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

TECHNICAL PROGRESS REPORT NO. 10

For The Period

1 October - 31 December 1996

Prepared by

Air Products and Chemicals, Inc.
Allentown, Pennsylvania

and

Eastman Chemical Company
Kingsport, Tennessee

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

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

Patents cleared by Chicago on 10 March 1997.

DISCLAIMER

This report was prepared by Air Products & Chemicals, Inc. and Eastman Chemical Company for the Air Products Liquid Phase Conversion Company, L.P., pursuant to a Cooperative Agreement partially funded by the U.S. Department of Energy, and neither Air Products & Chemicals, Inc., Eastman Chemical Company, the Air Products Liquid Phase Conversion Company, L.P., nor any of their subcontractors nor the U.S. Department of Energy, nor any person acting on behalf of either:

(A) Makes any warranty or representation, express or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or
(B) Assumes any liabilities with respect to the use of, or for damages resulting from the use of, any information, apparatus, method, or process disclosed in this report.

Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute its endorsement, recommendation, or favoring by the U.S. Department of Energy. The views and opinions of authors expressed herein does not necessarily state or reflect those of the U.S. Department of Energy.

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). The LPMEOH™ Process Demonstration Unit is being built at a site located at the Eastman Chemical Company (Eastman) complex in Kingsport.

During this quarter, the Cooperative Agreement was modified (Mod A011) on 8 October 1996, authorizing the transition from Budget Period No. 2 (Design and Construction) to the final Budget Period (Commissioning, Start-up, and Operation). A draft Topical Report on Process Economics Studies concludes that methanol coproduction, with integrated gasification combined cycle (IGCC) electric power utilizing the LPMEOH™ process technology, will be competitive in serving local market needs. Planning for a proof-of-concept test run of the liquid phase dimethyl ether (DME) process at the LaPorte Alternative Fuels Development Unit (AFDU) was recommended; and a decision to proceed is pending. Construction (Task 2.2) is 97% complete, as of 31 December 1996. Completion of pipe pressure testing has taken longer than expected. This will delay completion of construction by about three weeks. Commissioning activities (Task 2.3) commenced in mid-October of 1996, and the demonstration unit is scheduled to be mechanically complete on 24 January 1997.

Table of Contents

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
DME	-	dimethyl ether
DOE	-	United States Department of Energy
DOE-PETC	-	The DOE's Pittsburgh Energy Technology Center (Project Team)
DOE-HQ	-	The DOE's Headquarters - Clean Coal Technology (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
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.
KSCFH	-	Thousand Standard Cubic Feet per Hour
LaPorte PDU	-	The DOE-owned experimental unit (PDU) located adjacent to Air Product’s 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)
MTBE	-	methyl tertiary butyl ether
NEPA	-	National Environmental Policy Act
OSHA	-	Occupational Safety and Health Administration
Partnership	-	Air Products Liquid Phase Conversion Company, L.P.
PDU	-	Process Development Unit
PFD	-	Process Flow Diagram(s)
ppb	-	parts per billion
Project	-	Production of Methanol/DME Using the LPMEOH™ Process at an Integrated Coal Gasification Facility
psia	-	Pounds per Square Inch (Absolute)
psig	-	Pounds per Square Inch (gauge)
P&ID	-	Piping and Instrumentation Diagram(s)
SCFH	-	Standard Cubic Feet per Hour
Sl/hr-kg	-	Standard Liter(s) per Hour per Kilogram of Catalyst
Syngas	-	Abbreviation for Synthesis Gas
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
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). The LPMEOH™ Process Demonstration Unit is being built at a site located at the Eastman Chemical Company (Eastman) complex in Kingsport.

On 4 October 1994, Air Products and Chemicals, Inc. (Air Products) and Eastman Chemical Company (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 (Mod. 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 will also provide the engineering design, procurement, construction, and commissioning of the LPMEOH™ Process Demonstration Unit, and will provide the technical and engineering supervision needed to conduct the operational testing program required as part of the project. As subcontractor to Air Products, Eastman will be 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 construction 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 being 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 testing 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 will demonstrate the commercial application of the LPMEOH™ process, to allow utilities to manufacture and sell two products: electricity and methanol. 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 1000 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 1 June 1995 (Mod M009). After approval, the project initiated Design - Phase 1 - activities; and initiated Construction - Phase 2 - activities 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 demonstration unit is scheduled to be mechanically complete in January of 1997.

During this quarter, the Cooperative Agreement was modified (Mod A011) on 8 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.

A draft Topical Report on Process Economics Studies was issued for review. The study concludes that methanol coproduction, with IGCC electric power utilizing the LPMEOH™ process technology, will be competitive in serving local market needs. The study results are also being incorporated into a paper "Fuel and Power Coproduction", which will be presented in January at the DOE's 5th Annual Clean Coal Technology Conference.

A recommendation to continue with DME design verification testing was made. 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 beening developed. Planning for a proof-of-concept test run at the LaPorte Alternative Fuels Development Unit (AFDU) was recommended; and a decision to proceed is pending.

The off-site product-use test plan is to be updated by May of 1997. During this quarter, Air Products and Acurex initiated contacting prospective Federal, State, and University product-use test participants who are involved in fuel cell, transportation, and stationary power plant developments.

A project review meeting was held in Kingsport in early December. The equipment is installed, and installation of the demonstration unit showed tremendous progress since the project review meeting in September. Programming of the instrument control system was completed during this quarter, and design engineering is now completed. The construction and operation permits have been obtained. Operator training was completed during this quarter, and final plans for startup and initial operation have been made.

Construction (Task 2.2) is 97% complete, as of the end of December 1996. Completion of pipe pressure testing has taken longer than expected. This has impacted completion of insulation and will delay completion of construction by about three weeks. Commissioning activities (Task 2.3) commenced in mid-October of 1996, and will be completed late in January of 1997. Startup, of the utility systems, is also scheduled to begin late in January.

The demonstration unit is scheduled to be mechanically complete on 24 January 1997. Ninety-one percent (91%) of the $38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 31 December 1996.

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). A demonstration unit producing 80,000 gallons per day (260 TPD) of methanol was designed and is being constructed at a site located at the Eastman Chemical Company (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 will occupy an area of 0.6 acre, is being 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 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.

C. Process Description

The LPMEOH™ demonstration unit is being 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 fuel testing to determine its suitability as a transportation fuel and as a fuel for stationary applications in the power industry.

D. Project Status

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. During this quarter, the programming of the Honeywell Digital Control System (DCS) was completed.

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 fuel-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 fuel-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 fuel test plan under-represents new utility dispersed electric power developments, and possibly new mobile transport engine developments. The updated fuel-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 (e.g., economic) market applications (municipal, industrial and electric utility) replacing or supplementing (gasoline, diesel, natural gas) commercial fuels, based on expected (1998 to 2018) U.S. energy market needs when the technology is to be commercialized.

The fuel-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 fuel-use tests. Fuel-use tests will be targeted for an approximate 18 to 30-month period, commencing in the second year of demonstration operations. The methanol product will be available for shipment from the demonstration unit in Kingsport, Tennessee. Air Products, Acurex Environmental Corporation (Acurex), and the DOE will develop the final fuel-use test plan.

Activity during this quarter

- The fuel-use test plan is targeted to be updated by May of 1997. This will allow 12 months for proper implementation of the tests, which will be conducted for an 18 to 30 month period commencing in May of 1998. The Demonstration Test Plan (see Task 2.3) indicates methanol for testing (as-produced from CO-rich syngas) will first be produced in May of 1998.

- Air Products and Acurex met in November of 1996. The listing of fuel-use test prospects was reviewed, and responsibilities were divided up and assigned for initiating contact. In general, Air Products will contact the various Federal Departments (Defense, Transportation, Energy) who are involved in fuel cell, transportation, and stationary power plant developments. State and University contacts were divided between Acurex and Air Products, according to their local knowledge. The Methanol Applications - Product Test Plan document (see Appendix C) was prepared for mailing to interested product-use test participants. Milestones were established to complete initial contacts by January, and to have an initial listing of likely prospects by the end of February of 1997.

- The listing and result of initial contacts, especially those involved in Federal programs, will be prepared and reviewed with the DOE Federal Energy Technology Center (FETC) for possible follow-up action. DOE FETC is involved in other fuel cell and stationary power plant development programs, and the possibility of demonstrating coproduction from central power plants for distributed power generation facilities might be appropriate.

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

A number of 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 1800 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) fuel-use test plan update.

Activities during this quarter

- Part One of the Outline - "Coproduction of Methanol" was issued, for review and comment, as a draft Topical Report. The 15 October 1996 transmittal memo is included in Appendix D. 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. 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. Comments on the draft Topical Report have been received, and plans are to update, revise and reissue the next draft in April of 1997.

- The Demonstration Test Plan (see Task 2.3) has been updated to include all the important commercial aspects that were identified by this study.

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

- Part Two of the Outline - "Baseload Power and Methanol Coproduction", is now being developed for incorporation into the paper, "Fuel and Power Coproduction", that is being prepared for presentation at the DOE's Fifth Annual Clean Coal Technology Conference in January of 1997.

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 Design Verification Testing decision milestone, on whether to continue with DME DVT, is targeted for 1 December 1996. 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 of DME, and to proceed with planning a proof-of-concept test run at the DOE's AFDU at LaPorte, Texas. A copy of the recommendation (dated 2 December 1996) 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 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's FY-97 Cost Plan (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 of DME with proof-of-concept testing the LaPorte AFDU is now under consideration. LPDME is not applicable to H_2-rich syngas; and it is unlikely that a substantive LPDME demonstration will be recommended for Kingsport. Therefore, a convincing case that the test-run on CO-rich syngas at LaPorte will lead to successful commercialization must be made, prior to approving the final test-run plan. The strategy for commercialization must present the technical logic to combine the results of:

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. This definition and laboratory work are being undertaken, and review meetings on the DME implementation decision are planned for the next quarter.

Market Economic Studies

Work on the feasibility study for the coproduction of DME and 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. 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 were therefore 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

- Laboratory testing of the dual catalyst system containing the new aluminum-based (AB) dehydration catalyst continued to show promise. LPDME tests were conducted at 240°C using Texaco-type syngas and AB02 catalyst. The results showed that good productivity and DME selectivity can be obtained at 240°C. Better stability of the methanol catalyst was also observed (as compared to the standard 250°C).

- AB05, one of the best aluminum-based samples, was successfully reproduced in another laboratory preparation.

- The stability of this new AB05 material was further improved by nitridation with ammonia at elevated pressure. An LPDME life test using this material is on-going. After 450 hours on stream, the methanol synthesis activity is close to the baseline synthesis activity for methanol catalyst in the absence of any dehydration catalyst.

- Observation that catalyst aging is enhanced using (Texaco-type) syngas as the feed allows the aging experiments to be performed faster and so that long-term aging for catalysts previously tested can be examined. This more rapid aging, as compared to (Shell-type) syngas, has been attributed to the higher water content (0.2 vs. 0.04 mol%). However, tests at higher water concentrations (0.33%) showed no further increase in methanol catalyst deactivation rate.

Task 1.5.4 Administration and Reporting

The Cooperative Agreement was modified (Mod A011; 8 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. A copy of the approval memorandum, dated 3 October 1996, is included in Appendix F.

A project review meeting was held on 3 and 4 December 1996 in Kingsport. Attendees from Air Products, Eastman, and DOE participated. The construction and commissioning status was reviewed, as were plans for Startup and Initial Operation. The construction site was visited, and the demonstration unit showed tremendous progress since the project review meeting in September. The C-120 vent stack was being erected, and the small bore piping was nearly all installed. The status of the updated fuel-use test plan, the recommendation for continuation of DME design verification testing, and other matters were reviewed. The meeting agenda, extracts from the meeting handouts, and the meeting notes are included in Appendix G.

The Milestone Schedule Status Report and the Cost Management Report, through the period ending 31 December 1996, are included in Appendix H. These two reports show the current schedule, the percentage completion and the latest cost forecast for each of the Work Breakdown Structure (WBS) tasks. The demonstration unit is scheduled to be mechanically

complete on 24 January 1997. Construction progress photographs, taken on 3 December 1996, are included in Appendix H. Ninety-one percent (91%) of the $38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 2 tasks have been expended (as invoiced), as of 31 December 1996.

The schedule for completion of the major Phase 2 Construction Tasks, has changed. Completion of pipe pressure testing has taken longer than expected. This has impacted completion of insulation and will delay completion of startup. Construction (Task 2.2) is now forecast for completion by 24 January 1997 (formerly 31 December 1996). Commissioning activities (Task 2.3) commenced in mid-October of 1996, and will be completed 24 January 1997 (formerly 17 January 1997).

The monthly reports for October, November, and December were submitted. These reports include the Milestone Schedule Status Report, the Project Summary Report, and the Cost Management Report. The Annual Cost Plan for Fiscal Year 1997 was submitted in October.

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.

  - All the equipment items have been received at the site.
  - The prefabricated structural steel, for the reactor building and for the catalyst building, has been received at the site. The bulk materials (prefab piping, valves, instrumentation, and electrical) have also been received at the site.
  - 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.

  - Air Products' construction site manager, lead electrical superintendent, lead mechanical superintendent, clerk and secretary are continuing to oversee the construction activities at the site.

- 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.

- The equipment is installed. The vent stack installation was completed in December. The process building structural steel is installed, only minor punch list work remains. The catalyst building roofing and siding are being installed, and should be complete in mid-January. The Mechanical Contractor has installed all of the large and small bore piping, and has completed hydrostatic pressure testing of all of the pipe circuits, except one. Completion of pipe circuit pressure testing has taken longer than expected. This has impacted completion of insulation and will delay startup.

- The Instrument and Electrical (I&E) Contractor is 96% complete. The prefabricated analyzer building was set in place, and cable runs to the Distributed Control System building were completed. Permanent power to the Motor Control Center building was energized, and the instrument air dryer system was commissioned in order to begin instrument loop checking. Wiring of the reactor and reduction vessel Nuclear Density Gauges remains to be completed.

- The Insulation and Fireproofing Contractor has completed the equipment items, and should complete the piping insulation by 21 January 1997.

- Complete mechanical construction so that checkout and commissioning can be started in Budget Period No. 3.

  - Construction is 97% complete as of the end of December of 1996. The targeted Mechanical Completion date has slipped about three weeks, to 24 January 1997. Commissioning activities started in mid-October and should be complete by mid-January. Startup, of the utility systems, are scheduled to begin 20 January 1997.

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 during this quarter. Four, one-week sessions, were held in November and December.

Commissioning activities started in mid-October. The Commissioning and Startup Schedule is included in Appendix I. Instrument loop checking is 62% complete. Final function tests of the instrumentation and interlocks will begin in January. Commissioning of the steam and other utility systems, and final system cleaning will also be in mid-January. Mineral oil loading and the introduction of syngas are forecast to occur at the end of January.

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 LPMEOH™ 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 J). The main goal and objective for this second annual plan is to initiate Phase 3 - Operation of the LPMEOH™ 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 1.5.4 (Administration and Reporting).

E. Planned Activities for the Next Quarter

- Complete Phase 2, Task 2 Construction.
- Complete Phase 2, Task 3 Equipment Commissioning.
- Complete Phase 3, Task 1 demonstration unit startup , including the utility systems, carbonyl burnout, and catalyst reduction.
- Initiate Phase 3, Task 2.1 Methanol Operation.
- Initiate planning for a DME proof-of-concept test run at the LaPorte AFDU, and obtain approval to proceed.
- Prepare a draft update of the Off-site Product-use Test Plan, for review and comment.
- Present the "Fuel and Power Coproduction" paper at the 5th Annual Clean Coal Technology Program Conference.
- Hold a Project Review/Update Meeting in Kingsport in March, with the DOE, Eastman, and Air Products.

F. Summary

The Cooperative Agreement was modified (Mod A011) on 8 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.

A draft Topical Report on Process Economics Studies was issued for review. This shows that methanol coproduction, with IGCC electric power utilizing the LPMEOH™ process technology, will be competitive in serving local market needs. The study results are also being incorporated into a paper "Fuel and Power Coproduction", which will be presented in January at the DOE's 5th Annual Clean Coal Technology Conference.

A recommendation to continue with DME design verification testing was made. DME design verification testing studies show the 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 AFDU was recommended; and a decision to proceed is pending.

The off-site product testing plan is to be updated by May of 1997. During this quarter, Air Products and Acurex initiated contacting prospective Federal, State, and University product test participants who are involved in fuel cell, transportation, and stationary power plant developments.

A project review meeting was held in Kingsport in early December. The equipment is installed, and installation of the demonstration unit showed tremendous progress since the project review meeting in September. Programming of the instrument control system was completed during this quarter, and design engineering is now completed. The construction

and operation permits have been obtained. Operator training was completed during this quarter, and final plans for startup and initial operation have been made.

Construction (Task 2.2) is 97% complete, as of the end of December 1996. Completion of pipe pressure testing has taken longer than expected. This has impacted completion of insulation and will delay completion of construction by about three weeks. Commissioning activities (Task 2.3) commenced in mid-October of 1996, and will be completed late in January of 1997. Startup, of the utility systems, is also scheduled to begin late in January.

The demonstration unit is scheduled to be mechanically complete on 24 January 1997. Ninety-one percent (91%) of the $38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 31 December 1996.

APPENDICES

APPENDIX A - SIMPLIFIED PROCESS FLOW DIAGRAM
1 PAGE

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

APPENDIX C - TASK 1.4 - METHANOL APPLICATIONS - PRODUCT TEST PLAN
1 PAGE

APPENDIX D - TASK 1.5.2 - PROCESS ECONOMIC STUDY
6 PAGES

Process Economics Study - Outline
(Draft - 10/15/96 - four pages)

and

LPMEOH™ Process Economics - for IGCC Coproduction
(Memo - 15 October 1996 - two pages)

APPENDIX E - TASK 1.5.3 - DME DESIGN VERIFICATION TESTING
4 PAGES

DME Milestone Plan

and

DME Design VerificationTesting Recommendation
(Draft - 12/02/96 - three pages)

APPENDIX F - TASK 1.5.4 - APPROVAL FOR BUDGET PERIOD THREE
1 PAGE

APPENDIX G - TASK 1.5.4 - PROJECT REVIEW MEETING
(3 & 4 December 1996)
22 PAGES

APPENDIX H - TASK 1.5.4 - MILESTONE SCHEDULE STATUS AND COST MANAGEMENT REPORTS
5 PAGES

and

Construction Progress Photographs
(at 3 December 1996)

APPENDIX I - TASK 2.3 - COMMISSIONING & STARTUP SCHEDULE
(Update - Dec/31/96 - four pages)
4 PAGES

APPENDIX J - TASK 2.5 - PARTNERSHIP ANNUAL PLAN
(For FY - 97)
6 PAGES