>) to begin operation with the fresh catalyst at a temperature high enough to produce the nameplate capacity of the LPMEOH™ Demonstration Unit (80,000 gallons per day) with a reasonable conversion of the fresh syngas to methanol; for a Balanced Gas flow of 900 KSCFH, this would correspond to a purge of 50-100 KSCFH. Expectations are that, for fresh catalyst, the required temperature would be less than the 250°C value used during the initial start-up in April/May of 1997.</p> <p>The Air Products' Alternative Fuels Field Trailer Unit (AFFTU), which had been installed at the Kingsport site in May/June of 1995, was brought to Kingsport prior to the November restart attempt. The autoclave will be run in parallel with the reactor, and the analytical equipment will be used to measure the levels of carbonyls and sulfur species.</p>

**NOTES FROM MEETING
CONTINUATION**

Page three Of six

ITEM NO.	RESPONSIBLE PERSON (INITIALS)	TARGET DATE	DISCUSSION
2.	ECH/PJAT	Ongoing	<p>The spent catalyst which was drained from the reactor in November is awaiting shipment to the reclaimer. So far, no issues involving the acceptance of this material have been identified.</p> <p><u>DME Design Verification Testing</u></p> <p>The transfer of the dehydration catalyst production technique from Air Products to the commercial catalyst manufacturer (Calsicat) has proceeded smoothly to date. A 30 pound batch of the dehydration catalyst has been prepared at Calsicat's facilities. Tests on this material indicate that the catalyst has the proper chemical composition and initial activity. Work on optimizing the production technique is continuing. A production test at Calsicat's pilot plant is next, followed by operation in their commercial unit. The scheduled completion data for the production of the commercial catalyst is early Summer of 1998. All work at Calsicat is funded under the Alt. Fuels Program managed by Air Products. The status of the test at the LaPorte AFDU is still in doubt, due to the funding status of the Alt. Fuels Program. Once the funding status is resolved, Air Products will communicate this information to the participants in the Clean Coal Technology Program.</p>
	PJAT	1/16/98	<p>DOE asked if there was any published information on the cooking fuel tests being conducted at Penn State University. Air Products will check on the level (if any) of funding by the Clean Coal Technology Program, and the availability of information. DOE is receptive to cost-sharing the work at Penn State as part of the product-use testing task of the Clean Coal Technology project.</p>
3.	WCJ/ECH	1/19/98	<p><u>Public Design Report</u></p> <p>Air Products has been working on a revision to the Public Design Report, using Attachment 9 from Mod. 5 of the DOE Cooperative Agreement as the basis for the revision. Air Products has submitted an updated equipment breakdown by area to the DOE. A decision on accepting this change is pending. Eastman will review the most recent issue of the Report as well as the Contract Mod. regarding the operating cost information which can be published. A proposal on the publication of operating costs will be sent to DOE for review. Once this has been finalized, Air Products will issue a revised Public Design Report.</p>

**NOTES FROM MEETING
CONTINUATION**

ITEM NO.	RESPONSIBLE PERSON (INITIALS)	TARGET DATE	DISCUSSION
4.			<p><u>Topical Reports</u></p> <p><u>Process Economics Topical Report</u> - Air Products has the most recent comments from DOE (31 March 1997). This report continues to be on hold, pending resolution of capital and operating cost issues related to the Public Design Report.</p> <p><u>Peroxide Formation</u> - This report has been issued and approved.</p> <p><u>Catalyst Poisons</u> - The draft report has been issued by the Alt. Fuels Program. Air Products and DOE have agreed to publish a single report under two contract numbers. This report is on hold pending a review by the Alt. Fuels Program.</p>
4a.	VES	Target 1/98	<p><u>Reactor Design</u> - VanEric Stein has begun assembling this report. He is awaiting input from Dave Drown prior to releasing to DOE.</p>
	VES	Target 1/31/98	<p><u>Demonstration Technology Start-up Report</u></p> <p>DOE and Air Products agreed that the Demonstration Technology Start-up Report should focus on equipment issues, and that issues related to catalyst performance should be covered under a test series or Topical Report. DOE and Air Products will decide on the scope of the test series report. Air Products will finalize the Start-up Report and release to DOE.</p>
5.			<p><u>Reports - Quarterly. Other</u></p>
	ECH	1/19/98	<p>Technical Progress Report (TPR) No. 13 is being reviewed by DOE. Starting with TPR No. 14, the format for the report will be changed from a task breakdown to an activity breakdown; this will eliminate some of the boilerplate text. Air Products will submit a modified Table of Contents for review by DOE.</p>
	ECH	Done	<p>Comments from DOE on Environmental Monitoring Report (EMR) No. 1 have been received. Based on these remarks and additional information being collected by Eastman, this report will be sent to DOE for final review and approval. This also impacted the</p>
	ECH	Done	<p>information requirements for EMR No. 2; this report will be prepared and issued in draft form to DOE.</p>

**NOTES FROM MEETING
CONTINUATION**

Page five Of six

ITEM NO.	RESPONSIBLE PERSON (INITIALS)	TARGET DATE	DISCUSSION
6.	ECH	1/30/98	<p>The DOE Cooperative Agreement specifies that an updated Demonstration Test Plan be issued annually. Once the LPMEOH™ Demonstration Unit has been restarted, Air Products will send the revised Plan to DOE.</p> <p>The status of reports as tabulated by DOE is attached.</p> <p><u>Conferences/Papers</u></p> <p><u>World Methanol Conference (08 - 10 December 1997)</u> - This presentation was well received at the conference; Barry Street did an excellent job in assembling the paper and co-presenting with ECH.</p> <p><u>23rd International Technical Conference on Coal Utilization & Fuel Systems (09-13 March 1998)</u> - Air Products has accepted an invitation to present a paper at this forum. DOE and Air Products agreed that this paper will focus more on the past than any new data from Kingsport; new data will be presented at the Clean Coal Technology Conference. The final version of the paper is due in early February.</p> <p><u>Clean Coal Technology Conference (April/May 1998)</u> - Initial information on the conference has been received. The final paper is due in early February.</p>
			<p><u>Off-Site Testing (Product-Use Demonstrations) - Task 3.4</u></p> <p>Methanol from the LaPorte AFDU has been sent to 4 of the sites for the off-site product-use testing. A detailed status update will be included in TPR No. 14. A summary of the status will be included in the January monthly report.</p>
			<p>A review meeting with Acurex is still anticipated for April/May 1998. A series of dates will be proposed by Air Products and Acurex.</p>
8.			<p><u>On-Site Fitness-for-Use Testing - Task 3.3</u></p> <p>Barry Street has assembled statistical data on the quality of the Refined Methanol delivered from the LPMEOH™ Demonstration Unit to Eastman. This information will be included in TPR No. 14, and will complete the activities on On-Site Fitness-for-Use Testing.</p>

**NOTES FROM MEETING
CONTINUATION**

ITEM NO.	RESPONSIBLE PERSON (INITIALS)	TARGET DATE	DISCUSSION
9.	ECH/RMK	2/15/98	<p><u>Report Card - Project Success Factors</u></p> <p>All participants are pleased with the efforts to continue to meet the success factors. The successful restart of the LPMEOH™ Demonstration Unit is clearly the near-term goal, and monitoring of changes in catalyst activity will continue.</p>
10.			<p><u>Next Meeting</u></p> <p>Air Products suggested an interim Review Meeting at the FETC offices in Pittsburgh. A decision on the need for such a meeting will be made pending the operating status of the plant and the resolution of issues related to the Public Design Report.</p> <p>DOE suggested the possibility of holding Project Review Meetings on a rotating basis at Kingsport, Pittsburgh, and Allentown. The decision on the location of the March 1998 Meeting is pending.</p>
11.			<p><u>Miscellaneous</u></p> <p>Air Products presented the status of two plant improvement projects under consideration by the Partnership. A booster compressor is required to increase the pressure of the H₂ Gas stream to the LPMEOH™ Demonstration Unit. The operation of the existing methanol plant at reduced gas rates has dictated the lowering of operating pressure. If this had been known during the design phase, the booster compressor would have been included at that time. Also, Air Products is studying the feasibility of activating all of the catalyst at one time in the reactor instead of on a batch basis in the reduction equipment. This has implications for reducing the capital costs for future LPMEOH™ plants. Air Products will provide a technical overview of the trade-off between on-line catalyst addition/withdrawal and in-situ activation in the reactor at the next Project Review Meeting.</p> <p>Warren Miller has joined the Air Products team as Commercial Manager for the Liquid Phase Technologies area. He reports to Peter Tijm and will focus on the development of future opportunities for LPMEOH™ and related chemistries.</p>
	ECH/VES	3/98	

LPMEOH™ Reporting Status
December 15, 1997

Quarterly Technical Progress Reports

No. 1 (October 1, 1993 to June 30, 1994)	Approved
No. 2 (July 1, 1994 to September 30, 1994)	Approved
No. 3 (October 1, 1994 to March 31, 1995)	Approved
No. 4 (April 1, 1995 to June 30, 1995)	Approved
No. 5 (July 1, 1995 to September 30, 1995)	Approved
No. 6 (October 1, 1995 to December 31, 1995)	Approved
No. 7 (January 1, 1996 to March 31, 1996)	Approved
No. 8 (April 1, 1996 to June 30, 1996)	Approved
No. 9 (July 5, 1996 to September 30, 1996)	Approved
No. 10 (October 1, 1996 to December 31, 1996)	Approved
No. 11 (January 1, 1997 to March 31, 1997)	Approved
No. 12 (April 1, 1997 to June 30, 1997)	Approved
No. 13 (July 1, 1997 to September 30, 1997)	Submitted For DOE Review
No. 14 (October 1, 1997 to December 31, 1997)	First Draft Due February 15, 1998

Topical Reports

No. 1 (Peroxide Formation)	Approved
No. (DME Laboratory and Market Studies)	Need First Draft
No. (Catalyst Poisons)	Submitted For DOE Review
No. (Economic Analysis)	Need Revised Draft
No. (Liquid Phase Reactor Design)	Need First Draft

Environmental

Environmental Information Volume	Approved
Environmental Monitoring Plan	Approved
Environmental Monitoring Report	
No. 1 (April 1, 1997 to June 30, 1997)	Need Revised Draft
No. 2 (July 1, 1997 to September 30, 1997)	First Draft Due November 15, 1997
No. 2 (October 1, 1997 to December 31, 1997)	First Draft Due February 15, 1998

Technical

5 TH Clean Coal Technology Conference Paper	Approved
6 TH Clean Coal Technology Conference Paper	Due February 15, 1998
AIChE Spring 1997 Conference Paper	Approved
American Chemical Society Spring 1997 Paper	Approved
Power-Gen Europe '97 Conference Paper	Published
1997 Gasification Technologies Conference Paper	Approved
World Methanol Conference Paper	Approved
23 rd Coal Utilization & Fuel Systems Conference Paper	Due February 15, 1998
Industrial Catalysis News Article	Due February 15, 1998
Demonstration Test Plan	Approved
Final Report - Volume 1 - Public Design	Need Revised Draft
Demonstration Technology Start-Up Report	Due June 1, 1997
Test Series Report	As Required

Other

Project Management Plan	Approved
DME Decision Memo	Recommendation Accepted

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

MILESTONE SCHEDULE STATUS REPORT
LIQUID PHASE METHANOL DEMONSTRATION
DE-FC22-92PC90543

Task Name	Duration In Months	Start Date	End Date	% Com	% Sched	1993	1994	1995	1996	1997	1998	1999	2000	2001
PHASE 1: DESIGN														
PROJECT DEFINITION (TASK 1)	49.43mon	10/1/93	12/30/97	98	91%									
CONTINUATION APPLICATION (B.P.#2)	16.78mon	10/1/93	9/30/94	100	100%									
PERMITTING (TASK 2)	0.41mon	8/2/94	8/10/94	100	100%									
NEPA FONSI APPROVAL	47.31mon	11/17/93	9/10/96	100	100%									
DESIGN ENGINEERING (TASK 3)	6/30/95		6/30/95	100	100%									
VENDOR ENGINEERING	38.62mon	4/15/94	8/1/96	100	100%									
OFF-SITE TESTING (TASK 4)	33.15mon	8/10/94	7/30/96	100	100%									
UPDATED FUEL TEST PLAN APPROVAL	44.78mon	2/25/94	12/30/97	65	65%									
DECISION TO CONTINUE DME TESTING	8/29/97		8/29/97	100	100%									
PLANNING, ADMIN & DME DVT (TASK 5)	12/4/96		12/4/96	100	100%									
PHASE 2: CONSTRUCTION														
PROCUREMENT (TASK 1)	55.54mon	10/1/93	1/20/97	100	100%									
CONSTRUCTION (TASK 2)	41.24mon	10/17/94	5/1/98	97	91%									
TRAINING & COMMISSIONING (TASK 3)	30.02mon	10/17/94	7/30/96	100	100%									
OFF-SITE TESTING (TASK 4)	22.44mon	10/2/95	1/31/97	100	100%									
PLANNING & ADMINISTRATION (TASK 5)	24.92mon	9/5/95	2/27/97	100	100%									
CONTINUATION APPLICATION (B.P.#3)	8.74mon	8/1/97	5/1/98	0	0%									
PHASE 3: OPERATION														
START-UP (TASK 1)	34.02mon	6/1/95	5/1/98	100	100%									
METHANOL OPERATION (TASK 2.1)	4.46mon	5/31/96	9/4/96	100	100%									
DISMANTLE PLANT (TASK 2.3)	57.66mon	1/20/97	12/28/01	16	16%									
ON-SITE PRODUCT USE DEMO (TASK 3)	3.22mon	1/23/97	4/2/97	100	100%									
OFF-SITE PRODUCT USE DEMO (TASK 4)	46.48mon	4/2/97	3/28/01	19	19%									
DATA ANALYSIS/REPORTS (TASK 5)	6.76mon	6/1/01	12/28/01	0	0%									
PLANNING & ADMINISTRATIVE (TASK 6)	2.02mon	11/3/97	1/7/98	90	90%									
PROVISIONAL DME IMPLEMENTATION	19.26mon	12/1/97	7/27/99	5	5%									
DME DVT (PDU TESTS) (TASK 3.6)	54.53mon	1/20/97	9/21/01	17	17%									
DECISION TO IMPLEMENT	57.66mon	1/20/97	12/28/01	14	14%									
DESIGN, MODIFY & OPERATE (TASK 3.2.2)	49.70mon	4/1/97	7/5/01	0	0%									
	16.05mon	4/1/97	8/14/98	0	0%									
	6.94mon	3/1/98	10/1/98	0	0%									
	35.13mon	7/1/98	7/5/01	0	0%									

[illegible]

COPY

U.S. DEPARTMENT OF ENERGY
COST MANAGEMENT REPORT

1. TITLE Liquid Phase Methanol Demonstration		2. REPORTING PERIOD December 01, 1997 through December 31, 1997		3. IDENTIFICATION NUMBER DE-FC22-92PC90543	
2. PARTICIPANT NAME AND ADDRESS Air Products Liquid Phase Conversion Co., L.P. 7201 Hamilton Boulevard Allentown, PA 18195-4911		5. COST PLAN DATE October 29, 1997		6. START DATE January 1, 1990	
8. ELEMENT 9. REPORTING ELEMENT		7. COMPLETION DATE December 31, 2001			
10. ACCRUED COSTS		11. ESTIMATED ACCRUED COSTS			
Reporting Period		Cumulative to Date		12. Total Plan Value	
a. Actual	b. Plan	c. Actual	d. Plan	d. Subsequent FY's (4)	e. Total (3)
0	0	16,304	16,304		16,304
Project Definition		1,011			1,011
Permitting		248			248
Design Engr.		10,895			10,895
OFF-site Testing		76			76
Planning, Admin. & DME Verif. Testing		3,001			3,001
Procurement		10,232			10,232
Construction		11,730			11,730
Train. & Commissioning		585			585
OFF-Site Test - Proc. & Constr.		0			0
Planning & Admin		67			67
Startup		2			2
Operations		954			954
Methanol Operation		1,500			1,500
DME Design, Mod., Oper.		19,547			19,547
LPMEOH Dismantlement		0			0
On-Site Product Use Demo		0			0
Off-Site Product Use Demo		0			0
Data Analysis & Reports		0			0
Planning & Admin.		81			81
TOTAL		2,157			2,157
15. DOLLARS EXPRESSED IN:		Thousands			
16. SIGNATURE OF PARTICIPANT'S PROJECT MANAGER		2,629		78,867	
AND DATE		17 Jan 98		23,074	
17. SIGNATURE OF PARTICIPANTS AUTHORIZED FINANCIAL REPRESENTATIVE AND DATE		36,415		39,645	
DATE		17 Jan 98		35,035	
DATE		17 Jan 98		213,700	
DATE		17 Jan 98		213,700	