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

**TECHNICAL PROGRESS REPORT NO. 11**

**For The Period**

**1 January - 31 March 1997**

**Prepared by**

**Air Products and Chemicals, Inc.  
Allentown, Pennsylvania**

**and**

**Eastman Chemical Company  
Kingsport, Tennessee**

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

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

**Patents cleared by Chicago on 27 May 1997.**

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

During this quarter, the third draft of the Topical Report on Process Economics Studies was issued for review. A recommendation to continue with design verification testing on the coproduction of methanol and dimethyl ether (DME) was made. An liquid phase dimethyl ether (LPDME) catalyst system with reasonable long-term activity and stability is being developed, and a decision to proceed with a proof-of-concept test run at the LaPorte Alternative Fuels Development Unit (AFDU) is pending the release of a memo from Air Products on the catalyst targets and corresponding economics for a commercially successful LPDME catalyst. The off-site product-use test plan is to be updated in June of 1997.

During this quarter, Air Products and Acurex Environmental Corporation continued developing the listing of product-use test participants who are involved in fuel cell, transportation, and stationary power plant applications. All major Construction (Task 2.2) contract work was completed during the reporting period. Commissioning activities (Task 2.3) focused on checkout of the equipment, instrument and control, and data acquisition systems. All utility systems were brought online.

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

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

Ninety-seven percent (97%) of the \$38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 31 March 1997. One percent (1%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 March 1997.

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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 <sub>2</sub> ), carbon monoxide (CO), and carbon dioxide (CO <sub>2</sub> ) in stoichiometric balance for the production of methanol
Carbon Monoxide Gas	-	A syngas containing primarily carbon monoxide (CO); also called CO Gas
DME	-	dimethyl ether
DOE	-	United States Department of Energy
DOE-FETC	-	The DOE's Federal Energy Technology Center (Project Team)
DOE-HQ	-	The DOE's Headquarters - Coal Fuels and Industrial Systems (Project Team)
DTP	-	Demonstration Test Plan - The four-year Operating Plan for Phase 3, Task 2 Operation
DVT	-	Design Verification Testing
Eastman	-	Eastman Chemical Company
EIV	-	Environmental Information Volume
EMP	-	Environmental Monitoring Plan
EPRI	-	Electric Power Research Institute
HAPs	-	Hazardous Air Pollutants
Hydrogen Gas	-	A syngas containing an excess of hydrogen (H <sub>2</sub> ) over the stoichiometric balance for the production of methanol; also called H <sub>2</sub> Gas
IGCC	-	Integrated Gasification Combined Cycle, a type of electric power generation plant
IGCC/OTM	-	An IGCC plant with a "Once-Thru Methanol" plant (the LPMEOH™ Process) added-on
KSCFH	-	Thousands Standard Cubic Feet per Hour
LaPorte PDU	-	The DOE-owned experimental unit (PDU) located adjacent to Air Products' industrial gas facility at LaPorte, Texas, where the LPMEOH™ process was successfully piloted
LPDME	-	Liquid Phase DME process, for the production of DME as a mixed coproduct with methanol
LPMEOH™	-	Liquid Phase Methanol (the technology to be demonstrated)
MTBE	-	methyl tertiary butyl ether
NEPA	-	National Environmental Policy Act
OSHA	-	Occupational Safety and Health Administration
Partnership	-	Air Products Liquid Phase Conversion Company, L.P.
PDU	-	Process Development Unit
PFD	-	Process Flow Diagram(s)
ppbv	-	parts per billion (volume basis)
Project	-	Production of Methanol/DME Using the LPMEOH™ Process at an
psia	-	Integrated Coal Gasification Facility
psig	-	Pounds per Square Inch (Absolute)
P&ID	-	Pounds per Square Inch (gauge)
SCFH	-	Piping and Instrumentation Diagram(s)
SL/hr-kg	-	Standard Cubic Feet per Hour
Syngas	-	Standard Liter(s) per Hour per Kilogram of Catalyst
Synthesis Gas	-	Abbreviation for Synthesis Gas
	-	A gas containing primarily hydrogen (H <sub>2</sub> ) and carbon monoxide (CO), or mixtures of H <sub>2</sub> and CO; intended for "synthesis" in a reactor to form methanol and/or other hydrocarbons (synthesis gas may also contain CO <sub>2</sub> , water, and other gases)
Tie-in(s)	-	the interconnection(s) between the LPMEOH™ Process Demonstration Facility and the Eastman Facility
TPD	-	Ton(s) per Day
WBS	-	Work Breakdown Structure
wt	-	weight

## Executive Summary

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

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

The project involves the construction of an 80,000 gallons per day (260 tons per day (TPD)) methanol unit utilizing coal-derived syngas from Eastman's integrated coal gasification facility. The new equipment consists of syngas feed preparation and compression facilities, the liquid phase reactor and auxiliaries, product distillation facilities, and utilities.

The technology to be demonstrated is the product of a cooperative development effort by Air Products and DOE in a program that started in 1981. Developed to enhance electric power generation using integrated gasification combined cycle (IGCC) technology, the LPMEOH™ process is ideally suited for directly processing gases produced by modern day coal gasifiers. Originally tested at a small, DOE-owned experimental unit in LaPorte, Texas, the technology provides several improvements essential for the economic coproduction of methanol and electricity directly from gasified coal. This liquid phase process suspends fine catalyst particles in an inert liquid, forming a slurry. The slurry dissipates the heat of the chemical reaction away from the catalyst surface, protecting the catalyst and allowing the methanol synthesis reaction to proceed at higher rates.

At the Eastman complex, the technology is integrated with existing coal gasifiers. A carefully developed test plan will allow operations at Eastman to simulate electricity demand load-following in coal-based IGCC facilities. The operations will also demonstrate the enhanced stability and heat dissipation of the conversion process, its reliable on/off operation, and its ability to produce methanol as a clean liquid fuel without additional upgrading. An off-site product-use testing program will be conducted to demonstrate the

suitability of the methanol product as a transportation fuel and as a fuel for stationary applications for small modular electric power generators for distributed power.

The four-year operating test phase will demonstrate the commercial application of the LPMEOH™ process to allow utilities to manufacture and sell two products: electricity and methanol. A typical commercial-scale IGCC coproduction facility, for example, could be expected to generate 200 to 350 MW of electricity, and to also manufacture 45,000 to 300,000 gallons per day of methanol (150 to 1000 TPD). A successful demonstration at Kingsport will show the ability of a local resource (coal) to be converted in a reliable (storable) and environmentally preferable way to provide the clean energy needs of local communities for electric power and transportation.

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

The project was reinitiated in October of 1993, when DOE approved a site change to the Kingsport location. DOE conditionally approved the Continuation Application to Budget Period No. 2 (Design and Construction) in March of 1995 and formally approved it on 01 June 1995 (Mod M009). After approval, the project initiated Design - Phase 1 - activities; and initiated Construction - Phase 2 - activities in October of 1995. The project required review under the National Environmental Policy Act (NEPA) to move to the construction phase. DOE prepared an Environmental Assessment (DOE/EA-1029), and subsequently a Finding of No Significant Impact (FONSI) was issued on 30 June 1995. The Cooperative Agreement was modified (Mod A011) on 8 October 1996, authorizing the transition from Budget Period No. 2 (Design and Construction) to the final Budget Period (Commissioning, Start-up, and Operation). This modification provides the full \$213,700,000 of authorized funding, with 56.7% participant cost share and 43.3% DOE cost share.

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

A recommendation to continue with DME design verification testing was made. DME design verification testing studies show the liquid phase DME (LPDME) process will have a significant economic advantage for the coproduction of DME for local markets. The market applications for DME are large. An LPDME catalyst system with reasonable long-term activity and stability is being developed. Planning for a proof-of-concept test run at the

LaPorte Alternative Fuels Development Unit (AFDU) was recommended; and a decision to proceed is pending the release of a memo from Air Products on the catalyst targets and corresponding economics for a commercially successful LPDME catalyst.

The off-site, product-use test plan is to be updated in June of 1997. During this quarter, Air Products and Acurex continued developing the listing of product-use test participants who are involved in fuel cell, transportation, and stationary power plant developments.

A project review meeting was held in Kingsport in late March. The construction and commissioning status was reviewed, and results from activation of the first batch of catalyst were discussed. A tour of the completed demonstration unit was conducted.

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

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

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

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

Ninety-seven percent (97%) of the \$38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 31 March 1997. One percent (1%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 March 1997.

## **A. Introduction**

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

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

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

## **B. Project Description**

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

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

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

- *Storage/Utility Area* - Methanol product, slurry, and oil storage equipment.

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

- *Reaction Area*

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

- *Purification Area*

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

- *Catalyst Preparation Area*

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

- *Storage/Utility Area*

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

## **C. Process Description**

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

## **D. Results and Discussion**

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

### ***Task 1.2 Permitting***

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

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

### ***Task 1.3 Design Engineering***

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

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

### ***Task 1.4 Off-Site Testing (Definition and Design)***

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

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

## Discussion

The fuel-use test plan, developed in 1992 to support the demonstration at the original Cool Water Gasification Facility site, has become outdated. Since the site change to Eastman, the original fuel test plan under-represents new utility dispersed electric power developments, and possibly new mobile transport engine developments. The updated fuel-use test plan will attempt for broader market applications and for commercial fuels comparisons. The objective of the fuel-use test plan update will be to demonstrate commercial (e.g., economic) market applications (municipal, industrial and electric utility) replacing or supplementing (gasoline, diesel, natural gas) commercial fuels, based on expected (1998 to 2018) U.S. energy market needs when the technology is to be commercialized.

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

A limited quantity (up to 400,000 gallons) of the methanol product as produced from the demonstration unit will be made available for fuel-use tests. Fuel-use tests will be targeted for an approximate 18 to 30-month period, commencing in the second year of demonstration operations. The methanol product will be available for shipment from the demonstration unit in Kingsport, Tennessee. Air Products, Acurex Environmental Corporation (Acurex), and the DOE will develop the final fuel-use test plan.

## Activity during this quarter

- The fuel-use test plan is targeted to be updated in June of 1997. This will allow 12 months for proper implementation of the tests, which will be conducted for an 18 to 30 month period commencing in May of 1998. The Demonstration Test Plan (see Task 2.3) indicates methanol for testing (as-produced from carbon monoxide (CO)-rich syngas) will first be produced in May of 1998.
- Air Products and Acurex continued to develop the listing of fuel-use test prospects. These projects are being prioritized by their likelihood to proceed and the timing for the initial need of methanol. A review meeting between Air Products, Acurex, and the DOE is planned for mid-April. A status update of the fuel-use program will be an agenda item for a 29-30 April interim review meeting between Air Products and the DOE.

## **Task 1.5 Planning and Administration**

### **Task 1.5.1 Product-Use Test Plan**

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

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

### **Task 1.5.2 Commercialization Studies**

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

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

#### Discussion

A number of areas have been identified as needing development to support specific commercial design studies. These include: a) product purification options; b) front-end impurity removal options; c) catalyst addition/withdrawal options; and d) plant design configuration options. Plant sizes in the range of 300 TPD to 1800 TPD and plant design configurations for the range from 20% up to 70% syngas conversion will be considered. The Kingsport demonstration unit design and costs will be the basis for value engineering work to focus on specific cost reduction targets in developing the initial commercial plant designs.

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

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

will help set demonstration operating goals, and identify the important market opportunities for the liquid phase technology.

- c) to provide input to the Demonstration Test Plan (Task 2.3).
- d) to provide input to the Off-site Testing (Task 1.4) fuel-use test plan update.

#### Activities during this quarter

- Part One of the Outline - "Coproduction of Methanol" was reissued for review and comment, as a draft Topical Report. The 31 March 1997 transmittal memo is included in Appendix C. This Topical Report develops plant design options for the LPMEOH™ process, as an add-on to IGCC power plants for the coproduction of methanol and power. Part One also compares the LPMEOH™ (LP) process with gas phase (GP) methanol processes. Surprisingly, the LP technology can coproduce methanol at less than 50 cents per gallon, even at relatively small (400 to 1200 TPD) methanol plant sizes. LP's advantage over GP is 6 to 9 cents per gallon. Therefore, when baseload IGCC power is viable, the LP technology makes coproduction viable. Comments on this draft Topical Report are expected in early June of 1997.
- Part Two of the Outline - "Baseload Power and Methanol Coproduction", has been incorporated into the paper, "Fuel and Power Coproduction", that was presented at the DOE's Fifth Annual Clean Coal Technology Conference in January of 1997.
- Part Four of the Outline - "Methanol Fuel Applications", is being used as the basis to update the fuel-use test plan (Task 1.4).

#### **Task 1.5.3 DME Design Verification Testing**

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

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

#### Discussion

The first decision milestone, on whether to continue with DME DVT, was targeted for 1 December 1996. This milestone has been relaxed to July of 1997 to allow time for further development of the LPDME catalyst system. DVT is required to provide additional data for engineering design and demonstration decision-making. The essential steps required for decision-making are: a) confirm catalyst activity and stability in the laboratory, b) develop engineering data in the laboratory, and c) confirm market(s), including fuels and chemical feedstocks. The DME Milestone Plan, showing the DVT work and the decision and implementation timing, is included in Appendix D.

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

#### ***DME DVT Recommendation***

Air Products made a recommendation to continue with the design verification testing of DME, and to proceed with planning a proof-of-concept test run at the DOE's AFDU at LaPorte, Texas. A copy of the updated recommendation (dated 10 February 1997) is included in Appendix D. The recommendation was based on the results of the Market Economic Studies and on the LPDME catalyst system R&D work, and is summarized in the following.

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

- Planning for a DME test run at the LaPorte AFDU, in conjunction with other DOE Liquid Fuels Programs, should be initiated. Test plans, budgets, and a schedule for these LaPorte AFDU tests should now be developed. Up to \$875,000 of Clean Coal Technology Program budget support, from the LPMEOH™ Project's FY-97 Cost Plan (budget), could be made available to support a suitable LPDME test run at LaPorte.
- An implementation decision, made mutually by the DOE's Clean Coal Technology Program (DE-FC22-92PC90543) LPMEOH™ project participants, and by the DOE's Indirect Liquefaction Program (DE-FC22-95PC93052) project participants, should be made in time to implement testing at LaPorte.

The recommendation to continue design verification testing of DME with proof-of-concept testing the LaPorte AFDU is now under consideration. LPDME is not applicable to hydrogen (H<sub>2</sub>)-rich syngas; and it is unlikely that a substantive LPDME demonstration will be recommended for Kingsport. Therefore, a convincing case that the test-run on CO-rich syngas at LaPorte will lead to successful commercialization must be made, prior to approving the final test-run plan. The strategy for commercialization must present the technical logic to combine the results of the following two areas:

- 1) catalyst performance (productivity, selectivity, and life) for the LPDME catalyst system under CO-rich syngas from the proof-of-concept testing at the LaPorte AFDU; and

- 2) reactor performance (methanol catalyst activity and life, hydrodynamics, and heat transfer) from the LPMEOH™ Process Demonstration Unit

The productivity and life of an "acceptable" LPDME catalyst system must be better defined, and then confirmed in the laboratory. A memo from Air Products on the catalyst targets and corresponding economics for a commercially successful LPDME catalyst will be issued in April of 1997. This document, along with updated laboratory results (as discussed in the following section), will be the basis for discussion at the 29-30 April interim review meeting between Air Products and the DOE.

### ***Market Economic Studies***

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

### ***Laboratory R&D***

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

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

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

#### *Summary of Laboratory Activity and Results*

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

#### **Task 1.5.4 Administration and Reporting**

The Cooperative Agreement was modified (Mod A011 on 8 October 1996), authorizing the transition from Budget Period No. 2 (Design and Construction) to the final Budget Period (Commissioning, Start-up, and Operation). This modification provides the full DOE cost share of \$92,700,000 of authorized funding, with the remaining \$121,000,000 being provided by the participants. A copy of the approval memorandum, dated 3 October 1996, is included in Appendix E.

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

data acquisition system in the partnership trailer was used to view the progress and completion of the second catalyst batch. A tour of the demonstration unit was conducted. The status of the updated fuel-use test plan, the recommendation for continuation of DME design verification testing, and other matters were reviewed. The meeting agenda, extracts from the meeting handouts, and the meeting notes are included in Appendix F.

The Milestone Schedule Status Report and the Cost Management Report, through the period ending 31 March 1997, are included in Appendix G. These two reports show the current schedule, the percentage completion and the latest cost forecast for each of the Work Breakdown Structure (WBS) tasks. The demonstration unit was mechanically complete on 31 January 1997. Ninety-seven percent (97%) of the \$38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 31 March 1997. One percent (1%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 March 1997.

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

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

### ***Task 2.1 Procurement***

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

- Complete the bidding and procurement for all equipment and Air Products-supplied construction materials.
  - All the equipment items have been received at the site.
  - The prefabricated structural steel for the reactor building and for the catalyst building has been received at the site. The bulk materials (prefab piping, valves, instrumentation, and electrical) have also been received at the site.
  - Task 2.1 Procurement is complete.

### ***Task 2.2 Construction***

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

- Provide construction management for contractor coordination and compliance with design, construction, and quality control standards.
- Erect the major equipment and structural steel. Install the large bore piping, electrical, and insulation such that instrument check-out and equipment commissioning work can be completed during the 60-day Continuation Application approval period.
  - Pressure testing of the last piping circuit was completed on 05 February 1997. This circuit took additional time due to the need to replace manual isolation valves which did not meet the pressure test requirements after three attempts. Once the new valves were installed, the circuit passed the pressure test on the first attempt.
  - The hoist mechanisms for the two nuclear density gauges were also installed during the reporting period. The drives and gear assemblies for the hoists were reworked to provide the proper start-up and braking sequences. Structural beams were added to both applications in order to provide additional support to the pulley systems.
  - After a thorough review of the process piping, pressure relief circuits, and supports, the final piping circuit was accepted on 23 February 1997.
  - The installation of all instrument and electrical wiring was completed in late February. The last item completed was the wiring of the reactor and reduction vessel nuclear density gauges. A vendor representative from Texas Nuclear visited the site to supervise installation of the meters, perform a background radiation survey with Eastman Plant Protection personnel, and function-test the detectors.
  - The Insulation and Fireproofing Contractor completed all contract work on 10 March 1997.
- Complete mechanical construction so that check-out and commissioning can be started in Budget Period No. 3.
  - All major construction contract work has been completed. During the reporting period, commissioning activities (Task 2.3) were completed in parallel with the end of construction, and the demonstration unit was ready to receive syngas on 02 March 1997. Remaining construction activities include the completion of site paving/grading and the painting of large- and some small-bore piping systems. This work is being managed by an Air Products construction supervisor, and will be completed in May of 1997.

### **Task 2.3 Training and Commissioning**

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

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

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

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

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

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

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

to repeat the bench test which had been performed after initial assembly of the pump. A representative from Eastman was present to witness this test, during which this pump also could not develop more than 600 psig discharge pressure. Air Products and Eastman are working with the manufacturer (Ingersoll Dresser) to determine the cause of the loss of performance and identify a solution. Note that the condensed oil pumps cannot be tested at full suction pressure until the oil make-up pumps are able to function at rated conditions.

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

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

#### ***Task 2.4 Off-Site Testing (Procurement and Construction)***

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

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

#### ***Task 2.5 Planning and Administration***

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

- Prepare annually an updated (Partnership) plan for the remaining activities. The first annual plan will update the remaining Phase 1 and Phase 2 activities, and the second will include an update of the Phase 3 Demonstration Test Plan.
  - The first update of the Partnership Annual Operating Plan was prepared and submitted in September of 1995 (See Quarterly Technical Progress Report No. 5). The main goal and objective for this first annual plan was to continue construction so that the LPMEOH™ demonstration unit would be ready for commissioning and start-up in 1996; and to complete the Project Evaluation Report and to submit it to the DOE along with the Continuation Application for Budget Period No. 3.

- The second update of the Partnership Annual Operating Plan was prepared and submitted in November of 1996 (see Appendix I). The main goal and objective for this second annual plan is to initiate Phase 3 - Operation of the LPMEOH™ demonstration unit and to achieve 30 weeks of operation (Task 2.1.1 Operation) by September of 1997 in accordance with the Demonstration Test Plan. Other objectives include continuation of DME design verification testing, and updating the plan for off-site product-use testing.
- Submit all Project status, milestone schedule, and cost management reports as required by the Cooperative Agreement.
  - The DOE reporting tasks are being performed and reported under Task 1.5.4 (Administration and Reporting).

### ***Task 3.1 Start-up***

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

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

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

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

Catalyst activation began on 16 March with the first of nine 2250 lb batches required for the initial catalyst charge. Test Authorization #K0-AD for catalyst activation is attached in Appendix L. As expected, after typical "learning curve"-type problems during the first few batches, the procedure became quite routine. Eventually, the entire operation, including oil fill, catalyst loading, mixing, activation temperature ramp, cooldown, transfers, and flushes, was compressed into about 36 hours/batch.

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

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

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

#### **E. Planned Activities for the Next Quarter**

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

## F. Conclusion

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

A recommendation to continue with DME design verification testing was made. DME design verification testing studies show the liquid phase DME (LPDME) process will have a significant economic advantage for the coproduction of DME for local markets. The market applications for DME are large. An LPDME catalyst system with reasonable long-term activity and stability is being developed. Planning for a proof-of-concept test run at the LaPorte Alternative Fuels Development Unit (AFDU) was recommended; and a decision to proceed is pending the release of a memo from Air Products on the catalyst targets and corresponding economics for a commercially successful LPDME catalyst.

The off-site product-use test plan is to be updated in June of 1997. During this quarter, Air Products and Acurex continued developing the listing of product-use test participants who are involved in fuel cell, transportation, and stationary power plant developments.

A project review meeting was held in Kingsport in late March. The construction and commissioning status was reviewed, and results from activation of the first batch of catalyst were discussed. A tour of the completed demonstration unit was conducted.

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

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

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

Catalyst activation of the nine 2250 lb batches required for the initial catalyst charge began on 16 March and concluded on 30 March. As expected, after typical "learning curve"-type problems during the first few batches, the procedure became quite routine. Eventually, the

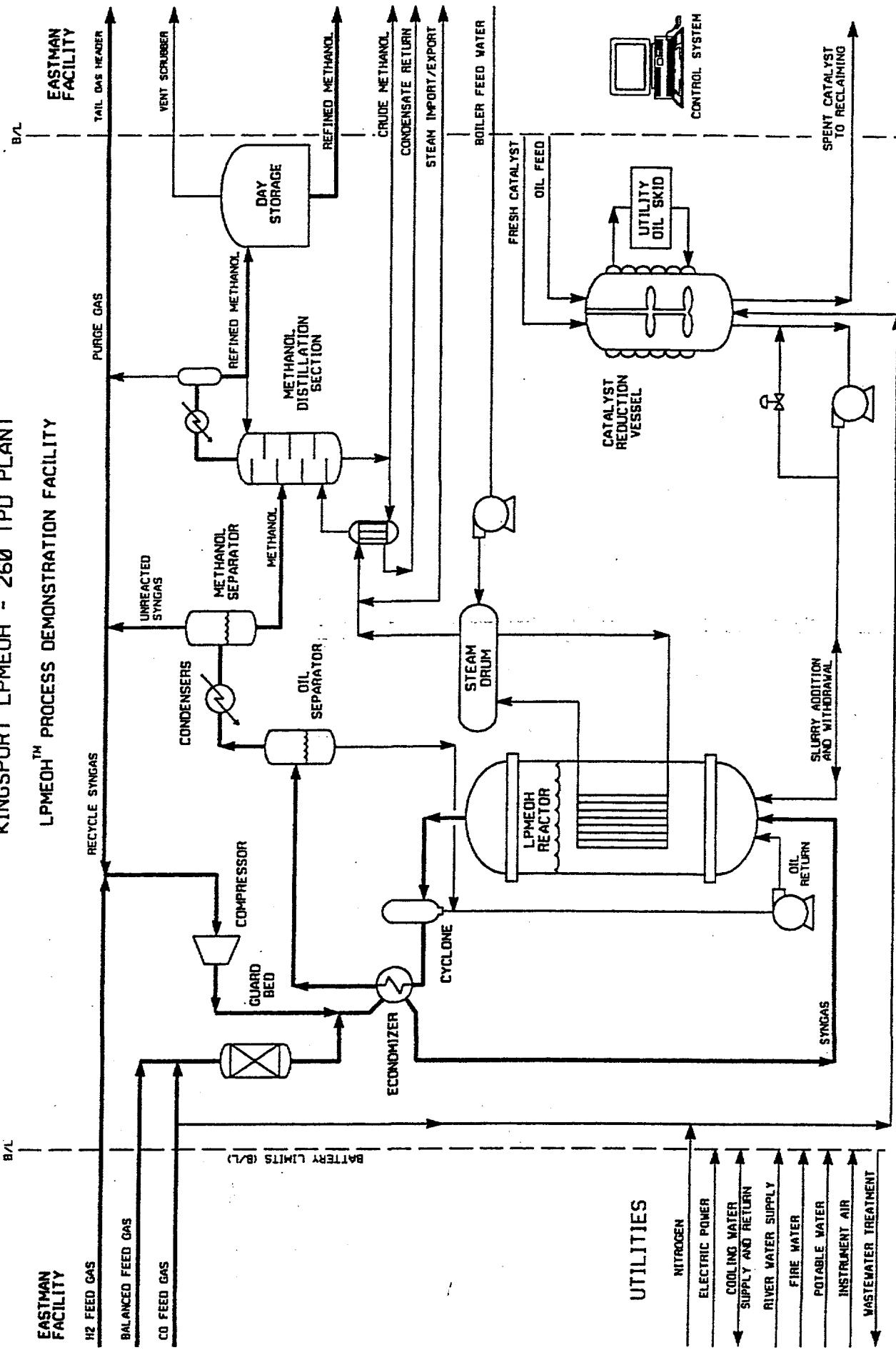
entire operation was compressed into about 36 hours/batch. All batches met or slightly exceeded the "theoretical maximum" uptake of 2.82 SCF of reducing gas/lb catalyst. Throughout the repetitive activation procedure, the growing charge of reduced catalyst was agitated under a slightly reducing atmosphere in the slurry storage vessel (29D-02). The first production of methanol at the demonstration unit is scheduled for April of 1997.

Ninety-seven percent (97%) of the \$38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 31 March 1997. One percent (1%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 March 1997.

**APPENDICES**

**APPENDIX A - SIMPLIFIED PROCESS FLOW DIAGRAM**

**SIMPLIFIED PROCESS DIAGRAM  
KINGSPORT LPMEOH - 260 TPD PLANT**



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

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

PROJECT EVALUATION PLAN FOR BUDGET PERIOD NO. 2

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

**1. Phase 1 - Task 2 - Permitting**

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

**2. Phase 1 - Task 3 - Design Engineering**

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

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

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

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

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

**5. Phase 2 - Task 1 - Procurement**

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

## 6. Phase 2 - Task 2- Construction

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

## 7. Phase 2 - Task 3 - Training and Commissioning

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

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

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

## 9. Phase 2 - Task 5 - Planning and Administration

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

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

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

WRB/jjs/Proeva.

**APPENDIX C - TASK 1.5.2 - PROCESS ECONOMIC STUDY**

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

**and**

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

# Process Economics Study - Outline

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

### Part One - Coproduction of Methanol

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

#### 1. Introduction

##### 1.. Process Design Options.

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

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

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

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

##### 2.2. Methanol Product Purification Cost.

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

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

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

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

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

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

# Process Economics Study - Outline LPMEOH™ Process, as an add-on to IGCC for Coproduction

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

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

## 2.6. Recommendations for Further Study.

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

## **Part Two - Baseload Power and Methanol Coproduction**

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

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

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

### **3.1 Gasification Plant Size Fixed**

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

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

### **3.2 Power Plant Size Fixed**

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

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

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

## **End of Part Two.**

**Process Economics Study - Outline**  
**LPMEOHT™ Process, as an add-on to IGCC for Coproduction**  
**Part Three - Coproduction for Intermediate Electric Load Following.**

**4. Intermediate Load Coproduction**

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

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

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

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

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

**4.2. Intermediate Load Coproduction - for Methanol Sales.**

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

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

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

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

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

**End of Part Three.**

# Process Economics Study - Outline LPMEOH™ Process, as an add-on to IGCC for Coproduction

## Part Four - Methanol Fuel Applications

### 5. Premium Methanol Fuel Applications

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

#### 5. . Hydrogen Source for:

- Hydrogen fuel cells, being developed for transportation applications, can achieve 65% system efficiency, as compared to 45% for diesel IC engines and 32% for gasoline IC engines. Methanol is a storable, transportable liquid fuel which can be reformed under mild conditions to provide H<sub>2</sub>. For small H<sub>2</sub> applications, *and at low utilization factors*, methanol reforming is a more economical source of hydrogen than : a) natural gas reforming, b) distillate (oil) reforming; and is cheaper than liquid H<sub>2</sub>.

##### 5.1.1. Fuel Cells for Transportation

##### 5.1.2. Fuel Cells for Stationary Power

(See also dispersed power below).

##### 5.1.3. Industrial Applications - Small Hydrogen Plants

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

#### 5.2. Dispersed Power

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

#### 5.3. *Dimethyl Ether as an Enhancement to Methanol in Premium Fuel Applications* Can coproduced mixtures of methanol and dimethyl ether improve upon methanol, in the above?

**End of Part Four.**

## Memorandum



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

### Distribution:

c: D. M. Brown - APE (Hersham)  
R. J. Allam - APE (Hersham)

APCI

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

R. M. Kornosky - DOE-FETC  
W. J. O'Dowd - DOE-FETC  
W. C. Jones - Eastman

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

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

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

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

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

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

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

Bill

**APPENDIX D - TASK 1.5.3 - DME DESIGN VERIFICATION TESTING**

**DME Milestone Plan**

**and**

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

**TABLE 5-2**  
**DME MILESTONE PLAN**

**1. Design Verification Testing:**

- A. Laboratory R&D, Verification
  - 1. Catalyst Activity and Stability Testing, and Engineering Data Mar. '95 - Aug. '96
- B. Market Verification
  - 1. Up to 3 wt% DME, as M-100 Diesel Replacement Engine Tests - (SCAQMD) - and Market Acceptance Study by Jan. '96  
\*\* Completed \*\*
  - 2. Up to 8 wt% DME for GCC Energy Storage
    - a. Re-review economics by Aug. '96
  - 3. About 80 wt% DME with Methanol, as a Diesel Replacement Fuel, or as Chemical Feedstock at Kingsport
    - a. Economics, process basis study by Aug. '96
- C. Decision to Continue or Drop Demonstration by Dec. '96

**2. Process Development Unit Design Verification Test (Provisional):**

- A. LaPorte AFDU Tests by Sep. '97
- B. Decision to Continue or Drop Demonstration by Mar. '98

**3. Implementation (Provisional) Plan:**

- A. Design, Procurement, and Construction at Kingsport, of Add-on Equipment, and Operation Start July '98 to Jan. '01

### **LPDME Design Verification Testing .**

From the Statement of Work : "Subject to Design Verification Testing (DVT), the Partnership proposes to enhance the Project by including the demonstration of the slurry reactor's capability to produce DME as a mixed co-product with methanol. The production of DME from synthesis gas is a natural extension of the LPMEOH™ process in that three reactions occur concurrently in a single liquid phase reactor, methanol synthesis, methanol dehydration and water-gas shift. This process enhancement can significantly improve the overall conversion of coal derived synthesis gas to a storable blend of methanol and DME. - - - the enhanced (DME production demonstration is complementary to ongoing studies being sponsored by DOE's Liquid Fuels Program --) -- . -- At the conclusion of each of the DVT steps, a joint Partnership/DOE decision will be made regarding continuation of methanol/DME demonstration.."

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

#### **The LPDME Process Concept: - Three Concurrent Reactions:**

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

The overall reaction, with CO-rich Syngas, in a single liquid phase (slurry) reactor:

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

This is the "once-through" CO-rich syngas concept for the LPDME process utilizing a single slurry reactor. Conversion per pass, with CO-rich syngas, can be higher than for the LPMEOH™ process. Methanol may also be produced, as a mixed co-product with the DME, and can easily be separated and recovered. The separation of DME from CO<sub>2</sub> will be necessary for certain market applications.

#### **Status of the LPDME DVT Work**

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

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

- Aerosol - Small, but established market. High purity DME is required.
- Cooking Fuel. Potentially a large market, to replace imported LPG. There is a lot of interest in China, and DME is on the DOE's CCT/FE China meeting (Sept. of 1997) agenda. Purity, of

about >95% DME, with <2% methanol, < 3% CO<sub>2</sub> is estimated. An unresolved application issue is CO emissions during cooking. How does DME purity impact this? Use testing is needed.

- Diesel Replacement Fuel. DME is an ultra clean (high Cetane) diesel fuel; and an 80% DME mixture with methanol and water is now being engine-tested by others (Amoco, et. al.). Market development (at least in the U.S.) faces a fuel distribution infrastructure problem. DME might more easily replace LPG in countries where LPG is already an engine fuel.
- DME Derivatives, as a Diesel Fuel Additive. Quotes from the DOE (Alt. Fuels R&D) Program quarterly report for April-June 1996: "Initial Cetane number (CN) testing of a three-component composition of 1,2-dimethoxy ethane, 1,1-dimethoxy methane and methanol blended with diesel fuel showed a 40% increase in the CN of the diesel fuel when the blend was 50/50." "The concept of adding a blend of oxygenated compounds to diesel fuel in order to enhance the Cetane value and cold start properties is being investigated. The blend of oxygenated compounds is derived from dimethyl ether chemistry, and builds on work conducted earlier --." It is early days for this DME feedstock chemistry, but CO<sub>2</sub> may not need to be separated from the DME.
- DME Derivatives, as Chemicals/Other Fuels. DME is a key intermediate in a commercial synthesis gas-to-gasoline process, and is being developed as an intermediate for other chemicals and fuels as part of the DOE's Liquid Fuels (Alt. Fuels R&D) Program.

**A. The economics studies**, for once-through coproduction (with an IGCC power plant for example) on synthesis gas rich in carbon oxides, show that the LPDME process will have an advantage greater than the LPMEOH™ process. A once-through LPDME reactor is able to convert greater than 50% of such a syngas, whereas a once-through LPMEOH™ reactor can convert only about 30%. The economics, of course, depend upon the end-use (purity) of the DME and upon the gasification plant's coproduct mix (amount of power, methanol, DME, etc.). The same liquid phase reactor design options to increase syngas conversion (see the CCT Tampa Conference Paper); such as feed gas compression and/or CO-rich gas recycle; are also be applicable for LPDME. So, the LPDME technology should have a significant advantage for the coproduction of DME to serve local markets.

As with the LPMEOH™ process, gas phase process technology must be considered as the economic competitor. The gas phase DME process (see Haldor's patent) must run with H<sub>2</sub>-rich syngas. In the IGCC coproduction flow sheet, gas phase technology is at an economic disadvantage, since separate shift and CO<sub>2</sub> removal are required. As is the case for methanol, inexpensive remote natural gas would therefore be the economic plant site choice for gas phase technology. A comparison, of IGCC/LPDME coproduction with DME imported from remote gas facilities, shows an advantage for locally produced DME relative to imported DME. The transportation cost to import DME is much higher than for methanol, and the LPDME coproduction advantage is even greater than that for LPMEOH™ (vs. methanol import; see the CCT Tampa Conference Paper). Dehydration of imported methanol to make DME is not competitive either. Therefore, for DME in local markets, LPDME coproduction should be a winner!

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



Since water inhibits the methanol dehydration reaction, the slurry reactor must be staged, with water removal between stages. Staging could be by high ratio gas recycle, and/or with multiple reactors; but the once-through simplicity is lost. Therefore, it is unlikely that the LPDME process would be developed for use in H<sub>2</sub>-rich syngas applications.

#### **B. Laboratory R&D Results. (CCT Project - ended 9/96).**

An LPDME catalyst system, with reasonable long-term activity, was identified and tested. The system exhibits best activity under CO-rich syngas conditions, i.e. those most likely for (IGCC) coproduction. Accelerated aging of the catalyst system is a remaining issue. Water concentrations in the LP reactor are higher with syngases richer in H<sub>2</sub>, and its effect needs to be evaluated.

Lab work has continued under the DOE's Liquid Fuels Program. The issues, to be addressed in the lab before a LaPorte test-run decision, are: 1. Understanding the LPDME catalyst system's accelerated aging; and modifying the catalyst and/or the system operating conditions. 2. Manufacturing scale-up of catalyst for a LaPorte run.

#### **Recommendations**

The catalyst system and the market applications/opportunities are sufficiently promising that proof-of-concept testing at the LaPorte AFDU is recommended. Kingsport is an unlikely site for the commercial size demonstration of LPDME, since there are limited times for CO-rich syngas testing; and H<sub>2</sub>-rich syngas would create water buildup. Therefore, the LaPorte AFDU test-run, hydrodynamic programs, and data from the scaleup of the LPMEOH™ reactor; must provide the basis for commercializing LPDME. Recommendations:

- An LPDME test run at the LaPorte AFDU, in conjunction with the DOE's Liquid Fuels Program, would be appropriate if the LPDME catalyst system development can be completed successfully. Up to \$875,000 of CCT Program budget support, from the LPMEOH™ Project's FY-97 Cost Plan (budget), should be made available to support a suitable LPDME test run at LaPorte.
- An implementation decision, made mutually by the DOE's CCT (DE-FC22-92PC90543) LPMEOH™ project participants, and by the DOE's Liquid Fuels (DE-FC22-95PC93052) program participants, should be made (by July of 1997 in time to implement testing at LaPorte in early 1998. *(Final dates should be recommended by the DOE's Liquid Fuels program, based on progress in developing the LPDME catalyst system)*). The DOE LPMEOH™ project participants should be kept informed of the LaPorte AFDU LPDME test-run plans, so that a timely final approval can be made.
- In the interim, some DME product-use testing may be appropriate for the LPMEOH™ Project's Off-site Product testing.

(end).

**APPENDIX E - TASK 1.5.4 - APPROVAL FOR BUDGET PERIOD THREE**



Department of Energy  
Washington, DC 20585

OCT 03 1996

**MEMORANDUM**

To: Sun Chon  
Director, Pittsburgh Energy Technology Center

From: Patricia Fry Godley *Patricia Fry Godley*  
Assistant Secretary for Fossil Energy

Subject: Approval of Request for Transition to the Final Budget Period Three for the  
Liquid Phase Methanol Process Demonstration Project Cooperative Agreement  
No. DE-FC22-92PC90543

You are authorized to transition from Budget Period Two (Design and Construction) to the final Budget Period Three (Commissioning, Startup, and Operation) on the demonstration project, "Commercial Demonstration of the Liquid Phase Methanol Process," Cooperative Agreement No. DE-FC22-92PC90543 in accordance with the Continuation Application Request of August 2, 1996, and supporting documentation. You are authorized to provide \$67,138,458 of funding as the Department of Energy's (DOE) share of the cost of the final Budget Period. The total estimated cost of the project remains at \$213,700,000 with a DOE cost share of \$92,708,370.

cc:  
C. L. Miller  
G. Kight  
G. Lynch  
D. Archer  
J. Strakosy, PETC  
R. Komosky, PETC

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

NOTES FROM PROJECT REVIEW MEETING - March 19 & 20, 1997



Page One Of Six

DISTRIBUTION (NAME/ORGANIZATION) *Unable to attend. **Chairman			COPIED FOR INFORMATION ONLY	
Bill Jones* - EMN	Barry Street* - EMN		Bernie Toseland* - APCI	
Bill Brown* - APCI	Peter Tijm* - APCI		Bob Moore* - APCI	
Bob Kornosky* - DOE	Ed Heydorn* - APCI		Dave Drown - APCI	
Ed Schmetz - DOE	Frank Frenduto* - APCI		Barry Halper* - APCI	
Bill O'Dowd - DOE	Van Eric Stein* - APCI		Laurie Paulonis - EMN	
Tom Sarkus - DOE	Doug Archer - DOE		Bharat Bhatt - APCI	
<b>Note: * by your name means you have action item(s).</b>				
FROM William R. Brown <i>MB 4/11</i>	ORGANIZATION APCI - Program Manager	EXTENSION 17584	TODAY'S DATE April 8, 1997	
DATE OF MEETING Mar. 19 & 20, 1997	WEEKDAY Weds. & Thurs.	TIME STARTED 12:15 PM	ENDED 11:45 AM	LOCATION LPMEOH Trailer, Kingsport.
SUBJECT AND/OR PURPOSE • Project Review Meeting, DOE, Eastman, and Air Products				
DESIRED RESULTS/OUTCOMES • Review Project status, prepare for Operation, review DME "go" decision.				
REFERENCE MATERIAL/OTHER • Handouts, attached.				
ITEM NO.	RESPONSIBLE PERSON (INITIALS)	TARGET DATE	DISCUSSION	
			<p>The Project Review Meeting was held on March 19 and 20, 1997; at the LPMEOH™ Demonstration Plant site in Kingsport.</p> <p>The meeting agenda is attached; with attendee's indicated by a (✓) mark. The Meeting Notes (pages two through six) follow the agenda, and cover the twelve agenda items.</p> <p><b>Note that * by your name indicates an action item.</b></p>	

## MEETING NOTICE

(Mar. 19 &amp; 20, 1997)

AIR  
PRODUCTS

LEASE NOTE: Security badges required for visitors in all buildings and employees in R&amp;D buildings.

DISTRIBUTION (NAME/ORGANIZATION) <i>(If unable to attend, contact originator)</i>		COPIED FOR INFORMATION ONLY	
Bill Jones - EMN ✓	Barry Street - EMN ✓	Dan Canning - APCI	
Bill Brown - APCI ✓	Peter Tijm - APCI ✓	Brenda Overton - APCI	
Bob Kornosky - DOE ✓	Ed Heydorn - APCI ✓	Dave Drown - APCI	
Ed Schmetz - DOE ✓	Frank Frenduto - APCI ✓	Barry Halper - APCI	
Bill O'Dowd - DOE ✓	Van Eric Stein - APCI ✓	Laurie Paulonis - EMN ✓	
Tom Sardino - DOE ; Doug Anderson - DOE ✓	Bharat Bhatt - APCI X		
FROM William R. Brown <i>MRB 2/2/97</i> ORGANIZATION APCI - Program Manager		EXTENSION 17584	TODAY'S DATE 12 February 1997
DATE OF MEETING Mar. 19 & 20, 1997	WEEKDAY Weds. & Thurs.	TIME FROM 12:15 PM	LOCATION TO 11:45 AM LPMEOH Trailer, Kingsport.
SUBJECT AND/OR PURPOSE <ul style="list-style-type: none"> <li>Project Review Meeting, DOE, Eastman, and Air Products</li> </ul>			
DESIRED RESULTS/OUTCOMES <ul style="list-style-type: none"> <li>Review Project status, prepare for Operation, review DME "go" decision.</li> </ul>			
REFERENCE MATERIAL/OTHER <ul style="list-style-type: none"> <li>(LP) DME DVT Recommendation (Attached)</li> </ul>			
AGENDA	<u>LPMEOH™ Project Review Meeting - Tentative Agenda*.</u>		
<u>A. Meet and Lunch. Wednesday - Mar. 19th - About 12:15.</u> (DOE flight arrives at 11:40 am)			
1. Project Team for Phase 3. - (Reorganization(s)/Additions/Deletions). 1. RMK/WRB/WCJ			
<u>B. Project Review - Part One 1:30PM</u>			
2. Construction, Commissioning - (Summary - Final Costs) 2. FSF/(ECH)			
3. Phase 3, Task 1 - Startup - (Summary - Costs) 3. ECH/BTS			
4. Phase 3, Task 2 - Operations - 4a). Status 4. ECH/BTS			
4b). Demo Test Plan - what to expect next quarter			
5. Data Collection - 5a). Status/Review 5. VES/(ECH)			
5b). Test Plan Overview: Major Goals/Objectives with Milestones.			
<u>C. Plant Operation - Visit - 4:00PM -</u>			
<u>D. End of (work) Day One - 5:30PM</u> Dinner and early to bed.			
<u>E. Project Review - Part Two 8:30AM Thurs. - Mar/ 20th</u>			
6. DME Design Verification Testing - Status/Recommendation 6. WRB/PJAT			
6a). Approval of decision to support LaPorte AFDU Test-run All			
7. Reporting Requirements/Other - Status 7 WRB/RMK			
8.. Press Release; "Operation Commences; Outstanding Success" - Review Draft - 8. WRB/RMK/All			
9. Conference Papers/Plans/Process Economics Update 9. WRB/PJAT			
10. Updated Fuel-use demo plan - Status/Schedule 10. WRB/PJAT			
11. Report Card - Project Success Factors 11. All			
12. Make Plans for: Next Project Meeting; Visitors: Other 12. All			
<u>F. Lunch and Depart - 12:00PM - Thurs.. - Mar. 20th</u> (Return flights depart at 2:00 PM)			
* Agenda Questions/Additions/Deletions to Bill Brown please - for Final Agenda, issue prior to meeting.			

NOTES FROM PROJECT REVIEW MEETING - March 19 & 20, 1997  
CONTINUATION



Page Two Of Six

ITEM NO.	RESPONSIBLE PERSON (INITIALS)	TARGET DATE	DISCUSSION
1			<p><b>Project Team Reorganization(s) for Phase 3.</b>  The DOE/Air Products/Eastman Project Team is being reorganized for Phase 3 (Operation). Effective April 1st, Ed Heydorn will be taking over from Bill Brown as Air Product's Program Manager for the Project. The Cooperative Agreement has been modified to reflect this change. Our two Project Managers, Dave Drown (APCI) and Laurie Paulonis (EMN) will be moving on to other (Project/Life) activities; after doing a great job of managing (and virtually completing) the design and construction activities. Bill Brown will be retiring (May 1 - after methanol operation has begun) after 36 years at Air Products, including a memorable last few years helping to develop the LPMEOH™ technology. Peter Tijm (APCI) now has overall technical and commercial responsibility.</p> <p>The DOE's Fossil Energy (FE) and Federal Energy Technology Center (FETC) organizations have also changed. Three sheets (attached) describe the changes: i) FE's New Look, ii) Who's Who in FE (Coal and Power Systems), and iii) Federal Energy Technology Center organization chart. Our Project will be directly managed by FETC's Office of Project Management (Major Projects &amp; Agreements Division); and will be overseen at HQ by "Coal Fuels &amp; Industrial Systems", directly by Ed Schmetz and John Shen. Welcome back Ed and John, and good luck to Doug Archer.</p>
1a.	RMK	Done 3/27/97	1a): Action; prepare updated Project Team list. <u>The revised Project Team (04-97) listing is attached.</u>
1b.	RMK PJAT/WCJ	Done (4/4/97)	1b): As an action item, FETC has requested that a high level DOE/ Participant contacts list be prepared. <u>The high level contacts listing (4/4/97) is attached.</u>
2.			<p><b>2. Construction/Commissioning (Phase 2) Status</b>  The Mechanical and Electrical Contracts were completed in February, and insulation work was completed in mid-March (see attachment 2). The remaining minor Phase 2 work (Painting, Paving, Clean-up, and Punch List Items) will be finished in April. The cost forecast (attached) is basically unchanged from our previous (Dec. 4th) meeting.</p>

NOTES FROM PROJECT REVIEW MEETING - March 19 & 20, 1997  
CONTINUATION



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ITEM NO.	RESPONSIBLE PERSON (INITIALS)	TARGET DATE	DISCUSSION
3.			<p><b>3. Phase 3, Task 1, Startup. Status.</b>          Phase 3 - Startup was initiated on Jan. 24th, with the introduction of steam. Syngas was introduced on Feb. 28th, and Carbonyl burnout was completed March 12th. The first (of nine) catalyst batches was introduced into the catalyst reduction vessel on March 16th. As of March 20th two batches had been successfully activated. The catalyst activation works well, and it is anticipated that the nine batches will be completed by April 2nd. The distillation system has been successfully run with methanol/water. Operation (Task 2.1) is expected to start April 5th.</p> <p>The Commissioning and Startup Schedule is attached (five pages). Compared to the milestone schedule plan at our Dec. 5th project review meeting, the start of Task 2.1 - Operation has slipped about six weeks. Key indicators show that about five weeks was due to late construction completion; and about one week was due to extra Startup/Catalyst Reduction time.</p>
4.			<p><b>4. Phase 3, Task 2.1 - Operations; what to expect.</b>          The bad news for initial operation is: the G-03 A/B Oil Makeup Pumps can not provide the 750 psi head required. One pump has been returned to the manufacturer, and will be tested on Mar. 21st. These pumps will probably not be repaired (to design conditions) until later in April. The good news is: we will start operation without these pumps. This will combine Test 7 (Free-Drain of entrained/ condensed oil to the Reactor) with the initial shakedown operation (Test 1). Eastman's biennial gasification facility "complex down", planned to start May 12th, will provide a two to three week window to install the repaired G-03 pump, and to do other maintenance and inspection.</p>
4a.	ECH/VES	5/25/97	<p><b>4a):</b> The monthly reports: "Milestone Schedule Status Report" and "Project Summary Report"; may be combined together with one cover letter. The operations test plan schedule should be developed; so that important upcoming milestones are identified and their achievement chronicled in the monthlies.</p>

NOTES FROM PROJECT REVIEW MEETING - March 19 & 20, 1997  
CONTINUATION



Page Four Of Six

ITEM NO.	RESPONSIBLE PERSON (INITIALS)	TARGET DATE	DISCUSSION
5.			<p><b>5. Data Collection.</b> The data collection system was in use, and was demonstrated during the meeting. Data from the catalyst reduction runs was compared (favorably) with the historical LaPorte PDU data. This computerized system can store data, and should be an extremely powerful tool for gathering and correlating the engineering data for commercial designs.</p>
5a.	ECH/VES/ BTS	April '97	<p>5a): Monitoring, per the Environmental Monitoring Plan, will be initiated in April as Task 2.1 - Methanol Operation commences.</p>
6.			<p><b>6. DME Design Verification Testing (DVT) - Status</b> Peter Tijm summarized the market applications for DME (see pages 1 and 2 of attachment 6). The fuel applications (cooking fuel, diesel replacement fuel, and "Liquid DME" (DME derivatives) as a diesel fuel additive) are especially exciting. The recommendation to proceed with proof-of-concept testing at the LaPorte AFDU remains unchanged from our Dec. 4th project review meeting. As noted in the Dec. 4th meeting notes: <i>LPDME is not applicable to H<sub>2</sub>-rich syngas; and it is unlikely that a substantive LPDME demonstration will be recommended for Kingsport. Therefore, there must be a convincing case that the test run at LaPorte will lead to successful commercialization.</i> The DVT decision timing, in conjunction with the DOE's other Liquid Fuels programs, has been relaxed (~ July 1997) to allow time for the LPDME catalyst system development. Action items:</p>
6a.	RMK/PAT	Done 4/10/97	<p>6a): A DME review meeting, at Air Products in Allentown, should be scheduled for late April. <u>Meeting is set for April 29 and 30.</u></p>
6b.	PJAT RBM/BAT	4/22/97	<p>6b): An action item remains from the Dec. 4th project review meeting: Air Products is to define "acceptable" catalyst system performance (life, productivity). "Acceptable" must define the range of catalyst productivity and life necessary for the commercial LPDME application. The definition must also include the range of acceptable operating conditions, including pressure, temperature and reactor syngas composition (CO, CO<sub>2</sub>, H<sub>2</sub>). "Once-through" and "once-through" with gas recycle should be considered. Background information should be sent to DOE one week in advance of meeting.</p>

NOTES FROM PROJECT REVIEW MEETING - March 19 & 20, 1997  
CONTINUATION



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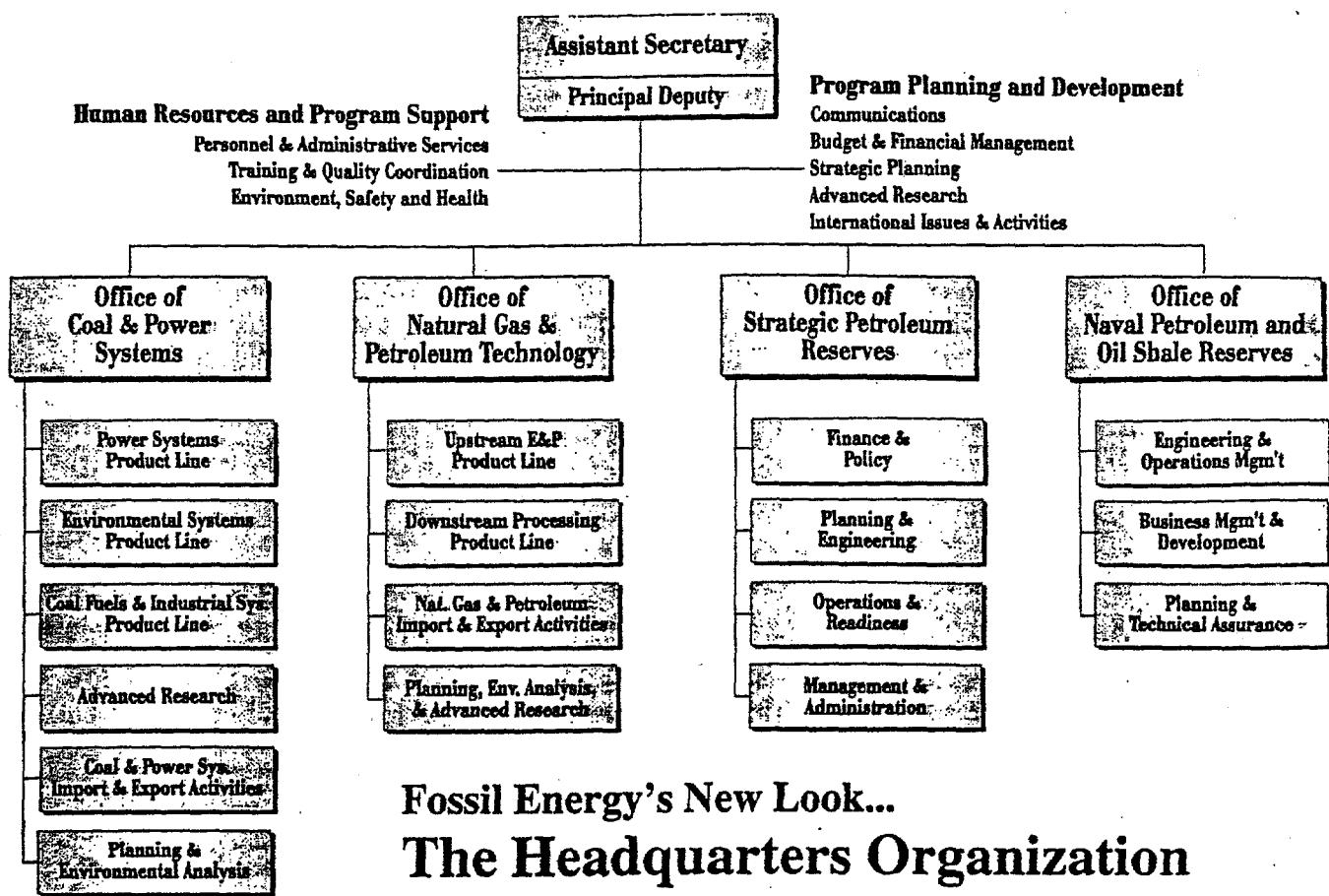
ITEM NO.	RESPONSIBLE PERSON (INITIALS)	TARGET DATE	DISCUSSION
7/9	WRB/RMK  FSF RBM/WRB WRB	4/25/97	<b>7 &amp; 9. Reporting Status / Paper Presentations</b> The reporting status and recent/pending papers are summarized in attachment 7. The quarterly reports are in fairly good shape, and should be near final by the end of April. The public design report is being developed (FSF). The third draft of the Topical on "Economic Analysis" should be ready in early April (RBM/WRB). The balance of the reports should be updated by the end of April (WRB).
8.			<b>8. Press Release</b> A draft TechLine, for release in April after successful initial operation, has been prepared and is circulating for review.
8a.	RMK/WRB	4/18/97	8a): A final release should be ready by mid-April.
10.			<b>10. Fuel-use Plan Update</b> A meeting with Acurex is planned for the week of April 13th, in conjunction with the ACS meeting in San Francisco (see papers, in attach. 7). a): A draft plan update is targeted for late in May. M. C. Williams (FETC) and Ed Schmetz are interested and should be involved in developing these plans. A DME cooking fuel test is being initiated at Penn State; as part of the overall plan. Methanol from LaPorte is still available (see ECH), if early setup/tests are required.
11.			<b>11. Report Card.</b> The Project "Success Factors" were reviewed. Now that Phase 2 is done, we can provide a good mid-term report card. The DOE/Air Products/Eastman <u>Participant relationships are excellent</u> . The Budget and <u>Schedule for Phases 1 &amp; 2</u> were good: the overall 24 month Design and Construction schedule, from the February of 1995 authorization; was <u>very good</u> , for a first-of-kind plant. The <u>Budget and Plan for Phase 3</u> are in <u>excellent</u> shape.
11a	ECH/BTS/ RMK	12/28/01	11a): Action item: Complete successful Phase 3 Operation, Obtain Industry Acceptance, and make the final report card "excellent".

NOTES FROM PROJECT REVIEW MEETING - March 19 & 20, 1997  
CONTINUATION



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ITEM NO.	RESPONSIBLE PERSON (INITIALS)	TARGET DATE	DISCUSSION
12.			<b>12. Plans</b>
12a	BJH/PJAT RMK WCJ	7/25/97	<p>12a): An LPMEOH™ Demonstration Plant dedication ceremony, for DOE/Air Products/Eastman participation, is desirable and should be targeted for the week of July 21st. Key participants (see high level contacts, in attachment 1) are to be contacted, and a date set so that plans can be made. <u>Dedication date has now been set for July 25th.</u> In conjunction with the Plant dedication, introduction/side-meetings for the high level contacts should be arranged, and also a tour of Eastman's Gasification Facility.</p>
12b	ECH	7/24/97	<p>12b): The next (full) project review meeting will be held in conjunction with the dedication. Smaller, individual meeting at Kingsport can be arranged through Ed Heydorn.</p> <p>- End - of March 19th &amp; 20th, 1997 Meeting Notes.</p> <p style="text-align: right;"><i>MB4/11</i></p>



**Fossil Energy's New Look...**  
**The Headquarters Organization**

# Who's Who in FE...and Where Are They Now?

With the Fossil Energy headquarters organization due to take effect November 1, here is the "crosswalk" of employees from the R&D and Strategic Petroleum Reserve offices into the

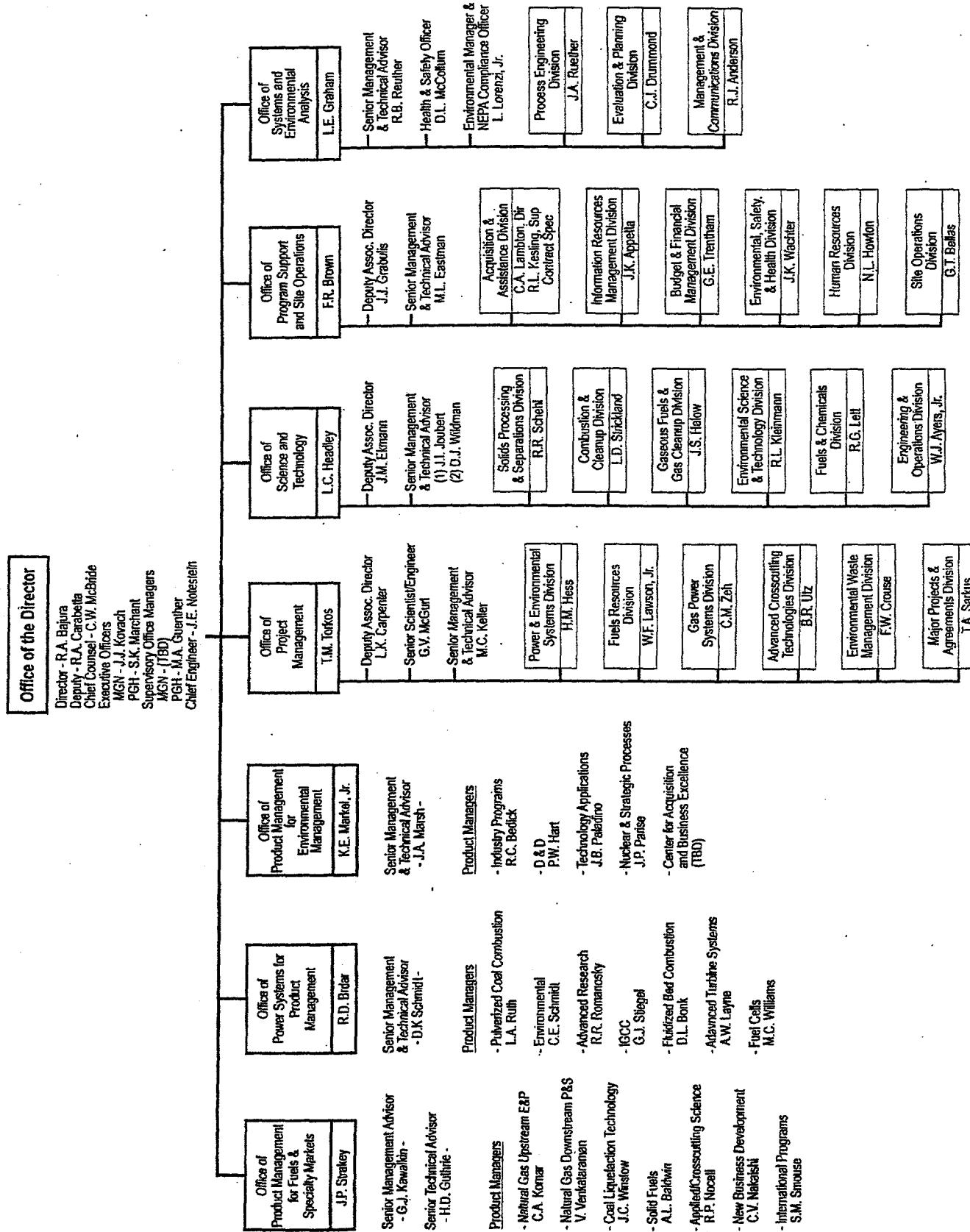
Fossil Energy's new offices and teams. The Naval Petroleum and Oil Shale Reserves Office is not affected by the organizational changes.

Assistant Secretary & Crosscutting Staff	Environment, Safety, Security & Health Team	Budget & Financial Management Team
<b>Assistant Secretary - P. Godley</b> B. Kripowicz - Principal Deputy C. Holmes - Executive Assistant J. Easton - Staff Assistant P. Hicks - Staff Analyst R. Coleman - Stay-in-School D. Hunter - Stay-in-School	C. Zamuda - Team Leader J. Johnson - NEPA I. Stern - Safeguards/Security A. White - OSHA T. Transtrum - TQM, Policy Studies V. Gardner - Secretary	C. Roy - Team Leader W. Musick - Budget Analyst P. Graham - Budget Analyst G. Stem - Budget Analyst M. Nicholson - Budget Assistant M. Roland - Procurement J. Panek - Procurement/Financial Management L. Mills - IG/GAO/Capital Equipment J. Gemma - Procurement C. Pinkney - Procurement W. Horseman - Secretary
<b>Personnel &amp; Administrative Services Team</b> E. Kilroy - Team Leader D. Wolfe - Administrative Officer L. Simons - Management Specialist D. Tolley - Personnel P. Gentel - Administrative Support S. Iannucci - Secretary	<b>Strategic Planning &amp; Analysis Team</b> J. Braitsch - Strategic Planning/Outreach/Coordination B. Kane - Environmental Issues D. Littleton - Environmental Analyst A. West - Secretary	<b>Advanced Research</b> Marvin Singer - Senior Advisor
<b>Training &amp; Quality Coordination Team</b> TBD	<b>Communications Team</b> R. Porter - Team Leader M. Biggerstaff - Program Analyst H. Wolfe - Technical Writer E. Hebron - Program Analyst J. Pryor - Management Assistant R. Ladesic - Information Resource Management M. Walsh - LAN Maintenance P. Dickinson - Systems Improvement R. Carter - Secretary	<b>International Issues &amp; Activities Team</b> TBD - Team Leader J. Neilson - International Coordination A. Dupont-Ewing - International Marketing of Technology R. Lynch - International Coordination

Coal and Power Systems	Coal Fuels & Industrial Systems	Import and Export Activities
<b>G. Rudins - Deputy Assistant Secretary</b> G. Kight - Sr. Financial/Procurement Director J. Lerch - Program Analyst F. Cline - Program Analyst G. Gordon - Secretary	L. Miller - Product Line Director E. Schmetz - Portfolio Mgr., Liquefaction W. Fernald - Portfolio Mgr., Industrial & Solid/Slurry J. Shen - Program Mgr., Indirect Liquefaction T. Simpson - Program Mgr., Direct Liquefaction P. Hamill - Program Mgr., Environmental & NEPA D. Archer - Program Mgr., Industrial Systems G. Benjamin - Program Analyst M. Powell - Program Assistant	F. Glaser - Environmental/Exploratory J. Shang - AR&TD/SBIR T. Summers, Program Assistant
<b>Power Systems</b> V. Der - Product Line Director S. Biondo - Portfolio Mgr., Natural Gas Systems G. Lynch - Portfolio Mgr., Gasification Systems R. Wright - Portfolio Mgr., Combustion Systems C. Kinney - Program Mgr., Advanced Turbines E. Beyma - Program Mgr., Fuel Cells R. Travers - Program Mgr., Pressurized Fluidized Bed M. Ortner - Engineer/Power Systems P. Davies - Program Assistant T. Michalski - Program Assistant	<b>Environmental Systems</b> D. Beccy - Product Line Director L. Saroff - Program Mgr., Air Toxics R. Pennington - Program Mgr., Environmental Systems F. Ferrell - Program Mgr., Waste Mgmt J. Summers - Program Mgr., PM2.5 E. Williams - Program Assistant G. Garner - Program Assistant	<b>Import and Export Activities</b> B. McKee - Director M. Greenbaum - Team Leader, Int'l Coordination T. Atwood - Int'l Trade & Finance J. Pell - Int'l Environmental Trade K. Hong - Int'l Trade Coordination T. Durdock - Program Assistant
	<b>Advanced Research</b> Howard Feibus - Director P. Muchunas - Advanced Concepts	<b>Planning &amp; Environmental Analysis</b> D. Carter - Team Leader T. Grahame - Policy and Environmental S. Clayton - Strategic Planning & Analysis J. Temchin - Technical and Scientific R. Dye - Environmental Analysis T. Scott - Program Assistant

Federal Energy Technology Center

February 18, 1997  
**DRAFT**



## CLEAN COAL TECHNOLOGY PROGRAM (CCT-3)

## PROJECT TEAM (04-97)

SPONSOR: Air Products Liquid Phase Conversion Company, L.P. (ID: 3K)  
 Air Products and Chemicals, Inc. (APCI) and Eastman Chemical Company (EMN)

ADDRESSES: Air Products and Chemicals, Inc. U.S. Department of Energy  
 7201 Hamilton Boulevard Federal Energy Technology Center  
 Allentown, PA 18195-1501 P.O. Box 10940  
 Eastman Chemical Company Building 920, Room 119  
 P.O. Box 511 626 Cochran Mill Road  
 201 South Wilcox Drive Pittsburgh, PA 15236-0940  
 Kingsport, TN 37662-5231

PROJECT: "Commercial-Scale Demonstration of the Liquid Phase Methanol (LPMEOH™) Process"

SITE: Kingsport, Sullivan County, Tennessee

		Phone Number	Fax
PETC Project Manager:	Robert M. Kornosky	412/892-4521	412/892-4775
APCI Program Manager:	Edward C. Heydorn	610/481-7099	610/706-7299 (A)
EMN Program Manager:	William C. Jones	423/229-2901	423/224-0364
APCI Operational Program Manager:	Edward C. Heydorn	423/229-2816	423/224-7674 (K)
EMN Operational Program Manager:	Barry T. Street	423/229-6062	423/224-7268
High-Level Participant Contact:	Peter J.A. Tijm	610/481-0513	610/706-7299
PETC Contract Specialist:	William R. Mundorf	412/892-4483	412/892-6216
APCI Contract Specialist:	Barry J. Halper	610/481-7685	610/481-2576
EMN Contract Specialist:	Gary R. Whitaker	423/229-8529	423/229-4137
PETC Legal Counsel:	Thomas J. Russial	412/892-4630	412/892-5949
APCI Legal Counsel:	Willard Jones, II	610/481-4587	610/481-8223
EMN Legal Counsel:	Gary R. Whitaker	423/229-8529	423/229-4137
PETC Environmental Coordinator:	Richard A. Hargis	412/892-6065	412/892-6204
APCI Environmental Coordinator:	Frank S. Frenduto	610/481-7857	610/481-5833
EMN Environmental Coordinator:	Barry T. Street	423/229-6062	423/224-7268
PETC Cost/Price Analyst:	John J. Wehner	412/892-4738	412/892-6216
APCI Cost/Price Analyst:	Susan J. Kasinecz	610/481-2658	610/481-8949
EMN Cost/Price Analyst:	Ron Jelle'y	423/229-2564	423/224-7386
PETC Patents Support (CH):	Robert J. Fisher	630/252-2176	630/252-2779
APCI Patents Support:	Willard Jones, II	610/481-4587	610/481-8223
EMN Patents Support:	Fred Thomsen	423/229-2282	423/229-1239
PETC Public Relations:	Patrice A. Leister	412/892-6126	412/892-6127
APCI Public Relations:	Greta C. Campbell	610/481-4986	610/481-6642
EMN Public Relations:	Thomas A. Dickens	423/229-3149	423/229-1008
PETC Business/Financial Advisor:	John C. McDowell	412/892-6237	412/892-4775
APCI Business/Financial Advisor:	Robert B. Moore	610/481-7513	610/481-5833
EMN Business/Financial Advisor:	William M. Fortenberry	423/229-2512	423/224-0648
PETC Technical Analyst:	William J. O'Dowd	412/892-4778	412/892-4775
APCI Technical Analyst:	Van Eric Stein	423/229-2841	423/224-7674 (K)
EMN Technical Analyst:	Barry T. Street	423/229-6062	423/224-7268
FE HQ Program Manager:	John Shen	301/903-4344	301/903-2406
FE HQ Portfolio Manager:	Edward Schmetz	301/903-3931	301/903-2406

April 4, 1997

LIQUID PHASE METHANOL (LPMBOH™) DEMONSTRATION PROJECT  
FETC/AIR PRODUCTS LIQUID PHASE CONVERSION COMPANY, L.P. CONTACTS

Name: Title: Address:	Stanley M. Morris Vice President, Technology Air Products and Chemicals, Inc. 7201 Hamilton Boulevard Allentown, PA 18195-1501	E.C. Horton Vice President and General Manager Industrial Intermediates Business Organization Eastman Chemical Company P.O. Box 431 Kingsport, TN 37662-5280
Telephone:  FAX:  E-Mail:	(412) 892-6122 - PGH (304) 285-3469 - MGN  (412) 892-6127 - PGH (304) 285-4408 - MGN  nbajur@fetc.doe.gov	(610) 481-8282  (610) 481-7009  morrism@apci.com
Telephone:  FAX:  E-Mail:	Thomas M. Tokos Associate Director Office of Project Management U.S. Department of Energy Federal Energy Technology Center <sup>1</sup>  (412) 892-6123 (412) 892-6127  tokos@fetc.doe.gov	Peter J.A. Tijm Manager, Syngas Conversion Systems Air Products and Chemicals, Inc. 7201 Hamilton Boulevard Allentown, PA 18195-1501  (610) 481-0531 (610) 706-7299  tijm@apci.com
Telephone:  FAX:  E-Mail:	Thomas A. Satus Director Major Projects & Agreements Division U.S. Department of Energy Federal Energy Technology Center <sup>2</sup>  (412) 892-5981 (412) 891-4775  satus@fetc.doe.gov	Peter J.A. Tijm Manager, Syngas Conversion Systems Air Products and Chemicals, Inc. 7201 Hamilton Boulevard Allentown, PA 18195-1501  (610) 481-0531 (610) 706-7299  tijm@apci.com
Telephone:  FAX:  E-Mail:	William C. Jones Senior Development Associate Eastman Chemical Company P.O. Box 511 Kingsport, TN 37662-5235-1  (423) 229-2901 (423) 224-0364  jwcmj@apci.com	

<sup>1</sup> 3610 Collins Ferry Road  
P.O. Box 380  
Morgantown, WV 26507-0080

<sup>2</sup> 626 Cochran's Mill Road  
P.O. Box 10940  
Pittsburgh, PA 15236-0940

WFB 3/10

# KINGSPORT LPMEOH PROJECT

## CONSTRUCTION STATUS - 17 MARCH 1997

### MECHANICAL AND ELECTRICAL CONTRACTS

- Complete in February 97

### INSULATION

- Complete March 17, 1997

### START-UP SUPPORT

- Complete March 14, 1997

### REMAINING WORK

- Clean-up construction trailer area-- April 97
- Complete Painting--April 97
- Complete Paving--April 97
- Review and complete Punch List Items-- April 97
- Provide as built P&IDs and Electrical Drawings

### DOE REPORTS

- Complete Public Design Report
- Provide Reactor Topical Report

✓ In April  
OPD - to do. ~ When?

uRB's 3/19

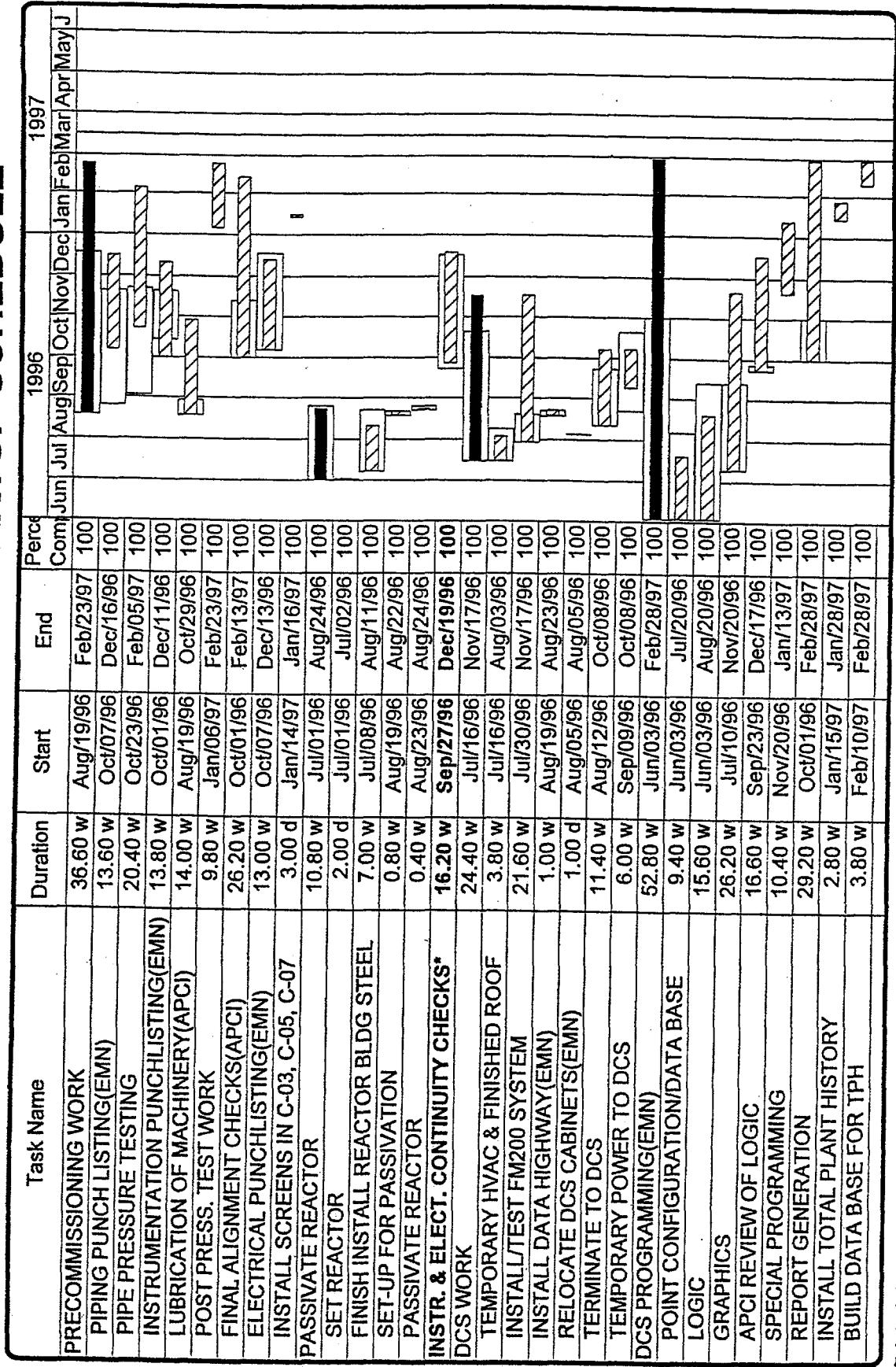
COST-397.XLS

**LPMEOH DEMONSTRATION PROJECT**  
**CURRENT SPENDING THRU 1/31/97**

		Cost Plan 10/22/96	Current Forecast	Spent to Date	% Spent
<b>PHASE 1</b>					
1.1.1	Project Definition	1,051	1,051	1,051	100
1.1.2	Permitting	238	257	257	100
1.1.3	Design Engineering	11,335	11,200	10,618	95
1.1.4	Off Site Testing	276	276	17	6
1.1.5	Planning, Admin & DME DVT	2,870	3,028	3,028	100
	Sub total	15,770	15,812	14,971	95
<b>PHASE 2</b>					
1.2.1	Procurement	9,703	10,130	9,994	99
1.2.2	Construction	11,550	11,550	10,614	92
1.2.3	Train & Commissioning	1,115	1,000	402	40
1.2.4	Off-Site Testing	256	256	0	0
1.2.5	Planning & Admin.	1,015	1,129	909	81
	Subtotal	23,639	24,065	21,919	91
<b>PHASE 3</b>					
1.3.1	Startup	680	680	99	15
1.3.2.1	Methanol Operation	146,485	146,021	397	0
1.3.2.2	Methanol Design Mod. Oper.	1,790	1,790	0	0
1.3.2.3	LPMEOH Dismantlement	515	515	0	0
1.3.3	On-Site Product Use Demo	4	4	0	0
1.3.4	Off Site Product Use Demo	3,451	3,451	0	0
1.3.5	Data Analysis & Reports	2,620	2,670	0	0
1.3.6	Planning & Admin.	2,392	2,392	11	0
	Subtotal	157,937	157,523	507	0
XXXXXX	Costs Prior to Mod 2	16,304 16,289	16,304	16,304	100
	<b>GRAND TOTAL</b>	<b>213,635</b>	<b>213,704</b>	<b>53,701</b>	<b>25</b>
	Total Phase 1&2	39,409	39,877	36,890	

NOTES: All dollar values in thousands; Forecast and Spending data updated from Jan. 97 Cost Management Report  
 Spending to date is actual not committed

# LPMEOH COMMISSIONING & STARTUP SCHEDULE

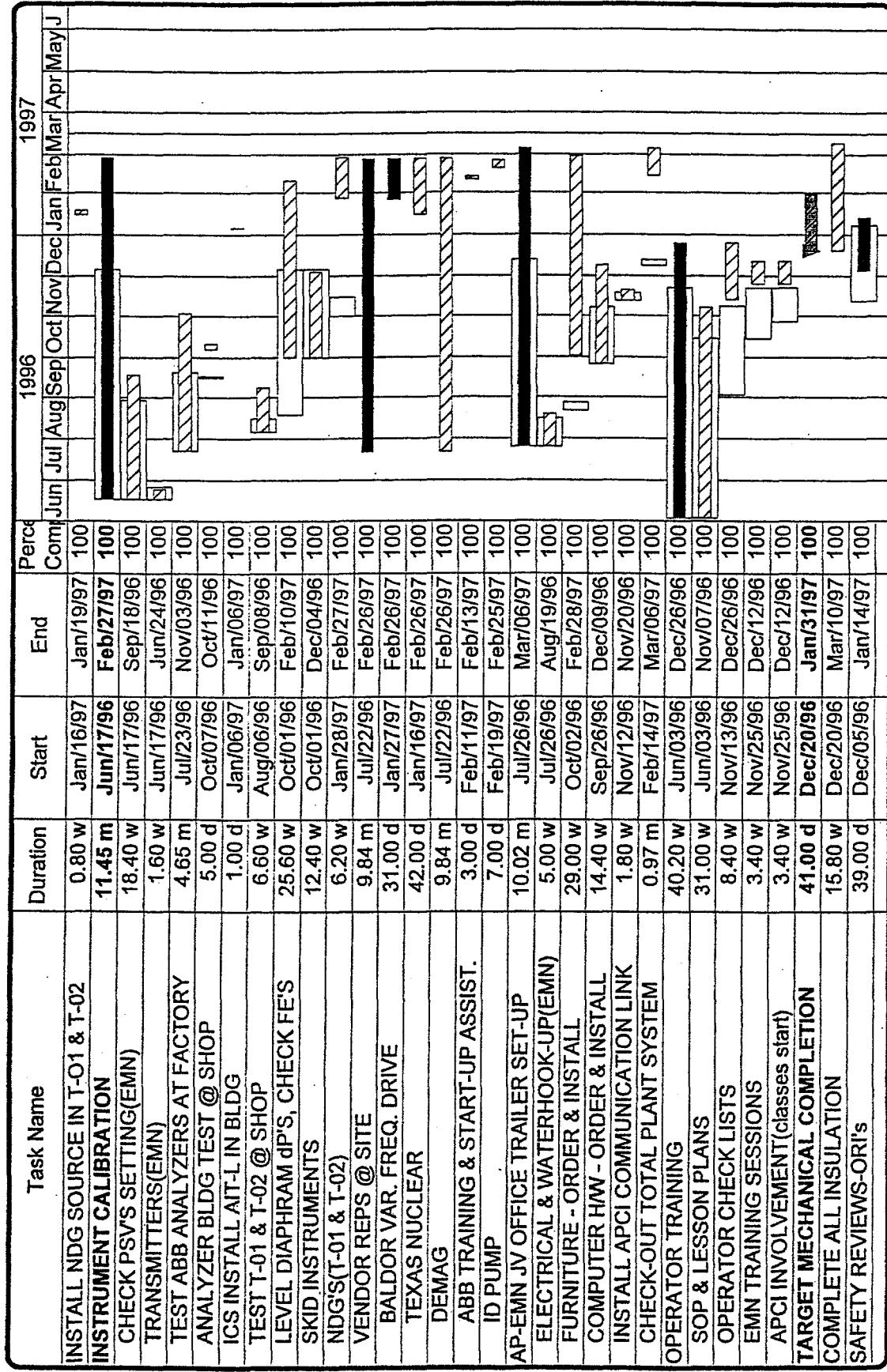


Printed: Mar/17/97  
Page 1

Milestone Summary

Attach (3)

# LPMEOH COMMISSIONING & STARTUP SCHEDULE



Printed: Mar/17/97  
Page 2

Attach(3) - 2

Milestone Delta Summary

# LPMEOH COMMISSIONING & STARTUP SCHEDULE

Task Name	Duration	Start	End	Percent Complete	1997												
					Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	
MECHANICAL	2.00 d	Dec/05/96	Dec/06/96	100													
PRESTART-UP	1.00 d	Jan/14/97	Jan/14/97	100													
INSTRUMENT LOOP CHECKING**	27.20 w	Oct/15/96	Mar/03/97	100													
ENERGIZE MCC	1.00 d	Oct/15/96	Oct/15/96	100													
EAST OF COLUMN "C"	9.60 w	Oct/22/96	Dec/10/96	100													
DISTILLATION AREA	7.80 w	Nov/04/96	Dec/14/96	100													
CATALYST PREP AREA	17.20 w	Dec/06/96	Mar/03/97	100													
REACTOR AREA	16.20 w	Dec/06/96	Feb/26/97	100													
ANALYZER SYSTEM CHECK-OUT(EMN)	10.20 w	Dec/23/96	Feb/13/97	100													
APCI FINAL FUNCTION TESTING	25.00 d	Jan/21/97	Feb/14/97	100													
POUR CONDUIT SEAL OFFS	14.20 w	Dec/09/96	Feb/19/97	100													
EAST OF COLUMN LINE "C"	7.80 w	Dec/09/96	Jan/18/97	100													
OTHER AREAS	10.00 w	Dec/31/96	Feb/19/97	100													
COMMISSIONING(PHASE 2, TASK 3)	28.40 w	Oct/16/96	Mar/10/97	100													
COMMISSION INSTR. AIR HEADERS	9.60 w	Oct/16/96	Dec/04/96	100													
EAST OF COLUMN "C"(incl. V-02)	4.00 d	Oct/16/96	Oct/19/96	100													
REMAINING PLANT AREAS	3.20 w	Nov/18/96	Dec/04/96	100													
COMMISSION OTHER UTILITIES	42.00 d	Jan/15/97	Feb/25/97	100													
PLANT AIR	22.00 d	Jan/21/97	Feb/11/97	100													
NITROGEN	1.00 d	Jan/30/97	Jan/30/97	100													
STEAM & CONDENSATE	32.00 d	Jan/24/97	Feb/24/97	100													
100 PSIG SYSTEM	1.00 d	Jan/24/97	Jan/24/97	100													
STEAM TRACING	14.00 d	Jan/27/97	Feb/09/97	100													
600 PSIG STEAM	17.00 d	Feb/08/97	Feb/24/97	100													
COOLING WATER	2.00 d	Feb/05/97	Feb/06/97	100													
CITY WATER	1.00 d	Jan/26/97	Jan/26/97	100													
RIVER WATER	1.00 d	Jan/15/97	Jan/15/97	100													
BOILER FEED WATER	2.00 d	Feb/24/97	Feb/25/97	100													
SYSTEM CLEANING	15.00 d	Jan/29/97	Feb/12/97	100													
CONNECT & CLEAN OUT C-50	2.00 d	Jan/29/97	Jan/30/97	100													
C-40 CATALYST GUARD BED	1.00 d	Jan/31/97	Jan/31/97	100													

# LPMEOH COMMISSIONING & STARTUP SCHEDULE

Task Name	Duration	Start	End	1997												
				Perce Com	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May
D-02 & D-30	1.00 d	Jan/31/97	Jan/31/97	100												
METHANOL STORAGE AREA	9.00 d	Feb/04/97	Feb/12/97	100												
DISTILLATION AREA	3.00 d	Feb/01/97	Feb/03/97	100												
LOAD CATALYST GUARD BED(EMN)***	3.00 d	Feb/01/97	Feb/05/97	100												
FILL D-30 WITH MINERAL OIL	5.00 d	Feb/06/97	Feb/10/97	100												
DRAKEOL SYSTEM START-UP	20.00 d	Feb/07/97	Feb/26/97	100												
FUNCTIONAL CHECK-OUT	13.00 d	Feb/07/97	Feb/19/97	100												
TEST RUN ID PUMPS	14.00 d	Feb/12/97	Feb/25/97	100												
TRANSFER THRU SLURRY LINES	12.00 d	Feb/15/97	Feb/26/97	100												
PUMP AROUND DISTILLATION	3.00 d	Mar/03/97	Mar/10/97	100												
K-01 LUBE OIL FLUSH(EMN)	1.00 w	Nov/18/96	Nov/22/96	100												
RESET K-01 MOTOR & ALIGN	1.00 w	Dec/16/96	Dec/20/96	100												
TURNOVER MCC & I/O BLDG.	18.00 d	Jan/26/97	Feb/12/97	100												
ANALYZER CALIBRATION(EMN)	3.00 d	Feb/11/97	Feb/13/97	100												
TEST & ACTIVATE FIRE SPRINKLER DELUGE	7.00 d	Feb/07/97	Feb/13/97	100												
EMN FUNCTIONAL CHECK-OUT	9.00 w	Jan/25/97	Mar/10/97	100												
UTILITIES	6.40 w	Jan/26/97	Feb/26/97	100												
OIL SYSTEM	4.80 w	Jan/25/97	Feb/17/97	100												
CATALYST PREPARATION	6.40 w	Jan/28/97	Feb/28/97	100												
DISTILLATION	7.00 w	Feb/04/97	Mar/10/97	100												
RUN REACTOR T-01 NDG HOIST	14.00 d	Feb/14/97	Feb/27/97	100												
RUN REDUCTION T-02 NDG HOIST	11.00 d	Feb/15/97	Feb/25/97	100												
OPEN-UP I.T. SEWER CONNECTION	1.00 d	Jan/31/97	Jan/31/97	100												
OPEN BIL FENCE GATE	1.00 d	Feb/25/97	Feb/25/97	100												
TAKE DOWN FENCE	3.00 d	Feb/25/97	Feb/27/97	100												
PLANT START-UP(PHASE 3-TASK 1)	63.00 d	Jan/30/97	Apr/02/97	86												
K-01 SYNGAS COMPRESSOR STARTUP	15.00 d	Jan/30/97	Feb/13/97	100												
2 HOUR RUN ON MOTOR	1.00 d	Jan/30/97	Jan/30/97	100												
CONNECT PIPING & FINAL ALIGNMENT	2.00 d	Feb/04/97	Feb/05/97	100												
FINAL SET-UP, PURGE PIPING	8.00 d	Feb/05/97	Feb/12/97	100												
RUN ON NITROGEN	1.00 d	Feb/13/97	Feb/13/97	100												

Printed: Mar/17/97  
Page 4

Milestone  Summary 

Attach (3) - 4



## LPDME Design Verification Testing.

From the Statement of Work: "Subject to Design Verification Testing (DVT), the Partnership proposes to enhance the Project by including the demonstration of the slurry reactor's capability to produce DME as a mixed co-product with methanol. The production of DME from synthesis gas is a natural extension of the LPMEOH™ process in that three reactions occur concurrently in a single liquid phase reactor, methanol synthesis, methanol dehydration and water-gas shift. This process enhancement can significantly improve the overall conversion of coal derived synthesis gas to a storable blend of methanol and DME. ----- the enhanced DME production demonstration is complementary to ongoing studies being sponsored by DOE's Liquid Fuels Program -- . -- At the conclusion of each of the DVT steps, a joint Partnership/DOE decision will be made regarding continuation of methanol/DME demonstration.."

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

### The LPDME Process Concept: - Three Concurrent Reactions:

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

The overall reaction, with CO-rich Syngas, in a single liquid phase (slurry) reactor:

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

This is the "once-through" CO-rich syngas concept for the LPDME process utilizing a single slurry reactor. Conversion per pass, with CO-rich syngas, can be higher than for the LPMEOH™ process. Methanol may also be produced, as a mixed co-product with the DME, and can easily be separated and recovered. The separation of DME from CO<sub>2</sub> will be necessary for certain market applications.

### Status of the LPDME DVT Work

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

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

- Aerosol - Small, but established market. High purity DME is required.
- Cooking Fuel. Potentially a large market, to replace imported LPG. There is a lot of interest in China, and DME is on the DOE's CCT/FE China meeting (Sept. of 1997) agenda. Purity, of

about >95% DME, with <2% methanol, < 3% CO<sub>2</sub> is estimated. An unresolved application issue is CO emissions during cooking. How does DME purity impact this? Use testing is needed.

- Diesel Replacement Fuel. DME is an ultra clean (high Cetane) diesel fuel; and an 80% DME mixture with methanol and water is now being engine-tested by others (Amoco, et. al.). Market development (at least in the U.S.) faces a fuel distribution infrastructure problem. DME might more easily replace LPG in countries where LPG is already an engine fuel.
- DME Derivatives, as a Diesel Fuel Additive. Quotes from the DOE (Alt. Fuels R&D) Program quarterly report for April-June 1996: "Initial Cetane number (CN) testing of a three-component composition of 1,2-dimethoxy ethane, 1,1-dimethoxy methane and methanol blended with diesel fuel showed a 40% increase in the CN of the diesel fuel when the blend was 50/50." "The concept of adding a blend of oxygenated compounds to diesel fuel in order to enhance the Cetane value and cold start properties is being investigated. The blend of oxygenated compounds is derived from dimethyl ether chemistry, and builds on work conducted earlier --." It is early days for this DME feedstock chemistry, but CO<sub>2</sub> may not need to be separated from the DME.
- DME Derivatives, as Chemicals/Other Fuels. DME is a key intermediate in a commercial synthesis gas-to-gasoline process, and is being developed as an intermediate for other chemicals and fuels as part of the DOE's Liquid Fuels (Alt. Fuels R&D) Program.

VCP

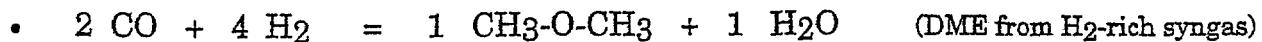
DME →

Ethylene  
Propylene

A. The economics studies, for once-through coproduction (with an IGCC power plant for example) on synthesis gas rich in carbon oxides, show that the LPDME process will have an advantage greater than the LPMEOH™ process. A once-through LPDME reactor is able to convert greater than 50% of such a syngas, whereas a once-through LPMEOH™ reactor can convert only about 30%. The economics, of course, depend upon the end-use (purity) of the DME and upon the gasification plant's coproduct mix (amount of power, methanol, DME, etc.). The same liquid phase reactor design options to increase syngas conversion (see the CCT Tampa Conference Paper); such as feed gas compression and/or CO-rich gas recycle; are also be applicable for LPDME. So, the LPDME technology should have a significant advantage for the coproduction of DME to serve local markets.

As with the LPMEOH™ process, gas phase process technology must be considered as the economic competitor. The gas phase DME process (see Haldor's patent) must run with H<sub>2</sub>-rich syngas. In the IGCC coproduction flow sheet, gas phase technology is at an economic disadvantage, since separate shift and CO<sub>2</sub> removal are required. As is the case for methanol, inexpensive remote natural gas would therefore be the economic plant site choice for gas phase technology. A comparison, of IGCC/LPDME coproduction with DME imported from remote gas facilities, shows an advantage for locally produced DME relative to imported DME. The transportation cost to import DME is much higher than for methanol, and the LPDME coproduction advantage is even greater than that for LPMEOH™ (vs. methanol import; see the CCT Tampa Conference Paper). Dehydration of imported methanol to make DME is not competitive either. Therefore, for DME in local markets, LPDME coproduction should be a winner!

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



Since water inhibits the methanol dehydration reaction, the slurry reactor must be staged, with water removal between stages. Staging could be by high ratio gas recycle, and/or with multiple reactors; but the once-through simplicity is lost. Therefore, it is unlikely that the LPDME process would be developed for use in H<sub>2</sub>-rich syngas applications.

**B. Laboratory R&D Results. (CCT Project - ended 9/96).**

An LPDME catalyst system, with reasonable long-term activity, was identified and tested. The system exhibits best activity under CO-rich syngas conditions, i.e. those most likely for (IGCC) coproduction. Accelerated aging of the catalyst system is a remaining issue. Water concentrations in the LP reactor are higher with syngases richer in H<sub>2</sub>, and its effect needs to be evaluated.

Lab work has continued under the DOE's Liquid Fuels Program. The issues, to be addressed in the lab before a LaPorte test-run decision, are: 1. Understanding the LPDME catalyst system's accelerated aging; and modifying the catalyst and/or the system operating conditions. 2. Manufacturing scale-up of catalyst for a LaPorte run.

**Recommendations**

The catalyst system and the market applications/opportunities are sufficiently promising that proof-of-concept testing at the LaPorte AFDU is recommended. Kingsport is an unlikely site for the commercial size demonstration of LPDME, since there are limited times for CO-rich syngas testing; and H<sub>2</sub>-rich syngas would create water buildup. Therefore, the LaPorte AFDU test-run, hydrodynamic programs, and data from the scaleup of the LPMEOH™ reactor; must provide the basis for commercializing LPDME. Recommendations:

- An LPDME test run at the LaPorte AFDU, in conjunction with the DOE's Liquid Fuels Program, would be appropriate if the LPDME catalyst system development can be completed successfully. Up to \$875,000 of CCT Program budget support, from the LPMEOH™ Project's FY-97 Cost Plan (budget), should be made available to support a suitable LPDME test run at LaPorte.
- An implementation decision, made mutually by the DOE's CCT (DE-FC22-92PC90543) LPMEOH™ project participants, and by the DOE's Liquid Fuels (DE-FC22-95PC93052) program participants, should be made (by July of 1997) in time to implement testing at LaPorte in early 1998. (*Final dates should be recommended by the DOE's Liquid Fuels program, based on progress in developing the LPDME catalyst system.*) The DOE LPMEOH™ project participants should be kept informed of the LaPorte AFDU LPDME test-run plans, so that a timely final approval can be made.
- In the interim, some DME product-use testing may be appropriate for the LPMEOH™ Project's Off-site Product testing.

(end).

Appendix A.

Why combine the CCT and Liquid Fuels Programs for an LPDME test-run at the LaPorte AFDU?

**The programs are related.** The DOE Liquid Fuels Program has as one of its objectives: to investigate potential technologies for the conversion of syngas to oxygenated and hydrocarbon fuels and industrial chemicals; and to demonstrate the most promising at the LaPorte AFDU. Three slurry reactor programs are at the LaPorte AFDU demonstration stage:

1. Syngas to Fischer-Tropsch (F-T) liquids. A test-run at LaPorte in October of 1996 was partially successful, but terminated early. Analysis is underway, and a recommendation for an additional test run at LaPorte has been made. Air Products and Shell are participants.
2. Syngas to DME. An earlier (Liquid Fuels Program) LPDME test run at LaPorte showed good promise. DME is an important intermediate chemical building block for many of the Liquid Fuels Program's promising ideas. Development of a stable and active LPDME catalyst system is therefore an important part of this program.
3. Slurry Reactor Hydrodynamics. The LaPorte AFDU is the ultimate test unit to confirm laboratory (University, National Labs, Other) hydrodynamic studies. A test, planned for October of 1996 at LaPorte, was only partially executed due to the early termination of the F-T run. This needs to be rescheduled.

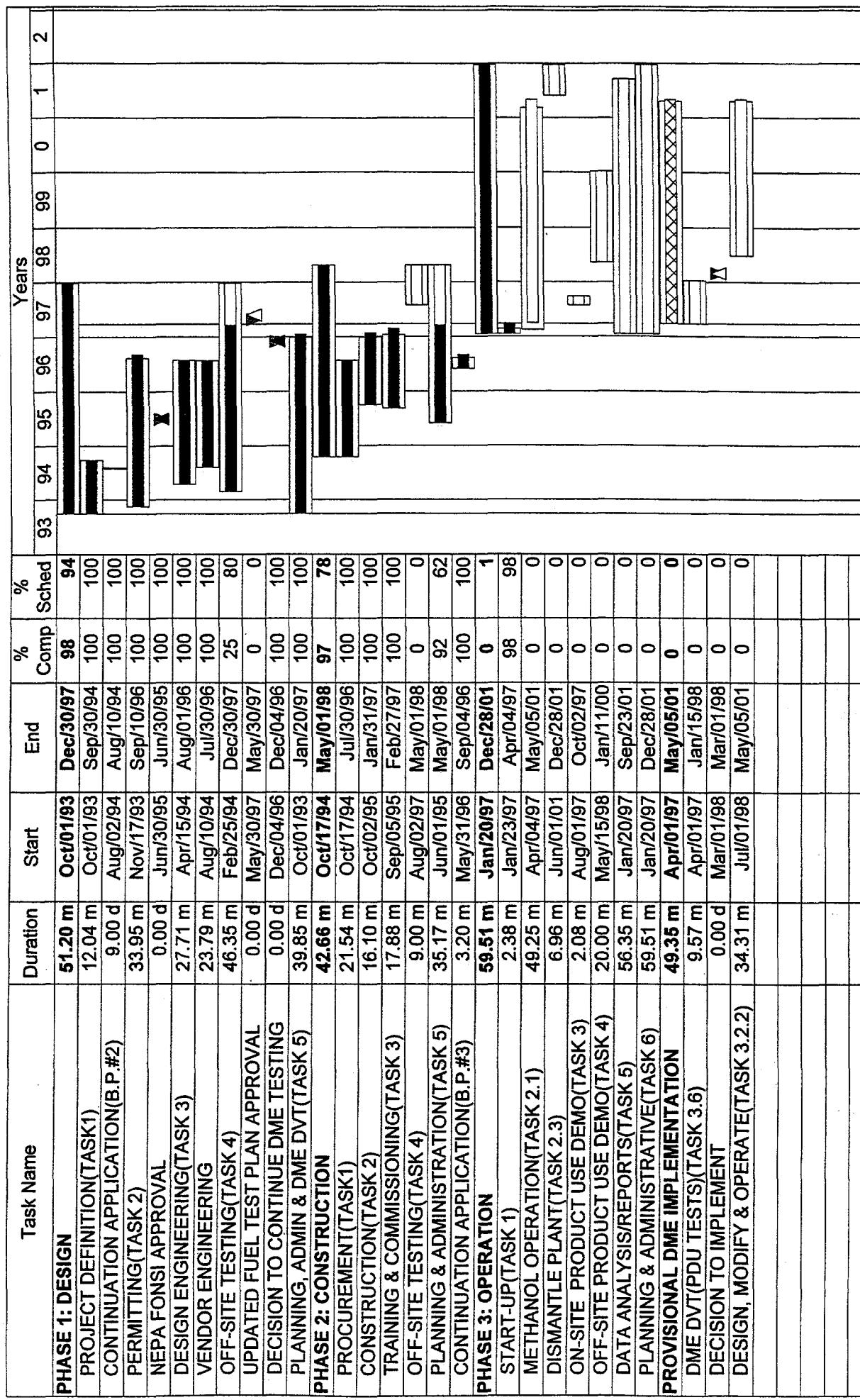
**Budgets are limited.** The DOE Liquid Fuels Program budget is limited. By combining the Liquid Fuels and the CCT Program budgets, the LPDME test-run at LaPorte can be made more extensive, and the CCT Program participants can provide experience from the Kingsport scaleup/demonstration to help in developing test-run plans.

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

# MILESTONE SCHEDULE STATUS REPORT

## LIQUID PHASE METHANOL DEMONSTRATION

### DE-FC22-92PC90543



U.S. DEPARTMENT OF ENERGY  
**COST MANAGEMENT REPORT**

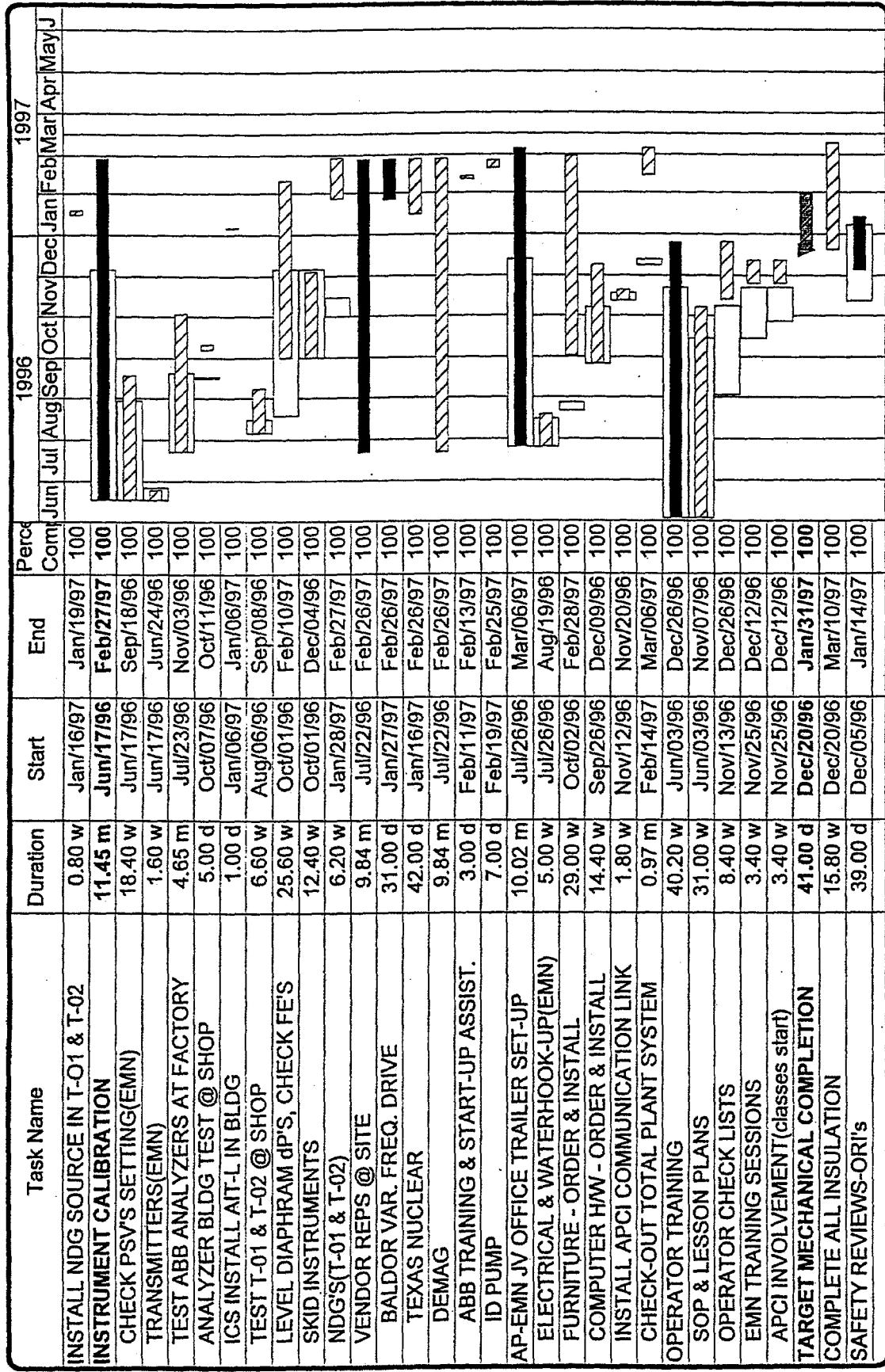
Form 1710-440												
1. TITLE Liquid Phase Methanol Demonstration		2. REPORTING PERIOD March 01, 1997 through March 31, 1997										
2. PARTICIPANT NAME AND ADDRESS Air Products Liquid Phase Conversion Co., L.P. 7201 Hamilton Boulevard Allentown, PA 18195-4911		3. IDENTIFICATION NUMBER DE-FC22-92PC90543										
4. ELEMENT 9. REPORTING ELEMENT		5. COST PLAN DATE October 22, 1996										
8. ELEMENT 10. ACCRUED COSTS		6. START DATE January 1, 1990										
		Reporting Period		Cumulative to Date		11. ESTIMATED ACCRUED COSTS		12. Total Contract Value		13. Variance		
		a. Actual	b. Plan	c. Actual	d. Plan	a. Subsequent Report- ing Period	b. Balance of Fiscal Year	c. FY 1998 (1)	d. FY 2000 (2)	e. FY 2000 (3)	f. Subse- quent FY's (4)	
Prior to Mod 2		0	0	16,304	16,289	0	0				16,304	
Project Definition		(10)	0	1,041	1,021	0	0	0	0	0	1,041	
Permitting		(7)	0	255	246	0	0	0	0	0	255	
Design Engr.		34	160	10,730	10,684	160	448	0	0	0	11,338	
Off-site Testing		3	15	26	119	15	160	75	0	0	276	
Planning, Admin. & DME Verif. Testing		(25)	0	2,996	2,055	0	0	0	0	0	2,996	
Procurement		44	0	10,102	9,000	224	0	0	0	0	10,326	
Construction		327	30	11,502	13,443	53	0	0	0	0	11,555	
Train. & Commissioning		(0)	0	586	2,162	0	0	0	0	0	586	
Off-Site Test - Proc. & Constr.		0	0	0	0	0	120	136	0	0	256	
Planning & Admin		53	10	971	813	50	50	0	0	0	1,071	
Startup		588	0	925	680	250	0	0	0	0	1,175	
Operations											3,495	
1.3.2.1. Methanol Operation		0	2,524	397	5,048	2,545	17,051	34,240	38,659	40,061	12,720	
1.3.2.2. DME Design, Mod., Oper.		0	0	0	0	0	0	0	567	1,223	0	
1.3.2.3. LPMEOH Dismantlement		0	0	0	0	0	0	0	0	515	515	
1.3.3. On-Site Product Use Demo		0	0	0	0	2	0	2	0	0	0	
1.3.4. Off-Site Product Use Demo		0	0	0	0	0	0	1,233	1,972	246	0	
Data Analysis & Reports		0	13	0	26	61	329	520	500	553	707	
Planning & Admin.		61	20	163	40	50	307	800	280	282	510	
14. TOTAL		1,068	2,772	55,998	61,628	3,408	18,467	37,006	42,008	42,365	14,452	
15. DOLLARS EXPRESSED IN:												213,700
Thousands												213,700
16. SIGNATURE OF PARTICIPANT'S PROJECT MANAGER AND DATE		17. SIGNATURE OF PARTICIPANT'S AUTHORIZED FINANCIAL REPRESENTATIVE AND DATE										
J. D. [Signature]		J. J. Kastner [Signature]										
DATE		DATE										
4/30/97		4/30/97										

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

# LPMEOH COMMISSIONING & STARTUP SCHEDULE

Task Name	Duration	Start	End	1996												1997												
				Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	
PRECOMMISSIONING WORK	36.60 w	Aug/19/96	Feb/23/97	100																								
PIPING PUNCHLISTING(EMN)	13.60 w	Oct/07/96	Dec/16/96	100																								
PIPE PRESSURE TESTING	20.40 w	Oct/23/96	Feb/05/97	100																								
INSTRUMENTATION PUNCHLISTING(EMN)	13.80 w	Oct/01/96	Dec/11/96	100																								
LUBRICATION OF MACHINERY(APCI)	14.00 w	Aug/19/96	Oct/29/96	100																								
POST PRESS. TEST WORK	9.80 w	Jan/06/97	Feb/23/97	100																								
FINAL ALIGNMENT CHECKS(APCI)	26.20 w	Oct/01/96	Feb/13/97	100																								
ELECTRICAL PUNCHLISTING(EMN)	13.00 w	Oct/07/96	Dec/13/96	100																								
INSTALL SCREENS IN C-03, C-05, C-07	3.00 d	Jan/14/97	Jan/16/97	100																								
PASSIVATE REACTOR	10.80 w	Jul/01/96	Aug/24/96	100																								
SET REACTOR	2.00 d	Jul/01/96	Jul/02/96	100																								
FINISH INSTALL REACTOR BLDG STEEL	7.00 w	Jul/08/96	Aug/1/96	100																								
SET-UP FOR PASSIVATION	0.80 w	Aug/19/96	Aug/22/96	100																								
PASSIVATE REACTOR	0.40 w	Aug/23/96	Aug/24/96	100																								
INSTR. & ELECT. CONTINUITY CHECKS*	16.20 w	Sep/27/96	Dec/19/96	100																								
DCS WORK	24.40 w	Jul/16/96	Nov/17/96	100																								
TEMPORARY HVAC & FINISHED ROOF	3.80 w	Jul/16/96	Aug/03/96	100																								
INSTALL/TEST FM200 SYSTEM	21.60 w	Jul/30/96	Nov/17/96	100																								
INSTALL DATA HIGHWAY(EMN)	1.00 w	Aug/19/96	Aug/23/96	100																								
RELOCATE DCS CABINETS(EMN)	1.00 d	Aug/05/96	Aug/05/96	100																								
TERMINATE TO DCS	11.40 w	Aug/12/96	Oct/08/96	100																								
TEMPORARY POWER TO DCS	6.00 w	Sep/09/96	Oct/08/96	100																								
DCS PROGRAMMING(EMN)	52.80 w	Jun/03/96	Feb/28/97	100																								
POINT CONFIGURATION/DATA BASE	9.40 w	Jun/03/96	Jul/20/96	100																								
LOGIC	15.60 w	Jun/03/96	Aug/20/96	100																								
GRAPHICS	26.20 w	Jul/10/96	Nov/20/96	100																								
APCI REVIEW OF LOGIC	16.60 w	Sep/23/96	Dec/17/96	100																								
SPECIAL PROGRAMMING	10.40 w	Nov/20/96	Jan/13/97	100																								
REPORT GENERATION	29.20 w	Oct/01/96	Feb/28/97	100																								
INSTALL TOTAL PLANT HISTORY	2.80 w	Jan/15/97	Jan/28/97	100																								
BUILD DATA BASE FOR TPH	3.80 w	Feb/10/97	Feb/28/97	100																								

# LPMEOH COMMISSIONING & STARTUP SCHEDULE



# LPMEOH COMMISSIONING & STARTUP SCHEDULE

Task Name	Duration	Start	End	Percent Complete												
				Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	
MECHANICAL	2.00 d	Dec/05/96	Dec/06/96	100												
PRESTART-UP	1.00 d	Jan/14/97	Jan/14/97	100												
<b>INSTRUMENT LOOP CHECKING**</b>	<b>27.20 w</b>	<b>Oct/15/96</b>	<b>Mar/03/97</b>	<b>100</b>												
ENERGIZE MCC	1.00 d	Oct/15/96	Oct/15/96	100												
EAST OF COLUMN "C"	9.60 w	Oct/22/96	Dec/10/96	100												
DISTILLATION AREA	7.80 w	Nov/04/96	Dec/14/96	100												
CATALYST PREP AREA	17.20 w	Dec/06/96	Mar/03/97	100												
REACTOR AREA	16.20 w	Dec/06/96	Feb/26/97	100												
ANALYZER SYSTEM CHECK-OUT(EMN)	10.20 w	Dec/23/96	Feb/13/97	100												
APCI-FINAL FUNCTION TESTING	25.00 d	Jan/21/97	Feb/14/97	100												
POUR CONDUIT SEAL OFFS	14.20 w	Dec/09/96	Feb/19/97	100												
EAST OF COLUMN LINE "C"	7.80 w	Dec/09/96	Jan/18/97	100												
OTHER AREAS	10.00 w	Dec/31/96	Feb/19/97	100												
COMMISSIONING(PHASE 2, TASK 3)	28.40 w	Oct/16/96	Mar/10/97	100												
COMMISSION INSTR. AIR HEADERS	9.60 w	Oct/16/96	Dec/04/96	100												
EAST OF COLUMN "C"(Incl. V-02)	4.00 d	Oct/16/96	Oct/19/96	100												
REMAINING PLANT AREAS	3.20 w	Nov/18/96	Dec/04/96	100												
COMMISSION OTHER UTILITIES	42.00 d	Jan/15/97	Feb/25/97	100												
PLANT AIR	22.00 d	Jan/21/97	Feb/11/97	100												
NITROGEN	1.00 d	Jan/30/97	Jan/30/97	100												
STEAM & CONDENSATE	32.00 d	Jan/24/97	Feb/24/97	100												
① 100 PSIG SYSTEM	1.00 d	Jan/24/97	Jan/24/97	100												
STEAM TRACING	14.00 d	Jan/27/97	Feb/09/97	100												
600 PSIG STEAM	17.00 d	Feb/08/97	Feb/24/97	100												
COOLING WATER	2.00 d	Feb/05/97	Feb/06/97	100												
CITY WATER	1.00 d	Jan/26/97	Jan/26/97	100												
RIVER WATER	1.00 d	Jan/15/97	Jan/15/97	100												
BOILER FEED WATER	2.00 d	Feb/24/97	Feb/25/97	100												
SYSTEM CLEANING	15.00 d	Jan/29/97	Feb/12/97	100												
CONNECT & CLEAN OUT C-50	2.00 d	Jan/29/97	Jan/30/97	100												
C-40 CATALYST GUARD BED	1.00 d	Jan/31/97	Jan/31/97	100												

# LPMEOH COMMISSIONING & STARTUP SCHEDULE

Task Name	Duration	Start	End	Percent Complete												
				Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	
D-02 & D-30	1.00 d	Jan/31/97	Jan/31/97	100												
METHANOL STORAGE AREA	9.00 d	Feb/04/97	Feb/12/97	100												
DISTILLATION AREA	3.00 d	Feb/01/97	Feb/03/97	100												
LOAD CATALYST GUARD BED(EMN)***	3.00 d	Feb/03/97	Feb/05/97	100												
FILL D-30 WITH MINERAL OIL	5.00 d	Feb/06/97	Feb/10/97	100												
DRAKEOL SYSTEM START-UP	20.00 d	Feb/07/97	Feb/26/97	100												
FUNCTIONAL CHECK-OUT	13.00 d	Feb/07/97	Feb/19/97	100												
TEST RUN ID PUMPS	14.00 d	Feb/12/97	Feb/25/97	100												
TRANSFER THRU SLURRY LINES	12.00 d	Feb/15/97	Feb/26/97	100												
PUMP AROUND DISTILLATION	3.00 d	Mar/08/97	Mar/10/97	100												
K-01 LUBE OIL FLUSH(EMN)	1.00 w	Nov/18/96	Nov/22/96	100												
RESET K-01 MOTOR & ALIGN	1.00 w	Dec/16/96	Dec/20/96	100												
TURNOVER MCC & I/O BLDG.	18.00 d	Jan/26/97	Feb/12/97	100												
ANALYZER CALIBRATION(EMN)	3.00 d	Feb/11/97	Feb/13/97	100												
TEST & ACTIVATE FIRE SPRINKLER DELUGE	7.00 d	Feb/07/97	Feb/13/97	100												
EMN FUNCTIONAL CHECK-OUT	9.00 w	Jan/25/97	Mar/10/97	100												
UTILITIES	6.40 w	Jan/26/97	Feb/26/97	100												
OIL SYSTEM	4.80 w	Jan/25/97	Feb/17/97	100												
CATALYST PREPARATION	6.40 w	Jan/28/97	Feb/28/97	100												
DISTILLATION	7.00 w	Feb/04/97	Mar/10/97	100												
RUN REACTOR T-01 NDG HOIST	14.00 d	Feb/14/97	Feb/27/97	100												
RUN REDUCTION T-02 NDG HOIST	11.00 d	Feb/15/97	Feb/25/97	100												
OPEN-UP I.T. SEWER CONNECTION	1.00 d	Jan/31/97	Jan/31/97	100												
OPEN BIL FENCE GATE	1.00 d	Feb/25/97	Feb/25/97	100												
TAKE DOWN FENCE	3.00 d	Feb/25/97	Feb/27/97	100												
<b>PLANT START-UP(PHASE 3-TASK 1)</b>	<b>63.00 d</b>	<b>Jan/30/97</b>	<b>Apr/02/97</b>	<b>86</b>												
K-01 SYNGAS COMPRESSOR STARTUP	15.00 d	Jan/30/97	Feb/13/97	100												
2 HOUR RUN ON MOTOR	1.00 d	Jan/30/97	Jan/30/97	100												
CONNECT PIPING & FINAL ALIGNMENT	2.00 d	Feb/04/97	Feb/05/97	100												
FINAL SET-UP, PURGE PIPING	8.00 d	Feb/05/97	Feb/12/97	100												
RUN ON NITROGEN	1.00 d	Feb/13/97	Feb/13/97	100												

# LPMEOH COMMISSIONING & STARTUP SCHEDULE

Task Name	Duration	Start	End	1997													
				Perce	Com	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May
CARBONYL BURNOUT	35.00 d	Feb/08/97	Mar/14/97	100													
STEAM TRACING	18.00 d	Feb/08/97	Feb/25/97	100													
BFW/IN	17.00 d	Feb/09/97	Feb/25/97	100													
CHECK REACTOR SYSTEMS	19.00 d	Feb/08/97	Feb/26/97	100													
FILL C-01 WITH OIL	12.00 d	Feb/15/97	Feb/26/97	100													
K-01 COMPRESSOR READY	5.00 d	Feb/10/97	Feb/14/97	100													
(1) BRING IN SYNGAS	12.00 d	Feb/28/97	Mar/11/97	100													
TEST FOR CARBONYLS	9.00 d	Mar/04/97	Mar/12/97	100													
DRAIN OIL FROM C-01	4.00 d	Mar/11/97	Mar/14/97	100													
HOT OIL WASH IN C-30 & CHECK-OUT	17.00 d	Feb/26/97	Mar/14/97	100													
REDUCE CATALYST BATCHES	18.00 d	Mar/16/97	Apr/02/97	20													
START DISTILLATION	6.00 d	Mar/28/97	Apr/02/97	0													
BEGIN PLANT OPERATION(PHASE 3-TASK 2)	21) 6.00 w	Apr/05/97	May/04/97	0													
(3) DTR - TEST #1 - SHAKEDOWN	6.00 w	Apr/05/97	May/04/97	0													
* 340 manhour estimate	1.00 d	Jun/01/96	Jun/01/96	100													
** durations based on 3 men @ 50 hrs/wk	1.00 d	Jun/01/96	Jun/01/96	100													
*** start 3 shift operation	1.00 d	Jun/01/96	Jun/01/96	100													
APCI CONSTRUCTION START-UP SUPPORT	8.00 w	Feb/03/97	Mar/14/97	100													
ELECTRICAL-GOSS	8.00 w	Feb/03/97	Mar/14/97	100													
MECHANICAL-SPARTAN	8.00 w	Feb/03/97	Mar/14/97	100													
DAN ZIGLER	7.60 w	Feb/03/97	Mar/12/97	100													
F. FRENDUTO	6.60 w	Feb/03/97	Mar/07/97	100													

Printed: Mar/17/97  
Page 5

Milestone Summary

Attach (3) - 5

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

## Memorandum



To: Distribution Dept./Loc.:  
From: W. R. Brown Dept./Ext.: PSED/A31E9, X17584  
Date: 11 November 1996  
Subject: Partnership Annual Operating Plan for FY-97

Distribution: cc: R. M. Kornosky/DOE/PETC

D. P. Drown/APCI      L. B. Paulonis/EMN  
E. C. Heydorn/APCI      V. E. Stein/APCI  
W. C. Jones/EMN      P. J. A. Tijm/APCI  
R. B. Moore/APCI

## Background

The Partnership Agreement requires that an Annual Operating Plan be prepared each Fiscal Year for the approval of the Partners. Article 5.2 of the Partnership Agreement sets forth the requirements. This memo constitutes the Partnership's Annual Operating Plan for FY-'97.

## Goals and Objectives for FY- '97

The goals and objectives for FY-'97 are to initiate Phase 3 operation of the LPMEOH™ demonstration plant in accordance with the Statement of Work. The Milestone Schedule (Attachment A), the Demonstration Test Plan (Attachment B), the FY-97 Cost Plan (Attachment C) and the Project Success Factors (Attachment D) are attached for reference. These attachments summarize the Phase 3, Operation activities, and the schedule for their performance. The Partnership's major FY-'97 objectives are:

- the LPMEOH™ demonstration plant will have successfully completed Test Runs #1 through #5 (by May-'97), and will have achieved 30 plus weeks of Task 2.1.1 operation (by Sept-'97).

• the decision to continue DME design verification testing, at the LaPorte AFDU in conjunction with the DOE Alternative Fuels R & D program, will have been made (by Dec. '96); and plans will have been made (by Apr '97) for completion of the operational proof of concept testing at LaPorte by December of 1997.

• the updated plan for Off-site Product-use Testing will have been completed (by May '97).

• the project Success Factors will continue to have been achieved during FY-97.



W. R. Brown

Approved:



Air Products/W.R. Brown



Eastman/W.C. Jones

DEPARTMENT OF DEFENSE STATUS REPORT  
LIQUID PHASE METHANOL DEMONSTRATION  
DE-FC22-92PC90543

## Attachment A

Printed: Jul/31/96  
Page 1

## Milestone $\Delta$ Summary $\boxtimes$

Test Run #	Test Run Description	Temp (Deg C)	Wt% Cat	H2/CO Ratio at Inlet	Space Velocity (SL/hr-kg)	Fresh Feed MeOH: (lpd)	Recycle CO Gas (KSCFH)	Inlet Sup. H2 Gas (KSCFH)	Time Velocity (ft/sec)	Elapsed Time (incl. outages) (weeks)	Start of Test
<b>Task 2.1.1 - Process Shakedown and Catalyst Aging:</b>											
1. Initial Shakedown; and Design Production Tests		250	28	2.42	8,000	260	900	50	40	1,800	0.64
2. Gassed Slurry Level	Part of other tests										Ongoing
3. Reactor Feed: Texaco-Type Syngas	250	28	0.67	9,240	202	650	95 (*)	0	2,612 (*)	0.77	2
4. Early Testing @ High Superficial Velocity	250	28	2.54	10,300	TBD	1,200 (**)	50	40	2,520 (*)	0.88	2
5. Check @ Test 1 Conditions	250	28	2.42	8,000	< 260	900	50	40	1,800	0.64	2
6. Catalyst Addition and Aging	250	28 - 40 or less	2.51	Dec. from 8,000	237	765	40	45	Max (2,700)	0.79	18
<i>(Note: Kingsport Complex Outage during this test)</i>											
7. Free-Drain Entrained/Condensed Oil to Reactor	250	28 - 40 or less	2.51	Dec. from 8,000	237	765	40	45	Max (2,700)	0.79	During Test 6
8. Operation @ Design Feed Gas Rates	250	40	2.42	4,000	260	900	50	40	1,800	0.64	2
9. Check for Limitation on Catalyst Slurry Concentration	250	> 40	2.51	Varies	TBD	765	40	45	Max (2,700)	0.79	6
10. Catalyst Addition to Reactor	250	Target 45	2.49	3,320	256	765	40	45	2,605	0.79	12
<i>(Note: Kingsport Complex Outage during this test)</i>											
Reach Max Productivity	or less	45	2.29	3,500	293	900	50	40	2,520	0.81	2
				TBD	TBD	1,110 (**)	50	40	2,520	0.86	2





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



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

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

Bob Kroskey

Bill Brown

Bill Jones

**APPENDIX J - TEST AUTHORIZATION K0-CB - CARBONYL BURNOUT**

**TEST AUTHORIZATION # K0-CB**

Kingsport LPMEOH Plant

Sheet: 1 of 3  
Date : 02/20/97  
By: VES

RUN NUMBER: K0-CB  
APPROX. START DATE: 26 February, 1997

**TITLE:** CARBONYL BURNOUT AND HOT-OIL TESTING PRIOR TO STARTUP OF  
CATALYST ACTIVATION AND METHANOL OPERATIONS

**OBJECTIVE:**

To passivate and test the synthesis loop and transfer circuits with oil and syngas at operating temperatures and pressures.

**SUMMARY:**

Approximately 5000 gallons of Drakeol-10 oil will be transferred to the 29C-01 reactor via various transfer circuits, intermediate tanks, and pumps. The reactor and 29C-02 steam drum will be heated to operating temperature with startup steam, and the synthesis loop will be pressurized up to operating pressure under syngas with the 29K-01 recycle compressor running. Over several days, a carbonyl concentration survey will be performed at various locations in the plant by using the Carbonyl Sample Stations. At the conclusion of the burnout period, the system will be drained and prepared for activation of catalyst batches. Approximate run time is 7 days.

**TEST DETAILS:** See pages 2 to 3 for details.

**ANALYTICAL COMMENTS:** See page 3.

**SAFETY IMPLICATIONS:**

Procedures are in place for leak-checking and purging all equipment before this test. However, this will be the first time that hot oil and syngas will be introduced to the plant, so operators should be aware that the usual safety implications of this operation are applicable hereafter. Air Products personnel will be required to wear Nomex in the plant when syngas is present.

The carbonyl sample handling will be conducted by Air Products' analytical chemist(s).

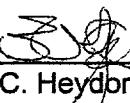
**ENVIRONMENTAL IMPLICATIONS:**

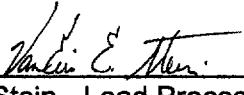
Minimal. The plant syngas purge will go to the Eastman boilers per normal operation.

**SPECIAL REMARKS:**

This test is designed to checkout the pumps and transfer circuits normally used to add oil (or slurry) to the reactor from 29D-02, 29C-05, and 29C-30. In addition, the pathways will be checked from the oil storage tank, 29D-30, to each of these vessels. Once the loop is under flowing syngas at temperature and pressure during the carbonyl burnout and sampling, the interim time provides an excellent opportunity to test GC's, flowmeters, and other instruments. Data will likely be available at two different flowrates and compositions.

**AUTHORIZATIONS:**

  
E. C. Heydorn - Program Manager

  
V. E. Stein - Lead Process Engineer

**TEST AUTHORIZATION # K0-CB**

Kingsport LPMEOH Plant

Sheet: 2 of 3  
Date : 02/20/97  
By: VES

**TEST DETAILS:**

1. This procedure assumes the oil storage tank, 29D-30, contains one full trailer load of Drakeol-10 mineral oil (approximately 6,000 gallons) at 38 °C.
2. Charge the 29C-30 catalyst reduction vessel with oil from 29D-30 (via 29G-34) per S.O.P. Section II C 3 Step C. Heat 29C-30 with utility oil from 29V-01 per Step D, and turn on agitator 29Y-30 per Step H to promote uniform heatup. Standby until reactor is at operating pressure to test a high pressure transfer via 29G-30.
3. Charge oil to 29C-05 secondary oil K.O. vessel and 29C-06 cyclone (via 29G-03) per S.O.P. Section II A 3 Step D.
4. Transfer the "remainder" of the oil in 29D-30 to 29D-02 (via 29G-34) per S.O.P. Section IV A 9 Step F (down to 5% to avoid tripping CP-03-S4 and shutting down 29G-01 and 29G-03).
5. If the 29D-02 utility systems have already been checked, proceed to the next step. Otherwise, this is a good opportunity to test the steam panel coils, 29Y-02 agitator (in oil), and H2 Makeup (including GC sample point #11) per S.O.P. Section IV A 9 Step J.
6. Follow the Reactor Area Start-Up Procedure S.O.P. Section II A 3, beginning at Step E.
  - E. Place 29K-01 syngas recycle compressor in service.
  - F. Transfer the oil in 29D-02 to the reactor (via 29G-02) per Section IV A 9 Steps K, L, and M.
  - 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 up reactor.
  - L. Prepare 29G-01 condensed oil circulation pumps for service.
  - M. Establish level control for 29C-05 secondary oil K.O. vessel.
  - N. Place 29G-01 condensed oil circulation pumps in service.
  - O. Start wall flush to 29C-06 cyclone.
  - P. Start fresh feed syngas to reactor.
7. Raise the reactor pressure and temperature to the normal operating conditions: 735 psig and 250 °C. Note that there will be no reaction or methanol production without catalyst in the system. Set the fresh feed flow rate (FIC-009) at 30-50 SCFH (probably cracked open in MANUAL). Set the compressor up for maximum flow.
8. Shutdown the 29Y-30 agitator, and transfer the oil in 29C-30 to the reactor (via 29G-30) per S.O.P. Section II C 3 Step P.
9. Shutdown and secure the utility oil skid per S.O.P. Section IV A 2 Step J.

**TEST AUTHORIZATION # K0-CB**

Kingsport LPMEOH Plant

Sheet: 3 of 3  
Date : 02/20/97  
By: VES

10. Establish 29T-01 nuclear density gauge hoist position and automatic level control on the reactor per S.O.P. Section II A 3 Step U. Note that the oil level at full gas rates will be more than double the initial height at Step 6F.
11. Maintain these conditions for several days to complete the carbonyl concentration survey and instrument checkout.
12. Additional tests will likely include a lower gas flow rate to the reactor, accomplished by decreasing the compressor flow rate (FIC-006). This may decrease the oil level in the reactor. If so, the 29T-01 must be repositioned to avoid drawing down the 29C-05 level. In addition, a test will be conducted with a more CO-rich feed to the reactor. This will be accomplished by blending in CO Makeup per S.O.P. Section II A 3 Step S.
13. At the conclusion of the carbonyl burnout test, purge, cool, and drain the reactor system according to the Reactor Area Extended Shutdown Procedure (S.O.P. Section II A 8).

TEST AUTHORIZATION #K0-CB is done, consult TEST AUTHORIZATION #K0-A1 for the next step.

**ANALYTICAL REQUIREMENTS:**

1. Carbonyl "wet chemistry" sampling requirements:
  - coordinated by Air Products' analytical chemist.
2. Carbonyl GC sampling requirements:
  - 29C-40 guard bed inlet (SP-12) and outlet (SP-15) as required to qualify instrument against "wet chemistry" results.
3. Process GC sampling requirements:
  - reactor inlet (SP-5) as required to setup proper loop composition;
  - all other applicable sample points as required to check out both GC's.
4. Flow measurement requirements:
  - all applicable FI's as required to check out instruments and compensation calculations.

## **APPENDIX K - TASK 3.1 - RESULTS OF CARBONYL BURNOUT**

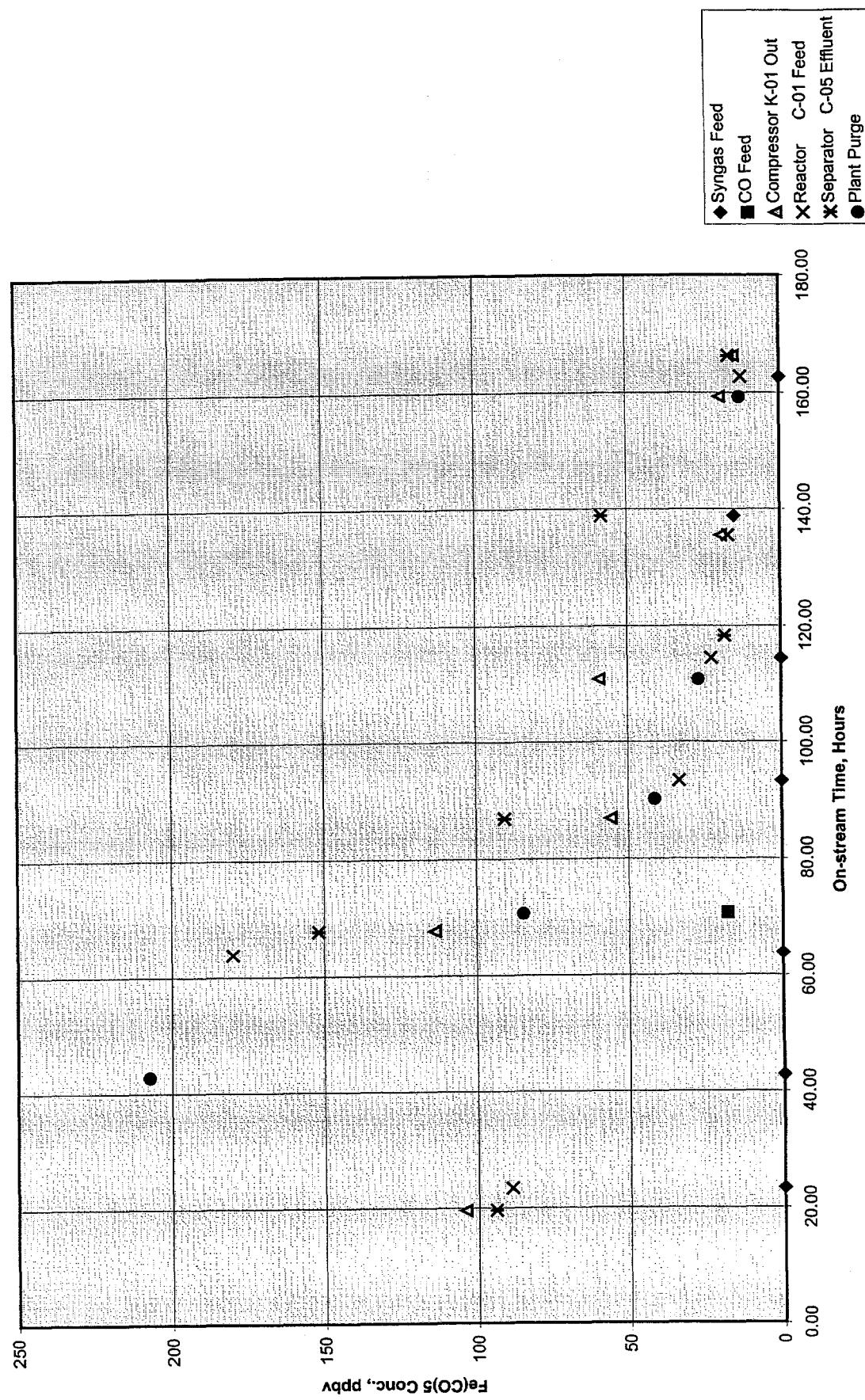
## Carbonyl Burnout at Kingsport LPMEOH Plant

Time Start	3/4/97 18:00	Sample Pt # 1	Sample Pt # 2	Sample Pt # 4	Sample Pt # 5	Sample Pt # 6	Sample Pt # 7
Fe(CO)5, ppbv		Syngas Feed	CO Feed	Compressor K-01 Out	Reactor C-01 Feed	Separator C-05 Effluent	Plant Purge
Sample Time	On-stream Time, Hrs						
3/5/97 13:34	19.58			104		94.2	
3/5/97 17:22	23.38	0			88.8		
3/6/97 12:45	42.75	0					207
3/7/97 9:46	63.78	0			180		
3/7/97 13:44	67.73			114		152	
3/7/97 16:46	70.77		17.7				84.7
3/8/97 8:51	86.87			55.8		90.7	
3/8/97 12:12	90.20						41.3
3/8/97 15:15	93.26	0			33.3		
3/9/97 8:46	110.78			59.3			26.9
3/9/97 12:21	114.36	0			22.5		
3/9/97 16:12	118.22					18.0	
3/10/97 9:30	135.51				19.1	16.8	
3/10/97 12:50	138.83	14.9				58.3	
3/11/97 9:15	159.27				19.2		12.9
3/11/97 12:42	162.70	0				12.3	
3/11/97 16:12	166.21				14.7		16.4

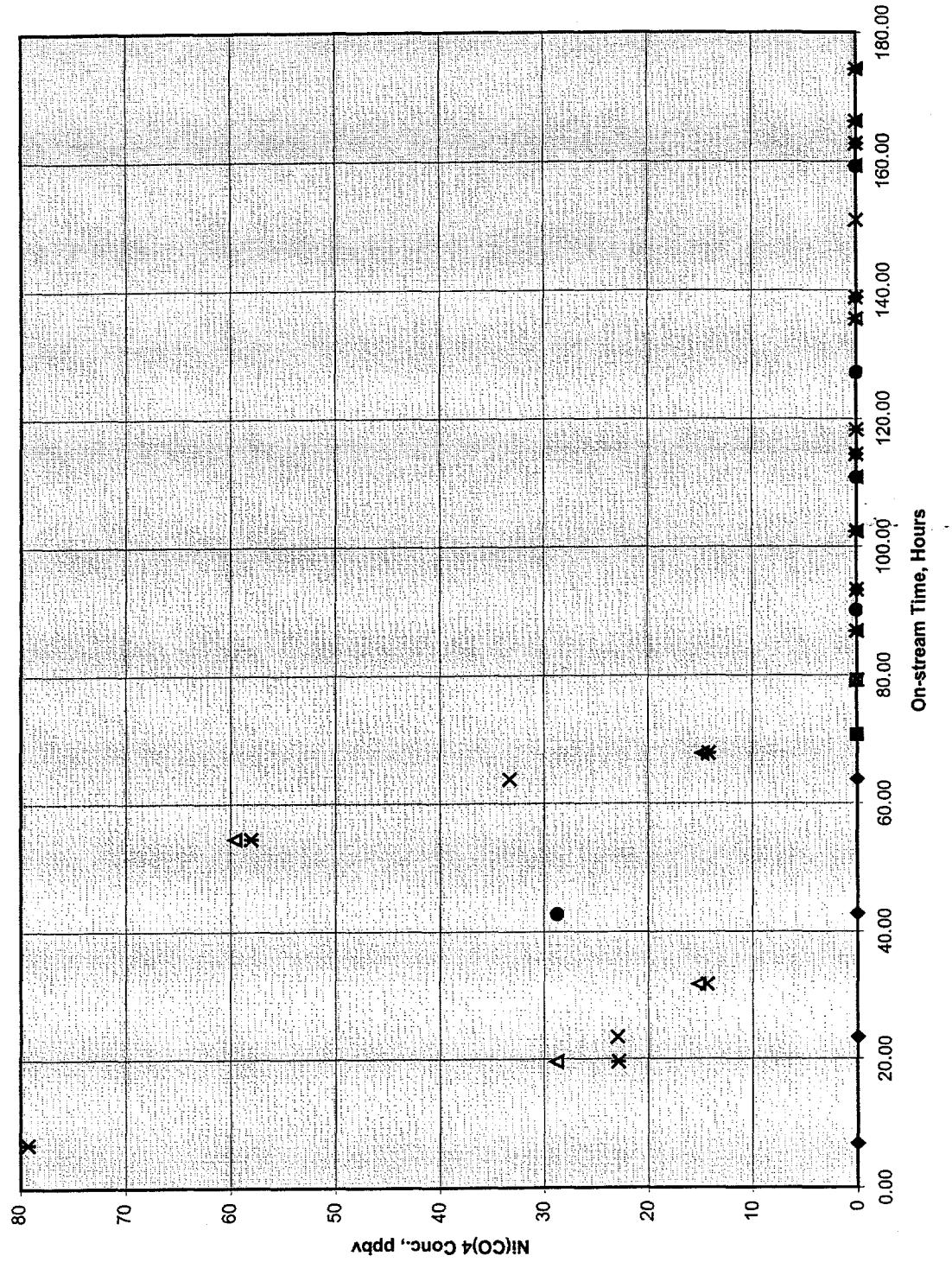
Carbonyl Burnout at Kingsport LPMEOH Plant

Time Start	3/4/97 18:00	Sample Pt # 1	Sample Pt # 2	Sample Pt # 4	Sample Pt # 5	Sample Pt # 6	Sample Pt # 7
Ni(CO)4, ppbv							
Sample Time	On-stream Time, Hrs	Syngas Feed	CO Feed	Compressor K-01 Out	Reactor C-01 Feed	Separator C-05 Effluent	Plant Purge
3/5/97 0:46	6.77	0					79.3
3/5/97 13:34	19.58			28.8			22.9
3/5/97 17:22	23.38	0					
3/6/97 1:39	31.65			15.2	14.4		
3/6/97 12:45	42.75	0					28.7
3/7/97 0:29	54.49			59.6		58.0	
3/7/97 9:46	63.78	0					
3/7/97 13:44	67.73			14.8		14.2	
3/7/97 16:46	70.77		0			0	
3/8/97 1:12	79.21		0		0		
3/8/97 8:51	86.87			0		0	
3/8/97 12:12	90.20					0	
3/8/97 15:15	93.26	0		0			
3/9/97 0:20	102.33			0		0	
3/9/97 8:46	110.78			0		0	
3/9/97 12:21	114.36	0		0			
3/9/97 16:12	118.22					0	
3/10/97 1:15	127.26					0	
3/10/97 9:30	135.51			0	0		
3/10/97 12:50	138.83	0			0		
3/11/97 0:48	150.82				0		
3/11/97 9:15	159.27			0		0	
3/11/97 12:42	162.70	0		0			
3/11/97 16:12	166.21			0		0	
3/12/97 0:16	174.27			0		0	

Carbonyl Burnout at Kingsport LPMEOH Plant



### Carbonyl Burnout at Kingsport LPMEOH Plant



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

**TEST AUTHORIZATION # K0-AD**

Kingsport LPMEOH™ Plant

Sheet: 1 of 3  
Date : 03/07/97  
By: VES

RUN NUMBER: K0-A1 thru K0-A?

APPROX. START DATE: 10 March, 1997

**TITLE:** ACTIVATION OF METHANOL CATALYST (IN NINE BATCHES) USING DILUTE CO  
PRIOR TO STARTUP OF METHANOL OPERATIONS

**OBJECTIVE:**

To activate the Liquid-Phase Methanol (LPMEOH™) synthesis catalyst.

**SUMMARY:**

Each "Design-sized" catalyst batch will comprise 2250 lbs of BASF S3-86 methanol catalyst slurried in 750 gallons of Drakeol-10 oil and activated with dilute CO (4% in nitrogen). At the completion of each activation procedure, the catalyst will be transferred to the 29D-02 slurry tank for temporary storage prior to startup. Approximate run time is 2 days/batch through nine successive "Design" batches. "Maximum-sized" batches are 50% larger, so if TEST AUTHORIZATION # K0-AM is used, fewer batches will be required.

**TEST DETAILS:** See pages 2 to 3 for details.

**ANALYTICAL COMMENTS:** See page 3.

**SAFETY IMPLICATIONS:**

While loading catalyst, operators should wear proper PPE to protect them from dust and the hot vapor which may be released from the loading hatch. Eastman industrial hygiene will perform a dust survey of the loading operation to determine if further mitigating controls must be engineered.

The catalyst building ventilation was designed for sufficient air change rate, and CO monitors situated under the roof will detect any accumulation. Air Products personnel will be required to wear Nomex in the plant when syngas is present.

**ENVIRONMENTAL IMPLICATIONS:**

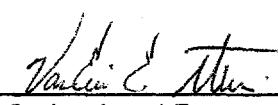
Minimal. The dilute syngas purge will go to the Eastman boilers per design. Byproduct water will be manually drained to the 29C-50 oil/water separator and ITS, and byproduct oil will be recycled to the process.

**SPECIAL REMARKS:**

CO and H<sub>2</sub> concentrations into and out of the 29C-30 catalyst reduction vessel must be monitored closely during the reduction procedure. Reduction temperature must be monitored closely and controlled per the attached TEST DETAILS. When adjusting flows or pressure, care should be taken to minimize catalyst carryover (caused by high gas velocity).

**AUTHORIZATIONS:**

  
E. C. Heydorn - Program Manager

  
V. E. Stein - Lead Process Engineer

**TEST AUTHORIZATION # K0-AD**

Kingsport LPMEOH™ Plant

Sheet: 2 of 3  
Date : 03/07/97  
By: VES

**TEST DETAILS:**

Follow the Catalyst Preparation Area Batch Preparation Procedure S.O.P. Section II C 3.

- C. After the normal preparation and isolation steps (A & B), charge the 29C-30 catalyst reduction vessel with 5250 lb of Drakeol-10 oil (750 gallons at 38°C). At Step 17 this corresponds to 41% level on LI-420. Search for level at about 8' 3" with the DI-454 nuclear density gauge (NDG).
- D. Heat 29C-30 to 82 °C at 15 °C/hour with utility oil from 29V-01.
- E. Depressurize reduction circuit.
- F. Charge 29C-30 with 2250 lb (6 drums) of S3-86 methanol catalyst powder.
- G. Purge reduction circuit.
- H. Mix the catalyst slurry with 29Y-30 agitator for 2 hours.
- I. Pressurize reduction circuit and set up nitrogen flow at 51,840 SCFH. (SCF evaluated at Eastman standard conditions 60 °F and 14.7 psia.)
- J. Introduce CO makeup at 2,160 SCFH (4% CO in N<sub>2</sub> @ 54,000 SCFH) and start temperature ramp to 240 °C at 8 °C/hour (approximately 20 hours). Note that composition as measured by GC will ultimately determine the proper flow setpoints.

Search for level at about 14' with the NDG. As the reduction proceeds, higher temperatures may cause the slurry to expand initially, but the prevailing trend will be decreasing level as oil evaporates out of 29C-30 and accumulates in 29C-31. Verify that the slurry is well-mixed by performing a NDG scan. Agitate with 29Y-30 if necessary. Check that the slurry temperatures are reasonably uniform.

At 200 °C, decrease the dilute CO reduction gas flow by 25% while maintaining composition (38,880 SCFH N<sub>2</sub> and 1,620 SCFH CO).

H<sub>2</sub> and CO concentrations should be measured continuously for the reduction feed and effluent streams (SP-9 and SP-10). If CO concentration in the reduction effluent (SP-10) falls below 0.1 mole %, increase the inlet CO concentration per instructions from the Air Products engineer. The objective here is to prevent starving the catalyst of reduction gas.

The catalyst reduction should be complete before reaching 240 °C. However, it may be necessary to hold the slurry at this temperature until the difference between inlet and outlet CO concentration falls below 0.05 mole %.

Record any indication of density or viscosity change, such as a change in the pressure drop across 29C-30 or shaking of 29C-30 during heat up and reduction.

**TEST AUTHORIZATION # K0-AD**

Kingsport LPMEOH™ Plant

Sheet: 3 of 3  
Date : 03/07/97  
By: VES

K. Cool catalyst slurry to 150-165 °C at 30 °C/hour under N<sub>2</sub>.

L. Transfer catalyst to 29D-02 slurry tank. Flush 29C-30 and transfer line to 29D-02.

Whenever 29D-02 contains catalyst slurry, follow S.O.P. Section IV A 9 for details about the H<sub>2</sub> Makeup purge, steam panel coils, and 29Y-02 agitator.

TEST AUTHORIZATION # K0-AD is complete. Repeat as necessary for additional "Design-sized" catalyst batches. Consult TEST AUTHORIZATION # K0-AM to reduce "Maximum-sized" catalyst batches, or refer to TEST AUTHORIZATION # K1 to begin methanol operations.

**ANALYTICAL REQUIREMENTS:**

1. GC sampling requirements:

- 29C-30 feed (SP-9) and effluent (SP-10) continuously;  
H<sub>2</sub> and CO are critical, and flows should be adjusted to maintain 4% CO in feed;  
CO<sub>2</sub> and N<sub>2</sub> are also important;
- 29D-02 atmosphere (SP-11) occasionally while catalyst is present.

2. Flow measurement requirements:

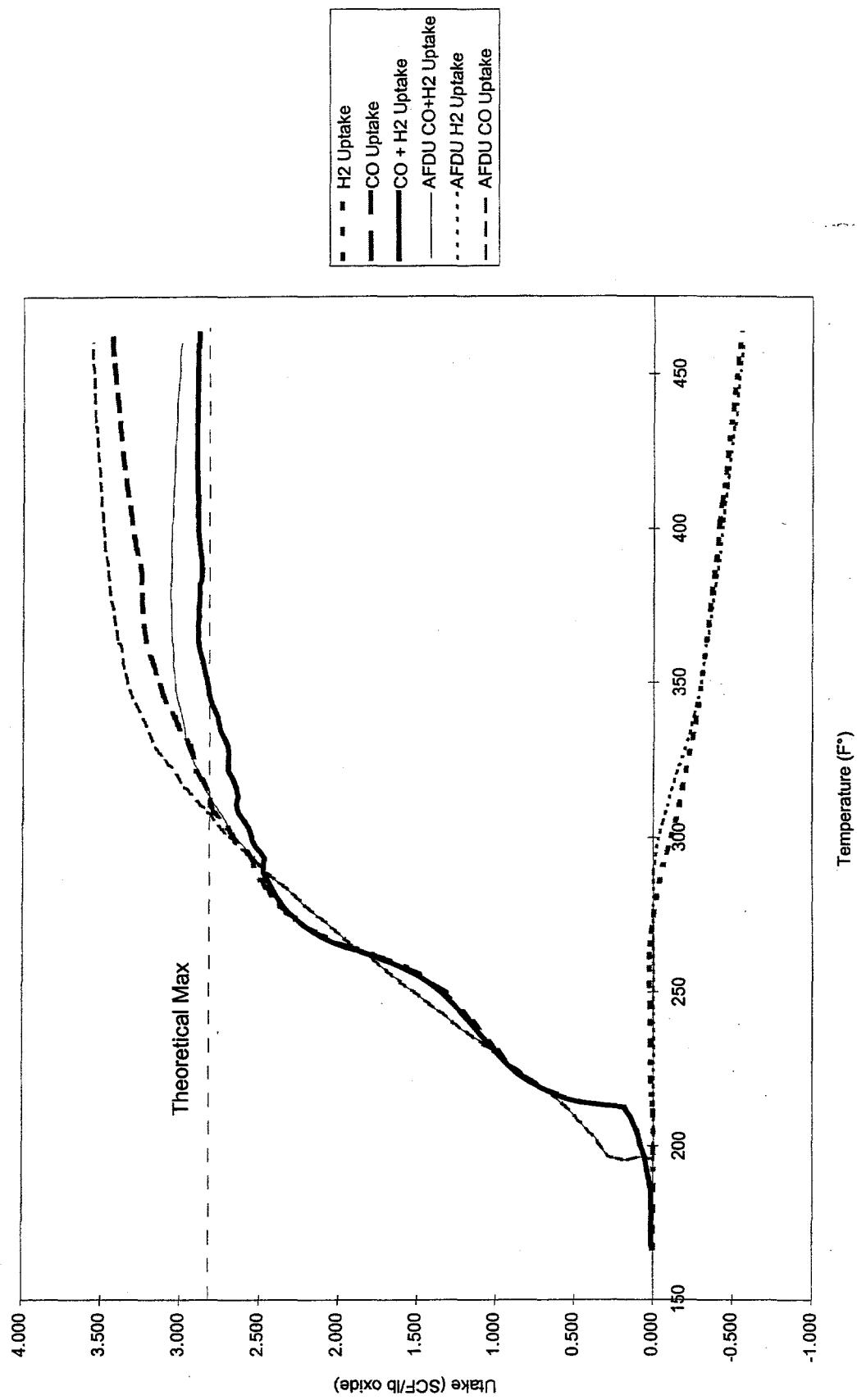
- N<sub>2</sub> to 29E-31 reduction preheater (FI-408);
- CO to 29E-31 reduction preheater (FI-400).

3. Catalyst sampling requirements:

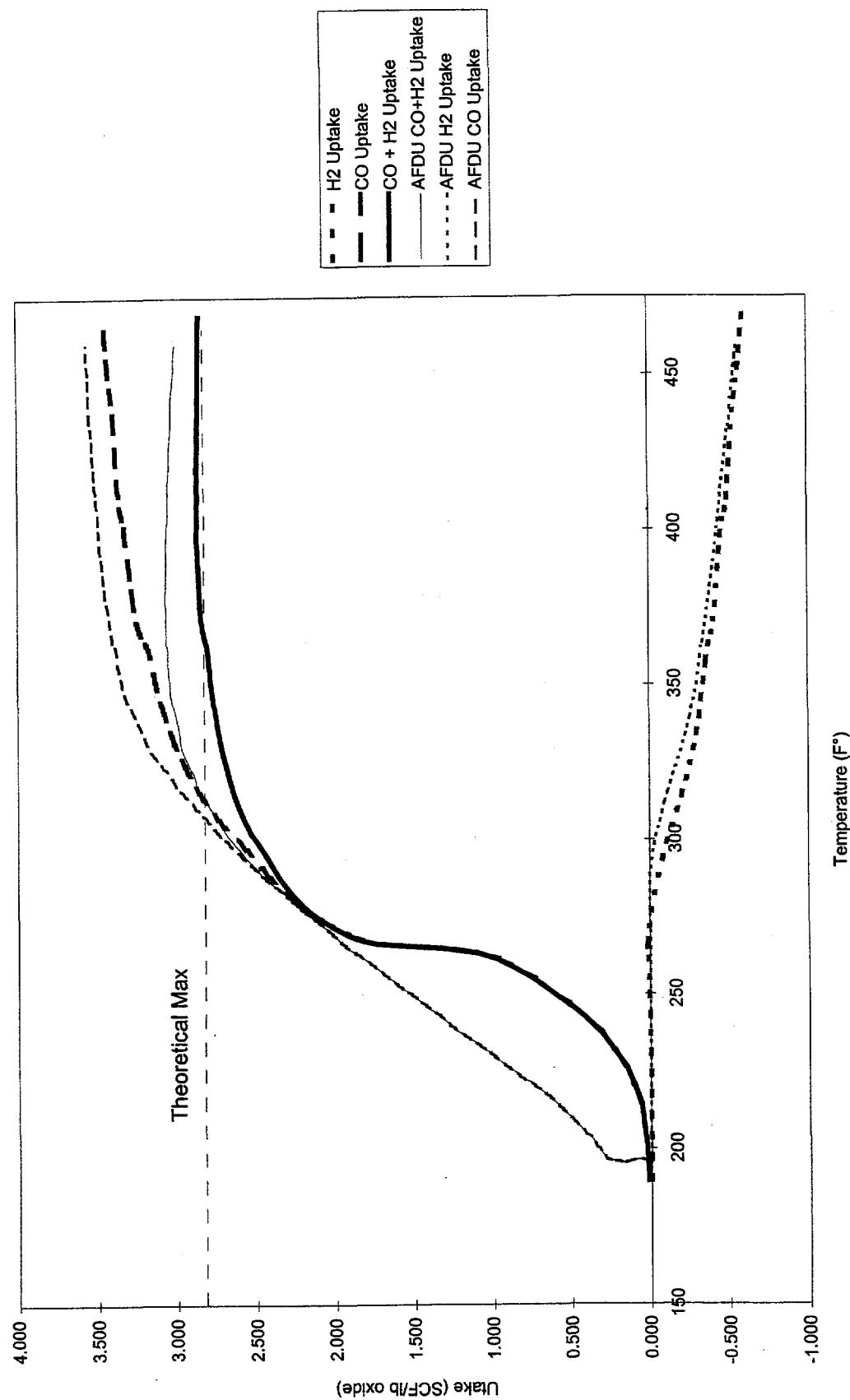
- slurry stub left between transfer to D-02 and flush (possibly through drain valve 1419 downstream of HV-418), as coordinated by Air Products.

## **APPENDIX M - TASK 3.1 - REDUCTION GAS UPTAKE CURVES**

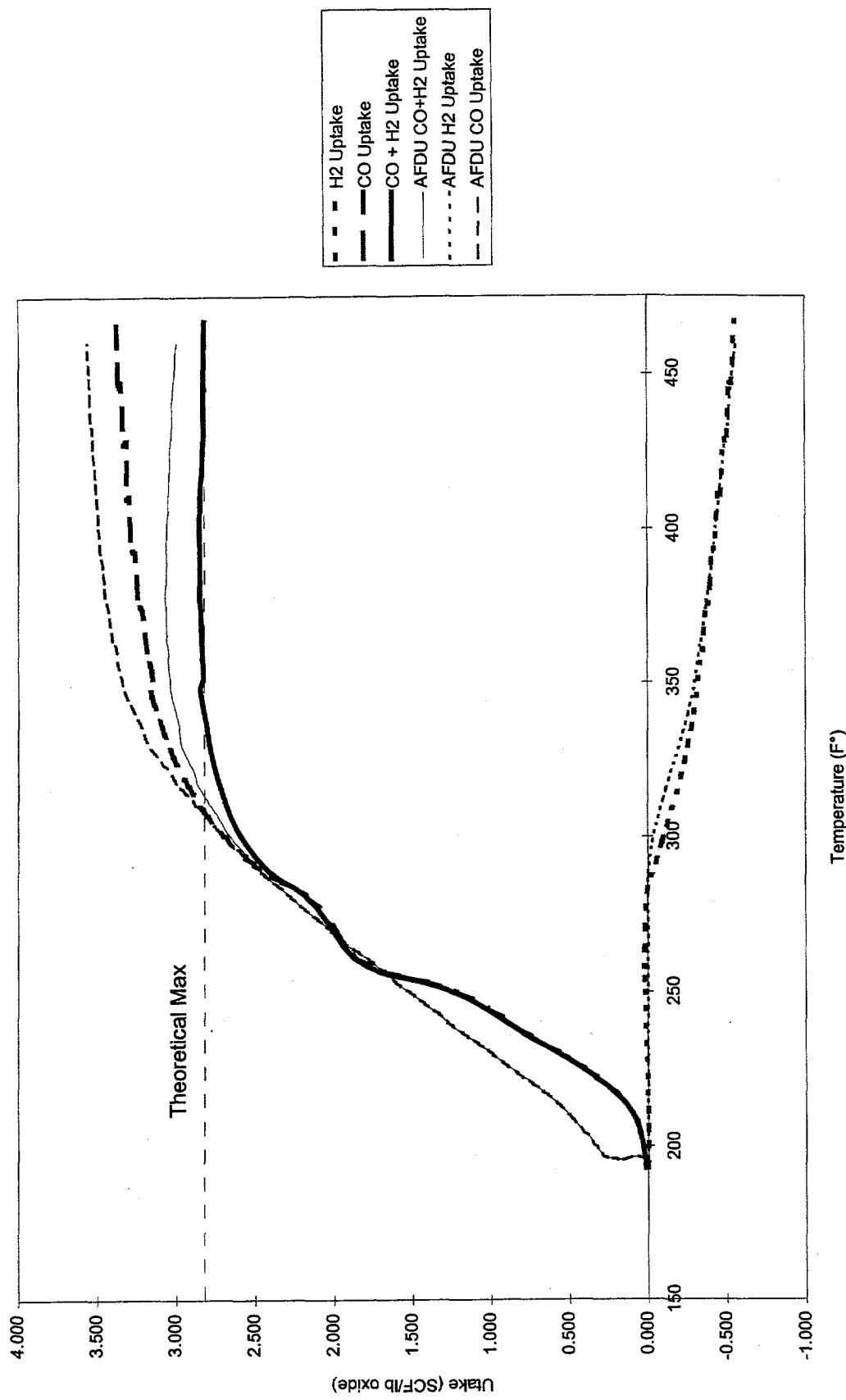
**Reduction Gas Uptake vs. Temperature**  
**Batch 1 March 16-17 1997**



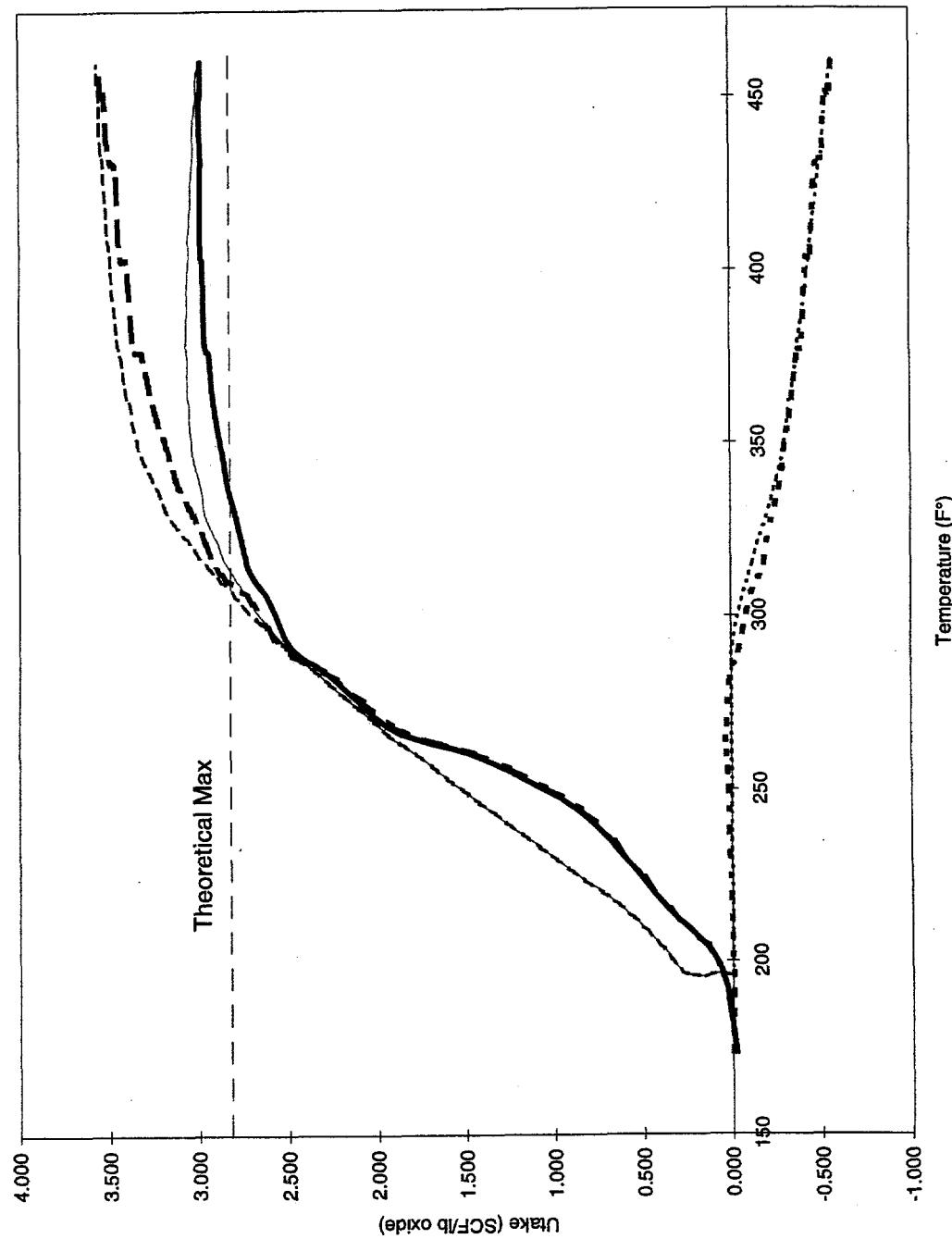
**Reduction Gas Uptake vs. Temperature**  
**Batch 2 March 19-20 1997**



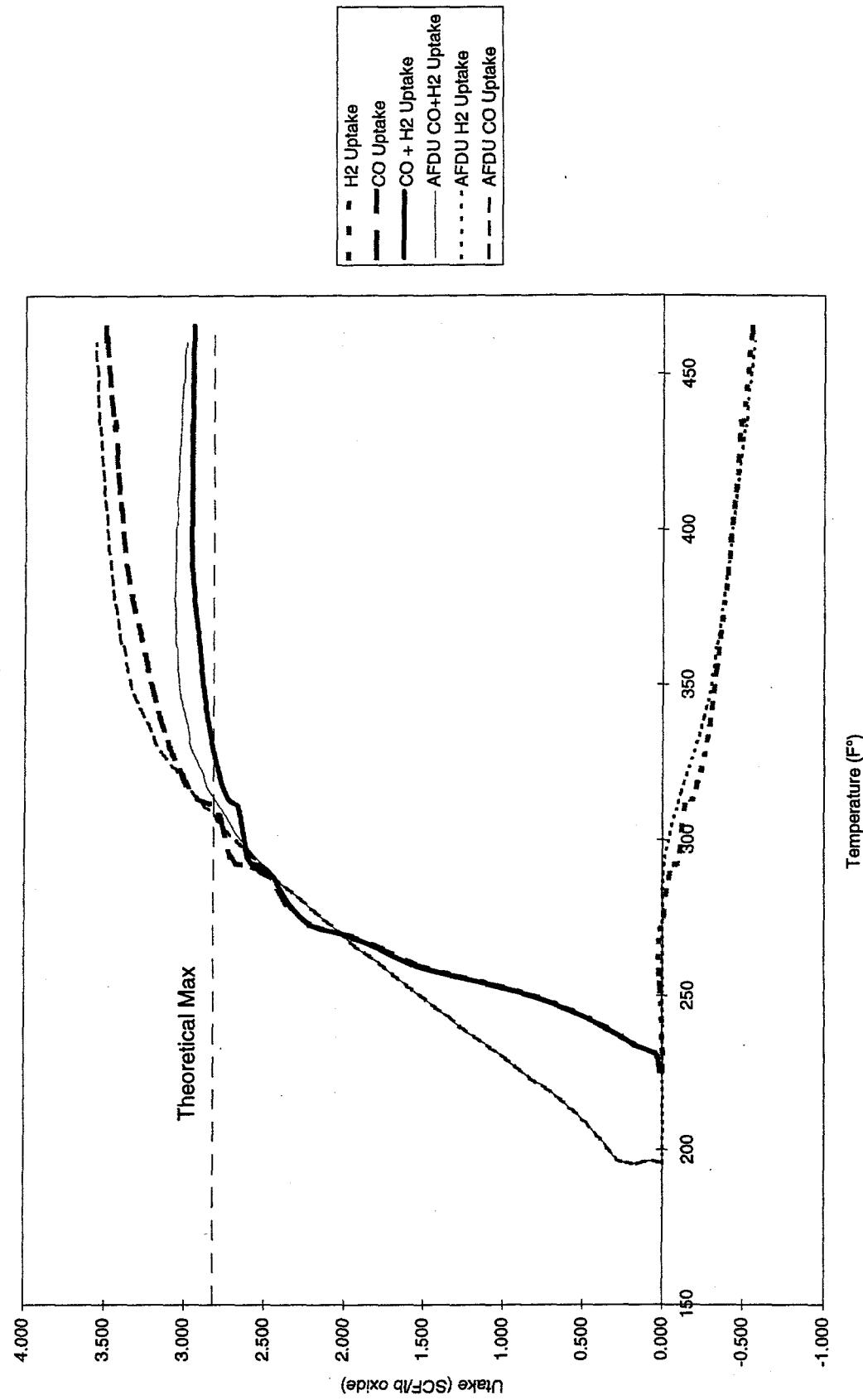
**Reduction Gas Uptake vs. Temperature**  
**Batch 3 March 20-21 1997**



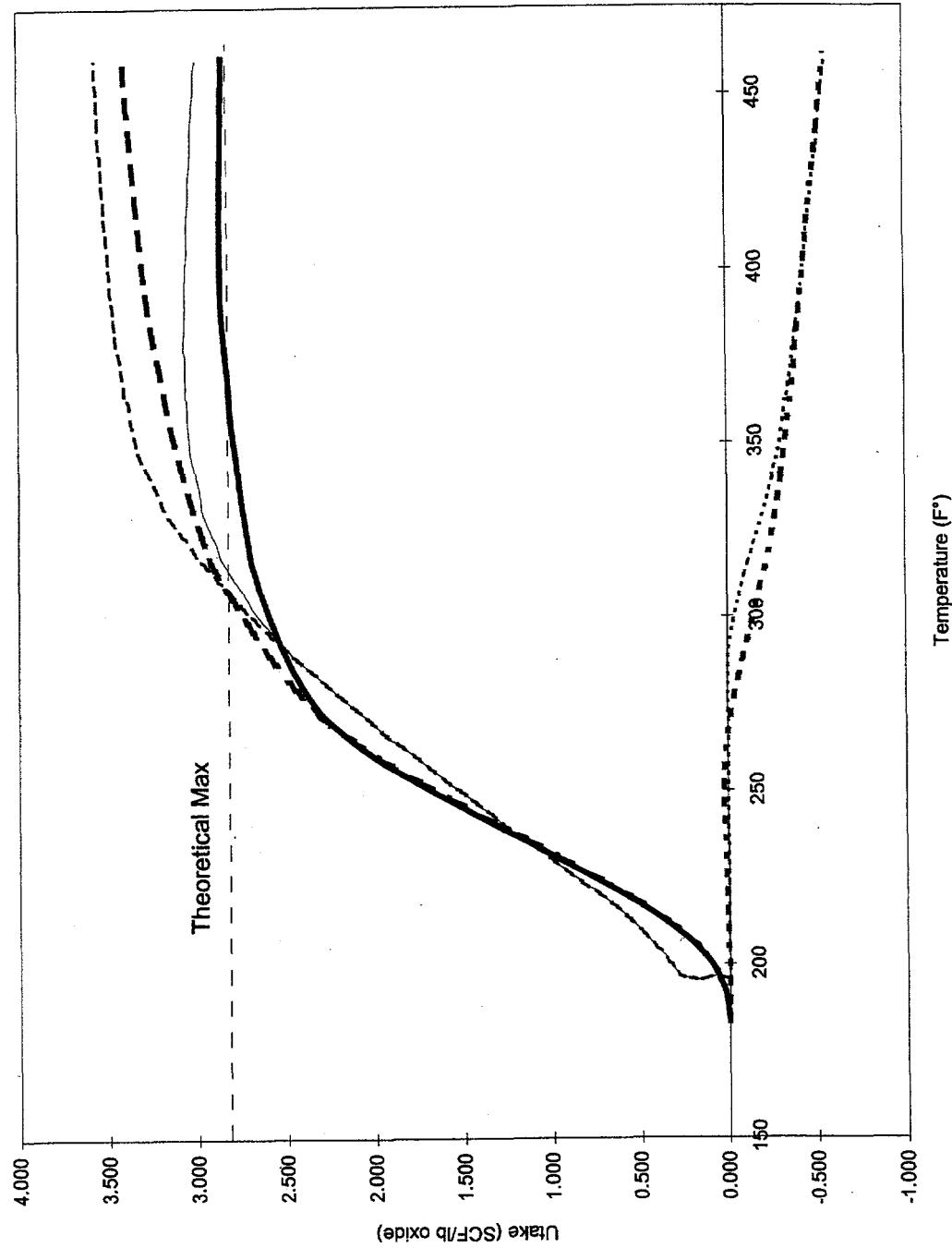
**Reduction Gas Uptake vs. Temperature**  
**Batch 4 March 22-23 1997**



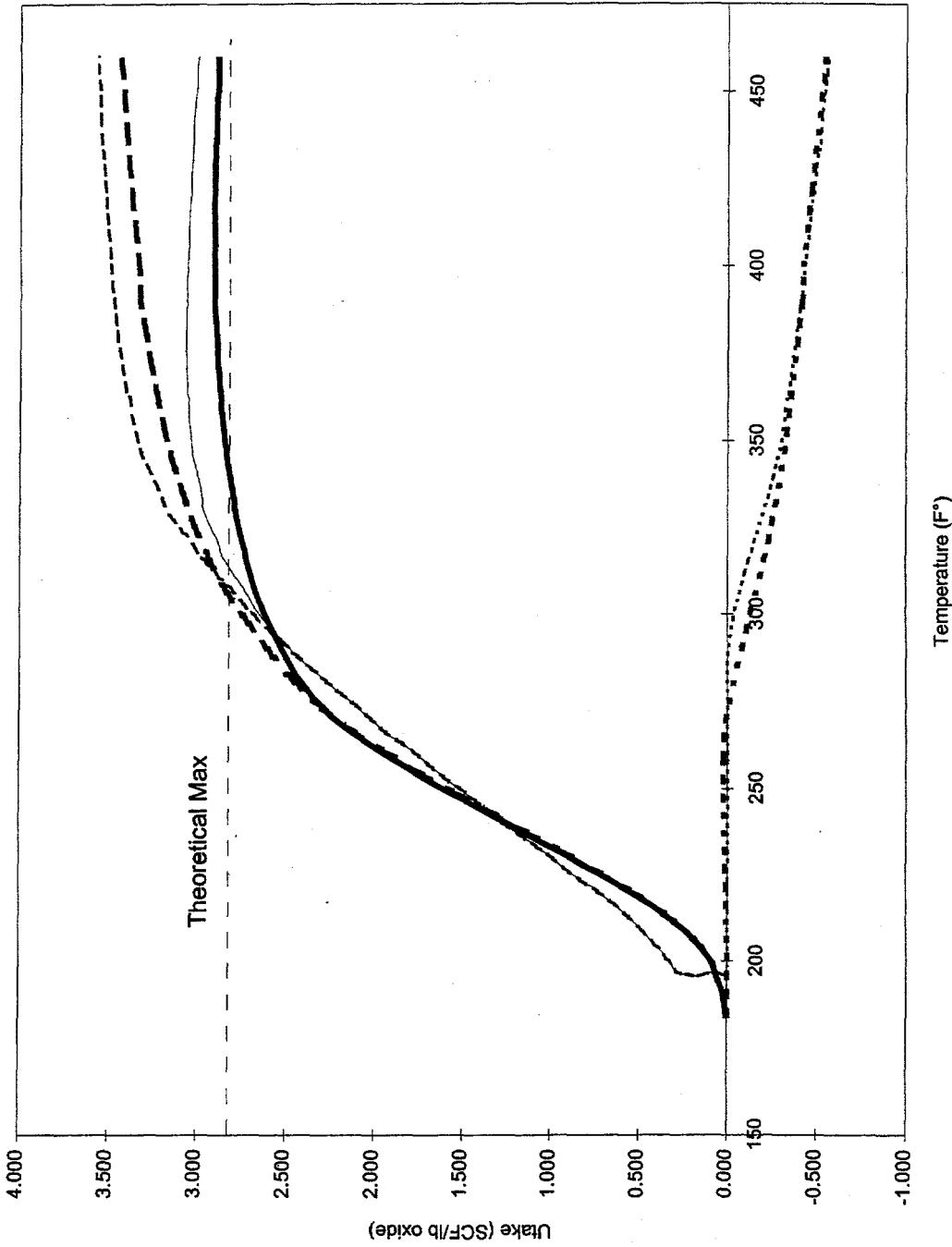
**Reduction Gas Uptake vs. Temperature**  
**Batch 5 March 23-24 1997**



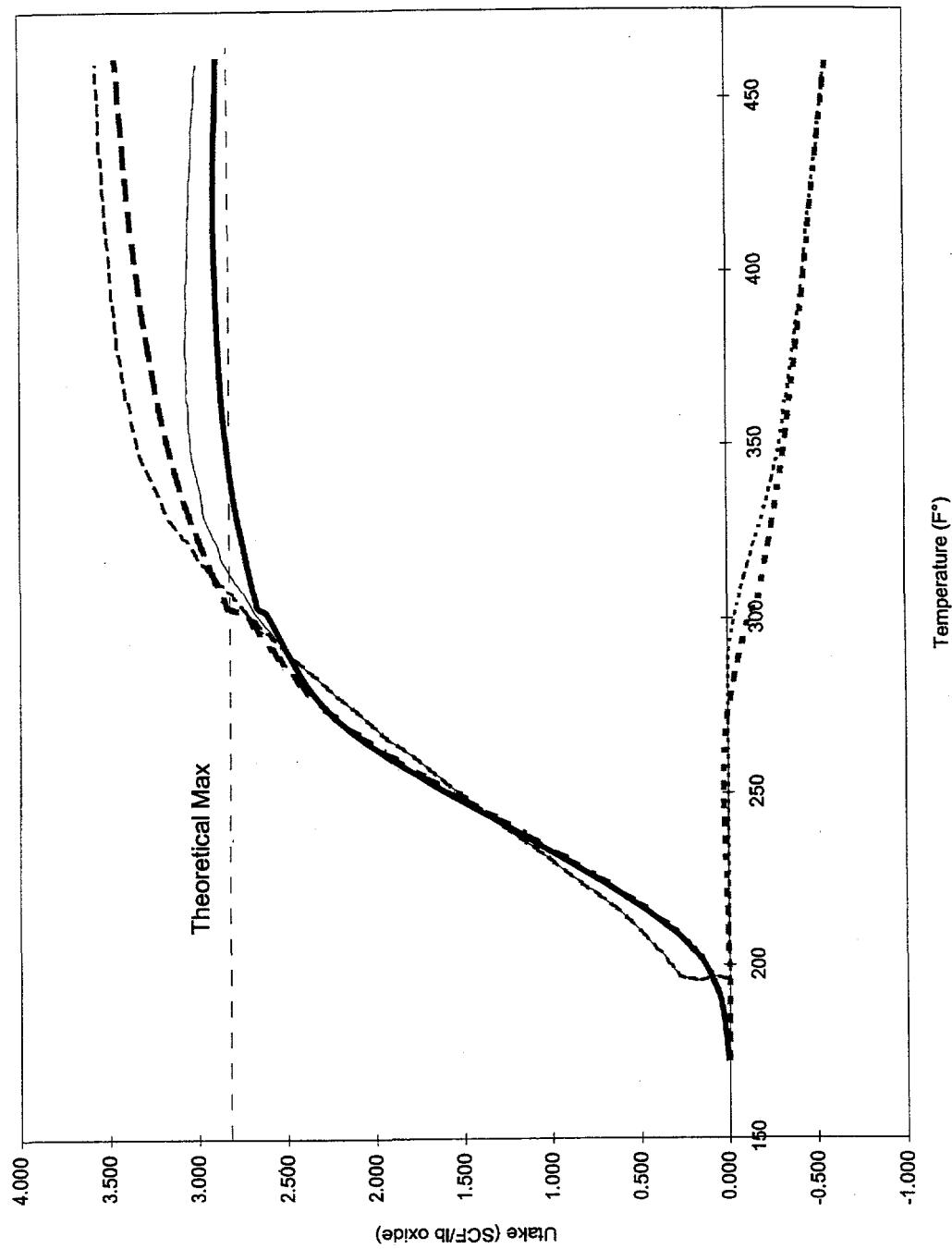
**Reduction Gas Uptake vs. Temperature**  
**Batch 6 March 25-26 1997**



**Reduction Gas Uptake vs. Temperature**  
**Batch 7 March 26-27 1997**



**Reduction Gas Uptake vs. Temperature**  
**Batch 8 March 27-28 1997**



**Reduction Gas Uptake vs. Temperature**  
**Batch 9 March 29-30 1997**

