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

DEMONSTRATION TECHNOLOGY STARTUP REPORT

Prepared by

Air Products and Chemicals, Inc.
Allentown, Pennsylvania

and

Eastman Chemical Company
Kingsport, Tennessee

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

September 1998

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

DISCLAIMER

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

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

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

Table of Contents

ACRONYMS AND DEFINITIONS

Air Products	-	Air Products and Chemicals, Inc.
AFDU	-	Alternative Fuels Development Unit - The "LaPorte PDU"
Balanced Gas	-	A syngas with a composition of hydrogen (H_2), carbon monoxide (CO), and carbon dioxide (CO_2) in stoichiometric balance for the production of methanol
Carbon Monoxide Gas	-	A syngas containing primarily carbon monoxide (CO); also called CO Gas
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
DCS	-	distributed control system
DME	-	dimethyl ether
DOE	-	United States Department of Energy
Eastman	-	Eastman Chemical Company
Fresh Feed	-	sum of Balanced Gas, H_2 Gas, and CO Gas
gpm	-	gallons per minute
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
K	-	sparger resistance coefficient (term used in calculation of pressure drop)
KSCFH	-	thousand standard cubic feet per hour
LPMEOH™	-	Liquid Phase Methanol (the technology to be demonstrated)
MeOH	-	methanol
MW	-	molecular weight, pound per pound mole
ρ	-	density, pounds per cubic foot
Partnership	-	Air Products Liquid Phase Conversion Company, L.P.
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)
Reactor Feed	-	sun of Fresh Feed and Recycle Gas
Recycle Gas	-	the portion of unreacted syngas effluent from the reactor "recycled" as a feed gas
SCF	-	standard cubic feet
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
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)
TPD	-	(short) ton(s) per day
V	-	volumetric flowrate, thousand standard cubic feet per hour
wt%	-	weight percent

A. Introduction

The Liquid Phase Methanol (LPMEOH™) demonstration project at Kingsport, Tennessee, is a $213.7 million cooperative agreement between the U.S. Department of Energy (DOE) and Air Products Liquid Phase Conversion Company, L. P. (the Partnership). Air Products and Chemicals, Inc. (Air Products) and Eastman Chemical Company (Eastman) formed the Partnership to execute the Demonstration Project. A demonstration unit producing 80,000 gallons per day (260 TPD) of methanol from coal-derived synthesis gas (syngas) was designed, constructed, and is operating at a site located at the Eastman complex in Kingsport. The Partnership will own and operate the facility for the four-year demonstration period.

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

The LPMEOH™ process is the product of a cooperative development effort by Air Products and the DOE in a program that started in 1981. It was successfully piloted at a 10 tons-per-day (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.

The project comprises 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 the practice of supporting process equipment in steel structures.

- *Reaction Area*

The reaction area includes the recycle gas compressor, a catalyst guard bed, the reactor and steam drum, separators, heat exchangers, and pumps. The equipment is supported by a matrix of structural steel. The most salient feature is the reactor, which is approximately 84-feet tall, including supports.

- *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 other columns in the surrounding process areas. This area also includes associated reboilers, condensers, air coolers, separators, and pumps.

- *Catalyst Preparation Area*

The catalyst preparation area consists of a building which houses the catalyst activation vessel, utility oil skid, and slurry handling equipment.

- *Storage/Utility Area*

The storage/utility area includes two diked lot tanks for methanol, an oil storage tank, a slurry holding tank, a trailer loading/unloading area, and an underground oil/water separator. An emergency vent stack and its associated knock-out drum are also 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 solid particles of methanol catalyst suspended in a mineral oil. The syngas dissolves through the oil, contacts the catalyst surface, and reacts to form methanol. The heat of reaction is absorbed by the slurry and removed from the reactor by steam coils. After disengaging from the slurry and exiting the reactor, the methanol vapor is condensed to a liquid, sent to distillation columns for removal of higher alcohols, water, and other impurities, and then stored in lot tanks for sampling before being transferred to Eastman's methanol storage. Most of the unreacted syngas is recycled back to the reactor by the recycle compressor, improving overall efficiency. The methanol is 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

This report focuses on startup activities at the LPMEOH™ Demonstration Unit, from the plant commissioning effort in early 1997 through completion of the first seven months of methanol production. In particular, changes to the original design because of difficulties encountered during commissioning and startup activities are noted.

D.1 Commissioning Activities - January-March 1997

Commissioning activities proceeded in earnest during the period January-March of 1997. The Commissioning and Startup Schedule is included in Appendix B. 100 psig steam and plant nitrogen were introduced to the facility during the last week of January. In addition, the distillation columns, methanol lot tanks, oil/slurry storage tanks, and carbonyl guard bed were chemically cleaned to remove the layer of rust which had formed on vessel and piping surfaces during the construction period.

Pressure testing of the last piping circuit was completed during the first week of February of 1997. This circuit required additional time to replace 54 manual isolation valves which did not meet the pressure test requirements. Once the new valves were installed, the circuit passed the pressure test on the first attempt. The cost of this change totaled $8,440, not including labor.

The 29K-01 recycle compressor was operated on nitrogen (80 psig suction pressure) in early February of 1997. During the nitrogen test, oil from the compressor gearbox migrated into the dry gas seal system, which minimizes leakage of syngas to the atmosphere. This upset was caused by improper location of a vent line from the gear box to a vacuum blower; oil filled this piping, and the vacuum system could not function properly. The compressor seal system was disassembled so that the oil could be cleaned from the system. The vent piping was relocated, and the nitrogen test of the recycle compressor was completed successfully.

All other rotating equipment (e.g. pumps, agitators) was function tested during February of 1997. The commissioning activities on several screw-type pumps (29G-01 condensed oil pumps, 29G-02 slurry return pump, and 29G-03 oil make-up pumps) are covered in Sections D.3 and D.4.

Throughout the commissioning period, Eastman personnel worked on control system functional check-out to ensure that equipment and instrumentation systems responded properly. The distributed control system (DCS) and redundant safety shutdown system were checked. Graphics for the DCS were developed and optimized during this time. Eastman and Air Products personnel worked together to install and configure the data acquisition system, which operates in parallel with the DCS.

Two process analyzers were included in the design to provide syngas compositions for material balance calculations. Minor modifications to the discharge piping system from these gas chromatographs to the Eastman boiler header were required to prevent backflow of condensed liquids into the analyzer columns. In addition, the sample handling system “hot box” was modified to prevent condensation of methanol in the tubing outside the analyzers.

Commissioning activities achieved a major milestone on 28 February 1997 with introduction of syngas for high-pressure leak checking. The recycle compressor ran successfully on syngas at line pressure on 02 March 1997, and reactor heatup for a hot function test with oil and syngas began later that day. This test, also known as “carbonyl burnout”, serves to passivate the synthesis loop against formation of metal carbonyls at operating temperatures and pressures. During the burnout period, Eastman also commissioned the catalyst activation equipment, including the feed gas control valves and flowmeters, the nuclear density gauge, and the utility oil skid. In addition, they commissioned the distillation equipment and test ran the system, first with water and then with methanol. Carbonyl burnout concluded on 12 March 1997.

Activation of nine 2,250 lb catalyst batches required for the initial reactor charge began on 16 March 1997 and concluded on 30 March 1997. As expected, after typical “learning curve”-type problems during the first few batches, the procedure became quite routine. Eventually, the entire operation was compressed into about 36 hours per batch. All batches met or slightly exceeded the “theoretical maximum” syngas uptake of 2.82 SCF per pound of catalyst. Throughout the repetitive activation procedure, the growing charge of reduced catalyst was agitated under a slightly reducing atmosphere in the 29D-02 slurry storage vessel.

Syngas was first introduced to activated catalyst in the LPMEOH™ Reactor on 02 April 1997. The startup was smooth, and the reactor performed well right from the start. After a series of coincidental interruptions in feed gas supply, the plant achieved the nameplate methanol capacity of 260 TPD on 06 April 1997, the first day that full feed rates were available.

D.2 LPMEOH™ Demonstration Unit Performance - April-November 1997

Appendix C contains the summary of performance data for the LPMEOH™ Demonstration Unit throughout the April-November 1997 operating period. These data represent daily averages, typically from a 24-hour material balance period. Those days with less than 12 hours of stable operation are omitted.

Appendix D contains the three summary tables of outages for the LPMEOH™ Demonstration Unit during this same period. These tables also calculate the availability of the LPMEOH™ Demonstration Unit for each operating quarter.

During the first seven months of operation, the LPMEOH™ Demonstration Unit produced a total of 8.95 million gallons of methanol. 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 period.

In the last month of this initial operating period, slurry concentration in the reactor exceeded the 40 wt% design level for the first time, and the LPMEOH™ Reactor operated in a stable hydrodynamic regime at this condition. In addition, the LPMEOH™ Demonstration Unit achieved its longest continuous operating campaign to that point (31 days).

The remainder of Section D deals with specific equipment- and process-related issues which have been encountered since startup of the LPMEOH™ Demonstration Unit, as well as the status as of mid-November of 1997.

D.3 Condensed Oil (29G-01) and Oil Makeup (29G-03) Pumps

During plant commissioning activities, two sets of pumps required additional attention and impacted the mode of operation of the LPMEOH™ Demonstration Unit during startup.

The 29G-01 condensed oil pumps return condensed and entrained oil and catalyst from the 29C-05 secondary oil knock-out drum and 29C-06 cyclone to the 29C-01 LPMEOH™ Reactor. These twin-screw pumps were sent to a factory repair shop to rebuild the seals which had been damaged by exposure to moisture, either from the weather or pressure testing during construction. After returning to site, these pumps ran successfully at 80 psig suction pressure.

The 29G-03 oil make-up pumps provide seal flush to the condensed oil pumps, oil addition to the reactor loop, and high-pressure (1,100 psig) piping flush oil. They also employ a twin-screw design with extremely tight clearances to generate high pressure differential. At the site, these pumps operated well during testing at 700-900 psig discharge pressure in late January of 1997. However, during the subsequent hot function test of the plant, both oil make-up pumps exhibited difficulty in delivering oil at the required pressure. One of the pumps was sent to a factory repair shop, and the seals were found to be damaged by exposure to moisture during construction. After rebuilding the seals, the pump still would not develop more than 600 psig discharge pressure at a dead-head condition. The second pump was sent to the same repair shop, rebuilt, and shipped to the factory in Canada to repeat the bench test which had been performed after initial assembly of the pump. An Eastman representative witnessed this test, and the second pump also failed to develop more than 600 psig discharge pressure. Notably, since the oil make-up pumps could not function at rated conditions to supply the necessary seal flush, the condensed oil pumps could never run at full suction pressure.

During the plant design phase, in anticipation of operating problems with the condensed oil pumps, the elevation of the slurry collection equipment (29C-05 secondary oil knock-out drum and 29C-06 cyclone) was raised high enough to permit any entrained or condensed material to gravity-drain back to the reactor. Because of the inability of the oil make-up pumps to provide seal flush for the condensed oil pumps, this test began at start-up, and initial results were positive. Fresh make-up oil was added to the process by using the 29G-30 slurry transfer pump, which was designed to transfer catalyst slurry from the 29C-30 catalyst reduction vessel to the LPMEOH™ Reactor. The slurry transfer pump packing also requires flush from the oil make-up pumps; however, it was determined that operation of the slurry transfer pump in clean-oil service without packing flush would not adversely affect the service life of the pump. Accordingly, make-up oil was batch-transferred from the 29D-30 oil storage tank to the catalyst reduction vessel, and then pumped to the reactor by the slurry transfer pump.

The gravity-drain line did exhibit intermittent plugging or vapor-locking during operation. Early in the operating campaign, blockages could be cleared by opening a utility line between the secondary oil knock-out drum and the catalyst reduction vessel and briefly blowing down to low pressure; piping connections to provide flush oil were rendered useless by the inoperable high-pressure oil make-up pumps. However, on 25 April 1997, a blockage occurred in the free-drain line that could not be removed by this method. Since the solids concentration of the condensed and entrained oil and catalyst slurry was relatively low, it was determined that the slurry transfer pump could pump this material without packing flush on the pump. Condensed oil was batch-transferred from the secondary oil knock-out drum to the catalyst reduction vessel, and then pumped to the reactor. The frequency of the transfer to the catalyst reduction vessel was about once every three hours, and the catalyst reduction vessel was pumped to the reactor about once every 10 hours. The calculated accumulation rate of condensed and entrained slurry (1.5 to 2.0 gpm) matched the expected liquid traffic within the oil/catalyst collection equipment.

The condensed oil pumps were also intended to supply an oil flush to the walls of the cyclone. At the LaPorte Alternative Fuels Development Unit (AFDU), liquid flush to the cyclone improved the efficiency of solids removal. During a complex-wide shutdown in May/June of 1997, the inlet to the tubesheet of the 29E-02 feed/product heat exchanger (immediately downstream of the cyclone) was removed to check for catalyst accumulation. The tubesheet was generally clean except for a small, off-center accumulation on the upper left quadrant. The catalyst slightly obstructed the entrance to these tubes, but did not completely block any tube. No catalyst was visible within any of the tubes. The surface catalyst was removed, and the exchanger was reassembled.

After an engineering review by Air Products and Eastman, a new 29G-03 oil make-up pump was specified with a simpler design that the original twin-screw configuration. Only one of these new pumps was purchased and installed to develop operational experience without risking too much capital. The new pump, costing $17,400 installed, came online in October of 1997 and met all operational requirements. The availability of this pump to flush out periodic blockages in the gravity-drain line establishes the viability of that concept to return condensed and entrained slurry to the LPMEOH™ Reactor, and as a result, the condensed oil pumps have not yet been put into service under operating conditions. The potential benefits are tremendous for future designs, including: significant capital cost savings from the elimination of two slurry pumps and their ancillaries (and perhaps the redundant oil make-up pump); increased operating flexibility; and, lower maintenance costs by eliminating the seal system for the slurry pumps.

Since the successful test of the gravity-draining slurry return eliminated the need for a continuous seal flush on the condensed oil pumps, the new oil make-up pump currently operates in batch mode only. In this mode it adds oil to the LPMEOH™ Process either to clear any blockages in slurry piping or to provide a regular (once every 2 to 4 days) addition of make-up oil to overcome the average oil loss rate with the methanol product (nominally 0.1 to 0.2 gpm). The new pump has operated well in over six months of operation in this batch mode. Future plans may call for a test of the condensed oil pumps, at which time the operability of the new oil make-up pump in continuous operation will be tested.

D.4 Slurry Return Pump (29G-02)

After activation of the original nine batches of methanol catalyst prior to startup, the entire charge was to be pumped from the 29D-02 slurry storage tank to the reactor using the 29G-02 slurry return pump. This pump included a packing design which required a continuous flush of clean oil from the oil storage tank using the 29G-34 oil feed pump. During the transfer operation, seal oil leaked through the packing at a significantly higher rate than expected. If this condition had continued throughout the transfer, the oil would have diluted the slurry considerably, perhaps to the point of overfilling the reactor vessel. The operations team on site decided to apply nitrogen at 45-50 psig to the slurry storage tank and pressure-transfer the remaining slurry to the reactor. This procedure was accomplished successfully, providing an opportunity for capital savings and improved operability by eliminating this pump from future plant designs.

D.5 Reactor Gas Sparger Resistance Coefficient

Because the performance of a slurry bubble column reactor depends heavily on the method of introduction of feed gas, the gas sparger design is a key feature of the overall LPMEOH™ Reactor design. The gas sparger design for the LPMEOH™ Demonstration Unit was based on the successful 10-TPD proof-of-concept run at the AFDU in LaPorte, TX. In general, the process scale-up from the LaPorte AFDU to commercial scale is a significant objective for the LPMEOH™ demonstration project, and the gas sparger is certainly one of the key components in this area.

Initial results from the LPMEOH™ Demonstration Unit indicated that the scaled-up gas sparger did provide the necessary distribution of syngas at the bottom of the reactor so that actual hydrodynamic behavior matched design expectations. In addition, the heat transfer coefficient of the internal heat exchanger exceed the design value, further indicating good mixing. However, the measured pressure drop across the gas sparger increased considerably throughout the first few weeks onstream. Pressure drop can be expressed by the following equation:

$$\Delta P = \frac{K * (V * MW)^2}{\rho}$$

where:

ΔP = pressure drop across sparger (psi)
K = sparger resistance coefficient (dimensionless)
V = vapor volumetric flowrate (KSCFH)
MW = vapor molecular weight (lb/lb mole)
ρ = vapor density (lb/ft^3)

This equation shows that changes in gas composition or flowrate can influence pressure drop measurements dramatically. Tracking the resistance coefficient (K) over time can indicate any change in the gas flow path through the gas sparger. For a given gas volumetric flowrate and density, an increase in K (caused by a restriction in the flow path, for example), will cause an increase in pressure drop.

Appendix E, Figure 1 show the change in K during the initial operating test of the LPMEOH™ Demonstration Unit in April/May of 1997. Note that K, as reported, contains an arbitrary factor to make the value more manageable, and therefore has meaning only in a relative sense. Pressure drop and resistance increased substantially with time on stream, and extended periods with no gas flow through the sparger (noted as shutdowns on Figure 1) appeared to have no impact on this trend. This observation contradicts the assumption that normal gas flow through the sparger actually prevents plugging by sweeping material out of the openings.

During a scheduled outage at the Eastman chemicals-from-coal complex in May/June of 1997, most of the activities in the LPMEOH™ Demonstration Unit focused on inspection of equipment associated with the reactor, particularly the gas sparger. About 800 pounds of residual catalyst was removed from the bottom head of the reactor during this exercise. A solid material, presumably methanol synthesis catalyst, appeared to block about 50% of the flow path through the sparger, in no particular pattern. A small amount of catalyst was found in the inlet piping to the sparger, but no significant construction debris was found in the inlet piping or in the sparger itself. The sparger was removed from the reactor and cleaned. Structural modifications increased the maximum allowable pressure differential across the sparger, but no changes were made to its flow distribution characteristics.

During the initial operating period in April/May of 1997, the blockage in the free-drain line between the entrained/condensed oil collection system and the reactor (as described in Section D.3) further confirmed that the ability to flush piping systems in slurry service was an important operability requirement. Since a replacement for the 29G-03 oil make-up pumps had not yet been identified, the 29G-30 slurry transfer pump was connected into the flush piping system originally designed to be supplied by the oil make-up pumps. An oil flush connection was also added to the gas inlet line to the reactor, upstream of the sparger. This connection could be used to flush out the piping and gas sparger during normal operation, when gas flow to the reactor is lost, or in preparation for maintenance.

Upon restarting the LPMEOH™ Demonstration Unit in June of 1997, the pressure drop across the gas sparger returned to its design value. However, the gas sparger pressure drop and resistance coefficient did continue to increase with time onstream, although not as rapidly as the April/May operation. The free-drain piping from the slurry collection equipment to the reactor plugged again shortly after restart, but flush oil from the slurry transfer pump successfully dislodged the blockage. On 26 June 1997, nine days after the restart, fresh oil from the slurry transfer pump was introduced for the first time to the new flush connection on the gas inlet line to the reactor. A brief 30-gpm flush decreased the sparger pressure drop from 5.5 psi to 4.5 psi. However, the effects were temporary, and the resistance coefficient continued to increase. Additional flushing with fresh oil is constrained by the average oil make-up requirement of only 0.1 - 0.2 gpm to match the rate of oil loss with the methanol product.

To overcome this constrain, a new flushing method was initiated, using the average internal rate of liquid traffic from the entrained/condensed slurry collection equipment to the LPMEOH™ Reactor (1.5 to 2.0 gpm). Since the condensed oil pumps were not in service, this flushing step was accomplished by shutting off the free-drain line back to the reactor and batch transferring condensed oil form the secondary knock-out drum to the catalyst reduction vessel through a utility connection. From there, the dilute slurry was returned to the reactor through the flush connection in the gas inlet line, 2 to 3 times per day via the 30-gpm slurry transfer pump. Flushing in this manner stabilized the flow resistance through the gas sparger at a manageable level over an extended operating period.

Appendix E, Figure 2 shows the average daily sparger resistance coefficient, K, from the restart of the LPMEOH™ Demonstration Unit after the complex-wide shutdown in June of 1997 until early October of 1997. The plot from the April/May 1997 operation is included for comparison. The resistance coefficient plateaued with the start of periodic flushing with internal oil traffic on Day 17 and remained relatively stable for approximately two months. This period included several interruptions in the flushing regimen, lasting 1 to 3 days, because of shutdowns or periods of on-line catalyst activation which occupied the reduction vessel. After a week-long shutdown in early September (Day 80), however, the sparger resistance exhibited a significant step-change increase. While some of this added resistance proved to be gradually reversible, some of it apparently was not.

On 09 October 1997 (Day 115), a new test began, whereby the condensed oil and entrained slurry gravity-drained continuously to the flush connection on the gas inlet line to the reactor at the normal rate of liquid traffic in the reactor loop (1 to 2 gpm), thus eliminating the batch-transfer steps. The required piping connection had been added during the May/June 1997 complex-wide outage. The gravity-drain line to the flush connection was placed in service after collecting an inventory of condensed oil in the secondary oil knock-out drum. Level in the oil knock-out drum dropped immediately and the temperature at the bottom of the reactor fell slightly, confirming the flowing path of oil to the reactor despite the already high pressure drop through the gas sparger. During subsequent operation, the gravity-drain line became obstructed about twice per day. This blockage, either resulting from accumulation of catalyst or from vapor-locking within the piping system, cleared easily with a brief flush of clean oil from the oil storage tank via the new oil make-up pump. At this frequency of operation, the amount of fresh oil added to the process during line flushing was less than the average oil loss rate with the methanol product (nominally 0.1 - 0.2 gpm).

Operation in this mode continued until the shutdown of the LPMEOH™ Demonstration Unit on 02 November 1997, and the results are plotted in Appendix E, Figure 3. Clearly, the low-flowrate continuous flush provided even more effective than the 30-gpm periodic flush at stabilizing flow resistance through the sparger. Notably, however, some component of the accumulated flow resistance continued to be irreversible.

Another turnaround of the LPMEOH™ Demonstration Unit began on 03 November 1997. After draining the LPMEOH™ Reactor, an attempt to flush and clean the bottom of the vessel with the gas sparger in place proved unsuccessful. The sparger was then removed, inspected, cleaned, and reinstalled. Apart from some residual catalyst, no other solid material was found in the bottom of the LPMEOH™ Reactor, the sparger, or the gas inlet piping.

The initial 7 months of operation of the LPMEOH™ Demonstration Unit provided valuable data on the performance of the gas sparger. Additional flush connections which had not been identified during the plant design phase were added, and initial results on the gravity-draining return of entrained/condensed slurry to the reactor were encouraging. This concept will continue to receive scrutiny during the ongoing execution of the Demonstration Test Plan.

D.6 Methanol Synthesis Catalyst Life

Another significant technical objective of the LPMEOH™ Demonstration project is to determine the long-term aging behavior of the catalyst in a coal-derived syngas environment. Because the proof-of-concept testing at the LaPorte AFDU used a CO-rich syngas produced from clean, natural gas feedstock, this concern was identified as the highest area of technical risk during the design phase.

The initial performance results of the LPMEOH™ Demonstration Unit were excellent. The first stable operation at nameplate methanol capacity of 260 TPD was achieved on 06 April 1997, within four days of the first introduction of syngas. During the first two weeks of operation, several coincidental interruptions in feed gas supply delayed extended, stable operation, so that the first stable 24-hour material balance period occurred on 12 April 1997. The highest methanol production rate over a 24-hour period occurred on 19 April 1997 (292 TPD); for shorter periods (approximately 12 hours), methanol production rates of 302 to 307 TPD were measured.

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 pre-exponential factor at any time to the design value for freshly reduced catalyst, using a proprietary kinetic model to eliminate the effects of changing feed composition or operating conditions. Appendix F, Figure 1 contains the plot for η versus days onstream for the April-November 1997 plant operation. Typical exponential decay will appear as a straight line on a log-plot, as shown.

For reference, the design target from the proof-of-concept testing at the LaPorte AFDU was 0.4% per day activity loss.

During the April/May 1997 operating period, up to Day 32, apparent catalyst activity exhibited a much faster decline than prior experience at the LaPorte AFDU. Performance following the restart in late June, after the sparger inspection and cleaning during Eastman's complex-wide outage, confirmed that this decline was not induced by poor hydrodynamics related to the aforementioned increase in sparger resistance coefficient. At that point, the activity decrease slowed but remained faster than predicted. With some slight variations, the deactivation rate remained relatively constant through early November of 1997 (Day 170). During July (Days 46-73), the first three additional catalyst batches were activated and added on-line to maintain the average catalyst activity within the reactor at just over 50% of fresh (the long-term design value). The increase in reactor performance for each fresh addition step roughly matched model predictions, and each step-change is noted in Appendix F, Figure 1.

A catalyst sample taken just before the restart in June of 1997, after 32 days on-stream, showed levels of arsenic and iron significantly above expectations (446 and 281 ppmw respectively). Averaged over the time on-stream, this arsenic loading equates to 87 ppbv arsine in the feed, assuming complete capture by the catalyst. Furthermore, the copper crystallite size had grown to 274 Angstroms, with a corresponding loss in catalyst surface area that correlates with the activity decline observed in the LPMEOH™ Demonstration Unit. This result focused attention on a guard bed within Eastman's battery limits, which was designed to remove trace arsine and sulfur from the Balanced Gas prior to its introduction into both the Eastman fixed-bed methanol plant and the LPMEOH™ Demonstration Unit.

Additional catalyst samples removed from the LPMEOH™ Reactor during operation in August and early September of 1997 continued to show increasing levels of arsenic, reaching a concentration of 779 ppmw. In addition, sulfur was detected for the first time on the August sample, and its loading increased markedly on the September sample, potentially indicating recent breakthrough of the aforementioned Eastman guard bed. Iron levels on the catalyst showed little or no increase with time after the original sample. No chlorides were detected at any time.

Sampling of the Balanced Gas entering the plant in August of 1997 confirmed the presence of arsine at levels similar to those measured during a plant survey in 1994 (>20 ppbv), prior to installation of Eastman's arsine-removal guard bed. Furthermore, at this arsine loading, the 29C-40 carbonyl guard bed within the LPMEOH™ Demonstration Unit boundary showed little ability to remove arsine, as expected.

During the aforementioned gas sampling, iron carbonyl concentrations were measured at: 13 ppbv in the CO Gas and reactor feed stream; 11-12 ppbv in the plant purge stream; and below the detectable limit of 10 ppbv in the Balanced Gas, carbonyl guard bed inlet, and carbonyl guard bed outlet. Nickel carbonyl was not detected at any time. These carbonyl levels are comparable with those measured during the carbonyl burnout period prior to startup in March of 1997 and within acceptable limits. Such results, coupled with the lack of increase in iron loading on successive catalyst samples, indicate that long-term carbonyl poisoning by iron was not an issue. The initially high iron levels on the catalyst most likely resulted from a one-time startup source, such as construction debris not cleaned out by the carbonyl burnout step.

Core samples taken from the 29C-40 carbonyl guard bed in early September of 1997 showed an iron front about two feet into the bed and no evidence of any nickel loading. In addition, the core samples indicated a significant arsenic gradient over the first four feet of the bed. This may be further evidence of intermittent periods of very high arsine loading, despite the apparent baseline levels of 20-30 ppbv in the Balanced Gas.

During plant operations in August of 1997 (Days 80-106), the catalyst deactivation rate remained relatively constant at about 1.6% per day, as shown in Appendix F, Figure 1. As a result, the average η in the reactor dropped well below its design level of 0.5. After a gasifier-related outage in early September of 1997 (Day 112), the plant could not be restarted because catalyst activity had declined to a point where the reaction would not initiate at the startup steam temperature. Consequently, one additional batch of fresh catalyst was activated and transferred to the reactor to facilitate the restart. A second catalyst batch was added shortly thereafter to further increase the value of η.

Based on the catalyst and gas sampling results, Eastman’s arsine- and sulfur-removal guard bed was changed out on 01-02 October 1997. A total of 75 ft^3 of arsine-removal catalyst and 155 ft^3 of sulfur-removal catalyst were removed from the guard bed and replaced with fresh material. Prior to restarting, an additional batch of fresh catalyst was activated and added to the LPMEOH™ Reactor.

Following the guard bed changeout, Balanced Gas was introduced to the LPMEOH™ Reactor on 03 October 1997 (Day 140). The increase in reactor performance from the addition of the last batch of catalyst roughly matched model predictions, and that last step-change is noted in Appendix F, Figure 1. A catalyst sample from early November showed negligible changes in arsenic or sulfur loading since late September, confirming the effectiveness of the guard bed changeout. All catalyst samples analyzed between April and November of 1997 are summarized in Appendix F, Table 1. During October, however, the catalyst deactivation rate remained essentially unchanged at 1.6% per day. Parallel testing in the laboratory using arsine-doped syngas, and subsequently arsine- and sulfur-doped syngas, also failed to prove that arsine alone or arsine/sulfur combinations were responsible for the catalyst deactivation in the plant.

Other laboratory testing, however, indicated that some sort of “migration” effect was occurring. Fresh catalyst, when mixed 1:1 with “poisoned” catalyst from the LPMEOH™ Demonstration Unit, exhibited accelerated deactivation in a clean syngas environment. This result suggested that even in the absence of additional poisons in the feed gas, the existing level of “poisons” in the reactor was harming fresh catalyst batches added over time. Thus, even the high loading of iron discovered on the catalyst shortly after startup, but not increasing appreciably since then, could have attributed to the unexpected high long-term aging rate of about 1.6% per day.

Based on all of these results, including the recently successful stabilization of the sparger resistance coefficient, DOE accepted a recommendation from Air Products and Eastman to drain the initial charge of catalyst from the reactor and replace it with fresh catalyst. By restarting the Demonstration Test Plan with a fresh charge of catalyst, any "migration" effects of poisons already existing in the reactor would be eliminated, and the full benefits of the guard bed changeout could be determined. Also, a total restart would confirm the viability of the continuous sparger flush, using gravity-drained internal oil traffic, to keep a "clean" sparger unobstructed through extended operation. The cost of a catalyst changeout had been anticipated during the design phase of the project, including procurement of an additional charge of catalyst to supply the LPMEOH™ Demonstration Unit during the first year of operation. However, the original Demonstration Test Plan (issued September 1996) did not call for a catalyst changeout until the third year of operation. Once additional data have been gathered on long-term catalyst aging characteristics, the Test Plan will be updated to reflect changes to the operating program.

Prior to the catalyst turnaround, a final test was performed to determine the impact of raising the operating temperature of the LPMEOH™ Reactor from 250°C to 260°C (Test 28 of the Demonstration Test Plan). As noted in Appendix C, the methanol productivity of the catalyst increased, indicating that the improvement in the rate of reaction was greater than the less favorable chemical equilibrium at the higher temperature. After completion of Test 28, the LPMEOH™ Demonstration Unit was shut down to drain the spent catalyst from the process and begin preparations for activating a new charge of catalyst.

On 11 November 1997, the 29C-40 carbonyl guard bed within the battery limits of the LPMEOH™ Demonstration Unit was recharged with approximately 5,000 pounds of fresh activated carbon. Samples taken in September of 1997 showed a significant arsenic loading at the top of this bed, so the material was replaced outright to avoid the risk of stripping the arsenic off with clean syngas. The cost of this work, covering the replacement charge of activated carbon and labor, totaled $17,900. Also, the inlet screen to the carbonyl guard bed had plugged with debris several time since startup. A new inlet screen was installed to improve accessibility for maintenance; cost for this modification totaled $7,360, including labor and materials.

During the LPMEOH™ Demonstration Unit's first seven months of operation, the study of catalyst life in the coal-derived syngas environment became the major technical challenge. Extensive laboratory testing and state-of-the-art analytical resources joined with plant-scale observations in an attempt to determine the variable(s) contributing to accelerated catalyst deactivation. The remaining elements of the Demonstration Test Plan will continue the study of catalyst aging characteristics in this syngas environment.

E. Conclusion

The LPMEOH™ Demonstration Unit completed a successful initial operating period in early-November of 1997. The first stable operation at nameplate methanol capacity of 260 TPD was achieved on 06 April 1997, within four days of the first introduction of syngas.

The highest methanol production rate over a 24-hour period occurred on 19 April 1997 (292 TPD); for shorter periods (approximately 12 hours), methanol production rates of 302 to 307 TPD were measured.

During the first seven months of operation, the LPMEOH™ Demonstration Unit produced a total of 8.95 million gallons of methanol. Eastman accepted all of this methanol for use in the production of methyl acetate, and ultimately cellulose acetate and acetic acid. In the last month of this initial operating period, the LPMEOH™ Demonstration Unit achieved its longest continuous operating campaign to that point (31 days).

No safety or environmental incidents were reported during this period.

Several problems encountered during plant commissioning and startup have provided an opportunity to improve the operability of the LPMEOH™ Demonstration Unit. In particular, the gravity-draining return of entrained and condensed slurry to the reactor has proven successful from the plant startup. This concept can result in capital savings in future plants as well as improved operability by eliminating the condensed oil pumps and their associated hardware (e.g. valves, instrumentation, seal oil system, etc.). A new oil make-up pump has performed well since its installation in October of 1997. Another pump, designed to transfer slurry from the slurry storage tank to the reactor, can be eliminated by using nitrogen to pressure-transfer the slurry between vessels.

Two major scale-up issues for the LPMEOH™ Demonstration Unit were encountered during this initial operating period. The gas sparger exhibited an increase in pressure drop which had not been observed in testing at the LaPorte AFDU. So far, flushing with entrained and condensed slurry has proven successful in stopping and even partially reversing the increase in resistance coefficient. The long-term aging behavior of the catalyst in the coal-derived syngas environment always was, and will continue to be, a primary focus of the Demonstration Test Plan.

Overall, the cost of changes to the original design during commissioning, startup, and initial operation of the LPMEOH™ Demonstration Project totaled just over $50,000. Impacts of the catalyst changeout in November of 1997 on the execution of the Demonstration Test Plan will be determined once additional data have been gathered on the long-term catalyst aging characteristics.

APPENDICES

APPENDIX A - SIMPLIFIED PROCESS FLOW DIAGRAM

APPENDIX B - COMMISSIONING AND STARTUP SCHEDULE

APPENDIX C - DATA SUMMARY FOR LPMEOH™ DEMONSTRATION UNIT

DATA SUMMARY FOR LPMEOH™ DEMONSTRATION UNIT

Case	Date	Gas Type	Temp (Deg C)	Pres. (psig)	Balanced Gas (KSCFH)	CO Gas (KSCFH)	H2 Gas (KSCFH)	Recycle 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")
1	6-Apr-97																						3.37	6.64
1	12-Apr-97	Balanced	248	719	758	0	0	1,375	0.50	6,203	30.5	50.2	54.9	20,300	1.30	54.8	33.1	37.6	242.3	30.74	0.105	181	6.23	10.87
1	13-Apr-97	Balanced	249	716	792	0	0	1,536	0.55	6,783	30.8	50.6	54.9	20,300	1.24	52.5	31.7	37.5	253.8	32.19	0.110	184	8.69	12.72
1	14-Apr-97	Balanced	249	705	789	0	0	1,619	0.58	7,014	30.9	52.0	56.1	20,300	1.18	50.5	30.5	37.5	252.4	32.00	0.107	172	10.50	14.17
1	15-Apr-97	Balanced	249	705	818	0	0	1,601	0.58	7,019	30.8	50.9	55.1	20,300	1.29	51.7	31.2	37.9	258.9	32.84	0.112	170	11.05	14.88
1	16-Apr-97	Balanced	248	705	904	0	0	1,527	0.59	7,094	30.9	50.7	54.5	20,300	1.78	55.8	33.6	38.5	281.9	35.82	0.123	168	14.17	18.78
1	17-Apr-97	Balanced	249	704	887	0	0	1,746	0.63	7,629	32.1	53.0	54.3	20,300	1.24	49.8	30.0	39.4	270.5	34.35	0.119	171	17.61	20.28
1	18-Apr-97	Balanced	249	705	841	0	0	1,843	0.64	7,762	32.0	51.9	53.4	20,300	1.14	48.2	29.1	37.8	267.0	33.86	0.119	173	19.17	21.35
1	18-Apr-97	Balanced	249	703	964	0	0	1,779	0.66	7,970	32.0	52.2	53.8	20,300	1.43	51.1	30.7	40.0	288.8	36.80	0.128	153	20.47	21.69
1	19-Apr-97	Balanced	249	709	958	0	0	1,994	0.70	8,562	32.2	51.5	52.4	20,300	1.17	48.3	29.2	39.4	292.2	36.60	0.133	158	21.59	19.36
1	20-Apr-97	Balanced	249	708	913	0	0	2,114	0.72	8,771	30.3	50.5	55.8	20,300	1.03	47.1	26.4	39.8	275.5	34.77	0.117	172	21.64	20.31
1	21-Apr-97	Balanced	249	709	859	0	0	2,108	0.71	8,635	30.2	47.5	53.0	20,300	0.94	47.5	25.6	39.5	261.1	32.83	0.117	176	22.29	21.90
1	22-Apr-97	Balanced	249	709	793	0	0	2,086	0.69	8,376	29.5	45.2	52.5	20,300	0.91	49.0	24.6	39.2	242.7	30.65	0.110	175	21.59	23.04
1	24-Apr-97	Balanced	249	702	865	0	0	2,028	0.70	8,419	29.9	43.1	49.5	20,300	0.93	44.6	26.2	39.8	261.0	33.17	0.126	186	24.25	23.16
1	25-Apr-97	Balanced	248	700	835	0	0	1,921	0.67	8,019	31.8	44.9	47.0	20,300	0.89	44.6	26.2	40.4	248.2	31.58	0.126	164	23.48	24.80
1	26-Apr-97	Balanced	246	694	864	0	0	1,878	0.67	7,980	32.8	45.6	45.5	20,300	0.83	41.6	26.0	41.9	247.8	31.38	0.130	147	25.38	26.81
1	27-Apr-97	Balanced	247	690	902	0	0	1,793	0.66	7,842	32.8	44.9	45.0	20,300	0.83	39.7	26.9	42.9	252.2	31.87	0.134	154	26.18	27.29
1	28-Apr-97	Balanced	247	696	783	0	0	1,903	0.65	7,817	30.9	43.3	47.5	20,300	0.75	42.7	24.4	41.9	224.4	28.35	0.113	157	24.49	27.11
1	29-Apr-97	Balanced	249	700	810	0	0	1,922	0.66	7,951	29.3	45.2	53.0	20,300	0.81	42.1	25.6	40.9	238.0	30.05	0.107	161	26.26	26.76
1	30-Apr-97	Balanced	249	699	794	0	0	1,912	0.66	7,872	29.1	43.6	52.0	20,300	0.79	43.0	25.1	41.3	230.8	29.24	0.106	163	26.13	27.67
1	1-May-97	Balanced	249	699	798	0	0	1,932	0.66	7,945	29.9	44.0	50.5	20,300	0.74	41.1	24.5	42.0	228.0	28.85	0.108	162	26.26	27.32
1	2-May-97	Balanced	249	700	757	0	0	1,898	0.64	7,728	29.7	43.0	50.0	20,300	0.71	42.8	23.7	42.2	215.4	27.32	0.103	162	25.91	29.72
1	3-May-97	Balanced	249	699	782	0	0	1,886	0.65	7,767	30.4	43.1	48.5	20,300	0.68	40.5	23.9	42.8	219.5	27.66	0.108	162	26.05	29.24
1	4-May-97	Balanced	249	700	789	0	0	1,901	0.65	7,831	30.5	44.0	49.0	20,300	0.64	38.9	23.4	43.4	218.1	27.43	0.106	156	26.24	29.58
1	5-May-97	Balanced	249	699	798	0	0	1,901	0.66	7,856	30.1	43.6	49.5	20,300	0.63	37.5	23.4	43.7	218.9	27.76	0.105	157	26.26	28.88
1	6-May-97	Balanced	249	700	776	0	0	1,923	0.65	7,835	30.4	42.5	48.5	20,300	0.65	38.1	23.3	42.8	217.3	27.85	0.108	161	26.26	28.83
1	8-May-97	CO-rich	249	700	215	40	0	1,051	0.32	3,813	30.8	36.9	43.0	20,300	0.64	8.6	15.1	44.4	69.0	8.83	0.038	128	14.81	33.22
5	18-Jun-97	Balanced	248	724	718	0	0	1,938	0.62	8062	25.6	45.9	61.4	19,500	0.61	39.3	22.5	42.0	205.3	26.95	0.079	161	2.99	3.37
5	19-Jun-97	Balanced	249	711	638	0	0	1,938	0.62	7905	26.6	45.3	57.8	19,500	0.63	42.6	21.4	40.7	188.3	24.77	0.077	175	3.05	3.52
5	20-Jun-97	Balanced	249	707	651	0	1	2,079	0.66	8294	27.1	44.9	56.0	19,500	0.59	39.2	20.2	41.3	189.4	24.99	0.080	171	3.45	3.71
5	21-Jun-97	Balanced	249	707	687	0	0	2,109	0.67	8465	27.6	45.2	55.0	19,500	0.58	35.5	20.8	41.4	198.9	26.14	0.086	170	4.00	3.90
5	22-Jun-97	Balanced	249	707	625	0	0	2,097	0.65	8203	28.2	44.2	52.7	19,500	0.50	33.4	19.5	41.3	181.5	23.85	0.082	168	3.96	4.11
5	23-Jun-97	Balanced	249	707	762	0	0	2,021	0.67	8456	28.4	43.6	51.6	19,500	0.57	33.6	21.5	43.8	208.8	27.35	0.096	171	4.51	4.38
5	24-Jun-97	Balanced	249	708	781	0	0	1,991	0.67	8412	29.0	44.5	50.9	19,500	0.56	33.2	21.2	45.6	205.4	26.90	0.096	154	5.04	4.93
5	25-Jun-97	Balanced	248	707	739	0	0	2,003	0.66	8338	29.2	43.3	49.4	19,500	0.57	33.7	21.0	44.0	201.5	26.62	0.097	160	5.33	5.31
5	26-Jun-97	Balanced	249	707	737	0	0	2,080	0.68	8559	28.9	48.3	54.8	19,500	0.55	33.0	20.7	43.4	203.5	26.65	0.088	163	5.64	5.38
5	27-Jun-97	Balanced	249	706	736	0	0	2,326	0.74	9252	26.7	46.6	59.0	19,500	0.53	29.7	19.5	42.8	206.5	26.68	0.083	161	7.34	5.74
5	28-Jun-97	Balanced	249	707	691	0	0	2,307	0.72	9079	27.5	45.8	56.0	19,500	0.53	31.0	19.3	41.5	199.6	25.84	0.085	168	8.12	6.67
5	29-Jun-97	Balanced	249	706	719	0	0	2,267	0.72	9042	27.7	43.9	53.5	19,500	0.53	30.0	19.5	42.6	202.3	26.24	0.090	170	9.33	7.65
5	30-Jun-97	Balanced	249	706	711	0	0	2,263	0.72	9019	28.1	43.9	52.5	19,500	0.51	29.9	19.1	43.0	198.4	25.77	0.090	168	9.29	7.90
5	1-Jul-97	Balanced	249	707	676	0	0	2,251	0.71	8944	26.1	45.3	59.4	19,500	0.52	31.5	19.0	42.0	193.2	24.68	0.077	163	9.47	8.15
5	2-Jul-97	Balanced	249	707	685	0	0	2,203	0.70	8827	26.5	45.4	58.4	19,500	0.54	32.5	19.6	41.9	195.9	25.39	0.080	166	10.66	9.21
5	3-Jul-97	Balanced	249	707	664	0	0	2,218	0.70	8794	27.5	43.9	54.0	19,500	0.54	32.8	19.4	41.1	193.9	25.48	0.085	168	12.10	10.78
6	4-Jul-97	Balanced	249	707	705	0	0	2,231	0.70	7939	30.0	44.1	54.0	21,800	0.56	37.3	21.0	40.6	208.4	23.96	0.092	162	12.21	10.89
6	5-Jul-97	Balanced	249	706	761	0	0	2,203	0.72	8060	30.7	43.1	51.5	21,800	0.56	35.8	21.1	42.3	216.0	25.11	0.100	159	11.09	9.73
6	6-Jul-97	Balanced	249	705	755	0	0	2,198	0.71	8031	30.2	42.5	52.0	21,800	0.55	36.2	20.9	42.6	212.8	24.85	0.097	163	11.49	10.12
6	8-Jul-97	Balanced	249	706	761	0	0	2,161	0.70	7926	31.1	41.9	49.5	21,800	0.53	35.4	20.5	44.2	206.5	24.25	0.099	161	11.94	10.85
6	9-Jul-97	Balanced	248	695	610	0	0	2,277	0.70	7788	31.3	42.0	49.0	21,800	0.50	34.3	19.0	40.2	182.1	21.28	0.089	166	11.02	9.60
6	10-Jul-97	Balanced	249	694	632	0	0	2,235	0.69	7705	29.9	42.3	52.5	21,800	0.47	33.9	18.5	41.9	181.1	21.09	0.082	161	12.05	11.35
6	11-Jul-97	Balanced	249	695	748	0	0	2,118	0.70	7013	32.0	42.4	53.0	24,100	0.53	39.8	21.0	43.4	206.6	21.54	0.093	157	11.34	10.83

DATA SUMMARY FOR LPMEOH™ DEMONSTRATION UNIT

Case	Date	Gas Type	Temp (Deg C)	Pres. (psig)	Balanced Gas (KSCFH)	CO Gas (KSCFH)	H2 Gas (KSCFH)	Recycle 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	12-Jul-97	Balanced	249	699	805	0	0	2,063	0.70	7036	31.5	41.8	53.5	24,100	0.52	40.0	21.0	46.0	209.8	22.17	0.093	157	11.30	11.24
6	13-Jul-97	Balanced	249	701	800	0	0	2,125	0.71	7177	32.0	41.4	52.0	24,100	0.54	37.7	21.8	43.4	221.2	23.42	0.101	157	12.00	10.65
6	14-Jul-97	Balanced	249	702	801	0	0	2,139	0.71	7209	32.6	41.3	50.5	24,100	0.53	36.8	21.5	43.8	219.6	23.24	0.104	151	11.89	10.57
6	15-Jul-97	Balanced	249	703	781	0	0	2,170	0.71	7228	32.3	42.1	52.0	24,100	0.52	36.1	21.4	42.8	218.9	23.14	0.100	155	12.27	10.63
6	16-Jul-97	Balanced	249	704	788	0	0	2,154	0.71	7217	32.3	41.6	51.5	24,100	0.51	35.9	21.4	43.2	219.1	23.07	0.101	153	12.53	11.13
6	17-Jul-97	Balanced	249	703	763	0	0	2,189	0.71	7231	32.5	41.6	51.0	24,100	0.50	35.0	21.1	42.5	215.6	22.71	0.101	152	12.80	11.01
6	18-Jul-97	Balanced	249	702	770	0	0	2,124	0.70	7102	33.2	40.3	48.5	24,100	0.49	36.1	20.5	44.6	207.3	21.92	0.102	152	11.99	11.30
6	19-Jul-97	Balanced	249	702	767	0	0	2,173	0.71	7213	33.4	40.2	48.0	24,100	0.48	34.1	20.5	43.8	210.2	22.25	0.104	153	12.05	10.52
6	20-Jul-97	Balanced	249	702	768	0	0	2,187	0.71	7240	33.2	40.9	49.0	24,100	0.48	32.7	20.6	43.5	212.0	22.50	0.103	152	12.26	10.33
6	21-Jul-97	Balanced	249	702	746	0	0	2,137	0.70	7067	31.6	42.1	53.5	24,100	0.48	35.4	20.8	42.8	209.1	22.15	0.093	160	11.61	10.81
6	22-Jul-97	Balanced	249	702	755	0	0	2,160	0.70	7083	31.1	40.6	53.5	24,100	0.46	34.0	20.5	43.9	206.6	21.67	0.092	159	13.32	12.33
6	23-Jul-97	Balanced	250	702	706	0	0	2,197	0.70	7058	31.1	42.5	55.0	24,100	0.46	32.8	20.4	41.6	203.5	21.30	0.088	152	14.71	13.01
6	24-Jul-97	Balanced	249	702	801	0	0	2,070	0.70	6426	33.4	40.8	53.0	26,400	0.51	39.0	22.3	43.3	222.0	21.42	0.100	156	13.17	12.43
6	25-Jul-97	Balanced	249	700	796	0	0	2,090	0.70	6460	33.7	39.8	51.5	26,400	0.51	37.3	22.4	42.6	224.3	21.64	0.104	153	13.80	12.41
6	26-Jul-97	Balanced	249	700	781	0	0	2,062	0.69	6361	33.6	39.6	51.5	26,400	0.52	38.1	22.7	41.9	223.9	21.62	0.103	152	13.32	12.43
6	27-Jul-97	Balanced	249	700	748	0	0	2,082	0.69	6334	33.6	39.4	51.5	26,400	0.50	37.9	22.1	41.6	215.7	20.58	0.100	146	13.02	12.31
6	28-Jul-97	Balanced	249	700	790	0	0	2,048	0.69	6354	33.1	39.5	52.5	26,400	0.52	38.3	22.8	42.1	225.3	21.71	0.102	154	13.45	12.58
3	4-Aug-97	Texaco	249	702	627	95	0	2,233	0.71	6600	33.5	47.5	59.5	26,400	0.56	12.1	18.1	45.0	192.4	18.11	0.077	139	17.86	9.01
3	5-Aug-97	Texaco	249	702	626	95	0	2,238	0.71	6600	33.7	46.2	57.7	26,400	0.57	12.3	18.2	45.1	192.0	18.25	0.079	140	18.10	9.08
3	6-Aug-97	Texaco	249	701	636	95	0	2,182	0.70	6507	33.8	45.4	56.5	26,400	0.56	12.8	18.5	45.5	193.0	18.56	0.081	139	17.07	8.95
3	7-Aug-97	Texaco	249	701	648	95	0	2,148	0.70	6459	34.4	45.4	55.0	26,400	0.54	13.3	18.8	45.8	194.8	18.75	0.084	138	16.52	9.00
3	8-Aug-97	Texaco	249	702	629	95	0	2,129	0.69	6376	33.4	45.1	57.0	26,400	0.55	13.0	18.6	45.7	190.2	18.36	0.079	140	17.05	9.33
3	9-Aug-97	Texaco	249	703	627	95	0	2,130	0.69	6383	33.4	45.4	57.5	26,400	0.53	12.8	18.4	46.1	187.9	18.08	0.078	138	17.30	9.46
3	10-Aug-97	Texaco	249	702	624	95	0	2,118	0.69	6364	33.8	45.9	57.0	26,400	0.52	13.2	18.6	46.0	187.7	17.98	0.078	136	17.25	9.47
3	11-Aug-97	Texaco	249	702	622	95	0	2,101	0.68	6320	34.9	45.9	54.5	26,400	0.50	13.0	18.3	46.3	186.0	17.89	0.081	137	16.94	9.52
3	12-Aug-97	Texaco	249	702	616	95	0	2,088	0.68	6284	34.0	42.4	53.0	26,400	0.50	12.9	18.2	46.0	185.6	17.95	0.083	139	16.71	9.56
6	15-Aug-97	Balanced	249	700	642	0	0	2,303	0.72	6602	34.0	39.4	50.5	26,400	0.37	31.9	18.1	41.5	185.5	17.82	0.087	151	10.37	9.38
6	16-Aug-97	Balanced	249	700	701	0	0	2,203	0.70	6491	34.1	39.0	50.0	26,400	0.37	31.4	18.6	44.1	190.8	18.46	0.091	151	11.05	10.64
6	17-Aug-97	Balanced	249	700	693	0	0	2,227	0.71	6537	34.2	40.4	51.0	26,400	0.37	29.7	18.9	42.6	195.5	18.78	0.091	150	12.19	11.20
6	18-Aug-97	Balanced	249	700	633	0	0	2,278	0.71	6508	34.7	41.1	50.5	26,400	0.36	29.4	18.5	40.4	188.0	18.03	0.089	152	12.29	11.09
6	19-Aug-97	Balanced	249	698	632	0	0	2,244	0.70	6454	34.8	40.3	49.5	26,400	0.34	28.7	17.9	41.8	181.6	17.56	0.087	151	12.04	11.26
6	20-Aug-97	Balanced	249	700	636	0	0	2,296	0.71	6554	35.3	43.2	51.0	26,400	0.34	27.7	18.2	41.0	186.3	17.89	0.087	157	12.40	10.87
6	21-Aug-97	Balanced	249	700	636	0	0	2,224	0.69	6383	36.4	41.7	47.5	26,400	0.34	29.5	17.7	42.8	178.3	17.20	0.089	154	11.34	11.33
6	22-Aug-97	Balanced	249	701	666	0	0	2,283	0.71	6567	35.7	45.8	52.5	26,400	0.32	26.7	17.8	43.6	183.1	17.36	0.083	148	13.77	12.23
6	23-Aug-97	Balanced	249	702	652	0	0	2,205	0.69	6368	35.5	41.2	49.0	26,400	0.31	28.1	17.2	45.1	173.8	16.59	0.084	148	13.49	13.78
6	24-Aug-97	Balanced	249	702	653	0	0	2,204	0.69	6381	35.8	40.1	47.5	26,400	0.30	27.4	16.8	45.7	171.3	16.36	0.086	146	11.79	12.11
6	25-Aug-97	Balanced	249	702	623	0	0	2,287	0.70	6469	36.3	40.7	47.0	26,400	0.30	25.3	17.0	42.7	175.0	16.65	0.089	147	12.09	11.19
6	26-Aug-97	Balanced	249	702	629	0	0	2,176	0.68	6275	35.1	39.0	48.0	26,400	0.29	27.6	16.8	45.0	167.7	16.03	0.083	148	12.21	13.01
6	27-Aug-97	Balanced	249	702	625	0	0	2,187	0.68	6291	35.7	39.3	47.0	26,400	0.29	26.4	16.7	44.9	167.1	16.13	0.085	149	14.31	14.30
6	28-Aug-97	Balanced	249	708	709	0	0	2,115	0.68	6331	35.5	39.3	47.5	26,400	0.29	25.3	17.0	49.5	172.1	16.61	0.086	145	15.22	15.04
6	29-Aug-97	Balanced	249	711	742	0	0	2,112	0.68	6403	36.4	40.3	46.5	26,400	0.31	25.7	16.8	51.1	174.2	16.88	0.089	140	15.16	15.72
6	30-Aug-97	Balanced	249	701	644	0	0	2,184	0.68	6311	36.5	39.3	45.5	26,400	0.30	24.6	17.0	45.3	170.8	16.52	0.090	146	16.66	15.99
6	1-Sep-97	Balanced	249	705	644	0	0	2,264	0.70	6499	35.1	38.5	47.5	26,400	0.28	23.7	15.8	46.9	164.7	15.84	0.083	149	10.65	10.00
6	2-Sep-97	Balanced	249	705	668	0	0	2,205	0.69	6418	35.1	38.9	46.0	26,400	0.28	23.4	16.1	48.1	166.9	16.14	0.087	147	13.24	12.64
6	3-Sep-97	Balanced	249	705	665	0	0	2,211	0.69	6419	39.3	46.4	46.0	26,400	0.28	23.3	16.0	48.2	165.6	15.93	0.086	142	13.01	12.81
6	4-Sep-97	Balanced	249	706	669	0	0	2,246	0.70	6516	37.9	42.8	45.5	26,400	0.27	22.4	15.7	49.0	163.6	15.62	0.086	143	12.77	12.02
6	15-Sep-97	Balanced	250	675	473	0	0	2,020	0.63	5147	36.4	38.6	49.0	28,700	0.24	28.3	15.7	41.6	136.4	12.07	0.066	166	14.82	19.56
6	16-Sep-97	Balanced	251	675	473	0	0	2,002	0.62	5093	35.4	39.0	51.5	28,700	0.23	27.1	15.7	42.2	134.5	11.99	0.062	151	16.90	21.67

DATA SUMMARY FOR LPMEOH™ DEMONSTRATION UNIT

Case	Date	Gas Type	Temp (Deg C)	Pres. (psig)	Balanced Gas (KSCFH)	CO Gas (KSCFH)	H2 Gas (KSCFH)	Recycle 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	17-Sep-97	Balanced	251	676	473	0	0	2,001	0.62	5075	35.1	39.5	52.5	28,700	0.23	25.2	16.0	41.4	137.1	12.26	0.062	147	20.28	24.41
6	18-Sep-97	Balanced	251	675	536	0	0	2,025	0.64	4872	37.5	38.5	50.5	31,000	0.29	35.6	17.6	41.3	155.8	12.91	0.073	147	15.76	20.30
6	19-Sep-97	Balanced	251	675	577	0	0	2,119	0.67	5047	37.3	38.6	51.0	31,000	0.30	33.3	18.0	41.7	165.9	13.78	0.077	155	16.11	18.56
6	20-Sep-97	Balanced	251	675	578	0	0	2,058	0.67	5054	38.1	40.5	51.0	31,000	0.28	31.6	17.7	42.4	163.5	13.51	0.076	148	16.80	19.28
6	21-Sep-97	Balanced	251	675	577	0	0	2,112	0.68	5115	38.8	39.2	48.5	31,000	0.28	29.4	17.7	42.0	164.7	13.64	0.081	151	18.24	19.61
6	22-Sep-97	Balanced	251	675	572	0	0	2,012	0.65	4922	36.7	38.3	52.0	31,000	0.26	30.3	17.5	43.5	157.8	12.94	0.072	148	16.17	19.52
6	23-Sep-97	Balanced	249	675	577	0	0	2,026	0.66	4971	39.1	41.1	49.5	31,000	0.26	29.5	17.7	43.1	160.8	13.15	0.077	143	19.31	22.79
6	24-Sep-97	Balanced	249	676	578	0	0	2,082	0.67	5076	39.6	39.2	47.0	31,000	0.25	27.7	17.2	43.6	159.1	12.96	0.081	140	17.14	18.95
6	25-Sep-97	Balanced	249	675	578	0	0	2,057	0.66	5031	38.8	36.6	46.5	31,000	0.25	28.1	17.2	43.9	157.8	13.00	0.081	147	15.76	17.72
6	26-Sep-97	Balanced	249	680	579	0	0	2,081	0.66	5065	37.6	37.5	49.5	31,000	0.26	28.0	17.2	43.6	159.4	13.35	0.077	153	16.91	18.54
6	27-Sep-97	Balanced	249	690	573	0	0	2,118	0.66	5120	37.2	36.9	50.0	31,000	0.25	27.5	17.3	42.8	160.7	13.51	0.077	154	17.50	18.41
6	28-Sep-97	Balanced	249	681	555	0	0	2,115	0.66	5085	40.1	40.8	47.5	31,000	0.25	26.8	17.0	42.3	157.5	13.25	0.079	155	17.24	18.21
6	3-Oct-97	Balanced	249	676	552	0	0	2,161	0.69	4847	41.0	40.7	49.0	33,300	0.28	34.1	17.6	39.8	166.4	13.02	0.081	143	15.23	17.11
6	4-Oct-97	Balanced	249	675	497	0	0	2,125	0.66	4671	39.2	39.5	51.5	33,300	0.28	35.1	17.2	38.4	155.2	12.15	0.072	146	15.08	17.88
6	5-Oct-97	Balanced	249	675	548	0	0	2,017	0.65	4584	40.1	38.0	48.5	33,300	0.28	34.4	18.2	40.2	163.6	12.80	0.080	141	17.92	22.81
6	6-Oct-97	Balanced	249	675	578	0	0	2,098	0.67	4749	40.0	38.2	49.0	33,300	0.27	31.5	18.2	40.8	170.0	13.31	0.083	139	15.37	17.23
6	7-Oct-97	Balanced	249	675	577	0	0	2,098	0.68	4766	40.2	38.0	48.5	33,300	0.27	29.9	18.2	40.6	170.5	13.35	0.084	140	16.12	17.47
6	8-Oct-97	Balanced	249	675	578	0	0	2,093	0.67	4763	40.9	37.1	46.5	33,300	0.27	30.0	18.0	40.9	169.3	13.25	0.087	142	14.49	16.14
6	9-Oct-97	Balanced	250	675	577	0	0	2,092	0.67	4753	37.0	40.9	57.5	33,300	0.27	30.2	18.3	40.6	170.6	13.36	0.071	135	13.97	15.19
6	10-Oct-97	Balanced	250	674	577	0	0	2,098	0.68	4768	37.0	40.9	57.5	33,300	0.26	29.7	18.1	40.9	169.3	13.25	0.070	136	13.37	14.35
6	11-Oct-97	Balanced	250	675	578	0	0	2,082	0.67	4743	38.1	39.8	54.0	33,300	0.26	29.7	18.0	41.1	168.6	13.20	0.074	140	13.01	14.31
6	12-Oct-97	Balanced	249	674	578	0	0	2,089	0.67	4750	38.2	40.5	54.5	33,300	0.26	28.9	18.0	41.2	168.1	13.16	0.073	143	13.28	14.25
6	13-Oct-97	Balanced	249	674	577	0	0	2,067	0.67	4698	38.2	39.4	53.5	33,300	0.25	29.3	17.5	42.8	161.7	12.66	0.072	142	12.83	14.66
6	14-Oct-97	Balanced	249	672	522	0	0	2,145	0.68	4760	40.5	42.0	51.0	33,300	0.24	27.2	16.7	40.4	154.8	12.12	0.072	154	14.08	14.85
6	15-Oct-97	Balanced	250	674	578	0	0	2,107	0.68	4785	39.0	39.6	52.0	33,300	0.24	26.7	17.1	43.2	160.7	12.58	0.074	144	14.45	15.03
6	16-Oct-97	Balanced	250	675	577	0	0	2,084	0.67	4747	38.7	39.4	52.5	33,300	0.24	27.6	17.3	42.7	162.1	12.69	0.073	147	13.34	14.44
6	17-Oct-97	Balanced	249	675	578	0	0	2,075	0.67	4717	39.5	39.0	50.5	33,300	0.24	28.1	17.2	43.1	160.9	12.59	0.076	146	12.54	14.36
6	18-Oct-97	Balanced	249	674	577	0	0	2,081	0.67	4742	39.5	38.9	50.5	33,300	0.24	27.1	17.4	42.5	163.2	12.78	0.077	146	12.72	13.68
6	19-Oct-97	Balanced	249	673	578	0	0	2,071	0.67	4733	39.9	39.3	50.0	33,300	0.24	26.6	17.3	42.8	161.8	12.67	0.077	149	12.79	13.66
6	21-Oct-97	Balanced	250	675	560	0	0	2,070	0.67	4692	40.7	38.6	48.0	33,300	0.21	26.5	15.9	45.6	147.2	11.53	0.073	149	12.73	14.49
6	22-Oct-97	Balanced	250	675	515	0	0	2,115	0.66	4687	41.5	39.8	47.5	33,300	0.21	25.4	15.8	42.7	144.7	11.33	0.073	152	12.88	14.09
6	23-Oct-97	Balanced	249	675	508	0	0	2,104	0.66	4662	41.6	38.9	46.5	33,300	0.20	24.9	15.5	43.1	141.6	11.09	0.073	152	12.61	13.85
6	24-Oct-97	Balanced	250	675	513	0	0	2,062	0.65	4596	40.2	39.4	49.5	33,300	0.21	24.8	15.9	43.0	143.2	11.22	0.069	150	12.98	14.58
28	25-Oct-97	Balanced	259	675	499	0	0	2,043	0.66	4540	40.3	37.2	48.0	33,300	0.21	29.6	16.7	40.2	149.0	11.67	0.074	145	12.12	14.74
28	26-Oct-97	Balanced	259	676	499	0	0	2,064	0.66	4583	39.7	38.3	50.0	33,300	0.21	28.7	16.8	39.7	150.9	11.81	0.072	150	12.77	14.70
28	27-Oct-97	Balanced	258	674	513	0	0	2,075	0.66	4598	41.7	42.4	49.5	33,300	0.20	28.0	16.5	41.6	148.0	11.59	0.071	159	13.15	15.03
28	28-Oct-97	Balanced	259	674	513	0	0	2,041	0.66	4550	40.8	39.1	48.5	33,300	0.19	28.0	16.2	42.5	144.8	11.34	0.071	157	12.31	14.42
28	29-Oct-97	Balanced	259	674	501	0	0	2,069	0.66	4568	41.6	38.4	46.5	33,300	0.19	26.9	16.1	41.3	145.4	11.38	0.075	156	13.12	14.93
28	30-Oct-97	Balanced	259	675	508	0	0	2,017	0.65	4501	40.2	38.2	49.0	33,300	0.19	28.0	16.0	42.8	142.7	11.17	0.069	153	11.66	14.24
28	31-Oct-97	Balanced	259	675	508	0	0	2,042	0.66	4534	40.4	36.8	47.5	33,300	0.18	27.2	15.6	43.5	140.2	10.97	0.070	152	12.16	14.70
28	1-Nov-97	Balanced	259	674	508	0	0	2,030	0.65	4512	41.4	38.4	47.0	33,300	0.18	27.1	15.7	43.5	140.4	10.99	0.071	155	12.27	14.99
28	2-Nov-97	Balanced	259	673	508	0	0	2,059	0.66	4566	41.5	38.2	46.5	33,300	0.18	26.4	15.5	43.4	140.4	11.00	0.072	159	12.59	15.08

APPENDIX D - SUMMARY OF LPMEOH™ DEMONSTRATION UNIT OUTAGES (APRIL-NOVEMBER 1997)

SUMMARY OF LPMEOH™ DEMONSTRATION UNIT OUTAGES - APRIL/JUNE 1997

Operation Start	Operation End	Operating Hours	Shutdown Hours		Reason for Shutdown
4/2/97 09:00	4/2/97 16:15	7.3	4.8		Syngas Unavailable to LPMEOH™ Demonstration Unit
4/2/97 21:05	4/2/97 21:25	0.3	23.3		Liquids to K-01
4/3/97 20:40	4/4/97 11:00	14.3	24.8		Syngas Unavailable to LPMEOH™ Demonstration Unit
4/5/97 11:45	4/6/97 01:45	13.0	5.8		C-03 Outlet Plugged
4/6/97 07:30	4/7/97 13:05	29.6	2.1		C-03 Outlet Plugged
4/7/97 15:10	4/8/97 06:30	15.3	21.5		Syngas Unavailable to LPMEOH™ Demonstration Unit
4/9/97 04:00	4/9/97 05:30	1.5	4.0		ESD on C-02 Level
4/9/97 09:30	4/9/97 14:20	4.8	9.7		Syngas Unavailable to LPMEOH™ Demonstration Unit
4/10/97 00:00	4/11/97 08:25	32.4	14.8		Syngas Unavailable to LPMEOH™ Demonstration Unit
4/11/97 23:15	4/18/97 18:05	162.8	0.7		Syngas Unavailable to LPMEOH™ Demonstration Unit
4/18/97 18:45	4/19/97 07:50	13.1	0.7		Syngas Unavailable to LPMEOH™ Demonstration Unit
4/19/97 08:30	4/23/97 00:20	87.8	20.7		Replace TV-101 Trim
4/23/97 21:00	4/23/97 21:00	0.0	12.0	*	Syngas Unavailable to LPMEOH™ Demonstration Unit
4/24/97 09:00	5/8/97 23:59	351.0	950.1	**	Syngas Unavailable to LPMEOH™ Demonstration Unit
6/17/97 14:05	6/30/97 23:59	321.9			

Total Operating Hours	1055.2
Syngas Available Hours	1112.0
Plant Availability, %	94.9

* Plant was ready to startup, but Eastman waited 12 hours to give the day crew training on startup procedures.

** Eastman complex outage.

SUMMARY OF LPMEOH™ DEMONSTRATION UNIT OUTAGES - JULY/SEPTEMBER 1997

Operation Start	Operation End	Operating Hours	Shutdown Hours		Reason for Shutdown
7/1/97 00:01	7/8/97 17:10	185.2	8.7		Syngas Unavailable to LPMEOH™ Demonstration Unit
7/9/97 01:50	7/29/97 00:25	478.6	68.3		Fix C-06 Flange Leak
7/31/97 20:40	8/12/97 21:05	288.4	51.2		Fix C-06 Flange Leak
8/15/97 00:15	8/31/97 13:30	397.3	10.0		Syngas Unavailable to LPMEOH™ Demonstration Unit
8/31/97 23:30	9/5/97 14:40	111.2	20.0		Syngas Unavailable to LPMEOH™ Demonstration Unit
9/6/97 10:40	9/6/97 10:40	0.0	149.3	*	Low Catalyst Activity
9/12/97 16:00	9/29/97 18:30	410.5	29.5		G-03 Electrical Tie-in and Eastman Guard Bed Change
9/30/97 23:59	9/30/97 23:59	0.0			End of Reporting Period

Total Operating Hours	1871.1
Syngas Available Hours	2169.3
Plant Availability, %	86.3

* Syngas became available, but Demonstration Unit would not restart because of low catalyst activity. Demonstration Unit was restarted after addition of one fresh batch of catalyst.

SUMMARY OF LPMEOH™ DEMONSTRATION UNIT OUTAGES - OCTOBER/DECEMBER 1997

Operation Start	Operation End	Operating Hours	Shutdown Hours	Reason for Shutdown
10/1/97 00:01	10/1/97 00:01	0.0	51.5	G-03 Electrical Tie-in and Eastman Guard Bed Change
10/3/97 03:30	11/3/97 14:50	756.3	1112.0	End of Catalyst Run
12/19/97 22:50	12/31/97 23:59	289.1		End of Reporting Period

Total Operating Hours	1045.5
Syngas Available Hours	1097.0 *
Plant Availability, %	95.3 *

* Excluding catalyst changeout to restart test program.

APPENDIX E - REACTOR GAS SPARGER RESISTANCE COEFFICIENT

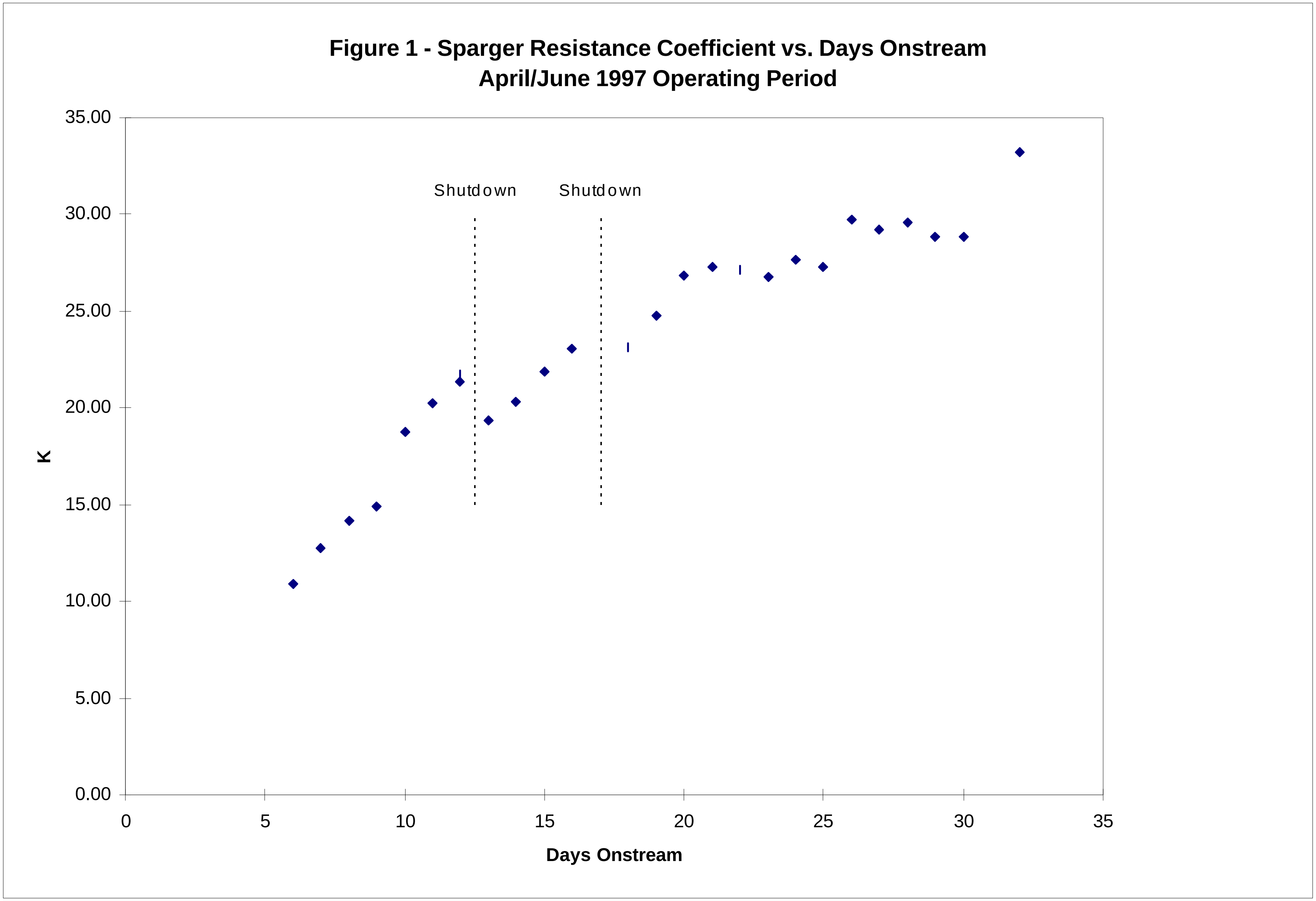

Figure 1 - Sparger Resistance Coefficient vs. Days Onstream
April/June 1997 Operating Period
Shutdown
Shutdown
K
35.00
30.00
25.00
20.00
15.00
10.00
5.00
0.00
0
5
10
15
20
25
30
35
Days Onstream

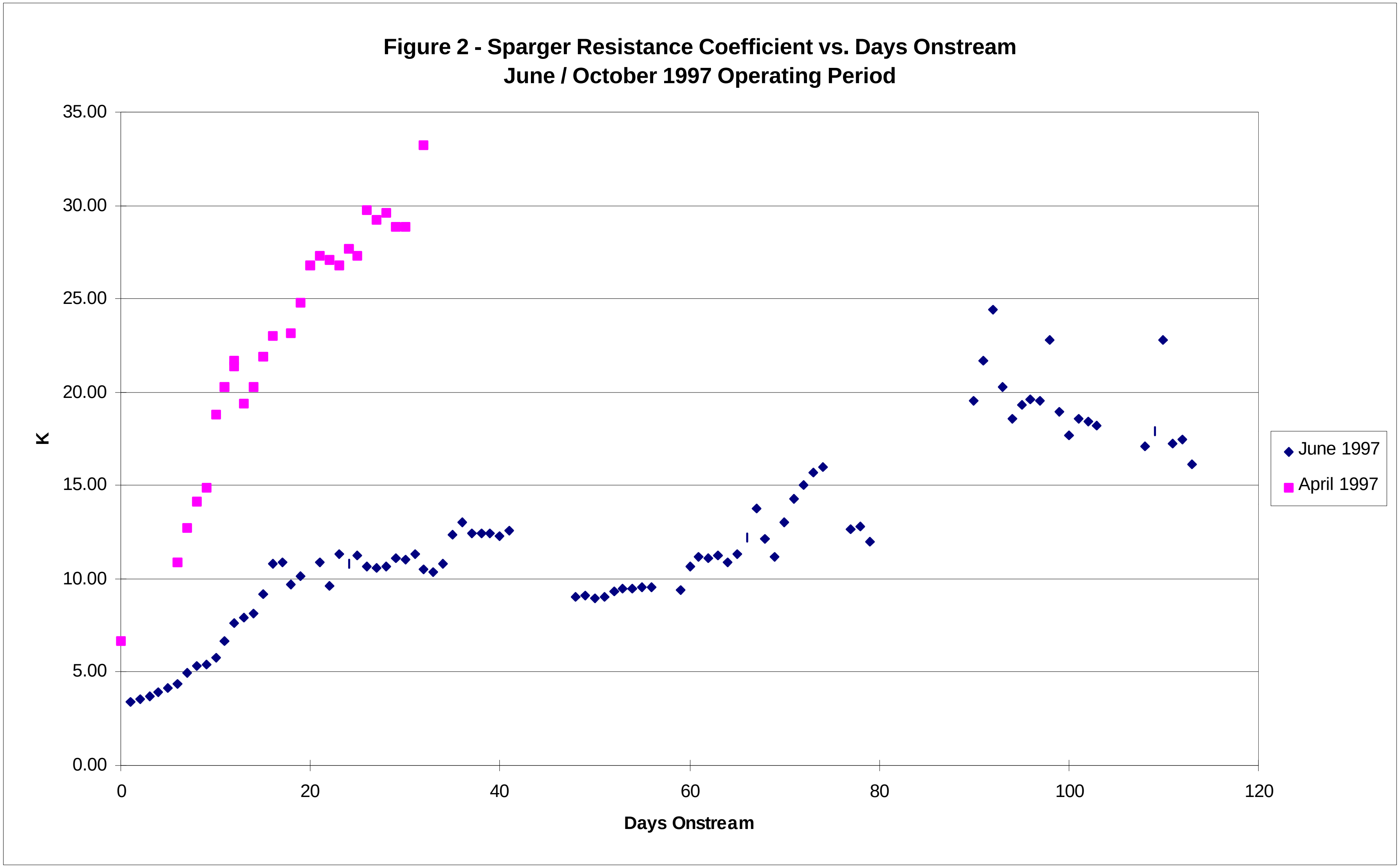
Figure 2 - Sparger Resistance Coefficient vs. Days Onstream
June / October 1997 Operating Period
35.00
30.00
25.00
20.00
15.00
10.00
5.00
0.00
K
0
20
40
60
80
100
120
Days Onstream
June 1997
April 1997

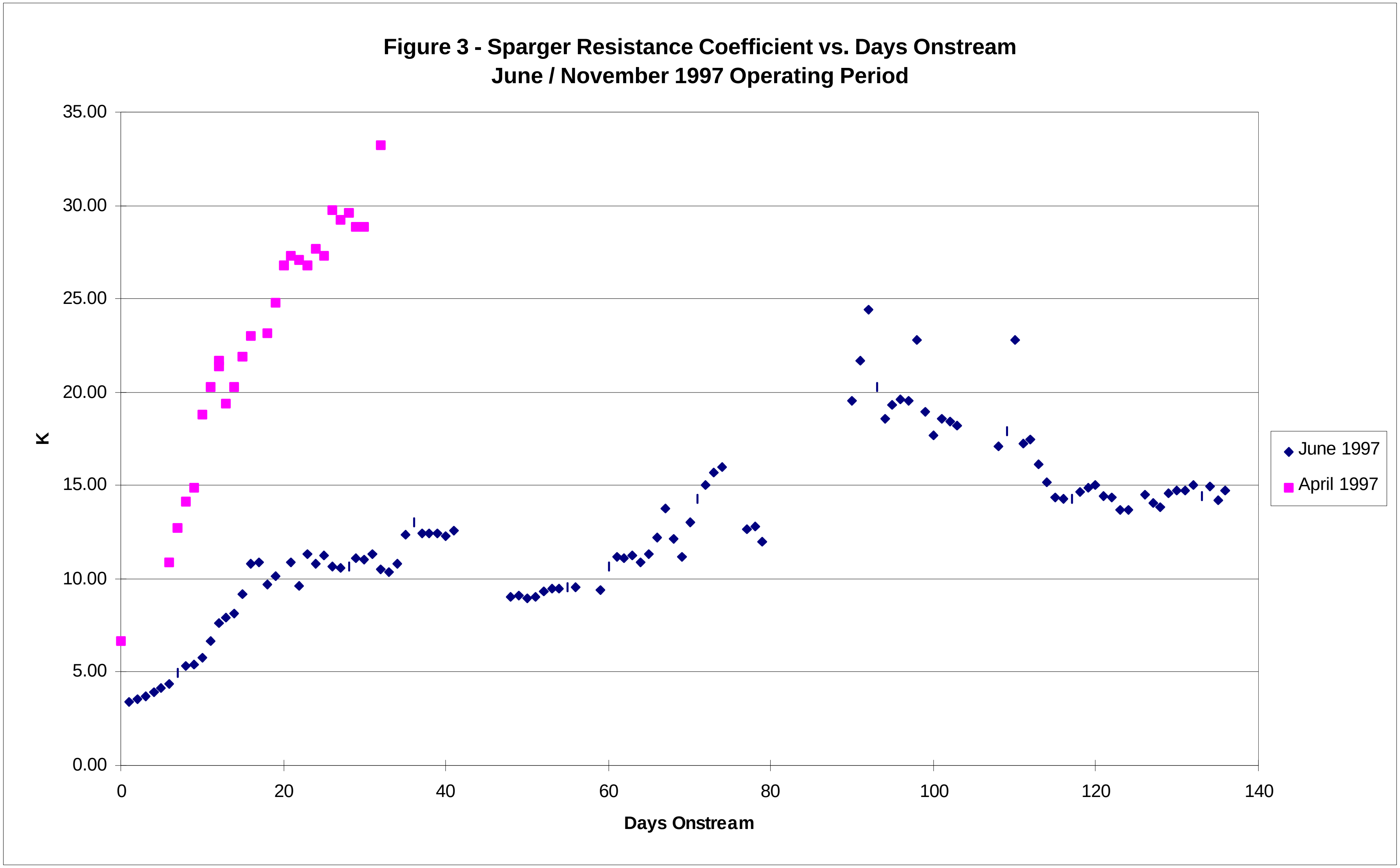

Figure 3 - Sparger Resistance Coefficient vs. Days Onstream
June / November 1997 Operating Period
K
35.00
30.00
25.00
20.00
15.00
10.00
5.00
0.00
0
20
40
60
80
100
120
140
Days Onstream
June 1997
April 1997

APPENDIX F - METHANOL SYNTHESIS CATALYST PERFORMANCE AND ANALYSIS

Figure 1 - Catalyst Life (η) vs. Days Onstream

Table 1 - Summary of Catalyst Samples

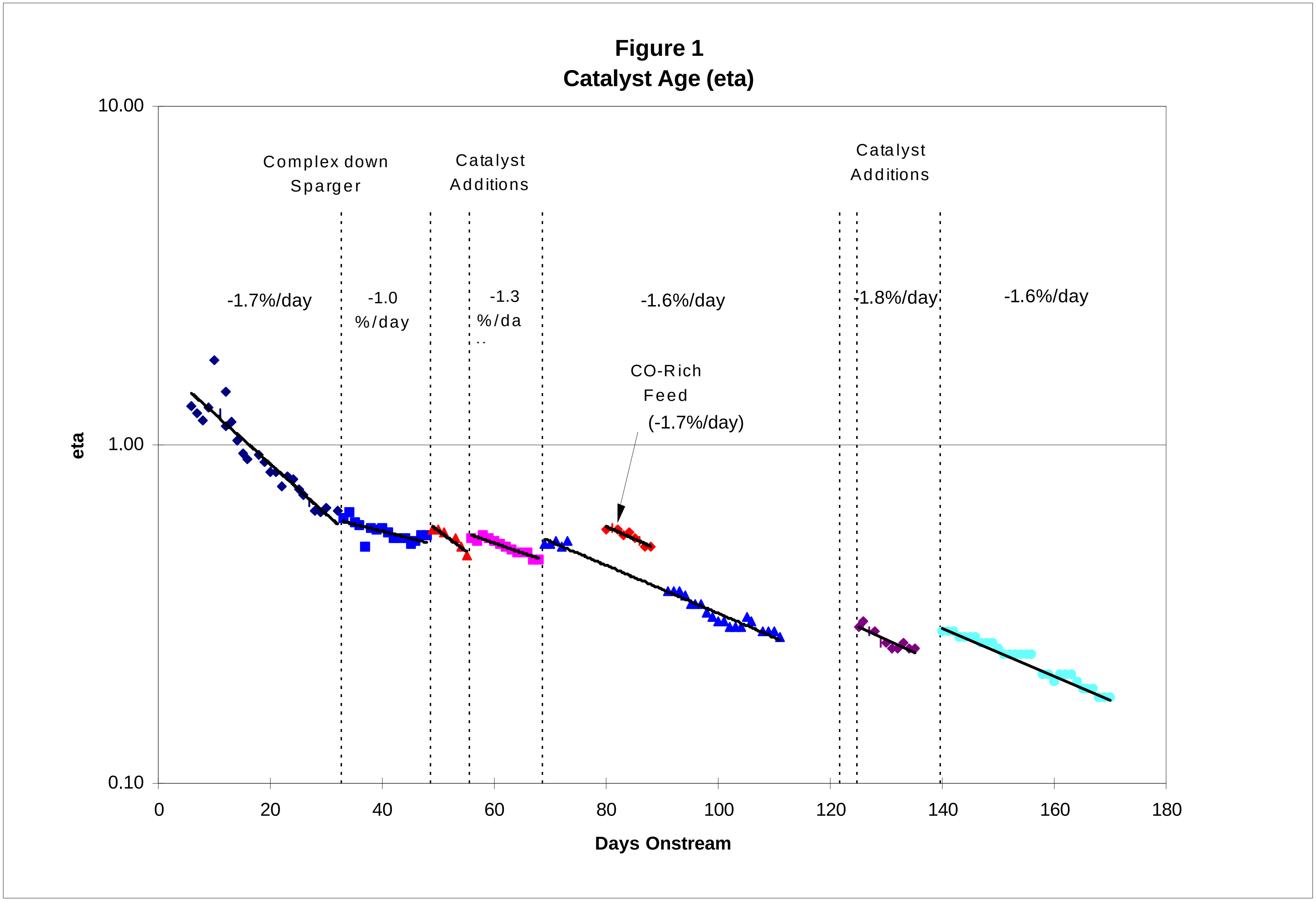
Figure 1
Catalyst Age (eta)
10.00
1.00
0.10
eta
Complex down
Sparger
Catalyst
Additions
Catalyst
Additions
-1.7%/day
-1.0
%/day
-1.3
%/da
-1.6%/day
-1.8%/day
-1.6%/day
CO-Rich
Feed
(-1.7%/day)
0
20
40
60
80
100
120
140
160
180
Days Onstream

Table 1
Summary of Catalyst Samples

Sample	Identity	XRD		BET	Analytical (ppm)				
		Cu	ZnO	m2/g	Fe	Ni	S	As	Cl
14987-54	Lab run using 383-4119 (450 hours)	175	74						
Trailer Run	AFFTU run in Kingsport (672 hours)	179	101		172	58	<=660	184	5570
Reduction #3	Reduction Batch sample from Kingsport	73	55	57	49	32	<=110	<25	
Reduction #4	Reduction Batch sample from Kingsport			73	83	28	<100	<25	
Reduction #6	Reduction Batch sample from Kingsport			90	29	18	<=150	<25	
Reduction #8	Reduction Batch sample from Kingsport			81	26	23	<=110	<25	
K0597-2	Reactor Sample 6/15/97 (30 days)	274	89	40	281	61	<=190	446	<200
K0897-1	Reactor Sample 8/19/97	283	87	43	169	<20	235	601	
K0997-1	Reactor Sample 9/5/97	281	118	42	261	37	575	779	
K1097-1	Reactor Sample 9/29/97	289	187	45	189	28	330	711	
K1197-1b	Reactor Sample 11/7/97	292	111	40	194	37	340	699	