

RECEIVED

JAN 19 1999

OSTI

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

**Quarterly Report
July 1 - September 30, 1997**

Work Performed Under Contract No.: DE-FC22-92PC90543

For
U.S. Department of Energy
Office of Fossil Energy
Federal Energy Technology Center
P.O. Box 880
Morgantown, West Virginia 26507-0880

By
Air Products and Chemicals, Inc.
Allentown, Pennsylvania

Disclaimer

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

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). 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 reporting period, DOE accepted the recommendation to continue with dimethyl ether (DME) design verification testing (DVT). DME design verification testing studies show the liquid phase DME (LPDME) process will have a significant economic advantage for the coproduction of DME for local markets. An LPDME catalyst system with reasonable long-term activity and stability is being developed. Planning for a proof-of-concept test run at the LaPorte Alternative Fuels Development Unit (AFDU) was recommended. DOE issued a letter dated 31 July 1997 accepting the recommendation to continue design verification testing. In order to allow for scale-up of the manufacturing technique for the dehydration catalyst from the pilot plant to the commercial scale, the time required to produce the catalyst to the AFDU has slipped. The new estimated delivery date is 01 June 1998.

During this quarter, eight sites were recommended to DOE as candidates for participation in the off-site, product-use test plan. Seven of the eight proposals have been defined in sufficient detail so that final planning and implementation should begin. DOE accepted Air Products' recommendation to proceed with the seven projects. Planning work on these tests has begun, and Air Products is preparing a recommended list of sites where methanol produced from carbon monoxide (CO)-rich synthesis gas (syngas) at the LaPorte AFDU can be used. This has become necessary since several of the off-site, product-use tests are scheduled to begin prior to the specific production campaign of methanol from CO-rich syngas at the demonstration unit.

The dedication ceremony for the LPMEOH™ Demonstration Unit was held on 25 July 1997. Senior management from DOE, Eastman, and Air Products participated.

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, but at a slower rate than during the April/May operation. Catalyst slurry samples taken from the LPMEOH™ Reactor have shown an increase in the levels of iron, arsenic, and sulfur as compared with fresh catalyst. The increase in iron appears to be the result of a one-time startup source, such as construction debris. Eastman has made plans to change out an upstream guard bed designed for the removal of arsine and sulfur. Laboratory work has confirmed that the process conditions during the April startup at the demonstration unit were not the cause of the decline in

catalyst activity during that period. Tests on the effects of possible poisons of methanol synthesis catalyst are ongoing.

Pressure drop and resistance coefficient across the gas sparger at the bottom of the reactor have been stabilized at a manageable level by flushing with entrained slurry collected within the process. This flush is applied through a connection at the gas inlet line to the reactor. The rate of addition of the entrained slurry was 30 gallons per minute, and can be performed 2 to 3 times per day.

A 12-day test on simulated Texaco-type synthesis gas ($H_2/CO = 0.8$) concluded on 12 August 1997. Methanol production averaged 57,100 gallons per day (190 TPD) during the period, and the crude product composition matched expectations for bulk components.

Over the reporting period, a total of 4,360,255 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. All methanol was used by Eastman in the production of methyl acetate, and ultimately cellulose acetate and acetic acid. No safety or environmental incidents were reported during this quarter. At the end of the quarter, slurry concentration in the reactor reached the design 40 wt% level for the first time.

Ninety-nine percent (99%) of the \$38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 30 September 1997. Ten percent (10%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 September 1997.

Table of Contents

Abstract	3
ACRONYMS AND DEFINITIONS.....	7
Executive Summary	9
A. Introduction.....	12
B. Project Description.....	13
C. Process Description.....	14
D. Results and Discussion.....	14
Task 1.2 Permitting.....	14
Task 1.3 Design Engineering.....	15
Task 1.4 Off-Site Testing (Definition and Design)	15
Task 1.5 Planning and Administration	16
Task 1.5.1 Product-Use Test Plan	16
Task 1.5.2 Commercialization Studies.....	17
Task 1.5.3 DME Design Verification Testing	18
Task 1.5.4 Administration and Reporting.....	21
Task 2.1 Procurement	22
Task 2.2 Construction	22
Task 2.3 Training and Commissioning	22
Task 2.4 Off-Site Testing (Procurement and Construction)	23
Task 2.5 Planning and Administration	23
Task 3.1 Start-up	24
Task 3.2 LPMEOH™ Process Demonstration Facility Operation.....	24
Task 3.2.1 Methanol Operation	24
Task 3.2.2 DME Design, Modification and Operation	30
Task 3.3 On-Site Testing (Product-Use Demonstration)	30
Task 3.4 Off-Site Testing (Product-Use Demonstration).....	30
Task 3.5 Data Analysis and Reports	30
Task 3.6 Planning and Administration.....	30
E. Planned Activities for the Next Quarter	31
F. Conclusion.....	31

Table of Contents (cont'd)

APPENDICES.....	34
APPENDIX A - SIMPLIFIED PROCESS FLOW DIAGRAM.....	34
APPENDIX B - PROJECT EVALUATION PLAN FOR BUDGET PERIOD NO. 2..	35
APPENDIX C - TASK 1.4 - OFF-SITE TESTING (DEFINITION AND DESIGN)...	36
APPENDIX D - TASK 1.5.2 - PROCESS ECONOMIC STUDY	37
APPENDIX E - TASK 1.5.3 - DME DESIGN VERIFICATION TESTING.....	38
APPENDIX F - TASK 1.5.4 - APPROVAL FOR BUDGET PERIOD THREE	39
APPENDIX G - TASK 2.5 - PARTNERSHIP ANNUAL PLAN	40
APPENDIX H - TASK 3.2.1 - SAMPLES OF DETAILED MATERIAL BALANCE REPORTS.....	41
APPENDIX I - TASK 3.2.1 - RESULTS OF DEMONSTRATION PLANT OPERATION	42
APPENDIX J - TEST AUTHORIZATION K3 - METHANOL SYNTHESIS WITH TEXACO-TYPE SYNGAS	43
APPENDIX K - TASK 3.6 - PROJECT REVIEW MEETING (24-25 JULY 1997).....	44
APPENDIX L - TASK 3.6 - PROJECT REVIEW MEETING (24-25 SEPTEMBER 1997)	45
APPENDIX M - TASK 3.6 - MILESTONE SCHEDULE STATUS AND COST MANAGEMENT REPORTS	46

ACRONYMS AND DEFINITIONS

Acurex	-	Acurex Environmental Corporation
Air Products	-	Air Products and Chemicals, Inc.
AFDU	-	Alternative Fuels Development Unit - The "LaPorte PDU"
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
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)
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
ΔP	-	pressure drop, psi
Partnership	-	Air Products Liquid Phase Conversion Company, L.P.
PDU	-	Process Development Unit

ACRONYMS AND DEFINITIONS (cont'd)

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
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). 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 synthesis gas (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 reporting period, DOE accepted the recommendation to continue with dimethyl ether (DME) design verification testing. DME design verification testing studies show the liquid phase DME (LPDME) process will have a significant economic advantage for the coproduction of DME for local markets. The market applications for DME are large. An LPDME catalyst system with reasonable long-term activity and stability is being developed. Planning for a proof-of-concept test run at the LaPorte Alternative Fuels Development Unit (AFDU) was recommended. DOE issued a letter dated 31 July 1997 accepting the recommendation to continue design verification testing.

The initial schedule for production of the dehydration catalyst for the test run called for use of pilot plant equipment used in multiple campaigns. In order to allow for scale-up of the catalyst manufacturing technique to the commercial scale, the time required to produce the catalyst to the AFDU has slipped. The new estimated delivery date 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.

During this quarter, eight project sites were recommended to DOE as candidates for participation in the off-site, product-use test plan. Seven of the eight proposals have been defined in sufficient detail so that final planning and implementation should begin. DOE accepted Air Products' recommendation to proceed with the seven projects. Planning work on these tests has begun, and Air Products is preparing a recommended list of project sites where methanol produced from carbon monoxide (CO)-rich syngas at the LaPorte AFDU can be used. This has become necessary since several of the off-site, product-use tests are scheduled to begin prior to the specific production campaign of methanol from CO-rich syngas at the demonstration unit.

The dedication ceremony for the LPMEOH™ Demonstration Unit was held on 25 July 1997. Senior management from DOE, Eastman, and Air Products participated.

Two project review meetings were held during the reporting period. Both meetings focused on reviewing the performance of the demonstration unit, the catalyst development work for the upcoming LPDME proof-of-concept test at the LaPorte AFDU, and the status of the off-site product-use test plan.

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, but at a slower rate than during the April/May operation. Catalyst slurry samples taken from the LPMEOH™ Reactor have shown an increase in the levels of iron, arsenic, and sulfur as compared with fresh catalyst. The increase in iron appears to be the result of a one-time startup source, such as construction debris. Eastman has made plans to change out an upstream guard bed designed for the removal of arsine and sulfur. Because of material delivery times, this work is scheduled for 01 October 1997. Laboratory work has confirmed that the process conditions during the April startup at the demonstration unit were not the cause of the decline in catalyst activity during that period. Tests on the effects of possible poisons of methanol synthesis catalyst are ongoing.

Pressure drop and resistance coefficient across the gas sparger at the bottom of the reactor have been stabilized at a manageable level by flushing with entrained slurry collected at the cyclone and secondary oil knock-out drum. This flush is applied through a connection at the gas inlet line to the reactor. The rate of addition of the entrained slurry was 30 gallons per minute, and can be performed 2 to 3 times per day at the average rate of liquid traffic through the cyclone and secondary oil knock-out drum.

A 12-day test on simulated Texaco-type synthesis gas ($H_2/CO = 0.8$) concluded on 12 August 1997. Methanol production averaged 57,100 gallons per day (190 TPD) during the period, and the crude product composition matched expectations for bulk components.

Over the reporting period, a total of 4,360,255 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, a total of 7,260,947 gallons of methanol has been produced. All methanol was used by Eastman in the production of methyl acetate, and ultimately cellulose acetate and acetic acid. No safety or environmental incidents were reported during this quarter. At the end of the quarter, slurry concentration in the reactor reached the design 40 wt% level for the first time.

Ninety-nine percent (99%) of the \$38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 30 September 1997. Ten percent (10%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 September 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, and then by the goals established by the Project Evaluation Plan for Budget Period No. 2 (see Appendix B). Major accomplishments during this period are as follows:

Task 1.2 Permitting

For this task the Project Evaluation Plan for Budget Period No. 2 establishes these goals:

- Issue the Final Environmental Information Volume (EIV) to support the DOE's Environmental Assessment/Finding of No Significant Impact.
 - The NEPA review was completed 30 June 1995 with the issuance of an Environmental Assessment (DOE/EA-1029) and Finding of No Significant Impact (FONSI). The Final Environmental Information Volume was approved by the DOE on 29 August 1996. Copies of the Final EIV were distributed in September of 1996.
- Obtain permits necessary for construction and operation.
 - The construction and operation permits have been obtained.

Task 1.3 Design Engineering

For this task the Project Evaluation Plan for Budget Period No. 2 establishes these goals:

- Prepare the Environmental Monitoring Plan (EMP).
 - The DOE approved the Draft Final EMP on 29 August 1996. Copies of the Final EMP were distributed in September of 1996.
- Complete the design engineering necessary for construction and commissioning. This includes Piping and Instrumentation Diagrams, Design Hazard Reviews, and the conduct of design reviews.
 - Task 1.3 Design Engineering is complete.

Task 1.4 Off-Site Testing (Definition and Design)

The Project Evaluation Plan for Budget Period No. 2 establishes the following goal for this task:

- Prepare the product-use demonstration plan for Phase 3, Task 4 Off-Site Product-Use Demonstration. This off-site test plan will be incorporated into an updated, overall (fuel and chemical) product-use test plan (in Phase 1, Task 5).

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 of-site tests may be shipped from the inventory held at the Alternative Fuels Development Unit in LaPorte, TX. Air Products, Acurex Environmental Corporation (Acurex), and the DOE will develop the final off-site, product-use test plan.

Activity during this quarter

- Acurex and Air Products have been working to identify a variety of sites and applications for product-use tests. During the 29-30 April 1997 interim review meeting, Air Products presented a status update on these activities to the DOE. A total of 22 projects have been screened by their likelihood to proceed and the timing for the initial methanol requirement. Eight sites from the list have met these criteria; these are summarized in Appendix C. At present, full proposals and cost breakdowns are being developed by Acurex and each of the eight possible participants. Due to the timing and quantities of methanol required by the earliest tests, Air Products and DOE are considering the use of methanol produced from carbon monoxide (CO)-rich syngas feeds from the LaPorte Alternative Fuels Development Unit (AFDU). This will allow for some initial testing to occur during calendar year 1997, when some of these projects will be ready to proceed. The Demonstration Test Plan indicates methanol for the remaining four tests (as-produced from CO-rich syngas) will first be produced in May of 1998. Air Products formally recommended that seven of the eight projects have been defined in sufficient detail so that final planning and implementation should begin. The eighth project, involving the testing of a water/naphtha/methanol emulsion as a transportation fuel, is awaiting final project definition. DOE accepted Air Products' recommendation to proceed with the seven projects in August of 1997. Planning work on these tests has begun, and Air Products is preparing a recommended list of sites where methanol from the LaPorte AFDU (as described above) can be used.

Task 1.5 Planning and Administration

Task 1.5.1 Product-Use Test Plan

The Project Evaluation Plan for Budget Period No. 2 establishes the following goal for this task:

- Update the (fuel and chemical) product-use test plan to better meet the technical objectives of the project and serve the needs of commercial markets.
- Air Products and Eastman have updated plans for the on-site product-use demonstrations. The schedule for on-site product-use tests was established for August to October of 1997. Methanol product from the LPMEOH™ Process Demonstration Unit will be used as a chemical feedstock. Eastman will perform fitness-for-use tests on the methanol product for use as a chemical feedstock and provide a summary of the results.

Task 1.5.2 Commercialization Studies

The Project Evaluation Plan for Budget Period No. 2 establishes the following goal for this task:

- Complete economic studies of important commercial aspects of the LPMEOH™ process to enhance IGCC electric power generation. These studies will be used to provide input to the LPMEOH™ Process Demonstration Unit's Demonstration Test Plan (Phase 2, Task 3).

Discussion

Several areas have been identified as needing 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 D. 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" were received during the reporting period. This Topical Report develops plant design options for the LPMEOH™ process, as an add-on to IGCC power plants for the coproduction of methanol and power. Part One also compares the LPMEOH™ (LP) process with gas phase (GP) methanol processes in the environment of coal-derived syngas. Surprisingly, the LP technology can coproduce methanol at less than 50 cents per gallon, even at relatively small (400 to 1200 TPD) methanol plant sizes. LP's advantage over GP is 6 to 9 cents per gallon. Therefore, when baseload IGCC power is viable, the LP technology makes coproduction viable. Air Products and DOE are reviewing this report so that the cost breakdown by plant area matches the format to be used in the Final Report - Volume 1 - Public Design, currently being reviewed by DOE. Once the area breakdown has been developed 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", 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).

Task 1.5.3 DME Design Verification Testing

The Project Evaluation Plan for Budget Period No. 2 establishes the following goal for this task:

- Perform initial Design Verification Testing (DVT) for the production of dimethyl ether (DME) as a mixed coproduct with methanol. This activity includes laboratory R&D and market economic studies.

Discussion

The first decision milestone, on whether to continue with DME DVT, was targeted for 01 December 1996. This milestone was relaxed to July of 1997 to allow time for further development of the 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 E.

Action during this quarter included a recommendation to continue with DME DVT, Market Economic Studies, and Laboratory R&D.

DME DVT Recommendation

Air Products made a 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 E. 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. The markets and this catalyst system is sufficiently promising that proof-of-concept planning for the LaPorte AFDU is 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 Indirect Liquefaction Program (DE-FC22-95PC93052) project participants, should be made in time to implement testing at LaPorte.

The recommendation to continue design verification testing to coproduce DME with methanol at the LaPorte AFDU is now under consideration. 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 productivity and life of an "acceptable" LPDME catalyst system must be better defined, and then confirmed in the laboratory. A recommendation document summarizing catalyst targets, experimental results, and the corresponding economics for a commercially successful LPDME catalyst was issued on 30 June 1997. DOE issued a letter dated 31 July 1997 accepting the recommendation to continue design verification testing.

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 the China and Pacific Rim regions. The results to date, are included in the DME recommendation in Appendix E.

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

- All of the LPDME runs at low space velocity show that the methanol equivalent productivity is much less sensitive to the catalyst activity (i.e., rate constant) at low space velocity than at high space velocity.
- A manufacturer for the dehydration catalyst has been selected by the Liquid Fuels Program. The initial schedule (contained in the DME Milestone Plan in Appendix E) showed a catalyst delivery date to the LaPorte AFDU of 01 March 1998. This date could be met assuming that the dehydration catalyst would be produced in a series of campaigns in a pilot plant. The Liquid Fuels Program has determined that it is important to complete the scale-up of the dehydration catalyst as part of the proposed LaPorte run. This will increase the time requirement, as a production test in the pilot plant is still required before operating the commercial catalyst production unit. The new estimated delivery date of dehydration catalyst to LaPorte is 01 June 1998. The DME DVT Recommendation will be updated during the next quarter 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.
- The entire procedure for preparing a batch of AB-type catalyst in the laboratory was videotaped with two purposes: to provide a clear example of the procedure for the commercial catalyst manufacturer, and to provide an internal record.
- LPDME runs using different feed gases are being carried out to help understand the correlation between gas phase composition and catalyst deactivation. Different deactivation patterns were observed under Balanced Gas.
- The commercial dehydration catalyst manufacturer has prepared the first two batches of AB-type catalyst in small-scale equipment. The immediate goals are to become familiar with the material and to optimize the material handling process during production.

Task 1.5.4 Administration and Reporting

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 DOE cost share of \$92,700,000 of authorized funding, with the remaining \$121,000,000 being provided by the participants. A copy of the approval memorandum, dated 03 October 1996, is included in Appendix F.

The remainder of the DOE reporting tasks are being performed and reported under Task 3.6 (Planning and Administration).

Task 2.1 Procurement

The Project Evaluation Plan for Budget Period No. 2 establishes the following goal for this task:

- Complete the bidding and procurement for all equipment and Air Products-supplied construction materials.
 - Task 2.1 Procurement is complete.

Task 2.2 Construction

The Project Evaluation Plan for Budget Period No. 2 establishes the following goal for this task:

- Provide construction management for contractor coordination and compliance with design, construction, and quality control standards.
- Erect the major equipment and structural steel. Install the large bore piping, electrical, and insulation such that instrument check-out and equipment commissioning work can be completed during the 60-day Continuation Application approval period.
- Complete mechanical construction so that check-out and commissioning can be started in Budget Period No. 3.
 - Task 2.2 Construction is complete.

Task 2.3 Training and Commissioning

The Project Evaluation Plan for Budget Period No. 2 establishes the following goals for this task:

- Prepare a four-year test plan for Phase 3, Task 2 - Operation.
 - The four-year Demonstration Test Plan (DTP) was approved and issued in September of 1996.
- Prepare the operating manual and initiate the operator training program.
 - The operator training was completed in December of 1996. Final additions to the operating manual were made in January of 1997.

- Task 2.3 Training and Commissioning is complete.

Task 2.4 Off-Site Testing (Procurement and Construction)

The Project Evaluation Plan for Budget Period No. 2 establishes the following goal for this task:

- Prepare the final off-site product-use test plan.
 - The off-site product-use test plan update is being reported under the Task 1.4 Off-Site Testing (Definition and Design).

Task 2.5 Planning and Administration

The Project Evaluation Plan for Budget Period No. 2 establishes the following goals for this task:

- Prepare annually an updated (Partnership) plan for the remaining activities. The first annual plan will update the remaining Phase 1 and Phase 2 activities, and the second will include an update of the Phase 3 Demonstration Test Plan.
 - The first update of the Partnership Annual Operating Plan was prepared and submitted in September of 1995 (See Quarterly Technical Progress Report No. 5). The main goal and objective for this first annual plan was to continue construction so that the LPMEOHTM Demonstration Unit would be ready for commissioning and start-up in 1996; and to complete the Project Evaluation Report and to submit it to the DOE along with the Continuation Application for Budget Period No. 3.
 - The second update of the Partnership Annual Operating Plan was prepared and submitted in November of 1996 (see Appendix G). The main goal and objective for this second annual plan is to initiate Phase 3 - Operation of the LPMEOHTM Demonstration Unit and to achieve 30 weeks of operation (Task 2.1.1 Operation) by September of 1997 in accordance with the Demonstration Test Plan. Other objectives include continuation of DME design verification testing, and updating the plan for off-site product-use testing.
- Submit all Project status, milestone schedule, and cost management reports as required by the Cooperative Agreement.
 - The DOE reporting tasks are being performed and reported under Task 3.6 (Planning and Administration).

Task 3.1 Start-up

- Start-up activities were completed on 02 April 1997 with the initial production of methanol.
- Task 3.1 Start-up is complete.

Task 3.2 LPMEOH™ Process Demonstration Facility Operation

Task 3.2.1 Methanol Operation

The summary table of performance data over the entire reporting period for the LPMEOH™ Demonstration Unit is included in Table 3.2.1-1. These data represent daily averages, typically from a 24-hour material balance period; those days with less than 12 hours of stable operation are omitted from this table. Appendix H contains samples of the detailed material balance report which are representative of the operation of the LPMEOH™ Demonstration Unit during the reporting period.

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

Over the reporting period, a total of 4,360,255 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. All methanol was used by Eastman in the production of methyl acetate, and ultimately cellulose acetate and acetic acid. No safety or environmental incidents were reported during this quarter. At the end of the quarter, slurry concentration in the reactor reached the design 40 wt% level for the first time.

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

Catalyst Life (η)

The activity of the methanol synthesis catalyst can be expressed in terms of a dimensionless variable η (η), which is defined as 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). Appendix I, Figure 1 plots $\log \eta$ versus days onstream since the start-up in April of 1997. Since catalyst activity typically follows a pattern of exponential decay, the plot of $\log \eta$ is fit to a series of straight lines.

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, confirms that this decline was not induced by poor hydrodynamics related to the sparger performance. Since that restart, the activity decrease slowed but remains faster than predicted. With some

TABLE 3.2.1-1

DATA SUMMARY FOR LPMEOH™ DEMONSTRATION UNIT

se	Date	Gas Type	Temp (Deg C)	Pres. (psig)	Balanced			Recycle Gas (KSCFH)	Inlet Sup. Velocity (ft/sec)	Space Velocity (1/hr-kg)	Slurry Conc. (wt% ox)	Gas Holdup (vol%)	Gassed Slurry Hgt (ft)	Catalyst Inventory (lb)	Catalyst Age (eta)	CO Conv. (%)	Reactor O-T-M Conv. (%)	Syngas Util. (SCF/lb)	Raw MeOH Production (TPD)	Catalyst MeOH Prod. (gmol/hr-kg)	Reactor Vol. Prod. (TPD/Cu ft)	Sparger dP (psi)	Sparger Resistance ("K)
					Gas (KSCFH)	CO Gas (KSCFH)	H2 Gas (KSCFH)																
1-Jul-97	249	707	676	0	0	0	2,251	0.71	8944	26.1	45.3	59.4	19,500	0.52	31.5	19.0	42.0	193.2	24.68	0.077	9.47	8.15	
2-Jul-97	249	707	685	0	0	0	2,203	0.70	8827	26.5	45.4	58.4	19,500	0.54	32.5	19.6	41.9	195.9	25.39	0.080	10.66	9.21	
3-Jul-97	249	707	664	0	0	0	2,218	0.70	8794	27.5	43.9	54.0	19,500	0.54	32.8	19.4	41.1	193.9	25.48	0.085	12.10	10.78	
4-Jul-97	249	707	705	0	0	0	2,231	0.70	7939	30.0	44.1	54.0	21,800	0.56	37.3	21.0	40.6	208.4	23.96	0.092	12.21	10.89	
5-Jul-97	249	705	761	0	0	0	2,203	0.72	8060	30.7	43.1	51.5	21,800	0.56	35.8	21.1	42.3	216.0	25.11	0.100	11.09	9.73	
6-Jul-97	249	705	755	0	0	0	2,198	0.71	8031	30.2	42.5	52.0	21,800	0.55	36.2	20.9	42.6	212.8	24.85	0.097	11.49	10.12	
8-Jul-97	249	706	761	0	0	0	2,161	0.70	7926	31.1	41.9	49.5	21,800	0.53	35.4	20.5	44.2	206.5	24.25	0.099	11.94	10.85	
9-Jul-97	248	695	610	0	0	0	2,277	0.70	7788	31.3	42.0	49.0	21,800	0.50	34.3	19.0	40.2	182.1	21.28	0.089	11.02	9.60	
10-Jul-97	249	694	632	0	0	0	2,235	0.69	7705	29.9	42.3	52.5	21,800	0.47	33.9	18.5	41.9	181.1	21.09	0.082	12.05	11.35	
11-Jul-97	249	695	748	0	0	0	2,118	0.70	7013	32.0	42.4	53.0	24,100	0.53	39.8	21.0	43.4	206.6	21.54	0.093	11.34	10.83	
12-Jul-97	249	699	805	0	0	0	2,063	0.70	7036	31.5	41.8	53.5	24,100	0.52	40.0	21.0	46.0	209.8	22.17	0.093	11.30	11.24	
13-Jul-97	249	701	800	0	0	0	2,125	0.71	7177	32.0	41.4	52.0	24,100	0.54	37.7	21.8	43.4	221.2	23.42	0.101	12.00	10.65	
14-Jul-97	249	702	801	0	0	0	2,139	0.71	7209	32.6	41.3	50.5	24,100	0.53	36.8	21.5	43.8	219.6	23.24	0.104	11.89	10.57	
15-Jul-97	249	703	781	0	0	0	2,170	0.71	7228	32.3	42.1	52.0	24,100	0.52	36.1	21.4	42.8	218.9	23.14	0.100	12.27	10.63	
16-Jul-97	249	704	788	0	0	0	2,154	0.71	7217	32.3	41.6	51.5	24,100	0.51	35.9	21.4	43.2	219.1	23.07	0.101	12.53	11.13	
17-Jul-97	249	703	763	0	0	0	2,189	0.71	7231	32.5	41.6	51.0	24,100	0.50	35.0	21.1	42.5	215.6	22.71	0.101	12.80	11.01	
18-Jul-97	249	702	770	0	0	0	2,124	0.70	7102	33.2	40.3	48.5	24,100	0.49	36.1	20.5	44.6	207.3	21.92	0.102	11.99	11.30	
19-Jul-97	249	702	767	0	0	0	2,173	0.71	7213	33.4	40.2	48.0	24,100	0.48	34.1	20.5	43.8	210.2	22.25	0.104	12.05	10.52	
20-Jul-97	249	702	768	0	0	0	2,187	0.71	7240	33.2	40.9	49.0	24,100	0.48	32.7	20.6	43.5	212.0	22.50	0.103	12.26	10.33	
21-Jul-97	249	702	746	0	0	0	2,137	0.70	7067	31.6	42.1	53.5	24,100	0.48	35.4	20.8	42.8	209.1	22.15	0.093	11.61	10.81	
22-Jul-97	249	702	755	0	0	0	2,160	0.70	7083	31.1	40.6	53.5	24,100	0.46	34.0	20.5	43.9	206.6	21.67	0.092	13.32	12.33	
23-Jul-97	250	702	706	0	0	0	2,197	0.70	7058	31.1	42.5	55.0	24,100	0.46	32.8	20.4	41.6	203.5	21.30	0.088	14.71	13.01	
24-Jul-97	249	702	801	0	0	0	2,070	0.70	6426	33.4	40.8	53.0	26,400	0.51	39.0	22.3	43.3	222.0	21.42	0.100	13.17	12.43	
25-Jul-97	249	700	796	0	0	0	2,090	0.70	6460	33.7	39.8	51.5	26,400	0.51	37.3	22.4	42.6	224.3	21.64	0.104	13.80	12.41	
26-Jul-97	249	700	781	0	0	0	2,062	0.69	6361	33.6	39.6	51.5	26,400	0.52	38.1	22.7	41.9	223.9	21.62	0.103	13.32	12.43	
27-Jul-97	249	700	748	0	0	0	2,082	0.69	6334	33.6	39.4	51.5	26,400	0.50	37.9	22.1	41.6	215.7	20.58	0.100	13.02	12.31	
28-Jul-97	249	700	790	0	0	0	2,048	0.69	6354	33.1	39.5	52.5	26,400	0.52	38.3	22.8	42.1	225.3	21.71	0.102	13.45	12.58	
4-Aug-97	249	702	627	95	0	0	2,233	0.71	6600	33.5	47.5	59.5	26,400	0.56	12.1	18.1	45.0	192.4	18.11	0.077	17.86	9.01	
5-Aug-97	249	702	626	95	0	0	2,238	0.71	6600	33.7	46.2	57.7	26,400	0.57	12.3	18.2	45.1	192.0	18.25	0.079	18.10	9.08	
6-Aug-97	249	701	636	95	0	0	2,182	0.70	6507	33.8	45.4	56.5	26,400	0.56	12.8	18.5	45.5	193.0	18.56	0.081	17.07	8.95	
7-Aug-97	249	701	648	95	0	0	2,148	0.70	6459	34.4	45.4	55.0	26,400	0.54	13.3	18.8	45.8	194.8	18.75	0.084	16.52	9.00	
8-Aug-97	249	702	629	95	0	0	2,129	0.69	6376	33.4	45.1	57.0	26,400	0.55	13.0	18.6	45.7	190.2	18.36	0.079	17.05	9.33	
9-Aug-97	249	703	627	95	0	0	2,130	0.69	6383	33.4	45.4	57.5	26,400	0.53	12.8	18.4	46.1	187.9	18.08	0.078	17.30	9.46	
10-Aug-97	249	702	624	95	0	0	2,118	0.69	6364	33.8	45.9	57.0	26,400	0.52	13.2	18.6	46.0	187.7	17.98	0.078	17.25	9.47	
11-Aug-97	249	702	622	95	0	0	2,101	0.68	6320	34.9	45.9	54.5	26,400	0.50	13.0	18.3	46.3	186.0	17.89	0.081	16.94	9.52	
12-Aug-97	249	702	616	95	0	0	2,088	0.68	6284	34.0	42.4	53.0	26,400	0.50	12.9	18.2	46.0	185.6	17.95	0.083	16.71	9.56	
15-Aug-97	249	700	642	0	0	0	2,303	0.72	6602	34.0	39.4	50.5	26,400	0.37	31.9	18.1	41.5	185.5	17.82	0.087	10.37	9.38	
16-Aug-97	249	700	701	0	0	0	2,203	0.70	6491	34.1	39.0	50.0	26,400	0.37	31.4	18.6	44.1	190.8	18.46	0.091	11.05	10.64	
17-Aug-97	249	700	693	0	0	0	2,227	0.71	6537	34.2	40.4	51.0	26,400	0.37	29.7	18.9	42.6	195.5	18.78	0.091	12.19	11.20	
18-Aug-97	249	700	633	0	0	0	2,278	0.71	6508	34.7	41.1	50.5	26,400	0.36	29.4	18.5	40.4	188.0	18.03	0.089	12.29	11.09	

TABLE 3.2.1-1

DATA SUMMARY FOR LPMEOH™ DEMONSTRATION UNIT

Case	Date	Gas Type	Temp (Deg C)	Pres. (psig)	Balanced			Recycle Gas Velocity (ft/sec)	Space Velocity (1/hr-kg)	Slurry Conc. (wt% ox)	Gas Holdup (vol%)	Gassed Slurry Hgt (ft)	Catalyst Inventory (lb)	Catalyst Age (eta)	CO Conv. (%)	Reactor O-T-M Conv. (%)	Syngas Util. (SCF/lb)	Raw MeOH Production (TPD)	Catalyst MeOH Prod. (g/mol/hr-kg)	Reactor Vol. Prod. (TPD/Cu ft)	Sparger dP (psi)	Sparger Resistance (K)	
					Gas (KSCFH)	CO Gas (KSCFH)	H2 Gas (KSCFH)																
6	19-Aug-97	Balanced	249	698	632	0	0	2,244	0.70	6454	34.8	40.3	49.5	26,400	0.34	28.7	17.9	41.8	181.6	17.56	0.087	12.04	11.26
6	20-Aug-97	Balanced	249	700	636	0	0	2,296	0.71	6554	35.3	43.2	51.0	26,400	0.34	27.7	18.2	41.0	186.3	17.89	0.087	12.40	10.87
6	21-Aug-97	Balanced	249	701	636	0	0	2,224	0.69	6383	36.4	41.7	47.5	26,400	0.34	29.5	17.7	42.8	178.3	17.20	0.089	11.34	11.33
6	22-Aug-97	Balanced	249	701	666	0	0	2,283	0.71	6567	35.7	45.8	52.5	26,400	0.32	26.7	17.8	43.6	183.1	17.36	0.083	13.77	12.23
6	23-Aug-97	Balanced	249	702	652	0	0	2,205	0.69	6368	35.5	41.2	49.0	26,400	0.31	28.1	17.2	45.1	173.8	16.59	0.084	13.49	13.78
6	24-Aug-97	Balanced	249	702	653	0	0	2,204	0.69	6381	35.8	40.1	47.5	26,400	0.30	27.4	16.8	45.7	171.3	16.36	0.086	11.79	12.11
6	25-Aug-97	Balanced	249	702	629	0	0	2,176	0.68	6275	35.1	39.0	48.0	26,400	0.29	27.6	16.8	45.0	167.7	16.03	0.083	12.21	13.01
6	26-Aug-97	Balanced	249	702	625	0	0	2,187	0.68	6291	35.7	39.3	47.0	26,400	0.29	26.4	16.7	44.9	167.1	16.13	0.085	14.31	14.30
6	28-Aug-97	Balanced	249	708	709	0	0	2,115	0.68	6331	35.5	39.3	47.5	26,400	0.29	25.3	17.0	49.5	172.1	16.61	0.086	15.22	15.04
6	29-Aug-97	Balanced	249	711	742	0	0	2,112	0.68	6403	36.4	40.3	46.5	26,400	0.31	25.7	16.8	51.1	174.2	16.88	0.089	15.16	15.72
6	30-Aug-97	Balanced	249	701	644	0	0	2,184	0.68	6311	36.5	39.3	45.5	26,400	0.30	24.6	17.0	45.3	170.8	16.52	0.090	16.66	15.99
6	1-Sep-97	Balanced	249	705	644	0	0	2,264	0.70	6499	35.1	38.5	47.5	26,400	0.28	23.7	15.8	46.9	164.7	15.84	0.083	10.65	10.00
6	2-Sep-97	Balanced	249	705	668	0	0	2,205	0.69	6418	35.1	38.9	46.0	26,400	0.28	23.4	16.1	48.1	166.9	16.14	0.087	13.24	12.64
6	3-Sep-97	Balanced	249	705	665	0	0	2,211	0.69	6419	39.3	46.4	46.0	26,400	0.28	23.3	16.0	48.2	165.6	15.93	0.086	13.01	12.81
6	4-Sep-97	Balanced	249	706	669	0	0	2,246	0.70	6516	37.9	42.8	45.5	26,400	0.27	22.4	15.7	49.0	163.6	15.62	0.086	12.77	12.02
6	15-Sep-97	Balanced	260	675	473	0	0	2,020	0.63	5147	36.4	38.6	49.0	28,700	0.24	28.3	15.7	41.6	136.4	12.07	0.066	14.82	19.56
6	16-Sep-97	Balanced	251	675	473	0	0	2,002	0.62	5093	35.4	39.0	51.5	28,700	0.23	27.1	15.7	42.2	134.5	11.99	0.062	16.90	21.67
6	17-Sep-97	Balanced	251	676	473	0	0	2,001	0.62	5075	35.1	39.5	52.5	28,700	0.23	25.2	16.0	41.4	137.1	12.26	0.062	20.28	24.41
6	18-Sep-97	Balanced	251	675	536	0	0	2,025	0.64	4872	37.5	38.5	50.5	31,000	0.29	35.6	17.6	41.3	155.8	12.91	0.073	15.76	20.30
6	19-Sep-97	Balanced	251	675	577	0	0	2,119	0.67	5047	37.3	38.6	51.0	31,000	0.30	33.3	18.0	41.7	165.9	13.78	0.077	16.11	18.56
6	20-Sep-97	Balanced	251	675	578	0	0	2,058	0.67	5054	38.1	40.5	51.0	31,000	0.28	31.6	17.7	42.4	163.5	13.51	0.076	16.80	19.28
6	21-Sep-97	Balanced	251	675	577	0	0	2,112	0.68	5115	38.8	39.2	48.5	31,000	0.28	29.4	17.7	42.0	164.7	13.64	0.081	18.24	19.61
6	22-Sep-97	Balanced	251	675	572	0	0	2,012	0.65	4922	39.1	38.3	52.0	31,000	0.26	30.3	17.5	43.5	157.8	12.94	0.072	16.17	19.52
6	23-Sep-97	Balanced	249	675	577	0	0	2,026	0.66	4971	39.1	41.1	49.5	31,000	0.26	29.5	17.7	43.1	160.8	13.15	0.077	19.31	22.79
6	24-Sep-97	Balanced	249	676	578	0	0	2,082	0.67	5076	39.6	39.2	47.0	31,000	0.25	27.7	17.2	43.6	159.1	12.96	0.081	17.14	18.95
6	25-Sep-97	Balanced	249	675	578	0	0	2,057	0.66	5031	38.8	36.6	46.5	31,000	0.25	28.1	17.2	43.9	157.8	13.00	0.081	15.76	17.72
6	26-Sep-97	Balanced	249	680	579	0	0	2,081	0.66	5065	37.6	37.5	49.5	31,000	0.26	28.0	17.2	43.6	159.4	13.35	0.077	16.91	18.54
6	27-Sep-97	Balanced	249	690	573	0	0	2,118	0.66	5120	37.2	36.9	50.0	31,000	0.25	27.5	17.3	42.8	160.7	13.51	0.077	17.50	18.41
6	28-Sep-97	Balanced	249	681	555	0	0	2,115	0.66	5085	40.1	40.8	47.5	31,000	0.25	26.8	17.0	42.3	157.5	13.25	0.079	17.24	18.21

slight variations, the deactivation rate has remained relatively constant throughout the reporting period. During July, the first three additional catalyst batches were reduced and added on-line to maintain the average catalyst activity within the reactor at just over 50% of fresh. The increase in reactor performance for each addition step roughly matched model predictions, and each step-change is noted in Appendix I, Figure 1.

A catalyst sample taken just before the restart in June, after 30 days on-stream, showed levels of arsenic and iron significantly above expectations (446 and 281 ppmw respectively). Averaged over the time on-stream, this arsenic loading equates to 87 ppbv arsine in the feed, assuming complete capture by the catalyst. Furthermore, the copper crystallite size had grown to 274 Angstroms, with a corresponding loss in catalyst surface area that correlates with the activity decline observed at the plant. In comparison, the 28-day autoclave test performed at Kingsport in May/June 1996 accumulated 184 ppmw arsenic and 172 ppmw iron, which correspond to an average gas concentration of 27 ppbv arsine and less than 10 ppbv iron carbonyl. The copper crystallite size at the conclusion of this autoclave test was 179 Angstroms. Under those conditions, the catalyst deactivation rate was no different from typical autoclave experiments with clean feed gas. A draft Topical Report has been issued on that study (Design and Construction of the Alternative Fuels Field Test Unit and Liquid Phase Methanol Feedstock and Catalyst Life Testing at Eastman Chemical Company (Kingsport, TN)).

Additional catalyst samples taken from the LPMEOH™ Reactor in August and early September continued to show increasing levels of arsenic, reaching a concentration of 779 ppmw. In addition, sulfur was detected for the first time on the August sample, and the loading increased markedly on the September sample, potentially indicating recent breakthrough of an upstream guard bed. Iron levels on the catalyst showed little or no increase with time since the original sample. No chlorides were detected at any time. Appendix I, Table 2 summarizes these results.

Sampling of the Balanced Gas entering the plant confirmed the presence of arsine at levels similar to those measured in 1994 (>20 ppbv), prior to installation of Eastman's arsine-removal guard bed. Furthermore, at this arsine loading, the 29C-40 carbonyl guard bed within the plant boundary showed little ability to remove arsine, as expected. Based on these catalyst and gas sampling results, Eastman made plans to change out the arsine- and sulfur-removal material in the guard bed. Because of material delivery times, that work is scheduled for 01 October 1997.

During the aforementioned gas sampling, iron carbonyl concentrations were measured at: 13 ppbv in the CO Gas and reactor feed stream; 11-12 ppbv in the plant purge stream; and below the detectable limit of 10 ppbv in the Balanced Gas, carbonyl guard bed inlet, and carbonyl guard bed outlet. These carbonyl levels are comparable with those measured during the carbonyl burnout period in March of 1997 and within acceptable limits. Such results, coupled with the lack of increase in iron loading on successive catalyst samples, indicate that long-term carbonyl poisoning by iron has not been an issue. The initially high iron levels on the catalyst most likely resulted from a one-time startup source, such as construction debris not cleaned out by the carbonyl burnout step.

Core samples taken from the carbonyl guard bed in early September showed an iron front about two feet into the bed and no evidence of any nickel loading. In addition, the core samples indicated a significant arsenic gradient over the first four feet of the bed. This may be further evidence of intermittent periods of very high arsine loading, despite the apparent baseline levels of 20-30 ppbv in the Balanced Gas.

While awaiting the replacement of Eastman's guard bed material on 01 October 1997, no fresh material was added to the reactor during August. Under these conditions, the catalyst deactivation rate remained relatively constant at about 1.6% per day, as shown in Appendix I, Figure 1. This rate is still significantly higher than the design rate of 0.4% per day, which was based on LaPorte data with clean CO-rich gas. As a result, the average η in the reactor dropped well below its design level of 0.5. After a gasifier-related outage in early September, the plant could not be restarted because catalyst activity had declined to a point where the reaction would not initiate at the startup steam temperature. Consequently, one additional batch of fresh catalyst was activated and transferred to the reactor to facilitate the restart. In addition, Eastman decreased plant operating pressure by an arbitrary 25 psi to improve the response of the Balanced Gas control valve. A second catalyst batch was added shortly thereafter to further increase the value of η .

Parallel to the extensive field sampling and analytical effort in search of catalyst poisons, several tests were accomplished in the laboratory autoclave. First, a run using a portion of the original June catalyst sample confirmed the field-calculated decline in activity. The sample also exhibited the baseline deactivation observed in the autoclave.

An autoclave test using fresh catalyst under the actual reactor feed gas composition from the LPMEOH™ Demonstration Unit confirmed the reactor effluent water concentration as back-calculated from product analyses at the plant; no prior database existed at conditions so hydrogen-rich. Since there was no evidence of accelerated catalyst deactivation, this test seemed to eliminate hydrothermal sintering as a potential cause of the rapid deactivation at the demonstration unit.

Next, half of the fresh catalyst was removed from the autoclave and replaced with poisoned catalyst from the original Kingsport sample. After several days of typical autoclave stability, this mixture did begin to exhibit accelerated deactivation. One of several possible explanations for this result is that some species migrates to the fresh catalyst, poisoning it after reaching some threshold concentration. Further tests are being designed to explore this effect in more detail.

Sparger Resistance

As reported in Technical Progress Report No. 12, flow resistance through the gas sparger of the LPMEOH™ Reactor had increased with time since the 17 June 1997 restart. A flush connection to the gas inlet line to the reactor had been added during the May/June outage. A flush by fresh oil had shown a decline in pressure drop through the gas sparger. However, the effects were temporary, and the flow resistance continued to increase. Additional

flushing with fresh oil is limited, because fresh oil can only be added to the process at an average of 0.1 - 0.2 gallons per minute to match the rate of oil loss with the methanol product.

A practice of flushing the gas sparger with entrained slurry from the 29C-06 cyclone and 29C-05 secondary oil knock-out drum was initiated; this can be supplied at the average rate of liquid traffic in the secondary oil knock-out drum and cyclone (1.5 to 2.0 gallons per minute). Flow resistance through the gas sparger has been stabilized at a manageable level by flushing in this manner. Since the 29G-01 condensed oil circulation pumps were not in service, this flushing step was accomplished by shutting off the free-drain line back to the reactor and batch transferring condensed oil and entrained slurry to the 29C-30 reduction vessel. From there, the entrained slurry is returned to the reactor via the 29G-30 slurry transfer pump through the flush connection at the gas inlet line to the reactor. The rate of addition of the flush was 30 gallons per minute, and can be performed 2 to 3 times per day at the average rate of liquid traffic through the cyclone and secondary oil knock-out drum.

Appendix I, Figure 2 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 pressure drop measurement, are included in Table 3.2.1-1. The resistance coefficient plateaus with the beginning of periodic flushing with internal oil traffic at approximately Day 20 and remains relatively stable for approximately two months. This period included several interruptions in the flushing regimen, lasting 1-2 days, because of shutdowns or periods of on-line catalyst activation which occupied the reduction vessel. During the week-long shutdown in early September, however, the sparger resistance experienced a significant step-change increase. While some of this added resistance proved to be gradually reversible, some of it apparently was not. Once the new 29G-03 oil makeup pump is operational, the effect of a 1-2 gpm continuous flush will be tested.

Other Issues

A 12-day test on simulated Texaco-type synthesis gas ($H_2/CO = 0.8$) concluded on 12 August 1997. The Test Authorization for this trial is included in Appendix J. Data from the test is seen as a step-change in the plot of $\log \eta$ in Appendix I, Figure 1, but the rate of deactivation remained unchanged. The offset results from the sensitivity of the rate equation model to changes in feed gas composition. Methanol production averaged 57,100 gallons per day (190 TPD) during the period, and the crude product composition matched expectations for bulk components. The results of an analysis of two samples of crude methanol from this operating period are provided in Appendix I, Table 3. Note that the Kingsport samples were stabilized, resulting in the removal of light components such as methyl acetate and methyl formate.

As noted in Technical Progress Report No. 11, the oil makeup pumps were unable to deliver fresh oil to the reactor loop at the required pressure of approximately 700 psig. These pumps also provide the required high pressure seal flush to the condensed oil circulation

pumps, which return oil and catalyst collected in the cyclone and the secondary oil knock-out drum to the reactor (refer to Appendix A for the simplified process flow diagram). A replacement oil makeup pump was ordered and installed, but testing has slipped to early October of 1997.

Task 3.2.2 DME Design, Modification and Operation

No activities occurred in this Task during the reporting period.

Task 3.3 On-Site Testing (Product-Use Demonstration)

No activities occurred in this Task during the reporting period.

Task 3.4 Off-Site Testing (Product-Use Demonstration)

No activities occurred in this Task during the reporting period.

Task 3.5 Data Analysis and Reports

The results of the data analysis for the operation of the LPMEOH™ Demonstration Unit are reported under Task 3.2.1 (Methanol Operation).

Task 3.6 Planning and Administration

The dedication ceremony for the LPMEOH™ Demonstration Unit was held on 25 July 1997. Senior management from DOE, Eastman, and Air Products participated. Press coverage by the local television stations and newspapers was arranged by Eastman.

Two project review meetings were held during the reporting period. The first, on 24 and 25 July 1997, was scheduled in conjunction with the dedication ceremony for the demonstration unit. The second meetings were held at Kingsport on 24 and 25 September 1997. Both meetings focused on reviewing the performance of the demonstration unit, the catalyst development work for the upcoming LPDME proof-of-concept test at the LaPorte AFDU, and the status of the off-site product-use test plan. Comments on the equipment breakdown used in the latest draft of the Final Technical Report, Volume 1, Public Design Report were received. The meeting agenda, extracts from the meeting handouts, and the meeting notes are included in Appendix K and Appendix L.

The Milestone Schedule Status Report and the Cost Management Report, through the period ending 30 September 1997, are included in Appendix M. These two reports show the current schedule, the percentage completion and the latest cost forecast for each of the Work Breakdown Structure (WBS) tasks. Ninety-nine percent (99%) of the \$38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 30 September 1997. Ten

percent (10%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 September 1997.

The monthly reports for July, August, and September 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

- Resolve any issues associated with the gas sparger in the reactor and with the oil make-up pumps. Upon resolution of these items, 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.
- 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.
- Begin execution of the Off-Site, Product-Use Test Plan (Phase 1, Task 1.4).
- Complete work on the on-site product-use tests (Phase 1, Task 1.5.1).
- Continue to incorporate DOE comments into the Topical Report on Process Economic Studies.
- Reach agreement with DOE on the equipment breakdown for use in the Final Technical Report, Volume 1, Public Design Report.
- Submit the third update of the Partnership Annual Operating Plan to DOE.

F. Conclusion

During the reporting period, DOE accepted the recommendation to continue with DME design verification testing. DME design verification testing studies show the liquid phase DME (LPDME) process will have a significant economic advantage for the coproduction of DME for local markets. The market applications for DME are large. An LPDME catalyst system with reasonable long-term activity and stability is being developed. Planning for a proof-of-concept test run at the LaPorte Alternative Fuels Development Unit (AFDU) was recommended. DOE issued a letter dated 31 July 1997 accepting the recommendation to continue design verification testing.

The initial schedule for production of the dehydration catalyst for the test run called for use of pilot plant equipment used in multiple campaigns. In order to allow for scale-up of the catalyst manufacturing technique to the commercial scale, the time required to produce the catalyst to the AFDU has slipped. The new estimated delivery date 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.

During this quarter, eight project sites were recommended to DOE as candidates for participation in the off-site, product-use test plan. Seven of the eight proposals have been defined in sufficient detail so that final planning and implementation should begin. DOE accepted Air Products' recommendation to proceed with the seven projects. Planning work on these tests has begun, and Air Products is preparing a recommended list of project sites where methanol produced from carbon monoxide (CO)-rich syngas at the LaPorte AFDU can be used. This has become necessary since several of the off-site, product-use tests are scheduled to begin prior to the specific production campaign of methanol from CO-rich syngas at the demonstration unit.

The dedication ceremony for the LPMEOH™ Demonstration Unit was held on 25 July 1997. Senior management from DOE, Eastman, and Air Products participated.

Two project review meetings were held during the reporting period. Both meetings focused on reviewing the performance of the demonstration unit, the catalyst development work for the upcoming LPDME proof-of-concept test at the LaPorte AFDU, and the status of the off-site product-use test plan.

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, but at a slower rate than during the April/May operation. Catalyst slurry samples taken from the LPMEOH™ Reactor have shown an increase in the levels of iron, arsenic, and sulfur as compared with fresh catalyst. The increase in iron appears to be the result of a one-time startup source, such as construction debris. Eastman has made plans to change out an upstream guard bed designed for the removal of arsine and sulfur. Because of material delivery times, this work is scheduled for 01 October 1997. Laboratory work has confirmed that the process conditions during the April startup at the demonstration unit were not the cause of the decline in catalyst activity during that period. Tests on the effects of possible poisons of methanol synthesis catalyst are ongoing.

Pressure drop and resistance coefficient across the gas sparger at the bottom of the reactor have been stabilized at a manageable level by flushing with entrained slurry collected at the cyclone and secondary oil knock-out drum. This flush is applied through a connection at the gas inlet line to the reactor. The rate of addition of the entrained slurry was 30 gallons per minute, and can be performed 2 to 3 times per day at the average rate of liquid traffic through the cyclone and secondary oil knock-out drum.

A 12-day test on simulated Texaco-type synthesis gas ($H_2/CO = 0.8$) concluded on 12 August 1997. Methanol production averaged 57,100 gallons per day (190 TPD) during the period, and the crude product composition matched expectations for bulk components.

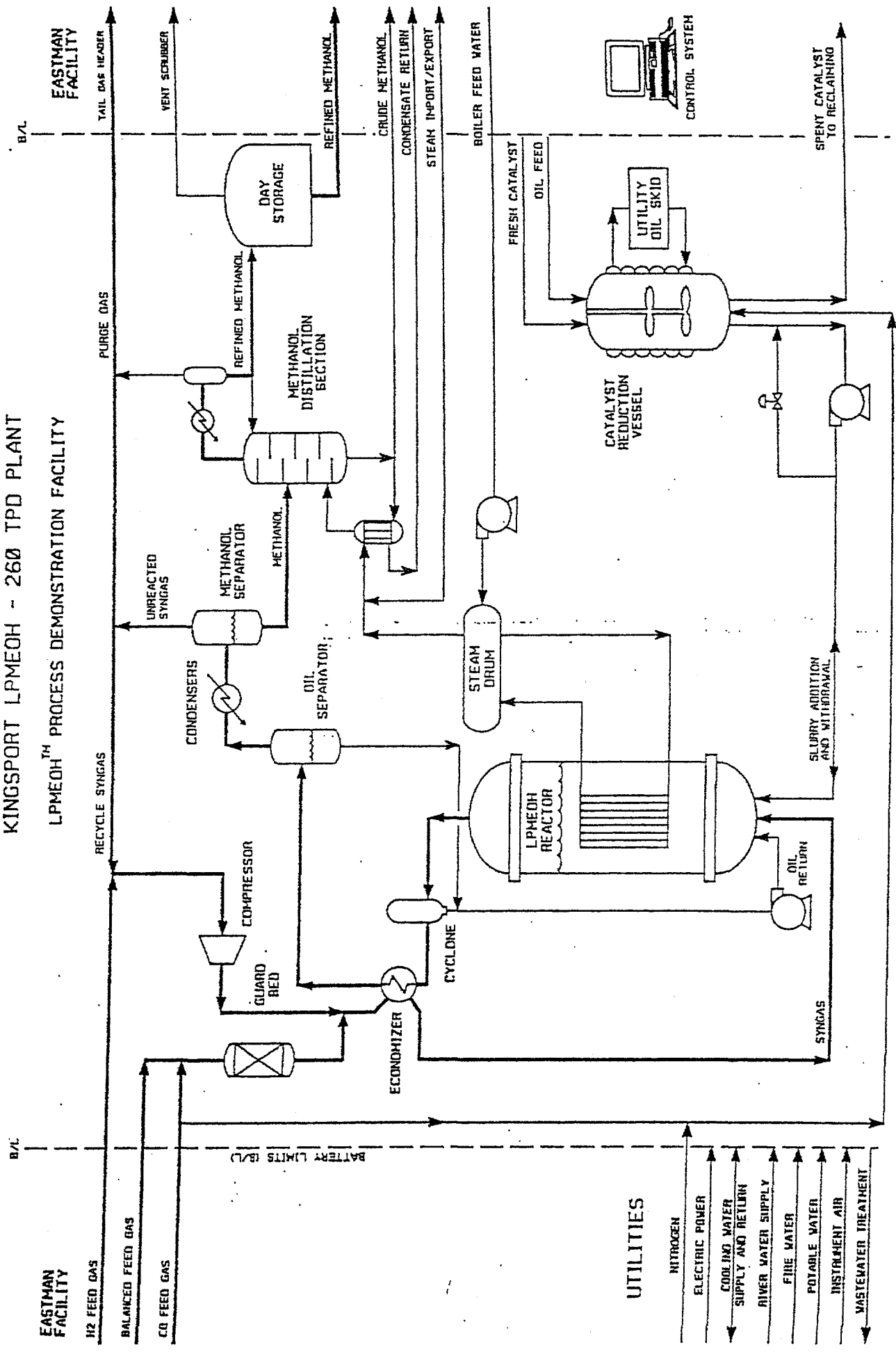
Over the reporting period, a total of 4,360,255 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Since startup, a total of 7,260,947 gallons of methanol has been produced. All methanol was used by Eastman in the production of methyl acetate, and ultimately cellulose acetate and acetic acid. No safety or environmental incidents were reported during this quarter. At the end of the quarter, slurry concentration in the reactor reached the design 40 wt% level for the first time.

Ninety-nine percent (99%) of the \$38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 30 September 1997. Ten percent (10%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 September 1997.

APPENDICES

APPENDIX A - SIMPLIFIED PROCESS FLOW DIAGRAM

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



APPENDIX B - PROJECT EVALUATION PLAN FOR BUDGET PERIOD NO. 2

COMMERCIAL-SCALE DEMONSTRATION
OF THE
LIQUID PHASE METHANOL (LPMEOH™) PROCESS
COOPERATIVE AGREEMENT
NO. DE-FC22-92PC90543

PROJECT EVALUATION PLAN FOR BUDGET PERIOD NO. 2

The work to be performed during Budget Period No. 2 consists of Phase 1 Design and Phase 2 Construction of the LPMEOH™ Process Demonstration Facility at Eastman Chemical Company's integrated coal gasification facility located in Kingsport, TN. Completion of these Budget Period No. 2 activities will essentially ready the LPMEOH™ Process Demonstration Facility for commissioning, startup, and operation to begin in the final Budget Period No. 3. The Statement of Work for the Project subdivides these Phase 1 and Phase 2 activities into Tasks. This Project Evaluation Plan for Budget Period No. 2 will meet the following criteria aligned by the Statement of Work tasks:

1. Phase 1 - Task 2 - Permitting

- Issue the final Environmental Information Volume to support the U.S. Department of Energy's (DOE's) Environmental Assessment/Finding of No Significant Impact.
- Obtain permits necessary for construction and operation.

2. Phase 1 - Task 3 - Design Engineering

- Complete the design engineering necessary for construction and commissioning. This includes Piping and Instrumentation Diagrams, Design Hazard Reviews, and conducting design reviews.
- Prepare the Environmental Monitoring Plan.

3. Phase 1 - Task 4 - Off-site Testing (Definition and Design)

- Prepare the fuel-use demonstration plan for Phase III, Task 4 Off-site Product Use Demonstration. This off-site test plan will be incorporated into the overall product-use test plan (in Phase 1, Task 5).

4. Phase 1 - Task 5 - Planning, Administration and DME Verification Testing

- Update the (fuel and chemical) product-use test plan, that will better meet the technical objectives of the Project and serve the needs of commercial markets.
- Complete economic studies of the important commercial aspects of the LPMEOH™ Process to enhance Integrated Gasification Combined Cycle (IGCC) electric power generation. These studies will be performed by Air Products and Chemicals, Inc. and the Electric Power Research Institute, and used to provide input to the LPMEOH™ Process Demonstration Facility operating test plan (Phase 2, Task 5).
- Perform initial Design Verification Testing for the production of dimethyl ether (DME) as a mixed coproduct with methanol. This activity includes laboratory R&D and market economic studies.
- Submit all Project status, milestone schedule, and cost management reports as required by the Cooperative Agreement.

5. Phase 2 - Task 1 - Procurement

- Complete the bidding and procurement for all equipment and Air Products supplied construction materials.

6. Phase 2 - Task 2 - Construction

- Complete mechanical construction so that checkout and commissioning can be started in Budget Period No. 3.
- Erect the major equipment and structural steel. Install the large bore piping, electrical, and insulation such that instrument checkout and equipment commissioning work can be completed during the 60-day Continuation Application approval period.
- Provide construction management for contractor coordination and compliance with design, construction, and quality control standards.

7. Phase 2 - Task 3 - Training and Commissioning

- Prepare a four (4)-year test plan for Phase 3, Task 2-Operation.
- Prepare the operating manual and initiate the operator training program.

8. Phase 2 - Task 4 - Off-Site Testing (Procurement and Construction)

- Prepare the final off-site product-use test plan.

9. Phase 2 - Task 5 - Planning and Administration

- Prepare annually an updated plan for the remaining activities. The first annual plan will update the remaining Phase I and Phase II tasks. The second annual plan will include an updated Phase III Operating Plan, identifying specific goals and milestones for the first twelve months of operation, and a general plan for the remaining years to achieve the Project's market penetration objectives.
- Submit all Project status, milestone schedule, and cost management reports as required by the Cooperative Agreement.

Completion of the above work activities will essentially ready the LPMEOH™ Process Demonstration Facility for commissioning, startup, and operation to begin in the final Budget Period No. 3. These criteria will be the basis of the Project Evaluation Report which shall be submitted to the DOE for approval along with the Project Continuation Application, at least 60 days before the end of Budget Period No. 2. Construction of the Facility will be essentially completed during the 60-day approval period for the Continuation Application.

At the time that the Project Evaluation Report for Budget Period No. 2 is submitted with the Continuation Application; Air Products will also prepare an update on the expected technical and economic performance of the mature unit. This update will demonstrate the commercial potential of the LPMEOH™ process technology to enhance IGCC electric power generation with coproduct methanol. This IGCC enhancement is expected to reduce the cost of electricity for retrofit, repowering, replacement, and new applications for electric power generation from coal.

WRB/jjs/Proeva

APPENDIX C - TASK 1.4 - OFF-SITE TESTING (DEFINITION AND DESIGN)

Summary Table of Eight Candidates (one page)

**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 D - TASK 1.5.2 - 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

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

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

Process Economics Study - Outline

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

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. 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)
R. J. Allam - APE (Hersham)

APCI

E. C. Heydorn
J. Klosek/E. R. Osterstock
R. B. Moore/D. P. Drown
V. E. Stein
P. J. A. Tijm

R. M. Kornosky - DOE-FETC
W. J. O'Dowd - DOE-FETC
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 than 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 E - TASK 1.5.3 - DME DESIGN VERIFICATION TESTING

Air Products and Chemicals, Inc.
7201 Hamilton Boulevard
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.

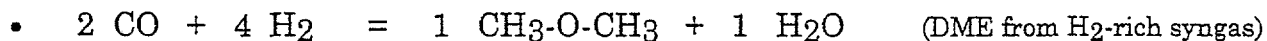
A-2. The economics studies, for once-through coproduction (with an integrated gasification combined cycle (IGCC) power plant, for example) on synthesis gas rich in carbon oxides, show that the LPDME process will have an economic advantage greater than the LPMEOHTM process. A once-through LPDME reactor 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).

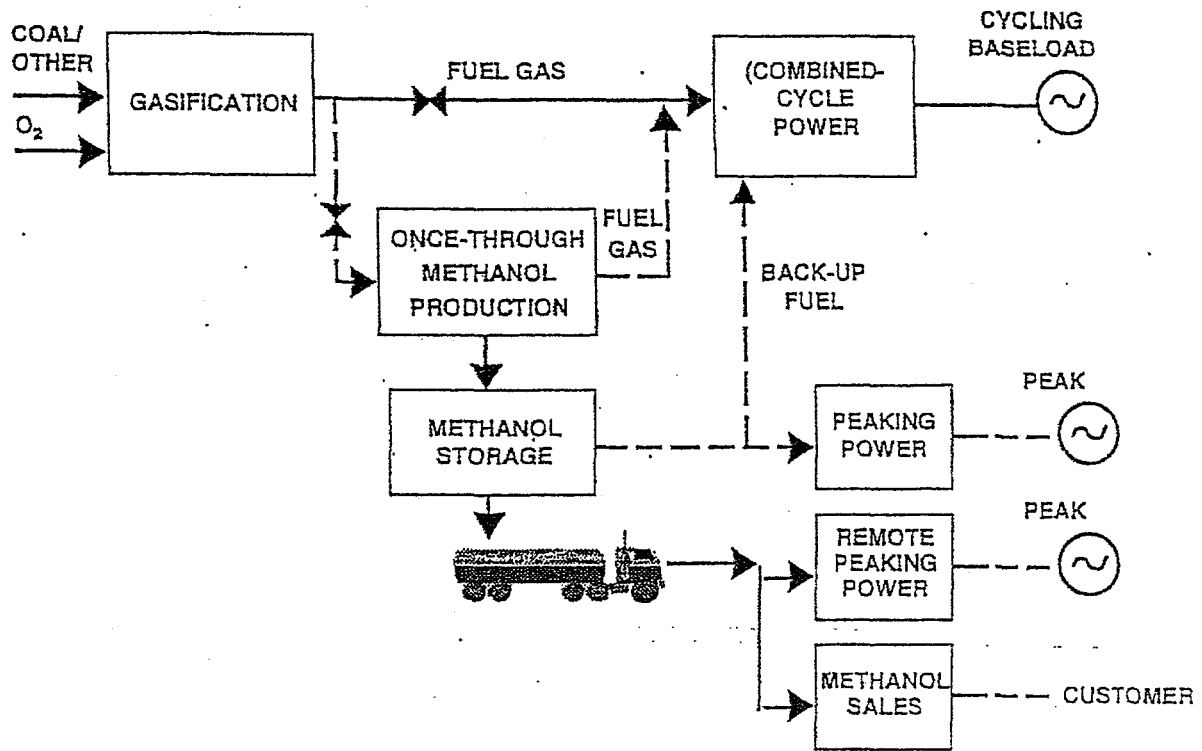
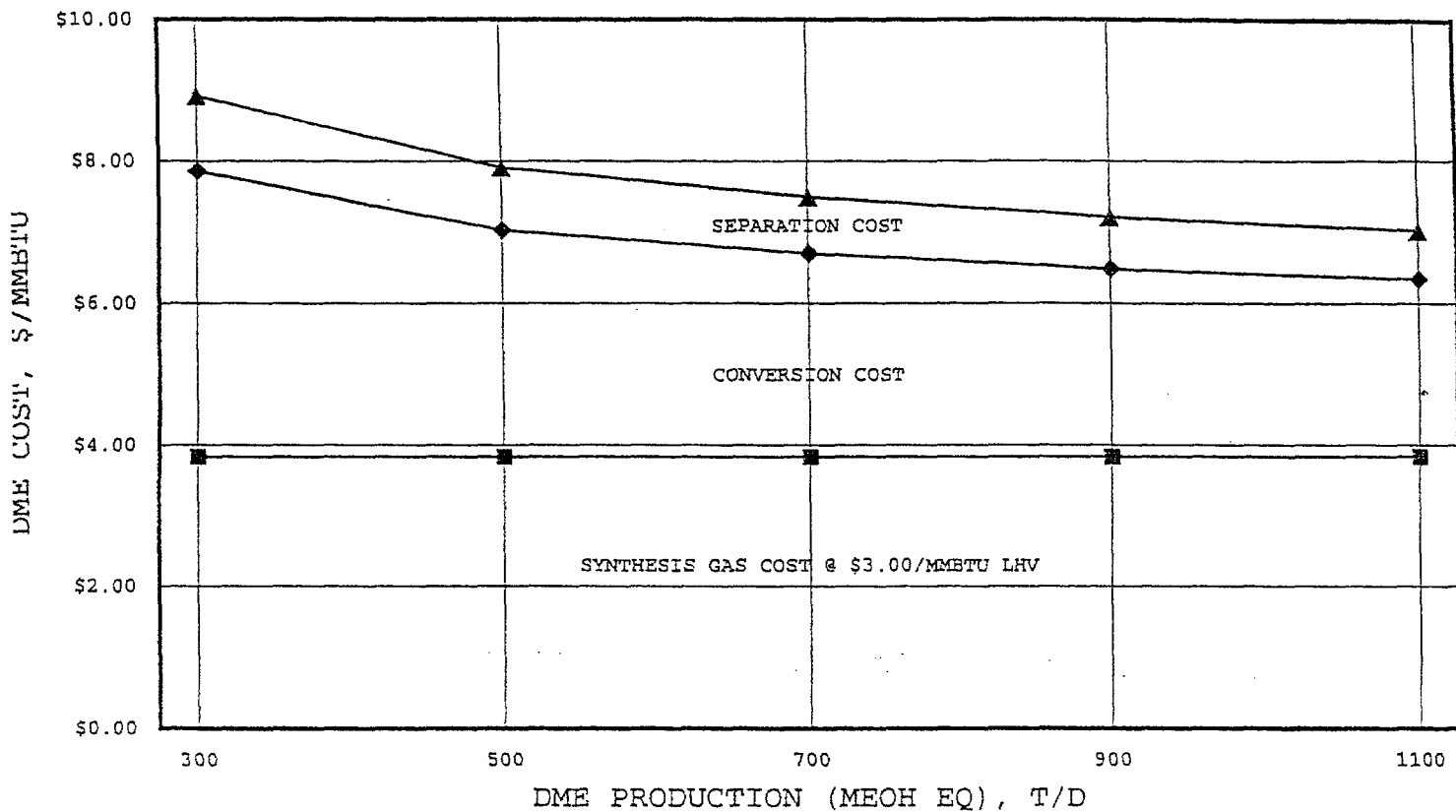


Figure 1. Once-through Methanol Coproduction with IGCC Electric Power

Figure 2

DME COST VERSUS SIZE

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



- SYNTHESIS GAS ONLY
- ◆ SYNTHESIS GAS PLUS CONVERSION
- ▲ TOTAL DME COST

GOALS

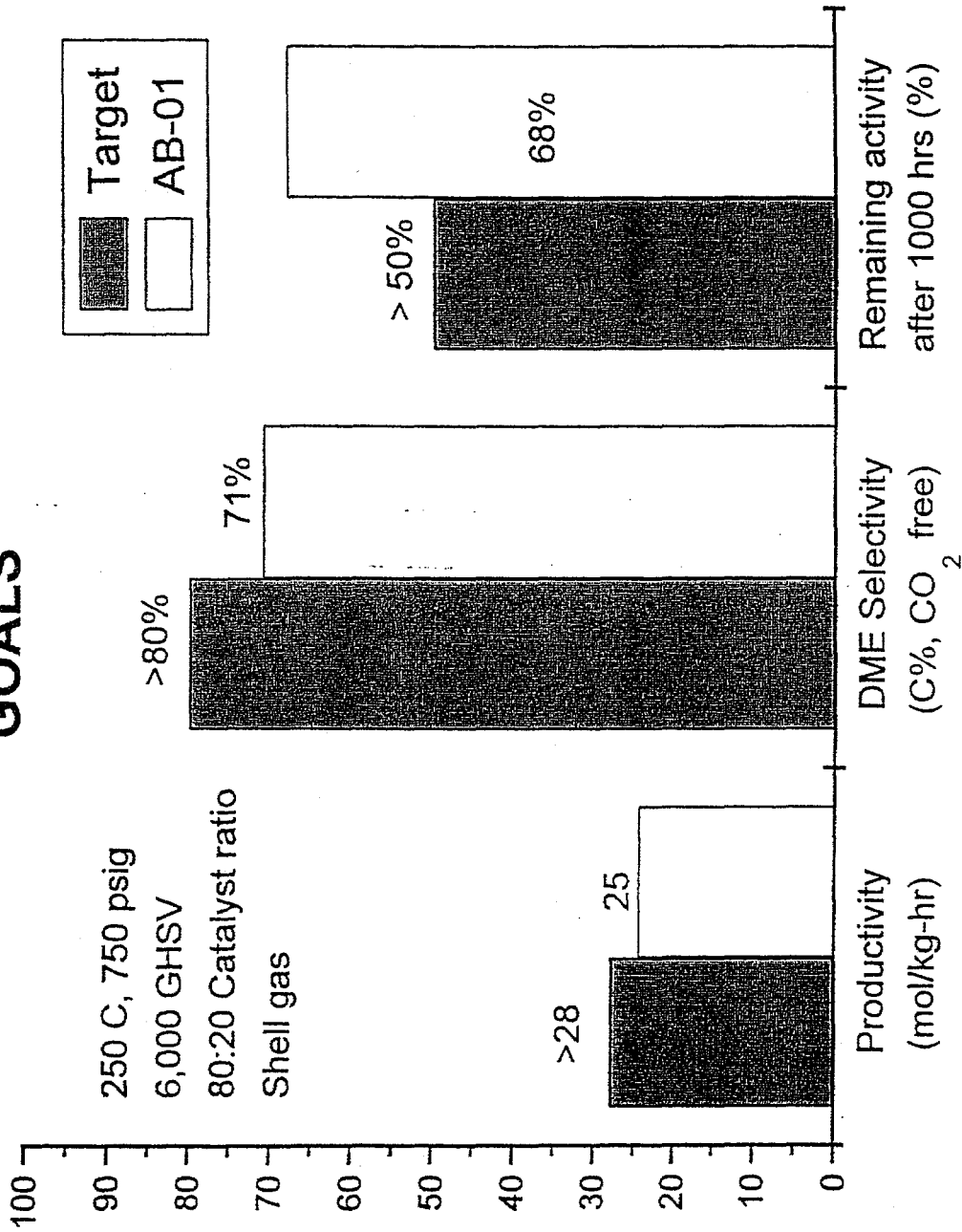
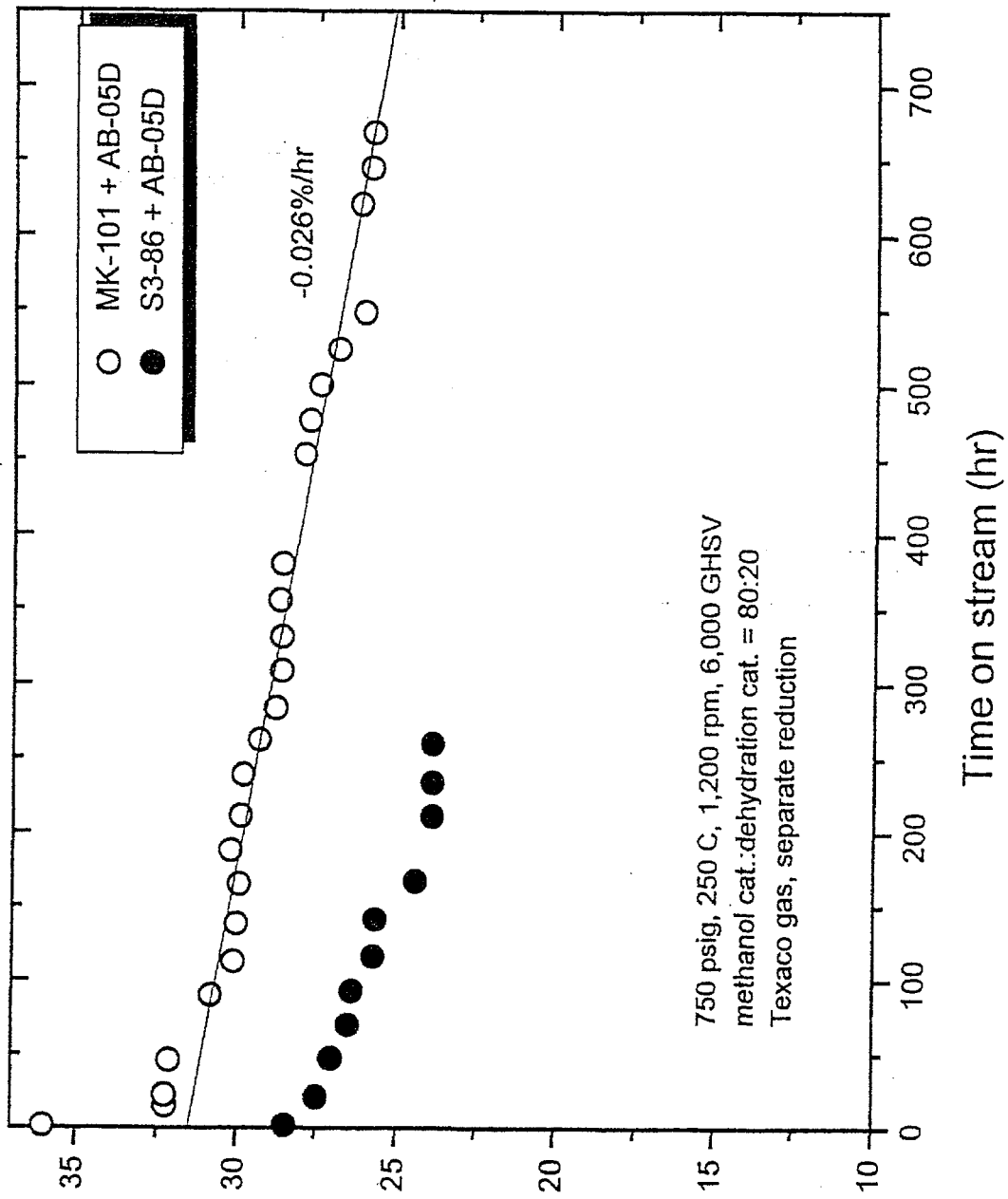


FIGURE 3

MEOH Equiv. Prod. (mol/kg-hr) **FIGURE 4**



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

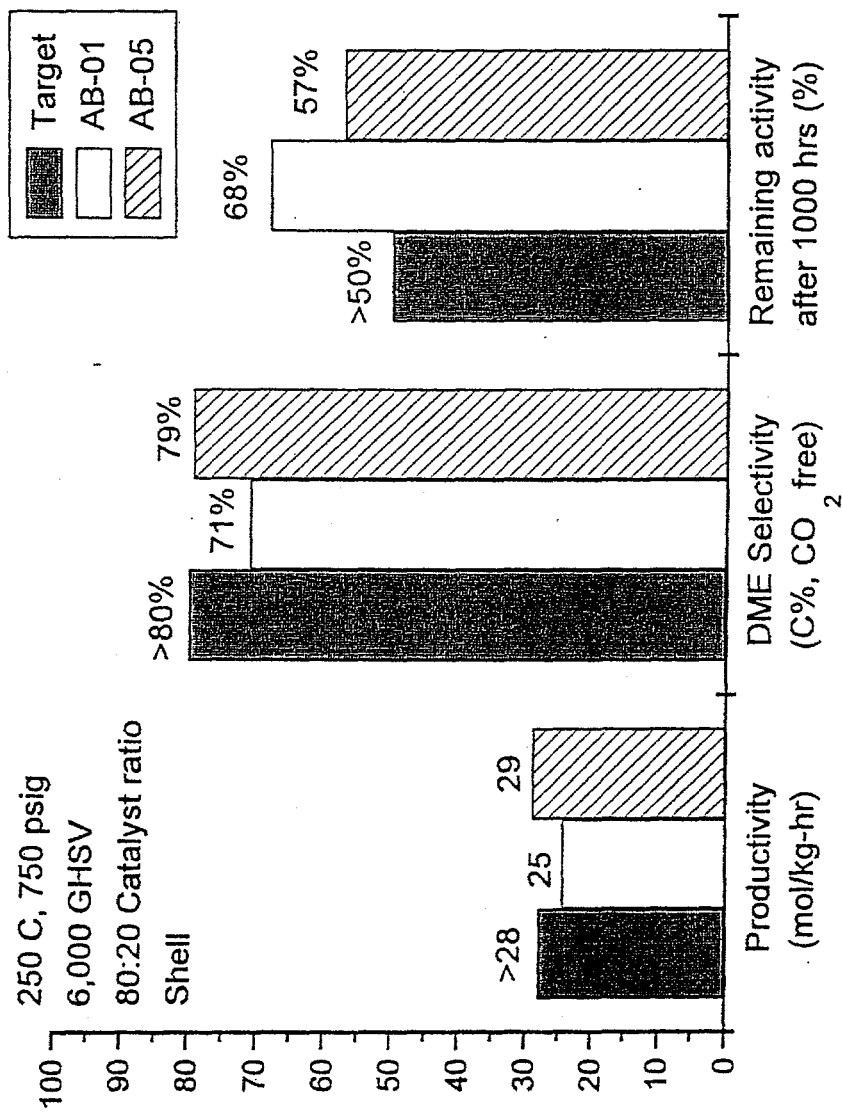


FIGURE 5