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

TECHNICAL PROGRESS REPORT NO. 6

For The Period

1 October - 31 December 1995

Prepared by

Air Products and Chemicals, Inc.
Allentown, Pennsylvania

and

Eastman Chemical Company
Kingsport, Tennessee

MASTER

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

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

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

Patents cleared by Chicago on October 22, 1996

DISCLAIMER

This report was prepared by Air Products & Chemicals, Inc. and Eastman Chemical Company for the Air Products Liquid Phase Conversion Company, L.P., pursuant to a Cooperative Agreement partially funded by the U.S. Department of Energy, and neither Air Products & Chemicals, Inc., Eastman Chemical Company, the Air Products Liquid Phase Conversion Company, L.P., nor any of their subcontractors nor the U.S. Department of Energy, nor any person acting on behalf of either:

(A) Makes any warranty or representation, express or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or
(B) Assumes any liabilities with respect to the use of, or for damages resulting from the use of, any information, apparatus, method, or process disclosed in this report.

Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute its endorsement, recommendation, or favoring by the U.S. Department of Energy. The views and opinions of authors expressed herein does not necessarily state or reflect those of the U.S. Department of Energy.

Table of Contents

ACRONYMS AND DEFINITIONS

Acurex	-	Acurex Environmental Corporation
Air Products	-	Air Products and Chemicals, Inc.
AFDU		Alternative Fuels Development Unit - The “LaPorte PDU.”
Balanced Gas		A syngas with a composition of hydrogen (H_2), carbon monoxide (CO), and carbon dioxide (CO_2) in stoichiometric balance for the production of methanol
Carbon Monoxide Gas		A syngas containing primarily carbon monoxide (CO); also called CO Gas
DME	-	dimethyl ether
DOE	-	United States Department of Energy
DOE-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 (H_2) over the stoichiometric balance for the production of methanol; also called H_2 Gas
IGCC	-	Integrated Gasification Combined Cycle, a type of electric power generation plant
IGCC/OTM		An IGCC plant with a "Once-Thru Methanol" plant (the LPMEOH™ Process) added-on.
KSCFH -		Thousand Standard Cubic Feet per Hour
LaPorte PDU	-	The DOE-owned experimental unit (PDU) located adjacent to Air 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 LPMEOH™ Process at an Integrated Coal Gasification Facility
psia	-	Pounds per Square Inch (Absolute)
psig	-	Pounds per Square Inch (gauge)
P&ID	-	Piping and Instrumentation Diagram(s)
SCFH	-	Standard Cubic Feet per Hour
Sl/hr-kg -		Standard Liter(s) per Hour per Kilogram of Catalyst
Syngas		Abbreviation for Synthesis Gas
Synthesis Gas		A gas containing primarily hydrogen (H_2) and carbon monoxide (CO), or mixtures of H_2 and CO; intended for "synthesis" in a reactor to form methanol and/or other hydrocarbons (synthesis gas may also contain CO_2, water, and other gases)
Tie-in(s)-		the interconnection(s) between the LPMEOH™ Process Demonstration Facility and the Eastman Facility
TPD	-	Ton(s) per Day
WBS	-	Work Breakdown Structure
wt	-	weight

Executive Summary

The Liquid Phase Methanol (LPMEOH™) Demonstration Project at Kingsport, Tennessee, is a $213.7 million cooperative agreement between the U.S. Department of Energy (DOE) and Air Products Liquid Phase Conversion Company, L. P. (the Partnership). The LPMEOH™ Process Demonstration Unit 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 LPMEOH™ Process Demonstration Unit, and will provide the technical and engineering supervision needed to conduct the operational testing program required as part of the project. As subcontractor to Air Products, Eastman will be responsible for operation of the LPMEOH™ Process Demonstration Unit, and for the interconnection and supply of synthesis gas, utilities, product storage, and other needed services.

The project involves the construction of an 80,000 gallons per day (260 TPD) 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 LPMEOH™ process is ideally suited for directly processing gases produced by modern-day coal gasifiers. Originally tested at a small, DOE-owned experimental unit in LaPorte, Texas, the technology provides several improvements essential for the economic coproduction of methanol and electricity directly from gasified coal. This liquid phase process suspends fine catalyst particles in an inert liquid, forming a slurry. The slurry dissipates the heat of the chemical reaction away from the catalyst surface, protecting the catalyst and allowing the methanol synthesis reaction to proceed at higher rates.

At the Eastman complex, the technology 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 Design - Phase 1 - activities; and moved also into Construction - Phase 2 - activities in October of 1995. The project required review under the National Environmental Policy Act (NEPA) to move to the construction phase. DOE prepared an Environmental Assessment (DOE/EA-1029), and subsequently a Finding of No Significant Impact (FONSI) was issued on 30 June 1995. The demonstration unit is scheduled to be mechanically complete in November of 1996.

During this quarter, site preparation construction work for the LPMEOH™ plant began on 2 October 1995. Air Products and Eastman held a status review with the DOE on 17 October 1995; following the Project groundbreaking ceremony held at the Kingsport site on 16 October 1995.

Detailed design of the facility is at a peak. Purchase of manual valves and of control valves and other instrumentation has begun. Equipment began arriving at the site. The majority of the piping tie-ins to the existing Eastman facility have been made. Site preparation work was completed, and foundation installation has begun.

An updated Project Management Plan was prepared. The updated plan reflects the restoration of three budget periods, as well as work completed since the previous plan. Activities to update the off-site Fuel-Use Test Plan were initiated. The draft Environmental Monitoring Plan (EMP) and the draft Demonstration Test Plan (DTP) were issued to DOE, comments received, and revised draft plans were being prepared.

Procurement of process equipment is nearly complete. Thirty-seven percent (37%) 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 31 December 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 is being 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 LPMEOH™ Process in conjunction with an integrated coal gasification facility." The project will also demonstrate the suitability of the methanol produced for use as a chemical feedstock or as a low-sulfur dioxide, low-nitrogen oxides alternative fuel in stationary and transportation applications. The project may also demonstrate the production of dimethyl ether (DME) as a mixed coproduct with methanol, if laboratory- and pilot-scale research and market verification studies show promising results. If implemented, the DME would be produced during the last six months of the four-year demonstration period.

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

B. Project Description

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 LPMEOH™ Process Demonstration. Three different feed gas streams (hydrogen gas, carbon monoxide gas, and balanced gas) will be diverted from existing operations to the LPMEOH™ demonstration unit, thus providing the range of coal-derived synthesis gas ratios (hydrogen to carbon monoxide) needed to meet the technical objectives of the demonstration project.

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

- *Reaction Area* - Synthesis gas preparation and methanol synthesis reaction equipment.
- *Purification Area* - Product separation and purification equipment.
- *Catalyst Preparation Area* - Catalyst and slurry preparation and disposal equipment.
- *Storage/Utility Area* - Methanol product, slurry, and oil storage equipment.

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

Reaction Area

The reaction area will include feed gas compressors, 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 B). Major accomplishments during this period are as follows:

Task 1.2 Permitting

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

- Issue the final Environmental Information Volume (EIV) to support the DOE's Environmental Assessment/Finding of No Significant Impact.

  - The NEPA review was completed 30 June 1995 with the issuance of an Environmental Assessment (DOE/EA-1029) and Finding of No Significant Impact (FONSI). The final 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).

  - The Draft EMP was submitted to DOE on 9 October 1995. DOE's comments were received, and a revised draft EMP is being prepared.

- Complete the design engineering necessary for construction and commissioning. This includedes Piping and Instrumentation Diagrams, Design Hazard Reviews, and the conduct of design reviews.

  - During this reporting period, Process Engineering work focused on:

    - Reviewing Piping Design
    - Designing Vent Scrubber (C-120)

  - Engineering work is focused on:

    - Designing the Vent Header System
    - Reviewing vendor data
    - Writing Specification for Instruments
    - Developing Distributed Control System (DCS) logic and documentation.

- Design Work focused on:

  - Completing the Foundation Bid Package
  - Completing the Fire Protection System Bid Package
  - Completing the Structural Steel Bid Package
  - Continuing Piping Design
  - Continuing Electrical Design

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 at the original Cool Water Gasification Facility in 1992, has become outdated. Since the site change to Eastman, the original fuel test plan under-represents new utility dispersed electric power developments, and possibly new mobile transport engine developments. The updated fuel 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.

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 LPMEOH™ 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

- Meetings with Acurex and with EPRI were held during the last week in October, in conjunction with EPRI's power generation (gasification) conference.

- The meeting with Acurex was held on 23 October 1995. Two agenda items were discussed: **1. The revisions needed to the Subcontract (dated 21 December 1992).** The main change needed was to incorporate the new Statement of Work (incorporated with Mod. A008 to the Cooperative Agreement) and to allow for the revised Project schedule that has been developed since the Project was reinitiated in October of 1993 at Kingsport. A draft amendment was to be prepared, for DOE review, which would also include minor changes to the Statement of Work, to clarify EPRI's role. **2. Fuel Test Plan Revisions.** The change in host site (to Kingsport) and the revised test schedule (from the original fuel test plan proposal) requires that a new fuel test plan be developed. Possibilities, including additional transportation (bus and automobile) and distributed power (electric utility) testing were discussed.

- Air Products and Acurex representatives met with with EPRI's Manager, Distributed Generation on 26 October 1995. This was an introductory meeting, to develop communication channels, and outline the possibilities for cooperative testing. The "Fuel Test Plan Update Objectives" and the exerpt from EPRI's brochure on Distributed Generation (Copy of each is in Appendix C.) was discussed. EPRI is involved in distributed generation benefit analysis, as well as in a number of fuel cell developments, which could be applicable to distributed generation. Since the fuel testing will not occur until 1998, immediate action by Acurex in developing the plan will be deferred. The possibilities for cooperation with EPRI in specific methanol product fuel-use testing will be explored as 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. No additional activity occurred during this reporting period.

Task 1.5.2 Commercialization Studies

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

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

Discussion

A number of areas have been identified as needing development to support specific commercial design studies. These include: a) Product Purification options; b) Front End Impurity Removal options; c) Catalyst Addition/Withdrawal options; and d) Plant Design Configuration options. These are summarized in the Process Engineering/Value Engineering Work Plan, included in Appendix D. Plant sizes in the range of 300 TPD to 1800 TPD and plant design configurations for the range from 20% up to 70% synthesis gas conversion will be considered. The Kingsport demonstration unit design and costs will be the basis for value engineering work to focus on specific cost reduction targets in developing the initial commercial plant designs.

Activities during this quarter

- During this quarter, the work on process economic studies was generally of lower priority than Task 1.3 Design Engineering. However, some work on this Task 1.5.2 was accomplished during the quarter:

  a) The design data base (from Labaratory R&D and the LaPorte PDU) for reactor temperature/catalyst life/productivity was reviewed. As a result, recommendations were made to include additional reactor productivity and catalyst life tests, at various reactor temperatures, in the Demonstration Test Plan (Task 2.3).

  b) Catalyst addition/withdrawal options were reviewed. The Kingsport demonstration unit design is capable of on-line partial addition/withdrawl (when the LPMEOH™ unit is operating), which provides maximum operating flexibility. Two other design options: Off-line partial addition/withdrawal (during planned outages of the LPMEOH^{TM} unit), and/or no partial addition/withdrawl capability; (like gas phase methanol units) would be less capital intensive systems, which could be applicable for certain commercial plants. The Demonstration Test Plan will be modified to include reactor temperature (ramping) data on fresh catalyst during startup; in order to provide the design data needed for these alternative catalyst addition/withdrawal options on future LPMEOH^{TM} unit designs.

  c) Process design work for the product purification options for three grades of methanol product: chemical, methyl tertiary butyl ether, and fuel; was partially completed.

- A few site-specific studies (Japan, Europe, EPRI), including dispatch (electric power) analysis and IGCC/design optimization will be developed. These studies would be done in parallel with the process economic study 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. Discussions are on-going with a few potential customers for add-on liquid phase methanol units, primarily for oil refinery gasification studies. These potential gasification projects are in the study phase. Site specific work will be deferred for now.

Task 1.5.3 DME Design Verification Testing

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

- Perform initial Design Verification Testing (DVT) for the production of dimethyl ether (DME) as a mixed coproduct with methanol. This activity includes laboratory R&D and market economic studies.
    - The project milestone schedule shows that the first decision point, on whether to continue with DME DVT, is targeted for 1 December 1996. DVT is required to provide additional data for engineering design and demonstration decision-making. The essential steps required for decision-making are: a) confirm catalyst activity and stability in the laboratory, b) develop engineering data in the laboratory, and c) confirm market(s), including fuels and chemical feedstocks. The DME Milestone Plan, showing the DVT work and the decision and implementation timing, is included in Appendix E.

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

Market Economic Studies

No specific work was done during this quarter. The market verification work (Items B. 2 and B. 3 in the DME Milestone Plan in Appendix E) will be deferred for at least another three months, pending positive results from the laboratory R & D program in finding a stable catalyst system.

Laboratory R&D

Initially, synthesis of DME concurrently with methanol in the same reactor was viewed as a way of overcoming the synthesis gas conversion limitations imposed by equilibrium in the LPMEOH™ process. Higher synthesis gas conversion would provide improved design flexibility for the coproduction of power and liquid fuels from an IGCC facility. The liquid phase DME (LPDME) process concept seemed ideally suited for the slurry-based liquid phase technology, since the second reaction (methanol to DME) could be accomplished by adding a second catalyst with dehydration activity to the methanol-producing reactor. Initial research work determined that two catalysts, a methanol catalyst and an alumina-based dehydration catalyst, could be physically mixed in different proportions to control the yield of DME and of methanol in the mixed product. Proof-of-concept runs, in the laboratory and at the 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 LPMEOH™ process catalyst system. Further studies of the LPDME catalyst deactivation phenomenon were therefore initially undertaken under DOE Contract No. DE-FC22-95PC93052, and are being continued under Task 1.5.3. 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--are being undertaken. During the last quarter, work concentrated on the screening of LPDME catalysts. Most catalysts exhibited poorer stability than the standard dual catalyst system. Efforts were also made to understand the nature of the detrimental interaction between the methanol synthesis and dehydration catalysts under LPDME conditions. The quarterly report, prepared for Contract DE-FC22-95PC93052 for the period July - September 1995, is included in Appendix D for reference.

Summary of Laboratory Activity and Results

- Baseline LPDME tests using a copper-lined reactor for both methanol and DME synthesis exhibited the same behavior as observed for the normal reactor. Thus, abrasion of the stainless steel walls was ruled out as a possible source of the faster aging observed in the laboratory reactors relative to the AFDU.

- At low levels of lithium doping, the alumina dehydration catalyst was not effective in reducing aging in LPDME synthesis. Higher levels had reduced methanol catalyst aging, but deactivated the dehydration function. Work to understand the correct level of doping continues.

- The bifunctional single particle DME catalyst patented by Shell deactivated rapidly in both slurry and gas phase reactions.

- Tetraglyme, a hydrophobic ether, is an unsuitable slurry media for DME service due to its reactivity with alumina at reaction temperatures.

- Encapsulation of alumina with silica oxide did not increase catalyst life. Fracture of the alumina particles is the most likely cause of this behavior.

- Modified Catapal B gamma-alumina, zinc ion-exchanged zeolite, and silicon ammonia phosphate were tested for use as the dehydration catalyst. Modification of the gamma-alumina did not help with the stability of the catalyst system. The activity of the latter two samples was nil. Although the silicon ammonia phosphate sample is inert in the LPDME run, fast long-term deactivation of the methanol catalyst was observed in this catalyst system.

- Life data reported in the literature using a copper oxide-modified alumina dehydration catalyst are conclusive because of (unreported) mass transfer limitations in their testing apparatus. The life run was repeated under the conditions free of mass transfer limitations in a 300cc autoclave using Air Products' version of this catalyst. The results show that the catalyst system deactivated at a rate similar to Air Products' standard dual catalyst system.

- Catalysts in both LPMEOH^{TM} and LPDME runs deactivate faster in the microclave (50cc) reactors with smaller volume-to-surface ratios. This suggests that there is an experimental artifact, or a deactivation mechanism, that becomes more pronounced in a reactor with smaller volume-to-surface ratio. Studies to identify what this artifact or deactivation mechanism is, and how much it contributes to the long-term deactivation of the catalysts in LPDME runs are on-going. Poisons in the feed gas have been ruled out.

Task 1.5.4 Administration and Reporting

An updated Project Management Plan dated 10 October 1995 was prepared and submitted. The updated plan reflects the restoration of three budget periods, as well as work completed since the previous Project

Management Plan was submitted. The roles of Acurex Environmental Corporation and of the Electric Power Research Institute were more clearly defined, including their relationship to the Partnership, Air Products, Eastman Chemical Company, and each other. DOE review found the updated plan to be detailed and well prepared. Some clarifications and revisions for the next update were suggested.

The Liquid Phase Methanol (LPMEOH™) Demonstration Project Groundbreaking Ceremony was held on 16 October 1995. The Project became the 31st in the U. S. Department of Energy's Clean Coal Technology Program to be under construction. The Air Products and Eastman teams planned and coordinated the ceremony. Acknowledgment letters to Congressional, U.S. Department of Energy and Corporate officials who participated in the ceremony are included in Appendix F. All the participants showed the commitment and dedication to the successful demonstration of the LPMEOH™ Process. The Kingsport area newspapers and local television stations covered the event. Extracts of the news coverage is also included in Appendix F.

A project review meeting was held on 17 October 1995 in Kingsport. Attendees from Air Products, Eastman, and DOE participated. The project status was reviewed. Piping design is fully underway, and site preparation construction work has started. Construction for foundation and underground work, began on 27 October 1995. The preparation of the Environmental Monitoring Plan and the Demonstration Test Plan was discussed. The design and construction schedule forecast for mechanical completion is 15 November 1996. The agenda, extracts from the meeting handouts, and the meeting notes are included in Appendix G.

The Milestone Schedule Status Report and the Cost Management Report, through the period ending 31 December 1995, are included in Appendix H. These two reports show the current schedule, the percentage completion and the latest cost forecast for each of the Work Breakdown Structure tasks. The demonstration unit is scheduled to be mechanically complete in November of 1996. Thirty-seven percent (37%) of the $36 million of funds budgeted for the Kingsport portion of the LPMEOH™ Process Demonstration Project through Budget Period Period No. 2 have been expended (as invoiced), as of 31 December 1995.

The 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 still forecast to commence on 31 October 1996, and to be completed by 01 February 1997.

The Cost Plan for fiscal year (FY) -1996 was submitted on 31 October 1995. A major cost reforecast, by Task, was included in the FY-96 Cost Plan. The reforecasted costs are shown in the Cost Management Report in Appendix H, and are summarized in the following table.

Cost Forecast Summary Table (Costs in $1,000's) Task // Date Submitted:	FY1996 Cost Plan 31 Oct. 1995	FY1995 Cost Plan 26 Sep. 1994	Change in Forecasted Costs
Non-Kingsport Portion of Project			
1.1 Pre-award & Proj. Def. (Prior to Mod 002.):	$16289	$ 16851	$ (562)
1.4 Off-site Testing (Plan and Design)	$ 320	$ 324	$ (4)
2.4 Off-site Testing (Construction)	$ 261	$ 305	$ (44)
3.4 Off-site Testing (Operation)	$ 3840	$ 3792	$ +48
Sub-Total; Non-Kingsport Portion of Project:	**$ 20710**	**$ 21272**	**$ (562)**
Kingsport Portion of Project:			
1.1 Project Definition	$ 1021	$ 1230	$ (209)
1.2 Permitting	$ 246	$ 288	$ (52)
1.3 Design Engineering	$ 9960	$ 8206	$ +1754
1.5 Planning, Admin. & DME DVT	$ 1892	$ 1663	$ +229
2.1 Procurement	$ 9783	$10953	$ (1170)
2.2 Construction	$11200	$11500	$ (300)
2.3 Training & Commissioning	$ 1197	$ 897	$ +300
2.5 Planning & Admin.	$ 681	$ 681	$ 0
Sub-Total; Kingsport Phase 1& 2 Portion:	**$35980**	**$35418**	**$ +562**
Sub-Total; Phase 3 (Operation at Kingsport)	**$157010**	**$157010**	**$ 0**
Total Project Cost Forecast	**$213700**	**$213700**	**$ 0**

Discussion of Cost Forecast changes

There was no change in the total cost forecast, however, there were variations within the WBS tasks.

- The Project Definition (Task 1.1) and Permitting (Task 1.2) tasks are essentially complete. Their forecasted costs (including costs prior to Mod. 002) were reduced by about $0.8 million. The prior to Mod. 002 cost reduction reflects lower actual costs, as audited, of the prior work. The reduction in Tasks 1.1 and 1.2 at Kingsport, reflect the good cooperation of the three (DOE/Eastman/Air Products) participants in defining the scope and the management basis for the Project at Kingsport, and the early completion of these Budget Period No. 1 tasks.

- The Procurement (Task 2.1) task is now more than 80% complete. The cost forecast was reduced by about $1.2 million, reflecting actual expenditures, commitments and an estimate of the costs to complete. The reduction in the cost forecast reflects the good initial process scope definition (from Task 1.1), and favorable competitive bidding.

- The Design Engineering (Task 1.3), the Planning, Administration, & DME -DVT (Tasks 1.5) tasks are about 70% completed. The cost forecast for these tasks was increased by about $2.0 million. This reflects:

  a) The impact of the unexpected 3 to 4 month delay in transitioning from Budget Period No. 1 to Budget Period No. 2. The project team was kept together during the transition approval period, in order to not lose momentum, and to try and hold the schedule for start-up of in December of 1996. Some initial process design work was accomplished during this intransition period, but the delay in initiating Procurement (Task 2.1) necessarily delayed the availability of the engineering details of the process equipment needed to initiate the foundation and structural engineering design. This required extra engineering design revision, as the final equipment design details differed from the preliminary designs incorporated in the construction bid packages.

  b) The detailed engineering design requirements were under-estimated in the initial Cost Plan budget. The first-of-a-kind, pioneer plant design effort that was needed to provide the scale-up to commercial size was underestimated. The acquisition of the operational data to support future engineering designs, as better defined in the Environmental Monitoring Plan and in the Demonstration Test Plan (DTP) documents, added an unanticipated engineering effort for the final plant design.

- There were small, off-setting, changes in the other Tasks. The commissioning work (Task 2.3) has been better defined during the DTP development, and some of the work estimated to be in the Start-up (Task 3.1) should really have been included in

Task 2.3. The Phase 3 cost reforecast will be done later (for the FY-97 Cost Plan), but will reflect lower costs for Task 3.1 (Start-up). The Construction costs (Task 2.2) were reduced due to favorable bidding on the early construction bid packages, and on the anticipation that the design and procurement deliverables schedule will be maintained, so that construction work can be completed as scheduled.

The monthly reports for October, November, and December were submitted. These reports include the Milestone Schedule Status Report, the Project Summary Report, and the Cost Management Report.

Task 2.1 Procurement

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

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

  - The status of equipment purchases is shown in the "Specification Status-Equipment" report included in Appendix I. This report, prepared on 2 Jan 1996, shows that orders have been placed for all equipment items except the Vent Scrubber.

  - The reactor fabrication continues at Joseph Oat Corporation in Camden, N.J. Three of the seven stainless steel clad plates were sensitized (to carbonyl formation) during an annealing step prior to rolling. This was due to their being left at the annealing temperature for too long a period of time. These plates were required to be reheattreated to restore the stainless steel to a non-sensitized condition. The time involved in analyzing the plates, deciding on an acceptable solution, reheat treating, and rolling has caused a 5-month delay in completing fabrication of the reactor. As of the end of December, the shell plates have been fit up and most of them welded together.

    The nozzles have been welded into the top and bottom heads. The bottom head has been welded to the shell course.

    The internal heat exchanger fabrication is progressing. The tubes have been laid in their support grid assembly and the header piping has been fabricated. The bottom headers have been welded to tube bundle.

    The inlet gas sparger components have been fabricated. Air Products has requested Joseph Oat to punch the sparger holes again to meet the tolerance requirements. The current ship promise date is March of 1996.

Task 2.2 Construction

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

- Provide construction management for contractor coordination and compliance with design, construction, and quality control standards.

    - Air Products' construction site manager relocated to Kingsport to oversee the Air Products' portion of construction activities at the site. In addition, a clerk and secretary were added to the Air Products on-site construction management. This team is supported by a home office (Allentown) construction team that prepares bid lists, writes and prepares bid packages, and evaluates and awards the construction contracts.

    - Eastman is using Midwest Technical for detailed design of the 'Tie-In Work' and Fluor Daniel for construction. Eastman has also assigned a construction coordinator.

- 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.

    - Site Preparation work began on 2 October 1995. This includes intital grading, soil fill, covering with stones, and compaction the north portion of site, and installation of area storm drains. Installation of foundations and undergrounds began on 27 October 1995. This work includes installation of most major foundations and tank farm dikes, underground process drains, and underground electrical grounding. The scheduled start date for structural steel and major equipment erection is 5 February 1996.

- Complete mechanical construction so that checkout and commissioning can be started in Budget Period No. 3.

    - Overall construction is 8% complete as of the end of December of 1995. The targeted Mechanical completion date remains 15 November 1996. Start-up activities are scheduled to begin 31 October 1996 and demonstration unit operation is scheduled to start 16 December 1996.

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.

  - An outline of the First Year Operating Plan was issued on 3 October 1995, for Partnership comment. This covers Phase 3, Task 2.1.1 - Process Shakedown and Catalyst Aging; and shows that the catalyst is still aging at the end of year 1. An early test (#2) at Texaco-type synthesis gas conditions and an early high superficial veloctiy test (#3) are proposed during the first few weeks of operation. This will provide a comparison with the LaPorte Process Development Unit's results for IGCC commercial applications. A thorough analysis of carbonyl levels in the fresh feed gas will be made before test #3, so that the confidence level in bypassing the carbonyl guard bed (necessary for high flow operation) is high. The Partnership discussed operating ideas for years 2 through 4, and an overall four-year operating test schedule is being prepared.

    The first draft (Rev. 0) of the four-year Demonstration Test Plan (DTP) for Phase 3, Task 2 Operation was issued for review and comment on 22 December 1995. A copy of the cover letter and of the Test Plan Table (4 pages) is included in Appendix J. The DTP will be iterated and improved over the next few months, with input from all the participants as well as from the Phase 1, Task 5 commercial economic studies. The final draft of the DTP is due to be submitted in August of 1996.

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

  - Work was initiated to prepare the operator training program outline and training schedule.

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 Demonstration Test Plan.

    - The first update of the Partnership Annual Operating Plan was prepared and submitted (See Quarterly Technical Progress Report No. 5). 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 1996 are:

        - Continue construction so that the LPMEOH^{TM} demonstration unit will be ready for commissioning and start-up 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.

- 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 Task 1.5.

E. Planned Activities for the Next Quarter

- Continue detailed engineering design and procurement
- Order prefabricated structural steel.
- Order prefabricated pipe.
- Bid and award the structural steel and Equipment Erection Construction package, and start erection work.
- Bid and award the General Mechanical Costruction package.
- Issue the Instrument and Electrical Construction bid package.
- Continue shipment of equipment to the site. Expedite reactor fabrication.
- Review and revise the draft Environmental Monitoring Plan.
- Review and revise the draft Demonstration Test Plan.
- Hold Project Review/Update Meeting with the DOE.

F. Summary

Site Preparation construction work for the LPMEOH™ demonstration unit began on 2 October 1995. Air Products and Eastman held a status review with the DOE on 17 October 1995; following the 16 October 1995, Project groundbreaking ceremony at the Kingsport site.

Detailed design of the facility is at a peak. Purchase of manual valves and of control valves and other instrumentation has begun. Equipment began arriving at the site. The majority of the piping tie-ins to the existing plant have been made. Site preparation work was completed, and foundation installation has begun.

An updated Project Management Plan was prepared. The updated plan reflects the restoration of three budget periods, as well as work completed since the previous plan.

Activities to update the off-site Fuel-use Test Plan were initiated. A meeting was held at Acurex to discuss timing for the development of a final fuel-use test plan. Guidance on distributed power generation applications to meet electric utility industry needs will be provided by EPRI.

DOE's comments on the draft Environmental Monitoring Plan (EMP) were received, and a revised EMP is being prepared. The draft Demonstration Test Plan was issued for review.

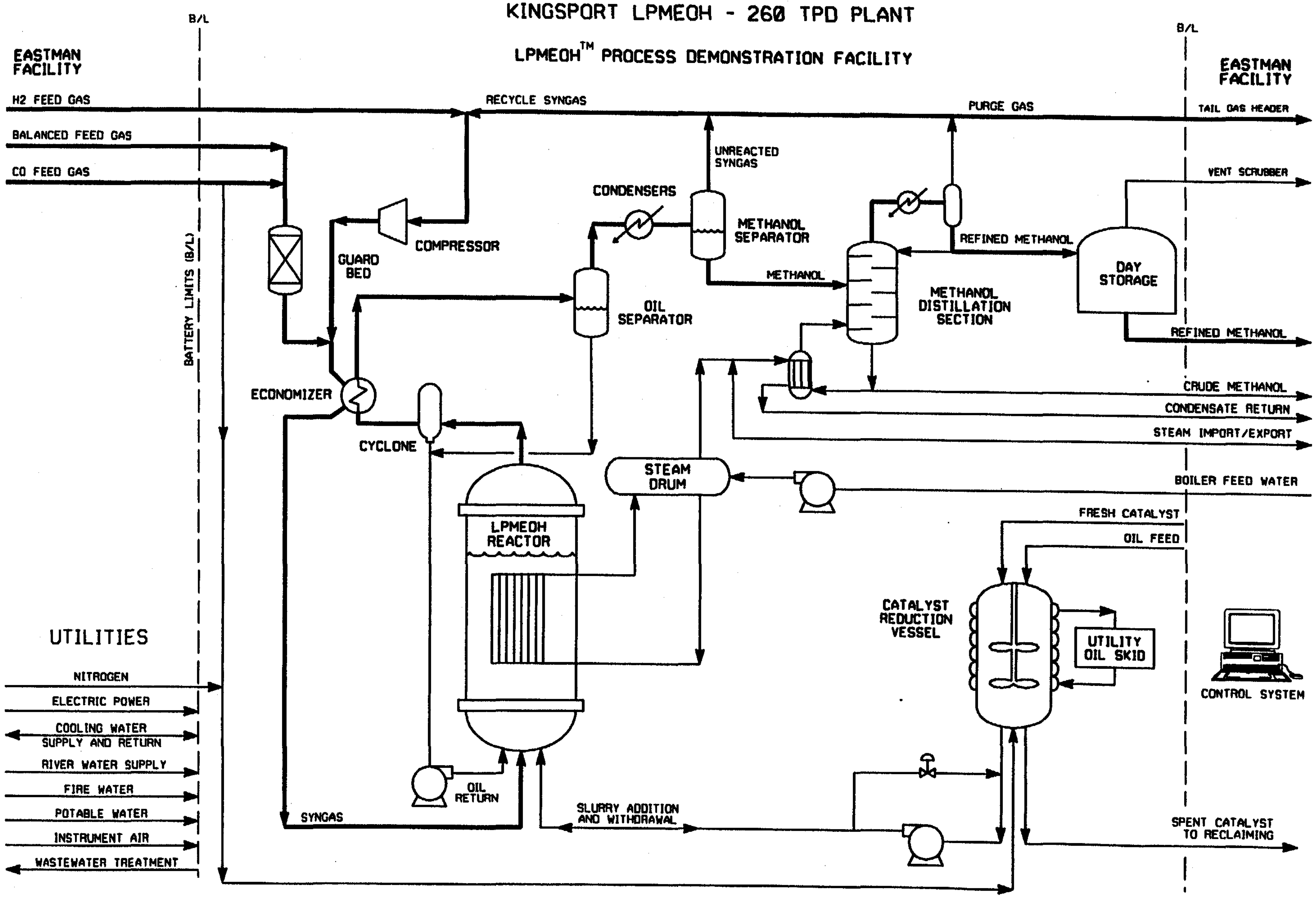
Procurement of process equipment is nearly complete. Thirty-seven percent (37%) of the $36 million in funds budgeted for the Kingsport portion of the LPMEOH™ Process Demonstration Project through Budget Period No. 2 have been expended (as invoiced) as of 31 December 1995. The demonstration unit is scheduled to be mechanically complete in November of 1996.

APPENDICES

APPENDIX A - SIMPLIFIED PROCESS FLOW DIAGRAM

SIMPLIFIED PROCESS DIAGRAM
KINGSPORT LPMEOH - 260 TPD PLANT

LPMEOH™ PROCESS DEMONSTRATION FACILITY



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

7/18/95

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

PROJECT EVALUATION PLAN FOR BUDGET PERIOD NO. 2

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

1. Phase 1 - Task 2 - Permitting

- Issue the final Environmental Information Volume to support the U.S. Department of Energy's (DOE's) Environmental Assessment/Finding of No Significant Impact.

- Obtain permits necessary for construction and operation.

2. Phase 1 - Task 3 - Design Engineering

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

- Prepare the Environmental Monitoring Plan.

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

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

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

- Update the (fuel and chemical) product-use test plan, that will better meet the technical objectives of the Project and serve the needs of commercial markets.

- Complete economic studies of the important commercial aspects of the LPMEOH™ Process to enhance Integrated Gasification Combined Cycle (IGCC) electric power generation. These studies will be performed by Air Products and Chemicals, Inc. and the Electric Power Research Institute, and used to provide input to the LPMEOH™ Process Demonstration Facility operating test plan (Phase 2, Task 5).

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

- Submit all Project status, milestone schedule, and cost management reports as required by the Cooperative Agreement.

5. Phase 2 - Task 1 - Procurement

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

6. Phase 2 - Task 2- Construction

- Complete mechanical construction so that checkout and commissioning can be started in Budget Period No. 3.

- Erect the major equipment and structural steel. Install the large bore piping, electrical, and insulation such that instrument checkout and equipment commissioning work can be completed during the 60-day Continuation Application approval period.

- Provide construction management for contractor coordination and compliance with design, construction, and quality control standards.

7. Phase 2 - Task 3 - Training and Commissioning

- Prepare a four (4)-year test plan for Phase 3, Task 2-Operation.

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

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

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

9. Phase 2 - Task 5 - Planning and Administration

- Prepare annually an updated plan for the remaining activities. The first annual plan will update the remaining Phase I and Phase II tasks. The second annual plan will include an updated Phase III Operating Plan, identifying specific goals and milestones for the first twelve months of operation, and a general plan for the remaining years to achieve the Project's market penetration objectives.

- Submit all Project status, milestone schedule, and cost management reports as required by the Cooperative Agreement.

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

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

WRB/jjs/Proeva.

APPENDIX C - TASK 1.4 - FUEL TEST PLAN UPDATE OBJECTIVES

and

Excerpt from EPRI Distributed Generation Brochure

Fuel Test Plan Update Objectives

The Technical Objective (No. 4) is:

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

(a) Analysis of samples of the fuels and combustion exhaust gas streams for emission levels of sulfur dioxide, nitrogen oxides, carbon monoxide, carbon dioxide and *hazardous air pollutants;* and

(b) A cost advantage for the use of methanol produced by the LPMEOH™ *demonstration facility* by means of a economic comparison with conventionally accepted fuels."

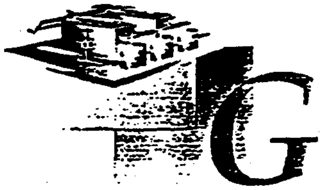
Discussion

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

Update Objective

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

WRB/jjs/6/27/94
Fuel Test Plan



Gas-Fired Distributed Generation

A new approach to meeting cost, environmental, and customer service objectives is distributed generation, which creates an integrated delivery network employing small, modular generators as well as central stations.

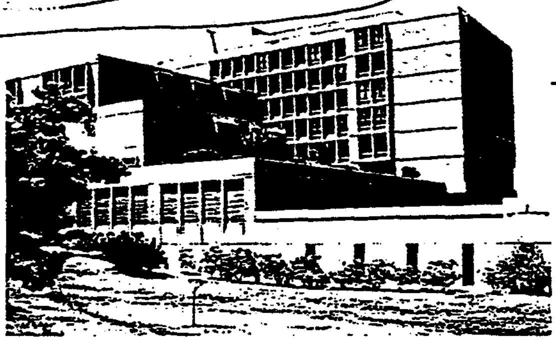
Such modular generators—typically hundreds of kW to tens of MW—can normally be sited, permitted, and assembled quickly, and even relocated should needs change. Strategic placement in the subtransmission and distribution system provides frequency control, voltage regulation, and local spinning reserve capacity, and may allow deferral or avoidance of T&D capacity upgrades.

Some distributed generating technologies—fuel cells in particular—operate quietly with extremely low emissions and are thus well suited for environmentally sensitive areas.

Moreover, placement of distributed generators near customer sites enables utilities to offer more reliable service to facilities with critical power needs. Such tailored energy services may prove paramount in meeting competitive challenges by other power producers.

Commercial technologies for distributed generation include internal combustion engines and small CTs. Emerging technologies include fuel cells, markedly improved CTs, and improved reciprocating engines.

Page Break



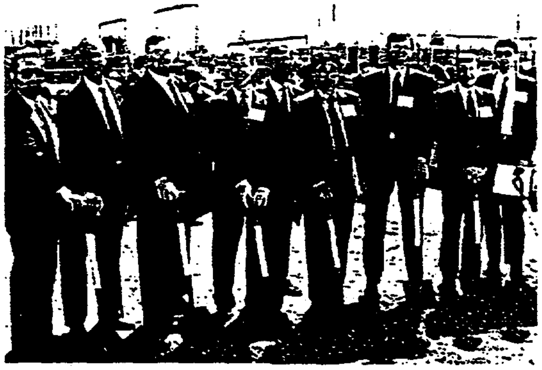
Locating modular generating units strategically within subtransmission and distribution systems or near customer sites allows utilities to offer tailored energy services to enhance customer value, such as exceptionally high reliability for hospitals. This distributed generation strategy may also defer or avoid the expense of T&D upgrades. The AFPS Business Unit supports this approach to competition based on "delivered energy service," rather than simple "busbar cost of electricity," with economic guides, technology development and assessment programs, and information on electrical interface requirements.

AFPS products support utility evaluation and implementation of natural-gas-fired distributed generation. The Business Unit's portfolio of key products includes:

- updated guides for identifying high-value applications of various distributed generation technologies
- a comprehensive commercialization program for molten carbonate fuel cells (MCFC) with efficiencies greater than 60% (LHV)
- small (<50 MW), low-emitting CTs with simple-cycle efficiencies greater than 50%

The prospect of two U.S. manufacturers offering commercial MCFC units in the next three to five years represents the culmination of more than a decade of fuel cell R&D at EPRI. A utility "buyers group" has placed advance orders for nearly 100 MW of initial commercial units in exchange for future royalties. As demand grows, the economies of standardization and mass production will reduce unit costs substantially, expanding cost-effective application.

If production cost targets are met, MCFC technology could already be cost-effective in some instances. Working with EPRI, the Los Angeles Department of Water & Power, Central and South West Services, and Oglethorpe Power evaluated the benefits of prototype 2-MW fuel cells in various distributed applications. Projected savings ranged from 2 to 71 mills/kWh.



AFPS staff provide technical management for a consortium of utilities and manufacturers that will demonstrate a commercial prototype of a modular 2-MW molten carbonate fuel cell. This quiet, efficient unit will serve customers of the City of Santa Clara, California. Groundbreaking ceremonies were held in April 1994; commercial generation will begin in 1996. The Business Unit also manages collaborative development programs for solid oxide fuel cells and monitors progress in phosphoric acid fuel cells, small advanced gas turbines, and high-efficiency reciprocating engines.

RB
5/11/95.

Excerpt from EPRI Advanced Fossil Power Systems (BR-103977 1994) Brochure
"Technologies to Minimize the Delivered Cost of Electricity"

APPENDIX D - TASK 1.5.2 - PROCESS DEVELOPMENT/VALUE ENGINEERING WORK PLAN

Process Development/Value Engineering

- (Task 5) Work Plan Worksheet (for Objective 1.c). -

Description of Task	Comments
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)	• 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.
A.2. Reactor Temperature/Catalyst Life/Production Study	• Review lab R&D data to optimize Catalyst Life/Product Cost.
B. To Support Site Specific Studies	
B1. Develop simplified (material balance) tools to answer LPMEOH screening bids.	• Eliminate the need for process support on preliminary requests. • Investigate Aspen "Optimizer" for optimization studies.
B2. Product Purification Options, Optimizations, "Standard" Designs.	Standardized designs for Chem grade, MTBE grade and fuel grade. Draw from Kingsport experience for equipment specifications, plot plan, etc.
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.	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?).
B4. Plant design configuration studies:	
1. Feed Compression (when and how much).	Different gas compositions; combined with different conversion studies (below).
2. Configuration for 20, 50, 70% conversion.	Different gas compositions; combined with different compression studies (above).
3. Plant size breakouts (single reactor vs. two).	

APPENDIX E - TASK 1.5.3 - DME MILESTONE PLAN

TASK 1.5.3 - DME (DE-FC22-95PC93052) QUARTERLY REPORT

(Excerpt from the July - September 1995 Report)

DME MILESTONE PLAN

1. Design Verification Testing:

A. Laboratory R&D, Verification

1. Catalyst Activity and Stability Testing, and Engineering Data	Mar. '95 - Aug. '96

B. Market Verification

1. Up to 3 wt% DME, as M-100 Diesel Replacement Engine Tests - (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 Kingsport a. Economics, process basis study	by Aug. '96

C. Decision to Continue or Drop Demonstration	by Dec. '96

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

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

3. Implementation (Provisional) Plan:

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

TASK 3: RESEARCH AND DEVELOPMENT

Task 3.1 Improved Processes for DME

This quarter's work concentrated on the screening of alternative LPDME catalysts. The candidates were chosen from the following categories based on our current understanding of catalyst deactivation:

- Chemically modified alumina to reduce the driving force of the interaction between methanol and dehydration catalysts.
- Physically modified alumina to eliminate the contact between the two catalysts.
- Other solid materials of moderate acidity.
- Alternative methanol catalysts such as single catalyst systems.

Most catalysts tested exhibited poorer stability than the standard dual catalyst system (S3-86 plus Catapal B g-alumina). One catalyst system exhibited improved long-term stability of the methanol catalyst.

A Japanese laboratory has recently reported a catalyst system that is stable under LPDME conditions. However, the life data are not reliable, or are at least inconclusive, since our analysis of the article showed that the life-test experimental conditons were subject to severe mass transfer limitations. We repeated the life run under the conditions free of mass transfer limitations. The results show that the catalyst system deactivates at a rate similar to our standard dual catalyst system.

Efforts were also made to understand the nature of the detrimental interaction between the methanol synthesis and dehydration catalysts under LPDME conditions. SEM (scanning electron microscopy) and EDS (energy dispersive X-ray spectrometry) analysis of different catalyst systems were used to determine if deactivation is due to the migration of zinc and copper from the methanol catalyst to the dehydration catalyst (e.g., g-alumina). Some of the results indicate that migration of zinc and copper from the methanol catalyst to alumina occurred under the reaction conditions. However, other inconsistent observations call for more complementary analyses before a definite conclusion can be made.

3.1.1 Screening of Alternative Catalyst Systems

3.1.1(i) δ-alumina

In the last quarterly we reported a dual catalyst system in which improved long-term stability of the methanol catalyst under LPDME conditions was observed for the first time. The dehydration catalyst in that system is a γ-alumina heavily doped with potassium (Run 14045-85). Improved long-term stability of the methanol catalyst was observed again this quarter when a δ-alumina (Condea Puralox scca-5/90) was used along with the S3-86 methanol catalyst (Run 14656-49). The run was carried out under the standard reaction conditions (250°C, 750 psig, 6,000 GHSV, 80:20 catalyst ratio) using Texaco gas. The results from this run are depicted in Figures 3.1.1a & b, along with those from the catalyst systems containing the K-doped γ-alumina (14045-85) and virgin γ-alumina (e.g., standard system, 11483-81). As can be seen in Figure 3.1.1a, the long-term stability of the methanol catalyst in this catalyst system is improved.

Currently, we are trying to understand why the long-term stability of the methanol catalyst is improved in this system and in the system that contains the K-doped γ-alumina. As shown in Figure 3.1.2b, the dehydration activity of both δ-alumina and K-doped γ-alumina are very much lower ($k_d \leq 3.0$ and 1.3, respectively) than that of γ-alumina. We have tested systems with similar and poorer dehydration activity, such as those using Chabazite ($k_d \leq 1.3$), Mg-Y ($k_d \leq 0.9$), and a silica alumina sample ($k_d \leq 0.6$) as dehydration catalysts (see April-June quarterly). However, the long-term deactivation of the methanol catalyst in these systems is similar to the relatively rapid deactivation of the standard system. This indicates that lower dehydration activity does not guarantee better long-term stability of the methanol catalyst. What we would like to understand is whether the low dehydration activity is a necessary condition for the long-term stability of the methanol catalyst. If it is, we should look for weakly acidic materials with high site density as dehydration catalysts, since it is assumed that the low activity seen in these two systems is related to the weak acid sites. If low activity is not a necessary condition, we need to understand what is special about these two systems so that screening guidelines can be developed for alternative dehydration catalysts.

3.1.1(ii) Modified γ-alumina

Three modified Catapal B γ-alumina catalysts have been tested. The modification methods include impregnation using potassium, silylation using $(Me_3Si\text{-})_2NH$, and encapsulation with SiO_2. The potassium-impregnated sample was prepared by the incipient wetness method using a KOH aqueous solution. This sample had higher dehydration activity due to a lower K loading than the K-doped sample discussed in section 3.1.1(i) (0.15 vs. 0.8 wt% of K). The goal of this new preparation was to determine if the improved long-term stability of the methanol catalyst seen in the sample with 0.8 wt% K loading could still be retained with a more active K-doped alumina. Silylation using $(Me_3Si\text{-})NH$ was aimed at removing the strong acid sites on Catapal B γ-alumina. The sample was prepared by refluxing Catapal B γ-alumina in $(Me_3Si\text{-})_2NH$. If an inert, porous layer of SiO_2 can be formed on the outside of alumina particles, intimate contact between the alumina and the methanol catalyst should be prevented. This, in turn, should lead to good stability of the catalyst system. The encapsulation of the alumina with SiO_2 was carried out according to a DuPont patent (US Patent 4,677,084). The LPDME runs using these three

Figure 3.1.1 LPDME Life Runs of Different Catalyst Systems

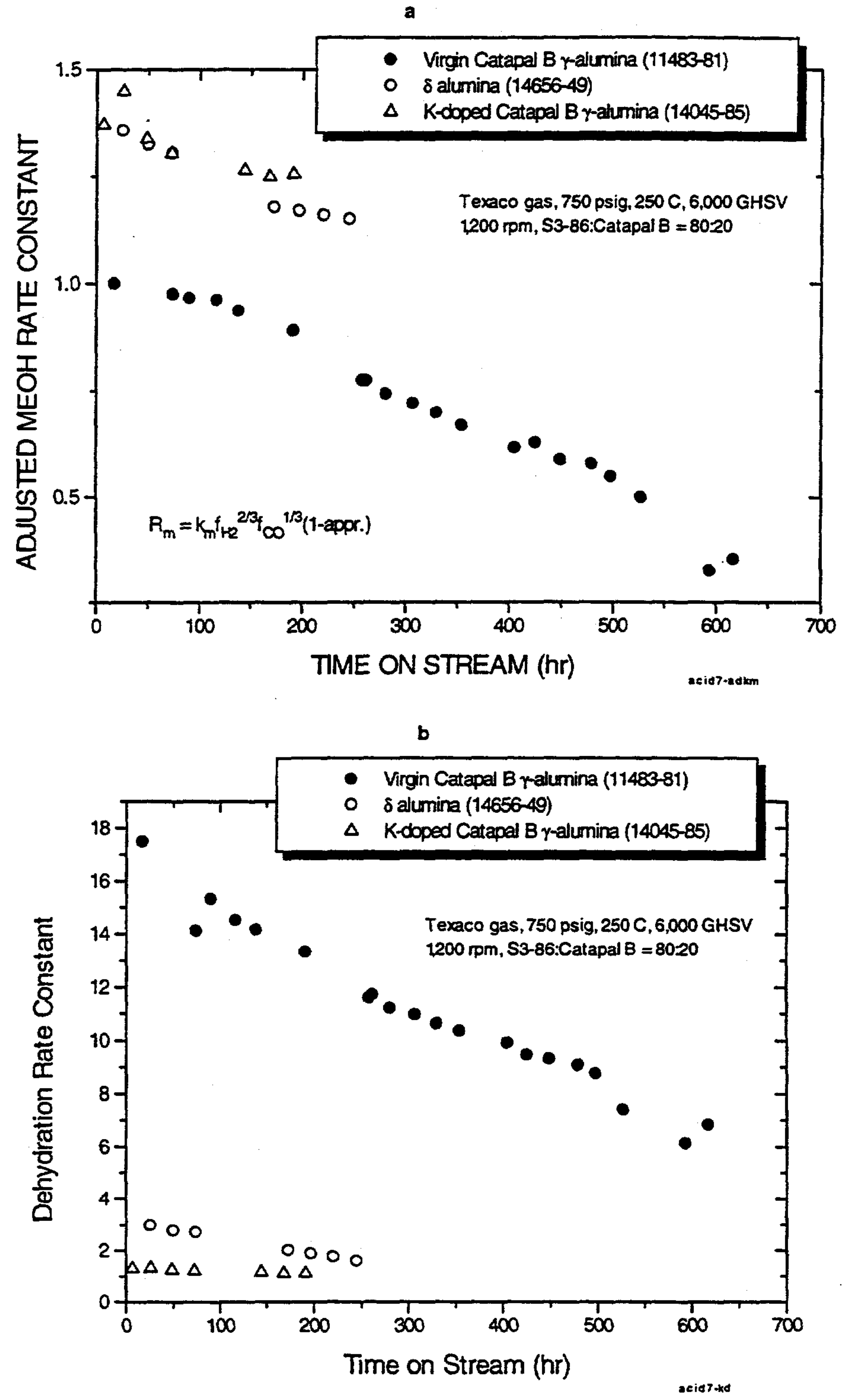
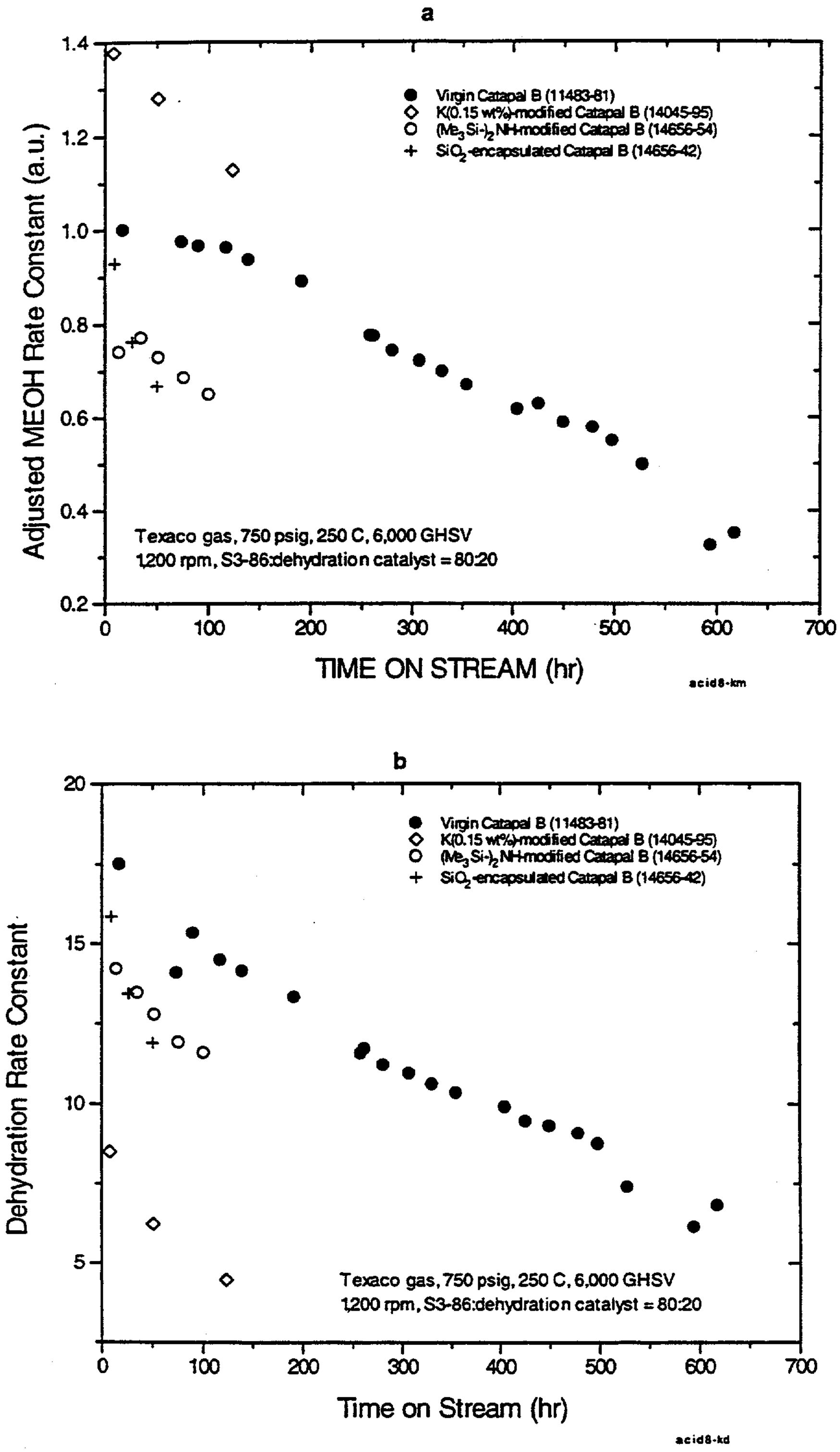


Figure 3.1.2 The Stability of Different Catalyst Systems under LPDME Conditions



dehydration catalysts were all conducted under the standard reaction conditions using BASF S3-86 as the methanol synthesis catalysts. The results are plotted in Fig. 3.1.2a and b against those from the standard catalyst system.

In contrast to the previous result obtained using 0.8 wt% K loading, doping the alumina with 0.15 wt% of K did not result in better long-term stability of the methanol catalyst. Figures 3.1.2a and b shows that the long-term stability of both methanol synthesis and dehydration catalysts in this system (14045-85) is poorer than that of the standard dual catalyst system (11483-81). We do not understand why the potassium doping at different levels leads to different effects on the long-term stability of the catalyst system. The effect cannot be attributed to the acidity of the dehydration catalyst because the dehydration activity of the 0.5 wt% sample ($k_d = 8.5$) lies between the virgin alumina ($k_d = 17.5$) and the 0.8 wt% sample ($k_d = 1.3$).

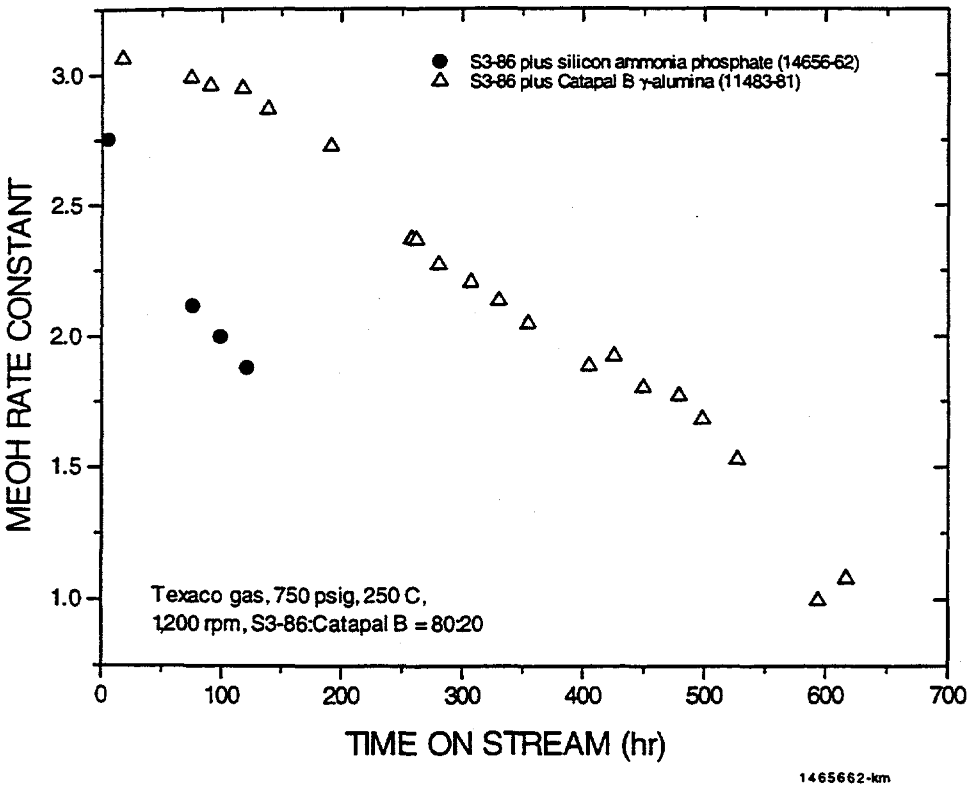
Figures 3.1.2a and b shows that silylation of Catapal B γ-alumina with $(Me_3Si-)_2NH$ does not result in better long-term stability. The initial deactivation of the methanol catalyst in this system is also greater than that in the standard system. Since silylation in general is believed to passivate the strong acid sites on acidic solid materials, this greater initial deactivation is probably due to the poisonous species in the $(Me_3Si-)_2NH$-modified alumina.

The SiO_2-encapsulated alumina sample was analyzed prior to the run by scanning electron microscope (SEM) and energy dispersive X-ray spectrometry (EDS). The results show that most of the silica, as desired, was on the exterior of the alumina particles. However, this sample did not bring about the better stability we expected. As shown in Figures 3.1.2a & b, the stability of this system is poorer than the standard catalyst system; both methanol and dehydration catalysts deactivated very rapidly. Another run (not shown) using the same sample calcined at a higher temperature yielded a similar result. The poor stability may have been due to the poor mechanical strength of the encapsulated alumina particles. Evidence of this hypothesis is the observation that during the SEM/EDS experiments the preparation of the cross section of alumina particles was much more difficult compared to virgin Catapal B γ-alumina because the particles broke apart readily. The SEM cross section of an alumina particle also appeared flaky. The exposure of the alumina to a fairly strong acidic environment (pH = 2) during the encapsulation may be the reason for the lower strength. If the particles cannot retain their integrity, a protective layer of SiO_2 will be useless. The cleavage of the particles under the reaction conditions (strong agitation) will create an active alumina surface, which then interacts with the methanol catalyst to cause the deactivation of both catalysts. This issue will be examined in greater depth, and further efforts will be made to prepare encapsulated alumina samples.

3.1.19(iii) Other Acidic Materials

Two samples we tested in this category are silica ammonia phosphate and zinc ion-exchanged zeolite Y (Zn-Y). The LPDME run using the silicon ammonium phosphate sample as the dehydration catalyst (Run 14656-62) was conducted under the standard conditions (250°C, 750 psig, 6,000 GHSV) using Texaco gas. It has been shown in Air Products' laboratory that the activity of this sample toward isobutanol dehydration at 300°C is similar to γ-alumina. Surprisingly, little dehydration activity was detected in the LPDME run. Increasing the temperature to 280°C had little effect. One explanation for the low activity is the low water concentration under LPDME conditions, which makes the active component in the catalyst, phosphoric acid, unstable. An interesting observation from this run is that the long-term deactivation of the methanol catalyst in this system is faster than in the standard catalyst system (Fig. 3.1.3), even though the phosphate sample did not show any dehydration activity. This again indicates that the dehydration activity, or acidity, is not necessarily the cause for the long-term deactivation of the methanol catalyst. When the reactor was taken apart after the run, it was observed that the spent catalyst stuck to the surface of the reactor hardware more strongly than it did in the standard catalyst system.

Figure 3.1.3 A LPDME Life Run Using Silicon Ammonia Phosphate as the Dehydration Catalyst



Zn-Y was chosen on the basis of the migration hypothesis. If Zn migration from the methanol catalyst to the dehydration catalyst is the cause of catalyst deactivation, it is unlikely that migration will take place in a dehydration catalyst that has already been saturated with Zn, such as Zn-Y. However, this sample did not exhibit any dehydration activity. The acidity of this sample might have been too weak to carry out methanol dehydration.

3.1.1(iv) Single Catalyst System — Cu and Zn Supported on γ-Alumina

It was reported in the April-June quarterly that a single particle catalyst system consisting of Cu, ZnO supported on γ-alumina has a poorer stability under LPDME conditions than the standard dual catalyst system. That catalyst was prepared by impregnating Catapal B γ-alumina powders with zinc and copper nitrate followed by calcination in air (sample # 14656-9), in accordance with a Shell patent (US 4,375,424, 1983). To find out if the poor stability is intrinsic to the catalyst or related to the slurry phase operation, a similar catalyst (sample # 14656-34) was prepared from Catapal B γ-alumina pellets and tested in a packed bed reactor. The final Cu and Zn content based on an elemental analyst was 4.0 and 4.6 wt%, respectively. The catalyst bed was diluted with quartz chips. The reduction was carried out using 2% H_2 in N_2 at 700 psig and a ramp from room temperature to 250°C in 3 hr, mimicking the Shell procedures. Two packed bed runs (14047-38 and 14047-44) were conducted because some equipment problems were encountered in the first run. The results from these two runs are displayed in Figure3.1.4, along with the runs conducted in the autoclave using the single catalyst and the standard dual catalyst system.

Figure 3.1.4 The Stability of a Single Catalyst System

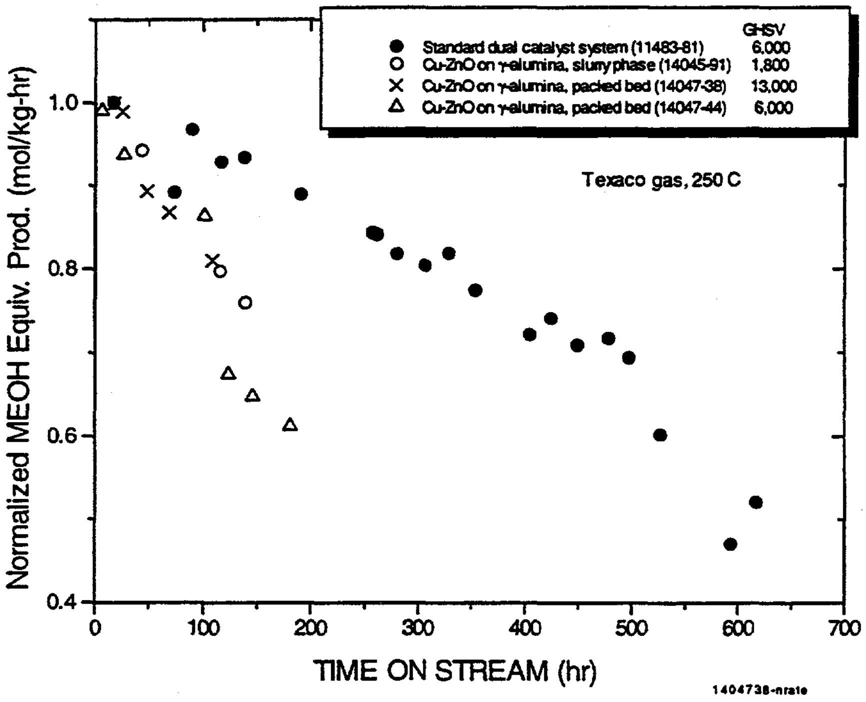


Figure 3.1.4 shows that the aging pattern of the single catalyst system in the packed bed runs is similar to that in the slurry phase run. This indicates that the poor stability is intrinsic to the catalyst sample, rather than to the slurry phase operation. In order to test the single catalyst concept, a DME catalyst that is at least stable in the gas phase operation is needed. Efforts are being made in this regard.

Several data points were collected at 270°C in order to compare the activity of the single catalyst system to that of the catalysts reported in the Shell patent. The results show that the catalyst made in the laboratory had about one-half of the activity of a corresponding sample in the patent. This may be partly due to 20% lower Cu loading and 10% Zn loading in our catalyst.

3.1.2 A Repeat Life Run of a Reported Dual Catalyst System for LPDME

According to a recent report from a Japanese laboratory, a dual catalyst system consisting of ICI 51-2 methanol catalyst and Cu-doped γ-alumina (5 wt%) powders, with hexadecane as the slurry fluid, exhibited no loss in activity under LPDME conditions for 700 hours. A close examination of the data reveals that the life run is suspect since the catalyst was subject to severe mass transfer limitations. (After our experiments were completed, the authors of the report acknowledged this problem and stated that the catalyst system may actually deactivate faster. No elaboration was offered.) We decided to examine the stability of the catalyst system in our 300 cc autoclave under the conditions free of mass transfer limitations (Run 14665-11). A Cu-doped γ-alumina sample was prepared according to the method reported in a patent by the Japanese authors. The same gas composition (50:50 H_2 to CO), catalyst ratio, solid concentration in the slurry, and slurry fluid were used. The run was conducted first at 250°C, 750 psig, and a space velocity of 1980 sl/kg-hr for 142 hr. Afterward the run was continued at 280°C, 426 psig, and 1980 sl/kg-hr for 43 hr, which are the exact conditions used in the reported life study. The activity of our version of the catalyst system was similar to that in the report, but stable activity was not observed. As shown in Fig. 3.1.5, the catalyst system deactivated under both sets of conditions. The rate of deactivation is similar to our standard catalyst system (S3-86 plus Catapal B γ-alumina). These results suggest that the LPDME run in the Japanese laboratory may also have had a stability problem, unless certain factors were not disclosed in their report and patent.

3.1.3 SEM/EDS Analysis of LPDME Catalysts — Examination of the Migration Hypothesis

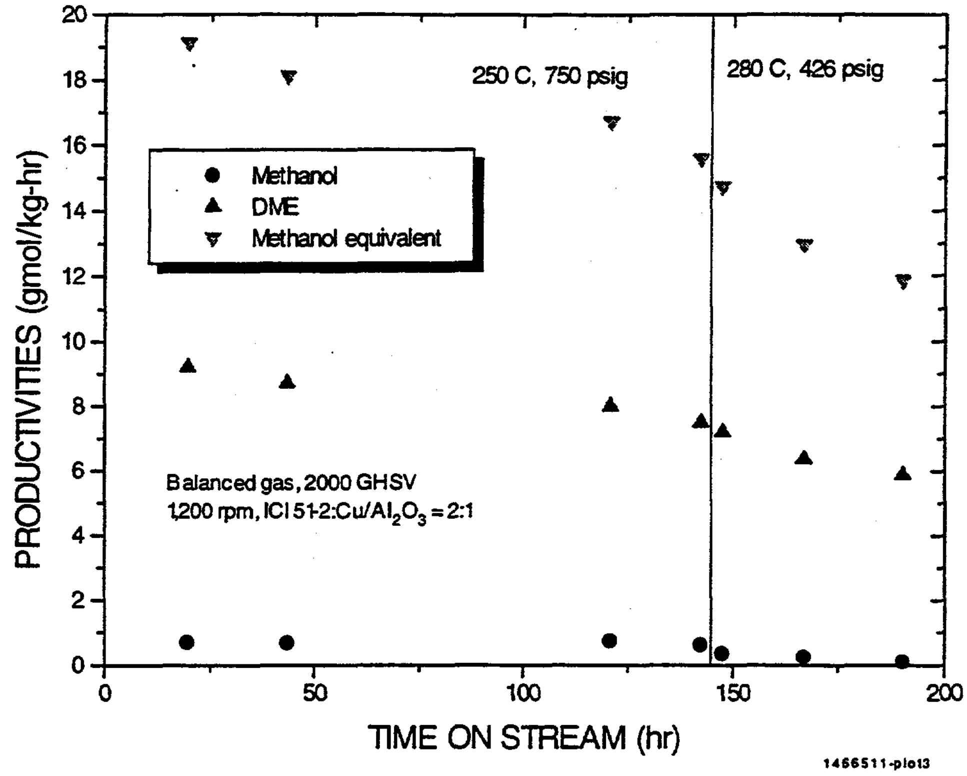
Summary

This section summarizes the analytical results of methanol catalyst and alumina mixtures using SEM and EDS. The analysis was performed to confirm the hypothesis that the deactivation of the LPDME catalyst system is caused by inter-catalyst migration of one or more types of atoms. The questions to be answered by this analysis are: Does the migration take place? And if it does, is the migration correlated to the catalyst deactivation?

Zinc and copper were detected on alumina particles in the spent catalyst mixtures, but not on virgin alumina, and the ratio of copper to zinc was significantly different from that found for the methanol catalyst itself. This indicates that migration occurs, at least in some cases. Furthermore, no migration of zinc or copper was found in the R-M???? experiment in which the catalyst did not deactivate rapidly. Unfortunately, the zinc and copper content of alumina particles in catalyst mixtures of different histories does not correlate with the activity of the catalyst systems. Several explanations for this behavior have been proposed:

1) methanol catalyst fines may exist in the alumina particles and thus, confound the analysis;
2) migration is only responsible for the initial deactivation of the catalyst system; or
3) the sampling may not be statistically sound.

Figure 3.1.5 The Stability of a Reported LPDME Catalyst System Tested in Air Products' Laboratory



This lack of correlation is not sufficient to reject the migration of zinc and/or copper as the primary cause of catalyst deactivation. Work on understanding the mechanism of migration continues.

Introduction

We reported previously that an interaction between a methanol synthesis catalyst (e.g., BASF S3-86 Cu-ZnO-based low pressure methanol catalyst) and a dehydration catalyst (e.g., γ-alumina) in the standard dual catalyst system is responsible for the deactivation of both catalysts under LPDME conditions. Several observations have suggested that this interaction could be due to *inter-catalyst migration of zinc and copper*, that is, the zinc- or/and copper-containing species from the methanol catalyst migrate onto the surface of the dehydration catalyst during reduction and/or under the reaction conditions. By this mechanism, the methanol catalyst deactivates because it loses its active components. The dehydration catalyst is poisoned by these migrating species by reacting with the acid sites. Possible driving forces for the inter-catalyst migration are:

1) *acid-base reactions* - solid state reactions such as acid-base reactions between ZnO in the methanol catalyst and the acid sites on the dehydration catalyst,
2) *ion exchange* - solid-state exchange of copper/zinc ions from the methanol catalyst with the protons (i.e., Bronsted acid sites) on the dehydration catalyst,

3) *spontaneous dispersion* - migration simply driven by the concentration gradient of copper and zinc between the two catalysts, considering that γ-alumina, and most other types of dehydration catalysts, for that matter, have good dispersing capability toward metal, metal oxide, and salts.

These processes must take place where two catalysts contact each other, that is, at the outer surface of alumina particles. The migration may or may not continue into the inner surface of the alumina particles. However, the extent of the deactivation we have seen suggests that the inner surface of the alumina should be affected, unless the alumina particles break apart considerably under the reaction conditions. Efforts have been made to confirm the hypothesis by analyzing the copper and zinc content in the alumina in spent catalyst mixtures. This report summarizes the results based on the analysis using SEM and EDS.

Method of Analysis

SEM and EDS were used because the different types of catalyst particles are inseparable. The catalyst system under study consists of a powder mixture of the two catalysts. The mean diameter of fresh methanol catalyst and alumina powders is around 60 microns. The particle size in the spent catalyst mixture is smaller because of the attrition in the reactor. This makes it virtually impossible to separate the alumina from the methanol catalyst and use it for conventional elemental analysis. SEM enables us to pick out individual alumina or methanol catalyst particles, while EDS provides elemental information on these particles. The smallest features that can be "seen" by SEM are 0.1 to 1 micron. The spatial resolution of EDS of 1 micron, and the lower detecting limit of EDS is believed to be 0.5 wt.%.

We looked at both the outer surface and the cross section of alumina particles. Inter-catalyst migration will initiate on the outer surface due to the contact of the two catalysts. Figure 3.1.6 is a SEM micrograph of the outer surface of an alumina particle in a spent catalyst mixture (13458-8). The bright spots are methanol catalyst powders, judging by EDS analysis and the brightness of these powders. (Heavier elements like Cu and Zn have a brighter image in SEM than lighter elements like Al.) These powders are physically attached to the alumina, a phenomenon that can be inferred since treating the sample with an organic removes most of the metal particles through dispersion. As shown in Figure 3.1.7, the outer surface of an alumina particle in a catalyst mixture dispersed using an organic solvent is much cleaner, although some methanol catalyst particles can still be seen. In both non-dispersed (or dry) and dispersed samples, "clean" areas where no methanol catalyst particles are found exist as illustrated in Figures 3.1.6 and 3.1.7. The "clean" area was assumed to be free of physically attached methanol catalyst particles, and used for the analysis.

We also looked at the cross section of the alumina particles as a way to circumvent the physical attachment problem. Moreover, this indicates if the migration into the inner surface of alumina particles has occurred. The cross section was prepared by freezing the sample in epoxy and cutting it to expose the cross section. Precautions were taken to make sure that copper and zinc from the methanol catalyst were not smeared onto the alumina cross section by the cutting. This was checked by EDS examination of the epoxy around the sample particles. Little copper and zinc were detected.

Figure 3.1.6 SEM Micrograph of an Alumina Particle in a Spent Catalyst Mixture.

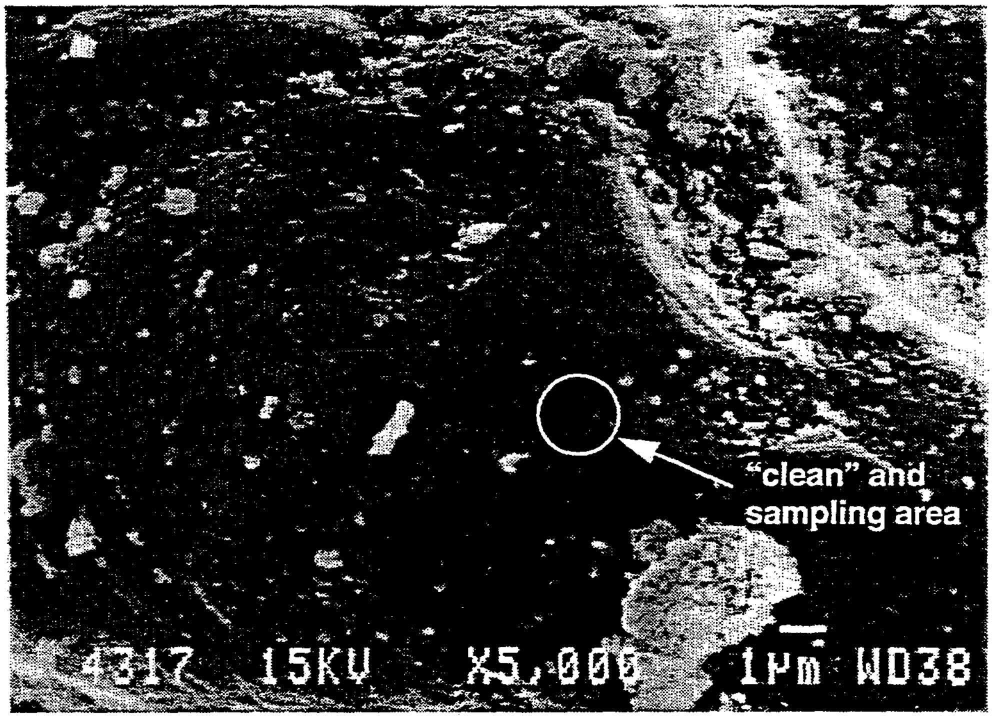
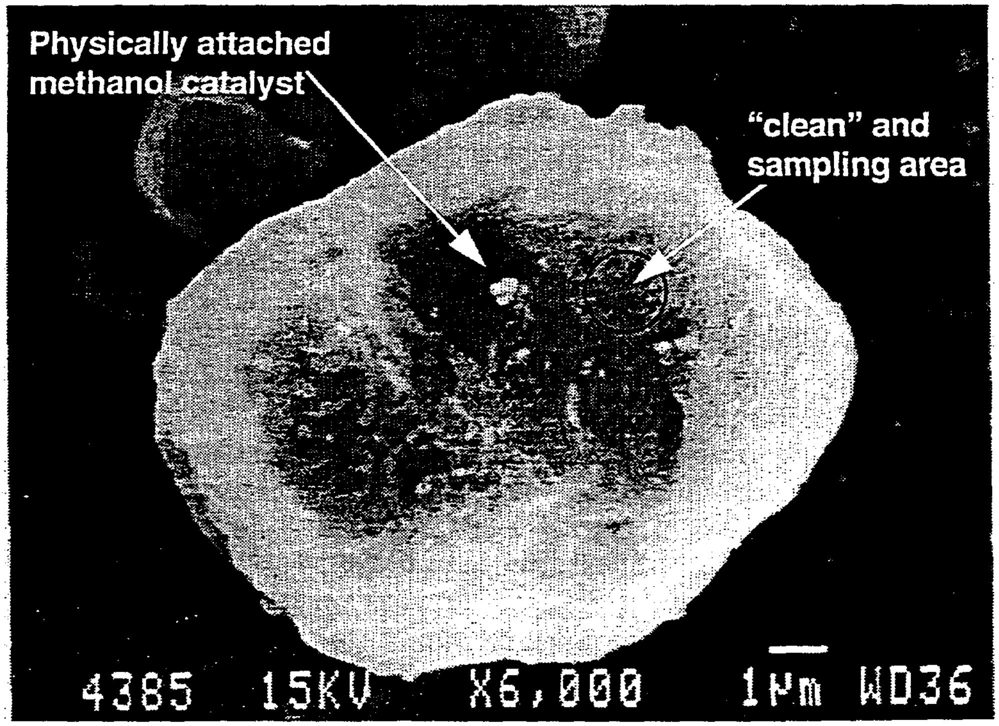


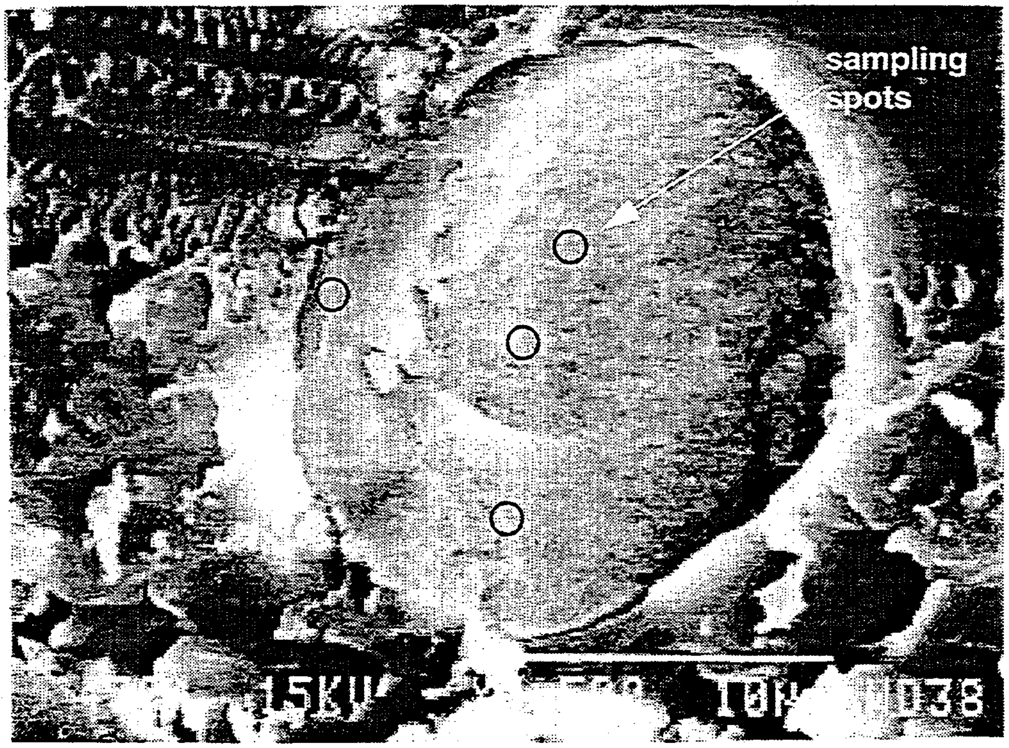
Figure 3.1.7 SEM Micrograph of an Alumina Particle in a Dispersed Spent Catalyst Mixture



sem1.ppt

Figure 3.1.8 illustrates how a typical analysis was conducted. An SEM micrograph of an alumina particle, a cross section in this case, was generated first. Then the electron beam was *parked* at different spots in this cross section, and an EDS elemental analysis was performed. The sampling volume in this mode of analysis was an order of magnitude smaller than the dimension of alumina particles. Alternatively, one could *raster* the beam across the sample to obtain integrated elemental information.

Figure 3.1.8 SEM Micrograph of the Cross Section of an Alumina Particle in a Spent Catalyst Mixture



The elemental analysis by EDS is based on the energy dispersion of the X-ray generated by the same electron beam that is used for SEM. Figure 3.1.9a illustrates how different elements in a methanol catalyst sample appear on an EDS spectrum. (The aluminum section of the spectrum is not shown.) The net peak area was used for quantitative analysis. The integration was conducted using graphics software called Origin, since the data processing software used by the EDS instrument tended to draw erroneous baselines, especially when the signal was weak. To make sure the quantitation was independent of analytical conditions, such as the size of sample particles and the electron beam, gold coating, and parking mode vs. rastering mode, we looked at methanol catalyst particles of different sizes, with and without gold coating (gold coating was used to remove the static charging on the sample), and of different origins. The particles included those in a pure, freshly reduced methanol catalyst sample (13458-90), in a spent catalyst mixture (13458-8), and those physically attached to the outer surface of alumina particles in the same spent catalyst mixture. As shown by the solid dots in Figure 3.1.10, the Zn-to-Cu ratio from these different samples falls into a narrow range around 0.29, indicating that the quantitation is insensitive to the analytical conditions.

Figure 3.1.9 EDS Spectra of Different Samples

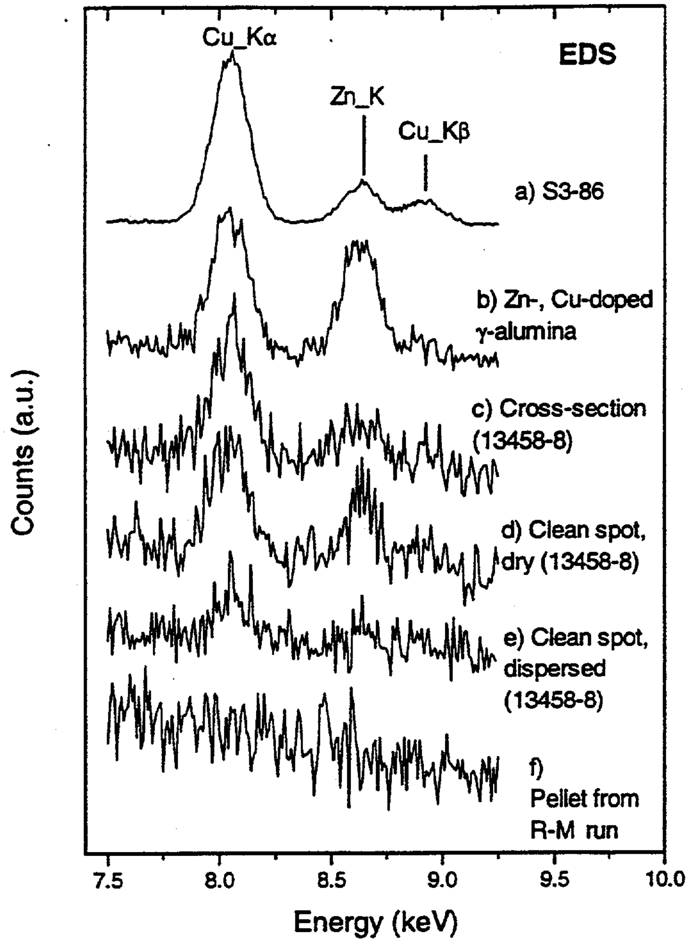
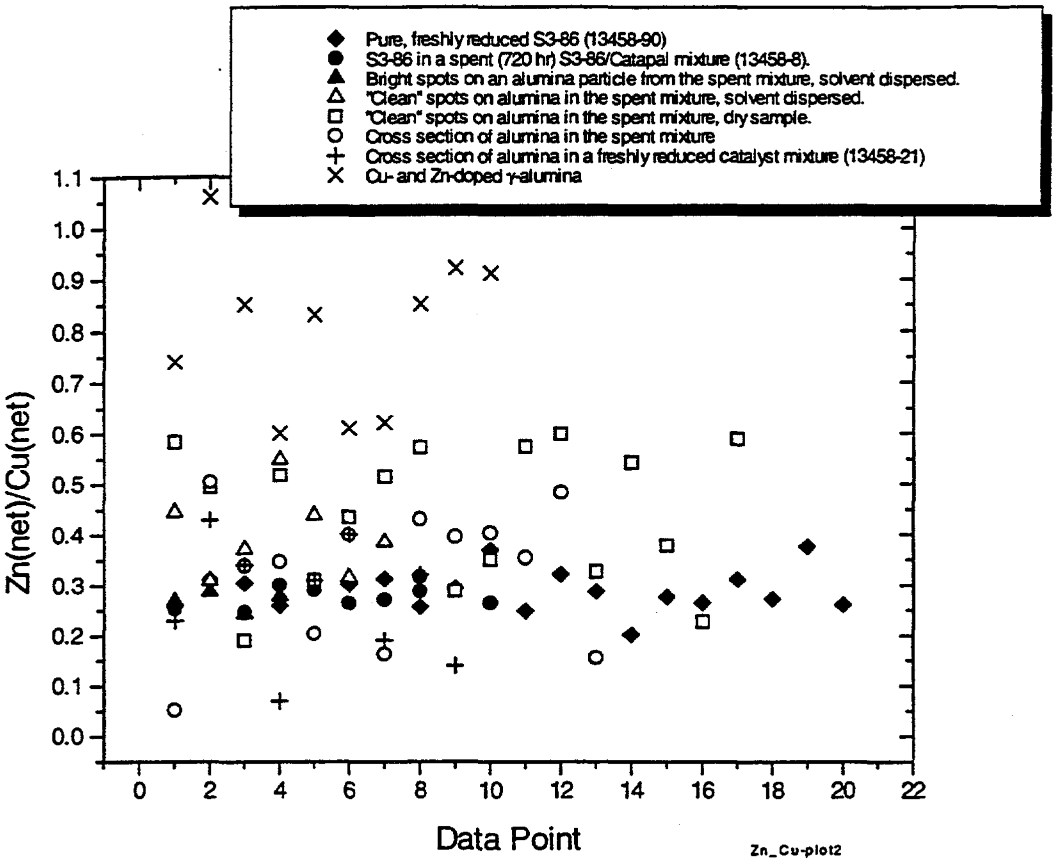


Figure 3.1.10 Zn-to-Cu EDS Peak Ratio of Different Samples



Since the absolute quantitation was difficult to measure, we relied on relative quantity of different elements for our analysis. These included ratios of (Zn+Cu) to Al and Zn to Cu. The (Zn+Cu) to Al ratio is intended as a measure of how much zinc and copper migrate onto the alumina, while the Zn to Cu ratio serves as a way to distinguish migrated zinc and copper from that in the physically attached methanol catalyst particles. We assume that zinc and copper deposition on the alumina due to the migration is unlikely to have the same ratio as in the methanol catalyst.

Results and Discussion

All EDS results, that is, the net peak area of Cu, Zn, and Al plus their ratios, are summarized in Table 3.1.1. Figure 3.1.9 displays typical EDS spectra from different samples. The Zn/Cu and (Zn+Cu)/Al ratios from different samples are shown in Figures 3.1.10 and 3.1.11. The three main samples discussed below are the alumina in a catalyst mixture that was on stream for 710 hours (13458-8, *the spent sample*), the alumina in a freshly reduced catalyst mixture (13458-21, *the freshly reduced sample*), and the alumina in a catalyst mixture used in a catalyst compatibility study (14045-26, *the compatibility sample*). The relative activities of these three catalyst systems, in terms of the methanol equivalent productivity, are 0.6, 1, and 0.5, respectively. The analytical results from these samples do not show a consistent picture. The data that appear to support the migration theory will be examined first.

Table 3.1-1: Summary of EDS Analysis Results

Spectrum	Sampled particle	Region	Description	Beam mode	net Cu_Ka	net Zn_K	net Al_K	Zn_K/Cu_K	(Cu_K+Zn_K)/Al_K
1. Spent pure methanol catalyst. On syngas stream for 30 hr, 250 C, 750 psig, Texaco gas. Run NB#: 13458-90.									
J959001	S3-86	1	individual particle	parking	233.47	60.4	30.85	0.26	9.526
J959002	S3-86	2	individual particle	parking	237.18	73.79	32.28	0.31	9.634
J959003	S3-86	3	individual particle	parking	212.63	64.76	37.94	0.30	7.311
J959004	S3-86	4	individual particle	parking	267.22	69.29	31.19	0.26	10.789
J959005	S3-86	5	individual particle	parking	242.14	74.78	41.64	0.31	7.611
J959006	S3-86	6	individual particle	parking	226.88	68.51	35.06	0.30	8.425
J959007	S3-86	7	individual particle	parking	217.82	67.92	49.19	0.31	5.809
J959008	S3-86	8	individual particle	parking	262.87	67.34	26.11	0.26	12.647
J959009	S3-86	9	individual particle	parking	242.56	71.16	30.91	0.29	10.149
J959010	S3-86	10	individual particle	parking	189.82	69.96	109.24	0.37	2.378
J959011	S3-86	11	individual particle	parking	242.91	60.18	34.8	0.25	8.709
J959012	S3-86	12	individual particle	parking	216.56	69.57	115.05	0.32	2.487
J959013	S3-86	13	individual particle	parking	234.61	67.22	64.22	0.29	4.700
J959014	S3-86	14	individual particle	parking	276.92	55.79	25.38	0.20	13.109
J959015	S3-86	15	individual particle	parking	261.17	71.98	36.04	0.28	9.244
J959016	S3-86	16	individual particle	parking	262.23	69.14	33.92	0.26	9.769
J959017	S3-86	17	individual particle	parking	232.69	72.38	54.52	0.31	5.596
J959018	S3-86	18	individual particle	parking	269.95	73.31	15.98	0.27	21.481
J959019	S3-86	19	individual particle	parking	191.81	71.92	147.92	0.37	1.783
J959020	S3-86	20	individual particle	parking	254.24	66.16	65.68	0.26	4.878
2. Catalyst mixture on syngas stream for 710 hours (250 C, 750 psig, H2-rich gas). Run NB#: 13458-8.									
j950804	alumina	4	dry, clean spot	parking	3.82	2.23	1046.73	0.58	0.006
j950805	alumina	5	dry, clean spot	parking	4.21	2.08	1053.04	0.49	0.006
j950806	alumina	6	dry, clean spot	parking	4.47	0.85	1203.49	0.19	0.004
j950807	alumina	7	dry, clean spot	parking	4.78	2.47	1093.26	0.52	0.007
j950808	alumina	8	dry, clean spot	parking	5.68	1.76	972.25	0.31	0.008
j950809	alumina	9	dry, clean spot	parking	9.88	4.29	719.93	0.43	0.020

Table 3.1-1: Summary of EDS Analysis Results (cont.)

Spectrum	Sampled particle	Region	Description	Beam mode	net Cu_Ka	net Zn_K	net Al_K	Zn_K/Cu_K	(Cu_K+Zn_K)/Al_K
j950810	alumina	10	dry, clean spot	parking	7.01	3.6	1037.1	0.51	0.010
j950811	alumina	11	dry, clean spot	parking	9.77	5.58	972.61	0.57	0.016
j950812	alumina	12	dry, clean spot	parking	9.4	5.53	947.93	0.59	0.016
j950813	alumina	13	dry, clean spot	parking	4.75	2.72	983.44	0.57	0.008
j950814	alumina	14	dry, clean spot	parking	5.42	3.24	1029.06	0.60	0.008
j950815	alumina	15	dry, clean spot	parking	9.38	5.08	584.03	0.54	0.025
j950816	alumina	16	dry, clean spot	parking	5.54	2.09	1068.74	0.38	0.007
j950817	alumina	17	dry, clean spot	parking	7.1	1.61	1142.79	0.23	0.008
j950818	alumina	18	dry, clean spot	parking	8.62	2.81	1188.99	0.33	0.010
j950819	alumina	19	dry, clean spot	parking	9.35	2.69	1118.31	0.29	0.011
j950820	alumina	20	dry, clean spot	parking	13.71	4.79	839.51	0.35	0.022
d0801b	alumina	1	dispersed, clean spot	parking	1.64	0.73	682.36	0.45	0.003
d0802b	alumina	2	dispersed, clean spot	parking	n.d.	n.d.	960.9		
d0804b	alumina	4	dispersed, clean spot	parking	n.d.	n.d.	1084.2		
d0805b	alumina	5	dispersed, clean spot	parking	2.42	0.75	972.97	0.31	0.003
d0806b	alumina	6	dispersed, clean spot	parking	n.d.	n.d.	1043.1		
d0807b	alumina	7	dispersed, clean spot	parking	3.41	1.87	999.15	0.55	0.005
d0808b	alumina	8	dispersed, clean spot	parking	little	little	982		
d0809b	alumina	9	dispersed, clean spot	parking	3.67	1.38	880.64	0.38	0.006
d0810b	alumina	10	dispersed, clean spot	parking	little	little	996.7		
d0811b	alumina	11	dispersed, clean spot	parking	little	little	996.7		
d0812b	alumina	12	dispersed, clean spot	parking	little	little	996.7		
d0813b	alumina	13	dispersed, clean spot	parking	n.d.	n.d.	1727.9		
d0814b	alumina	14	dispersed, clean spot	parking	2.83	1.24	1721.33	0.44	0.002
d0815b	alumina	15	dispersed, clean spot?	parking	8.47	2.67	1467.21	0.32	0.008
d0816b	alumina	16	dispersed, clean spot	parking	1.38	0.53	1481.3	0.38	0.001
d0817b	alumina	17	dispersed, clean spot	parking	n.d.	n.d.	1417.5		

Table 3.1-1: Summary of EDS Analysis Results (cont.)

Spectrum	Sampled particle	Region	Description	Beam mode	net Cu_Ka	net Zn_K	net Al_K	Zn_K/Cu_K	(Cu_K+Zn_K)/Al_K
d0818b	alumina	18	dispersed, clean spot	parking	n.d.	n.d.	1152.5		
d0819b	alumina	19	dispersed, clean spot	parking	n.d.	n.d.	1000.1		
d0820b	alumina	20	dispersed, clean spot	parking	n.d.	n.d.	1329.1		
d0803b	alumina	3	dispersed, bright spot	parking	160.84	46.24	193	0.29	1.073
d0821b	alumina	21	dispersed, bright spot	parking	219.57	58.95	100.81	0.27	2.763
d0822b	alumina	22	dispersed, bright spot	parking	223.47	54.32	75.68	0.24	3.671
d0823b	alumina	23	dispersed, bright spot	parking	204.59	56.83	70.89	0.28	3.688
x0801b	alumina	1	cross section, near center	parking	10.03	0.52	1991.62	0.05	0.005
x0802b	alumina	2	cross section, near center	parking	6.12	2.07	1958.28	0.34	0.004
x0803b	alumina	3	cross section, near center	parking	6.35	2.2	1939.4	0.35	0.004
x0804b	alumina	4	cross section, inclusion	parking	7.22	1.47	2399.42	0.20	0.004
x0805b	alumina	5	cross section, within section	parking	4.74	2.39	2100.33	0.50	0.003
x0806b	alumina	6	cross section, near edge	parking	10.6	4.23	2254.42	0.40	0.007
x0807b	alumina	7	cross section, near center	parking	4.6	0.71	174.13	0.15	0.030
x0808b	alumina	8	cross section, within section	parking	3.2	0.52	1137.66	0.16	0.003
x0809b	alumina	9	cross section, near edge	parking	2.35	1.01	1209.88	0.43	0.003
x0810b	alumina	10	cross section, near center	parking	2.38	0.94	1282.14	0.39	0.003
x0811b	alumina	11	cross section, near edge	parking	9.32	3.75	826.58	0.40	0.016
x0812b	alumina	12	cross section, within section	parking	3.23	1.14	1123.36	0.35	0.004
x0813b	alumina	13	cross section, near center	parking	1.91	0.92	1031.17	0.48	0.003
xr0801	alumina	1	individual cross section	rastering	7.29	0.92	1806.7	0.13	0.005
xr0802	alumina	2	individual cross section	rastering	9.46	3.15	1127.2	0.33	0.011
xr0803	alumina	3	individual cross section	rastering	9.75	2.02	1771.6	0.21	0.007
xr08e	epoxy	4	epoxy	rastering	n.d.	little	little		

Table 3.1-1: Summary of EDS Analysis Results (cont.)

Spectrum	Sampled particle	Region	Description	Beam mode	net Cu_Ka	net Zn_K	net Al_K	Zn_K/Cu_K	(Cu_K+Zn_K)/Al_K
3. Freshly reduced catalyst mixture. Run NB#: 13458-21.									
x2101	alumina	1	cross section region1	parking	5.15	1.2	2241.3	0.23	0.003
x2102	alumina	2	cross section region 2	parking	2.54	1.09	1341.8	0.43	0.003
x2103	alumina	3	cross section region 3	parking	11	3.72	1380.7	0.34	0.011
x2104	alumina	4	cross section region4	parking	0.59	0.04	1322.9	0.07	0.001
x2105	alumina	5	cross section region5	parking	13.61	4.22	1394.9	0.31	0.013
x2106	alumina	6	cross section region 6	parking	7.67	3.09	1519	0.40	0.007
xr2101	alumina	1	individual cross section	rastering	6.355	1.23	1721.3	0.19	0.004
xr2102	alumina	2	individual cross section	rastering	39.05	12.47	1822.4	0.32	0.028
xr2103	alumina	3	individual cross section	rastering	8.58	1.18	1097	0.14	0.009
xr21e	epoxy	4	epoxy area	rastering	n.d.	n.d.	n.d.		
d2101	alumina	1	dispersed, clean spot	packing	n.d.	n.d.	1395.4		
d2102	alumina	2	dispersed, clean spot	packing	n.d.	n.d.	2134.4		
d2103	alumina	3	dispersed, clean spot	packing	n.d.	n.d.	1868.2		
d2104	alumina	4	dispersed, clean spot	packing	n.d.	n.d.	1493.9		
d2105	alumina	5	dispersed, clean spot	packing	n.d.	n.d.	1721.8		
4. Catalyst mixture used in the catalyst compatibility experiment. Under 2% H2 for 120 hr, 250 C, 750 psig. Run NB#: 14045-26.									
x2601	alumina	1	cross section region1	parking	9.33	3.55	1728.3	0.38	0.007
x2602	alumina	2	cross section region 2	parking	6.49	1.77	1819.2	0.27	0.005
x2603	alumina	3	cross section region 3	parking	6.64	1.77	1867.1	0.27	0.005
x2604	alumina	4	cross section region4	parking	4.37	1.03	1592.3	0.24	0.003
x2605	alumina	5	cross section region5	parking	7.71	1.82	783.8	0.24	0.012
x2606	alumina	6	cross section region 6	parking	3.49	1.11	1537.9	0.32	0.003

Table 3.1-1: Summary of EDS Analysis Results (cont.)

Spectrum	Sampled particle	Region	Description	Beam mode	net Cu_Ka	net Zn_K	net Al_K	Zn_K/Cu_K	(Cu_K+Zn_K)/Al_K
5. Alumina pellets used in the run with R-M basket internals. On stream 503 hr, 250 C 750 psig, Shell gas. Run NB#: 14045-43									
p4301g	alumina	1	outer surface, grey particle	parking	little	little			
p4301w	alumina	1	outer surface, white particle	parking	little	little			
p4302g	alumina	2	outer surface, grey particle	parking	0	0			
p4302w	alumina	2	outer surface, white particle	parking	0	0			
p4303g	alumina	3	outer surface, grey particle	parking	0	0			
p4303w	alumina	3	outer surface, white particle	parking	0	0			
px4301g	alumina	1	cross section, grey particle	parking	0	0			
px4301w	alumina	1	cross section, white particle	parking	3.01	1.81	1315.6	0.60	0.004
px4302g	alumina	2	cross section, grey particle	parking	0	0			
px4302w	alumina	2	cross section, white particle	parking	0	0			
px4303g	alumina	3	cross section, grey particle	parking	0	0			
px4303w	alumina	3	cross section, white particle	parking	0	0			
px4304g	alumina	4	cross section, grey particle	parking	4.94	1.39	1019.6	0.28	0.006
px4304w	alumina	4	cross section, white particle	parking	0	0			
px4305g	alumina	5	cross section, grey particle	parking	0	0			
px4305w	alumina	5	cross section, white particle	parking	0	0			
px4306g	alumina	6	cross section, grey particle	parking	0	0			
px4306w	alumina	6	cross section, white particle	parking	0	0			
6. Cu and Zn-doped gama-alumina (Sample NB# 14656-34, 3.95 wt% Cu, 4.55 wt% Zn)									
x3401		1	cross section	parking	9.94	7.37	646.29	0.74	0.027
x3402		2	cross section	parking	22.97	24.38	1395.28	1.06	0.034
x3403		3	cross section	parking	23.59	20.01	1140.02	0.85	0.038
x3404		4	cross section	parking	17.08	10.25	676.88	0.60	0.040
x3405		5	cross section	parking	9.65	8.02	451.21	0.83	0.039
x3406		6	cross section	parking	16.17	9.83	557.89	0.61	0.047

Table 3.1-1: Summary of EDS Analysis Results (cont.)

Spectrum	Sampled particle	Region	Description	Beam mode	net Cu_Ka	net Zn_K	net Al_K	Zn_K/Cu_K	(Cu_K+Zn_K)/Al_K
x3407		1	cross section	parking	11.22	7.01	421.39	0.62	0.043
x3408		2	cross section	parking	20.42	17.34	890.79	0.85	0.042
x3409		3	cross section	parking	30.73	28.18	1382.98	0.92	0.043
x3410		4	cross section	parking	29.14	26.44	1934.22	0.91	0.029

little: Barely above the noise level.
n.d.: Not detectable.

Sample 13458-8 is a catalyst mixture that was on stream for 710 hr in a LPDME run. The methanol equivalent productivity dropped by 39% at the end of the run. Both the cross section and the "clean" area on the outer surface of the alumina particles in this spent mixture were analyzed with EDS. Zinc and copper were detected from all spots on the cross section and on the "clean" area of the outer surface of alumina in a *dry* sample. On the "clean" area of the outer surface of alumina in a *dispersed* sample, zinc and copper were detected on more than half of the spots (Figure 3.1.9 and Table 3.1.1).

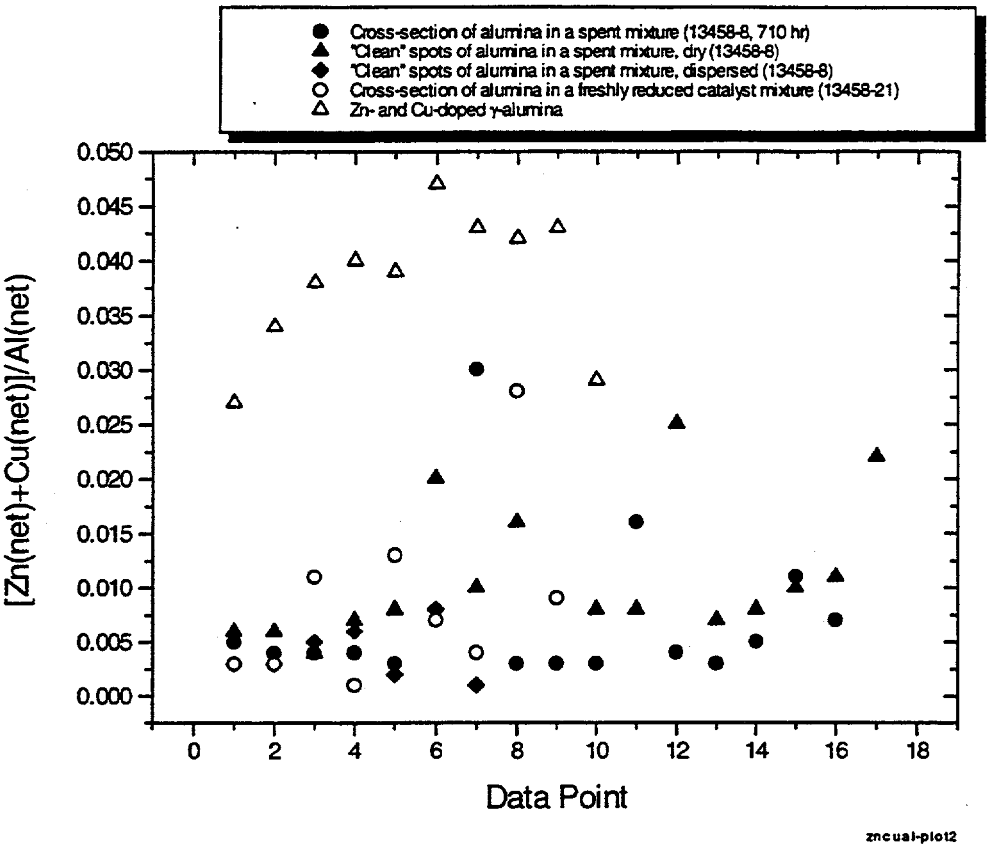
The method used to distinguish the zinc and copper by migration from that in the methanol catalyst fines is to examine the Zn-to-Cu ratio because it is very unlikely that zinc and copper from the migration will have the same stoichiometry as that in the methanol catalyst. The open dots in Figure 3.1.10 show the Zn-to-Cu ratio from the spent sample. The data are very scattered compared to those from the methanol catalyst. However, they are not scattered evenly around those from the methanol catalyst, but, instead most of the data points lie above. The number average is 0.38 compared to 0.29 from the methanol catalyst. This suggests that the Zn and Cu detected from the spent sample derive from the migration of zinc- and copper-containing species during the run.

Other information supporting the migration theory is obtained from the alumina pellets used in the LPDME employing Robinson-Mahoney basket internals and a mixture of catalyst pellets (14045-43). The methanol catalyst did not deactivate after 508 hr on stream in that run. The outer surface and the cross section of the alumina pellets from the run were analyzed by EDS. As shown in Table 3.1.1, Zn and Cu were not detected in most spots (16 of 18) examined. A typical EDS spectrum from this sample is shown in Figure 3.1.9f. A traditional elemental analysis using chemical digestion and atomic absorption also showed no zinc and copper buildup on the alumina. This indicates indirectly that the deactivation is related to zinc and copper migration.

Other results were puzzling. According to the migration hypothesis, the catalyst deactivation should be proportional to the extent of zinc or/and copper migration. It follows that the zinc and copper content in the freshly reduced sample (with a relative activity of 1) should be much lower than that in the spent sample (with a relative activity of 0.6). Therefore, experiments were conducted to measure the zinc and copper contents of the freshly reduced sample and compare them to that in the spent sample.

The solid dots in Figure 3.1.11 show the (Zn+Cu)/Al ratio from the cross section and "clean" area of the spent sample. The data are very scattered, ranging from 0.001 to 0.03, with a number average of 0.008. The three data points collected by rastering the electron beam across the cross-section of three alumina particles do not show better consistency, ranging from 0.005 to 0.01 (Table 3.1.1). No concentration gradient across alumina particles can be detected (Table 3.1.1). To estimate the concentration of zinc and copper in the alumina, a calibration sample was prepared by impregnating γ-alumina with 4.55 wt % zinc and 3.95 wt % of copper. The EDS results from this sample are shown in Figures 3.1.9b, 3.1.10 and 3.1.11. The (Zn+Cu)/Al ratios from this sample, as shown in Figure 3.1.11, are also scattered, ranging from 0.027 to 0.047 with a number average of 0.038. Based on this standard, the concentration of zinc plus copper in the spent sample ranges from 0.2 to 6.7 wt %, with an average of 1.8 wt %.

Figure 3.1.11 (Cu+Zn)-to-Al EDS Ratios of Different Samples



The open circles in Figure 3.1.11 are the (Zn+Cu)/Al ratios measured from the cross section of the freshly reduced sample. Zinc and copper were detected on all spots examined with EDS (Table 3.1.1). Again the data are scattered, but the range they cover is similar to that from the spent sample, with an average of 0.009. The zinc and copper contents in these two samples are similar to each other, although their activity is different by 40%. Further confusion comes from the Zn-to-Cu ratio measured from the freshly reduced sample. As shown in Figure 3.1.10, the data, with an average ratio of 0.27, are scattered around those from the methanol catalyst, with an average of 0.29, not above them as seen with the spent sample.

Also examined was the cross section of alumina particles in the catalyst mixture used in the catalyst compatibility experiment (14045-46). In this experiment, the methanol catalyst and alumina were held together under 2% H_2 in N_2 and the reaction temperature (250°C) and pressure (750 psig) for 120 hours. The activity of the catalyst system, in terms of methanol productivity, dropped by 50%. Zinc and copper were detected from all spots examined on the cross section. As shown in Table 3.1.1, the average (Zn+Cu)/Al ratio from this sample is 0.006. This ratio is lower than that from the spent sample (0.008), although the spent catalyst system shows lower deactivation. The Zn-to-Cu ratio from the sample used in the compatibility study is 0.25 on average, again close to, but not greater than, that from the methanol catalyst (0.29).

It is certain from all of these analyses that zinc and copper are detected in the alumina particles in the catalyst mixture samples. However, it is not certain where they originate and how they are related to the catalyst deactivation. The detected zinc and copper in the cross sections could arise from several possible causes:

1) Migration from the methanol catalyst
2) Methanol catalyst fines smeared over the cross section by the cutting
3) Methanol catalyst fines that are small enough to enter the defects and even the pores (with a mean diameter of 60 angstroms) of the alumina particles.

The first possibility is the working hypothesis. The second possibility, that of smear-over, is very unlikely because little zinc and copper were detected on the epoxy around the alumina cross sections; therefore smear-over was ignored. The third possibility is important because if it occurs, adhering methanol catalyst particles will interfere with or may overshadow analysis of the presence of any copper and/or zinc that has migrated. Thus, when the Zn-to-Cu ratio is near that of the methanol catalyst, a definitive conclusion about migration cannot be made.

The best way to determine the third possibility is to examine the morphology of the zinc and copper inside the alumina particle (i.e., the cross section) using other techniques such as high resolution Transmission Electron Microscopy (TEM). This technique allows us to distinguish the presence of fines from atomically dispersed zinc and copper. The atomically dispersed zinc and copper arise most likely from the migrated species. If the methanol catalyst fines work their way into alumina particles, it would be impossible to verify the existence of migrated zinc and copper by elemental analysis. Nevertheless even if the third possibility were proven, it is strong evidence of intimate contact between the methanol catalyst and alumina which would allow the possibility of migration. Experiments have been designed to examine this issue using TEM.

The results discussed above demonstrate that catalyst deactivation is not correlated with (Zn+Cu)/Al ratio. Note, however, that if the zinc and copper detected derive partially or mainly from the methanol catalyst fines in the alumina, we would not expect such a correlation. *If the presence of copper and zinc arises only from the migration,* there could be two explanations for the lack of correlation. First, the migration may be responsible only for the initial deactivation of the catalyst system. It has been shown that deactivation during reduction contributes substantially to the initial catalyst deactivation. Therefore, once a catalyst mixture passes through the reduction stage, the zinc and copper level in the alumina will be similar. Second, the migration did occur, but it does not account for the deactivation of both catalysts. In other words, the migration hypothesis is not correct.

Finally, the quality of the EDS data was examined. As shown in Figures 3.1.10 and 3.1.11, both Zn/Cu and (Zn+Cu)/Al ratios from the alumina samples are very scattered. Is this due to the inhomogeneous nature of the samples, or to the noise level of the EDS spectra (see Fig. 3.1.9a-e)? The inhomogeneous nature of the samples may be the more likely reason. Figures 3.1.10 and 3.1.12 show that similar scattering is observed from the Zn- and Cu-doped alumina sample, while the signal-to-noise ratio in its EDS spectrum is satisfactorily low (Fig. 3.1.9b).

APPENDIX F - TASK 1.5.4 - GROUNDBREAKING CEREMONY

Air Products and Chemicals, Inc.
7201 Hamilton Boulevard
Allentown, PA 18195-1501
Telephone (610) 481-8000

H. A. Wagner
Chairman, President and
Chief Executive Officer

AIR PRODUCTS

19 October 1995

Mr. G. Alan Edwards
Principal Deputy Assistant Secretary
for Fossil Energy
U.S. Department of Energy
Washington, DC 20585

Dear Alan:

Your presence at Monday's groundbreaking ceremonies in Kingsport made the day a very memorable one for all of us. We've been most appreciative of the DOE's support of the innovative liquid phase methanol technology over the past 14 years. Without that support, this project simply would not have moved forward.

We are very proud to be partners with the DOE in developing innovative ways to use our nation's vast coal reserves to lessen our dependence on foreign oil imports and meet our environmental objectives. I am confident that when this demonstration project has been completed, we will have been successful on both counts.

Warmest regards,

HAWagner

H. A. Wagner

bcc: Greta Campbell
Bill Brown
Barry Halper

Air Products and Chemicals, Inc.
7201 Hamilton Boulevard
Allentown, PA 18195-1501
Telephone (610) 481-8000

H. A. Wagner
Chairman, President and
Chief Executive Officer

AIR PRODUCTS

cc'd RMKornosky at PETC 11/2/95 ✓
(Fax)

19 October 1995

Mr. Thomas M. Torkos
Deputy Director
Pittsburgh Energy Technology Center
Office of Fossil Energy
U.S. Department of Energy
P.O. Box 10940
Pittsburgh, PA 15236-0940

Dear Tom:

It was a pleasure to meet you at the groundbreaking ceremonies at Kingsport on Monday. As a long-time supporter of the liquid phase methanol technology, I'm sure you are as proud as we are to see this project move from the pilot plant stage to commercial scale.

My personal thanks to you and the PETC office for your perseverance over the years. I firmly believe that your efforts ultimately will greatly benefit our nation.

Warmest regards,

HAWagner
H. A. Wagner

Air Products and Chemicals, Inc.
7201 Hamilton Boulevard
Allentown, PA 18195-1501
Telephone (610) 481-8000

H. A. Wagner
Chairman, President and
Chief Executive Officer

AIR PRODUCTS

20 October 1995

The Honorable James H. Quillen
U.S. House of Representatives
102 Cannon House Office Building
Washington, DC 20515-1101

Dear Congressman Quillen:

We were greatly honored by your presence at the groundbreaking ceremonies for our liquid phase methanol demonstration facility in Kingsport last Monday. It was a particularly memorable day for us, since you had joined Dex Baker 14 years ago at the same site when we broke ground for an industrial gas facility for Eastman's pioneering chemicals-from-coal project.

I'm sure you share our pride in seeing this coal gasification enhancement project move from the pilot plant stage to commercial scale. I am confident that our efforts ultimately will greatly benefit our nation by lessening the country's dependence on foreign oil imports while meeting our environmental objectives.

Warmest regards,

HAWagner

H. A. Wagner

Air Products and Chemicals, Inc.
7201 Hamilton Boulevard
Allentown, PA 18195-1501
Telephone (610) 481-8000

H. A. Wagner
Chairman, President and
Chief Executive Officer

AIR
PRODUCTS

19 October 1995

Mr. Earnest W. Deavenport, Jr.
Chairman of the Board and
Chief Executive Officer
Eastman Chemical Company
P.O. Box 511
Kingsport, TN 37662

Dear Earnie:

You were a superb host at the groundbreaking ceremonies on Monday, making the day a very memorable one for all of us.

We are very proud to be partnering with Eastman on this innovative liquid phase methanol technology project. It is another example of our continuing commercial relationship. In this instance, our collaboration will not only benefit our two companies but ultimately the nation.

Warmest regards,

Hap

H. A. Wagner

Memorandum

AIR PRODUCTS

To: DISTRIBUTION
Dept./Loc.:

From: G. Campbell
Dept./Ext.: Corp./Fin. Comm./14986

Date: 26 October 1995

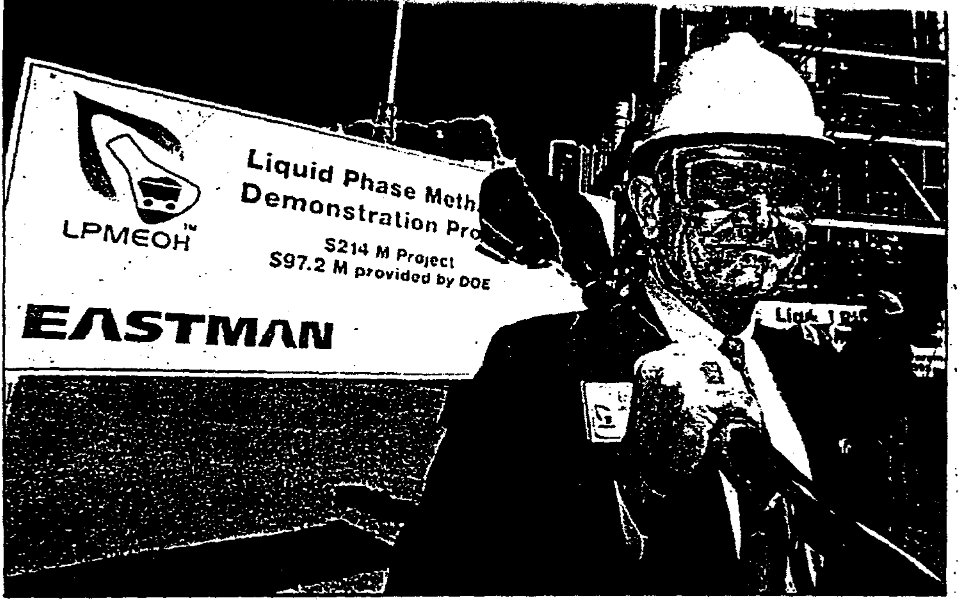
Subject: MEDIA COVERAGE /KINGSPORT GROUNDBREAKING CEREMONY

DISTRIBUTION:
C. E. Anderson
~~W. R. Brown~~ B 10/30
J. A. Castello
N. Chatterjee
B. J. Halper
J. J. Holliday
S. A. Morth
K. J. Ramundo
J. C. Sorensen
J. F. Strecansky
H. A. Wagner
P. J. Witmer

cc: RM Kornosky - PETC

Attached is the newspaper coverage we received in the Kingsport area newspapers on the groundbreaking ceremony for the liquid phase methanol facility. The local TV stations also covered the event . Let me know if you would like to borrow the videotapes of that coverage.

Attach.



Times-News photo — David Grace

Eastman Chairman and CEO Earnie Deavenport stands on the site of the liquid phase methanol process demonstration facility, scheduled to be completed by the end of 1996.

Eastman holds groundbreaking for methanol producing facility

By AMY STEVENS
Times-News Business Writer

Officials from Eastman Chemical Co., Air Products and Chemicals Inc. and the U.S. Department of Energy on Monday broke ground for a facility that Eastman Chairman and Chief Executive Officer Earnie Deavenport said "confirms that technology is a viable solution to our environmental problems."

The liquid phase methanol process demonstration facility, scheduled to be completed at Eastman's Kingsport complex by the end of 1996, will make methanol from coal-derived synthesis gas. The project is part of the DOE's Clean Coal Technology Program and will be the first facility of its kind in existence.

Projected cost for the facility is $214 million. The DOE will provide $93 million, or 43 percent of the costs. The remaining $121 million will be funded through a joint venture between Eastman and Air Products and Chemicals.

"This first-of-a-kind facility ... will demonstrate a cost-effective, environmentally friendly route for producing liquid fuels and chemical feedstocks from coal," said H.A. Wagner, chairman, president and CEO of Air Products and Chemicals. "This project illustrates just how well government and industry can work together to address our nation's goals for energy independence and a cleaner environment."

At full output, the liquid phase methanol facility will produce 260 tons of methanol per day. Although most methanol today is produced from natural gas, officials said, Eastman has pioneered commercial technology for producing methanol from coal.

Methanol produced at the facility will be used by Eastman as a chemical feedstock. The methanol will also be tested off-site as an alternative fuel in packaged electric generators and as a transportation fuel in buses and van pools in California and West Virginia.

Test operations at the facility are scheduled to begin in early 1997 and continue through January 2001. The facility is the 31st project in the DOE's Clean Coal Technology Program.

"Today is indeed a momentous occasion, because it's a day of confirmation," Deavenport said. "Today confirms the beginning of a new era in synthesis gas technology. The day that a new technology takes the giant step from pilot plant to commercial scale is certainly a day for celebration.

"And to the scientists and engineers who have brought this technology to this stage in its development, I extend my heartfelt congratulations and a special thanks for applying your knowledge and your skills toward making our environment a little greener."

Rep. Jimmy Quillen was on hand for the groundbreaking and said he is "proud of what's going to happen" at the facility, citing a need to use coal to its maximum potential for developing new energies.

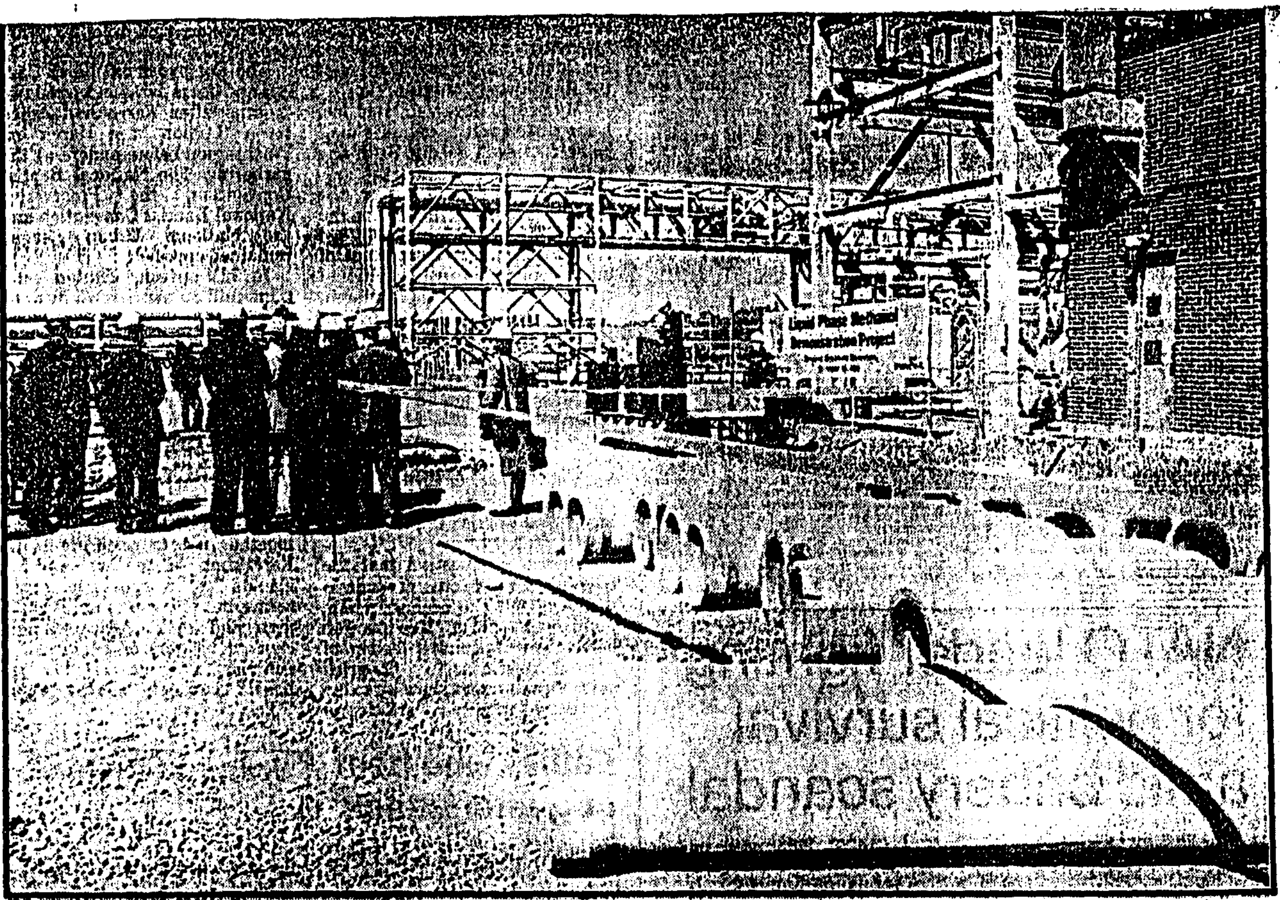
And Alan Edwards, principal deputy assistant secretary of fossil energy for the DOE, said the groundbreaking is a milestone that "will reshape the role of coal in the 21st century."

Projects such as the new facility, Edwards said, are the return on the federal government's $6.7 billion investment in the Clean Coal program.

"When we go to Congress to explain what the American people are getting for their Clean Coal investment, we can talk about brick and mortar and steel rather than just promises and possibilities," Edwards said. "We can talk about a new way to produce liquid fuels to power our automobiles and light our homes.

"We can talk about the affordable energy that can't be embargoed or shut off from our shores. This energy is ours, and we can control it. It's part of our destiny...

"That is why when the American people ask, 'What are we doing to improve the quality of our lives and the lives of our children?' we can point here to Kingsport, Tennessee, to the brick and steel of an innovative facility ... and we can say to them, 'This is what we're building for you, for your future and the future of our children.' "



Staff Photo by Lee Talbert

Officials break ground on the Liquid Phase Methanol Demonstration Project at Tennessee Eastman

Coal extraction plant launched in joint project

By Phyllis Johnson

Press Business Writer

KINGSPORT — Construction has begun here on a unique facility to extract methanol from coal, a process that eventually could lessen the country's dependence on foreign oil and produce cleaner-burning fuels.

The first of its kind anywhere, the Liquid Phase Methanol Demonstration Project is a joint venture among Eastman Chemical Co., Air Products & Chemicals Inc. and the Department of Energy's Clean Coal Technology Program.

The DOE is funding 43 percent of the $213.7 million project in the belief it will lead to broad-based commercial applications of the technology, which Eastman pioneered.

The methanol derived in Eastman's low-impact process is clean-burning and can be used in electricity-generating turbines, as a fuel for automobiles and other vehicles, or as feedstock for a variety of chemicals.

The process provides a way to tap into the nation's vast coal reserves without damaging the environment, said Alan Edwards, DOE assistant secretary of fossil energy, during a groundbreaking ceremony Monday.

"Coal is our largest energy resource. We can't turn our backs on it. (This technology) could provide a secure, affordable energy source for our country."

Test operations are targeted to begin in December 1996, when construction is complete, and continue until January 2001.

In full production, the demonstration facility will produce 260 tons per day of methanol.

Eastman will use a portion in its plastics and chemicals manufacturing processes.

The methanol also will be tested off-site as an alternative fuel in packaged electric generators and as a transportation fuel in buses and van pools in California and West Virginia. Off-site testing will begin in January 1998.

The long-term implications of the project are considerable, said Eastman Chairman and CEO Earnie Deavenport.

Please see FUEL, Page 9

Fuel

Continued from Page 1

"Since the days of the oil embargoes of the 1970s, the DOE has been on a mission to enhance our national security by identifying and developing domestic sources of energy that will reduce America's dependence on foreign oil. Eastman is proud to be a part of this undertaking."

The Clean Coal Technology Program is an ambitious joint government-industry initiative to develop environmentally-clean energy options for the nation's abundant coal reserves.

The Kingsport facility is the

gram to move into construction. Currently, 26 of the projects are either operating or have completed their test runs.

It's no conincidence the project has drawn together APCI, Eastman and the DOE.

APCI came here 14 years ago to build an industrial gas facility to produce oxygen and nitrogen for Eastman's chemicals from coal project, said H.A. Wagner, APCI president and CEO.

"Over the years, that project — designed to save about 1.5 million barrels of oil annually — has showcased Eastman's mastery of the complex chemistry of coal con-

Ground broken for methanol project

By RICK WAGNER
Bristol Herald Courier

KINGSPORT — Coal-derived gas will be converted to methanol through a joint project between Eastman Chemical Co. and Air Products and Chemicals Inc., kicked off here Monday.

The $214 million liquid phase methanol demonstration project, the 31st project in the U.S. Department of Energy's Clean Coal Technology Program, underwent a ground-breaking by officials from Eastman, Allentown, Pa.-based Air Products, DOE and U.S. Rep. James H. Quillen, R-1st.

DOE is providing $97.2 million in funding for the project, which has been in the works for more than a decade. The two partners are providing the rest. The two companies formed Air Products Liquid Phase Conversion Co., L.P., which is DOE's industrial partner in the project.

Quillen used the occasion to reemphasize his support of DOE, which has been the target of Republican congressional efforts to curtail or abolish the federal agency.

"There's a feeling in Washington maybe it should be abolished," Quillen said. "I don't share that feeling."

The project, located in the Long Island section of Kingsport, is to take about 14 months for construction and is to be completed by December 1996. It is to begin operations in the first quarter of 1997 and release its first product early in the second quarter of 1997, of-

Please see METHANOL, Page 10A

METHANOL

From Page 1A

ficials said.

The liquid phase methanol process is scheduled to continue at the demonstration facility until 2001. At full output, the facility will produce 260 tons of methanol a day.

The new technology converts coal-derived synthesis gas to methanol in a single vessel containing catalyst particles suspended in mineral oil. Conventional technology, in contrast, sends the synthesis gas through a fixed bed of dry catalyst particles. The new technology has greater stability and heat dissipation than conventional technology, reliable on/off operation and the ability to use the methanol directly as fuel without further upgrading.

APPENDIX G - TASK 1.5.4 - PROJECT REVIEW MEETING

AIR PRODUCTS

MEETING NOTICE

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

DISTRIBUTION (NAME/ORGANIZATION) *(If unable to attend, contact originator)*

DOE	ACPI	EMN	COPIED FOR INFORMATION ONLY
D. Archer	W. Brown/A12B2	W. Jones	E. Schaub/A12A3
~~K. Khonsari~~	D. Drown/A12B2	L. Paulonis	
R. Kornosky	F. Frenduto/A12B2	B. Street	
~~R. Noceti~~ W. Mundorf	E. Heydorn/MC83		
~~J. Reuther~~	R. Moore/A32G3		
T. Torkos			

FROM	ORGANIZATION	EXTENSION	TODAY'S DATE
D. Drown	PSE- Project Engineering	6143	11 October 1995

DATE OF MEETING	WEEKDAY	TIME FROM	TIME TO	LOCATION
10/17/95	Tuesday	8:30 AM	11:45 AM	Kingsport, TN

SUBJECT AND/OR PURPOSE

Kingsport Liquid Phase Methanol Demonstration Project
DOE Project Status - Review Meeting (Start of Construction)

Desired Results/Outcomes

Project Update

REFERENCE MATERIAL/OTHER

APCI Draft EMP (10/09/95)
Project Evaluation Plan

AGENDA

• Introductions	All		8:30-8:45 AM
• Project Status			8:45-10:00 AM
Overview	WRB	Success Factors Board. on Partnerships FY-96 plan.	
Engineering	DPD		
Procurement	DPD		
Construction	DPD		
Eastman Scope of Work	LBP		
Project Schedule	DPD		
Current Spending	DPD		
• Environmental Monitoring Plan	FSF		10:00-10:45 AM
• Demonstration Plan	ECH		10:45-11:00 AM
• Commercialization Update	WRB		11:00-11:10 AM
• DME/Off Site Testing Update ✓	WRB	DME Milestones - Annex Letter - Attachment.	11:10-11:20 AM
• Wrap-Up	All		11:20-11:45 AM
• Lunch	All		12:00-12:30 PM

MN31

KINGSPORT LPMEOH PROJECT STATUS

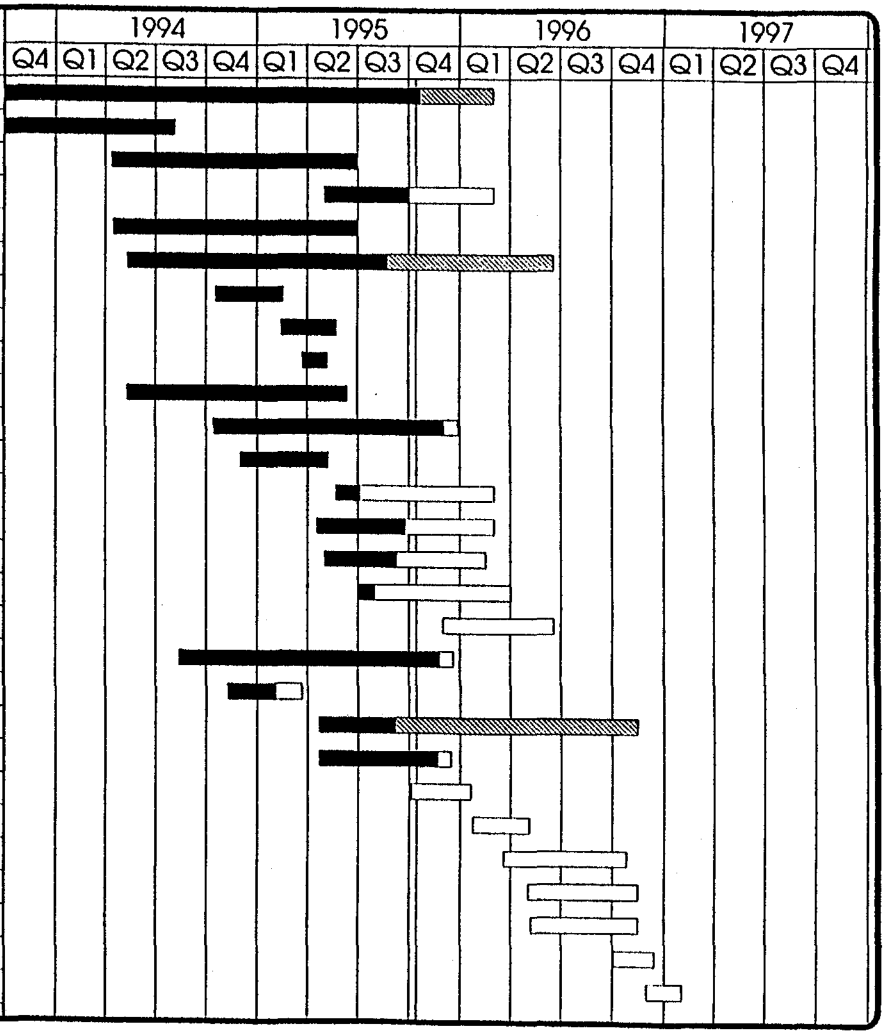
SUMMARY JULY - SEPT. 1995

- PIPING DESIGN TEAM FULLY ENGAGED
- MOST CONTROL VALVES SIZED
- REACTOR FABRICATION RESTARTED
- MAJORITY OF PIPING TIE-IN WORK COMPLETE AT SITE
- SITE PREPARATION WORK STARTED
- FOUNDATION AND UNDERGROUND CONSTRUCTION FOR BIDS IN

S079
10/13/95

LPMEOH DEMONSTRATION PROJECT
PHASE 1-2 SUMMARY SCHEDULE

Task Name	Start	End	Perce Comp
PROCESS DESIGN	Oct/01/93	Mar/01/96	85
PFD/HEAT & MAT'L BALANCE	Oct/01/93	Aug/05/94	100
PROCESS EQUIPMENT SPECS	Apr/13/94	Jun/30/95	100
REVIEW DETAIL DESIGN	May/01/95	Mar/01/96	50
NEPA FONSI REVIEW	Apr/15/94	Jun/30/95	100
DETAILED DESIGN	May/11/94	Jun/17/96	61
P&ID'S, REV. PC	Oct/17/94	Feb/17/95	100
OPERABILITY/HAZARDS REVIEWS	Feb/13/95	May/24/95	100
P&ID'S, REV. P1	Mar/23/95	May/08/95	100
PROCESS EQUIP. MECH. SPECS	May/11/94	Jun/13/95	100
EQUIPMENT VENDOR PRINTS	Oct/14/94	Dec/29/95	94
EQUIPMENT ARRANGEMENTS	Dec/01/94	May/08/95	100
INSTRUMENT SPECS	May/22/95	Mar/01/96	15
CIVIL/STRUCTURAL DESIGN	Apr/17/95	Mar/01/96	50
PIPING DESIGN	May/01/95	Feb/15/96	45
INSTRUMENT/ELECTRICAL DESIGN	Jul/03/95	Apr/01/96	10
DCS PROGRAMMING	Dec/01/95	Jun/17/96	0
EQUIPMENT PROCUREMENT	Aug/12/94	Dec/20/95	95
REACTOR DELIVERY	Nov/08/94	Mar/22/95 96	65
CONSTUCTION	Apr/21/95	Nov/15/96	
EASTMAN OSBL WORK	Apr/21/95	Dec/15/95	90
CIVIL/UNDERGROUND	Oct/02/95	Jan/19/96	2
STEEL & EQUIPMENT ERECTION	Jan/22/96	May/03/96	0
MECHANICAL	Mar/18/96	Oct/25/96	0
INSTRUMENT/ELECTRICAL	May/01/96	Nov/15/96	0
TRAINING	No May/06/96	Nov/15/96	0
CHECK-OUT/COMMISSIONING	Oct/01/96	Dec/13/96	0
START-UP	Nov/29/96	Jan/31/97	0



Printed: Oct/13/95
Page 1

Commissioning
to be done by areas.

Milestone △ Summary ▇▨
Fixed Delay · · ·

NOTES FROM MEETING

DISTRIBUTION (NAME/ORGANIZATION) *Unable to attend. **Chairman

DOE	ACPI	EMN	COPIED FOR INFORMATION ONLY
D. Archer	W. Brown/A12B2	W. Jones	E. Schaub/Process TeamA12A3
R. Kornosky	D. Drown/A12B2	L. Paulonis	D. Brown/A31E4
W. Mundorf	F. Frenduto/A12B2	B. Street	
T. Torkos	E. Heydorn/MC83		
	R. Moore/A32G3		

FROM	ORGANIZATION	EXTENSION	TODAY'S DATE
D. Drown	PSE- Project Engineering	16143	20 October 1995

DATE OF MEETING	WEEKDAY	TIME FROM	TIME TO	LOCATION
10/17/95	Tuesday	8:30 AM	Noon	Kingsport, TN

SUBJECT AND/OR PURPOSE
Kingsport Liquid Phase Methanol Demonstration Project
DOE Project Status - Review Meeting (Start of Construction)

ITEM NO.	RESPONSIBLE PERSON (INITIALS)	TARGET DATE	DISCUSSION
			These notes highlight action items and discussion from the DOE Project Status review meeting held at Eastman's plant on 17 October 1995.
1.	E. Heydorn	12/95	APCI will issue to PETC a draft of the 4 year Demonstration Plan in December.
2.	D. Drown	12/1/95	PETC would like a drawing showing reactor internals.
3.	D. Drown	10/18/95	Remind APCI Transportation that the compressor should be delivered on a U.S. flag ship.
4.	F. Frenduto	11/1/95	APCI should define other areas in plant design that may be of concern in producing noise above 85 dBA (i.e., let down control valves, venting startup steam, etc.).
5.			The on-site delivery dates for the reactor is estimated to be 3/22/96.
6.			APCI plans to submit the Budget Period #3 Continuation Application on or before 8/1/96. The current end date for Budget Period #2 is 9/30/96. We need to better define for the DOE the commissioning and startup schedule and plan as of 8/1/96.

NOTES FROM MEETING
CONTINUATION

AIR PRODUCTS

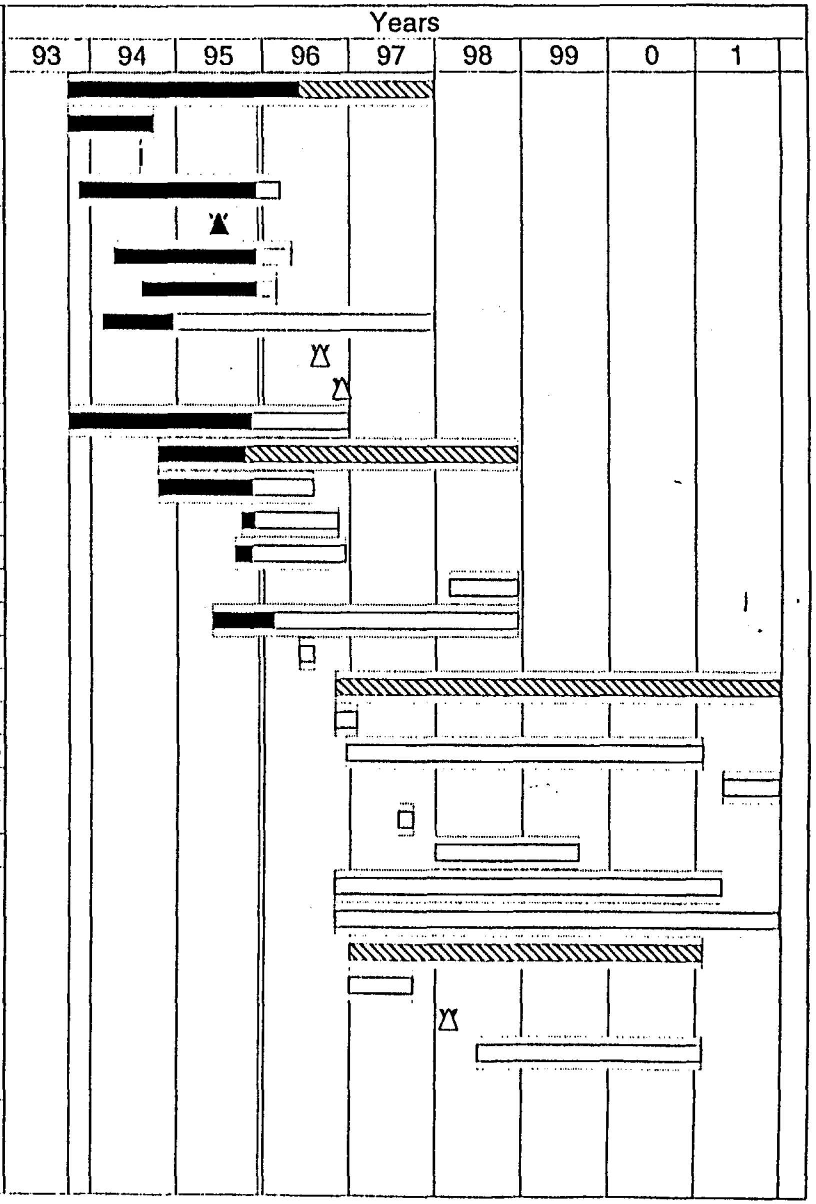
Page Two Of Two

ITEM NO.	RESPONSIBLE PERSON (INITIALS)	TARGET DATE	DISCUSSION
7.	F. Frenduto	11/17/96	APCI will discuss what analysis will be done on each stream to be monitored in Section 7 of the EMP. APCI will mark-up a simplified PFD or BFD with the stream numbers to be monitored. Streams of environmental concern for this plant include: - fugitive emissions - spent catalyst removed from the plant - spent absorbents removed from the plant - higher alcohols to existing Lurgi plant - liquids (spent lubricants, etc.) The EMP should also describe the environmental streams of concern to an audience that may not know anything about the LPMEOH process.
8.	E. Schaub	8/11/96	It was suggested that we write a Topical Report on the work done to date on analyzing the feed synthesis gas streams for catalyst poisons and on the additional work scheduled to be started in May 1996.
9.			APCI is talking to a few potential customers for this technology as an add-on plant in the U.S. (TVA, Koch Refining) and a few potentials overseas. These potential projects are in the study phase. We would not initiate a project until the Kingsport plant is up and running for at least a year.
	W. Brown	1//20/96	APCI will update our commercialization plan next year. We are looking at future energy forecasts to aid in this projection.
10.	W. Brown	1/20/96	APCI needs to write up our conclusions on the market analysis of 3 weight percent DME as a M-100 Diesel replacement or put in the Quarterly report.
11.	W. Brown	10/25/95	APCI will be meeting with Acurex and EPRI next week to reinstate discussions on Off-site Product Fuel-use Testing.
	MN031		

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

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

Task Name	Duration	Start	End	% Comp	% Sched
PHASE 1: DESIGN	**51.20 m**	**Oct/01/93**	**Dec/30/97**	**60**	**63**
PROJECT DEFINITION(TASK1)	12.04 m	Oct/01/93	Sep/30/94	100	100
CONTINUATION APPLICATION(B.P.#2)	9.00 d	Aug/02/94	Aug/10/94	100	100
PERMITTING(TASK 2)	28.04 m	Nov/17/93	Mar/15/96	91	88
NEPA FONSI APPROVAL	0.00 d	Jun/30/95	Jun/30/95	100	100
DESIGN ENGINEERING(TASK 3)	24.68 m	Apr/15/94	May/01/96	70	80
VENDOR ENGINEERING	18.80 m	Aug/10/94	Mar/01/96	90	85
OFF-SITE TESTING(TASK 4)	46.35 m	Feb/25/94	Dec/30/97	6	21
UPDATED FUEL TEST PLAN APPROVAL	0.00 d	Aug/31/96	Aug/31/96	0	0
DECISION TO CONTINUE DME TESTING	0.00 d	Dec/01/96	Dec/01/96	0	0
PLANNING, ADMIN & DME DVT(TASK 5)	39.16 m	Oct/01/93	Dec/30/96	76	65
PHASE 2: CONSTRUCTION	**50.18 m**	**Oct/17/94**	**Dec/15/98**	**10**	**24**
PROCUREMENT(TASK1)	21.61 m	Oct/17/94	Aug/01/96	83	60
CONSTRUCTION(TASK 2)	13.56 m	Oct/02/95	Nov/15/96	6	13
TRAINING & COMMISSIONING(TASK 3)	15.44 m	Sep/05/95	Dec/15/96	4	15
OFF-SITE TESTING(TASK 4)	9.57 m	Mar/01/98	Dec/15/98	0	0
PLANNING & ADMINISTRATION(TASK 5)	42.69 m	Jun/01/95	Dec/15/98	20	20
CONTINUATION APPLICATION(B.P.#3)	2.08 m	May/31/96	Aug/01/96	0	0
PHASE 3: OPERATION	**62.19 m**	**Oct/31/96**	**Dec/28/01**	**0**	**0**
START-UP(TASK 1)	3.10 m	Oct/31/96	Feb/01/97	0	0
METHANOL OPERATION(TASK 2.1)	49.78 m	Dec/16/96	Feb/01/01	0	0
DISMANTLE PLANT(TASK 2.3)	7.98 m	May/01/01	Dec/28/01	0	0
ON-SITE PRODUCT USE DEMO(TASK 3)	2.08 m	Aug/01/97	Oct/02/97	0	0
OFF-SITE PRODUCT USE DEMO(TASK 4)	20.02 m	Jan/02/98	Aug/31/99	0	0
DATA ANALYSIS/REPORTS(TASK 5)	54.10 m	Oct/31/96	Apr/27/01	0	0
PLANNING & ADMINISTRATIVE(TASK 6)	62.19 m	Oct/31/96	Dec/28/01	0	0
PROVISIONAL DME IMPLEMENTATION	**49.25 m**	**Jan/02/97**	**Feb/02/01**	**0**	**0**
DME DVT(PDU TESTS)(TASK 3.6)	8.97 m	Jan/02/97	Sep/30/97	0	0
DECISION TO IMPLEMENT	0.00 d	Mar/01/98	Mar/01/98	0	0
DESIGN, MODIFY & OPERATE(TASK 3.2.2)	31.27 m	Jul/01/98	Feb/02/01	0	0



Printed: Dec/12/95
Page 1

Milestone △ Summary ▨

DOE F 1332.9
(11-84)

U.S. DEPARTMENT OF ENERGY

COST MANAGEMENT REPORT

Page 1 of 1
FORM APPROVED
OMB 1910-1400

1. TITLE Liquid Phase Methanol Demonstration	2. REPORTING PERIOD December 01, 1995 through December 31, 1995	3. IDENTIFICATION NUMBER DE-FC22-92PC90543
2. PARTICIPANT NAME AND ADDRESS Air Products Liquid Phase Conversion Co., L.P. 7201 Hamilton Boulevard Allentown, PA 18195-4911	5. COST PLAN DATE October 01, 1995	6. START DATE January 1, 1990 7. COMPLETION DATE December 31, 2001

8. ELEMENT	9. REPORTING ELEMENT	10. ACCRUED COSTS				11. ESTIMATED ACCRUED COSTS							12.	13.
		Reporting Period		Cumulative to Date		a. Subsequent Reporting Period	b. Balance of Fiscal Year	c.			d. Subsequent FY's	e.	Total Contract Value	Variance
		a. Actual	b. Plan	c. Actual	d. Plan			FY 1997 (1)	FY 1998 (2)	FY 1999 (3)	(4)	Total		
	Prior to Mod 2	0	0	16,289	16,289	0	0					16,289	16,289	0
1.1.1	Project Definition	0	0	1,021	1,021	0	0	0	0	0	0	1,021	1,021	0
1.1.2	Permitting	0	8	221	238	8	17	0	0	0	0	246	246	0
1.1.3	Design Engr.	504	622	6,037	6,231	565	3,075	283	0	0	0	9,960	9,960	0
1.1.4	Off-site Testing	0	6	12	14	6	56	246	0	0	0	320	320	0
1.1.5	Planning, Admin, & DME Verif. Testing	97	43	1,697	1,508	43	152	0	0	0	0	1,892	1,892	0
1.2.1	Procurement	307	552	3,254	2,715	829	4,572	1,128	0	0	0	9,783	9,783	0
1.2.2	Construction	138	738	1,015	1,660	1,108	7,475	1,602	0	0	0	11,200	11,200	0
1.2.3	~~Proj. Mgmt.~~ Train + Commiss.	0	10	1	31	80	1,082	34	0	0	0	1,197	1,197	0
1.2.4	Off-Site Test - Proc. & Constr.	0	0	0	0	0	0	180	81	0	0	261	261	0
1.2.5	Planning & Admin	18	46	104	197	46	463	68	0	0	0	681	681	0
1.3.1	Startup	0	0	0	0	0	0	3,435	0	0	0	3,435	3,435	0
1.3.2	Operations													0
1.3.2.1	Methanol Operation	0	0	0	0	0	0	33,753	36,822	36,822	39,890	147,287	147,287	0
1.3.2.2	DME Design, Mod., Oper.	0	0	0	0	0	0	351	509	680	800	2,340	2,340	0
1.3.2.3	LPMEOH Dismantlement	0	0	0	0	0	0	0	0	0	425	425	425	0
1.3.3	On-Site Demo.	0	0	0	0	0	0	0	0	2	2	4	4	0
1.3.4	Off-Site Demo.	0	0	0	0	0	0	427	2,773	340	300	3,840	3,840	0
1.3.5	Data Analysis & Reports	0	0	0	0	0	0	385	380	500	661	1,926	1,926	0
1.3.6	Planning & Admin.	0	0	0	0	0	0	245	252	260	836	1,593	1,593	0
14. TOTAL		1,064	2,025	29,652	29,904	2,685	16,891	42,137	40,817	38,604	42,914	213,700	213,700	0

15. DOLLARS EXPRESSED IN: Thousands	16. SIGNATURE OF PARTICIPANT'S PROJECT MANAGER AND DATE D. P. Drown 1/26/96 DATE	17. SIGNATURE OF PARTICIPANT'S AUTHORIZED FINANCIAL REPRESENTATIVE AND DATE Susan J. Kasinecz S. J. Kasinecz 1/19/96 DATE

APPENDIX I - TASK 2.1 - PROCUREMENT SPECIFICATION STATUS - EQUIPMENT

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

Tag #	Description	Process Engineer	Process Spec Released(Rev)	Equipment Specifier	Equipment Spec Released(Rev)	Eastman Comments	Vendor List*	Released to Purchasing	Bids Due	Bids Received	Order Placement	Vendor Prints Due/Received
C-01	LPMEOH Reactor	Schaub	6/10/94 (0)	Koeller	11/02/94 (1)	8/18/94	Y	8/29/94	9/29/94	10/3/94	11/8/94	Rec'd 1/3/95
C-02	Steam Drum	Schaub	10/19/94 (1)	Koeller	11/07/94 (1)	10/19/94	Y	11/8/94	12/6/94	12/7/94	3/16/95	Rec'd 4/18/95
C-03	HP Methanol Separator	Schaub	11/18/94 (1)	Koeller	01/09/95 (2)	12/15/94	Y	1/5/95	2/2/95	2/10/95	3/15/95	Rec'd 4/04/95
C-05	Secondary Oil Knockout Vessel	Schaub	11/18/94 (2)	Koeller	1/09/95 (2)	12/15/94	Y	1/5/95	2/2/95	2/10/95	3/15/95	Rec'd 4/04/95
C-06	Reactor Cyclone	Schaub	11/21/94 (2)	Koeller	11/30/94 (1)	11/30/94	Y	12/2/94	12/28/94	12/16/94	3/21/95	Rec'd 4/18/95
C-07	Syngas Compressor Knockout Separator	Mazdai	03/22/95 (0)	Koeller	3/23/95 (0)	4/5/95	Y	4/25/95	5/17/95	5/17/95	6/5/95	Rec'd 7/10/95
C-10	Methanol Stabilizer Column	Stein	11/11/94 (2)	Koeller	11/11/94 (2)	9/21/94	Y	10/4/94	11/1/94	11/1/94	12/20/94	Rec'd 1/25/95
C-10T	Methanol Stabilizer Column Trays	Stein	11/11/94 (2)	Koeller	11/11/94 (2)	10/11/94	Y	9/7/94	10/14/94	10/7/94	1/10/95	Rec'd 3/02/95
C-11	Methanol Stabilizer Reflux Drum	Stein	12/30/94 (2)	Koeller	01/09/95 (1)	12/15/94	Y	1/10/95	2/7/95	2/17/95	3/14/95	Rec'd 4/18/95
C-12	Methanol Stabilizer Feed Vessel	Stein	11/18/94 (0)	Koeller	01/09/95 (1)	12/15/94	Y	1/10/95	2/7/95	2/17/95	3/14/95	Rec'd 4/26/95
C-13	Methanol Stabilizer Condensate Pot	Stein	03/09/95 (0)	Koeller	3/27/95 (1)	3/27/95	Y	3/28/95	4/18/95	4/20/95	5/15/95	Rec'd 6/6/95
C-20	Methanol Rectifier Column	Stein	11/11/94 (2)	Koeller	11/11/94 (2)	9/21/94	Y	10/4/94	11/1/94	11/1/94	12/20/94	Rec'd 1/25/95
C-20T	Methanol Rectifier Column Trays	Stein	11/11/94 (2)	Koeller	11/11/94 (2)	10/11/94	Y	9/7/94	10/14/94	10/7/94	1/10/95	Rec'd 3/9/95
C-21	Methanol Rectifier Reflux Drum	Stein	12/30/94 (2)	Koeller	01/09/95 (1)	12/15/94	Y	1/10/95	2/7/95	2/17/95	3/14/95	Rec'd 4/18/95
C-23	Methanol Rectifier Condensate Pot	Stein	03/09/95 (0)	Koeller	3/27/95 (1)	3/27/95	Y	3/28/95	4/18/95	4/20/95	5/15/95	Rec'd 6/6/95
C-30	Catalyst Reduction Vessel	Bhatt	8/23/94 (0)	Koeller	10/03/94 (1)	9/21/94	Y	10/4/94	11/1/94	11/1/94	12/16/94	Rec'd 1/12/95
C-31	Reduction Condensate Accumulator	Mazdai	3/07/95 (2)	Koeller	01/09/95 (1)	12/15/94	Y	1/10/95	2/7/95	2/17/95	3/14/95	Rec'd 4/26/95
C-32	Utility Oil Surge Tank (V-01 Skid)	Bhatt	9/29/94 (0)	Koeller	12/02/94 (1)	11/23/94	Y	12/5/94	1/12/95	1/9/95	3/29/95	Rec'd 6/17/95
C-40	Guard Bed	Chen	03/10/95 (0)	Koeller	3/27/95 (1)	3/27/95	Y	3/28/95	4/20/95	5/3/95	5/19/95	Rec'd 6/26/95
C-50	Oil-Water Separator/Coalescer	N/A		Skuchas	6/13/95 (0)	6/26/95	Y	6/14/95	7/10/95	7/11/95	8/16/95	Rec'd 8/31/95
C-120	Vent Scrubber											
D-01	Safety Relief Knockout Drum	Schaub	10/30/95 (0)	Skuchas	11/14/95 (0)		Y	11/15/95	12/8/95	12/8/95	12/15/95	Due 1/5/96
D-02	Slurry Tank	Mazdai	3/07/95 (2)	Koeller	3/08/95 (2)	3/3/95	Y	3/9/95	4/3/95	4/7/95	6/5/95	Rec'd 7/13/95
D-20	Methanol Lot Tank	Stein	3/07/95 (1)	Koeller	3/09/95 (1)	3/8/95	Y	3/13/95	4/10/95	4/12/95	8/1/95	Rec'd 8/23/95
D-21	Methanol Lot Tank	Stein	3/07/95 (1)	Koeller	3/09/95 (1)	3/8/95	Y	3/13/95	4/10/95	4/12/95	8/1/95	Rec'd 8/23/95
D-25	Methanol Drain Tank	Frenduto/Stein	4/21/95 (1)	Skuchas	6/13/95 (0)	6/26/95	Y	6/14/95	7/10/95	7/11/95	8/11/95	Rec'd 9/8/95
D-30	Fresh Oil Storage Tank	Mazdai	3/30/95 (2)	Koeller	3/08/95 (2)	3/3/95	Y	3/9/95	4/3/95	4/7/95	6/5/95	Rec'd 7/13/95
D-60	Caustic Mix Tank	Stein	2/03/95 (1)	Koeller	3/08/95 (2)	3/3/95	Y	3/9/95	4/3/95	4/7/95	6/5/95	Rec'd 7/13/95
D-70	Compressor L.O. Sump (K-01 Skid)	Schaub	4/22/94 (1)	Fleischer	08/29/94 (1)	5/17/94	Y	5/17/94	6/12/94	6/12/94	8/12/94	Rec'd 10/14/94
E-01	Syngas Compressor Cooler	Chen	03/31/95 (5)	Koeller	4/26/95 (4)	10/11/94	Y	11/8/94	12/21/94	12/20/94	3/28/95	Rec'd 5/10/95
E-02	Syngas Feed/Product Economizer	Schaub	8/29/94 (1)	Koeller	9/16/94 (1)	9/7/94	Y	9/19/94	10/14/94	10/24/94	12/2/94	Rec'd 1/27/95
E-03	Methanol Product Air-Cooled Condenser	Chen	10/14/94 (0)	Koeller	4/10/95 (2)	12/6/94	Y	12/1/94	1/5/95	1/9/95	3/24/95	Rec'd 4/20/95
E-04	Methanol Product C.W. Condenser	Chen	10/21/94 (1)	Koeller	11/7/94 (2)	11/1/94	Y	11/8/94	12/21/94	12/20/94	3/28/95	Rec'd 5/19/95
E-10	Methanol Stabilizer Reboiler	Stein	10/10/94 (2)	Koeller	11/07/94 (1)	10/19/94	Y	11/8/94	12/21/94	12/20/94	3/28/95	Rec'd 6/5/95
E-11	Methanol Stabilizer Condenser	Stein	10/17/94 (3)	Koeller	11/7/94 (2)	10/11/94	Y	11/8/94	1/9/95	12/28/94	3/28/95	Rec'd 5/10/95
E-20	Methanol Rectifier Reboiler	Stein	10/10/94 (1)	Koeller	11/07/94 (1)	10/19/94	Y	11/8/94	12/21/94	12/20/94	3/28/95	Rec'd 6/5/95
E-21	Methanol Rectifier Air Cooler	Chen	03/31/95 (2)	Koeller	11/30/94 (1)	12/6/94	Y	12/1/94	1/5/95	1/9/95	3/24/95	Rec'd 4/20/95
E-22	Methanol Rectifier C.W. Condenser	Stein	10/18/94 (2)	Koeller	11/7/94 (2)	11/1/94	Y	11/8/94	1/9/95	12/28/94	3/28/95	Rec'd 5/11/95
E-23	Crude Methanol Cooler	Stein	10/09/94 (1)	Koeller	11/7/94 (2)	11/1/94	Y	11/8/94	1/9/95	12/28/94	3/28/95	Rec'd 5/11/95
E-31	Reduction Vessel Overhead Condenser	Mazdai	10/14/94 (0)	Koeller	11/7/94 (2)	11/1/94	Y	11/8/94	1/9/95	12/28/94	3/28/95	Rec'd 5/11/95
E-32A/B/C	Utility Oil Heaters (V-01 Skid)	Bhatt	9/29/94 (0)	Koeller	12/02/94 (1)	11/23/94	Y	12/5/94	1/12/95	1/9/95	3/29/95	Rec'd 9/19/95

* preferred vendor list issued to Eastman

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

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

* preferred vendor list issued to Eastman

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

Tag #	Description	Process Engineer	Process Spec Released(Rev)	Equipment Specifier	Equipment Spec Released(Rev)	Eastman Comments	Vendor List*	Released to Purchasing	Bids Due	Bids Received	Order Placement	Vendor Prints Due/Received
Y-10	Methanol Product Filter	N/A	N/A	Browning	4/26/95 (2)	4/10/95	Y	4/27/95	5/15/95	5/18/95	7/19/95	Rec'd 8/19/95
Y-30	Catalyst Reduction Agitator	Bhatt	10/24/94 (1)	Fleischer	2/23/95 (2)	10/19/94	Y	11/11/94	12/9/94	12/14/94	3/20/95	Rec'd 4/25/95
Y-35A/B	Seal Oil Filters	N/A	*data sheet issued*	Browning	4/26/95 (2)	4/10/95	Y	4/27/95	5/15/95	5/18/95	7/19/95	Rec'd 8/19/95
Y-60	Caustic Tank Agitator	Stein	11/29/94 (0)		2/23/95 (1)	12/15/94	Y	12/8/94	1/9/95	1/17/95	3/20/95	Rec'd 4/28/95
Y-70A/B	Compressor L.O. Filters (K-01 Skid)	Schaub	4/22/94 (1)	Fleischer	08/29/94 (1)	5/17/94	Y	5/17/94	6/12/94	6/12/94	8/12/94	Rec'd 10/14/94
Y-71A/B	Compressor Seal Gas Filters (K-01 Skid)	Schaub	4/22/94 (1)	Fleischer	08/29/94 (1)	5/17/94	Y	5/17/94	6/12/94	6/12/94	8/12/94	Rec'd 10/14/94
------	Trailer Loading Platform	N/A	N/A	Frenduto				8/31/95		4/26/95	9/5/95	Rec'd 10/4/95

* preferred vendor list issued to Eastman

APPENDIX J - TASK 2.3 - DEMONSTRATION TEST PLAN

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

Telephone (610) 481-4911
Telex: 847416

AIR PRODUCTS

December 22, 1995

WRB's
1/29/96
with comments

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

Cooperative Agreement DE-FC22-92PC90543
Liquid Phase Methanol Demonstration Project
Subject: Operating Test Plan

Dear Bob:

Attached for your review and comments is the first draft (Rev. 0) of the four year test plan for Phase 3, Task 2 (LPMEOH™ Process Demonstration Facility) Operation. We plan to iterate this plan over the next few months, with input from all the participants as well as from our Phase 1, Task 5 commercial economic studies. The final draft of this plan is due to be submitted in August 1996, 60 days prior to the commencement of Phase 3.

Task 2.1.1 now includes a few (high inlet velocity, high catalyst concentration) commercially important tests from Task 2.2.2. These changes provides a better overall use of the four year test plan time.

We have focused on the first 12 to 18 months of operation in developing this first four year plan. We will be preparing annual test plans throughout Phase 3 operation. These annual test plans updates will provide detailed plans for test runs for the coming twelve months, and a general plan for the remaining years.

We look forward to receiving your comments and suggestions on this draft of the Operating Test Plan.

Very truly yours,

E. C. Heydorn
DOE/CCT Operational Program Manager
LPMEOH Demonstration Project

Attachments - 1) Operation Plan (8 Pages) - 2) Glossary (2 Pages) - 3) Test Plan Table (4 pages)
ECH/L-001

Test					Space		Fresh Feed		Compressed Feed		Inlet Sup.	Time	Start
Run	Test Run	Temp	Wt%	Recycle	Velocity	MeOH	Balanced	CO Gas	H2 Gas	Recycle	Velocity	Period	of
#	Description	(Deg C)	Cat	Ratio	(Sl/hr-kg)	(tpd)	(KSCFH)	(KSCFH)	(KSCFH)	(KSCFH)	(ft/sec)	(weeks)	Test
Task 2.1.1 - Process Shakedown and Catalyst Aging:													
1.	Initial Shakedown; and	250	28	1.82	8000	260	900	50	40	1800	0.64	6	Jan-97
	Design Production Tests.						(varies, to maintain syngas utilization.)						
2.	Gassed Slurry Level	Part of other tests										Ongoing	
	Tests												
3.	Reactor Feed: Texaco-	250	28	High (*)	High	TBD	TBD (650)	TBD (*)	0	TBD	TBD	2	Feb-97
	Type Syngas												
4.	Early Testing @ High	250	28	High (*)	TBD	TBD	1200 (**)	TBD (**)	TBD	TBD	High	2	Mar-97
	Superficial Velocity												
5.	Check @ Test 1	250	28	1.82	8000	< 260	900	50	40	1800	0.64	2	Mar-97
	Conditions												
6.	Catalyst Addition and	250	28 - 40	Max	Dec. from	>237	765	40	45	Max(2700)	0.79	18	Apr-97
	Aging	or less			8000	TBD	900	50	40	Max	TBD	6	to
													Oct-97
7.	Free-Drain Entrained/	250	28 - 40	Max	Dec. from	237	765	40	45	Max	0.79	During	
	Condensed Oil to	or less			8000							Test 6	
	Reactor												
8.	Operation @ Design	250	40	1.82	4000	260	900	50	40	1800	0.64	2	Oct-97
	Feed Gas Rates												
9.	Check for Limitation on	250	> 40	Max	Varies	TBD	765	40	45	Max(2700)	0.79	6	Oct-97
	Catalyst Slurry Concentration.												
10.	Catalyst Addition to	250	Target	Max	TBD	TBD	765	40	45	Max(TBD)	0.79	16	Dec-97
	Reach Max Productivity.		Max				900	50	40	Max(TBD)			April 96

Test Run #	Test Run Description	Temp (Deg C)	Wt% Cat	Recycle Ratio	Space Velocity (Sl/hr-kg)	MeOH (tpd)	Fresh Feed		Compressed Feed		Inlet Sup. Velocity (ft/sec)	Time Period (weeks)	Start of Test
							Balanced (KSCFH)	CO Gas (KSCFH)	H2 Gas (KSCFH)	Recycle (KSCFH)			
Task 2.1.2 - Process Operational Test Phase:													
	Note: At this time, need to produce some "typical" product methanol for off-site fuel tests. Also need to reassess the optimum operating conditions for the remaining tests. e.g. for commercial design/optimal performance).												
11.	Catalyst Addition/ Withdrawal Test	250	Target	Max	TBD	TBD	765	40	45	Max(TBD)	0.79	6	Apr-98
12.	Test 11 Conditions with No CO Make-up	250	Target	Max	TBD	TBD	765	0	45	Max(TBD)	TBD	2	May-98
13.	Test 11 Conditions with No H2 Make-up	250	Target	Max	TBD	TBD	765	40	0	Max(TBD)	TBD	2	Jun-98
14.	Test 11 Conditions with No H2 or CO Make-up	250	Target	Max	TBD	TBD	765	0	0	Max(TBD)	TBD	2	Jul-98
15.	Repeat of Test 11 Conditions	250	Target	Max	TBD	TBD	765	40	45	Max(TBD)	0.79	2	Jul-98
16.	Design Fresh Feed Operation Test	250	Target	Max	TBD	TBD	900	50	40	Max(TBD)	TBD	2	Jul-98
17.	Testing @ High Superficial Velocity	250	Target	High (*)	TBD	TBD	1200 (**)	TBD	TBD	TBD	High	2	Aug-98
18.	Turndown (50% of Design)	250	Target	TBD	TBD	TBD	450	25	TBD	TBD	TBD	2	Sep-98
19.	Load-Following, Cyclone, & On/Off Tests			To be defined								4	Sep-98
20.	Reactor Feed: Texaco-Type Syngas	250	Target	High (*)	High	TBD	TBD (650)	TBD (**)	0	High	TBD	4	Oct-98

Test Run #	Test Run Description	Temp (Deg C)	Wt% Cat	Recycle Ratio	Space Velocity (Sl/hr-kg)	MeOH (tpd)	Fresh Feed		Compressed Feed		Inlet Sup. Velocity (ft/sec)	Time Period (weeks)	Start of Test
							Balanced (KSCFH)	CO Gas (KSCFH)	H2 Gas (KSCFH)	Recycle (KSCFH)			
21.	Reactor Feed: Destec-Type Syngas	250	Target	To be defined				(***)				3	Nov-98
22.	Reactor Feed: BGL-Type Syngas	250	Target	To be defined				(***)				3	Dec-98
23.	Repeat of Test 15 Conditions	250	Target	Max	TBD	TBD	765	40	45		0.79	2	Dec-98
24	Reactor Feed: Nat.Gas Reformer-Type Syngas	250	Target	To be defined								3	Jan-99
25.	Reactor Feed: Shell-Type Syngas with Steam Injection and 1:1 Recycle	250	Target	To be defined				(***)				3	Feb-99
26.	Operation with Two Methanol Synthesis Catalysts	250	Target	Max	TBD	TBD	765	40	45	Max(TBD)	0.79	8	Mar-99
							900	50	40		TBD	2	
27.	Reactor Operation @ 500 deg F	260	Target	To be defined								2	May-99
28.	Reactor Inspection											4	Jun-99
	(Then, Continue Operational Tests - with alternative catalyst):												
29.	Plant Shakedown	240	TBD	Max	TBD	260	900	50	40	Max(TBD)	TBD	6	Jul-99
30.	Reactor Feed: Texaco-Type Syngas	240	To be defined					(***)				2	Aug-99
31.	Catalyst Aging	240	TBD	Max	TBD							16	Sep-99
							900	50	40	Max(TBD)	TBD	4	

Test Run #	Test Run Description	Temp (Deg C)	Wt% Cat	Recycle Ratio	Space Velocity (Sl/hr-kg)	MeOH (tpd)	Fresh Feed		Compressed Feed		Inlet Sup. Velocity (ft/sec)	Time Period (weeks)	Start of Test
							Balanced (KSCFH)	CO Gas (KSCFH)	H2 Gas (KSCFH)	Recycle (KSCFH)			
32.	Catalyst Addition/	240 -	Target	Max	To be					Max(TBD)		6	Feb-00
	Withdrawal to Achieve	250			defined								
	Target Slurry Concentration												
33.	Reactor Feed: Texaco-	250	Target	To be				(***)				4	Mar-00
	Type Syngas			defined									
Task 2.1.3 - Extended Optimum Operation:													
34.	Stable Operation	250	Target	Max	TBD	TBD	765	40	45		TBD	16	Apr-00
		250	Target	Max	TBD	TBD	900	50	40		TBD	8	
35.	DME Demo (Task 2.2) or			To be								8	Oct-00
	Continuous Test Run (Task 2.1.3)			defined									to
													Dec-00
	Syngas Outages (5%)											10	
	Planned LPMEOH Outages (incl. 2 1-wk. Carbonyl Burnouts and Reactor Inspection aft. Test 26)											12	
	Unplanned LPMEOH Outages (10/yr @ 8 hrs.)											2	

Notes:

(*) - 700 HP motor on 29K-01 Compressor allows higher recycle gas flow than in the Rev. 5 C. Chen memo (for Texaco case, the CO usage could be reduced from 100 KSCFH to 80 - 90 KSCFH).

(**) - 1200 KSCFH of Fresh Feed Syngas can be made available for testing (per Eastman debottlenecking of gasification area). Final decision on test will depend upon carbonyl concentrations in Fresh Feed and CO Make-up, since 29C-40 Carbonyl Guard Bed will have to be bypassed. For this condition, test execution is subject to availability of CO-makeup stream.

(***) - Subject to availability of CO-makeup stream.