Commercial-Scale Demonstration of the Liquid Phase Methanol (LPMEOTH™) Process

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Abstract

The Liquid Phase Methanol (LPMEOH™) Demonstration Project at Kingsport, Tennessee, is a \$213.7 million cooperative agreement between the U.S. Department of Energy (DOE) and Air Products Liquid Phase Conversion Company, L.P. (the Partnership) to produce methanol from coal-derived synthesis gas (syngas). Air Products and Chemicals, Inc. (Air Products) and Eastman Chemical Company (Eastman) formed the Partnership to execute the Demonstration Project. The LPMEOH™ Process Demonstration Unit was built at a site located at the Eastman coal-to-chemicals complex in Kingsport.

During this quarter, initial planning and procurement work continued on the seven project sites which have been accepted for participation in the off-site, product-use test program. Approximately 12,000 gallons of fuel-grade methanol (98+ wt% methanol, <1 wt% water) produced during operation on carbon monoxide (CO)-rich syngas at the LPMEOH™ Demonstration Unit was loaded into trailers and shipped off-site for future product-use testing. At one of the projects, three buses have been tested on chemical-grade methanol and on fuel-grade methanol from the LPMEOH™ Demonstration Project.

During the reporting period, planning for a proof-of-concept test run of the Liquid Phase Dimethyl Ether (LPDME™) Process at the Alternative Fuels Development Unit (AFDU) in LaPorte, TX continued. The commercial catalyst manufacturer (Calsicat) has prepared the first batch of dehydration catalyst in large-scale equipment. Air Products will test a sample of this material in the laboratory autoclave.

Catalyst activity, as defined by the ratio of the rate constant at any point in time to the rate constant for freshly reduced catalyst (as determined in the laboratory autoclave), was monitored for the initial extended operation at the lower initial reactor operating temperature of 235°C. At this condition, the decrease in catalyst activity with time from the period 20 December 1997 through 27 January 1998 occurred at a rate of 1.0% per day, which represented a significant improvement over the 3.4% per day decline measured during the initial six weeks of operation in April and May of 1997. The deactivation rate also improved from the longer-term rate of 1.6% per day calculated throughout the summer and autumn of 1997.

Based on this improvement, DOE accepted a recommendation by Air Products and Eastman to further reduce the reactor temperature to 225°C. The initial operation at this temperature (from 31 January through 18 February 1998) showed a modest improvement in the deactivation rate to 0.7% per day. However, most of the activity decline occurred during a test period on CO-rich feed gas and immediately after an interruption in the Balanced Gas supply at the end of that test case. During two additional stable operating periods between 19 February and 31 March 1998, deactivation rates of 0.27% and 0.36% per day were measured. A discontinuity occurred in the data on 15 March 1998, again coincidental with an excursion in the Balanced Gas supply. If the current results prove to be correct, the current baseline activity decline of about 0.4% per day matches the original target from the 4-month proof-of concept run on a natural-gas derived syngas at the LaPorte

AFDU. Additional operating time is necessary to further quantify the catalyst deactivation rate and the effects of operating conditions and syngas supply excursions.

The weight of catalyst in the LPMEOH™ Reactor has reached 67% of the design value, and the slurry concentration approached 40 wt%. Catalyst slurry samples from the LPMEOH™ Reactor have been taken on a regular basis to correlate any change in plant performance with changes in the physical properties of the catalyst. So far, samples from mid-February and mid-March of 1998 have shown an increase in arsenic loading, although not nearly to the levels measured in the summer of 1997. Copper crystallite size measurements are still pending, as are analyses from more recent samples which will help complete the picture.

The performance of the alternative gas sparger, which was designed by Air Products and installed into the LPMEOH™ Reactor prior to the restart of the LPMEOH™ Demonstration Unit in December of 1997, was monitored throughout the reporting period. Pressure drop through the gas sparger was stabilized using a continuous flush of condensed oil and entrained slurry which were gravity-drained from downstream process equipment. As with the original gas sparger design, the return of the internal oil and slurry streams via the flush connection was required to stabilize the pressure drop after extended interruptions of the flush fluid. When compared to the original gas sparger, the alternative gas sparger may have greater flexibility in maintaining stable pressure drop after interruptions of the syngas supply; this may also be a result of greater attention to achieving a proper standby condition during shutdowns. The most recent results provide an initial confirmation of the encouraging data reported for the first two weeks of operation at the end of the prior reporting period.

The Alternative Fuels Field Test Unit (AFFTU), a transportable laboratory equipped with an autoclave and analytical equipment, was shipped from the Air Products' Iron Run laboratory in Allentown, PA to Kingsport to perform additional testing on the reactor feed gas at the site. Performance results from the 31-day campaign on coal-derived syngas at Kingsport were generally consistent with other laboratory experiments on poison-free syngas. A post-mortem analysis on the catalyst revealed no unusual levels of catalyst poisons or significant changes in catalyst physical properties. The AFFTU test concluded on 20 January 1998, and the equipment was returned to the Air Products' Iron Run laboratory.

During the reporting period, a total of 5,762,047 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Apart from 12,000 gallons shipped off-site for product-use testing, Eastman accepted all of this methanol for use in the production of methyl acetate, and ultimately cellulose acetate and acetic acid. During a portion of the operating period on CO-rich syngas, approximately 181,800 gallons (600 tons) of stabilized methanol (99.3 wt% methanol, 0.3 wt% water) was utilized directly from the LPMEOH™ Demonstration Unit in Eastman's methyl acetate plant, bypassing the normal route through the distillation equipment which is designed to remove water, higher alcohols, and process oil. This provides another indication of the flexibility of the LPMEOH™ Process. No safety or environmental incidents were reported during this quarter. Availability exceeded 99%, as the demonstration unit continued to operate through the longest continuous campaign to date (45 days) as of 31 March 1998.

Ninety-nine percent (99%) of the \$38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 31 March 1998. Nineteen percent (19%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 March 1998.

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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"
AFFTU	-	Alternative Fuels Field Trailer Unit
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
Catalyst Age (η - eta)	-	the ratio of the rate constant at any point in time to the rate constant for a freshly reduced catalyst (as determined in the laboratory autoclave)
Catalyst Concentration	-	Synonym for Slurry Concentration
Catalyst Loading	-	Synonym for Slurry Concentration
CO Conversion	-	the percentage of CO consumed across the reactor
Crude Grade Methanol	-	Underflow from rectifier column (29C-20), defined as 80 wt% minimum purity; requires further distillation in existing Eastman equipment prior to use
DME	-	dimethyl ether
DOE	-	United States Department of Energy
DOE-FETC	-	The DOE's Federal Energy Technology Center (Project Team)
DOE-HQ	-	The DOE's Headquarters - Coal Fuels and Industrial Systems (Project Team)
DTP	-	Demonstration Test Plan - The four-year Operating Plan for Phase 3, Task 2 Operation
DVT	-	Design Verification Testing
Eastman	-	Eastman Chemical Company
EIV	-	Environmental Information Volume
EMP	-	Environmental Monitoring Plan
EPRI	-	Electric Power Research Institute
FFV	-	flexible fuel vehicle
Fresh Feed	-	sum of Balanced Gas, H ₂ Gas, and CO Gas
Gas Holdup	-	the percentage of reactor volume up to the Gassed Slurry Height which is gas
Gassed Slurry Height	-	height of gassed slurry in the reactor
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
Inlet Superficial Velocity	-	the ratio of the actual cubic feet of gas at the reactor inlet (calculated at the reactor temperature and pressure) to the reactor cross-sectional area (excluding the area contribution by the internal heat exchanger); typical units are feet per second
K	-	Sparger resistance coefficient (term used in calculation of pressure drop)
KSCFH	-	Thousand Standard Cubic Feet per Hour
LaPorte PDU	-	The DOE-owned experimental unit (PDU) located adjacent to Air Products' industrial gas facility at LaPorte, Texas, where the LPMEOH™ process was successfully piloted
LPDME™	-	Liquid Phase DME process, for the production of DME as a mixed coproduct with methanol
LPMEOH™	-	Liquid Phase Methanol (the technology to be demonstrated)
M85	-	a fuel blend of 85 volume percent methanol and 15 volume percent unleaded gasoline
MeOH	-	methanol
Methanol Productivity	-	the gram-moles of methanol produced per hour per kilogram catalyst (on an oxide basis)
MTBE	-	methyl tertiary butyl ether
MW	-	molecular weight, pound per pound mole
NEPA	-	National Environmental Policy Act
OSHA	-	Occupational Safety and Health Administration
ρ	-	density, pounds per cubic foot

ACRONYMS AND DEFINITIONS (cont'd)

Partnership	-	Air Products Liquid Phase Conversion Company, L.P.
PDU	-	Process Development Unit
PFD	-	Process Flow Diagram(s)
ppbv	-	parts per billion (volume basis)
ppmw	-	parts per million (weight basis)
Project	-	Production of Methanol/DME Using the LPMEOH™ Process at an Integrated Coal Gasification Facility
psi	-	Pounds per Square Inch
psia	-	Pounds per Square Inch (Absolute)
psig	-	Pounds per Square Inch (gauge)
P&ID	-	Piping and Instrumentation Diagram(s)
Raw Methanol	-	sum of Refined Grade Methanol and Crude Grade Methanol; represents total methanol which is produced after stabilization
Reactor Feed	-	sum of Fresh Feed and Recycle Gas
Reactor O-T-M Conversion	-	percentage of energy (on a lower heating value basis) in the Reactor Feed converted to methanol (Once-Through-Methanol basis)
Reactor Volumetric Productivity	-	the quantity of Raw Methanol produced (tons per day) per cubic foot of reactor volume up to the Gassed Slurry Level
Recycle Gas	-	the portion of unreacted syngas effluent from the reactor "recycled" as a feed gas
Refined Grade Methanol	-	Distilled methanol, defined as 99.8 wt% minimum purity; used directly in downstream Eastman processes
SCFH	-	Standard Cubic Feet per Hour
Slurry Concentration	-	percentage of weight of slurry (solid plus liquid) which is catalyst (on an oxide basis)
Sl/hr-kg	-	Standard Liter(s) per Hour per Kilogram of Catalyst
Syngas	-	Abbreviation for Synthesis Gas
Syngas Utilization	-	defined as the number of standard cubic feet of Balanced Gas plus CO Gas to the LPMEOH™ Demonstration Unit required to produce one pound of Raw Methanol
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
V	-	volumetric flowrate, thousand standard cubic feet per hour
VOC	-	volatile organic compound
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) to produce methanol from coal-derived synthesis gas (syngas). Air Products and Chemicals, Inc. (Air Products) and Eastman Chemical Company (Eastman) formed the Partnership to execute the Demonstration Project. The LPMEOH™ Process Demonstration Unit was designed, constructed, and is in operation at a site located at the Eastman coal-to-chemicals complex in Kingsport.

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

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

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

At the Eastman complex, the technology is integrated with existing coal gasifiers. A carefully developed test plan will allow operations at Eastman to simulate electricity demand load-following in coal-based IGCC facilities. The operations will also demonstrate the enhanced stability and heat dissipation of the conversion process, its reliable on/off

operation, and its ability to produce methanol as a clean liquid fuel without additional upgrading. An off-site, product-use test 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 and off-site product-use test program will demonstrate the commercial viability of the LPMEOH™ process and allow utilities to evaluate the application of this technology in the coproduction of methanol with electricity. 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 1,000 TPD). A successful demonstration at Kingsport will show the ability of a local resource (coal) to be converted in a reliable (storable) and environmentally preferable way to provide the clean energy needs of local communities for electric power and transportation.

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

The project was reinitiated in October of 1993, when DOE approved a site change to the Kingsport location. DOE conditionally approved the Continuation Application to Budget Period No. 2 (Design and Construction) in March of 1995 and formally approved it on 01 June 1995 (Modification No. M009). After approval, the project initiated Phase 1 - Design - activities. Phase 2 - Construction - activities were initiated in October of 1995. The project required review under the National Environmental Policy Act (NEPA) to move to the construction phase. DOE prepared an Environmental Assessment (DOE/EA-1029), and subsequently a Finding of No Significant Impact (FONSI) was issued on 30 June 1995. The Cooperative Agreement was modified (Modification No. A011) on 08 October 1996, authorizing the transition from Budget Period No. 2 (Design and Construction) to the final Budget Period (Commissioning, Start-up, and Operation). This modification provides the full \$213,700,000 of authorized funding, with 56.7% participant cost share and 43.3% DOE cost share.

During this quarter, initial planning and procurement work continued on the seven project sites which have been accepted for participation in the off-site, product-use test program. Approximately 12,000 gallons of fuel-grade methanol (98+ wt% methanol, <1 wt% water) produced during operation on carbon monoxide (CO)-rich syngas at the LPMEOH™ Demonstration Unit was loaded into trailers and shipped off-site for future product-use testing. At one of the projects, three buses have been tested on chemical-grade methanol and on fuel-grade methanol from the LPMEOH™ Demonstration Project. At two other project sites (a flexible fuel vehicle and a fuel cell application), testing on fuel-grade methanol from

the LPMEOH™ Demonstration Project is scheduled for the second quarter of calendar year 1998.

During the reporting period, planning for a proof-of-concept test run of the Liquid Phase Dimethyl Ether (LPDME™) Process at the Alternative Fuels Development Unit (AFDU) in LaPorte, TX continued. The commercial catalyst manufacturer (Calsicat) has prepared the first batch of dehydration catalyst in large-scale equipment. Air Products will test a sample of this material in the laboratory autoclave.

Catalyst activity, as defined by the ratio of the rate constant at any point in time to the rate constant for freshly reduced catalyst (as determined in the laboratory autoclave), was monitored for the initial extended operation at the lower initial reactor operating temperature of 235°C. At this condition, the decrease in catalyst activity with time from the period 20 December 1997 through 27 January 1998 occurred at a rate of 1.0% per day, which represented a significant improvement over the 3.4% per day decline measured during the initial six weeks of operation in April and May of 1997. The deactivation rate also improved from the longer-term rate of 1.6% per day calculated throughout the summer and autumn of 1997.

Based on this improvement, DOE accepted a recommendation by Air Products and Eastman to further reduce the reactor temperature to 225°C. The initial operation at this temperature (from 31 January through 18 February 1998) showed a modest improvement in the deactivation rate to 0.7% per day. However, most of the activity decline occurred during a test period on CO-rich feed gas and immediately after an interruption in the Balanced Gas supply at the end of that test case. This observation more likely indicated a real change in catalyst activity, and this type of behavior seems to have occurred after other excursions in the Balanced Gas supply. During two additional stable operating periods between 19 February and 31 March 1998, deactivation rates of 0.27% and 0.36% per day were measured. A discontinuity occurred in the data on 15 March 1998, again coincidental with an excursion in the Balanced Gas supply. If the current results prove to be correct, the current baseline activity decline of about 0.4% per day is a measurable improvement over the 1% per day rate seen at 235°C in January of 1998 and matches the original target from the 4-month proof-of-concept run on a natural-gas derived syngas at the LaPorte AFDU. Additional operating time is necessary to further quantify the catalyst deactivation rate and the effects of operating conditions and syngas supply excursions.

The weight of catalyst in the LPMEOH™ Reactor has reached 67% of the design value, and the slurry concentration approached 40 wt% . Catalyst slurry samples from the LPMEOH™ Reactor have been taken on a regular basis to correlate any change in plant performance with changes in the physical properties of the catalyst. So far, samples from mid-February and mid-March of 1998 have shown an increase in arsenic loading, although not nearly to the levels measured in the summer of 1997. Copper crystallite size measurements are still pending, as are analyses from more recent samples which will help complete the picture.

The performance of the alternative gas sparger, which was designed by Air Products and installed into the LPMEOH™ Reactor prior to the restart of the LPMEOH™ Demonstration Unit in December of 1997, was monitored throughout the reporting period. Pressure drop through the gas sparger of the LPMEOH™ Reactor was stabilized using a continuous flush

of condensed oil and entrained slurry which were gravity-drained from the 29C-05 secondary oil knock-out drum and 29C-06 cyclone. As with the original gas sparger design, the return of the internal oil and slurry streams via the flush connection was required to stabilize the pressure drop after extended interruptions of the flush fluid. When compared to the original gas sparger, the alternative gas sparger may have greater flexibility in maintaining stable pressure drop after interruptions of the syngas supply; this may also be a result of greater attention to achieving a proper standby condition during shutdowns. The most recent results provide an initial confirmation of the encouraging data reported for the first two weeks of operation at the end of the prior reporting period. This parameter will continue to be closely monitored for any change in flow resistance.

The Alternative Fuels Field Test Unit (AFFTU), a transportable laboratory equipped with an autoclave and analytical equipment, was shipped from the Air Products' Iron Run laboratory in Allentown, PA to Kingsport to perform additional testing on the reactor feed gas at the site. Analytical results from the AFFTU showed less than 10 ppbv concentrations of metal carbonyls and hydrogen sulfide within the reactor loop. Carbonyl sulfide was typically less than 10 ppbv in the loop, but occasionally drifted higher; these excursions could not be correlated with any changes in the feed gas cleanup operations upstream of the LPMEOH™ facility. Performance results from the 31-day campaign on coal-derived syngas at Kingsport were generally consistent with other laboratory experiments on poison-free syngas. A post-mortem analysis on the catalyst revealed no unusual levels of catalyst poisons or significant changes in catalyst physical properties. The AFFTU test concluded on 20 January 1998, and the equipment was returned to the Air Products' Iron Run laboratory.

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Ninety-nine percent (99%) of the \$38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 31 March 1998. Nineteen percent (19%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 March 1998.

A. Introduction

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

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

B. Project Description

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

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

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

- *Catalyst Preparation Area* - Catalyst and slurry preparation and disposal equipment.
- *Storage/Utility Area* - Methanol product, slurry, and oil storage equipment.

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

- *Reaction Area*

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

- *Purification Area*

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

- *Catalyst Preparation Area*

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

- *Storage/Utility Area*

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

C. Process Description

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

D. Results and Discussion

The project status is reported by task, covering those areas in which activity took place during the reporting period. Major accomplishments during this period are as follows:

D.1 Off-Site Testing (Product-Use Demonstration)

Discussion

The product-use test program, developed in 1992 to support the demonstration at the original Cool Water Gasification Facility site, became outdated due in large part to changes within the power and chemical industries. This original product test program under-represented new utility dispersed electric power developments, and possibly new mobile transport engine developments. The updated product-use test program attempts for broader market applications and for commercial fuels comparisons. The objective of the product-use test program is 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.

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

A limited quantity (up to 400,000 gallons) of the methanol product as produced from the demonstration unit will be made available for product-use tests. Product-use tests were targeted for an approximate 18 to 30-month period, and commenced during the first year of demonstration operations. An initial inventory of approximately 12,000 gallons of stabilized methanol was produced at LPMEOH™ Demonstration Unit in February of 1998 to supply the needs of the product-use test program; due to the pre-1998 timing for certain tests, methanol was shipped from the inventory held at the Alternative Fuels Development Unit (AFDU) in LaPorte, TX. Air Products, ARCADIS, Geraghty & Miller (formerly Acurex

Environmental Corporation), and the DOE have worked together to select the projects to be included in the off-site, product-use test program.

Activity during this quarter

Eight sites involving a variety of product-use tests have been selected to participate in this task. The sites and project titles are listed in Appendix B-1. In a letter to the DOE dated 31 July 1997, Air Products formally recommended that seven of the eight projects had been defined in sufficient detail so that final planning and implementation should begin. DOE accepted Air Products' recommendation to proceed with the seven projects in August of 1997. The eighth project, involving the testing of a water/naphtha/methanol emulsion as a transportation fuel, is awaiting final project definition.

All of the remaining product-use test projects have begun planning and equipment procurement. Methanol produced from carbon monoxide (CO)-rich syngas at the LaPorte AFDU has been shipped to three of the project sites. Appendix B-2 through B-8 contain summary reports from the approved projects. Highlights from these reports include:

Acurex Flexible Fuel Vehicle (FFV) - The first drum of M85 fuel using methanol supplied from the inventory at the LaPorte AFDU was prepared. Operation is scheduled for May of 1998.

Stationary Turbine for Volatile Organic Carbon (VOC) Control - ARCADIS, Geraghty & Miller is continuing to work on selecting a host site for the project. Allied Signal is the leading candidate using their existing 525 kilowatt gas turbine.

West Virginia University (WVU) Stationary Gas Turbine - The gas turbine has been run successfully on jet fuel. Work is focusing on overcoming a flame-out when the turbine is switched from jet fuel to methanol fuel at idle speed. Methanol from inventory at the LaPorte AFDU is being used in this program.

Aircraft Ground Equipment Emulsion - Tyndall Air Force Base will begin scoping tests in May of 1998 to determine the best emulsion composition.

University of Florida Fuel Cell - Testing is scheduled to begin in June of 1998 pending results of the analysis of the fuel-grade methanol from the LPMEOH™ Demonstration Project and an assessment of the impact (if any) of impurities on the fuel cell system.

West Virginia University Tri-Boro Bus - Three buses have been tested on chemical-grade methanol and on fuel-grade methanol from the inventory at the LaPorte AFDU. Data reduction is underway, and a full report is expected in July of 1998.

Florida Institute of Technology Bus & Light Vehicle - Both vehicles remain operational, and fuel-grade methanol from the LPMEOH™ Demonstration Project has been received. Emissions data will be collected and processed during the next reporting period

D.2 Commercialization Studies

Discussion

Several areas have been identified for development to support specific commercial design studies. These include: a) product purification options; b) front-end impurity removal options; c) catalyst addition/withdrawal options; and d) plant design configuration options. Plant sizes in the range of 300 TPD to 1,800 TPD and plant design configurations for the range from 20% up to 70% syngas conversion will be considered. The Kingsport demonstration unit design and costs will be the basis for value engineering work to focus on specific cost reduction targets in developing the initial commercial plant designs.

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

- a) to provide process design guidance for commercial plant designs.
- b) to meet the Cooperative Agreement's technical objectives requirement for comparison with gas phase methanol technology. This preliminary assessment will help set demonstration operating goals, and identify the important market opportunities for the liquid phase technology.
- c) to provide input to the Demonstration Test Plan (Task 2.3).
- d) to provide input to the Off-Site Testing (Task 1.4) product-use test program.

Recent Activities

- Part One of the Outline - "Coproduction of Methanol" has been written for release as a Topical Report. Comments from DOE on the 31 March 1997 draft of the Topical Report "Economic Analysis - LPMEOH™ Process as an Add-on to IGCC for Coproduction" are the current basis for discussion. As part of reviewing this report, Air Products has submitted a recommendation that the cost breakdown by plant area matches the format to be used in the Final Report - Volume 1 - Public Design. The Topical Report on the Economic Analysis of LPMEOH™ will be updated and sent to DOE for further comment.
- Part Two of the Outline - "Baseload Power and Methanol Coproduction", has been incorporated into the paper, "Fuel and Power Coproduction - The Liquid Phase Methanol (LPMEOH™) Process Demonstration at Kingsport ", that was presented at the DOE's Fifth Annual Clean Coal Technology Conference in January of 1997.
- Part Four of the Outline - "Methanol Fuel Applications", was used as the basis to update the product-use test program (Task 1.4).

D.3 DME Design Verification Testing

Discussion

The first decision milestone, on whether to continue with dimethyl ether (DME) Design Verification Testing (DVT), was targeted for 01 December 1996. This milestone was relaxed to July of 1997 to allow time for further development of the Liquid Phase Dimethyl Ether (LPDME™) catalyst system. DVT is required to provide additional data for engineering design and demonstration decision-making. The essential steps required for decision-making are: a) confirm catalyst activity and stability in the laboratory, b) develop engineering data in the laboratory, and c) confirm market(s), including fuels and chemical feedstocks. The DME Milestone Plan, showing the DVT work and the decision and implementation timing, is included in Appendix D.

Prior work in this task included a recommendation to continue with DME DVT and Market Economic Studies. Ongoing activity is focusing on Laboratory R&D.

DME DVT Recommendation

DOE issued a letter dated 31 July 1997 accepting Air Products' recommendation to continue with the design verification testing to coproduce DME with methanol, and to proceed with planning a proof-of-concept test run at the DOE's AFDU in LaPorte, Texas. A copy of the recommendation (dated 30 June 1997) is included in Appendix D. The recommendation was based on the results of the Market Economic Studies and on the LPDME™ catalyst system R&D work, and is summarized in the following.

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

Based upon the potential size of the markets and the promise of the LPDME™ catalyst system, proof-of-concept planning for the LaPorte AFDU was recommended. A summary of the DME DVT recommendation is:

- Planning for a DME test run at the LaPorte AFDU, in conjunction with other DOE Liquid Fuels Programs, should be initiated. Test plans, budgets, and a schedule for these LaPorte AFDU tests should now be developed. Up to \$875,000 of Clean Coal Technology Program budget support from the LPMEOH™ Project budget could be made available to support a suitable LPDME™ test run at LaPorte.

- An implementation decision, made mutually by the DOE's Clean Coal Technology Program (DE-FC22-92PC90543) LPMEOH™ project participants, and by the DOE's Liquid Fuels Program (DE-FC22-95PC93052) project participants, will be made in time to meet the schedule for testing at LaPorte.

LPDME™ is not applicable to hydrogen (H₂)-rich syngas; and it is unlikely that a substantive LPDME™ demonstration will be recommended for Kingsport. Therefore, a convincing case that the test-run on CO-rich syngas at LaPorte will lead to successful commercialization must be made, prior to approving the final test-run plan. The strategy for commercialization must present the technical logic to combine the results of the following two areas:

- 1) catalyst performance (productivity, selectivity, and life) for the LPDME™ catalyst system under CO-rich syngas from the proof-of-concept testing at the LaPorte AFDU; and
- 2) reactor performance (methanol catalyst activity and life, hydrodynamics, and heat transfer) from the LPMEOH™ Process Demonstration Unit at Kingsport.

The DME DVT recommendation summarizes the catalyst targets, experimental results, and the corresponding economics for a commercially successful LPDME™ catalyst.

Market Economic Studies

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

Laboratory R&D

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

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

Summary of Laboratory Activity and Results

- A manufacturer for the dehydration catalyst (Calsicat) was selected by the Liquid Fuels Program. The initial schedule (contained in the DME Milestone Plan in Appendix D) showed a catalyst delivery date to the LaPorte AFDU of 01 March 1998. This date could be met assuming that the dehydration catalyst would be produced in a series of campaigns in a pilot plant. The Liquid Fuels Program has determined that it is important to complete the scale-up of the dehydration catalyst as part of the proposed LaPorte run. This will increase the time requirement, as a production test in the pilot plant is still required before operating the commercial catalyst production unit. The new estimated delivery date of dehydration catalyst to LaPorte is 01 June 1998. The DME DVT Recommendation will be updated to reflect the change in schedule and the impact (if any) on the implementation of the coproduction of DME with methanol at the LPMEOH™ Demonstration Unit.
- Recent activities have focused on the scale-up of the manufacturing technique for the dehydration catalyst. The commercial catalyst manufacturer (Calsicat) has prepared the first batch of dehydration catalyst in large-scale equipment. Air Products will test a sample of this material in the laboratory autoclave.

D.4 LPMEOH™ Process Demonstration Facility - Methanol Operation

Table D.4-1 contains the summary table of performance data for the LPMEOH™ Demonstration Unit during the reporting period. These data represent daily averages, typically from a 24-hour material balance period, and those days with less than 12 hours of stable operation are omitted. Appendix E contains samples of the detailed material balance reports which are representative of the operation of the LPMEOH™ Demonstration Unit during the reporting period.

TABLE D.4-1

DATA SUMMARY FOR LPMEOH™ DEMONSTRATION UNIT

Case	Date	Days Onstream	Gas Type	Temp (°C)	Pres. (psig)	Fresh Feed (KSCFH)	Recycle Gas (KSCFH)	Reactor Feed (H ₂ CO)	Purge Gas (KSCFH)	Inlet Sup. Velocity (ft/sec)	Space Velocity (hr ⁻¹ kg)	Slurry Conc. (wt% ox)	Gas Holdup (vol%)	Gas Slurry Hgt (ft)	Catalyst Inventory (lb)	Catalyst Age (eta)	CO Conv. (%)	Reactor O-T-M Conv. (%)	Syngas Util. (SCF/lb)	Raw MeOH Production (TPD)	Catalyst MeOH Prod. (gmol/hr-kg)	Reactor Vol. Prod. (TPD/Cu ft)	U Overall (BTU/hr ft ² F)	Sparger dP (psi)	Sparger Resistance (K)
6	1-Jan-98	12	Balanced	235	680	804	2,211	5.34	59.5	0.73	9,089	32.1	50.6	49.0	19,500	1.28	55.4	23.2	39.9	241.9	32.33	0.118	164	4.22	4.75
6	2-Jan-98	13	Balanced	235	680	906	2,264	3.70	92.0	0.77	9,601	31.0	52.5	53.5	19,500	1.14	43.2	24.2	40.7	267.2	35.73	0.119	153	5.54	4.58
6	3-Jan-98	14	Balanced	235	680	918	2,181	3.98	119.3	0.76	9,434	30.5	51.0	53.0	19,500	1.15	45.9	24.3	41.4	265.9	35.57	0.119	155	5.12	4.68
6	4-Jan-98	15	Balanced	235	680	772	2,289	4.43	49.7	0.74	9,222	30.5	50.4	52.5	19,500	1.12	47.9	22.9	38.1	238.8	31.68	0.107	168	5.27	4.73
6	5-Jan-98	16	Balanced	234	680	735	2,223	5.28	53.5	0.72	8,939	28.9	47.7	51.0	19,500	1.16	52.5	22.1	39.3	224.8	30.05	0.105	169	4.43	4.77
6	6-Jan-98	17	Balanced	235	681	734	2,191	5.37	50.1	0.71	8,843	28.1	50.6	58.8	19,500	1.22	54.8	22.7	39.1	225.5	30.21	0.091	161	4.43	4.72
6	7-Jan-98	18	Balanced	235	680	732	2,168	5.55	53.3	0.70	8,748	28.4	47.7	60.5	19,500	1.27	58.4	22.9	39.2	224.1	30.00	0.088	165	4.32	4.65
6	8-Jan-98	19	Balanced	235	680	727	2,253	4.77	39.8	0.72	8,995	28.8	48.7	62.0	19,500	1.20	50.8	23.2	38.4	227.4	30.42	0.087	170	5.17	4.60
6	9-Jan-98	20	Balanced	235	681	727	2,252	5.08	47.5	0.72	8,905	27.9	50.0	58.5	19,500	1.11	50.8	22.1	39.2	222.7	29.76	0.090	166	4.79	4.77
6	10-Jan-98	21	Balanced	235	680	735	2,221	5.14	52.9	0.72	8,964	28.4	49.8	57.0	19,500	1.08	50.9	22.0	39.5	223.4	29.88	0.093	168	4.59	4.85
6	11-Jan-98	22	Balanced	235	680	735	2,239	4.75	48.2	0.72	8,960	28.4	50.3	57.5	19,500	1.08	48.4	22.2	39.3	224.6	30.03	0.093	168	4.87	4.89
6	12-Jan-98	23	Balanced	235	681	725	2,284	4.55	44.5	0.72	8,991	28.0	48.8	58.0	19,500	1.05	48.9	22.3	38.8	224.5	30.01	0.092	165	5.04	4.83
6	13-Jan-98	24	Balanced	235	680	728	2,273	4.41	43.7	0.73	9,071	28.5	49.9	57.0	19,500	1.04	45.8	22.3	38.7	225.9	30.20	0.094	163	5.18	4.85
6	14-Jan-98	25	Balanced	235	680	728	2,289	5.07	50.3	0.73	9,174	28.5	48.7	55.5	19,500	1.07	48.2	21.4	39.2	223.0	29.81	0.096	167	5.58	5.58
6	15-Jan-98	26	Balanced	235	696	732	2,375	5.06	43.7	0.73	9,388	29.0	48.5	55.0	19,500	1.04	47.8	21.2	39.4	223.1	29.82	0.098	170	5.69	5.30
6	16-Jan-98	27	Balanced	235	700	727	2,360	4.88	45.3	0.73	9,320	28.1	48.2	54.5	19,500	1.01	47.8	21.4	39.0	223.6	29.89	0.098	171	5.83	5.24
6	17-Jan-98	28	Balanced	235	701	731	2,360	4.87	51.9	0.73	9,345	28.5	50.0	54.5	19,500	0.97	48.8	21.1	39.5	221.9	29.87	0.097	172	5.54	5.29
6	18-Jan-98	29	Balanced	235	700	728	2,342	4.84	53.4	0.72	9,278	28.5	49.8	54.0	19,500	0.95	48.2	21.0	39.8	219.7	29.38	0.097	167	5.46	5.34
6	19-Jan-98	30	Balanced	235	700	729	2,336	5.24	67.5	0.72	9,254	30.1	48.5	51.5	19,500	0.95	48.5	20.6	40.0	218.5	29.22	0.101	168	5.03	5.38
6	20-Jan-98	31	Balanced	235	700	729	2,402	4.37	57.9	0.73	9,422	30.4	50.3	52.5	19,500	0.90	42.3	20.8	39.7	220.3	29.45	0.100	163	5.93	5.27
6	21-Jan-98	32	Balanced	235	700	725	2,312	4.75	60.6	0.71	9,185	30.1	48.0	51.0	19,500	0.92	45.2	20.8	39.8	218.8	29.22	0.102	167	5.19	5.34
6	22-Jan-98	33	Balanced	234	710	728	2,397	4.62	50.2	0.72	9,387	30.8	48.7	51.0	19,500	0.93	44.4	20.8	39.1	222.6	29.78	0.104	170	5.45	5.29
6	23-Jan-98	34	Balanced	235	709	734	2,367	4.83	52.2	0.72	9,436	30.7	51.8	53.5	19,500	0.94	45.3	20.5	39.4	221.9	29.65	0.099	155	4.84	4.86
6	24-Jan-98	37	Balanced	234	709	734	2,397	4.81	53.5	0.72	9,391	29.6	48.4	53.5	19,500	0.98	46.1	20.9	39.0	226.0	30.20	0.100	164	4.81	4.89
6	25-Jan-98	38	Balanced	235	711	727	2,385	4.99	54.3	0.72	9,450	30.1	48.8	51.5	19,500	0.95	45.8	20.3	39.6	220.2	29.43	0.102	165	4.59	4.79
6	26-Jan-98	41	Balanced	228	700	735	2,217	5.31	53.7	0.68	7,030	31.1	48.2	61.5	24,800	1.00	52.2	21.7	39.3	224.4	23.63	0.087	160	4.18	4.93
6	31-Jan-98	42	Balanced	225	701	728	2,338	4.34	50.7	0.70	7,255	31.0	47.2	60.5	24,800	0.99	43.3	21.0	39.4	221.7	23.38	0.087	155	4.83	4.76
6	1-Feb-98	43	Balanced	225	700	728	2,315	4.35	55.4	0.70	7,171	31.7	48.2	60.0	24,800	0.97	43.0	20.9	39.9	218.7	22.99	0.087	151	4.82	4.78
6	2-Feb-98	44	Balanced	225	700	732	2,271	4.51	65.0	0.69	7,112	32.0	47.6	58.5	24,800	0.87	44.4	21.2	39.9	220.2	23.16	0.089	154	4.59	4.79
6	3-Feb-98	45	Balanced	225	700	734	2,256	4.42	62.4	0.69	7,118	32.2	47.7	58.0	24,800	0.87	43.5	21.3	40.1	219.5	23.07	0.090	154	4.72	4.92
6	4-Feb-98	46	Balanced	225	700	723	2,337	4.03	51.2	0.70	7,243	31.9	47.0	58.0	24,800	0.87	40.8	21.4	39.2	221.3	23.28	0.091	162	5.18	4.76
6	5-Feb-98	47	Balanced	225	700	732	2,323	3.99	56.1	0.70	7,203	32.9	49.7	58.5	24,800	0.88	40.4	21.4	39.6	221.9	23.32	0.090	159	5.07	4.84
6	6-Feb-98	48	Balanced	225	700	727	2,300	4.18	56.5	0.69	7,148	33.3	48.3	57.0	24,800	0.88	41.7	21.3	39.5	221.0	23.24	0.092	154	4.78	4.85
6	7-Feb-98	49	Balanced	225	702	721	2,037	4.77	65.3	0.63	6,532	33.3	47.8	55.5	24,800	0.89	47.6	21.9	41.0	210.9	22.17	0.090	162	4.11	5.59
3	8-Feb-98	50	Texaco	225	710	735	2,295	0.74	133.6	0.69	7,216	33.7	54.2	62.0	24,800	0.89	12.4	17.8	44.2	198.7	21.15	0.076	147	8.96	4.78
3	9-Feb-98	51	Texaco	225	711	724	2,320	0.76	128.4	0.70	7,288	35.1	53.0	57.0	24,800	0.88	12.2	17.3	44.3	196.0	20.77	0.082	139	8.03	4.78
3	10-Feb-98	52	Texaco	224	710	708	2,343	0.84	123.7	0.70	7,283	37.0	49.4	49.0	24,800	0.84	12.5	16.9	44.2	192.4	20.39	0.094	136	8.87	4.93
3	11-Feb-98	53	Texaco	224	710	700	2,343	0.81	131.1	0.70	7,287	38.8	49.8	48.0	24,800	0.85	12.2	16.8	44.0	190.8	20.25	0.099	135	8.57	5.25
3	12-Feb-98	54	Texaco	224	710	700	2,348	0.74	152.5	0.70	7,314	40.7	46.9	40.5	24,800	0.81	10.9	15.7	46.4	181.0	19.08	0.107	127	10.13	5.45
6	15-Feb-98	57	Balanced	223	710	738	2,218	5.68	133.6	0.67	7,020	38.5	40.6	39.5	24,800	0.85	49.0	16.7	44.7	197.6	20.80	0.120	159	4.64	6.18
6	16-Feb-98	58	Balanced	224	710	728	2,479	3.92	89.9	0.73	7,603	37.9	43.0	42.0	24,800	0.78	35.5	18.6	41.5	210.8	22.15	0.120	149	6.12	5.42
6	17-Feb-98	59	Balanced	224	710	721	2,437	3.49	78.3	0.72	7,519	37.8	45.2	44.0	24,800	0.78	33.7	18.3	40.5	213.6	22.47	0.116	151	6.28	5.23
6	18-Feb-98	60	Balanced	225	710	728	2,413	3.93	83.4	0.71	7,450	37.0	45.3	45.5	24,800	0.78	38.4	18.1	41.2	211.9	22.29	0.111	147	5.63	5.27
6	19-Feb-98	61	Balanced	225	710	728	2,349	4.46	54.7	0.70	6,616	37.1	45.2	50.0	27,450	0.81	43.8	20.4	39.4	221.8	21.07	0.108	148	5.54	5.42
6	20-Feb-98	62	Balanced	225	711	728	2,344	4.11	53.4	0.70	6,616	37.0	46.3	50.0	27,450	0.79	41.2	20.6	39.4	222.5	21.13	0.106	148	5.87	5.38
6	21-Feb-98	63	Balanced	225	710	712	2,328	4.43	54.9	0.69	6,528	37.4	44.2	48.5	27,450	0.78	43.3	20.2	39.4	217.1	20.62	0.107	146	5.32	5.38
6	22-Feb-98	64	Balanced	224	710	728	2,292	4.84	68.5	0.69	6,441	37.2	44.9	49.5	27,450	0.79	45.3	19.9	40.2	212.2	20.16	0.102	142	4.89	5.44
6	23-Feb-98	65	Balanced	225	710	728	2,304	4.46	82.0	0.69	6,524	38.0	47.8	50.5	27,450	0.81	44.1	20.5	39.7	220.0	21.11	0.104	153	5.31	5.42
6	24-Feb-98	66	Balanced	225	710	728	2,281	4.52	61.1	0.68	6,427	37.4	47.0	51.0	27,450	0.80	44.8	20.4	39.8	219.3	20.84	0.102	155	5.17	5.47
6	25-Feb-98	67	Balanced	225	710	728	2,258	4.47	61.8	0.68	6,409	38.0	47.3	50.0	27,450	0.79	44.6	20.8	39.7	218.7	20.87	0.105	156	5.16	5.47
6	26-Feb-98	68	Balanced	225	710	724	2,249	4.49	65.2	0.68	6,397	36.5	43.4	49											

TABLE D.4-1

DATA SUMMARY FOR LPMEOH™ DEMONSTRATION UNIT

Case	Date	Days Onstream	Gas Type	Temp (Deg C)	Pres. (psig)	Fresh Feed (KSCFH)	Recycle Gas (KSCFH)	Reactor Feed (H ₂ CO)	Purge Gas (KSCFH)	Inlet Sup. Velocity (ft/sec)	Space Velocity (1/hr-kg)	Slurry Conc. (wt% ox)	Gas Holdup (vol%)	Gas Slurry Hgt (ft)	Catalyst Inventory (lb)	Catalyst Age (eta)	CO Conv. (%)	Reactor O-T-M Conv. (%)	Syngas Util. (SCF/lb)	Raw MeOH Production (TPD)	Catalyst MeOH Prod. (gmol/hr-ft ² F)	Reactor Vol. Prod. (TPD/Cu ft)	U Overall (BTU/hr-ft ² F)	Sparger dP (psi)	Sparger Resistance (°K)
6	2-Mar-98	72	Balanced	225	710	721	2,287	4,21	50.7	0.88	6,421	35.1	51.0	60.5	27,450	0.81	43.7	21.8	38.8	223.4	21.22	0.088	148	5.39	5.38
6	3-Mar-98	73	Balanced	225	710	722	2,288	4,21	53.0	0.88	6,427	34.6	48.3	58.5	27,450	0.78	42.8	21.2	39.1	221.4	21.03	0.090	148	5.32	5.48
6	4-Mar-98	74	Balanced	225	710	729	2,234	4,25	58.8	0.87	6,364	32.8	47.2	62.0	27,450	0.78	43.2	21.2	39.7	220.4	20.93	0.084	144	5.28	5.53
6	5-Mar-98	75	Balanced	225	710	728	2,217	4,44	61.0	0.87	6,334	32.3	47.0	63.0	27,450	0.79	44.8	21.4	39.5	221.3	21.02	0.083	143	5.07	5.55
6	6-Mar-98	76	Balanced	225	710	728	2,194	4,53	61.7	0.88	6,262	32.3	48.2	62.0	27,450	0.79	45.4	21.3	39.8	218.0	20.80	0.084	143	4.80	5.58
6	7-Mar-98	77	Balanced	225	710	728	2,212	4,32	61.9	0.87	6,295	33.5	45.8	58.5	27,450	0.79	44.1	21.4	39.7	220.3	20.93	0.089	144	5.09	5.50
6	8-Mar-98	78	Balanced	225	710	731	2,202	4,33	64.7	0.88	6,261	33.8	44.4	56.5	27,450	0.80	44.5	21.8	39.9	218.9	20.90	0.093	148	5.05	5.34
6	9-Mar-98	79	Balanced	225	710	728	2,250	4,02	59.2	0.88	6,411	38.4	48.4	55.5	27,450	0.76	41.1	21.2	39.5	221.4	21.03	0.085	150	5.29	5.39
6	10-Mar-98	80	Balanced	225	710	720	2,260	4,37	63.3	0.88	6,401	38.2	50.8	57.5	27,450	0.75	42.6	20.8	40.0	215.9	20.50	0.089	145	5.01	5.48
6	11-Mar-98	81	Balanced	225	709	742	2,257	4,13	73.9	0.88	6,436	35.8	48.9	58.5	27,450	0.75	41.4	20.8	40.8	219.6	20.86	0.092	148	5.28	5.45
6	12-Mar-98	82	Balanced	225	710	756	2,244	3.88	75.9	0.88	6,441	35.9	48.2	56.5	27,450	0.74	39.7	21.0	41.0	221.1	21.01	0.093	151	5.40	5.40
6	13-Mar-98	83	Balanced	225	710	741	2,215	4.15	84.3	0.87	6,347	35.7	47.8	55.5	27,450	0.73	41.0	20.7	41.2	215.9	20.51	0.093	154	5.08	5.45
6	14-Mar-98	84	Balanced	225	709	735	2,228	4.11	85.5	0.87	6,343	35.4	48.2	54.5	27,450	0.74	40.8	20.7	40.7	218.9	20.81	0.095	159	5.06	5.48
6	15-Mar-98	85	Balanced	225	709	733	2,319	3.27	87.5	0.70	6,569	35.9	48.4	53.5	27,450	0.70	33.6	20.1	40.8	217.0	20.62	0.097	158	6.16	5.45
6	16-Mar-98	86	Balanced	225	710	715	2,189	4.08	82.9	0.85	6,113	38.0	45.4	52.5	27,450	0.70	40.3	20.4	41.5	206.5	19.82	0.094	158	5.23	6.09
6	17-Mar-98	87	Balanced	225	709	728	2,283	3.91	83.8	0.89	6,474	38.8	45.2	50.5	27,450	0.69	37.4	18.8	41.5	208.7	19.93	0.099	152	5.51	5.53
6	18-Mar-98	88	Balanced	225	709	727	2,271	3.38	82.0	0.88	6,462	37.3	48.7	51.0	27,450	0.67	34.0	20.2	41.1	212.1	20.16	0.089	148	5.80	5.48
6	19-Mar-98	89	Balanced	225	709	733	2,242	3.48	85.5	0.88	6,396	37.0	45.5	50.5	27,450	0.70	35.2	20.4	41.3	212.9	20.24	0.100	151	5.85	5.41
6	20-Mar-98	90	Balanced	225	710	733	2,268	3.24	82.2	0.88	6,444	38.7	47.1	48.5	27,450	0.69	33.3	20.4	41.2	213.3	20.27	0.105	154	5.81	5.31
6	21-Mar-98	91	Balanced	224	710	730	2,274	3.33	82.6	0.88	6,447	40.0	47.0	48.0	27,450	0.69	33.6	20.3	41.1	213.2	20.28	0.111	159	5.84	5.37
6	22-Mar-98	92	Balanced	224	710	728	2,247	3.40	85.5	0.88	6,413	40.3	48.5	45.0	27,450	0.68	34.0	20.1	41.3	211.8	20.12	0.112	156	5.55	5.43
6	23-Mar-98	93	Balanced	224	710	728	2,219	3.63	96.5	0.87	6,358	38.7	43.9	48.5	27,450	0.68	35.4	19.9	41.9	208.4	19.88	0.100	138	5.27	5.48
6	24-Mar-98	94	Balanced	224	710	715	2,223	3.88	98.5	0.87	6,298	38.1	43.4	48.5	27,450	0.68	38.5	18.6	42.2	203.2	19.32	0.104	143	5.04	5.49
6	25-Mar-98	95	Balanced	224	710	674	2,235	4.02	74.0	0.88	6,231	38.8	42.4	44.5	27,450	0.67	37.2	19.3	40.9	197.9	18.81	0.103	148	4.98	5.52
6	26-Mar-98	96	Balanced	224	710	685	2,187	4.12	80.5	0.85	6,189	37.4	41.8	46.5	27,450	0.69	38.7	19.7	41.0	200.7	19.08	0.103	149	4.87	5.82
6	27-Mar-98	97	Balanced	224	710	675	2,225	3.58	88.1	0.86	6,243	37.3	41.7	48.5	27,450	0.68	34.3	19.5	40.8	198.6	18.88	0.102	146	5.47	5.80
6	28-Mar-98	98	Balanced	224	710	674	2,111	4.57	90.0	0.83	6,003	37.8	40.2	44.5	27,450	0.68	41.3	19.5	41.9	182.9	18.35	0.103	150	4.47	5.78
6	29-Mar-98	99	Balanced	224	710	682	2,223	3.63	80.6	0.88	6,242	38.2	41.8	43.0	27,450	0.65	34.4	18.3	41.2	198.6	18.89	0.110	150	5.23	5.51
6	30-Mar-98	100	Balanced	224	710	681	2,175	3.98	84.7	0.85	6,153	38.8	41.2	43.5	27,450	0.67	36.8	18.4	41.5	196.8	18.72	0.108	148	4.88	5.51
6	31-Mar-98	101	Balanced	224	709	681	2,176	3.93	79.4	0.85	6,152	38.4	41.8	44.5	27,450	0.68	37.2	19.7	41.0	199.4	18.96	0.107	147	4.91	5.48

Appendix F, Table 1 contains the summary of outages for the LPMEOH™ Demonstration Unit during this quarter. Availability exceeded 99%, as the plant continued to operate through the longest continuous campaign to date (45 days) as of 31 March 1998.

During the reporting period, a total of 5,762,047 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Apart from 12,000 gallons shipped off-site for product-use testing, Eastman accepted all of this methanol for use in the production of methyl acetate, and ultimately cellulose acetate and acetic acid. No safety or environmental incidents were reported during this quarter.

Operations focused on resolution of key issues identified during prior operating periods.

Catalyst Life (η) - December 1997 - March 1998

The “age” of the methanol synthesis catalyst can be expressed in terms of a dimensionless variable η , which is defined as the ratio of the rate constant at any time to the rate constant for freshly reduced catalyst (as determined in the laboratory autoclave). Appendix F, Figure 1 plots $\log \eta$ versus days onstream from the restart in December of 1997 through the end of the reporting period. Since catalyst activity typically follows a pattern of exponential decay, the plot of $\log \eta$ is fit to a series of straight lines, with step-changes whenever fresh catalyst was added to the reactor.

As reported in Technical Progress Report No. 14, the operating temperature of the LPMEOH™ Reactor was set at 235°C in December of 1997. At this temperature the nameplate capacity of 80,000 gallons-per-day (260 TPD) of methanol could be achieved at a low syngas utilization (i.e. high overall conversion of syngas to methanol). This result demonstrated the excellent initial activity of the catalyst, in that the design production capacity of methanol could be achieved at a lower reactor temperature and pressure than during the April 1997 operating period (refer to Technical Progress Report No. 12 for these results). In fact, at 235°C the unit initially produced 280 TPD of methanol.

The Balanced Gas flowrate was reduced to a nominal 700 KSCFH on 04 January 1998 to maintain a low syngas utilization for the remainder of the initial material balance period. As shown in Appendix F, Figure 1, the decrease in η with time from the period 20 December 1997 through 27 January 1998 occurred at a rate of 1.0% per day, which represented a significant improvement over the 3.4% per day decline measured during the initial six weeks of operation in April and May of 1997. The deactivation rate also improved from the longer-term rate of 1.6% per day seen throughout the summer and autumn of 1997.

Based on this improvement, DOE accepted a recommendation by Air Products and Eastman to further reduce the reactor temperature to 225°C. The subsequent change in conditions began on 28 January 1998, when two consecutive batches of fresh catalyst were activated and transferred to the LPMEOH™ Reactor, bringing the total inventory to 24,800 pounds, or about 60% of design. Reductant gas uptake for both batches approached the theoretical value to within 1%. Following addition of the second catalyst batch on 30 January, the reactor temperature was reduced to 225°C. Subsequent performance results, as regressed

with the reactor kinetic model, showed a slight increase in η on the temperature transition day, followed by a significant decrease in η at 225°C. No operating transients occurred during this time which could have resulted in such a drastic performance decline. It appears, therefore, that the lower calculated catalyst age is a temperature-related artifact of the reactor kinetic model induced by further departure from the autoclave baseline temperature of 250°C.

On 08 February 1998, a planned five-day outage in Eastman's shift reactor provided an opportunity to continue operations with a Texaco-type (CO-rich) reactor feed, while Eastman's gas-phase methanol plant was forced to shut down. The results from this test are included in Table D.4-1 and Appendix F, Figure 1. Furthermore, Eastman took the opportunity to shut down their methanol distillation equipment for maintenance, leaving no flow path for the crude methanol underflow from the second, rectifier column in the LPMEOH™ Demonstration Unit. However, because of the CO-rich feed, the rectifier column could be shut down for the production of stabilized, "fuel-grade" methanol (98+ wt% methanol, <1 wt% water). Approximately 12,000 gallons of "fuel-grade" methanol was loaded into trailers and shipped off-site for future product-use testing; Appendix G contains a series of analytical reports on the purity of this product. During the remainder of this operating period, approximately 181,800 gallons (600 tons) of stabilized methanol (99.3 wt% methanol, 0.3 wt% water) was pumped directly from the LPMEOH™ Demonstration Unit to Eastman's methyl acetate plant, bypassing the normal route through the distillation equipment. This methanol was considered acceptable by Eastman because of the low levels of all impurities. In particular, the water concentration of the stabilized methanol was significantly lower than what is produced during operation on the normal Balanced Gas supply (typically 3 - 4 wt% water). This is a result of operation on a CO-rich syngas with a relatively low CO₂ concentration. This test was the first attempt to feed a stabilized methanol stream with low water content directly into one of Eastman's downstream chemical processes, providing another indication of the flexibility of the LPMEOH™ Process. Eastman's only alternative was to flare the syngas stream, which was undesirable both environmentally and economically.

On 19 February 1998, a third batch of catalyst was activated and transferred to the LPMEOH™ Reactor, bringing the total catalyst inventory to 27,450 pounds, or about 67% of design. Prior to the addition of fresh catalyst, the average deactivation rate at 225°C was 0.7% per day, which represented a modest improvement over the 1% per day rate calculated at 235°C. However, most of the activity decline occurred during the period on CO-rich feed gas and immediately after an interruption in the Balanced Gas supply at the end of that test case. This observation more likely indicates a real change in catalyst activity, as opposed to an anomaly of the kinetic model, because deviations from steady-state operation in the syngas generation area could also cause upsets in the level of poisons carried by the gas. This type of behavior seems to have occurred before, and the more frequent catalyst sampling protocol in effect since the December restart may provide clues about the types of poisons that become prevalent during such upsets. So far, samples from mid-February and mid-March of 1998 have shown an increase in arsenic loading, although not nearly to the levels measured in the summer of 1997. Copper crystallite size measurements are still pending, as are analyses from more recent samples which will help complete the picture.

Through the remainder of the reporting period, the Balanced Gas flow rate was maintained at approximately 700 KSCFH, with reactor temperature fixed at 225°C. As shown in Appendix F, Figure 1, two periods of very stable operation ensued, with average activity declines of 0.36% and 0.27% per day. A discontinuity occurred in the data on 15 March 1998, again coincidental with an excursion in the Balanced Gas feed supply. Regressing the entire data set from the catalyst addition on 19 February through 31 March 1998 yields a deactivation rate of 0.60% per day. However, the data were plotted in two separate sets to show the possibility of an improved steady-state baseline deactivation rate, exclusive of any additive effects related to excursions in the Balanced Gas supply. If this conclusion is viable, the current baseline activity decline of about 0.4% per day is a measurable improvement over the 1% per day rate calculated at 235°C in January of 1998 and matches the original target from the 4-month proof-of concept run on a natural-gas derived syngas at the LaPorte AFDU. Additional operating time is necessary to further quantify the catalyst deactivation rate and the effects of operating conditions and syngas supply excursions.

Sparger Resistance

As reported in Technical Progress Report No. 14, flow resistance through the gas sparger of the LPMEOH™ Reactor had been stabilized using a continuous flush of condensed oil and entrained slurry from the 29C-05 secondary oil knock-out drum and 29C-06 cyclone. These streams were gravity-drained back to the reactor through a flush connection at the gas inlet line to the reactor, thus eliminating a batch-transfer operation which had been used during prior operation. The flow rate of the flush was equivalent to the average rate of liquid traffic in the reactor loop (1 to 2 gallons per minute).

This technique was first applied to a clean sparger at the restart of operations on 19 December 1997. Appendix F, Figure 2 plots the average daily sparger resistance coefficient since then, and provides an initial confirmation of the encouraging results reported for the first two weeks of operation at the end of the prior reporting period. As with the original gas sparger design, the return of the internal oil and slurry streams via the flush connection was required to stabilize the pressure drop after extended interruptions of the flush fluid. When compared to the original gas sparger, the alternative gas sparger may have greater flexibility in maintaining stable pressure drop after interruptions of the syngas supply; this may also be a result of greater attention to achieving a proper standby condition during shutdowns, which has inhibited any plugging caused by slurry backflow and stagnation. The data for this plot, along with the corresponding average pressure drop, are included in Table D.4-1. Appendix F, Figure 3 shows the progressive improvement in sparger operation seen since the original startup in April of 1997. This parameter will continue to be closely monitored for any change in flow resistance.

Alternative Fuels Field Trailer Unit (AFFTU) Results - December 1997 - January 1998

The Alternative Fuels Field Test Unit (AFFTU), a transportable laboratory equipped with an autoclave and analytical equipment, was shipped from the Air Products' Iron Run laboratory in Allentown, PA to Kingsport to perform additional testing on the reactor feed gas at the

site. From the restart on 19 December 1998, the autoclave operated in parallel with the Demonstration Unit on the same reactor feed gas, although at 250°C, the typical laboratory baseline condition. Analytical results from the AFFTU showed less than 10 ppbv concentrations of metal carbonyls and hydrogen sulfide within the reactor loop. Carbonyl sulfide was typically less than 10 ppbv in the loop, but occasionally drifted higher; these excursions could not be correlated with any changes in the feed gas cleanup operations upstream of the LPMEOH™ facility. Performance results from the 31-day campaign on coal-derived syngas at Kingsport were generally consistent with other laboratory experiments on poison-free syngas. A post-mortem analysis on the catalyst revealed no unusual levels of catalyst poisons or significant changes in catalyst physical properties. The AFFTU test concluded on 20 January 1998, and the equipment was returned to the Air Products' Iron Run laboratory.

D.5 Planning and Administration

Work has continued on the Final Report - Volume 1 - Public Design. Air Products received the latest comments from DOE (letter dated 12 October 1997). In response to these comments, Air Products has submitted a cost breakdown for both capital and operating costs within the LPMEOH™ Demonstration Unit. These will ultimately be incorporated into an updated version of this report which will be sent to DOE for comment.

The Milestone Schedule Status Report and the Cost Management Report, through the period ending 31 March 1998, 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 (WBS) tasks. Ninety-nine percent (99%) of the \$38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 31 March 1998. Nineteen percent (19%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 March 1998.

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.

A paper entitled "Commercial-Scale Demonstration of the Liquid Phase Methanol (LPMEOH™) Process: Initial Operating Experience" was submitted for presentation at the Clean Coal Technology Conference in Reno, Nevada on April 29, 1998.

A draft topical report entitled "Design and Fabrication of the First Commercial-Scale LPMEOH™ Reactor" was circulated for internal Air Products review prior to formal submission.

E. Planned Activities for the Next Quarter

- Write and submit the Demonstration Technology Start-up Report to DOE.
- Continue to analyze catalyst slurry samples and gas samples to determine causes for deactivation of methanol synthesis catalyst.
- Continue executing Phase 3, Task 2.1 Methanol Operation per the Demonstration Test Plan. Focus activities on increasing catalyst concentration in the LPMEOH™ Reactor to determine the maximum slurry concentration (Test 9 of Test Plan).
- Reissue the DVT Recommendation for a DME proof-of-concept test run at the LaPorte AFDU to reflect the final delivery date for the dehydration catalyst and the actual schedule for the test run.
- Continue execution of the Off-Site, Product-Use Test Program (Phase 1, Task 1.4).
- Continue to incorporate DOE comments into the Topical Report on Process Economic Studies.
- Reach agreement with DOE on the equipment breakdown and operating cost summary for use in the Final Technical Report, Volume 1, Public Design Report.
- Issue the Topical Report on Liquid Phase Reactor Design to DOE for review and comment.
- Present the paper "Commercial-Scale Demonstration of the Liquid Phase Methanol (LPMEOH™) Process: Initial Operating Experience" at the Clean Coal Technology Conference in Reno, Nevada on April 29, 1998.

F. Conclusion

During this quarter, initial planning and procurement work continued on the seven project sites which have been accepted for participation in the off-site, product-use test program. Approximately 12,000 gallons of fuel-grade methanol (98+ wt% methanol, <1 wt% water) produced during operation on carbon monoxide (CO)-rich syngas at the LPMEOH™ Demonstration Unit was loaded into trailers and shipped off-site for future product-use testing. At one of the projects, three buses have been tested on chemical-grade methanol and on fuel-grade methanol from the LPMEOH™ Demonstration Project. At two other project sites (a flexible fuel vehicle and a fuel cell application), testing on fuel-grade methanol from the LPMEOH™ Demonstration Project is scheduled for the second quarter of calendar year 1998.

During the reporting period, planning for a proof-of-concept test run of the Liquid Phase Dimethyl Ether (LPDME™) Process at the Alternative Fuels Development Unit (AFDU) in LaPorte, TX continued. The commercial catalyst manufacturer (Calsicat) has prepared the first batch of dehydration catalyst in large-scale equipment. Air Products will test a sample of this material in the laboratory autoclave.

Catalyst activity, as defined by the ratio of the rate constant at any point in time to the rate constant for freshly reduced catalyst (as determined in the laboratory autoclave), was monitored for the initial extended operation at the lower initial reactor operating temperature of 235°C. At this condition, the decrease in catalyst activity with time from the period 20 December 1997 through 27 January 1998 occurred at a rate of 1.0% per day, which represented a significant improvement over the 3.4% per day decline measured during the initial six weeks of operation in April and May of 1997. The deactivation rate also improved from the longer-term rate of 1.6% per day calculated throughout the summer and autumn of 1997.

Based on this improvement, DOE accepted a recommendation by Air Products and Eastman to further reduce the reactor temperature to 225°C. The initial operation at this temperature (from 31 January through 18 February 1998) showed a modest improvement in the deactivation rate to 0.7% per day. However, most of the activity decline occurred during a test period on CO-rich feed gas and immediately after an interruption in the Balanced Gas supply at the end of that test case. This observation more likely indicated a real change in catalyst activity, and this type of behavior seems to have occurred after other excursions in the Balanced Gas supply. During two additional stable operating periods between 19 February and 31 March 1998, deactivation rates of 0.27% and 0.36% per day were measured. A discontinuity occurred in the data on 15 March 1998, again coincidental with an excursion in the Balanced Gas supply. If the current results prove to be correct, the current baseline activity decline of about 0.4% per day is a measurable improvement over the 1% per day rate seen at 235°C in January of 1998 and matches the original target from the 4-month proof-of-concept run on a natural-gas derived syngas at the LaPorte AFDU. Additional operating time is necessary to further quantify the catalyst deactivation rate and the effects of operating conditions and syngas supply excursions.

The weight of catalyst in the LPMEOH™ Reactor has reached 67% of the design value, and the slurry concentration approached 40 wt%. Catalyst slurry samples from the LPMEOH™ Reactor have been taken on a regular basis to correlate any change in performance with changes in the physical properties of the catalyst. So far, samples from mid-February and mid-March of 1998 have shown an increase in arsenic loading, although not nearly to the levels measured in the summer of 1997. Copper crystallite size measurements are still pending, as are analyses from more recent samples which will help complete the picture.

The performance of the alternative gas sparger, which was designed by Air Products and installed into the LPMEOH™ Reactor prior to the restart of the LPMEOH™ Demonstration Unit in December of 1997, was monitored throughout the reporting period. Pressure drop through the gas sparger of the LPMEOH™ Reactor was stabilized using a continuous flush of condensed oil and entrained slurry which were gravity-drained from the 29C-05 secondary oil knock-out drum and 29C-06 cyclone. As with the original gas sparger design, the return of the internal oil and slurry streams via the flush connection was required to stabilize the pressure drop after extended interruptions of the flush fluid. When compared to the original gas sparger, the alternative gas sparger may have greater flexibility in maintaining stable pressure drop after interruptions of the syngas supply; this may also be a result of greater attention to achieving a proper standby condition during shutdowns. The most recent results provide an initial confirmation of the encouraging data reported for the

first two weeks of operation at the end of the prior reporting period. This parameter will continue to be closely monitored for any change in flow resistance.

The Alternative Fuels Field Test Unit (AFFTU), a transportable laboratory equipped with an autoclave and analytical equipment, was shipped from the Air Products' Iron Run laboratory in Allentown, PA to Kingsport to perform additional testing on the reactor feed gas at the site. Analytical results from the AFFTU showed less than 10 ppbv concentrations of metal carbonyls and hydrogen sulfide within the reactor loop. Carbonyl sulfide was typically less than 10 ppbv in the loop, but occasionally drifted higher; these excursions could not be correlated with any changes in the feed gas cleanup operations upstream of the LPMEOH™ facility. Performance results from the 31-day campaign on coal-derived syngas at Kingsport were generally consistent with other laboratory experiments on poison-free syngas. A post-mortem analysis on the catalyst revealed no unusual levels of catalyst poisons or significant changes in catalyst physical properties. The AFFTU test concluded on 20 January 1998, and the equipment was returned to the Air Products' Iron Run laboratory.

During the reporting period, a total of 5,762,047 gallons of methanol was produced at the LPMEOH™ Demonstration Unit. Apart from 12,000 gallons shipped off-site for product-use testing, Eastman accepted all of this methanol for use in the production of methyl acetate, and ultimately cellulose acetate and acetic acid. During a portion of the operating period on CO-rich syngas, approximately 181,800 gallons (600 tons) of stabilized methanol (99.3 wt% methanol, 0.3 wt% water) was pumped directly from the LPMEOH™ Demonstration Unit to Eastman's methyl acetate plant, bypassing the normal route through the distillation equipment designed to remove water, higher alcohols, and process oil. This test was the first attempt to feed a stabilized methanol stream with low water content directly into one of Eastman's downstream chemical processes, providing another indication of the flexibility of the LPMEOH™ Process. No safety or environmental incidents were reported during this quarter. Availability exceeded 99%, as the demonstration unit continued to operate through the longest continuous campaign to date (45 days) as of 31 March 1998.

Ninety-nine percent (99%) of the \$38 million of funds forecast for the Kingsport portion of the LPMEOH™ Process Demonstration Project for the Phase 1 and Phase 2 tasks have been expended (as invoiced), as of 31 March 1998. Nineteen percent (19%) of the \$158 million of funds for the Phase 3 tasks have been expended (as invoiced), as of 31 March 1998.

APPENDICES

APPENDIX A - SIMPLIFIED PROCESS FLOW DIAGRAM

SIMPLIFIED PROCESS DIAGRAM KINGSPORT LPMEOH - 260 TPD PLANT LPMEOH™ PROCESS DEMONSTRATION FACILITY

APPENDIX B - OFF-SITE TESTING (DEFINITION AND DESIGN)

Appendix B-1 - Summary Table of Eight Candidates (one page)

Quarterly Reports:

Appendix B-2 - Acurex FFV (one page)

Appendix B-3 - Stationary Turbine for VOC Control (one page)

Appendix B-4 - West Virginia University Stationary Gas Turbine (two pages)

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Appendix B-6 - University of Florida Fuel Cell (one page)

Appendix B-7 - West Virginia University Tri-Boro Bus (three pages)

Appendix B-8 - Florida Institute of Technology Bus & Light Vehicle (twenty-two pages)

ARCADIS GERAGHTY & MILLER

ARCADIS Geraghty & Miller, Inc.
555 Clyde Avenue
Mountain View
California 94043
Tel 650 961 5700
Fax 650 254 2496

MEMO

To:
Larry Waterland

Copies:

TRANSPORTATION
TECHNOLOGY

From:
Doug Coleman

Date:
29 April 1998

Subject:
LPMEOH™ FFV Demonstration Status Report

Progress on the FFV demonstration continues to be hampered in part by problems with the California Energy Commission (CEC) methanol fuel dispensers. The Taurus has often been unable to refuel at sites which were supposedly operational. And in many instances it has not been possible to obtain a readout on the amount of fuel dispensed. Both of these factors have affected the ability to develop as much fuel economy data as desired. Thus, since the inception of this program, while the FFV has traveled roughly 8,000 miles, only 1,300 of these miles have yielded acceptable fuel economy data. The FFV has been achieving a 16.3 mpg fuel economy on regular M85 during this time period.

With the CEC's recent announcement to phase out all of the methanol stations by the end of the year, it appears that the stations will continue to be in a state of disrepair and are no longer usable. Thus, operation on LPMEOH™ will be initiated before data for the originally planned 3,200 miles on regular M85 have been acquired. The first drum of LPMEOH™-based M85 has been prepared and this phase of the project will now begin. Before doing so, however, the FFV will undergo emissions testing on regular M85 during the week of April 27.

During the LPMEOH™ phase, ARCADIS Geraghty & Miller will mix 8.25 gallons of RUL with 46.75 gallons of LPMEOH™ to obtain a LPMEOH™-based M85 mixture. The fuel will be measured during dispensing with the use of a scale. When the LPMEOH™ phase is completed and the FFV has undergone emissions testing on that fuel, the 1,900 miles remaining on regular M85 will be completed using fuel purchased in drums and stored at ARCADIS Geraghty & Miller in the same manner that was used to fuel with LPMEOH™-based M85.

ARCADIS GERAGHTY & MILLER

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MEMO

To:
Larry Waterland

Copies:

ENGINEERING SYSTEMS

From:
Carlo Castaldini

Date:
29 April 1998

Subject:
Project update report: LPMEOH™ Testing in a Gas Turbine for Control

The objective of this project is to demonstrate the use of a mini gas turbine for the destruction of VOC from an emission source while burning LPMEOH™. Mini gas turbines, with a gross generating capacity of 75 kW or less, promise to be one of the most successful technologies for distributed power generation in the new deregulated power industry. There are two main considerations in the use of these mini turbines. The first is the cost of their operation and the second is their emissions. When the mini turbine is also used to destroy VOC from vented process air, the economics improve. The use of methanol for remote applications or for emission control may alleviate other key obstacles to commercial development.

Efforts since the previous progress report have focused on selecting a host site for the demonstration. Of the four candidate sites identified in the previous report, the one most interested in supporting the project was the Allied Signal facility in Phoenix, Arizona. Allied Signal has agreed to be the host site for the LPMEOH™ tests using their 525 kW ASE8-1000 gas turbine engine in a laboratory set up. On March 5, Carlo Castaldini, the ARCADIS Geraghty & Miller Project Manager for this project, met at the Allied Signal Phoenix plant with Paul Dodge, Program Manager of Industrial Engines, and Jeff Zimmerman, Project Engineer, Industrial/Marine applications, to discuss the scope and the time frame for the demonstration tests. Allied Signal agreed to the overall objective and confirmed their plans to support the project. Several discussions were held after the meeting to further clarify the proposed approach and logistics for methanol storage, test duration, and measurements. Their laboratory facility can be readily modified to store and handle methanol.

Allied Signal is currently reviewing the project scope in greater detail and will provide suggestions regarding the specific testing they would recommend. A followup meeting at Allied Signal is scheduled for May 14 to discuss testing and other project-specific details.

WVU Progress Report

Dated May 1st, 1998

For

Air Products and Chemical Inc.

Methanol Utilization Demonstration Project

WVU Emission Studies on a
Dual-Fuel, Methanol/Jet Fuel
Stationary Gas Turbine.
350 HP Type GTC-85
Manufactured by Allied Signals Inc.

Report Prepared for

Robert J. Senn
Air Products and Chemical Inc
7201 Hamilton Blvd
Allentown PA 18195-1501

Tel: (610) 481-7042
Fax: (610) 706-7299

by

John L. Loth and Nigel N. Clark

Department of Mechanical and Aerospace Engineering
West Virginia University

P. O. Box 6106, Morgantown WV 26505-6106

Status Summary

The GTC gas turbine has been fully instrumented and the fuel system has been converted to allow convenient fuel type change over between Jet fuel and Methanol. Our operating procedure is geared to prevent methanol cold starting problems. Therefore we always ignite on jet fuel and only after an adequate warm up period (5 minutes), is the fuel change over implemented. Five minutes before shut down, the fuel type is changed back to jet fuel. The turbine has been run successfully on jet fuel and all the instrumentation checked out to be working as planned. Three attempts were made to change over to methanol fuel at idle speed. All three attempts resulted in a flame-out. In view of this problem the gas-turbine combustor and the fuel controller were taken to Pratt and Whitney aircraft for refurbishing. A standard Pratt and Whitney PT6 dual glow plug ignition system was welded into the combustor can. This will be used in addition to the existing spark ignition. Another measure to prevent gas turbine flame-out in the future, is to limit fuel type change to operations at high exhaust gas temperature (EGT at least 1000°F). We have been having some difficulty in procuring a new fuel shut-off solenoid, as the old one leaks. However we are confident to be back on line by June 1 1998. We may have to request a no-cost extension for contract completion till September 15, 1998. Note this seems not unreasonable as WVU did not receive official contract authorization till September 22, 1997.

ARCADIS GERAGHTY & MILLER

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MEMO

To:
Larry Waterland

Copies:
Copies

ENGINEERING SYSTEMS

From:
Shyam Venkatesh

Date:
May 1, 1998

Subject:
Project update report: LPMEOH™ testing with -86 generators at Tyndall AFB

In January 1998, A-55 LP withdrew their participation from the program. The reasons given by A-55 for their withdrawal were specified in the previous progress report. Applied Research Associates (ARA), the operating contractor at Tyndall AFB coordinating the LPMEOH™ program, requested that they be allowed to test their proprietary water-based emulsion fuel. The ARA emulsion fuel is JP-8 based. JP-8 was selected instead of diesel because of the Air Force's desire to universalize its fuels in the future.

ARA began experimentation with preliminary formulations of their fuel in March and April. ARCADIS Geraghty & Miller consulted with ARA in the development of an emulsion mixing system. ARCADIS Geraghty & Miller suggested that a high shear-rate centrifugal pump system be used instead of a more conventional mixer. ARCADIS Geraghty & Miller also procured for ARA the emulsifying agents and other proprietary chemical additives. Due to scheduling conflicts, and prior commitments, ARA was not able to conduct preliminary testing with the emulsion in April. ARA expects to perform scoping tests in May, based on which they will determine the best emulsion composition. Currently the emulsion fuel is designed to use up to 10 percent methanol. The best percentage of methanol to actually use will be determined after the scoping tests. Barring any further schedule conflicts, scoping tests are expected to be completed in May. Following these, the actual testing phase of the project will begin immediately.

To : Dr. V.P. Roan
From : Jim Fletcher
Concerning : Air Products "Coal-based Methanol" Contract Progress Report
Date : May 10, 1998

This memo details the progress accomplished towards the completion of the Air Products "Coal-based Methanol as a Fuel for PAFC Systems" contract for the February through April time period.

Two 55 gallon barrels of Air Products methanol were delivered at the end of April. The barrels were received, clearly marked, and moved into the fuel storage area.

A second testing laboratory, Intertek Testing Services, has been identified. It is envisioned that both labs will perform a comprehensive analysis of the Air Products methanol. In addition, Intertek will also perform analysis to identify any sulfur or chlorides within the methanol. These two elements have been identified as problems for the fuel cell system. Intertek has performed similar tests for Georgetown University on the methanol to be used in the 40 ft. bus program. The required paperwork is underway and testing should begin within the next week or so. Work is also continuing to further identify any problem constituents.

It was determined that the fuel cell data acquisition system had been damaged during a recent power outage. The system was sent to the manufacturer for repair, and has been returned in working order. Various thermocouples, an air flow meter, and a fuel flow meter have been added to the system. The labview data acquisition software has been upgraded to incorporate the new hardware. Preliminary baseline testing of the fuel cell system has begun using fuel grade methanol.

The gas chromatograph has been ordered and received. Preliminary testing and debugging of the system has begun using known gases (air, etc.). The design of the gas collection system is complete and will be ordered in the near future. The remaining concern is the exact nature of the interface between the gas chromatograph and the collection bottles. The interface will be completed within the next week.

The fuel cell system is operational with the upgrading of various components within the system. The upgrades include the installation of a pressure relief valve on the CO₂ line. The method of in-lab storage and fuel delivery for the Air Products methanol is complete and purchase orders for the necessary equipment have been written.

It is expected that testing of the Air Products methanol within the fuel cell system will commence in June pending successful results of the chemical analysis.

FUEL GRADE METHANOL IN TRANSIT BUS APPLICATIONS

PROGRESS REPORT, APRIL 1998

Submitted by: Nigel N. Clark, Principal Investigator
Department of Mechanical & Aerospace Engineering
West Virginia University
Morgantown, WV 26506-6106

The objectives of this program are to quantify the emissions and evaluate the performance of full size transit buses with compression ignition engines using Air Products fuel grade methanol. A comparison will be made from the results of the two fuels, current in use methanol and Air Products fuel grade methanol. This project centers around an intensive experimental effort presently taking place in New York City.

Prior to departing to perform the emissions research in New York City, the test equipment was subjected to a quality audit by performing, propane, alcohol and aldehyde injections to determine the respective recovery percentages from the dilution tunnel. Appropriate sampling flow-rates were established to perform the testing.

A research team transported one of the two West Virginia University's Heavy Duty Vehicle Emissions Transportable Laboratories to the test site at Command Bus Corporation Depot, in New York City. Three methanol powered buses were tested: details are given in Table 1. The buses were subjected to testing using both in-use methanol and the Air Products' fuel grade methanol. The fuel change was accomplished by connecting into the bus's current fuel system. Two fuel lines were routed from the Air Products' fuel container to the bus's fuel pump and fuel return. At least three gallons of the Air Products' fuel grade methanol was pumped through the fuel system before for the return line was placed into the fuel grade container. The test weight (tw) for the vehicles was calculated to be 34,500 lbs. using the following equation;

$$tw = (\text{curbweight} + 150 + (\text{seating} + \text{standing})/2 * 150)$$

All three buses were tested using the Central Business District cycle (CBD) and one was also tested using the WVU 5 Mile Route. Figure 1 shows the CBD cycle while figure 2 shows the continuous oxides of nitrogen (NO_x) emissions from bus # 2145 fueled with the Air Products methanol. At this time the laboratory has not yet left New York City and the data processing is not complete. A full report on data is expected within six weeks. Future plans will therefore be centered around data processing and report writing.

Bus Details

Table 1

Bus Number	Engine Type	Test Weight	Cycle
2145	6V92 DDC II	34,408	CBD
2139	6V92 DDC II	34,408	CBD
2143	6V92 DDC II	34,408	CBD 5 Mile Route

Central Business District Cycle, Bus 2145

Figure 1:

Continuous Oxides of Nitrogen CBD Bus 2145

Figure 2:

**Quarterly Report
(Nov. 25, 1997 – Mar. 11, 1998)**

to

**Mr. Robert Senn
consultant to**

Air Products and Chemicals, Inc.

from

**Dr. John J. Thomas
Florida Tech
150 W. University Blvd.
Melbourne, Florida 32901
Ph 407-674-7252
Fax 407-984-8461**

Fleet Trails for Fuel Grade Methanol

under

**USDOE Cooperative Agreement No. DE-FC22-92PC90543
Performance Period 8/11/97 – 10/10/98**

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1. Introduction

During this period the methanol car was re-commissioned and the operator, Dr. John Thomas, drove the car on a routine basis between 3325 W. New Haven Ave. and the Florida Tech main campus at 150 W. University Blvd.(please see Appendix). He also drove the car between his house and the above addresses. Before and after these trips he recorded all the gauge readings, the date, the time, and comments. This allowed several MPG calculations. The gear shift did not work properly from the beginning and eventually stopped functioning at all. The problem turned out to be caused by some student modification of a key bearing made some nine years ago. The problem was easily repaired. When the gear shift was operating at its poorest, the mileage dropped dramatically. The electronic temperature gauge was replaced with a mechanical one which now allows us to record the exact temperature in degrees F. The car is now operating ideally. In the meantime we intend to proceed with collection of emission data.

The bus required more extensive work but it too is now operational. Apparently the old Avocet we obtained as ignition enhancer decomposed over the years since the mechanics could not determine any performance difference between methanol containing Avocet and methanol without Avocet. As of now we are relying on glow plug warming to produce ignition. Up to now the system appears to work well. It is now operating on methanol that was put in its tanks five years ago. The vehicle is running as well as can be expected according to our consultants. In the meantime, we intend to proceed with collection of emission data and miscellaneous upgrades

2. Activities (As Recorded by the Staff)

1. To use cold start:
1st - Try to start car, if car does not start then -
2nd - Stop cranking engine but leave the key on, and press injector switch in until it clicks. The switch is located on the underside of the dash board near the radio.
3rd - Try to start engine by normal starting procedures
4th - If the engine does not start, repeat steps 2 and 3
2. Replaced the shift cable on the car because of very hard shifting after warming up. However, shifting was still unacceptable.
3. The clutch pedal only disengages in the last inch or so of travel resulting in hard shifting.
4. The car was taken to Jim Rathmann's for repair.
5. The bus was started with the help of starting fluid. It idled all right but did not rev up.
6. The bus is missing very badly when running or idling. Dirt in the gas is suspected or the fuel is too old. Injectors that were just put in might be already clogged due to sediments in the lines. Further testing was needed.
7. The bus started again on starting fluid after the governor and other controls were readjusted and freed up. The oil was also changed. Controls worked erratically at first; after familiarization they worked well.
8. The bus eventually ran smoothly. It was necessary to replace two clogged injectors. It takes about 20 - 25 minutes to warm up and run smoothly. It will not let you rev the engine until a certain temperature is reached. It blows white smoke until warmed up and all condensation from system has been burnt off.
9. The bus was driven on I-95 to test. It ran well but is not really designed for highway due to low gear ratio. Max speed was about 55 mph.
10. The car transmission was inspected due to suspicion of bad clutch. Clutch was fine, however the clutch engaging arm and bearings were bad resulting in the clutch not fully engaging. Problem was fixed and the car is now running and working well as a result. More details below.
11. An air leak in bus was fixed, all panels were put back on and tightened down. Bus graffiti was removed.

12. Several cleaning materials were tried on the seats, however most either had no result or began eating or rubbing off covers.
13. Throwout bearings in the car were replaced in transmission along with clutch fork arm and the bushings. The play that resulted from worn bearings caused the clutch not to fully disengage and resulted in hard shifting. Alternative Automotive in West Melbourne did the work on the transmission.
14. Modification to both vehicles for on-board emission samplings were discussed and designed.
15. Car air-conditioner was inspected for leaks.
16. The ether start system was installed in the bus. It is identical to the one in the car and was detailed in the last quarterly report. After installation, the time for the bus to properly warm up only took a few minutes.

3. Results and Discussion

The methanol powered car was operated routinely and the total miles traveled after re-commissioning is 1176 over approximately 3 months. The mileage varied from a high of 12.8 mpg to a low of 5.5 mpg. The low mileage data was obtained when the shifting problem was at its worse. A mechanical temperature gauge was installed which now allows us to determine if any overheating takes place. The old electronic temp gauge never did work well. All the gauges (oil, fuel, and battery) now work well and show nothing except normal readings. The cold-starting system continues to work very well. At this time the car is undergoing repairs because of a defect in the air conditioning system. The operational log is shown in the Appendix.

We are currently installing systems to allow us to determine on-board emissions.

The bus is now operating satisfactorily but we can not operate it routinely because the fuel shipment has not arrived. It did, however, make a 50 mile trip on I-95. An ethyl ether starting system was installed on the bus and it works very well. This system allows operation after less than five minutes of warm-up. This is comparable to the performance of a regular diesel engine. The ether starting system is exactly the same as on the car.

As it turned out, only lack of familiarization caused any problems putting the bus into operation. The only real problem was two clogged injections which were replaced.

4. Work Schedule for Thlrđ Quarter

1. Complete installation of on-board sampling and emissions data collecting systems.
2. Complete air-conditioning repair of car and return it to routine operation.
3. Complete installation of on-board sampling and emission data collecting systems for the bus.
4. Compile the emissions data at 0, 10, 20, 30, 40, and 50 miles/hour for the bus.
5. Compile the emissions data at 0,10, 20, 30, 40, 50, and 60 miles/hour for the car.

6. Return the car to routine operation and collect operating data.
7. Re-commission the bus and use it for biology field trips as other university activities while collecting operating data.

The work scheduled for the second quarter was completed on schedule. The only variance was caused by a decision to determine emissions using on-board systems instead of installing a chassis dynamometer.

Listing of Project Personnel

1. Dr. John Thomas, Principal Investigator
2. Frank Aransky, Research Assistant
3. Greg Palubin, Research Assistant
4. Richard MacKenzie, Director, Florida Tech Vehicle Maintenance
5. Bert Austin
6. David Cash
7. Jeff Reilly

5. Appendix

Road Data for the Methanol Car

METHANOL CAR OPERATIONS DATA

Operator	Date	Time	Mileage 1 Mileage 2	Temp (°F)	Oil (psi)	Fuel	Battery (V)	Start (S) Finish(F)	Place	Comments
JT	1/6/98	11:30AM	6946.5 89.5	1/4	40	1/2+	13	S	Blowest	Service Engine Soon Light On
JT	1/6/98	11:49	6954.3	1/4	20	1/2+	12	F	Rooney's	Started Right Up, Oil Gauge Jumps Around
JT	1/6/98	12:40PM	97.4							Service Engine Soon Light On
JT	1/6/98	1:00	6964.2	1/8	50	1/2+	14	S	Rooney's	Service Engine Soon Light On
JT	1/6/98		107.3	3/8	32	1/2-	13	F	Blowest	Service Engine Soon Light On
JT	1/6/98	1:30	"	1/4	39	1/2-	13	S	Blowest	Service Engine Soon Light Not On
JT	1/6/98	1:39	6969.1	3/8	30	1/2-	13	F	FIT	Service Engine Soon Light On
JT	1/6/98	2:40	112.3							
JT	1/6/98	2:56	6974.0	1/8	45	1/2-	13	S	FIT	Service Engine Soon Light On
JT	1/6/98	3:12	117.1	3/8	30	3/8	13	F	Blowest	Service Engine Soon Light On
JT	1/6/98	3:32	6978.6	1/4	39	1/4	13	S	Blowest	Service Engine Soon Light On
JT	1/6/98		121.7	3/8	30	1/4	14	F	FIT	Service Engine Soon Light On
JT	1/7/98	11:16AM	"	0	60	3/8	15	S	FIT	Added 6 Gal of Methanol Prior to Travel
JT	1/7/98	11:35	6983.3	3/8	30	5/8	14	F	Blowest	Service Engine Soon Light Not On
JT	1/7/98	1:03PM	126.4							Add 2 Gal of Methanol
JT	1/7/98	1:20	6991.3	1/8	50	7/8	14	S	Blowest	Service Engine Soon Light Not On
JT	1/7/98		134.4	1/4	30	3/4+	13	F	Rooney's	Service Engine Soon Light Not On
JT	1/7/98	1:58	"							
JT	1/7/98	2:10	6995.3	1/8	42	3/4+	14	S	Rooney's	Service Engine Soon Light Not On
JT	1/9/98	9:55AM	138.4	1/4	30	3/4+	13	F	FIT	Service Engine Soon Light Not On
JT	1/9/98	10:10	6995.6	1/16	65	5/8	15	S	FIT	Service Engine Soon Light Not On
JT	1/9/98		138.7							Used Starter Button
JT	1/9/98	10:10	7000.2	1/16	39	5/8	15	F	Blowest	Service Engine Soon Light Not On
JT	1/9/98		143.3							
JT	1/9/98	11:28	"	3/4	55	5/8	16	S	Blowest	HOT Gauge
JT	1/9/98	11:47	7008.3	Out	32	5/8	13	F	Rooney's	Temperature Gauge Out
JT	1/9/98		151.4							
JT	1/9/98	12:40PM	"	Out	50	5/8	14	S	Rooney's	Temperature Gauge Out

METHANOL CAR OPERATIONS DATA

Operator	Date	Time	Mileage 1 Mileage 2	Temp (°F)	Oil (psi)	Fuel	Battery (V)	Start (S) Finish(F)	Place	Comments
JT	1/9/98	1:03AM	7018.1 161.3	Out	30	5/8	14	F	Blowest	Temperature Gauge Out
JT	1/9/98	1:47	7018.2 161.3	Out	45	1/2+	14	S	Blowest	Temperature Gauge Out
JT	1/9/98	2:48	7022.8 165.9	Out	45	1/2+	14	S	FIT	Temperature Gauge Out
JT	1/9/98	3:05	7027.5 170.6	Out	30	1/2+	13	F	Blowest	Temperature Gauge Out
JT	1/9/98	4:37	"	Out	60	1/2+	15	S	Blowest	Temperature Gauge Out
JT	1/9/98	5:53	7032.1 175.2	Out	30	1/2+	13	F	FIT	Temperature Gauge Out
JT	1/9/98	8:22	"	Out	60	1/2+	15	S	FIT	Temperature Gauge Out
JT	1/9/98	8:50	7044.9 188	Out	38	1/2-	15	F	419B	Temperature Gauge Out
JT	1/10/98	1:44PM	"	Out	60	1/2-	15	S	419B	Temperature Gauge Out
JT	1/10/98	1:56	7050.1 193.2	Out	40	1/2-	13	F	Walmart	Temperature Gauge Out
JT	1/10/98	2:42	"	Out	60	1/2-	15	S	Walmart	Temperature Gauge Out
JT	1/10/98	3:08	7062.0 205.1	Out	30	1/4	14	F	419B	Temperature Gauge Out
JT	1/17/98	4:00	"	"	"	"	"	"	419B	Added 3.5 Gal of Methanol
JT	1/18/98	12:13PM	7062.0 205.1	Out	63	5/8	14	S	419B	Very Difficult Starting, Pressed Starter 4x
JT	1/18/98	12:53	7082.8 225.9	Out	30	1/2	13	F	419B	Service Engine Soon Light On
JT	1/19/98	8:38AM	"	Out	65	1/2-	15	S	419B	Very Difficult Starting, Presses Starter 6x
JT	1/19/98	9:08	7095.2 233.3	Out	30	3/8	14	F	Blowest	Service Engine Soon Light On
JT	1/19/98	10:00	"	Out	"	"	"	"	"	Added 3.0 Gal of Methanol
JT	1/19/98	10:10	"	Out	60	1/2+	14	S	Blowest	Easy Start, Service Eng. Soon Light Not On
JT	1/19/98	10:25	7100.0 243.1	Out	35	1/2+	14	F	FIT	Service Engine Soon Light Not On

METHANOL CAR OPERATIONS DATA

Operator	Date	Time	Mileage 1 Mileage 2	Temp (°F)	Oil (psi)	Fuel	Battery (V)	Start (S) Finish(F)	Place	Comments
JT	1/19/98	11:38	"	Out	60	1/2+	14	S	FIT	Service Engine Soon Light Not On
JT	1/19/98	11:50	7103.8	Out	38	1/2+	14	F	Rooney's	Service Engine Soon Light Not On
JT	1/19/98	12:35PM	245.9							
JT	1/19/98	12:35PM	7103.8	Out	55	1/2+	15	S	Rooney's	Service Engine Soon Light Not On
JT	1/19/98	1:12	240.9							Instant Start
JT	1/19/98	1:12	7118.2	Out	30	1/2+	14	F	*JT Via Biowe	Service Engine Soon Light Not On
JT	1/19/98		261.5							
JT	1/19/98	4:00	"	Out	60	1/2+	15	S	FIT	Starter Fixed at 3:00PM
JT	1/19/98	4:15	7123.1	Out	30	1/2-	14	F	Biowest	Service Engine Soon Light Not On
JT	1/19/98		265.2							
JT	1/19/98	4:55	"	Out	55	1/2-	14	S	Biowest	Service Engine Soon Light Not On
JT	1/19/98	5:24	7136.7	Out	35	1/4	14	F	419B	Service Engine Soon Light Not On
JT	1/19/98		279.8							
JT	1/20/98	11:23AM	"	Out	60	3/4	15	S	419B	Very Difficult Starting, Presses Starter 2x
JT	1/20/98	11:38	7143.1	Out	45	5/8	14	F/S	1082B/STT	Added 5.25 Gal Methanol
JT	1/20/98	12:05PM	7154.6	Out	40	5/8	14	F/S	Rooney's	
JT	1/20/98		297.9							
JT	1/20/98	1:20	"	Out	60	5/8	14	S	Rooney's	
JT	1/20/98	2:05	7172.7	Out	35	1/2+	13	F	419B	
JT	1/20/98		315.8							
JT	1/21/98	2:44PM	"	Out	70	1/2+	15	S	419B	Not Bad Starting
JT	1/21/98	2:44	7188.4	Out	35	1/2+	14	F	Biowest	
JT	1/21/98		331.5							
JT	1/21/98	3:00								
JT	1/21/98	3:32	"	Out	60	F+	14	S	Biowest	Filled Fuel Tank ~8.2 Gal
JT	1/21/98	3:47	7192.7	Out	40	F+	14	F	FIT	Service Engine Soon Light Not On
JT	1/21/98		335.8							
JT	1/21/98	4:29	"	Out	55	F+	14	S	FIT	
JT	1/21/98	4:47	7197.5	Out	35	F+	14	F	Biowest	
JT	1/21/98		340.6							
JT	1/21/98	5:03	"	Out	40	F+	14	S	Biowest	

METHANOL CAR OPERATIONS DATA

Operator	Date	Time	Mileage 1 Mileage 2	Temp (°F)	Oil (psi)	Fuel	Battery (V)	Start (S) Finish(F)	Place	Comments
JT	1/21/98	5:37	7210	Out	30	F+	13	F	419B	
			353.1							
JT	1/22/98	8:20AM	"	Out	70	F-	15	S	419B	Started Fairly Easy, No Button
JT	1/22/98	8:50	7222	Out	35	F-	13	F	FIT	
			365.1							
JT	1/22/98	11:25	"	Out	60	F-	14	S	FIT	
JT	1/22/98	11:38	7225.8	Out	40	F-	14	F	Rooney's	
			368.9							
JT	1/22/98	12:33PM	"	Out	60	F-	14	S	Rooney's	Easy Start
JT	1/22/98	12:53	7235.7	Out	35	F-	14	F	Biowest	
			378.8							
JT	1/22/98	1:17	"	Out	45	3/4+	13	S	Biowest	
JT	1/22/98	1:32	7240	Out	38	3/4+	13	F	FIT	
			383.1							
JT	1/22/98	2:49	"	Out	60	3/4+	14	S	FIT	
JT	1/22/98	3:05	7244.8	Out	38	3/4	13	F	Biowest	
			387.9							
JT	1/22/98	4:43	"	Out	60	3/4	14	S	Biowest	
JT	1/22/98	5:13	7258.4	Out	35	3/4-	13	F	419B	
			401.5							
JT	1/23/98	8:52AM	"	Out	60	3/4-	15	S	419B	Difficult Start - Rain
JT	1/23/98	9:20	7270.8	Out	35	1/2+	13	F	Biowest	
			413.9							
JT	1/23/98	9:33	"	Out	42	1/2+	14	S	Biowest	
JT	1/23/98	9:45	7275.1	Out	32	1/2+	13	F	FIT	
			418.2							
JT	1/23/98	10:18	"	Out	50	1/2+	14	S	FIT	
JT	1/23/98	10:54	7299	Out	35	1/2-	13	F	Rockledge	1 - 95 in the rain (192)
			442.1							
JT	1/23/98	11:18	"	Out	45	1/2-	13	S	Rockledge	
JT	1/23/98	11:49	7315.6	Out	35	1/4	13	F	419B	1 - 95 to Wickham
			458.7							
JT	1/26/98	8:37AM	"	Out	70	1/4	15	S	419B	Very Difficult

METHANOL CAR OPERATIONS DATA

Operator	Date	Time	Mileage 1 Mileage 2	Temp (°F)	Oil (psi)	Fuel	Battery (V)	Start (S) Finish (F)	Place	Comments
JT	1/26/98	9:05AM	7329.2	Out	35	E	14	F	Blowest	Made it! Total miles since 1/16/98 - 382.8 miles 12 driving days
			472.3							
JT	1/26/98	1:00PM								
JT	1/26/98	7:25	7329.8	Out	60	F+	15	S	Blowest	Filled with 11.0 Gal MeOH => 12.8 mpg
			472.3							
JT	1/26/98	7:52	7342.8	Out	35	F+	14	F	419B	
			483.9							
JT	1/27/98	8:47AM	"	Out	70	F+	15	S	"	Pressed starter button 4x
JT	1/27/98	9:13	7354.6	Out	35	F-	14	F	FIT	
			497.7							
JT	1/27/98	11:28	"	Out	60	F-	14	S	FIT	Started immediately
JT	1/27/98	11:42	7358.6	Out	40	F-	14	F	Rooney's	
			501.7							
JT	1/27/98	12:40PM	"	Out	60	F-	14	S	Rooney's	
JT	1/27/98	12:58	7368.4	Out	35	F-	14	F	Blowest	
			511.5							
JT	1/27/98	1:10	"	Out	40	F-	14	S	Blowest	
JT	1/27/98	1:37	7377.4	Out	35	F-	13	F	Blowest	Had to return to Biowest to Steno pad
			520.5							
JT	1/27/98	1:43	"	Out	38	3/4+	13	S	Blowest	
JT	1/27/98	1:55	7381.7	Out	30	3/4	13	F	FIT	
			524.8							
JT	1/27/98	2:53	"	Out	60	3/4+	14	S	FIT	Started immediately
JT	1/27/98	3:10	7386.5	Out	35	3/4	13	F	Blowest	
			529.6							
JT	1/27/98	3:28	"	Out	45	3/4-	14	S	Blowest	
JT	1/27/98	3:39	7390	Out	35	3/4	14	F	ARL	
			533.1							
JT	1/27/98	4:26	"	Out	60	3/4	15	S	ARL	
JT	1/27/98	4:37	7393.5	Out	40	3/4-	14	F	Blowest	
			536.6							
JT	1/27/98	4:50	"	Out	45	3/4-	14	S	Blowest	

METHANOL CAR OPERATIONS DATA

Operator	Date	Time	Mileage 1 Mileage 2	Temp (°F)	Oil (psi)	Fuel	Battery (V)	Start (S) Finish(F)	Place	Comments
JT	1/27/98	5:12PM	7401.5	Out	30	5/8	13	F	Rooney's	
			544.6							
JT	1/27/98	5:50	"	Out	60	5/8	14	S	Rooney's	
JT	1/27/98	6:00	7405.5	Out	35	5/8	13	F	FIT	
			548.6							
JT	1/27/98	8:03	"	Out	60	3/8	14	S	FIT	
JT	1/27/98	8:28	7417.2	Out	35	1/2+	14	F	419B	Overhead light out
			560.4							
JT	1/28/98	8:18AM	"	Out	70	1/2+	15	S	419B	Presses starter button 6x, Cold (55°F)
JT	1/28/98	8:48	7429.6	Out	38	1/2-	14	F	Biowest	
			572.7							
JT	1/28/98	9:03	"	Out	42	1/2-	14	S	Biowest	
								F	FIT	
JT	1/28/98	9:55	7434	Out	60	1/2-	15	S	FIT	
			577.1							
JT	1/28/98	10:03	7435.7	Out	40	1/2-	14	F	ARL	
			578.8							
JT	1/28/98	10:08	"	Out	45	1/2-	14	S	ARL	
JT	1/28/98	10:18	7439.3	Out	38	1/2-	14	F	Biowest	
			582.4							
JT	1/28/98	11:38	"	Out	60	1/2-	14	S	Biowest	
JT	1/28/98	11:56	7447.3	Out	30	1/4	14	F	Rooney's	
			590.4							
JT	1/28/98	12:42PM	"	Out	60	1/4	14	S	Rooney's	
JT	1/28/98	1:03	7457.2	Out	38	Red Mark	14	F	Biowest	
			600.3							
JT	1/28/98	1:40	"							Filled with 11 Gal Methanol => 11.64 mpg
JT	1/28/98	2:00	"	Out	60	F+	14	S	Biowest	
JT	1/28/98	2:15	7461.6	Out	35	F-	13	F	FIT	
			604.7							
JT	1/28/98	3:20	"	Out	60	F+	14	S	FIT	
JT	1/28/98	3:40	7466.6	Out	38	F+	14	S	Biowest	
			609.7							

METHANOL CAR OPERATIONS DATA

Operator	Date	Time	Mileage 1 Mileage 2	Temp (°F)	Oil (psi)	Fuel	Battery (V)	Start (S) Finish(F)	Place	Comments
JT	1/28/98	5:08PM	7466.6 609.7	Out	60	F+	14	S	Biowest	
JT	1/28/98	5:45	7480.2 623.3	Out	35	F-	14	F	419B	
JT	1/29/98	8:09AM	" 7492.6	Out	70	F-	15	S	419B	Very difficult start (46°F). More than 10 attempt
JT	1/29/98	9:08	" 635.7	Out	60	F-	14	S	Biowest	
JT	1/29/98	9:20	7496.8 639.9	Out	40	3/4+	13	F	FIT	
JT	1/29/98	10:02	"	Out	60	3/4+	14	S	FIT	
JT	1/29/98	10:20	7502 645.1	Out	35	3/4+	14	F	Biowest	Very difficult shifting in low gears
JT	1/29/98	11:55	"	Out	60	3/4+	14	S	Biowest	
JT	1/29/98	12:15PM	7510 653.1	Out	35	3/4+	13	F	Rooney's	Very difficult shifting in low gears
JT	1/29/98	1:03	"	Out	50	3/4+	14	S	Rooney's	
JT	1/29/98	1:22	7519.8 662.9	Out	35	3/4-	13	F	Biowest	
JT	1/29/98	1:53	"	Out	45	3/4-	14	S	"	
JT	1/29/98	2:12	7525.8 668.9	Out	35	3/4-	13	F	FIT	
JT	1/29/98	2:50	"	Out	50	5/8	13	S	"	Tough Shifting
JT	1/29/98	3:08	7530.3 673.4	Out	35	5/8	13	F	Biowest	
JT	1/29/98	5:26	"	Out	60	5/8	14	S	"	Easy start
JT	1/29/98	5:42	7534.4 677.5	Out	45	1/2+	13	F	FIT	
JT	1/29/98	8:00	"	Out	60	1/2+	14	S	FIT	Smell of burning
JT	1/29/98	8:25	7546.3 689.4	Out	35	1/2+	14	F	419B	
JT	2/2/98	10:35AM	"	Out	60	1/2+	15	S	419B	Easy start. Clutch no problem

METHANOL CAR OPERATIONS DATA

Operator	Date	Time	Mileage 1 Mileage 2	Temp (°F)	Oil (psi)	Fuel	Battery (V)	Start (S) Finish(F)	Place	Comments
JT	2/2/98	11:00AM	7558.5	Out	35	1/2-	14	F	FIT	
			701.6							
JT	2/2/98	11:32	"	Out	50	1/2-	14	S	FIT	Easy start, Clutch no problem
JT	2/2/98	11:42	7562.4	Out	35	1/2-	13	F	Rooney's	Easy start, Clutch no problem
			705.3							
JT	2/2/98	12:28PM	"	Out	55	1/2-	14	S	"	Easy start, Clutch no problem
JT	2/2/98	12:47PM	7572.3	Out	35	3/8	13	F	Blowest	
			715.4							
JT	2/2/98	1:00								Filled with 10.2 Gal Methanol => 11.27 mpg
JT	2/2/98	2:00	"	Out	60	F+	14	S	Blowest	
JT	2/2/98	2:17	7576.5	Out	35	F+	13	F	FIT	
			719.6							
JT	2/2/98	3:29	"	Out	60	F+	14	S	FIT	Very tough shifting
JT	2/2/98	3:50	7581.4	Out	35	F+	13	F	Blowest	Shifting not too bad
			724.5							
JT	2/3/98	2:28PM	"	Out	60	F+	14	S	Blowest	
JT	2/3/98	2:48	7586.1	Out	40	F+	14	F	FIT	Car shipped out for repairs
			729.2							
JT	3/2/98	10:25AM	7602.2	Out	70	3/4	15	S	FIT	Starting a little difficult, Clock not right
			745.3							
JT	3/2/98	10:42	7606.8	Out	40	3/4-	13	F	Blowest	
			749.9							
JT	3/2/98	11:35	"	Out	60	3/4-	13	S	Blowest	Instant start
JT	3/2/98	11:49	7611.1	Out	38	1/2+	13	F	FIT	
			Can't Read							
JT	3/2/98	1:05PM	7611.1	Out	60	1/2+	14	S	FIT	
			754.2							
JT	3/2/98	1:23	7615.9	Out	42	1/2+	13	F	Blowest	Filled with 7.0 Gal Methanol
			759							
JT	3/2/98	2:28	"	Out	55	F+	13	S	Blowest	
JT	3/2/98	2:44	7620.2	Out	55	F+	13	F	FIT	
			763.3							
JT	3/2/98	4:13	"	Out	60	F+	14	S	FIT	

METHANOL CAR OPERATIONS DATA

Operator	Date	Time	Mileage 1 Mileage 2	Temp (°F)	Oil (psi)	Fuel	Battery (V)	Start (S) Finish (F)	Place	Comments
JT	3/2/98	4:31	7625 768.1	Out	35	F+	13	F	Biowest	
Filled with 7 Gal of Methanol => 5.54 mpg										
JT	3/2/98	5:13	7625	Out	60	F+	14	S	Biowest	
JT	3/2/98	5:30	7529.5	Out	35	F+	13	F	FIT	
			772.6							
JT	3/3/98	"	"	Out		F		S	FIT	Very hard starting. too much Ether
JT	3/3/98	10:28AM	7634.2	Out	40	F-	13	F	Biowest	
			777.3							
JT	3/3/98	11:18	"	Out	60	F-	14	S	Biowest	Instant start
JT	3/3/98	11:43	7647.8	Out	35	F-	13	F	419B	By Wickham Road
			790.9							
JT	3/3/98	12:50PM	"	Out	60	F-	14	S	419B	Instant start
JT	3/3/98	1:33	7664.4	Out	35	3/4	13	F	Biowest	Via Riverside and FIT
			807.5	Out						
JT	3/3/98	1:55	"	Out	45	3/4	13	S	Biowest	
JT	3/3/98		7668.7	Out		3/4+		F	FIT	
			811.8							
JT	3/3/98	3:10	"	Out	50	3/4+	13	S	FIT	
JT	3/3/98	3:25	7673.6	Out	40	3/4-	13	F	Biowest	
			816.7							
JT	3/3/98	3:33	"	Out	40	3/4-	13	S	Biowest	
JT	3/3/98	4:08	7687.2	Out	35	1/2+	13	F	419B	
			830.3							
JT	3/3/98	6:05	"	Out	60	1/2+	15	S	419B	
JT	3/3/98	6:29	7699.3	Out	39	1/2	14	F	FIT	
			842.4							
JT	3/3/98	8:14	"	Out	60	1/2+	14	S	FIT	Instant start
JT	3/3/98	8:40	7711.3	Out	35	1/2	14	F	419B	
			854.4							
JT	3/4/98	8:19AM	"	Out	70	1/2-	16	S	419B	Tough starting, Pressed starter 3x
JT	3/4/98	8:53	7723.7	Out	35	1/4	14	F	Biowest	
			866.8							

METHANOL CAR OPERATIONS DATA

Operator	Date	Time	Mileage 1 Mileage 2	Temp (°F)	Oil (psi)	Fuel	Battery (V)	Start (S) Finish (F)	Place	Comments
JT	3/4/98	9:00AM	7723.7	Out	40	1/4	14	S	Biowest	
			866.8							
JT	3/4/98	9:12	7728	Out	40	1/4	14	F	FIT	
			871.1							
JT	3/4/98	10:15	"	Out	60	1/4	15	S	"	
JT	3/4/98	10:30	7732.6	Out	35	Red Mark	13	F	Biowest	Performed well at low fuel
			873.9							Filled with 11.6 Gal of Methanol => 9.28 mpg
JT	3/4/98	1:23PM	"	Out	60	F+	14	S	Biowest	
JT	3/4/98	1:37	7737.3	Out	40	F+	13	F	FIT	
			880.4							
JT	3/4/98	2:43	"	Out	60	F+	14	S	FIT	
JT	3/4/98	2:58	7742.1	Out	40	F+	13	F	Biowest	
			885.2							
JT	3/4/98	3:53	"	Out	60	F+	14	S	Biowest	
JT	3/4/98	4:08	7746.4	Out	35	F+	13	F	FIT	
			889.5							
JT	3/4/98	4:20	"	Out	45	F+	13	S	FIT	
JT	3/4/98	4:38	7751.2	Out	35	F	13	F	Biowest	
			894.4							
JT	3/4/98	4:51	"	Out	40	F-	14	S	Biowest	
JT	3/4/98	5:13	7759.3	Out	35	F-	13	F	Rooney's	
			902.4							
JT	3/4/98	5:48	"	Out	42	F-	13	S	Rooney's	
JT	3/4/98	6:16	7773.7	Out	35	F-	13	F	419B	
			916.8							
JT	3/5/98	12:33PM	"	Out	70	F-	15	S	419B	Fairly easy starting
JT	3/5/98	1:13	7790.2	Out	30	3/4	13	F	Biowest	Via FIT and South Patrick
			933.3							
JT	3/5/98	1:25	"	Out	40	3/4	13	S	Biowest	
JT	3/5/98	1:40	7794.7	Out	40	3/4-	13	F	FIT	
			937.8							
JT	3/5/98	3:43	"	Out	65	3/4-	14	S	FIT	

METHANOL CAR OPERATIONS DATA

Operator	Date	Time	Mileage 1 Mileage 2	Temp (°F)	Oil (psi)	Fuel	Battery (V)	Start (S) Finish(F)	Place	Comments
JT	3/5/98	4:03AM	7799.4 942.3	Out	35	3/4-	13	F	Blowest	At idle: 1000 RPMs
JT	3/5/98	4:20	"	Out	45	3/4-	13	S	Blowest	
JT	3/5/98	4:40	7807.5	Out	40	1/2+	13	F	Rooney's	
			950.6							
JT	3/5/98	5:17	"	Out	60	1/2+	14	S	Rooney's	
JT	3/5/98	5:28	7811.5	Out	40	1/2+	13	F	FIT	
			954.6							
JT	3/5/98	8:05	"	Out	60	1/2+	14	S	FIT	
JT	3/5/98	8:16	7815.4	Out	45	1/2+	13	F	Rooney's	
			958.5							
JT	3/5/98	8:52	"	Out	60	1/2+	14	S	Rooney's	
JT	3/5/98	9:20	7829.1	Out	35	1/2	13	F	419B	
			973.2							
JT	3/18/98	3:33PM	7937	165	60	F+	14	S	Blowest	New temperature gauge installed
			80.1							Filled with 11.05 Gal Methanol
JT	3/18/98	3:51	7944.7	190	40	F+	13	F	FIT	
			84.8							
JT	3/18/98	4:18	"	120	55	F+	13	S	FIT	
JT	3/18/98	4:33	7946.3	198	40	F+	13	F	Blowest	
			89.4							
JT	3/18/98	9:05	"	100	60	F+	13	S	Blowest	
JT	3/18/98	9:29	7959.8	190	35	F-	12	F	419B	Via Wickham Rd.
			102.9							
JT	3/19/98	7:34AM	"	10	60	F-	15	S	419B	
JT	3/19/98	8:09	7972.2	195	35	F-	13	F	Blowest	Via South Patrick
			115.3							
JT	3/19/98	8:21	"	190	45	3/4+	13	S	Blowest	
JT	3/19/98	8:35	7976.9	190	35	3/4	13	F	FIT	
			120							
JT	3/19/98	10:40	"	125	60	3/4+	14	S	FIT	
JT	3/19/98	10:53	7981.6	192	45	3/4	13	F	Blowest	
			124.7							

METHANOL CAR OPERATIONS DATA

Operator	Date	Time	Mileage 1 Mileage 2	Temp (°F)	Oil (psi)	Fuel	Battery (V)	Start (S) Finish (F)	Place	Comments
JT	3/19/98	11:35AM	7981.6 124.7	160	60	3/4	14	S	FIT	
JT	3/19/98	11:54	7989.6 132.7	203	35	3/4-	13	F	Rooney's	
JT	3/19/98	1:15PM	"	150	60	3/4	14	S	Rooney's	
JT	3/19/98	1:35	7999.5 142.6	195	35	3/4-	13	F	Blowest	
JT	3/19/98	2:03	"	172	50	3/4-	14	S	Blowest	
JT	3/19/98	2:17	8003.8 146.9	200	35	1/2+	13	F	FIT	
JT	3/19/98	3:13	"	165	60	1/2+	13	S	FIT	
JT	3/19/98	3:33	8008.8 151.9	200	35	1/2+	13	F	Blowest	
JT	3/19/98	4:24	"	160	60	1/2+	14	S	Blowest	
JT	3/19/98	4:28	8009.2 152.3	185	50	1/2+	13	F	Rom Bakery	
JT	3/19/98	4:58	"	160	60	1/2+	13	S	Rom Bakery	
JT	3/19/98	5:10	8013.1 156.2	190	40	1/2	13	F	FIT	
JT	3/19/98	7:42	"	110	60	1/2+	14	S	FIT	
JT	3/19/98	8:07	8025.7 168.8	195	35	1/2	13	F	419B	
JT	3/23/98	8:18AM	"	100	70	1/2-	15	S	419B	Turned key, hit button, instant start
JT	3/23/98	8:47	8032.2 181.3	193	30	1/4	13	F	Blowest	
JT	3/23/98	8:53	"	193	40	1/4+	14	S	Blowest	
JT	3/23/98	9:08	8042.1 185.9	198	38	1/4	13	F	FIT	
JT	3/23/98	10:03	"	160	60	1/4	14	S	FIT	
JT	3/23/98	10:16	8047.5 190.6	190	40	Red Mark	13	F	Blowest	Filled with 11.6 Gal Methanol => 11.48 mpg
JT	3/23/98	3:08PM	"	100	60	F+	15	S	Blowest	

METHANOL CAR OPERATIONS DATA

Operator	Date	Time	Mileage 1 Mileage 2	Temp (°F)	Oil (psi)	Fuel	Battery (V)	Start (S) Finish(F)	Place	Comments
JT	3/23/98	3:20PM	8051.3	190	45	F+	14	F	FIT	
			194.8							
JT	3/23/98	4:28	"	150	60	F+	14	S	FIT	
JT	3/23/98	4:45	8056.6	195	35	F+	13	F	Blowest	
			199.7							
JT	3/23/98	5:08	8056.7	176	60	F+	13	S	Blowest	
			199.7							
JT	3/23/98	5:40	8070.2	198	35	F-	13	F	419B	
			213.3							
JT	3/24/98	7:29AM	"	100	60	F-	14	S	419B	Instant Start
JT	3/24/98	8:25	8082.6	203	35	F-	13	F	Blowest	
			225.7							
JT	3/24/98	9:38	"	150	60	3/4+	14	S	Blowest	
JT	3/24/98	9:55	8087	203	40	3/4+	13	F	FIT	
			230.1							
JT	3/24/98	12:33PM	8091.5	170	60	3/4+	13	S	Rooney's	
			234.6							
JT	3/24/98	12:49	8101.5	195	35	3/4	13	F	Blowest	
			244.6							
JT	3/24/98	1:30	"	170	50	3/4	13	S	Blowest	
JT	3/24/98	1:44	8105.7	203	35	3/4-	13	F	FIT	
			248.8							
JT	3/24/98	2:40	"	165	60	3/4-	14	S	FIT	
JT	3/24/98	2:56	8110.4	203	35	1/2+	13	F	Blowest	
			256.7							
JT	3/24/98	4:35	"	150	60	1/2+	14	S	Blowest	
JT	3/24/98	4:58	8118.7	203	35	1/2+	13	F	Rooney's	
JT	3/24/98	5:27	"	180	50	1/2+	13	S	Rooney's	
			261.7							
JT	3/24/98	5:40	8122.7	195	35	1/2+	13	F	FIT	
			265.7							
JT	3/24/98	7:57	"	150	60	1/2+	14	S	FIT	

METHANOL CAR OPERATIONS DATA

Operator	Date	Time	Mileage 1 Mileage 2	Temp (°F)	Oil (psi)	Fuel	Battery (V)	Start (S) Finish(F)	Place	Comments
JT	3/24/98	8:21PM	8134.3	192	40	1/2	13	F	419B	
			277.4							
JT	3/25/98	8:04AM	"							
JT	3/25/98	8:40	8146.7	205	35	1/2-	13	F	Blowest	Instant start, key, button, ignition (order)
			289.8							
JT	3/25/98	9:15	8146.7	170	50	1/2-	14	S	Blowest	
			289.8							
JT	3/25/98	9:29	8151	200	40	1/4	13	F	FIT	
			294.1							
JT	3/25/98	10:10	"	170	55	1/4	13	S	FIT	
JT	3/25/98	10:23	8155.8	199	35	Red Mark	13	F	Blowest	Filled with 11.0 Gal Methanol => 9.85 mpg
			298.9							
JT	3/25/98	2:07PM	"	110	60	F+	14	S	Blowest	
JT	3/25/98	2:20	8160.7	192	55	F+	13	F	FIT	
			306.7							
JT	3/25/98	3:25	"	150	60	F+	14	S	FIT	
JT	3/25/98	3:39	8165.1	200	35	F+	13	F	Blowest	
			308.2							
JT	3/25/98	4:50	"	160	60	F+	14	S	Blowest	
JT	3/25/98	5:20	8178.7	195	35	F	13	F	419B	Via Wickham Rd.
			321.8							
JT	3/27/98	8:21AM	"	100	70	F-	15	S	419B	Instant start with Ether
JT	3/27/98	8:51	8191.2	193	35	F-	13	F	Blowest	
			334.3							
JT	3/27/98	9:00	"	190	45	F-	13	S	Blowest	
JT	3/27/98	9:11	8195.5	193	40	3/4	13	F	FIT	
			338.6							
JT	3/27/98	11:31	"	120	60	3/4+	13	S	FIT	
JT	3/27/98	11:43	8199.4	195	35	3/4+	13	F	Rooney's	
			342.3							
JT	3/27/98	12:45PM	"	160	60	3/4+	13	S	Rooney's	
JT	3/27/98	1:06	8209.3	195	35	3/4	13	F	Blowest	
JT	3/27/98		362.4							

APPENDIX C - PROCESS ECONOMIC STUDY

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

and

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

Process Economics Study - Outline

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

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

1. Introduction

1.1. Process Design Options.

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

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

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

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

2.2. Methanol Product Purification Cost.

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

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

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

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

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

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

Process Economics Study - Outline

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

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

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

2.6. Recommendations for Further Study.

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

Part Two - Baseload Power and Methanol Coproduction

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

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

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

3.1 Gasification Plant Size Fixed

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

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

3.2 Power Plant Size Fixed

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

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

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

End of Part Two.

Process Economics Study - Outline

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

Part Three - Coproduction for Intermediate Electric Load Following.

4. Intermediate Load Coproduction

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

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

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

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

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

4.2. Intermediate Load Coproduction - for Methanol Sales.

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

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

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

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

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

End of Part Three.

Process Economics Study - Outline

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

Part Four - Methanol Fuel Applications

5. Premium Methanol Fuel Applications

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

5.1. Hydrogen Source for:

- Hydrogen fuel cells, being developed for transportation applications, can achieve 65% system efficiency, as compared to 45% for diesel IC engines and 32% for gasoline IC engines. Methanol is a storable, transportable liquid fuel which can be reformed under mild conditions to provide 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 liquid H₂.

5.1.1. Fuel Cells for Transportation

5.1.2. Fuel Cells for Stationary Power

(See also dispersed power below).

5.1.3. Industrial Applications - Small Hydrogen Plants

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

5.2. Dispersed Power

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

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

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

End of Part Four.

Memorandum

To: Distribution Dept./Loc.:

From: W. R. Brown Dept./Ext.: PSED, X17584

Date: 31 March 1997

Subject: LPMEOH™ Process Economics - for IGCC Coproduction

Distribution:

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

APCI

E. C. Heydorn
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V. E. Stein
P. J. A. Tijm

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

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

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

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

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

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

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

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

Bill

APPENDIX D - DME DESIGN VERIFICATION TESTING

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

Telephone (610) 481-4911

30 June 1997

Mr. Robert M. Kornosky
Technical Project Manager
Mail Stop 920-L
U. S. Department of Energy
Federal Energy Technology Center
P. O. Box 10940
Pittsburgh, PA 15236-0940

Subject: Cooperative Agreement DE-FC22-92PC90543
Liquid Phase Methanol Demonstration Project
Liquid Phase Dimethyl Ether Design Verification Testing -
Recommendation

Dear Bob:

The updated version of the Recommendation to proceed with Design Verification Testing of the Liquid Phase Dimethyl Ether Process is attached. This document will be used during the Project Review Meeting on 24-25 July, at which time final approval by DOE and the Partnership will be requested.

Very truly yours,

A handwritten signature in dark ink, appearing to read "E. Heydorn", with a long horizontal line extending to the right.

Edward C. Heydorn
Program Manager
LPMEOH™ Demonstration Project

Enclosure

cc: Mr. William C. Jones - Eastman Chemical Co.
Mr. William J. O'Dowd - DOE-FETC
Mr. Edward Schmetz - DOE-FE-HQ
Dr. John Shen - DOE-FE-HQ
Mr. Barry T. Street - Eastman Chemical Co.
Mr. Peter Tijm - Air Products & Chemicals, Inc.

LPDME Recommendation

Summary

From the Statement of Work, "Commercial-Scale Demonstration of the Liquid Phase Methanol (LPMEOH™) Process," selected under Round 3 of the U.S. Department of Energy's (DOE's) Clean Coal Technology (CCT) Program: "Subject to Design Verification Testing (DVT), the Partnership proposes to enhance the Project by including the demonstration of the slurry reactor's capability to produce DME (*dimethyl ether*) as a mixed co-product with methanol." The first DVT step (Phase 1, Task 5), to address issues such as catalyst activity and stability, to provide data for engineering design, and to verify the market through engine tests and through market and economic study, is now complete. The market potential for DME is large, and progress in the laboratory toward developing a catalyst system whose performance meets the economic targets of a methanol equivalent productivity of 14 mol/kg catalyst-hr after 6 months of operation, producing at least 75% (by heating value) DME and 25% methanol.

A test of the Liquid Phase Dimethyl Ether (LPDME) at the LaPorte Alternative Fuels Development Unit (AFDU), in conjunction with the DOE's Liquid Fuels Program, would be appropriate if the catalyst system development can be completed successfully. An implementation decision, made mutually by the DOE's Clean Coal Technology LPMEOH™ project participants, and by the DOE's Liquid Fuels Program participants, should be made (by July of 1997) to implement testing at LaPorte in early 1998. (*Final dates should be recommended by the DOE's Liquid Fuels Program, based on progress in developing the LPDME catalyst system*).

Liquid Phase Dimethyl Ether (LPDME) Design Verification Testing (DVT)

From the Statement of Work, DOE's CCT LPMEOH™ project (Cooperative Agreement No. DE-FC22-92PC90543): "Subject to Design Verification Testing (DVT), the Partnership proposes to enhance the Project by including the demonstration of the slurry reactor's capability to produce DME as a mixed co-product with methanol. The production of DME from synthesis gas is a natural extension of the LPMEOH™ process in that three reactions occur concurrently in a single liquid phase reactor, methanol synthesis, methanol dehydration and water-gas shift. This process enhancement can significantly improve the overall conversion of coal derived synthesis gas to a storable blend of methanol and DME. -- -- -- the enhanced (DME production demonstration is complementary to ongoing studies being sponsored by DOE's Liquid Fuels Program --) -- . -- At the conclusion of each of the DVT steps, a joint Partnership/DOE decision will be made regarding continuation of methanol/DME demonstration.."

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

LPDME Recommendation

The LPDME Process Concept: - Three Concurrent Reactions:

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

The overall reaction, with carbon monoxide (CO)-rich synthesis gas (syngas), in a single liquid phase (slurry) reactor:

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

This is the "once-through" CO-rich syngas concept for the LPDME process utilizing a single slurry reactor.

Conversion per pass, with CO-rich syngas, can be higher than for the LPMEOHTM process. Methanol may also be produced, as a mixed co-product with the DME, and can easily be separated and recovered. The separation of DME from carbon dioxide (CO₂) will be necessary for certain market applications.

Status of the LPDME DVT Work

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

A-1. The market applications for DME are extensive. DME is, or may be, used as:

- Aerosol - Small, but established market. High purity DME is required.
- Cooking Fuel - Potentially a large market, to replace imported liquefied petroleum gas (LPG). There is a lot of interest in China, and DME is on the agenda for DOE's Pittsburgh Coal Conference in China (Sept. of 1997). Purity, of about >95% DME, with <2% methanol, < 3% CO₂ is estimated. An unresolved application issue is CO emissions during cooking. How does DME purity impact this? Use testing is needed.

Our contacts with representatives from the Institute of Coal Chemistry of the Chinese Academy of Sciences in Shanxi has provided the following assessment of the potential market for DME as a cooking fuel:

Of the 1.2 billion people in China, 0.3 billion live in cities. Of these, 1/3 currently use natural gas or LPG. Assuming 4 people per family, the 0.2 billion people who do not use gas or LPG converts to 50 million families. If DME captures 20-30% of the market share for these new applications, and the DME consumption is 200 kg per family per year, the demand for DME would be 2.4-3.0 million tons per year.

- Diesel Replacement Fuel. DME is an ultra clean (high Cetane) diesel fuel; and an 80% DME mixture with methanol and water is now being engine-tested by others (Amoco, et. al.). Market development (at least in the U.S.) faces a fuel distribution infrastructure problem. DME might

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more easily replace LPG in countries where LPG is already an engine fuel. Diesel use in the U.S. is projected to increase by 1.5 percent a year, assuming an economic growth of 1.9 percent a year. This will raise consumption from over 4 quadrillion BTU to approaching 6 quadrillion BTU (Reference 1). This corresponds to an annual increase of almost 1.4 million gallons per year of diesel consumption.

- DME Derivatives as a Diesel Fuel Additive. Quotes from the DOE Liquid Fuels Program (Contract No. DE-FC22-95PC93052) quarterly report for April-June 1996: "Initial Cetane number (CN) testing of a three-component composition of 1,2-dimethoxy ethane, 1,1-dimethoxy methane and methanol blended with diesel fuel showed a 40% increase in the CN of the diesel fuel when the blend was 50/50." "The concept of adding a blend of oxygenated compounds to diesel fuel in order to enhance the Cetane value and cold start properties is being investigated. The blend of oxygenated compounds is derived from dimethyl ether chemistry, and builds on work conducted earlier --." The testing of this DME feedstock chemistry is in its early days, but it is possible that CO₂ may not need to be separated from the DME prior to the production of DME derivatives. The 50/50 blend referenced above would therefore provide a large market opportunity for the projected U.S. market growth (Reference 1), let alone for the present consumption.
- DME Derivatives as Chemicals/Other Fuels. DME is a key intermediate in a commercial synthesis gas-to-gasoline process, and is being developed as an intermediate for other chemicals and fuels as part of the DOE's Liquid Fuels Program. The fit for DME here is long-term.

A-2. The economics studies, for once-through coproduction (with an integrated gasification combined cycle (IGCC) power plant, for example) on synthesis gas rich in carbon oxides, show that the LPDME process will have an economic advantage greater than the LPMEOHTM process. A once-through LPDME reactor is able to convert greater than 50% of such a syngas, whereas a once-through LPMEOHTM reactor can convert only about 30%. The economics, of course, depend upon the end-use (purity) of the DME and upon the gasification plant's coproduct mix (amount of power, methanol, DME, etc.). The same liquid phase reactor design options to increase syngas conversion (Reference 2); such as feed gas compression and/or CO-rich gas recycle; are also be applicable for LPDME. So, the LPDME technology has the potential to improve on the 5-10 cents per gallon (methanol equivalent) advantage over the LPMEOHTM process for the coproduction of DME to serve local markets.

As with the LPMEOHTM process, gas phase process technology must be considered as the economic competitor. The gas phase DME process (Reference 3) must run with hydrogen (H₂)-rich syngas. In the IGCC coproduction flow sheet (shown in Figure 1), gas phase technology is at an economic disadvantage, since separate shift and CO₂ removal are required. As is the case for methanol, inexpensive remote natural gas would therefore be the economic plant site choice for gas phase technology. A comparison, of IGCC/LPDME coproduction with DME imported from remote gas facilities, shows an advantage of 20-30% for locally produced DME relative to

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imported DME. The transportation cost to import DME is much higher than for methanol, and the LPDME coproduction advantage is even greater than that for LPMEOH™ (vs. methanol import) (Reference 2). Dehydration of imported methanol to make DME is not competitive either. Therefore, for DME in local markets, LPDME coproduction should be a winner!

With H₂-rich syngas, the LPDME process loses its (once-through, high conversion per pass) economic advantage. The overall reaction, with (> 2:1) H₂-rich syngas is:

Since water inhibits the methanol dehydration reaction, the slurry reactor must be staged, with water removal between stages. Staging could be by high ratio gas recycle, and/or with multiple reactors; but the once-through simplicity is lost. Therefore, it is unlikely that the LPDME process would be developed for use in H₂-rich syngas applications.

A cost estimate of commercial-scale LPDME plants has been performed. This work has helped quantify the targets for the laboratory R&D program (summarized in Part B). From these studies, a commercially successful LPDME system is defined for a Texaco-type synthesis gas (35 mol% H₂, 51 mol% CO, 13 mol% CO₂) available at 500 PSIG. At a reactor operating pressure of 950 PSIG and a space velocity of 4,000 liters/hr-kg catalyst, the LPDME catalyst system must have a methanol equivalent productivity of 14 mol/kg catalyst-hr after 6 months of operation, producing at least 75% (by heating value) DME and 25% methanol. Figure 2 shows the effect of plant size on DME cost. These costs are competitive with LPG in China (Section A-1).

B. Laboratory R&D Results

Summary of work through end of funding by CCT LPMEOH™ Project (9/96): An LPDME catalyst system, with reasonable long-term activity (57% of initial activity after 1000 hours), productivity (equivalent methanol productivity of 29 mol/kg catalyst-hr), and selectivity (79% carbon selectivity to DME, CO₂-free basis), was identified and tested. The system exhibits best activity under CO-rich syngas conditions, i.e. those most likely for (IGCC) coproduction. Accelerated aging of the catalyst system is a remaining issue. Water concentrations in the liquid phase reactor are higher with syngases richer in H₂, and its effect needs to be evaluated.

Laboratory work has continued under the DOE's Liquid Fuels Program. The issues, to be addressed in the lab before a decision on a test run at the DOE's AFDU in LaPorte, are:

- 1) Understanding the LPDME catalyst system's accelerated aging; and modifying the catalyst and/or the system operating conditions; and
- 2) Manufacturing scale-up of catalyst for a LaPorte AFDU run.

LPDME Recommendation

Progress has been made in the laboratory effort. Figure 3 shows the performance for the first DME catalyst which was tested; goals from the Liquid Fuels Program are provided for reference. After further study, an improved DME catalyst (AB-05) was tested with two LPMEOH™ catalysts (S8-86 and MK-101); the results of a 700 hour life study are presented in Figure 4. When compared with the program goals (summarized in Figure 5), the catalyst performance of the newer catalyst is approaching the commercial targets defined in Section A. The status of the laboratory program is summarized in the following table:

	Liquid Fuels Program Goals	Commercial Targets	Laboratory Results
Catalyst Productivity, mol/kg catalyst-hr (MeOH-equivalent)	> 28 (Initial Productivity)	> 14 (productivity for aged catalyst)	28 (Initial Productivity)
Catalyst Selectivity	DME Selectivity > 80% (% Carbon, CO ₂ -free)	DME = 75%, Methanol = 25% (heating value basis)	DME Selectivity = 79% (% Carbon, CO ₂ -free)
Catalyst Life	> 50% Remaining Activity after 1000 hours	Target Productivity after 6 months of operation	57% Remaining Activity after 1000 hours

Initial discussions with catalyst manufacturers have been held. Once a manufacturer is selected, a laboratory-scale catalyst batch will be produced and tested in the autoclave to verify the production technique developed at Air Products. An interim 1 lb batch will then be produced and tested. Once the catalyst production techniques have been verified at this scale, the 200 lb LaPorte batch will be produced using the same methodology as for a full commercial batch. An autoclave check of this material will be performed prior to the start of the LaPorte AFDU run.

Recommendations

The catalyst system and the market applications/opportunities are sufficiently promising that proof-of-concept testing at the LaPorte AFDU is recommended. Kingsport is an unlikely site for the commercial size demonstration of LPDME, since there are limited times for CO-rich syngas testing; and H₂-rich syngas would create water buildup. Therefore, the basis for commercializing LPDME must come from:

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

LPDME Recommendation

- 2) continuing work in hydrodynamics of slurry reactors (other ongoing DOE programs); and
- 3) reactor performance (methanol catalyst activity and life, hydrodynamics, and heat transfer) from the LPMEOH™ Process Demonstration Unit.

The tie-in between the laboratory and the LaPorte AFDU is important. Historically, the rate of deactivation of methanol synthesis catalyst has been greater in the autoclave than at the AFDU; this may be a result of loss of catalyst from the autoclave, or due to greater carbonyl poisoning as a result of the higher surface-to-volume ratio at the laboratory scale. Testing at the engineering scale of the LaPorte AFDU can eliminate this variable. Operation of the LPMEOH™ Process Demonstration Unit will provide data on catalyst life under coal-derived syngas and at the larger engineering scale (the tie-in to the LaPorte AFDU for commercialization).

The recommendations for proceeding with DVT of the LPDME catalyst system are:

- An LPDME test run at the LaPorte AFDU, in conjunction with the DOE's Liquid Fuels Program, would be appropriate if the LPDME catalyst system development can be completed successfully. Up to \$875,000 of CCT LPMEOH™ Project budget support, from the Cost Plan (22 October 1996), should be made available to support a suitable LPDME test run at LaPorte.
- An implementation decision, made mutually by the DOE's CCT (DE-FC22-92PC90543) LPMEOH™ Project participants, and by the DOE's Liquid Fuels (DE-FC22-95PC93052) Program participants, should be made (by July of 1997) in time to implement testing at LaPorte in early 1998. (*Final dates should be recommended by the DOE's Liquid Fuels Program, based on progress in developing the LPDME catalyst system*). The CCT LPMEOH™ Project participants shall be kept informed (via review meetings and status reports) by Air Products of the development by the DOE Liquid Fuels Program participants of the LaPorte AFDU LPDME test-run plans, so that a timely final approval can be made
- In the interim, some DME product-use testing may be appropriate for the LPMEOH™ Demonstration Project's off-site product-use testing.

The schedule for the proposed LPDME testing at the LaPorte AFDU and possible implementation at the Kingsport LPMEOH™ Process Demonstration Facility is summarized below:

DME DVT Decision Made	July 1997
Commercial-Scale DME Catalyst Produced/Tested in Laboratory Autoclave	January 1998
LaPorte AFDU Test	February/March 1998
Kingsport Decision Made	March/April 1998
Kingsport Implementation (Provisional) Plan	July 1998 - March 2001

LPDME Recommendation

Impact on CCT Project

Technical: The commercialization of the LPDME Process can be successfully achieved by the combination of the activities at the LaPorte AFDU and the LPMEOH™ Process Demonstration Unit described previously.

Cost: Up to \$875,000 of Project funds would be available to support a suitable LPDME run. An update of the CCT Project's Cost Plan (22 October 1996), based upon the DVT Recommendation, will be performed following the joint Partnership/DOE decision.

Schedule: If the DVT Recommendation is approved by the Partnership and DOE, the operating schedule for the LPMEOH™ Process Demonstration Unit will remain unchanged from the current Demonstration Test Plan (September 1996). The DVT would proceed according to the September 1996 DME Milestone Plan (included in the Demonstration Test Plan) and the schedule of the Liquid Fuels Program.

References

1. Transportation energy consumption by fuel, 1975, 1995 and 2015: History: Energy Information Administration, *Short-Term Energy Outlook*, DOE/EIA-0202(96/4Q) (Washington, DC, October 1996), and *State Energy Data Report 1994*, DOE/EIA-0214(98). Projections: Table A2. Internet access at <http://www/eia.doe.gov/oiaf/aec97/figure.html#fig46>.
2. "Fuel and Power Coproduction - The Liquid Phase Methanol™ Process Demonstration at Kingsport", paper presented at Fifth Annual DOE Clean Coal Technology Conference, Tampa, FL, January 7-9, 1997.
3. Haldor Topsoe AS, "Preparation of Fuel Grade Dimethyl Ether", International Publication Number WO9623755, World International Property Organization, 08 August 1996.

(end).

Figure 1. Once-through Methanol Coproduction with IGCC Electric Power

Figure 2

DME COST VERSUS SIZE

TEXACO-TYPE SYNTHESIS GAS, 1:1 FEED:RECYCLE

—■— SYNTHESIS GAS ONLY

—◆— SYNTHESIS GAS PLUS CONVERSION

—▲— TOTAL DME COST

GOALS

FIGURE 3

MEOH Equiv. Prod. (mol/kg-hr)

FIGURE 4

- Six catalyst samples (#1 - #6) were developed with good stability and decent activity.

FIGURE 5

APPENDIX E - SAMPLES OF DETAILED MATERIAL BALANCE REPORTS

RUN NO: K6-01/02

TITLE: Catalyst Addition and Aging

Balance Period:

Start Date	02Jan98 07:00:00
End Date	03Jan98 07:00:00

Time From Start of Run (days)

Start	13.3
End	14.3

Reaction Conditions

Temperature (°F)	454
Pressure (psig)	680
Space Velocity (sL/kg-hr)	9601
Vg (inlet)	0.77

Slurry Data

Catalyst Weight (lb oxide)	19,500
Slurry Concentration (wt %)	31.0
Slurry Level (ft)	53.5
Gas Holdup (vol %)	52.5
Gassed Slurry Volume (ft3)	2249

Performance Results

Raw MeOH Production (ton/day)	279.8
Raw MeOH Production (ton/day)	267.2
Syngas Utilization (SCF/lb MeOH)	40.7
Catalyst Life (eta)	1.14

(gas measurements)	279.8
(liquid measurements)	267.2

Energy Balance

Steam Production (lb/hr)	21015
Steam Drum Pressure (psig)	203
Steam Import/Export (lb/hr)	1687
Reactor O-T-M Conversion (% LHV)	24.2
Weighted Tube Length (ft)	49.7
Heat Transfer Area (ft2)	1929
Reactor Overall U (BTU/hr-ft2-F)	153

CO Conversion (total) (%)	41.4
---------------------------	------

CO Conversion to MeOH (%)	43.2
---------------------------	------

CO Conversion to H2 (%)	-1.97
-------------------------	-------

Syngas Conversion (% LHV)	79.8
---------------------------	------

Syngas Usage (BTU/gallon MeOH)	68,296
--------------------------------	--------

Recycle Ratio	2.50
---------------	------

MeOH Productivity (gmol/kg-hr)	35.73
--------------------------------	-------

Rxr Volumetric Productivity (ton/day-ft3)	0.119
---	-------

Sparger "K"-value	4.58
-------------------	------

Atom/Mass Balance Closure (% of reactor Inlet)

C	99.42
H	99.91
O	98.85
N	102.25
Total Mass	99.19

Liquid Product Analysis (wt%)

Methanol	99.96
Ethanol	0.01
Water	0.03
Oil	0.00
Total	100.00

REFINED PRODUCT

1/2	1/3
19:00	7:00

CRUDE PRODUCT

1/2	1/3
19:00	7:00
91.73	91.93
0.10	0.10
8.13	8.00
0.06	0.06
100.02	100.09

RUN NO: K6-01/02 TITLE: Catalyst Addition and Aging

	FRESH FEED	CO MAKEUP	H2 MAKEUP	K-01 OUTLET	REACTOR FEED	C-05 OUTLET	MAIN PURGE	DISTILL. PURGE	CRUDE PRODUCT	REFINED PRODUCT
T	80	38	39	109	291	249	89	55	224	74
P	743	870	552	708	700	670	656	8	185	133
Comp	67.47	1.93	77.66	73.52	71.44	65.24	73.52	8.00	0.00	0.00
(mol %)	29.47	97.05	9.12	15.11	19.29	13.58	15.11	7.00	0.00	0.00
N2	0.43	0.97	6.50	4.20	3.08	3.70	4.20	2.00	0.00	0.00
CH4	0.03	0.05	0.92	0.32	0.24	0.28	0.32	0.00	0.00	0.00
CO2	2.60	0.00	3.76	6.64	5.42	6.05	6.64	62.00	0.00	0.00
DME	0.00	0.00	0.01	0.00	0.00	0.01	0.00	4.00	0.00	0.00
MeOH	0.00	0.00	0.06	0.21	0.54	10.66	0.21	10.00	86.43	99.93
EtOH	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.07	0.01
H2O	0.00	0.00	0.00	0.00	0.00	0.47	0.00	0.00	13.50	0.06
others	0.00	0.00	1.97	0.00	0.00	0.01	0.00	7.00	0.01	0.00
TOTAL	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Mole Wt	10.885	27.501	7.770	9.934	10.304	12.372	9.934	39.217	30.175	32.032
LHV	253.2	0.0	0.0	575.5	835.3	809.0	23.4	0.9	62.7	199.3
Enthalpy	-43.93	0.00	0.00	-110.24	-144.74	-175.70	-4.51	-1.67	-25.54	-49.38
Flow	906,498	0	0	2,264,298	3,168,812	2,639,532	91,981	4,858	94,616	181,945
lbmol/hr	2,389.6	0.0	0.0	5,969.0	8,353.4	6,958.1	242.5	12.8	249.4	479.6
lb/hr	26,011	0	0	59,297	86,075	86,084	2,409	502	7,526	15,364

RUN NO: K3-02/08

TITLE: Texaco-type Syngas and Fuel-Grade Methanol Production

Balance Period:

Start Date
End Date

08/feb98 19:00:00
09/feb98 07:00:00

Time From Start of Run (days)

Start
End

50.8
51.3

Reaction Conditions

Temperature (°F)
Pressure (psig)
Space Velocity (sL/kg-hr)
Vg (inlet)

437
710
7216
0.69

Slurry Data

Catalyst Weight (lb oxide)
Slurry Concentration (wt %)
Slurry Level (ft)
Gas Holdup (vol %)
Gassed Slurry Volume (ft3)

24,800
33.7
62.0
54.2
2611

Performance Results

Raw MeOH Production (ton/day)
Raw MeOH Production (ton/day)
Syngas Utilization (SCF/lb MeOH)
Catalyst Life (eta)

211.7
199.7
44.2
0.89

(gas measurements)
(liquid measurements)

Energy Balance

Steam Production (lb/hr)
Steam Drum Pressure (psig)
Steam Import/Export (lb/hr)
Reactor O-T-M Conversion (% LHV)
Wetted Tube Length (ft)
Heat Transfer Area (ft2)
Reactor Overall U (BTU/hr-ft2-F)

15186
218
3308
17.8
57.0
2212
147

CO Conversion (total) (%)
CO Conversion to MeOH (%)
CO Conversion to H2 (%)

12.5
12.4
-0.03

Syngas Conversion (% LHV)
Syngas Usage (BTU/gallon MeOH)
Recycle Ratio
MeOH Productivity (gmol/kg-hr)
Rrx Volumetric Productivity (ton/day-ft3)
Spatger "K"-value

72.1
68,422
3.12
21.15
0.076
4.78

Atom/Mass Balance Closure (% of reactor inlet)

C
H
O
N
Total Mass

99.64
100.90
99.70
100.01
99.78

Liquid
Product
Analysis
(wt%)

FUEL-Grade PRODUCT

	2/8	2/9	2/9
	21:26	0:13	5:42
Methanol	99.20	99.27	99.30
Ethanol	0.26	0.24	0.25
Water	0.34	0.30	0.29
Oil	0.20	0.20	0.20
Total	100.00	100.01	100.04

RUN NO: K3-02/08 TITLE: Texaco-type Syngas and Fuel-Grade Methanol Production

	FRESH FEED	CO MAKEUP	H2 MAKEUP	K-01 OUTLET	REACTOR FEED	C-05 OUTLET	MAIN PURGE	DISTILL. PURGE	CRUDE PRODUCT	FUEL-Grade PRODUCT
T	61	43	44	269	287	244	87	57		213
P	776	870	566	750	738	694	673	8		80
Comp (mol %)										
H2	59.96	1.93	77.66	32.67	39.16	29.88	32.20	8.00		0.00
CO	37.88	97.05	9.12	57.97	52.88	53.33	57.40	7.00		0.00
N2	0.46	0.97	6.50	2.47	1.97	2.27	2.45	2.00		0.00
CH4	0.02	0.05	0.92	0.10	0.08	0.09	0.10	0.00		0.00
CO2	1.69	0.00	3.76	6.55	5.33	6.12	6.73	62.00		0.00
DME	0.00	0.00	0.01	0.00	0.00	0.01	0.00	4.00		0.00
MeOH	0.00	0.00	0.06	0.24	0.57	8.23	1.12	10.00		99.25
EtOH	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.00		0.17
H2O	0.00	0.00	0.00	0.00	0.00	0.04	0.00	0.00		0.55
others	0.00	0.00	1.97	0.00	0.00	0.01	0.00	7.00		0.02
TOTAL	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00		100.00
Mole Wt	12.695	27.501	7.770	20.564	18.698	21.542	20.751	39.217		31.980
LHV	209.9	0.0	0.0	637.0	852.6	832.3	37.6	0.9		151.4
Enthalpy	-40.69	0.00	0.00	-227.16	-265.20	-289.77	-14.01	-1.68		-52.32
Flow										
SCFH	735,255	0	0	2,294,723	3,029,198	2,629,414	133,624	4,899		198,497
lbmol/hr	1,938.2	0.0	0.0	6,049.2	7,985.3	6,931.4	352.2	12.9		523.3
lb/hr	24,605	0	0	124,397	149,307	149,320	7,310	506		16,734

RUN NO: K6-03/07

TITLE: Catalyst Addition and Aging

Balance Period:

Start Date
End Date

07mar98 07:00:00
08mar98 07:00:00

Time From Start of Run (days)

Start
End

77.3
78.3

Reaction Conditions

Temperature (°F)
Pressure (psig)
Space Velocity (sL/kg-hr)
Vg (inlet)

437
710
6295
0.67

Slurry Data

Catalyst Weight (lb oxide)
Slurry Concentration (wt %)
Slurry Level (ft)
Gas Holdup (vol %)
Gassed Slurry Volume (ft3)

27,450
33.5
58.5
45.8
2462

Performance Results

Raw MeOH Production (ton/day)
Raw MeOH Production (ton/day)
Syngas Utilization (SCF/lb MeOH)
Catalyst Life (eta)

230.4 (gas measurements)
220.3 (liquid measurements)
39.7
0.79

Energy Balance

Steam Production (lb/hr)
Steam Drum Pressure (psig)
Steam Import/Export (lb/hr)
Reactor O-T-M Conversion (% LHV)
Wetted Tube Length (ft)
Heat Transfer Area (ft2)
Reactor Overall U (BTU/hr-ft2-F)

16796
195
3812
21.4
54.7
2123
144

Syngas Conversion (% LHV)
Syngas Usage (BTU/gallon MeOH)
Recycle Ratio
MeOH Productivity (gmol/kg-hr)
Rrx Volumetric Productivity (ton/day-ft3)
Sparger "K"-value

81.9
67,536
3.04
20.93
0.089
5.50

Atom/Mass Balance Closure (% of reactor inlet)

C
H
O
N
Total Mass

100.23
101.04
99.81
100.95
100.26

Liquid Product Analysis (wt%)

Methanol
Ethanol
Water
Oil
Total

REFINED PRODUCT

3/7 19:00
3/8 7:00
99.99 0.01 0.00 0.00 100.00

CRUDE PRODUCT

3/7 19:00
3/8 7:00
88.76 0.09 11.20 0.06 100.11
88.99 0.09 10.96 0.06 100.10

RUN NO: K6-03/07 TITLE: Catalyst Addition and Aging

	FRESH FEED	CO MAKEUP	H2 MAKEUP	K-01 OUTLET	REACTOR FEED	C-05 OUTLET	MAIN PURGE	DISTILL. PURGE	CRUDE PRODUCT	REFINED PRODUCT
T	87	58	59	269	283	244	90	64	221	79
P	745	867	690	738	731	702	691	6	185	138
Comp	67.57	1.93	77.66	75.23	73.12	67.79	75.23	8.00	0.00	0.00
(mol %)	29.22	97.05	9.12	12.82	16.91	11.58	12.82	7.00	0.00	0.00
	0.43	0.97	6.50	4.91	3.80	4.46	4.91	2.00	0.00	0.00
	0.09	0.05	0.92	1.03	0.80	0.94	1.03	0.00	0.00	0.00
	2.69	0.00	3.76	5.01	4.41	4.67	5.01	62.00	0.00	0.00
	0.00	0.00	0.01	0.00	0.00	0.01	0.00	4.00	0.00	0.00
	0.00	0.00	0.06	0.29	0.56	9.40	0.29	10.00	81.80	99.99
	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.06	0.01
	0.00	0.00	0.00	0.00	0.00	0.51	0.00	0.00	18.14	0.01
	0.00	0.00	1.97	0.69	0.41	0.64	0.69	7.00	0.01	0.00
TOTAL	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Mole Wt	10.867	27.501	7.770	9.161	9.647	11.293	9.161	39.217	29.523	32.040
LHV	203.4	0.0	0.0	572.1	777.5	756.0	16.0	0.7	45.6	121.0
Enthalpy	-35.31	0.00	0.00	-80.80	-114.07	-139.95	-2.47	-1.41	-19.81	-42.81
Flow	728,230	0	0	2,211,719	2,924,595	2,491,955	61,909	4,132	72,675	157,909
	1,919.7	0.0	0.0	5,830.4	7,709.6	6,569.1	163.2	10.9	191.6	416.3
	20,861	0	0	53,410	74,375	74,183	1,495	427	5,656	13,337

APPENDIX F - RESULTS OF DEMONSTRATION PLANT OPERATION

**Table 1 - Summary of LPMEOH™ Demonstration Unit Outages -
January/March 1998**

Figure 1 - Catalyst Age (η) vs. Days Onstream - Second Catalyst Batch

**Figure 2 - Sparger Resistance Coefficient vs. Days Onstream
(Post-19 December 1997 Restart)**

**Figure 3 - Sparger Resistance Coefficient vs. Days Onstream
(Since April 1997 Startup)**

Table 1 - Summary of LPMEOH™ Demonstration Plant Outages - January/March 1998

Operation Start	Operation End	Operating Hours	Shutdown Hours	Reason for Shutdown
1/1/98 00:01	1/14/98 05:23	317.4	0.2	C-03 Outlet Plugged
1/14/98 05:35	1/14/98 05:43	0.1	0.1	ESD on C-02 Level
1/14/98 05:49	1/14/98 07:53	2.1	0.4	ESD on C-02 Level
1/14/98 08:17	1/23/98 00:20	208.1	55.9	Syngas Outage
1/25/98 08:15	2/13/98 07:15	455.0	29.0	Syngas Outage
2/14/98 12:15	2/14/98 15:55	3.7	0.2	ESD on C-02 Level
2/14/98 16:10	3/31/98 23:59	1087.8		End of Reporting Period
Total Operating Hours				2074.1
Syngas Available Hours				2075.1
Plant Availability, %				99.95

Figure 1

Catalyst Age (η)

Figure 2

Sparger Resistance Coefficient (Post-December 1997 Restart)

Figure 3

Sparger Resistance Coefficient (since April 1997 Startup)

**APPENDIX G - ANALYTICAL REPORTS - FUEL-GRADE METHANOL FROM
LPMEOH™ DEMONSTRATION UNIT**

Analysis Report

To: Robert R. Broekhuis Dept./Loc.: GEG / I7066
From: Patrick J. Clark Dept./Ext.: CRSD-ATC / 16504
Date: 06 April 1998 Lab Name: Separations
Subject: Gas Chromatographic Analysis of Methanol Product from LPMeOH Process
Sample No.: See Tables 1-2

c: CS File, LB File, A.J. Di Gioia

SUMMARY:

Samples of methanol product from the Liquid Phase Methanol process were analyzed by gas chromatography to quantify the amount of impurities present. The weight percent concentrations of the impurities present in each sample are listed in Tables 1-2. A representative chromatogram of one of the samples is attached.

PROBLEM DEFINITION:

The identification and concentration of the impurities in methanol product from the LPMeOH process is of interest.

ANALYTICAL PROCEDURES:

The gas chromatographic analysis was performed using a 60 meter Supelco SPB-5 capillary column interfaced to a flame ionization detector. The gas chromatographic operating conditions are listed in Table 3.

The sample was analyzed neat without dilution with solvent. Quantitation of the impurities was obtained using an external standardization calculation. A calibration standard was prepared to contain a number of available alcohol components at concentration levels ranging from 0.1 to 0.5 wt% in methanol. The response factor of the nearest alcohol present in the calibration standard was used to calculate the concentration of the esters and branched higher alcohols in the sample.

Request No.:	049553 / 050058	Analyst:	PJC
Charge No.:	LRKAPOTH34		
Notebook No.:	16235		
Method No.:			
Phone Date:			
Sample Receipt Date:	09 March / 01 April 1998	Doc. Name:	049553broekhuis.doc 050058broekhuis.doc

Table 1

Concentration of Impurities (weight percent)

Compound	Retention Time (min)	tanker 268 drum 1	1302 / 10	1302 / 40	1302 / 80
ethanol	6.90	0.255	0.251	0.257	0.241
2-propanol	7.72	0.0072	0.0072	0.0072	0.0068
1-propanol	9.36	0.0896	0.0880	0.0894	0.0840
2-butanol	10.78	0.0178	0.0176	0.0179	0.0168
isobutanol	11.75	0.0086	0.0084	0.0086	0.0081
methyl propanoate	11.93	0.0023	0.0022	0.0023	0.0022
1-butanol	13.17	0.0483	0.0475	0.0484	0.0456
2-pentanone	14.27	0.0018	0.0014	0.0014	0.0014
3-pentanol	14.68	0.0086	0.0085	0.0086	0.0081
2-pentanol	14.76	0.0049	0.0048	0.0049	0.0046
methyl butanoate	15.77	0.0031	0.0030	0.0031	0.0029
2-methyl-1-butanol	16.23	0.0023	0.0028	0.0024	0.0022
3-methyl-1-butanol	16.43	0.0106	0.0104	0.0106	0.0099
1-pentanol	17.66	0.0290	0.0285	0.0290	0.0273
3-hexanol	19.02	0.0038	0.0038	0.0038	0.0036
2-hexanol	19.16	0.0047	0.0046	0.0047	0.0044
methyl pentanoate	20.29	0.0021	0.0020	0.0021	0.0020
2-methyl-1-pentanol	20.67	0.0050	0.0050	0.0050	0.0047
1-hexanol	22.18	0.0160	0.0157	0.0160	0.0150
3-heptanol	23.46	0.0021	0.0020	0.0020	0.0019
2-heptanol	23.58	0.0025	0.0024	0.0025	0.0023
methyl hexanoate	24.63	0.0012	0.0012	0.0013	0.0012
2-methyl-1-hexanol	24.98	0.0039	0.0038	0.0038	0.0036
1-heptanol	26.50	0.0081	0.0079	0.0080	0.0075
1-octanol	30.56	0.0037	0.0036	0.0037	0.0035
1-nonanol	34.34	0.0019	0.0019	0.0019	0.0018
total impurities (incl. Others)		0.585	0.579	0.591	0.548
methanol purity (by difference)		99.415%	99.421%	99.409%	99.452%

The values listed above are the average of two determinations with an approximate percent relative standard deviation of 0.5-3%.

Table 2

Concentration of Impurities (weight percent)

Compound	Retention Time (min)	1302 / last	268 / partial	268 / 40	268 / 80
ethanol	6.90	0.270	0.254	0.249	0.278
2-propanol	7.72	0.0076	0.0071	0.0070	0.0078
1-propanol	9.36	0.0943	0.0884	0.0867	0.0965
2-butanol	10.78	0.0189	0.0177	0.0174	0.0193
isobutanol	11.75	0.0091	0.0085	0.0083	0.0093
methyl propanoate	11.93	0.0024	0.0023	0.0022	0.0025
1-butanol	13.17	0.0512	0.0480	0.0470	0.0524
2-pentanone	14.27	0.0015	0.0015	0.0017	0.0016
3-pentanol	14.68	0.0091	0.0085	0.0084	0.0093
2-pentanol	14.76	0.0052	0.0048	0.0048	0.0053
methyl butanoate	15.77	0.0032	0.0030	0.0030	0.0033
2-methyl-1-butanol	16.23	0.0023	0.0023	0.0023	0.0026
3-methyl-1-butanol	16.43	0.0111	0.0105	0.0103	0.0114
1-pentanol	17.66	0.0305	0.0288	0.0282	0.0314
3-hexanol	19.02	0.0040	0.0038	0.0037	0.0042
2-hexanol	19.16	0.0050	0.0047	0.0046	0.0051
methyl pentanoate	20.29	0.0022	0.0021	0.0020	0.0023
2-methyl-1-pentanol	20.67	0.0052	0.0050	0.0049	0.0055
1-hexanol	22.18	0.0168	0.0159	0.0156	0.0174
3-heptanol	23.46	0.0021	0.0020	0.0020	0.0022
2-heptanol	23.58	0.0026	0.0025	0.0024	0.0027
methyl hexanoate	24.63	0.0013	0.0012	0.0012	0.0014
2-methyl-1-hexanol	24.98	0.0040	0.0038	0.0038	0.0042
1-heptanol	26.50	0.0088	0.0079	0.0076	0.0085
1-octanol	30.56	0.0039	0.0037	0.0036	0.0040
1-nonanol	34.34	0.0020	0.0018	0.0019	0.0021
total impurities (incl. Others)		0.623	0.584	0.574	0.638
methanol purity (by difference)		99.377%	99.416%	99.426%	99.362%

The values listed above are the average of two determinations with an approximate percent relative standard deviation of 0.5-3%.

Table 3

Operating Conditions for Gas Chromatographic Analysis

Column: Supelco SPB-5 capillary column, 60 meter x 0.25 mm ID, 1.0 micron film thickness

Oven Temperature Program

Initial Temperature	40 °C
Initial Time	2 min
Program Rate	5 °C/min
Final Temperature	280 °C
Final Time	5 min
Total Analysis Time	55 min

Carrier Gas Helium

Carrier Gas Flow Rate (at 50 °C)	2 mL/min
Column Headpressure (at 50 °C)	25 psig
Splitter Flow Rate	30 mL/min

Injector Temperature 250 °C

Injection Mode / Volume Hewlett-Packard 7673 Autosampler / 1 microliter

Detector Type Flame Ionization Detector

Detector Temperature	300 °C
Detector Range	1

Analysis Report

To: R. Broekhuis Dept./Loc.: GEG/I7066
From: C. Mengel-Smith Dept./Ext.: CRSD/16973
Date: 13 May 1998 Lab Name: Wet Organic
Subject: Crude Methanol for Water Content Analysis
Sample No.: see table

c: E. Heydorn; S. Reidy; CMS/lb

SUMMARY:

Samples of crude fuel-grade methanol were analyzed by coulometric titration for the water content. The samples were run in duplicate, both results are reported. If the difference between the two determinations was greater than one standard deviation, a third determination was made and the average of the three reported.

Sample ID	Wt. % Water
1302/10	0.456, 0.459
1302/last	0.482(0.036)*
268/partial	0.473(0.022)*
1302/40	0.461, 0.461
268/40	0.462, 0.456
1302/80	0.465, 0.476
268/80	0.470, 0.463

* The value is reported in the format X(s), where X is the mean of three determinations and s is the standard deviation.

ANALYTICAL PROCEDURES:

Water was determined by coulometric Karl Fischer titration of the weighed samples following analytical method CRSD-A-002-90. The Mitsubishi CA-05 Moisture Meter and the Sartorius research balance were used for the analysis. Based on historical data ranging from 0.1 to 1.0 wt.% water, one standard deviation by this method is estimated to be 0.015 wt.% water.

Request No.:050058
Charge No.:LRKAPOTH34
Notebook No.:16498-49
Method No.:CRSD-A-002-90
Phone Date;
Sample Receipt Date:4/1/98

Analyst:CMS

Doc. Name:r:/wetorg/050000/050058

**APPENDIX H - MILESTONE SCHEDULE STATUS AND COST MANAGEMENT
REPORTS**

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

LIQUID PHASE METHANOL DEMONSTRATION - PHASE III

Task Name	Start Date	Duration in Weeks	End Date	Percent Complete
Task 2.1.1 - Process Shutdown and Catalyst Aging	4/2/97	68.00w	7/22/98	74%
Test 1 - Initial Shutdown, and Design Production Tests	4/2/97	5.43w	5/10/97	100%
Test 2 - Gassed Slurry Level	4/2/97	28.28w	10/17/97	100%
Test 3 - Reactor Feed: Texaco-Type Syngas	2/10/98	0.71w	2/13/98	100%
Test 4 - Early Testing @ High Superficial Velocity	2/13/98	3.00w	3/6/98	0%
Test 5 - Check @ Test 1 Conditions	6/10/97	3.43w	7/4/97	100%
Test 6 - Catalyst Addition and Aging	12/7/98	13.29w	4/30/99	66%
Test 7 - Free-Drain Entrained/ Condensed Oil to Reactor	4/2/97	53.00w	4/8/98	87%
Test 8 - Operation @ Design Feed Gas Rates	5/1/98	2.00w	5/15/98	0%
Test 9 - Check for Limitation on Catalyst Slurry Concentration	5/15/98	7.00w	7/3/98	0%
Test 10 - Catalyst Addition to Reach Max Productivity	5/15/98	9.71w	7/22/98	0%
Task 2.1.2 - Process Operational Test Phase	7/22/98	105.00w	7/25/00	1%
Test 11 - Catalyst Addition/ Withdrawal Test	7/22/98	6.00w	9/2/98	0%
Test 12 - Test 11 Conditions with No CO Make-up	9/2/98	2.00w	9/16/98	0%
Test 13 - Test 11 Conditions with No H ₂ Make-up	9/16/98	3.00w	10/7/98	0%
Test 14 - Test 11 Conditions with No H ₂ or CO Make-up	10/7/98	2.00w	10/21/98	0%
Test 15 - Repeat of Test 11 Conditions	10/21/98	2.00w	11/4/98	0%
Test 16 - Design Fresh Feed Operation Test	11/4/98	3.00w	11/25/98	0%
Test 17 - Testing @ High Superficial Velocity	11/25/98	2.00w	12/6/98	0%
Test 18 - Turndown and Ramping	12/6/98	4.00w	1/6/99	0%
Test 19 - Load-Following, Cyclone, & On/Off Tests	1/6/99	7.00w	2/24/99	0%
Test 20 - Reactor Feed: Texaco-Type Syngas	2/24/99	4.00w	3/24/99	0%
Test 21 - Reactor Feed: Distec-Type Syngas	3/24/99	3.00w	4/14/99	0%
Test 22 - Reactor Feed: BGL-Type Syngas	4/14/99	3.00w	5/5/99	10%
Test 23 - Repeat of Test 15 Conditions	5/5/99	3.00w	5/26/99	0%
Test 24 - Reactor Feed: Nat. Gas Reformate-Type Syngas	5/26/99	3.00w	6/16/99	0%
Test 25 - Reactor Feed: Shell-Type Syngas with Steam Injection and 1:1 Recycle	6/16/99	3.00w	7/7/99	0%
Test 26 - Repeat of Test 15 Conditions	7/7/99	3.00w	7/28/99	0%
Test 27 - Repeat of Test 16 Conditions	7/28/99	2.00w	8/11/99	0%
Test 28 - Reactor Operation @ 260 dog C	8/11/99	2.00w	8/25/99	25%
Test 29 - Repeat of Test 28 Conditions	8/25/99	2.00w	9/8/99	0%
Test 30 - Reactor Inspection - Then, Continue with Alternative Catalyst	9/8/99	4.00w	10/6/99	0%
Test 31 - Plant Shutdown	10/6/99	6.00w	11/17/99	0%
Test 32 - Reactor Feed: Texaco-Type Syngas	11/17/99	3.00w	12/6/99	0%
Test 33 - Catalyst Aging	12/6/99	22.00w	5/10/00	0%
Test 34 - Catalyst Addition/Withdrawal to Achieve Target Slurry Concentration	5/10/00	6.00w	6/21/00	0%
Test 35 - Reactor Feed: Texaco-Type Syngas	6/21/00	5.00w	7/26/00	0%
Task 2.1.3 - Extended Optimum Operation	7/26/00	35.00w	3/28/01	0%
Test 36 - Stable Operation	7/26/00	27.00w	1/31/01	0%
Test 37 - Commercial Test Run	1/31/01	8.00w	3/28/01	0%

COPY

U.S. DEPARTMENT OF ENERGY
COST MANAGEMENT REPORT

1. TITLE		2. REPORTING PERIOD		3. IDENTIFICATION NUMBER															
Liquid Phase Methanol Demonstration		March 01, 1998 through March 31, 1998		DE-FC22-92PC90543															
2. PARTICIPANT NAME AND ADDRESS		5. COST PLAN DATE		6. START DATE															
Air Products Liquid Phase Conversion Co., L.P. 7201 Hamilton Boulevard Allentown, PA 18195-4911		October 29, 1997		January 1, 1990															
8. ELEMENT		9. REPORTING ELEMENT		7. COMPLETION DATE															
				December 31, 2001															
		10. ACCRUED COSTS				11. ESTIMATED ACCRUED COSTS				12. Total Plan Value		13. Variance							
		Reporting Period		Cumulative to Date		a. Subsequent Reporting Period		b. Balance of Fiscal Year		c. FY 1999 (1)		FY 2000 (2)		FY 2001 (3)		d. Subsequent FY's (4)		e. Total	
		a. Actual	b. Plan	c. Actual	d. Plan	a. Actual	b. Plan	a. Actual	b. Plan	a. Actual	b. Plan	a. Actual	b. Plan	a. Actual	b. Plan	a. Actual	b. Plan	a. Actual	b. Plan
Prior to Mod 2		0	0	16,304	16,304	0	0	0	0	0	0	0	0	0	0	0	0	0	0
1.1.1 Project Definition		0	0	1,011	1,011	0	0	0	0	0	0	0	0	0	0	0	0	0	0
1.1.2 Permitting		0	0	248	248	0	0	0	0	0	0	0	0	0	0	0	0	0	0
1.1.3 Design Engr.		15	0	10,916	10,916	0	0	0	0	0	0	0	0	0	0	0	0	0	0
1.1.4 Off-site Testing		15	106	164	541	54	646	0	0	0	0	0	0	0	0	0	0	0	0
1.1.5 Planning, Admin, & DMB Verif. Testing		0	4	3,001	3,018	2	27	0	0	0	0	0	0	0	0	0	0	0	0
1.2.1 Procurement		0	0	10,232	10,226	0	0	0	0	0	0	0	0	0	0	0	0	0	0
1.2.2 Construction		0	0	11,730	11,728	0	0	0	0	0	0	0	0	0	0	0	0	0	0
1.2.3 Train. & Commissioning		0	46	585	864	0	279	0	0	0	0	0	0	0	0	0	0	0	0
1.2.4 Off-Site Test - Proc. & Constr.		81	67	117	398	66	612	0	0	0	0	0	0	0	0	0	0	0	0
1.2.5 Planning & Admin		0	2	954	966	2	22	0	0	0	0	0	0	0	0	0	0	0	0
1.3.1 Startup		1	0	1,507	1,497	0	0	0	0	0	0	0	0	0	0	0	0	0	0
1.3.2 Operations		0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
1.3.2.1 Methanol Operation		2,137	2,374	26,249	27,595	3,154	13,217	34,635	36,137	33,489	0	146,822	146,862	0	1,767	1,767	0	0	0
1.3.2.2 DME Design, Mod., Oper.		0	0	0	0	0	0	560	1,207	0	0	0	0	0	0	0	0	0	0
1.3.2.3 LPMEOH Dismantlement		0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
1.3.3 On-Site Product Use Demo		0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
1.3.4 Off-Site Product Use Demo		0	2	0	12	2	22	24	1,595	45	0	1,688	1,688	0	0	0	0	0	0
1.3.5 Data Analysis & Reports		14	3	148	85	4	34	47	49	14	0	296	296	0	0	0	0	0	0
1.3.6 Planning & Admin.		35	81	903	947	81	449	1,149	652	936	0	4,170	4,170	0	0	0	0	0	0
14. TOTAL		2,297	2,685	84,069	86,335	3,365	15,308	36,415	39,645	34,956	0	213,700	213,700	0	0	0	0	0	0
15. DOLLARS EXPRESSED IN:		17. SIGNATURE OF PARTICIPANT'S AUTHORIZED FINANCIAL REPRESENTATIVE AND DATE																	
Thousands		<div style="display: flex; justify-content: space-between;"> <div> SIGNATURE OF PARTICIPANT'S PROJECT MANAGER AND DATE H. C. Hightower 4/13/98 </div> <div> SIGNATURE OF PARTICIPANT'S AUTHORIZED FINANCIAL REPRESENTATIVE AND DATE Susan J. Kennedy 4/13/98 </div> </div>																	