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

TECHNICAL PROGRESS REPORT NO. 15

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and

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for the
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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 - sun 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.

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.

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 (H_2)-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 (Deg C)	Pres. (psig)	Fresh Feed (KSCFH)	Recycle Gas (KSCFH)	Reactor Feed (H2:CO)	Purge Gas (KSCFH)	Inlet Sup. Velocity (ft/sec)	Space Velocity (l/hr-kg)	Slurry Conc. (wt% ox)	Gas Holdup (vol%)	Gassed 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 ft2 F)	Sparger dP (psi)	Sparger Resistance ("K")
6	20-Dec-97	0	Balanced	225	680	772	2,193	4.11	66.1	0.71	8,994	27.3	49.3	59.0	19,500	1.24	44.4	23.3	39.3	236.0	31.53	0.095	171	3.98	3.90
6	21-Dec-97	1	Balanced	233	680	907	2,276	4.16	74.4	0.77	9,688	26.2	44.6	57.5	19,500	1.27	48.2	25.1	39.9	272.8	36.45	0.113	144	4.67	3.84
6	22-Dec-97	2	Balanced	235	680	908	2,250	4.27	71.6	0.77	9,587	29.0	51.6	57.6	19,500	1.34	50.9	25.9	39.5	275.8	36.85	0.114	146	4.67	3.79
6	23-Dec-97	3	Balanced	235	680	920	2,259	4.29	78.0	0.77	9,657	27.7	49.8	59.0	19,500	1.32	50.7	25.7	39.8	277.5	37.08	0.112	162	4.76	3.90
6	24-Dec-97	4	Balanced	235	680	921	2,288	4.02	82.0	0.77	9,558	27.5	49.8	59.5	19,500	1.31	48.6	25.6	39.8	277.7	37.10	0.111	166	5.21	4.31
6	25-Dec-97	5	Balanced	235	680	918	2,255	4.05	72.9	0.77	9,625	28.1	49.8	58.0	19,500	1.29	48.5	25.5	39.4	279.5	37.35	0.115	164	5.27	4.45
6	26-Dec-97	6	Balanced	235	680	929	2,276	4.05	84.4	0.77	9,664	28.9	50.0	56.0	19,500	1.27	48.2	25.4	39.9	279.0	37.28	0.118	163	5.32	4.46
6	27-Dec-97	7	Balanced	235	679	914	2,280	3.79	81.7	0.78	9,714	30.0	50.8	54.0	19,500	1.21	45.4	25.4	39.3	279.3	37.32	0.123	163	5.42	4.44
6	29-Dec-97	9	Balanced	235	680	919	2,199	4.20	94.6	0.76	9,462	30.7	51.9	53.5	19,500	1.23	48.7	24.9	40.8	270.7	36.18	0.120	159	4.93	4.51
6	31-Dec-97	11	Balanced	234	681	733	2,303	5.61	46.8	0.73	9,111	32.1	51.2	49.0	19,500	1.22	54.2	21.7	38.8	226.8	30.35	0.110	154	4.17	4.75
6	1-Jan-98	12	Balanced	235	680	804	2,211	5.34	59.5	0.73	9,069	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	3.99	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	4.66
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	47.4	22.9	39.1	236.8	31.66	0.107	166	5.27	4.73
6	5-Jan-98	16	Balanced	234	680	735	2,223	5.28	53.5	0.72	8,939	29.9	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	54.6	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	26.4	47.7	60.5	19,500	1.27	56.4	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	8,989	26.6	49.7	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	9,005	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	29.86	0.093	166	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	1.06	48.4	22.2	39.3	224.6	30.03	0.093	166	4.87	4.89
6	12-Jan-98	23	Balanced	235	681	725	2,264	4.55	44.5	0.72	8,991	28.0	49.8	58.0	19,500	1.05	46.9	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	49.2	21.4	39.2	223.0	29.81	0.096	167	5.56	5.56
6	15-Jan-98	26	Balanced	235	696	732	2,375	5.06	43.7	0.73	9,388	29.0	49.5	55.0	19,500	1.04	48.7	21.2	39.4	223.1	29.82	0.096	170	5.69	5.30
6	16-Jan-98	27	Balanced	235	700	727	2,360	4.88	45.3	0.73	9,320	29.1	49.2	54.5	19,500	1.01	47.8	21.4	39.0	223.6	29.89	0.098	171	5.63	5.24
6	17-Jan-98	28	Balanced	235	701	731	2,360	4.87	51.9	0.73	9,345	29.5	50.0	54.5	19,500	0.97	46.8	21.1	39.5	221.9	29.67	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	29.5	49.6	54.0	19,500	0.95	46.2	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	5.36
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	218.6	29.22	0.102	167	5.19	5.34
6	22-Jan-98	33	Balanced	234	710	726	2,397	4.62	50.2	0.72	9,387	30.8	49.7	51.0	19,500	0.93	44.4	20.8	39.1	222.6	29.76	0.104	170	5.45	5.29
6	25-Jan-98	36	Balanced	235	710	729	2,382	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	4.94	4.96
6	26-Jan-98	37	Balanced	234	709	734	2,367	4.81	50.1	0.72	9,391	29.6	49.4	53.5	19,500	0.98	46.1	20.9	39.0	226.0	30.20	0.100	164	4.81	4.89
6	27-Jan-98	38	Balanced	235	711	727	2,385	4.99	54.3	0.72	9,450	30.1	48.6	51.5	19,500	0.95	45.8	20.3	39.6	220.2	29.43	0.102	165	4.59	4.79
6	30-Jan-98	41	Balanced	226	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	0.89	43.3	21.0	39.4	221.7	23.38	0.087	155	4.93	4.76
6	1-Feb-98	43	Balanced	225	700	726	2,315	4.35	55.4	0.70	7,171	31.7	48.2	60.0	24,800	0.87	43.0	20.9	39.9	218.7	22.99	0.087	151	4.82	4.76
6	2-Feb-98	44	Balanced	225	700	732	2,271	4.51	65.6	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	23.26	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	0.86	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	49.3	57.0	24,800	0.88	41.7	21.3	39.5	221.0	23.24	0.092	154	4.76	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	199.7	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	7,286	35.1	53.0	57.0	24,800	0.86	12.2	17.3	44.3	196.0	20.77	0.082	139	9.03	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	46.0	24,800	0.85	12.2	16.8	44.0	190.8	20.25	0.099	135	9.57	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

Table D.4-1. Data Summary for LPMEOH™ Demonstration Unit (continued)

6	15-Feb-98	57	Balanced	223	710	736	2,218	5.86	133.6	0.67	7,020	38.5	40.6	39.5	24,800	0.85	49.0	18.7	44.7	197.6	20.80	0.120	159	4.64	6.16
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	210.6	22.15	0.120	149	6.12	5.42
6	17-Feb-98	59	Balanced	224	710	721	2,437	3.49	76.3	0.72	7,519	37.8	45.2	44.0	24,800	0.78	33.7	19.3	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	36.4	19.1	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	0.106	148	5.54	5.42
6	20-Feb-98	62	Balanced	225	711	728	2,344	4.11	53.4	0.70	6,613	37.6	46.3	50.0	27,450	0.79	41.2	20.6	39.3	222.5	21.13	0.106	148	5.67	5.36
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	43.1	20.2	39.4	217.1	20.62	0.107	146	5.32	5.38
6	22-Feb-98	64	Balanced	224	710	710	2,292	4.84	66.5	0.68	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	62.0	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	2,261	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	726	2,256	4.47	61.8	0.68	6,409	38.0	47.3	50.0	27,450	0.79	44.6	20.6	39.7	219.7	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	6,387	36.5	43.4	49.5	27,450	0.80	44.6	20.7	39.8	218.0	20.72	0.105	152	5.12	5.39
6	27-Feb-98	69	Balanced	225	710	734	2,184	4.83	69.1	0.67	6,275	34.9	47.6	57.0	27,450	0.84	48.3	21.3	40.1	219.4	20.85	0.091	148	4.87	5.46
6	28-Feb-98	70	Balanced	225	711	727	2,234	4.59	61.5	0.68	6,372	34.5	48.5	59.0	27,450	0.82	46.4	21.2	39.5	221.3	21.03	0.089	146	5.13	5.39
6	1-Mar-98	71	Balanced	225	710	727	2,223	4.60	59.5	0.67	6,333	33.5	48.8	62.0	27,450	0.82	46.5	21.3	39.6	220.6	20.95	0.084	146	5.02	5.41
6	2-Mar-98	72	Balanced	225	710	721	2,267	4.21	50.7	0.68	6,421	35.1	51.0	60.5	27,450	0.81	43.7	21.6	38.8	223.4	21.22	0.088	146	5.39	5.36
6	3-Mar-98	73	Balanced	225	710	722	2,268	4.21	53.0	0.68	6,427	34.6	48.3	58.5	27,450	0.78	42.8	21.2	39.1	221.4	21.03	0.090	146	5.32	5.46
6	4-Mar-98	74	Balanced	225	710	729	2,234	4.25	56.6	0.67	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	5.26	5.53
6	5-Mar-98	75	Balanced	225	710	728	2,217	4.44	61.0	0.67	6,334	32.3	47.0	63.0	27,450	0.79	44.8	21.4	39.5	221.3	21.02	0.083	145	5.07	5.55
6	6-Mar-98	76	Balanced	225	710	726	2,194	4.53	61.7	0.66	6,262	32.3	46.2	62.0	27,450	0.79	45.4	21.3	39.8	219.0	20.80	0.084	143	4.90	5.58
6	7-Mar-98	77	Balanced	225	710	728	2,212	4.32	61.9	0.67	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	0.66	6,261	33.8	44.4	56.5	27,450	0.80	44.5	21.6	39.9	219.9	20.90	0.093	148	5.05	5.34
6	9-Mar-98	79	Balanced	225	710	728	2,250	4.02	59.2	0.68	6,411	36.4	49.4	55.5	27,450	0.76	41.1	21.2	39.5	221.4	21.03	0.095	150	5.29	5.39
6	10-Mar-98	80	Balanced	225	710	720	2,260	4.37	63.3	0.68	6,401	36.2	50.8	57.5	27,450	0.75	42.6	20.5	40.0	215.9	20.50	0.089	145	5.01	5.46
6	11-Mar-98	81	Balanced	225	709	742	2,257	4.13	73.9	0.68	6,436	35.8	48.9	56.5	27,450	0.75	41.4	20.8	40.6	219.6	20.86	0.092	148	5.26	5.45
6	12-Mar-98	82	Balanced	225	710	756	2,244	3.88	75.9	0.68	6,441	35.9	49.2	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	0.67	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	5.06	5.45
6	14-Mar-98	84	Balanced	225	709	735	2,226	4.11	85.5	0.67	6,343	35.4	46.2	54.5	27,450	0.74	40.8	20.7	40.7	216.9	20.61	0.095	159	5.06	5.48
6	15-Mar-98	85	Balanced	225	709	733	2,319	3.27	67.5	0.70	6,569	35.9	46.4	53.5	27,450	0.70	33.6	20.1	40.6	217.0	20.62	0.097	156	6.16	5.45
6	16-Mar-98	86	Balanced	225	710	715	2,139	4.08	82.9	0.65	6,113	36.0	45.4	52.5	27,450	0.70	40.3	20.4	41.5	206.5	19.62	0.094	158	5.23	6.09
6	17-Mar-98	87	Balanced	225	709	726	2,283	3.91	83.8	0.69	6,474	36.8	45.2	50.5	27,450	0.69	37.4	19.8	41.5	209.7	19.93	0.099	152	5.51	5.53
6	18-Mar-98	88	Balanced	225	711	727	2,271	3.38	82.0	0.68	6,462	37.3	46.7	51.0	27,450	0.67	34.0	20.2	41.1	212.1	20.16	0.099	148	5.90	5.48
6	19-Mar-98	89	Balanced	225	709	733	2,242	3.48	85.5	0.68	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	5.65	5.41
6	20-Mar-98	90	Balanced	225	710	733	2,268	3.24	82.2	0.68	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	0.68	6,447	40.0	47.0	46.0	27,450	0.69	33.6	20.3	41.1	213.2	20.26	0.111	159	5.64	5.37
6	22-Mar-98	92	Balanced	224	710	729	2,247	3.40	85.5	0.68	6,413	40.3	46.5	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	0.67	6,358	36.7	43.9	49.5	27,450	0.68	35.4	19.9	41.9	208.4	19.88	0.100	138	5.27	5.46
6	24-Mar-98	94	Balanced	224	710	715	2,223	3.88	98.5	0.67	6,298	38.1	43.4	46.5	27,450	0.68	36.5	19.6	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	0.66	6,231	38.8	42.4	44.5	27,450	0.67	37.2	19.3	40.9	197.9	18.81	0.106	148	4.96	5.52
6	26-Mar-98	96	Balanced	224	710	685	2,187	4.12	80.5	0.65	6,169	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	5.62
6	27-Mar-98	97	Balanced	224	710	675	2,225	3.56	68.1	0.66	6,243	37.3	41.7	46.5	27,450	0.66	34.3	19.5	40.6	199.6	18.98	0.102	146	5.47	5.60
6	28-Mar-98	98	Balanced	224	710	674	2,111	4.57	90.0	0.63	6,003	37.8	40.2	44.5	27,450	0.68	41.3	19.5	41.9	192.9	18.35	0.103	150	4.47	5.78
6	29-Mar-98	99	Balanced	224	710	682	2,223	3.63	80.6	0.66	6,242	39.2	41.6	43.0	27,450	0.65	34.4	19.3	41.2	198.6	18.89	0.110	150	5.23	5.51
6	30-Mar-98	100	Balanced	224	710	681	2,175	3.96	84.7	0.65	6,153	38.8	41.2	43.5	27,450	0.67	36.9	19.4	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	0.65	6,152	38.4	41.6	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 (eta) - December 1997 - March 1998

The "age" of the methanol synthesis catalyst can be expressed in terms of a dimensionless variable eta (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 η 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 η 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 CO_2 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

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)
Appendix B-5 - Aircraft Ground Equipment Emulsion (one page)
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)

APPENDIX C - PROCESS ECONOMIC STUDY

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

and

LPMEOH™ Process Economics - for IGCC Coproduction
(Memo - 31 March 1997 - two pages)

APPENDIX D - DME DESIGN VERIFICATION TESTING

APPENDIX E - SAMPLES OF DETAILED MATERIAL BALANCE REPORTS

APPENDIX F - RESULTS OF DEMONSTRATION PLANT OPERATION

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

Table 2

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

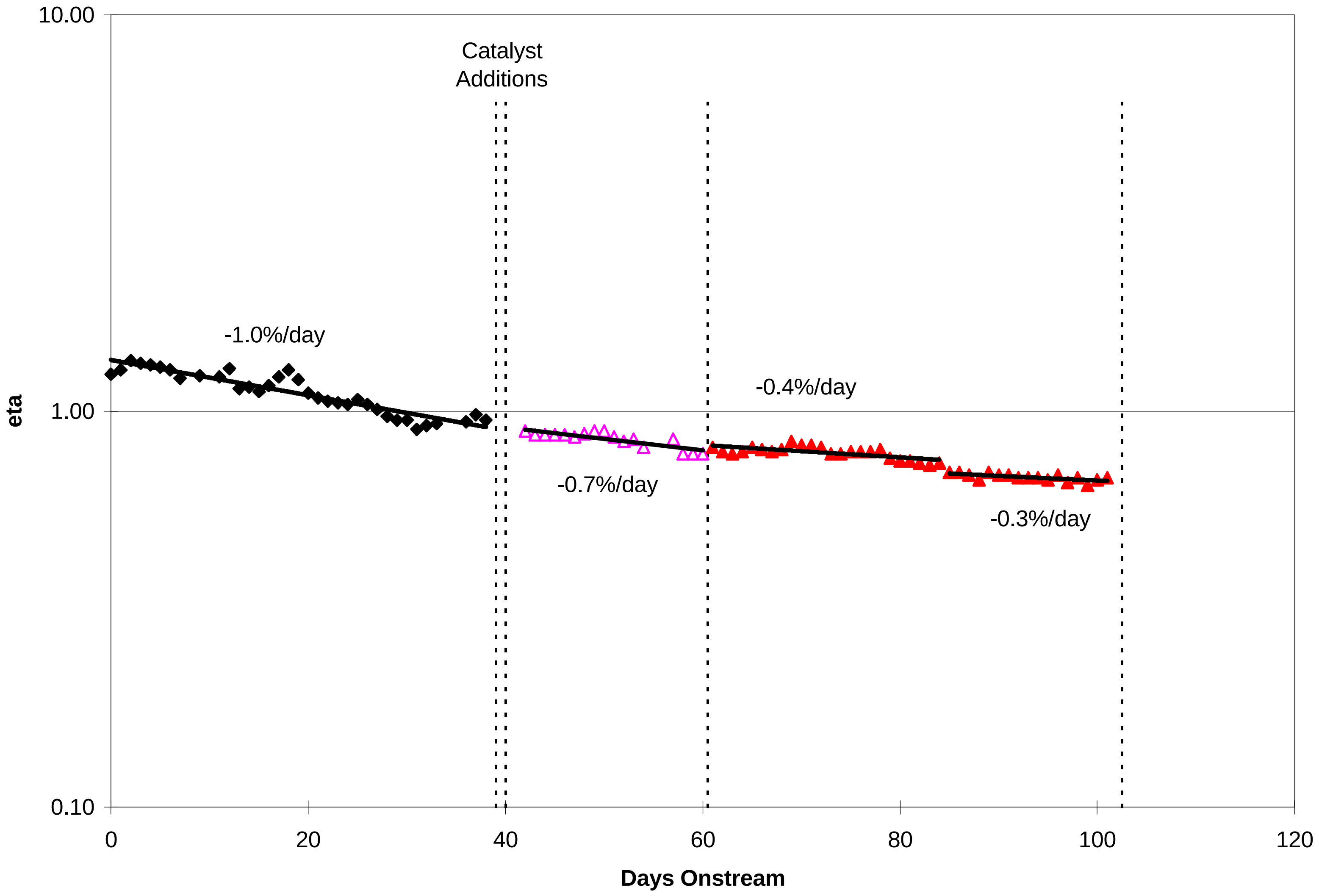


Figure 2 - Sparger Resistance Coefficient vs. Days Onstream (Post-19 December 1997 Restart)

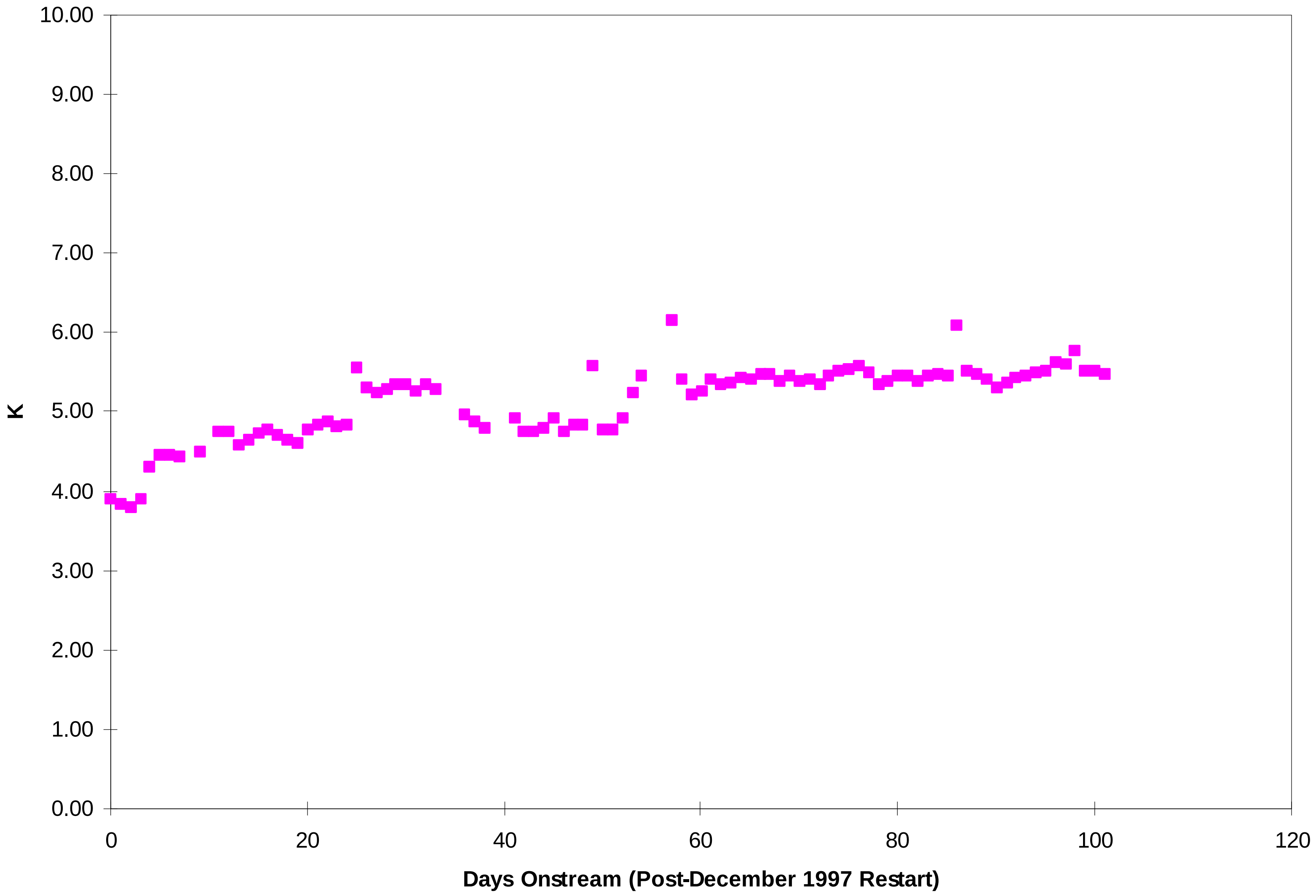
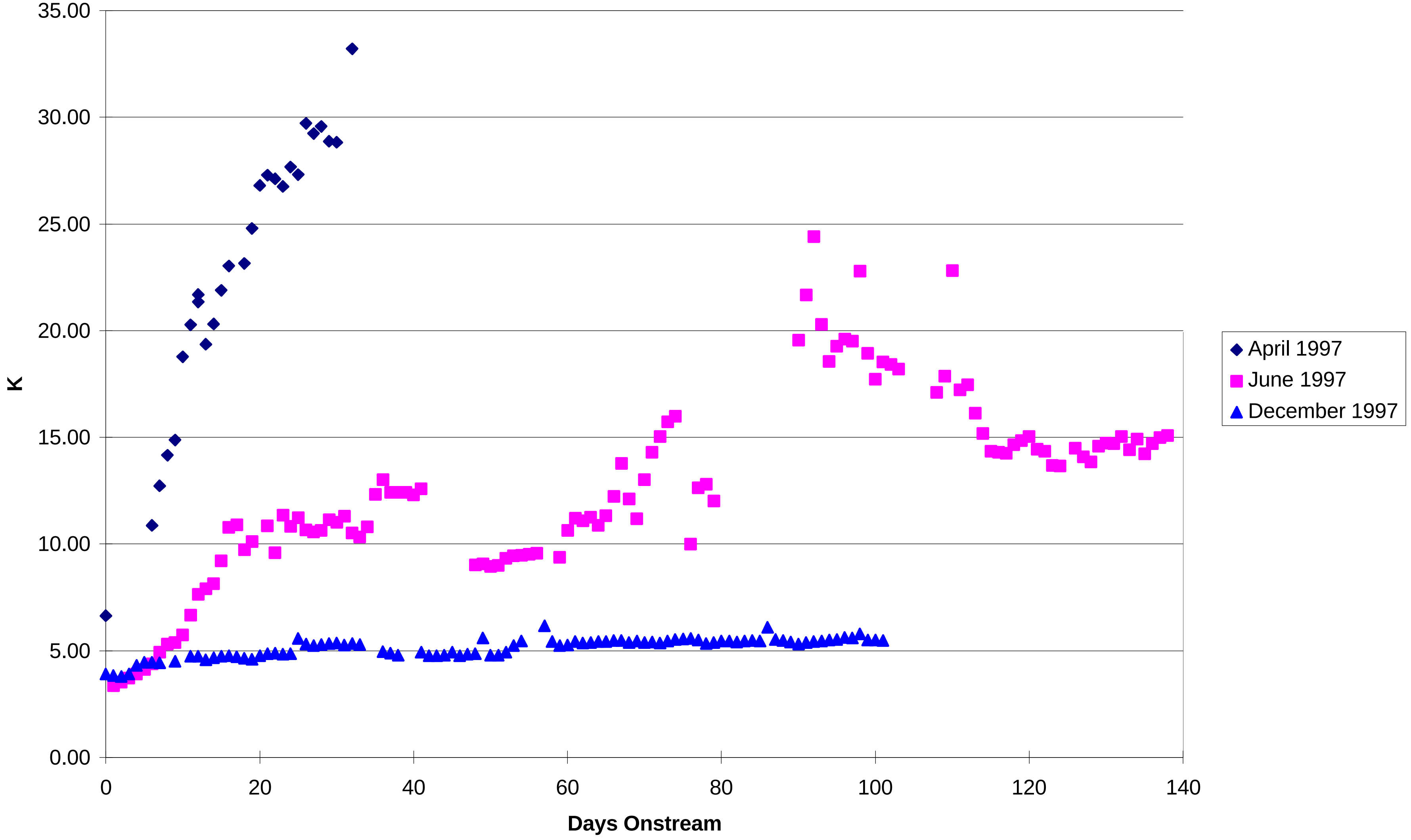


Figure 3 - Sparger Resistance Coefficient vs. Days Onstream (Since April 1997 Startup)



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

APPENDIX H - MILESTONE SCHEDULE STATUS AND COST MANAGEMENT REPORTS