

## Liquid Phase Methanol (LPMEOH™) Project Operational Experience

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### Abstract

The Liquid Phase Methanol (LPMEOH™) process uses a slurry bubble column reactor to convert synthesis gas (syngas), primarily a mixture of carbon monoxide and hydrogen, to methanol. Because of its superior heat management, the process can utilize *directly* the carbon monoxide (CO)-rich syngas characteristic of the gasification of coal, petroleum coke, residual oil, wastes, or other hydrocarbon feedstocks. When added to an integrated gasification combined cycle (IGCC) power plant, the LPMEOH™ process converts a portion of the CO-rich syngas produced by the gasifier to methanol, and the unconverted gas is used to fuel the gas turbine combined-cycle power plant. In addition, the LPMEOH™ process has the flexibility to operate in a daily load-following pattern, coproducing methanol during periods of low electricity demand, and idling during peak times. Coproduction of power and methanol via IGCC and the LPMEOH™ process provides opportunities for energy storage for electrical demand peak shaving, clean fuel for export, and/or chemical methanol sales.

Construction of the LPMEOH™ Process Demonstration Plant was completed in January of 1997 at Eastman Chemical Company's chemicals-from-coal complex in Kingsport, Tennessee. Following commissioning and shakedown activities, the first production of methanol from the 260 tons-per-day (TPD) plant occurred on April 2, 1997. Nameplate capacity was reached for the

first time on April 6, 1997, and production rates of over 300 TPD of methanol were achieved shortly after startup. Throughout 1998, availability for the LPMEOH™ Demonstration Plant has exceeded 99%, and the longest continuous operating campaign reached 65 days.

This paper provides a description of the LPMEOH™ process, the commercial applications for the technology, and a review of the startup and plant performance results at the Kingsport site.

## **Introduction**

The LPMEOH™ technology was developed during the 1980's with the financial support of the U. S. Department of Energy (DOE). The concept was proven in over 7,400 hours of test operation in a DOE-owned, 10 tons-per-day (TPD) Process Development Unit (PDU) located at LaPorte, Texas.<sup>1</sup> The first commercial-scale demonstration plant for the technology was sited at Eastman Chemical Company's (Eastman's) coal gasification facility in Kingsport, Tennessee, with the help of a \$92.7 million award under the DOE's Clean Coal Technology Program. Construction began in October of 1995 and concluded in January of 1997. After commissioning and startup activities were completed, operation began in April of 1997. During a four-year operating program, the LPMEOH™ Process Demonstration Plant will meet or exceed the design production capacity of 260 TPD of methanol, and will simulate operation for the integrated gasification combined cycle (IGCC) coproduction of power and methanol application. The test plan will also seek to establish commercial acceptance of the technology and verify the fitness of the as-produced methanol product (about 98 wt% purity) through a series of off-site, product-use tests. Total cost of the project, including the four-year demonstration test program, is forecast at \$213.7 million.

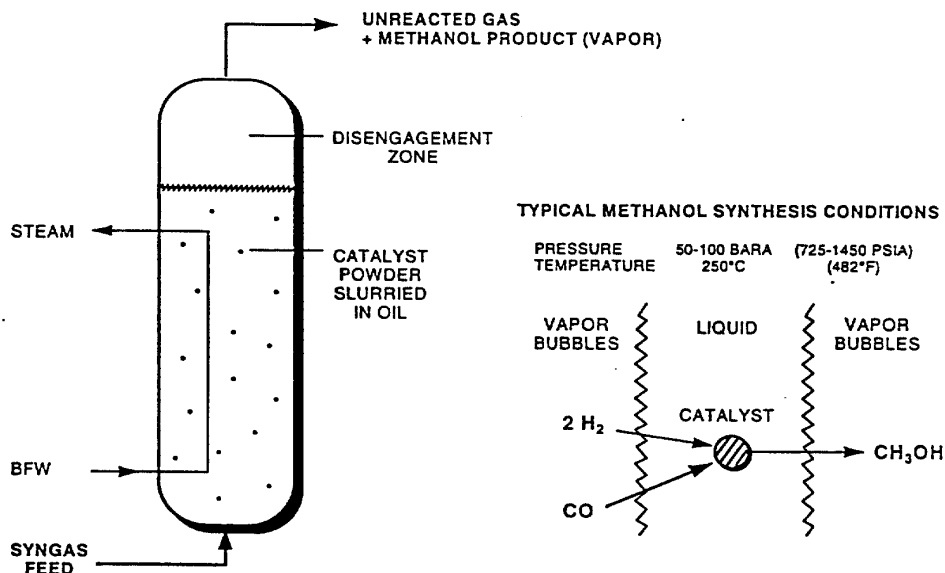
Air Products and Chemicals, Inc. (Air Products) and Eastman formed the "Air Products Liquid Phase Conversion Co., L.P." partnership to execute the project and own the LPMEOH™ Demonstration Plant. Air Products manages the overall project and provides technology analysis and direction for the demonstration. Air Products also provided the design, procurement, and construction of the plant (i.e., a turnkey facility). Eastman provides the host site, acquired the necessary permits, operates the demonstration plant, supplies the supporting auxiliaries and the synthesis gas (syngas), and takes the product methanol. Most of the product methanol is refined to chemical-grade quality (99.85 wt% purity) via distillation and used by Eastman as chemical feedstock elsewhere in their commercial facility. A portion of the product methanol will be withdrawn prior to purification for use in off-site, product-use tests.

This paper reviews: the **Commercial Application** for the LPMEOH™ process technology, and the **Demonstration Plant - Current Performance Results**, reviewing the demonstration test plan and highlighting the operating results achieved to date.

## Commercial Application

### Technology Description

The heart of the LPMEOH<sup>TM</sup> process is the slurry bubble column reactor (Figure 1).



**Figure 1. LPMEOH<sup>TM</sup> Reactor and Reaction Schematics**

Conventional methanol reactors use fixed beds of catalyst pellets and operate in the gas phase. The LPMEOH<sup>TM</sup> reactor uses catalyst in powder form, slurred in an inert mineral oil. The mineral oil acts as a temperature moderator and a heat removal medium, transferring the heat of reaction from the catalyst surface via the liquid slurry to boiling water in an internal tubular heat exchanger. Since the heat transfer coefficient on the slurry side of the exchanger is relatively large, the heat exchanger occupies only a small fraction of the cross-sectional area of the reactor. As a result of this capability to remove heat and maintain a constant, highly uniform temperature through the entire length of the reactor, the slurry reactor can achieve much higher syngas conversion per pass than its gas-phase counterparts.

Furthermore, because of the LPMEOH<sup>TM</sup> reactor's unique temperature control capabilities, it can *directly* process syngas that is rich in carbon oxides (carbon monoxide and carbon dioxide). Gas-phase methanol technology would require that similar feedstocks undergo stoichiometry adjustment by the water gas shift reaction, to increase the hydrogen content, and subsequent carbon dioxide (CO<sub>2</sub>) removal. In a gas-phase reactor, temperature moderation is achieved by recycling large quantities of hydrogen (H<sub>2</sub>)-rich gas. Typically, a gas-phase process is limited to about 16% CO in the reactor inlet, as a means of constraining the conversion per pass to avoid excess heating. In contrast, for the LPMEOH<sup>TM</sup> reactor, CO concentrations in excess of 50% have been tested routinely in the laboratory and at the PDU in LaPorte, without any adverse effect on catalyst activity.

A second distinctive feature of the LPMEOH™ reactor is its robust character. The slurry reactor is suitable for rapid ramping, idling, and even extreme stop/start actions. The thermal moderation provided by the liquid inventory in the reactor acts to buffer sharp transient operations that would not normally be tolerable in a gas-phase methanol synthesis reactor. This characteristic is especially advantageous in the environment of electricity demand load-following in IGCC facilities.

A third differentiating feature of the LPMEOH™ process is that a high quality methanol product is produced directly from syngas rich in carbon oxides. Gas-phase methanol synthesis, which must rely on H<sub>2</sub>-rich syngas, yields a crude methanol product with 4% to 20% water by weight. The product from the LPMEOH™ process, using CO-rich syngas, typically contains only 1% water by weight. As a result, raw methanol coproduced with electric power in an IGCC facility would be suitable for many applications at a substantial savings in purification costs. The steam generated in the LPMEOH™ reactor is suitable for purification of the methanol product to a higher quality or for use in the IGCC power generation cycle.

Another unique feature of the LPMEOH™ process is the ability to withdraw spent catalyst slurry and add fresh catalyst slurry on-line periodically. This capability facilitates uninterrupted operation and also allows perpetuation of high productivity in the reactor. Furthermore, choice of replacement rate permits optimization of reactor productivity versus catalyst replacement cost.

### ***IGCC Coproduction Options***

The LPMEOH™ process is a very effective technology for converting a portion of an IGCC electric power plant's coal-derived syngas to methanol<sup>2</sup>, as depicted in Figure 2. The process has the flexibility to handle wide variations in syngas composition. It can be designed to operate in a continuous, baseload manner, converting syngas from oversized gasifiers or from a spare gasifier. Alternatively, the process can be designed to operate only during periods of off-peak electric power demand, consuming a portion of the excess syngas and reducing the electricity output from the combined-cycle power unit. In this scenario, the gasification unit continues to operate at full baseload capacity, so that the IGCC facility's major capital asset is always fully utilized.

In either baseload or cycling operation, partial conversion of between 20% and 33% of the IGCC plant's syngas is optimal, and conversion of up to 50% is feasible. The required degree of conversion of syngas, or the quantity of methanol relative to the power plant size, determines the design configuration for the LPMEOH™ plant. In its simplest configuration, syngas at maximum available pressure from the IGCC electric power plant passes once-through the LPMEOH™ plant and is partially converted to methanol without recycle, water-gas shift, or CO<sub>2</sub> removal. The unreacted gas is returned to the IGCC power plant's combustion turbines. If greater syngas conversion is required, different plant design options are available.<sup>3</sup>

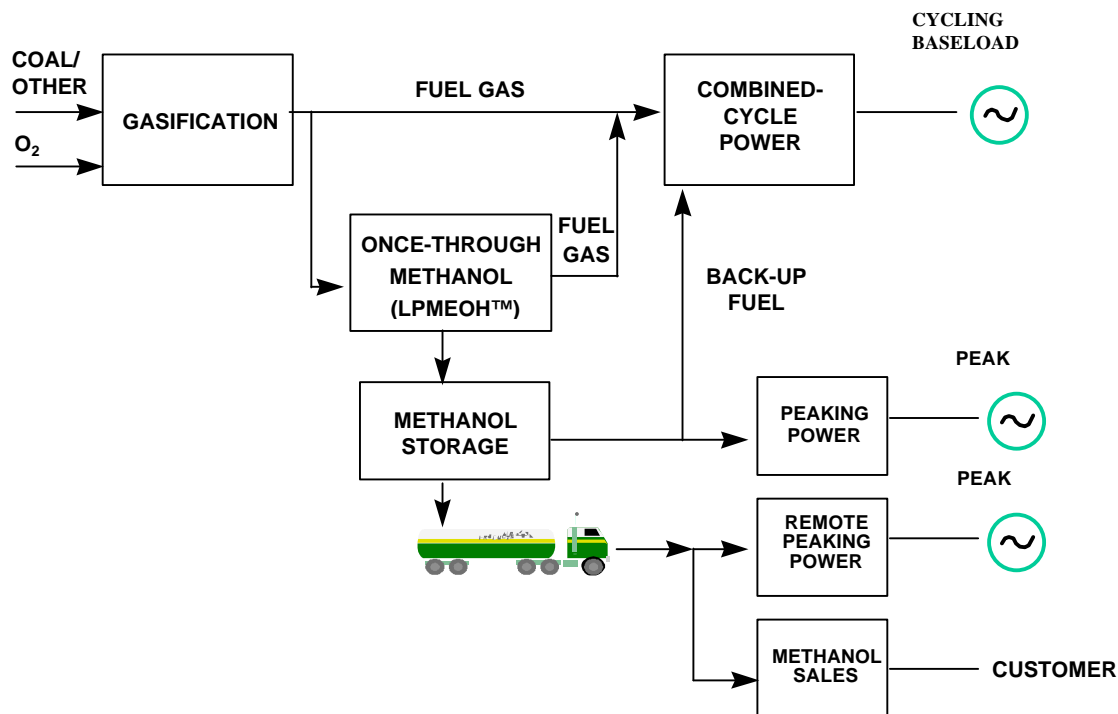


Figure 2.  
Once-through Methanol Coproduction with IGCC Electric Power

### *Economics for Coproduction of Methanol and Power*

Design studies for the LPMEOH™ process have focused principally on the aforementioned IGCC applications. For a given gasification plant size, the IGCC coproduction plant can be designed to accommodate a range of methanol to power output ratios. For example,<sup>4,5</sup> a gasification plant, with two gasifiers of 1735 million Btu (HHV) per hour output each, could be sized for baseload power output of 426 megawatts of electricity (MWe) and for baseload methanol coproduction of 500 TPD. If the baseload fuel gas value is \$4.00 per million Btu, then 500 TPD of methanol can be coproduced from coal for under 50 cents per gallon.<sup>3</sup> Methanol coproduction, by IGCC and the once-through LPMEOH™ process, does not require large methanol plant sizes to achieve good economies of scale. The gasification plant is necessarily at a large economical scale for power generation, so the syngas manufacturing economies are already achieved. Methanol storage and transport economies are also achieved by serving local markets, and realizing freight savings over competing methanol, which is usually shipped from the U. S. Gulf Coast.

The economics of an IGCC coproduction facility can also be improved if a syngas-fueled peaking turbine is installed in parallel with the LPMEOH™ process. Either unit is dispatched based upon the relative, real-time economics of power versus methanol production. This allows the IGCC facility to take maximum advantage of both the off-peak and peak costs of electric power. Economies of scale are improved by increasing the size of the gasification system and optimizing the delivery of syngas to either power or chemicals. The system economics could also be

improved by retrofitting an existing peaking combustion turbine to burn syngas instead of low-sulfur fuel oil; the low dispatch costs of syngas fuel from an IGCC could yield both environmental benefits and the potential to increase the capacity factor of these units when compared to fuel oil.<sup>6</sup>

## **Demonstration Plant - Current Performance Results**

### ***Kingsport Site***

Eastman began coal gasification operations at Kingsport in 1983. Texaco gasification converts about 1,000 tons-per-day of high-sulfur, Eastern bituminous coal to syngas for the manufacture of methanol, acetic anhydride, and associated products. Air Products provides the oxygen for gasification by a pipeline from an over-the-fence air separation unit. The crude syngas is quenched, partially shifted, treated for acid gas removal (hydrogen sulfide, carbonyl sulfide, and CO<sub>2</sub>) via Rectisol, and partially processed in a cryogenic separation unit to produce separate H<sub>2</sub> and CO streams. The H<sub>2</sub> stream is combined with clean syngas to produce stoichiometrically balanced feed for a conventional gas-phase methanol synthesis unit, which is further polished in an arsine- and sulfur-removal guard bed. The methanol product reacts with recovered acetic acid to produce methyl acetate. Finally, the methyl acetate reacts with the pure CO stream to produce the prime product, acetic anhydride (and acetic acid for recycle).

Because the gasification facility produces individual streams of clean balanced syngas (Balanced Gas), CO (CO Gas), and H<sub>2</sub>-rich gas (H<sub>2</sub> Gas), the LPMEOH™ Demonstration Plant design includes the capability to blend these streams into a wide range of syngas compositions. This flexibility enables the plant to simulate the feed gas composition available from any commercial gasifier.

### ***Process Description***

Figure 3 shows a simplified process flow diagram of the LPMEOH™ Demonstration Plant. Approximately half of the Balanced Gas fresh feed to the existing methanol unit is diverted to the LPMEOH™ Demonstration Plant, where it can be combined with the high-purity CO Gas and passes through an activated carbon guard bed. This bed removes iron and nickel carbonyls, which are poisons to methanol synthesis catalyst, down to ppb levels. Since the third feed stream, H<sub>2</sub> Gas, is at lower pressure than the other two feed streams, it can be combined when appropriate with the Recycle Gas stream, made up of unconverted syngas from the LPMEOH™ reactor, and compressed in the recycle compressor.

These streams are then combined to form a single high pressure reactor feed gas stream that is preheated in the feed/product economizer against the reactor effluent. The feed gas is then sparged into the LPMEOH™ reactor (7.5' diameter), where it mixes with the catalyst slurry and is partially converted to methanol vapor, releasing the heat of reaction to the slurry. The slurry temperature is controlled by varying the steam temperature within the heat exchanger tubes, which is accomplished by adjusting the steam pressure.

Disengagement of the effluent gas (methanol vapor and unreacted syngas) from the catalyst/oil slurry occurs in the freeboard region of the reactor. Any entrained slurry droplets leaving the top of the reactor are collected in the cyclone separator. The product gas passes through the tubeside of the feed/product economizer, where it is cooled against the reactor inlet gas stream. Any condensed oil droplets are collected in the high-pressure oil separator and then returned to the reactor with the entrained slurry from the cyclone separator.

The product gas is cooled further in a series of air-cooled and cooling water exchangers, whereupon the product methanol condenses and collects in the high pressure methanol separator. Most of the unreacted syngas returns to the reactor after undergoing compression in the recycle compressor. The balance of the unreacted syngas is purged to the Eastman fuel gas system.

The condensed methanol contains dissolved gases, water, trace oil, and some higher alcohols. These impurities are removed in a two-column distillation train that produces a methyl acetate feed-grade methanol product. The bottom draw from the second column is a crude methanol stream heavy in higher alcohols, water, and any oil carried over from the reactor. This stream is sent to the existing distillation system for recovery of the methanol and disposal of the byproducts. Stabilized, fuel-grade methanol for off-site product-use testing will be produced at limited times during the demonstration period by using only the first distillation column.

Catalyst slurry is activated in the catalyst reduction vessel, which is equipped with a heating/cooling jacket, utility oil skid, and agitator. Pure CO, diluted in nitrogen, acts as the reducing agent. During the activation procedure, slurry temperature is carefully increased while monitoring consumption of CO to determine when the catalyst is completely reduced. At the end of this procedure, the catalyst is fully active and can be pumped directly to the reactor. Prior to adding the fresh catalyst slurry to the LPMEOH<sup>TM</sup> reactor, an equivalent amount of partially deactivated or spent slurry is removed to maintain the total catalyst inventory.

### ***Demonstration Test Plan***

During the design and construction phase of the LPMEOH<sup>TM</sup> Demonstration Plant, a four-year demonstration test plan was developed in order to demonstrate the commercial advantages of the LPMEOH<sup>TM</sup> process. Although generation of electric power is not a feature at the Kingsport site, the demonstration test plan was structured to provide technical data related to the coproduction of electric power with value-added liquid transportation fuels and/or chemical feedstocks from coal. The demonstration test plan will also simulate the energy load-following capability which is required in IGCC applications to allow conversion of off-peak energy, at attendant low value, into peak energy commanding a higher value.

The initial LPMEOH<sup>TM</sup> operating test plan outline, by year, is summarized in Table 1. The demonstration test plan encompasses the range of conditions and operating circumstances anticipated for methanol coproduction with electric power in an IGCC power plant. Since Kingsport does not have a combined-cycle power generation unit, the tests will simulate the

IGCC application. In addition, the test program will emphasize extended periods of steady-state operation.

The ultimate goal of the demonstration period is to reach a stable, optimized operating condition, with the best combination of the most aggressive operating parameters. These parameters, such as reactor superficial gas velocity, slurry concentration, and reactor level, will allow maximum reactor productivity to be achieved. Catalyst life in the environment of coal-derived syngas will also be determined. Confirmation of equipment scale-up will be used to develop the engineering data needed for future commercial designs. Debottlenecking limitations of the demonstration plant will be an ongoing goal during the demonstration period.

Table 1. Initial LPMEOH™ Demonstration Test Plan Outline	
<u>Year 1</u>	Catalyst Aging Catalyst Life vs. LaPorte process development unit and Lab Autoclaves Process Optimization / Maximum Reactor Productivity Catalyst Slurry Concentration (increasing to 40 wt%) Reactor Slurry Level Catalyst Slurry Addition Frequency Test Gas Superficial Velocity Establishment of Baseline Condition
<u>Years 2 &amp; 3</u>	Catalyst Slurry Addition and Withdrawal at Baseline Condition Catalyst Attrition/Poisons/Activity/Aging Tests Simulation of IGCC Coproduction for: <ol style="list-style-type: none"> <li>1. Syngas Composition Studies for Commercial Gasifiers                  Texaco, Shell, Destec, British Gas/Lurgi, Other Gasifiers</li> <li>2. IGCC Electrical Demand Load Following:                  Rapid Ramping, Stop/Start (Hot and Cold Standby).</li> <li>3. Additional Industry User Tests</li> </ol> Maximum Catalyst Slurry Concentration (exceeding 40 wt%) Maximum Throughput/Production Rate
<u>Year 4</u>	Stable, extended Operation at Optimum Conditions 99% Availability Potential Alternative Catalyst Test Additional Industry User Tests



### ***Initial Operation - April/October 1997***

Table 2 summarizes the commissioning and startup milestones at the LPMEOH™ Demonstration Plant during the first year of operation.

Table 2

#### LPMEOH™ Demonstration Plant Milestones - 1997

• Groundbreaking/Construction Begun	October 1995
• Plant Mechanically Complete	January 1997
• Commissioning Begun	February 1997
• Catalyst Activation	March 1997
• Syngas In	April 2, 1997
• Design Production of 260 TPD MeOH	April 6, 1997
• Greater Than 300 TPD MeOH Achieved	April 10, 1997
• Availability in Calendar Year 1997	> 92%

After activation of nine 1-ton batches of methanol synthesis catalyst, the reduced catalyst slurry was pressure-transferred from a maintenance tank to the LPMEOH™ reactor on April 1, 1997. In less than two weeks of operation, the LPMEOH™ Demonstration Plant met several of its short-term performance goals. Methanol production reached the nameplate capacity of 260 TPD, and a stable test period at over 300 TPD of methanol revealed no system limitations, either in the reactor or distillation areas. The rapid progression from first introduction of syngas to stable operation at greater than nameplate capacity is an indication of the robust nature of the LPMEOH™ process. The startup also proceeded without injury or environmental incidents.

As reported in last year's conference,<sup>7</sup> the two high-pressure oil make-up pumps failed to generate the differential pressure required for their service. These pumps were designed in part to provide seal flush to two slurry pumps which return entrained process oil and catalyst, recovered in the cyclone and high-pressure oil separator, to the reactor. During the design phase, in anticipation of operating problems with the slurry return pumps, the elevation of this slurry collection equipment was raised high enough to permit any entrained material to gravity-drain back to the reactor. Because of the pump problems, this test began at startup; initial results were positive, but the connection at the reactor became blocked with catalyst after 3 weeks. An existing piping connection was then used to pressure-transfer this entrained slurry to the catalyst reduction equipment, and a positive displacement pump on the catalyst reduction vessel was used to return this stream to the reactor on a batch basis.

An inspection of the reactor internals in May of 1997 revealed greater than expected accumulation of solids in the bottom head of the reactor. These solids were primarily below the nozzle to which the entrained slurry was returned. Upon restarting in June, the entrained slurry was pumped batch-wise to a new flush connection below the original gravity-drain nozzle. This procedure was

used successfully over the next 5 months of operation. In September of 1997, a less expensive high-pressure oil make-up pump, of alternative design, was installed and tested.

During the initial operating period, the LPMEOH™ Demonstration Plant achieved an availability in excess of 92%. The H<sub>2</sub>/CO ratio in the reactor feed stream was varied from 0.4 to 5.6 with no negative effects on performance. The gas holdup (the volume fraction of the reactor occupied by gas), an important design parameter for slurry reactors, matched the predictions of an in-house correlation developed from pilot plant data. Important parameters such as high inlet superficial velocity of reactor feed gas, maximum gassed slurry level, and the overall heat transfer coefficient of the internal heat exchanger were demonstrated at 115 to 120% of design levels.

### ***Ongoing Performance Results - October 1997 - August 1998***

As noted earlier, the batch return of entrained slurry to the reactor allowed for plant operation to continue through the summer of 1997. However, this procedure needed to be simplified to provide an adequate long-term solution.

In October of 1997, a new test began to gravity-drain the entrained slurry to the new flush connection at the bottom of the reactor. This trial combined the aforementioned benefits of eliminating the slurry pumps with minimizing the accumulation of solids in the bottom of the reactor. Initial results showed that the gravity-drain line became obstructed about twice a day. This blockage, either resulting from solids or from vapor-locking within the piping system, cleared easily with a brief flush of clean process oil from the new high-pressure oil make-up pump. In more recent operation, the frequency of formation of the obstruction within this piping system has decreased to about twice a week. As with the batch flushing, the pressure drop at the bottom of the reactor has shown no increase since the test began, which indicates that the accumulation of solids has been minimized.

The potential benefits of the gravity-draining slurry return to the reactor are significant for future designs, including: significant capital cost savings from the elimination of two slurry pumps and their ancillaries; increased operating flexibility; and, lower maintenance costs by eliminating the seal system for the slurry pumps. The less expensive high-pressure oil make-up pump can be successfully used in this intermittent mode of operation, as demonstrated by its trouble-free performance since its installation in September of 1997.

One of the long-term demonstration issues for the project is to determine the change in catalyst performance with time exposed to the trace poisons present in coal-derived syngas. During the design of the plant, a number of design and analytical steps were taken to characterize the presence of trace contaminants at the parts-per-billion level and to minimize their introduction to or generation within the LPMEOH™ Demonstration Plant.

In addition, a transportable laboratory was shipped to Kingsport in May of 1996 to test the long-term performance of a continuous stirred-tank autoclave on the coal-derived syngas at the Eastman complex. Over the past 20 years, Air Products has developed the skills and analytical

techniques to sample syngas streams and detect concentrations of specific components at the parts-per-billion level. These tests indicated no unusually high levels of known catalyst poisons, and the autoclave produced a typical laboratory catalyst activity curve over a 28-day campaign.<sup>7</sup>

Figure 4 shows performance results from the LPMEOH™ reactor during the first several months of operation at the design reactor temperature of 250°C. The data are reduced to a ratio of rate constant pre-exponential factors (actual vs. design value for fresh catalyst), using an in-house kinetic model, to eliminate the effects of changing feed composition or operating conditions. Typical exponential decay will appear as a straight line on a log-plot, as shown. The curve fit to data from a 4-month test at the LaPorte PDU in 1988/89 is included for reference. The plant results from the initial start-up in April of 1997 showed excellent initial activity, verifying the activation procedure for the catalyst. During the first month of operation, however, an accelerated change in performance occurred; whereas, the remaining operation from June through November matched the typical activity loss measured in the laboratory. This included the performance during the ongoing addition of fresh catalyst batches to the reactor to build inventory and maintain a viable overall level of activity. In fact, the eventual replacement rate of spent catalyst should maintain the average activity in the reactor at about half the fresh value, although that choice is ultimately an economic optimization of catalyst usage rate vs. reactor productivity. Notably, operations at the LaPorte PDU used natural gas feedstock for the generation of the CO-rich syngas fed to the reactor. In this "clean" environment, the methanol catalyst exhibited a very slow loss of activity with time.

An important feature of the LPMEOH™ process is the ability to remove spent catalyst from the reactor during operation; this also affords the opportunity to examine samples for changes in the microscopic structure and/or chemical make-up of the catalyst with time. Analyses of such samples from Kingsport indicated a step-change in the concentration of iron on the catalyst surface during the initial six weeks which cannot be correlated to the presence of iron carbonyl in the feed gas streams. This finding may be related to the detection of post-construction debris within various parts of the facility, or an incipient production of iron carbonyl within the new piping systems, characteristic of a passivation-like mechanism which decreased rapidly with time. Higher than expected levels of arsenic were also found on the catalyst samples. However, a subsequent changeout of an Eastman arsine-removal guard bed, and laboratory tests using arsine-doped syngas, failed to prove that arsine alone was responsible for the catalyst deactivation in the plant. Regardless, the plant originally came on-stream with less than a full charge of catalyst to mitigate the risk of exposure to anomalous contaminants during the initial start-up.

Based on these results, the reactor was drained and another partial charge of fresh catalyst was activated during December of 1997. The calculated catalyst activity curve since the restart is included in Figure 5, along with additional data from the transportable laboratory which was returned to the Kingsport site and operated in parallel on the same reactor feed gas. The initial catalyst performance was excellent, and methanol production again exceeding nameplate capacity at a lower reactor temperature of 235°C. Also, a rapid decrease in activity did not occur during the initial month on-stream, as compared to the results from April of 1997, and the activity maintenance in the LPMEOH™ reactor actually exceeded the results from the parallel laboratory run. As illustrated in Figure 5, several operating periods have shown catalyst performance

meeting or exceeding the results from the 1988/89 campaign at the LaPorte PDU. The variability in the catalyst performance with time is thought to be caused by the presence of trace levels of catalyst poisons (iron, sulfur, arsenic, etc.) in syngas generated from coal. During the eight months of operation, reactor temperature has been varied from 220°C to 250°C; ultimately, a temperature programming protocol will be developed for the LPMEOH™ catalyst system.

For the calendar year to date, availability of the LPMEOH™ Demonstration Unit has exceeded 99%. Catalyst slurry concentration in the LPMEOH™ reactor has reached the design value of 40 wt%, with no instability or loss of performance due to mass transfer limitations. A highlight of this year was the completion of a 65-day continuous operating test ending on 21 April 1998. These stable operating periods provide an indication of the durability of the LPMEOH™ process, in that the performance in the second year of operation is already meeting the targets for mature technologies. Since startup, Eastman has accepted all methanol produced at the LPMEOH™ Demonstration Plant (in excess of 20 million gallons) for use in downstream chemical processes.

### ***Off-Site Product Test Plan***

As part of the LPMEOH™ demonstration project, an off-site test plan involving the use of as-produced methanol in various fuel and energy applications has been implemented. This program focuses on potential of using stabilized methanol produced from CO-rich syngas in the LPMEOH™ process. This as-produced methanol contains about 1 wt% water, which is in contrast to methanol produced from a H<sub>2</sub>-rich syngas (4 to 20% water by weight). If the as-produced methanol can be used directly in downstream fuel or chemical processes, the expensive second distillation step can be avoided, resulting in a savings of 3 to 5 cents per gallon.

Two test runs using a CO-rich feed gas to the LPMEOH™ reactor have been completed. The H<sub>2</sub>/CO ratio of the reactor feed for these cases varied between 0.4 and 0.8. Methanol production matched the predicted quantity for the reactor operating conditions, and the catalyst deactivation rate under CO-rich syngas was equivalent to the H<sub>2</sub>-rich rates before and after. The as-produced methanol composition from two tests simulating feed from a Texaco coal gasifier (H<sub>2</sub>/CO ratio = 0.8) is shown in Table 3. The results from a test campaign at the LaPorte PDU are also presented; this material has already been used successfully in several fuel and chemical applications. The differences in the water and higher alcohol content of the two Kingsport samples is related to the age of the catalyst and the amount of CO<sub>2</sub> in the reactor feed gas.

At present, seven projects have been selected to participate in the off-site product-use test plan. Five projects have completed the planning phase and have begun testing. During the February 1998 campaign on CO-rich syngas, approximately 12,000 gallons of as-produced methanol was collected for future use by these test sites. Three of these tests are studying the operating performance and emissions characteristics of as-produced methanol in bus and automobile applications. A stationary gas turbine has begun operation, and testing in a methanol fuel cell is scheduled to begin in the fall of 1998.

Table 3

## As-Produced Methanol Composition - Texaco-Type Feed Gas

	Kingsport Sample August 1997 (wt%)	Kingsport Sample February 1998 (wt%)	LaPorte PDU Sample (wt%)
Methanol	98.08	99.26	98.40
Ethanol	0.31	0.25	0.60
C3+	0.40	0.18	0.46
Water	1.22	0.31	0.54

Due to maintenance activities within Eastman's facility, an unplanned test of as-produced methanol in a chemical application occurred at the Kingsport site in February of 1998. Approximately 600 tons of as-produced methanol was pumped directly from the LPMEOH™ Demonstration Plant 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 4 wt% water). This is a result of operation on a CO-rich syngas with a relatively low CO<sub>2</sub> concentration. This test was the first attempt to feed an as-produced 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. During this 5-day trial, no operational problems were encountered. Although not a definitive test, this experience may prove valuable as other potential chemical applications for as-produced methanol are identified.

### ***Future Activities***

Over the next 12 months, efforts will continue to sample the catalyst from the reactor and monitor plant performance to quantify the long-term catalyst aging characteristics under coal-derived syngas. Additional studies on the effects of reactor temperature on catalyst life will be performed. The slurry concentration in the reactor will be increased beyond the design value to determine the maximum volumetric productivity of methanol. Additional operations with CO-rich syngas and other reactor feed gas compositions are planned. Operability at each of these conditions will be evaluated.

### **Conclusion**

The LPMEOH™ process is now being demonstrated at commercial scale under the DOE Clean Coal Technology Program. The demonstration plant, located at Eastman Chemical Company's

Kingsport, Tennessee coal gasification facility, has produced in excess of the 260 TPD of methanol nameplate capacity from coal-derived syngas. During calendar year 1997, overall availability exceeded 92%, while the performance in 1998 has achieved greater than 99% availability. The startup and operation to date proceeded without injury or environmental incidents, and Eastman has accepted all methanol produced at the LPMEOH™ Demonstration Plant (over 20 million gallons) for use in downstream chemical processes. A wide variety of syngas compositions and reactor operating conditions have been tested to date, and the 65-day continuous operating campaign in the winter and spring of 1998 provides an indication of the operability of the LPMEOH™ process.

Successful demonstration of the LPMEOH™ technology will add significant flexibility and dispatch benefits to IGCC electric power plants, which traditionally have been viewed as strictly a baseload power generation technology. Now, central clean coal technology processing plants, making coproducts of electricity and methanol, can meet the needs of local communities for dispersed power and transportation fuel. The LPMEOH™ process provides competitive methanol economics at small methanol plant sizes, and a freight and cost advantage in local markets vis-à-vis large offshore remote gas methanol. Methanol coproduction studies show that methanol can be produced at less than 50 cents per gallon from an abundant, non-inflationary local fuel source, such as coal. The coproduced methanol may be an economical hydrogen source for small fuel cells, and an environmentally advantaged fuel for dispersed electric power.

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