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

TECHNICAL PROGRESS REPORT NO. 5

For The Period

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and

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

MASTER

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ACRONYMS AND DEFINITIONS

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 (H₂), carbon monoxide (CO), and

carbon dioxide (CO₂) in stoichiometric balance for the production of methanol

Carbon Monoxide Gas A syngas containing primarily carbon monoxide (CO); also called CO Gas

DME - dimethyl ether

DOE - United States Department of Energy

DOE-PETC The DOE's Pittsburgh Energy Technologh Center (Project Team)
DOE_HQ The DOE's Headquarters - Clean Coal Technology (Project Team)

DTP Demonstration Test Plan - The four year Operating Plan for Phase 3, Task 2 Operation

DVT - Design Verification Testing
Eastman Eastman Chemical Company
EIV - Environmental Information Volume
EMP Environmental Monitoring Plan
EPRI - Electric Power Research Institute

HAPs - Hazardous Air Pollutants

Hydrogen Gas A syngas containing an excess of hydrogen (H₂) over the stoichiometric balance for

the production of methanol; also called H₂ Gas

IGCC - Integrated Gasification Combined Cycle, a type of electric power generation plant IGCC/OTM An IGCC plant with a "Once-Thru Methanol" plant (the LPMEOH™ Process) added-on.

KSCFH - Thousand Standard Cubic Feet per Hour

LaPorte PDU - The DOE-owned experimental unit (PDU) located adjacent to Air Product's industrial

gas facility at LaPorte, Texas, where the LPMEOH™ process was successfully piloted.

LPDME - Liquid Phase DME process, for the production of DME as a mixed coproduct with

methanol

LPMEOH™ - Liquid Phase Methanol (the technology to be demonstrated)

MTBE - methyl tertiary butyl ether

NEPA - National Environmental Policy Act

OSHA Occupational Safety and Health Administration
Partnership - Air Products Liquid Phase Conversion Company, L.P.

PDU - Process Development Unit PFD Process Flow Diagram(s)

ppb parts per billion

Project - Production of Methanol/DME Using the LPMEOHTM Process at an

Integrated Coal Gasification Facility

psia - Pounds per Square Inch (Absolute)
psig - Pounds per Square Inch (gauge)
P&ID - Piping and Instrumentation Diagram(s)

SCFH - Standard Cubic Feet per Hour

Sl/hr-kg - Standard Liter(s) per Hour per Kilogram of Catalyst

Syngas Abbreviation for Synthesis Gas

Synthesis Gas A gas containing primarily hydrogen (H₂) and carbon monoxide (CO), or mixtures of

H₂ and CO; intended for "synthesis" in a reactor to form methanol and/or other

hydrocarbons (synthesis gas may also contain CO₂, 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 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. As subcontractor to the Partnership, Air Products will also provide the engineering design, procurement, construction, and commissioning of the LPMEOHTM 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 LPMEOHTM Process Demonstration Unit, and for the interconnection and supply of synthesis gas, utilities, product storage, and other needed services.

The project involves the construction of a 80,000 gallons per day (260TPD) methanol unit utilizing coal-derived synthesis gas from Eastman's integrated coal gasification facility. The new equipment consists of synthesis gas 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 LPMEOHTM 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 will be integrated with existing coal-gasifiers. A carefully developed test plan will allow operations at Eastman to simulate electricity demand load-following in coal-based IGCC facilities. The operations will also demonstrate the enhanced stability and heat dissipation of the conversion process, its reliable on/off operation, and its ability to produce methanol as a clean liquid fuel without additional upgrading. An off-site product testing program will be conducted to demonstrate the suitability of the methanol product as a transportation fuel and as a fuel for stationary applications for small modular electric power generators for distributed power.

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

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

The project was reinitiated in October of 1993, when DOE approved a site change to the Kingsport location. DOE conditionally approved the Continuation Application to Budget Period No. 2 (Design and Construction) in March of 1995, and formally approved it on 1 June 1995 (Mod M009). Since then the project has been in the project definition phase. During this last quarter the project transitioned to the design phase. 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 a Finding of No Significant Impact (FONSI) was issued on 30 June 1995. Construction of the demonstration unit is scheduled to begin in October of 1995, and the unit will be mechanically complete in November of 1996.

During this quarter, the main process design work was completed, and work was started on secondary systems (e.g., utilities, vents). Piping design and structural design are at peak levels, and electrical design has started.

A draft "Process Development/Value Engineering" plan was issued identifying specific needs for site-specific commercial plant studies. Work on this plan will be done in parallel with development of the operation test plan during October of 1995 through June of 1996.

Results of the California South Coast Air Quality Management District (SCAQMD) testing of 3 wt% DME as a cold-start additive to methanol as a diesel fuel replacement were not encouraging. Although DME failed as a cold-start additive, the tests did indicate DME was a promising fuel.

Procurement of process equipment is nearly complete. Twenty-seven percent of the \$36 million in funds authorized for the LPMEOH™ Process Demonstration Project through Budget Period No. 2 have been expended as of 30 September 1995.

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 will be designed and constructed at a site located at the Eastman Chemical Company (Eastman) complex in Kingsport. The Partnership will own and operate the facility for the four-year demonstration period.

This project is sponsored under the DOE's Clean Coal Technology Program, and its primary objective is to "demonstrate the production of methanol using the LPMEOHTM 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

Existing Site

The demonstration unit, which will occupy an area of 0.6 acre, 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 LPMEOHTM Process Demonstration. Three different feed gas streams (hydrogen gas, carbon monoxide gas, and balanced gas) will be diverted from existing operations to the LPMEOHTM demonstration unit, 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 a synthesis gas recycle compressor, 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.

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.

Storage/Utility Area

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

C. Process Description

The LPMEOH™ demonstration unit will be integrated with Eastman's coal gasification facility. A simplified process flow diagram is included in Appendix A. Synthesis gas is 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 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 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 F). 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 EA/FONSI.
 - The NEPA review was completed 30 June 1995 with the issuance of an Environmental Assessment (DOE/EA-1029) and a FONSI. The final EIV is being prepared.
- 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).
 - Work was begun on the draft EMP.
- Complete the design engineering necessary for construction and commissioning. This included Piping and Instrumentation Diagrams, Design Hazard Reviews, and the conduct of design reviews.
 - During this reporting period, Process Engineering work focused on:
 - Completion of specifications for instruments and relief devices
 - Vent header design
 - Small bore piping sizing
 - Engineering work focused on:
 - Review of vendor data
 - Completion of valve specification
 - Design of utility system piping
 - Initiation of mechanical specifications for safety relief valves
 - Control valve detailed design to support piping design information needs
 - Initial work on distributed control system (DCS) design and vendor selection
 - Design work focused on:
 - Completion of the site preparation (e.g., storm sewers, roads, temporary power) bid package, which was sent out for bids

- Completion of the foundation bid package
- Design of structural steel in all areas
- Design of piping. The effort peaked with nine designers actively involved.
- Initiation of electrical design
- Safety focused on continuing to resolve issues that were identified in the Hazards reviews.

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 test plan developed to support the demonstration project at the original Cool Water Gasification Facility in 1992 has become outdated. Since the site change to Eastman, the original fuel test plan now 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.

The objective of the fuel 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 following fuel-use test plan basis has been 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 unit will be made available for fuel-use tests. Fuel-use tests will be targeted for an approximate 18-month period starting in January of 1998. The methanol product will be available ex-works from the demonstration unit 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 LPMEOHTM technology. The Electric Power Research Institute (EPRI) will provide guidance in identifying the application (e.g., 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 (Acurex) will develop the final fuel-use test plan, which is to be prepared by June of 1996.

Activity during this quarter:

- Tentative plans were made to meet with Acurex and with EPRI during October of 1995, and in conjunction with EPRI's power generation (gasification) conference. The agenda with Acurex will include Fuel-Use Test Plan Update Planning and Subcontract Revisions needed to conform to the Statement of Work (incorporated with Mod. 008 to the Cooperative Agreement) and to the revised project schedule that has been developed since the project was relocated to Kingsport in October of 1993.

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

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 will update plans for the on-site product use demonstrations. The schedule for on-site product use tests was established for August to October of 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 of 1996.

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 operating test plan (Phase 2, Task 3).

- The draft Process Development/Value Engineering plan was issued. A number of areas were identified as needing development to support site specific commercial plant studies. These include:
 - Product Purification options
 - Front End Impurity Removal options
 - Catalyst Addition/Withdrawal options
 - Plant Design Configuration options, and
 - Tools for use in screening bids

The draft plan (dated 14 July 1995) is included in Appendix B. Work on this plan will be done in parallel with development of the Kingsport operation test plan (Phase 2, Task 3), during October of 1995 through June of 1996.

- The Kingsport design will be used as the basis for developing the first commercial LPMEOH™ plant designs. Items which are specific to the Kingsport site will be identified and deleted. The process flowsheets (PFD's and P&ID's) will be broken out into smaller sections (e.g., catalyst preparation, reaction, distillation) so that value engineering work can focus on essential cost reduction targets. A memo (dated 14 August 1995) transmitting the initial work is included in Appendix C.

A few site-specific studies (Japan, Europe, EPRI), including dispatch (electric power) analysis and IGCC design optimization will be developed. These studies could be done in parallel with the value engineering work and would help establish "real" commercial design targets. Deregulation of the electric power generation industry has changed the traditional U.S. utility's focus on longer term planning and has delayed interest in these studies.

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 first decision point, on whether to continue with DME DVT, is targeted for December of 1996. DVT is required to provide additional data for engineering design and demonstration decision-making. The essential steps required for decision-making are: a) confirm catalyst activity and stability in the laboratory, b) develop engineering data in the laboratory, and c) confirm market(s), including fuels and chemical feedstocks. The DME Milestone Plan, showing the

DVT work and the decision and implementation timing, is included in Appendix D.

Action during this quarter included Market Economic Studies and Laboratory R&D.

Market Economic Studies

Testing of up to 3 wt% DME as a cold-start additive to methanol as a diesel engine replacement fuel was completed by the Los Angeles Rapid Transport District (LARTD). The testing was performed for the California South Coast Air Quality Management District (SCAQMD) and for the DOE (Contract No. DE-AC22-91PC90018). The Technical Report for the tests is included in Appendix D. The results, for DME as a cold-start additive, were not encouraging. A summary of the report follows:

In 1993, Air Products proposed to SCAQMD the use of DME as a possible cold-start additive to methanol for compression ignition diesel engines. Measurements on cetane number were encouraging and the vapor pressure characteristics, similar to propane, were deemed appropriate. DME can be concentrated in methanol up to 3 wt% under ambient conditions. At this concentration the vapor is essentially pure DME. Maintaining DME at a constant (3 wt%) level in the base-line fuel methanol, with (hopefully) cold-start capabilities but without fuel vapor-lock problems, would be the easiest DME cold-start option. More difficult options, such as generating DME "on-board" only at the time of cold-start requirements by dehydrating fuel methanol to DME using an electronically heated catalytic device, had already been considered, but with mixed results.

The means of production of DME and associated cost were obviously vital for the decision to test DME as a cold-start additive. Conventionally, DME is made from the dehydration of methanol, with DME product price reflecting the (historically highly variable) price of feedstock methanol. Air Products and the DOE had pioneered the one-step synthesis of DME from synthesis gas with proof-of-concept testing at the DOE's process development unit in LaPorte, Texas. A particularly attractive feature of this new process, Liquid Phase DME (LPDME), is the ability to vary the concentration of DME with methanol in the final product. It is possible to produce the DME via this technology for less than the equivalent cost of producing methanol.

In 1994, DME, under terms of a SCAQMD contract, was evaluated as a cold-start additive. LaPorte fuel-grade M-100 methanol, which had up to 3 wt% of DME added to it, was tested in the Los Angeles Rapid Transport District (LARTD) bus fleet. The fuel was evaluated in those buses fitted with Detroit Diesel Corporation (DCC) custom methanol engines. Although "rough" ignition was apparent, the engine failed to start and run on a regular basis. Later tests with 5 wt% DME were tried, but the engine did not start convincingly. LARTD returned to the AVOCET (their regular additive for cold start) spiked methanol, which gave instant cold-start ignition and smooth running.

Further testing of DME as an additive was abandoned, since the cold-start requirements of the custom-built DDC methanol engines could not be met.

Although DME failed as a cold-start additive, the tests did indicate DME was a promising fuel. Once started, increased amounts of DME did lead to increasing improvements in engine smoothness and power. Some emission testing was also done, indicating that the bus-engine ran cleaner (less nitrogen oxides) than on methanol with DME additive, than on methanol with normal AVOCETTM additive. More recent work by AMOCO and others (see Quarterly Technical Progress Report No. 4) has shown that much higher concentrations (up to 90 wt%) of DME works excellently as a diesel fuel alternative, with very good cold-start capability, improved tail-pipe emissions, and easy retrofit capability to existing diesel engines. Higher concentrations of DME is included as item B. 3. in the DME milestone plan in Appendix D; and will be investigated by August of 1996, in time for the DVT decision expected in December of 1996.

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 LPMEOHTM 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 Alternative Fuels Development Unit (AFDU), 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 LPMEOHTM process catalyst system. Further studies of the LPDME catalyst deactivation phenomenon were therefore undertaken under DOE Contract No. DE-FC22-95PC93052. 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. Seven dehydration catalysts were previously screened and none exhibited attractive performance. Several distinct modes of deactivation were identified, suggesting new directions for the continuing alternate dehydration catalyst research. The quarterly report, prepared under Contract DE-FC22-95PC93052 for the period April - June 1995, is included in Appendix E for reference and is summarized in the following:

Summary of Laboratory Activity and Results

- Deactivation of the DME catalyst systems is associated with some physical features
 of a slurry phase reactor, since both methanol and dehydration catalysts do not suffer
 long-term deactivation in the LPDME run with Robinson-Mahoney basket internals
 and pelletized catalysts. Most likely, the intimate contact between the two catalysts is
 necessary for catalyst deactivation of the two-component system. This suggests new
 directions in future research to better understand the deactivation mechanism and to
 develop solutions.
- A repeat of the LPDME run in the Robinson-Mahoney reactor confirmed the result that the methanol catalyst does not deactivate significantly when only a small amount of physical contact is allowed by the catalysts. In addition, the long-term deactivation of the dehydration catalyst is eliminated.
- Modification of the alumina catalyst surface with silica (Si) reduced the initial steep deactivation seen in LPDME synthesis. However, the deactivation rate was still relatively high due to long-term deactivation. In another LPDME approach, a Sibased dispersion aid gave the desired dispersion of the mixed catalysts in the slurry reactor, but the material polymerized during the run. More stable dispersants will be tried. A Si-modified Catapal B δ-alumina sample with a very high silica oxide content (35 wt%) was prepared using a polymeric Si-containing material. However, the modification resulted in little improvement in the long-term stability of the methanol catalyst.

In LPDME studies BASF S3-86 methanol catalyst exhibited a better long-term stability when used with a potassium-doped Catapal B δ -alumina. This is the first time that an improved long-term stability has been observed. However, the dehydration activity of this alumina was low due to high Potassium (K) loading. K-doped alumina samples of lower loading, and therefore higher dehydration activity, will be tested in the coming quarter to see if a similar stability can be obtained.

Task 1.5.4 Administration and Reporting

An updated Project Management Plan was being prepared. The updated plan will reflect the restoration of three budget periods, which is a change from the previous Project Management Plan submitted in October of 1994.

A ground breaking ceremony is planned for 16 October 1995, at the site in Kingsport. Invitations have been sent to federal, state, and local authorities, as well as to industrial customers and the media. Final plans are in progress. A copy of the invitation, and the agenda, are included in Appendix G.

A Project Review meeting is also being planned for 17 October 1995 at Kingsport.

The Milestone Schedule Status Report and the Cost Management Report, through September 30, 1995, are included in Appendix H. These two reports show the schedule, percent completion, and cost forecast for each of the Work Breakdown Structure tasks.

The overall Milestone Schedule, for completion of the major Budget Period No. 2 Tasks, is unchanged. Construction (Task 2.2) is still forecast for completion by 15 November 1996. Start-up activities (Task 3.1) are forecast to commence on 31 October 1996, and to be completed by 01 February 1997.

The cost forecast is unchanged. A number of the tasks are now completed, or are well underway. The Project Definition (Task 1.1) and Permitting (Task 1.2) tasks are essentially complete. The Design Engineering (Task 1.3), the Planning, Administration, & DME -DVT (Tasks 1.5) and Procurement (Task 2.1) tasks are more than 67% complete. Therefore, an updated cost estimate of each of the WBS tasks is now being undertaken. This reforecast, by task, will be submitted next quarter as part of the Cost Plan for fiscal year 1996. No change in the total cost forecast is anticipated, although there will be variations within the tasks.

The monthly reports for July, August and September 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 Productssupplied construction materials.
 - The status of equipment purchases is shown in the "Specification Status-Equipment" report included in Appendix I. This report, prepared in mid-September, shows that orders have been placed for all but 3 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.
 - Eastman completed the remaining tie-ins of process and utility lines to their existing facilities. Site preparation work is scheduled to begin in the next quarter.

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.

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.

- The update of the Partnership annual plan was prepared and submitted. The goal and objective for the fiscal year 1996 annual plan is to continue the Phase 1 and Phase 2 tasks required by the Statement of Work. The major objectives for the 1996 plan are:

- the LPMEOHTM demonstration unit will be ready for commissioning and startup in the 4th quarter of calendar year 1996.
- the Project Evaluation Report for Budget Period No. 2 is to be completed and submitted to the DOE along with the Continuation Application for Budget Period No. 3.

Specific Partnership Tasks (milestones, responsibilities) to meet these Partnership goals and objectives are included in Appendix J:

- Submit all Project status, milestone schedule, and cost management reports as required by the Cooperative Agreement.
 - The DOE reporting tasks are currently being performed and reported under Task1.5.

E. Planned Activities for the Next Quarter

- Award Site Preparation and Foundation contracts
- Conduct Ground-Breaking Ceremony, set for 16 October 1995
- Complete design of vent header system
- Award Prefab Piping contract
- Award Prefab Steel contract
- Purchase Distribute Control System
- Oversee arrival of some equipment on site
- Prepare and submit an updated Project Management Plan
- Prepare an updated cost estimate and submit the Cost Plan for Fiscal Year 1996.

F. Summary

The main process design work is complete, and work has started on secondary systems (e.g., utilities, vents). Piping design and structural design are at peak levels, and electrical design has started.

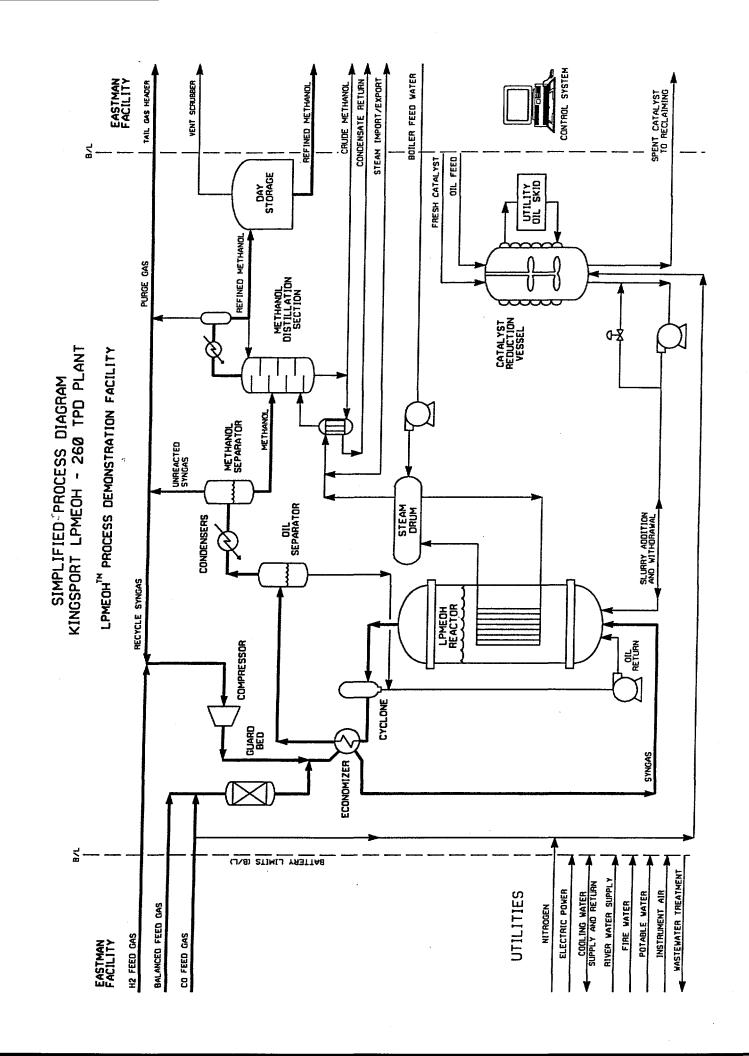
A draft "Process Development/Value Engineering" plan was issued identifying specific needs for site-specific commercial plant studies.

Results of testing of 3 wt% DME as a cold-start additive to methanol as a diesel fuel replacement were not encouraging.

Procurement of process equipment is nearly complete. Twenty-seven percent of the \$36 million in funds authorized for the Kingsport portion of the LPMEOH™ Process Demonstration Project through Budget Period No. 2 have been expended (as invoiced) as of 30 September 1995. The overall schedule for completion of the major Budget Period No. 2 tasks, is unchanged. Construction (Task 2.2) is still forecast for completion on 15 November 1996.

APPENDICES

APPENDIX A SIMPLIFIED PROCESS FLOW DIAGRAM



APPENDIX B TASK 1.5.2 - DRAFT PROCESS DEVELOPMENT/VALUE ENGINEERING PLAN

<u>Process Development/Value Engineering</u> - (Task 5) Work Plan Worksheet (for Objective 1.c). -

Description of Task

A. To Support the second Generic Plant Design

- A.1. Catalyst Addition/Withdrawal Options.
 - a) Periodic Partial Replacement (PPR): On-Line (Kingsport)
 - b) Periodic Partial Replacement (PPR): Off-Line
 - c) Replace All (like GP process)
- A.2. Reactor Temperature/Catalyst Life/Production Study
- B. To Support Site Specific Studies
- B1. Develop simplified (material balance) tools to answer LPMEOH screening bids.
- B2. Product Purification Options, Optimizations, Frandard Designs.
- B3. Front End Impurity Removal Options
 - Sulfur Guard Bed Design Basis
 - Economic Breakpoint for Guard Bed Alone (no solvent system)
 - Other Contaminants Guard Bed Options or Solvent Systems.
 - Need grass-roots and retrofit (add LPM-on later) options.
- B4. Plant design configuration studies:
 - 1. Feed Compression (when and how much).
 - 2. Configuration for 20, 50, 70% conversion.
 - 3. Plant size breakouts (single reactor vs. two).

Comments

- The Capital cost for PPR is high.
- PPR is operator intensive.
- Catalyst replacement costs for PPR is high (relative to GP process).
- J. Klosek feel constant nameplate production is important for Option c.
- Review lab R&D data to optimize Catalyst Life/Product Cost.
- Eliminate the need for process support on preliminary requests.
- Investigate Aspen "Optimizer" for . optimization studies.

Standardized designs for Chem grade, MTBE grade and fuel grade. Draw from Kingsport experience for equipment specifications, plot plan, etc.

Major issue for future sites. Kingsport is not providing any design basis. Currently using a 5 ppm "grab" number in SE's for grass-roots. Retrofit could be 40 ppm (?). Need to identify absorbents for other likely contaminants besides S and carbonyls. Review of literature, lab data, (work with Bechtel?).

Different gas compositions; combined with different conversion studies (below). Different gas compositions; combined with different compression studies (above).

APPENDIX C TASK 1.5.2-GENERIC PLANT PROCESS DESIGN

Memorandum



To:

R. B. Moore

Dept/Loc.:

Project Development/A12B2

From:

E. C. Heydorn

Dept/Ext:

GEG Technology - LaPorte

Date:

14 August 1995

Subject

LPMEOH Commercial Studies - Initial Cost Estimate from Kingsport Flowsheet

cc: D. M. Brown (PFD only)

W.:R.:Brown (PFD only)

J. Klosek (PFD only)

E. S. Schaub (PFD only)

As part of the activities under Task 5 for the Kingsport LPMEOH Clean Coal Demonstration project, I have made an an initial pass at marking up the Kingsport P&ID's to remove items which are specific to the Kingsport site. I have sent the entire flowsheet to RBM, and I have marked up the PFD for those on distribution for reference. Major scope differences include:

Runge

- one fresh feed gas commercially
- a single stabilizer column to produce fuel-grade methanol
- less instrumentation (removed site-specific and demonstration needs)

This mark-up is a starting point for your estimating work. Plant costs will be based upon the Kingsport Class 3 estimate. As we discussed, you are planning to break the flowsheet into smaller sections (Reaction, Catalyst Preparation, Distillation) so that we can identify those areas which may be cost problems in future bids. Once the costs have been segmented, we can start looking at areas where additional savings will be required.

ESS - Could you please calculate the maximum production rate minus 10% at 1 ft/sec inlet superficial velocity, 45 wt% slurry concentration, Fresh Feed Syngas only, and maximum slurry level that we could expect from the 29C-01? We should match the syngas utilization as Plant 19. RBM will adjust costs for the piping and equipment to reach the maximum production rate.

V (1) EMN. Belineselbe

(2) Co-ruch

After the Kingsport case, we will develop a cost for CO-rich syngas. In order to will need to have both cases completed by 31 October.

E. C. Heydorn

APPENDIX D TASK 1.5.3 - DME Milestone Plan TASK 1.5.3 - Technical Report (DE-AC22-91PC90018)

DME MILESTONE PLAN

1.	Design V	Verification Testing:		
	, A.	Laboratory R&D, Verification		
	1.	Catalyst Activity and Stability Testing, and Engineering Data	Mar. '95 - Aug. '96	
	В.	Market Verification		
	1.	Up to 3 wt% DME, as M-100 Diesel Replacement Engine Tests - (SCAQMID) - 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 Kin a. Economics, process basis study	by Aug. '96 gsport	
	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	
-	В.	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 to Jan. '01

<u>Technical Report</u>: DOE Contract No. <u>DE-AC22-91PC90018</u> and SCAQMD Contract No. <u>AB2766/C93046</u>

"USE OF DME AS A COLD-START ADDITIVE FOR FUEL METHANOL"

Background:

In the late 80's to early 90's, methanol was promulgated as a desirable alternative to conventional diesel fuel. Particularly in the inner cities and on the highways of the United States, the abilities of diesel-burning buses and trucks were viewed as doubtful in meeting the increasing emission standards that EPA and state agencies intended putting into place. The processing costs of cleaning up crude oil fractions to provide the necessary clean diesel fuel were considered prohibitive and this led to the search for alternatives which met performance and expected emission standards. Methanol was a favourite candidate for many. Available, immediately in the short term from natural gas and in the longer term (through DOE developed technology) from domestic coal reserves, and clean burning (compared to diesel), it offered a cost effective alternative. Although energy density was considerably less than diesel fuel, the environmental premium warranted its close consideration as a potential replacement. Unfortunately, for a variety of reasons, methanol in its pure form M-100, could not easily be used in existing diesel engines and extensive modifications were necessary. Some organisations, such as the Detroit Diesel Corporation (DDC) even developed whole new power units geared to the requirements of fuel methanol. Although these units overcame the problems of low cetane number, the problem of cold-start capability remained.

Conventional diesel engines had long used the glow-plug as a device for enabling cold-starts and this application could be extended to methanol fueled power units. However, compression ignition additives, such as AVOCETTM, had found their way into diesel engines, obviating the need for glow-plugs, and both the retrofit market and the new methanol-based engines were keen to avoid the complexity and expense of re-introducing the additional cold-start hardware. Cold-start additives for diesel engines have different characteristics to those expected for a gasoline engine. Whereas octane number and Reid Vapor Pressure are considered important parameters for ease of starting a gasoline power unit, little is publicly known about the starting requirements of a diesel or compression ignition engine. Vapour pressure and detonation capability under pressure are thought to be important.

In 1993, Air Products and Chemicals proposed to the California South Coast Air Quality Management District (SCAQMD) the use of dimethyl ether (DME) as a possible cold-start additive for both methanol and diesel engines. Measurements on cetane number were encouraging (approx. 50) and the vapour pressure characteristics, similar to propane, were deemed appropriate, particularly as it was known that, in practice, externally introduced squirts of regular 'ether' (diethyl ether) worked for cold-starting M-100 methanol power units. After the initial question 'does it work?', issues such as cost, how best to introduce it to the fuel-power unit, would need to be addressed. Would it be necessary to introduce it only on a 'cold' start - or should it be present in the fuel at all times?

The means of production of DME and associated costs were obviously vital. Conventionally, DME is made from the dehydration of methanol, with the DME product price reflecting the (historically highly variable) price of feedstock methanol. Air Products and the DOE had pioneered the one-step synthesis of DME from synthesis-gas with proof of concept demonstrated at the DOE's Alternative Fuels Development Unit (AFDU) in LaPorte, Texas (1991). A particularly attractive feature of this new process, Liquid Phase DME (LPDME), is the ability to vary the concentration of DME in the final product from 1% to 100%. It is possible to produce DME via this technology for less than the equivalent cost of producing methanol, making it a very attractive alternative in those technologies which use methanol as a feedstock.

DME can be concentrated in methanol up to 3 wt% under ambient conditions. At this concentration the vapour is essentially pure DME. This represents the easiest option - maintaining DME at a constant level in the base-line fuel methanol with (hopefully) cold-start capabilities but without fuel vapour-lock problems. The more difficult option would be to generate the DME 'on-board' only at the time of cold-start requirements. A number of people have already looked at this alternative, dehydrating fuel methanol to DME using an electronically heated catalytic device, with mixed results.

Tests at Los Angeles Rapid Transport District

In 1994, DME, under the terms of a SCAQMD contract and with the cooperation of EPRI, was evaluated as a cold-start additive at the down-town Bus Depot of the Los Angeles Rapid Transport District (LARTD). The objective was to test LaPorte fuel-grade M-100 methanol (minimum 96 wt% purity), which had up to 3 wt% of DME added to it, as fuel for a number of the LARTD bus fleet. The fuel would be evaluated in those buses fitted with DDC custom methanol engines - those buses did not have glow-plugs. Several 55 gallon drums of fuel methanol containing 3 wt% DME were shipped to Los Angeles from LaPorte, Texas. One of the LARTD methanol-powered bus fleet was made available for cold-start testing. After flushing through of the regular methanol fuel (laced with AVOCETTM, the additive LARTD use for cold-start capability) the DME-enhanced methanol fuel was introduced to the power unit. Although 'rough' ignition was apparent, the engine failed to start and run on a regular basis. A squirt of externally-introduced diethyl ether kicked-off the engine, and then re-introduction of the DME/MEOH fuel mixture led to continued, but very rough, running of the power unit. Later tests tried the addition of extra DME to the methanol fuel, up to 5 wt%, but, although cold-starting appeared imminent, the bus did not start convincingly. Once started, however, the increased amounts of DME led to ever-increasing improvements in engine smoothness and power demand. A return to AVOCETTM spiked methanol gave instant cold-start ignition and smooth running.

The indication that higher levels of DME addition were leading to improved capability were encouraging and although there was insufficient fuel available for road-trials, some emission testing was carried out at the LARTD EPA-approved laboratories. Results on both engine and chassis dynamometers were clear: a methanol-powered bus engine, once started, ran cleaner (less NOx) on methanol with DME additive than on methanol with normal AVOCETTM additive.

Conclusions and Future Work

A 3 wt% DME-in-methanol fuel did not satisfactorily address the cold-start requirements of the custom-built DDC methanol engine. Once running, however, tail-pipe emissions seem improved over M-100 methanol fuel spiked with conventional cold-start enhancers.

Increasing the concentration of DME in the fuel continues to improve the running and operability of the power unit. At the time however, further increases in DME level were not considered practical for DDC's methanol-powered engine - at least by considering its addition to the base-line fuel.

Which is a pity, as recent work by AMOCO and others has shown that much higher concentrations (> 90 wt%) of DME on its own, works excellently as a diesel alternative, with very good cold-start capability, improved tail-pipe emissions, and, best of all perhaps, ease of retrofit capability to existing diesel engines.

APPENDIX E TASK 1.5.3 - DME (DE-FC22-95PC93052) QUARTERLY REPORT (Excerpt from April - June 1995 Quarterly Technical Report, pages 31-47)

TASK 3: RESEARCH AND DEVELOPMENT

3.1 New Processes for DME

3.1.1 DME Catalyst Activity and Maintenance

The progress made in this quarter features more advanced understanding of the mechanism of catalyst deactivation under LPDME conditions. This understanding was obtained by analyzing the results from 1) screening runs using different dehydration catalysts, and 2) the experiments using Robinson-Mahoney basket internals and pelletized catalysts. This understanding provides new directions in solving the catalyst stability problem. The details of the analysis and the work conducted based on the new understanding are given below.

Analysis of the Trends in Catalyst Deactivation

We have reported previously that an interaction between the methanol synthesis and dehydration catalysts is responsible for catalyst deactivation under LPDME conditions. Since then, we have been screening for alternative dehydration catalysts that could be compatible with a standard methanol catalyst (e.g., BASF S3-86). One need for efficient screening is an idea of the properties required in a dehydration catalyst. Part of the answer to this question has been

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obtained by analyzing the results from the previous screening runs, including 14 samples described in the last two quarterly reports.

One of the key ideas developed during this analysis is that the catalyst deactivation is divided into four modes: the initial and long term deactivation of the methanol catalyst, and the initial and long term deactivation of the dehydration catalysts.

Major conclusions so far include:

- 1. The initial deactivation of the methanol catalyst is caused by the strong acid sites on the dehydration catalyst.
- 2. The initial deactivation of the dehydration catalyst is related to the type as well as the strength of the acid sites. Both strong acid sites and sites of Bronsted acid nature appear to deactivate rapidly.
- 3. The long term deactivation of both methanol and dehydration catalysts is not directly correlated to the acidity (e.g., the dehydration activity) of dehydration catalysts. Most dual catalyst systems show a similar rate of long term deactivation for the methanol catalyst. This rate is about a factor of 2 greater than that of the methanol catalyst-only system.

The following are the details of the analysis.

a. Observations of Deactivation of the Methanol Catalyst

Observations were made according to groups consisting of different dehydration catalysts. All systems used BASF S3-86 methanol catalyst as the other catalyst in the dual system.

Catalyst systems based on Catapal B g-alumina. Figures 3.1.1 and 3.1.2 display the activity of methanol catalyst and dehydration catalyst, respectively, as a function of time on stream for Catapal B-based catalyst systems, namely S3-86 plus virgin Catapal B or Catapal B modified by Si, WO₃, and ZnO. (Activity is measured by the rate constants of the methanol synthesis and dehydration reactions. The rate expressions for these rate constants and the adjustment performed to make the comparison between the different catalysts possible are given in the Appendix.) All data were obtained under standard conditions (i.e., 250°C, 750 psig, 6000 GHSV, Shell gas, and a methanol-to-dehydration catalyst ratio of 80:20).

As shown in Figure 3.1.1, the deactivation of the methanol catalyst can be divided into two stages: an *initial*, fast deactivation followed by a stage of slower but continuous, *long-term* deactivation. The division between the initial and long-term deactivation is the point at which the deactivation starts to slow down and the deactivation rate becomes almost constant. For example, for the catalyst system containing virgin alumina, the initial deactivation period stops at ca. 80 hr on stream. For the system containing ZnO-modified alumina, it stops at ca. 40 hr. The first observation from these two figures is that the methanol catalyst deactivates at a similar rate in the second stage, regardless of the activity of the dehydration catalysts. The deactivation in this stage is referred to as long-term deactivation hereafter.

Figure 3.1.1 "Normalized" Methanol Synthesis Rate Constant as a Function of Time on Stream for Different Catalyst Systems

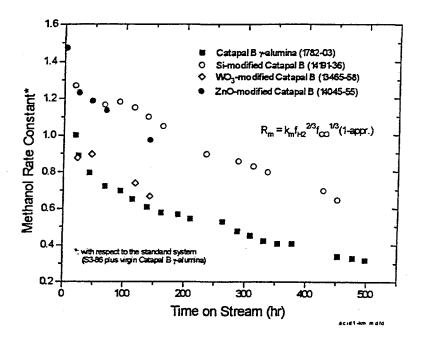
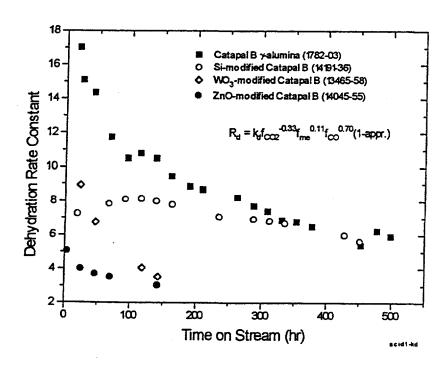


Figure 3.1.2 Dehydration Rate Constant as a Function of Time on Stream for Different Catalysts



The second observation is that deactivation of the methanol catalyst can occur during reduction, as suggested by the different initial activities of the same methanol catalysts in different catalyst systems. Rapid deactivation continues into the early period when the system is switched to syngas. The deactivation in this stage is referred to as initial deactivation, hereafter. In the standard catalyst system (S3-86 plus virgin Catapal B alumina), the methanol catalyst loses 20-30% of its activity in the initial stage.

Systems of other traditional solid acids. The deactivation patterns of the catalyst systems consisting of S3-86 and silica alumina or zeolites are shown in Figures 3.1.3 and 3.1.4, along with that of the standard catalyst system. Again, all the data were collected under the standard reaction conditions. The two observations mentioned above hold true for these four additional systems, that is, the rate of the long-term deactivation of the methanol catalyst is similar among different systems, and the initial deactivation of the methanol catalyst varies from one system to another.

Systems of inert materials. A number of metal oxides we have tested, including silica gel, titania, zirconia, and, zirconia-doped silica gel, exhibited nil or negligibly small dehydration activity. The most important observation from these runs, as shown in Figure 3.1.5, is that there is no significant initial deactivation of the methanol catalyst when the dehydration component is inert. The system containing the silica gel shows a long-term methanol catalyst deactivation similar to that of S3-86 in a LPMEOH run (no dehydration catalyst). The experiments using other systems are too short to establish a trend in the long-term deactivation. However, the system containing the ZrO₂-doped silica gel appears to undergo long-term deactivation of the methanol catalyst. This suggests that for some systems the long-term deactivation of the methanol catalyst in the dual system may even occur in the presence of an inert material.

Figure 3.1.3 "Normalized" Methanol Synthesis Rate Constant as a Function of Time On-Stream for Different Catalyst Systems

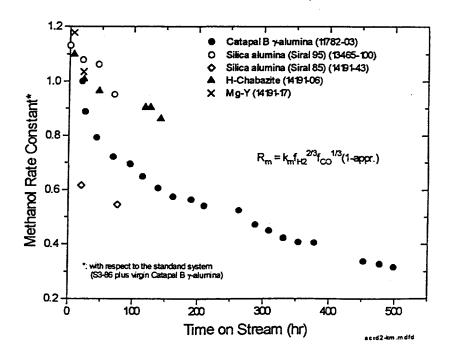


Figure 3.1.4 Dehydration Rate Constant as a Function of Time On-Stream for Different Catalysts

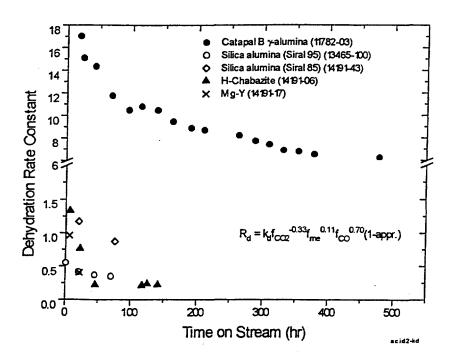
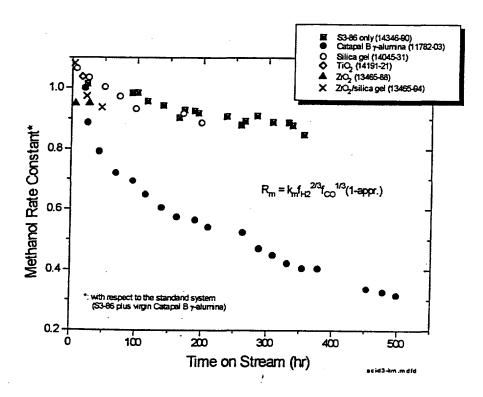


Figure 3.1.5 "Normalized" Methanol Synthesis Rate Constant as a Function of Time On-Stream for Different Catalyst Systems



b. Trends in Deactivation of the Methanol Catalyst

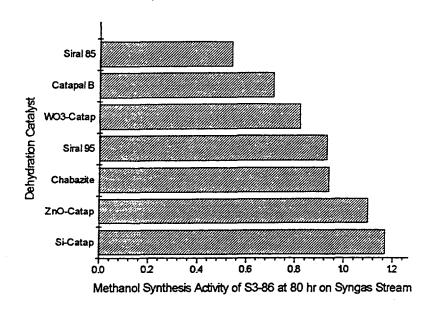
With the exception of the system containing silica gel, long-term deactivation of the methanol catalyst is observed in all dual systems, and does not depend on the activity of dehydration catalysts. The methanol catalyst deactivates at a rate of $\sim 0.082\%$ hr⁻¹, which is about a factor of 2 greater than that of the S3-86-only system (0.045% hr⁻¹).

It is not straightforward to correlate the initial deactivation of the methanol catalyst to the activity of dehydration catalysts, because the dehydration catalysts may also have deactivated during reduction. Thus, the dehydration activity shown by the first data point in Figures 3.1.2 and 3.1.4 may not be a fair indication of the initial or intrinsic activity of a dehydration catalyst. The best way to determine the activity of a dehydration catalyst is to conduct a measurement using only the dehydration catalyst with methanol as feed gas. Since few measurements have been made in this regard, we have to use literature and general principles to estimate the initial dehydration activity.

Figure 3.1.6 displays the *initial deactivation* of the methanol catalyst against different dehydration catalysts. Dehydration catalysts are arranged in increasing dehydration activity order with the least active catalyst at the bottom. The initial deactivation of the methanol catalyst is measured approximately by methanol synthesis activity at 80 hours on syngas stream. Although we do not have direct measurements of the initial or intrinsic activity of these dehydration catalysts as mentioned above, a correlation can be established based on the following discussion.

First, silylation (i.e., Si-modification) has been known in the literature as a means to passivate strong acid sites on metal oxides. Modification of Catapal B by ZnO was also aimed at passivating the catalyst through acid-base reaction between ZnO (a base) and acid sites on Catapal B. Therefore, these two catalysts should have less dehydration activity than virgin Catapal B g-alumina. Second, WO₃ has been reported in the literature to have greater or similar dehydration activity compared to g-alumina. Third, silica alumina is generally more acidic than g-alumina. Siral 85, a silica alumina containing 85 wt % of SiO₂, has shown higher isobutanol dehydration activity in this lab than a g-alumina comparable to Catapal B. While the dehydration activity of the Chabazite and Siral 95 samples remains to be determined, the available data indicate that the initial deactivation of the methanol catalyst is correlated to the activity of dehydration catalysts. The greater the dehydration activity is, the larger is the initial deactivation of the methanol catalyst.

Figure 3.1.6 Correlation Between the Initial Deactivation of the Methanol Catalyst and the Activity of Dehydration Catalysts



c. Patterns in the Deactivation of Dehydration Catalysts

There are several different patterns in the deactivation of different dehydration catalysts. The first pattern includes MgY, Chabazite, Siral 85, and WO₃-Catapal. This group of catalysts is characterized by rapid initial deactivation to a small residual activity. Significant deactivation occurs during reduction. For example, although Siral 85 has a known higher dehydration activity than g-alumina, its measured initial activity is much lower than that of Catapal B (1.2 vs. 17 mol/kg-hr, see Fig. 3.1.4). The Chabazite and MgY samples were expected to have activities comparable to g-alumina, but their measured initial activities were much lower than expected (Fig. 3.1.4). This rapid deactivation continued when the system was switched to syngas, and the dehydration activity dropped to a residual level within 100 hr on syngas stream.

Si-modified Catapal B g-alumina represents another extreme. No significant initial deactivation was observed. Instead, there was only a slow, long-term deactivation (Fig. 3.1.2).

Catapal B g-alumina undergoes a two-stage deactivation: an initial rapid deactivation (~ 40% loss in its activity) followed by a slower but continuous deactivation. ZnO-modified Catapal also falls into this pattern.

The rapid initial deactivation of dehydration catalysts may depend on the type as well as the strength of acid sites on the catalysts. MgY and Chabazite are typical Bronsted acids. Their fast deactivation may be indicative of the extra vulnerability of the Bronsted acid under LPDME conditions. Siral 85 and Catapal B should contain both Bronsted and Lewis acid sites according to conventional knowledge. Without solid evidence, it is assumed for the time being that the fast deactivation of Siral 85 and of Catapal B in the initial stage is due to the acid sites of great strength and/or Bronsted nature. This assumption is supported by the following observation.

The initial fast deactivation of Catapal B can be eliminated by modifying the surface with silica (Si-modified Catapal B), a process called silylation, and known for eliminating strong and Bronsted acid sites. It may not be just a coincidence that this passivated catalyst starts with an activity similar to that of the virgin Catapal B alumina after the first stage of deactivation.

The long-term deactivation of dehydration catalysts does not follow any clear pattern. The deactivation varies from one system to another. The higher dehydration activity (i.e., greater acidity) does not necessarily result in faster deactivation of the dehydration catalyst, or vice versa. This is clearly illustrated by the results from the Si- and WO₃-modified Catapal B samples shown in Figure 3.1.2.

d. Deactivation of the Methanol Catalyst vs. That of Dehydration Catalysts

In the standard catalyst system the initial deactivation of the methanol catalyst is accompanied by a corresponding deactivation of Catapal B alumina (Figs. 3.1.1 and 3.1.2). Relatively stable Si-Catapal B and ZnO-Catapal B correspond to smaller initial deactivation of the methanol catalyst. However, the almost complete deactivation of Chabazite and MgY samples is not accompanied by significant initial deactivation of the methanol catalyst (Figs. 3.1.3 and 3.1.4). And the initial loss in the methanol activity is totally outweighed by that of the dehydration activity in the case of Siral 85.

This lack of correlation between the deactivation of the methanol catalyst and the deactivation of dehydration catalysts can be further illustrated. Figures 3.1.7A and 3.1.7B plot the ratio of the methanol synthesis rate constant to the dehydration rate constant, both normalized by their initial values, for different catalyst systems. This serves as a measurement of the relative deactivation rates of two catalysts in a given catalyst system. It can be seen that the ratio is about 1 for the standard catalyst system, the systems containing silica alumina (Siral 85 and 95), and ZnO-modified Catapal B. In the case of Si-modified Catapal, the dehydration catalyst deactivates more slowly than the methanol catalyst. For the rest of the catalyst systems, the dehydration catalyst deactivates much faster than the methanol catalyst. This variation suggests that the deactivation of methanol and dehydration catalysts does not have to be a concerted process. Furthermore, different deactivation mechanisms may be operational for different catalyst systems.

Figure 3.1.7A The Deactivation Rate of the Methanol Catalyst Relative to that of Dehydration Catalysts in Different Catalyst Systems

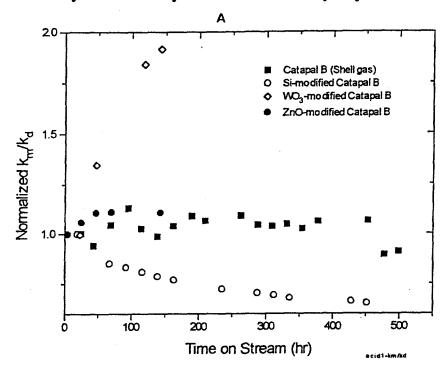
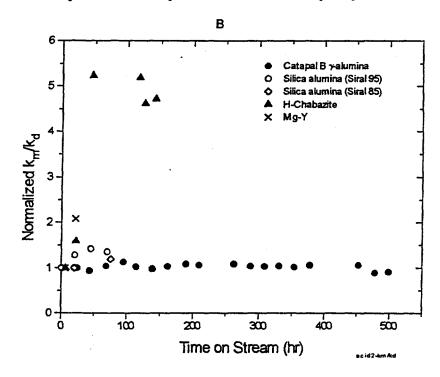


Figure 3.1.7B The Deactivation Rate of the Methanol Catalyst Relative to that of Dehydration Catalysts in Different Catalyst Systems



e. Mechanistic Considerations

The initial deactivation of the methanol catalyst is possibly driven by the acid-base interaction between the two catalysts, since it correlates with the dehydration activity (Fig. 3.1.6). Among the possible mechanisms are the inter-catalyst mass transfer and inter-catalyst solid state reaction. For instance, ion exchange could take place between Cu- and Zn-containing species from the methanol catalyst and the protons on the dehydration catalyst. Or the deactivation could be due to a reaction between ZnO (a base) in the methanol catalyst and the acid sites on a dehydration catalyst. The same acid-base interaction may also be responsible for the initial deactivation of dehydration catalysts.

Surprisingly, the initial deactivation of the methanol catalyst is not correlated to the initial deactivation of dehydration catalysts, considering that both may be due to the acid-base interaction. However, this lack of correlation can be understood if acid sites of different natures (Bronsted or Lewis) undergo deactivation through different routes. Furthermore, we still have not ruled out the possibility that coke formation deactivates, as a parallel route, dehydration catalysts. If it is, certainly it will complicate the pattern of the deactivation of dehydration catalysts.

The mechanism for the long-term deactivation of both methanol and dehydration catalysts is not clear. For one thing, it is not directly related to the dehydration activity. The long-term deactivation may still be due to inter-catalyst mass transfer or solid state reactions, but is not likely acid-base in nature. For example, the migration of Zn- and Cu-containing species from the methanol catalyst to dehydration catalysts can be *driven by the concentration gradient* of these species between the methanol catalyst and dehydration catalysts. Note that most of the metal oxides tested as dehydration catalysts are also good catalyst supports with dispersing capability for metal, metal oxides, and salts, and the dispersing capability is not necessarily related to the acidity of the materials.

3.1.2 What We Learned from the Experiments using Robinson-Mahoney Basket Internals

3.1.2.1 A Repeated Run Using Robinson-Mahoney Basket Internals

A second LPDME run using Robinson-Mahoney (R-M) basket internals was conducted this quarter. The goal of this experiment was twofold: first, to confirm the results from the first experiment using the basket internals because of their important implication for future work, and second, to determine when the deactivation of the dehydration catalyst occurs under this setup.

The current run (14045-69) was carried out at the same conditions as the first one (Shell gas, 750 psig, 250°C). Two different space velocities and stirrer speeds were used in the previous R-M run: 6,000 GHSV and 1600 rpm vs. 1500 GHSV and 2000 rpm. The second set of conditions was used in the current run, which was also shorter, 121 hr compared to 500 hr for the earlier run. As for the first experiment, due to the mass transfer limitations under the R-M setup, the activity of the catalysts could only be checked in a subsequent run using the normal LPDME setup and the powdered catalyst mixture made from the spent pellets. The previous experiment showed that the powders from the R-M experiment were reduced (i.e., hydrogen uptake of the

powder was minimal). Therefore, the system was brought to the reaction conditions without reduction in the run of activity measurement.

Table 3.1.1 lists the activity results of the catalysts from both R-M runs, along with those from a standard LPDME life run (powder mixture, 11782-3). There are some differences between the two R-M runs; mainly, the second run exhibits higher methanol activity and lower dehydration activity. This difference is likely due to the experimental variability. As mentioned previously, the spent pellets were ground into powders, then reloaded into the autoclave to check activity. In both runs, only a portion (1/4 in the first and 1/2 in the second run) of the catalyst mixture was ground, saving the other portion for analytical purposes. Since the pellet mixture might not have been perfectly mixed, the ratio of the two catalysts in the ground samples may have been different from the nominal values, resulting in the uncertainty in data analysis.

Given the experimental variability, the same conclusion can be drawn from the two R-M runs. Judging by the rate constant, the spent methanol catalysts from the R-M runs have almost the same activity as the fresh methanol catalyst in the standard run, and much higher activity than the methanol catalyst at a similar time on stream in the standard run. Therefore, both runs indicate that the methanol catalyst is stable under the R-M setup. The dehydration catalyst deactivated in the R-M runs by 37-59%. Since the longer time on stream in the initial experiment did not result in greater deactivation in the dehydration activity, it can be concluded that the deactivation of the dehydration catalyst occurred only in the earlier stage of the run (< 127 hr). That is, there is no long-term deactivation of the dehydration catalyst under the R-M setup.

Table 3.1.1 Activity of the Catalysts Used in the LPDME Runs Using Robinson-Mahoney
Basket Internals

Reaction conditions: 250°C, 750 psig, Shell gas.

Run	Catalyst	Time On-Stream	MEOH Equiv. Prod.	Concen		Rate Constant		
·	S3-86:A1203	(hr)	(mol/kg-hr)	MEOH	DME	kma	kdb	
1st R-M (14045-52)	82.2:17.8	508	28.1	1.59	6.13	2.7	10.7	
2nd R-M (14045-75)	80:20	127	27.1	2.74	4.87	3.1	7.0	
Standard (11782-3)	81.3:18.7	20	30.7	1.01	6.95	3.0	17.0	
		115	23.6	0.83	4.89	19	10.8	
		499	30.37	0.49	2.67	1.0	5.9	

- 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: ethanol 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.

3.1.2.2 The Role of Intimate Contact Between the Two Catalysts in Catalyst Deactivation There are two important observations from the experiments using Robinson-Mahoney basket internals and pelletized catalysts. First, this system is free of methanol catalyst deactivation,

indicating that the presence of an active dehydration catalyst in the system does not necessarily cause the deactivation of the methanol catalyst. This is true for even the potential initial deactivation stage, a stage associated with dehydration activity. It also indicates that the slurry fluid does not participate in deactivation of the methanol catalyst.

Second, we did see initial deactivation of the dehydration catalyst under this setup. This deactivation may be due to either inter-catalyst mass transfer or coking. The analysis of the spent samples from this experiment will enable us to distinguish between these two mechanisms. If inter-catalyst mass transfer is the cause, the slurry fluid must have served as the mass transfer medium. And apparently the methanol catalyst has some "free" Zn- and/or Cu-containing species to spare before its activity starts to suffer. In summary, among four modes of catalyst deactivation under the standard LPDME conditions, three of them do not occur under the Robinson-Mahoney setup.

Why is the catalyst deactivation pattern so different between a normal slurry phase operation and the run using Robinson-Mahoney basket internals and pelletized catalysts? If one assumes that the inter-catalyst mass transfer or reaction causes the deactivation of both catalysts, this process requires a **driving force** and the **intimate contact** between two catalysts. The driving force, as discussed above, could be acid-base interaction between the methanol catalyst and dehydration catalysts (the initial deactivation of both catalysts), or simply the concentration gradient between the two catalysts and the dispersion capability of dehydration catalysts (the long-term deactivation of both catalysts). The driving force alone is not sufficient; intimate contact between the two catalysts is necessary to provide the time and area for the mass transfer or/and reaction to take place.

One can envision that the solid state reaction between the two catalysts can only occur when they touch each other and remain that way for a long enough time. Under the slurry phase operation conditions, this intimate contact can be provided by: 1) collision between the catalyst particles, 2) the attachment of small particles to the large ones, either on the outside surface or inside the pores, and 3) the agglomeration of small particles. Collision and attrition continuously generate particles of smaller and smaller size, resulting in large and fresh (therefore active) contact area.

3.1.3 Efforts in Developing Stable LPDME Catalysts

According to the above analysis, the stability of LPDME catalyst systems should be improved by eliminating or reducing either the driving force for the inter-catalyst mass transfer/reaction or the intimate physical contact between the two catalysts. In principle, these can be accomplished by using:

- Chemically modified alumina or methanol catalysts to reduce the driving force of the interaction between methanol and dehydration catalysts, such as removal of strong acid sites.
- Physically modified alumina or slurry system to eliminate the contact between the two catalysts.

Alternative methanol catalysts such as one-component catalyst systems.

The following are the efforts we have made in these directions this past quarter.

3.1.3.1 Improvement in the Long-Term Stability of the Methanol Catalyst using K-Doped Alumina

A potassium-doped Catapal B g-alumina sample was prepared by impregnating Catapal B g-alumina with a KOH solution. This catalyst was used in a LPDME run along with S3-86 methanol catalyst (19045-85). As shown in Figure 3.1.8, the stability of this catalyst system is better than the standard system (S3-86 plus virgin Catapal B g-alumina). However, its productivity is lower due to the low dehydration activity of the K-doped alumina (Figure 3.1.9). This low activity apparently is due to the high K loading.

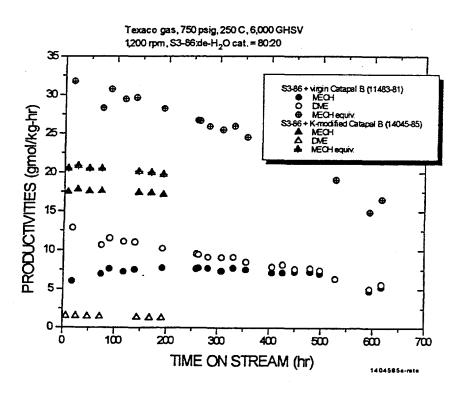
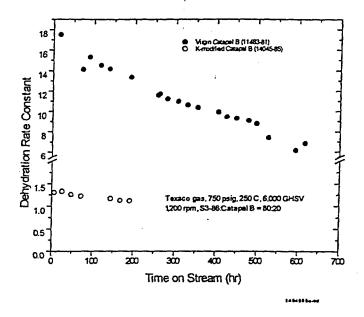


Figure 3.1.8 Productivity as a Function of Time

Figure 3.1.9 Dehydration Rate Constants of Different Catalyst Systems as a Function of Time



An important observation from this experiment is the improved stability of the methanol catalyst. As shown in Figure 3.1.10, the methanol catalyst has a higher initial activity (i.e., smaller initial deactivation) and slower long-term deactivation, compared to the standard system. Smaller initial deactivation of the methanol catalyst was observed previously whenever a dehydration catalyst without strong acidic sites was used. However, this is the first time that improvement in the long-term stability of the methanol catalyst has been observed. This improvement cannot be simply attributed to the low dehydration activity of the K-doped alumina. For example, H-Chabazite, Mg-Y, and two silica alumina catalysts (Siral 85 and 95) have a similar or lower dehydration activity than the K-doped alumina upon initial deactivation. However, little improvement in the long-term stability of the methanol catalyst was observed in these systems (Figs. 3.1.3 and 3.1.4).

We have tried to increase the productivity of the current catalyst system by adding more K-doped alumina into the system (from a ratio of 20:80 to 43:57). The productivity is still too low to be attractive. In the coming quarter K-doped alumina samples with lower loading, therefore, higher dehydration activity, will be tested to see if a similar stability can still be obtained.

Another observation from this experiment is that the production of high alcohols, e.g., isobutanol, from this catalyst system is not any higher than a typical LPMEOH run. This suggests that there is little migration of K from the K-doped alumina to the methanol catalyst.

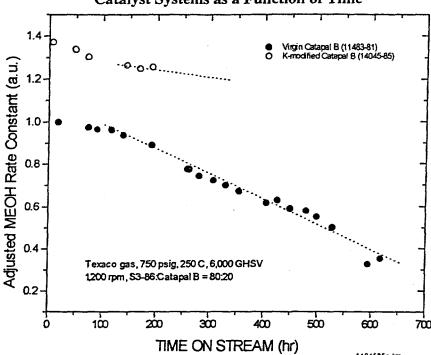


Figure 3.1.10 Methanol Rate Constants of Different Catalyst Systems as a Function of Time

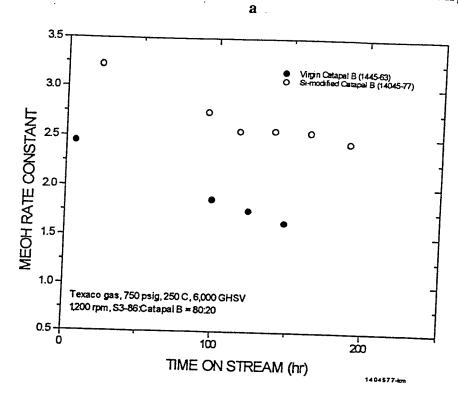
3.1.3.2 Efforts in Passivating the Exterior of Catapal B g-Alumina Particles

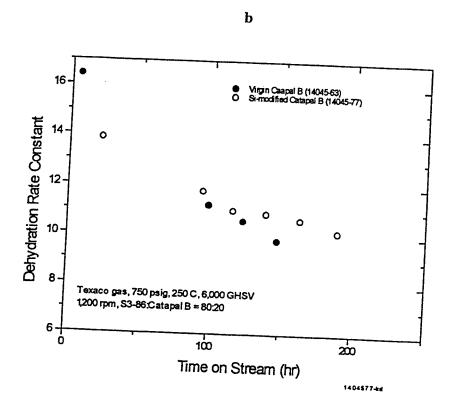
A Si-modified Catapal B g-alumina (14191-104) was prepared using a polymeric siloxane precursor. Because of their large size, the siloxane molecules will most likely attach to the outer surface of the alumina. Therefore, when the siloxane is converted into silica upon calcination, only the outer surface of the alumina will be modified or passivated. (This is a technique mentioned in the literature to passivate the strong acid sites on the exterior of zeolites.) Only the outer surface of the alumina is concerned here, because that is where the deactivation of the methanol and dehydration catalysts probably occurs.

Figures 3.1.11a and b display the results of a LPDME run using sample 14045-77, along with samples of a virgin Catapal B g-alumina (14045-63). It can be seen that silica modification of the outer alumina surface results in smaller initial deactivation of the methanol catalyst, as indicated by the greater rate constant for the modified sample compared to that of the unmodified sample (Fig. 3.1.11a). This agrees with our theory that the initial deactivation of the methanol catalyst is due to the strong acid sites on the dehydration catalyst. Figure 3.1.11a shows that the long-term deactivation of the methanol catalyst is also improved somewhat.

Figure 3.1.11b shows that the initial dehydration rate constant of the Si-modified sample is smaller than that of the virgin Catapal B g-alumina, apparently due to reduction in the number of acid sites by the passivation. The modified alumina, however, deactivates more slowly than the virgin one, and becomes more active after 100 hr on stream. In brief, the modification results in significant improvement in the stability of the catalyst system, but not enough as far as the long-term stability of the system is concerned.

Figure 3.1.11 Stability of LPDME Catalyst Systems: Si-Modified vs. Virgin Catapal B



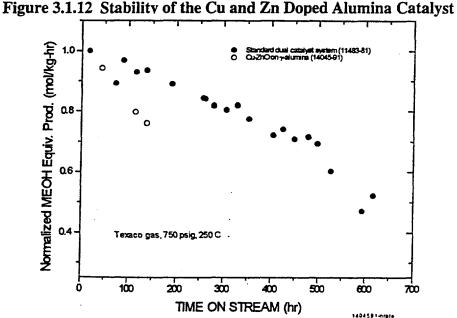


A second sample was prepared in a similar manner, but with much higher Si loading (35 wt % SiO₂). High loading was used so that alumina particles would not be only passivated on the outside surface, but hopefully encapsulated or embedded inside SiO₂. This in turn might reduce the contact between the methanol catalyst and alumina, therefore, improving the stability of the catalyst system. The sample was tested along with S3-86 methanol catalyst at the standard conditions using Texaco gas (14656-17). The dehydration activity of the sample was fairly low, about 19% of the activity of the virgin Catapal B g-alumina. No improvement in the long-term stability of the methanol catalyst was observed from this catalyst system. Note that the technique used in the preparation of this sample is best suited for silylation, but not for encapsulation and embedment.

3.1.3.3 A Single Particle DME Catalyst

A single particle DME catalyst was prepared by impregnating Catapal B g-alumina with zinc and copper (14656-9). The preparation was based on a Shell patent (US 4,375,424, 1983) for a syngas-to-DME catalyst. The catalyst is claimed to perform both methanol synthesis and dehydration functions, and is therefore referred to as single particle catalyst to distinguish it from the dual catalyst mixture in our standard LPDME system.

The standard liquid phase reduction procedure was used to activate this catalyst using 2% H₂ in N₂. The activity of this catalyst was checked using Texaco syngas and standard reaction conditions (250°C, 750 psig, and 6,000 GHSV, run 14045-91). The methanol productivity of this catalyst was found to be an order of magnitude lower than that of our standard dual catalyst system (S3-86 plus Catapal B g-alumina). It is not straightforward to compare this catalyst with the ones reported in Shell's patent, because their test reaction was run at 280°C and 1700 psig. However, catalyst stability is that which is of greatest concern. Figure 3.1.12 depicts the normalized methanol equivalent productivity of this catalyst in comparison with that of our standard dual catalyst system. It can be seen that the rate of deactivation of this one component catalyst is greater than that of our standard dual catalyst system. In the coming months, other one-component catalysts will be examined.



APPENDIX F TASK 1.5.4 - 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.

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 LPMEOHTM 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 G TASK 1.5.4 - GROUND BREAKING CEREMONY

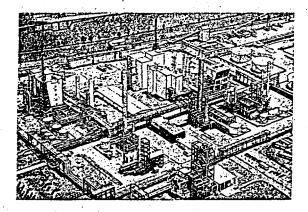
The U.S. Department of Energy, through its Clean Coal Technology Program, is supporting the joint venture between Air Products and Eastman to demonstrate Air Products' novel liquid phase methanol technology. This first-of-a-kind demonstration project will be constructed at Eastman's integrated coal gasification facility in Kingsport. The project, which will produce 260 tons per day of methanol, will be a preview of future commercial facilities in which coproduct methanol is made from coal-derived synthesis gas as an adjunct to integrated gasification combined cycle (IGCC) power plants.



EASTMAN

PRODUCTS 1

ou are cordially invited to attend
the Liquid Phase Methanol Demonstration Project
groundbreaking ceremony
on Monday, October 16, 1995
at Eastman Chemical Company
in Kingsport, Tennessee.
The ceremony will begin at 11:30 a.m.
in the Research Laboratory Building
and will conclude at 2:30 p.m.
at the construction site.



Liquid Phase Methanol

Demonstration Project

Groundbreaking Ceremony

October 16, 1995



11:30 a.m.-12:30 p.m.

Lunch in the Eastman Research Building

4th floor cafeteria

12:40-1:10 p.m.

Remarks from Dignitaries

Mr. Earnest Deavenport

Chairman and CEO, Eastman Chemical Company

Mr. H. A. Wagner

Chairman, President, and CEO, Air Products and Chemicals, Inc.

Mr. Alan Edwards

Principal Deputy Assistant Secretary, Fossil Energy,

U.S. Department of Energy

The Honorable James Quillen

U.S. Representative

1:20 p.m.

Depart for the Construction Site

1:35 p.m.

Groundbreaking Ceremony

Mr. Harry Holliman

President, Tennessee Eastman Division

Mr. Thomas Torkos

Deputy Director, Pittsburgh Energy Technology Center

1:45 p.m.

Groundbreaking Photographs

2:15 p.m.

Return to Research Laboratory Building

2:30 p.m.

Conclusion of Ceremony





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

LIQUID PHASE METHANOL DEMONSTRATION MILESTONE SCHEDULE STATUS REPORT DE-FC22-92PC90543

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Task Name		PHASE 1: DESIGN	PROJECT DEFINITION(TASK1)	CONTINUATION APPLICATION(B.P.#2)	PERMITTING(TASK 2)	NEPA FONSI APPROVAL	DESIGN ENGINEERING(TASK 3)	VENDOR ENGINEERING	OFF-SITE TESTING(TASK 4)	UPDATED FUEL TEST PLAN APPROVAL	DECISION TO CONTINUE DIME TESTING	PLANNING, ADMIN & DME DVT(TASK 5)	PHASE 2: CONSTRUCTION	PROCUREMENT(TASK1)	CONSTRUCTION(TASK 2)	TRAINING & COMMISSIONING(TASK 3)	OFF-SITE TESTING(TASK 4)	PLANNING & ADMINISTRATION(TASK 5)	CONTINUATION APPLICATION(B.P.#3)	PHASE 3: OPERATION	START-UP(TASK 1)	METHANOL OPERATION(TASK 2.1)	DISMANTLE PLANT(TASK 2.3)	ON-SITE PRODUCT USE DEMO(TASK 3)	OFF-SITE PRODUCT USE DEMO(TASK 4)	DATA ANALYSIS/REPORTS(TASK 5)	PLANNING & ADMINISTRATIVE(TASK 6)	PROVISIONAL DME IMPLEMENTATION	DME DVT(PDU TESTS)(TASK 3.6)	DECISION TO IMPLEMENT	DESIGN, MODIFY & OPERATE(TASK 3.2.2)		

Printed: Sep/29/95 Page 1

Summary

Milestone Δ

COST MANAGEMENT REPORT U.S. DEPARTMENT OF ENERGY

DOE F 1332.9

FORM APPROVED Page 1 of 1

249 (249) Variance 50/16/01 OMB 1910-1400 2340 3,435 3,792 1,926 1,593 10,953 11,500 897 8 147,287 \$ 213,700 1,230 8,206 1,663 681 288 324 Contract 16,851 DATE Total Value 3. IDENTIFICATION NUMBER 17. SIGNATURE OF PARTICIPANT'S AUTHORIZED FINANCIAL 7. COMPLETION DATE 3,435 10,953 2,340 3,792 1,926 1,593 213,700 1,663 11,500 33 147,536 425 DE-FC22-92PC90543 16,602 1,230 88 8,206 324 897 83 Total December 31, 2001 6. START DATE January 1, 1990 JULDAUM TO DATE 1,480 1,096 quent FY's 0 76,701 474 1,161 81,341 \$ d. Subse-€ 37,401 1,896 380 252 ş 40,438 FY 1998 S. J. Kasinecz ପ ESTIMATED ACCRUED COSTS 3,435 33,434 40,328 103 \$ 432 8 1,422 385 245 351 September 01, 1995 through September 30, 1995 FY 1997 2 11,060 0 0 25,891 9,182 88 2,869 ŝ 919 ŝ 324 \$ FY 1996 ε of Fiscal 0 2. REPORTING PERIOD b. Balance 0 0 0 0 0 0 0 0 S. COST PLAN DATE DATE Year September 26, 1994 0 quent Report-Ing Period 16. SIGNATURE OF PARTICIPANT'S PROJECT MANAGER a. Subse-1,230 2,070 16,851 6,478 1,032 6,159 88 324 324 34,756 MANY Cumulative to Date d. Plan 16,602 5,234 1,366 1,021 214 1,197 ક 25,702 c. Actual ACCRUED COSTS 2,422 0 482 1 1,407 <u>ફ</u> 0 ¥ 0 o 0 0 Reporting Period b. Plan AND DATE 8 837 741 አ 2 a. Actual Planning, Admin, & DME Verif. Testing Proj. Mount - Train + Commus. Air Products Liquid Phase Conversion Co., L.P. 8. ELEMENT 9. REPORTING ELEMENT 2. PARTICIPANT NAME AND ADDRESS Off-Site Test . Proc. & Constr. Liquid Phase Methanol Demonstration DME Design, Mod., Oper. LPMEOH Dismantlement Data Analysis & Reports Methanol Operation Planning & Admin. Thousands Planning & Admin 15. DOLLARS EXPRESSED IN: Project Definition Off-site Testing On-Site Demo. Off-Site Demo. Allentown, PA 18195-4911 Prior to Mod 2 7201 Hamilton Boulevard Design Engr. Procurement Construction Permitting Operations Startup 1.3.2.1 1,3,2,2 1,3.2.3 14. TOTAL 1.3.3 13.4 1.1.5 121 1.2.3 1.2.4 13.1 13.2 1.3.5 1.3.6 1.1.2 1.1.3 1.1.4 1.2.5 1. TITLE 1.1.1 1.2.2 (11-84)

APPENDIX I TASK 2.1 - SPECIFICATION STATUS - EQUIPMENT

Vendor Prints	Rec'd 1/3/95 Rec'd 4/18/95 Rec'd 4/04/95 Rec'd 4/04/95 Rec'd 4/18/95 Rec'd 4/18/95	Rec'd J/25/95 Rec'd 4/18/95 Rec'd 4/26/95 Rec'd 4/26/95 Rec'd 1/25/95 Rec'd 1/25/95 Rec'd 1/18/95	Rec'd 6/6/95 Rec'd 4/26/95 Rec'd 6/17/95 Rec'd 6/17/95 Rec'd 8/31/95	Rec'd 7/13/95 Rec'd 8/23/95 Rec'd 8/23/95 Rec'd 9/95 Rec'd 7/13/95 Rec'd 7/13/95	Rec'd 10/14/94 Rec'd 5/10/95 Rec'd 1/27/95 Rec'd 4/20/95 Rec'd 5/19/95 Rec'd 5/19/95	Rec'd 6/5/95 Rec'd 4/20/95 Rec'd 5/11/95 Rec'd 5/11/95 Rec'd 5/11/95
Order Placement	11/8/94 3/16/95 3/15/95 3/15/95 3/21/95 6/5/95	1/10/95 3/14/95 3/14/95 5/15/95 1/2/20/94 1/10/95	5/15/95 12/16/94 3/14/95 3/29/95 6/19/95 8/16/95	6/5/95 8/1/95 8/1/95 8/11/95 6/5/95	8/12/94 3/28/95 12/2/94 3/24/95 3/28/95 3/28/95 3/28/95	3/28/95 3/24/95 3/28/95 3/28/95 3/28/95 3/28/95
Bids Received	10/3/94 12/7/94 2/10/95 2/10/95 12/16/94 5/17/95	107/94 2/17/95 2/17/95 2/17/95 11/1/94 107/94 2/17/95	4/20/95 11/1/94 2/17/95 1/9/95 5/3/95	47/95 4/12/95 4/12/95 7/11/95 47/95	6/12/94 12/20/94 10/24/94 1/9/95 12/20/94 12/20/94	12/20/94 1/9/95 12/28/94 12/28/94 12/28/94
Bids Due	9/29/94 12/6/94 2/2/95 2/2/95 12/28/94 5/17/95	10/14/94 27/95 27/95 4/18/95 11/1/94 10/14/94 27/95	4/18/95 11/1/94 2/7/95 1/12/95 4/20/95	4/3/95 4/10/95 4/10/95 7/10/95 4/3/95	6/12/94 12/21/94 10/14/94 1/5/95 12/21/94 12/21/94 1/9/95	12/21/94 1/5/95 1/9/95 1/9/95 1/12/95
Released to Purchasing	8/29/94 11/8/94 1/5/95 1/5/95 12/2/94 4/25/95	977/94 1/10/95 1/10/95 3/28/95 10/4/94 977/94	3/28/95 10/4/94 1/10/95 12/5/94 3/28/95 6/14/95	3/9/95 3/13/95 3/13/95 6/14/95 3/9/95 3/9/95	5/17/94 11/8/94 9/19/94 12/1/94 11/8/94 11/8/94	11/8/94 12/1/94 11/8/94 11/8/94 12/5/94
Vendor List*	***************************************	***************************************	***************************************	***************************************	***************************************	***************************************
Eastman Comments	8/18/94 10/19/94 12/15/94 12/15/94 11/30/94 4/5/95 9/21/94	10/11/94 12/15/94 12/15/94 3/27/95 9/21/94 10/11/94 12/15/94	3/27/95 9/21/94 12/15/94 11/23/94 3/27/96 6/26/95	3/3/95 3/8/95 3/8/95 6/26/95 3/3/95 3/3/95	5/17/94 10/11/94 9/7/94 12/6/94 11/1/94 10/19/94	10/19/94 12/6/94 11/1/94 11/1/94 11/1/94
Equipment Spec Released(Rev)	11/02/94 (1) 11/07/94 (1) 01/09/95 (2) 1/09/95 (2) 11/30/94 (1) 3/23/95 (0) 11/11/94 (2)	11/11/94 (2) 01/09/95 (1) 01/09/95 (1) 3/27/95 (1) 11/11/94 (2) 11/11/94 (2) 01/09/95 (1)	3/27/95 (1) 10/03/94 (1) 01/09/95 (1) 12/02/94 (1) 3/27/95 (1) 6/13/95 (0)	3/08/95 (2) 3/09/95 (1) 3/09/95 (1) 6/13/95 (0) 3/08/95 (2) 3/08/95 (2)	08/29/94 (1) 4/26/95 (4) 9/16/94 (1) 4/10/95 (2) 11/7/94 (2) 11/7/94 (2)	11/07/94 (1) 11/30/94 (1) 11/7/94 (2) 11/7/94 (2) 11/7/94 (2)
Equipment Specifier	Koeller Koeller Koeller Koeller Koeller	Koeller Koeller Koeller Koeller Koeller Koeller	Koeller Koeller Koeller Koeller Koeller	Koeller Koeller Koeller Skuchas Koeller	Fleischer Koeller Koeller Koeller Koeller	Koeller Koeller Koeller Koeller Koeller
Process Spec Released(Rev)	6/10/94 (0) 10/19/94 (1) 11/18/94 (1) 11/18/94 (2) 11/21/94 (2) 03/22/95 (0) 11/11/94 (2)	. 11/11/94 (2) 12/30/94 (2) 11/18/94 (0) 03/09/95 (0) 11/11/94 (2) 12/30/94 (2)	03/09/95 (0) 8/23/94 (0) 3/07/95 (2) 9/29/94 (0) 03/10/95 (0)	3/07/95 (2) 3/07/95 (1) 3/07/95 (1) 4/21/95 (1) 3/30/95 (2) 2/03/95 (1)	4/22/94 (1) 03/31/95 (5) 8/29/94 (1) 10/14/94 (0) 10/21/94 (1) 10/10/94 (2) 10/17/94 (3)	10/10/94 (1) 03/31/95 (2) 10/18/94 (2) 10/09/94 (1) 10/14/94 (0) 9/29/94 (0)
Process Engineer	Schaub Schaub Schaub Schaub Schaub Mazdai	Stein Stein Stein Stein Stein Stein	Stein Bhatt Mazdai Bhatt Chen N/A	Mazdai Stein Stein Stein Frenduto/Stein Mazdai	Schaub Chen Schaub Chen Chen Chen Stein	Stein Chen Stein Stein Mazdai Bhatt
Description	LPMEOH Reactor Steam Drum HP Methanol Separator Secondary Oil Knockout Vessel Reactor Cyclone Syngas Compressor Knockout Separator Methanol Stabilizer Column	Methanol Stabilizer Column Trays Methanol Stabilizer Redux Drum Methanol Stabilizer Feed Vessel Methanol Stabilizer Condensate Pot Methanol Rectifier Column Methanol Rectifier Column Methanol Rectifier Reflux Drum	Methanol Rectifier Condensate Pot Catalyst Reduction Vessel Reduction Condensate Accumulator Utility Oil Surge Tank (V-01 Skid) Guard Bed Oil-Water Separator/Coalescer Vent Scrubber	Safety Relief Knockout Drum Slurry Tank Methanol Lot Tank Methanol Lot Tank Methanol Drain Tank Fresh Oil Storage Tank Caustic Mix Tank	Compressor L.O. Sump (K-01 Skid) Syngas Compressor Cooler Syngas FeedProduct Economizer Methanol Product Air-Cooled Condenser Methanol Product C.W. Condenser Methanol Stabilizer Reboiler Methanol Stabilizer Condenser	Methanol Rectifier Reboiler Methanol Rectifier Air Cooler Methanol Rectifier C.W. Condenser Crude Methanol Cooler Reduction Vessel Overhead Condenser Utility Oil Henters (V-01 Skid)
Tag.#	C C 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0	C-10T C-11 C-12 C-13 C-20 C-20T C-21	C-23 C-30 C-31 C-32 C-40 C-120	D-01 D-02 D-20 D-25 D-30	D-70 E-01 E-03 E-04 E-10	E-20 E-21 E-22 E-23 E-31

\$6/61/6

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

Vendor Prints Due/Received	Rec'd 6/27/95 Rec'd 8/16/95 Rec'd 10/14/94 Rec'd 10/14/95 Rec'd 5/26/95	Rec'd 4/25/95 Rec'd 4/19/95	Rec'd 7/12/95 Rec'd 6/30/95 Rec'd 4/19/95 Rec'd 7/14/95 Rec'd 10/14/94 Rec'd 10/14/94		Due 9/8/95 Due 9/8/95 Rec'd 6/21/95 Rec'd 8/19/95 Rec'd 4/28/95	Rec'd 8/19/95 Rec'd 4/25/95 Rec'd 8/19/95 Rec'd 10/14/94 Rec'd 10/14/94
Order Placement	3/29/95 8/12/94 8/12/94 8/12/94 3/22/95 3/29/95	3/24/95 3/24/95 3/27/95 3/27/95 3/27/95 3/27/95 7/6/95	6/20/95 3/22/95 3/22/95 3/27/95 6/16/95 8/12/94 8/12/94	8/12/94 8/12/94 6/13/95 Boaluation Required Evaluation Required Evaluation Required	8/25/95 8/25/95 3/29/95 7/19/95 3/20/95	7/19/95 3/20/95 7/19/95 3/20/95 8/12/94 8/12/94
Bids Received	1/9/95 5/18/95 6/12/94 6/12/94 12/22/94 12/22/94	12/22/94 11/9/95 11/9/95 11/9/95 11/9/95 17/9/95 5/25/95	12/22/94 12/6/94 1/9/95 12/22/94 6/12/94 6/12/94		6/8/95 6/8/95 N/A 1/9/95 5/18/95 1/17/95	5/18/95 12/14/94 5/18/95 1/17/95 6/12/94 4/26/95
Bids Due	1/12/95 5/12/95 6/12/94 6/12/94 11/28/94 12/30/94	12/30/94 1/6/95 1/6/95 1/6/95 1/6/95 6/1/95	12/30/94 11/28/94 1/6/95 12/30/94 6/12/94 6/12/94	6/12/94 6/12/95 8/18/95 9/11/95 5/24/95	5/24/95 5/24/95 N/A 1/12/95 5/15/95 1/9/95	9/19/95 5/15/95 12/9/94 5/15/95 1/9/95 6/12/94
Released to Purchasing	12/5/94 4/27/95 5/17/94 5/17/94 11/9/94 12/8/94 11/9/94	12/8/94 12/19/94 12/19/94 12/19/94 12/19/94 5/17/95	6/12/95 12/8/94 11/9/94 12/19/94 12/8/94 5/17/94	5/17/94 5/4/95 8/7/95 8/28/95 5/9/95	5/9/95 5/9/95 8/10/95 12/5/94 4/27/95 12/7/94	9/1/95 4/27/95 11/1/194 4/27/95 12/8/94 5/17/94 8/31/95
Vendor List*	******	********	*******	Sole X	** ***	*******
Eastman Comments	11/23/94 4/10/95 5/17/94 5/17/94 11/30/94 11/30/94	12/21/94 11/30/94 11/30/94 11/30/94 11/30/94 12/12/94 5/4/95	5/26/95 12/15/94 11/30/94 1/10/95 12/15/94 5/17/94	5/17/94 7/10/95 9/11/95 9/11/95 5/11/95	5/11/95 5/11/95 11/23/94 4/10/95 12/15/94	4/10/95 10/19/94 4/10/95 12/15/94 5/17/94 5/17/94 8/28/95
Equipment Spec Released(Rev)	12/02/94 (1) 8/18/95 (4) 08/29/94 (1) 08/29/94 (1) 03/15/95 (2) 03/15/95 (2)	12/8/94 (2) 5/16/95 (5) 5/16/95 (6) 03/01/95 (4) 03/01/95 (5) 03/01/95 (4) 5/16/95 (0)	5/26/95 (0) 03/15/95 (1) 2/23/95 (2) 03/15/95 (2) 2/23/95 (1) 08/29/94 (1)	08/29/94 (1) 4/26/95 (memo) 8/4/95 (1) 8/22/95 (0) 8/22/95 (0) 4/17/95 (0)	4/17/95 (0) 4/17/95 (0) 8/10/95 (memo) 7/26/95 (2) 4/26/95 (2) 2/23/95 (1)	8/31/95 (0) 4/26/95 (2) 2/23/95 (2) 4/26/95 (2) 2/23/95 (1) 08/29/94 (1) 8/7/95 (P)
Equipment Specifier	Koeller Browning Fleischer Fleischer Fleischer Fleischer	Fleischer Fleischer Fleischer Fleischer Fleischer	Reed Fleischer Fleischer Fleischer Fleischer Fleischer	Fleischer Gressman Gressman Gressman Browning	Browning Browning Leenhouts N/A Browning Browning	Skuchas Browning Fleischer Browning Fleischer Fleischer Stoltz/Schnyder Frenduto
Process Spec Released(Rev)	9/29/94 (0) 7/25/95 (2) 4/22/94 (1) 4/22/94 (1) 10/20/94 (0) 03/14/95 (2) 03/14/95 (1)	11/10/94 (1) 5/9/95 (2) 5/9/95 (2) 12/13/94 (1) 12/15/94 (2) 02/28/95 (2) 04/10/95 (0)	03/14/95 (2) 9/29/94 (0) 03/14/95 (1) 11/29/94 (0) 4/22/94 (1)	4/22/94 (1)	3/17/95 (0) 3/17/95 (0) N/A N/A 9/29/94 (0) 03/22/95 (0) 11/29/94 (0)	N/A N/A 10/24/94 (1) data sheet issued 11/29/94 (0) 4/22/94 (1) N/A N/A
Process Engineer	Bhatt Chen Schaub Schaub Schaub Mazdai Mazdai	Schaub Stein Stein Stein Stein Stein	N/A Mazdai Bhatt Mazdai Stein Schaub	Schaub N/A N/A N/A N/A	N/A N/A Bhatt Mazdai Mazdai	N/A N/A Bhatt N/A Stein Schaub N/A N/A
Description	Utility Oil Cooler (V-01 Skid) GuardBed Electric Heater Compressor L.O. Cooler (K-01 Skid) Compressor L.O. Heater (K-01 Skid) Condensed Oil Circulation Pumps Slurry Return Pump Oil Makeup Pumps	BFW Pumps Methanol Stabilizer Underflow Pumps Methanol Stabilizer Reflux Pumps Methanol Rectifier Underflow Pumps Methanol Rectifier Reflux Pumps Methanol Transfer Pumps Methanol Drain Tank Lift Pump	Distillation Area Sump Pump Slurry Transfer Pump Utility Oil Circulating Pump Oil Feed Pump Caustic Metering Pumps Comp Main L.O. Pump (K-01 Skid)	Syngas Compressor Catalyst Building Fan Lube Oil Demister/Blower Motor Control Center Oil Heater SCR Regeneration Heater SCR BFW Blowdown Sample Cooler	Stabilizer Column Understow Sample Cooler Rectifier Column Understow Sample Cooler Fresh Catalyst Loading Chute Fresh Catalyst Drum Dumper Utility Oil Skid Fresh Feed Syngas Filters Slurry Tank Agitator	Instrument Air Dryer Methanol Product Filter Catalyst Reduction Agitator Seal Oil Filters Caustic Trank Agitator Compressor L.O. Filters (K-01 Skid) Compressor Seal Gas Filters (K-01 Skid) Density Sensor Hoist Trailer Loading Platform
Tag#	E-33 E-40 E-70 E-71 G-01 A/B G-03 A/B	G-04 A/B G-10 A/B G-11 A/B G-20 A/B G-21 A/B G-25 A/B	G-26 G-30 G-32 G-34 G-60A/B G-70	K-01 K-02 K-70 F-01 P-32 P-40 SP-001	SP-002 SP-003 T-30 T-31 V-01 Y-01A/B	Y-03 Y-10 Y-36AB Y-60 Y-70AB Y-71AB

APPENDIX J TASK 2.5 - SPECIFIC PARTNERSHIP 1996 TASKS

ATTACHMENT D

Specific Partnership FY-'96 Tasks

In addition to the demonstration plant construction and commissioning, the following

Partnership tasks, responsibilities and milestones are required for the Continuation Application submitted.

- 1. Prepare a four year test plan for Phase 3, Task 2-Operation.
 - a. The detailed first year plan and general four year plan will be initially issued by November. (ECH 11/13/95).
 - b. Bi-monthly Partnership reviews and re-issues will be done, with the final plan completed in March. (ECH 3/15/96).
- 2. Prepare the cost plan for the Continuation Application.
 - a. The project capital cost will be re-forecast in March. (DPD 3/15/96).
 - b. The Eastman operational cost inputs for the demonstration plant will be re-forecast in March. (WCJ 3/15/96).
 - c. The Partnership's Operating Plan Budget for Phase 3 (R. B. Moore's memo format) will be issued by the end of March. (RBM 3/25/96).
 - d. Monthly Partnership reviews (April, May) will be done, and the final DOE cost plan completed in May. (RBM 5/20/95).
- 3. Prepare the DOE Continuation Application for submittal. (WRB -