

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

TECHNICAL PROGRESS REPORT NO. 11

For The Period

1 January - 31 March 1997

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 27 May 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 was built at a site located at the Eastman Chemical Company (Eastman) complex in Kingsport.

During this quarter, the third draft of the Topical Report on Process Economics Studies was issued for review. A recommendation to continue with design verification testing on the coproduction of methanol and dimethyl ether (DME) was made. An liquid phase dimethyl ether (LPDME) catalyst system with reasonable long-term activity and stability is being developed, and a decision to proceed with a proof-of-concept test run at the LaPorte Alternative Fuels Development Unit (AFDU) is pending the release of a memo from Air Products on the catalyst targets and corresponding economics for a commercially successful LPDME catalyst. The off-site product-use test plan is to be updated in June of 1997. During this quarter, Air Products and Acurex Environmental Corporation continued developing the listing of product-use test participants who are involved in fuel cell, transportation, and stationary power plant applications. All major Construction (Task 2.2) contract work was completed during the reporting period. Commissioning activities (Task 2.3) focused on checkout of the equipment, instrument and control, and data acquisition systems. All utility systems were brought online.

Start-up activities (Task 3.1) began during the reporting period, and coal-derived synthesis gas (syngas) was introduced to the demonstration unit on 28 February. The recycle compressor was tested successfully on syngas at line pressure of 700 psig on 02 March, and the reactor loop reached 220°C for carbonyl burnout at 18:00 on 04 March. Iron carbonyl in the balanced gas feed remained below the 10 ppbv detection limit for all samples but one. Within the reactor loop, iron carbonyl levels peaked out near 200 ppbv after about 40 hours on-stream, before decreasing to between 10-20 ppbv at 160 hours on-stream. Nickel carbonyl measurements reached a peak of about 60 ppbv, and decreased at all sampling locations to below the 10 ppbv detection limit by 70 hours on-stream. Carbonyl sampling concluded on 12 March.

Catalyst activation of the nine 2250 lb batches required for the initial catalyst charge began on 16 March and concluded on 30 March. All batches met or slightly exceeded the "theoretical maximum" uptake of 2.82 SCF of reducing gas/lb catalyst. The first production of methanol at the demonstration unit is scheduled for April of 1997.

Ninety-seven percent (97%) 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 March 1997. One percent (1%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 March 1997.

Table of Contents

Abstract.....	3
ACRONYMS AND DEFINITIONS.....	5
Executive Summary	6
A. Introduction	9
B. Project Description.....	9
C. Process Description.....	10
D. Results and Discussion.....	11
Task 1.2 Permitting	11
Task 1.3 Design Engineering.....	11
Task 1.4 Off-Site Testing (Definition and Design)	11
Task 1.5 Planning and Administration	13
Task 1.5.1 Product-Use Test Plan.....	13
Task 1.5.2 Commercialization Studies	13
Task 1.5.3 DME Design Verification Testing.....	14
Task 1.5.4 Administration and Reporting.....	17
Task 2.1 Procurement.....	18
Task 2.2 Construction.....	18
Task 2.3 Training and Commissioning.....	20
Task 2.4 Off-Site Testing (Procurement and Construction).....	21
Task 2.5 Planning and Administration	21
Task 3.1 Startup.....	22
E. Planned Activities for the Next Quarter	23
F. Conclusion	24
APPENDICES	26
APPENDIX A - SIMPLIFIED PROCESS FLOW DIAGRAM	26
APPENDIX B - PROJECT EVALUATION PLAN FOR BUDGET PERIOD NO. 2.....	27
APPENDIX C - TASK 1.5.2 - PROCESS ECONOMIC STUDY	28
APPENDIX D - TASK 1.5.3 - DME DESIGN VERIFICATION TESTING.....	29
APPENDIX E - TASK 1.5.4 - APPROVAL FOR BUDGET PERIOD THREE	30
APPENDIX F - TASK 1.5.4 - PROJECT REVIEW MEETING	31
APPENDIX G - TASK 1.5.4 - MILESTONE SCHEDULE STATUS AND COST MANAGEMENT REPORTS	32
APPENDIX H - TASK 2.3 - COMMISSIONING & STARTUP SCHEDULE.....	33
APPENDIX I - TASK 2.5 - PARTNERSHIP ANNUAL PLAN	34
APPENDIX J - TEST AUTHORIZATION K0-CB - CARBONYL BURNOUT	35
APPENDIX K - TASK 3.1 - RESULTS OF CARBONYL BURNOUT	36
APPENDIX L - TEST AUTHORIZATION K0-AD - ACTIVATION OF METHANOL CATALYST	37
APPENDIX M - TASK 3.1 - REDUCTION GAS UPTAKE CURVES	38

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-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
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 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)
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)
ppbv	-	parts per billion (volume basis)
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 was designed, constructed, and has begun startup at a site located at the Eastman Chemical Company (Eastman) complex in Kingsport.

On 04 October 1994, Air Products and Chemicals, Inc. (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 (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 provided 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 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 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 01 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 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.

During this quarter, the third draft of the 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 were incorporated into a paper "Fuel and Power Coproduction", which was 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 being 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 release of a memo from Air Products on the catalyst targets and corresponding economics for a commercially successful LPDME catalyst.

The off-site, product-use test plan is to be updated in June of 1997. During this quarter, Air Products and Acurex continued developing the listing of 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 late March. The construction and commissioning status was reviewed, and results from activation of the first batch of catalyst were discussed. A tour of the completed demonstration unit was conducted.

All major Construction (Task 2.2) contract work was completed during the reporting period. The site paving and final painting will be completed in May of 1997. Commissioning activities (Task 2.3) focused on checkout of the equipment, instrument and control, and data acquisition systems. All utility systems were brought online. Activity in this task was completed on 28 February 1997 with initial introduction of syngas to the demonstration unit..

Start-up activities (Task 3.1) achieved a major milestone on 28 February with introduction of syngas for high pressure leak checking. The recycle compressor was tested successfully on syngas at line pressure of 700 psig on 02 March, and the reactor loop reached 220°C for carbonyl burnout at 18:00 on 04 March.

Iron carbonyl in the balanced gas feed remained below the 10 ppbv (parts per billion by volume) detectable limit for all samples but one. Within the reactor loop, iron carbonyl levels peaked at near 200 ppbv after about 40 hours on-stream, before decreasing rapidly over the next 40 hours. From 120-160 hours on-stream the concentrations remained steady between 10-20 ppbv. Nickel carbonyl measurements exhibited similar trends, with peak levels at about 60 ppbv and all points below the 10 ppbv detectable limit by 70 hours on-stream. In view of these results, carbonyl sampling concluded on 12 March.

Catalyst activation of the nine 2250 lb batches required for the initial catalyst charge began on 16 March and concluded on 30 March. As expected, after typical "learning curve"-type problems during the first few batches, the procedure became quite routine. Eventually, the entire operation was compressed into about 36 hours/batch. All batches met or slightly exceeded the "theoretical maximum" uptake of 2.82 SCF of reducing gas/lb catalyst. Throughout the repetitive activation procedure, the growing charge of reduced catalyst was agitated under a slightly reducing atmosphere in the slurry storage vessel (29D-02). The first production of methanol at the demonstration unit is scheduled for April of 1997.

Ninety-seven percent (97%) 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 March 1997. One percent (1%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 March 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). A demonstration unit producing 80,000 gallons per day (260 TPD) of methanol was designed, constructed, and has begun start-up 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 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 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 fuel 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 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 LPMEOHTM 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 in June 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 carbon monoxide (CO)-rich syngas) will first be produced in May of 1998.
- Air Products and Acurex continued to develop the listing of fuel-use test prospects. These projects are being prioritized by their likelihood to proceed and the timing for the initial need of methanol. A review meeting between Air Products, Acurex, and the DOE is planned for mid-April. A status update of the fuel-use program will be an agenda item for a 29-30 April interim review meeting between Air Products and the DOE.

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 C. This Outline addresses several needs for this Task 1.5.2 Commercialization Study:

- a) to provide process design guidance for commercial plant designs.
- b) to meet the Cooperative Agreement's technical objectives requirement for comparison with gas phase methanol technology. This preliminary assessment

- will help set demonstration operating goals, and identify the important market opportunities for the liquid phase technology.
- c) to provide input to the Demonstration Test Plan (Task 2.3).
 - d) to provide input to the Off-site Testing (Task 1.4) fuel-use test plan update.

Activities during this quarter

- Part One of the Outline - "Coproduct of Methanol" was reissued for review and comment, as a draft Topical Report. The 31 March 1997 transmittal memo is included in Appendix C. 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 this draft Topical Report are expected in early June of 1997.
- 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 fuel-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 1 December 1996. This milestone has been 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 D.

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 updated recommendation (dated 10 February 1997) is included in Appendix D. The recommendation was based on the results of the Market Economic Studies and on the LPDME catalyst system R&D work, and is summarized in the following.

The Market Economic Studies show that the LPDME process should have a significant economic advantage for the coproduction of DME 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 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 memo from Air Products on the catalyst targets and corresponding economics for a commercially successful LPDME catalyst will be issued in April of 1997. This document, along with updated laboratory results (as discussed in the following section), will be the basis for discussion at the 29-30 April interim review meeting between Air Products and the DOE.

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 D.

Laboratory R&D

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

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

laboratory work continued on developing an LPDME catalyst system based on the AB series of catalysts.

Summary of Laboratory Activity and Results

- Laboratory testing of the dual catalyst system containing the new aluminum-based (AB) dehydration catalyst continued to show promise. An LPDME run using AB05-C reported in Technical Progress Report #10 was continued up to 1027 hours on stream. The acceleration in the deactivation of the methanol catalyst with time was again observed. However, this acceleration was not accompanied by an increase in water concentration. This observation points to the conclusion that the physical properties of the slurry may result in catalyst deactivation.
- A second test with AB05-C was completed after 925 hours on Texaco-type syngas. Methanol synthesis activity was as stable as in LPMEOH™ runs. No increase in deactivation rate was observed after 700 hours. These results show that nitridation of the AB material can eliminate the accelerated loss of methanol synthesis activity.
- A new AB sample (AB05-D) was produced after a series of experiments were performed to study the effect of preparation techniques on catalyst performance. In an experiment with AB05-D, the methanol catalyst showed a stability similar to the LPMEOH™ baseline under the standard test conditions (250°C, 750 psig, 6,000 SI/hr-kg space velocity), the deactivation became very rapid when the space velocity was decreased to 2,000. This provides additional evidence that physical factors, such as agglomeration, may play an important role in this rapid deactivation. In another experiment, a particle-size analysis of spent slurry from a 1000-hour autoclave run revealed the presence of agglomerates.
- An LPDME run using methanol catalyst S3-86 with material AB-05C did not show accelerated deactivation when the space velocity was lowered. The key differences in this run (compared to previous experiments that have shown the effect of space velocity) were: (1) separate reduction of the S3-86 prior to introduction of the AB-05C; and (2) Texaco-type syngas rather than Shell-type.

Task 1.5.4 Administration and Reporting

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 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 3 October 1996, is included in Appendix E.

A project review meeting was held on 19 and 20 March 1997 in Kingsport. Attendees from Air Products, Eastman, and DOE participated. The construction and commissioning status was reviewed, and results from activation of the first batch of catalyst were discussed. The

data acquisition system in the partnership trailer was used to view the progress and completion of the second catalyst batch. A tour of the demonstration unit was conducted. 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 F.

The Milestone Schedule Status Report and the Cost Management Report, through the period ending 31 March 1997, are included in Appendix G. 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 was mechanically complete on 31 January 1997. Ninety-seven percent (97%) of the \$38 million of funds forecast for the Kingsport portion of the LPMEOHTM Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 31 March 1997. One percent (1%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 March 1997.

With the completion of construction (Task 2.2) on 31 January 1997, commissioning activities (Task 2.3) continued into February. Start-up activities (Task 3.1) were on-going during the reporting period, with the milestone of syngas introduction into the demonstration unit reached on 02 March 1997. The first batch of catalyst was activated on 16-17 March 1997; and by 31 March 1997, the remaining 8 batches had been activated. Initial methanol production is planned for 05 April 1997.

The monthly reports for January, February, and March were submitted. These reports include the Milestone Schedule Status Report, the Project Summary Report, and the Cost Management Report.

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.
- 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.
 - Pressure testing of the last piping circuit was completed on 05 February 1997. This circuit took additional time due to the need to replace manual isolation valves which did not meet the pressure test requirements after three attempts. Once the new valves were installed, the circuit passed the pressure test on the first attempt.
 - The hoist mechanisms for the two nuclear density gauges were also installed during the reporting period. The drives and gear assemblies for the hoists were reworked to provide the proper start-up and braking sequences. Structural beams were added to both applications in order to provide additional support to the pulley systems.
 - After a thorough review of the process piping, pressure relief circuits, and supports, the final piping circuit was accepted on 23 February 1997.
 - The installation of all instrument and electrical wiring was completed in late February. The last item completed was the wiring of the reactor and reduction vessel nuclear density gauges. A vendor representative from Texas Nuclear visited the site to supervise installation of the meters, perform a background radiation survey with Eastman Plant Protection personnel, and function-test the detectors.
 - The Insulation and Fireproofing Contractor completed all contract work on 10 March 1997.
- Complete mechanical construction so that check-out and commissioning can be started in Budget Period No. 3.
 - All major construction contract work has been completed. During the reporting period, commissioning activities (Task 2.3) were completed in parallel with the end of construction, and the demonstration unit was ready to receive syngas on 02 March 1997. Remaining construction activities include the completion of site paving/grading and the painting of large- and some small-bore piping systems. This work is being managed by an Air Products construction supervisor, and will be completed in May of 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 in December of 1996. Final additions to the operating manual were made in January of 1997.

Commissioning activities continued during the reporting period. The Commissioning and Start-up Schedule is included in Appendix H. Steam (100 psig) and plant nitrogen were introduced to the demonstration unit during the last week of January. Chemical cleaning of the distillation columns, methanol lot tanks, oil/slurry storage tanks, and the carbonyl guard bed was then performed to remove the layer of rust which had formed on vessel and piping surfaces during the construction period.

The recycle compressor (29K-01) was operated on nitrogen (80 psig suction pressure) in early February. The compressor is designed with a dry gas seal system which minimizes leakage of syngas to the atmosphere. During the nitrogen test, oil from the compressor gearbox migrated into the seal system as a result of the improper location of a vent line from the gear box to a vacuum blower. With oil filling this piping, the vacuum system could not function properly. The compressor seal system had to be disassembled so that the oil could be cleaned from the seal system. The vent piping was relocated, and the nitrogen test of the recycle compressor was completed.

All other rotating equipment (pumps, agitators) were function tested during February of 1997. Two pumps required attention during initial operation.

The condensed oil pumps (29G-01) were sent to a factory repair shop to clean/replace the pump seals which had been damaged due to exposure to moisture resulting from either the weather or pressure testing during construction. These pumps were then operated at 80 psig suction pressure.

The oil make-up pumps (29G-03) provide seal flush to the condensed oil pumps (29G-01), oil addition to the reactor loop, and high-pressure (1100 psig) piping flush oil. These pumps were tested at 700-900 psig discharge pressure, and initially operated well. Subsequently, the both oil make-up pumps exhibited difficulty in delivering oil at the required pressure. One of the pumps was sent to a factory repair shop, and the seals were found to be damaged as a result of exposure to moisture during construction. After this maintenance, the pump would not develop more than 600 psig discharge pressure at a dead-head condition. The second pump was sent to the same repair shop, rebuilt, and shipped to the factory in Canada to repeat the bench test which had been performed after initial assembly of the pump. A representative from Eastman was present to witness this test, during which this pump also

could not develop more than 600 psig discharge pressure. Air Products and Eastman are working with the manufacturer (Ingersoll Dresser) to determine the cause of the loss of performance and identify a solution. Note that the condensed oil pumps cannot be tested at full suction pressure until the oil make-up pumps are able to function at rated conditions.

Throughout the reporting period, Eastman personnel worked on system functional check-out to ensure that equipment and instrument systems operated properly. The distributed control system (DCS) and redundant safety shutdown system were checked. Graphics for the DCS were developed and optimized during this time. Eastman and Air Products personnel were working to install the data acquisition system, which operates concurrently with the DCS. Spreadsheets were developed so that historical data can be averaged, downloaded, and fed into report programs which calculate the heat and material balance for the demonstration unit. These systems were then tested during the demonstration unit start-up (Task 3.1).

Activity in this task was completed on 28 February 1997 with initial introduction of syngas to the demonstration unit.

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 I). 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).

Task 3.1 Start-up

Start-up activities began during the reporting period, and achieved a major milestone on 28 February with introduction of syngas for high pressure leak checking. The recycle compressor was tested successfully on syngas at line pressure on 02 March, and reactor heat-up for carbonyl burnout began later that day. Test Authorization #K0-CB for carbonyl burnout is attached in Appendix J.

The heat-up step became quite protracted by instrumentation troubleshooting, compressor trips, and other typical start-up problems. In addition, the heat-up itself, from a very cold start, provided several challenges in learning the dynamics of the whole heat exchange system (tube bundle, steam drum, level control, pressure control, high pressure steam flow, boiler feed water circulation pattern, etc.). The reactor reached 220°C at 18:00 on 04 March, and carbonyl sampling began immediately thereafter. CO Gas was introduced on 07 March to increase the partial pressure of CO in the reactor loop and speed formation and burnout of metal carbonyls. Unfortunately, CO availability problems limited CO flow to a 22 hour period.

Iron and nickel carbonyl measurements are summarized in tabular and graphical form in Appendix K. Iron carbonyl in the balanced gas feed remained below the 10 ppbv (parts per billion by volume) detectable limit for all samples but one. Within the reactor loop, iron carbonyl levels peaked at near 200 ppbv after about 40 hours on-stream, before decreasing rapidly over the next 40 hours. From 120-160 hours on-stream the concentrations remained steady between 10-20 ppbv. Nickel carbonyl measurements exhibited similar trends, with peak levels at about 60 ppbv and all points below the 10 ppbv detectable limit by 70 hours on-stream.

In view of these results, carbonyl sampling concluded on 12 March. During the burnout period, Eastman also commissioned the catalyst reduction equipment, including the feed gas control valves and flowmeters, the nuclear density gauge, and the utility oil skid. In addition, they commissioned the distillation equipment and test ran the system, first with water and then with methanol.

Catalyst activation began on 16 March with the first of nine 2250 lb batches required for the initial catalyst charge. Test Authorization #K0-AD for catalyst activation is attached in Appendix L. As expected, after typical "learning curve"-type problems during the first few batches, the procedure became quite routine. Eventually, the entire operation, including oil

fill, catalyst loading, mixing, activation temperature ramp, cooldown, transfers, and flushes, was compressed into about 36 hours/batch.

Curves of the uptake of reducing gas for each of the nine batches are included in Appendix M. All batches met or slightly exceeded the "theoretical maximum" uptake of 2.82 SCF of reducing gas/lb catalyst. Notably, the temperature control scheme, via the jacketed vessel and utility oil skid, was somewhat unstable throughout various tuning attempts during the first five batches. During the fifth batch, Eastman's start-up engineer with the most controls experience returned to shift duty and completely modified the control scheme to a split-range mode. This conceptual change dramatically improved the temperature control of the process, resulting in very uniform reducing gas uptake profiles for the final four batches.

Nuclear density gauge (NDG) scans were taken at approximately 190°C for each catalyst batch, and somewhat more frequently during the last two batches. At 190°C the holdup calculations varied from 19-24 vol% for catalyst concentrations between 29-33 wt%. NDG profiles were always uniform throughout the slurry.

Activation of the ninth and final start-up batch of catalyst was completed on 30 March. Throughout the repetitive activation procedure, the growing charge of reduced catalyst was agitated under a slightly reducing atmosphere in the slurry storage vessel (29D-02).

E. Planned Activities for the Next Quarter

- Complete Phase 2, Task 2 Construction, including the site paving and painting contracts.
- Begin Phase 3, Task 2.1 Methanol Operation.
- Complete any maintenance activities as part of Eastman's biannual complex outage (scheduled to begin 10 May 1997).
- Continue planning for a DME proof-of-concept test run at the LaPorte AFDU, and issue the Final DVT Recommendation document to DOE.
- Submit the draft update of the Off-Site Product-Use Test Plan to DOE.
- Submit the Demonstration Technology Start-up Report to DOE.
- Hold an interim Project Review/Update Meeting in Allentown in April, with the DOE and Air Products.
- Incorporate DOE comments into the Topical Report on Process Economic Studies.
- Make plans for the dedication ceremony at the demonstration unit.

F. Conclusion

The third draft of the Topical Report on Process Economic 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 were incorporated into a paper "Fuel and Power Coproduction", which was 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 being 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 release of a memo from Air Products on the catalyst targets and corresponding economics for a commercially successful LPDME catalyst.

The off-site product-use test plan is to be updated in June of 1997. During this quarter, Air Products and Acurex continued developing the listing of 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 late March. The construction and commissioning status was reviewed, and results from activation of the first batch of catalyst were discussed. A tour of the completed demonstration unit was conducted.

All major Construction (Task 2.2) contract work was completed during the reporting period. The site paving and final painting will be completed in May of 1997. Commissioning activities (Task 2.3) focused on check-out of the equipment, instrument and control, and data acquisition systems. All utility systems were brought on-line. Activity in this task was completed on 28 February 1997 with initial introduction of syngas to the demonstration unit.

Start-up activities (Task 3.1) achieved a major milestone on 28 February with introduction of syngas for high pressure leak checking. The recycle compressor was tested successfully on syngas at line pressure on 02 March, and the reactor loop reached 220°C for carbonyl burnout at 18:00 on 04 March.

Iron carbonyl in the balanced gas feed remained below the 10 ppbv (parts per billion by volume) detectable limit for all samples but one. Within the reactor loop, iron carbonyl levels peaked at near 200 ppbv after about 40 hours on-stream, before decreasing rapidly over the next 40 hours. From 120-160 hours on-stream the concentrations remained steady between 10-20 ppbv. Nickel carbonyl measurements exhibited similar trends, with peak levels at about 60 ppbv and all points below the 10 ppbv detectable limit by 70 hours on-stream. In view of these results, carbonyl sampling concluded on 12 March.

Catalyst activation of the nine 2250 lb batches required for the initial catalyst charge began on 16 March and concluded on 30 March. As expected, after typical "learning curve"-type problems during the first few batches, the procedure became quite routine. Eventually, the entire operation was compressed into about 36 hours/batch. All batches met or slightly exceeded the "theoretical maximum" uptake of 2.82 SCF of reducing gas/lb catalyst.

Throughout the repetitive activation procedure, the growing charge of reduced catalyst was agitated under a slightly reducing atmosphere in the slurry storage vessel (29D-02). The first production of methanol at the demonstration unit is scheduled for April of 1997.

Ninety-seven percent (97%) 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 March 1997. One percent (1%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 March 1997.

APPENDICES

APPENDIX A - SIMPLIFIED PROCESS FLOW DIAGRAM

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

APPENDIX C - 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)**

APPENDIX D - TASK 1.5.3 - DME DESIGN VERIFICATION TESTING

DME Milestone Plan

and

**DME Design Verification Testing Recommendation
(Draft - 2/10/97 - three pages)**

APPENDIX E - TASK 1.5.4 - APPROVAL FOR BUDGET PERIOD THREE

**APPENDIX F - TASK 1.5.4 - PROJECT REVIEW MEETING
(19 & 20 March 1997)**

**APPENDIX G - TASK 1.5.4 - MILESTONE SCHEDULE STATUS AND COST
MANAGEMENT REPORTS**

APPENDIX H - TASK 2.3 - COMMISSIONING & STARTUP SCHEDULE
(Update - 3/17/97 - five pages)

**APPENDIX I - TASK 2.5 - PARTNERSHIP ANNUAL PLAN
(For FY - 97)**

APPENDIX J - TEST AUTHORIZATION K0-CB - CARBONYL BURNOUT

APPENDIX K - TASK 3.1 - RESULTS OF CARBONYL BURNOUT

**APPENDIX L - TEST AUTHORIZATION K0-AD - ACTIVATION OF METHANOL
SYNTHESIS CATALYST**

APPENDIX M - TASK 3.1 - REDUCTION GAS UPTAKE CURVES