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

## TECHNICAL PROGRESS REPORT NO. 4

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**1 April-30 June 1995** We have no objection from a patent standpoint to the publication or dissemination of this material.

*Mark Dvorscak* 3-22-96  
Office of Intellectual Property Counsel  
DOE Field Office, Chicago

Prepared by

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

and

**Eastman Chemical Company**  
**Kingsport, Tennessee**

for the

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

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

Patents cleared by Chicago on \_\_\_\_\_

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## ACRONYMS AND DEFINITIONS

Acurex	-	Acurex Environmental Corporation
Air Products	-	Air Products and Chemicals, Inc.
AFDU	-	Alternative Fuels Development Unit
DME	-	dimethyl ether
DOE	-	United States Department of Energy
DVT	-	Design Verification Testing
Eastman	-	Eastman Chemical Company
EIV	-	Environmental Information Volume
EPRI	-	Electric Power Research Institute
HAPs	-	Hazardous Air Pollutants
IGCC	-	Integrated Gasification Combined Cycle
KSCFH -	-	Thousand Standard Cubic Feet per Hour
LPMEOH™	-	Liquid Phase Methanol (the technology to be demonstrated)
MTBE	-	methyl tertiary butyl ether
NEPA	-	National Environmental Policy Act
Partnership	-	Air Products Liquid Phase Conversion Company, L.P.
Project	-	Production of Methanol/DME Using the LPMEOH™ Process at an Integrated Coal Gasification Facility
psia	-	Pounds per Square Inch (Absolute)
psig	-	Pounds per Square Inch (gauge)
P&ID	-	Piping and Instrumentation Diagram(s)
SCFH	-	Standard Cubic Feet per Hour
Sl/hr-kg -	-	Standard Liter(s) per Hour per Kilogram of Catalyst
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 Facility will be built at a site located at the Eastman Chemical Company (Eastman) complex in Kingsport.

On 4 October 1994, Air Products and Chemicals, Inc. (Air Products) and Eastman Chemical Company (Eastman) signed the agreements that would form the Partnership, secure the demonstration site, and provide the financial commitment and overall project management for the Project. These Partnership agreements became effective on 15 March 1995, when DOE authorized the commencement of Budget Period No. 2 (Mod. A008 to the Cooperative Agreement). The Partnership has subcontracted with Air Products to provide the overall management of the Project, and to act as the primary interface with DOE. Air Products, as subcontractor to the Partnership, will also provide the engineering design, procurement, construction, and commissioning of the LPMEOH™ Process Demonstration Facility, 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 Facility, and for the interconnection and supply of synthesis gas, utilities, product storage, and other needed services.

The project involves the construction of a 260 tons-per-day (TPD) methanol plant utilizing coal-derived synthesis gas from Eastman's integrated coal gasification

facility. The new equipment consists of synthesis gas feed preparation and compression, liquid phase reactor and auxiliaries, product distillation, 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), the LPMEOH™ process is ideally suited for directly processing gases produced by modern-day coal gasifiers. Originally tested at a small, DOE-owned experimental facility 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 Chemical complex, the technology will be integrated with existing coal-gasifiers, and a carefully developed test plan will allow operations to simulate electricity demand load-following in coal-based IGCC facilities. The operations will also demonstrate the enhanced stability and heat dissipation of the conversion process, its reliable on/off operation, and its ability to produce methanol as a clean liquid fuel without additional upgrading. An off-site product testing program will be conducted to demonstrate the suitability of the 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 tons/day). 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, and formally approved it on 1 June 1995 (Mod 009). Since then the project has been in the project definition phase. During this last quarter the project transitioned to the design phase. The project requires review under the National Environmental Policy Act (NEPA) to move to the construction phase, which is scheduled to begin in August of 1995. DOE has prepared an Environmental Assessment (EA), and a Finding of No Significant Impact (FONSI) was issued during this quarter. The facility is scheduled to be mechanically complete in November of 1996.

Process Engineering and Equipment Engineering activities have peaked in this period, and essentially all of the equipment is out for bid or has been purchased. Equipment vendors are beginning to supply design information. Piping and Instrument Diagram (P&ID) development has progressed rapidly, and a civil/piping design start has started. A program to test synthesis gas for catalyst poisons is being considered.

**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 facility producing 260 TPD of methanol will be designed and constructed at a site located at the Eastman Chemical (Eastman) complex in Kingsport, Tennessee. The Partnership will own and operate the facility for the four-year demonstration facility operational 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 shows promising results. If implemented, the DME would be produced during the last six months of the operations phase.



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 facility at Air Products' LaPorte, Texas, site. This demonstration project is the culmination of that extensive cooperative development effort.

## **B. Project Description**

### *Existing Site*

The 0.6 acre demonstration facility will be integrated into the existing 4,000-acre Eastman complex located in Kingsport, Tennessee. The Eastman complex employs approximately 12,000 people. In 1983 Eastman constructed a coal gasification facility utilizing Texaco technology. The synthesis gas 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, balanced gas) will be diverted from existing operations to the LPMEOH™ facility, thus providing the range of coal-derived synthesis gas 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* - Synthesis gas 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 will closely resemble the adjacent Eastman process plants, including process equipment in steel structures.

#### *Reaction Area*

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

#### *Purification Area*

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

#### *Storage/Utility Area*

The storage/utility area will include two diked lot-tanks for methanol, two tanks for oil storage, a slurry holdup tank, trailer loading/unloading area, and an underground oil/water separator.

#### *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 will be housed. In addition, a hot oil utility system is included in the area.

### **C. Process Description**

The LPMEOH™ plant will be integrated with Eastman's coal gasification facility. A simplified process flow diagram is included in Appendix G. Synthesis gas will be introduced into the slurry reactor, which contains a slurry of liquid mineral oil with suspended solid particles of catalyst. The synthesis gas dissolves through the 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 synthesis gas is recycled back to the reactor with the synthesis gas recycle compressor, improving cycle efficiency. The methanol will be used for downstream feedstocks and in off-site fuel testing to determine its suitability as a transportation fuel and as a fuel for stationary applications in the power industry.

### **D. Project Status**

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

### ***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 U.S. DOE's Environmental Assessment/Finding of No Significant Impact.
- The NEPA review was completed 30 June 1995 with the issuance of an Environmental Assessment and a Finding of No Significant Impact (FONSI). The final EIV is being prepared.
- Obtain permits necessary for construction and operation. The construction 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. Status: no work was done during this reporting period.
- Complete the design engineering necessary for construction and commissioning. This included Piping and Instrumentation Diagrams, Design Hazard Reviews, and the conduct of design reviews.

### ***Status of design engineering work***

Process Engineering work focused on:

- Completing specifications for remaining process equipment.
- Reviewing vendor drawings.
- Preparing process specifications for control valves and flow elements.

Engineering work is focused on:

- Completing mechanical specifications for process and electrical equipment.
- Reviewing mechanical vendor data.

- Writing specifications for valves, instruments, fire protection system and insulation.

Design Work is focused on:

- Completing site survey and soils investigation.
- Starting structural steel design.

Piping design is working on piping in the reactor structure and on completing facility plans.

The Design Hazards Review was completed in May.

#### ***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).

The product-use test plan is to be updated to better meet the technical objectives of the Project and serve the needs of municipal, industrial and electric utility markets. The Fuel Test Plan Update Objective, dated 27 June 1994, is included in Appendix A for reference.

Activity during this quarter included formulation of the Planning and Administrative Plan for this task. Acurex and Air Products will update plans for the off-site product-use demonstration. The Electric Power Research Institute (EPRI) will act as an advisor, to ensure that the demonstration obtains commercial acceptance by the electric power industry. EPRI may also help arrange for electric utility host sites, in coordination with Acurex for specific off-site fuel-use demonstrations

The following fuel-use test plan basis was prepared for discussion with potential fuel-use test participants:

A limited quantity (up to 400,000 gallons) of the methanol product as produced from the demonstration plant will be made available for fuel-use tests. Fuel-use tests will be targeted for an approximate 18 month period starting in January 1998. The methanol product will be available ex-works from the demonstration facility in Kingsport, Tennessee, at below market pricing. The objective of these fuel-use tests is to demonstrate the suitability of the product for use in applications that would enhance the commercial acceptance of the Liquid Phase Methanol™ (LPMEOH™) technology. The Electric Power Research Institute will provide guidance in identifying the application (e.g., dual firing with syngas in combustion turbines, or direct use in distributed power generators such as fuel cells, diesels and internal combustion engines) which would best meet the electric utility industry needs. Air Products and Acurex Environmental Corporation will develop the final fuel-use test plan, which needs to be prepared by June 1996.

Since the fuel-testing will not occur until 1998, further action by Acurex, Air Products, and EPRI in developing the plan will be deferred until late in 1995.

### ***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, that will better meet the technical objectives of the Project and serve the needs of commercial markets.

Air Products and Eastman will update plans for the on-site product use demonstrations.

The schedule for on-site product use tests was established for August to October 1997.

Product-use test plan details will be developed in 1996, in parallel with the operating test plan (Phase 2, Task 3); and in combination with the off-site fuel-use test plan (Phase 1, Task 4). These are to be developed by June 1996. No additional action occurred during this reporting period.

### ***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 Facility operating test plan (Phase 2, Task 3 ).

Activity during this quarter included development of a plan for commercial LPMEOH™ plant designs. The plan will utilize the detail engineering design of the Kingsport facility as a starting point for a "first" Generic Plant Design. The first Generic Plant Design will then be used as a basis for value engineering and process engineering development work to develop a "second" Generic Plant Design, which will embody design improvements and performance targets for commercial LPMEOH™ plant offerings. The Kingsport facility design is sufficiently far along that marking up the P&IDs, PFDs , Layout, and Scope Report will be undertaken in the next quarter. We will issue a work plan for the process engineering development and value engineering work in July, so that the overall engineering economics work can be used as input in time for the June 1996 completion for the operating test plan.

### ***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.

The project milestone schedule dictates that the first decision point, on whether to continue with DME DVT, is targeted for 1 December 1996. DVT is required to provide

additional data for engineering design and demonstration decision making. The essential steps required for decision making are: a) confirm catalyst activity and stability in the laboratory, b) develop engineering data in the laboratory, and c) confirm market(s), including fuels and chemical feedstocks.

A schedule to complete the initial DVT work by August 1996 has been developed. This schedule will allow time for reviews and necessary rework for the December 1996 decision. The DME Milestone Plan, with the latest target completion dates, is included in Appendix B for reference. Parallel market economic studies and laboratory R&D work are being undertaken, and are reported on separately below.

#### *Market Economic Studies*

Papers recently published by Amoco and Haldor Topsoe indicate that DME, as an 80 wt% mixture with methanol and water, has a high cetane number and may therefore be an excellent diesel engine fuel. This extends the range of market possibilities for DME beyond that of the up to 20 wt % contemplated in the demonstration plan. Air Products is analyzing the Amoco and Haldor Topsoe papers in order to determine if this finding could impact the final decision.

#### *Laboratory R&D*

Initially, synthesis of DME concurrently with methanol in the same reactor was viewed as a way of overcoming the synthesis gas conversion limitations imposed by equilibrium in the LPMEOH™ process. Higher synthesis gas conversion would provide improved design flexibility for the coproduction of power and liquid fuels from an IGCC facility. The liquid phase DME (LPDME) process concept seemed ideally suited for the slurry based liquid phase technology, since the second reaction (methanol to DME) could be accomplished by adding a second catalyst with dehydration activity to the methanol-producing reactor.



Initial research work determined that two catalysts, a methanol catalyst and an alumina-based dehydration catalyst, could be physically mixed in different proportions to control the yield of DME and of methanol in the mixed product. Proof-of-concept runs, in the laboratory and at the LaPorte process development unit, confirmed that a higher synthesis gas conversion could be obtained when a mixture of DME and methanol is produced in the liquid phase reactor.

Subsequent catalyst activity-maintenance experiments have shown the catalyst system utilized in the proof-of concept runs experienced relatively fast deactivation compared to the LPMEOH™ process catalyst system. Further studies of the LPDME catalyst deactivation phenomenon were therefore undertaken under DOE Contract No. FC-22-95PC93052. 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--were undertaken. During the January-March 1995 quarter, seven dehydration catalysts were screened. None exhibited attractive performance. Several distinct modes of deactivation were identified, suggesting new directions for alternate dehydration catalyst research. The January-March 1995 quarterly report, prepared under Contract FC-22-95PC93052, is included in Appendix C for reference. Continuation of LPDME catalyst system research is now being undertaken as part of the design verification testing (Task 1.5.3) of this Project, and will be reported in subsequent quarterly reports.

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

The Cooperative Agreement was modified on 15 March 1995 (Modification A008). This modification acknowledged the Novation Agreement by which the project participant was transferred from Air Products to the Air Products Liquid Phase Conversion Company,

L.P., extended the Project completion date to 31 December 2001, and allowed the Project to proceed into Budget Period No. 2. The modification also incorporated an updated Statement of Work and Statement of Joint Objectives, in addition to the Eastman and Air Products Agreements as submitted with the initial Continuation Application. A subsequent modification on 1 June 1995 (Modification M009) clarified the Novation Agreement.

The Project Evaluation Plan for Budget Period No. 2 was drafted, reviewed and formally submitted on 13 June 1995 for DOE review and approval. Final DOE approval was obtained in July, and the final approved plan, dated 18 July 1995, is included in Appendix D for reference.

The 40% design completion project review meeting was held on 22 June 1995 at PETC. Attendees from Air Products, Eastman, DOE HQ, and PETC participated. The project status was reviewed. The delay in approving the Continuation Application caused extra project expenditure, and delayed the orderly commencement of detail design engineering by about 3 months. The schedule delay is expected to be made up by careful planning, with startup still targeted for the fourth quarter of 1996. This schedule is tight, and allows no slippage in procurement, design engineering, construction bidding, or construction execution. The agenda, some meeting handouts, and the meeting notes are included in Appendix E. Appendix E shows the schedule (page E.4) and the cost forecast (page E.5) of \$36.0 million for the project at Kingsport, with 12% spent as of 30 April 1995.

The monthly reports for April, May and June were prepared and submitted. This includes 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.

The status of equipment purchases is shown in the "Specification Status-Equipment" report included in Appendix E. This report, prepared in mid-June, shows that orders have been placed for all but 11 of the 80 equipment items.

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

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

- 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.

The only construction activities at the site are the piping tie-in to Eastman's process and the extension of these lines to the LPMEOH plant. Fourteen of twenty-three tie-ins were completed in May. All 23 of the lines were extended to the LPMEOH plant battery limits.

### ***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.
- Prepare the operating manual and initiate the operator training program.

There was no activity for this task during this quarter.

#### ***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 Phase 1, Task 4 Off-Site Testing work. No procurement or construction work is planned for this task during Budget Period No. 2.

#### ***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 updated Phase 3 Operating Plan.
- Submit all Project status, milestone schedule, and cost management reports as required by the Cooperative Agreement.

No work was done on the updated annual plan during this reporting period.

The DOE reporting tasks are currently being performed and reported under Task 1.5.

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

- Complete site preparation and foundation designs
- Add piping design staff
- Start electrical design
- Update the Project Management Plan
- Prepare the (Partnership) first annual plan

**F. Summary**

Process Engineering and Equipment Engineering activities have peaked in this period, and essentially all of the equipment is out for bid or has been purchased. Vendor information is just starting to arrive. P&ID development has progressed rapidly, and a civil/piping design start has started. A program to test synthesis gas for catalyst poisons is being considered. DOE gave final approval of the Continuation Application to Budget Period No. 2 (Design and Construction). The schedule delay is expected to be made up by careful planning, with startup targeted for the fourth quarter of 1996. Twelve percent of the \$36.0 million of funds authorized for Kingsport through Budget Period No. 2 have been expended as of 30 April 1995.

## **APPENDICES**

### **APPENDIX A      TASK 1.4 - FUEL TEST PLAN UPDATE OBJECTIVES**

## Fuel Test Plan Update Objectives

The Technical Objective (No. 4) is:

"Demonstration in end-use tests of the suitability of the methanol produced by the LPMEOH™ process as a fuel for mobile and stationary applications by providing:

- (a) Analysis of samples of the fuels and combustion exhaust gas streams for emission levels of sulfur dioxide, nitrogen oxides, carbon monoxide, carbon dioxide and *hazardous air pollutants*; and
- (b) A cost advantage for the use of methanol produced by the LPMEOH™ *demonstration facility* by means of a economic comparison with conventionally accepted fuels."

### Discussion

The original (Cooperative Agreement) fuel test plan has become outdated, and under-represents new Utility dispersed electric power developments, and possibly new mobile transport engine developments. The updated fuel test plan will attempt for broader market applications and for commercial fuels comparisons.

### Update Objective

The objective of the fuel test plan update will be to demonstrate commercial (e.g., economic) market applications (municipal, industrial and electric utility) replacing (gasoline, diesel, natural gas) commercial fuels, based on expected (1998 to 2018) U.S., energy markets needs when the technology is to be commercialized.

**APPENDIX B      TASK 1.5.3 - DME MILESTONE PLAN**



## 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
2. Up to 8 wt.% DME for GCC energy storage by Aug. '96
  - a. Re-review economics
3. About 80 wt.% DME with methanol, as a diesel replacement fuel; or as chemical feedstock at Kingsport. by Aug. '96
  - a. Economics, process basis study

- #### C. Decision to continue or drop by Dec. '96

### 2. Process Development Unit Design Verification Test:

- #### A. LaPorte Process Development Unit tests by Sep. '97

- #### B. Decision to Implement 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 Feb.'01

**APPENDIX C      TASK 1.5.3-DME R&D EXCERPT FROM JANUARY-  
MARCH 1995 QUARTERLY TECHNICAL REPORT, PAGES 17-31**

**DME R&D**

**Excerpt from January - March 1995 Quarterly Technical Report prepared under  
DOE Contract FC22-95PC93052**

## DME R&D

Excerpt from January - March 1995 Quarterly Technical Report prepared under DOE Contract FC22-95PC93052

### TASK 3: RESEARCH AND DEVELOPMENT

#### 3.1 New Processes for DME

The work this quarter focused on particular aspects of LPDME activity maintenance.

##### Dehydration Catalyst Screening Runs

In the previous quarterly we reported that an interaction between BASF S3-86 methanol catalyst and Catapal B  $\gamma$ -alumina is the cause of catalyst deactivation under LPDME conditions. This finding resulted in our active screening for alternative dehydration catalysts. In this quarter, nine more dehydration catalysts were examined, including two silica alumina, two modified Catapal B  $\gamma$ -alumina, a fumed alumina, two metal phosphate, and two  $ZrO_2$ -based samples. However, none exhibited better performance than  $\gamma$ -alumina.

##### *Silica Alumina*

Silica based materials are the preferred dehydration catalysts, since a previous experiment (14045-31) showed that a high surface area silica gel, while inert toward methanol dehydration, did not cause premature aging of the BASF S3-86 methanol catalyst. Two silica-aluminas of high silica content, i.e., Siral 85 (85% silica and 15% alumina) and Siral 95 from Condea, were

tested. The results for these two samples are shown in Table 3.1.1 and Figure 3.1.1 below, along with the results from a run using our standard catalyst system (S3-86 plus Catapal B  $\gamma$ -alumina). The methanol equivalent productivity of the systems containing Siral 85 and 95 is very low, e.g., at 20 hours onstream, 12.9 and 15.0 mol/kg-hr, respectively, compared to 30.7 mol/kg-hr for the standard catalyst system. This is due to both an extremely low dehydration activity and low methanol synthesis activity. The rate constants in Table 3.1.1 quantitate this observation. The results from these two experiments indicate that silica alumina is not a better dehydration catalyst than  $\gamma$ -alumina for our application.

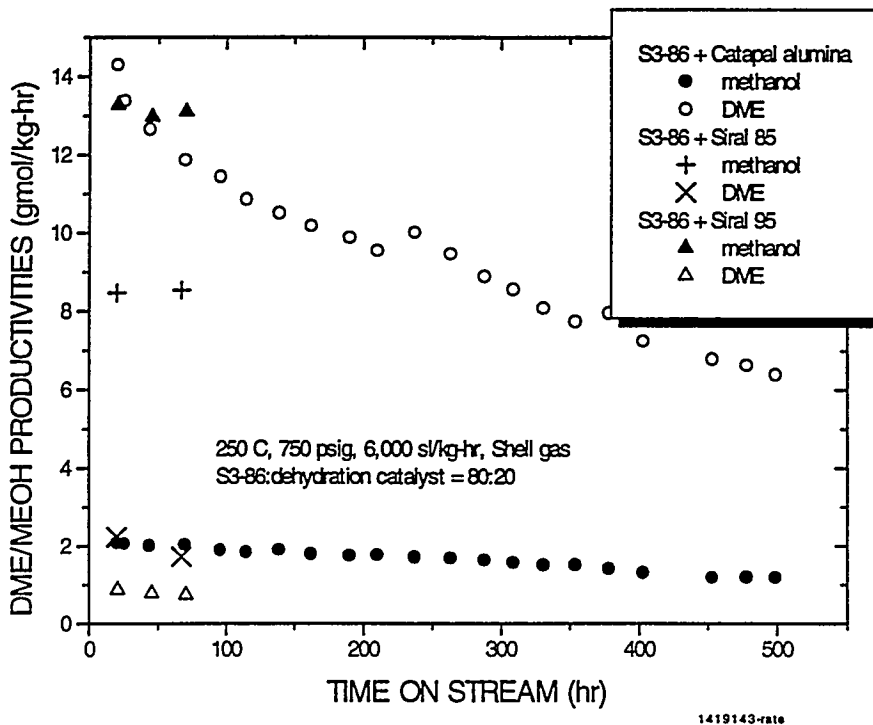
**Table 3.1.1. Kinetic Results from the Runs using S3-86 along with Siral 85 (14191-43), Siral 95 (13465-100), and Catapal B  $\gamma$ -alumina (11782-3), respectively.**

Run	Catalyst S3-86:Al <sub>2</sub> O <sub>3</sub>	Time on Stream (hr)	Productivity (mol/kg-hr)			Rate Constant	
			MEOH	DME	Equiv.	$k_m^a$	$k_d^b$
14191-43	80:20	20	8.5	2.2	12.9	1.0	1.2
13465-100	80:20	20	13.3	0.9	15.0	1.8	0.4
11782-3	82:18	20	2.1	14.3	30.7	2.6	17.0

a: Methanol synthesis rate constant calculated from  $R_m = k_m f_{H_2}^{2/3} f_{CO}^{1/3} (1 - appr.)$ , based on methanol catalyst weight.

b: Methanol dehydration rate constant calculated from  $R_d = k_d f_{CO_2}^{-0.33} f_{MEOH}^{0.11} f_{CO}^{0.70} (1 - appr.)$ , based on dehydration catalyst weight.

**Figure 3.1.1 Catalyst screening using Siral 85 and Siral 95 plus S3-86**



Two important observations should be noted, especially since they appear frequently in the series screening runs: First, different dehydration catalysts have different impacts on the methanol catalyst. The lower initial activity of the methanol catalyst in the catalyst systems containing silica alumina (see Table 3.1.1) indicates faster deactivation of the methanol catalyst in the presence of silica alumina than in the presence of  $\gamma$ -alumina. It is likely that this deactivation started during catalyst reduction. This occurs during the activation procedure for the methanol catalyst which runs 24 hr from room temperature to 240°C under diluted hydrogen before syngas is introduced into the system.

Second, different dehydration catalysts deactivate differently. The low dehydration activity from the silica alumina samples was unexpected, since silica alumina is normally more acidic than  $\gamma$ -alumina, and therefore should be more active toward dehydration. This low activity is attributed to the fast deactivation of silica alumina based on the following observation. We monitored the exit gas flow rate in the early hours of the run using Siral 85. Since the synthesis reaction is molecule-reducing, lower flow rate means greater activity. Judging by the flow rate shown in Table 3.1.2, the activity of the Siral 85 system at 1.2 hr on stream is fairly high, i.e., corresponding to a methanol equivalent productivity near 30 mol/kg-hr. Thus, we infer that the silica alumina had an initial dehydration activity at least comparable to the  $\gamma$ -alumina. However, this activity dropped considerably in the first 20 hr on stream as can be seen from the increasing flow rate. Again, it is likely that the activity of the silica alumina already started to drop during the reduction.

**Table 3.1.2. Exit Gas Flow Rate as a Function of Time On Stream for the Run using Siral 85**

<u>Time on stream (hr)</u>	<u>Normalized exit flow rate<sup>a</sup></u>
1.2	0.79
4.3	0.85
20	0.89

a: Normalized by the inlet flowrate. For the run using S3-86 and Catapal  $\gamma$ -alumina, the normalized exit flowrate at 20 hr on stream is 0.77, corresponding to a methanol equivalent productivity of 30.7 mol/kg-hr.

#### *Modified Catapal B $\gamma$ -Alumina*

One of our hypotheses about the interaction between the methanol and dehydration catalysts is that ZnO from the methanol catalyst might migrate onto alumina under the reaction conditions. The effect of this migration could be twofold: ZnO may deactivate the dehydration catalyst by reacting with the acid sites, and/or the methanol catalyst may lose its activity by losing its active component. If this is true, one would expect that:

- 1) the activity of the alumina would drop considerably if it is doped with ZnO; and
- 2) the doped Catapal B would result in a better stability of the methanol catalyst due to the smaller driving force for the migration.

Based on these considerations, a ZnO-doped Catapal B sample (14191-65) was prepared by impregnating the alumina with zinc nitrate, followed by calcination at 560°C for 4 hr to convert  $Zn(NO_3)_2$  into ZnO and disperse ZnO on the alumina surface. The loading of ZnO is 40 wt%. According to the literature [Xie, et al., in: *Adv. Catal.*, V37 (1990) p1], this corresponds to the highest loading of ZnO on alumina in atomically dispersed form. Further loading will result in ZnO crystallite formation.

Figures 3.1.2 and 3.1.3 display the activity of the catalyst system consisting of BASF S3-86 methanol catalyst and ZnO-Catapal B in comparison with that of the standard catalyst system (S3-86 plus virgin Catapal B). The reaction was run at the standard conditions (250°C, 750 psig, 6,000 GHSV, methanol:dehydration catalyst =80:20) using Shell gas. As expected, the ZnO-doped Catapal B has a lower dehydration activity than the virgin Catapal B. However, it shows little improvement in the stability of the methanol catalyst.

A  $WO_3$ -modified Catapal B  $\gamma$ -alumina was prepared by impregnating Catapal B  $\gamma$ -alumina with an aqueous solution of ammonium meta tungstate, followed by calcination at 700°C. Tungsten oxide has been reported in the literature to have dehydration activity at least as high as  $\gamma$ -alumina. This sample was tested along with BASF S3-86 methanol catalyst under the standard conditions using Shell gas (13465-58). Figures 3.1.2 and 3.1.3 show that the catalyst system does not exhibit better activity or stability than the standard catalyst system (S3-86 plus virgin Catapal B  $\gamma$ -alumina).

Figure 3.1.2. Methanol Synthesis Rate Constant as a Function of Time On Stream for Different Catalyst Systems

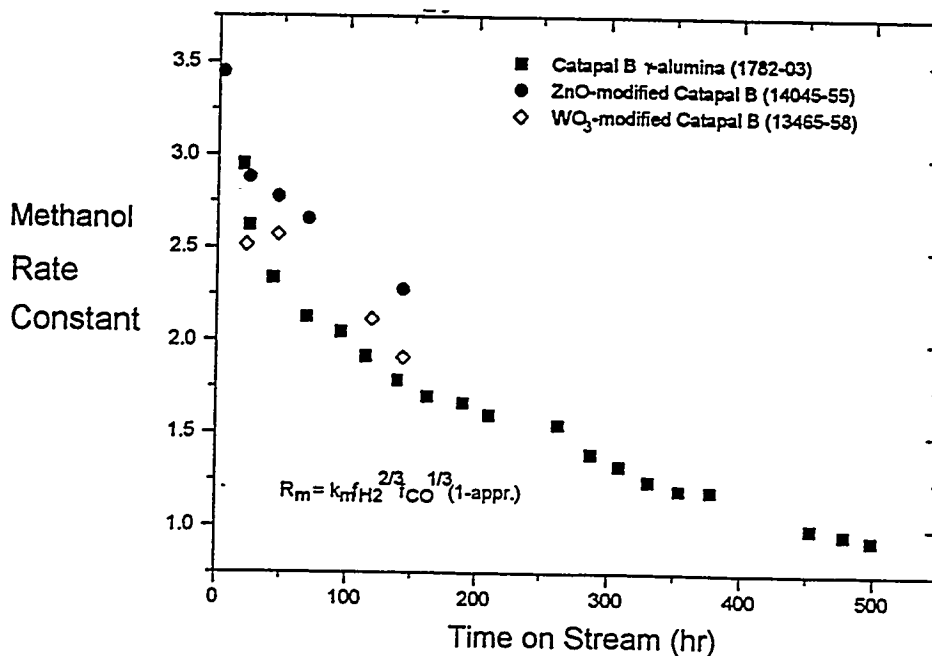
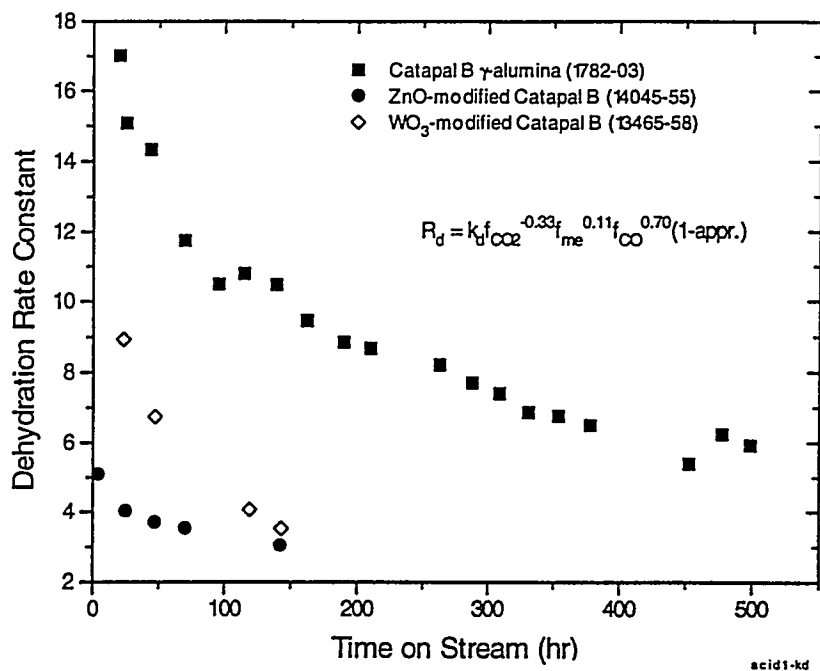


Figure 3.1.3. Dehydration Rate Constant as a Function of Time On Stream for Different Catalysts

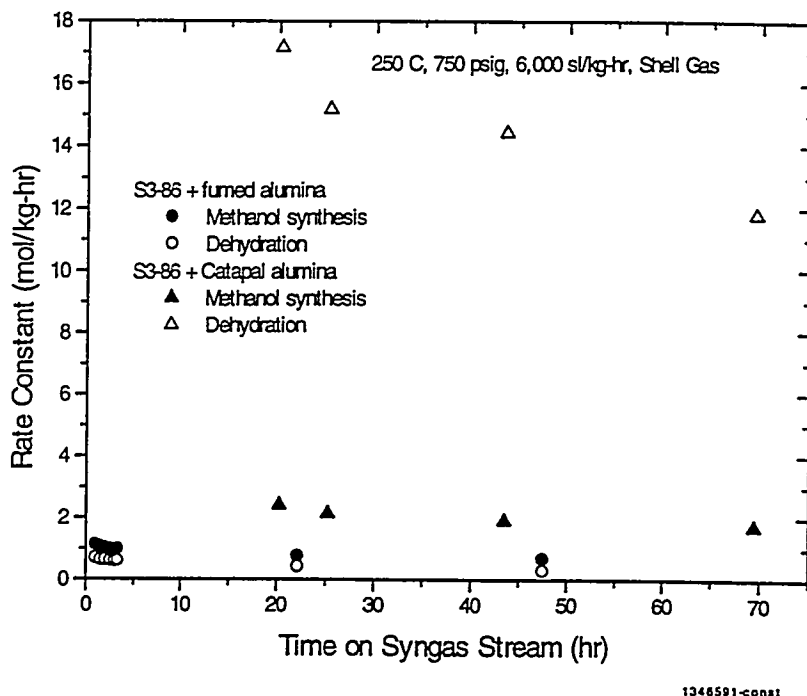


### Fumed Alumina

Fumed alumina from Degussa (aluminum oxide C, average particle size = 13 nm) was tested along with BASF S3-86 methanol catalyst as an alternative dehydration catalyst (13465-91). Figure 3.1.4 shows that both initial methanol synthesis and methanol dehydration activities, in terms of rate constant, are low for this catalyst system as compared to the standard one, followed by continuous drop in both activities.



Figure 3.1.4. Activity of the Catalyst System Consisting of S3-86 and Fumed Alumina (13465-91)



The interpretation of the poor activity and stability of this system is complicated by the following two factors. First, when the slurry was discharged from the reactor, clumps of the catalyst mixture were observed on the stirring rod and the walls of the reactor. This is due to low sedimentation of the very fine powders in the oil. If the sediment was also formed under the reaction conditions, it could lead to poor catalyst performance. The second complication is the fact that the fumed alumina contains a small amount of HCl (<0.5% by specification). The uncertainty is that  $\text{Cl}^-$  may migrate onto the methanol catalyst under the reaction conditions, leading to methanol catalyst deactivation, since  $\text{Cl}^-$  is a known methanol catalyst poison.

### ***Metal Phosphates***

Metal salts such as phosphates, sulfates, and chlorides have been used as industrial dehydration catalysts. Therefore, two phosphate samples were examined this month: one calcium phosphate, called hydroxyapatite, sample (Ca:P = 1.58), and a silica doped with 34% phosphorous acid. Owing to the high acid loading, the acid-doped silica contains some free  $\text{H}_3\text{PO}_4$ . Therefore, we would also like to use this sample to probe the possibility of using homogeneous acids as dehydration catalysts for LPDME. The runs were conducted under the standard LPDME conditions (250°C, 750 psig, 6,000 GHSV, 80:20 catalyst ratio). As shown in Figure 3.1.5, both samples have almost nil dehydration activity. This lack of activity could be due to the reaction temperature (dehydration using metal phosphates usually takes place at higher temperatures, e.g., > 300°C), the low water level in our system (high water level is needed to prevent  $\text{H}_3\text{PO}_4$  from being dehydrated, therefore losing its acidity, above 200°C), or incompatibility with the methanol catalyst.

Figure 3.1.5. Dehydration Rate Constant as a Function of Time On Stream for Different Catalyst Systems

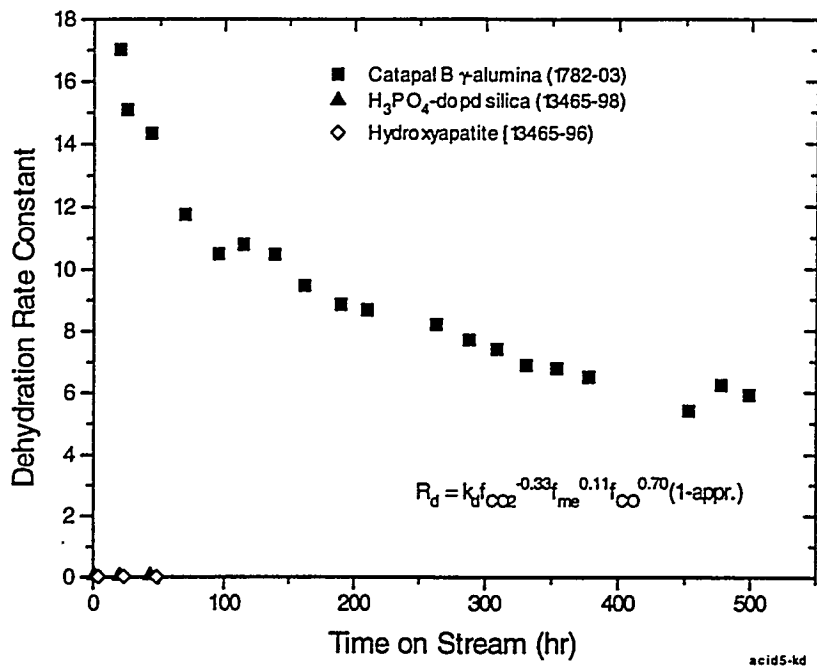
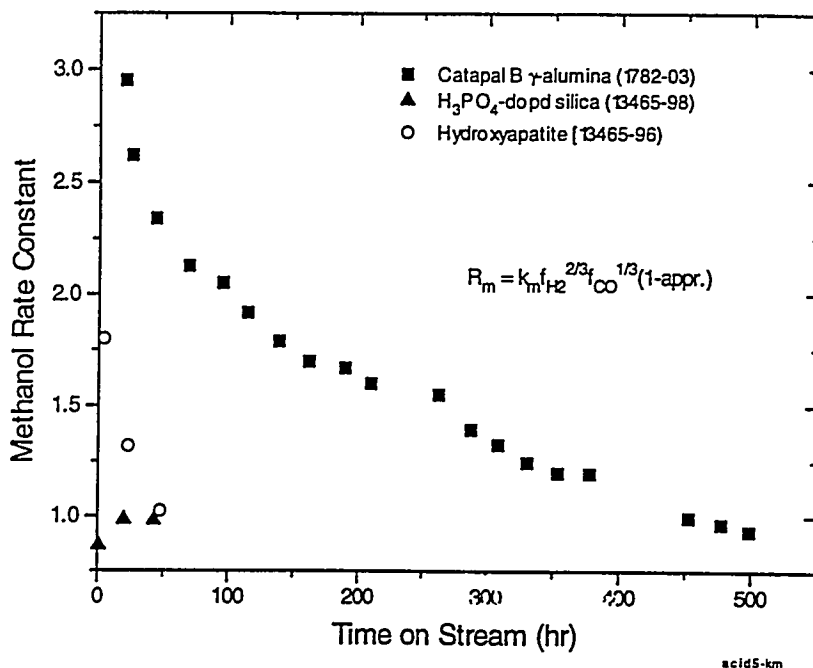


Figure 3.1.6. Methanol Synthesis Rate Constant as a Function of Time On Stream for Different Catalyst Systems



Both catalysts have significant impact on methanol catalyst activity, although they did not exhibit any dehydration activity. As shown in Figure 3.1.6, the methanol catalyst, when used along with the hydroxyapatite sample, deactivates at a higher rate than in the standard catalyst system. A different effect on the stability of the methanol catalyst was observed in the case of  $H_3PO_4$ -doped silica. The activity of the methanol catalyst must have been severely diminished during the reduction, as indicated by the low initial activity of the catalyst. The long-term impact of this sample on the methanol catalyst is not clear since the run was terminated at 43 hr on stream.

#### *ZrO<sub>2</sub> and ZrO<sub>2</sub>-Modified Silica Gel*

Two  $ZrO_2$ -based materials were tested because dehydration of alcohols using  $ZrO_2$  has been reported in the literature. A bulk  $ZrO_2$  sample was obtained by calcining  $Zr(OH)_4$  at  $600^\circ C$  for 6 hr. The surface area of the calcined sample was  $38.1 \text{ m}^2/\text{g}$ . The second sample,  $ZrO_2$ -modified silica, was prepared by impregnating a silica gel (Davison grade 55) with zirconium ethoxide, followed by calcination. Both test runs, conducted under the standard conditions (13465-88 and 14191-33), show that the  $ZrO_2$  samples had essentially zero dehydration activity. While the bulk zirconia sample had little impact on the activity of the methanol catalyst, the  $ZrO_2$ -modified silica gel caused a continuous drop in the methanol catalyst activity.

#### **Experiments using Robinson-Mahoney Basket Internals and Pelletized Catalysts**

Robinson-Mahoney basket internals were purchased for our 300 cc autoclave from Autoclave Engineers Group. Figure 3.1.7 shows the assembly of this system. An annular basket, which sits

stationary inside the autoclave reactor, is used to hold pelletized catalyst samples. An agitator, connected to the shaft of our current reactor system, provides the necessary agitation. Baffles are built inside and outside the basket to prevent vortexing. This setup is designed to help understand the mechanism of catalyst deactivation under LPDME conditions. First, it enables us to separate spent methanol and dehydration catalysts for characterization. Second, it creates a physical environment different from the slurry such as pellets vs. powders, and there is an absence of collisions between catalyst particles. Whether this results in a different deactivation pattern will provide insight into the mechanism of catalyst deactivation.

### ***Reactor Shakedown***

A shakedown run of this reactor system was conducted using the BASF S3-86 methanol catalyst (#ZU 553-5072) alone. The catalyst pellets were between 1 and 3.35 mm. Before the run, the reactor system was passivated using flowing syngas at 300 C and 1,200 psig for 20 hr. The catalyst was reduced using the standard procedure. As shown in Figure 3.1.8, the standard heating ramp was too fast for methanol catalyst pellets in this setup, resulting in incomplete reduction; the total H<sub>2</sub> uptake during this reduction was 1.7 scf/lb, 61% of the normal uptake. The system was switched to Shell gas when the reduction temperature reached 240°C.

As shown in Figure 3.1.9, the methanol productivity of this system (9 mol/kg-hr) was much lower than that of a normal slurry phase run (15 mol/kg-hr). The apparent rate constant of this system was 70% smaller. The low activity was due to a mass transfer limitation in the system. Figure 3.1.10 shows that productivity increased with stirring speed. No attempt to overcome this limitation by exceeding 2,000 rpm was made because of potential damage to the system. The activity of the system appeared to be stable except for a small initial drop. However, it should be mentioned that the apparent activity of this system will not be sensitive to catalyst deactivation because the reaction rate is mass transfer limited.

The catalysts used in the LPDME run were BASF S3-86 tablets (#ZU 553 5072) crushed into a 1.0-1.7 mm size and Catapal B 1/8"x1/8" tablets from Calsicat (02E-60A) crushed into a smaller size (about four pieces from every tablet). The S3-86 and Catapal catalyst pellets were mixed first in a 80:20 ratio and loaded into the basket. Since the shakedown run showed much slower H<sub>2</sub> uptake by the pellets during reduction and incomplete reduction of the catalyst using the temperature ramp designed for powder S3-86, a new reduction scheme was employed in the current run, based on the BASF activation procedures for packed bed application. The reduction results are shown in Figure 3.1.11. Again, H<sub>2</sub> uptake was slower compared to the powder case. However, the final H<sub>2</sub> uptake (2.6 scf/lb) was close to the acceptable value (2.8 - 3.0 scf/lb), which is near the stoichiometric uptake.

Figure 3.1.7. Schematic of Robinson-Mahoney Stationary Catalyst Basket

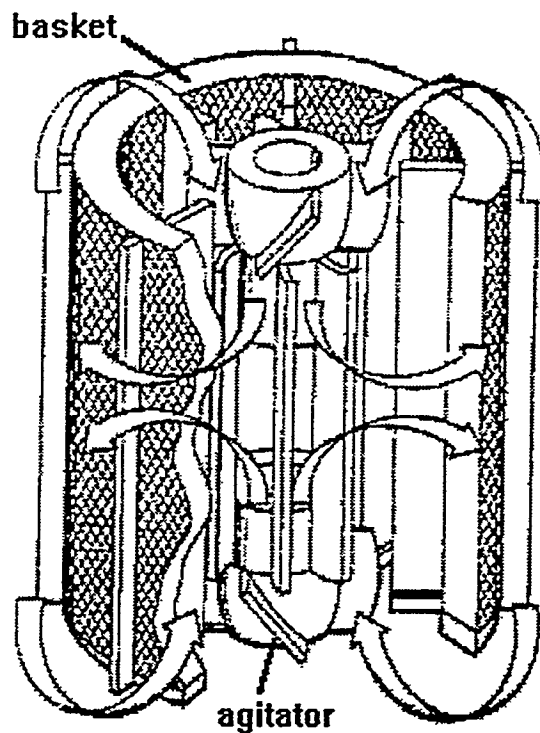
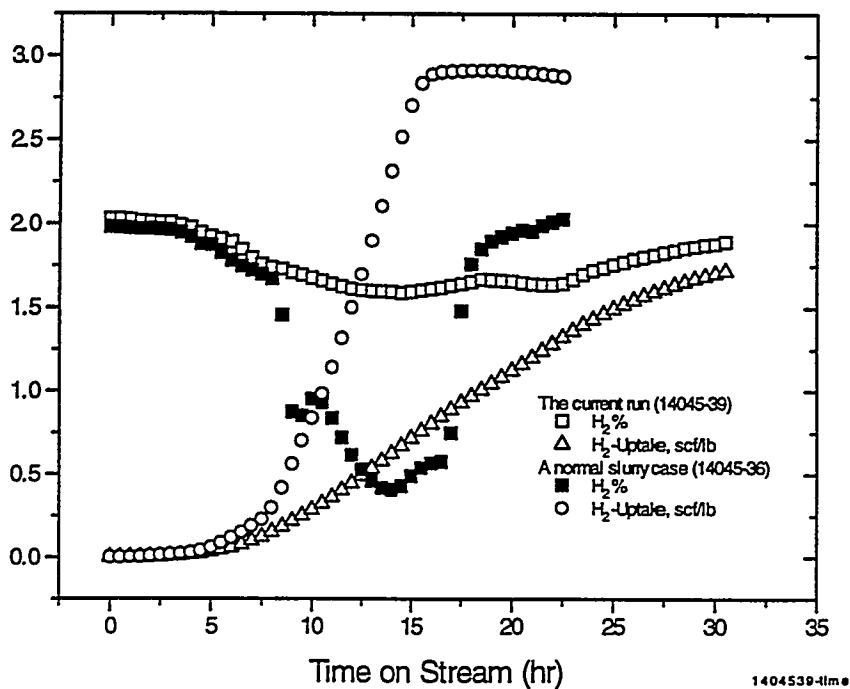
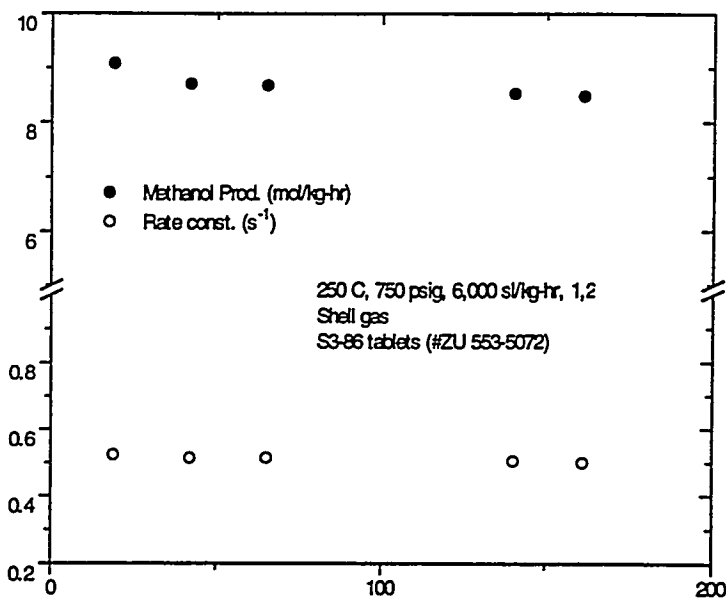


Figure 3.1.8. Profiles of Catalyst Reduction



**Figure 3.1.9. Shakedown Run of the 300 cc Autoclave with Robinson-Mahoney Basket Internals**



**Figure 3.1.10. Activity as a Function of Stirring Speed**

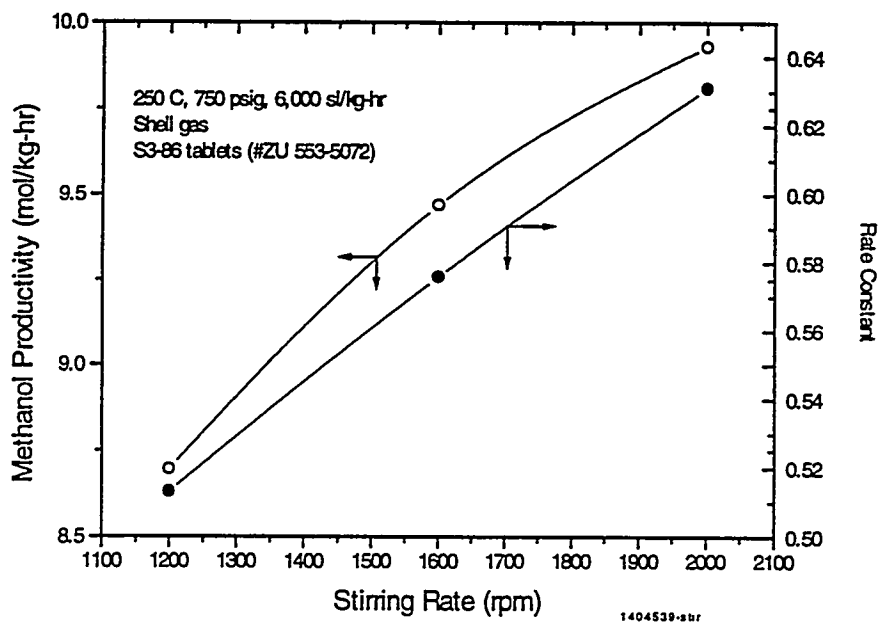
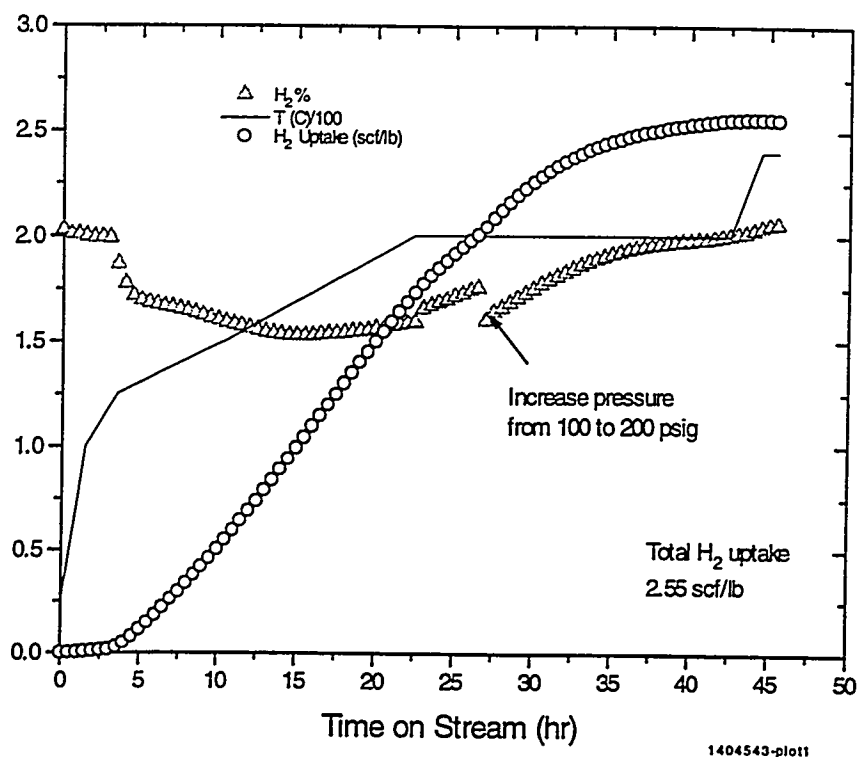


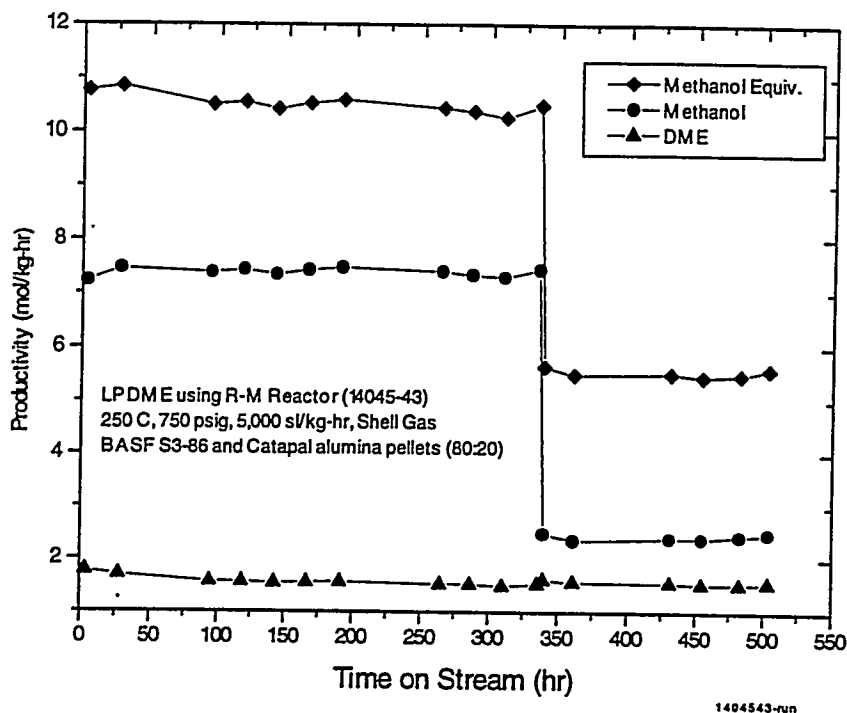
Figure 3.1.11. Reduction Profiles of the Robinson-Mahoney Run (10454-43)



### LPDME Run

The first part of the LPDME run was carried out at 250°C, 750 psig and 5000 GHSV using Shell gas. The stirring rate was higher than that used in slurry phase runs (1,600 vs. 1,200 rpm) to enhance mass transfer. As shown in Figure 3.1.12, the productivity of the system (10.5 mol/kg-hr) was only one third of the initial productivity (30 mol/kg-hr) of a mixture of S3-86 and Catapal alumina powders, indicating that the reaction was mass transfer limited. However, if the catalyst system deactivated, the reaction rate would eventually be comparable to the mass transfer rate, and from then on catalyst deactivation would be observable. The run was allowed to proceed for 350 hr more than needed for the reaction rate to slow down below the mass transfer rate, but no deactivation was observed. The stirring rate was then increased to 2,000 rpm to boost mass transfer rate, and space velocity was decreased from 6,000 to 1,500 sl/kg-hr to decrease the reaction rate, giving a better chance to observe catalyst deactivation. However, still little deactivation was detected up to 500 hr on stream.

Figure 3.1.12. LPDME Run Using Robinson-Mahoney Basket Internals (14045-43)



The run was stopped at this point. Part of the spent catalyst pellet mixture was preserved for analysis, and part was ground into powder in a nitrogen box and charged back in the autoclave reactor to check its activity. The reactor was operated in the slurry phase mode and free of mass transfer limitations. The powder mixture underwent the standard reduction. The minimal  $H_2$  uptake, 0.17 scf/lb, occurred mainly between 100 and 130°C. The activity was checked under the standard conditions, and the results (14045-52) are summarized in Table 3.1.3.

Also listed in Table 3.1.3 for comparison are the activity data from two other experiments. The first one, 14045-58, was conducted using the powders prepared by crushing the *fresh* S3-86 and Catapal B pellets from the same batches used for the Robinson-Mahoney run. This measured the initial activity of the catalyst system used for the Robinson Mahoney run. The second experiment (11782-3) was a standard LPDME life run using genuine powder samples. As shown in Table 3.1.3, the S3-86 powder prepared from the fresh S3-86 pellets had the same activity as the genuine S3-86 powder. The dehydration activity of the Catapal B powder prepared from the fresh pellets was only slightly lower (11%) than that of the genuine Catapal B powder.



**Table 3.1.3. The Activity of the Catalysts used in the LPDME Run using Robinson-Mahoney Basket Internals. Reaction conditions: 250°C, 750 psig, 6,000 sl/kg-hr, Shell Gas**

Run	Catalyst	Time on Stream, hr	MEOH Equiv. Prod. (mol/kg-hr)	Concentration (%)		Rate Constant	
				MEOH	DME	$k_m^b$	$k_d^c$
14045-52	powders from spent pellets	508	27.1	1.53	5.96	2.7	10.2
14045-58	powders from fresh pellets	20.5	30.6	0.78	7.06	2.9	15.1
11782-03	genuine powders	20	30.7	1.01	6.95	3.0	17.0
"	"	499	14.0	0.49	2.67	0.9	5.9

a: Methanol synthesis rate constant calculated from  $R_m = k_m k_{CO_2} f_{H_2}^{2/3} f_{CO}^{1/3} (1 - appr.)$ , based on methanol catalyst weight.

b: Methanol dehydration rate constant calculated from  $R_d = k_d f_{CO_2}^{-0.33} f_{MEOH}^{0.11} f_{CO}^{0.70} (1 - appr.)$ , based on alumina weight.

The results in Table 3.1.3 indicate that 500 hours in the Robinson-Mahoney run did not cause a significant change in the methanol catalyst activity (a 7% drop). For a similar period in the normal LPDME life run in a slurry phase reactor (11782-3), a much greater drop in activity was observed (70%). In fact, the activity of the methanol catalyst pellets dropped at a rate of only 0.013% hr<sup>-1</sup> in the Robinson-Mahoney run. This rate is even smaller than the S3-86 powder under LPMEOH conditions in a 300 cc autoclave (0.045% hr<sup>-1</sup>).

However, deactivation in the dehydration catalyst was observed in the Robinson-Mahoney run. The Catapal B pellets lost 32% of their activity upon 508 hours on stream. From this single experiment, there is no way to tell if this deactivation occurred only in the early period of the run or throughout the run. Note that in the normal LPDME life run (11782-3), the dehydration activity dropped by 65% in a similar period.

This experiment shows two important phenomena:

- 1) Point contact between methanol catalyst and alumina *pellets* does not cause deactivation of the methanol catalyst.
- 2) The fluid phase (mineral oil) neither causes nor transports anything that causes deactivation.

This suggests that certain physical features in a slurry phase operation, such as attrition which leads to the formation of methanol catalyst and alumina fines and good mixing between two catalyst powders, may be the cause or a necessary step for methanol catalyst to deactivate. For instance, these features may provide large contact area and long contact time, which are needed to foul the methanol catalyst or to go through solid phase reactions between methanol catalyst and alumina.

In contrast, the physical features associated with a slurry phase operation are not necessary for the alumina catalyst to deactivate. If one assumes that this deactivation is caused by migration of

Cu- and/or Zn-containing species from the methanol catalyst to the alumina, then this migration is conducted either by surface diffusion through the point-contact between the pellets of the two catalysts or mass transfer through the fluid medium, or both. More severe deactivation in dehydration activity occurred in the powder case for a similar period (Table 3.1.3), indicating that either this process can be accelerated by good mixing in a slurry phase operation, or there exists an additional mechanism for alumina deactivation, e.g., a process in concert with the deactivation of the methanol catalyst. In addition to this migration hypothesis, coking could be the cause of alumina deactivation. Elemental and coke analysis of the spent alumina sample will be conducted to resolve this issue.

**APPENDIX D      TASK 1.5.4 - PROJECT EVALUATION PLAN FOR B.P. NO.2**

Air Products and Chemicals, Inc.  
7201 Hamilton Boulevard  
Allentown, PA 18195-1501

Telephone (610) 481-4911  
Telex: 847416



July 18, 1995

Mr. Robert M. Kornosky  
PETC Technical Project Manager  
CT-10, Mail Stop 920-L  
U.S. Department of Energy/PETC  
P.O. Box 10940  
Pittsburgh, PA 15236

**Subject: Cooperative Agreement DE-FC22-92PC90543  
Liquid Phase Methanol Demonstration Project  
Project Evaluation Plan for Budget Period No. 2.**

Dear Bob:

The Project Evaluation Plan for Budget Period No. 2 is enclosed for your records. This final plan incorporates the minor editorial changes identified in your July 12, 1995 approval letter.

Very truly yours,

A handwritten signature in cursive script that reads "Bill".

William R. Brown  
DOE/CCT Program Manager  
LPMEOH Demonstration Project

Enclosure

cc: William C. Jones - Eastman Chemical Co.  
David P. Drown - APCI  
DOE Address List: A(2), B, F, J, L

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

**PROJECT EVALUATION PLAN FOR BUDGET PERIOD NO. 2**

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

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

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

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

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

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

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

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

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

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

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

**6. Phase 2 - Task 2- Construction**

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

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

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

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

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

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

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

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

---

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

WRB/jjs/Proeva.



**APPENDIX E      TASK 1.5.4 - PROJECT STATUS REVIEW MEETING (6/22/95)**

# LPMEOH DEMONSTRATION PROJECT

## 6/22/95 STATUS REVIEW MEETING WITH DOE

Introductions	ALL
Project Status	
Overview	WRB
Engineering, Design, and Equipment	DPD
Status Eastman Tie-in Work	LBP
Schedule	DPD
Current Spending and Current Capital Cost Estimate	DPD
NEPA	KMK
Project Management Plan Update	DPD
Reporting	DPD/RMK
Project Evaluation Plan	WRB
Commercialization Update	WRB
Wrap-up	All

### Attendees:

#### APCI

W. Brown

D. Drown

#### Eastman Chemical

W. Jones

L. Paulonis

S073

6/16/95

# LPMEOH DEMONSTRATION PROJECT STATUS

## SUMMARY APRIL-JUNE 1995

- COMPLETED EQUIPMENT ARRANGEMENTS
- COMPLETED DESIGN HAZARD REVIEWS
- STARTED CIVIL/STRUCTURAL DETAIL DESIGN IN MAY
- STARTING DETAIL PIPING LAYOUTS IN JUNE
- 53 OUT OF 66 EQUIPMENT ITEMS PURCHASED
- COMPLETED CLASS III COST ESTIMATE
- START OF CONSTRUCTION - SEPTEMBER 1995

# LPMEOH DEMONSTRATION PROJECT

## PLANS FOR JULY - AUGUST 1995

- Develop civil/structural schedule and provide manpower to assure that civil and underground work in the field is completed before 1/15/96.
- Process Controls to provide control valve sizing to support Piping Design needs. Start other instrument specs.
- Build Piping Design staff.
- Start electrical/instrument detail design.
- Manual valves out for bids.
- Complete EMP and EIV update.
- Update detail design and construction schedules.
- Establish bid list for civil and underground construction contractors.
- Further investigate reactor shipment.

*255,000 lb.*



# LPMEOH DEMONSTRATION PROJECT

## PHASE 1&2 CURRENT SPENDING THRU 4/30/95 POST MOD 003

<u>Phase 1</u>		<u>Contract Value, \$M*</u>	<u>Spent to Date, \$M**</u>	<u>% Spent</u>
1.1.1	Project Definition	1,230	1,010	82
1.1.2	Permitting	288	192	67
1.1.3	Design Engineering	8,206	1,976	24
1.1.4	Off-site Testing	324	0	0
1.1.5	Planning, Admin., & DME DVT	<u>1,663</u>	<u>688</u>	<u>41</u>
	Subtotal:	11,711	3,866	33
<u>Phase 2</u>				
1.2.1	Procurement	10,953	427	4
1.2.2	Construction	11,500	0	0
1.2.3	Training and Commissioning	897	0	0
1.2.4	Off-site Testing	305	0	0
1.2.5	Planning and Admin.	<u>681</u>	<u>0</u>	<u>0</u>
	Subtotal:	24,336	427	2
	Total:	36,047	4,293	12

\*From 9/26/94 Cost Plan

\*\*Actual spending and not committed.

NOTES FROM MEETING

DISTRIBUTION (NAME/ORGANIZATION) *Unable to attend. **Chairman			COPIED FOR INFORMATION ONLY
<u>JOE</u>	<u>APCI</u>	<u>Eastman</u>	F. Frenduto/A12B2 E. Schaub/A12B3 E. Heydorn/MC83
J. Archer	W. Brown/A12B2	W. Jones	
M. Dean	D. Drown/A12B2	L. Paulonis	
K. Khonsari			
L. Kornosky			
S. Rao			
T. Reuther			
L. Schehl			

FROM J. Drown		ORGANIZATION ACPI - Project Engineering	EXTENSION 1-6143	TODAY'S DATE 11 October, 1995
DATE OF MEETING 10/22/95	WEEKDAY Thursday	TIME STARTED 8:30 AM ENDED 3:00 PM		LOCATION PETC - Pittsburgh

SUBJECT AND/OR PURPOSE  
 1. PMEOH Demonstration Project  
 10/22/95 Status Review Meeting with DOE

ITEM NO.	RESPONSIBLE PERSON (INITIALS)	TARGET DATE	DISCUSSION
1.			The following notes highlight action items from the DOE Status Report meeting held at PETC's offices on 22 June, <del>1996</del> 1995.
2.			There were no comments received on the Environmental Assessment (EA) issued by PETC. A copy of the EA was placed in the Kingsport Library Reading Room. A notice of this copy was in the local newspaper on Mother's Day. It looks like a FONSI should be issued for this project.
3.	F. Frenduto		Jacobs Engineering requested a copy of the EA. The EIV should be updated after the FONSI is issued. The updated EIV will be a public document.
4.		3/1/98	The scope of DME production will not be discussed in the EMP because we do not know what we will design for this potential add-on. A separate EA write-up will be done after we decide what we will produce.
5.			A copy of the DESTEC Plaquemine, LA air-toxics report will be forwarded to APCI to review for our EMP. The DOE is interested in what air toxics may be in the incoming syngas, in the spent catalyst and/or in the methanol product.

NOTES FROM MEETING  
CONTINUATION

ITEM NO.	RESPONSIBLE PERSON (INITIALS)	TARGET DATE	DISCUSSION
6.	W. Brown D. Drown		APCI should define some topical reports to write on the LPMEOH Demonstration Project.
7.	W. Brown		APCI gave <sup>a</sup> summary of our commercialization plans for the LPMEOH process. The Kingsport LPMEOH plant will be started up in late 1996. There is interest in Europe & Japan for our process. We should look at updating the 1989 Commercial Plans.
8.			<p>Opportunities for future meetings with the DOE include:</p> <ul style="list-style-type: none"> <li>Ground Breaking (10/96)</li> <li>Review of 3-D CADD Plant Model (10/96)</li> <li>90% Design Completion (12/95)</li> <li>Update on DME (11/95)</li> <li>20% Construction Completion (3/96)</li> <li>Update of Off-site Fuel Testing (4/96)</li> </ul> <p>A copy of the meeting handouts is attached.</p> <p>DJD/MM024</p>



**APPENDIX F**

**TASK 2.1 - EQUIPMENT PURCHASE STATUS**

Kingsport Liquid Phase Methanol Project 00-3-8215  
Specification Status - Equipment

Tag #	Description	Process Engineer	Process Spec Released(Rev)	Equipment Specifier	Equipment Released(Rev)	Eastman Comments	Vendor List*	Released to Purchasing	Bids Due	Bids Received	Order Placement	Vendor Prints Due/Received
C-01	LPMEOH Reactor	Schaub	6/10/94 (0)	Koeller	11/02/94 (1)	9/18/94	Y	8/29/94	9/29/94	10/3/94	1/18/94	Rec'd 1/3/95
C-02	Steam Drum	Schaub	10/19/94 (1)	Koeller	11/07/94 (1)	0/19/94	Y	11/8/94	12/6/94	12/7/94	3/16/95	Rec'd 4/18/95
C-03	HP Methanol Separator	Schaub	11/18/94 (1)	Koeller	01/09/95 (2)	12/15/94	Y	1/5/95	2/2/95	2/10/95	3/15/95	Rec'd 4/04/95
C-05	Secondary Oil Knockout Vessel	Schaub	11/18/94 (1)	Koeller	1/09/95 (2)	12/15/94	Y	1/5/95	2/2/95	2/10/95	3/15/95	Rec'd 4/04/95
C-06	Reactor Cyclone	Schaub	11/23/94 (2)	Koeller	11/30/94 (1)	11/30/94	Y	12/2/94	12/28/94	12/16/94	3/21/95	Rec'd 4/18/95
C-07	Syngas Compressor Knockout Separator	Mazdai	03/22/95 (0)	Koeller	3/23/95 (0)	4/5/95	Y	4/25/95	5/17/95	5/17/95	6/5/95	Due 7/3/95
C-10	Methanol Stabilizer Column	Stein	11/11/94 (2)	Koeller	11/11/94 (2)	9/21/94	Y	10/4/94	11/1/94	11/1/94	12/20/94	Rec'd 1/25/95
C-10T	Methanol Stabilizer Column Trays	Stein	11/11/94 (2)	Koeller	11/11/94 (2)	10/11/94	Y	9/7/94	10/14/94	10/7/94	1/10/95	Rec'd 3/02/95
C-11	Methanol Stabilizer Reflux Drum	Stein	12/30/94 (2)	Koeller	01/09/95 (1)	12/15/94	Y	1/10/95	2/7/95	2/17/95	3/14/95	Rec'd 4/18/95
C-12	Methanol Stabilizer Feed Vessel	Stein	11/18/94 (0)	Koeller	01/09/95 (1)	12/15/94	Y	1/10/95	2/7/95	2/17/95	3/14/95	Rec'd 4/26/95
C-13	Methanol Stabilizer Condensate Pot	Stein	03/09/95 (0)	Koeller	3/27/95 (1)	3/27/95	Y	3/28/95	4/18/95	4/20/95	5/15/95	Rec'd 6/6/95
C-20	Methanol Rectifier Column	Stein	11/11/94 (2)	Koeller	11/11/94 (2)	9/21/94	Y	10/4/94	11/1/94	11/1/94	12/20/94	Rec'd 1/25/95
C-20T	Methanol Rectifier Column Trays	Stein	11/11/94 (2)	Koeller	11/11/94 (2)	10/11/94	Y	9/7/94	10/14/94	10/7/94	1/10/95	Rec'd 3/9/95
C-21	Methanol Rectifier Reflux Drum	Stein	12/30/94 (2)	Koeller	01/09/95 (1)	12/15/94	Y	1/10/95	2/7/95	2/17/95	3/14/95	Rec'd 4/18/95
C-23	Methanol Rectifier Condensate Pot	Stein	03/09/95 (0)	Koeller	3/27/95 (1)	3/27/95	Y	3/28/95	4/18/95	4/20/95	5/15/95	Rec'd 6/6/95
C-30	Catalyst Reduction Vessel	Bhatt	8/23/94 (0)	Koeller	10/03/94 (1)	9/21/94	Y	10/4/94	11/1/94	11/1/94	12/16/94	Rec'd 1/12/95
C-31	Reduction Condensate Accumulator	Mazdai	3/07/95 (2)	Koeller	01/09/95 (1)	12/15/94	Y	1/10/95	2/7/95	2/17/95	3/14/95	Rec'd 4/26/95
C-32	Utility Oil Surge Tank (V-01 Skid)	Bhatt	9/29/94 (0)	Koeller	12/02/94 (1)	11/23/94	Y	12/5/94	1/12/95	1/9/95	3/29/95	Due 4/26/95
C-40	Guard Bed	Chen	03/10/95 (0)	Koeller	3/27/95 (1)	3/27/95	Y	3/28/95	4/20/95	5/3/95	5/19/95	Due 6/16/95
C-45	Oil-Water Separator/Coalescer	N/A		Skuchas								
C-120	Vent Scrubber											
D-01	Safety Relief Knockout Drum											
D-02	Slurry Tank	Mazdai	3/07/95 (2)	Koeller	3/08/95 (2)	3/3/95	Y	3/9/95	4/3/95	4/7/95	6/26/95	
D-20	Methanol Lot Tank	Stein	3/07/95 (1)	Koeller	3/09/95 (1)	3/8/95	Y	3/13/95	4/10/95	4/12/95	Tech Eval Required	
D-21	Methanol Lot Tank	Stein	3/07/95 (1)	Koeller	3/09/95 (1)	3/8/95	Y	3/13/95	4/10/95	4/12/95	Tech Eval Required	
D-25	Methanol Drain Tank	Frenduto/Stein	4/21/95 (1)	Skuchas								
D-30	Fresh Oil Storage Tank	Mazdai	3/30/95 (2)	Koeller	3/08/95 (2)	3/3/95	Y	3/9/95	4/3/95	4/7/95	6/26/95	
D-60	Caustic Mix Tank	Stein	2/03/95 (1)	Koeller	3/08/95 (2)	3/3/95	Y	3/9/95	4/3/95	4/7/95	6/26/95	
D-70	Compressor L.O. Sump (K-01 Skid)	Schaub	4/22/94 (1)	Fleischer	08/29/94 (1)	5/17/94	Y	5/17/94	6/12/94	6/12/94	8/12/94	Rec'd 10/14/94
E-01	Syngas Compressor Cooler	Chen	03/31/95 (5)	Koeller	4/26/95 (4)	10/11/94	Y	11/8/94	12/21/94	12/20/94	3/28/95	Rec'd 5/10/95
E-02	Syngas Feed/Product Economizer	Schaub	8/29/94 (1)	Koeller	9/16/94 (1)	9/7/94	Y	9/19/94	10/14/94	10/24/94	12/22/94	Rec'd 1/27/95
E-03	Methanol Product Air-Cooled Condenser	Chen	10/14/94 (0)	Koeller	4/10/95 (2)	12/6/94	Y	12/1/94	1/5/95	1/9/95	3/24/95	Rec'd 4/20/95
E-04	Methanol Product C.W. Condenser	Chen	10/21/94 (1)	Koeller	11/7/94 (2)	11/1/94	Y	11/8/94	12/21/94	12/20/94	3/28/95	Rec'd 5/19/95
E-10	Methanol Stabilizer Reboiler	Stein	10/10/94 (2)	Koeller	11/07/94 (1)	10/19/94	Y	11/8/94	12/21/94	12/20/94	3/28/95	Rec'd 6/5/95
E-11	Methanol Stabilizer Condenser	Stein	10/17/94 (3)	Koeller	11/7/94 (2)	10/11/94	Y	11/8/94	1/9/95	12/28/94	3/28/95	Rec'd 5/10/95
E-20	Methanol Rectifier Reboiler	Stein	10/10/94 (1)	Koeller	11/07/94 (1)	10/19/94	Y	11/8/94	12/21/94	12/20/94	3/28/95	Rec'd 6/5/95
E-21	Methanol Rectifier Air Cooler	Chen	03/31/95 (2)	Koeller	11/30/94 (1)	12/6/94	Y	12/1/94	1/5/95	1/9/95	3/24/95	Rec'd 4/20/95
E-22	Methanol Rectifier C.W. Condenser	Stein	10/18/94 (1)	Koeller	11/7/94 (2)	11/1/94	Y	11/8/94	1/9/95	12/28/94	3/28/95	Rec'd 5/11/95
E-23	Grude Methanol Cooler	Stein	10/09/94 (1)	Koeller	11/7/94 (2)	11/1/94	Y	11/8/94	1/9/95	12/28/94	3/28/95	Rec'd 5/11/95
E-31	Reduction Vessel Overhead Condenser	Mazdai	10/14/94 (0)	Koeller	11/7/94 (2)	11/1/94	Y	11/8/94	1/9/95	12/28/94	3/28/95	Rec'd 5/11/95
E-32	Utility Oil Heater (V-01 Skid)	Bhatt	9/29/94 (0)	Koeller	12/02/94 (1)	11/23/94	Y	12/5/94	1/12/95	1/9/95	3/29/95	Due 4/26/95



**APPENDIX G      SIMPLIFIED PROCESS FLOW DIAGRAM**

# SIMPLIFIED PROCESS DIAGRAM KINGSPORT LPMEOH - 260 TPD PLANT

## LPMEOH™ PROCESS DEMONSTRATION FACILITY

