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Commercial-Scale Demonstration of the Liquid Phase Methanol (LPMEOH^{TM}) Process

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Office of Fossil Energy
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ACRONYMS AND DEFINITIONS

Acurex	-	Acurex Environmental Corporation
Air Products	-	Air Products and Chemicals, Inc.
AFDU	-	Alternative Fuels Development Unit - The "LaPorte PDU."
Balanced Gas	-	A syngas with a composition of hydrogen (H_2), carbon monoxide (CO), and carbon dioxide (CO_2) in stoichiometric balance for the production of methanol
Carbon Monoxide Gas	-	A syngas containing primarily carbon monoxide (CO); also called CO Gas
DME	-	dimethyl ether
DOE	-	United States Department of Energy
DOE-PETC	-	The DOE's Pittsburgh Energy Technology 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 is being 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 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 tons-per-day (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 3,200 gallons per day, DOE-owned experimental unit in LaPorte, Texas, the technology provides several improvements essential for the economic coproduction of methanol and electricity directly from gasified coal. This liquid phase process suspends fine catalyst particles in an inert liquid, forming a slurry. The slurry dissipates the heat of the chemical reaction away from the catalyst surface, protecting the catalyst and allowing the methanol synthesis reaction to proceed at higher rates.

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

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

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

The project was reinitiated in October of 1993, when DOE approved a site change to the Kingsport location. DOE conditionally approved the Continuation Application to Budget Period No. 2 (Design and Construction) in March, and formally approved it on 1 June 1995 (Mod M009). Since then the project has been in Design - Phase 1 - activities; and also moved 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.

Construction work for the LPMEOH™ plant began in October of 1995. The foundation and underground work was completed in January. The erection of the pipe rack steel and equipment items has begun, and piping installation in the pipe rack area should begin in April. The fabrication of the reactor continues, and is being expedited. The reactor ship date has slipped to 3 May 1996.

DOE's comments on the draft Environmental Monitoring Plan (EMP) and on the draft Demonstration Test Plan (DTP) were received. Revised EMP and DTP drafts were prepared, and a meeting to review and finalize both is planned for April.

Procurement of process equipment is essentially complete and construction work is well underway. Mechanical completion has slipped two weeks due to late reactor and structural steel delivery dates. Commissioning work is expected to start in mid-October, with plant start-up in late December. Fifty-two percent (52%) 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 March 1996.

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 of methanol (260 TPD) is being designed and constructed at a site located at the Eastman Chemical Company (Eastman) complex in Kingsport, Tennessee. The Partnership will own and operate the facility for the four-year demonstration facility operational period.

This project is sponsored under the DOE's Clean Coal Technology Program, and its primary objective is to "demonstrate the production of methanol using the LPMEOH™ Process in conjunction with an integrated coal gasification facility." The project will also demonstrate the suitability of the methanol produced for use as a chemical feedstock or as a low-sulfur dioxide, low-nitrogen oxides alternative fuel in stationary and transportation applications. The project may also demonstrate the production of dimethyl ether (DME) as a mixed coproduct with methanol, if laboratory- and pilot-scale research 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 reactionequipment.
- *Purification Area* - Product separation and purification equipment.
- *Catalyst Preparation Area* - Catalyst and slurry preparation and disposal equipment.
- *Storage/Utility Area* - Methanol product, slurry and oil storage equipment.

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

Reaction Area

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

Purification Area

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

Catalyst Preparation Area

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

Storage/Utility Area

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

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, against the goals established by the Project Evaluation Plan for Budget Period No. 2 (see Appendix B). The status, and the 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 Significant Impact (FONSI). The draft final EIV was submitted on 31 Jan 1996.

- Obtain permits necessary for construction and operation.

  - The construction permits have been obtained.

Task 1.3 Design Engineering

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

- Prepare the Environmental Monitoring Plan (EMP).

  - The initial draft EMP was submitted on 9 October 1995. DOE's comments were received, and revised draft EMP was issued 27 February 1995. A meeting with DOE is scheduled on 25 April to review the EMP, and also the Demonstration Test Plan (see Task 2.3), since both plans are interrelated.

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

- Process Engineering work focused on:
  - Completing Design of C-120 Vent Scrubber
  - Piping Design Reviews
  - Documentation of Vent Header Design

- Engineering work is focused on:
  - Starting work on Pressure Testing definition
  - Working on Distributed Control System logic and documentation.
  - Writing Specification for Instruments
  - Continuing Work on Distributed Control System (DCS) logic and documentation.

- Design Work is focused on:
  - Completing the Mechanical Bid Package
  - Completing the Electrical Bid Package
  - Completing the Bid Package for Miscellaneous Buildings

Task 1.4 Off-Site Testing (Definition and Design)

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

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

Discussion

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

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

- The draft amendment to the 21 December 1992, contract between Air Products and Acurex Environmental Corporation (Acurex) was issued for consideration and approval. The amendment will incorporate the current Statement of Work and Milestone Schedule for the request.

- Air Products' Program Manager attended a seminar on fuel cell developments. Of particular interest was a presentation by DOE's Manager, Fuel Cell Systems R&D, Office of Transportation Technologies. Methanol is being considered in some of the longer-term developments for transportation vehicles. Methanol would be used as the on-board storable fuel, which would be reformed to provide the hydrogen for the fuel cell/electric powered vehicle. A fuel test plan outline was drafted, based on the concept of methanol coproduced at centrally located IGCC power plants providing liquid transportation fuel for local markets. The draft of this fuel test plan outline is included in Appendix C.

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 later 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).

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).

- 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) Initiated process design work of the Product Purification Options for three alternative grades of product: Chemical, MTBE, and Fuel.

  b) Completed work on a methanol production matrix for a given (e.g. Kingsport) liquid phase reactor volume. The results are included in Appendix D. These show that a given reactor size is capable of a wide range methanol production rates (100 TPD to 600 TPD for the Kingsport reactor size), dependent upon four inter-related process design variables. These design variables are i) reactor pressure, ii) inlet superficial velocity, iii) recycle ratio; and iv) percent Btu conversion. These interesting results of the Kingsport reactor's capability will be utilized in the next quarter to prepare the outline of the Process Economics Study.

  c) Reviewed the methanol production matrix and determined that percent Btu conversion for the Texaco-type synthesis gas was limited. This conclusion has led to the inclusion of other design parameters in the Process Economics Study, such as feed gas composition and water addition to the reactor feed gas. A comparison with gas phase reactor technology, which requires treatment of the reactor feed gas with shift and carbon dioxide (CO_2) removal equipment, will also be explored.

  d) Plans and resource commitments for Task 1.5.2 Commercialization Studies work in the next quarter were made.

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.

Action during this quarter included:

Market Economic Studies

No further work will be done, until Labratory R&D confirmation of a stable liquid phase DME (LPDME) catalyst system is obtained.

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 electric power and liquid fuels from an IGCC facility. The 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, 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 October - December 1995, is included in Appendix E for reference, and is summarized in the following:

Summary of Laboratory Activity and Results

- Stability of the DME catalyst system was greatly improved to near that of a LPMEOH™ catalyst system when a laboratory prepared alumina based dehydration catalyst was used. The productivity of this catalyst system is 30% higher than a normal LPMEOH™ system, but is still lower than the initial productivity of a standard LPDME catalyst so that the DME selectivity is rather low. The data show once again that there is a chance for a modification of the alumina to lead to long life.

- The new aluminum-based dehydration catalyst showed good stability in an LPDME run. The activity was low and the methanol catalyst deactivated. However, the results are interesting enough so that additional runs are planned. The ratio of Lewis to Bronsted acid sites can be changed for this catalyst so that it is an interesting probe catalyst for determination of the deactivation mechanism.

- In continued testing of candidate dehydration catalysts, mesoporous silica alumina (MCM-41) and hydrotalcite ($MgOAl_2O_3$) were tested. Neither were suitable dehydration catalysts. The hydrotalcite exhibited no dehydration activity while catalyst system using MCM-41 exhibited poor stability.

- Changing solvents may lead to a greater degree of dispersion of the two catalysts and thus a decrease in the interaction which causes deactivation. The use of perfluoropolyether (FOMBLIN Y HVAC) resulted in very poor catalyst life probably due to decomposition of the solvent.

- Two single component, dual functional catalysts which were obtained from BASF were tested. Copper oxide (CuO) on alumina gave high selectivity to DME at low rates with about the same stability as the standard DME catalyst system. Copper oxide (CuO) on magnesium silicate had low activity and showed little dehydration activity so that the main product was methanol.

- The use of a lanthanum oxide modified BASF methanol catalyst with the standard dehydration catalyst did not exhibit improved stability.

Task 1.5.4 Administration and Reporting

A project review meeting was held in Air Products' offices in Allentown, PA, on March 4th, and a trip was made to Joseph Oat Corporation in Camden, NJ, on March 5th, to observe the LPMEOH™ reactor fabrication. Attendees from Air Products and DOE participated. The meeting notes, agenda, and some of the meeting handouts are included in Appendix F. The project status was reviewed. Detailed design is 85% complete. The general mechanical construction package is out for bid, and the instrument and electrical construction bid package was released March 25th. Construction on steel and equipment erection has started (see photo in Appendix F). The Environmental Monitoring and Demonstration Test Plans were reviewed, and plans were made for a review meeting with DOE in April. A visit to Air Products Research Lab in Iron Run was made, where the DME catalyst life testing research is being done. The status of the DME laboratory Research (Task 1.5.3) was reviewed. The Alternate Fuels Field Development Unit (AFFDU) trailer, which is being prepared for a synthesis gas catalyst poisons study at Kingsport in May 1996, was also visited.

The Milestone Schedule Status and the Cost Management reports, through March 31, 1996, are inlcuded in Appendix G. The demonstration unit is scheduled to be mechanically complete in November of 1996. Fifty-two percent (52%) of the $36 million of funds authorized for the Kingsport portion of the LPMEOH™ Process Demonstration Project through Budget Period No. 2 have been expended, as invoiced through 31 March 1996.

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

Task 2.1 Procurement

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

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

  - All Equipment except for the C-120 Vent Scrubber have been purchased.

Reactor Status

- The reactor fabrication continues at Joseph Oat Corporation in Camden, NJ. Some time was lost during this quarter due to required weld repairs on a few of the shell circumferential welds. Oat also encountered problems using an orbital welder to weld the two sections of the internal heat exchanger tubes in the middle. The weld procedure was revised to achieve an acceptable weld. Air Products assigned a full time inspector at Joseph Oats' shop to expedite the order and insure that good quality is maintained.

  The vessel shell and heads were post weld heat treated at the beginning of March prior to inspection of the internal heat exchanger.

  The DOE joined Air Products for a progress review meeting at Joseph Oat's shop on 5 March. The reactor ship date has slipped to 3 May 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 added a lead Mechanical Superintendent to the site construction management staff in late January. His main area of concentration will be working with the Structural Steel Erector who started construction work on 29 January 1996 and then the Mechanical Contractor.

- 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 was completed at the end of 1995. This included initial grading and covering with gravel, soil fill and compaction on the north side of the job site, and installation of the storm sewers. The foundation and underground work was completed on schedule at the end of January 1996. This included installation of all major structural steel and equipment foundations, installation of tank farm dikes, process area slabs, underground drain lines, the oil-water separator and underground electrical grounding.

- On-site fabrication of the large product methanol lot tanks started in November 1995 and was completed in December prior to erecting on their foundations in March 1996.

- Erection of the Pipe Rack area structural steel and equipment items east of the process building started 29 January. This work was awarded to Spartan Constructors of Atlanta, GA. There is approximately 47 tons of steel to erect east of the process structure. The process building steel is scheduled to begin arriving on site in May, therefore, construction work will concentrate on the east half of the plant. This contract was 22% complete as of the end of March.

- The reactor and the process building structural steel deliveries have slipped to the end of May. This will delay completion of construction for a major section of the demonstation unit.

- Prefabricated large bore piping was sent out for bidding at the end of November 1995. This work was awarded to Connex of Troutville, VA in mid-December. Initial release of piping spools drawings to Connex began on 5 January 1996. The large bore piping should be delivered to the job site to support construction.

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

  - The Mechanical Completion date has slipped two weeks to the end of November 1996. This is due to the late reactor and structural steel delivery dates. Air Products is investigating ways to expedite delivery of these items and minimize their impact. The revised estimated start-up date is 27 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.

  - A second draft of the Demonstration Test Plan (DTP) for Phase 3, Task 2 Operation was issued for review and comment on 29 March. A copy of the cover letter and of Table 5-1 - Operation Test Plan (4 pages) is included in Appendix H. A meeting with DOE is scheduled for 25 April to review the DTP, and also the Environmental Monitoring Plan, since both plans are interrelated. 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.

  - Eastman began preparing a rough draft of the LPMEOH™ Standard Operating Procedures and developed a plan for the start-up team. The operator training program outline was also being prepared and the schedule for the start of operator training was tentatively set for October of 1996.

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 (Definition and Design).

Task 2.5 Planning and Administration

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

- Prepare annually an updated (Partnership) plan for the remaining activities. The first annual plan will update the remaining Phase 1 and Phase 2 activities, and the second will include an updated Phase 3 Operating 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 fiscal year 1996 are:

    - the LPMEOH™ 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 1.5.4 - Administration and Reporting.

E. Planned Activities for the Next Quarter

- Complete the detailed engineering design and procurement.
- Continue shipment of equipment to the site. Expedite reactor fabrication, ship reactor by rail, off-load and transport the reactor to the site.
- Continue erection of equipment and structural steel.
- Issue and award the Instrument and Electrical Construction bid package and start work.
- Issue revised drafts of the Demonstration Test Plan and of the Environmental Monitoring Plan. Conduct a review meeting of both plans in late April with DOE. Issue final drafts of both plans.
- Hold a Project Review/Update meeting at the site in June.
- Complete Part One of the Process Economics Study; on co-production of methanol with IGCC power.
- Issue updated fuel-use test program plan.

F. Summary

Construction work for the LPMEOH™ demonstration unit began in October of 1995. The foundation and underground work was completed in January of 1996. The erection of the pipe rack steel and equipment items has begun, and piping installation in the pipe rack area should begin in April.

The fabrication of the reactor continues, and is being expedited. The reactor ship date has slipped to 3 May 1996.

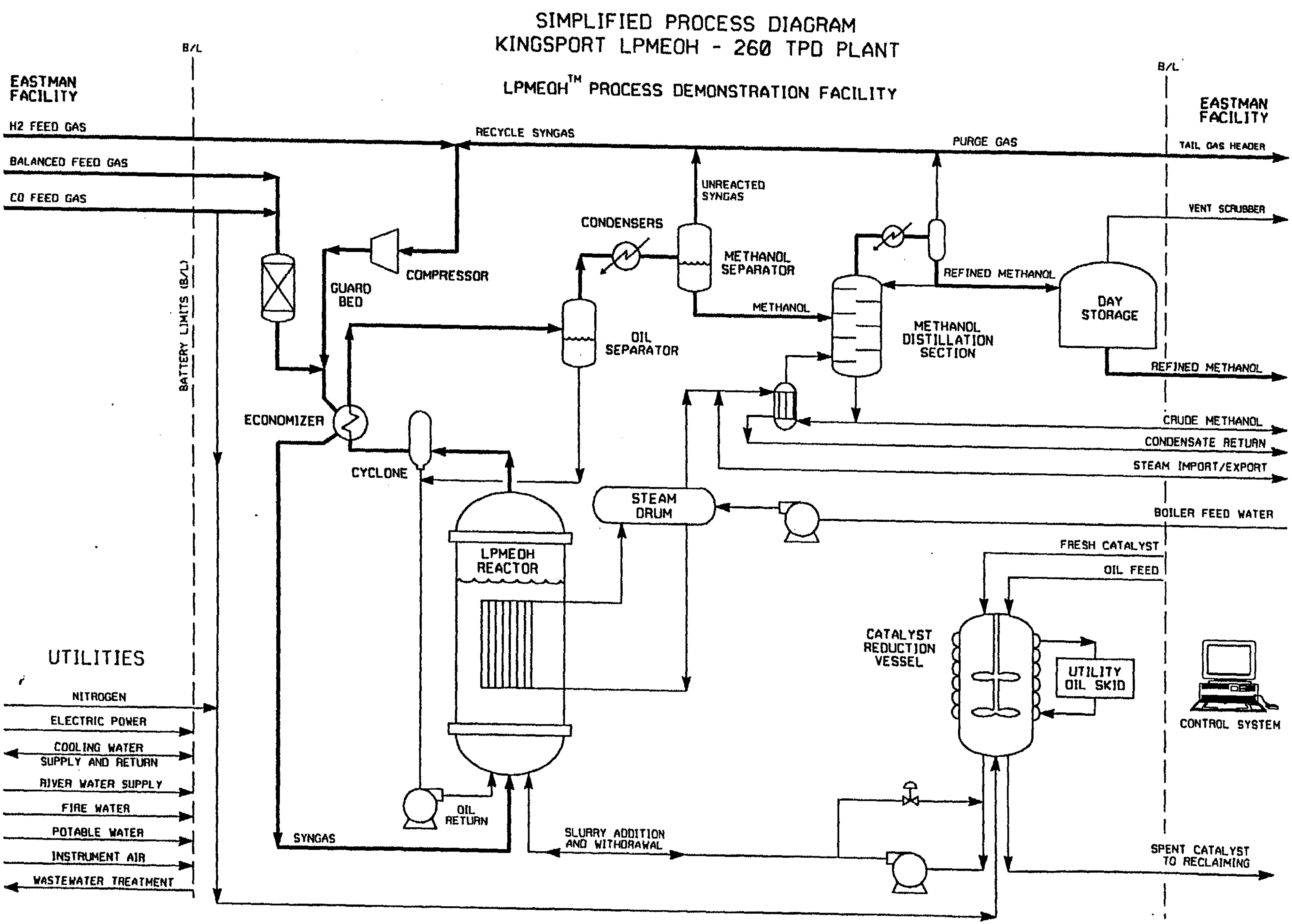
A draft amendment to the off-site product-use testing subcontract between Acurex Environmental Corporation and Air Products has been prepared. The amendment incorporates the current Statement of Work and milestone schedule for the project.

DOE's comments on the draft Environmental Monitoring Plan (EMP) and on the draft Demonstration Test Plan (DTP) were received. Revised EMP and DTP drafts were prepared, and a meeting to review and finalize both is planned for April with DOE.

Procurement of process equipment is essentially complete and construction work is well underway. Mechanical completion has slipped two weeks due to late reactor and structural steel delivery dates. Commissioning work is expected to start in mid-October, with plant start-up in late December of 1996. Fifty-two percent (52%) 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 March 1996.

APPENDICES

APPENDIX A - SIMPLIFIED PROCESS FLOW DIAGRAM


SIMPLIFIED PROCESS DIAGRAM
KINGSPORT LPMEOH - 260 TPD PLANT
LPMEOH™ PROCESS DEMONSTRATION FACILITY
B/L
EASTMAN FACILITY
H2 FEED GAS
BALANCED FEED GAS
CO FEED GAS
BATTERY LIMITS (B/L)
RECYCLE SYNGAS
PURGE GAS
TAIL GAS HEADER
UNREACTED SYNGAS
VENT SCRUBBER
CONDENSERS
METHANOL SEPARATOR
REFINED METHANOL
COMPRESSOR
GUARD BED
DAY STORAGE
METHANOL
OIL SEPARATOR
METHANOL DISTILLATION SECTION
ECONOMIZER
CRUDE METHANOL
CONDENSATE RETURN
STEAM IMPORT/EXPORT
CYCLONE
STEAM DRUM
BOILER FEED WATER
FRESH CATALYST
OIL FEED
LPMEOH REACTOR
CATALYST REDUCTION VESSEL
UTILITY OIL SKID
UTILITIES
NITROGEN
ELECTRIC POWER
COOLING WATER SUPPLY AND RETURN
RIVER WATER SUPPLY
FIRE WATER
POTABLE WATER
INSTRUMENT AIR
WASTEWATER TREATMENT
CONTROL SYSTEM
OIL RETURN
SYNGAS
SLURRY ADDITION AND WITHDRAWAL
SPENT CATALYST TO RECLAIMING

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

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

PROJECT EVALUATION PLAN FOR BUDGET PERIOD NO. 2

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

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

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

   - Obtain permits necessary for construction and operation.

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

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

   - Prepare the Environmental Monitoring Plan.

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

Fuel Use Test Program. Draft for Discussion.

1. Premium Methanol Fuel Applications

- At 40 cents per gallon, methanol as a fuel ($6.00 per mmBtu) will not compete with oil in most applications ($20/bbl crude = $3.30/mmBtu; $24/bbl diesel = $4.00 /mmBtu). However, methanol coproduced at a central IGCC power station, may be a valuable premium fuel for two evolving developments: as an economical Hydrogen source for small fuel cells, and as an environmentally advantaged fuel for dispersed electric power.

- "Central clean coal technology processing plants, making coproducts of electricity and methanol; to meet the needs of local communities for dispersed power and transportation fuel" - meets the DOE Clean Coal Technology Program's objectives. Serving (initially) small local fuel markets also builds on LP's (the LPMEOHTM process) strengths; good economics at small methanol plant sizes, fuel grade product distillation savings, and a freight advantage in local markets vis a vis large off-shore remote gas methanol. Baseload methanol coproduction studies show that (40) cent per gallon methanol can be provided from an abundant, non-inflationary local fuel source.. *We need to show when (at what oil price) we can compete, and to arrange fuel tests to confirm the dispersed energy environmental advantage.*

1.1. Hydrogen Source for Fuel Cells

- Hydrogen fuel cells, being developed for transportation applications, can achieve 65% system efficiency, as compared to 45% for diesel IC engines and 32% for gasoline IC engines. Methanol is a storable, transportable liquid fuel which can be reformed under mild conditions to provide H2. For small H2 applications, *and at low utilization factors,* methanol reforming is a more economical source of hydrogen than : a) natural gas reforming, b) distillate (oil) reforming; and is cheaper than LH2.

1.1.1. Fuel Cells for Transportation

1.1.2. Fuel Cells for Stationary Power

(See also dispersed power below).

1.1.3. Small Hydrogen Applications

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

1.2. Dispersed Power

- Dispersed power is getting a lot of favorable publicity. . The world wide package (0.2 MW to 10 MW) power plant market is large. A variety of technologies (combustion turbine, internal combustion engine, fuel cell) are being packaged to provide power and heat locally, at the use point. Environmental and Economic advantages include Methanol for Fuel Cells = clean stationary local power; no need for natural gas pipelines; no new high voltage power lines.

1.3. Dimethyl Ether as an Enhancement to Methanol in Premium Fuel Applications

Can coproduced mixtures of methanol and dimethyl ether improve upon methanol, in the above?

APPENDIX D - TASK 1.5.2 - METHANOL PRODUCTION MATRIX

Assumptions
for
Methanol Production Matrix

- Same size reactor as at Kingsport
  (7.5 foot inside diameter, 76 foot tangent-tangent length)

- "Texaco-Type" coal gas fresh feed
  (35% H_2, 51% CO, 13% CO_2, 1% inerts)

- Aged catalyst, with addition and withdrawal

- Catalyst slurry concentration of 45 wt % (oxide basis)

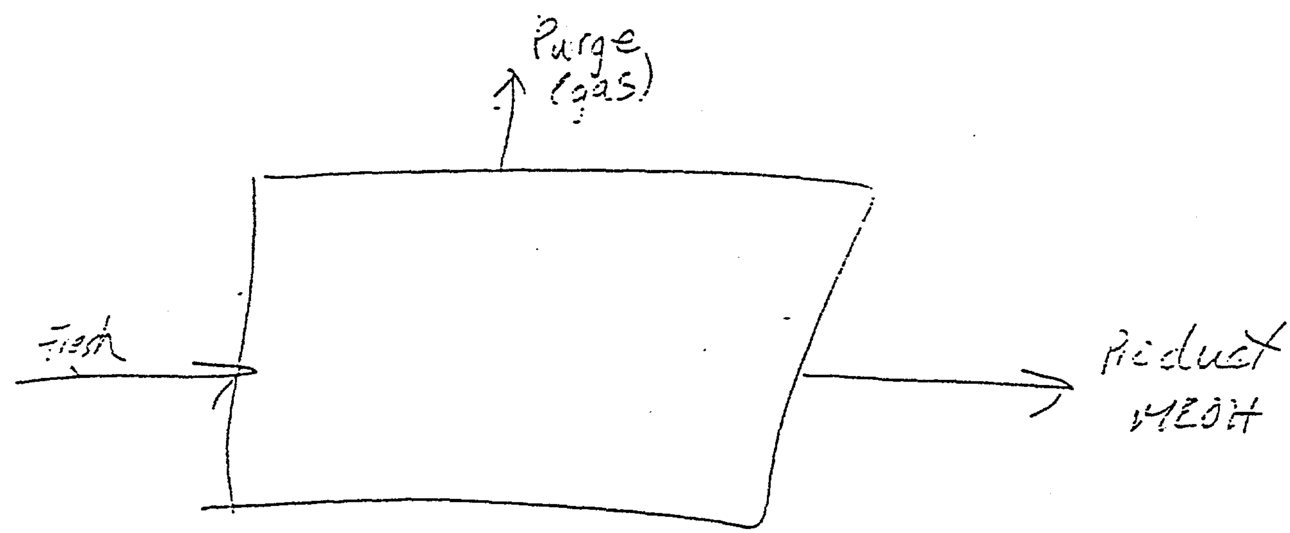
- Gassed Slurry Level maintained at 65.5 feet

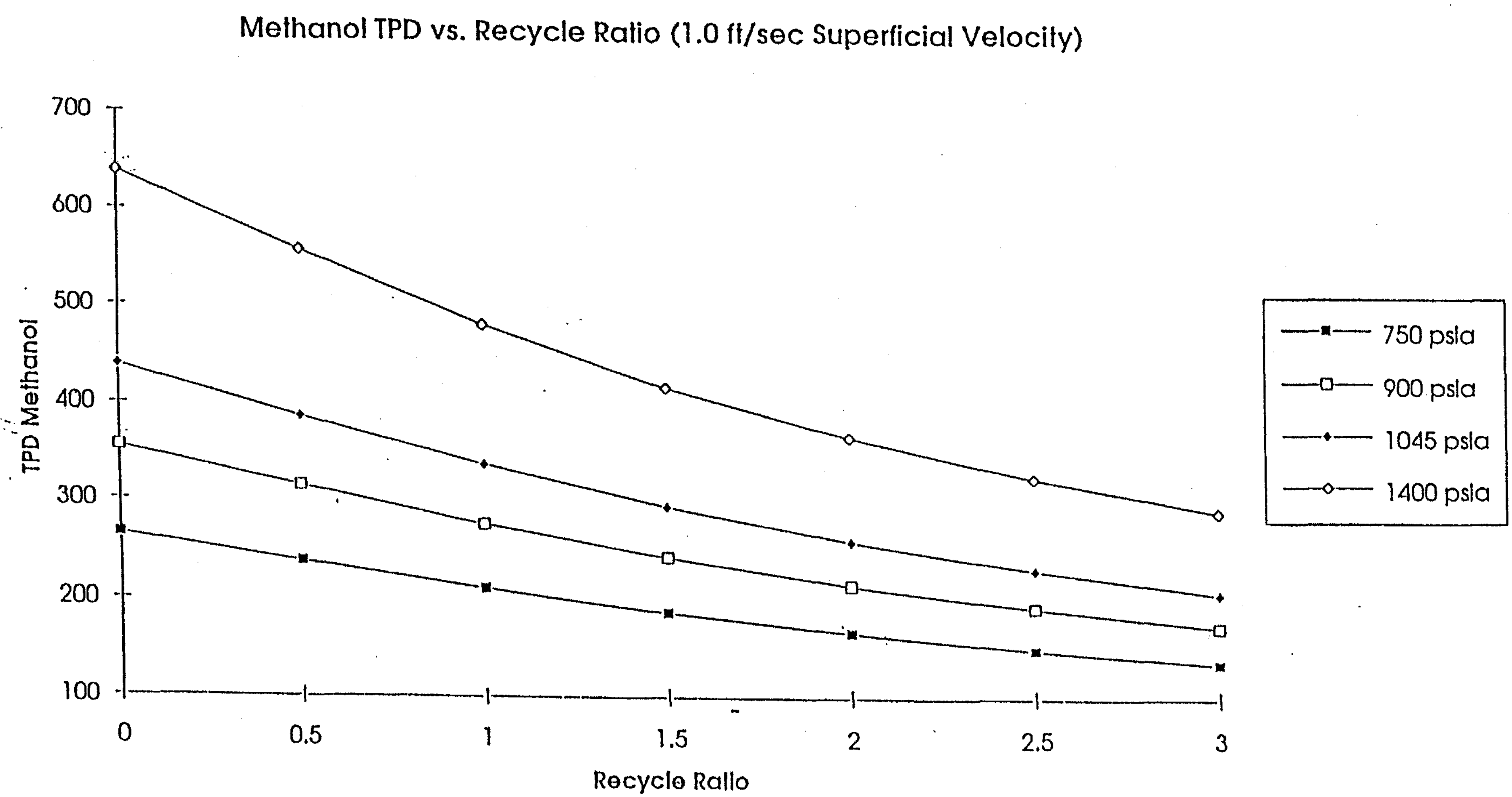
- Inlet Superficial Velocity; 0.6, 0.8, 1.0 foot per second

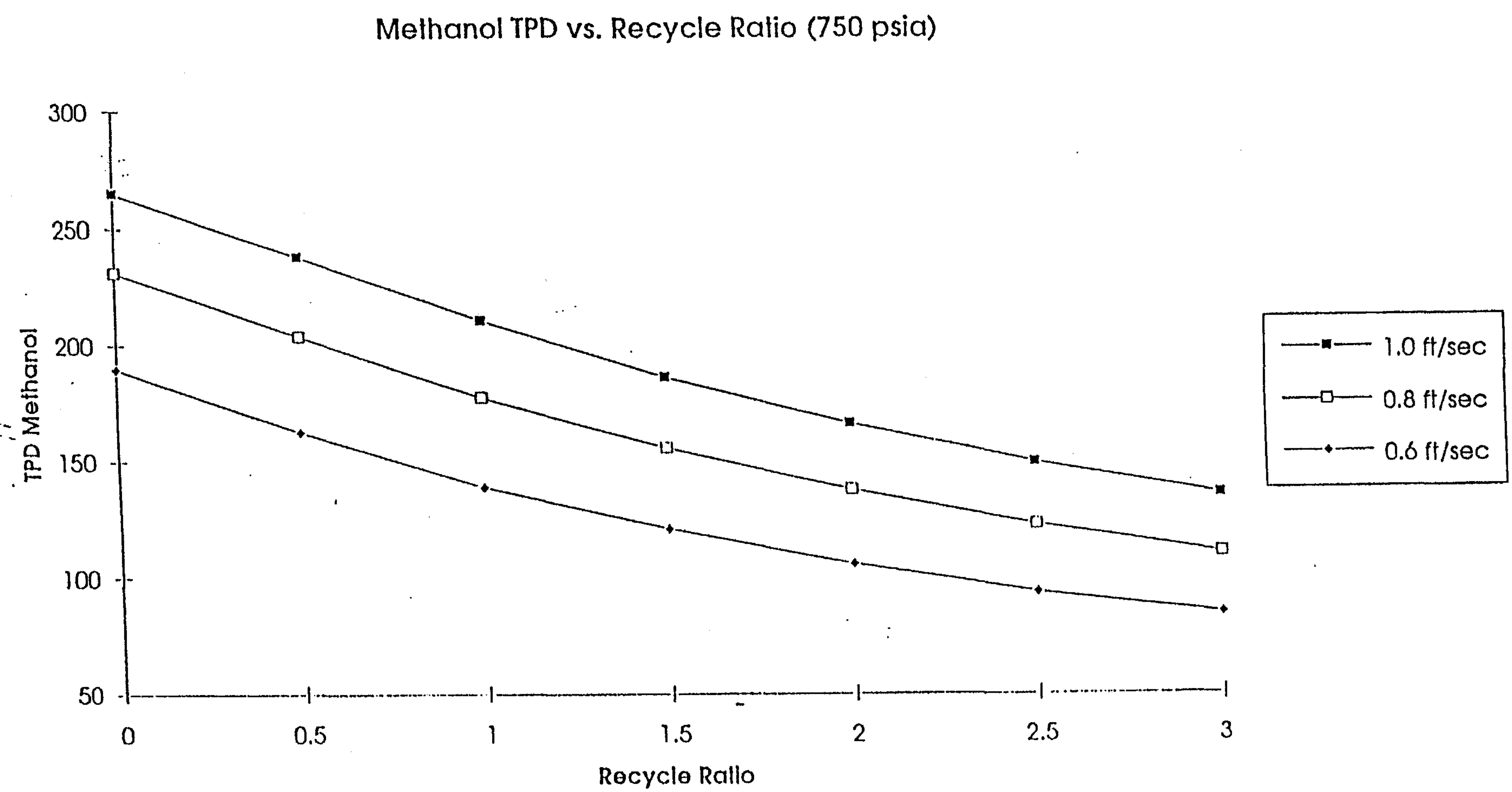
Notes: These Kingsport reactor design constraints: a) maximum operating pressure (about 1045 psia), and b) heat removal (size per about 300 TPD of methanol) would require changes for certain of cases.

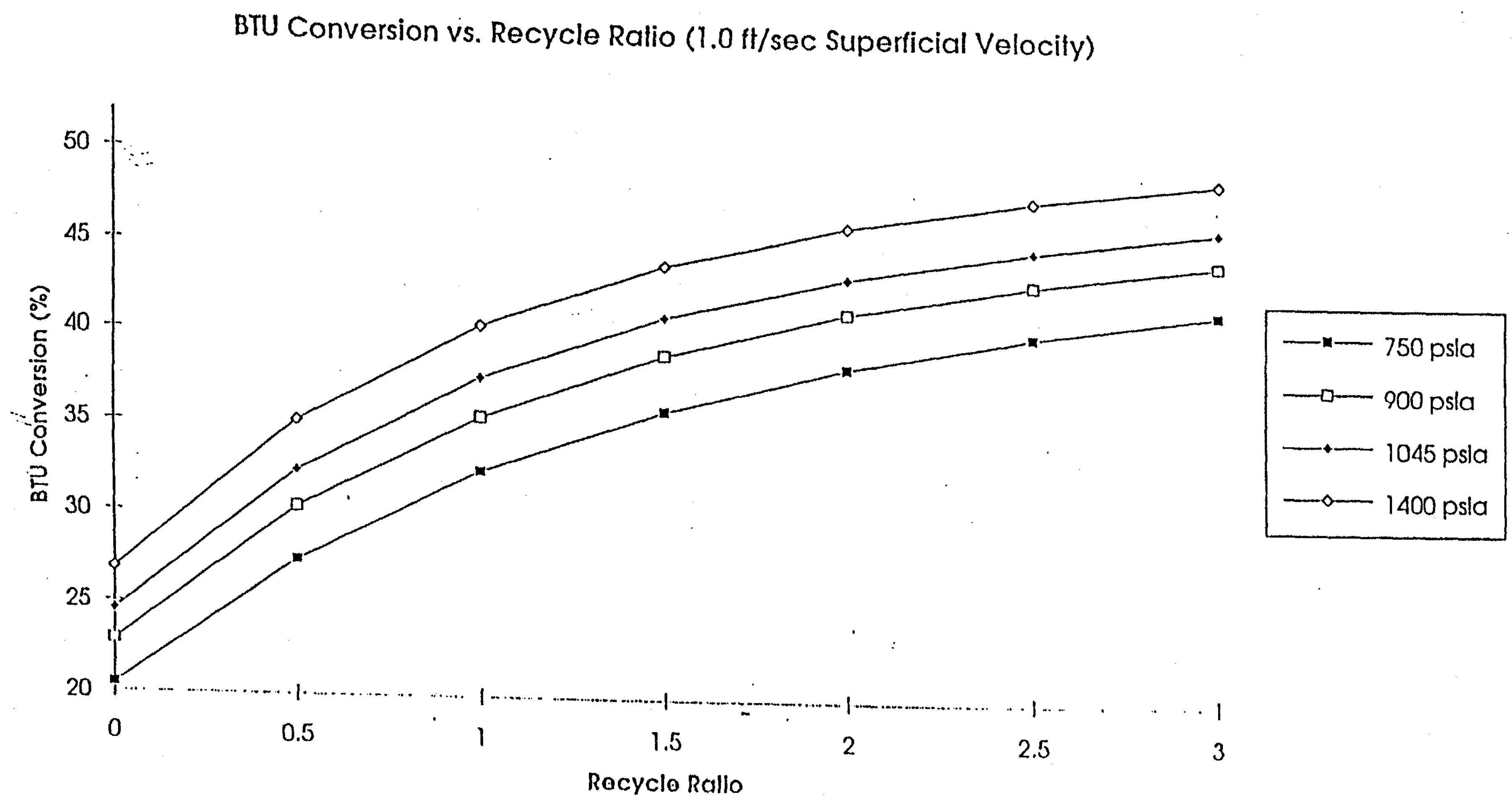
Glossary of Terms

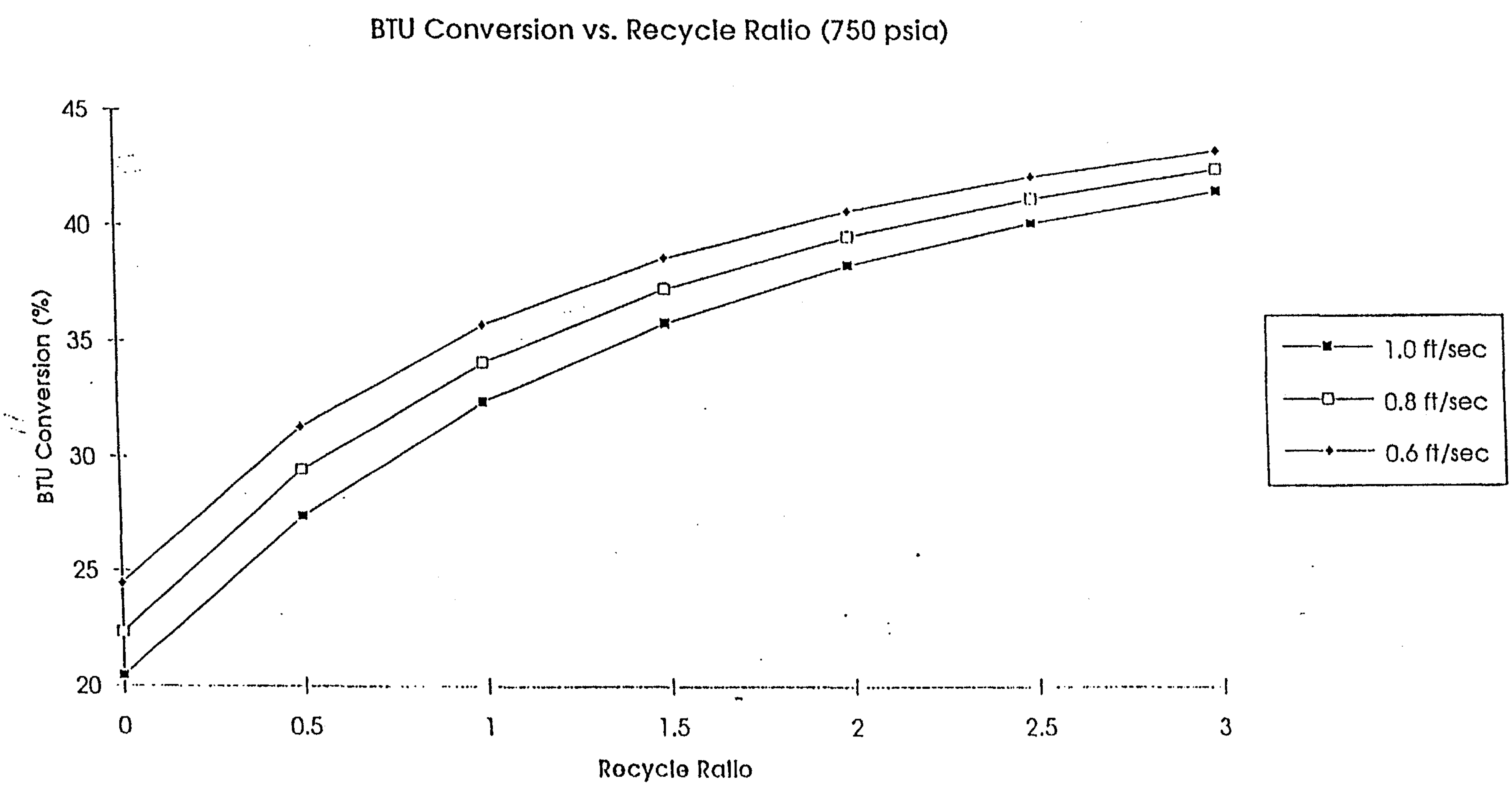
BTU Conversion	Difference of the lower heating value LHV of the fresh feed and the LHV of the gas purge stream divided by the LHV of the fresh feed.
Catalyst Age	Described in terms of the ratio of the catalyst rate constant at a given time to the rate constant of fresh catalyst; this is a design parameter used to set the catalyst addition/withdrawal frequency for a given methanol production rate.
Gassed Slurry Level	Level of gassed slurry; one of the major process control loops; typical units are feet.
Inlet Superficial Velocity	Ratio of the actual cubic feet of gas at the reactor inlet to the reactor cross-sectional area (excluding the area contribution by the internal heat exchanger); typical units are feet per second.
KSCF	Acronym for 1,000 standard cubic feet; for this term, standard conditions are 14.7 psia and 60°F (379.43 scf/lbmol).
LHV	Lower heating value
Slurry Concentration	Percentage of weight of slurry (solid + liquid) which is catalyst. Catalyst weight is defined on an oxide (unreduced) basis.




Methanol TPD vs. Recycle Ratio (1.0 ft/sec Superficial Velocity)
TPD Methanol
700
600
500
400
300
200
100
0
0.5
1
1.5
2
2.5
3
Recycle Ratio
750 psia
900 psia
1045 psia
1400 psia


Methanol TPD vs. Recycle Ratio (750 psia)
TPD Methanol
300
250
200
150
100
50
0
0.5
1
1.5
2
2.5
3
Recycle Ratio
1.0 ft/sec
0.8 ft/sec
0.6 ft/sec


BTU Conversion vs. Recycle Ratio (1.0 ft/sec Superficial Velocity)
BTU Conversion (%)
50
45
40
35
30
25
20
0
0.5
1
1.5
2
2.5
3
Recycle Ratio
750 psia
900 psia
1045 psia
1400 psia



BTU Conversion vs. Recycle Ratio (750 psia)
BTU Conversion (%)
45
40
35
30
25
20
0
0.5
1
1.5
2
2.5
3
Recycle Ratio
1.0 ft/sec
0.8 ft/sec
0.6 ft/sec

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

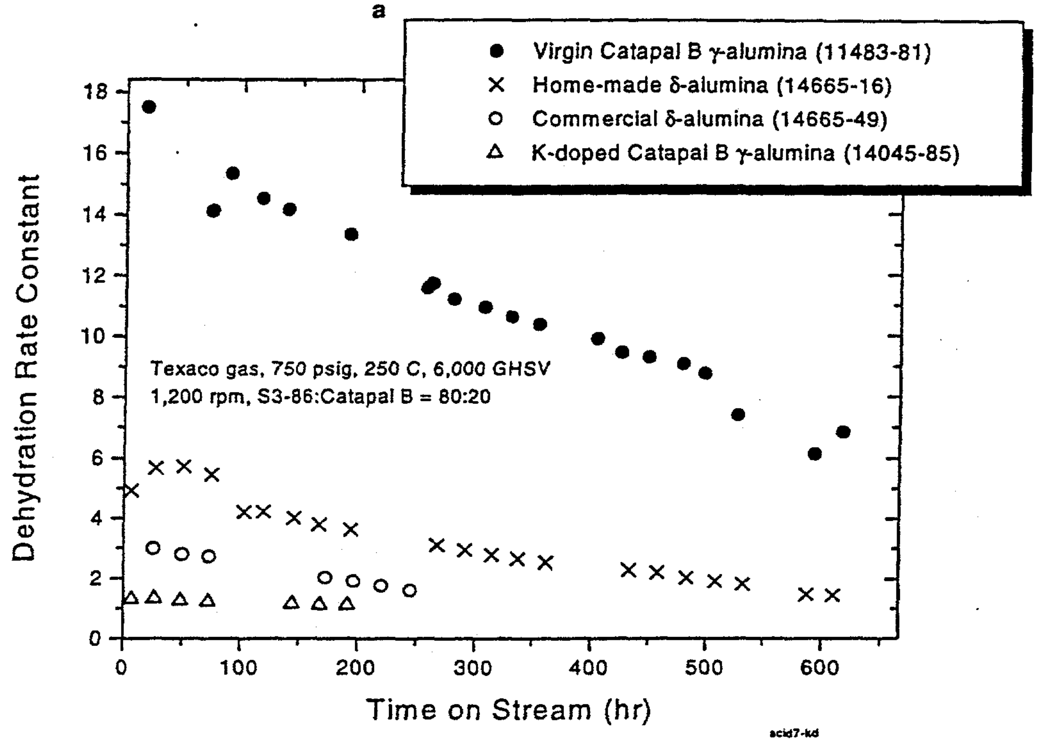
DME R + D QUARTERLY

A new alumina sample, # 14656-54, was tested this quarter. It was prepared by sililyzing Catapal B g-alumina with tetraethyl orthosilicate ($Si(OC_2H_5)_4$), followed by calcination at 1000°C. The XRD pattern of the sample shows that it is a mixture of d- and g-alumina. This sample was tested along with BASF S3-86 methanol catalyst in a standard LPDME run (14665-16) using Texaco gas. The results from the run are depicted in Figures 3.1.6 and 3.1.7 against those from virgin g-alumina and the two dehydration catalysts mentioned above. Figure 3.1.6a shows that the initial dehydration activity of the sample is still low compared to virgin Catapal B g-alumina, but higher than the other two dehydration catalysts. The catalyst deactivates continuously with time on stream, but at a rate lower than the virgin g-alumina. Figure 3.1.6b shows that the long-term stability of the methanol catalyst is improved. Namely, the methanol catalyst deactivated rapidly for the first 170 hours on stream, then decreased to a rate 33% lower than that in the standard dual catalyst system. This improved dehydration activity and long-term methanol catalyst stability results in a catalyst system with promising stability and activity. As shown in Figure 3.1.7, the overall long-term stability of this system is similar to a LPMEOH run, and its long-term productivity is significantly higher than that of a LPMEOH run (by ~30%). However, the DME-to-methanol molar ratio from this system after stabilization is 1:7.7, much lower than that from the standard catalyst system (changing from 2:1 to 1:1 with increasing time on stream).

As in the other two catalyst systems that have exhibited better long-term stability, dehydration activity is still the bottleneck in the current catalyst system. Increasing the reaction temperature to 255 and 265°C, as shown in Figure 3.1.8, did not result in increased productivity. Apparently, the increased rate constants for both reactions were not large enough to compensate for the more unfavorable equilibrium conditions for the methanol synthesis reaction at higher temperatures.

The stability of both catalysts in this system is also not sufficiently satisfactory. The methanol catalyst deactivated at a rate greater than that in a LPMEOH run, and the alumina deactivation was low but continuous. This indicates that detrimental interaction between the two catalysts still exists. A LPDME run (#14665-29) was conducted at a lower temperature (240°C) using a similar laboratory-prepared d-alumina (Sample #14656-72). At this temperature, the alumina was very stable, and the long-term stability of the methanol catalyst was only 10-15% poorer than in a LPMEOH run. However, the productivity suffered (18 mol/kg-hr) because of the lower temperature.

Figure 3.1.6 The Stability of Different Catalyst Systems under LPDME Conditions



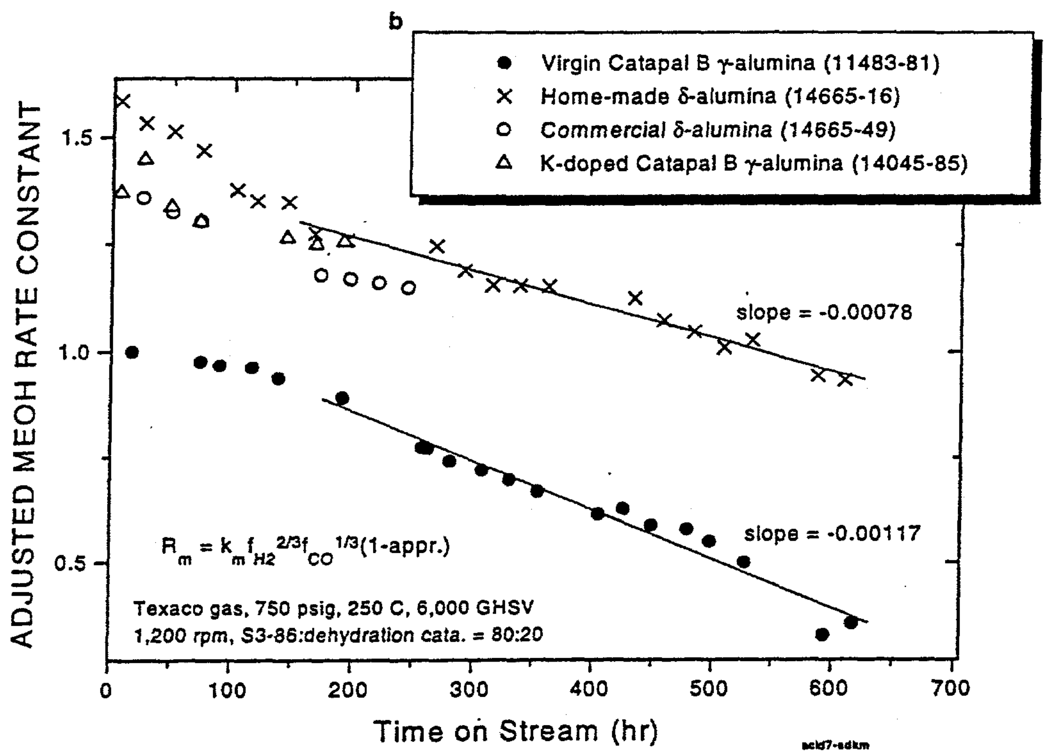


Figure 3.1.7 The Methanol Equivalent Productivity of Different Catalyst Systems

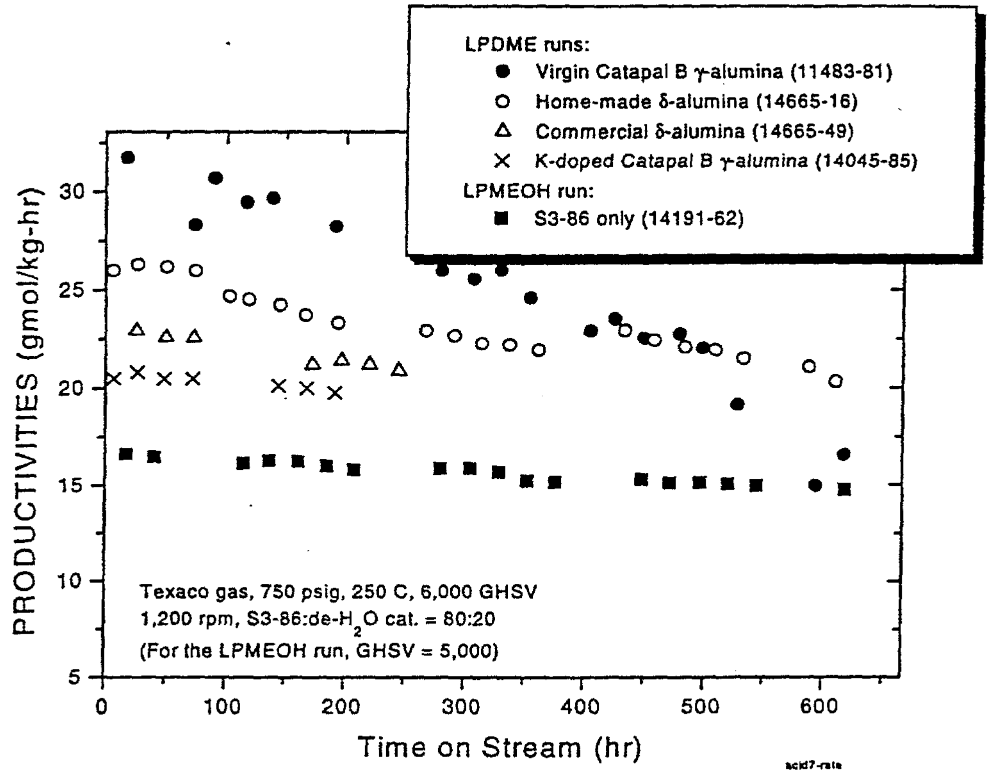
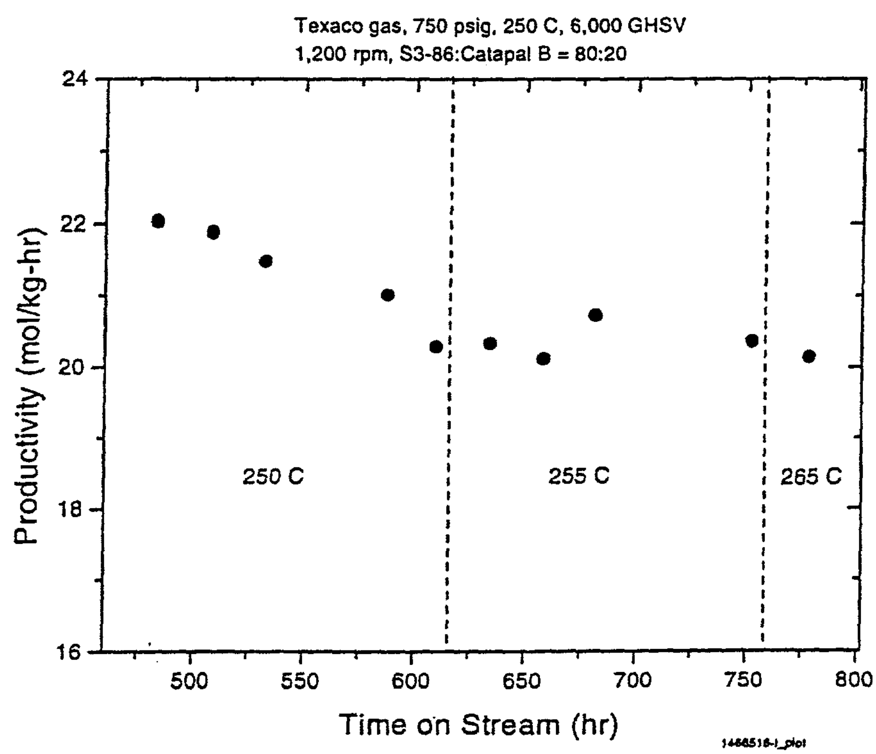


Figure 3.1.8 Methanol Equivalent Productivity as a Function of Temperature



3.1.3. Other Catalyst Systems

Mesoporous Silica Alumina

A mesoporous silica alumina (MCM-41) was tested as an alternative dehydration catalyst for several reasons. First, it has a mesoporous structure, different from the macroporous materials examined most frequently, such as g-alumina. Second, the particle size of the sample powder is very small; in fact, the powders could not be observed once they were in the slurry oil. Therefore, this system is likely to have better intimate contact between the two catalysts, and little mechanical impact on the methanol catalyst powders through collisions. We wanted to determine how these differences impact the deactivation pattern of the catalyst system.

The run was conducted under standard conditions (Run #14656-66). As shown in Figure 3.1.9a, both initial and long-term deactivation of the methanol catalyst in this system was faster than in the standard catalyst system. The long-term deactivation of the dehydration catalyst was also very rapid. (The initial deactivation of the dehydration catalyst could not be determined from the available data.) These results may serve as further evidence of the negative effect of intimate contact between the two catalysts.

Hydrotalcite

A hydrotalcite (i.e., mixed MgO-Al_2O_3 oxide) sample was obtained from LaRoche, and was calcined at 500°C prior to use. This material is essentially a solid base, and did not show any activity toward methanol dehydration (Run # 14656-69).

La_2O_3-Modified g-Alumina

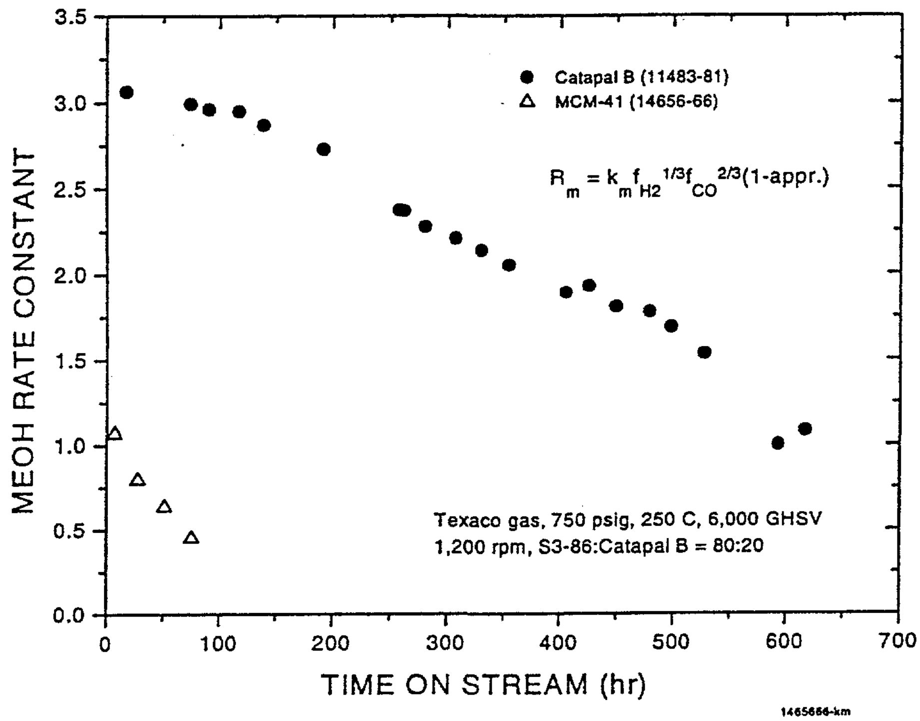
A La_2O_3-modified g-alumina (Sample #14656-30) was prepared by impregnating Catapal B g-alumina with a lanthanum nitrate aqueous solution to incipient wetness, followed by drying at 90°C and calcination at 560°C to convert lanthanum nitrate into oxide. The La_2O_3 loading was 34 wt %, corresponding to 88% of a monolayer coverage. This sample was tested in a 300 cc autoclave under the standard LPDME conditions using Texaco gas (Run # 14656-36). Possibly due to the high loading used, the sample did not exhibit any dehydration activity. The interesting observation from this run was that, despite the lack of dehydration activity of this sample, the methanol catalyst deactivated in this 76 hour run at least as fast as in the run using virgin g-alumina. This shows again that other factors in addition to the acidity of the dehydration catalyst may play a role in the deactivation of the methanol catalyst.

La_2O_3-Modified BASF S3-86

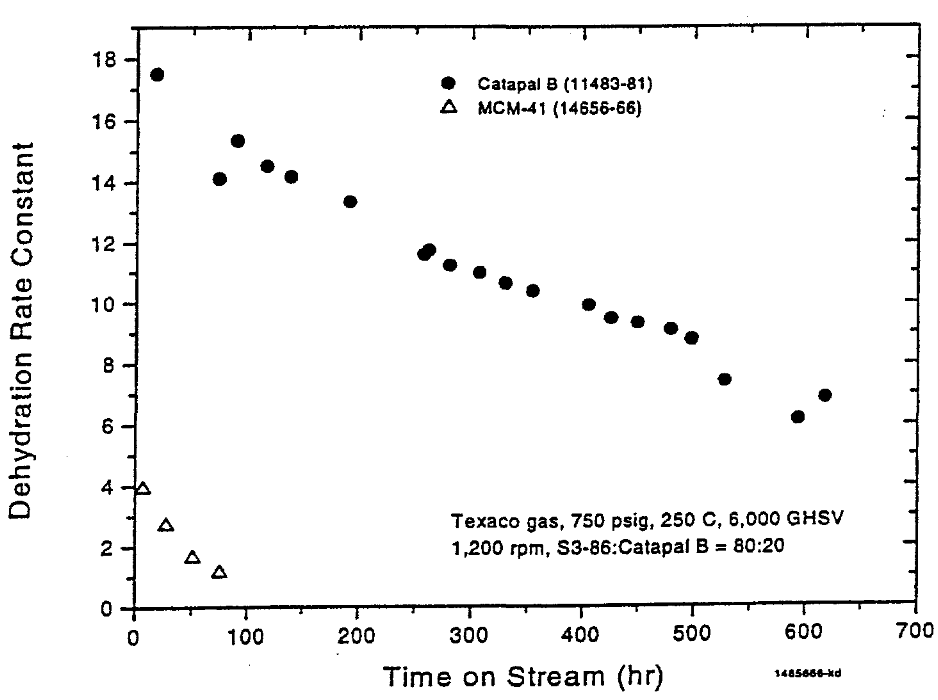
The BASF S3-86 methanol catalyst was impregnated with lanthanum nitrate, followed by calcination at 350°C to convert lanthanum nitrate into the oxide (Sample #13467-69). This modification was attempted to improve the stability of the methanol catalyst. A LPMEOH run (14656-72) showed that the modification caused a 15% decrease in the activity of the methanol catalyst; however, the catalyst was stable. A LPDME run (14656-74) followed using standard conditions with Shell gas as the feed and Catapal B g-alumina as the dehydration catalyst. During the LPDME run, the catalyst system exhibited much worse stability than the standard

Figure 3.1.9 The Stability of the Catalyst System Containing MCM-41

a



b



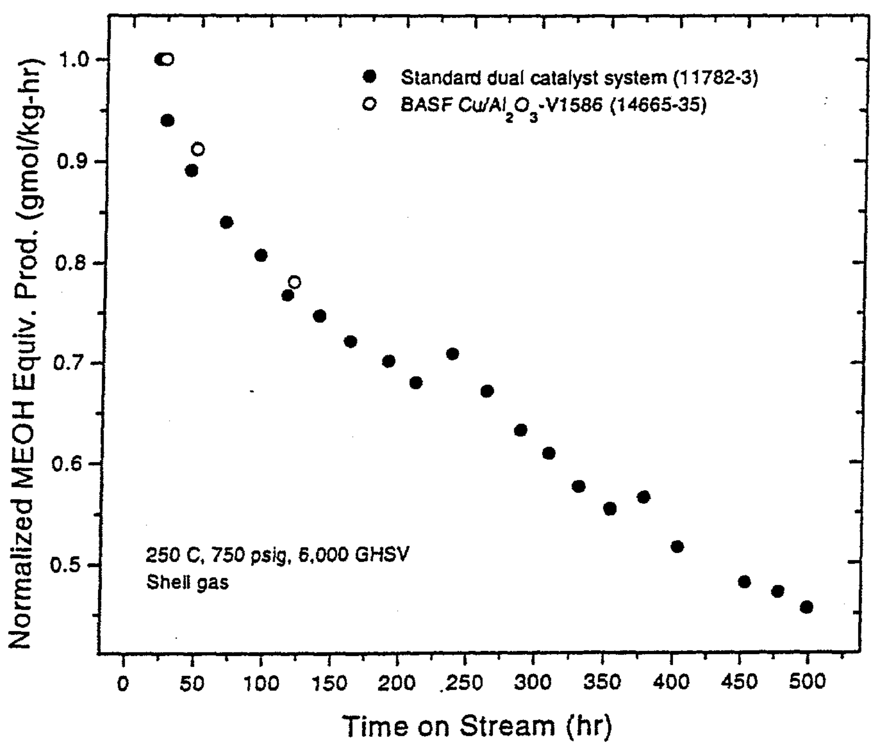
dual catalyst system (virgin S3-86 plus Catapal B g-alumina). Both catalysts suffered a large initial deactivation. Their long-term deactivation was similar to the standard dual catalyst system.

Dual Function DME Catalysts from BASF

A dual function DME catalyst is defined as one that performs both methanol synthesis and methanol dehydration functions, but is not a physical mixture of a methanol catalyst and a dehydration catalyst. Dual function DME catalysts are being considered as one of the options to solve the catalyst stability problem for the LPDME process. Three copper-based catalysts from BASF were examined: CuO on alumina (V1586), CuO on magnesium silicate (V1585), and copper-chromite on titania (V1583). All samples were reduced using 2% H_2 in N_2 and the standard heating ramp.

Under the typical reaction conditions (250°C, 750 psig, 6000 GHSV) using Shell gas, the initial productivity of Sample V1586 was 6.1 mol/kg-hr, one-fifth of the initial activity of a LPDME run using the standard dual catalyst system. The concentration of DME and methanol in the effluent was 1.14 and 0.15 mol%, respectively. This catalyst has a strong dehydration activity , but a low methanol synthesis activity. The stability of the catalyst is plotted in Figure 3.1.10 against that of the standard dual catalyst system in terms of the normalized methanol equivalent productivity. It can be seen that the stability of this catalyst is similar to that of the standard dual catalyst system.

Figure 3.1.10 Stability of Different Catalyst Systems



Sample V1585 exhibited both low methanol synthesis and methanol dehydration activity. The DME and methanol concentrations in the effluent were 0.24 and 1.7 mol%, respectively, corresponding to a methanol equivalent productivity of 5.7 mol/kg-hr. Increasing the reactor temperature to 270°C increased the DME and methanol concentration to 0.93 and 2.2%, respectively, and the productivity to 10.6 mol/kg-hr. After 67 more hours on stream at 270°C, the productivity had dropped to 3.8 mol/kg-hr. In brief, the activity, DME selectivity, and stability of this catalyst do not make it a promising one-component DME catalyst.

Sample V1583 did not show any hydrogen uptake during the reduction, suggesting that copper in this catalyst either is in the reduced form or cannot be reduced below 250°C. In any event, the catalyst showed no activity toward methanol synthesis and dehydration.

3.1.4 *Alternative Slurry Fluids*

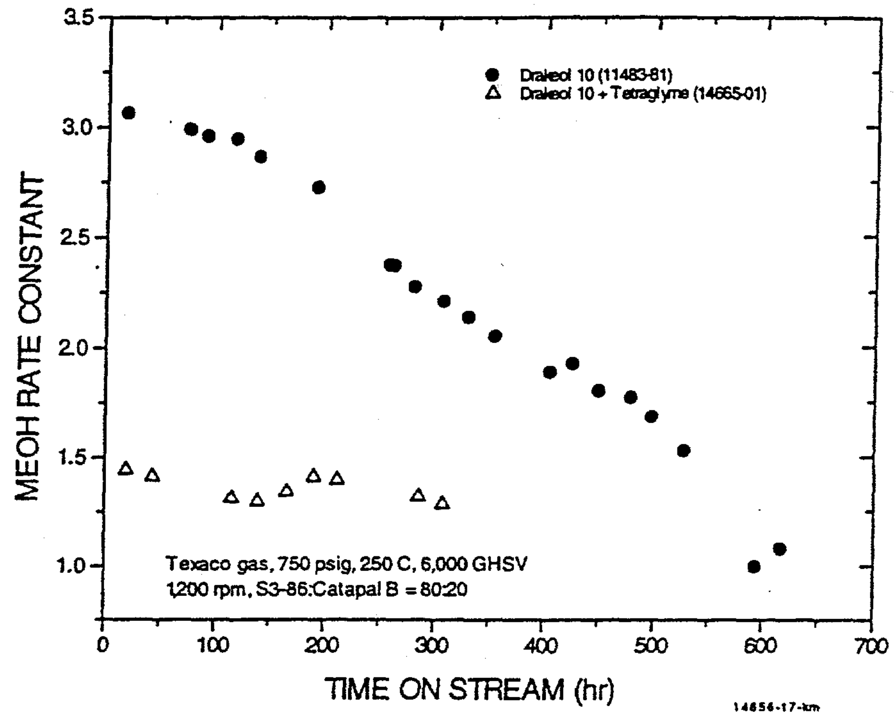
In the following experiments we attempted to determine if a different solvent would diminish the interaction between methanol synthesis and methanol dehydration catalysts in a dual catalyst system, thus improving catalyst stability. Tetraglyme (tetraethylene glyco dimethyl ether) is a hydrophilic solvent. It has been previously tested for LPMEOH in our lab (RRRS report by Hsiung et al., 1990) and showed performance similar to hydrocarbon oils. We replaced one third of Drakeol 10 oil, our current slurry fluid, with tetraglyme in a LPDME run (14656-1) to see if addition of a hydrophilic solvent to the slurry would better disperse catalyst powders, resulting in less contact between them and greater stability. The standard dual catalysts, S3-86 plus virgin Catapal B g-alumina, and reaction conditions were used in this run.

Hydrogen uptake during the reduction in this run was slower than in a standard run using Drakeol 10 oil only, and the final H_2 uptake was smaller (2.1 vs. 3 scf/lb). As shown in Figures 3.1.11a and b, the initial activity of the catalyst system, especially the alumina, was much lower than that in a standard run. However, the catalysts did not deactivate further with time on stream; essentially, a stable productivity was observed (not shown). When the autoclave was taken apart at the end of the run, some differences from a standard run were observed. First, the catalyst mixture looked brownish, instead of black in a standard run. Second, more clumps of catalyst stuck on the stirrer and the walls of the reactor. Third, the slurry recovery was about one third lower. These observations indicate that tetraglyme is not stable under the reduction and LPDME conditions. Most likely it underwent both decomposition and polymerization in the presence of g-alumina. This is evidenced by a much higher production of ethanol, a likely decomposition product, in the early stage of the run. Apparently, the fast, initial deactivation of both catalysts is related to the degradation of tetraglyme. For the alumina, coking may be the major cause, leaving only the very weak acid sites intact. One noticeable occurrence in this experiment is the stable activity of the catalyst system after the initial deactivation.

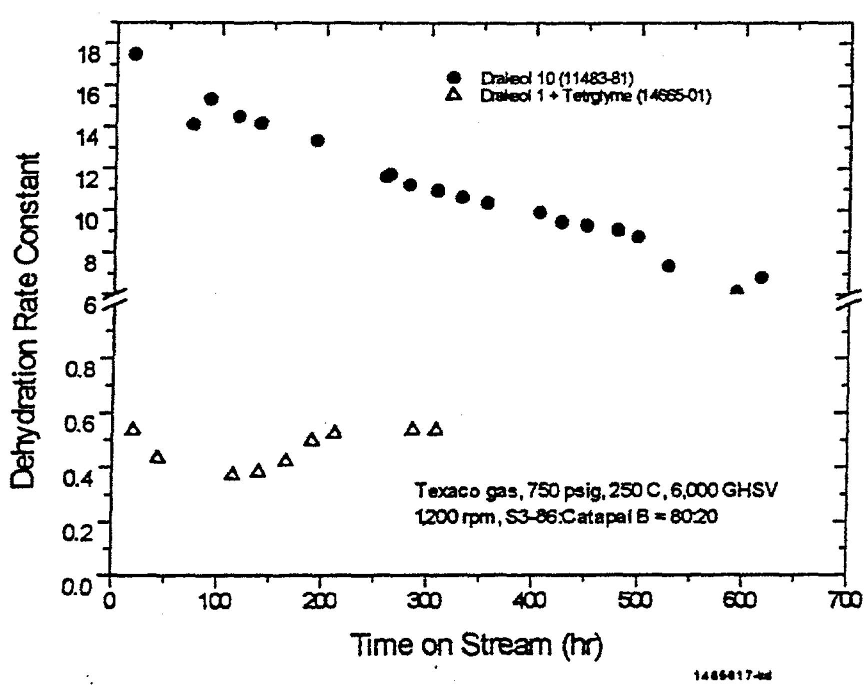
A perfluoropolyether solvent (FOMBLIN Y HVAC from Aldrich) was also tested as an alternative slurry fluid. This solvent has been used in laboratory Fischer-Tropsch synthesis experiments. The LPDME run using this solvent was conducted in a 50 cc microclave with a standard catalyst system (Run # 14667-11). Both catalysts died quickly, possibly again due to the decomposition of the solvent in the presence of g-alumina, which releases poisonous species such as fluorine-containing moieties.

Figure 3.1.11 Catalyst Stability in the System Containing Tetraglyme

a



b



APPENDIX F - TASK 1.5.4 - PROJECT REVIEW MEETING

NOTES FROM MEETING

AIR PRODUCTS

DISTRIBUTION (NAME/ORGANIZATION)			COPIED FOR INFORMATION ONLY
Attendees:			W. Brown/A31E9
APCI		DOE	W. Jones/EMN
T. Dahl*/I7066	D. Polyak*/A32K1	D. Archer	L. Paulonis/EMN
D. Drown/A31E9	E. Schaub*/A12A3	W. Mundorf	B. Street /EMN
F. Frenduto/A12B2	V. Stein*/A12A3	R. Kornosky	
V. Hallowell*/A32K1	B. Toseland*/I7066		
B. Halper*/A6222	A. Wang*/A6213		
E. Heydorn/A31E9	*Part-time		

FROM	ORGANIZATION	EXTENSION	TODAY'S DATE
D. P. Drown	PSE Project Engineering	16143	15 March, 1996

DATE OF MEETING	WEEKDAY	TIME STARTED	TIME ENDED	LOCATION
4 March 1996	Monday	10:30 a.m.	5:00 p.m.	CR111B & Iron Run
5 March 1996	Tuesday	8:00 a.m.	2:00 p.m.	Joseph Oat Shop

SUBJECT AND/OR PURPOSE

Kingsport LPMEOH Demonstration Project
DOE Status Review Meeting

ITEM NO.	RESPONSIBLE PERSON (INITIALS)	TARGET DATE	DISCUSSION
			A project status review meeting for the Kingsport LPMEOH Demonstration Project was held at APCI's offices in Allentown, PA A copy of the agenda for this meeting is attached (Attachment No. 1). A copy of the slides presented are also attached (Attachment No. 2). The following notes document action items from this meeting.
			GENERAL
1			Air Products was awarded the Construction Industry Business Roundtable Owners Safety Award for 1995-1996. This award is presented to APCI based on our construction management safety performance. A copy of the award certificate is attached (Attachment No. 3).
2			Air Products intends to submit a draft of the Budget Period 3 Continuation Application in June 1996.
3	B. Street		R. Kornosky requested a picture of the LPMEOH Project sign in front of the APCI Construction trailer.
4	W. Brown		The DOE expressed concern with cost sharing a DME demonstration run at LaPorte from both the Alternative Fuels funding and the Clean Coal program. APCI will submit an explanation of the cost sharing basis for this potential run. The benefits of a DME demonstration run at LaPorte (vs. Kingsport) were explained.

AIR PRODUCTS

NOTES FROM MEETING
CONTINUATION

ITEM NO.	RESPONSIBLE PERSON (INITIALS)	TARGET DATE	DISCUSSION
5			<u>ENVIRONMENTAL MONITORING PLAN</u> R. Kornosky commented on the recent draft of the EMP a) Request for pertinent coal gasifier information b) Need frequency for crude methanol analysis c) Explain process data vs. supplemental data d) Describe or refer to the Operating Plan
6	F. Frenduto	April	It was suggested that a meeting at PETC be held to resolve open items after the next submittal of APCI's Operating Plan.
7	R. Kornosky	ASAP	The DOE will collect comments on the EMP and submit them to APCI.
8	F. Frenduto		APCI should put a lead page in the EMP with the phrase "patents cleared by Chicago on (date)."
9	F. Frenduto		The EIV discusses two numbers for peak construction manpower, 110 and 135 (use 135).
10	F. Frenduto		The DOE prefers unbound or loose leaf copies of the EMP.
11			<u>DEMONSTRATION PLAN</u> A copy of the paper "Fuel Cells in Transportation" presented at the Fuel Cell Conference attended by W. Brown was given to R. Kornosky. Acurex and APCI are considering a stationary power and a transportation power fuel cell demonstration as part of the off-site product testing.
12			<u>DME RESEARCH</u> The slides presented at Iron Run on DME Research are Attachment No. 4 (Note; Proprietary information has been removed).
13			APCI is currently doing catalyst life testing. We have looked at 27 catalysts to date.
14			APCI recommends a run at LaPorte on the DME catalyst to give confidence before a larger scale up.
15			The Alternate Fuels Field Development Unit (AFFDU) trailer is being prepared for a catalyst poisons study at Kingsport in May 1996.

AIR PRODUCTS

NOTES FROM MEETING
CONTINUATION

ITEM NO.	RESPONSIBLE PERSON (INITIALS)	TARGET DATE	DISCUSSION
			<u>VISIT TO JOSEPH OAT'S SHOP IN CAMDEN, NJ (3/5/95)</u>
16			A trip was made to Joseph Oat's Shop in Camden, NJ to observe the LPMEOH reactor. The internal tube bundle is being prepared for insertion into the shell after the shell goes through Post Weld Heat Treatment.
17			R. Kornosky requested copies of APCI's pictures of the reactor (Attachment No. 5).
18	D. Drown	3/25/96	R. Kornosky requested a copy of the railroad routing to Kingsport, TN.
			<u>ATTACHMENTS</u> 1) 3/4/96 Meeting Agenda 2) Slides from Status Review Meeting 3) Business Roundtable Owners Safety Award Certificate 4) Slides from DME Presentation 5) Kingsport LPMEOH Reactor Pictures taken 11/29/95 (and 1 from 3/96)

MM025

Attachment No. 1

MEETING NOTICE

AIR PRODUCTS

☐ Check if this meeting was scheduled through Schedule+

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)*		COPIED FOR INFORMATION ONLY
APCI	DOE	D. Brown/APE ✓
F. Frenduto/A32G3 ✓	D. Archer ✓	W. Brown/A31E9 ✓
V. Hallowell/A32K1	R. Kornosky ✓	G. Cattell/A32E2
B. Halper/A6222 ✓	W. Mundorf ✓	R. Moore/A32G3 ✓
E. Heydorn/A31E9 ✓		E. Schaub/A12A3
X.D. Peng/I7066		V. Stein/A12A3 ✓
D. Polyak/A32K1		L. Paulino/B.J./W.J. ✓
B. Toseland/I7066 ✓		
A. Wang/I7066		

FROM	ORGANIZATION	EXTENSION	TODAY'S DATE
D. Drown	LPT - Project Management	1-6143	21 February 1996

DATE OF MEETING	WEEKDAY	TIME FROM	TIME TO	LOCATION
March 4, 1996	Monday	10:30 AM	4:30 PM	CR111B & Iron Run

SUBJECT AND/OR PURPOSE
Kingsport LPMEOH Project
DOE/PETC Update

DESIRED RESULTS/OUTCOMES
Project Update

REFERENCE MATERIAL/OTHER
Minutes of 10/17/95 DOE Meeting

AGENDA	Who	Time
• Project Status (CR 111B)		
Project Overview	D. Drown	10:30 - 10:45 AM
Status of Design	F. Frenduto	10:45 - 11:00 AM
Outstanding Equipment Items	F. Frenduto	11:00 - 11:10 AM
Status of Construction	D. Drown	11:10 - 11:30 AM
Lunch	All	11:30 - 12:20 PM
Eastman Scope of Work	D. Drown	12:20 - 12:40 PM
Schedule Update	D. Drown	12:40 - 1:00 PM
Current Spending	D. Drown	1:00 - 1:15 PM
• Environmental Monitoring Plan	F. Frenduto	1:15 - 2:00 PM
• Demonstration/Operating Plan	E. Heydorn	2:00 - 2:30 PM
• Review of Plant CADD Model	F. Frenduto/V. Hallowell	2:30 - 3:10 PM
Drive to Iron Run		3:10 - 3:25 PM
• DME Update	B. Toseland	3:30 - 4:00 PM
• May 1996 Catalyst Poisons Test at Kingsport	A. Wang	4:00 - 4:15 PM
• Tour of Iron Run Lab and Test Trailer	B. Toseland	4:15 - 4:30 PM

MN046

FORM 3583 (REV. 2/91)

LPMEOH DEMONSTRATION PROJECT
FEBRUARY 1996 STATUS OVERVIEW

DESIGN

85% COMPLETE DETAIL DESIGN
97% PREFAB PIPE RELEASED TO CONNEX PIPE SYSTEMS, INC.
90-95% PACKAGE OUT TO BID FOR GENERAL MECHANICAL CONSTRUCTION
I&E BID PACKAGE READY MARCH 22, 1996
DESIGN VERIFICATION SAFETY REVIEW (DVR) SCHEDULED FOR MARCH 11 - 15, 1996

EQUIPMENT

ALL PROCESS EQUIPMENT ITEMS TO BE ON SITE IN MARCH EXCEPT:

REACTOR
SAFETY RELIEF K.O. DRUM
PUMP SEAL OIL COOLER
SAFETY RELIEF VENT STACK

CONSTRUCTION

STEEL AND EQUIPMENT ERECTION STARTED
RECEIVING EQUIPMENT AND MATERIALS

COST FORECAST

PHASE 1 & 2 - POST MOD 3
AIR PRODUCTS-EMN INCLUDING G&A - $36.5MM

SCHEDULE

DESIGN SCHEDULE OKAY EXCEPT FOR PSV SPECIFICATION AND COMPLETING STEEL DESIGN
STARTED STEEL AND EQUIPMENT ERECTION ON SCHEDULE
EVALUATING NOVEMBER MECHANICAL COMPLETION DATE

3/01/96
DPD/SLIDE

Attachment No. 2

LPMEOH PROJECT

DESIGN STATUS - 4 MARCH 1996

PROCESS ENGINEERING

- Working with Radian Corporation on the Vent Stack design
- Review of piping designs

P&ID

- Rev 1 +(as designed) in drafting for issue this week

PROCESS CONTROLS

- Most Instrument items released for purchase
  -transmitters, analyzers and PLC to be released week of 3/4
- DCS Slot Assignments have been prepared
- Work continues on providing data needed for EMN to program the DCS
- Decided to purchase the Honeywell Data Acquisition System (DAS).
  Purchase spec. to be complete by 3/31

VALVES AND MATERIALS

- Pressure Test Flowsheet is the largest unfinished work
- A few steam valves and small strainers to complete
- Review of Fire Protection vendor drawings still in future
- Approx. 1 1/2 man months work left

PIPING/LAYOUT

- Sent 97+% complete large bore prefab package to Connex Piping Systems for fabrication last week
- Will send Bulletin #2 to Mechanical contractors this week
- Work to complete
  - Get remaining FCN's (about 12) on the drawings
  -provide package(changes that might affect cost)to two low bidders on 3/12
- Staffing: 6 people through March, 3 through mid April, 1 ? thereafter

Attachment No. 2

CIVIL STRUCTURAL

- Steel Engineering continues through end of March
- Steel Design continues through mid April
- Misc. Building Package out 3/4
- HVAC package out 3/4
- Staffing: 2 Engineers through March, 1 ? thereafter
  6 Designers through mid April, 1-2 thereafter

EQUIPMENT ENGINEERING

- Essentially complete

ELECTRICAL/INSTRUMENT DESIGN

- Working toward bid package by 3/15
- Issue "for construction" package by mid April
- Complete Analyzer Bldg. Design by mid April for mid August delivery
- Instrument Loop Diagrams to complete for July
- Electrical Staffing: 4 people through mid March, 3 people mid March-mid April, 1 person thereafter
- Instrument Staffing: 2 people through March, 1 thereafter

DESIGN HAZARD REVIEW

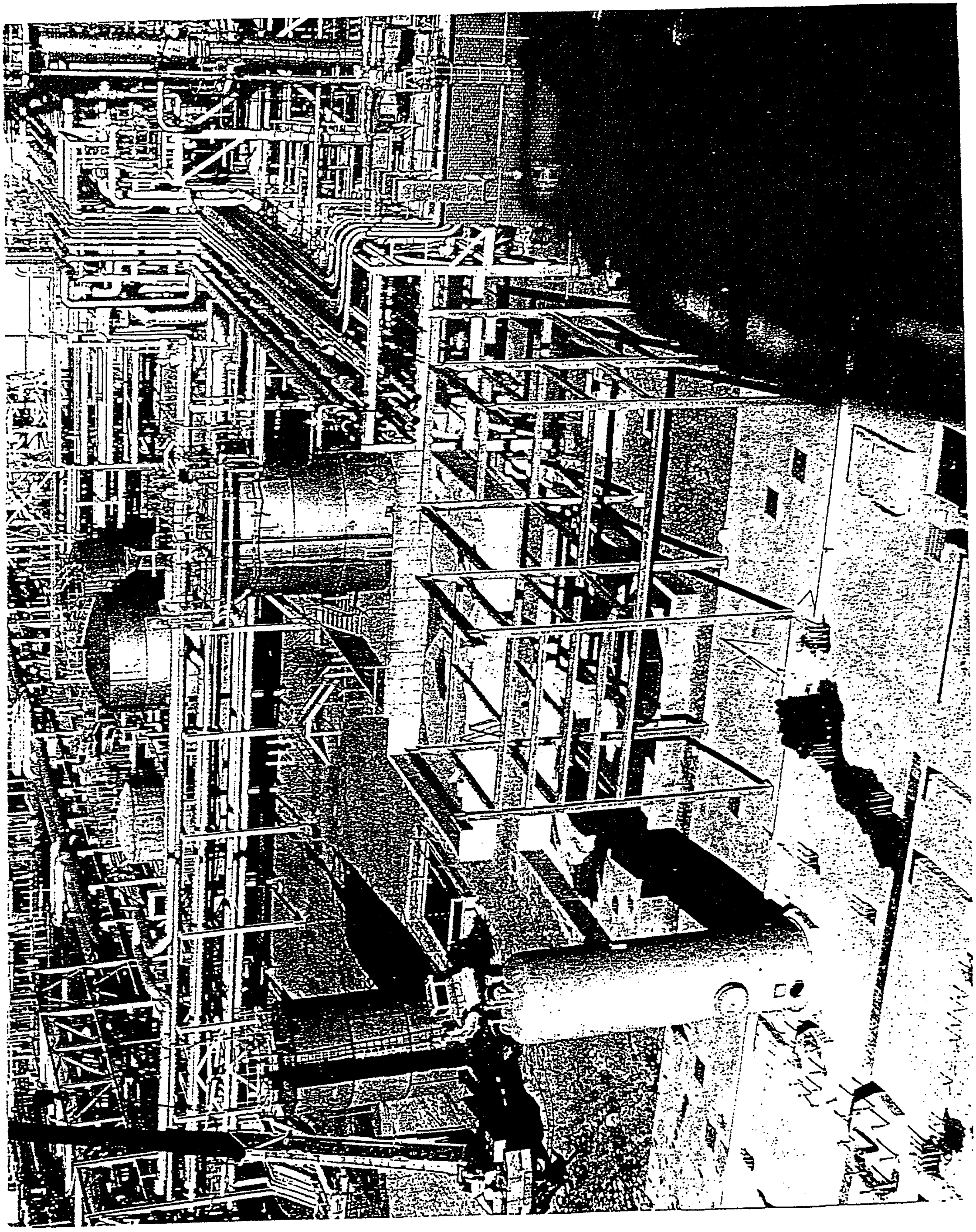
- Joint effort with Eastman scheduled for 11 March- 15 March

MAJOR UNRESOLVED ISSUES

- Vent Stack Design

FSF 2/29/96

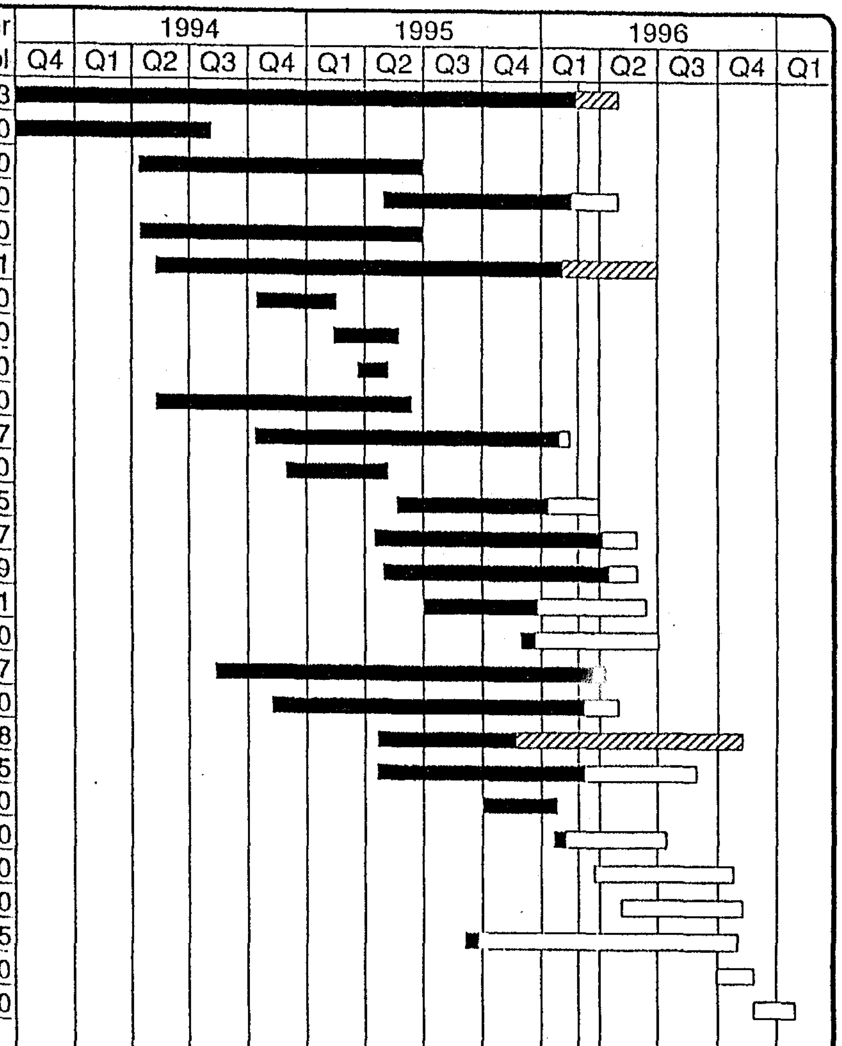
Attachment No. 2



Attachment No. 2

LPMEOH DEMONSTRATION PROJECT
PHASE 1-2 SUMMARY SCHEDULE

Task Name	Start	End	Percent Compl
PROCESS DESIGN	Oct/01/93	May/01/96	93
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	May/01/96	80
NEPA FONSI REVIEW	Apr/15/94	Jun/30/95	100
DETAILED DESIGN	May/11/94	Jul/03/96	81
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	Feb/15/96	97
EQUIPMENT ARRANGEMENTS	Dec/01/94	May/08/95	100
INSTRUMENT SPECS	May/22/95	Apr/01/96	75
CIVIL/STRUCTURAL DESIGN	Apr/17/95	May/30/96	87
PIPING DESIGN	May/01/95	May/31/96	89
INSTRUMENT/ELECTRICAL DESIGN	Jul/03/95	Jun/14/96	51
DCS PROGRAMMING	Dec/01/95	Jul/03/96	10
EQUIPMENT PROCUREMENT	Aug/12/94	Apr/10/96	97
REACTOR DELIVERY	Nov/08/94	May/01/96	90
CONSTUCTION	Apr/21/95	Nov/08/96	38
EASTMAN OSBL WORK	Apr/21/95	Aug/30/96	65
CIVIL/UNDERGROUND	Oct/02/95	Jan/26/96	100
STEEL & EQUIPMENT ERECTION	Jan/22/96	Jul/15/96	10
MECHANICAL	Mar/25/96	Oct/25/96	0
INSTRUMENT/ELECTRICAL	May/06/96	Nov/08/96	0
PROCEDURES/TRAINING	Sep/05/95	Nov/01/96	5
CHECK-OUT/COMMISSIONING	Oct/01/96	Nov/27/96	0
START-UP	Nov/27/96	Jan/31/97	0



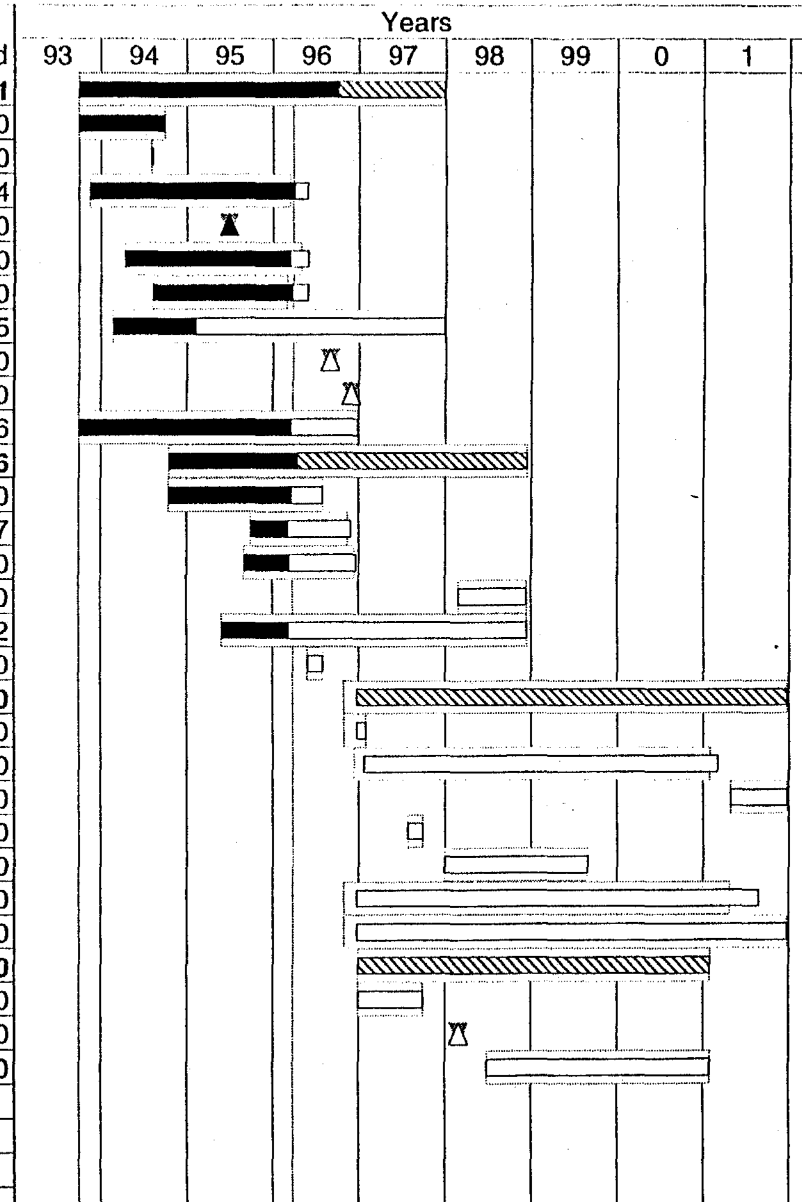
Printed: Feb/28/96

Milestone △ Summary
Fixed Delay · · · ·

APPENDIX G - 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**	**76**	**71**
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)	30.55 m	Nov/17/93	May/30/96	95	94
NEPA FONSI APPROVAL	0.00 d	Jun/30/95	Jun/30/95	100	100
DESIGN ENGINEERING(TASK 3)	25.70 m	Apr/15/94	Jun/01/96	85	90
VENDOR ENGINEERING	21.84 m	Aug/10/94	Jun/01/96	97	90
OFF-SITE TESTING(TASK 4)	46.35 m	Feb/25/94	Dec/30/97	7	25
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	91	76
PHASE 2: CONSTRUCTION	**50.18 m**	**Oct/17/94**	**Dec/15/98**	**15**	**36**
PROCUREMENT(TASK1)	21.61 m	Oct/17/94	Aug/01/96	92	80
CONSTRUCTION(TASK 2)	14.02 m	Oct/02/95	Nov/29/96	17	37
TRAINING & COMMISSIONING(TASK 3)	15.70 m	Sep/05/95	Dec/23/96	6	40
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	22	22
CONTINUATION APPLICATION(B.P.#3)	2.08 m	May/31/96	Aug/01/96	0	0
PHASE 3: OPERATION	**60.31 m**	**Dec/27/96**	**Dec/28/01**	**0**	**0**
START-UP(TASK 1)	1.22 m	Dec/27/96	Feb/01/97	0	0
METHANOL OPERATION(TASK 2.1)	49.52 m	Jan/26/97	Mar/06/01	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)	56.35 m	Dec/27/96	Aug/30/01	0	0
PLANNING & ADMINISTRATIVE(TASK 6)	60.31 m	Dec/27/96	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



Milestone △ Summary

DOE F 1332.9

(11-84)

FORM APPROVED

OMB 1910-1400

U.S. DEPARTMENT OF ENERGY

COST MANAGEMENT REPORT

1. TITLE	2. REPORTING PERIOD	3. IDENTIFICATION NUMBER
Liquid Phase Methanol Demonstration	March 01, 1996 through March 31, 1996	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 Reporting Period a. Actual	b. Plan	Cumulative to Date c. Actual	d. Plan	11. ESTIMATED ACCRUED COSTS a. Subsequent Reporting Period	b. Balance of Fiscal Year	c. FY 1997 (1)	FY 1998 (2)	FY 1999 (3)	d. Subsequent FY's (4)	e. Total	12. Total Contract Value	13. Variance
	Prior to Mod 2	0	0	16,289	16,289	0	0					16,289	16,289	0
1.1.1	Project Definition	0	0	1,044	1,021	0	0	0	0	0	0	1,044	1,021	23
1.1.2	Permitting	4	0	224	246	0	0	0	0	0	0	224	246	(22)
1.1.3	Design Engr.	612	565	7,957	7,926	565	1,155	283	0	0	0	9,960	9,960	0
1.1.4	Off-site Testing	0	8	12	34	8	54	246	0	0	0	320	320	0
1.1.5	Planning, Admin, & DME Verif. Testing	102	43	2,023	1,637	43	200	0	0	0	0	2,266	1,892	374
1.2.1	Procurement	1,349	829	5,241	5,202	829	2,209	1,128	0	0	0	9,407	9,783	(376)
1.2.2	Construction	442	1,108	2,167	4,984	1,108	6,323	1,602	0	0	0	11,200	11,200	0
1.2.3	~~Proj. Mgmt.~~ Train + Commission	0	80	1	271	148	1,014	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	15	47	176	337	46	391	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		2,525	2,680	35,137	37,947	2,747	11,345	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 (signature) — DATE: 5/2/96

17. SIGNATURE OF PARTICIPANT'S AUTHORIZED FINANCIAL REPRESENTATIVE AND DATE: Susan J. Kasinecz (signature), S. J. Kasinecz — DATE: 5/2/96

APPENDIX H - 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

WRB 4/1/96

3/29/96

DOE DISTRIBUTION:

Mr. Robert M. Kornosky (2 copies)
Mr. William R. Mundorf
Mr. William J. O'Dowd (2 copies)

Subject: Cooperative Agreement DE-FC22-92PC90543
Kingsport Liquid Phase Methanol Demonstration
Demonstration Test Plan (Second Draft)

Attached is the Demonstration Test Plan (Second Draft). We have incorporated many of the suggestions from your earlier correspondence (Letter, Kornosky (DOE) to Heydorn (APCI), "Draft Test Plan," 09 February 1996).

This revision will be used as the basis for the review meeting scheduled for 25 April 1996 in Pittsburgh. If you have any comments in advance, please give me a call at (610) 481-7099.

Yours truly,

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

TABLE 5-1 - OPERATION TEST PLAN

Test Run #	Test Run Description	Temp (Deg C)	Wt% Cat	H2/CO Ratio at Inlet	Space Velocity (Sl/hr-kg)	MeOH (tpd)	Fresh Feed Balanced (KSCFH)	Fresh Feed CO Gas (KSCFH)	Compressed Feed H2 Gas (KSCFH)	Compressed Feed Recycle (KSCFH)	Inlet Sup. Velocity (ft/sec)	Time Period (weeks)	Cumulative On Stream Time (wks)	Start of Test
Task 2.1.1 - Process Shakedown and Catalyst Aging:														
1.	Initial Shakedown; and Design Production Tests	250	28	2.42	8,000	260	900	50	40	1,800	0.64	6	6	Feb-97
							(varies, to maintain syngas utilization.)							
2.	Gassed Slurry Level	Part of other tests										Ongoing		
3.	Reactor Feed: Texaco-Type Syngas	250	28	0.67	9,240	202	650	95 (*)	0	2,612 (*)	0.77	2	9	Mar-97
4.	Early Testing @ High Superficial Velocity	250	28	2.54	10,300	325	1,200 (**)	50	40	2,520 (*)	0.88	2	12	Apr-97
5.	Check @ Test 1 Conditions	250	28	2.42	8,000	< 260	900	50	40	1,800	0.64	2	15	Apr-97
6.	Catalyst Addition and Aging	250	28 - 40	2.51	Dec. from	237	765	40	45	Max	0.79	18	41	May-97
		or less		2.30	8,000	260	900	50	40	(2,700)	TBD	6		to
	(Note: Kingsport Complex Outage during this test)													Nov-97
7.	Free-Drain Entrained/ Condensed Oil to Reactor	250 or less	28 - 40	2.51	Dec. from 8,000	237	765	40	45	Max	0.79	During Test 6		
8.	Operation @ Design Feed Gas Rates	250	40	2.42	4,000	260	900	50	40	1,800	0.64	2	43	Nov-97
9.	Check for Limitation on Catalyst Slurry Concentration	250	> 40	2.51	Varies	TBD	765	40	45	Max (2,700)	0.79	6	50	Nov-97
10.	Catalyst Addition to	250	Target	2.49	3,320	256	765	40	45	2,605	0.79	12	68	Jan-98
	Reach Max Productivity	or less	45	2.29	3,500	293	900	50	40	2,520	0.81	2		
				TBD	TBD	TBD	1,110 (**)	50	40	2,520	0.86	2		

5-3

3-29-96

TABLE 5-1 - OPERATION TEST PLAN

Test Run #	Test Run Description	Temp (Deg C)	Wt% Cat	H2/CO Ratio at Inlet	Space Velocity (Sl/hr-kg)	MeOH (tpd)	Fresh Feed Balanced (KSCFH)	Fresh Feed CO Gas (KSCFH)	Compressed Feed H2 Gas (KSCFH)	Compressed Feed Recycle (KSCFH)	Inlet Sup. Velocity (ft/sec)	Time Period (weeks)	Cumulative On Stream Time (wks)	Start of Test
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. feed gas allocation for commercial design/optimal performance).													
11.	Catalyst Addition/ Withdrawal Test	250	Target 45	2.49	3,320	256	765	40	45	2,605	0.79	6	74	May-98
12.	Test 11 Conditions with No CO Make-up	250	Target 45	4.97	3,282	229	765	0	45	2,605	0.78	2	76	Jul-98
13.	Test 11 Conditions with No H2 Make-up	250	Target 45	1.98	3,277	252	765	40	0	2,605	0.78	2	79	Jul-98
14.	Test 11 Conditions with No H2 or CO Make-up	250	Target 45	5.03	3,238	232	765	0	0	2,605	0.77	2	81	Aug-98
15.	Repeat of Test 11 Conditions	250	Target 45	2.49	3,320	256	765	40	45	2,605	0.79	2	83	Aug-98
16.	Design Fresh Feed Operation Test	250	Target 45	2.29	3,500	293	900	50	40	2,520	0.81	2	86	Sep-98
17.	Testing @ High Superficial Velocity	250	Target 45	TBD	TBD	TBD	1,110 (**)	50	40	2,520	0.86	2	88	Sep-98
18.	Turndown and Ramping	250	Target 45	3.30	1,825	151	450	25	60	1,364	0.44	2	90	Oct-98
19.	Load-Following, Cyclone, & On/Off Tests		Target 45	To be Defined								4	95	Oct-98
20.	Reactor Feed: Texaco-Type Syngas	250	Target 45	0.69	2,870	207	650	85 (**)	0	2,195	0.67	4	99	Nov-98

3-29-96

TABLE 5-1 - OPERATION TEST PLAN

Test Run #	Test Run Description	Temp (Deg C)	Wt% Cat	H2/CO Ratio at Inlet	Space Velocity (Sl/hr-kg)	MeOH (tpd)	Fresh Feed Balanced (KSCFH)	Fresh Feed CO Gas (KSCFH)	Compressed Feed H2 Gas (KSCFH)	Compressed Feed Recycle (KSCFH)	Inlet Sup. Velocity (ft/sec)	Time Period (weeks)	Cumulative On Stream Time (wks)	Start of Test
21.	Reactor Feed: Destec-Type Syngas	250	Target 45	1.01	2,770	215	670	65 (***)	0	2,147	0.67	3	102	Dec-98
22.	Reactor Feed: BGL-Type Syngas	250	Target 45	0.52	2,165	137	485	200 (***)	0	1,568	0.43	3	105	Jan-99
23.	Repeat of Test 15 Conditions	250	Target 45	2.49	3,320	256	765	40	45	2,605	0.79	2	108	Feb-99
24.	Reactor Feed: Nat. Gas Reformer-Type Syngas	250	Target 45	4.98	1,978	197	765	0	30	1,264	0.48	3	111	Feb-99
25.	Reactor Feed: Shell-Type Syngas with Steam Injection and 1:1 Recycle	250	Target 45	0.53	1,471	101	238	400 (***)	50	842	0.35	3	114	Mar-99
26.	Operation with Two Methanol Synthesis Catalysts	250	Target 45	2.49	3,320	256	765	40	45	2,605	0.79	8	125	Apr-99
				2.29	3,500	293	900	50	40	2,520	0.81	2		
27.	Reactor Operation @ 500 deg F	260	Target 45	2.51	3,320	248	765	40	45	2,605	0.79	2	127	Jun-99
28.	Reactor Inspection (Then, Continue Operational Tests - with alternative catalyst):											4	131	Jul-99
29.	Plant Shakedown	240	TBD	2.42	TBD	260	900	50	40	Max(TBD)	TBD	6	137	Aug-99
30.	Reactor Feed: Texaco-Type Syngas	240	TBD	0.67	TBD	202	650	95 (*)	0	2,612 (*)	0.77	2	140	Sep-99
31.	Catalyst Aging	240	TBD	2.50	TBD	237	765	40	45	2,605		16	162	Oct-99
				2.31	TBD	260	900	50	40	2,520	TBD	4		

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Test Run #	Test Run Description	Temp (Deg C)	Wt% Cat	H2/CO Ratio at Inlet	Space Velocity (Sl/hr-kg)	MeOH (tpd)	Fresh Feed Balanced (KSCFH)	Fresh Feed CO Gas (KSCFH)	Compressed Feed H2 Gas (KSCFH)	Compressed Feed Recycle (KSCFH)	Inlet Sup. Velocity (ft/sec)	Time Period (weeks)	Cumulative On Stream Time (wks)	Start of Test
32.	Catalyst Addition/ Withdrawal to Achieve Target Slurry Concentration	240 - 250	Target 45		To be defined					Max(TBD)		6	168	Mar-00
33.	Reactor Feed: Texaco-Type Syngas	250	Target 45	0.69	2,870	207	650	85 (**)	0	2,195	0.67	4	173	Apr-00
Task 2.1.3 - Extended Optimum Operation:														
34.	Stable Operation	250	Target 45	2.49	3320	256	765	40	45	2605	0.79	16	200	May-00
		250		2.29	3500	293	900	50	40	2520	0.81	6		
		250		TBD	TBD	TBD	1,110 (**)	50	40	2520	0.86	2		
35.	DME Demo (Task 2.2) or Commercial Test Run (Task 2.1.3)											TBD		Dec-00 to
												8	208	Mar-01
	Syngas Outages (5%, including Kingsport complex outage during Test 6)											10		
	Planned LPMEOH Outages (including Reactor Inspection and Fresh Catalyst Charge in Test 28)											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.

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