

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

TECHNICAL PROGRESS REPORT NO. 12

For The Period

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for the

Air Products Liquid Phase Conversion Company, L.P.

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Abstract

The Liquid Phase Methanol (LPMEOH™) Demonstration Project at Kingsport, Tennessee, is a \$213.7 million cooperative agreement between the U.S. Department of Energy (DOE) and Air Products Liquid Phase Conversion Company, L.P. (the Partnership). Air Products and Chemicals, Inc. (Air Products) and Eastman Chemical Company (Eastman) formed the Partnership to execute the Demonstration Project. The LPMEOH™ Process Demonstration Unit was built at a site located at the Eastman complex in Kingsport.

During this quarter, comments from the DOE on the Topical Report "Economic Analysis - LPMEOH™ Process as an Add-on to IGCC for Coproduction" were received. A recommendation to continue with design verification testing for the coproduction of dimethyl ether (DME) and methanol 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. An LPDME catalyst system with reasonable long-term activity and stability is being developed. A recommendation document summarizing catalyst targets, experimental results, and the corresponding economics for a commercially successful LPDME catalyst was issued on 30 June 1997.

The off-site, product-use test plan was updated in June of 1997. During this quarter, Acurex Environmental Corporation and Air Products screened proposals for this task by the likelihood of the projects to proceed and the timing for the initial methanol requirement. Eight sites from the list have met these criteria. The formal submission of the eight projects for review and concurrence by the DOE will be made during the next reporting period.

The site paving and final painting were completed in May of 1997. Start-up activities were completed during the reporting period, and the initial methanol production from the demonstration unit occurred on 02 April 1997. The first extended stable operation at the nameplate capacity of 80,000 gallons per day (260 tons per day) took place on 06 April 1997.

Pressure drop and resistance coefficient across the gas sparger at the bottom of the reactor increased over this initial operating period. The demonstration unit was shut down from 08 May - 17 June 1997 as part of a scheduled complex outage for the Kingsport site. During this outage, the gas sparger was removed, cleaned, and reinstalled. After completion of other maintenance activities, the demonstration unit was restarted, and maintained stable operation through the remainder of the reporting period. Again, the gas sparger showed an increase in pressure drop and resistance since the restart, although not as rapidly as during the April-May operation. Fresh oil was introduced online for the first time to a new flush connection on the gas inlet line to the reactor; the flush lowered the pressure drop by 1 psi. However, the effects were temporary, and the sparger resistance coefficient continued to increase. Additional flushing with both fresh oil and entrained slurry recovered in the cyclone and secondary oil knock-out drum will be attempted in order to stabilize the sparger resistance coefficient.

Catalyst activity, as defined by the ratio of the rate constant at any point in time to the rate constant for a freshly reduced catalyst (as determined in the laboratory autoclave), declined more rapidly than expected. A catalyst slurry sample was taken during the May/June 1997 complex outage for analysis.

Overall, the LPMEOH™ Demonstration Unit operated well during the initial campaign. The availability of the LPMEOH™ Demonstration Unit was 94.9% during the reporting period. All methanol produced (a total of 2,900,692 gallons) was used by Eastman in the production of methyl acetate, and ultimately cellulose acetate and acetic acid. The start-up was successfully completed in a safe and environmentally sound manner.

Ninety-eight percent (98%) of the \$38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 30 June 1997. Five percent (5%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 June 1997.

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ACRONYMS AND DEFINITIONS

Acurex	-	Acurex Environmental Corporation
Air Products	-	Air Products and Chemicals, Inc.
AFDU	-	Alternative Fuels Development Unit - The "LaPorte PDU"
Balanced Gas	-	A syngas with a composition of hydrogen (H ₂), carbon monoxide (CO), and carbon dioxide (CO ₂) in stoichiometric balance for the production of methanol
Carbon Monoxide Gas	-	A syngas containing primarily carbon monoxide (CO); also called CO Gas
Catalyst Age (η -eta)	-	the ratio of the rate constant at any point in time to the rate constant for a freshly reduced catalyst (as determined in the laboratory autoclave)
Catalyst Concentration	-	Synonym for Slurry Concentration
Catalyst Loading	-	Synonym for Slurry Concentration
CO Conversion	-	the percentage of CO consumed across the reactor
Crude Grade Methanol	-	Underflow from rectifier column (29C-20), defined as 80 wt% minimum purity; requires further distillation in existing Eastman equipment prior to use
DME	-	dimethyl ether
DOE	-	United States Department of Energy
DOE-FETC	-	The DOE's Federal Energy Technology Center (Project Team)
DOE-HQ	-	The DOE's Headquarters - Coal Fuels and Industrial Systems (Project Team)
DTP	-	Demonstration Test Plan - The four-year Operating Plan for Phase 3, Task 2 Operation
DVT	-	Design Verification Testing
Eastman	-	Eastman Chemical Company
EIV	-	Environmental Information Volume
EMP	-	Environmental Monitoring Plan
EPRI	-	Electric Power Research Institute
Fresh Feed	-	sum of Balanced Gas, H ₂ Gas, and CO Gas
Gas Holdup	-	the percentage of reactor volume up to the Gassed Slurry Height which is gas
Gassed Slurry Height	-	height of gassed slurry in the reactor
HAPs	-	Hazardous Air Pollutants
Hydrogen Gas	-	A syngas containing an excess of hydrogen (H ₂) over the stoichiometric balance for the production of methanol; also called H ₂ Gas
IGCC	-	Integrated Gasification Combined Cycle, a type of electric power generation plant
IGCC/OTM	-	An IGCC plant with a "Once-Thru Methanol" plant (the LPMEOH™ Process) added-on
Inlet Superficial Velocity	-	the ratio of the actual cubic feet of gas at the reactor inlet (calculated at the reactor temperature and pressure) to the reactor cross-sectional area (excluding the area contribution by the internal heat exchanger); typical units are feet per second
K	-	Sparger resistance coefficient (term used in calculation of pressure drop)
KSCFH	-	Thousand Standard Cubic Feet per Hour
LaPorte PDU	-	The DOE-owned experimental unit (PDU) located adjacent to Air Products' industrial gas facility at LaPorte, Texas, where the LPMEOH™ process was successfully piloted
LPDME	-	Liquid Phase DME process, for the production of DME as a mixed coproduct with methanol
LPMEOH™	-	Liquid Phase Methanol (the technology to be demonstrated)
MeOH	-	methanol
Methanol Productivity	-	the gram-moles of methanol produced per hour per kilogram catalyst (on an oxide basis)
MTBE	-	methyl tertiary butyl ether
MW	-	molecular weight, pound per pound mole
NEPA	-	National Environmental Policy Act
OSHA	-	Occupational Safety and Health Administration
ρ	-	density, pounds per cubic foot
ΔP	-	pressure drop, psi
Partnership	-	Air Products Liquid Phase Conversion Company, L.P.

PDU - Process Development Unit

ACRONYMS AND DEFINITIONS (cont'd)

PFD	-	Process Flow Diagram(s)
ppbv	-	parts per billion (volume basis)
Project	-	Production of Methanol/DME Using the LPMEOH™ Process at an Integrated Coal Gasification Facility
psi	-	Pounds per Square Inch
psia	-	Pounds per Square Inch (Absolute)
psig	-	Pounds per Square Inch (gauge)
P&ID	-	Piping and Instrumentation Diagram(s)
Raw Methanol	-	sum of Refined Grade Methanol and Crude Grade Methanol; represents total methanol which is produced after stabilization
Reactor Feed	-	sum of Fresh Feed and Recycle Gas
Reactor O-T-M Conversion	-	percentage of energy (on a lower heating value basis) in the Reactor Feed converted to methanol (Once-Through-Methanol basis)
Reactor Volumetric Productivity	-	the quantity of Raw Methanol produced (tons per day) per cubic foot of reactor volume up to the Gassed Slurry Level
Recycle Gas	-	the portion of unreacted syngas effluent from the reactor “recycled” as a feed gas
Refined Grade Methanol	-	Distilled methanol, defined as 99.8 wt% minimum purity; used directly in downstream Eastman processes
SCFH	-	Standard Cubic Feet per Hour
Slurry Concentration	-	percentage of weight of slurry (solid plus liquid) which is catalyst (on an oxide basis)
Sl/hr-kg	-	Standard Liter(s) per Hour per Kilogram of Catalyst
Syngas	-	Abbreviation for Synthesis Gas
Syngas Utilization	-	defined as the number of standard cubic feet of Balanced Gas plus CO Gas to the LPMEOH™ Demonstration Unit required to produce one pound of Raw Methanol
Synthesis Gas	-	A gas containing primarily hydrogen (H ₂) and carbon monoxide (CO), or mixtures of H ₂ and CO; intended for "synthesis" in a reactor to form methanol and/or other hydrocarbons (synthesis gas may also contain CO ₂ , water, and other gases)
Tie-in(s)	-	the interconnection(s) between the LPMEOH™ Process Demonstration Facility and the Eastman Facility
TPD	-	Ton(s) per Day
V	-	volumetric flowrate, thousand standard cubic feet per hour
WBS	-	Work Breakdown Structure
wt	-	weight

Executive Summary

The Liquid Phase Methanol (LPMEOH™) Demonstration Project at Kingsport, Tennessee, is a \$213.7 million cooperative agreement between the U.S. Department of Energy (DOE) and Air Products Liquid Phase Conversion Company, L.P. (the Partnership). Air Products and Chemicals, Inc. (Air Products) and Eastman Chemical Company (Eastman) formed the Partnership to execute the Demonstration Project. The LPMEOH™ Process Demonstration Unit was designed, constructed, and has begun start-up at a site located at the Eastman complex in Kingsport.

On 04 October 1994, Air Products and Eastman signed the agreements that would form the Partnership, secure the demonstration site, and provide the financial commitment and overall project management for the project. These partnership agreements became effective on 15 March 1995, when DOE authorized the commencement of Budget Period No. 2 (Modification No. A008 to the Cooperative Agreement). The Partnership has subcontracted with Air Products to provide the overall management of the project, and to act as the primary interface with DOE. As subcontractor to the Partnership, Air Products provided the engineering design, procurement, construction, and commissioning of the LPMEOH™ Process Demonstration Unit, and is providing the technical and engineering supervision needed to conduct the operational testing program required as part of the project. As subcontractor to Air Products, Eastman is responsible for operation of the LPMEOH™ Process Demonstration Unit, and for the interconnection and supply of synthesis gas (syngas), utilities, product storage, and other needed services.

The project involves the 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 test program will be conducted to demonstrate the suitability of the methanol product as a transportation fuel and as a fuel for stationary applications for small modular electric power generators for distributed power.

The four-year operating test phase 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 1,000 TPD). A successful demonstration at Kingsport will show the ability of a local resource (coal) to be converted in a reliable (storable) and environmentally preferable way to provide the clean energy needs of local communities for electric power and transportation.

This project may also demonstrate the production of dimethyl ether (DME) as a mixed coproduct with methanol if laboratory- and pilot-scale research and market verification studies show promising results. If implemented, the DME would be produced during the last six months of the four-year demonstration period. DME has several commercial uses. In a storable blend with methanol, the mixture can be used as a peaking fuel in gasification-based electric power generating facilities, or as a diesel engine fuel. Blends of methanol and DME can be used as chemical feedstocks for synthesizing chemicals, including new oxygenated fuel additives.

The project was reinitiated in October of 1993, when DOE approved a site change to the Kingsport location. DOE conditionally approved the Continuation Application to Budget Period No. 2 (Design and Construction) in March of 1995 and formally approved it on 01 June 1995 (Mod M009). After approval, the project initiated Phase 1 - Design - activities. Phase 2 - Construction - activities were initiated in October of 1995. The project required review under the National Environmental Policy Act (NEPA) to move to the construction phase. DOE prepared an Environmental Assessment (DOE/EA-1029), and subsequently a Finding of No Significant Impact (FONSI) was issued on 30 June 1995. The Cooperative Agreement was modified (Modification No. A011) on 08 October 1996, authorizing the transition from Budget Period No. 2 (Design and Construction) to the final Budget Period (Commissioning, Start-up, and Operation). This modification provides the full \$213,700,000 of authorized funding, with 56.7% participant cost share and 43.3% DOE cost share.

During this quarter, comments from the DOE on the Topical Report "Economic Analysis - LPMEOH™ Process as an Add-on to IGCC for Coproduction" were received. The study concludes that methanol coproduction, with IGCC electric power utilizing the LPMEOH™ process technology, will be competitive in serving local market needs.

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 Alternative Fuels Development Unit (AFDU) in LaPorte, TX was recommended. A recommendation document summarizing catalyst targets, experimental results, and the

corresponding economics for a commercially successful LPDME catalyst was issued on 30 June 1997.

The off-site, product-use test plan was updated in June of 1997. During this quarter, Acurex Environmental Corporation (Acurex) and Air Products screened proposals for this task by the likelihood of the projects to proceed and the timing for the initial methanol requirement. Eight sites from the list have met these criteria. The formal submission of the eight projects for review and concurrence by the DOE will be made during the next reporting period.

An interim project review meeting was held in Allentown in late April of 1997. An update on the performance of the demonstration unit was provided, and the status of the DME recommendation and the off-site, product-use test plan were discussed.

The site paving and final painting were completed in May of 1997. Start-up activities were completed during the reporting period, and the initial methanol production from the demonstration unit occurred on 02 April 1997. The first extended stable operation at the nameplate capacity of 80,000 gallons per day (260 TPD) took place on 06 April 1997. Pressure drop and resistance coefficient across the gas sparger at the bottom of the reactor increased over this initial operating period. The demonstration unit was shut down on 08 May 1997 as part of a scheduled complex outage for the Kingsport site. During this outage, the gas sparger was removed, cleaned, and reinstalled. After completion of other maintenance activities, the demonstration unit was restarted on 17 June 1997, and maintained stable operation through the remainder of the reporting period. Again, the gas sparger showed an increase in pressure drop and resistance since the restart, although not as rapidly as during the April/May operation. Fresh oil was introduced online for the first time to a new flush connection on the gas inlet line to the reactor; the flush lowered the pressure drop by 1 psi. However, the effects were temporary, and the sparger resistance coefficient continued to increase. Additional flushing with both fresh oil and entrained slurry recovered in the cyclone and secondary oil knock-out drum will be attempted in order to stabilize the sparger resistance coefficient.

Catalyst activity, as defined by the ratio of the rate constant at any point in time to the rate constant for a freshly reduced catalyst (as determined in the laboratory autoclave), declined more rapidly than expected. A catalyst slurry sample was taken during the May/June 1997 complex outage for analysis.

Overall, the LPMEOHTM Demonstration Unit operated well during the initial campaign. The availability of the LPMEOHTM Demonstration Unit was 94.9% during the reporting period. All methanol produced (a total of 2,900,692 gallons) was used by Eastman in the production of methyl acetate, and ultimately cellulose acetate and acetic acid. The start-up was successfully completed in a safe and environmentally sound manner.

Ninety-eight percent (98%) 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 30 June 1997. Five percent (5%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 June 1997.

A. Introduction

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

This project is sponsored under the DOE's Clean Coal Technology Program, and its primary objective is to “demonstrate the production of methanol using the LPMEOH™ Process in conjunction with an integrated coal gasification facility.” The project will also demonstrate the suitability of the methanol produced for use as a chemical feedstock or as a low-sulfur dioxide, low-nitrogen oxides alternative fuel in stationary and transportation applications. The project may also demonstrate the production of dimethyl ether (DME) as a mixed coproduct with methanol, if laboratory- and pilot-scale research and market verification studies show promising results. If implemented, the DME would be produced during the last six months of the four-year demonstration period.

The LPMEOH™ process is the product of a cooperative development effort by Air Products and the DOE in a program that started in 1981. It was successfully piloted at a 10-TPD rate in the DOE-owned experimental unit at Air Products' LaPorte, Texas, site. This demonstration project is the culmination of that extensive cooperative development effort.

B. Project Description

The demonstration unit, which occupies an area of 0.6 acre, is integrated into the existing 4,000-acre Eastman complex located in Kingsport, Tennessee. The Eastman complex employs approximately 12,000 people. In 1983, Eastman constructed a coal gasification facility utilizing Texaco technology. The synthesis gas (syngas) generated by this gasification facility is used to produce carbon monoxide and methanol. Both of these products are used to produce methyl acetate and ultimately cellulose acetate and acetic acid. The availability of this highly reliable coal gasification facility was the major factor in selecting this location for the LPMEOH™ Process Demonstration. Three different feed gas streams (hydrogen gas, carbon monoxide gas, and balanced gas) will be diverted from existing operations to the LPMEOH™ demonstration unit, thus providing the range of coal-derived syngas ratios (hydrogen to carbon monoxide) needed to meet the technical objectives of the demonstration project.

For descriptive purposes and for design and construction scheduling, the project has been divided into four major process areas with their associated equipment:

- *Reaction Area* - Syngas preparation and methanol synthesis reaction equipment.
- *Purification Area* - Product separation and purification equipment.

- *Catalyst Preparation Area* - Catalyst and slurry preparation and disposal equipment.
- *Storage/Utility Area* - Methanol product, slurry, and oil storage equipment.

The physical appearance of this facility closely resembles the adjacent Eastman process plants, including process equipment in steel structures.

- *Reaction Area*

The reaction area includes feed gas compressors, catalyst guard beds, the reactor, a steam drum, separators, heat exchangers, and pumps. The equipment is supported by a matrix of structural steel. The most salient feature is the reactor, since with supports, it is approximately 84-feet tall.

- *Purification Area*

The purification area features two distillation columns with supports; one is approximately 82-feet tall, and the other 97-feet tall. These vessels resemble the columns of the surrounding process areas. In addition to the columns, this area includes the associated reboilers, condensers, air coolers, separators, and pumps.

- *Catalyst Preparation Area*

The catalyst preparation area consists of a building with a roof and partial walls, in which the catalyst preparation vessels, slurry handling equipment, and spent slurry disposal equipment are housed. In addition, a hot oil utility system is included in the area.

- *Storage/Utility Area*

The storage/utility area includes two diked lot-tanks for methanol, two tanks for oil storage, a slurry holdup tank, a trailer loading/unloading area, and an underground oil/water separator. A vent stack for safety relief devices is located in this area.

C. Process Description

The LPMEOH™ demonstration unit is integrated with Eastman's coal gasification facility. A simplified process flow diagram is included in Appendix A. Syngas is introduced into the slurry reactor, which contains a slurry of liquid mineral oil with suspended solid particles of catalyst. The syngas dissolves through the mineral oil, contacts the catalyst, and reacts to form methanol. The heat of reaction is absorbed by the slurry and is removed from the slurry by steam coils. The methanol vapor leaves the reactor, is condensed to a liquid, sent to the distillation columns for removal of higher alcohols, water, and other impurities, and is then stored in the day tanks for sampling before being sent to Eastman's methanol storage. Most of the unreacted syngas is recycled back to the reactor with the syngas recycle compressor, improving cycle efficiency. The methanol will be used for downstream feedstocks and in off-site, product-use testing to determine its suitability as a transportation fuel and as a fuel for stationary applications in the power industry.

D. Results and Discussion

The project status is reported by task, and then by the goals established by the Project Evaluation Plan for Budget Period No. 2 (see Appendix B). Major accomplishments during this period are as follows:

Task 1.2 Permitting

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

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

Task 1.3 Design Engineering

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

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

Task 1.4 Off-Site Testing (Definition and Design)

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

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

Discussion

The product-use test plan, developed in 1992 to support the demonstration at the original Cool Water Gasification Facility site, has become outdated. Since the site change to Eastman, the original product test plan under-represents new utility dispersed electric power developments, and possibly new mobile transport engine developments. The updated product-use test plan will attempt for broader market applications and for commercial fuels comparisons. The objective of the product-use test plan update will be to demonstrate commercial market applications for the “as produced” methanol as a replacement fuel and as a fuel supplement. Fuel economics will be evaluated for the “as produced” methanol for use in municipal, industrial, and utility applications and as fuel supplements for gasoline, diesel, and natural gas. These fuel evaluations will be based on the U.S. energy market needs projected during the 1998 to 2018 time period when the LPMEOH™ technology is expected to be commercialized.

The product-use test plan will be developed to enhance the early commercial acceptance of central clean coal technology processing facilities, coproducing electricity and methanol to meet the needs of the local community. One of the advantages of the LPMEOH™ process for coproduction from coal-derived syngas is that the as-produced, stabilized (degassed) methanol product is of unusually high quality (e.g. less than 1 wt. % water) which may be suitable for the premium fuel applications. Cost savings (10 to 15%) of several cents per gallon of methanol can be achieved, if the suitability of the stabilized product as a fuel can be demonstrated. The applications: as a hydrogen source for fuel cells, and as a clean transportable, storable fuel for dispersed power, will require testing of the product to confirm its suitability.

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

Activity during this quarter

- Acurex and Air Products have been working to identify a variety of sites and applications for product-use tests. During the 29-30 April 1997 interim review meeting, Air Products presented a status update on these activities to the DOE. A

total of 22 projects have been screened by their likelihood to proceed and the timing for the initial methanol requirement. Eight sites from the list have met these criteria. Appendix C contains a synopsis of all projects screened, and a table summarizing the best eight candidates. At present, full proposals and cost breakdowns are being developed by Acurex and each of the eight possible participants. Due to the timing and quantities of methanol required by the earliest four tests, Air Products and DOE are considering the use of methanol produced from carbon monoxide (CO)-rich syngas feeds from the LaPorte Alternative Fuels Development Unit (AFDU). This will allow for some initial testing to occur during calendar year 1997, when some of these projects will be ready to proceed. The Demonstration Test Plan indicates methanol for the remaining four tests (as-produced from CO-rich syngas) will first be produced in May of 1998. The formal submission of the eight projects for review and approval by the DOE will be made during the next reporting period.

Task 1.5 Planning and Administration

Task 1.5.1 Product-Use Test Plan

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

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

Task 1.5.2 Commercialization Studies

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

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

Discussion

Several areas have been identified as needing development to support specific commercial design studies. These include: a) product purification options; b) front-end impurity

removal options; c) catalyst addition/withdrawal options; and d) plant design configuration options. Plant sizes in the range of 300 TPD to 1,800 TPD and plant design configurations for the range from 20% up to 70% syngas conversion will be considered. The Kingsport demonstration unit design and costs will be the basis for value engineering work to focus on specific cost reduction targets in developing the initial commercial plant designs.

The Process Economics Study - Outline has been prepared to provide guidance for the overall study work. The four part Outline is included in Appendix D. This Outline addresses several needs for this Task 1.5.2 Commercialization Study:

- a) to provide process design guidance for commercial plant designs.
- b) to meet the Cooperative Agreement's technical objectives requirement for comparison with gas phase methanol technology. This preliminary assessment will help set demonstration operating goals, and identify the important market opportunities for the liquid phase technology.
- c) to provide input to the Demonstration Test Plan (Task 2.3).
- d) to provide input to the Off-Site Testing (Task 1.4) product-use test plan update.

Activities during this quarter

- Part One of the Outline - "Coproduction of Methanol" has been written for release as a Topical Report. Comments from DOE on the 31 March 1997 draft of the Topical Report "Economic Analysis - LPMEOH™ Process as an Add-on to IGCC for Coproduction" were received during the reporting period. This Topical Report develops plant design options for the LPMEOH™ process, as an add-on to IGCC power plants for the coproduction of methanol and power. Part One also compares the LPMEOH™ (LP) process with gas phase (GP) methanol processes in the environment of coal-derived syngas. Surprisingly, the LP technology can coproduce methanol at less than 50 cents per gallon, even at relatively small (400 to 1200 TPD) methanol plant sizes. LP's advantage over GP is 6 to 9 cents per gallon. Therefore, when baseload IGCC power is viable, the LP technology makes coproduction viable. An update of this draft Topical Report is expected to be released for comment in September 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 product-use test plan (Task 1.4).

Task 1.5.3 DME Design Verification Testing

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

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

Discussion

The first decision milestone, on whether to continue with DME DVT, was targeted for 01 December 1996. This milestone was relaxed to July of 1997 to allow time for further development of the LPDME catalyst system. DVT is required to provide additional data for engineering design and demonstration decision-making. The essential steps required for decision-making are: a) confirm catalyst activity and stability in the laboratory, b) develop engineering data in the laboratory, and c) confirm market(s), including fuels and chemical feedstocks. The DME Milestone Plan, showing the DVT work and the decision and implementation timing, is included in Appendix E.

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

DME DVT Recommendation

Air Products made a recommendation to continue with the design verification testing to coproduce DME with methanol, and to proceed with planning a proof-of-concept test run at the DOE's AFDU in LaPorte, Texas. A copy of the recommendation (dated 30 June 1997) is included in Appendix E. The recommendation was based on the results of the Market Economic Studies and on the LPDME catalyst system R&D work, and is summarized in the following.

The Market Economic Studies show that the LPDME process should have a significant economic advantage for the coproduction of DME with methanol for local markets. The studies show that the market applications for DME are large. DME is an ultra clean diesel fuel; and an 80% DME mixture with methanol and water is now being developed and tested by others. DME is a key intermediate in a commercial syngas-to-gasoline process, and is being developed as an intermediate for other chemicals and fuels. An LPDME catalyst system with reasonable long-term activity and stability has been developed from the Laboratory R&D work. The markets and this catalyst system is sufficiently promising that proof-of-concept planning for the LaPorte AFDU is recommended. A summary of the DME DVT recommendation is:

- Planning for a DME test run at the LaPorte AFDU, in conjunction with other DOE Liquid Fuels Programs, should be initiated. Test plans, budgets, and a schedule for these LaPorte AFDU tests should now be developed. Up to \$875,000 of Clean Coal Technology Program budget support from the LPMEOH™ Project budget could be made available to support a suitable LPDME test run at LaPorte.
- An implementation decision, made mutually by the DOE's Clean Coal Technology Program (DE-FC22-92PC90543) LPMEOH™ project participants, and by the DOE's

Indirect Liquefaction Program (DE-FC22-95PC93052) project participants, should be made in time to implement testing at LaPorte.

The recommendation to continue design verification testing to coproduce DME with methanol at the LaPorte AFDU is now under consideration. LPDME is not applicable to hydrogen (H₂)-rich syngas; and it is unlikely that a substantive LPDME demonstration will be recommended for Kingsport. Therefore, a convincing case that the test-run on CO-rich syngas at LaPorte will lead to successful commercialization must be made, prior to approving the final test-run plan. The strategy for commercialization must present the technical logic to combine the results of the following two areas:

- 1) catalyst performance (productivity, selectivity, and life) for the LPDME catalyst system under CO-rich syngas from the proof-of-concept testing at the LaPorte AFDU; and
- 2) reactor performance (methanol catalyst activity and life, hydrodynamics, and heat transfer) from the LPMEOH™ Process Demonstration Unit

The productivity and life of an "acceptable" LPDME catalyst system must be better defined, and then confirmed in the laboratory. A recommendation document summarizing catalyst targets, experimental results, and the corresponding economics for a commercially successful LPDME catalyst was issued on 30 June 1997.

Market Economic Studies

Work on the feasibility study for the coproduction of DME and methanol with electric power continued. The product DME would be used as a domestic liquid cooking fuel, to replace imported Liquid Petroleum Gas, for the China and Pacific Rim regions. The results to date, are included in the DME recommendation in Appendix E.

Laboratory R&D

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

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

Summary of Laboratory Activity and Results

- Experiments using an alternative methanol catalyst with the AB dehydration catalyst have given the highest productivity seen for a stable catalyst system. A new reduction procedure, one which reflects plant procedure, was also used. No sign of the accelerated long-term catalyst deactivation was observed following 1030 stream hours of testing.
- This new reduction procedure has given good stability in a run at low feed rates on a syngas typically produced by a Shell coal gasifier. This run is part of a matrix of experiments to understand the effects of space velocity and feed gas composition on catalyst stability.
- Air Products has begun discussing scale-up of the production of the AB dehydration catalyst with two catalyst manufacturers. The key technical issue at this point is whether nitridation is (a) commercially feasible and (b) technically desirable in light of recent laboratory successes in improving the stability of non-nitrided material.

Task 1.5.4 Administration and Reporting

The Cooperative Agreement was modified (Modification No. A011 on 08 October 1996), authorizing the transition from Budget Period No. 2 (Design and Construction) to the final Budget Period (Commissioning, Start-up, and Operation). This modification provides the full DOE cost share of \$92,700,000 of authorized funding, with the remaining \$121,000,000 being provided by the participants. A copy of the approval memorandum, dated 03 October 1996, is included in Appendix F.

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

Task 2.1 Procurement

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

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

Task 2.2 Construction

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

- Provide construction management for contractor coordination and compliance with design, construction, and quality control standards.
- Erect the major equipment and structural steel. Install the large bore piping, electrical, and insulation such that instrument check-out and equipment commissioning work can be completed during the 60-day Continuation Application approval period.
- Complete mechanical construction so that check-out and commissioning can be started in Budget Period No. 3.
 - All major construction contract work has been completed. During the reporting period, site paving/grading and the painting of large- and some small-bore piping systems was 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.
 - Task 2.3 Training and Commissioning is complete.

Task 2.4 Off-Site Testing (Procurement and Construction)

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

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

Task 2.5 Planning and Administration

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

- Prepare annually an updated (Partnership) plan for the remaining activities. The first annual plan will update the remaining Phase 1 and Phase 2 activities, and the second will include an update of the Phase 3 Demonstration Test Plan.
 - The first update of the Partnership Annual Operating Plan was prepared and submitted in September of 1995 (See Quarterly Technical Progress Report No. 5). The main goal and objective for this first annual plan was to continue construction so that the 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 G). 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 3.6 (Planning and Administration).

Task 3.1 Start-up

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

Task 3.2 LPMEOH™ Process Demonstration Facility Operation

Task 3.2.1 Methanol Operation

Upon completion of the activation of the nine batches of methanol synthesis catalyst (reported in Technical Progress Report No. 11), the catalyst slurry was transferred from the 29D-02 slurry storage tank to the 29C-01 reactor (refer to Appendix A for the simplified process flow diagram). A portion of the slurry was pumped by the 29G-02 slurry return pump; the remainder was pressure-transferred using nitrogen at 45-50 psig on the slurry storage tank. Heat-up of the catalyst slurry by injecting 600 psig steam into the risers of the internal heat exchanger on the reactor proceeded smoothly. Balanced Gas was introduced to the LPMEOH™ demonstration unit at 0900 hours on 02 April 1997, but several coincidental interruptions in feed gas supply delayed extended, stable operation for several more days. The first stable operation at the nameplate methanol capacity of 80,000 gallons per day (260 TPD) was achieved on 06 April 1997. The Test Authorization for the initial operating campaign at the LPMEOH™ demonstration unit is provided in Appendix H.

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

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

The following discussion of performance results will focus on the distinct operating periods during the quarter and detailed reporting of specific performance parameters.

Initial Operating Period - 02 April - 08 May 1997

The frequent feed gas interruptions continued for several more days, so that the first stable 24-hour material balance period occurred on 12 April 1997. The highest methanol production rate over a 24-hour period occurred on 19 April 1997 (89,900 gallons per day, or

Table 3.2.1-1 - DATA SUMMARY FOR LPMEOH DEMONSTRATION UNIT
April/June 1997 Operating Period

Case	Date	Gas Type	Temp (Deg C)	Pres. (psig)	Balanced			Recycle Gas (KSCFH)	Inlet Sup. Velocity (ft/sec)	Space Velocity (l/hr-kg)	Slurry Conc. (wt% ox)	Gas Holdup (vol%)	Gassed Slurry Hgt (ft)	Catalyst Inventory (lb)	Catalyst Age (eta)	CO Conv. (%)	Reactor O-T-M Conv. (%)	Syngas Util. (SCF/lb)	Raw MeOH Production (TPD)	Catalyst MeOH Prod. (gmol/hr-kg)	Reactor Vol. Prod. (TPD/Cu ft)	Sparger dP (psi)	Sparger Resistance ("K")
					Gas (KSCFH)	CO Gas (KSCFH)	H2 Gas (KSCFH)																
1	12-Apr-97	Balanced	248	719	758	0	0	1,375	0.50	6,203	30.5	50.2	54.9	20,300	1.30	54.8	33.1	37.6	242.3	30.74	0.105	6.23	10.87
1	13-Apr-97	Balanced	249	716	792	0	0	1,536	0.55	6,783	30.8	50.6	54.9	20,300	1.24	52.5	31.7	37.5	253.8	32.19	0.110	8.69	12.72
1	14-Apr-97	Balanced	249	705	789	0	0	1,619	0.58	7,014	30.9	52.0	56.1	20,300	1.18	50.5	30.5	37.5	252.4	32.00	0.107	10.50	14.17
1	15-Apr-97	Balanced	249	705	818	0	0	1,601	0.58	7,019	30.8	50.9	55.1	20,300	1.29	51.7	31.2	37.9	258.9	32.84	0.112	11.05	14.88
1	16-Apr-97	Balanced	248	705	904	0	0	1,527	0.59	7,094	30.9	50.7	54.5	20,300	1.78	55.8	33.6	38.5	281.9	35.82	0.123	14.17	18.78
1	17-Apr-97	Balanced	249	704	887	0	0	1,746	0.63	7,629	32.1	53.0	54.3	20,300	1.24	49.8	30.0	39.4	270.5	34.35	0.119	17.61	20.28
1	18-Apr-97	Balanced	249	705	841	0	0	1,843	0.64	7,762	32.0	51.9	53.4	20,300	1.14	48.2	29.1	37.8	267.0	33.86	0.119	19.17	21.35
1	18-Apr-97	Balanced	249	703	964	0	0	1,779	0.66	7,970	32.0	52.2	53.8	20,300	1.43	51.1	30.7	40.0	288.8	36.80	0.128	20.47	21.69
1	19-Apr-97	Balanced	249	709	958	0	0	1,994	0.70	8,562	32.2	51.5	52.4	20,300	1.17	48.3	29.2	39.4	292.2	36.60	0.133	21.59	19.36
1	20-Apr-97	Balanced	249	708	913	0	0	2,114	0.72	8,771	30.3	50.5	55.8	20,300	1.03	47.1	26.4	39.8	275.5	34.77	0.117	21.64	20.31
1	21-Apr-97	Balanced	249	709	859	0	0	2,108	0.71	8,635	30.2	47.5	53.0	20,300	0.94	47.5	25.6	39.5	261.1	32.83	0.117	22.29	21.90
1	22-Apr-97	Balanced	249	709	793	0	0	2,086	0.69	8,376	29.5	45.2	52.5	20,300	0.91	49.0	24.6	39.2	242.7	30.65	0.110	21.59	23.04
1	24-Apr-97	Balanced	249	702	865	0	0	2,028	0.70	8,419	29.9	43.1	49.5	20,300	0.93	44.6	26.2	39.8	261.0	33.17	0.126	24.25	23.16
1	25-Apr-97	Balanced	248	700	835	0	0	1,921	0.67	8,019	31.8	44.9	47.0	20,300	0.89	44.6	26.2	40.4	248.2	31.58	0.126	23.48	24.80
1	26-Apr-97	Balanced	246	694	864	0	0	1,878	0.67	7,980	32.8	45.6	45.5	20,300	0.83	41.6	26.0	41.9	247.8	31.38	0.130	25.38	26.81
1	27-Apr-97	Balanced	247	690	902	0	0	1,793	0.66	7,842	32.8	44.9	45.0	20,300	0.83	39.7	26.9	42.9	252.2	31.87	0.134	26.18	27.29
1	28-Apr-97	Balanced	247	696	783	0	0	1,903	0.65	7,817	30.9	43.3	47.5	20,300	0.75	42.7	24.4	41.9	224.4	28.35	0.113	24.49	27.11
1	29-Apr-97	Balanced	249	700	810	0	0	1,922	0.66	7,951	29.3	45.2	53.0	20,300	0.81	42.1	25.6	40.9	238.0	30.05	0.107	26.26	26.76
1	30-Apr-97	Balanced	249	699	794	0	0	1,912	0.66	7,872	29.1	43.6	52.0	20,300	0.79	43.0	25.1	41.3	230.8	29.24	0.106	26.13	27.67
1	1-May-97	Balanced	249	699	798	0	0	1,932	0.66	7,945	29.9	44.0	50.5	20,300	0.74	41.1	24.5	42.0	228.0	28.85	0.108	26.26	27.32
1	2-May-97	Balanced	249	700	757	0	0	1,898	0.64	7,728	29.7	43.0	50.0	20,300	0.71	42.8	23.7	42.2	215.4	27.32	0.103	25.91	29.72
1	3-May-97	Balanced	249	699	782	0	0	1,886	0.65	7,767	30.4	43.1	48.5	20,300	0.68	40.5	23.9	42.8	219.5	27.66	0.108	26.05	29.24
1	4-May-97	Balanced	249	700	789	0	0	1,901	0.65	7,831	30.5	44.0	49.0	20,300	0.64	38.9	23.4	43.4	218.1	27.43	0.106	26.24	29.58
1	5-May-97	Balanced	249	699	798	0	0	1,901	0.66	7,856	30.1	43.6	49.5	20,300	0.63	37.5	23.4	43.7	218.9	27.76	0.105	26.26	28.88
1	6-May-97	Balanced	249	700	776	0	0	1,923	0.65	7,835	30.4	42.5	48.5	20,300	0.65	38.1	23.3	42.8	217.3	27.85	0.108	26.26	28.83
22	8-May-97	BGL	249	700	215	40	0	1,051	0.32	3,813	30.8	36.9	43.0	20,300	0.64	8.6	15.1	44.4	69.0	8.83	0.038	14.81	33.22
5	18-Jun-97	Balanced	248	724	718	0	0	1,938	0.62	8062	25.6	45.9	61.4	19,500	0.61	39.3	22.5	42.0	205.3	26.95	0.079	2.99	3.37
5	19-Jun-97	Balanced	249	711	638	0	0	1,938	0.62	7905	26.6	45.3	57.8	19,500	0.63	42.6	21.4	40.7	188.3	24.77	0.077	3.05	3.52
5	20-Jun-97	Balanced	249	707	651	0	0	2,079	0.66	8294	27.1	44.9	56.0	19,500	0.59	39.2	20.2	41.3	189.4	24.99	0.080	3.45	3.71
5	21-Jun-97	Balanced	249	707	687	0	0	2,109	0.67	8465	27.6	45.2	55.0	19,500	0.58	35.5	20.8	41.4	198.9	26.14	0.086	4.00	3.90
5	22-Jun-97	Balanced	249	707	625	0	0	2,097	0.65	8203	28.2	44.2	52.7	19,500	0.50	33.4	19.5	41.3	181.5	23.85	0.082	3.96	4.11
5	23-Jun-97	Balanced	249	707	762	0	0	2,021	0.67	8456	28.4	43.6	51.6	19,500	0.57	33.6	21.5	43.8	208.8	27.35	0.096	4.51	4.38
5	24-Jun-97	Balanced	249	708	781	0	0	1,991	0.67	8412	29.0	44.5	50.9	19,500	0.56	33.2	21.2	45.6	205.4	26.90	0.096	5.04	4.93
5	25-Jun-97	Balanced	248	707	739	0	0	2,003	0.66	8338	29.2	43.3	49.4	19,500	0.57	33.7	21.0	44.0	201.5	26.62	0.097	5.33	5.31
5	26-Jun-97	Balanced	249	707	737	0	0	2,080	0.68	8559	28.9	48.3	54.8	19,500	0.55	33.0	20.7	43.4	203.5	26.65	0.088	5.64	5.38
5	27-Jun-97	Balanced	249	706	736	0	0	2,326	0.74	9252	26.7	46.6	59.0	19,500	0.53	29.7	19.5	42.8	206.5	26.68	0.083	7.34	5.74
5	28-Jun-97	Balanced	249	707	691	0	0	2,307	0.72	9079	27.5	45.8	56.0	19,500	0.53	31.0	19.3	41.5	199.6	25.84	0.085	8.12	6.67
5	29-Jun-97	Balanced	249	706	719	0	0	2,267	0.72	9042	27.7	43.9	53.5	19,500	0.53	30.0	19.5	42.6	202.3	26.24	0.090	9.33	7.65
5	30-Jun-97	Balanced	249	706	711	0	0	2,263	0.72	9019	28.1	43.9	52.5	19,500	0.51	29.9	19.1	43.0	198.4	25.77	0.090	9.29	7.90

292.2 TPD); for shorter balance periods (approximately 12 hours), methanol production rates of 92,900 to 94,500 gallons per day (302 to 307 TPD) were measured.

During the first days of operation, several strainers in the reactor loop became blocked with debris remaining in the piping systems from construction and hydrotesting. Outages were taken to clean screens at the inlet to the 29C-40 carbonyl guard bed and the 29C-03 high-pressure methanol separator. The carbonyl guard bed was bypassed from 04 April 1997 until 18 April 1997; the decision to bypass the carbonyl guard bed was based upon the results of the carbonyl survey completed in March (as reported in Technical Progress Report No. 11) and an autoclave test performed at the Kingsport site in May/June 1996. A draft Topical Report has been issued on that study (Design and Construction of the Alternative Fuels Field Test Unit and Liquid Phase Methanol Feedstock and Catalyst Life Testing at Eastman Chemical Company (Kingsport, TN)).

As noted in Technical Progress Report No. 11, the 29G-03 oil make-up pumps were unable to deliver fresh oil to the reactor loop at the required pressure of approximately 700 psig. These pumps also provide the required high pressure seal flush to the 29G-01 condensed oil circulation pumps, which return oil and catalyst collected in the 29C-06 cyclone and the 29C-05 secondary oil knock-out drum to the reactor (refer to Appendix A for the simplified process flow diagram). One of the features included in the design of the LPMEOH™ Demonstration Unit was the capability to free-drain condensed and entrained oil and catalyst slurry back to the reactor. Furthermore, fresh make-up oil could be added to the process by using the 29G-30 slurry transfer pump, which was designed to transfer catalyst slurry from the 29C-30 catalyst reduction vessel to the reactor. Oil was batch-transferred from the 29D-30 oil storage tank to the catalyst reduction vessel, and then pumped to the reactor by the slurry transfer pump. The slurry transfer pump has packing which also requires flush from the oil make-up pumps; however, it was determined that operation of the slurry transfer pump in services with clean oil or low solids concentration would not adversely affect the service life of the pump.

The free-drain line showed intermittent plugging or vapor-locking during operation. Early in the operating campaign, blockages could be cleared by opening a transfer line between the secondary oil knock-out drum and catalyst reduction vessel and briefly blowing down to low pressure; piping connections to provide flush oil were rendered useless by the inoperable oil make-up pumps. However, on 25 April 1997, a blockage in the free-drain line occurred in a location which could not be removed by this method. Since the slurry concentration of the entrained oil and catalyst was relatively low, it was determined that the slurry transfer pump could pump this material without packing flush on the pump. Condensed oil was batch-transferred from the secondary oil knock-out drum to the catalyst reduction vessel, and then pumped to the reactor. The frequency of the transfer to the catalyst reduction vessel was about every 3 hours, and the catalyst reduction vessel was pumped to the reactor about every 10 hours. The rate of accumulation of entrained/condensed slurry (1.5 to 2.0 gallons per minute) matched the expected liquid traffic within the oil/catalyst collection equipment.

A two-day test using a CO-rich reactor feed ($H_2/CO = 0.43$) was performed on 07-08 May 1997. The Test Authorization for this trial is included in Appendix K. At the conclusion of this test, the LPMEOH™ Demonstration Unit was shut down in preparation for a biannual

outage at the Eastman coal-to-chemicals facility. Catalyst slurry was pressure-transferred from the reactor to the slurry storage tank for storage under a reducing atmosphere during the outage.

Throughout this initial operating period, pressure-drop measurements across the gas sparger at the bottom of the reactor showed a steady increase during normal operation. Pressure drop can be expressed in the following equation:

$$\Delta P = \frac{K * (V * MW)^2}{\rho}$$

where:

- ΔP = pressure drop across sparger, pounds per square inch
- K = sparger resistance coefficient
- V = vapor volumetric flowrate, thousand standard cubic feet per hour
- MW = vapor molecular weight, pounds per pound mole
- ρ = vapor density, pounds per cubic foot

This equation shows that pressure drop readings can be influenced by changes in gas flowrate and/or gas composition. The resistance coefficient (K) can be used to determine any change in the vapor flow path through the gas sparger. For a given vapor volumetric flowrate and density, an increase in K (caused by a restriction in the flow path, for example), will result in an increase in pressure drop.

Appendix I, Figure 1 plots K over time since the start-up of the LPMEOH™ Demonstration Unit. (Note that K as reported contains an arbitrary factor to make the value more manageable, and therefore has meaning only in a relative sense.) The data for this plot, along with the corresponding pressure drop measurement, are included in Table 3.2.1-1. Pressure drop and resistance increased with time on stream, and extended periods with no vapor flow through the gas sparger (noted on Figure 1) appear to have no impact on this trend.

Maintenance Activities During May/June 1997 Complex Outage

Most of the activities in the LPMEOH™ Demonstration Unit during the complex outage focused on the inspection of equipment associated with the reactor, particularly the gas sparger. About 800 pounds of residual catalyst was removed from the bottom head of the reactor during this exercise. A solid material (presumably methanol synthesis catalyst) appeared to block about 50% of the flow path through the sparger; a small amount of catalyst was found in the inlet piping to the sparger. There was no discernible pattern to the blockage by the catalyst, and no significant construction debris was found in the inlet piping or in the sparger. The sparger was removed from the reactor and cleaned. The only modifications to the sparger itself were changes to increase the maximum allowable pressure drop; no change to the flow distribution characteristics was made.

Another effect of the commissioning problems associated with the oil make-up pumps is the loss of oil flush provided by the condensed oil circulation pumps to the walls of the cyclone. At the LaPorte AFDU, liquid flush to the cyclone improved the efficiency of solids removal. During the complex outage, the inlet to the tubesheet of the 29E-02 feed/product heat

exchanger (immediately downstream of the cyclone) was removed to check for catalyst accumulation. The tubesheet was generally clean except for a small, off-center accumulation on the upper left quadrant. The catalyst slightly obstructed the entrance to these tubes, but did not completely block any tube. No catalyst was visible within any of the tubes. The surface catalyst was removed, and the feed/product heat exchanger was reassembled.

During the initial operating period, the blockage in the free-drain line provided evidence that the ability to flush piping systems in slurry service was an important operability requirement. Since a replacement for the oil make-up pumps had not yet been identified, the slurry transfer pump was connected into the flush piping system originally designed to be supplied by the oil make-up pumps. A flush connection was also added to the gas inlet line to the reactor; this could be used to flush out the piping and gas sparger during normal operation, at those times when gas flow to the reactor is lost, or in preparation for maintenance.

Other maintenance activities focused on repair of minor leaks in the steam system.

Unit Restart and Operation - 17-30 June 1997

After the catalyst slurry was pressure-transferred from the slurry storage tank to the reactor, the reactor was heated using 600 psig steam in the same manner as the April start-up. Balanced Gas was introduced to the LPMEOH™ Demonstration Unit at 1400 hours on 17 June 1997. Operation of the facility has continued uninterrupted since the restart. The free-drain piping from the secondary oil knock-out drum and cyclone to the reactor plugged again shortly after restart, but flush oil from the slurry transfer pump successfully dislodged the blockage.

Again, the gas sparger has shown an increase in pressure drop and resistance since the restart, although not as rapidly as during the April-May operation. The plot of sparger resistance coefficient with time for both operating periods is provided in Appendix I, Figure 1. The value for the resistance coefficient is lower for the latest start-up of the reactor; this may be a result of additional attention to maintaining vapor flow through the sparger during the slurry transfer operation. On 26 June 1997, fresh oil from the slurry transfer pump was introduced for the first time to the new flush connection on the gas inlet line to the reactor; the flush lowered the pressure drop from 5.5 psi to 4.5 psi. However, the effects were temporary, and the resistance coefficient continued to increase. Additional flushing with both fresh oil and entrained slurry will be attempted in order to stabilize the resistance coefficient. Fresh oil can only be added to the process at an average of 0.1 - 0.2 gallons per minute to match the rate of oil loss with the methanol product; entrained slurry can be supplied at the rate of liquid traffic in the secondary oil knock-out drum and cyclone (1.5 to 2.0 gallons per minute).

Catalyst Life (η)

The activity of the methanol synthesis catalyst can be expressed in terms of a dimensionless variable η , which is defined as the ratio of the rate constant at any point in time to the rate constant for a freshly reduced catalyst (as determined in the laboratory autoclave). Appendix I, Figure 2 contains the plot for η versus days onstream since the start-up in April of 1997; shutdowns of the LPMEOH™ Demonstration Unit are indicated and match the

longer interruptions in operation from Appendix I, Table 1. During the April/May 1997 operating period, the evidence was unclear whether the decline in η was a result of a decline in catalyst activity or hydrodynamic effects related to the increase in resistance coefficient for the gas sparger. Upon restarting the LPMEOH™ Demonstration Unit in June of 1997, the value of η was determined to be unaffected by the magnitude of the sparger resistance coefficient. It appears that catalyst activity is declining more rapidly than expected.

A catalyst slurry sample was taken during the May/June 1997 complex outage. Due to a change in procedures for handling reduced catalyst in the laboratory, analysis of this sample for copper crystallite size, surface area, and the presence of catalyst poisons will not be performed until July of 1997.

Overall, the LPMEOH™ Demonstration Unit operated well during the initial campaign. The availability of the LPMEOH™ Demonstration Unit was 94.9% during the reporting period. All methanol produced (a total of 2,900,692 gallons) was used by Eastman in the production of methyl acetate, and ultimately cellulose acetate and acetic acid. The start-up was successfully completed in a safe and environmentally sound manner.

Methods of Calculation

As described in Section 6.2 of the Demonstration Test Plan, a comprehensive set of the formulas used to calculate key performance parameters of the LPMEOH™ Process was to be included in the first Technical Progress Report for Task 3.2.1 - Methanol Operation. These calculations are provided in Appendix L.

Task 3.2.2 DME Design, Modification and Operation

No activities occurred in this Task during the reporting period.

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

No activities occurred in this Task during the reporting period.

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

No activities occurred in this Task during the reporting period.

Task 3.5 Data Analysis and Reports

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

Task 3.6 Planning and Administration

An interim project review meeting was held on 29 and 30 April 1997 in Allentown. Attendees from Air Products and DOE participated. An update on the performance of the demonstration unit was provided. The catalyst targets and corresponding economics for a commercially successful LPDME catalyst were reviewed; these and other comments from DOE were incorporated into the DME recommendation (issued 30 June 1997). The status of the updated product-use test plan was also discussed. The meeting agenda, extracts from the meeting handouts, and the meeting notes are included in Appendix M.

The Milestone Schedule Status Report and the Cost Management Report, through the period ending 30 June 1997, are included in Appendix N. 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-eight percent (98%) of the \$38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 30 June 1997. Five percent (5%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 June 1997.

Start-up activities were completed during the reporting period, and the initial methanol production from the demonstration unit occurred on 02 April 1997. The first extended stable operation at the nameplate capacity of 80,000 gallons per day (260 TPD) took place on 06 April 1997. The demonstration unit was shut down on 08 May 1997 as part of a scheduled complex outage for the Kingsport site. After completion of maintenance activities, the demonstration unit was restarted on 17 June 1997, and maintained stable operation through the remainder of the reporting period. Details of the operating activities are provided under Task 3.2 of this report.

Preparations for the plant dedication ceremony, scheduled for 25 July 1997, began in earnest. Participants are expected to include senior management from Air Products, Eastman, and DOE.

A press release on the start-up of the LPMEOH™ Demonstration Facility was issued on 21 May 1997. A copy of the press release, as well as a sample of other publications which reported on the start-up of the demonstration unit, are included in Appendix O.

An update of the Project Management Plan was submitted to DOE on 30 June 1997. This version summarizes the reporting structure during Tasks 1 and 2, and lists the current team members for Air Products, Eastman, and Acurex.

The monthly reports for April, May, and June were submitted. These reports include the Milestone Schedule Status Report, the Project Summary Report, and the Cost Management Report. All Quarterly Technical Progress Reports through 31 March 1997 have been approved by DOE. DOE and Air Products agreed to delay the publication of the Demonstration Technology Start-up Report until issues related to the oil make-up pump and the reactor sparger have been resolved (refer to Task 3.2 for the status of these items).

E. Planned Activities for the Next Quarter

- Resolve any issues associated with the gas sparger in the reactor and with the oil make-up pumps. Upon resolution of these items, write and submit the Demonstration Technology Start-up Report to DOE.
- Analyze catalyst slurry sample taken during May/June 1997 complex outage to determine causes for deactivation of methanol synthesis catalyst.
- Continue executing Phase 3, Task 2.1 Methanol Operation per the Demonstration Test Plan.
- Receive concurrence from DOE on the DVT Recommendation for a DME proof-of-concept test run at the LaPorte AFDU.
- Receive concurrence from DOE on the Off-Site, Product-Use Test Plan (Phase 1, Task 1.4).
- Hold a Project Review Meeting in Kingsport in July, in conjunction with the 25 July dedication ceremony.
- Incorporate DOE comments into the Topical Report on Process Economic Studies.

F. Conclusion

During this quarter, comments from the DOE on the Topical Report "Economic Analysis - LPMEOH™ Process as an Add-on to IGCC for Coproduction" were received. The study concludes that methanol coproduction, with IGCC electric power utilizing the LPMEOH™ process technology, will be competitive in serving local market needs.

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. A recommendation document summarizing catalyst targets, experimental results, and the corresponding economics for a commercially successful LPDME catalyst was issued on 30 June 1997.

The off-site, product-use test plan was updated in June of 1997. During this quarter, Acurex and Air Products screened proposals for this task by the likelihood of the projects to proceed and the timing for the initial methanol requirement. Eight sites from the list have met these criteria. The formal submission of the eight projects for review and concurrence by the DOE will be made during the next reporting period.

An interim project review meeting was held in Allentown in late April of 1997. An update on the performance of the demonstration unit was provided, and the status of the DME recommendation and the off-site product-use test plan were discussed.

The site paving and final painting were completed in May of 1997. Start-up activities were completed during the reporting period, and the initial methanol production from the demonstration unit occurred on 02 April 1997. The first extended stable operation at the nameplate capacity of 80,000 gallons per day (260 TPD) took place on 06 April 1997. Pressure drop and resistance coefficient across the gas sparger at the bottom of the reactor increased over this initial operating period. The demonstration unit was shut down on 08 May 1997 as part of a scheduled complex outage for the Kingsport site. During this outage, the gas sparger was removed, cleaned, and reinstalled. After completion of other maintenance activities, the demonstration unit was restarted on 17 June 1997, and maintained stable operation through the remainder of the reporting period. Again, the gas sparger showed an increase in pressure drop and resistance since the restart, although not as rapidly as during the April-May operation. Fresh oil was introduced for the first time to a new flush connection on the vapor inlet line to the reactor; the flush lowered the pressure drop by 1 psi. However, the effects were temporary, and the sparger resistance coefficient continued to increase. Additional flushing with both fresh oil and entrained slurry will be attempted in order to stabilize the sparger resistance coefficient.

Catalyst activity, as defined by the ratio of the rate constant at any point in time to the rate constant for a freshly reduced catalyst (as determined in the laboratory autoclave), declined more rapidly than expected. A catalyst slurry sample was taken during the May/June 1997 complex outage for analysis.

Overall, the LPMEOHTM Demonstration Unit operated well during the initial campaign. The availability of the LPMEOHTM Demonstration Unit was 94.9% during the reporting period. All methanol produced (a total of 2,900,692 gallons) was used by Eastman in the production of methyl acetate, and ultimately cellulose acetate and acetic acid. The start-up was successfully completed in a safe and environmentally sound manner.

Ninety-eight percent (98%) 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 30 June 1997. Five percent (5%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 30 June 1997.

APPENDICES

APPENDIX A - SIMPLIFIED PROCESS FLOW DIAGRAM

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

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

Synopsis of All Proposals (twenty pages)

and

Summary Table of Eight Candidates (one page)

APPENDIX D - TASK 1.5.2 - PROCESS ECONOMIC STUDY

**Process Economics Study - Outline
(Draft - 3/31/97 - four pages)**

and

**LPMEOH™ Process Economics - for IGCC Coproduction
(Memo - 31 March 1997 - two pages)**

APPENDIX E - TASK 1.5.3 - DME DESIGN VERIFICATION TESTING

APPENDIX F - TASK 1.5.4 - APPROVAL FOR BUDGET PERIOD THREE

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

**APPENDIX H - TEST AUTHORIZATION #K1 - INITIAL SHAKEDOWN AND
DESIGN PRODUCTION TESTS**

TEST AUTHORIZATION # K1

Kingsport LPMEOH™ Plant

Sheet: 1 of 3
Date : 03/31/97
By: VES

RUN NUMBER: K1 (also incorporating K7)
APPROX. START DATE: 1 April, 1997

TITLE: METHANOL SYNTHESIS WITH BASELINE CATALYST:
INITIAL SHAKEDOWN AND DESIGN PRODUCTION TESTS

OBJECTIVE:

To start-up the LPMEOH™ facility, test the design methanol production rate, and shakedown various systems critical to successful long-term operation of the plant.

SUMMARY:

After activation of the initial catalyst charge (approximately 20,000 lbs of catalyst oxide, or about 1/2 of the design catalyst loading in the reactor), the start-up of methanol operations will initiate a total system shakedown and test of the design methanol production rate of 260 TPD. This 6-week operating period prior to Eastman's complex outage will facilitate testing of systems which must operate on a continuous basis (e.g. recycle compression, carbonyl guard bed, reactor/steam system, oil collection and return, methanol collection and distillation, data acquisition, analytical, etc.). The process control strategy will be validated and tuned, and material balance calculations will be performed. During this test, catalyst activity will decrease slowly due to normal catalyst aging. Fresh Feed flowrates will be adjusted to maintain Syngas Utilization at its initial value.

TEST DETAILS: See pages 2 to 3 for details.

ANALYTICAL COMMENTS: See page 3.

SAFETY IMPLICATIONS:

Air Products personnel will be required to wear Nomex in the plant when syngas is present. Otherwise, Eastman safety rules (including M.O.C.) are in effect. All visitors to the facility must follow the Visitor Safety Guidelines issued by the Joint Venture.

ENVIRONMENTAL IMPLICATIONS:

Minimal. The plant syngas purge will go to the Eastman boilers as designed.

SPECIAL REMARKS:

Because of ongoing problems with the 29G-03 Oil Make-up Pumps, which provide high pressure seal flush to the 29G-01 Condensed Oil Circulation Pumps and batch make-up oil into the reactor loop, Test #K1 will also incorporate Test #K7. The objective of Test #K7 is to test the capability of the system to free-drain condensed and entrained oil/catalyst from the 29C-06 Cyclone and 29C-05 Secondary Oil K.O. Separator back to the reactor. During this period the 29G-30 Slurry Transfer Pump will provide make-up oil to the process in batches as necessary. It requires only low pressure packing flush oil, which the 29G-03 can provide.

AUTHORIZATIONS:

E. C. Heydorn - Program Manager

V. E. Stein - Lead Process Engineer

TEST AUTHORIZATION # K1

Kingsport LPMEOH™ Plant

Sheet: 2 of 3
Date : 03/31/97
By: VES

TEST DETAILS:

1. Set up N2 purges to the vent header at 100 SCFH on each of the rotameters by the 29E-01 (FI-1970) and 29C-02 (FI-1115).
2. Follow the Reactor Area Start-Up Procedure S.O.P. Section II A 3. After the start-up preparation steps (A-C), continue with Step D.
 - D. Charge fresh oil from 29D-30 to 29C-05 secondary oil K.O. vessel and 29C-06 cyclone.
 - E. Place 29K-01 syngas recycle compressor in service.
 - F. Transfer reduced catalyst slurry from 29D-02 slurry tank to the reactor (via 29G-02) per Section IV A 9 Steps K, L, M, and N. **The slurry temperature must not exceed ambient temperature by more than 165 °C.**
 - G. Start N2 Flow from 29K-01 to reactor.
 - H. Start BFW to 29C-02 steam drum and reactor tubes.
 - I. Start CW flow to 29E-04 MeOH product CW condenser.
 - J. Start fans on 29E-03 MeOH product air-cooled condenser.
 - K. Heat reactor to 204 °C at <30 °C/hr. **Initially BFW / steam temperature should not exceed slurry temperature by more than 150 °C. Once the slurry temperature exceeds 125 °C, BFW / steam temperature should not exceed slurry temperature by more than 40 °C.**
3. Because condensed and entrained oil/catalyst will free-drain back to the reactor, omit Steps L-O. Instead, ensure that automatic valves HV-184 and HV-185 are both shut.
4. Continue with Step P.
 - P. Start fresh feed syngas to plant.
 - Q. Establish level control for 29C-03 high pressure MeOH separator.
5. At Step R, raise the reactor pressure (PIC-150) and temperature (TIC-109) to the design operating conditions: 735 psig and 250 °C. Set the syngas flow rate (FIC-009) at 990 KSCFH, and skip Steps S and T until Plant 19 lines out at reduced rates and the H2 Makeup composition reaches its new steady state. Eventually, new feed setpoints will be calculated for the CO and H2 Makeup streams. Then, FIC-009 will be reduced by that combined flow rate to maintain total fresh feed at 990 KSCFH. Set the compressor flow (FIC-008) at 1,760 KSCFH.
6. During the first 24 hours, the syngas conversion across the reactor may decrease as the catalyst loses its initial hyperactivity. As a result, the purge flow (FI-157) may increase. Eventually, the purge rate should be about 160 KSCFH.
7. To free-drain condensed and entrained oil/catalyst from 29C-05 and 29C-06 to the reactor, open HV-185 and the necessary manual valves. Monitor levels in the 29C-05 (LI-102) and 29C-06 (LI-152), as well as the reactor NDG.

TEST AUTHORIZATION # K1

Kingsport LPMEOH™ Plant

Sheet: 3 of 3
Date : 03/31/97
By: VES

8. Until the 29G-03 pumps are repaired, oil will be batch transferred into the system as needed by the 29G-30 pump per S.O.P. Section II C 3 Step P.
9. The shakedown period will likely conclude with the Eastman complex outage in mid-May. In that event, purge, cool, and drain the reactor system according to the Reactor Area Extended Shutdown Procedure (S.O.P. Section II A 8).

TEST AUTHORIZATION #K1 is complete.

ANALYTICAL REQUIREMENTS:

1. Process GC sampling requirements:
 - SP-1: syngas feed;
 - SP-4: K-01 outlet;
 - SP-5: reactor feed (highest frequency);
 - SP-6: C-05 outlet (highest frequency);
 - SP-7: main purge;
 - SP-8: distillation purge;

 - SP-2 and SP-3 can remain valved out until required.
2. Carbonyl GC sampling requirements:
 - SP-12: 29C-40 guard bed inlet;
 - SP-13: 29C-40 guard bed intermediate #1;
 - SP-14: 29C-40 guard bed intermediate #2;
 - SP-15: 29C-40 guard bed outlet.
3. Liquid sampling requirements:
 - all identified liquid sampling points per standard Eastman routine.

**APPENDIX I - TASK 3.2.1 - RESULTS OF DEMONSTRATION PLANT
OPERATION**

**Table 1 - Summary of LPMEOH™ Demonstration Unit Outages -
April/June 1997**

**Figure 1 - Sparger Resistance Coefficient vs. Days Onstream -
April/June 1997 Operating Period**

Figure 2 - Catalyst Life (η) vs. Days Onstream

Table 1 - Summary of LPMEOH™ Demonstration Unit Outages - April/June 1997

Operation Start	Operation End	Operating Hours	Shutdown Hours	Reason for Shutdown
4/2/97 09:00	4/2/97 16:15	7.3	4.8	Syngas Unavailable to LPMEOH™ Demonstration Unit
4/2/97 21:05	4/2/97 21:25	0.3	23.3	Liquids to K-01
4/3/97 20:40	4/4/97 11:00	14.3	24.8	Syngas Unavailable to LPMEOH™ Demonstration Unit
4/5/97 11:45	4/6/97 01:45	13.0	5.8	C-03 Outlet Plugged
4/6/97 07:30	4/7/97 13:05	29.6	2.1	C-03 Outlet Plugged
4/7/97 15:10	4/8/97 06:30	15.3	21.5	Syngas Unavailable to LPMEOH™ Demonstration Unit
4/9/97 04:00	4/9/97 05:30	1.5	4.0	ESD on C-02 Level
4/9/97 09:30	4/9/97 14:20	4.8	9.7	Syngas Unavailable to LPMEOH™ Demonstration Unit
4/10/97 00:00	4/11/97 08:25	32.4	14.8	Syngas Unavailable to LPMEOH™ Demonstration Unit
4/11/97 23:15	4/18/97 18:05	162.8	0.7	Syngas Unavailable to LPMEOH™ Demonstration Unit
4/18/97 18:45	4/19/97 07:50	13.1	0.7	Syngas Unavailable to LPMEOH™ Demonstration Unit
4/19/97 08:30	4/23/97 00:20	87.8	20.7	Replace TV-101 Trim
4/23/97 21:00	4/23/97 21:00	0.0	12.0	* Syngas Unavailable to LPMEOH™ Demonstration Unit
4/24/97 09:00	5/8/97 23:59	351.0	950.1	** Syngas Unavailable to LPMEOH™ Demonstration Unit
6/17/97 14:05	6/30/97 23:59	321.9		End of Reporting Period
Total Operating Hours			1055.2	
Syngas Available Hours			1111.0	
Plant Availability, %			95.0	

* Plant was ready to startup, but Eastman waited 12 hours to give the day crew training on startup procedures.

** Eastman complex outage.

**APPENDIX J - TASK 3.2.1 - SAMPLES OF DETAILED MATERIAL BALANCE
REPORTS**

**APPENDIX K - TEST AUTHORIZATION K22 - METHANOL SYNTHESIS WITH
BGL-TYPE SYNGAS**

TEST AUTHORIZATION # K22

Kingsport LPMEOH™ Plant

Sheet: 1 of 2
Date : 05/07/97
By: VES

RUN NUMBER: K22
APPROX. START DATE: 7 May, 1997

TITLE: METHANOL SYNTHESIS WITH BGL-TYPE SYNGAS

OBJECTIVE:

To evaluate the performance of the LPMEOH™ facility when fed with BGL-type (CO-rich) syngas per a typical IGCC application.

SUMMARY:

At a time when approximately 200 KSCFH of CO is available for use in Plant 29 over a multi-week period, the reactor feed composition will be adjusted to match the typical syngas composition exiting a BGL gasifier. The performance data from this test will be more directly comparable to the extensive database from the LaPorte pilot plant and more indicative of LPMEOH™'s expected market in IGCC facilities. The test will best be accomplished by maintaining essentially constant feed gas composition (SP-5), and the operators may adjust the Balanced Gas, CO Gas, and Recycle flow rates to achieve that. Liquid samples from the 29C-10 underflow should be collected periodically to test the stabilized product's suitability for fuel-grade applications.

TEST DETAILS: See page 2.

ANALYTICAL COMMENTS: See page 2.

SAFETY IMPLICATIONS:

Air Products personnel will be required to wear Nomex in the plant when syngas is present. Otherwise, Eastman safety rules (including M.O.C.) are in effect. All visitors to the facility must follow the Visitor Safety Guidelines issued by the Joint Venture.

ENVIRONMENTAL IMPLICATIONS:

Minimal. The plant syngas purge will go to the Eastman boilers as designed.

SPECIAL REMARKS:

Because of ongoing problems with plugging in the free-draining oil return line to the reactor, the current off-design operating mode will be continued. Condensed oil will be batch transferred from 29C-05 to 29C-30 and then returned to the reactor with the 29G-30 pump.

AUTHORIZATIONS:

E. C. Heydorn - Program Manager

V. E. Stein - Lead Process Engineer

TEST AUTHORIZATION # K22

Kingsport LPMEOH™ Plant

Sheet: 2 of 2
Date : 05/07/97
By: VES

TEST DETAILS:

1. Call the PIA's and have them set up SP-2 (CO Makeup) and add it to the analysis sequence.
2. Follow the Reactor Area Start-Up Procedure S.O.P. Section II A 3 Step T - start CO makeup feed.
3. Line up CO flow through FE-010B (open valve 2025) and shut off flow through FE-010A (close valve 2026). Make sure the Honeywell DCS is set up to recognize FE-010B.
4. Slowly increase the flow on FC-010 to approximately 200 KSCFH while decreasing the flow on FC-009 to approximately 485 KSCFH. As the MW of the recycle stream begins to rise, the compressor flow will increase. Adjust it to maintain approximately 1570 KSCFH on FI-100.
5. The target feed gas composition (SP-5) in mol% (Honeywell schematic AnalD or AnalB) is: 32% H₂, 61% CO, 5% CO₂, 1% N₂. In wt% (Honeywell schematic Anal_D or Anal_B), this corresponds to 3% H₂, 83% CO, 11% CO₂, 2% N₂. Be patient when adjusting flows to match the target composition; Air Products personnel will advise.

TEST AUTHORIZATION #K22 is complete.

ANALYTICAL REQUIREMENTS:

1. Process GC sampling requirements:
 - SP-1: syngas feed;
 - SP-2: CO makeup
 - SP-4: K-01 outlet;
 - SP-5: reactor feed (highest frequency);
 - SP-6: C-05 outlet (highest frequency);
 - SP-7: main purge;
 - SP-8: distillation purge;

 - SP-3 can remain valved out.
2. Carbonyl GC sampling requirements:
 - SP-12: 29C-40 guard bed inlet;
 - SP-13: 29C-40 guard bed intermediate #1;
 - SP-14: 29C-40 guard bed intermediate #2;
 - SP-15: 29C-40 guard bed outlet.
3. Liquid sampling requirements:
 - all identified liquid sampling points per standard Eastman routine;

TEST AUTHORIZATION # K22

Kingsport LPMEOH™ Plant

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Date : 05/07/97
By: VES

- 29C-10 underflow samples may be shipped to Allentown for by-product analysis.

**APPENDIX L - METHODS OF CALCULATION FOR KEY PROCESS
PARAMETERS**

$$\text{Catalyst Age } (\eta) = k_0/k_0(t=0)$$

where:

k_0 = the pre-exponential kinetic rate constant for the methanol synthesis reaction at any time

$k_0(t=0)$ = the pre-exponential kinetic rate constant for the methanol synthesis reaction for fresh catalyst

The rate constants are determined using a proprietary model which computes the kinetic rate constant for the methanol synthesis reaction.

$$\text{CO Conversion to Methanol [\%]} =$$

$$\frac{(\text{Raw Methanol Flow} + \text{Methanol in Main Plant Purge} + \text{Methanol in Distillation Purge [all lbmol/hr]})}{\text{Carbon Monoxide in Reactor Feed [lbmol/hr]} * 0.01}$$

where:

$$\text{Methanol in Main Plant Purge [lbmol/hr]} = \text{Main Plant Purge Flow [lbmol/hr]} * \text{Methanol Concentration in Main Plant Purge [mol\%]}$$

$$\text{Methanol in Distillation Purge [lbmol/hr]} = \text{Distillation Purge Flow [lbmol/hr]} * \text{Methanol Concentration in Distillation Purge [mol\%]}$$

$$\text{Carbon Monoxide in Reactor Feed [lbmol/hr]} = \text{Reactor Feed Flow [lbmol/hr]} * \text{Carbon Monoxide Concentration in Reactor Feed [mol\%]}$$

$$\text{Gas Holdup (vol\%)} = \frac{(\text{Liquid-Solid Density [lb/ft}^3\text{]} - \text{3-Phase Density [lb/ft}^3\text{]}) * 100}{(\text{Liquid-Solid Density [lb/ft}^3\text{]} - \text{Vapor Density [lb/ft}^3\text{]})}$$

where:

Vapor Density [lb/ft³] is calculated using the composition at the Reactor Effluent, and at the measured temperature and pressure of the Reactor

$$\text{3-Phase Density [lb/ft}^3\text{]} = \frac{\text{Pressure Drop [psi]} * 144 \text{ [in}^2\text{/ft}^2\text{]}}{\text{Span between Taps of Pressure Drop Measurement [ft]}}$$

Liquid-Solid Phase Density [lb/ft³] is a function of the catalyst concentration [wt%], which is iterated until the calculated weight of catalyst matches the catalyst weight in the Reactor.

Notes:

1. All terms in *Italics* refer to measured variables, either by field instruments or gas chromatographs.

$$\text{Gassed Slurry Volume [ft}^3\text{]} = \text{Reactor Cylindrical Volume [ft}^3\text{]} + \text{Reactor Head Volume [ft}^3\text{]}$$

where: Reactor Cylindrical Volume [ft³] = A [ft²] * Cylindrical Height [ft]

$$A \text{ [ft}^2\text{]} = \frac{\pi}{4} * (\text{Reactor ID [ft]})^2 * (\text{Fraction Open Area})$$

$$\text{Reactor ID} = \text{Reactor Inside Diameter} = 7.5 \text{ ft}$$

Fraction Open Area = 0.9652 (3.48% of the cross-sectional area of the Reactor is occupied by the tubes of the internal heat exchanger)

$$\text{Cylindrical Height [ft]} = \text{Gassed Slurry Height [ft]} - \frac{(\text{Reactor ID [ft]})}{4}$$

Note: The term “(Reactor ID)/4” is the depth of the bottom head of the Reactor.

$$\text{Reactor Head Volume [ft}^3\text{]} = \frac{\pi * (\text{Reactor ID [ft]})^3}{24} - 8 \text{ [ft}^3\text{]}$$

Note: 8 cubic feet is subtracted from the Reactor Head Volume to account for the displacement of the gas sparger and the header piping for the internal heat exchanger.

$$\text{Inlet Superficial Velocity [ft/s]} = \frac{\text{Reactor Feed Flow [lbmol/hr]} * V \text{ [ft}^3\text{/lbmol]}}{3,600 \text{ [s/hr]} * A \text{ [ft}^2\text{]}}$$

where: $V \text{ [ft}^3\text{/lbmol]} = \frac{10.73 * (\text{Reactor Temperature [}^\circ\text{F]} + 459.67)}{(\text{Reactor Pressure [psig]} + 14.1)}$

$$A \text{ [ft}^2\text{]} = \frac{\pi}{4} * (\text{Reactor ID [ft]})^2 * (\text{Fraction Open Area})$$

$$\text{Reactor ID} = \text{Reactor Inside Diameter} = 7.5 \text{ ft}$$

Fraction Open Area = 0.9652 (3.48% of the cross-sectional area of the Reactor is occupied by the tubes of the internal heat exchanger)

Notes:

1. All terms in *Italics* refer to measured variables, either by field instruments or gas chromatographs.

$$\text{Methanol Productivity [gmol/kg-hr]} = \frac{(\text{Methanol in Reactor Effluent [lbmol/hr]} - \text{Methanol in Reactor Feed [lbmol/hr]}) * 1,000 \text{ [g/kg]}}{\text{Catalyst Weight (lb oxide)}}$$

where:

$$\text{Methanol in Reactor Effluent [lbmol/hr]} = \text{Reactor Effluent Flow [lbmol/hr]} * \text{Methanol Concentration in Reactor Effluent Stream [mol\%]}$$

$$\text{Methanol in Reactor Feed [lbmol/hr]} = \text{Reactor Feed Flow [lbmol/hr]} * \text{Methanol Concentration in Reactor Feed Stream [mol\%]}$$

$$\text{Reactor O-T-M Conversion [\%]} = \frac{\text{Lower Heating Value of Raw Methanol} * 100}{\text{Lower Heating Value of Reactor Feed}}$$

Note: Lower heating values for Raw Methanol and Reactor Feed are calculated from compositions of each stream, in units of million Btu per hour.

$$\text{Reactor Volumetric Productivity [TPD/ft}^3\text{]} = \frac{\text{Raw Methanol Flow [TPD]}}{\text{Gassed Slurry Volume [ft}^3\text{]}}$$

$$\text{Space Velocity [sL/kg-hr]} = \frac{\text{Reactor Feed Flow [lbmol/hr]} * 10,175 \text{ [sL/lbmol @ 0}^\circ\text{C]}}{\text{Catalyst Weight [lb oxide]} * 0.454 \text{ [kg/lb]}}$$

$$\text{Sparger Resistance Coefficient "K"} = \frac{\text{Sparger Pressure Drop [psi]} * \text{Reactor Feed Density [lb/ft}^3\text{]} * 10^9}{(\text{Reactor Feed Flow [KSCFH]} * \text{Reactor Feed Molecular Weight [lb/lbmol]})^2}$$

where: 10^9 is an arbitrary factor.

$$\text{Syngas Usage [Btu/gallon Methanol]} = \frac{\text{Syngas LHV to Methanol [Btu/hr]} * 24 \text{ [hr/day]} * 6.642 \text{ [lb/gallon Methanol]}}{\text{Raw Methanol Flow [TPD]} * 2,000 \text{ [lb/ton]}}$$

where:

Syngas LHV to Methanol [Btu/hr] = the difference between the Lower Heating Value of the three feed gas streams (Balanced Gas, CO Gas, H₂ Gas) and the two purge gas streams (Main Plant Purge, Distillation Purge). Lower heating values are calculated from the compositions of each stream.

Notes:

1. All terms in *Italics* refer to measured variables, either by field instruments or gas chromatographs.

$$\text{Syngas Utilization [SCF/lb Methanol]} = \frac{(\text{Balanced Gas Flow [SCFH]} + \text{CO Gas Flow [SCFH]}) * 24 \text{ [hr/day]}}{\text{Raw Methanol Flow [TPD]} * 2,000 \text{ [lb/ton]}}$$

Notes:

1. All terms in *Italics* refer to measured variables, either by field instruments or gas chromatographs.

**APPENDIX M - TASK 3.6 - INTERIM PROJECT REVIEW MEETING
(29 & 30 April 1997)**

**APPENDIX N - TASK 3.6 - MILESTONE SCHEDULE STATUS AND COST
MANAGEMENT REPORTS**

APPENDIX O - PRESS RELEASE (21 MAY 1997) AND PRESS COVERAGE