# LIQUID PHASE DIMETHYL ETHER DEMONSTRATION IN THE LAPORTE ALTERNATIVE FUELS DEVELOPMENT UNIT

**Topical Report**

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**Prepared by**

**Air Products and Chemicals, Inc.**
**Allentown, Pennsylvania**

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

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## ABSTRACT

A demonstration of the co-production of dimethyl ether (DME) with methanol from synthesis gas by Air Products and Chemicals, Inc.'s Liquid Phase Dimethyl Ether (LPDME) Process was successfully completed in a slurry bubble column reactor at the U. S. Department of Energy's (DOE's) Alternative Fuels Development Unit (AFDU) in LaPorte, Texas. The plant was operated for 25 days to compare catalyst aging in a pilot-scale (10 tons per day) slurry bubble column with that in a laboratory autoclave. The deactivation rate, on a rate constant basis, for both the methanol and the dehydration catalyst was estimated at 0.7% per day. This is lower than the 1.2% per day observed for both the catalyst in the autoclave. However, the rate is slightly higher than 0.5% per day rate achieved for the Liquid Phase Methanol (LPMEOH™) Process after 3 weeks of operation at LaPorte. This demonstration represents a significant step forward in the development of the LPDME Process. The 0.7% per day aging rate achieved in the AFDU is a large improvement over the 4% per day autoclave deactivation of the previous catalyst system. The results of an economic screening study indicate that DME can be produced in an Integrated Gasification Combined Cycle (IGCC) system at costs approaching liquified petroleum gas (LPG) in China. A report on the market analysis for DME and the economics of the LPDME Process is planned. Further research and development and economic studies are suggested to continue the developmental work.

The methanol catalyst was successfully activated with dilute hydrogen, resulting in an expected hydrogen uptake. The initial productivities of methanol and DME were higher than the laboratory, perhaps due to the effect of multiple continuous stirred tank reactors (CSTRs) in the slurry bubble column. The reactor operated hydrodynamically stable with uniform temperature profile and gas holdups. Gas, liquid, and solid phase mixing were studied using radioactive tracer injections. Both short-term and long-term observations of irradiated dehydration catalyst suggest no alumina settling in the reactor. The tracer data will be analyzed with help of personnel from Washington University as part of the Hydrodynamic Program with DOE. The oxygenates system containing the high pressure reactor was operated after a 4-year gap; the operations were smooth. The initial start-up was very quick with the baseline condition reached in 12 hours after the introduction of synthesis gas. A re-start after a synthesis gas outage only took 4 hours, demonstrating the ease and flexibility of the slurry technology to respond to changes.

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ACRONYMS AND DEFINITIONS

| | | |
|---|---|---|
| Air Products | - | Air Products and Chemicals, Inc. |
| AFDU | - | Alternative Fuels Development Unit - The “LaPorte PDU” |
| Btu | - | British Thermal Unit |
| Catalyst Concentration | - | Synonym for Slurry Concentration |
| Catalyst Loading | - | Synonym for Slurry Concentration |
| cc | - | cubic centimeter |
| CO Conversion | - | the percentage of CO consumed across the reactor |
| CSTR | - | continuous stirred tank reactor |
| DME | - | dimethyl ether |
| DP | - | differential pressure |
| DOE | - | United States Department of Energy |
| DOE-NETL | - | The DOE's National Energy Technology Laboratory (Project Team) |
| DOE-HQ | - | The DOE's Headquarters - Coal Fuels and Industrial Systems (Project Team) |
| DVT | - | Design Verification Testing |
| Eastman | - | Eastman Chemical Company |
| FID | - | flame ionization detector |
| F-T | - | Fischer-Tropsch |
| Gas Holdup | - | the percentage of reactor volume up to the Gassed Slurry Height which is gas |
| GC | - | gas chromatograph |
| GHSV | - | gas hourly space velocity |
| hr | - | hour |
| IGCC | - | Integrated Gasification Combined Cycle, a type of electric power generation plant |
| Inlet Superficial Velocity | - | the ratio of the actual cubic feet of gas at the reactor inlet (calculated at the reactor temperature and pressure) to the reactor cross-sectional area (excluding the area contribution by the internal heat exchanger); typical units are feet per second |
| LaPorte PDU | - | The DOE-owned experimental unit (PDU) located adjacent to Air Products’ industrial gas facility at LaPorte, Texas, where the LPMEOH™ Process was successfully piloted |
| LPDME | - | Liquid Phase DME, the technology being demonstrated |
| LPG | - | liquified petroleum gas |
| LPMEOH™ | - | Liquid Phase Methanol |
| MeOH | - | methanol |
| Methanol Equivalent Productivity | - | the methanol productivity plus two times the DME productivity in terms of moles per hour per kilogram of total catalysts (on an oxide basis) |
| MW | - | molecular weight, pound per pound mole |
| NDG | - | nuclear density gauge |
| 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) |
| psi | - | pounds per square inch |
| psia | - | pounds per square inch (absolute) |
| psig | - | pounds per square inch (gauge) |
| 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 |

ACRONYMS AND DEFINITIONS (cont'd)

| | | |
|---|---|---|
| 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) |
| TCD | - | thermal conductivity detector |
| TNRCC | - | Texas Natural Resource Conservation Commission |
| TPD | - | short tons per day |
| vol% | - | volume % |
| wt | - | weight |

# EXECUTIVE SUMMARY

A demonstration of the production of dimethyl ether (DME) by Air Products and Chemicals, Inc.'s (Air Products') Liquid Phase Dimethyl Ether (LPDME) Process was successfully completed in a slurry bubble column reactor at the U. S. Department of Energy's (DOE's) Alternative Fuels Development Unit (AFDU) in LaPorte, Texas. The demonstration was conducted at pilot-scale of 10 tons per day (TPD) to evaluate the commercial viability of the LPDME Process. Synthesis gas or syngas (a mixture of primarily hydrogen and carbon monoxide) produced from coal as well as natural gas can be converted to form DME. DME has potential applications as a diesel substitute, a domestic fuel or a chemical building block. Alternatively, DME synthesis can be incorporated into an Integrated Gasification Combined Cycle (IGCC). The Air Products' LPDME Process uses a physical mixture of a commercial methanol catalyst and a commercial dehydration catalyst in a single slurry reactor to co-produce DME and methanol. This process provides high syngas conversion, efficient heat transfer, and directly converts a variety of feed gas compositions. While the proof-of-concept was demonstrated in 1991 at the AFDU, the catalyst system needed improvements in stability. Researchers at Air Products significantly improved the life of the catalyst system in a study conducted starting in 1994, and, the next step was to demonstrate these improvements in a slurry bubble column at a larger scale. This run was co-funded by the Alternative Fuels and Chemicals II program and the Clean Coal Technology Program's Liquid Phase Methanol (LPMEOH™) Process Demonstration Project.

The plant was operated for 25 days to compare catalyst aging in a pilot-scale slurry bubble column with that in a laboratory autoclave. The catalyst life study was extended instead of a planned process variable study to obtain additional data on catalyst aging. The proportion of two catalysts corresponded to a 95:5 methanol to dehydration catalyst ratio by weight. The deactivation rate for both the catalysts was estimated at 0.7% per day. This is lower than the 1.2% per day observed for both the catalysts in the autoclave. However, the rate is slightly higher than 0.5% per day rate achieved for the LPMEOH™ Process after 3 weeks of operation at LaPorte. This demonstration represents a significant step forward in the development of the LPDME Process. The 0.7% per day aging rate achieved in the AFDU is a large improvement over the 4% per day autoclave deactivation of the previous catalyst system (81:19 methanol to dehydration catalyst ratio by weight). The results of an economic screening study indicate that DME can be produced in an Integrated Gasification Combined Cycle (IGCC) system at costs approaching liquified petroleum gas (LPG) in China. A report on the market analysis for DME and the economics of the LPDME Process is planned. Further research and development (R&D) and economic studies are suggested to continue the developmental work.

The methanol catalyst was successfully activated with dilute hydrogen, resulting in an expected hydrogen uptake. The initial productivities of methanol and DME were higher than the laboratory, perhaps due to the effect of multiple continuous stirred tank reactors (CSTRs) in the slurry bubble column. The DME production rate started at 5.1 TPD and declined to 4.1 TPD in 25 days on-stream, while the methanol production showed a scatter within the 3.1 to 3.8 TPD range through the run. The scatter in data decreased significantly after a gas chromatograph (GC) problem and a sampling problem were discovered and resolved. The reactor operated hydrodynamically stable with uniform temperature profile and gas holdups. Differential pressure measurements indicated about 42 vol% gas holdup and 36 wt% catalyst concentration.

Gas, liquid, and solid phase mixing was studied using radioactive tracer injections. A large amount of data was collected using 34 detectors around the reactor. Several repeat injections were made during the gas and liquid injections to evaluate variability with time. Manganese oxide doped gamma alumina was injected at four different locations to look at the dehydration catalyst mixing. Both short-term and long-term observations of irradiated dehydration catalyst suggest no alumina settling in the reactor. A post-run inspection of the reactor bottom head did not show any settled catalyst around the sparger, in contrast to the 1991 DME run when significant settled catalyst was found at the bottom. The tracer data will be analyzed with help of personnel from Washington University as part of the Hydrodynamic Program with DOE.

The oxygenates system containing the high pressure reactor was operated after a 4-year gap; the operations were smooth. The initial start-up was very quick with the baseline condition reached in 12 hours after the introduction of syngas. A re-start after a syngas outage only took 4 hours. This demonstrates the ease and flexibility of the slurry technology to respond to changes. Approximately 27,200 gallons of liquid product (89 wt% methanol, 7 wt% DME) was collected during the run.

# INTRODUCTION

The generation of synthesis gas or syngas (a mixture of primarily hydrogen ($H_2$) and carbon monoxide (CO)) from a variety of carbon sources including coal, natural gas, biomass, and environmentally-distressed materials such as petroleum-derived coke/residues, has emerged as a promising method of providing our nation with environmentally benign, alternative sources of chemicals and energy. Syngas is a potential raw material for chemical intermediates including both oxygenated and hydrocarbon alternative fuels, octane enhancers, and a myriad of other industrial chemicals such as acetyls, alcohols, ethers, and olefins. Processing syngas generated by coal gasification has been the principle focus of the U. S. Department of Energy (DOE) program on the indirect liquefaction of coal. Emergence of these new sources of syngas demonstrates the potential importance of syngas processing technology to the current energy policy. One example of this technology in an industrial setting is the successful demonstration of the Liquid Phase Methanol (LPMEOH™) technology under the Clean Coal Technology Program at Eastman Chemical Company's (Eastman's) chemicals-from-coal complex in Kingsport, Tennessee, using the slurry bubble-column technology developed at the DOE's Alternative Fuels Development Unit (AFDU) in LaPorte, Texas.

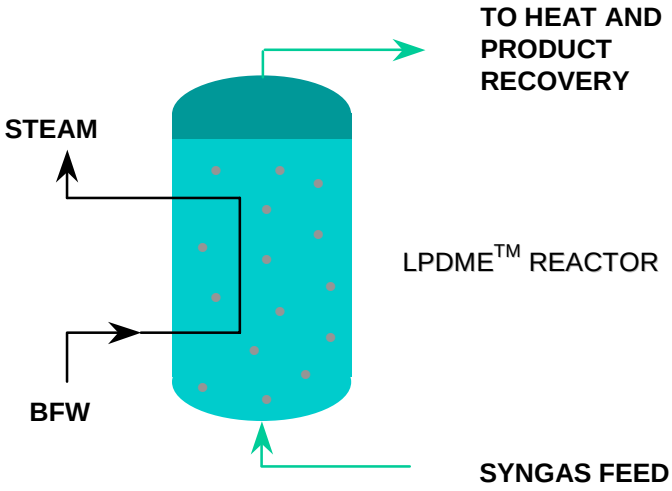
The LPMEOH™ Process Demonstration Project at Kingsport, Tennessee, is a $213.7 million cooperative agreement between the DOE and Air Products Liquid Phase Conversion Company, L. P. (the Partnership). Air Products and Chemicals, Inc. (Air Products) and Eastman formed the Partnership to execute the Demonstration Project. A demonstration unit producing 80,000 gallons per day (260 TPD) of methanol was designed, constructed, and is operating at a site located at the Eastman chemicals-from-coal complex in Kingsport. The Partnership owns and is operating the facility for the 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 has been demostrating 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 demonstration unit has been onstream for about 4 years, and the operating results have been excellent (1).

This project has also been evaluating the demonstration of the production of dimethyl ether (DME) as a mixed coproduct with methanol. DME has potential applications as a diesel substitute, domestic fuel or chemical building block. As part of this activity, a run was conduted to co-produce DME with methanol at the LaPorte AFDU and evaluate the commercial viability of the Liquid Phase Dimethyl Ether (LPDME) Process.

The Air Products' LPDME Process uses a physical mixture of a commercial methanol catalyst and a commercial dehydration catalyst in a single slurry reactor to co-produce DME with methanol. This process provides high syngas conversion, efficient heat transfer, and directly converts a variety of feed gas compositions. Fine catalyst particles are entrained in an inert hydrocarbon liquid, usually a 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. 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 manage the high syngas conversion per pass that be achieved in the LPDME process.




In addition, the LPDME process is particularly well suited to coal-derived syngas which is rich in carbon monoxide. These capabilities make the LPDME process a potentially lower-cost conversion route to DME, especially when DME coproduction is added to a coal-based Integrated Gasification Combined Cycle (IGCC) power plant. Syngas mixtures produced from coal as well as natural gas can be converted to form DME.

While the proof-of-concept was demonstrated in 1991 at the AFDU (2), the catalyst system needed improvements in stability. Researchers at Air Products significantly improved the life of the catalyst system in a study conducted starting in 1994 (3,4,5), and, the next step was to demonstrate these improvements at a pilot scale in a slurry bubble column. DOE sponsorship was provided from the Alternative Fuels and Chemicals II Program, and the Clean Coal Technology Program's LPMEOH™ Process Demonstration Project. Some perspective on the involvement of the LPMEOH™ Demonstration Project is provided for reference.

An excerpt from Commercialization Objectives from the LPMEOH™ Demonstration Project Statement of Work is included here to provide the global perspective regarding the demonstration of the coproduction of DME with methanol:

> "Secondary Objective
>
> A secondary objective of the Project is to demonstrate the production of DME (Dimethyl ether) as a mixed coproduct with methanol...
>
> Subject to Design Verification Testing (DVT), the Partnership proposes to enhance the Project by including the demonstration of the slurry reactor's capability to produce DME as a mixed co-product with methanol...
>
> DVT is required to address issues such as catalyst activity and stability and to provide data for engineering design and demonstration decision making...

At the conclusion of the DVT Steps, a joint Partnership/DOE decision will be made regarding continuation of the methanol/DME demonstration. Timing of the final decision must ensure that the necessary design, procurement, construction and commissioning can be completed to allow for (Phase 3, Task 2.2) operation at the end of the primary LPMEOH™ process demonstration period."

The first decision milestone, on whether to continue with DVT activities, was targeted for 01 December 1996. This milestone was relaxed to July of 1997 to allow time for further development of the LPDME catalyst system. DVT was considered necessary to provide additional data for engineering design and demonstration decision-making. The essential steps required for decision-making were: a) confirm catalyst activity and stability in the laboratory, b) develop engineering data in the laboratory, and c) confirm market(s), including fuels and chemical feedstocks.

DOE issued a letter dated 31 July 1997 accepting Air Products' recommendation to continue with the design verification testing to co-produce DME with methanol, and to proceed with planning a design verification test run at the LaPorte AFDU. A copy of the recommendation (dated 30 June 1997) is included in Appendix A-1. The recommendation was based on the results of the Market Economic Studies and on the LPDME catalyst system development work.

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

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

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

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

LPDME was not applicable to $H_2$-rich syngas; and, at the time of development of the recommendation, it was considered unlikely that a substantive LPDME demonstration would be recommended for Kingsport. Therefore, a convincing case that the test-run on CO-rich syngas at LaPorte would lead to successful commercialization had to be made, prior to approving the final test-run plan. The strategy for commercialization needed to present the technical logic to combine the results of the following two areas:

1) catalyst performance (productivity, selectivity, and life) for the LPDME

catalyst system under CO-rich syngas from the design verification testing at the LaPorte AFDU; and

2) reactor performance (methanol catalyst activity and life, hydrodynamics, and heat transfer) from the LPMEOH™ Process Demonstration Unit at Kingsport.

The 1997 DME DVT recommendation summarizes the catalyst targets, experimental results, and the corresponding economics for a commercially successful LPDME catalyst.

Work on the feasibility study for the coproduction of DME and methanol with electric power was completed. The product DME would be used as a domestic liquid cooking fuel, to replace imported liquid petroleum gas (LPG), for China and the Pacific Rim regions. The results are included in the 1997 DME recommendation in Appendix A-1.

At a review meeting for the DOE's Liquid Fuels Program on 09 June 1999, members of the LPMEOH™ Project Team from Air Products and DOE were given an update on the activities regarding the status of catalyst development and the economics for the LPDME Process. The participants agreed that the next test for the LPDME Process at the LaPorte AFDU should be treated as an interim campaign, with the primary objective being the determination of a tie-point between catalyst performance in the autoclave and the pilot plant scale.

Following this meeting, a formal recommendation to proceed with the interim campaign at the LaPorte AFDU was issued by Air Products to DOE. A copy of this letter (dated 06 August 1999) is included in Appendix A-2. DOE issued a letter dated 10 August 1999 accepting Air Products' recommendation to proceed with DME DVT activities at the LaPorte AFDU.

## RUN OBJECTIVES

The objectives of the run were:

- to demonstrate the operation of the LPDME process with improved catalyst life at a 10 tons per day (TPD) scale, using commercially produced catalysts,
- to obtain information to correlate the scale-up of catalyst aging from autoclave to bubble column,
- to conduct process variable testing, and,
- to perform experiments to better understand the reactor hydrodynamics.

## ENGINEERING AND MODIFICATIONS

With the plant in the Fischer-Tropsch (F-T) mode after the Fischer-Tropsch IV run (6), and the oxygenates system, containing the high pressure reactor, last used in 1995 for a hydrodynamic/methanol run (7), a significant set-up effort was needed, even though, no major modifications were required for this run.,. Since this was the first DME run in the high pressure reactor system as well as with the new distributed control system and data acquisition system, heat and mass balance spreadsheets had to be developed, and the data acquisition system had to be set up for DME synthesis. In addition, the analytical set-up required reconfiguration. Also, blinds needed to be switched from F-T to Oxygenates and common pumps needed to be relocated. Additionally, the following oxygenates systems were reactivated:

- Reduction Circuit
- Carbon Dioxide ($CO_2$) removal system

Process Description

The process flow diagrams are shown in Figures 1-3. The operation of the plant is described as follows:

$H_2$, CO, and nitrogen ($N_2$) are blended and compressed in the 01.10 feed gas compressor to about 800 psig. This stream is then mixed with recycle gas and additional hydrogen from a high pressure pipeline to obtain the desired syngas composition and flow. The 01.30 booster compressor was bypassed as higher pressure operation was not desired. The mixed feed then passes through the 01.34 aftercooler used to control the inlet temperature to the 21.11 feed/product economizer, which preheats the feed against the reactor effluent. The 10.95 high pressure liquid injection pump was not used during this run. The mixed feed is further preheated against high pressure steam in the 02.63 high pressure feed steam preheater before introducing the syngas blend to the bottom of the 27.20 high pressure slurry reactor.

The syngas flows upward through the slurry of catalyst and mineral oil as the reaction proceeds. The heat of reaction is absorbed by the slurry and removed through the internal heat exchanger, which uses a heat transfer fluid. The product gas passes through the reactor freeboard with the unconverted syngas, and the gross reactor effluent cools against the feed in the 21.11 economizer. Any traces of slurry oil entrained or vaporized in the effluent condense and are returned to the bottom of the reactor by the 10.52.02 pumps. The vapor leaving the 21.11 economizer: flows through the 27.14 vapor-liquid separator to remove any left-over condensed oil; chills against cooling water in the 21.30 hairpin exchangers; and passes into the 22.10 separator where liquid products (methanol, water, higher alcohols, some DME) collect. The liquids flash to near atmospheric pressure in the 22.11 degasser and collect in the 22.15 low pressure separator before passing on to the 22.16 day tank and eventually to a trailer for storage.

To minimize the amount of gas sent to the flare, most of the syngas leaving the 22.10 separator is recycled to the reactor. Since $CO_2$ is a byproduct of DME synthesis with CO-rich syngas, it was necessary to remove $CO_2$ from the 22.10 separator overheads before recycling this stream. The closed-loop $CO_2$-removal system uses methanol to preferentially absorb the $CO_2$ and DME from the syngas. The vapor from the 22.10 separator cools against returning $CO_2$-lean syngas in the 21.10 gas-gas economizer. It then feeds into the bottom of the 07.10

absorber and contacts against chilled methanol introduced at the top of the column. The $CO_2$-lean syngas leaves the top of the absorber and rewarms to ambient temperatures in the 21.10 gas-gas economizer before being recompressed in the 01.20 recycle compressor. A small portion of this gas is purged to flare to prevent the buildup of inerts. The $CO_2$-rich liquid collects in the bottom of the 07.10 absorber, de-pressurizes across a valve, and heats up against returning methanol in the 21.45 hairpin exchangers. This liquid then passes into the top of the 07.20 stripper where it is reboiled to remove the dissolved gases such as $CO_2$ and DME. The overhead cooling water condenser reduces the amount of methanol solvent lost in the overhead stream, which goes to flare. The liquid from the bottom of the 07.20 stripper cools in the 21.45 hairpin exchangers prior to recompression in the 10.80 pump. The methanol then chills against liquid $CO_2$ in the 21.80 kettle evaporator before recycling to the top of the 07.10 absorber.

The syngas from the 22.10 separator includes equilibrium amounts of methanol, water, and other hydrocarbons which will build up in the methanol solvent. Methanol is also lost in the 07.20 stripper overheads. As a result, the $CO_2$-removal system operates in an unsteady state as the composition of the solvent changes. Since this change affects the level of $CO_2$ removal, the system includes a solvent purge and fresh methanol makeup lines.

Bubble-Column Reactor

The high pressure 27.20 bubble-column reactor for oxygenate synthesis measures 50 feet flange-to-flange and 18 inches inside diameter (Figure 4). Its design slurry level is 40 feet from the bottom flange with the remainder being vapor disengagement space. The reactor contains an internal heat exchanger consisting of twelve 3/4" U- tubes occupying 8% of the reactor cross-section. In addition, thirteen thermocouples measure the longitudinal temperature profile at 4-foot intervals. A nuclear density gauge (NDG), mounted on an external hoist mechanism, spans the space occupied by the internal exchanger to measure slurry level and density. Six differential pressure (DP) transmitters have been installed on the reactor wall for more accurate slurry density measurements. Detectors are temporarily set-up at various locations outside the reactor during a radio-active tracer injection study. The design pressure of the reactor is 2,000 psig at 700°F.

Analytical Set-up

The analytical system was configured for DME synthesis. Two gas chromatographs (GC's) with Flame Ionization Detectors (FID's) monitored hydrocarbon and alcohol concentrations in the reactor feed and effluent streams. Two other GC's with Thermal Conductivity Detectors (TCD's) measured $H_2$, $N_2$, CO, $CO_2$, water ($H_2O$), methanol ($CH_3OH$ or MeOH), and DME ($CH_3OCH_3$) in feed, product, purge, and intermediate streams. A small amount of $N_2$ (approximately 1 mole%) was added to the reactor feed as an internal standard to verify flow measurements. Details of the analytical methods are given in Appendix B.

Hazards Review

Process Engineering, Operations and Safety personnel conducted a hazards review on August 23 and 24, 1999 to revalidate the AFDU for the DME operations starting in October of 1999. Changes since the initial hazards review in 1994 were discussed and safety documents were updated. The AFDU was approved for syngas operations starting 01 October 1999.

Environmental Reviews

A meeting was held with Air Products' Environmental personnel in November of 1997 to discuss air emission permit requirements for the planned DME operations. The original 1991 permit covered the DME operations in the old reactor (27.10) only, while the exemptions obtained in 1994 for the new reactor (27.20) were valid solely for methanol and isobutanol operations. As a result, at least an exemption was needed from the Texas Natural Resource Conservation Commission (TNRCC) to operate DME synthesis in the new reactor. Material balances were generated and air permit exemption calculations were conducted to verify the qualification for the exemption. Two cases with a maximum DME production rate of about 14 TPD (AFDU limit) were simulated. An application for the air permit exemption was prepared by Air Products' Environmental group. An approval from TNRCC was obtained in May of 1998.

## CATALYST DEVELOPMENT

A research and development (R&D) program was initiated by Air Products in 1994, with DOE's sponsorship under the Alternative Fuels and Chemicals II Program, to investigate the cause of accelerated catalyst deactivation and the underlying mechanism (2). Traditional causes such as leaching, hydrothermal sintering, coking, and poisoning were investigated and subsequently ruled out. Interaction between the methanol synthesis and the methanol dehydration catalysts was identified as a cause of deactivation. Screening of alternative dehydration catalysts suggested increased deactivation with highly acidic catalysts, particularly the ones with higher Bronsted acid sites. Better stability was obtained with low activity dehydration catalysts. Several classes of molecular sieves were investigated as methanol dehydration catalysts (4). Molecular sieves offered a number of attractive features including a wide range of acid strengths, diverse architectures and channel connectivities, high active site density, well-investigated syntheses and characterization, and commercial availability in some cases. Many molecular sieves were confirmed to possess very high intrinsic activity for methanol dehydration to DME. However, no molecular sieve catalysts were found which provided a combination of activity, stability, and compatibility with methanol catalyst superior to the existing LPDME catalysts. Therefore, it was concluded that zeolites and related molecular sieves are not suitable for use in the LPDME process.

Additional R&D work resulted in an invention of aluminum phosphate as the dehydration catalyst (8). Due to its moderate acidity, the aluminum phosphate avoided coke formation and catalyst interaction problems, and provided an active and stable catalyst system. The system met the program targets under standard test process conditions of an initial methanol equivalent productivity of 28 gmole/kg catalyst - hr, a $CO_2$-free, carbon selectivity of 80% to DME, and stability of both catalysts equivalent to that of the methanol catalyst in the absence of the aluminum phosphate. Because the aluminum phosphate catalyst was not commercially available, a scale-up project was initiated with a commercial catalyst vendor to prepare a total of 800 pounds of aluminum phosphate to provide for two reactor charges and some additional material for testing (3). Eight 160 pound pilot batches of uncalcined catalyst were prepared. The reproducibility of the aluminum to phosphate (Al/P) ratio, an important physical property of the catalyst, from batch to batch was poor. Of these eight lots, two were qualified as yielding acceptable performance under the proposed LaPorte trial life test conditions. One of the batches was tested under conditions which allow direct comparison to the lab benchmark aluminum phosphate. This batch gave faster deactivation of the methanol catalyst than did the lab material, showing it to be of inferior quality. Two additional batches were prepared at half-dilution to alleviate any mixing problems which might have caused the poor reproducibility in the original eight batches. However, the Al/P ratio of both of these batches was high. One was tested, and shown to give unacceptable performance. All scale-up work on aluminum phosphate was suspended in November of 1998, as a commercially-available catalyst discussed below was identified which performed equivalently.

The deactivation rates of the methanol and the dehydration catalyst were correlated to reaction conditions. The correlations suggested a dependence of the deactivation rates on reaction conditions. The trends suggested by the correlations were confirmed by additional LPDME experiments. Better stability was obtained using the aluminum phosphate catalyst system by operating at favorable process conditions. The same improvement was also demonstrated with the gamma alumina catalyst system. However, there was a 10-20% trade-off in the productivity when shifting from the optimal conditions to the stable conditions for both the catalyst systems.

As the gamma alumina was off-the-shelf commercial material, it was chosen for the LaPorte test. A methanol catalyst to gamma alumina ratio of 95:5 by weight was found to be optimum for catalyst stability.

## DEMONSTRATION RUN PLAN

Kick-off meetings were held in November of 1996 to discuss plans to demonstrate DME synthesis at the AFDU in 1997. It was decided not to install equipment for product recovery but to rely on analysis instead to reduce capital expenditure. Since the timing of the demonstration was not critical relative to the schedule for potential implementation at Kingsport, it was later decided to postpone the run until 1998 to give Air Products' R&D more time to improve the catalyst system further and scale up the preparation and make batches for LaPorte. The most important objective for the run was to demonstrate improved stability over the 1991 LaPorte catalyst system. A key question was the run duration required at LaPorte to achieve this objective. Data from the 1988-89 four-month proof-of-concept run of the LPMEOH™ Process at the LaPorte AFDU (E-7) were statistically analyzed to answer that question. Standard errors for deactivation rates were calculated for different on-stream times as shown in Table 1. The results indicated that data for a minimum of ~15 days were needed to check stability, assuming the DME catalyst system has stability similar to the methanol system. Data for more than 3 weeks on-stream would be required to quantify the deactivation rate. Data for 60 days on-stream were enough to get a fairly accurate deactivation rate.

Due to delays experienced in producing the required 800 pounds of dehydration catalyst, the run was further postponed until 1999. The material produced at the larger scale appeared to be inferior to the laboratory-scale preparations. Air Products' R&D group followed up on leads based on their work and came up with the alternative gamma-alumina catalyst system as discussed in the Catalyst Selection section.

The DME run plan was finalized at a meeting with Air Products' R&D group in August of 1999 (see Table 2). It included 18 days of life study as well as a process variable study to evaluate LPDME at a lower space velocity, a higher superficial velocity, at stochiometric feed composition, and at a higher catalyst concentration. In addition, tracer studies were planned at two different velocities. Since the autoclave data indicated potential performance issues at higher concentration, it was decided to conduct the majority of run at a lower slurry concentration (35 wt%) compared to a typical LPMEOH™ run (40 wt%), and then check the higher concentration as part of the process variable study. The run plan was consistent with the objectives agreed upon with DOE. Revised material and energy balance calculation were conducted for the baseline condition (reactor space velocity = 6,000 gmole/hr-kg and slurry concentration = 35 wt%) as well as other process conditions. The methanol and DME productivities determined experimentally compared well with the productivities calculated by simulation as shown in Table 3. The simulations predicted maximum productivities based on fresh catalyst activity and two continuous stirred tank reactors (CSTRs) in series. Hence, they were somewhat higher than experimentally determined values.

A meeting was held with Synetix (formerly known as ICI Tracerco) to make plans for the hydrodynamic work. Radial gamma scans as well as radioactive tracer injections were planned. A fan type of source-detector arrangement was selected to improve reproducibility of the data. Detectors would be permanently installed to avoid alignment problems. The system would be checked out with empty tube and cold oil scans first, to determine if there were significant collimation issues with this type of arrangement. Gas and liquid as well as solid mixing were planned at two different conditions using three different tracer materials. As done in previous studies, argon would be used as the tracer in the gas phase, and manganese oxide ($Mn_2O_3$) fine particles would be used in the liquid for both liquid as well as methanol catalyst mixing. A

$Mn_2O_3$ doped gamma alumina sample was prepared by Air Products' R&D group and sent to Texas A&M for irradiation to check the gamma alumina mixing. The manganese loading measured by Texas A&M was very close to the target of 4.6 wt%. The particle size distribution of the tracer material was almost identical to that of the dehydration catalyst to be used in the trial.

Test Authorizations for the run are included in Appendix C.

## RESULTS AND DISCUSSION

Set-up activities for the DME run were conducted in September of 1999. The analytical and data acquisition system were set-up and checked out for the DME synthesis operating mode. Calibration of the NDG on the reactor was completed. A function test of the $CO_2$ removal system revealed no major problems; several pressure relief valves were removed and sent for maintenance, as the valves lifted at pressures below their respective set pressures. Set-up of the AFDU was completed and carbonyl burnout was started on 08 October 1999. A detailed Run Chronology for the entire run is given in Appendix D.

### Carbonyl Burnout

The reactor was loaded with oil and heated up on 08 October 1999 to start a carbonyl burnout. In addition to conducting a metal carbonyl survey, this also served as a hot function test with oil in the reactor and a syngas flow. Levels of iron and nickel carbonyl were both well below 10 parts per billion by volume (ppbv) by 10 October 1999. A summary of the carbonyl data is shown in Table 4. Syngas was then removed from the plant, and the plant was purged, cooled and drained in preparation of catalyst loading and reduction.

### Slurry Preparation

A 35 wt% oxide catalyst slurry was mixed in the 28.30 Prep Tank as per Test Authorization #56 (see Appendix C). The Prep Tank was charged with 1,747 pounds of Drakeol-10 mineral oil, 894 pounds of methanol catalyst, and 47 pounds of gamma alumina on 11 October 1999. The proportion of two catalysts corresponds to a 95:5 methanol to dehydration catalyst ratio by weight. The slurry was heated and agitated in the Prep Tank for about two hours prior to transfer to the reactor.

### Catalyst Reduction

Catalyst reduction began at 18:00 on 11 October 1999. The reduction gas (3 mole % $H_2$ in $N_2$) was set at 12,400 SCFH with the reactor pressure at 67 psig (Run # A13, Table 2). The heat up commenced at 18:50 hours and proceeded from 200°F to 464°F, as planned (see Figure 5). The reduction appeared normal, as shown in Figure 6, and reached a cumulative uptake very close to the theoretical maximum value of 2.68 SCF $H_2$/lb oxide. The reduction was essentially complete at 390°F or 17 hours on-stream. The uptake curve was always above the minimum curve, as the reduction in the bubble column was faster compared to the autoclave. NDG measurements indicated an average gas holdup of 36.8 vol% with a catalyst concentration of 40.1 wt% at 392°F during the reduction.

### Catalyst Life Study

Reduction was completed at 18:00 hours on 12 October 1999 and syngas was brought in the reactor at 19:30. The start-up with syngas was smooth and the operating conditions for Run 17.1 were reached within 12 hours after the introduction of syngas. The operating condition of Run 17.1 were: A reactor feed simulating Shell gas with a $H_2$/CO ratio of 0.46 and 3 mole % $CO_2$, a gas hourly space velocity (GHSV) of 6,000 sl/hr-kg, 750 psig reactor pressure, and 482° F average reactor temperature. Stable reactor temperature and pressure were successfully achieved. The reactor feed composition was then fine tuned and a mass balance period began

at 16:00 hours on 13 October 1999. The catalysts appeared to have good initial activity with DME and methanol productivity slightly exceeding expectations. This confirmed that the catalyst activation was proper. The DME production rate was calculated to be about 5 TPD compared to an expectation of 4.8 TPD, while the methanol production rate was 3.6 TPD vs 3.5 TPD expected. The DME and methanol production rates throughout the run are shown in Figure 7. The performance also met the productivity and selectivity targets. The initial methanol equivalent productivity exceeded the target of 28 gmole/kg catalyst - hr and the DME selectivity was at the target of 65% on a carbon basis (see Figure 8). Calculations based on differential pressure measurements indicated the catalyst concentration in the reactor was about 36 wt% with a gas holdup of 41-42 vol%.

A 13-hour interruption was experienced on 15 October 1999 due to a loss of syngas supply. The AFDU was put in a stand-by mode with $N_2$ flowing through the reactor at a lower temperature. The syngas became available at midnight on 15 October 1999 and the plant was brought back to the baseline condition in 8 hours. Operations continued at the baseline conditions and reached 320 hours on-stream as of 27 October 1999. Several material balances were generated for this period to track the catalyst performance. The methanol and DME productivities are shown in Figure 9. The methanol and dehydration catalyst activities, expressed as 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), are plotted in Figure 10. These normalized rate constants are estimated based on a reaction model developed from laboratory data (9, 10). After the expected initial aging, the catalysts appeared to be stabilizing, but there was significant scatter in the data. Gas chromatograhic as well as sampling problems were discovered with methanol analysis that required use of liquid balance for calculations, contributing to the scatter. The problems were corrected in two days. The deactivation rate appeared high: 0.08% per hr (2% per day) for methanol catalyst with a 0.05% per hr standard error and 0.03% per hr (0.6% per day) for dehydration catalyst with a 0.009% per hr standard error. It was decided to extend the aging run to get a better estimate on catalyst deactivation rate, which was the main objective. In order to stay within the budget, the process variable study was eliminated. An extensive tracer study at the baseline condition was still planned.

Operations continued at the baseline conditions into early November. At that point, 500 hours on-stream were completed. The AFDU data appeared to follow the autoclave trends, with somewhat higher conversions than the autoclave. The higher conversions were perhaps due to the multiple-CSTR effect in the bubble column. The methanol productivity level remained relatively constant, while the DME productivity showed a slight decline. The scatter in data decreased significantly after the GC and the sampling problems were resolved (350 hours on-stream). The deactivation rate for both the catalysts was estimated at 0.7% per day. This is lower than the 1.2% per day autoclave rate and only slightly higher than 0.5% per day observed during the first three weeks of the 4-month proof-of-concept run of the LPMEOH™ Process at the LaPorte in 1988/89. Due to the initial scatter, the standard error was still high for the methanol catalyst: 0.34% per day.

Tracer Study

As planned, an extensive tracer study was conducted at the baseline condition to evaluate gas, liquid and solid mixing. Synetix personnel started setting up on 02 November 1999. Due to a delay in obtaining the radioactive materials, the study was split over two days (03 and 04 November 1999). Detectors were set-up at various locations outside the reactor as shown in Figure 4. Sets of four detectors at 90° angles were set-up at seven different heights. In addition, detectors were set-up at the reactor inlet, reactor outlet, vapor space near the reactor

top, and recycle feed line. An extra detector was set-up on the reactor outlet piping to measure gas velocity. During the liquid injection, a detector was set-up at the liquid injection nozzle to monitor the injection pulse.

A vapor residence time distribution study was initiated on 03 November 1999 by injecting Argon-41 into the inlet gas line and monitoring its progress through the reactor. The first injection indicated a bad detector. The detector was repaired and seven additional injections were made. Time was allowed between injections for the recycle to stabilize. Excellent pulses were obtained at the inlet and sharp responses were observed at other locations.

The liquid and solid mixing study was conducted on 4 November. Five injections of radioactive $Mn_2O_3$ mixed in Drakeol-10 were made in the reactor slurry to study liquid phase mixing. All the injections were made at the same location injections to evaluate variability with time. The location used was nozzle N2-4.5" from wall. Data periods following the injections were varied during the study to look for longer-term trends as shown below:

Injection 1: 5 minutes.
Injection 2: Not enough intensity, data did not get saved.
Injection 3: 10 minutes.
Injection 4: 15 minutes.
Injection 5: 60 minutes.

Four injections of a slurry of radioactive $Mn_2O_3$ doped gamma alumina in Drakeol-10 were made to look at gamma alumina mixing. The injections were made at four different locations: (1) nozzle N2-4.5" from wall, (2) nozzle N2-wall, (3) nozzle N1-4.5" from wall, and (4) nozzle N1-wall. Both short-term and long-term observations of irradiated dehydration catalyst suggested no alumina settling in the reactor. A post-run inspection of the reactor bottom head did not show any settled catalyst around the sparger, in contrast to the 1991 DME run when significant settled catalyst was found at the bottom. The tracer data will be analyzed with help of personnel from Washington University as part of the Hydrodynamic Program with DOE.

A decision was made to continue the life study after the tracer work instead of moving on to the high velocity condition. It was considered to be more important to get additional life data at the baseline condition instead of more tracer data at a higher velocity. The catalyst life study was continued for two more days after completion of the tracer work. Totally, data for approximately 600 hours on-stream were obtained. The additional data resulted in improved statistics with no change in the deactivation rates. Following a shut-down test conducted to get a better estimate of the gas holdup, the plant was shut down at 10:00 hours on 06 November 1999. The slurry was cooled down under a $N_2$ flow and then drained from the reactor.

Mass Balance

A run time table, which provides a cross-reference between run numbers, actual times, and on-stream times, is given in Table 5. Mass balance was performed for 29 different data periods during the run. A summary of the results is given in Table 6. Detailed data for each data period are included in Appendix E.

Corrections were applied to flow meters which are generally inaccurate in their particular service. The recycle flow was calculated by the balance of dry feed gas vs. fresh make-up gas plus high-pressure $H_2$. Known chemistry was used along with measured gas concentrations to

calculate reactor effluent flow and composition. The 22.10 seperator vapor flow was calculated using $N_2$ balance on the 22.10 seperator vapor plus Liquid product plus the 22.11 degasser purge vs. Reactor effluent. Elemental and mass balances are summarized in Table 7. Over the run, the carbon, oxygen, and nitrogen balance averaged around 100%, while the hydrogen balance was about 94%.

Catalyst Deactivation Rates Estimates

To evaluate the deactivation rates of the LPDME Process, it is useful to look at historic data from prior tests of the LPMEOH™ Process for reference. The data for the LPMEOH™ Process are summarized in Table 8. A baseline deactivation rate of 1.2% per day is typically observed in the autoclave, while a deactivation rate of 0.4% per day was observed in the 120-day run at LaPorte in 1988/89. At the Kingsport LPMEOH™ Process Demonstration Unit, deactivation in the range of 1.0 to 3.4% per day was estimated in Campaign # 1, and it improved to 0.2 to 1.3% per day in the second campaign. The Kingsport catalysts had evidence of poisoning. The LPDME data from the autoclave indicated a deactivation rate of 1.2% per day, which was comparable to the LPMEOH™ Process in the autoclave. However, the stability depended on reaction conditions, and the rates were higher at more severe conditions. A key question to be answered was whether the deactivation rate would be lower than 1.2% per day at LaPorte and how would it compare with the LPMEOH™ deactivation rate at LaPorte.

The deactivation rates from the current run are compared with LPDME autoclave data and LPMEOH™ LaPorte data in Table 9. The deactivation rate was lower for both the catalysts at LaPorte compared to the autoclave, but it was higher than LPMEOH™ LaPorte. There was significantly higher scatter in the LPDME LaPorte data compared to the other two sets of data for the methanol synthesis catalyst as indicated by the standard errors. In contrast, the standard errors are low and comparable for the dehydration catalysts. Thus, the deactivation rate of 0.65% per day for the dehydration catalyst is a reliable estimate. In addition, the laboratory data indicate a similar deactivation rates for both the catalyst. Hence, the 0.7% per day deactivation rate for the methanol catalyst may also be more accurate than the standard errors indicate.

Gas Holdup and Catalyst Concentration Estimates

The average gas holdup in the reactor was estimated using both DP measurements as well as NDG readings. The estimates are shown in Figure 11. The holdup based on DP measurements, which is more accurate, was in the range of 41 to 44 vol%, while the NDG readings indicated a higher holdup (49 to 55 vol%). The NDG based holdup is typically higher than actual as it is an integrated measurement through the centerline with equal weighting for the center core as well as outer annulus, and generally the holdup is higher at the center. Two shut-down tests conducted during the run indicated a holdup of about 36 vol%.

Catalyst concentrations in the reactor were estimated based on the DP as well as the NDG readings and are shown in Figure 12. The more accurate DP estimate indicated the catalyst concentration in the 36 to 37 wt% range, comparable to the planned concentration of 35 wt%.

Spent Catalyst Analysis

The activity of the methanol and dehydration catalysts in the spent slurry from the run was checked in a lab 300 cubic centimeter (cc) autoclave reactor under the same conditions as

used at LaPorte (482$^{o}$F, 750 psig, 6,000 GHSV, Shell gas). The goal was to see if the results would back up the plant observation. It would also provide some information about the reactor model of the LaPorte bubble column reactor. The experiment showed that the methanol rate constant was 2.6 ($k_m$)and the dehydration rate constant 17.7 ($k_d$). These numbers are comparable to rate constants of 2.8 and 19.9 obtained at the end of a laboratory run at the LaPorte trial conditions. With on-stream times of 380 hours for the lab run vs. 600 hours for the LaPorte run, it substantiates the conclusion that the deactivation rate was lower at LaPorte. The DME selectivity was 59.5%, very close to that observed in the laboratory run at the LaPorte trial conditions, 60.2%. This indicates that the catalyst ratio in this slurry sample was indeed representative. In contrast to the rate constants measured in the lab from the LaPorte spent slurry ($k_m$ = 2.6, $k_d$ = 17.7), the rate constants calculated using one CSTR model from the LaPorte data at the end of the run were 3.3 for methanol synthesis and 19.5 for methanol dehydration. The discrepancy between the lab and LaPorte results indicates that the LaPorte reactor is more than 1 CSTR. Thus, the LaPorte bubble column reactor outperforms the lab autoclave (1 CSTR).

X-ray diffraction (XRD) results of the spent methanol catalyst showed that the copper (Cu) and zinc oxide (ZnO) crystallite size of the LaPorte spent methanol catalyst is 88.9 and 54.7 Å, respectively. Both were smaller than the crystallites from a spent lab sample with similar on-stream time but were within the expected range (see Table 10). This indicates no unexpected sintering at LaPorte. Elemental analysis of the spent catalysts was also performed. The results are shown in the Table 10. The Al/Cu ratio of the spent catalysts is very close to that expected from the catalyst mixture used in the run (95% methanol catalyst and 5% gamma alumina). Therefore, the spent sample was representative of the slurry in the reactor. It also suggests that the two catalysts were well mixed in the reactor, in agreement with the tracer-study observation. The concentrations of minor components in the spent catalysts, especially those known as methanol catalyst poisons, were very close to those in the fresh methanol catalyst, indicating a poison-free environment.

Economics for Production of DME by the LPDME Process

During a Project Review Meeting on 12-13 January 2000, the preliminary results from the LPDME design verification test were presented to the Clean Coal Technology project team. Table 11 was presented at this meeting, and contains a comparison of the performance of the LPDME catalyst system during the Design Verification Test Run with results from the recent work in the laboratory autoclave and the economic targets for the LPDME Process. The results from the laboratory work had indicated that the targets for catalyst productivity and life can be met at lower selectivity to DME.

The results from the Design Verification Test Run were applied to a cost estimate for a commercial-scale LPDME plant. The case that was considered was the retrofit of an existing Texaco gasifier in China which is currently used in the production of ammonia. The results of this initial work were that, for this syngas (35 vol% $H_2$, 51 vol% CO, 13 vol% $CO_2$) available at 500 psig, the DME costs are competitive with LPG in China ($7 - $8 per million Btu's). The cost of syngas assumes constant utilization for each plant size, and economies of scale are indicated by changes in the conversion and separation costs. The results from this screening study are presented in Figure 13. The target DME cost can likely be achieved at larger plant sizes by extrapolating the costs to the 1,100 TPD production rates. The sensitivity of the cost of production of DME to the life of the LPDME catalyst system was shown for the results from

LaPorte (0.7% per day deactivation), as well as the impact of changes to the value for catalyst life on the process economics.

As stated in the 30 July 1997 letter which transmitted Air Products' recommendation to proceed with LPDME Design Verification Testing, the basis for commercialization of the technology must come from:

1) the results from testing of the LPDME catalyst system at the LaPorte AFDU; and
2) the reactor performance (methanol catalyst activity and life, hydrodynamics, and heat transfer) from the LPMEOH™ Demonstration Unit.

Ongoing activities at the LPMEOH™ Demonstration Unit have been focused on determining the causes for the varying rates of catalyst deactivation which have been calculated over the initial three years of operation. Given the remaining time in that program, the Clean Coal Technology Program participants decided that testing of the LPDME Process will not be performed at the LPMEOH™ Demonstration Unit. The available resources should be directed toward improving the catalyst performance for the LPMEOH™ Process, without which the commercialization of the LPDME Process becomes more difficult.

## CONCLUSIONS AND RECOMMENDATIONS

- Operation of the LPDME Process with improved catalyst life was successfully demonstrated on a 10 TPD scale, using commercially produced catalysts. The AFDU was operated for 25 days to compare catalyst aging in a pilot-scale slurry bubble column with that in a laboratory autoclave. The catalyst life study was extended instead of a planned process variable study to obtain additional data on catalyst aging. Hydrodynamic information was obtained at the baseline conditions by conducting a detailed survey of the reactor with radioactive tracer injections.

- The catalysts were activated successfully with an expected hydrogen uptake. The initial productivities of methanol and DME were higher than the laboratory, perhaps due to the multiple-CSTR effect in the bubble column. The DME production rate started at 5.1 TPD and declined to 4.1 TPD in 25 days on-stream while the methanol production showed a scatter within 3.1 to 3.8 TPD range through the run. The scatter in data decreased significantly after a GC problem and a sampling problem were discovered and resolved (350 hours on-stream).

- The deactivation rate for both the catalysts was estimated at 0.7% per day. This is lower than the 1.2% per day rate observed for both the catalyst in the autoclave. However, the rate is slightly higher than 0.5% per day rate achieved for the LPMEOH™ process after 3 weeks of operation at LaPorte. As the stability of the catalyst was better than the lab, further developmental work is recommended. Also, economic studies are suggested to determine the impact of higher catalyst costs compared to those for the LPMEOH™ process. The methanol productivity level remained relatively constant throughout the run, while the DME productivity showed a slight decline, consistent with laboratory observations. The standard error for the methanol catalyst deactivation rate was high (0.25% per day) due to scatter in methanol data. The dehydration catalyst activity data are much tighter with a standard error of 0.06% per day.

- Spent catalysts from the run showed expected properties. The activity results of the spent catalysts supported the stable performance observed during the run. XRD analysis indicated that the size of the Cu and ZnO crystallites in the spent methanol catalyst were in the expected range indicating no sintering. Elemental analysis of the spent catalyst samples showed a typical composition, with no evidence of accumulation of catalyst poisons.

- The reactor operated hydrodynamically stable with uniform temperature profile and gas holdups. Differential pressure measurements indicated about 42 vol% gas holdup and 36 wt% catalyst concentration.

- Mass balance calculations showed good closure.

- The initial start-up was very quick with the baseline condition reached in 12 hours after the introduction of syngas. A re-start after a syngas outage only took 4 hours. This demonstrates the ease and flexibility of the slurry technology.

- Gas, liquid, and solid phase mixing was studied at the baseline conditions using radioactive materials. A large amount of data were collected using 34 detectors around

the reactor. Several repeat injections were made during the gas and liquid injections to evaluate variability with time. Manganese oxide doped gamma alumina was injected at four different locations to look at the dehydration catalyst mixing. Both short-term and long-term observations of irradiated dehydration catalyst suggest no alumina settling in the reactor. A post-run inspection of the reactor bottom head did not show any settled catalyst around the sparger, in contrast to the 1991 DME run at the AFDU when significant settled catalyst was found at the bottom. The tracer data will be analyzed with help of personnel from Washington University as part of the Hydrodynamic Program with DOE.

- The oxygenates system was operated after a 4-year gap; the operations were smooth.

- Approximately 27,200 gallons of liquid product (89 wt% methanol, 7 wt% DME) was collected during the run.

- This demonstration represents a significant step forward in the development of the LPDME process. The 0.7% per day aging rate achieved in the AFDU is a large improvement over the 4% per day autoclave deactivation of the previous catalyst system. Further R&D and economic studies as part of ongoing or future programs are suggested based upon the results of the DVT activities to continue the developmental work. A report on the market analysis for DME and the economics of the LPDME Process is planned.

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Table 1

**Analysis of 4-month LPMEOH™ Life Run at LaPorte**

| Days On-stream | Deact. Rate (a) of $k_0$, %/day | 2 times Std Error of (a) | 2 times % Std Error of (a) |
|---|---|---|---|
| 113 | -0.38 | 0.01 | 3 |
| 60 | -0.36 | 0.04 | 11 |
| 45 | -0.29 | 0.06 | 21 |
| 30 | -0.44 | 0.10 | 24 |
| 22 | -0.52 | 0.18 | 34 |
| 15 | -0.25 | 0.26 | 106 |
| 7 | 0.44 | 0.59 | 132 |

| Days On-stream | Deact. Rate (b) of Productivity, %/day | # of Days needed to see a 2% drop |
|---|---|---|
| 113 | -0.17 | 12 |

Table2

| | | | | | | FINAL RUN PLAN | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | DME RUN AT THE LAPORTE AFDU - OCTOBER / NOVEMBER 1999 | | | | | |
| | | | | | | | | | | | |
| | | | | Reactor | Reactor | | | Inlet | | Methanol | DME |
| Run No. | No. of | Comment | Gas Type | Pressure | Temp. | Space Vel. | React. Fd. | Sup. Vel. | Slurry wt% | Production | Production |
| | Days | | | psia | deg F | sl/kg-hr | lbmol/hr | ft/sec | oxide | TPD | TPD |
| | | | | | | | | | | | |
| | | | | | | | | | | | |
| MEOH + DEHYDRATION CATALYST (% MEOH CAT = 95%) | | | | | | | | | | | |
| | 1 | Catalyst Loading | | | | | | | | | |
| AF-A13 | 1 | Reduction | 3% H2 in N2 | 82 | | 800 | 32 | 0.62 | 35 | | |
| AF-R17.1 | 18 | Life Study + Tracer1 (on-stream Day 18) | Shell | 765 | 482 | 6000 | 248 | 0.56 | 35.9 | 3.5 | 4.8 |
| AF-R17.2 | 1.5 | Low Space Velocity | Shell | 765 | 482 | 3100 | 128 | 0.29 | 34.6 | 1.1 | 4.2 |
| AF-R17.3 | 1.5 | Stoch. Feed | 1:1 H2/CO | 765 | 482 | 6000 | 248 | 0.56 | 35.1 | 8.5 | 4.0 |
| AF-R17.4 | 2.5 | High Velocity + Tracer2 (on-stream Day 23) | Shell | 765 | 482 | 8000 | 331 | 0.75 | 36.5 | 5.0 | 5.1 |
| AF-R17.5 | 1.5 | High Concentration | Shell | 765 | 482 | 8000 | 331 | 0.75 | 40 | 5.0 | 5.1 |
| TOTAL | 27 | | | | | | | | | | |

Table 3

| Comparison of Experimental and Simulated Productivities (gmole/hr kg) | | | | |
|---|---|---|---|---|
| | Methanol Productivity | | DME Productivity | |
| | Experimental | Simulation | Experimental | Simulation |
| Baseline | 9.5 | 9.9 | 8.9 | 9.4 |
| Low Velocity | 3.1 | 3 | 6.3 | 8.1 |
| Stoichiometric Feed | 21.7 | 23.6 | 8.2 | 7.7 |
| High Velocity | 13.7 | 14.1 | 8.5 | 9.8 |
| High Concentration | 13.7 | 14.1 | 8.5 | 9.8 |

Table 4

**Carbonyl Burn-Out Data (ppbv)**
**Oct'99 LPMEOH/DME Run**

| 8 Oct. 1999 | Time | $Ni(CO)_4$ | $Fe(CO)_5$ |
|---|---|---|---|
| SP # 4 | 16:33 | N.D. | 1.2 |
| SP # 3 | 16:40 | N.D. | 1.0 |
| SP # 4 | 16:47 | N.D. | 1.0 |
| SP # 3 | 16:55 | N.D. | 0.6 |
| SP # 4 | 17:05 | N.D. | 0.7 |
| SP # 3 | 17:12 | N.D. | 0.3 |
| | | | |
| 9 Oct. 1999 | Time | $Ni(CO)_4$ | $Fe(CO)_5$ |
| SP # 4 | 07:52 | N.D. | 0.5 |
| SP # 3 | 08:02 | N.D. | 0.2 |
| SP # 4 | 08:24 | N.D. | 0.5 |
| SP # 3 | 08:47 | N.D. | 0.3 |
| SP # 4 | 09:30 | N.D. | 0.4 |
| SP # 3 | 14:45 | N.D. | 0.3 |
| SP # 4 | 15:00 | | |
| SP # 3 | 15:05 | N.D. | 0.2 |
| SP # 4 | 15:10 | N.D. | 0.4 |
| SP # 3 | 15:20 | N.D. | 0.3 |
| SP # 4 | 15:30 | N.D. | 0.4 |
| | | | |
| 10 Oct. 1999 | Time | $Ni(CO)_4$ | $Fe(CO)_5$ |
| SP # 3 | 07:30 | N.D. | 0.3 |
| SP # 4 | 07:50 | N.D. | 0.4 |
| SP # 3 | 07:55 | N.D. | 0.3 |
| SP # 4 | 08:03 | N.D. | 0.5 |

Note: SP # 4 - Reactor Feed
SP # 3 - Reactor Effluent
N. D. - Not Detected

Table 5

| | | | | Run Time Table | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | | | | | | |
| | LPDME-99: | | Activation Started | | 11-Oct-99 | 18:00 | | |
| | | | Syngas Started | | 12-Oct-99 | 20:00 | | |
| | | | | | | | | |
| | **Avg. Time** | **Start** | **Start** | **Time** | **End** | **End** | **Time** | **Time** |
| **Run No.** | **On-stream,** | **Date** | **Time** | **On-stream,** | **Date** | **Time** | **On-stream,** | **Period,** |
| | **Hrs.** | | | **Hrs.** | | | **Hrs.** | **Hrs.** |
| | | | | | | | | |
| R17.1A | 29 | 13-Oct | 16:00 | 20 | 14-Oct | 10:00 | 38 | 18 |
| R17.1B | 47 | 14-Oct | 10:00 | 38 | 15-Oct | 4:00 | 56 | 18 |
| R17.1C | 59.5 | 15-Oct | 4:00 | 56 | 15-Oct | 11:00 | 63 | 7 |
| R17.1D | 95 | 16-Oct | 10:00 | 86 | 17-Oct | 4:00 | 104 | 18 |
| R17.1E | 114.5 | 17-Oct | 7:00 | 107 | 17-Oct | 22:00 | 122 | 15 |
| R17.1F | 132 | 17-Oct | 22:00 | 122 | 18-Oct | 18:00 | 142 | 20 |
| R17.1G | 156 | 18-Oct | 23:00 | 147 | 19-Oct | 17:00 | 165 | 18 |
| R17.1H | 174 | 19-Oct | 17:00 | 165 | 20-Oct | 11:00 | 183 | 18 |
| R17.1I | 192 | 20-Oct | 11:00 | 183 | 21-Oct | 5:00 | 201 | 18 |
| R17.1J | 210 | 21-Oct | 5:00 | 201 | 21-Oct | 23:00 | 219 | 18 |
| R17.1K | 240 | 22-Oct | 11:00 | 231 | 23-Oct | 5:00 | 249 | 18 |
| R17.1L | 258 | 23-Oct | 5:00 | 249 | 23-Oct | 23:00 | 267 | 18 |
| R17.1M | 276 | 23-Oct | 23:00 | 267 | 24-Oct | 17:00 | 285 | 18 |
| R17.1N | 293 | 24-Oct | 17:00 | 285 | 25-Oct | 9:00 | 301 | 16 |
| R17.1O | 313 | 25-Oct | 12:00 | 304 | 26-Oct | 6:00 | 322 | 18 |
| R17.1P | 344.5 | 27-Oct | 0:00 | 340 | 27-Oct | 9:00 | 349 | 9 |
| R17.1Q | 365 | 27-Oct | 17:00 | 357 | 28-Oct | 9:00 | 373 | 16 |
| R17.1R | 379 | 28-Oct | 9:00 | 373 | 28-Oct | 21:00 | 385 | 12 |
| R17.1S | 391 | 28-Oct | 21:00 | 385 | 29-Oct | 9:00 | 397 | 12 |
| R17.1T | 411 | 29-Oct | 14:00 | 402 | 30-Oct | 8:00 | 420 | 18 |
| R17.1U | 429 | 30-Oct | 8:00 | 420 | 31-Oct | 2:00 | 438 | 18 |
| R17.1V | 447 | 31-Oct | 2:00 | 438 | 31-Oct | 20:00 | 456 | 18 |
| R17.1W | 466.5 | 31-Oct | 23:00 | 459 | 1-Nov | 14:00 | 474 | 15 |
| R17.1X | 483 | 1-Nov | 14:00 | 474 | 2-Nov | 8:00 | 492 | 18 |
| R17.1Y | 499.5 | 2-Nov | 8:00 | 492 | 2-Nov | 23:00 | 507 | 15 |
| R17.1Z | 517 | 2-Nov | 23:00 | 507 | 3-Nov | 19:00 | 527 | 20 |
| R17.1AA | 537.5 | 4-Nov | 0:00 | 532 | 4-Nov | 11:00 | 543 | 11 |
| R17.1BB | 563 | 4-Nov | 22:00 | 554 | 5-Nov | 16:00 | 572 | 18 |
| R17.1CC | 581 | 5-Nov | 16:00 | 572 | 6-Nov | 10:00 | 590 | 18 |

Table 6 (p. 1 of 2)

| | | | | Summary of Results for LPDME-99 Demonstration Run at LaPorte | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Run No. | Time On-stream, Hrs | Temperature (average), deg F | Pressure, psig | Space Velocity, sl/kg-hr | Superficial Gas Vel. (Inlet), ft/sec | Slurry Conc. based on NDG, wt % | Slurry Conc. based on DP, wt % | Gas Holdup based on NDG, vol % | Gas Holdup based on DP, vol % |
| AF-R17.1a | 29 | 481.7 | 748.7 | 5889 | 0.56 | 39.1 | 35.8 | 48.7 | 41.2 |
| AF-R17.1b | 47 | 481.7 | 748.6 | 5850 | 0.56 | 39.7 | 36.1 | 49.9 | 41.9 |
| AF-R17.1c | 59.5 | 481.7 | 748.0 | 5919 | 0.56 | 39.9 | 36.3 | 50.3 | 42.3 |
| AF-R17.1d | 95 | 481.6 | 748.9 | 5837 | 0.55 | 40.2 | 36.4 | 50.9 | 42.5 |
| AF-R17.1e | 114.5 | 481.6 | 749.1 | 5828 | 0.55 | 40.5 | 36.5 | 51.4 | 42.9 |
| AF-R17.1f | 132 | 481.5 | 751.8 | 5933 | 0.56 | 40.8 | 37.1 | 52.1 | 44.1 |
| AF-R17.1g | 156 | 481.5 | 752.5 | 5897 | 0.56 | 40.8 | 37.1 | 51.9 | 44.2 |
| AF-R17.1h | 174 | 481.5 | 752.2 | 5866 | 0.55 | 40.8 | 37.1 | 52.0 | 44.2 |
| AF-R17.1i | 192 | 481.6 | 750.5 | 5788 | 0.55 | 40.8 | 36.7 | 52.0 | 43.3 |
| AF-R17.1j | 210 | 481.7 | 749.9 | 5770 | 0.55 | 40.9 | 36.6 | 52.2 | 43.1 |
| AF-R17.1k | 240 | 481.6 | 749.2 | 5744 | 0.55 | 41.0 | 36.4 | 52.4 | 42.7 |
| AF-R17.1l | 258 | 481.6 | 750.2 | 5808 | 0.55 | 41.4 | 36.6 | 53.1 | 43.0 |
| AF-R17.1m | 276 | 481.6 | 752.9 | 6081 | 0.57 | 41.7 | 37.0 | 53.5 | 44.0 |
| AF-R17.1n | 293 | 481.6 | 753.5 | 6097 | 0.58 | 41.9 | 37.1 | 53.8 | 44.2 |
| AF-R17.1o | 313 | 481.6 | 751.3 | 6052 | 0.57 | 41.8 | 36.9 | 53.9 | 43.6 |
| AF-R17.1p | 344.5 | 481.6 | 750.8 | 6016 | 0.57 | 41.8 | 36.9 | 53.9 | 43.6 |
| AF-R17.1q | 365 | 481.5 | 752.4 | 5964 | 0.56 | 42.1 | 36.7 | 54.3 | 43.3 |
| AF-R17.1r | 379 | 481.5 | 751.2 | 5949 | 0.56 | 42.0 | 36.7 | 54.2 | 43.2 |
| AF-R17.1s | 391 | 481.4 | 753.0 | 6027 | 0.57 | 41.9 | 36.9 | 54.9 | 43.7 |
| AF-R17.1t | 411 | 481.3 | 750.9 | 5991 | 0.57 | 42.1 | 36.3 | 54.5 | 42.4 |
| AF-R17.1u | 429 | 481.3 | 751.6 | 6014 | 0.57 | 42.3 | 36.4 | 54.7 | 42.6 |
| AF-R17.1v | 447 | 481.3 | 752.5 | 5947 | 0.56 | 42.3 | 36.5 | 54.7 | 42.8 |
| AF-R17.1w | 466.5 | 481.5 | 752.2 | 5876 | 0.56 | 42.1 | 36.5 | 54.4 | 42.7 |
| AF-R17.1x | 483 | 481.4 | 751.8 | 5810 | 0.55 | 42.1 | 36.3 | 54.4 | 42.3 |
| AF-R17.1y | 499.5 | 481.3 | 751.8 | 5874 | 0.56 | 42.4 | 36.4 | 54.8 | 42.6 |
| AF-R17.1z | 517 | 481.3 | 751.5 | 5911 | 0.56 | 42.5 | 36.6 | 55.0 | 42.9 |
| AF-R17.1aa | 537.5 | 481.5 | 750.0 | 5819 | 0.55 | 42.5 | 36.3 | 55.0 | 42.3 |
| AF-R17.1bb | 563 | 481.4 | 749.1 | 5819 | 0.55 | 42.2 | 35.9 | 54.6 | 41.5 |
| AF-R17.1cc | 581 | 481.4 | 749.7 | 5861 | 0.56 | 42.5 | 36.1 | 55.0 | 41.8 |
| Shut-down Test #1 | 397 | | | | | | | 36.4 | |
| Shut-down Test #2 | 590 | | | | | | | 36.4 | |

Table 6 (p. 2 of 2)

| | | | | | Summary of Results for LPDME-99 Demonstration Run at LaPorte | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Run No. | Time On-stream, Hrs | CO Conversion to MeOH (%) | CO Conversion to DME (%) | Methanol Production (Ton/day) | DME Production (Ton/day) | Methanol Productivity (gmole/kg-hr) | DME Productivity (gmole/kg-hr) | DME Selectivity on Carbon Basis (%) | Methanol Equv. Productivity (gmole/kg-hr) | MeOH Rate Constant (1-CSTR) | DME Rate Constant (1-CSTR) |
| AF-R17.1a | 29 | 6.31 | 17.59 | 3.84 | 5.14 | 10.63 | 9.87 | 65.02 | 30.37 | 5.06 | 26.41 |
| AF-R17.1b | 47 | 5.87 | 16.42 | 3.65 | 4.89 | 10.08 | 9.39 | 65.09 | 28.86 | 5.20 | 24.38 |
| AF-R17.1c | 59.5 | 5.31 | 16.09 | 3.35 | 4.86 | 9.25 | 9.34 | 66.89 | 27.94 | 3.95 | 24.41 |
| AF-R17.1d | 95 | 4.94 | 15.02 | 3.11 | 4.54 | 8.60 | 8.72 | 66.96 | 26.05 | 3.52 | 22.46 |
| AF-R17.1e | 114.5 | 5.16 | 14.72 | 3.25 | 4.44 | 8.98 | 8.54 | 65.54 | 26.07 | 3.83 | 21.91 |
| AF-R17.1f | 132 | 5.02 | 14.43 | 3.22 | 4.44 | 8.91 | 8.53 | 65.71 | 25.97 | 3.66 | 21.68 |
| AF-R17.1g | 156 | 5.50 | 14.70 | 3.47 | 4.44 | 9.58 | 8.53 | 64.05 | 26.65 | 4.17 | 21.90 |
| AF-R17.1h | 174 | 5.63 | 14.80 | 3.53 | 4.44 | 9.75 | 8.54 | 63.67 | 26.83 | 4.30 | 22.00 |
| AF-R17.1i | 192 | 5.13 | 15.27 | 3.17 | 4.52 | 8.76 | 8.69 | 66.49 | 26.13 | 3.27 | 22.01 |
| AF-R17.1j | 210 | 5.85 | 15.10 | 3.59 | 4.44 | 9.93 | 8.54 | 63.25 | 27.01 | 4.20 | 21.84 |
| AF-R17.1k | 240 | 5.41 | 15.31 | 3.29 | 4.47 | 9.11 | 8.59 | 65.36 | 26.29 | 3.24 | 22.10 |
| AF-R17.1l | 258 | 5.01 | 14.75 | 3.13 | 4.42 | 8.65 | 8.49 | 66.25 | 25.64 | 3.24 | 21.63 |
| AF-R17.1m | 276 | 5.27 | 13.98 | 3.42 | 4.35 | 9.46 | 8.37 | 63.88 | 26.20 | 3.30 | 21.23 |
| AF-R17.1n | 293 | 5.79 | 13.64 | 3.77 | 4.25 | 10.41 | 8.17 | 61.10 | 26.75 | 3.84 | 20.37 |
| AF-R17.1o | 313 | 5.48 | 13.90 | 3.52 | 4.28 | 9.74 | 8.23 | 62.84 | 26.20 | 3.27 | 20.86 |
| AF-R17.1p | 344.5 | 5.92 | 13.40 | 3.79 | 4.12 | 10.49 | 7.91 | 60.14 | 26.31 | 3.69 | 19.56 |
| AF-R17.1q | 365 | 5.59 | 14.21 | 3.59 | 4.37 | 9.91 | 8.40 | 62.89 | 26.71 | 3.83 | 20.74 |
| AF-R17.1r | 379 | 5.63 | 14.34 | 3.58 | 4.37 | 9.90 | 8.41 | 62.94 | 26.72 | 3.61 | 20.84 |
| AF-R17.1s | 391 | 5.40 | 13.74 | 3.52 | 4.29 | 9.73 | 8.25 | 62.91 | 26.22 | 3.56 | 20.19 |
| AF-R17.1t | 411 | 5.42 | 13.95 | 3.49 | 4.31 | 9.65 | 8.28 | 63.18 | 26.20 | 3.43 | 20.43 |
| AF-R17.1u | 429 | 5.54 | 14.00 | 3.57 | 4.33 | 9.87 | 8.32 | 62.75 | 26.50 | 3.58 | 20.92 |
| AF-R17.1v | 447 | 5.42 | 14.09 | 3.45 | 4.30 | 9.55 | 8.27 | 63.41 | 26.09 | 3.32 | 20.24 |
| AF-R17.1w | 466.5 | 5.45 | 14.32 | 3.41 | 4.29 | 9.42 | 8.25 | 63.66 | 25.92 | 3.23 | 20.52 |
| AF-R17.1x | 483 | 5.56 | 14.17 | 3.46 | 4.22 | 9.56 | 8.12 | 62.95 | 25.80 | 3.40 | 20.04 |
| AF-R17.1y | 499.5 | 5.58 | 13.93 | 3.51 | 4.20 | 9.71 | 8.08 | 62.47 | 25.86 | 3.38 | 19.77 |
| AF-R17.1z | 517 | 5.56 | 13.74 | 3.52 | 4.17 | 9.72 | 8.01 | 62.23 | 25.74 | 3.24 | 19.65 |
| AF-R17.1aa | 537.5 | 5.52 | 14.06 | 3.42 | 4.18 | 9.46 | 8.03 | 62.94 | 25.53 | 3.24 | 19.88 |
| AF-R17.1bb | 563 | 5.73 | 14.03 | 3.55 | 4.16 | 9.80 | 8.00 | 62.01 | 25.81 | 3.40 | 19.87 |
| AF-R17.1cc | 581 | 5.74 | 13.70 | 3.59 | 4.11 | 9.92 | 7.89 | 61.41 | 25.70 | 3.39 | 19.50 |

Table 7

| | | | Elemental / Mass Balance (based on Reactor Inlet) Summary for LPDME-99 | | | |
|---|---|---|---|---|---|---|
| | | | | | | |
| **Run No.** | **Time On-stream, Hrs** | **C, %** | **H, %** | **O, %** | **N, %** | **Total Mass, %** |
| AF-R17.1a | 29 | 95.8 | 91.6 | 97.1 | 99.9 | 96.4 |
| AF-R17.1b | 47 | 100.0 | 94.1 | 101.2 | 100.8 | 100.5 |
| AF-R17.1c | 59.5 | 99.5 | 90.2 | 100.9 | 101.2 | 100.0 |
| AF-R17.1d | 95 | 99.4 | 88.1 | 101.1 | 107.8 | 100.1 |
| AF-R17.1e | 114.5 | 99.6 | 92.1 | 100.8 | 101.8 | 100.1 |
| AF-R17.1f | 132 | 99.4 | 89.0 | 100.8 | 102.0 | 99.9 |
| AF-R17.1g | 156 | 99.5 | 92.7 | 100.5 | 101.5 | 99.8 |
| AF-R17.1h | 174 | 103.6 | 103.7 | 103.3 | 99.9 | 103.4 |
| AF-R17.1i | 192 | 99.2 | 91.8 | 99.3 | 99.8 | 99.0 |
| AF-R17.1j | 210 | 101.3 | 97.2 | 101.7 | 100.2 | 101.4 |
| AF-R17.1k | 240 | 100.9 | 94.7 | 101.2 | 100.0 | 100.9 |
| AF-R17.1l | 258 | 100.3 | 93.7 | 101.0 | 100.3 | 100.5 |
| AF-R17.1m | 276 | 99.2 | 91.0 | 100.1 | 100.1 | 99.4 |
| AF-R17.1n | 293 | 97.1 | 91.6 | 97.8 | 99.9 | 97.3 |
| AF-R17.1o | 313 | 100.0 | 94.2 | 100.6 | 100.5 | 100.2 |
| AF-R17.1p | 344.5 | 97.9 | 94.2 | 98.3 | 100.4 | 98.0 |
| AF-R17.1q | 365 | 98.3 | 92.0 | 99.0 | 100.0 | 98.6 |
| AF-R17.1r | 379 | 100.5 | 93.5 | 101.1 | 100.9 | 100.6 |
| AF-R17.1s | 391 | 100.6 | 95.3 | 101.2 | 100.4 | 100.8 |
| AF-R17.1t | 411 | 99.1 | 95.5 | 99.9 | 101.6 | 99.5 |
| AF-R17.1u | 429 | 97.7 | 99.7 | 98.8 | 100.4 | 98.4 |
| AF-R17.1v | 447 | 100.2 | 94.5 | 100.8 | 101.8 | 100.4 |
| AF-R17.1w | 466.5 | 99.3 | 93.1 | 99.9 | 100.4 | 99.4 |
| AF-R17.1x | 483 | 99.9 | 93.2 | 100.5 | 100.5 | 100.0 |
| AF-R17.1y | 499.5 | 100.7 | 94.2 | 101.3 | 99.7 | 100.8 |
| AF-R17.1z | 517 | 101.5 | 95.5 | 102.1 | 100.3 | 101.6 |
| AF-R17.1aa | 537.5 | 105.5 | 98.9 | 106.2 | 100.3 | 105.6 |
| AF-R17.1bb | 563 | 101.0 | 95.6 | 101.5 | 100.2 | 101.1 |
| AF-R17.1cc | 581 | 100.5 | 94.8 | 101.1 | 100.4 | 100.7 |
| Average | | 99.9 | 94.0 | 100.7 | 100.8 | 100.1 |

Table 8

CATALYST DEACTIVATION RATES

| | Deactivation Rate (Rate Constant Pre-exponential Factor Decay), %/day | Significant Contaminants found on Catalyst | Bulk Gas Composition | Temperature Range, ºC |
|---|---|---|---|---|
| | | | | |
| LaPorte | 0.4 | None | CO-Rich | 250 |
| | | | | |
| Autoclaves | 1.2 | None | CO-Rich<br>$H_2$-Rich | 250 |
| | | | | |
| Kingsport:<br>Campaign # 1 | 1.0 - 3.4 | Fe<br>As<br>S | $H_2$-Rich | 250 |
| | | | | |
| Kingsport:<br>Campaign # 2 | 0.2 - 1.3 | As<br>S | $H_2$-Rich | 220 - 235 |

Table 9

| | | | Deactivation Rate Comparison | | |
|---|---|---|---|---|---|
| | | | | | |
| | **No. of Days On-stream** | **Deactivation Rate (a) of $k_0$, %/day** | **Std Error of (a), %/day** | **Deactivation Rate (b) of $k_0$, %/day** | **Std Error of (b), %/day** |
| | | Methanol Catalyst | | DME Catalyst | |
| | | | | | |
| LPDME (LaPorte - 99) | 25 | 0.70 | 0.25 | 0.65 | 0.06 |
| | | | | | |
| LPDME (Lab - 99) | 17 | 1.20 | 0.14 | 1.20 | 0.08 |
| | | | | | |
| LPMEOH™ - (LaPorte - 89) | 22 | 0.52 | 0.09 | | |

Table 10

**XRD Crystallite Size Analysis**

| Run | Time On-stream (hours) | Crystallite size (Å) | |
|---|---|---|---|
| | | Cu | ZnO |
| Spent Catalyst from LaPorte LPDME run | 600 | 88.9 | 54.7 |
| Spent Catalyst from Lab LPDME run | 688 | 120.5 | 61.2 |

**Results of Elemental Analysis of Selected Samples.**

| Run | Elemental (ppmw) | | | |
|---|---|---|---|---|
| | Fe | Ni | Cl | S |
| Spent Catalyst from LaPorte LPDME run | <13 | <5 | <260 | <50 |
| Fresh Methanol Catalyst | <10 | <10 | n.a. | <55 |

Table 11

LPDME Goals and Performance Results (Laboratory and AFDU)

| | **Liquid Fuels Program Goals** | **Commercial Targets** | **Laboratory Results (July 1997)** | **Laboratory Results (June 1999)** | **AFDU Results (Oct./Nov. 1999)** |
|---|---|---|---|---|---|
| **Catalyst Productivity, mol/kg catalyst-hr (MeOH-equivalent)** | > 28 (Initial Productivity) | > 14 (productivity for aged catalyst) | 28 (Initial Productivity) | 28 (Initial Productivity) | 30.5 (Initial Productivity) |
| **Catalyst Selectivity** | DME Selectivity > 80% (% Carbon, $CO_2$-free) | DME = 75%, Methanol = 25% (heating value basis) | DME Selectivity = 79% (% Carbon, $CO_2$-free) | DME Selectivity = 65% (% Carbon, $CO_2$-free) | DME Selectivity = 66 (start) - 61 (end)% (% Carbon, $CO_2$-free) |
| **Catalyst Life** | > 50% Initial Productivity after 1000 hours | Target Productivity after 6 months of operation | 57% of Initial Productivity after 1000 hours | 61% of Initial Productivity after 1000 hours | Calculated Target Productivity after 3-1/3 months of operation |

Figure 1

LaPorte AFDU Oxygenates Modification - Feed Compression and Synthesis

(NOT AVAILABLE ELECTRONICALLY)

Figure 2

LaPorte AFDU Oxygenates Modification - Product Collection and $CO_2$ Removal

(NOT AVAILABLE ELECTRONICALLY)

Figure 3

Utility Oil System

(NOT AVAILABLE ELECTRONICALLY)

Figure 4

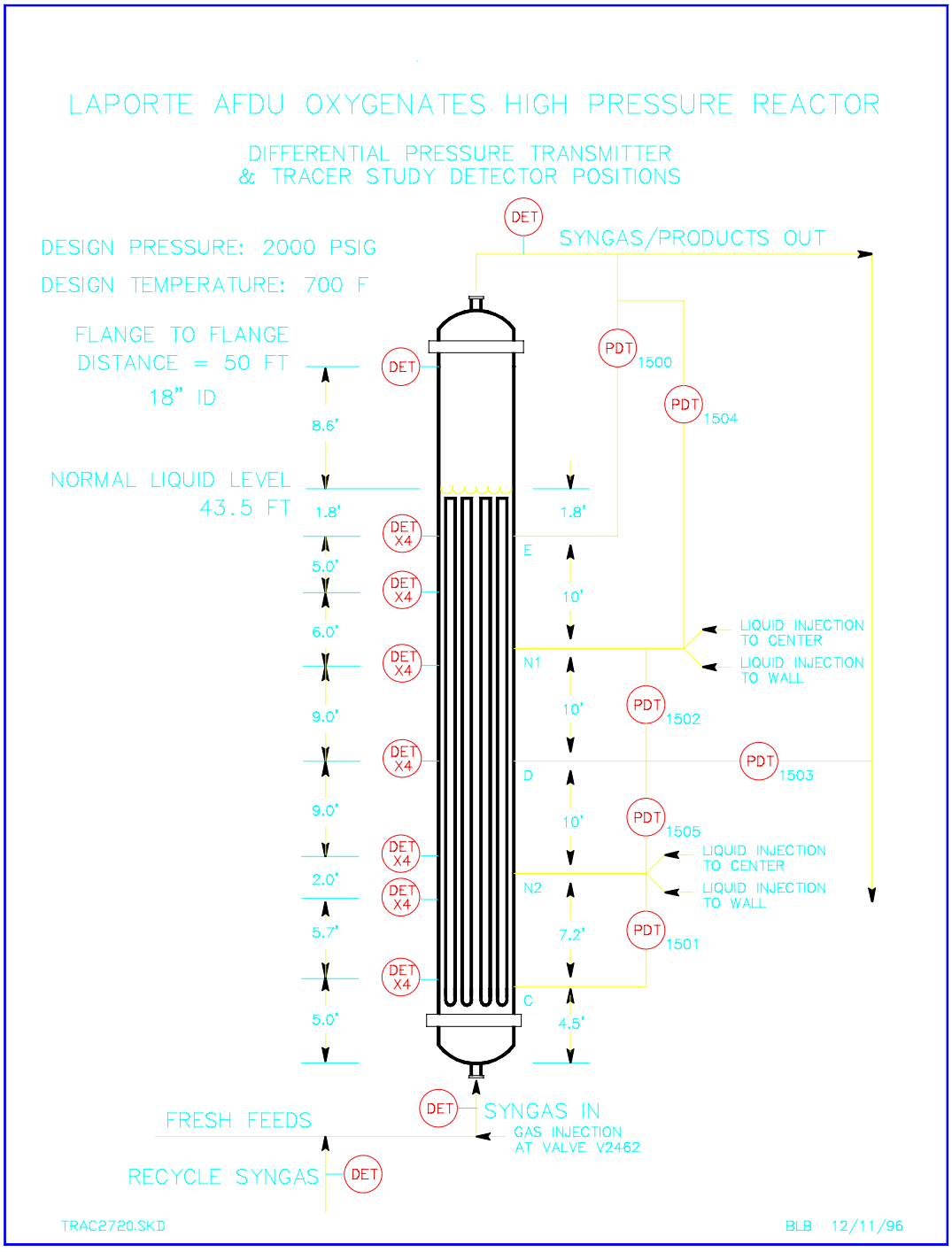

LAPORTE AFDU OXYGENATES HIGH PRESSURE REACTOR
DIFFERENTIAL PRESSURE TRANSMITTER
& TRACER STUDY DETECTOR POSITIONS
DESIGN PRESSURE: 2000 PSIG
DESIGN TEMPERATURE: 700 F
FLANGE TO FLANGE
DISTANCE = 50 FT
18" ID
NORMAL LIQUID LEVEL
43.5 FT
SYNGAS/PRODUCTS OUT
DET
PDT 1500
PDT 1504
8.6'
1.8'
5.0'
6.0'
9.0'
9.0'
2.0'
5.7'
5.0'
DET X4
E
10'
N1
10'
D
10'
N2
7.2'
C
4.5'
LIQUID INJECTION TO CENTER
LIQUID INJECTION TO WALL
PDT 1502
PDT 1503
PDT 1505
LIQUID INJECTION TO CENTER
LIQUID INJECTION TO WALL
PDT 1501
SYNGAS IN
GAS INJECTION AT VALVE V2462
FRESH FEEDS
RECYCLE SYNGAS
TRAC2720.SKD
BLB 12/11/96

Figure 5

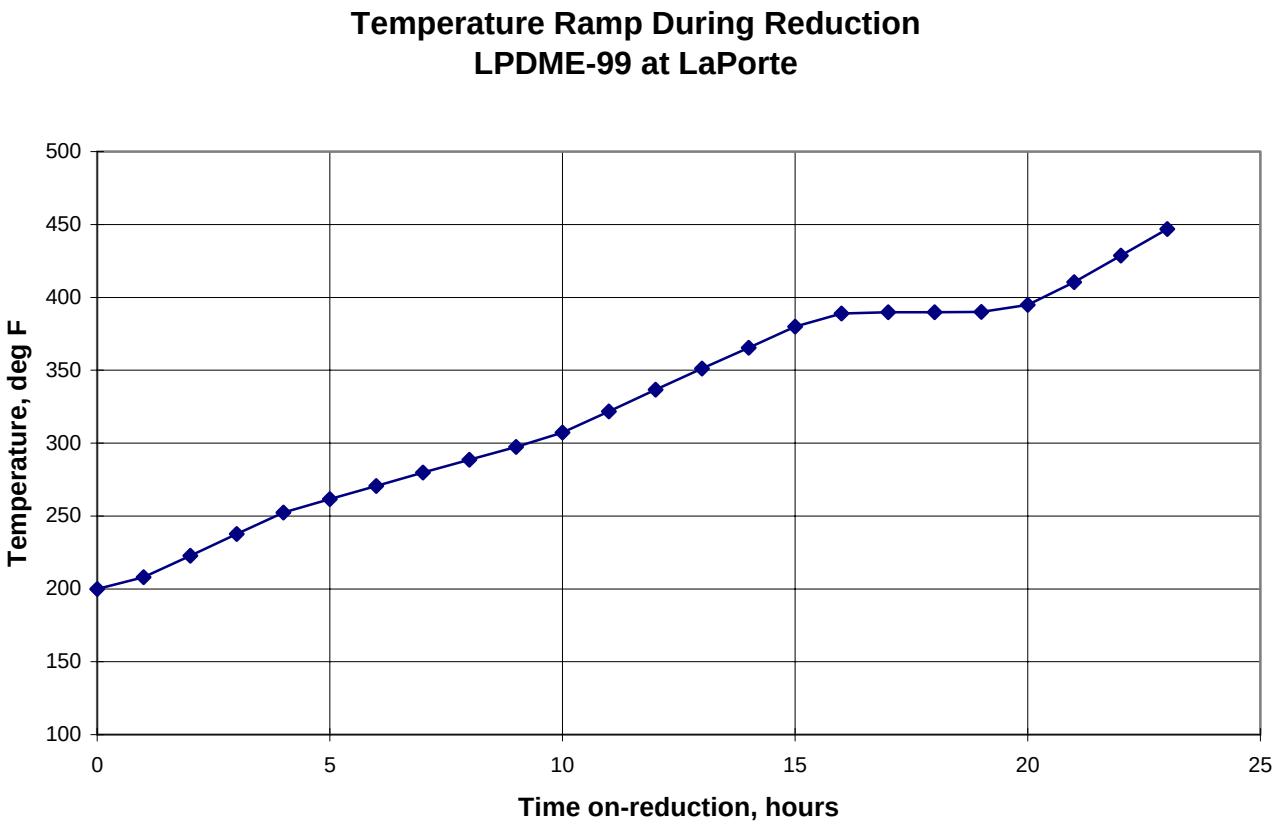

Temperature Ramp During Reduction
LPDME-99 at LaPorte
Temperature, deg F
500
450
400
350
300
250
200
150
100
0
5
10
15
20
25
Time on-reduction, hours

Figure 6

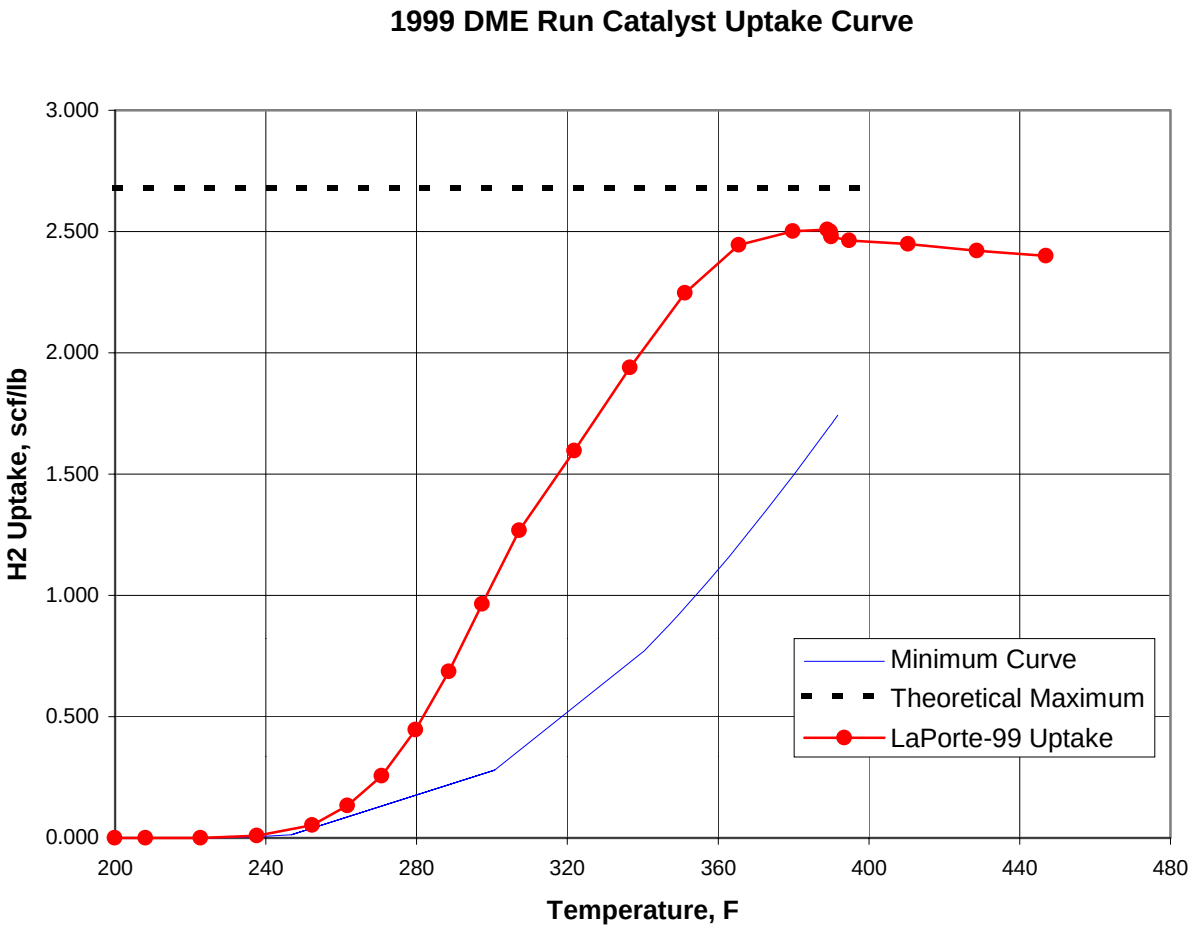

1999 DME Run Catalyst Uptake Curve
H2 Uptake, scf/lb
3.000
2.500
2.000
1.500
1.000
0.500
0.000
200
240
280
320
360
400
440
480
Temperature, F
Minimum Curve
Theoretical Maximum
LaPorte-99 Uptake

Figure 7

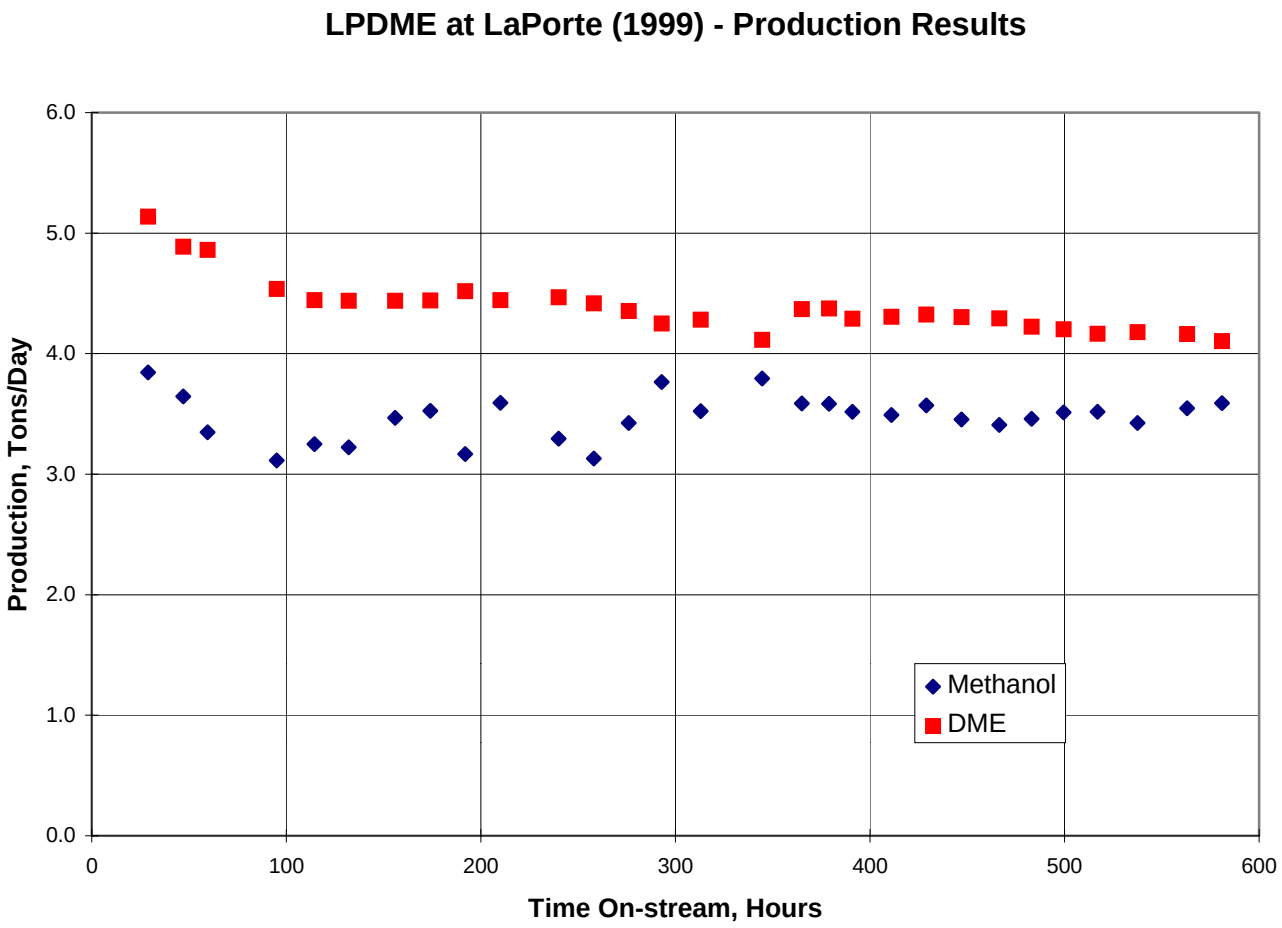

LPDME at LaPorte (1999) - Production Results
Production, Tons/Day
6.0
5.0
4.0
3.0
2.0
1.0
0.0
0
100
200
300
400
500
600
Time On-stream, Hours
Methanol
DME

Figure 8

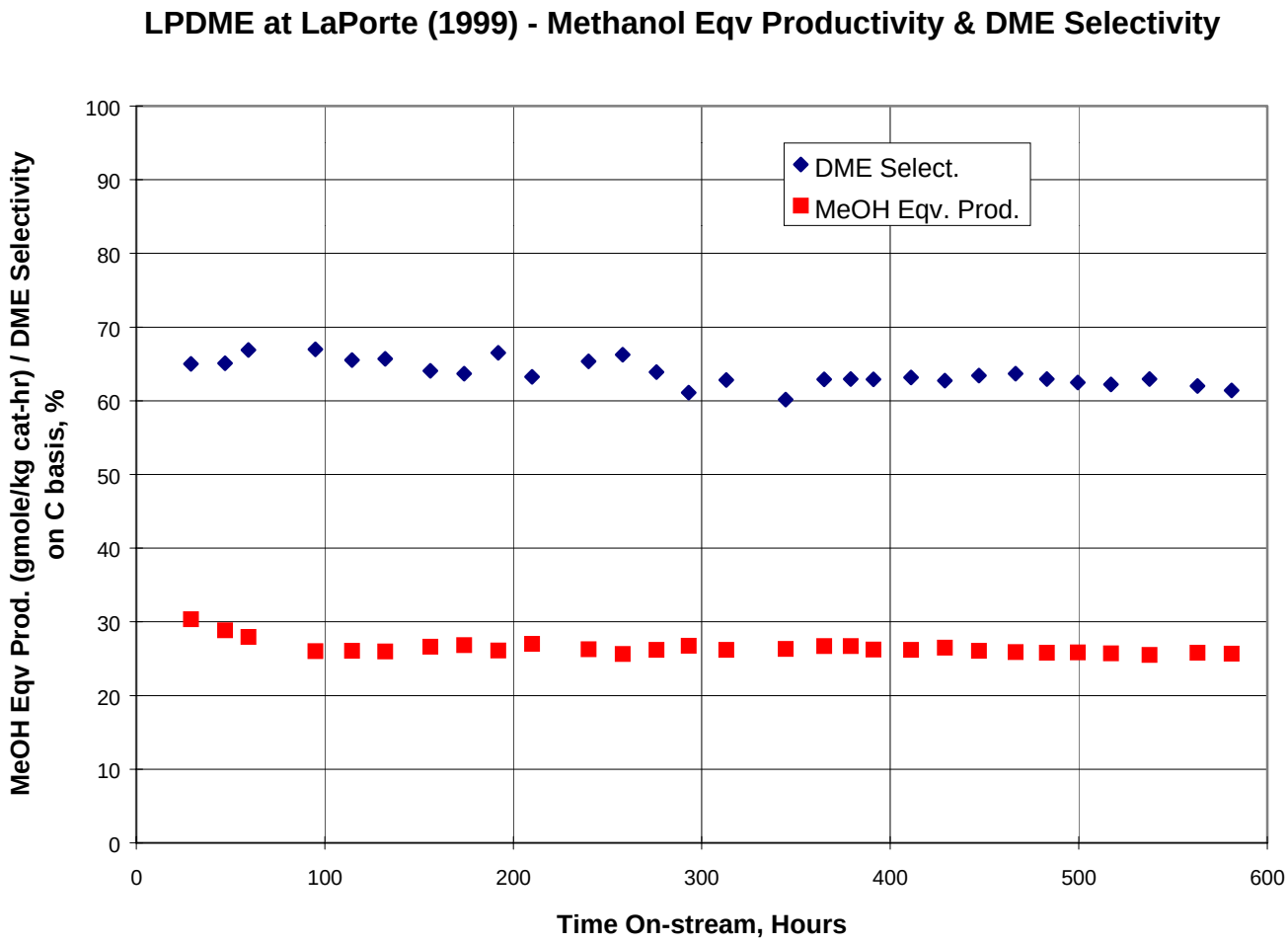

LPDME at LaPorte (1999) - Methanol Eqv Productivity & DME Selectivity
MeOH Eqv Prod. (gmole/kg cat-hr) / DME Selectivity on C basis, %
Time On-stream, Hours
DME Select.
MeOH Eqv. Prod.
0
10
20
30
40
50
60
70
80
90
100
0
100
200
300
400
500
600

Figure 9

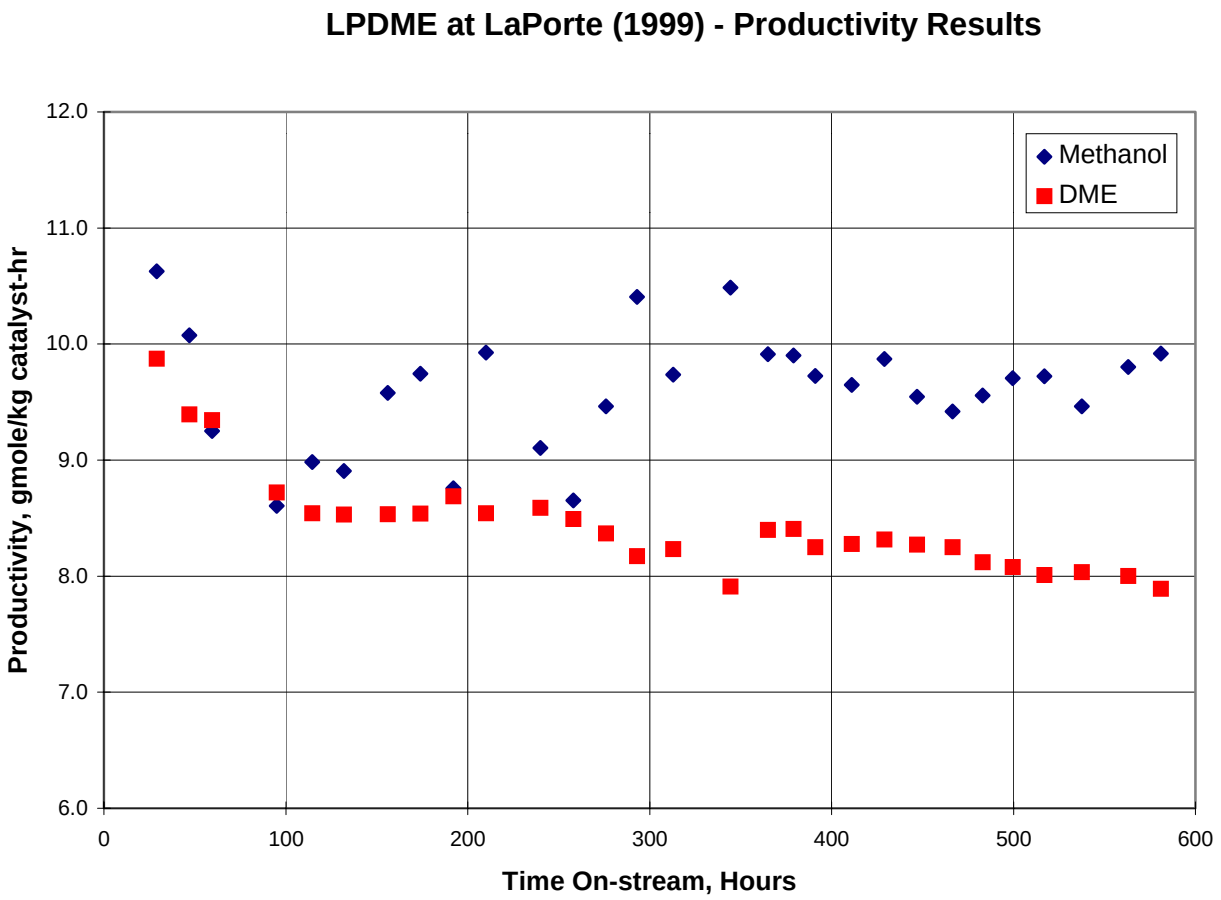

LPDME at LaPorte (1999) - Productivity Results
Productivity, gmole/kg catalyst-hr
12.0
11.0
10.0
9.0
8.0
7.0
6.0
0
100
200
300
400
500
600
Time On-stream, Hours
Methanol
DME

Figure 10

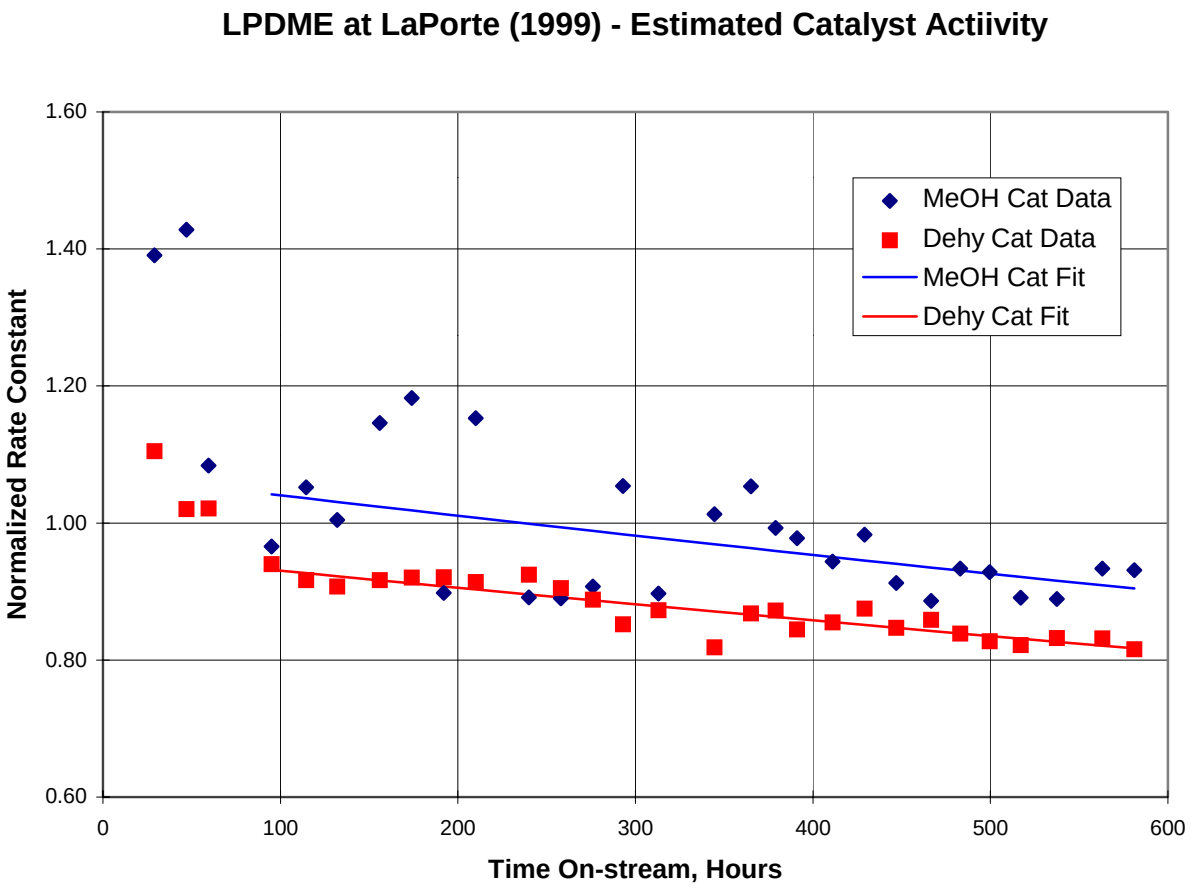

LPDME at LaPorte (1999) - Estimated Catalyst Actiivity
Normalized Rate Constant
1.60
1.40
1.20
1.00
0.80
0.60
0
100
200
300
400
500
600
Time On-stream, Hours
MeOH Cat Data
Dehy Cat Data
MeOH Cat Fit
Dehy Cat Fit

Figure 11

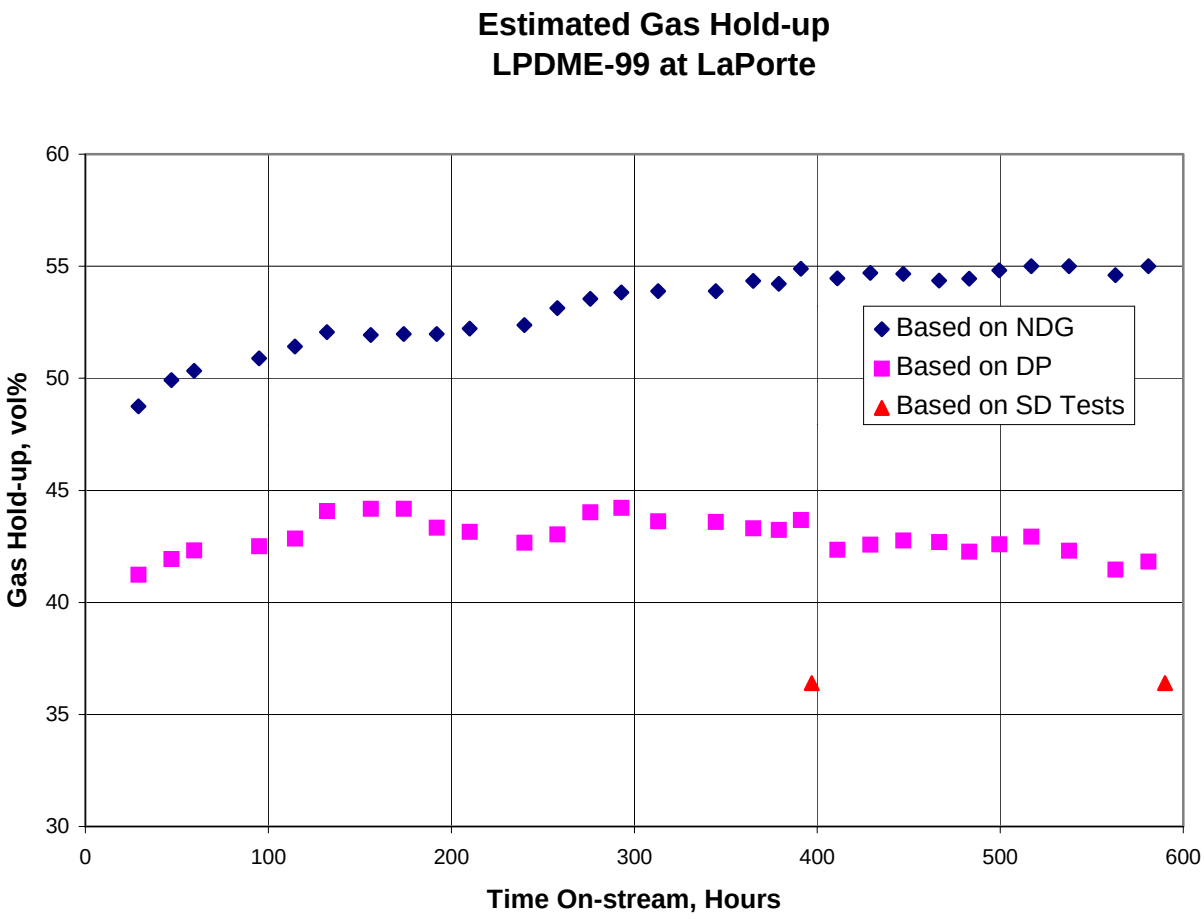

Estimated Gas Hold-up
LPDME-99 at LaPorte
Gas Hold-up, vol%
60
55
50
45
40
35
30
0
100
200
300
400
500
600
Time On-stream, Hours
Based on NDG
Based on DP
Based on SD Tests

Figure 12

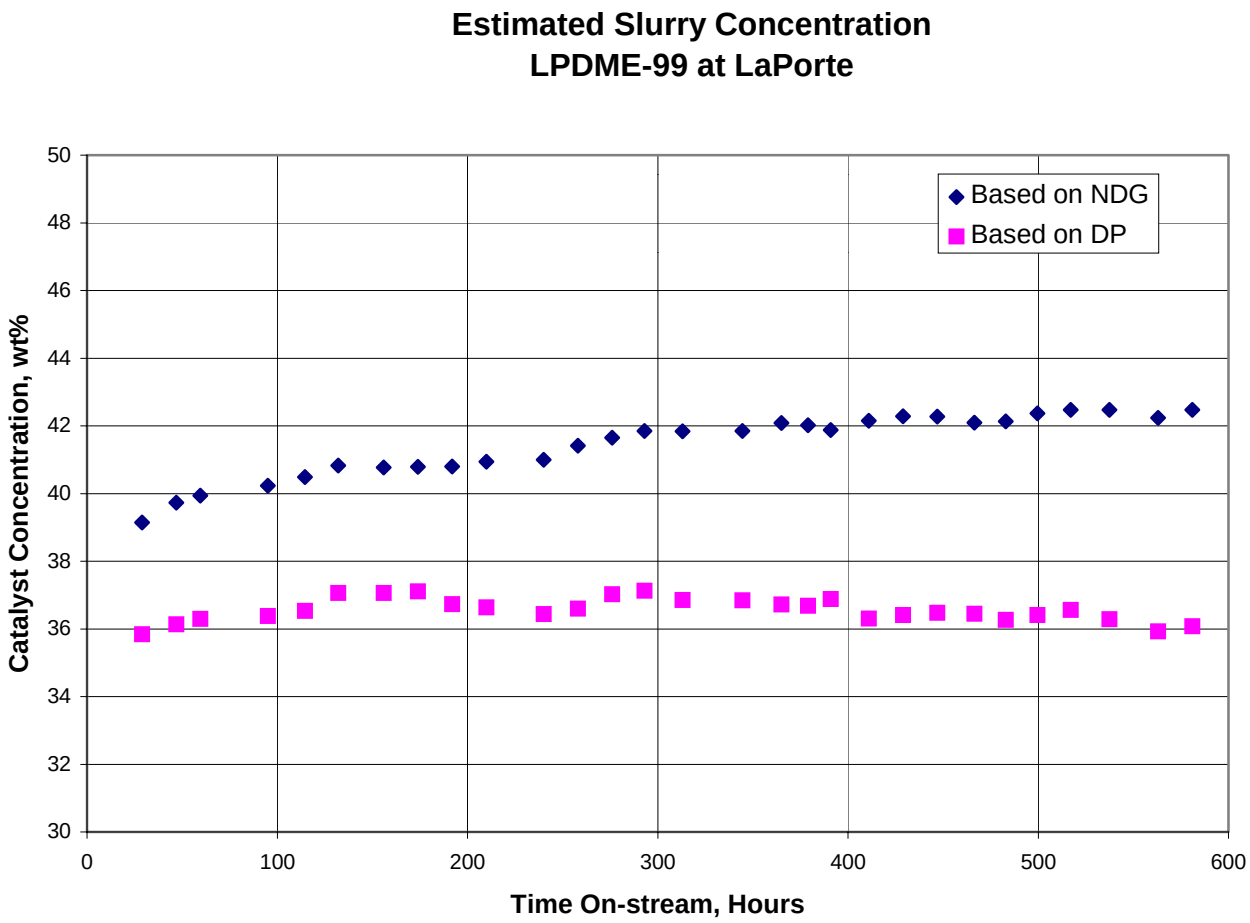
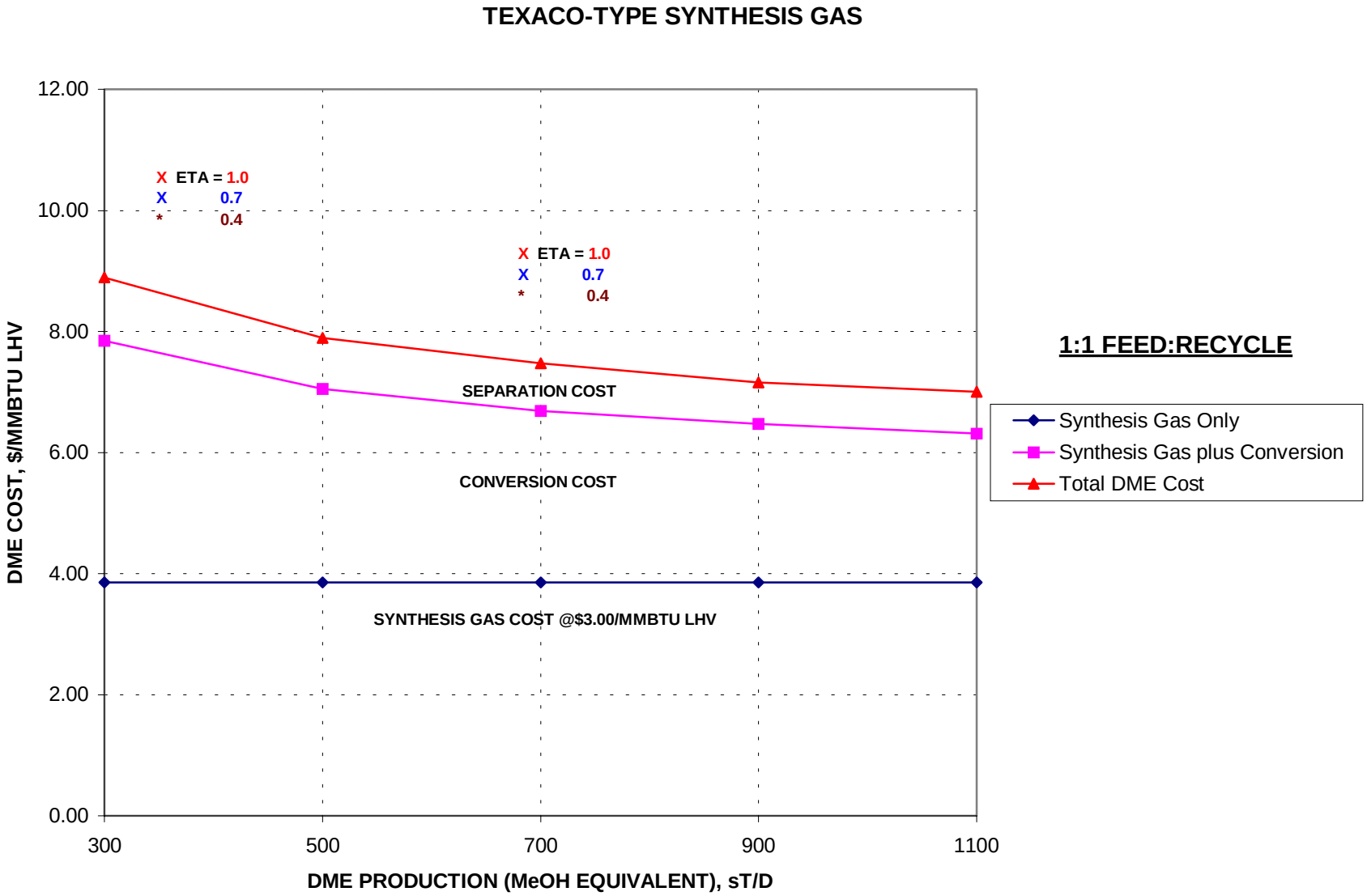

Estimated Slurry Concentration
LPDME-99 at LaPorte
Based on NDG
Based on DP
Catalyst Concentration, wt%
Time On-stream, Hours
50
48
46
44
42
40
38
36
34
32
30
0
100
200
300
400
500
600

FIGURE 13 - LPDME PROCESS ECONOMICS



# APPENDIX A

## DME Design Verification Testing

APPENDIX A-1 - JULY 1997 DME Design Verification Testing Recommendation

## Summary

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

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

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

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

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

## The LPDME Process Concept: - Three Concurrent Reactions:

- $2\ CO + 4\ H_2 = 2\ CH_3OH$ (Methanol Synthesis).
- $2\ CH_3OH = 1\ CH_3\text{-}O\text{-}CH_3 + 1\ H_2O$ (Methanol Dehydration).
- $1\ CO + 1\ H_2O = 1\ CO_2 + 1\ H_2$ (Water-gas Shift).

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

- $3\ CO + 3\ H_2 = 1\ CH_3\text{-}O\text{-}CH_3 + 1\ CO_2$ (DME from CO-rich syngas)

This is the "once-through" CO-rich syngas concept for the LPDME process utilizing a single slurry reactor. Conversion per pass, with CO-rich syngas, can be higher than for the LPMEOH™ process. Methanol may also be produced, as a mixed co-product with the DME, and can easily be separated and recovered. The separation of DME from carbon dioxide ($CO_2$) will be necessary for certain market applications.

## Status of the LPDME DVT Work

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

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

- Aerosol - Small, but established market. High purity DME is required.
- Cooking Fuel - Potentially a large market, to replace imported liquefied petroleum gas (LPG). There is a lot of interest in China, and DME is on the agenda for DOE's Pittsburgh Coal Conference in China (Sept. of 1997). Purity, of about >95% DME, with <2% methanol, < 3% $CO_2$ is estimated. An unresolved application issue is CO emissions during cooking. How does DME purity impact this? Use testing is needed.

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

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

- Diesel Replacement Fuel. DME is an ultra clean (high Cetane) diesel fuel; and an 80% DME mixture with methanol and water is now being engine-tested by others (Amoco, et. al.). Market development (at least in the U.S.) faces a fuel distribution infrastructure problem. DME might more easily replace LPG in countries where LPG is already an engine fuel. Diesel use in the U.S. is projected to increase by 1.5 percent a year, assuming an economic growth of 1.9 percent a year. This will raise consumption from over 4 quadrillion BTU to approaching 6 quadrillion BTU (Reference 1). This corresponds to an annual increase of almost 1.4 million gallons per year of diesel consumption.
- DME Derivatives, as a Diesel Fuel Additive. Quotes from the DOE Liquid Fuels Program (Contract No. DE-FC22-95PC93052) quarterly report for April-June 1996: "Initial Cetane number (CN) testing of a three-component composition of 1,2-dimethoxy ethane, 1,1-dimethoxy methane and methanol blended with diesel fuel showed a 40% increase in the CN of the diesel fuel when the blend was 50/50." "The concept of adding a blend of oxygenated compounds to diesel fuel in order to enhance the Cetane value and cold start properties is being investigated. The blend of oxygenated compounds is derived from dimethyl ether chemistry, and builds on work conducted earlier --." The testing of this DME feedstock chemistry is in its early days, but it is possible that $CO_2$ may not need to be separated from the DME prior to the production of DME derivatives. The 50/50 blend referenced above would therefore provide a large market opportunity for the projected U.S. market growth (Reference 1), let alone for the present consumption.
- DME Derivatives, as Chemicals/Other Fuels. DME is a key intermediate in a commercial synthesis gas-to-gasoline process, and is being developed as an intermediate for other chemicals and fuels as part of the DOE's Liquid Fuels Program. The fit for DME here is long-term.

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

As with the LPMEOH™ process, gas phase process technology must be considered as the economic competitor. The gas phase DME process (Reference 3) must run with hydrogen ($H_2$)-rich syngas. In the IGCC coproduction flow sheet (shown in Figure 1), gas phase technology is at an economic disadvantage, since separate shift and $CO_2$

removal are required. As is the case for methanol, inexpensive remote natural gas would therefore be the economic plant site choice for gas phase technology. A comparison, of IGCC/LPDME coproduction with DME imported from remote gas facilities, shows an advantage of 20-30% for locally produced DME relative to imported DME. The transportation cost to import DME is much higher than for methanol, and the LPDME coproduction advantage is even greater than that for LPMEOH™ (vs. methanol import) (Reference 2). Dehydration of imported methanol to make DME is not competitive either. Therefore, for DME in local markets, LPDME coproduction should be a winner!

With $H_2$-rich syngas, the LPDME process loses its (once-through, high conversion per pass) economic advantage. The overall reaction, with (> 2:1) $H_2$-rich syngas is:

- $2\ CO + 4\ H_2 = 1\ CH_3\text{-}O\text{-}CH_3 + 1\ H_2O$ (DME from $H_2$-rich syngas)

Since water inhibits the methanol dehydration reaction, the slurry reactor must be staged, with water removal between stages. Staging could be by high ratio gas recycle, and/or with multiple reactors; but the once-through simplicity is lost. Therefore, it is unlikely that the LPDME process would be developed for use in $H_2$-rich syngas applications.

A cost estimate of commercial-scale LPDME plants has been performed. This work has helped quantify the targets for the laboratory R&D program (summarized in Part B). From these studies, a commercially successful LPDME system is defined for a Texaco-type synthesis gas (35 mol% $H_2$, 51 mol% CO, 13 mol% $CO_2$) available at 500 PSIG. At a reactor operating pressure of 950 PSIG and a space velocity of 4,000 liters/hr-kg catalyst, the LPDME catalyst system must have a methanol equivalent productivity of 14 mol/kg catalyst-hr after 6 months of operation, producing at least 75% (by heating value) DME and 25% methanol. Figure 2 shows the effect of plant size on DME cost. These costs are competitive with LPG in China (Section A-1).

**B. Laboratory R&D Results**

Summary of work through end of funding by CCT LPMEOH™ Project (9/96): An LPDME catalyst system, with reasonable long-term activity (57% of initial activity after 1000 hours), productivity (equivalent methanol productivity of 29 mol/kg catalyst-hr), and selectivity (79% carbon selectivity to DME, $CO_2$-free basis), was identified and tested. The system exhibits best activity under CO-rich syngas conditions, i.e. those most likely for (IGCC) coproduction. Accelerated aging of the catalyst system is a remaining issue. Water concentrations in the liquid phase reactor are higher with syngases richer in $H_2$, and its effect needs to be evaluated.

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

1) Understanding the LPDME catalyst system's accelerated aging; and modifying the catalyst and/or the system operating conditions; and

2) Manufacturing scale-up of catalyst for a LaPorte AFDU run.

Progress has been made in the laboratory effort. Figure 3 shows the performance for the first DME catalyst which was tested; goals from the Liquid Fuels Program are provided for reference. After further study, an improved DME catalyst (AB-05) was tested with two LPMEOH™ catalysts (S3-86 and MK-101); the results of a 700 hour life study are presented in Figure 4. When compared with the program goals (summarized in Figure 5), the catalyst performance of the newer catalyst is approaching the commercial targets defined in Section A. The status of the laboratory program is summarized in the following table:

| | **Liquid Fuels Program Goals** | **Commercial Targets** | **Laboratory Results** |
|---|---|---|---|
| **Catalyst Productivity, mol/kg catalyst-hr (MeOH-equivalent)** | > 28 (Initial Productivity) | > 14 (productivity for aged catalyst) | 28 (Initial Productivity) |
| **Catalyst Selectivity** | DME Selectivity > 80% (% Carbon, $CO_2$-free) | DME = 75%, Methanol = 25% (heating value basis) | DME Selectivity = 79% (% Carbon, $CO_2$-free) |
| **Catalyst Life** | > 50% Remaining Activity after 1000 hours | Target Productivity after 6 months of operation | 57% Remaining Activity after 1000 hours |

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

## Recommendations

The catalyst system and the market applications/opportunities are sufficiently promising that proof-of-concept testing at the LaPorte AFDU is recommended. Kingsport is an unlikely site for the commercial size demonstration of LPDME, since there are limited times for CO-rich syngas testing; and $H_2$-rich syngas would create water buildup. Therefore, the basis for commercializing LPDME must come from:

1) catalyst performance (productivity, selectivity, and life) for the LPDME catalyst system under CO-rich syngas from the proof-of-concept testing at the LaPorte AFDU;
2) continuing work in hydrodynamics of slurry reactors (other ongoing DOE programs); and
3) reactor performance (methanol catalyst activity and life, hydrodynamics, and heat transfer) from the LPMEOH™ Process Demonstration Unit.

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

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

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

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

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

## Impact on CCT Project

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

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

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

## References


1. **Transportation energy consumption by fuel, 1975, 1995 and 2015: History:** Energy Information Administration, *Short-Term Energy Outlook*, DOE/EIA-0202(96/4Q) (Washington, DC, October 1996), and *State Energy Data Report 1994*, DOE/EIA-0214(93). **Projections:** Table A2. Internet access at http://www/eia.doe.gov/oiaf/aeo97/figure.html#fig46.

2. "Fuel and Power Coproduction - The Liquid Phase Methanol™ Process Demonstration at Kingsport", paper presented at Fifth Annual DOE Clean Coal Technology Conference, Tampa, FL, January 7-9, 1997.

3. Haldor Topsoe AS, "Preparation of Fuel Grade Dimethyl Ether", International Publication Number WO9623755, World International Property Organization, 08 August 1996.

Figure 1

(NOT AVAILABLE ELECTRONICALLY)

Figure 2

(NOT AVAILABLE ELECTRONICALLY)

Figure 3

(NOT AVAILABLE ELECTRONICALLY)

Figure 4

(NOT AVAILABLE ELECTRONICALLY)

Figure 5

(NOT AVAILABLE ELECTRONICALLY)

06 August 1999

Mr. Robert M. Kornosky
Technical Project Manager
Mail Stop 922-342C
U. S. Department of Energy
Federal Energy Technology Center
P. O. Box 10940
Pittsburgh, PA 15236-0940

**Subject: Cooperative Agreement DE-FC22-92PC90543**
**Liquid Phase Methanol Demonstration Project**
**Liquid Phase Dimethyl Ether LPDME Design Verification Testing - Recommendation to Proceed with Fall 1999 Test at LaPorte AFDU**

Dear Bob:

As defined in the Statement of Work for the subject Project:

*Subject to Design Verification Testing (DVT), the Partnership proposes to enhance the Project by including the demonstration of the slurry reactor's capability to produce DME (dimethyl ether) as a mixed co-product with methanol.*

Catalyst research for the Liquid Phase Dimethyl Ether (LPDME) Process has been ongoing under sponsorship of the U. S. Department of Energy's (DOE's) Liquid Fuels Program. Based upon results of the catalyst development program, a recommendation letter was sent to your attention on 30 June 1997. In the Summary, the recommendation states that a test of the LPDME Process at the LaPorte Alternative Fuels Development Unit (AFDU), in conjunction with the DOE's Liquid Fuels Program, would be appropriate if the catalyst system development can be completed successfully. An implementation decision, made mutually by the DOE's Clean Coal Technology Liquid Phase Methanol (LPMEOH™) project participants, and by the DOE's Liquid Fuels Program participants, should be made to implement testing at LaPorte. Your letter of 31 July 1997 accepted this recommendation based upon the status of the research which had been completed.

During your visit to Allentown on 09 June 1999, we participated in a portion of the review meeting for the Liquid Fuels Program which covered the activities regarding the status of catalyst development for the LPDME Process. Over the past year, the research team has made significant progress in understanding the operating parameters which can impact the performance and life for the methanol synthesis and dehydration catalysts. This work

indicates that the targets for catalyst productivity and life can be met at lower selectivity to DME. The results of the laboratory program are summarized in Table I.

Within the 30 June 1997 Recommendation, a cost estimate for a commercial-scale LPDME plant was performed. The case that was considered was the retrofit of an existing Texaco gasifier in China which is currently used in the production of ammonia. The results of this initial work were that, for this synthesis gas (35 mol% $H_2$, 51 mol% CO, 13 mol% $CO_2$) available at 500 psig, the DME costs are competitive with liquefied petroleum gas (LPG) in China ($7 - $8 per million Btu's). Figure I shows an updated estimate for these costs based upon the changes in productivity and selectivity as given in Table I. The cost of synthesis gas assumes constant utilization for each plant size, and economies of scale are indicated by changes in the conversion and separation costs. The target DME cost can likely be achieved at larger plant sizes by extrapolating the costs to the 1,100 short tons-per-day production rates. (Note that the conversion and separation costs of DME produced from Shell-type synthesis gas (30 mol% $H_2$, 66 mol% CO, 3 mol% $CO_2$) are typically less than the cost from Texaco-type synthesis gas. Given the recent financial difficulties in Asia, the construction of new gasification capacity within China is unlikely; therefore, the study referenced in the 30 June 1997 Recommendation focused on the likely initial entry of the LPDME Process as a retrofit to an existing gasification system.)

Based upon these summaries of the laboratory program and economic analysis, the Liquid Fuels Program has recommended that the goals and objectives for the next operating campaign for the LPDME Process at the LaPorte AFDU (scheduled to begin on or about 01 October 1999) should be modified. Table II, which was used in the presentation on the upcoming LPDME run, accurately reflects this change. For planning purposes, the next test for the LPDME Process at the LaPorte AFDU is being treated as an interim campaign, with the primary objective being the determination of a tie-point between catalyst performance in the autoclave and the pilot plant scale.

A draft of the test plan for the operation of the LaPorte AFDU during this upcoming trial is included in Table III. (Note that this table has been updated since the 8 June 1999 review meeting to reflect the latest input from the laboratory and from DOE's Hydrodynamics Program.) Run time is provided to study catalyst life and to perform process variable scans on other operating conditions which are of potential commercial interest. Most of the operating conditions during the plant trial will be performed with Shell-type synthesis gas; this composition was used for the majority of the tests in the laboratory autoclaves. Also, as noted above, the conversion and separation costs for the LPDME Process are expected to be better when Shell-type synthesis gas is used as the feed. Detailed test authorizations for the conditions will be prepared prior to the commencement of operations.

A description of key objectives of the run conditions is provided below:

Run AF-13 - Reduction - The methanol synthesis catalyst will be reduced (4% hydrogen in nitrogen) in the presence of dehydration catalyst per the procedure developed in the laboratory.

Run AF-R17.1 - Life Study + Tracer 1 - In the laboratory, much of the work on catalyst life has used Shell-type synthesis gas as the feed to the autoclave. During this 18-day test, catalyst life in the slurry bubble column reactor (SBCR) will be monitored in an effort to develop the tie-point with the autoclave. A radioactive tracer study will also be performed in order to enhance the level of understanding of flow patterns within the SBCR and provide data to the modeling work within DOE's Hydrodynamics Program.

Run AF-R17.2 - Low Velocity with Methanol Injection - Operation of the LPDME Process at low space velocity results in higher total conversion of synthesis gas, which is important in all-DME applications. Recycle of methanol to the SBCR will increase the selectivity to DME.

Run AF-R17.3 - Stoichiometric Feed ($H_2$/CO = 1) with Methanol Injection - Simulations have indicated that operation of the LPDME Process with a $H_2$ to CO ratio at the feed to the SBCR of 1.0 provides the optimal kinetic conditions and the best utilization of synthesis gas. Results from this test will provide additional data on this commercially attractive condition.

Run AF-R17.4 - High Velocity + Tracer 2 - A process variable scan will be performed to determine the performance results at the maximum gas superficial velocity which can be tested at the LaPorte AFDU. It was proposed during a Hydrodynamic Program Review Meeting on 9 June 1999 to conduct the second radioactive tracer study at the highest velocity instead of the low velocity. It is important to check the fluid dynamics at velocities of commercial relevance.

Run AF-R17.5 - High Concentration - During the initial trial of the LPDME Process at the LaPorte AFDU in 1991, indications of catalyst settling during normal operation were detected. Due to system limitations, the maximum slurry concentration which can be tested in the laboratory autoclaves is about 20 weight percent (wt%). As a consequence, the initial 4 tests of this upcoming trial will be performed at a lower slurry concentration (35 wt%) as compared with prior tests of the LPMEOH™ Process (40 - 45 wt%). During this final test, liquid level in the SBCR will be lowered to study the effects of slurry concentration on catalyst performance. It is expected that the maximum slurry concentration which can be achieved during this run is 40 wt%.

Once the results from the plant trial have been analyzed, information will be used to give additional direction to the ongoing research program within the Liquid Fuels Program. Also, an assessment of the results as they relate to the possible implementation of the LPDME catalyst system at Kingsport will be performed. During this review, one of the following three options will be selected:

1) The performance of the LPDME catalyst system during this test approaches the Commercial Targets as defined in Table I. Upon agreement between the

participants, operation of the LPDME Process at Kingsport will be incorporated into the Demonstration Test Plan.

2) The results on catalyst performance fail to reach the Commercial Targets, but provide an indication that additional DVT, including the potential need for additional testing at the LaPorte AFDU, may be warranted. The use of funding from the LPMEOH™ Demonstration Project for future DVT (such as additional laboratory experiments) will then be addressed by the participants.

3) The participants may conclude that the current LPDME catalyst system is not likely to meet the Commercial Targets, and that additional basic research and development is required. In this case, no additional DVT activities will be undertaken under sponsorship of the LPMEOH™ Demonstration Project.

Air Products recommends that the funds currently allocated within the LPMEOH™ Demonstration Project for DVT of the LPDME Process should be allocated to the execution of the trial at the LaPorte AFDU in order to meet the objectives as defined above. I have assessed the impact of the change of objectives for this run on the implementation of the LPDME Process at the LPMEOH™ Process Demonstration Unit. For reference, the original assessment in the 30 June 1997 Recommendation states:

*The catalyst system and the market applications/opportunities are sufficiently promising that proof-of-concept testing at the LaPorte AFDU is recommended. Kingsport is an unlikely site for the commercial-size demonstration of LPDME, since there are limited times for CO-rich syngas testing; and $H_2$-rich syngas would create water buildup. Therefore, the basis for commercializing LPDME must come from:*

1) *catalyst performance (productivity, selectivity, and life) for the LPDME catalyst system under CO-rich syngas from the proof-of-concept testing at the LaPorte AFDU;*
2) *continuing work in hydrodynamics of slurry reactors (other ongoing DOE programs); and*
3) *reactor performance (methanol catalyst activity and life, hydrodynamics, and heat transfer) from the LPMEOH™ Process Demonstration Unit.*

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

The strategy for commercialization of the LPDME Process remains unchanged; the timing for commercialization will depend upon the resolution of performance issues related to the methanol synthesis catalyst at the LPMEOH™ Process Demonstration Unit and the results of the upcoming trial at the LaPorte AFDU.

Reporting on the efforts in developing the LPDME Process will be as follows:

1) Results of the laboratory program will be published by the DOE's Liquid Fuels Program.
2) Results of the upcoming DVT test at the LaPorte AFDU will be published under the LPMEOH™ Demonstration Project.
3) A market analysis for DME and review of the economics of the LPDME Process will be prepared by the LPMEOH™ Demonstration Project following the completion of the upcoming campaign at the LaPorte AFDU.

Very truly yours,

Edward C. Heydorn
Program Manager
LPMEOH™ Demonstration Project

Attachments

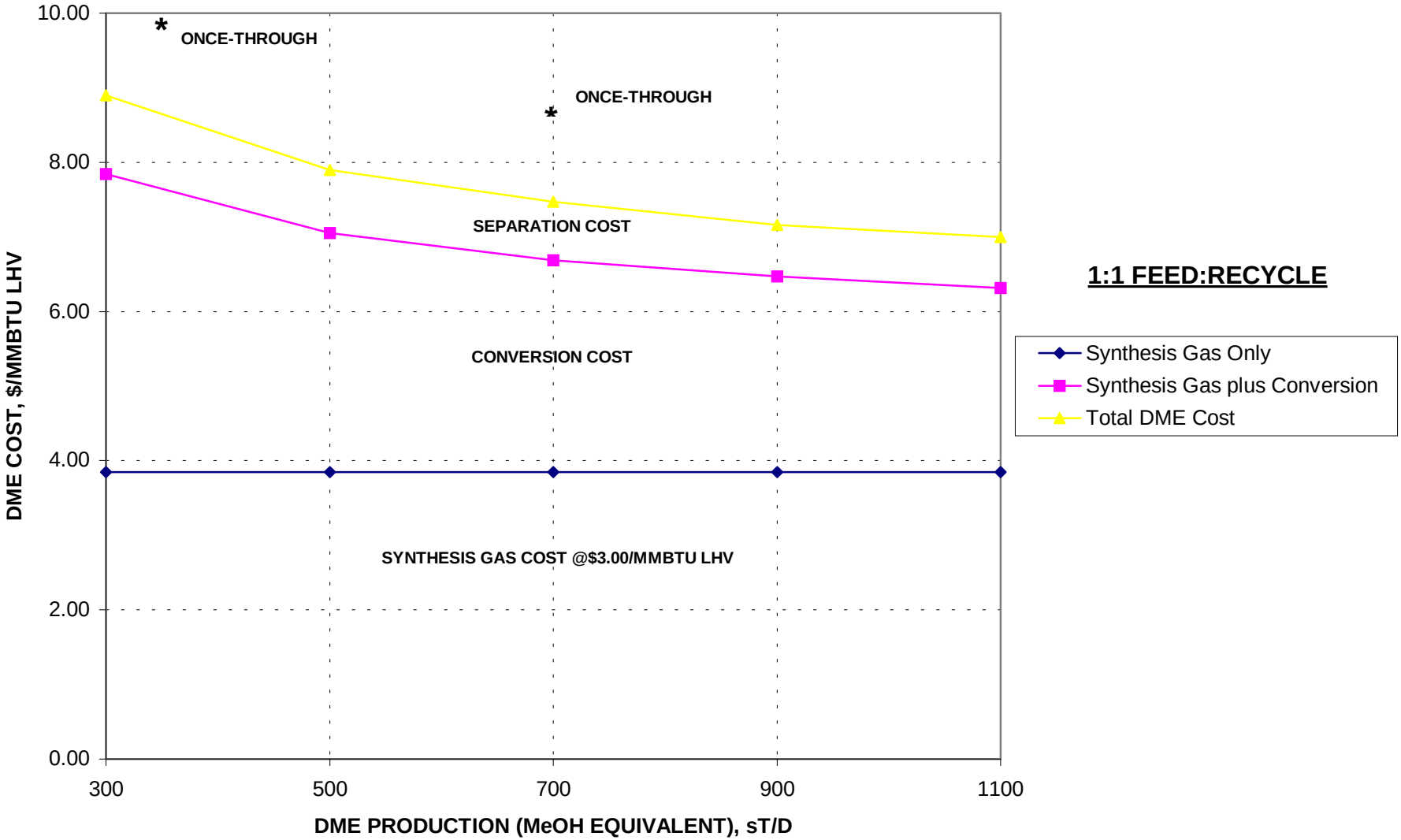
cc: P. J. A. Tijm
W. C. Jones (Eastman)

TABLE I

**LPDME GOALS AND LABORATORY PERFORMANCE**

| | **Liquid Fuels Program Goals** | **Commercial Targets** | **Laboratory Results (July 1997)** | **Laboratory Results (June 1999)** |
|---|---|---|---|---|
| **Catalyst Productivity, mol/kg catalyst-hr (MeOH-equivalent)** | > 28 (Initial Productivity) | > 14 (productivity for aged catalyst) | 28 (Initial Productivity) | 28 (Initial Productivity) |
| **Catalyst Selectivity** | DME Selectivity > 80% (% Carbon, $CO_2$-free) | DME = 75%, Methanol = 25% (heating value basis) | DME Selectivity = 79% (% Carbon, $CO_2$-free) | DME Selectivity = 65% (% Carbon, $CO_2$-free) |
| **Catalyst Life** | > 50% Initial Productivity after 1000 hours | Target Productivity after 6 months of operation | 57% of Initial Productivity after 1000 hours | 61% of Initial Productivity after 1000 hours |

**FIGURE I**
**LPDME - DME COST VERSUS PLANT SIZE - TEXACO-TYPE SYNTHESIS GAS**



## TABLE II

## LIQUID PHASE DME SYNTHESIS RUN OBJECTIVES

DETERMINE COMMERCIAL VIABILITY OF THE LPDME PROCESS ON A 10 T/D SCALE, USING COMMERCIALLY PRODUCED CATALYSTS

OBTAIN INFORMATION TO CORRELATE SCALE-UP OF CATALYST AGING FROM AUTOCLAVE TO BUBBLE COLUMN

CONDUCT PROCESS VARIABLE TESTING

PERFORM EXPERIMENTS TO BETTER UNDERSTAND REACTOR HYDRODYNAMICS

| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | | **TABLE III** | | | |
| | | | | | | **PRELIMINARY RUN PLAN** | | | |
| | | | **DME RUN AT THE LAPORTE AFDU - OCTOBER / NOVEMBER 1999** | | | | | | |
| | | | | | | | | | |
| | | | | **Reactor** | **Reactor** | | | **Inlet** | |
| **Run No.** | **No. of** | **Comment** | **Gas Type** | **Pressure** | **Temp.** | **Space Vel.** | **React. Fd.** | **Sup. Vel.** | **Slurry wt%** |
| | **Days** | | | **psia** | **deg F** | **sl/kg-hr** | **lbmol/hr** | **ft/sec** | **oxide** |
| | | | | | | | | | |
| | | | | | | | | | |
| **MEOH + DEHYDRATION CATALYST (% MEOH CAT = 95%)** | | | | | | | | | |
| | 1 | Catalyst Loading | | | | | | | |
| AF-A13 | 1 | Reduction | 4% H2 in N2 | 67 | | 793 | 32.3 | 0.62 | 35 |
| AF-R17.1 | 18 | Life Study + Tracer1 | Shell | 765 | 482 | 6000 | 247 | 0.56 | 35 |
| AF-R17.2 | 2.5 | Low Vel. w/ MeOH Inj. + Tracer2 | Shell | 765 | 482 | 3100 | 128 | 0.29 | 35 |
| AF-R17.3 | 1.5 | Stoch. Feed + MeOH Inj. | 1:1 H2/CO | 1000 | 482 | 3250 | 134 | 0.23 | 35 |
| AF-R17.4 | 1.5 | High Velocity | Shell | 765 | 482 | 8000 | 330 | 0.74 | 35 |
| AF-R17.5 | 1.5 | High Concentration | Shell | 765 | 482 | 8000 | 330 | 0.74 | 40 |
| TOTAL | 27 | | | | | | | | |

# APPENDIX B

## Gas Chromatographic Analytical Methods

# Analysis Report

| | | | |
|---|---|---|---|
| To: | Bharat Bhatt | Dept./Loc.: | GEG Centrl PST: Process / A12A3 |
| From: | Dean Chin-Fatt | Dept./Ext.: | CRSD-ATC / 13666 |
| Date: | 8 March 2001 | Lab Name: | Separations Laboratory |

Subject: Gas Chromatographic Analysis of AFDU Process Samples

Sample No.: Samples taken during Oct'99 LPMEOH/DME run

cc: CS File; CS Circ.; F. K. Schweighardt; LB File


SUMMARY:
A 5 cc. gas tight syringe was used to inject 2 cc. of process syn gas, from the AFDU LaPorte facility, for analysis by gas chromatography to determine the iron and nickel carbonyl concentrations. The mole concentrations of iron and nickel carbonyl in the samples taken are listed in Table B-1.

This report also summarizes the gas chromatographic conditions used to analyze the final liquid product produced during the LPMEOH/DME run. Modifications made to the gas chromatographs for this run are also summarized below.


PROBLEM DEFINITION:
Prior to the LPMEOH/DME run a "burn-out" of the plant is performed. This procedure brings syngas into the plant with the reactor at operational temperature. This process syngas to the reactor inlet and reactor outlet are tested for the presence of iron and nickel carbonyls prior to catalyst being loaded. Iron and nickel carbonyls are known catalyst poisons and have to be present at sufficiently low levels in the reactor for good catalyst performance.

ANALYTICAL PROCEDURES:
The carbonyl analysis is performed on a HP-5890 Series II gas chromatograph equipped with a packed glass column interfaced to an electron capture detector. The gas chromatographic conditions are listed in Table B-2.

An external standard procedure was used for quantification. A calibration gas standard containing 240 ppbv nickel carbonyl and 110 ppbv iron carbonyl in nitrogen was used to calibrate the instrument.

The liquid product analysis is performed on the same instrument, however, with a combined DB-225/DB-1701 capillary column interfaced to a thermal conductivity detector. The gas chromatographic conditions are listed in Table B-3.

The bulk gas GCs (Gary and Dennis) were not modified in anyway in preparation for the LPMEOH/DME run. These gas chromatographs were setup to control the automatic Valco sample stream select valves according to the attached "GC Setup" listed in Table B-4. The only changes made to the bulk gas GCs was to install new HayeSep DIP (Acid Wash) packed columns to get base line resolution of methanol and DME.

Modifications to the alcohol GCs (Bharat and Rocco) were made to get methanol on the second FID channel. The DME analysis was performed on the alcohol capillary column already installed in the GCs. This had to be done so that the components could be sent to the DEC according to the component list (Table B-5). To accomplish this the FT Alumina PLOT columns and associated packed pre-columns were removed. The packed pre-columns were replaced with blank copper tubing and the Alumina PLOT capillary columns were replaced with J&W DB-1 15 meter megabore columns.

Table B-1

**Carbonyl Burn-Out Data (ppbv)**
**Oct'99 LPMEOH/DME Run**

| 8 Oct. 1999 | Time | $Ni(CO)_4$ | $Fe(CO)_5$ |
|---|---|---|---|
| SP # 4 | 16:33 | N.D. | 1.2 |
| SP # 3 | 16:40 | N.D. | 1.0 |
| SP # 4 | 16:47 | N.D. | 1.0 |
| SP # 3 | 16:55 | N.D. | 0.6 |
| SP # 4 | 17:05 | N.D. | 0.7 |
| SP # 3 | 17:12 | N.D. | 0.3 |
| 9 Oct. 1999 | Time | $Ni(CO)_4$ | $Fe(CO)_5$ |
| SP # 4 | 07:52 | N.D. | 0.5 |
| SP # 3 | 08:02 | N.D. | 0.2 |
| SP # 4 | 08:24 | N.D. | 0.5 |
| SP # 3 | 08:47 | N.D. | 0.3 |
| SP # 4 | 09:30 | N.D. | 0.4 |
| SP # 3 | 14:45 | N.D. | 0.3 |
| SP # 4 | 15:00 | | |
| SP # 3 | 15:05 | N.D. | 0.2 |
| SP # 4 | 15:10 | N.D. | 0.4 |
| SP # 3 | 15:20 | N.D. | 0.3 |
| SP # 4 | 15:30 | N.D. | 0.4 |
| 10 Oct. 1999 | Time | $Ni(CO)_4$ | $Fe(CO)_5$ |
| SP # 3 | 07:30 | N.D. | 0.3 |
| SP # 4 | 07:50 | N.D. | 0.4 |
| SP # 3 | 07:55 | N.D. | 0.3 |
| SP # 4 | 08:03 | N.D. | 0.5 |

Table B-2

## Gas Chromatographic Conditions

**Instrument:** Hewlett-Packard 5890 Series II Gas Chromatograph.

**Columns:** 6' x ¼" x 2mm ID glass column packed with 10 % squalane on 100/120 mesh on Chromosorb W.

**Oven Temperature Program:** ISOTHERMAL

| | | |
|---|---|---|
| Initial Temperature | 50 | $^{o}$C |
| Initial Time | 5 | min |

**Carrier Gas:** P5

| | | |
|---|---|---|
| Flow Rate | 7.5 | ml/min |
| Column Headpressure | 22 | psig |
| Splitter Flow Rate | 40 | ml/min |

**Detector Type:** Electron Capture Detector

| | | |
|---|---|---|
| Detector Temperature | 100 | $^{o}$C |
| Anode Purge Flow | ON | |
| Valve / Injector Temperature | 120 | $^{o}$C |

**Injection Volume:** 2 cc. at atmospheric pressure

**Injection Mode:** Gas tight syringe

**Data System:** Hewlett-Packard 3396 integrator

**Quantitation Method:** External Standard

**Sample Prep.:** None

Table B-3

**Gas Chromatographic Conditions**

**Instrument:** Hewlett-Packard 5890 Series II Gas Chromatograph.

**Columns:** J&W DB-225 FSOT capillary column, 30 m x 0.25 mm x 0.25 um film/J&W DB-1701 FSOT capillary column, 30 m x 0.32 mm x 1 um film.

*Note: DB-225 Cat. # 122-2232, SN: 8855147 DB-1701 Cat.# 123-0733, SN: 1780516.*

**Oven Temperature Program:**

| | | |
|---|---|---|
| Initial Temperature | 40 | $^{o}$C |
| Initial Time | 5 | min |
| Program Rate | 10 | $^{o}$C/min |
| Final Temperature | 180 | $^{o}$C |
| Final Time | 1 | min |

**Carrier Gas:** Helium

| | | |
|---|---|---|
| Flow Rate | 1.8 | ml/min |
| Column Headpressure | 16 | psig |
| Splitter Flow Rate | 50 | ml/min |

**Detector Type:** Thermal Conductivity

| | | |
|---|---|---|
| Detector Temperature | 200 | $^{o}$C |
| TCD Reference Flow | 22.5 | ml/min |
| TCD Aux. Flow | | ml/min |
| TCD Signal A | 41.5 | |
| TCD Sensitivity | HIGH | |
| Valve / Injector Temperature | 200 | $^{o}$C |

**Injection Volume:** 1 microliter

**Injection Mode:** HP-7673 Autosampler Tower

**Data System:** Hewlett-Packard 3396 integrator

**Quantitation Method:** External Standard

**Sample Prep.:** None

Table B-4

## GC SETUP FOR LPMEOH/DME (OCT'99)

| **GC GARY** | |
|---|---|
| Detectors: TCDs (BULK GAS/H2) | |
| Sampling: AUTO | |
| Sample Port | Sample Tag |
| 01 | Std. 3 (Shell Gas) |
| 02 | SP15 or SP6 (22.11) |
| 03 | SP3a (Rx. Effluent) |
| 04 | SP4 (Reactor Feed) |
| 05 | SP8 (21.10 Vapor) |
| 06 | SP1 (Fresh Feed) |

| **GC DENNIS** | |
|---|---|
| Detectors: TCDs (BULK GAS/H2) | |
| Sampling: AUTO | |
| Sample Port | Sample Tag |
| 01 | Std. 3 (Shell Gas) |
| 02 | SP2 (22.10 Vapor) |
| 03 | SP3a (Rx. Effluent) |
| 04 | SP4 (Reactor Feed) |
| 05 | SP8 (21.10 Vapor) |
| 06 | SP10 (07.20 Vapor) |

| **GC ROCCO** | |
|---|---|
| Detectors: FIDs (Alcohols) | |
| Sampling: MANUAL | |
| Sample Port | Sample Tag |
| 01 | Std. 5 (MeOH/DME Gas) |
| 02 or | SP3a (Rx. Effluent) |
| 03 | SP15 or SP6 (22.11) |

| **GC BHARAT** | |
|---|---|
| Detectors: FIDs (Alcohols) | |
| Sampling: MANUAL | |
| Sample Port | Sample Tag |
| 01 | Std. 5 (MeOH/DME Gas) |
| 02 | SP3a (Rx. Effluent) |

Table B-5

## COMPONENT LIST FOR LPMEOH/DME (OCT'99)

| COMPONENT # | COMPONENT NAME | CHEMICAL FORMULA | MOLECULAR WT. |
|---|---|---|---|
| 1 | Hydrogen | $H_2$ | 2.02 |
| 2 | Nitrogen | $N_2$ | 28.01 |
| 3 | Carbon Monoxide | CO | 28.01 |
| 4 | Methane | $CH_4$ | 16.04 |
| 5 | Carbon Dioxide | $CO_2$ | 44.01 |
| 6 | Water | $H_2O$ | 18.02 |
| 7 | Ethane | $C_2H_6$ | 30.07 |
| 8 | Ethylene | $C_2H_4$ | 28.05 |
| 9 | Porpane | $C_3H_8$ | 44.10 |
| 10 | Methanol | $CH_3OH$ | 32.03 |
| 11 | DME | ME | 46.07 |
| 12 | Methyl Formate | MF | 60.05 |
| 13 | Ethanol | L2 | 46.07 |
| 14 | Isopropanol | | 60.10 |
| 15 | Pentane | | 72.15 |
| 16 | Methyl Acetate | MEAC | 74.08 |
| 17 | 1-Propanol | L3 | 60.10 |
| 18 | 2-Butanol | SBOH | 74.12 |
| 19 | Ethyl Acetate | ETAC | 88.11 |
| 20 | Isobutanol | IBOH | 74.12 |
| 21 | 1-Butanol | L4 | 74.12 |
| 22 | Methyl Isobutyrate | | 102.00 |
| 23 | 2-Methyl-1-Butanol | | 88.15 |
| 24 | 1-Pentanol | L5 | 88.15 |
| 25 | 2-Methyl-1-Pentanol | | 102.18 |
| 26 | 1-Hexanol | $C_6H_{14}O$ | 102.18 |

Table B-6

## STANDARD LIST FOR LPMEOH/DME (OCT'99)

| Cyl. # SG282464 | REDUCTION GAS |
|---|---|
| Bar Code: DRM353 | |
| Hydrogen | 2.04 % |
| Nitrogen | 97.96 % |

| Cyl. # SG896067ALB | TEXACO GAS |
|---|---|
| Bar Code: DRM354 | |
| Hydrogen | 34.10 % |
| Nitrogen | 1.13 % |
| Carbon Dioxide | 13.70 % |
| Carbon Monoxide | 51.07 % |

| Cyl. # SG24175 | MeOH/DME |
|---|---|
| Bar Code: DVE494 | Gas |
| Methanol | 2.93 % |
| DME | 2.99 % |
| Nitrogen | Balance |

| Cyl. # SG915085BAL | SHELL GAS |
|---|---|
| Bar Code: DLU146 | |
| Hydrogen | 29.40 % |
| Nitrogen | 1.13 % |
| Carbon Dioxide | 3.08 % |
| Carbon Monoxide | 66.39 % |

| Cyl. # SGN40137 | MeOH/DME |
|---|---|
| | Gas |
| Methanol | 2.93 % |
| DME | 2.99 % |
| Nitrogen | Balance |

# APPENDIX C

## LPDME Test Authorizations

## Test Authorization No. 56

RUN NUMBER: AF-A13
APPROX. START DATE: 11 October, 1999

TITLE: IN-SITU METHANOL CATALYST ACTIVATION USING DILUTE $H_2$ PRIOR TO DME SYNTHESIS RUN

OBJECTIVE:
To activate the Liquid-Phase Methanol (LPMEOH) synthesis catalyst.

SUMMARY:
Approximately 893 lbs of a methanol catalyst and 47 lbs of a dehydration catalyst are to be slurried with Drakeol-10 oil, transferred to the 27.20 reactor and activated with dilute $H_2$ (3% in nitrogen). Approximate run time is 2 days.

SAFETY IMPLICATIONS:
Operators should wear protective gear while loading catalyst to protect them from the dust and hot vapor which may be released from the loading nozzle. Protective gear including face shield should be worn during slurry sampling.

This operation will require the venting of unreacted $H_2$. During a previous activation (performed under TEST AUTHORIZATION #29) the off-gas was blended with methane and burned in the flare. Previous calculations (for TA #23) indicated that in the event a combustible mixture could not be maintained, there would be no danger to personnel from venting. The reduction gas flow rates to be used in this run are less than those used in TA #23.

ENVIRONMENTAL IMPLICATIONS:
Minimal, a flame will be maintained at the flare. At 98% destruction efficiency, the $H_2$ emission rate would be 0.04 lb/hr.

SPECIAL REMARKS:
$H_2$ concentrations in and out of the reactor must be monitored closely during the reduction. Reactor temperature must be closely monitored and controlled per the attached TEST DETAILS. The utility oil inlet temperature (TI 1246) to the 27.20 internal heat exchanger must not exceed a 200°F difference from the utility oil outlet temperature (TI-1244) or the reactor slurry temperature. When adjusting flows or pressure, care should be taken to minimize catalyst carryover (caused by high gas velocity).

AUTHORIZATIONS:

E. C. Heydorn, Plant Mgr

B. L. Bhatt, Process Engr

TEST DETAILS:

1. This reduction procedure follows previous methanol catalyst reductions from the LPIII ER-6 reduction (TEST AUTHORIZATION #23), 1991 DME run (#25), 1992 LPSHIFT run (#29), 1994 methanol run (#37), 1995 hydrodynamic run with baseline catalyst (#46) and, 1995 hydrodynamic run with an alternate catalyst (#48).

2. Charge the 28.30 prep tank with 1746 lb of oil (247 gallons of Drakeol-10 at 80°F). The oil should be transferred to drums and weighed using the scale for accurate measurement. Heat this oil to 150-200°F.

3. Fill the 27.14 intermediate V/L separator to 25 nuts on LG-358 with approximately 100 gallons of Drakeol-10 oil from storage.

4. When the prep tank oil is at 150-200°F, add 893 lbs of the methanol catalyst (4 full drums and a portion from a fifth drum) and 47 lbs of the dehydration catalyst. Add the catalysts very slowly to make a 35 wt% oxide slurry. Keep the slurry well stirred to prevent agglomeration of the catalyst.

5. Heat the slurry to 200°F and continue agitation, under nitrogen, for at least 2 hours to ensure good mixing.

6. Establish gas flow through the reactor using nitrogen through V-2627 to prevent slurry back-flow into the distributor. Vent the gas through PV-1261.

7. Pressure transfer the slurry to the reactor and verify operation by noting level with the nuclear density gauge (NDG- estimated level: 22 to 26 ft.)

8. Flush out the prep tank with 283 lb of oil (40 gallons of Drakeol-10 at 80°F). Pressure transfer the flush oil to the reactor and verify level with the NDG (LI-1242).

9. Close V-645 to prevent utility oil flow back to the prep tank and establish full utility oil flow through the 27.20 internal heat exchanger.

10. Pressurize the reactor loop to 67 psig.

11. Begin heating the slurry to 200°F, following TAVR on the DEC console. Check that the slurry temperatures are in reasonable agreement. Verify that the slurry is well mixed by performing a NDG scan.

12. Establish Dilute $H_2$ reduction gas flow at 12400 SCFH (on FI-126) and vent the flow through PV-170. Establish the following composition:

| | Composition | Est. Flows (SCFH) |
|---|---|---|
| $H_2$ | 3.0 | 370 |
| $N_2$ | 97.0 | 12030 |
| | 100.0 | 12400 |

MW = 27.2, SCF evaluated at 70°F, 14.7 psia
Target space velocity = 800 sL/h-kg; Target starting inlet superficial velocity = 0.62 ft/sec

13. When the reactor temperature reaches 200°F, bring reduction gas to the reactor slowly and close the nitrogen purge (V-2627). Establish a final flow to the reactor of 12,400 SCFH. Maintain flow and reducing gas composition as specified in step 13. The temperature-programmed activation consists of the following steps:

| Initial Temperature °F | Final Temperature °F | Heat-up Rate °F/hr | Time Required Hours |
|---|---|---|---|
| | | | |
| 200 | 257 | 15 | 4 |
| 257 | 302 | 9 | 5 |
| 302 | 392 | 15 | 6 |
| 392 | 392 | 0 | 4 |
| 392 | 464 | 15 | 5 |
| | | | Total = 24 |

$H_2$ concentrations are to be measured continuously for the feed and effluent streams. As long as the $H_2$ consumption at a given temperature is equal to or greater than the autoclave reduction data then the activation is proceeding well. Figure C-1 shows the consumption profile vs temperature from the labs. If the cumulative consumption curve falls below the autoclave curve, consult the process or research engineer to reduce the heat up rate.

If the $H_2$ concentration in the effluent falls below 0.1 mole %, increase the inlet $H_2$ concentration to 4% after consulting the process engineer. The objective here is to prevent reduction gas starvation. The reduction is expected to be complete before reaching 464°F. It may become necessary to hold slurry at this temperature until the difference between inlet and outlet $H_2$ concentration falls below 0.05 mole %.

14. The slurry level should be maintained between 90 and 95% of NDG range (approximately 40 ft.) by using LIC 1242 to control the makeup oil rate. Note that as the reactor is heated to 464 F, the slurry will expand. At the same time, some of the oil will be lost in the reactor effluent. If authorized by the process engineer or the plant manager, additional makeup oil

can be added to the system via the 27.14 by following the standard procedure; FQI-334 readings and the change in level of the 27.14 should be recorded before and after each addition. It is important to note that the discharge valve of the 10.52.01 and 02 pumps should be used to throttle to the 67 psig reactor pressure. The pressure in the sump of the 21.11 should be at 150 psig or less.

15. Record any indication of density or viscosity change, such as a change in the pressure drop across the reactor or shaking of the reactor during heat up and reduction.

16. During the reduction, scan the reactor with the NDG. At the end of the reduction, add fresh oil to 27.14 to bring the level up to 25 nuts on LG-358. This charge should be drawn from storage.

TA #56 is done, consult TEST AUTHORIZATION #57 for the next step.

ANALYTICAL REQUIREMENTS:

1. Catalyst sampling requirements:

    - slurried oxide catalyst from prep tank before reduction,

    Exact quantities to be determined by operations, process, and research.

2. Composition sampling requirements:

    - reactor in and out continuously
    - $H_2$ is critical
    - $CO_2$ and $N_2$ are also required

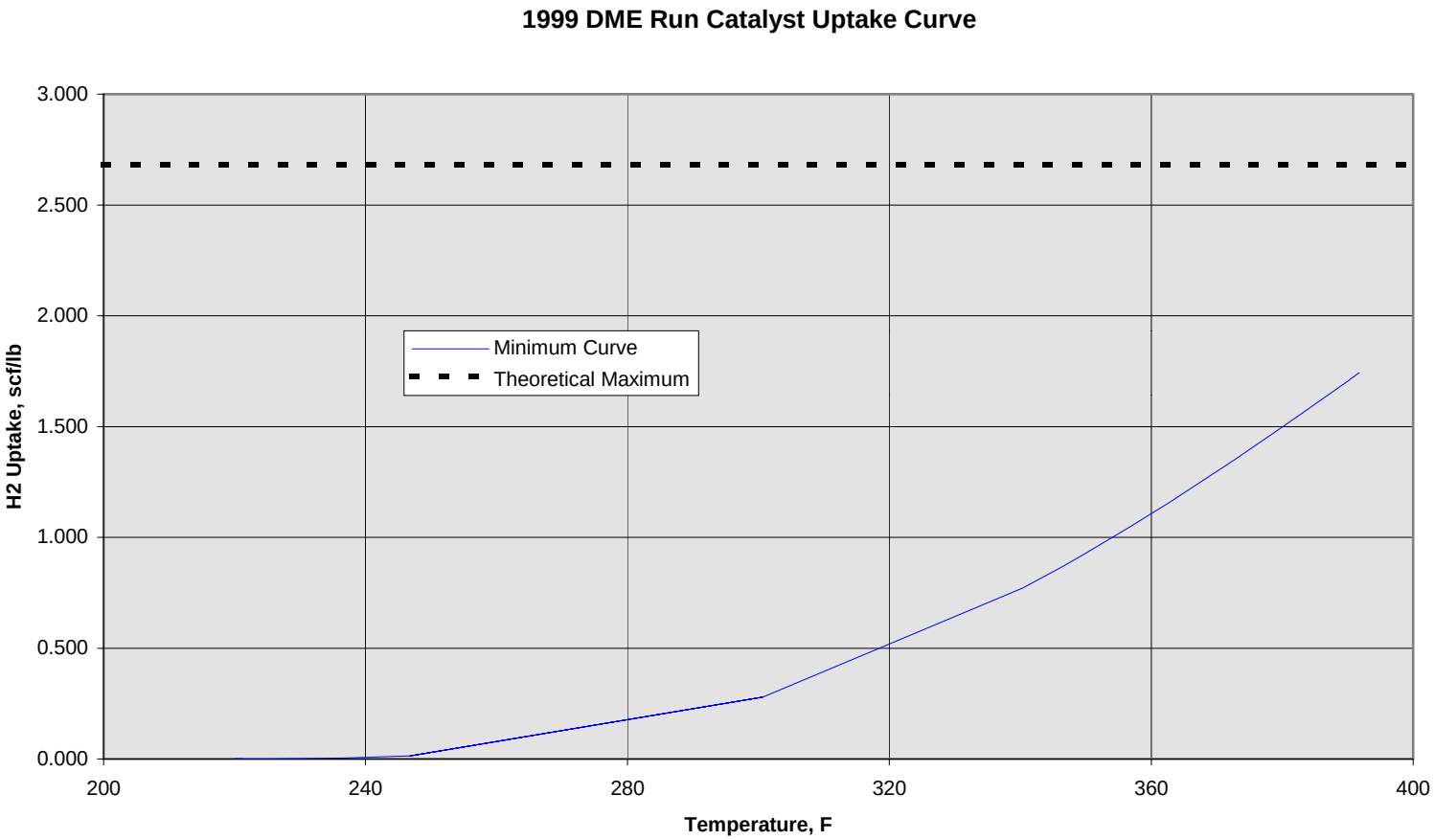
3. Flow measurement requirements:

    - reactor in at FI-126 and FI-299

REFERENCES:

1. TEST AUTHORIZATION # 23 : Procedure for previous in-situ activation.

Figure C-1


1999 DME Run Catalyst Uptake Curve
3.000
2.500
2.000
1.500
1.000
0.500
0.000
H2 Uptake, scf/lb
Minimum Curve
Theoretical Maximum
200
240
280
320
360
400
Temperature, F

## Test Authorization No. 57

RUN NUMBER: AF-R17
APPROX. START DATE: 13 October, 1999

TITLE: DIMETHYL ETHER SYNTHESIS WITH A MIXTURE OF METHANOL AND DEHYDRATION CATALYSTS

OBJECTIVE: To study the liquid phase dimethyl ether process in the 27.20 pilot plant reactor using commercially produced catalysts.

SUMMARY:
Upon completion of the activation step (AF-A13), the reactor feed will be adjusted to a Shell gas composition (30% $H_2$, 66% CO, 3% $CO_2$, 1% $N_2$). For approximately 18 days, the conditions will be targeted at 750 psig, 482°F, 6,000 sL/kg-hr space velocity (GHSV), and 35 wt% oxide in oil for a catalyst life study. A tracer scan will be conducted on the last day of the life study. Radioactive gas and solid tracers will be injected to study the mixing of the gas, liquid as well as the solid phases. The space velocity will then be lowered to 3,100 GHSV to evaluate a high conversion case. The third condition would evaluate a stochiometric feed composition (1:1 $H_2$/CO, 3% $CO_2$) at 6,000 GHSV. A high velocity condition will be tested next (0.74 ft/sec) with Shell gas. A second radioactive tracer test will be conducted at this condition. The fifth operating condition would evaluate the reactor performance at a higher concentration (40 wt%) by operating at a lower slurry level in the reactor. The unit will be shut down following the fifth condition after a total operating period of approximately 25 days.

SAFETY IMPLICATIONS:
Protective gear including face shield should be worn during slurry sampling. Safety information available from ICI Tracerco is attached (Letters from D. A. Bucior to B. L. Bhatt, "Radiation Safety Analysis of Proposed Methanol Reactor Residence Time and Distribution Study", 3 June 1993; "Radiation Safety Analysis of Proposed LaPorte Pilot Plant Radioactive Tracer Study", 10 February 1994; "Tracerco Radiation Analysis", 15 June 1995). Barricades will be erected by ICI Tracerco to prevent access to areas containing radioactive materials. Radiation film badges will be worn by all personnel present during the study.

ENVIRONMENTAL IMPLICATIONS: Minimal.

SPECIAL REMARKS:
The high pressure hydrogen pipe line will be in use during most of the run. The $CO_2$ removal system will be in operation throughout the run. The radioactive tracer injection will be performed by ICI Tracerco personnel using their injection equipment. APCI personnel will be present during the injection and operate AFDU equipment.

AUTHORIZATIONS:

E. C. Heydorn, Plant Mgr

B. L. Bhatt, Process Engr

TEST DETAILS:

1. Prior to completion of TA #56 (run AF-A13), the $CO_2$ removal section is charged with approximately 638 gallons of methanol . Follow the procedures set forth by operations. The breakdown of methanol inventory in the back-end is:

| | | |
|---|---|---|
| 07.10 | 130 gal | (54 gals above normal liquid level of 5 ft) |
| 07.20 | 207 gal | (61 gals above normal liquid level of 6 ft) |
| 07.22 | 132 gal | (full) |
| 21.45/21.65 | 117 gal | (full) |
| piping | 52 gal | |

   The inventory numbers in the 07.10 and 07.20 include the normal sump liquid level plus the liquid which will be held up in the packing once the 10.80 circulation pump is started. Upon completion of the TA #56 (run AF-A6), the 22.10, 22.15 and 22.16 should be empty, the reactor loop should be de-pressurized, and the $CO_2$ removal section should be blocked-in.

2. Upon completion of the catalyst activation (AF-A13), increase the reactor pressure to 750 psig and introduce syngas while the reactor is approximately 430-460 F. The target for the slurry temperature at 482°F. Slowly increase the reactor feed rate to 25,000 SCFH, while maintaining slurry level at 95% of NDG span (480" on tape). The reactor feed composition should correspond to run AF-R17.1 conditions (Shell gas, refer to Table B-1) and the effluent should be directed to flare (once-through operation). In the event of a premature shut-down, consult TA #20 (RUN E-05) for appropriate standby conditions.

3. Once reactor feed flows have been established, bring the $CO_2$ removal section on-line by opening valves V-2001,V-2003,V-2004,V-2006 and shutting V-2000. When the $CO_2$ removal section is functioning properly, recycle flow may be slowly brought in and the fresh feed flows reduced so that they match the target in Table C-1 for AF-R17.1. Note that the HP hydrogen pipeline is in service during all the cases except run AF-R17.2.

4. When the target feed rate has been achieved, put LIC-1242 in automatic to control slurry level at 95% (480" on tape). Adjust the fresh feed flow to achieve an initial purge flow rate of approximately 3,000 SCFH. Maintain reactor feed flow and reactor temperature and pressure at the case AF-R17.1 values for next 18 days.

5. During the first 24 hours, the syngas conversion across the reactor will fall as the catalyst loses its hyperactivity. The purge flow will increase and the reactor feed composition will be changing during this period. When these rates of change diminish, fine tune the fresh feed flow to reach the desired reactor feed composition as specified for case AF-R17.1. The ultimate purge rate should be around 4,200 SCFH.

6. Monitor the air-cooler loading and temperature difference between the utility oil and the slurry and utility oil inlet & outlet using TI-1246 and TI-1244. Both of these temperature differences must be below 200°F.

7. The composition of the methanol product is to be monitored every 8 hours. The target oil content of the methanol product should be <=0.2 wt%. If the oil content is higher, lower the 21.11 effluent outlet TIC-1260 set point.

8. SPECIAL CONSIDERATIONS:

WATER BUILD-UP IN $CO_2$ REMOVAL SECTION: Water and higher alcohols enter the back-end of the plant through the vapor off the 22.10 and remain. Additionally, methanol escapes from the back-end with the vapors off the 07.20. As a result, the concentration of water in the circulating methanol increases with time-on-stream. Water is a poor solvent for $CO_2$ and the effectiveness of the circulating liquid will diminish with time. Therefore, it will be necessary to increase 10.80 flow and/or increase plant purge during any given run. The composition of the methanol solvent in the $CO_2$ removal section will be monitored regularly and, if necessary, methanol will be drained out (via the 22.11 and 22.15) and fresh methanol will be added using the 10.85 pump.

WATER FREEZE-OUT IN 21.10 TUBESIDE: Although unlikely, it is possible for the water contained in the 22.10 overhead to plate-out on the tubes of the 21.10. This process, if it occurs at all, will be slow. Indications of freeze-out include: excessive pressure drop, colder temperature at TI-233, and warmer temperature at TI-188. Corrective action is to operate the 10.85 pump to inject methanol into the 21.10 tubeside inlet.

9. Maintain conditions for approximately 18 days.

10. Preparation and initial testing of the tracer study equipment will be conducted on the 17$^{th}$ day of the Catalyst Life Study. A vapor residence time distribution study will be performed by injecting Argon-41 into the inlet gas line and monitoring its progress through the reactor by several detectors. Also, four injections each of radioactive $Mn_2O_3$ fine particles as well as manganese oxide doped gamma alumina will be made in the reactor slurry to study liquid and solid phase mixing. The electronic equipment includes 28 detectors connected to a data acquisition system. The process equipment includes a gas sample cylinder with adequate valves to allow filling of the cylinder with Ar-41 and subsequent injection into the reactor via a nitrogen flush. The Ar-41 will be injected thru valve V-2462. The radioactive manganese as well as catalyst will be sluried in Drakeol-10 and injected with a hand-powered piston pump. The liquid injections will be made at two locations on the side of the reactor: Top N1-nozzle and Bottom N2-nozzle. The electronic equipment will be connected and power supplied to it. A preliminary calibration will be performed to verify the equipment is operational. Arrangements will be made to support the detectors in their proper location, detectors will not be placed until the next day. Personnel available during the study will include two persons from Tracerco, one operator, one GEG process engineer and the plant manager.

ICI Tracerco is licensed to conduct these tests and will conform to guidelines prescribed by the Texas Department of Health. Texas A&M will irradiate the argon, the manganese oxide and the catalyst on the morning of the tests, and they will be delivered to the LaPorte site by courier. The radioactive Ar-41 has a half life of 1.8 hrs and will be injected into the reactor and vented to the atmosphere in levels acceptable to the Texas Department of Health. The radioactive manganese is expected to have a half life of 2.5 hours; only small amounts acceptable to the Texas Department of Health will be injected. An irradiation test will be performed on $Mn_2O_3$ and the catalyst by Texas A&M to ensure that the radiation will decay to very low levels within several days.

Conduct a shut-down test to determine catalyst inventory and verify the gas hold-up estimates using the nuclear density gauge and differential pressure measurements with no gas flow through the reactor.

11. The radioactive tracer experiments will be conducted on the 18$^{th}$ day of the life study. Prior arrangements will be made to irradiate Ar-41, $Mn_2O_3$ and, the manganese doped catalyst in a reactor at Texas A&M. The irradiation will take place on the morning of the day of experiment and be transported to LaPorte by 1 pm. The radioactive materials produced can only be used during that day since the half life of these compounds is less than three hours. During the morning, the Tracerco crew will calibrate and hang the 28 detectors at the LaPorte AFDU. Each detector will be subjected to a gamma-ray source, and the response will be measured. All the detectors will then be normalized relative to the most sensitive detector.

    After the calibration is complete, the detectors will be placed at specified locations. Two Ar-41 injections will be made into the feed gas. A reasonable amount of time must exist between injections so that either Ar-41 has left the system or a steady level of radiation is available to use as a baseline. Four liquid injections will be made at the top nozzle (one near the wall and another half way between the wall and the center). Four more injections (wall and center) will be made at the bottom.

    At the beginning and at the end of the experiment, liquid level, gas hold up and slurry concentration will be measured with the nuclear density gauge (NDG). Two sets of detectors below the liquid level will have to be removed for the NDG measurements. The slurry level should be maintained between 80 and 100% of NDG range, as specified in Table 1. Leave the Nuclear Density Gauge at the normal controlling reactor height. Shut off the gauge for 10-15 minutes during the injections as cross-interference is expected. Put LIC-1242 on manual during the injections but bring it back on auto between runs. Pump 10.52.02 should be on all the time to bring oil back to the reactor. Pump 10.52.01 should be on all the time to circulate oil thru 27.14. Maintain 27.14 separator between 270-290$^{O}$F. Maintain 22.10 separator between 90-115$^{O}$F.

12. After conferring with the process engineer or plant manager, switch to AF-R17.2 run conditions (Low Velocity). Run this data period for approximately 1 day.

13. After conferring with the process engineer or plant manager, switch to AF-R17.3 run conditions (Stochiometric Feed Gas). Run this data period for approximately 1 day.

14. After conferring with the process engineer or plant manager, switch to AF-R17.4 run conditions (High Velocity). Run this data period for approximately 2 days. A second tracer experiment will be conducted on the 2$^{nd}$ day of this condition. Tracer experiments similar to those described in step 8 will be repeated at this condition. After completing the study, Tracerco will remove their equipment from the LaPorte site.

15. After conferring with the process engineer or plant manager, switch to AF-R17.5 run conditions (High Concentration). Run this data period for approximately 1 day. Conduct a second shut-down test at the end of the run to determine catalyst inventory and verify the

gas hold-up estimates using the nuclear density gauge and differential pressure measurements with no gas flow through the reactor.

16. Upon completion of the shut-down test at the end of case AF-R17.5, this test run is done. De-pressurize the plant and proceed with shut-down.

ANALYTICAL COMMENTS:

1. Catalyst sampling requirements:
   - slurried catalyst at the end-of-run.

   Exact quantities to be determined by operations, process, and research.

2. Continuous composition sampling requirements (GC):
   - fresh feed,
   - reactor in,
   - reactor out,
   - recycle
   - 22.10 overheads
   - 07.20 offgas

3. Periodic composition sampling requirements (GC):
   - 22.11 off-gas (frequency to be determined by operations & process)

   Periodic composition sampling requirements (LC):
   - methanol product (every 8 hours during first two days, twice a day thereafter)
   - 10.80 suction liquid (once every two days or as required)
   - 27.14 and 21.11 oil samples (as directed by operations & process)

4. Flow measurement requirements:
   - fresh feed,
   - reactor in,
   - reactor out,
   - recycle,
   - purge,
   - 22.11 off-gas,
   - methanol product (by level)
   - 22.10 overhead
   - 07.20 off gas
   - 10.80 flow

REFERENCES:

1. TEST AUTHORIZATION #20 - Procedures for reactor standby during shutdown.
2. STANDARD STARTUP PROCEDURES FOR MeOH-ONLY OPERATION
3. STARTUP PROCEDURES FOR OPERATION WITH $CO_2$ REMOVAL.

Table C-1

## Run Plan for Fall 1999 DME Demonstration

| **RUN** | | | AF-A13 | AF-R17.1 | AF-R17.2 | AF-R17.3 | AF-R17.4 | AF-R17.5 |
|---|---|---|---|---|---|---|---|---|
| Description | - - - | - - - | Reduction | Baseline | Low Velocity | Stoich Feed | High Velocity | High Cat. |
| Duration | - - - | days | 2 | 18 | 1.5 | 1.5 | 2.5 | 1.5 |
| Syngas | - - - | - - - | - - - | SHELL | SHELL | 1:1 H2/CO | SHELL | SHELL |
| Inlet Space Velocity | - - - | sL/kg-hr | 800 | 6000 | 3100 | 6000 | 8000 | 8000 |
| Reactor Pressure | PIC-1247 | psig | 67 | 750 | 750 | 750 | 750 | 750 |
| | | | | | | | | |
| **REACTOR** | | | | | | | | |
| Pressure | PIC-1247 | psig | 67 | 750 | 750 | 750 | 750 | 750 |
| Temperature | TI-1233 | F | - - - | 482 | 482 | 482 | 482 | 482 |
| Heat Duty | | MM BTU/hr | - - - | -1.260 | -0.907 | -1.675 | -1.430 | -1.430 |
| Inlet Superficial Velocity | - - - | ft/sec | 0.62 | 0.56 | 0.29 | 0.56 | 0.75 | 0.75 |
| Outlet Superficial Velocity | - - - | ft/sec | - - - | 0.52 | 0.28 | 0.46 | 0.69 | 0.69 |
| Liquid Level | LI-2142 | % span | | 100 | 100 | 100 | 100 | 81 |
| Catalyst Load | - - - | lb | 940 | 940 | 940 | 940 | 940 | 940 |
| Cat Concentration | - - - | wt% | 35 | 36 | 35 | 35 | 37 | 40 |
| Vapor Void Fraction | - - - | vol% | | 40 | 35 | 37 | 43 | 39 |
| | | | | | | | | |
| **FEED FLOWS** | | | | | | | | |
| LP H2 | FIC-101 | scfh | | 16271 | 11686 | 14205 | 14165 | 14165 |
| CO | FIC-104 | scfh | | 18462 | 13869 | 20512 | 20573 | 20572 |
| CO2 | FIC-107 | scfh | | 0 | 0 | 0 | 0 | 0 |
| N2 | FIC-111 | scfh | | 0 | 0 | 0 | 0 | 0 |
| N2 | FIC-111 | scfh | | 83 | 82 | 99 | 79 | 79 |
| 01.10 Total Flow | FI-726 | scfh | | 34817 | 25638 | 34817 | 34817 | 34817 |
| | | | | | | | | |
| HP H2 | FIC-1200 | scfh | | 2213 | 0 | 13756 | 7960 | 7960 |
| 01.20 Recycle | FIC-246 | scfh | | 58806 | 23961 | 47246 | 85363 | 85364 |
| | | | | | | | | |
| **10.95 PUMP INJECTION** | | | | | | | | |
| Total Flow | FI-1221 | gpm | zero | zero | zero | zero | zero | zero |
| MEOH | - - - | wt% | - - - | - - - | - - - | - - - | - - - | - - - |
| C2OH | - - - | wt% | - - - | - - - | - - - | - - - | - - - | - - - |
| C3OH | - - - | wt% | - - - | - - - | - - - | - - - | - - - | - - - |
| | | | | | | | | |
| **REACTOR FEED** | | | | | | | | |
| Target Feed Temp | TI-1253 | F | - - - | 400 | 400 | 400 | 400 | 400 |
| Feed Dewpoint | - - - | F | - - - | 6.9 | 18.9 | 20.6 | 26.0 | 26.0 |
| Total Dry Flow | FI-1216 | scfh | | 95835 | 49602 | 95822 | 128144 | 128144 |
| H2 | - - - | mol% | 4 | 29.9 | 29.9 | 47.9 | 29.9 | 29.9 |
| CO | - - - | mol% | 0 | 65.8 | 65.9 | 47.9 | 65.8 | 65.8 |
| N2 | - - - | mol% | | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 |
| N2 | - - - | mol% | 96 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 |
| CO2 | - - - | mol% | 0 | 3.0 | 3.0 | 3.0 | 3.0 | 3.0 |
| MEOH | - - - | mol% | 0 | 0.1 | 0.0 | 0.1 | 0.1 | 0.1 |
| DME | - - - | mol% | 0 | 0.2 | 0.1 | 0.1 | 0.2 | 0.2 |
| C1 | - - - | mol% | 0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| | | | 100 | 100.0 | 100.0 | 99.9 | 100.0 | 100.0 |
| | | | | | | | | |

Table C-1 (Contd.)

## Run Plan for Fall 1999 DME Demonstration

| **RUN** | | | AF-A13 | AF-R17.1 | AF-R17.2 | AF-R17.3 | AF-R17.4 | AF-R17.5 |
|---|---|---|---|---|---|---|---|---|
| Description | - - - | - - - | Reduction | Baseline | Low Velocity | Stoich Feed | High Velocity | High Cat. |
| | | | | | | | | |
| **21.11 Feed/Product Exchanger** | | | | | | | | |
| Feed Inlet Temp | TI-1257 | F | - - - | 150 | 150 | 150 | 150 | 150 |
| Feed Outlet Temp | TI-1263 | F | - - - | 400 | 400 | 400 | 400 | 400 |
| Total Feed to 02.63 Temp | TI-1216 | F | - - - | 364 | 360 | 357 | 366 | 366 |
| Reactor Eff. Inlet Temp | TI-1262 | F | - - - | 482 | 482 | 482 | 482 | 482 |
| Reactor Eff. Outlet Temp | TIC-1260 | F | - - - | 250 | 250 | 250 | 250 | 250 |
| Reactor Eff. Dew Temp | - - - | F | - - - | 177 | 155 | 238 | 179 | 179 |
| | | | | | | | | |
| **REACTOR EFFLUENT** | | | | | | | | |
| Total Flow | FI-196 | scfh | - - - | 74783 | 35545 | 66858 | 103359 | 103359 |
| H2 | - - - | mol% | - - - | 14.8 | 10.6 | 29.7 | 16.6 | 16.6 |
| CO | - - - | mol% | - - - | 65.7 | 63.8 | 42.6 | 66.0 | 66.0 |
| N2 | - - - | mol% | - - - | 1.3 | 1.4 | 1.4 | 1.2 | 1.2 |
| CO2 | - - - | mol% | - - - | 8.4 | 12.5 | 8.6 | 7.2 | 7.2 |
| MEOH | - - - | mol% | - - - | 4.9 | 3.1 | 12.9 | 5.1 | 5.1 |
| DME | - - - | mol% | - - - | 4.8 | 8.4 | 4.4 | 3.7 | 3.7 |
| C1 | - - - | mol% | - - - | 0.0 | 0.0 | 0.1 | 0.0 | 0.0 |
| | | | - - - | 99.8 | 99.8 | 99.6 | 99.8 | 99.8 |
| **PRODUCT RECOVERY** | | | | | | | | |
| Syngas to Backend Flow | FI-682 | scfh | - - - | 71354 | 34498 | 57482 | 98548 | 98548 |
| 22.11 to Flare Flow | FI-237 | scfh | - - - | 294 | 164 | 752 | 330 | 330 |
| Main Flare Flow | FI-245 | scfh | - - - | 4567 | 4225 | 4653 | 4312 | 4312 |
| Product Flow | - - - | gpd | - - - | 980 | 280 | 2695 | 1395 | 1395 |
| BACK-END | | | | | | | | |
| MEOH Circulation | FIC-814 | gpm | None | 13.1 | 6.3 | 9.5 | 18.1 | 18.1 |
| MEOH to 07.10 Temp | TI-814 | F | - - - | 9.2 | -20.0 | 17.3 | 22.9 | 22.9 |
| | | | | | | | | |
| 07.10 OH Temp | TI-1275 | F | - - - | 17.6 | -4.9 | 26.9 | 29.6 | 29.6 |
| 07.10 Pressure (top) | PI-1280 | psig | | 695 | 695 | 695 | 695 | 695 |
| 07.20 Pressure (top) | PIC-7291 | psig | | 165 | 165 | 165 | 165 | 165 |
| 07.20 DME to Flare Flow | FI-7291 | scfh | - - - | 7462 | 6096 | 5211 | 8161 | 8161 |
| 07.20 Reboiler Temp | TIC-7339 | F | - - - | 293 | 294 | 293 | 294 | 294 |
| | | | | | | | | |
| 07.22 Steam Pressure | PIC-7338 | psig | - - - | 550 | 550 | 550 | 550 | 550 |
| 07.22 Steam Usage | FI-7338 | lb/hr | - - - | 989 | 559 | 501 | 1272 | 1234 |
| 21.80 CO2 Usage | - - - | TPD | - - - | 25.2 | 15.5 | 19.3 | 34.2 | 34.2 |
| Total CO2 Usage | Fd+21.80 | TPD | 0 | 25.2 | 15.5 | 19.3 | 34.2 | 34.2 |
| | | | | | | | | |
| **RECYCLE FEED** | | | | | | | | |
| H2 | - - - | mol% | - - - | 17.3 | 13.2 | 37.9 | 18.9 | 18.9 |
| CO | - - - | mol% | - - - | 75.8 | 78.5 | 53.7 | 74.6 | 74.6 |
| N2 | - - - | mol% | - - - | 1.5 | 1.7 | 1.8 | 1.4 | 1.4 |
| CO2 | - - - | mol% | - - - | 4.9 | 6.2 | 6.1 | 4.5 | 4.5 |
| MEOH | - - - | mol% | - - - | 0.1 | 0.1 | 0.1 | 0.2 | 0.2 |
| DME | - - - | mol% | - - - | 0.3 | 0.2 | 0.3 | 0.3 | 0.3 |
| C1 | - - - | mol% | - - - | 0.1 | 0.0 | 0.1 | 0.0 | 0.0 |
| | | | - - - | 99.9 | 99.9 | 99.9 | 99.9 | 99.9 |
| | | | | | | | | |

# APPENDIX D

## LPDME Run Chronology

**Liquid Phase DME Demonstration - October/November 1999**

Monday, Oct. 4, 1999

8:00 Checked nuke level on reactor structure versus control room readout at 31 points
9:00 NDG calibration at atmosphere pressure w/ $N_2$
10:45 Nuke locked out in preparation for radial detector installation
11:00 Radial detector installation begins
1530 Source installed & background test began

Tuesday, Oct. 5, 1999

Radial Nuke Scan

Determined that sampling frequency of 1 data point every six seconds gave data w/ least noise. Total sampling time was 5 min.

Reproducibility checked by collecting data from North side, then moving source to East side, and finally moving back to North side. Reproducibility was within 8%.

Next experiment was moving the source away from the reactor wall (0"; 2"; 4"). Counts decreased by the expected amount. Best to keep source flush w/ wall to ensure high counts for all the detectors. This was done for both North side source & East side source. In addition, 2 background measurements were taken & tests also done with extra shielding in some instances.

All Tracerco scans were done @ 16 ft. on tape.

NDG Calibration

1400 Calibration done @ 100 psig with $N_2$
1550 Calibration done with $N_2$ ~ 262 psig

Wednesday, Oct. 6, 1999

718 NDG Calibration done w /$N_2$ @ 765 psig
830 Depressurize & leak check & prepare for oil change & 4 additional radial scans
1145 Oil pressure-transferred from 28.30
Resulting level in reactor between 319" & 340" (on tape)
1200 Oil Temp in RXT = 70°F
Tracerco doing the gamma scan.

Thursday, Oct. 7, 1999

Morn. Ramping up utility oil @ 60°F/hr up to 250°F
1000 Utility oil reaches 250°F
1130 Slurry oil level between 340" & 351.9" on tape, T= 79°F
1144 As gas flow rate increased to reactor, level rose to between 424 & 445" on tape
1330 Liquid level returned to 320" vicinity in reactor due to rupture disk failure in CW line
1600 Liquid level near 424" on tape
1630 Liquid filled to top of reactor (530" on tape)

Friday, Oct. 8, 1999

Heat-up overnight was hampered by leakage around 21.40 bypass valves.
0813 UO system briefly shutdown to repair valves (stroke)

0925 UO system back in service and heating up
1030 Liquid level between 510" and 528"
UO inlet temp = 431°F Avg. Reactor temp = 422°F
1110 Start syngas to plant (3,000 sfch $H_2$/4,000 scfh CO)
(purging $N_2$ from system)
1245 Inc. $H_2$ to 4.5 and CO to 5.5
1255 Inc. $H_2$ to 5.0 and CO to 6.0
1315 Reactor level ~ 529.8" (full), Inc. $H_2$ to 6.0 and CO to 7.5
1325 Inc. CO to 8.0
1620 Liquid level @ 510" (NDG Reading = 136)
Reading @ 528" = 210, Reading @ 498" = 100

Sat., Oct. 9, 1999

0705 Nuke readings @ 528" ~ 240
511" ~ 136
498" ~ 212
460" ~ 229
299" ~ 65
Level @ ~ 297" 319" ~ 167
0830 Increasing 01"20 flow (recycle flow) & pumping oil from 21.11 & 27.14
to raise reactor level
1025 Level near 318" on tape (NDG Reading = 89.2)
1040 Level near 339" on tape (NDG Reading = 115)
1045 Level near 351" on tape (NDG Reading = 113)
1053 Level near 372" on tape (NDG Reading ~ 108)
1130 Level near 444" on tape (NDG Reading ~ 123)
1138 Level near 459" on tape
1145 Level near 468" on tape (NDG Reading ~ 125)
1153 Level near 484" on tape (NDG Reading ~ 120)
1421 FI-101 6 Mscfh
FI-104 8 Mscfh
FI-126 Mscfh 14.1 MW = 21.7 everywhere
FI-246 96.4 Mscfh
FI-1216A 101.2 Mscfh
FI-1216B 94.3 Mscfh
FI-1216C 6.4 Mscfh
FI-196 112.8 Mscfh
FI-682-1 104.9 FI- 682-2 75.7 Mscfh
FI-701A 104.4 Mscfh
FI-701B 0 Mscfh MW = 17 for these
FI-245A 0 B 0.07 Mscfh C 0 A= 12.2 Mscfh B= 0.07 C= 0
FI-237A 0.02 Mscfh B 0 C 0 Readings
FI-7291 0.1 Mscfh
FI-111 0 Mscfh FI-299 5.7 Mscfh
FI-1200 0 Mscfh

Sunday, Oct. 10, 1999

820 Nuke Scan
FT-1216A = 76.6" FI-1216A = 115.7 Mscfh
FT-1216B = 70.2" FI-1216B = 110.8 Mscfh

FT-1216C = 5.4" FI-1216C = 7.6 Mscfh
G06MW = 0 GO5MW = GO4MW = 17.2
FIC - 246 = 109.5

1030 Correct GO6MW tag in DEC to reference GO6Ave instead of GO5Ave
(See file Var617.dat) (found because GO6MW = 0)

| | | | MSCFH |
|---|---|---|---|
| 1255 | A CO Feed Flow | FIC-104 | 8.0 |
| | A $H_2$ Feed Flow | FIC-101 | 5.7 |
| | A 0110 Suct Flow A | FI-126A | 16.0 |
| | B | B | 16.1 |
| | A 0120 Disch Flow | FIC-246 | 108.3 |
| | A 2720 Dryfeed Flow A | FI-1216A | 114.4 |
| | B | B | 111.0 |
| | C | C | 3.6 |
| | A 2714 In Flow | FI-196 | 133.6 |
| | A 2210 Ovrhd Flow A | FI-701A | 117.3 |
| | B | B | 0 |
| | A 2210 Tube In Flow 682-1 | FI-682-1 | 117.7 |
| | -2 | -2 | 84.9 |
| | A Recyc Purg Flow A | FI-245-A | 11.6 |
| | B | B | 63.5 |
| | C | C | 0 |
| | A 2211 Ovrhd Flow A | FI-237-A | 0.15 |
| | B | B | 0 |
| | C | C | 0 |
| | A 0720 $CO_2$ Out Flow | FI-7291 | 0.2 |

SP1O MW = 17.32 SP6MW = 17.10

| | | | |
|---|---|---|---|
| 15 | 17.10 | 8 | 17.08 |
| 1 | 17.24 | | |
| 2 | 16.99 | | |
| 3A | 17.37 | | |
| 4 | 17.06 | | |

1300 Cooling Down Reactor. Syngas Shut-off. Burnout Done.

Monday, Oct. 11, 1999

1000 Began loading Drakeol - 10 into 28.30. The hose filling the drums was flushed w/Drakeol-10 prior to filling. Total of 1747 lbs. oil loaded into 28.30.

1300 Added 894 lbs MK-101P catalyst (Haldor-Topsoe) & 47 lbs $A1_2 O_3$ dehydration catalyst (Englehard A1-3916P) - Gamma Alumina
Average oil temp during addition 170°F

1430 Prepared 283 lbs of Drakeol - 10 in barrel to be used as 28.30 flush

1540 Transferred oil to 27.14 28 nuts

1545 28.30 Temp = 205F

1600 Pressured transferred slurry from 28.30 to 27.20
NDG readings: 151.3 @ 298.1 (LI)
169.2 @ 319.8 (LI)
222.6 @ 340.2 (LI)
141 @ 280.8 (LI)

1640 Pressure transferred oil wash from 28.30 to 27.20

1644 Nuke scan level near 280.4" (LI) = 279 on tape (recorded on log)
NDG reading = 6.3
1657 Flare lit
1730 Receiving GC date on Dennis3, Dennis 6, Gary 6 & Gary 3
1816 Starting to introduce reduction gas (FI-126A - 13,000 scfh)
NDG reading 21.7 @ 352 (LI)
205.3 @ 373
Dennis 6 & Gary 6 (Hydrogen in feed) = 1 mol%
Temp = 200.1 (Avg.)
1840 FI-126A = 12464 scfh
NDG reading 35@ 351.6 (LI) 206 @ 372.7 (LI)
Dennis 6 = 4.7 mol% $H_2$
Ave Temp = 200.5 P= 67.4 psig (PIC-1247)
1845 Adjusted $H_2$ rotameter to achieve 3% $H_2$
1850 Beginning temperature ramp @ 15°F/hr.
1900 FI-126A = 12470 scfh
Dennis 6 = 3.0 mol% $H_2$ (feed) Gary 3 = 2.96 mol% $H_2$ (effluent)
Tare = 202.2 P=68.1 psig
NDG; 206.6 @ 372.7 (LI)
38 @ 352.3 (LI)
2000 NDG: 200 @ 372.8 (LI)
@ 351.9 (LI)
Targ - 216.8°F
FI-126A = 12,216 scfh
FI-299 = 13089 scfh
Report press - 68.1 psig
LO3001 ($H_2$) = 3.00 %
Out
G06001 ($H_2$ IN) = 2.94%
G06MW = 27.21
G03MW = 27.21
DO3001 ($H_2$ IN) = 3.09%
DO3MW = 27.20
DO6MW = 27.20
SP1MW = 27.21
SP3AMW = 27.42
2105 NDG: 20.5 @ 373 LI
@ 352.1 LI
2200 NDG: 203.1 @ 373 LI
2250 Downloaded Data:
10/11/99 18:00 to 19:00 A1301.TXT
19:00 to 20:00 A1302.TXT
20:00 to 21:00 A1303.TXT
21:00 to 22:00 A1304. TXT
2255 Changed oil temp ramp to 9°F/hr. from 262 to 307°F
2300 NDG: 21.7 @ 351.8 LI
@ 373 LI
2305 Downloaded Data:
10/11/99 22:00 to 23:00 A1305.TXT

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0020 NDG: 48.3 @ 373.1 LI
Download Data:
10/11/99 23:00 to 24:00 A1306.TXT

0040 NDG: 34.4 @ 373.1 LI
209.5 @ 392.9 LI

0100 Download Data:
10/12/99 00:00 to 01:00 A1307.TXT

0145 NDG: 26.9 @ 373.1 LI
207.4 @ 393.3 LI
Download Data:
10/12/99 01:00 to 02:00 A1308.TXT

0255 NDG: 208.7 @ 393.3 LI
25.0 @ 373.9 LI

0302 Download Data:
10/12/99 02:00 to 03:00 A1309.TXT

0401 NDG: 28.9 @ 372.9 LI
@ 392.9 LI
Changed oil temp ramp to 15°F/hr. from 370°F to 392°F

0407 Download Data:
10/12/99 03:00 to 04:00 A1310.TXT

0443 NDG: 209.2 @ 392.9 LI
25.1 @ 373.1 LI

0515 Download Data:
10/12/99 04:00 to 05:00 A1311.TXT

0535 NDG: 24.9 @ 373.1 LI
208 @ 393.3 LI

0610 Download Data:
10/12/99 05:00 to 06:00 A1312. TXT

0635 NDG: 207.8 @ 393.3 LI
25.7 @ 373.2 LI
26.8 @ 352.1 LI
26.9 @ 340.4 LI
19.8 @ 319.4 LI

0705 Download Data: 10/12/99 06:00 to 07:00 A1313.TXT

0807 Download 07:00 to 08:00 A1314.TXT

0830 NDG reading 26.3 @ 372.5 (LI)
160 @ 393.2 (LI)

0907 Download 08:00 to 09:00 A1315.TXT
NDG reading 28.9 @ 351.5 (LI)
26 @ 373 (LI)
~162 @ 393.1 (LI)

1010 Download 09:00 to 10:00 A1316.TXT
NDG reading 26.3 @ 373.8 (LI)

1015 Beginning 4 hour hold (reactor @ 390F)

1055 NDG reading 27@ 373.8 (LI)
210.7 @ 393.7 (LI)
28.3 @ 351.9 (LI)

1106 Download 10:00-11:00 A1317.TXT

1130 NDG Scan Level near 351.9 (LI)

1209 Download 11:00→12:00 A1318.TXT

1255 Adding oil to 27.20 to bring level up near 393 on tape in preparation for temp ramp to 464F (not completed due to possible problem w/pump) (Level remains near 352 (LI)

1310 Download 12:00→13:00 A1319.TXT

1405 Began 18°F/hr ramp to 464°F

1408 Download 13:00→14:00 A1320.TXT

1800 Download 14:00→15:00 A1321.TXT
Download 15:00→16:00 A1322.TXT
Download 16:00→17:00 A1323.TXT
Download 17:00→18:00 A1324.TXT

1820 Reduction completed

1915 NDG Scan Reading Level (LI)
38.6 340.1
232 352.2
27.4 319.6

1930 Begin to switch over to syngas
Receiving GC data from Dennis 2; Gary 6; Dennis 3

2000 Started with once-thru operation

2100 Starting up with back-end

2200 Starting Recycle Gas

2300 Rxt level @ 511". Will wait for 6 cl to come down before increasing recycle.
FI-126A = 20,600 SCFH (CO) FIC-104 = 13 MSCFH
FIC-246 = 23.5 MSCFH ($H_2$) FIC-101 = 6.4 MSCFH
FI-1216A = 36.87 MSCFH FIC-1200 = 0
FI-196 = 22.3 MSCFH
FI-245A = 10,6000 MSCFH
FI-7291 = 3.76 MSCFH
SP10MW = 25.7
SP1MW = 20.13
SP2MW = 29.56
SP3AMW = 33.77
SP4MW = 23.05
SP6MW = 28
SP8MW = 2615
Rxt Avg. Temp = 446°F
Rxt Out Press = 758.6 psig
Fresh Feed: $H_2$ = 31.3, (0 = 63.5, $CO_2$ = 2.7.1
Rxt Feed: $H_2$ = 22.2, CO = 70.8, $CO_2$ = 4.7.1

2340 Increasing $H_2$ in Fresh Feed
6.5 → 6.8 MSCFH → 7.2 → 7.8 → 9.2 → 10.0

<u>Oct. 13, 1999</u>

0050 Increased Recycle 22 → 26 MSCFH → 27.5 → 31 → 32.7
$CO_2$ turned off completely in fresh feed.

0240 Increased Recycle → 40 → 50 MSCFH

0550 We are now at full flows.
NDG controlling level @ 480"
Rxt temp on control @ 482°F
Need fine tuning on Rxtor Feed, also need to bring $CO_2$ level down.

1215 Inc. $H_2$ & CO fresh feed flow rates in order to increase reactor pressure. (currently @ 717 psig)
1400 Field log taken
1430 Lost $CO_2$ flow to 21.80 (Reactor feed $CO_2$ at 4.3%, 1510 $CO_2$ back on line.
1530 Dennis 6 was indicating syngas exiting at top of 7.20.
Dean said that he had no pressure from sample pt. #10.
Work begun on Sample pt. #10.
1620 Pressure restored to sample point 10.
1640 Nuke scan
1800 Field log taken
1900 MeOH sample taken from 22.11 (First Sample)
Oil sample taken from 27.14 (First Sample)
1930 Checked GC printers
1945 Reactor feed $CO_2$ = 2.8 mol%

NDG:

| Reading | Level (LI) |
|---|---|
| 41.2 | 460.9 |
| 41.5 | 445.8 |
| 36.9 | 424.4 |
| 168 | 470.3 |

2100 Increasing CO & Reducing $H_2$ to fine tune reactor feed
2200 Full Field Log
2300 Nuke scan

Oct. 14, 1999

0200 Liq. lends field log
Reactor Feed Composition is now perfect.
0500 Nuke scan
0600 Full field log
1100 Liquid level field log
22.11 sample taken
Reactor P.T., compositions and flow rate look very good
1210 Nuke scan
1400 Transferred 1484 gal MeOH from 22.16 to trailer #70967 (transfer #1)
1530 Full field log
1700 Reactor P.T. compositions & flow rates are very good & steady
1800 Nuke scan
1900 Liquid level field log
Downloaded 1st MMB Period Data; 16:00 10/13/99 to 10:00 10/14/99
2130 22.11 sample taken AFR171A.TXT
22.14 sample taken: This is the second 27.14 oil sample. The first one was not representative, as the line was blocked off.
2400 Full field log

Oct. 15, 1999

0500 Downloaded 2nd MMB Period: 10:00 10/14 to 04:00 10/15 AFR171B.TXT
0515 Nuke scan
0600 Liquid level field log
Plant very steady throughout the night.
1045 POX trains in HyCO plant next door went down; Operators putting ™ on standby (12-15 hrs. before syngas resumes)

1130 Liquid level field log
1140 NDG: 242.4 @ 445.7 (LI)
34.6 @ 424.6 (LI)
33.2 @ 415.9 (LI)

Saturday 10/16/99

0005 Bringing syngas back into plant.
0020 CO at 10 KSCFH; $H_2$ @ 7 KSCFH. Backing out $N_2$. Adding $CO_2$.
0045 Dean setting up GC Gary to shoot SP #1 & #4. GC Dennis is on normal sequence.
0315 Gas feed back to original valves. Reactor temp. set point at 482°F.
$N_2$ in reactor feed at 6% & falling. $CO_2$ at 3.6% & backed out of fresh feed. Level at 444" & rising. Pressure at 750 psig.
0730 Downloaded HMB AFR171C.TXT; 04:00 10/15 to 11:00 10/15
0800 Transferred liquid from 22.16 to trailer
2nd transfer: 129 gallons
total in trailer: 2753 gallons
trailer capacity: 6300 gallons
930 Both GCS now analyzing all streams
1000 Adding oil to 27.14 & transferring oil from 27.14 to 22.11
(8 bolts added = 20 bolts - 12 bolts) = 76-49 = 27 gal
1115 Full field log
1440 NK Scan
1515 Liquid Level Field Log
1630 27.14 oil sample & 22.11. Methanol sample taken.
1900 Full Field Log
1945 Nuke Scan

Sunday, Oct. 17, 1999

0200 Nuke Scan. No significant changes; very steady readings
0600 Found that GC's Bharat & Dennis had both run out of paper (Bharat just as I watched; Dennis sometime before, although not too long). Correspondingly, FI-1216A dropped from ~ 95 KSCFH to ~ 80 KSCFH for about an hour, then returned to it's original value. Raw dp does not change. FI-1216A corrected by Dennis also drops from ~ 95 KSCFH to ~ 80 KSCFH.
0630 FI-1216A corrected by Dennis returned to correct valve of ~ 95 KSCFH.
0650 Liquid Level Log
0730 22-11 sample taken
Downloaded HMB AFR171D.TXT 10:00 10/16 - 04:00 10/17
0900 NK Scan
1030 Dcg Tank Transfer (33) 84.5" to 2" → 1137 gallons
total in trailer: 3890 gallons
capacity: 6300 gallons
1115 Changed printer cartridge on DEC Printer & reprinted last two hourly reports.
*Need to order more black ink cartridges (3)
1135 Full Field Log
1215 *Now take 21.11 oil sample instead of 27.14 oil sample (more representative)
1530 Nuke Scan
1600 Liquid Level Log
2130 Mast caught MeOH & oil samples → (21.11; shows s catalyst

Oct. 18, 1999

0700 Downloaded HMB AFR171E.TXT 07:00 10/17 - 22:00 10/17
0830 Liquid Level Field Logs
0930 NK Scan
1200 Full field logs
Added 10.80 Barrier fluid level to list (report in bolts)
1530 NK Scan
1600 Downloaded HMB AF171F.TXT 27:00 10/17 - 16:00 10/18
Transferred 1237 gal MeOH to trailer 70967.
Next transfer should be @ 80" level on 22.16 to ensure proper filling
2000 Noticed $CO_2$ conc. In reactor feed jumped > 4:5% according to Dennis
Dennis is printing part 1 & part 2 of each port out of sequence
(D11; D21; D31; D12. . .) - problem started around 19:45 pm (may have started as early as 19:00). Dean has been called.
2030 Full Field Log
2100 Oil & MeOH samples taken
2110 Dean showed up to stop GC Dennis & resequence.
2240 GC Dennis has cycled then one complete sequence.
2400 Liquid Level Log taken

Oct. 19, 1999

400 Full Field Log taken
0730 Redownloaded HMB AFR171F.TXT 22:00 10/17 - 18:00 10/18
0800 Liquid Level Log taken
1030 NK Scan
1250 Full Field Log taken
1545 $CO_2$ in reactor feed rising (~ 3.3%); increased MeOH flow rate to 07.10 from 14 gpm to 14.5 gpm
1600 Liquid Level Log taken
Transferred 1058 gallons of MeOH to the trailer.
1630 NK Scan taken
Downloaded AFR171A 23:00 10/18 - 17:00 10/19
1935 Drained MeOH from 10.80 suction to 22.16 because of recurring T&P control problems around 07:20. Transferred 326 gallons to day tank.
Decreased MeOH flow rate to 07.10 from 14.5 gpm to 14 gpm
2000 Full Field Log taken
2150 Matt caught methanol & oil samples
2320 Nuke Scan

Oct. 20, 1999

0000 Liquid Level Logs taken
0400 Full Field Logs taken
0500 Nuke Scan
0900 Liquid Level Logs taken
1010 Increased MeOH flow rate from 14 to 14.5 gpm
1100 Nuke Scan
1111 Downloaded AFR171H.TXT 10/19/99 17:00 → 10/20/99 11:00
1240 Transferred 2- 55gal drums of MeOH from the knockout pot to trailer #70900
1400 Full log taken.
Transferred 14.7 gal. (69" to 61") from 07.20 sump to 22.10 & 33 gal. (61" to 43") from

07.20 sump to 22.16 (day tank). Level in 07.20 is now 43". Check during field logs.
1500 Transferred 1384 gals to tanker #70900 (total amount in tanker is 1494 gals)
1700 Nuke Scan
Liquid Level Log
2130 Gary caught methanol & oil samples.
2155 Full log taken
2340 Nuke Scan taken

Oct. 21, 1999
150 Liquid Level Logs taken
520 Full Log taken
550 Nuke Scan taken
910 Liquid Level Log taken
1200 Nuke Scan
1300 Full Field Log taken. Downloaded AFR171I, TXT 10/20/99 16:00→ 10/21/99 05:00
1430 MeOH sample taken from 22.11
1530 Transferred 61.5 gallons of MeOH for 7020 to 22.16
1615 Transferred 1163 gallons of MeOH for day tank to trailer.
1645 Liquid Level Log taken
1818 Nuke Scan
2100 Full Field Log taken
May need to add oil to 27.14 tomorrow as level has steadily drop (currently near 7 bolts = 33 gal)
2300 Gary caught MeOH & oil samples.

Oct. 22, 1999
010 Nuke Scan taken
110 Liquid Level Log taken
430 Full Log taken
600 Nuke Scan taken
900 Liquid Level Log taken. Day tank level was lower than expected.
920 Dean noticed this morning the flow in the surplus line SP6 (off 22.11) was eratic due to stickiness inside the flow meter. This may make the GC reading inaccurate. Dean is replacing the flow meter.
935 It was found that the valve between the 22.11 and 22.15 was closed. Most likely, it was closed when the liquid sample was taken at 23:00 on 10/21/99 and forgotten to be reopened. As a result, some of the MeOH product when to flare. This also explains the problem seen by Dean on SP6.
Day tank level increased about 10 inches every 4 hours at 13:00, 10/20/99 to 5:20, 10/21/99. It increased about 10 inches every 4 hours from 16:45, 10/21/99 to 1:10, 10/22/99. In the next two ~ 4 hour periods, it increased 2 and 4 inches, respectively. This indicates significant amount of product MeOH went to the flare when the valve was closed.
G02(SP6) reading

| | Time | DME | MeOH %/Area |
|---|---|---|---|
| 10/21 | 22:37 | 33.69 | 4.14 |
| | 23.54 | 33.05 | 3.79 |
| 10/22 | 1:10 | 33.32 | 3.78/21889 |
| | 2:27 | 14:85 | -/507680 |
| | 3:44 | 10:61 | -/528522 |

7:34 34.73 53.68/310778

The increase in (MeOH) between 1:10 and 2:27, 10/22 indicates when 22:11 was flooded.

**- DAY TANK data of this period should be used for MB (23:00, 10/21 - 10:00, 10/22)

1210 Nuke Scan

1355 Downloaded AFR171J.TXT 05:00 10/21 → 23:00 10/21/99

1407 Full Log taken

1430 Dean fixed the flow meter for SP6 at 11:30. The results look reasonable afterward and can be used for MB.

** - SP6 data from 2:27 to 10:30, 10/22/99 can not be used for MB.

1440 Decreased LP $H_2$ cetpoint from 9.0 to 8.8 to try to bring rising $H_2$ and sliding CO compositions back into line.

1500 MeOH sample taken from 22.11

1611 Transferred MeOH from 22.16 to trailer #70900. 811 gallons transferred (total in trailer = 3468 gallons)

Transferred 62 gallons of MeOH from 7.20 to 22.16.

1700 Decreased LP $H_2$ from 8.8 to 8.6 to try to bring $H_2$ & CO back in line

May need to crease chilled MeOH flow rate to 07.10 later to bring $CO_2$ up.

1800 Nuke Scan

1900 Gary caught methanol & oil samples.

2235 Full Log taken

Oct. 23, 1999

000 Nuke Scan taken

115 Liquid Level Log taken

440 Full Log taken

700 Nuke Scan taken

755 110 gallons of MeOH in 255 gallon drums was transferred to trailer 70997.

900 Liquid Level Log taken. 27.14 (replenishing oil tank) at 16.20 ($CO_2$ tank) are running low. Both are covered by the operators.

1300 Full Log taken

1320 Nuke Scan taken

1414 Transferred 70 gallons of MeOH from 7.20 to 22.16

1607 Transferred 1069 gallons from Day Tank to trailer

1640 Liquid Level Log taken.

1900 Increased recycle flow (1800 MeOH & oil sample taken) to bring reactor flow up.

1845 Nuke Scan Increased $H_2$ in fresh feed.

2010 Full Field Log

2240 Increased $H_2$ in fresh feed. 22.11 & 21.11 sample taken

Oct. 24, 1999

0120 Increased $H_2$ in fresh feed

0510 Increased $H_2$ in fresh feed again.

720 Nuke Scan taken

800 Liquid Level Log taken

1200 Full Log taken, 22.11 sample taken

1340 Nuke Scan taken

1230 13:15, FI-7291 & PIC-7291 not working, being repaired.

1400 Downloaded AFR171L.TXT, 05:00 10/23 - 23:00 - 10/23

1508 Transferred 75 gallon MeOH from 7.20 to 22.16
1640 Transferred 1185 gallons from Day Tank to trailer.
578 gallons left in the trailer. Need to push after ~ 12 hrs. (at 53-1/2" mark)
1800 Downloaded AFR171M.TXT, 23:00 10/23 - 17:00 10/24
1930 Gary caught methanol & oil sample.
2000 Full Field Log
2030 Nuke Scan

Oct. 25, 1999

0435 Day tank transfer to trailer #70900. 47-1/2" to 20".
485 gallons transferred. Trailer is full.
800 Liquid Level Log taken
830 Nuke Scan taken
905 Dean is taking the sample point #3A off the line to fix a problem (out of alignment) in the selection valve.
1217 Full Log taken
1330 Transferred 76 gallons for 7.20 to 22.16
1519 Nuke Scan taken, 16:00 liquid level loss taken
1630 Transferred 674 gals. From day tank to trailer #70997
(total amt. In trailer = 784 gals.)
2000 Full Log taken. 1930 Gary caught methanol & oil samples.
2030 Nuke Scan

Oct. 26, 1999

0815 Downloaded AFR171N. TXT 17:00 10/24 - 09:00 10/25
AFR171O.TXT 17:00 10/25 - 06:00 10/26
800 Liquid Level Log taken
830 Nuke Scan taken
1200 Full Field Log taken
1500 Transferred 79.5 gal (75" to 79.5" on 22.16) from 07:20 sump to 22.16
1530 Transferred 1049 gal. From 22.16 to trailer #70997
(total in trailer = 1833 gal)
1630 Liquid Level Log taken
1830 Nuke Scan, 1930 MeOH & oil samples
1930 Noticed DEC downloads indicate no MeOH & DME in reactor effluent = consistent with Bharat & Rocco's GC's. Also ~ 10 load files received from GC in past 3 hrs. Called Dean & Bharat.
2030 Full Field Log taken
2115 Dean is returning to AFDU. The problem w/the bad files that that Part 2 of Rocco or Bharat report was disabled because it was a second DME peak not a MeOH peak as originally thought. But disabling part 2 of report will be enabled & BLB will apply a factor to the 2nd DME peak to convert it to MeOH peak.
2310 GC Rocco back on-line & sending DME values to DEC. Dean still setting up & calibrating GC Bharat.

Oct. 27, 1999

0030 GC Bharat back on-line & sending data to the DEC.
820 Nuke Scan taken.
850 Liquid Level Log taken.
1310 Full Field Log taken 13:30 MeOH sample taken from 22.11

14600 Transferred 64.4 gals. (77.5" to 42.5" on 7.20 sump) to day tank
1445 Downloaded AFR171P.TXT 0:00 10/27 → 9:00 10/27
1445 Nuke Scan
1530 Transferred 1181 gal. From day tank to trailer #70997
(total in trailer = 3014 gal.)
1610 Rocco modified & recalibrated & back on.
Start new HMB at 17:00.
1700 Liquid levels taken
1930 MeOH & oil samples taken.
2100 Full Field Logs taken
2130 Nuke Scan

Oct. 28, 1999

0210 GC Gary ran out of paper at approx. 1:30. Problem caught and fixed 30-40 minutes later. Gary has resumed its normal sequence, but with the ½ hour delay, its sequence is now pretty much back in phase with GC Dennis.
0930 Downloaded AFR171Q: 17:00 10/27 - 09:00 10/28
900 Liquid Level Log taken
930 Nuke Scan taken
1300 Full Field Log taken. Transferred 85 gallons from 7.20 to 22.16
1530 Nuke Scan
1600 Transferred 1155 gal. to trailer #70997 (total in trailer = 4169 gal.).
1700 Liquid Level Log taken
1930 MeOH & oil samples taken
2100 Full Field Logs taken.
2130 Nuke Scan - Discovered that NDG was not returned to its original spot on reactor (480" LI) after previous scan has been @ 511" (LI) for six hours.
Current level between 498" & 511" (LI)
2220 Nuke Scan completed. Detector set to 480" (LI)

Oct. 29, 1999

0000 Reactor level back to 480".
914 Flow to the reactor stopped for shutdown test/leak fixing.
Start Nuke Scan.
945 Reactor temp = 458.3F Reactor pressure = 693.0 psig
TI-1235 = 467.4F TI-1244- TI-1246 = 423.5
956 Reactor temp = 455.7 Reactor pressure = 625.6 psig
TI-1235 = 462.9
957 Syngas starts to flow into the reactor
1043 RT = 476.0 F, RP = 751 psig. Feed line = 102.1
▲P = 62.4
1140 Inlet at exit compositions came close to those prior to shutdown.
1145 A full log taken - the system may not be at steady state yet.
1400 A Full Log taken.
Nuke Scan
1550 Transferred 69 gals from 07.20 sump to 22.16 (81-3/4" to 44" on 07.20 sump)
1600 MeOH sample taken from 22.11
1615 Transferred 1150 gals from 22.16 to trailer #70997
Total in trailer = 5319
Should transfer before reaching 75" level on day tank in order to fill trailer

1630 Downloaded AFR171R ((:00 10/28 → 21:00 10/28) and AFR171S (21:00 10/28 → 9:00 10/29
2000 Nuke Scan
2200 Full Field Log

Oct. 30, 1999

0200 Liquid Level Field Log
0300 N/C Scan 06:00 Full Log taken
0900 Nuke Scan taken
1030 MeOH sample taken from 22.11
1340 Downloaded AFR171T: 14:00 10/29 → 8:00 10/30
1340 Transferred 987 gallons from 22.16 to the trailer.
Transferred 82 gallons from 82 gallons from 7.20 to 22.16.
Full log taken.
1500 Nuke Scan
1530 Ed found GC data printer out-of-ink. Cartridge replaced.
Lost ~ 30 mins of hard copy GC data. No effect on Dec seen.
1800 Liquid Level Log taken
2100 Nuke Scan
2200 Full Field Log. MeOH & oil samples taken.

Oct. 31, 1999

0200 - CDT Time change from CDT to CST (02.00 → 01200)
Bailey changed time automatically.
We will NOT change DEC time.
0100 - CST Liquid Logs
0130 Downloaded AFR171V.TXT 08:00 10/30 - 02:00 10/31 DEC TIME
0200 Nuke Scan
0230 Decreased recycle flow slightly to reduce reactor flow.
Rx flow reduced from 99.5 to 98.0 MSCFH
0330 Another move to 97.5 MSCFH
0500 Full Field Log
0830 Nuke Scan taken
0900 Liquid Level Log
0930 MeOH Sample taken
1250 Full Log taken
1300 Transferred 1234 gallons from 22.16 to trailer.
1400 Nuke Scan taken.
1630 Transferred 57 gals. From 07.20 to 22.16 (72-3/4” to 42” on 07.20 sight glass)
1700 Liquid Level Logs taken
2000 DEC crashed & had to be rebooted. (Probable cause: downloading data during time periods spanning past & future)
(Lost data from 21:00 to 22:30 DEC time)
2100 MeOH & oil samples
2130 Full Field Logs taken
2200 Decreased MeOH flow rate to 14 gpm from 14.5 gpm
2115 Downloaded AFR171V.TXT (2:00 10/31 - 20:00 10/31 DEC time)
2300 Nuke Scan

Nov. 1, 1999

130 Liquid Level Logs taken
530 Full Field Logs taken
600 Nuke Scan
800 MeOH sample taken
930 Liquid Level Logs taken
1015 Decreased LP $H_2$ from 8.4 to 8.2 & CO decreased from 18.6 to 18.2
(move made because $H_2$ reactor feed rising)
1200 Nuke Scan
1330 Full Log taken
1350 Transferred 1169 gallons from 22.16 to the trailer.
1700 Downloaded AFR171W.TXT 23:00 10/31 - 14:00 11/1
1707 Transferred 71 gallons from 7.20 to 22.16
1730 Liquid Level Log taken
1800 Nuke Scan taken
2100 MeOH & oil samples taken
2130 Full Log taken

Nov. 2, 1999

0700 Nuke Scan
0800 Downloaded AFR171X.TXT 14:00 11/1 - 08:00 11/2
1300 Full Log taken
1450 Transferred 58 gals. from 7.20 to 22.16
1500 Transferred 1251 gals. from 22.16 to the trailer
1730 Liquid Log taken
2100 MeOH & oil samples
2125 Full Log taken

Nov. 3, 1999

~0115 DEC shows that GC Rocco (02) dropped the DME valve for about the next 40 minutes (1 shot). Printouts in lab show no problem, all DME valves normal.
0725 Nuke Scan
0730 Downloaded AFR171Y.TXT 08:00 11/2 - 23:00 11/2
1120 Transferred 07.20 liquid, 66-1/4" to 41" into 22.16, 74-3/4" to 77-1/4" (44 gallons)
1130 Transferred 22.16 liquid to trailer (1008.5 gals.)
Total in trailer now: 4663 gallons
15001 Full Log taken
1800 Liquid Level Log taken
1835 GC's stopped surplus to allow tracer experiment.
1830 Downloaded AFR171Z.TXT 23:00 11/2 19:00 11/3
1830 Radioactive gas on site, GC sampling & samples flow off
1902 First gas injection. One bad detector. Fixed
1945 3rd injection (2nd good one). The following injection will be made every 3 minutes. It actually took much longer between injections to wait for the recycle to stabilize.
2140 Last injeciton was made (8th)
2150 Resume GC shots
2300 MeOH & oil samples taken
2315 - CST Last shot clearned thru to DEC on each GC.

Nov. 4, 1999

0555 Transferring from 07:20 to 22.16. ^1-1/2" to 40" gallons
22.16 level between sight glasses, but starting level was 68-3/4" = 1106 gallons + 40 gallons = 1146 gallons = 71" = B.C.

0605 Transferring from 22.16 to trailer #70999. 71" to 20" = 899 gallons
Trailer #70999 now at 5562 gallons. Next transfer at 62" will top it off at 6300 gallons.

0930 Full Field Log

1020 Downloaded AFR171AA.TXT 11/4 00:00 to 11/4 11:00

1110 First $Mn_22_3$ shot thru center bottom ($N_2$) (5 min)

1150 Second $Mn_2O_3$ shot thru center bottom ($N_2$) (Not enough not get saved)

1228 Third $Mn_2O_3$ shot thru bottom ($N_2$) center (10 min)

1254 Fourth $Mn_2O_3$ shot thru bottom ($N_2$) center (15 min)

1340 Fifth $Mn_2O_3$ shot thru bottom center (60 minutes)

1526 1st Manganese Oxide doped gamma alumina catalyst shot thru bottom ($N_2$) center (1 hr)

1705 2nd $Mn_2O_3$ doped gamma alumina catalyst shot thru bottom ($N_2$) center (15 min)

1823 3rd $Mn_2O_3$ doped gamma alumina catalyst shot thru upper center (N1) port (15 min)

1850 4th $Mn_2O_3$ coped gamma alumina catalyst shot into N1 nozzle (15 min)

1940 GC valves reopened

| LI | Reading | |
|---|---|---|
| 484.3 | >220 | Level at or above 470" (LI) |
| 470 | 48.1 | 480 ~130 |
| 460.8 | 49.8 | |

2045 Full Field Logs taken

2110 Gary has completed one cycle (22.10 DEC time)

2113 Dennis has completed one cycle (22.13 DEC time)

2345 Day tank transfer 64-1/2" to 20" = 784 gallons
Trailer #70999 is now full!

Nov. 5, 1999

0225 Transferring from 07.20 to 22.16. 70-1/2" to 36" = 63 gallons
22.16 level between sight glasses, but starting level was 26-1/2" = 362 gallons + 63 gallons = 425 gallons = 30" = B.C.

0430 Gary collected a MeOH sample.

0700 Nuke Scan

0900 Liquid Level Logs.

1300 Full Field Logs taken

1420 Nuke Scan

1645 Transferred 36 gal from 07.20 sump (57" to 37.5") & ~ 74 gal from 22.15 to get an accurate reading in the 22.16 sight glass.

1700 Transferred 916 gal from 22.16 to trailer #70996
(total in trailer = 916 gal)

1730 Liquid level readings taken

2045 Nuke Scan

2130 Full Field Log taken

Nov. 6, 1999

0110 Liquid Level Log taken

0130 MeOH & oil samples collected

0245 Nuke Scan
0430 Full Field Log
0820 Liquid Level Log
0830 Nuke Scan
930 Plant shutdown, shut down test @ 9:33.
1600 Drained 2624 lbs slurried catalyst & Drakeol 10 (5 full drums & 1 partial drum filled).
Collected 4 catalyst/oil samples (one from each of first four drums).

Nov. 7, 1999

Transferred 771 gal from 22.16 (day tank) to trailer #70996
(total in trailer = 1687 gal)
115 Open Drum 1 of spent catalyst/oil - showed no signs of settling.
No signs of settling in any of the 4 samples taken either.
1200 Transferred 719 gal from 22>16 to trailer #70996
(total in trailer = 2406 gal)

# APPENDIX E

## LPDME Mass Balances

**RUN NO: AF-R17.1a** **TITLE: LPDME with Shell Syngas : SV=6000**

**Balance Period:**

| | | |
|---|---|---|
| Start Date | 10/13/1999 | 16:00 |
| End Date | 10/14/1999 | 10:00 |

**Reaction Conditions**

| | |
|---|---|
| Temperature (°F) | 481.69 |
| Pressure (psig) | 748.71 |
| Space Velocity (sL/kg-hr) | 5889 |
| Vg (inlet) | 0.56 |

**Performance Results**

| | |
|---|---|
| CO Conversion to H2 (%) | -0.1 |
| CO Conversion to MeOH (%) | 6.3 |
| CO Conversion to DME (%) | 17.6 |
| Methanol Production (Ton/day) | 3.84 |
| DME Production (Ton/day) | 5.14 |
| Methanol Productivity (gmole/kg-hr) | 10.63 |
| DME Productivity (gmole/kg-hr) | 9.87 |
| H2O Productivity (gmole/kg-hr) | 0.04 |
| MeOH Rate Constant (1-CSTR) | 5.06 |
| DME Rate Constant (1-CSTR) | 26.41 |

**Time From Start of Run (hr)**

| | |
|---|---|
| Start | 20.00 |
| End | 38.00 |

**Slurry Data**

| | |
|---|---|
| Catalyst Weight (lb oxide) | 941 |
| Slurry Level (Reactor Height, ft) | 43.5 |
| Slurry Conc. based on NDG (wt %) | 39.1 |
| Slurry Conc. based on DP (wt %) | 35.8 |
| Gas Holdup based on NDG (vol %) | 48.7 |
| Gas Holdup based on DP (vol %) | 41.2 |

**Atomic/Mass Balance (% of reactor inlet)**

| | |
|---|---|
| C | 95.8 |
| H | 91.6 |
| O | 97.1 |
| N | 99.9 |
| Total Mass | 96.4 |

**Liquid Product Analysis (wt%)**

| | Sample# 19:00 | Sample# 11:00 |
|---|---|---|
| Methanol | 85.394 | 83.881 |
| Ethanol | 1.763 | 1.915 |
| 1-Propanol | 0.689 | 0.788 |
| iso-Propanol | 0.03 | 0.071 |
| 1-Butanol | 0.491 | 0.03 |
| 2-Butanol | 0.064 | 0.062 |
| iso-Butanol | 0.13 | 0.168 |
| 2-Methyl-1-Butanol | 0 | 0 |
| 1-Pentanol | 0.126 | 0.14 |
| 2-Methyl-1-Pentanol | 0 | 0 |
| 1-Hexanol | 0.014 | 0.015 |
| 2-Methyl-1-Isobutyrate | 0 | 0 |
| Methyl Acetate | 0.416 | 0.416 |
| Ethyl Acetate | 0 | 0 |
| Methyl Formate | 0.778 | 0.773 |
| DME | 6.747 | 7.177 |
| CO2 | 0 | 0 |
| Water | 0.278 | 0.286 |
| Oil | 0.2 | 0.2 |
| Total | 97.12 | 95.922 |

**RUN NO:** **AF-R17.1a** **TITLE:** **LPDME with Shell Syngas : SV=6000**

| | | FRESH MAKE-UP | RECYCLE | HP H2 MAKEUP | DRY FEED | ALCOHOL INJECT. | REACT FEED | REACT EFFL | 22.10 VAPOR | PURGE 1 PIC-201 | PURGE 2 22.11 | PURGE 3 07.20 | LIQUID PROD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **T** | **F** | 288.8 | 119.0 | 80.3 | 272.2 | 100.0 | 264.8 | 453.4 | 86.7 | 82.3 | 78.4 | 80.2 | 80.0 |
| **P** | **psig** | 815.2 | 807.0 | 783.8 | 777.6 | 777.6 | 777.6 | 748.7 | 738.0 | 706.7 | 29.0 | 164.9 | 1.0 |
| | | | | | | | | | | 9.19 | | | |
| **Comp** | **H2** | 37.00 | 17.81 | 100.00 | 32.27 | 0.00 | 32.27 | 15.55 | 15.74 | 17.81 | 2.40 | 2.07 | 0.00 |
| **(mole%)** | **CO** | 62.78 | 76.01 | 0.00 | 64.09 | 0.00 | 64.09 | 63.27 | 67.32 | 76.01 | 23.46 | 18.13 | 0.00 |
| | **N2** | 0.22 | 1.47 | 0.00 | 0.90 | 0.00 | 0.90 | 1.20 | 1.32 | 1.47 | 0.40 | 0.41 | 0.00 |
| | **CH4** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **CO2** | 0.00 | 4.55 | 0.00 | 2.73 | 0.00 | 2.73 | 9.19 | 10.23 | 4.55 | 34.98 | 44.57 | 0.00 |
| | **DME** | 0.00 | 0.16 | 0.00 | 0.02 | 0.00 | 0.02 | 5.04 | 4.68 | 0.16 | 34.03 | 33.60 | 5.23 |
| | **MeAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.19 |
| | **EtAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **MeFm** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.45 |
| | **MeOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 5.40 | 0.70 | 0.00 | 4.72 | 1.22 | 91.45 |
| | **H2O** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.09 | 0.00 | 0.00 | 0.01 | 0.00 | 0.54 |
| | **Etoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.08 | 0.00 | 0.00 | 0.00 | 0.00 | 1.38 |
| | **1-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 | 0.00 | 0.00 | 0.00 | 0.00 | 0.43 |
| | **iso-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 |
| | **IBOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.07 |
| | **1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.12 |
| | **2-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 |
| | **2-Methyl 1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 |
| | **2-Methyl 1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-hexanol** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **2-Methyl 1-Isobutyrate** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **others** | 0.00 | 0.00 | 0.00 | 0.00 | 100.00 | 0.00 | 0.16 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **TOTAL** | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| | | 1839.444 | 2413.743 | 202.000 | 2006.152 | 0.000 | 2006.152 | 2653.261 | 2642.586 | 2413.743 | 3929.731 | 4069.988 | 3310.900 |
| **Mole Wt** | **lb/lb mole** | 18.394 | 24.137 | 2.020 | 20.062 | 57.799 | 20.062 | 26.627 | 26.426 | 24.137 | 39.297 | 40.700 | 33.355 |
| **Flow** | **SCFH** | 35676 | 51426 | 8502 | 95605 | 0 | 95605 | 71576 | 64837 | 5821 | 289 | 8926 | 3732 |
| | **lb mole/hr** | 92.27 | 133.01 | 21.99 | 247.27 | 0.00 | 247.27 | 185.12 | 167.69 | 15.06 | 0.75 | 23.09 | 9.65 |
| | **lb/hr** | 1697.3 | 3210.4 | 44.4 | 4960.6 | 0.0 | 4960.6 | 4929.3 | 4431.4 | 363.4 | 29.3 | 939.6 | 321.9 |

**RUN NO: AF-R17.1b** **TITLE: LPDME with Shell Syngas : SV=6000**

**Balance Period:**

| | | |
|---|---|---|
| Start Date | 10/14/1999 | 10:00 |
| End Date | 10/15/1999 | 4:00 |

**Time From Start of Run (hr)**

| | |
|---|---|
| Start | 38.00 |
| End | 56.00 |

**Reaction Conditions**

| | |
|---|---|
| Temperature (°F) | 481.70 |
| Pressure (psig) | 748.56 |
| Space Velocity (sL/kg-hr) | 5850 |
| Vg (inlet) | 0.56 |

**Slurry Data**

| | |
|---|---|
| Catalyst Weight (lb oxide) | 941 |
| Slurry Level (Reactor Height, ft) | 43.5 |
| Slurry Conc. based on NDG (wt %) | 39.7 |
| Slurry Conc. based on DP (wt %) | 36.1 |
| Gas Holdup based on NDG (vol %) | 49.9 |
| Gas Holdup based on DP (vol %) | 41.9 |

**Performance Results**

| | |
|---|---|
| CO Conversion to H2 (%) | -0.1 |
| CO Conversion to MeOH (%) | 5.9 |
| CO Conversion to DME (%) | 16.4 |
| Methanol Production (Ton/day) | 3.65 |
| DME Production (Ton/day) | 4.89 |
| Methanol Productivity (gmole/kg-hr) | 10.08 |
| DME Productivity (gmole/kg-hr) | 9.39 |
| H2O Productivity (gmole/kg-hr) | 0.04 |
| MeOH Rate Constant (1-CSTR) | 5.20 |
| DME Rate Constant (1-CSTR) | 24.38 |

**Atomic/Mass Balance (% of reactor inlet)**

| | |
|---|---|
| C | 100.0 |
| H | 94.1 |
| O | 101.2 |
| N | 100.8 |
| Total Mass | 100.5 |

**Liquid Product Analysis (wt%)**

| | Sample# 21:30 |
|---|---|
| Methanol | 81.584 |
| Ethanol | 1.842 |
| 1-Propanol | 0.742 |
| iso-Propanol | 0.067 |
| 1-Butanol | 0.345 |
| 2-Butanol | 0.061 |
| iso-Butanol | 0.156 |
| 2-Methyl-1-Butanol | 0 |
| 1-Pentanol | 0.132 |
| 2-Methyl-1-Pentanol | 0 |
| 1-Hexanol | 0.032 |
| 2-Methyl-1-Isobutyrate | 0 |
| Methyl Acetate | 0.378 |
| Ethyl Acetate | 0 |
| Methyl Formate | 0.724 |
| DME | 7.247 |
| CO2 | 0 |
| Water | 0 |
| Oil | 0.2 |
| Total | 93.51 |

**RUN NO:** AF-R17.1b **TITLE:** LPDME with Shell Syngas : SV=6000

| | | FRESH MAKE-UP | RECYCLE | HP H2 MAKEUP | DRY FEED | ALCOHOL INJECT. | REACT FEED | REACT EFFL | 22.10 VAPOR | PURGE 1 PIC-201 | PURGE 2 22.11 | PURGE 3 07.20 | LIQUID PROD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **T** | **F** | 291.9 | 120.5 | 85.4 | 276.8 | 100.0 | 269.1 | 454.5 | 86.6 | 82.9 | 80.2 | 82.0 | 80.0 |
| **P** | **psig** | 816.3 | 807.8 | 783.5 | 778.1 | 778.1 | 778.1 | 748.6 | 739.2 | 705.9 | 29.0 | 165.0 | 1.0 |
| | | | | | | | | | | 8.86 | | | |
| **Comp** | **H2** | 35.13 | 16.47 | 100.00 | 30.81 | 0.00 | 30.81 | 14.56 | 14.47 | 16.47 | 2.19 | 1.32 | 0.00 |
| **(mole%)** | **CO** | 64.64 | 77.75 | 0.00 | 65.76 | 0.00 | 65.76 | 65.50 | 69.70 | 77.75 | 23.37 | 16.49 | 0.00 |
| | **N2** | 0.23 | 0.98 | 0.00 | 0.63 | 0.00 | 0.63 | 0.83 | 0.88 | 0.98 | 0.29 | 0.18 | 0.00 |
| | **CH4** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **CO2** | 0.00 | 4.57 | 0.00 | 2.74 | 0.00 | 2.74 | 8.86 | 9.84 | 4.57 | 32.66 | 45.52 | 0.00 |
| | **DME** | 0.00 | 0.22 | 0.00 | 0.06 | 0.00 | 0.06 | 4.82 | 4.53 | 0.22 | 36.92 | 35.13 | 5.65 |
| | **MeAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.18 |
| | **EtAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **MeFm** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.43 |
| | **MeOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 5.08 | 0.57 | 0.00 | 4.56 | 1.36 | 91.46 |
| | **H2O** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.09 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **Etoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.07 | 0.00 | 0.00 | 0.00 | 0.00 | 1.44 |
| | **1-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 | 0.00 | 0.00 | 0.00 | 0.00 | 0.44 |
| | **iso-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.04 |
| | **IBOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.08 |
| | **1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.17 |
| | **2-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 |
| | **2-Methyl 1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 |
| | **2-Methyl 1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-hexanol** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.01 |
| | **2-Methyl 1-Isobutyrate** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **others** | 0.00 | 0.00 | 0.00 | 0.00 | 100.00 | 0.00 | 0.16 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **TOTAL** | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| **Mole Wt** | **lb/lb mole** | 18.880 | 24.500 | 2.020 | 20.451 | 58.006 | 20.451 | 26.777 | 26.662 | 24.500 | 39.497 | 41.328 | 33.524 |
| **Flow** | **SCFH** | 34850 | 51618 | 8501 | 94969 | 0 | 94969 | 72108 | 68361 | 7583 | 247 | 9052 | 3549 |
| | **lb mole/hr** | 90.13 | 133.50 | 21.99 | 245.62 | 0.00 | 245.62 | 186.50 | 176.81 | 19.61 | 0.64 | 23.41 | 9.18 |
| | **lb/hr** | 1701.8 | 3270.8 | 44.4 | 5023.3 | 0.0 | 5023.3 | 4993.8 | 4714.0 | 480.5 | 25.2 | 967.6 | 307.7 |

**RUN NO: AF-R17.1c** **TITLE: LPDME with Shell Syngas : SV=6000**

**Balance Period:**

| | | |
|---|---|---|
| Start Date | 10/15/1999 | 4:00 |
| End Date | 10/15/1999 | 11:00 |

**Time From Start of Run (hr)**

| | |
|---|---|
| Start | 56.00 |
| End | 63.00 |

**Reaction Conditions**

| | |
|---|---|
| Temperature (°F) | 481.65 |
| Pressure (psig) | 748.04 |
| Space Velocity (sL/kg-hr) | 5919 |
| Vg (inlet) | 0.56 |

**Slurry Data**

| | |
|---|---|
| Catalyst Weight (lb oxide) | 941 |
| Slurry Level (Reactor Height, ft) | 43.5 |
| Slurry Conc. based on NDG (wt %) | 39.9 |
| Slurry Conc. based on DP (wt %) | 36.3 |
| Gas Holdup based on NDG (vol %) | 50.3 |
| Gas Holdup based on DP (vol %) | 42.3 |

**Performance Results**

| | |
|---|---|
| CO Conversion to H2 (%) | -0.1 |
| CO Conversion to MeOH (%) | 5.3 |
| CO Conversion to DME (%) | 16.1 |
| Methanol Production (Ton/day) | 3.35 |
| DME Production (Ton/day) | 4.86 |
| Methanol Productivity (gmole/kg-hr) | 9.25 |
| DME Productivity (gmole/kg-hr) | 9.34 |
| H2O Productivity (gmole/kg-hr) | 0.04 |
| MeOH Rate Constant (1-CSTR) | 3.95 |
| DME Rate Constant (1-CSTR) | 24.41 |

**Atomic/Mass Balance (% of reactor inlet)**

| | |
|---|---|
| C | 99.5 |
| H | 90.2 |
| O | 100.9 |
| N | 101.2 |
| Total Mass | 100.0 |

**Liquid Product Analysis (wt%)**

| | Sample# 21:30 |
|---|---|
| Methanol | 81.584 |
| Ethanol | 1.842 |
| 1-Propanol | 0.742 |
| iso-Propanol | 0.067 |
| 1-Butanol | 0.345 |
| 2-Butanol | 0.061 |
| iso-Butanol | 0.156 |
| 2-Methyl-1-Butanol | 0 |
| 1-Pentanol | 0.132 |
| 2-Methyl-1-Pentanol | 0 |
| 1-Hexanol | 0.032 |
| 2-Methyl-1-Isobutyrate | 0 |
| Methyl Acetate | 0.378 |
| Ethyl Acetate | 0 |
| Methyl Formate | 0.724 |
| DME | 7.247 |
| CO2 | 0 |
| Water | 0 |
| Oil | 0.2 |
| Total | 93.51 |

**RUN NO:** **AF-R17.1c** **TITLE:** **LPDME with Shell Syngas : SV=6000**

| | | FRESH MAKE-UP | RECYCLE | HP H2 MAKEUP | DRY FEED | ALCOHOL INJECT. | REACT FEED | REACT EFFL | 22.10 VAPOR | PURGE 1 PIC-201 | PURGE 2 22.11 | PURGE 3 07.20 | LIQUID PROD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **T** | **F** | 290.6 | 118.8 | 78.2 | 279.1 | 100.0 | 271.5 | 455.5 | 85.6 | 80.8 | 75.7 | 84.0 | 80.0 |
| **P** | **psig** | 814.6 | 807.7 | 785.2 | 777.8 | 777.8 | 777.8 | 748.0 | 739.5 | 704.3 | 28.9 | 164.7 | 1.0 |
| | | | | | | | | | | 8.60 | | | |
| **Comp** | **H2** | 35.09 | 16.39 | 100.00 | 30.53 | 0.00 | 30.53 | 15.29 | 14.47 | 16.39 | 2.26 | 1.29 | 0.00 |
| **(mole%)** | **CO** | 64.66 | 77.87 | 0.00 | 65.97 | 0.00 | 65.97 | 65.67 | 70.30 | 77.87 | 24.26 | 16.33 | 0.00 |
| | **N2** | 0.24 | 0.96 | 0.00 | 0.62 | 0.00 | 0.62 | 0.80 | 0.86 | 0.96 | 0.28 | 0.17 | 0.00 |
| | **CH4** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **CO2** | 0.00 | 4.51 | 0.00 | 2.74 | 0.00 | 2.74 | 8.60 | 9.70 | 4.51 | 32.71 | 44.91 | 0.00 |
| | **DME** | 0.00 | 0.27 | 0.00 | 0.14 | 0.00 | 0.14 | 4.77 | 4.23 | 0.27 | 36.17 | 36.19 | 5.65 |
| | **MeAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.18 |
| | **EtAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **MeFm** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.43 |
| | **MeOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.55 | 0.44 | 0.00 | 4.32 | 1.11 | 91.46 |
| | **H2O** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.09 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **Etoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.07 | 0.00 | 0.00 | 0.00 | 0.00 | 1.44 |
| | **1-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 | 0.00 | 0.00 | 0.00 | 0.00 | 0.44 |
| | **iso-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.04 |
| | **IBOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.08 |
| | **1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.17 |
| | **2-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 |
| | **2-Methyl 1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 |
| | **2-Methyl 1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-hexanol** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.01 |
| | **2-Methyl 1-Isobutyrate** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **others** | 0.00 | 0.00 | 0.00 | 0.00 | 100.00 | 0.00 | 0.14 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **TOTAL** | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| **Mole Wt** | **lb/lb mole** | 18.889 | 24.520 | 2.020 | 20.540 | 58.006 | 20.540 | 26.512 | 26.580 | 24.520 | 39.340 | 41.421 | 33.524 |
| **Flow** | **SCFH** | 34182 | 53520 | 8388 | 96090 | 0 | 96090 | 74053 | 69671 | 7753 | 240 | 8787 | 3341 |
| | **lb mole/hr** | 88.41 | 138.42 | 21.69 | 248.52 | 0.00 | 248.52 | 191.53 | 180.19 | 20.05 | 0.62 | 22.73 | 8.64 |
| | **lb/hr** | 1670.0 | 3394.1 | 43.8 | 5104.5 | 0.0 | 5104.5 | 5077.7 | 4789.6 | 491.7 | 24.5 | 941.4 | 289.7 |

**RUN NO: AF-R17.1d** **TITLE: LPDME with Shell Syngas : SV=6000**

**Balance Period:**

| | | |
|---|---|---|
| Start Date | 10/16/1999 | 10:00 |
| End Date | 10/17/1999 | 4:00 |

**Time From Start of Run (hr)**

| | |
|---|---|
| Start | 86.00 |
| End | 104.00 |

**Reaction Conditions**

| | |
|---|---|
| Temperature (°F) | 481.62 |
| Pressure (psig) | 748.93 |
| Space Velocity (sL/kg-hr) | 5837 |
| Vg (inlet) | 0.55 |

**Slurry Data**

| | |
|---|---|
| Catalyst Weight (lb oxide) | 941 |
| Slurry Level (Reactor Height, ft) | 43.5 |
| Slurry Conc. based on NDG (wt %) | 40.2 |
| Slurry Conc. based on DP (wt %) | 36.4 |
| Gas Holdup based on NDG (vol %) | 50.9 |
| Gas Holdup based on DP (vol %) | 42.5 |

**Performance Results**

| | |
|---|---|
| CO Conversion to H2 (%) | -0.1 |
| CO Conversion to MeOH (%) | 4.9 |
| CO Conversion to DME (%) | 15.0 |
| Methanol Production (Ton/day) | 3.11 |
| DME Production (Ton/day) | 4.54 |
| Methanol Productivity (gmole/kg-hr) | 8.60 |
| DME Productivity (gmole/kg-hr) | 8.72 |
| H2O Productivity (gmole/kg-hr) | 0.04 |
| MeOH Rate Constant (1-CSTR) | 3.52 |
| DME Rate Constant (1-CSTR) | 22.46 |

**Atomic/Mass Balance (% of reactor inlet)**

| | |
|---|---|
| C | 99.4 |
| H | 88.1 |
| O | 101.1 |
| N | 107.8 |
| Total Mass | 100.1 |

**Liquid Product Analysis (wt%)**

| | Sample# 16:30 |
|---|---|
| Methanol | 82.208 |
| Ethanol | 1.94 |
| 1-Propanol | 0.864 |
| iso-Propanol | 0.071 |
| 1-Butanol | 0.342 |
| 2-Butanol | 0.121 |
| iso-Butanol | 0.172 |
| 2-Methyl-1-Butanol | 0 |
| 1-Pentanol | 0.075 |
| 2-Methyl-1-Pentanol | 0 |
| 1-Hexanol | 0.033 |
| 2-Methyl-1-Isobutyrate | 0 |
| Methyl Acetate | 0.137 |
| Ethyl Acetate | 0 |
| Methyl Formate | 0.819 |
| DME | 7.453 |
| CO2 | 0 |
| Water | 0 |
| Oil | 0.2 |
| Total | 94.435 |

**RUN NO:** **AF-R17.1d** **TITLE:** **LPDME with Shell Syngas : SV=6000**

| | | FRESH MAKE-UP | RECYCLE | HP H2 MAKEUP | DRY FEED | ALCOHOL INJECT. | REACT FEED | REACT EFFL | 22.10 VAPOR | PURGE 1 PIC-201 | PURGE 2 22.11 | PURGE 3 07.20 | LIQUID PROD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **T** | **F** | 295.0 | 120.5 | 83.9 | 280.5 | 100.0 | 273.2 | 455.8 | 86.6 | 82.3 | 80.4 | 80.6 | 80.0 |
| **P** | **psig** | 816.6 | 808.5 | 806.7 | 778.9 | 778.9 | 778.9 | 748.9 | 739.5 | 705.4 | 28.9 | 164.9 | 1.0 |
| | | | | | | | | | | 8.35 | | | |
| **Comp** | **H2** | 33.09 | 15.82 | 100.00 | 29.53 | 0.00 | 29.53 | 15.05 | 14.18 | 15.82 | 2.32 | 1.31 | 0.00 |
| **(mole%)** | **CO** | 66.68 | 78.23 | 0.00 | 66.88 | 0.00 | 66.88 | 66.85 | 71.35 | 78.23 | 24.27 | 17.33 | 0.00 |
| | **N2** | 0.23 | 0.86 | 0.00 | 0.56 | 0.00 | 0.56 | 0.72 | 0.81 | 0.86 | 0.25 | 0.15 | 0.00 |
| | **CH4** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **CO2** | 0.00 | 4.75 | 0.00 | 2.85 | 0.00 | 2.85 | 8.35 | 9.43 | 4.75 | 32.21 | 45.35 | 0.00 |
| | **DME** | 0.00 | 0.34 | 0.00 | 0.18 | 0.00 | 0.18 | 4.50 | 3.72 | 0.34 | 36.14 | 34.58 | 5.75 |
| | **MeAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.07 |
| | **EtAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **MeFm** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.49 |
| | **MeOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.22 | 0.51 | 0.00 | 4.80 | 1.28 | 91.28 |
| | **H2O** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.09 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **Etoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.07 | 0.00 | 0.00 | 0.00 | 0.00 | 1.50 |
| | **1-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 | 0.00 | 0.00 | 0.00 | 0.00 | 0.51 |
| | **iso-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.04 |
| | **IBOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.08 |
| | **1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.16 |
| | **2-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.06 |
| | **2-Methyl 1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 |
| | **2-Methyl 1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-hexanol** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.01 |
| | **2-Methyl 1-Isobutyrate** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **others** | 0.00 | 0.00 | 0.00 | 0.00 | 100.00 | 0.00 | 0.14 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **TOTAL** | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| **Mole Wt** | **lb/lb mole** | 19.410 | 24.720 | 2.020 | 20.824 | 57.129 | 20.824 | 26.467 | 26.523 | 24.720 | 39.259 | 41.201 | 33.532 |
| **Flow** | **SCFH** | 33665 | 52587 | 8508 | 94761 | 0 | 94761 | 74193 | 70334 | 9137 | 215 | 8170 | 3008 |
| | **lb mole/hr** | 87.07 | 136.01 | 22.01 | 245.08 | 0.00 | 245.08 | 191.89 | 181.91 | 23.63 | 0.55 | 21.13 | 7.78 |
| | **lb/hr** | 1690.0 | 3362.1 | 44.5 | 5103.7 | 0.0 | 5103.7 | 5078.8 | 4824.8 | 584.2 | 21.8 | 870.6 | 260.9 |

**RUN NO: AF-R17.1e** **TITLE: LPDME with Shell Syngas : SV=6000**

**Balance Period:**

| | | |
|---|---|---|
| Start Date | 10/17/1999 | 7:00 |
| End Date | 10/17/1999 | 22:00 |

**Time From Start of Run (hr)**

| | |
|---|---|
| Start | 107.00 |
| End | 122.00 |

**Reaction Conditions**

| | |
|---|---|
| Temperature (°F) | 481.61 |
| Pressure (psig) | 749.09 |
| Space Velocity (sL/kg-hr) | 5828 |
| Vg (inlet) | 0.55 |

**Slurry Data**

| | |
|---|---|
| Catalyst Weight (lb oxide) | 941 |
| Slurry Level (Reactor Height, ft) | 43.5 |
| Slurry Conc. based on NDG (wt %) | 40.5 |
| Slurry Conc. based on DP (wt %) | 36.5 |
| Gas Holdup based on NDG (vol %) | 51.4 |
| Gas Holdup based on DP (vol %) | 42.9 |

**Performance Results**

| | |
|---|---|
| CO Conversion to H2 (%) | -0.1 |
| CO Conversion to MeOH (%) | 5.2 |
| CO Conversion to DME (%) | 14.7 |
| Methanol Production (Ton/day) | 3.25 |
| DME Production (Ton/day) | 4.44 |
| Methanol Productivity (gmole/kg-hr) | 8.98 |
| DME Productivity (gmole/kg-hr) | 8.54 |
| H2O Productivity (gmole/kg-hr) | 0.04 |
| MeOH Rate Constant (1-CSTR) | 3.83 |
| DME Rate Constant (1-CSTR) | 21.91 |

**Atomic/Mass Balance (% of reactor inlet)**

| | |
|---|---|
| C | 99.6 |
| H | 92.1 |
| O | 100.8 |
| N | 101.8 |
| Total Mass | 100.1 |

**Liquid Product Analysis (wt%)**

| | Sample# 7:30 |
|---|---|
| Methanol | 83.694 |
| Ethanol | 1.961 |
| 1-Propanol | 0.824 |
| iso-Propanol | 0.072 |
| 1-Butanol | 0.087 |
| 2-Butanol | 0.062 |
| iso-Butanol | 0.179 |
| 2-Methyl-1-Butanol | 0 |
| 1-Pentanol | 0.075 |
| 2-Methyl-1-Pentanol | 0 |
| 1-Hexanol | 0.011 |
| 2-Methyl-1-Isobutyrate | 0 |
| Methyl Acetate | 0.307 |
| Ethyl Acetate | 0 |
| Methyl Formate | 0.893 |
| DME | 7.603 |
| CO2 | 0 |
| Water | 0 |
| Oil | 0.2 |
| Total | 95.968 |

**RUN NO:** **AF-R17.1e** **TITLE:** **LPDME with Shell Syngas : SV=6000**

| | | FRESH MAKE-UP | RECYCLE | HP H2 MAKEUP | DRY FEED | ALCOHOL INJECT. | REACT FEED | REACT EFFL | 22.10 VAPOR | PURGE 1 PIC-201 | PURGE 2 22.11 | PURGE 3 07.20 | LIQUID PROD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **T** | **F** | 292.5 | 119.0 | 81.6 | 277.2 | 100.0 | 269.3 | 456.1 | 85.3 | 80.9 | 78.3 | 79.9 | 80.0 |
| **P** | **psig** | 815.4 | 808.7 | 820.1 | 779.0 | 779.0 | 779.0 | 749.1 | 739.8 | 705.4 | 28.8 | 164.9 | 1.0 |
| | | | | | | | | | | 8.39 | | | |
| **Comp** | **H2** | 32.74 | 15.66 | 100.00 | 29.35 | 0.00 | 29.35 | 14.69 | 13.97 | 15.66 | 2.23 | 1.29 | 0.00 |
| **(mole%)** | **CO** | 67.02 | 78.23 | 0.00 | 66.94 | 0.00 | 66.94 | 67.06 | 71.04 | 78.23 | 24.25 | 17.18 | 0.00 |
| | **N2** | 0.24 | 0.82 | 0.00 | 0.55 | 0.00 | 0.55 | 0.70 | 0.75 | 0.82 | 0.24 | 0.15 | 0.00 |
| | **CH4** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **CO2** | 0.00 | 4.93 | 0.00 | 2.98 | 0.00 | 2.98 | 8.39 | 9.52 | 4.93 | 32.46 | 44.52 | 0.00 |
| | **DME** | 0.00 | 0.36 | 0.00 | 0.19 | 0.00 | 0.19 | 4.43 | 4.15 | 0.36 | 36.08 | 35.73 | 5.77 |
| | **MeAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.14 |
| | **EtAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **MeFm** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.52 |
| | **MeOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.41 | 0.57 | 0.00 | 4.75 | 1.14 | 91.35 |
| | **H2O** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.09 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **Etoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.06 | 0.00 | 0.00 | 0.00 | 0.00 | 1.49 |
| | **1-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 | 0.00 | 0.00 | 0.00 | 0.00 | 0.48 |
| | **iso-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.04 |
| | **IBOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.08 |
| | **1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.04 |
| | **2-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 |
| | **2-Methyl 1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 |
| | **2-Methyl 1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-hexanol** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **2-Methyl 1-Isobutyrate** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **others** | 0.00 | 0.00 | 0.00 | 0.00 | 100.00 | 0.00 | 0.14 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **TOTAL** | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| **Mole Wt** | **lb/lb mole** | 19.500 | 24.794 | 2.020 | 20.892 | 56.589 | 20.892 | 26.560 | 26.673 | 24.794 | 39.310 | 41.275 | 33.498 |
| **Flow** | **SCFH** | 32980 | 53129 | 8505 | 94614 | 0 | 94614 | 74048 | 69913 | 8061 | 227 | 7611 | 3122 |
| | **lb mole/hr** | 85.30 | 137.41 | 22.00 | 244.71 | 0.00 | 244.71 | 191.52 | 180.82 | 20.85 | 0.59 | 19.69 | 8.07 |
| | **lb/hr** | 1663.3 | 3406.9 | 44.4 | 5112.4 | 0.0 | 5112.4 | 5086.6 | 4823.1 | 516.9 | 23.1 | 812.5 | 270.4 |

**RUN NO: AF-R17.1f** **TITLE: LPDME with Shell Syngas : SV=6000**

**Balance Period:**

| | | |
|---|---|---|
| Start Date | 10/17/1999 | 22:00 |
| End Date | 10/18/1999 | 18:00 |

**Time From Start of Run (hr)**

| | |
|---|---|
| Start | 122.00 |
| End | 142.00 |

**Reaction Conditions**

| | |
|---|---|
| Temperature (°F) | 481.48 |
| Pressure (psig) | 751.85 |
| Space Velocity (sL/kg-hr) | 5933 |
| Vg (inlet) | 0.56 |

**Slurry Data**

| | |
|---|---|
| Catalyst Weight (lb oxide) | 941 |
| Slurry Level (Reactor Height, ft) | 43.5 |
| Slurry Conc. based on NDG (wt %) | 40.8 |
| Slurry Conc. based on DP (wt %) | 37.1 |
| Gas Holdup based on NDG (vol %) | 52.1 |
| Gas Holdup based on DP (vol %) | 44.1 |

**Performance Results**

| | |
|---|---|
| CO Conversion to H2 (%) | -0.1 |
| CO Conversion to MeOH (%) | 5.0 |
| CO Conversion to DME (%) | 14.4 |
| Methanol Production (Ton/day) | 3.22 |
| DME Production (Ton/day) | 4.44 |
| Methanol Productivity (gmole/kg-hr) | 8.91 |
| DME Productivity (gmole/kg-hr) | 8.53 |
| H2O Productivity (gmole/kg-hr) | 0.04 |
| MeOH Rate Constant (1-CSTR) | 3.66 |
| DME Rate Constant (1-CSTR) | 21.68 |

**Atomic/Mass Balance (% of reactor inlet)**

| | |
|---|---|
| C | 99.4 |
| H | 89.0 |
| O | 100.8 |
| N | 102.0 |
| Total Mass | 99.9 |

**Liquid Product Analysis (wt%)**

| | Sample# 21:30 |
|---|---|
| Methanol | 82.379 |
| Ethanol | 1.829 |
| 1-Propanol | 0.738 |
| iso-Propanol | 0.073 |
| 1-Butanol | 0.346 |
| 2-Butanol | 0.058 |
| iso-Butanol | 0.156 |
| 2-Methyl-1-Butanol | 0 |
| 1-Pentanol | 0.162 |
| 2-Methyl-1-Pentanol | 0 |
| 1-Hexanol | 0.196 |
| 2-Methyl-1-Isobutyrate | 0 |
| Methyl Acetate | 0.386 |
| Ethyl Acetate | 0 |
| Methyl Formate | 0.832 |
| DME | 7.766 |
| CO2 | 0 |
| Water | 0 |
| Oil | 0.2 |
| Total | 95.121 |

**RUN NO:** **AF-R17.1f** **TITLE:** **LPDME with Shell Syngas : SV=6000**

| | | FRESH MAKE-UP | RECYCLE | HP H2 MAKEUP | DRY FEED | ALCOHOL INJECT. | REACT FEED | REACT EFFL | 22.10 VAPOR | PURGE 1 PIC-201 | PURGE 2 22.11 | PURGE 3 07.20 | LIQUID PROD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **T** | **F** | 282.1 | 109.6 | 65.5 | 266.4 | 100.0 | 258.5 | 456.2 | 77.0 | 71.0 | 65.2 | 73.8 | 80.0 |
| **P** | **psig** | 814.8 | 812.1 | 773.4 | 781.3 | 781.3 | 781.3 | 751.8 | 743.6 | 706.2 | 28.4 | 139.1 | 1.0 |
| | | | | | | | | | | 8.39 | | | |
| **Comp** | **H2** | 33.13 | 15.67 | 100.00 | 29.12 | 0.00 | 29.12 | 14.72 | 14.11 | 15.67 | 2.31 | 1.37 | 0.00 |
| **(mole%)** | **CO** | 66.62 | 78.15 | 0.00 | 67.01 | 0.00 | 67.01 | 67.09 | 71.86 | 78.15 | 25.25 | 18.36 | 0.00 |
| | **N2** | 0.25 | 0.91 | 0.00 | 0.61 | 0.00 | 0.61 | 0.77 | 0.83 | 0.91 | 0.26 | 0.17 | 0.00 |
| | **CH4** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **CO2** | 0.00 | 4.80 | 0.00 | 2.99 | 0.00 | 2.99 | 8.39 | 9.22 | 4.80 | 33.82 | 45.69 | 0.00 |
| | **DME** | 0.00 | 0.47 | 0.00 | 0.26 | 0.00 | 0.26 | 4.44 | 3.66 | 0.47 | 34.78 | 33.62 | 5.97 |
| | **MeAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.18 |
| | **EtAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **MeFm** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.49 |
| | **MeOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.28 | 0.32 | 0.00 | 3.58 | 0.78 | 91.05 |
| | **H2O** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.09 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **Etoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.06 | 0.00 | 0.00 | 0.00 | 0.00 | 1.41 |
| | **1-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 | 0.00 | 0.00 | 0.00 | 0.00 | 0.43 |
| | **iso-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.04 |
| | **IBOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.07 |
| | **1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.17 |
| | **2-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 |
| | **2-Methyl 1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.07 |
| | **2-Methyl 1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-hexanol** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.07 |
| | **2-Methyl 1-Isobutyrate** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **others** | 0.00 | 0.00 | 0.00 | 0.00 | 100.00 | 0.00 | 0.14 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **TOTAL** | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| **Mole Wt** | **lb/lb mole** | 19.399 | 24.792 | 2.020 | 20.967 | 59.069 | 20.967 | 26.549 | 26.488 | 24.792 | 39.226 | 41.048 | 33.623 |
| **Flow** | **SCFH** | 31861 | 55949 | 8503 | 96313 | 0 | 96313 | 75652 | 71621 | 7282 | 229 | 7844 | 3314 |
| | **lb mole/hr** | 82.40 | 144.70 | 21.99 | 249.10 | 0.00 | 249.10 | 195.66 | 185.24 | 18.83 | 0.59 | 20.29 | 8.57 |
| | **lb/hr** | 1598.5 | 3587.5 | 44.4 | 5223.0 | 0.0 | 5223.0 | 5194.6 | 4906.5 | 466.9 | 23.3 | 832.8 | 288.2 |

**RUN NO: AF-R17.1g** **TITLE: LPDME with Shell Syngas : SV=6000**

**Balance Period:**

| | | |
|---|---|---|
| Start Date | 10/18/1999 | 23:00 |
| End Date | 10/19/1999 | 17:00 |

**Time From Start of Run (hr)**

| | |
|---|---|
| Start | 147.00 |
| End | 165.00 |

**Reaction Conditions**

| | |
|---|---|
| Temperature (°F) | 481.48 |
| Pressure (psig) | 752.47 |
| Space Velocity (sL/kg-hr) | 5897 |
| Vg (inlet) | 0.56 |

**Slurry Data**

| | |
|---|---|
| Catalyst Weight (lb oxide) | 941 |
| Slurry Level (Reactor Height, ft) | 43.5 |
| Slurry Conc. based on NDG (wt %) | 40.8 |
| Slurry Conc. based on DP (wt %) | 37.1 |
| Gas Holdup based on NDG (vol %) | 51.9 |
| Gas Holdup based on DP (vol %) | 44.2 |

**Performance Results**

| | |
|---|---|
| CO Conversion to H2 (%) | -0.1 |
| CO Conversion to MeOH (%) | 5.5 |
| CO Conversion to DME (%) | 14.7 |
| Methanol Production (Ton/day) | 3.47 |
| DME Production (Ton/day) | 4.44 |
| Methanol Productivity (gmole/kg-hr) | 9.58 |
| DME Productivity (gmole/kg-hr) | 8.53 |
| H2O Productivity (gmole/kg-hr) | 0.04 |
| MeOH Rate Constant (1-CSTR) | 4.17 |
| DME Rate Constant (1-CSTR) | 21.90 |

**Atomic/Mass Balance (% of reactor inlet)**

| | |
|---|---|
| C | 99.5 |
| H | 92.7 |
| O | 100.5 |
| N | 101.5 |
| Total Mass | 99.8 |

**Liquid Product Analysis (wt%)**

| | Sample# 21:00 |
|---|---|
| Methanol | 83.452 |
| Ethanol | 1.692 |
| 1-Propanol | 0.67 |
| iso-Propanol | 0.072 |
| 1-Butanol | 0.294 |
| 2-Butanol | 0.054 |
| iso-Butanol | 0.141 |
| 2-Methyl-1-Butanol | 0 |
| 1-Pentanol | 0.142 |
| 2-Methyl-1-Pentanol | 0 |
| 1-Hexanol | 0.054 |
| 2-Methyl-1-Isobutyrate | 0 |
| Methyl Acetate | 0.29 |
| Ethyl Acetate | 0 |
| Methyl Formate | 0.774 |
| DME | 8.817 |
| CO2 | 0 |
| Water | 0 |
| Oil | 0.2 |
| Total | 96.652 |

**RUN NO:** **AF-R17.1g** **TITLE:** **LPDME with Shell Syngas : SV=6000**

| | | FRESH MAKE-UP | RECYCLE | HP H2 MAKEUP | DRY FEED | ALCOHOL INJECT. | REACT FEED | REACT EFFL | 22.10 VAPOR | PURGE 1 PIC-201 | PURGE 2 22.11 | PURGE 3 07.20 | LIQUID PROD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **T** | **F** | 281.2 | 111.5 | 62.5 | 263.8 | 100.0 | 255.1 | 454.6 | 81.5 | 73.5 | 58.9 | 77.4 | 80.0 |
| **P** | **psig** | 812.6 | 812.2 | 783.7 | 780.8 | 780.8 | 780.8 | 752.5 | 746.9 | 707.4 | 28.1 | 125.3 | 1.0 |
| | | | | | | | | | | 8.75 | | | |
| **Comp** | **H2** | 34.43 | 16.11 | 100.00 | 29.66 | 0.00 | 29.66 | 14.63 | 14.26 | 16.11 | 2.63 | 1.37 | 0.00 |
| **(mole%)** | **CO** | 65.31 | 77.31 | 0.00 | 66.20 | 0.00 | 66.20 | 66.19 | 70.37 | 77.31 | 26.82 | 17.83 | 0.00 |
| | **N2** | 0.25 | 1.08 | 0.00 | 0.71 | 0.00 | 0.71 | 0.92 | 0.98 | 1.08 | 0.32 | 0.20 | 0.00 |
| | **CH4** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **CO2** | 0.00 | 5.00 | 0.00 | 3.16 | 0.00 | 3.16 | 8.75 | 9.53 | 5.00 | 33.35 | 44.93 | 0.00 |
| | **DME** | 0.00 | 0.50 | 0.00 | 0.28 | 0.00 | 0.28 | 4.53 | 4.25 | 0.50 | 33.42 | 34.62 | 6.66 |
| | **MeAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.14 |
| | **EtAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **MeFm** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.45 |
| | **MeOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.68 | 0.61 | 0.00 | 3.46 | 1.04 | 90.72 |
| | **H2O** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.09 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **Etoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.06 | 0.00 | 0.00 | 0.00 | 0.00 | 1.28 |
| | **1-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 | 0.00 | 0.00 | 0.00 | 0.00 | 0.39 |
| | **iso-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.04 |
| | **IBOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.07 |
| | **1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.14 |
| | **2-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 |
| | **2-Methyl 1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.06 |
| | **2-Methyl 1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-hexanol** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **2-Methyl 1-Isobutyrate** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **others** | 0.00 | 0.00 | 0.00 | 0.00 | 100.00 | 0.00 | 0.14 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **TOTAL** | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| **Mole Wt** | **lb/lb mole** | 19.061 | 24.712 | 2.020 | 20.857 | 58.221 | 20.857 | 26.660 | 26.617 | 24.712 | 38.818 | 41.116 | 33.601 |
| **Flow** | **SCFH** | 30733 | 56498 | 8504 | 95735 | 0 | 95735 | 74404 | 70536 | 6379 | 90 | 7835 | 3354 |
| | **lb mole/hr** | 79.49 | 146.12 | 21.99 | 247.60 | 0.00 | 247.60 | 192.43 | 182.43 | 16.50 | 0.23 | 20.27 | 8.67 |
| | **lb/hr** | 1515.1 | 3611.0 | 44.4 | 5164.3 | 0.0 | 5164.3 | 5130.2 | 4855.7 | 407.7 | 9.0 | 833.2 | 291.5 |

**RUN NO: AF-R17.1h** **TITLE: LPDME with Shell Syngas : SV=6000**

**Balance Period:**

| | | |
|---|---|---|
| Start Date | 10/19/1999 | 17:00 |
| End Date | 10/20/1999 | 11:00 |

**Time From Start of Run (hr)**

| | |
|---|---|
| Start | 165.00 |
| End | 183.00 |

**Reaction Conditions**

| | |
|---|---|
| Temperature (°F) | 481.54 |
| Pressure (psig) | 752.24 |
| Space Velocity (sL/kg-hr) | 5866 |
| Vg (inlet) | 0.55 |

**Slurry Data**

| | |
|---|---|
| Catalyst Weight (lb oxide) | 941 |
| Slurry Level (Reactor Height, ft) | 43.5 |
| Slurry Conc. based on NDG (wt %) | 40.8 |
| Slurry Conc. based on DP (wt %) | 37.1 |
| Gas Holdup based on NDG (vol %) | 52.0 |
| Gas Holdup based on DP (vol %) | 44.2 |

**Performance Results**

| | |
|---|---|
| CO Conversion to H2 (%) | -0.1 |
| CO Conversion to MeOH (%) | 5.6 |
| CO Conversion to DME (%) | 14.8 |
| Methanol Production (Ton/day) | 3.53 |
| DME Production (Ton/day) | 4.44 |
| Methanol Productivity (gmole/kg-hr) | 9.75 |
| DME Productivity (gmole/kg-hr) | 8.54 |
| H2O Productivity (gmole/kg-hr) | 0.04 |
| MeOH Rate Constant (1-CSTR) | 4.30 |
| DME Rate Constant (1-CSTR) | 22.00 |

**Atomic/Mass Balance (% of reactor inlet)**

| | |
|---|---|
| C | 103.6 |
| H | 103.7 |
| O | 103.3 |
| N | 99.9 |
| Total Mass | 103.4 |

**Liquid Product Analysis (wt%)**

| | Sample# 21:50 |
|---|---|
| Methanol | 85.283 |
| Ethanol | 1.736 |
| 1-Propanol | 0.672 |
| iso-Propanol | 0.06 |
| 1-Butanol | 0.34 |
| 2-Butanol | 0.53 |
| iso-Butanol | 0.135 |
| 2-Methyl-1-Butanol | 0 |
| 1-Pentanol | 0.116 |
| 2-Methyl-1-Pentanol | 0 |
| 1-Hexanol | 0.022 |
| 2-Methyl-1-Isobutyrate | 0 |
| Methyl Acetate | 0.348 |
| Ethyl Acetate | 0 |
| Methyl Formate | 0.785 |
| DME | 6.851 |
| CO2 | 0 |
| Water | 0 |
| Oil | 0.2 |
| Total | 97.078 |

**RUN NO:** **AF-R17.1h** **TITLE:** **LPDME with Shell Syngas : SV=6000**

| | | FRESH MAKE-UP | RECYCLE | HP H2 MAKEUP | DRY FEED | ALCOHOL INJECT. | REACT FEED | REACT EFFL | 22.10 VAPOR | PURGE 1 PIC-201 | PURGE 2 22.11 | PURGE 3 07.20 | LIQUID PROD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **T** | **F** | 283.3 | 113.0 | 61.7 | 267.2 | 100.0 | 258.6 | 455.1 | 83.4 | 75.1 | 59.2 | 80.7 | 80.0 |
| **P** | **psig** | 812.0 | 811.4 | 789.1 | 780.5 | 780.5 | 780.5 | 752.2 | 747.5 | 707.6 | 27.9 | 125.0 | 1.0 |
| | | | | | | | | | | 8.76 | | | |
| **Comp** | **H2** | 34.74 | 16.25 | 100.00 | 29.77 | 0.00 | 29.77 | 14.65 | 14.15 | 16.25 | 2.70 | 1.42 | 0.00 |
| **(mole%)** | **CO** | 64.97 | 77.31 | 0.00 | 66.13 | 0.00 | 66.13 | 66.09 | 69.08 | 77.31 | 25.98 | 18.09 | 0.00 |
| | **N2** | 0.29 | 1.26 | 0.00 | 0.84 | 0.00 | 0.84 | 1.08 | 1.10 | 1.26 | 0.34 | 0.22 | 0.00 |
| | **CH4** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **CO2** | 0.00 | 5.02 | 0.00 | 3.16 | 0.00 | 3.16 | 8.76 | 9.68 | 5.02 | 33.97 | 46.87 | 0.00 |
| | **DME** | 0.00 | 0.17 | 0.00 | 0.10 | 0.00 | 0.10 | 4.32 | 5.36 | 0.17 | 33.52 | 32.27 | 5.14 |
| | **MeAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.16 |
| | **EtAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **MeFm** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.45 |
| | **MeOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.79 | 0.63 | 0.00 | 3.49 | 1.14 | 91.98 |
| | **H2O** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.09 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **Etoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.06 | 0.00 | 0.00 | 0.00 | 0.00 | 1.30 |
| | **1-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 | 0.00 | 0.00 | 0.00 | 0.00 | 0.39 |
| | **iso-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 |
| | **IBOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.06 |
| | **1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.16 |
| | **2-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.25 |
| | **2-Methyl 1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 |
| | **2-Methyl 1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-hexanol** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.01 |
| | **2-Methyl 1-Isobutyrate** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **others** | 0.00 | 0.00 | 0.00 | 0.00 | 100.00 | 0.00 | 0.15 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **TOTAL** | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| **Mole Wt** | **lb/lb mole** | 18.981 | 24.621 | 2.020 | 20.796 | 59.287 | 20.796 | 26.629 | 26.871 | 24.621 | 38.918 | 40.995 | 33.488 |
| **Flow** | **SCFH** | 30599 | 56137 | 8494 | 95230 | 0 | 95230 | 73980 | 71958 | 6338 | 26 | 7819 | 3352 |
| | **lb mole/hr** | 79.14 | 145.19 | 21.97 | 246.30 | 0.00 | 246.30 | 191.34 | 186.11 | 16.39 | 0.07 | 20.22 | 8.67 |
| | **lb/hr** | 1502.1 | 3574.7 | 44.4 | 5122.1 | 0.0 | 5122.1 | 5095.2 | 5001.0 | 403.6 | 2.7 | 829.1 | 290.3 |

**RUN NO: AF-R17.1i** **TITLE: LPDME with Shell Syngas : SV=6000**

**Balance Period:**

| | | |
|---|---|---|
| Start Date | 10/20/1999 | 11:00 |
| End Date | 10/21/1999 | 5:00 |

**Time From Start of Run (hr)**

| | |
|---|---|
| Start | 183.00 |
| End | 201.00 |

**Reaction Conditions**

| | |
|---|---|
| Temperature (°F) | 481.61 |
| Pressure (psig) | 750.50 |
| Space Velocity (sL/kg-hr) | 5788 |
| Vg (inlet) | 0.55 |

**Slurry Data**

| | |
|---|---|
| Catalyst Weight (lb oxide) | 941 |
| Slurry Level (Reactor Height, ft) | 43.5 |
| Slurry Conc. based on NDG (wt %) | 40.8 |
| Slurry Conc. based on DP (wt %) | 36.7 |
| Gas Holdup based on NDG (vol %) | 52.0 |
| Gas Holdup based on DP (vol %) | 43.3 |

**Performance Results**

| | |
|---|---|
| CO Conversion to H2 (%) | -0.1 |
| CO Conversion to MeOH (%) | 5.1 |
| CO Conversion to DME (%) | 15.3 |
| Methanol Production (Ton/day) | 3.17 |
| DME Production (Ton/day) | 4.52 |
| Methanol Productivity (gmole/kg-hr) | 8.76 |
| DME Productivity (gmole/kg-hr) | 8.69 |
| H2O Productivity (gmole/kg-hr) | 0.04 |
| MeOH Rate Constant (1-CSTR) | 3.27 |
| DME Rate Constant (1-CSTR) | 22.01 |

**Atomic/Mass Balance (% of reactor inlet)**

| | |
|---|---|
| C | 99.2 |
| H | 91.8 |
| O | 99.3 |
| N | 99.8 |
| Total Mass | 99.0 |

**Liquid Product Analysis (wt%)**

| | Sample# 21:30 |
|---|---|
| Methanol | 85.662 |
| Ethanol | 1.697 |
| 1-Propanol | 0.665 |
| iso-Propanol | 0.055 |
| 1-Butanol | 0.334 |
| 2-Butanol | 0.056 |
| iso-Butanol | 0.149 |
| 2-Methyl-1-Butanol | 0 |
| 1-Pentanol | 0.113 |
| 2-Methyl-1-Pentanol | 0 |
| 1-Hexanol | 0.02 |
| 2-Methyl-1-Isobutyrate | 0 |
| Methyl Acetate | 0.333 |
| Ethyl Acetate | 0 |
| Methyl Formate | 0.742 |
| DME | 7.081 |
| CO2 | 0 |
| Water | 0 |
| Oil | 0.2 |
| Total | 97.107 |

**RUN NO:** **AF-R17.1i** **TITLE:** **LPDME with Shell Syngas : SV=6000**

| | | FRESH MAKE-UP | RECYCLE | HP H2 MAKEUP | DRY FEED | ALCOHOL INJECT. | REACT FEED | REACT EFFL | 22.10 VAPOR | PURGE 1 PIC-201 | PURGE 2 22.11 | PURGE 3 07.20 | LIQUID PROD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **T** | **F** | 292.7 | 118.4 | 74.7 | 275.7 | 100.0 | 267.4 | 455.9 | 86.8 | 80.5 | 68.7 | 80.6 | 80.0 |
| **P** | **psig** | 812.5 | 809.2 | 801.1 | 779.2 | 779.2 | 779.2 | 750.5 | 745.4 | 707.5 | 29.1 | 125.0 | 1.0 |
| | | | | | | | | | | 8.48 | | | |
| **Comp** | **H2** | 34.72 | 16.12 | 100.00 | 30.26 | 0.00 | 30.26 | 15.84 | 14.69 | 16.12 | 2.68 | 1.34 | 0.00 |
| **(mole%)** | **CO** | 65.04 | 78.10 | 0.00 | 66.10 | 0.00 | 66.10 | 65.91 | 70.96 | 78.10 | 25.90 | 16.67 | 0.00 |
| | **N2** | 0.24 | 1.03 | 0.00 | 0.67 | 0.00 | 0.67 | 0.86 | 0.92 | 1.03 | 0.29 | 0.17 | 0.00 |
| | **CH4** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **CO2** | 0.00 | 4.75 | 0.00 | 2.97 | 0.00 | 2.97 | 8.48 | 8.81 | 4.75 | 32.98 | 43.96 | 0.00 |
| | **DME** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.30 | 4.27 | 0.00 | 33.54 | 36.26 | 5.29 |
| | **MeAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.15 |
| | **EtAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **MeFm** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.43 |
| | **MeOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.34 | 0.36 | 0.00 | 4.61 | 1.59 | 92.12 |
| | **H2O** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.08 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **Etoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.06 | 0.00 | 0.00 | 0.00 | 0.00 | 1.27 |
| | **1-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 | 0.00 | 0.00 | 0.00 | 0.00 | 0.38 |
| | **iso-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 |
| | **IBOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.07 |
| | **1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.16 |
| | **2-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 |
| | **2-Methyl 1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.04 |
| | **2-Methyl 1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-hexanol** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.01 |
| | **2-Methyl 1-Isobutyrate** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **others** | 0.00 | 0.00 | 0.00 | 0.00 | 100.00 | 0.00 | 0.12 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **TOTAL** | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| **Mole Wt** | **lb/lb mole** | 18.986 | 24.580 | 2.020 | 20.620 | 58.121 | 20.620 | 26.243 | 26.386 | 24.580 | 38.815 | 41.286 | 33.399 |
| **Flow** | **SCFH** | 30680 | 54775 | 8504 | 93959 | 0 | 93959 | 73477 | 68491 | 6234 | 126 | 7956 | 3175 |
| | **lb mole/hr** | 79.35 | 141.67 | 21.99 | 243.01 | 0.00 | 243.01 | 190.04 | 177.14 | 16.12 | 0.33 | 20.58 | 8.21 |
| | **lb/hr** | 1506.5 | 3482.1 | 44.4 | 5010.8 | 0.0 | 5010.9 | 4987.2 | 4674.1 | 396.3 | 12.6 | 849.6 | 274.2 |

**RUN NO: AF-R17.1j** **TITLE: LPDME with Shell Syngas : SV=6000**

**Balance Period:**

| | | |
|---|---|---|
| Start Date | 10/21/1999 | 5:00 |
| End Date | 10/21/1999 | 23:00 |

**Time From Start of Run (hr)**

| | |
|---|---|
| Start | 201.00 |
| End | 219.00 |

**Reaction Conditions**

| | |
|---|---|
| Temperature (°F) | 481.69 |
| Pressure (psig) | 749.91 |
| Space Velocity (sL/kg-hr) | 5770 |
| Vg (inlet) | 0.55 |

**Slurry Data**

| | |
|---|---|
| Catalyst Weight (lb oxide) | 941 |
| Slurry Level (Reactor Height, ft) | 43.5 |
| Slurry Conc. based on NDG (wt %) | 40.9 |
| Slurry Conc. based on DP (wt %) | 36.6 |
| Gas Holdup based on NDG (vol %) | 52.2 |
| Gas Holdup based on DP (vol %) | 43.1 |

**Performance Results**

| | |
|---|---|
| CO Conversion to H2 (%) | -0.1 |
| CO Conversion to MeOH (%) | 5.9 |
| CO Conversion to DME (%) | 15.1 |
| Methanol Production (Ton/day) | 3.59 |
| DME Production (Ton/day) | 4.44 |
| Methanol Productivity (gmole/kg-hr) | 9.93 |
| DME Productivity (gmole/kg-hr) | 8.54 |
| H2O Productivity (gmole/kg-hr) | 0.04 |
| MeOH Rate Constant (1-CSTR) | 4.20 |
| DME Rate Constant (1-CSTR) | 21.84 |

**Atomic/Mass Balance (% of reactor inlet)**

| | |
|---|---|
| C | 101.3 |
| H | 97.2 |
| O | 101.7 |
| N | 100.2 |
| Total Mass | 101.4 |

**Liquid Product Analysis (wt%)**

| | Sample# 14:30 | Sample# 23:00 |
|---|---|---|
| Methanol | 86.074 | 84.906 |
| Ethanol | 1.659 | 1.62 |
| 1-Propanol | 0.648 | 0.634 |
| iso-Propanol | 0.053 | 0.054 |
| 1-Butanol | 0.322 | 0.321 |
| 2-Butanol | 0.055 | 0.052 |
| iso-Butanol | 0.129 | 0.121 |
| 2-Methyl-1-Butanol | 0 | 0 |
| 1-Pentanol | 0.114 | 0.117 |
| 2-Methyl-1-Pentanol | 0 | 0 |
| 1-Hexanol | 0.021 | 0.027 |
| 2-Methyl-1-Isobutyrate | 0 | 0 |
| Methyl Acetate | 0.326 | 0.32 |
| Ethyl Acetate | 0 | 0 |
| Methyl Formate | 0.772 | 0.792 |
| DME | 6.621 | 7.699 |
| CO2 | 0 | 0 |
| Water | 0 | 0 |
| Oil | 0.2 | 0.2 |
| Total | 96.994 | 96.863 |

**RUN NO:** **AF-R17.1j** **TITLE:** **LPDME with Shell Syngas : SV=6000**

| | | FRESH MAKE-UP | RECYCLE | HP H2 MAKEUP | DRY FEED | ALCOHOL INJECT. | REACT FEED | REACT EFFL | 22.10 VAPOR | PURGE 1 PIC-201 | PURGE 2 22.11 | PURGE 3 07.20 | LIQUID PROD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **T** | **F** | 295.8 | 119.5 | 77.0 | 281.6 | 100.0 | 273.8 | 457.7 | 87.2 | 81.3 | 68.9 | 81.0 | 80.0 |
| **P** | **psig** | 812.6 | 808.6 | 789.6 | 778.8 | 778.8 | 778.8 | 749.9 | 745.0 | 707.3 | 29.3 | 125.0 | 1.0 |
| | | | | | | | | | | 8.54 | | | |
| **Comp** | **H2** | 35.09 | 16.82 | 100.00 | 30.50 | 0.00 | 30.50 | 15.16 | 14.66 | 16.82 | 2.72 | 1.35 | 0.00 |
| **(mole%)** | **CO** | 64.68 | 77.49 | 0.00 | 65.91 | 0.00 | 65.91 | 65.87 | 69.66 | 77.49 | 25.82 | 16.69 | 0.00 |
| | **N2** | 0.23 | 0.99 | 0.00 | 0.65 | 0.00 | 0.65 | 0.84 | 0.88 | 0.99 | 0.27 | 0.16 | 0.00 |
| | **CH4** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **CO2** | 0.00 | 4.70 | 0.00 | 2.94 | 0.00 | 2.94 | 8.54 | 9.49 | 4.70 | 32.79 | 43.92 | 0.00 |
| | **DME** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.29 | 4.61 | 0.00 | 33.83 | 36.17 | 5.36 |
| | **MeAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.15 |
| | **EtAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **MeFm** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.45 |
| | **MeOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.99 | 0.70 | 0.00 | 4.58 | 1.71 | 92.10 |
| | **H2O** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.09 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **Etoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.06 | 0.00 | 0.00 | 0.00 | 0.00 | 1.23 |
| | **1-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 | 0.00 | 0.00 | 0.00 | 0.00 | 0.37 |
| | **iso-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 |
| | **IBOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.06 |
| | **1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.15 |
| | **2-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **2-Methyl 1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 |
| | **2-Methyl 1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-hexanol** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.01 |
| | **2-Methyl 1-Isobutyrate** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **others** | 0.00 | 0.00 | 0.00 | 0.00 | 100.00 | 0.00 | 0.14 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **TOTAL** | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| **Mole Wt** | **lb/lb mole** | 18.890 | 24.392 | 2.020 | 20.554 | 58.223 | 20.554 | 26.460 | 26.578 | 24.392 | 38.823 | 41.266 | 33.398 |
| **Flow** | **SCFH** | 30783 | 54386 | 8504 | 93674 | 0 | 93674 | 72363 | 69131 | 6327 | 15 | 7893 | 3405 |
| | **lb mole/hr** | 79.62 | 140.66 | 21.99 | 242.27 | 0.00 | 242.27 | 187.16 | 178.80 | 16.36 | 0.04 | 20.41 | 8.81 |
| | **lb/hr** | 1504.0 | 3431.0 | 44.4 | 4979.7 | 0.0 | 4979.7 | 4952.1 | 4752.0 | 399.1 | 1.5 | 842.4 | 294.1 |

**RUN NO: AF-R17.1k** **TITLE: LPDME with Shell Syngas : SV=6000**

**Balance Period:**

| | | |
|---|---|---|
| Start Date | 10/22/1999 | 11:00 |
| End Date | 10/23/1999 | 5:00 |

**Time From Start of Run (hr)**

| | |
|---|---|
| Start | 231.00 |
| End | 249.00 |

**Reaction Conditions**

| | |
|---|---|
| Temperature (°F) | 481.61 |
| Pressure (psig) | 749.21 |
| Space Velocity (sL/kg-hr) | 5744 |
| Vg (inlet) | 0.55 |

**Slurry Data**

| | |
|---|---|
| Catalyst Weight (lb oxide) | 941 |
| Slurry Level (Reactor Height, ft) | 43.5 |
| Slurry Conc. based on NDG (wt %) | 41.0 |
| Slurry Conc. based on DP (wt %) | 36.4 |
| Gas Holdup based on NDG (vol %) | 52.4 |
| Gas Holdup based on DP (vol %) | 42.7 |

**Performance Results**

| | |
|---|---|
| CO Conversion to H2 (%) | -0.1 |
| CO Conversion to MeOH (%) | 5.4 |
| CO Conversion to DME (%) | 15.3 |
| Methanol Production (Ton/day) | 3.29 |
| DME Production (Ton/day) | 4.47 |
| Methanol Productivity (gmole/kg-hr) | 9.11 |
| DME Productivity (gmole/kg-hr) | 8.59 |
| H2O Productivity (gmole/kg-hr) | 0.04 |
| MeOH Rate Constant (1-CSTR) | 3.24 |
| DME Rate Constant (1-CSTR) | 22.10 |

**Atomic/Mass Balance (% of reactor inlet)**

| | |
|---|---|
| C | 100.9 |
| H | 94.7 |
| O | 101.2 |
| N | 100.0 |
| Total Mass | 100.9 |

**Liquid Product Analysis (wt%)**

| | Sample# 15:05 | Sample# 19:00 |
|---|---|---|
| Methanol | 86.574 | 87.442 |
| Ethanol | 1.601 | 1.644 |
| 1-Propanol | 0.623 | 0.647 |
| iso-Propanol | 0.047 | 0.022 |
| 1-Butanol | 0.323 | 0.324 |
| 2-Butanol | 0.053 | 0.056 |
| iso-Butanol | 0.124 | 0.132 |
| 2-Methyl-1-Butanol | 0 | 0 |
| 1-Pentanol | 0.119 | 0.137 |
| 2-Methyl-1-Pentanol | 0 | 0 |
| 1-Hexanol | 0.024 | 0.052 |
| 2-Methyl-1-Isobutyrate | 0 | 0 |
| Methyl Acetate | 0.296 | 0.297 |
| Ethyl Acetate | 0 | 0 |
| Methyl Formate | 0.743 | 0.717 |
| DME | 6.605 | 4.944 |
| CO2 | 0 | 0 |
| Water | 0.258 | 0.257 |
| Oil | 0.2 | 0.2 |
| Total | 97.59 | 96.871 |

**RUN NO:** **AF-R17.1k** **TITLE:** **LPDME with Shell Syngas : SV=6000**

| | | FRESH MAKE-UP | RECYCLE | HP H2 MAKEUP | DRY FEED | ALCOHOL INJECT. | REACT FEED | REACT EFFL | 22.10 VAPOR | PURGE 1 PIC-201 | PURGE 2 22.11 | PURGE 3 07.20 | LIQUID PROD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **T** | **F** | 296.7 | 122.1 | 81.5 | 281.6 | 100.0 | 273.2 | 456.1 | 90.0 | 84.4 | 75.6 | 83.3 | 80.0 |
| **P** | **psig** | 812.3 | 807.2 | 795.2 | 778.4 | 778.4 | 778.4 | 749.2 | 743.1 | 706.9 | 28.9 | 125.0 | 1.0 |
| | | | | | | | | | | 8.29 | | | |
| **Comp** | **H2** | 34.33 | 16.98 | 100.00 | 30.83 | 0.00 | 30.83 | 16.30 | 14.59 | 16.98 | 3.21 | 1.36 | 0.00 |
| **(mole%)** | **CO** | 65.42 | 77.46 | 0.00 | 65.67 | 0.00 | 65.67 | 65.45 | 69.73 | 77.46 | 26.48 | 16.83 | 0.00 |
| | **N2** | 0.25 | 1.00 | 0.00 | 0.66 | 0.00 | 0.66 | 0.85 | 0.90 | 1.00 | 0.31 | 0.16 | 0.00 |
| | **CH4** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **CO2** | 0.00 | 4.56 | 0.00 | 2.84 | 0.00 | 2.84 | 8.29 | 9.22 | 4.56 | 30.94 | 43.49 | 0.00 |
| | **DME** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.30 | 4.80 | 0.00 | 33.71 | 36.13 | 4.28 |
| | **MeAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.14 |
| | **EtAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **MeFm** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.42 |
| | **MeOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.55 | 0.75 | 0.00 | 5.34 | 2.03 | 92.78 |
| | **H2O** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.08 | 0.00 | 0.00 | 0.00 | 0.00 | 0.49 |
| | **Etoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.04 | 0.00 | 0.00 | 0.00 | 0.00 | 1.20 |
| | **1-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 | 0.00 | 0.00 | 0.00 | 0.00 | 0.36 |
| | **iso-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **IBOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.06 |
| | **1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.15 |
| | **2-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 |
| | **2-Methyl 1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 |
| | **2-Methyl 1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-hexanol** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.01 |
| | **2-Methyl 1-Isobutyrate** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **others** | 0.00 | 0.00 | 0.00 | 0.00 | 100.00 | 0.00 | 0.12 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **TOTAL** | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| **Mole Wt** | **lb/lb mole** | 19.087 | 24.327 | 2.020 | 20.451 | 58.334 | 20.451 | 26.099 | 26.586 | 24.327 | 38.408 | 41.202 | 33.160 |
| **Flow** | **SCFH** | 30427 | 54331 | 8495 | 93253 | 0 | 93253 | 72739 | 68399 | 6640 | 137 | 8011 | 3009 |
| | **lb mole/hr** | 78.70 | 140.52 | 21.97 | 241.19 | 0.00 | 241.19 | 188.13 | 176.90 | 17.17 | 0.35 | 20.72 | 7.78 |
| | **lb/hr** | 1502.1 | 3418.4 | 44.4 | 4932.5 | 0.0 | 4932.5 | 4910.0 | 4703.3 | 417.8 | 13.6 | 853.6 | 258.0 |

**RUN NO: AF-R17.1l** **TITLE: LPDME with Shell Syngas : SV=6000**

**Balance Period:**

| | | |
|---|---|---|
| Start Date | 10/23/1999 | 5:00 |
| End Date | 10/23/1999 | 23:00 |

**Time From Start of Run (hr)**

| | |
|---|---|
| Start | 249.00 |
| End | 267.00 |

**Reaction Conditions**

| | |
|---|---|
| Temperature (°F) | 481.56 |
| Pressure (psig) | 750.20 |
| Space Velocity (sL/kg-hr) | 5808 |
| Vg (inlet) | 0.55 |

**Slurry Data**

| | |
|---|---|
| Catalyst Weight (lb oxide) | 941 |
| Slurry Level (Reactor Height, ft) | 43.5 |
| Slurry Conc. based on NDG (wt %) | 41.4 |
| Slurry Conc. based on DP (wt %) | 36.6 |
| Gas Holdup based on NDG (vol %) | 53.1 |
| Gas Holdup based on DP (vol %) | 43.0 |

**Performance Results**

| | |
|---|---|
| CO Conversion to H2 (%) | -0.1 |
| CO Conversion to MeOH (%) | 5.0 |
| CO Conversion to DME (%) | 14.8 |
| Methanol Production (Ton/day) | 3.13 |
| DME Production (Ton/day) | 4.42 |
| Methanol Productivity (gmole/kg-hr) | 8.65 |
| DME Productivity (gmole/kg-hr) | 8.49 |
| H2O Productivity (gmole/kg-hr) | 0.04 |
| MeOH Rate Constant (1-CSTR) | 3.24 |
| DME Rate Constant (1-CSTR) | 21.63 |

**Atomic/Mass Balance (% of reactor inlet)**

| | |
|---|---|
| C | 100.3 |
| H | 93.7 |
| O | 101.0 |
| N | 100.3 |
| Total Mass | 100.5 |

**Liquid Product Analysis (wt%)**

| | Sample# 9:30 | Sample# 20:00 |
|---|---|---|
| Methanol | 87.119 | 87.289 |
| Ethanol | 1.686 | 1.635 |
| 1-Propanol | 0.674 | 0.645 |
| iso-Propanol | 0.049 | 0.046 |
| 1-Butanol | 0.333 | 0.314 |
| 2-Butanol | 0.055 | 0.054 |
| iso-Butanol | 0.127 | 0.122 |
| 2-Methyl-1-Butanol | 0 | 0 |
| 1-Pentanol | 0.144 | 0.137 |
| 2-Methyl-1-Pentanol | 0 | 0 |
| 1-Hexanol | 0.024 | 0.026 |
| 2-Methyl-1-Isobutyrate | 0 | 0 |
| Methyl Acetate | 0.298 | 0.291 |
| Ethyl Acetate | 0 | 0 |
| Methyl Formate | 0.744 | 0.757 |
| DME | 5.44 | 5.591 |
| CO2 | 0 | 0 |
| Water | 0.251 | 0.243 |
| Oil | 0.2 | 0.2 |
| Total | 97.144 | 97.35 |

**RUN NO:** **AF-R17.1l** **TITLE:** **LPDME with Shell Syngas : SV=6000**

| | | FRESH MAKE-UP | RECYCLE | HP H2 MAKEUP | DRY FEED | ALCOHOL INJECT. | REACT FEED | REACT EFFL | 22.10 VAPOR | PURGE 1 PIC-201 | PURGE 2 22.11 | PURGE 3 07.20 | LIQUID PROD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **T** | **F** | 291.6 | 119.8 | 77.6 | 277.6 | 100.0 | 269.2 | 456.7 | 87.2 | 81.3 | 71.7 | 81.0 | 80.0 |
| **P** | **psig** | 812.9 | 809.3 | 778.9 | 779.4 | 779.4 | 779.4 | 750.2 | 743.7 | 706.3 | 29.1 | 122.9 | 1.0 |
| | | | | | | | | | | 8.18 | | | |
| **Comp** | **H2** | 33.88 | 16.42 | 100.00 | 29.66 | 0.00 | 29.66 | 15.55 | 14.64 | 16.42 | 2.38 | 1.36 | 0.00 |
| **(mole%)** | **CO** | 65.85 | 77.84 | 0.00 | 66.65 | 0.00 | 66.65 | 66.63 | 70.73 | 77.84 | 26.65 | 17.08 | 0.00 |
| | **N2** | 0.27 | 1.15 | 0.00 | 0.76 | 0.00 | 0.76 | 0.97 | 1.02 | 1.15 | 0.29 | 0.18 | 0.00 |
| | **CH4** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **CO2** | 0.00 | 4.59 | 0.00 | 2.92 | 0.00 | 2.92 | 8.18 | 9.09 | 4.59 | 32.41 | 43.39 | 0.00 |
| | **DME** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.16 | 4.20 | 0.00 | 33.38 | 36.23 | 4.09 |
| | **MeAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.14 |
| | **EtAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **MeFm** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.43 |
| | **MeOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.23 | 0.31 | 0.00 | 4.88 | 1.75 | 92.93 |
| | **H2O** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.08 | 0.00 | 0.00 | 0.00 | 0.00 | 0.47 |
| | **Etoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.06 | 0.00 | 0.00 | 0.00 | 0.00 | 1.23 |
| | **1-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 | 0.00 | 0.00 | 0.00 | 0.00 | 0.37 |
| | **iso-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 |
| | **IBOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.06 |
| | **1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.15 |
| | **2-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 |
| | **2-Methyl 1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 |
| | **2-Methyl 1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-hexanol** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.01 |
| | **2-Methyl 1-Isobutyrate** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **others** | 0.00 | 0.00 | 0.00 | 0.00 | 100.00 | 0.00 | 0.11 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **TOTAL** | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| **Mole Wt** | **lb/lb mole** | 19.205 | 24.475 | 2.020 | 20.769 | 58.185 | 20.769 | 26.237 | 26.428 | 24.475 | 38.781 | 41.193 | 33.147 |
| **Flow** | **SCFH** | 30065 | 55717 | 8499 | 94281 | 0 | 94281 | 74344 | 70498 | 6606 | 4 | 7919 | 3167 |
| | **lb mole/hr** | 77.76 | 144.10 | 21.98 | 243.84 | 0.00 | 243.84 | 192.28 | 182.33 | 17.09 | 0.01 | 20.48 | 8.19 |
| | **lb/hr** | 1493.4 | 3526.9 | 44.4 | 5064.4 | 0.0 | 5064.4 | 5044.9 | 4818.6 | 418.2 | 0.4 | 843.7 | 271.5 |

**RUN NO: AF-R17.1m** **TITLE: LPDME with Shell Syngas : SV=6000**

**Balance Period:**

| | | |
|---|---|---|
| Start Date | 10/23/1999 | 23:00 |
| End Date | 10/24/1999 | 17:00 |

**Time From Start of Run (hr)**

| | |
|---|---|
| Start | 267.00 |
| End | 285.00 |

**Reaction Conditions**

| | |
|---|---|
| Temperature (°F) | 481.56 |
| Pressure (psig) | 752.93 |
| Space Velocity (sL/kg-hr) | 6081 |
| Vg (inlet) | 0.57 |

**Slurry Data**

| | |
|---|---|
| Catalyst Weight (lb oxide) | 941 |
| Slurry Level (Reactor Height, ft) | 43.5 |
| Slurry Conc. based on NDG (wt %) | 41.7 |
| Slurry Conc. based on DP (wt %) | 37.0 |
| Gas Holdup based on NDG (vol %) | 53.5 |
| Gas Holdup based on DP (vol %) | 44.0 |

**Performance Results**

| | |
|---|---|
| CO Conversion to H2 (%) | -0.1 |
| CO Conversion to MeOH (%) | 5.3 |
| CO Conversion to DME (%) | 14.0 |
| Methanol Production (Ton/day) | 3.42 |
| DME Production (Ton/day) | 4.35 |
| Methanol Productivity (gmole/kg-hr) | 9.46 |
| DME Productivity (gmole/kg-hr) | 8.37 |
| H2O Productivity (gmole/kg-hr) | 0.04 |
| MeOH Rate Constant (1-CSTR) | 3.30 |
| DME Rate Constant (1-CSTR) | 21.23 |

**Atomic/Mass Balance (% of reactor inlet)**

| | |
|---|---|
| C | 99.2 |
| H | 91.0 |
| O | 100.1 |
| N | 100.1 |
| Total Mass | 99.4 |

**Liquid Product Analysis (wt%)**

| | Sample# 12:00 |
|---|---|
| Methanol | 86.438 |
| Ethanol | 1.513 |
| 1-Propanol | 0.661 |
| iso-Propanol | 0.049 |
| 1-Butanol | 0.284 |
| 2-Butanol | 0.057 |
| iso-Butanol | 0.116 |
| 2-Methyl-1-Butanol | 0 |
| 1-Pentanol | 0.129 |
| 2-Methyl-1-Pentanol | 0 |
| 1-Hexanol | 0.05 |
| 2-Methyl-1-Isobutyrate | 0 |
| Methyl Acetate | 0.278 |
| Ethyl Acetate | 0 |
| Methyl Formate | 0.778 |
| DME | 6.647 |
| CO2 | 0 |
| Water | 0.247 |
| Oil | 0.2 |
| Total | 97.447 |

**RUN NO:** AF-R17.1m **TITLE:** LPDME with Shell Syngas : SV=6000

| | | FRESH MAKE-UP | RECYCLE | HP H2 MAKEUP | DRY FEED | ALCOHOL INJECT. | REACT FEED | REACT EFFL | 22.10 VAPOR | PURGE 1 PIC-201 | PURGE 2 22.11 | PURGE 3 07.20 | LIQUID PROD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| T | F | 290.4 | 118.0 | 71.1 | 279.3 | 100.0 | 271.3 | 458.5 | 84.7 | 77.8 | 63.9 | 78.5 | 80.0 |
| P | psig | 817.2 | 816.0 | 819.6 | 783.1 | 783.1 | 783.1 | 752.9 | 744.6 | 705.0 | 29.2 | 118.3 | 1.0 |
| | | | | | | | | | | 8.28 | | | |
| Comp | H2 | 34.83 | 16.81 | 100.00 | 29.72 | 0.00 | 29.72 | 15.77 | 15.11 | 16.81 | 2.63 | 1.41 | 0.00 |
| (mole%) | CO | 64.86 | 76.88 | 0.00 | 66.19 | 0.00 | 66.19 | 66.20 | 70.76 | 76.88 | 27.88 | 17.11 | 0.00 |
| | N2 | 0.31 | 1.38 | 0.00 | 0.92 | 0.00 | 0.92 | 1.17 | 1.24 | 1.38 | 0.37 | 0.22 | 0.00 |
| | CH4 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | CO2 | 0.00 | 4.94 | 0.00 | 3.17 | 0.00 | 3.17 | 8.28 | 9.02 | 4.94 | 33.46 | 43.52 | 0.00 |
| | DME | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 3.90 | 3.59 | 0.00 | 31.56 | 36.24 | 4.93 |
| | MeAc | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.13 |
| | EtAc | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | MeFm | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.44 |
| | MeOH | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.41 | 0.28 | 0.00 | 4.10 | 1.51 | 92.21 |
| | H2O | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.08 | 0.00 | 0.00 | 0.00 | 0.00 | 0.47 |
| | Etoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 | 0.00 | 0.00 | 0.00 | 0.00 | 1.12 |
| | 1-Proh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 | 0.00 | 0.00 | 0.00 | 0.00 | 0.38 |
| | iso-Proh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 |
| | IBOH | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 |
| | 1-Buoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.13 |
| | 2-Buoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 |
| | 2-Methyl 1-Buoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | 1-Peoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 |
| | 2-Methyl 1-Peoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | 1-hexanol | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | 2-Methyl 1-Isobutyrate | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | others | 0.00 | 0.00 | 0.00 | 0.00 | 100.00 | 0.00 | 0.11 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | TOTAL | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| Mole Wt | lb/lb mole | 18.959 | 24.431 | 2.020 | 20.792 | 58.660 | 20.792 | 26.157 | 26.184 | 24.431 | 38.525 | 41.191 | 33.246 |
| Flow | SCFH | 30516 | 59696 | 8499 | 98712 | 0 | 98712 | 78073 | 73493 | 6217 | 7 | 7677 | 3499 |
| | lb mole/hr | 78.93 | 154.39 | 21.98 | 255.30 | 0.00 | 255.30 | 201.92 | 190.08 | 16.08 | 0.02 | 19.86 | 9.05 |
| | lb/hr | 1496.3 | 3772.1 | 44.4 | 5308.4 | 0.0 | 5308.4 | 5281.7 | 4977.1 | 392.9 | 0.7 | 817.9 | 300.9 |

**RUN NO: AF-R17.1n** **TITLE: LPDME with Shell Syngas : SV=6000**

**Balance Period:**

| | | |
|---|---|---|
| Start Date | 10/24/1999 | 17:00 |
| End Date | 10/25/1999 | 9:00 |

**Time From Start of Run (hr)**

| | |
|---|---|
| Start | 285.00 |
| End | 301.00 |

**Reaction Conditions**

| | |
|---|---|
| Temperature (°F) | 481.58 |
| Pressure (psig) | 753.47 |
| Space Velocity (sL/kg-hr) | 6097 |
| Vg (inlet) | 0.58 |

**Slurry Data**

| | |
|---|---|
| Catalyst Weight (lb oxide) | 941 |
| Slurry Level (Reactor Height, ft) | 43.5 |
| Slurry Conc. based on NDG (wt %) | 41.9 |
| Slurry Conc. based on DP (wt %) | 37.1 |
| Gas Holdup based on NDG (vol %) | 53.8 |
| Gas Holdup based on DP (vol %) | 44.2 |

**Performance Results**

| | |
|---|---|
| CO Conversion to H2 (%) | -0.1 |
| CO Conversion to MeOH (%) | 5.8 |
| CO Conversion to DME (%) | 13.6 |
| Methanol Production (Ton/day) | 3.77 |
| DME Production (Ton/day) | 4.25 |
| Methanol Productivity (gmole/kg-hr) | 10.41 |
| DME Productivity (gmole/kg-hr) | 8.17 |
| H2O Productivity (gmole/kg-hr) | 0.04 |
| MeOH Rate Constant (1-CSTR) | 3.84 |
| DME Rate Constant (1-CSTR) | 20.37 |

**Atomic/Mass Balance (% of reactor inlet)**

| | |
|---|---|
| C | 97.1 |
| H | 91.6 |
| O | 97.8 |
| N | 99.9 |
| Total Mass | 97.3 |

**Liquid Product Analysis (wt%)**

| | Sample# 19:30 |
|---|---|
| Methanol | 86.397 |
| Ethanol | 1.485 |
| 1-Propanol | 0.644 |
| iso-Propanol | 0.047 |
| 1-Butanol | 0.277 |
| 2-Butanol | 0.051 |
| iso-Butanol | 0.11 |
| 2-Methyl-1-Butanol | 0 |
| 1-Pentanol | 0.122 |
| 2-Methyl-1-Pentanol | 0 |
| 1-Hexanol | 0.11 |
| 2-Methyl-1-Isobutyrate | 0 |
| Methyl Acetate | 0.27 |
| Ethyl Acetate | 0 |
| Methyl Formate | 0.759 |
| DME | 6.58 |
| CO2 | 0 |
| Water | 0.25 |
| Oil | 0.2 |
| Total | 97.302 |

**RUN NO:** AF-R17.1n **TITLE:** LPDME with Shell Syngas : SV=6000

| | | FRESH MAKE-UP | RECYCLE | HP H2 MAKEUP | DRY FEED | ALCOHOL INJECT. | REACT FEED | REACT EFFL | 22.10 VAPOR | PURGE 1 PIC-201 | PURGE 2 22.11 | PURGE 3 07.20 | LIQUID PROD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| T | F | 291.2 | 117.2 | 66.0 | 282.4 | 100.0 | 275.2 | 459.0 | 85.2 | 77.5 | 61.9 | 78.5 | 80.0 |
| P | psig | 817.3 | 816.6 | 795.9 | 783.7 | 783.7 | 783.7 | 753.5 | 745.5 | 705.2 | 28.8 | 120.0 | 1.0 |
| | | | | | | | | | | 8.28 | | | |
| Comp | H2 | 35.36 | 16.93 | 100.00 | 29.85 | 0.00 | 29.85 | 15.37 | 15.22 | 16.93 | 2.73 | 1.43 | 0.00 |
| (mole%) | CO | 64.34 | 76.82 | 0.00 | 66.07 | 0.00 | 66.07 | 66.22 | 70.40 | 76.82 | 27.99 | 17.25 | 0.00 |
| | N2 | 0.30 | 1.35 | 0.00 | 0.91 | 0.00 | 0.91 | 1.16 | 1.25 | 1.35 | 0.37 | 0.22 | 0.00 |
| | CH4 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | CO2 | 0.00 | 4.90 | 0.00 | 3.16 | 0.00 | 3.16 | 8.28 | 8.87 | 4.90 | 33.40 | 43.58 | 0.00 |
| | DME | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 3.82 | 3.58 | 0.00 | 31.64 | 36.10 | 4.89 |
| | MeAc | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.12 |
| | EtAc | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | MeFm | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.43 |
| | MeOH | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.87 | 0.68 | 0.00 | 3.87 | 1.42 | 92.28 |
| | H2O | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.08 | 0.00 | 0.00 | 0.00 | 0.00 | 0.47 |
| | Etoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 | 0.00 | 0.00 | 0.00 | 0.00 | 1.10 |
| | 1-Proh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 | 0.00 | 0.00 | 0.00 | 0.00 | 0.37 |
| | iso-Proh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 |
| | IBOH | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 |
| | 1-Buoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.13 |
| | 2-Buoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | 2-Methyl 1-Buoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | 1-Peoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 |
| | 2-Methyl 1-Peoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | 1-hexanol | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.04 |
| | 2-Methyl 1-Isobutyrate | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | others | 0.00 | 0.00 | 0.00 | 0.00 | 100.00 | 0.00 | 0.12 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | TOTAL | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| Mole Wt | lb/lb mole | 18.820 | 24.394 | 2.020 | 20.757 | 59.012 | 20.757 | 26.268 | 26.144 | 24.394 | 38.497 | 41.167 | 33.239 |
| Flow | SCFH | 30582 | 59897 | 8495 | 98973 | 0 | 98973 | 77778 | 71898 | 6271 | 13 | 7664 | 3577 |
| | lb mole/hr | 79.10 | 154.91 | 21.97 | 255.98 | 0.00 | 255.98 | 201.16 | 185.95 | 16.22 | 0.03 | 19.82 | 9.25 |
| | lb/hr | 1488.6 | 3779.0 | 44.4 | 5313.4 | 0.0 | 5313.4 | 5284.0 | 4861.6 | 395.7 | 1.3 | 816.0 | 307.5 |

**RUN NO: AF-R17.1o** **TITLE: LPDME with Shell Syngas : SV=6000**

**Balance Period:**

| | | |
|---|---|---|
| Start Date | 10/25/1999 | 12:00 |
| End Date | 10/26/1999 | 6:00 |

**Time From Start of Run (hr)**

| | |
|---|---|
| Start | 304.00 |
| End | 322.00 |

**Reaction Conditions**

| | |
|---|---|
| Temperature (°F) | 481.56 |
| Pressure (psig) | 751.28 |
| Space Velocity (sL/kg-hr) | 6052 |
| Vg (inlet) | 0.57 |

**Slurry Data**

| | |
|---|---|
| Catalyst Weight (lb oxide) | 941 |
| Slurry Level (Reactor Height, ft) | 43.5 |
| Slurry Conc. based on NDG (wt %) | 41.8 |
| Slurry Conc. based on DP (wt %) | 36.9 |
| Gas Holdup based on NDG (vol %) | 53.9 |
| Gas Holdup based on DP (vol %) | 43.6 |

**Performance Results**

| | |
|---|---|
| CO Conversion to H2 (%) | -0.1 |
| CO Conversion to MeOH (%) | 5.5 |
| CO Conversion to DME (%) | 13.9 |
| Methanol Production (Ton/day) | 3.52 |
| DME Production (Ton/day) | 4.28 |
| Methanol Productivity (gmole/kg-hr) | 9.74 |
| DME Productivity (gmole/kg-hr) | 8.23 |
| H2O Productivity (gmole/kg-hr) | 0.04 |
| MeOH Rate Constant (1-CSTR) | 3.27 |
| DME Rate Constant (1-CSTR) | 20.86 |

**Atomic/Mass Balance (% of reactor inlet)**

| | |
|---|---|
| C | 100.0 |
| H | 94.2 |
| O | 100.6 |
| N | 100.5 |
| Total Mass | 100.2 |

**Liquid Product Analysis (wt%)**

| | Sample# 10:30 | Sample# 19:30 |
|---|---|---|
| Methanol | 86.386 | 86.414 |
| Ethanol | 1.484 | 1.443 |
| 1-Propanol | 0.643 | 0.539 |
| iso-Propanol | 0.049 | 0.044 |
| 1-Butanol | 0.28 | 0.275 |
| 2-Butanol | 0.053 | 0.048 |
| iso-Butanol | 0.115 | 0.105 |
| 2-Methyl-1-Butanol | 0 | 0 |
| 1-Pentanol | 0.125 | 0.119 |
| 2-Methyl-1-Pentanol | 0 | 0 |
| 1-Hexanol | 0.055 | 0.081 |
| 2-Methyl-1-Isobutyrate | 0 | 0 |
| Methyl Acetate | 0.275 | 0.299 |
| Ethyl Acetate | 0 | 0 |
| Methyl Formate | 0.765 | 0.794 |
| DME | 6.817 | 6.758 |
| CO2 | 0 | 0 |
| Water | 0.25 | 0.253 |
| Oil | 0.2 | 0.2 |
| Total | 97.497 | 97.372 |

**RUN NO:** AF-R17.1o **TITLE:** LPDME with Shell Syngas : SV=6000

| | | FRESH MAKE-UP | RECYCLE | HP H2 MAKEUP | DRY FEED | ALCOHOL INJECT. | REACT FEED | REACT EFFL | 22.10 VAPOR | PURGE 1 PIC-201 | PURGE 2 22.11 | PURGE 3 07.20 | LIQUID PROD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| T | F | 294.1 | 119.6 | 73.1 | 283.5 | 100.0 | 276.1 | 458.4 | 87.1 | 80.2 | 66.9 | 80.4 | 80.0 |
| P | psig | 816.1 | 814.0 | 792.5 | 782.0 | 782.0 | 782.0 | 751.3 | 743.6 | 704.2 | 29.3 | 120.0 | 1.0 |
| | | | | | | | | | | 8.17 | | | |
| Comp | H2 | 35.19 | 17.23 | 100.00 | 30.15 | 0.00 | 30.15 | 16.14 | 15.49 | 17.23 | 2.64 | 1.45 | 0.00 |
| (mole%) | CO | 64.48 | 76.54 | 0.00 | 65.80 | 0.00 | 65.80 | 65.78 | 69.66 | 76.54 | 26.50 | 17.02 | 0.00 |
| | N2 | 0.33 | 1.42 | 0.00 | 0.96 | 0.00 | 0.96 | 1.21 | 1.28 | 1.42 | 0.37 | 0.23 | 0.00 |
| | CH4 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | CO2 | 0.00 | 4.81 | 0.00 | 3.09 | 0.00 | 3.09 | 8.17 | 8.96 | 4.81 | 31.94 | 43.33 | 0.00 |
| | DME | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 3.86 | 3.91 | 0.00 | 30.77 | 36.36 | 5.03 |
| | MeAc | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.13 |
| | EtAc | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | MeFm | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.44 |
| | MeOH | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.57 | 0.70 | 0.00 | 7.78 | 1.62 | 92.17 |
| | H2O | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.08 | 0.00 | 0.00 | 0.00 | 0.00 | 0.48 |
| | Etoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 | 0.00 | 0.00 | 0.00 | 0.00 | 1.09 |
| | 1-Proh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 | 0.00 | 0.00 | 0.00 | 0.00 | 0.34 |
| | iso-Proh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 |
| | IBOH | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 |
| | 1-Buoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.13 |
| | 2-Buoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | 2-Methyl 1-Buoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | 1-Peoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 |
| | 2-Methyl 1-Peoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | 1-hexanol | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | 2-Methyl 1-Isobutyrate | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | others | 0.00 | 0.00 | 0.00 | 0.00 | 100.00 | 0.00 | 0.11 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | TOTAL | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| Mole Wt | lb/lb mole | 18.864 | 24.300 | 2.020 | 20.668 | 58.882 | 20.668 | 26.043 | 26.150 | 24.300 | 38.287 | 41.176 | 33.244 |
| Flow | SCFH | 30642 | 59099 | 8500 | 98241 | 0 | 98241 | 77550 | 73570 | 6243 | 38 | 7723 | 3282 |
| | lb mole/hr | 79.25 | 152.85 | 21.98 | 254.09 | 0.00 | 254.09 | 200.57 | 190.28 | 16.15 | 0.10 | 19.98 | 8.49 |
| | lb/hr | 1495.0 | 3714.3 | 44.4 | 5251.6 | 0.0 | 5251.6 | 5223.4 | 4975.8 | 392.3 | 3.8 | 822.5 | 282.2 |

**RUN NO: AF-R17.1p** **TITLE: LPDME with Shell Syngas : SV=6000**

**Balance Period:**

| | | |
|---|---|---|
| Start Date | 10/27/1999 | 0:00 |
| End Date | 10/27/1999 | 9:00 |

**Time From Start of Run (hr)**

| | |
|---|---|
| Start | 340.00 |
| End | 349.00 |

**Reaction Conditions**

| | |
|---|---|
| Temperature (°F) | 481.61 |
| Pressure (psig) | 750.76 |
| Space Velocity (sL/kg-hr) | 6016 |
| Vg (inlet) | 0.57 |

**Slurry Data**

| | |
|---|---|
| Catalyst Weight (lb oxide) | 941 |
| Slurry Level (Reactor Height, ft) | 43.5 |
| Slurry Conc. based on NDG (wt %) | 41.8 |
| Slurry Conc. based on DP (wt %) | 36.9 |
| Gas Holdup based on NDG (vol %) | 53.9 |
| Gas Holdup based on DP (vol %) | 43.6 |

**Performance Results**

| | |
|---|---|
| CO Conversion to H2 (%) | -0.1 |
| CO Conversion to MeOH (%) | 5.9 |
| CO Conversion to DME (%) | 13.4 |
| Methanol Production (Ton/day) | 3.79 |
| DME Production (Ton/day) | 4.12 |
| Methanol Productivity (gmole/kg-hr) | 10.49 |
| DME Productivity (gmole/kg-hr) | 7.91 |
| H2O Productivity (gmole/kg-hr) | 0.04 |
| MeOH Rate Constant (1-CSTR) | 3.69 |
| DME Rate Constant (1-CSTR) | 19.56 |

**Atomic/Mass Balance (% of reactor inlet)**

| | |
|---|---|
| C | 97.9 |
| H | 94.2 |
| O | 98.3 |
| N | 100.4 |
| Total Mass | 98.0 |

**Liquid Product Analysis (wt%)**

| | Sample# 19:30 | Sample# 13:30 |
|---|---|---|
| Methanol | 87.18 | 87.242 |
| Ethanol | 1.439 | 1.455 |
| 1-Propanol | 0.542 | 0.581 |
| iso-Propanol | 0.042 | 0.042 |
| 1-Butanol | 0.276 | 0.279 |
| 2-Butanol | 0.053 | 0.051 |
| iso-Butanol | 0.111 | 0.113 |
| 2-Methyl-1-Butanol | 0 | 0 |
| 1-Pentanol | 0.126 | 0.129 |
| 2-Methyl-1-Pentanol | 0 | 0 |
| 1-Hexanol | 0.044 | 0.109 |
| 2-Methyl-1-Isobutyrate | 0 | 0 |
| Methyl Acetate | 0.287 | 0.273 |
| Ethyl Acetate | 0 | 0 |
| Methyl Formate | 0.768 | 0.748 |
| DME | 6.418 | 6.098 |
| CO2 | 0 | 0 |
| Water | 0.25 | 0.253 |
| Oil | 0.2 | 0.2 |
| Total | 97.736 | 97.573 |

**RUN NO:** **AF-R17.1p** **TITLE:** **LPDME with Shell Syngas : SV=6000**

| | | FRESH MAKE-UP | RECYCLE | HP H2 MAKEUP | DRY FEED | ALCOHOL INJECT. | REACT FEED | REACT EFFL | 22.10 VAPOR | PURGE 1 PIC-201 | PURGE 2 22.11 | PURGE 3 07.20 | LIQUID PROD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **T** | **F** | 296.0 | 121.0 | 71.3 | 288.2 | 100.0 | 281.0 | 459.6 | 89.5 | 81.9 | 66.5 | 82.5 | 80.0 |
| **P** | **psig** | 814.7 | 812.8 | 795.9 | 781.2 | 781.2 | 781.2 | 750.8 | 742.9 | 703.8 | 29.6 | 120.0 | 1.0 |
| | | | | | | | | | | 8.06 | | | |
| **Comp** | **H2** | 34.96 | 17.27 | 100.00 | 30.09 | 0.00 | 30.09 | 15.72 | 15.58 | 17.27 | 2.84 | 1.47 | 0.00 |
| **(mole%)** | **CO** | 64.74 | 76.70 | 0.00 | 65.99 | 0.00 | 65.99 | 66.18 | 70.08 | 76.70 | 28.42 | 17.24 | 0.00 |
| | **N2** | 0.29 | 1.24 | 0.00 | 0.84 | 0.00 | 0.84 | 1.06 | 1.15 | 1.24 | 0.35 | 0.20 | 0.00 |
| | **CH4** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **CO2** | 0.00 | 4.79 | 0.00 | 3.08 | 0.00 | 3.08 | 8.06 | 8.65 | 4.79 | 32.51 | 43.14 | 0.00 |
| | **DME** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 3.74 | 3.79 | 0.00 | 31.50 | 36.27 | 4.62 |
| | **MeAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.13 |
| | **EtAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **MeFm** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.43 |
| | **MeOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.96 | 0.75 | 0.00 | 4.37 | 1.68 | 92.64 |
| | **H2O** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.08 | 0.00 | 0.00 | 0.00 | 0.00 | 0.47 |
| | **Etoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 | 0.00 | 0.00 | 0.00 | 0.00 | 1.07 |
| | **1-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 | 0.00 | 0.00 | 0.00 | 0.00 | 0.32 |
| | **iso-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **IBOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 |
| | **1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.13 |
| | **2-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **2-Methyl 1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 |
| | **2-Methyl 1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-hexanol** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 |
| | **2-Methyl 1-Isobutyrate** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **others** | 0.00 | 0.00 | 0.00 | 0.00 | 100.00 | 0.00 | 0.12 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **TOTAL** | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| **Mole Wt** | **lb/lb mole** | 18.923 | 24.289 | 2.020 | 20.681 | 59.005 | 20.681 | 26.133 | 26.057 | 24.289 | 38.318 | 41.126 | 33.176 |
| **Flow** | **SCFH** | 30486 | 58673 | 8499 | 97657 | 0 | 97657 | 76873 | 71430 | 6324 | 36 | 7596 | 3541 |
| | **lb mole/hr** | 78.85 | 151.75 | 21.98 | 252.58 | 0.00 | 252.58 | 198.82 | 184.74 | 16.36 | 0.09 | 19.65 | 9.16 |
| | **lb/hr** | 1492.0 | 3685.7 | 44.4 | 5223.6 | 0.0 | 5223.6 | 5195.7 | 4814.0 | 397.3 | 3.6 | 808.0 | 303.9 |

**RUN NO: AF-R17.1q** **TITLE: LPDME with Shell Syngas : SV=6000**

**Balance Period:**

| | | |
|---|---|---|
| Start Date | 10/27/1999 | 17:00 |
| End Date | 10/28/1999 | 9:00 |

**Time From Start of Run (hr)**

| | |
|---|---|
| Start | 357.00 |
| End | 373.00 |

**Reaction Conditions**

| | |
|---|---|
| Temperature (°F) | 481.55 |
| Pressure (psig) | 752.44 |
| Space Velocity (sL/kg-hr) | 5964 |
| Vg (inlet) | 0.56 |

**Slurry Data**

| | |
|---|---|
| Catalyst Weight (lb oxide) | 941 |
| Slurry Level (Reactor Height, ft) | 43.5 |
| Slurry Conc. based on NDG (wt %) | 42.1 |
| Slurry Conc. based on DP (wt %) | 36.7 |
| Gas Holdup based on NDG (vol %) | 54.3 |
| Gas Holdup based on DP (vol %) | 43.3 |

**Performance Results**

| | |
|---|---|
| CO Conversion to H2 (%) | -0.1 |
| CO Conversion to MeOH (%) | 5.6 |
| CO Conversion to DME (%) | 14.2 |
| Methanol Production (Ton/day) | 3.59 |
| DME Production (Ton/day) | 4.37 |
| Methanol Productivity (gmole/kg-hr) | 9.91 |
| DME Productivity (gmole/kg-hr) | 8.40 |
| H2O Productivity (gmole/kg-hr) | 0.04 |
| MeOH Rate Constant (1-CSTR) | 3.83 |
| DME Rate Constant (1-CSTR) | 20.74 |

**Atomic/Mass Balance (% of reactor inlet)**

| | |
|---|---|
| C | 98.3 |
| H | 92.0 |
| O | 99.0 |
| N | 100.0 |
| Total Mass | 98.6 |

**Liquid Product Analysis (wt%)**

| | Sample# 19:30 |
|---|---|
| Methanol | 88.118 |
| Ethanol | 1.488 |
| 1-Propanol | 0.588 |
| iso-Propanol | 0.042 |
| 1-Butanol | 0.3 |
| 2-Butanol | 0.051 |
| iso-Butanol | 0.104 |
| 2-Methyl-1-Butanol | 0 |
| 1-Pentanol | 0.122 |
| 2-Methyl-1-Pentanol | 0 |
| 1-Hexanol | 0.044 |
| 2-Methyl-1-Isobutyrate | 0 |
| Methyl Acetate | 0.275 |
| Ethyl Acetate | 0 |
| Methyl Formate | 0.732 |
| DME | 5.596 |
| CO2 | 0 |
| Water | 0.25 |
| Oil | 0.2 |
| Total | 97.91 |

**RUN NO:** AF-R17.1q **TITLE:** LPDME with Shell Syngas : SV=6000

| | | FRESH MAKE-UP | RECYCLE | HP H2 MAKEUP | DRY FEED | ALCOHOL INJECT. | REACT FEED | REACT EFFL | 22.10 VAPOR | PURGE 1 PIC-201 | PURGE 2 22.11 | PURGE 3 07.20 | LIQUID PROD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| T | F | 298.1 | 123.6 | 74.0 | 288.5 | 100.0 | 281.4 | 459.0 | 92.1 | 84.4 | 71.1 | 85.1 | 80.0 |
| P | psig | 817.0 | 814.4 | 782.0 | 783.2 | 783.2 | 783.2 | 752.4 | 743.5 | 705.8 | 29.9 | 120.0 | 1.0 |
| | | | | | | | | | | 8.17 | | | |
| Comp | H2 | 34.05 | 16.97 | 100.00 | 29.78 | 0.00 | 29.78 | 15.18 | 15.43 | 16.97 | 2.80 | 1.43 | 0.00 |
| (mole%) | CO | 65.73 | 77.54 | 0.00 | 66.64 | 0.00 | 66.64 | 66.84 | 71.00 | 77.54 | 28.41 | 17.23 | 0.00 |
| | N2 | 0.23 | 0.88 | 0.00 | 0.60 | 0.00 | 0.60 | 0.76 | 0.81 | 0.88 | 0.29 | 0.16 | 0.00 |
| | CH4 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | CO2 | 0.00 | 4.61 | 0.00 | 2.98 | 0.00 | 2.98 | 8.17 | 8.70 | 4.61 | 31.43 | 42.04 | 0.00 |
| | DME | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.02 | 3.76 | 0.00 | 31.35 | 36.17 | 4.11 |
| | MeAc | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.13 |
| | EtAc | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | MeFm | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.41 |
| | MeOH | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.75 | 0.30 | 0.00 | 5.71 | 2.98 | 93.14 |
| | H2O | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.08 | 0.00 | 0.00 | 0.00 | 0.00 | 0.47 |
| | Etoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 | 0.00 | 0.00 | 0.00 | 0.00 | 1.09 |
| | 1-Proh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 | 0.00 | 0.00 | 0.00 | 0.00 | 0.33 |
| | iso-Proh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | IBOH | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 |
| | 1-Buoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.14 |
| | 2-Buoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | 2-Methyl 1-Buoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | 1-Peoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 |
| | 2-Methyl 1-Peoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | 1-hexanol | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.01 |
| | 2-Methyl 1-Isobutyrate | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | others | 0.00 | 0.00 | 0.00 | 0.00 | 100.00 | 0.00 | 0.12 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | TOTAL | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| Mole Wt | lb/lb mole | 19.161 | 24.337 | 2.020 | 20.748 | 58.629 | 20.748 | 26.330 | 26.079 | 24.337 | 38.182 | 40.995 | 33.100 |
| Flow | SCFH | 30105 | 58207 | 8503 | 96815 | 0 | 96815 | 75952 | 71626 | 6512 | 3 | 7700 | 3371 |
| | lb mole/hr | 77.86 | 150.54 | 21.99 | 250.40 | 0.00 | 250.40 | 196.44 | 185.25 | 16.84 | 0.01 | 19.91 | 8.72 |
| | lb/hr | 1491.9 | 3663.7 | 44.4 | 5195.3 | 0.0 | 5195.3 | 5172.2 | 4831.1 | 409.9 | 0.3 | 816.4 | 288.6 |

**RUN NO: AF-R17.1r** **TITLE: LPDME with Shell Syngas : SV=6000**

**Balance Period:**

| | | |
|---|---|---|
| Start Date | 10/28/1999 | 9:00 |
| End Date | 10/28/1999 | 21:00 |

**Reaction Conditions**

| | |
|---|---|
| Temperature (°F) | 481.50 |
| Pressure (psig) | 751.25 |
| Space Velocity (sL/kg-hr) | 5949 |
| Vg (inlet) | 0.56 |

**Performance Results**

| | |
|---|---|
| CO Conversion to H2 (%) | -0.1 |
| CO Conversion to MeOH (%) | 5.6 |
| CO Conversion to DME (%) | 14.3 |
| Methanol Production (Ton/day) | 3.58 |
| DME Production (Ton/day) | 4.37 |
| Methanol Productivity (gmole/kg-hr) | 9.90 |
| DME Productivity (gmole/kg-hr) | 8.41 |
| H2O Productivity (gmole/kg-hr) | 0.04 |
| MeOH Rate Constant (1-CSTR) | 3.61 |
| DME Rate Constant (1-CSTR) | 20.84 |

**Liquid Product Analysis (wt%)**

| | Sample# 15:30 | Sample# 19:30 |
|---|---|---|
| Methanol | 87.182 | 87.634 |
| Ethanol | 1.449 | 1.485 |
| 1-Propanol | 0.596 | 0.646 |
| iso-Propanol | 0.04 | 0.037 |
| 1-Butanol | 0.275 | 0.282 |
| 2-Butanol | 0.05 | 0.06 |
| iso-Butanol | 0.084 | 0.111 |
| 2-Methyl-1-Butanol | 0 | 0 |
| 1-Pentanol | 0.124 | 0.14 |
| 2-Methyl-1-Pentanol | 0 | 0 |
| 1-Hexanol | 0.115 | 0.043 |
| 2-Methyl-1-Isobutyrate | 0 | 0 |
| Methyl Acetate | 0.259 | 0.27 |
| Ethyl Acetate | 0 | 0 |
| Methyl Formate | 0.752 | 0.752 |
| DME | 6.22 | 5.949 |
| CO2 | 0 | 0 |
| Water | 0.243 | 0.241 |
| Oil | 0.2 | 0.2 |
| Total | 97.589 | 97.85 |

**Time From Start of Run (hr)**

| | |
|---|---|
| Start | 373.00 |
| End | 385.00 |

**Slurry Data**

| | |
|---|---|
| Catalyst Weight (lb oxide) | 941 |
| Slurry Level (Reactor Height, ft) | 43.5 |
| Slurry Conc. based on NDG (wt %) | 42.0 |
| Slurry Conc. based on DP (wt %) | 36.7 |
| Gas Holdup based on NDG (vol %) | 54.2 |
| Gas Holdup based on DP (vol %) | 43.2 |

**Atomic/Mass Balance (% of reactor inlet)**

| | |
|---|---|
| C | 100.5 |
| H | 93.5 |
| O | 101.1 |
| N | 100.9 |
| Total Mass | 100.6 |

**RUN NO:** **AF-R17.1r** **TITLE:** **LPDME with Shell Syngas : SV=6000**

| | | FRESH MAKE-UP | RECYCLE | HP H2 MAKEUP | DRY FEED | ALCOHOL INJECT. | REACT FEED | REACT EFFL | 22.10 VAPOR | PURGE 1 PIC-201 | PURGE 2 22.11 | PURGE 3 07.20 | LIQUID PROD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **T** | **F** | 299.6 | 125.2 | 84.1 | 287.0 | 100.0 | 279.5 | 458.7 | 92.5 | 86.5 | 76.8 | 85.9 | 80.0 |
| **P** | **psig** | 817.3 | 813.1 | 812.3 | 782.2 | 782.2 | 782.2 | 751.2 | 742.4 | 705.9 | 29.8 | 120.0 | 1.0 |
| | | | | | | | | | | 8.11 | | | |
| **Comp** | **H2** | 32.76 | 17.33 | 100.00 | 30.24 | 0.00 | 30.24 | 15.68 | 15.63 | 17.33 | 2.85 | 1.45 | 0.00 |
| **(mole%)** | **CO** | 67.00 | 77.28 | 0.00 | 66.27 | 0.00 | 66.27 | 66.36 | 70.66 | 77.28 | 28.23 | 17.10 | 0.00 |
| | **N2** | 0.23 | 0.89 | 0.00 | 0.60 | 0.00 | 0.60 | 0.77 | 0.81 | 0.89 | 0.29 | 0.16 | 0.00 |
| | **CH4** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **CO2** | 0.00 | 4.50 | 0.00 | 2.89 | 0.00 | 2.89 | 8.11 | 8.66 | 4.50 | 31.02 | 41.97 | 0.00 |
| | **DME** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.05 | 3.88 | 0.00 | 31.70 | 36.29 | 4.49 |
| | **MeAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.12 |
| | **EtAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **MeFm** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.43 |
| | **MeOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.77 | 0.36 | 0.00 | 5.91 | 3.04 | 92.75 |
| | **H2O** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.08 | 0.00 | 0.00 | 0.00 | 0.00 | 0.46 |
| | **Etoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 | 0.00 | 0.00 | 0.00 | 0.00 | 1.08 |
| | **1-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 | 0.00 | 0.00 | 0.00 | 0.00 | 0.35 |
| | **iso-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **IBOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.04 |
| | **1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.13 |
| | **2-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 |
| | **2-Methyl 1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 |
| | **2-Methyl 1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-hexanol** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 |
| | **2-Methyl 1-Isobutyrate** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **others** | 0.00 | 0.00 | 0.00 | 0.00 | 100.00 | 0.00 | 0.12 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **TOTAL** | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| **Mole Wt** | **lb/lb mole** | 19.496 | 24.224 | 2.020 | 20.611 | 58.894 | 20.611 | 26.197 | 26.045 | 24.224 | 38.178 | 41.002 | 33.166 |
| **Flow** | **SCFH** | 30093 | 57982 | 8501 | 96576 | 0 | 96576 | 75609 | 72556 | 6469 | 3 | 7776 | 3387 |
| | **lb mole/hr** | 77.83 | 149.96 | 21.99 | 249.78 | 0.00 | 249.78 | 195.55 | 187.66 | 16.73 | 0.01 | 20.11 | 8.76 |
| | **lb/hr** | 1517.4 | 3632.7 | 44.4 | 5148.3 | 0.0 | 5148.3 | 5122.9 | 4887.5 | 405.3 | 0.3 | 824.6 | 290.6 |

**RUN NO: AF-R17.1s** **TITLE: LPDME with Shell Syngas : SV=6000**

**Balance Period:**

| | | |
|---|---|---|
| Start Date | 10/28/1999 | 21:00 |
| End Date | 10/29/1999 | 9:00 |

**Time From Start of Run (hr)**

| | |
|---|---|
| Start | 385.00 |
| End | 397.00 |

**Reaction Conditions**

| | |
|---|---|
| Temperature (°F) | 481.43 |
| Pressure (psig) | 752.99 |
| Space Velocity (sL/kg-hr) | 6027 |
| Vg (inlet) | 0.57 |

**Slurry Data**

| | |
|---|---|
| Catalyst Weight (lb oxide) | 941 |
| Slurry Level (Reactor Height, ft) | 43.5 |
| Slurry Conc. based on NDG (wt %) | 41.9 |
| Slurry Conc. based on DP (wt %) | 36.9 |
| Gas Holdup based on NDG (vol %) | 54.9 |
| Gas Holdup based on DP (vol %) | 43.7 |

**Performance Results**

| | |
|---|---|
| CO Conversion to H2 (%) | -0.1 |
| CO Conversion to MeOH (%) | 5.4 |
| CO Conversion to DME (%) | 13.7 |
| Methanol Production (Ton/day) | 3.52 |
| DME Production (Ton/day) | 4.29 |
| Methanol Productivity (gmole/kg-hr) | 9.73 |
| DME Productivity (gmole/kg-hr) | 8.25 |
| H2O Productivity (gmole/kg-hr) | 0.04 |
| MeOH Rate Constant (1-CSTR) | 3.56 |
| DME Rate Constant (1-CSTR) | 20.19 |

**Atomic/Mass Balance (% of reactor inlet)**

| | |
|---|---|
| C | 100.6 |
| H | 95.3 |
| O | 101.2 |
| N | 100.4 |
| Total Mass | 100.8 |

**Liquid Product Analysis (wt%)**

| | Sample# 19:30 |
|---|---|
| Methanol | 87.634 |
| Ethanol | 1.485 |
| 1-Propanol | 0.646 |
| iso-Propanol | 0.037 |
| 1-Butanol | 0.282 |
| 2-Butanol | 0.06 |
| iso-Butanol | 0.111 |
| 2-Methyl-1-Butanol | 0 |
| 1-Pentanol | 0.14 |
| 2-Methyl-1-Pentanol | 0 |
| 1-Hexanol | 0.043 |
| 2-Methyl-1-Isobutyrate | 0 |
| Methyl Acetate | 0.27 |
| Ethyl Acetate | 0 |
| Methyl Formate | 0.752 |
| DME | 5.949 |
| CO2 | 0 |
| Water | 0.241 |
| Oil | 0.2 |
| Total | 97.85 |

**RUN NO:** **AF-R17.1s** **TITLE:** **LPDME with Shell Syngas : SV=6000**

| | | FRESH MAKE-UP | RECYCLE | HP H2 MAKEUP | DRY FEED | ALCOHOL INJECT. | REACT FEED | REACT EFFL | 22.10 VAPOR | PURGE 1 PIC-201 | PURGE 2 22.11 | PURGE 3 07.20 | LIQUID PROD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **T** | **F** | 290.1 | 116.1 | 70.2 | 281.3 | 100.0 | 274.5 | 459.6 | 82.6 | 76.2 | 69.7 | 75.3 | 80.0 |
| **P** | **psig** | 817.3 | 815.8 | 787.6 | 783.9 | 783.9 | 783.9 | 753.0 | 743.2 | 705.3 | 30.0 | 120.0 | 1.0 |
| | | | | | | | | | | 7.96 | | | |
| **Comp** | **H2** | 33.70 | 16.86 | 100.00 | 29.48 | 0.00 | 29.48 | 15.28 | 15.33 | 16.86 | 2.80 | 1.44 | 0.00 |
| **(mole%)** | **CO** | 66.06 | 77.76 | 0.00 | 66.98 | 0.00 | 66.98 | 67.26 | 71.48 | 77.76 | 29.36 | 17.70 | 0.00 |
| | **N2** | 0.23 | 0.88 | 0.00 | 0.61 | 0.00 | 0.61 | 0.77 | 0.81 | 0.88 | 0.30 | 0.15 | 0.00 |
| | **CH4** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **CO2** | 0.00 | 4.50 | 0.00 | 2.94 | 0.00 | 2.94 | 7.96 | 8.49 | 4.50 | 32.64 | 42.47 | 0.00 |
| | **DME** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 3.88 | 3.61 | 0.00 | 30.25 | 36.02 | 4.38 |
| | **MeAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.12 |
| | **EtAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **MeFm** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.42 |
| | **MeOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.58 | 0.28 | 0.00 | 4.63 | 2.21 | 92.84 |
| | **H2O** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.08 | 0.00 | 0.00 | 0.00 | 0.00 | 0.45 |
| | **Etoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 | 0.00 | 0.00 | 0.00 | 0.00 | 1.09 |
| | **1-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 | 0.00 | 0.00 | 0.00 | 0.00 | 0.36 |
| | **iso-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **IBOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 |
| | **1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.13 |
| | **2-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 |
| | **2-Methyl 1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 |
| | **2-Methyl 1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-hexanol** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.01 |
| | **2-Methyl 1-Isobutyrate** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **others** | 0.00 | 0.00 | 0.00 | 0.00 | 100.00 | 0.00 | 0.11 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **TOTAL** | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| **Mole Wt** | **lb/lb mole** | 19.250 | 24.349 | 2.020 | 20.819 | 58.727 | 20.819 | 26.236 | 26.044 | 24.349 | 38.137 | 41.003 | 33.155 |
| **Flow** | **SCFH** | 29985 | 59360 | 8493 | 97837 | 0 | 97837 | 77282 | 74077 | 6448 | 3 | 7403 | 3709 |
| | **lb mole/hr** | 77.55 | 153.53 | 21.96 | 253.04 | 0.00 | 253.04 | 199.88 | 191.59 | 16.68 | 0.01 | 19.15 | 9.59 |
| | **lb/hr** | 1492.9 | 3738.1 | 44.4 | 5268.2 | 0.0 | 5268.2 | 5244.0 | 4989.9 | 406.0 | 0.3 | 785.1 | 318.1 |

**RUN NO: AF-R17.1t** **TITLE: LPDME with Shell Syngas : SV=6000**

**Balance Period:**

| | | |
|---|---|---|
| Start Date | 10/29/1999 | 14:00 |
| End Date | 10/30/1999 | 8:00 |

**Time From Start of Run (hr)**

| | |
|---|---|
| Start | 402.00 |
| End | 420.00 |

**Reaction Conditions**

| | |
|---|---|
| Temperature (°F) | 481.35 |
| Pressure (psig) | 750.88 |
| Space Velocity (sL/kg-hr) | 5991 |
| Vg (inlet) | 0.57 |

**Slurry Data**

| | |
|---|---|
| Catalyst Weight (lb oxide) | 941 |
| Slurry Level (Reactor Height, ft) | 43.5 |
| Slurry Conc. based on NDG (wt %) | 42.1 |
| Slurry Conc. based on DP (wt %) | 36.3 |
| Gas Holdup based on NDG (vol %) | 54.5 |
| Gas Holdup based on DP (vol %) | 42.4 |

**Performance Results**

| | |
|---|---|
| CO Conversion to H2 (%) | -0.1 |
| CO Conversion to MeOH (%) | 5.4 |
| CO Conversion to DME (%) | 14.0 |
| Methanol Production (Ton/day) | 3.49 |
| DME Production (Ton/day) | 4.31 |
| Methanol Productivity (gmole/kg-hr) | 9.65 |
| DME Productivity (gmole/kg-hr) | 8.28 |
| H2O Productivity (gmole/kg-hr) | 0.04 |
| MeOH Rate Constant (1-CSTR) | 3.43 |
| DME Rate Constant (1-CSTR) | 20.43 |

**Atomic/Mass Balance (% of reactor inlet)**

| | |
|---|---|
| C | 99.1 |
| H | 95.5 |
| O | 99.9 |
| N | 101.6 |
| Total Mass | 99.5 |

**Liquid Product Analysis (wt%)**

| | Sample# 16:00 | Sample# 4:00 |
|---|---|---|
| Methanol | 87.093 | 87.349 |
| Ethanol | 1.477 | 1.538 |
| 1-Propanol | 0.557 | 0.564 |
| iso-Propanol | 0.049 | 0.049 |
| 1-Butanol | 0.277 | 0.27 |
| 2-Butanol | 0.05 | 0.051 |
| iso-Butanol | 0.105 | 0.11 |
| 2-Methyl-1-Butanol | 0 | 0 |
| 1-Pentanol | 0.11 | 0.128 |
| 2-Methyl-1-Pentanol | 0 | 0 |
| 1-Hexanol | 0.047 | 0.05 |
| 2-Methyl-1-Isobutyrate | 0 | 0 |
| Methyl Acetate | 0.268 | 0.273 |
| Ethyl Acetate | 0 | 0 |
| Methyl Formate | 0.836 | 0.813 |
| DME | 6.479 | 6.126 |
| CO2 | 0 | 0 |
| Water | 0.225 | 0.227 |
| Oil | 0.2 | 0.2 |
| Total | 97.773 | 97.748 |

**RUN NO:** **AF-R17.1t** **TITLE:** **LPDME with Shell Syngas : SV=6000**

| | | FRESH MAKE-UP | RECYCLE | HP H2 MAKEUP | DRY FEED | ALCOHOL INJECT. | REACT FEED | REACT EFFL | 22.10 VAPOR | PURGE 1 PIC-201 | PURGE 2 22.11 | PURGE 3 07.20 | LIQUID PROD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **T** | **F** | 292.3 | 117.4 | 75.6 | 277.6 | 100.0 | 270.4 | 456.8 | 83.7 | 77.9 | 75.4 | 76.8 | 80.0 |
| **P** | **psig** | 815.3 | 813.0 | 792.6 | 781.9 | 781.9 | 781.9 | 750.9 | 739.3 | 702.8 | 29.9 | 120.0 | 1.0 |
| | | | | | | | | | | 7.85 | | | |
| **Comp** | **H2** | 34.02 | 17.44 | 100.00 | 29.82 | 0.00 | 29.82 | 15.63 | 15.94 | 17.44 | 2.79 | 1.47 | 0.00 |
| **(mole%)** | **CO** | 65.70 | 59.45 | 0.00 | 66.59 | 0.00 | 66.59 | 66.77 | 70.38 | 59.45 | 28.58 | 17.63 | 0.00 |
| | **N2** | 0.28 | 18.76 | 0.00 | 0.77 | 0.00 | 0.77 | 0.98 | 1.05 | 18.76 | 0.32 | 0.19 | 0.00 |
| | **CH4** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **CO2** | 0.00 | 4.36 | 0.00 | 2.82 | 0.00 | 2.82 | 7.85 | 8.53 | 4.36 | 32.29 | 42.70 | 0.00 |
| | **DME** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 3.93 | 3.71 | 0.00 | 31.06 | 35.75 | 4.65 |
| | **MeAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.12 |
| | **EtAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **MeFm** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.47 |
| | **MeOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.58 | 0.39 | 0.00 | 4.95 | 2.26 | 92.59 |
| | **H2O** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.08 | 0.00 | 0.00 | 0.00 | 0.00 | 0.43 |
| | **Etoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 | 0.00 | 0.00 | 0.00 | 0.00 | 1.11 |
| | **1-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 | 0.00 | 0.00 | 0.00 | 0.00 | 0.32 |
| | **iso-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 |
| | **IBOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 |
| | **1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.13 |
| | **2-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **2-Methyl 1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 |
| | **2-Methyl 1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-hexanol** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **2-Methyl 1-Isobutyrate** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **others** | 0.00 | 0.00 | 0.00 | 0.00 | 100.00 | 0.00 | 0.12 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **TOTAL** | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| **Mole Wt** | **lb/lb mole** | 19.168 | 24.176 | 2.020 | 20.711 | 58.504 | 20.711 | 26.137 | 25.915 | 24.176 | 38.240 | 40.986 | 33.192 |
| **Flow** | **SCFH** | 29448 | 59308 | 8502 | 97259 | 0 | 97259 | 76696 | 72535 | 6336 | 170 | 7620 | 3543 |
| | **lb mole/hr** | 76.16 | 153.39 | 21.99 | 251.54 | 0.00 | 251.54 | 198.36 | 187.60 | 16.39 | 0.44 | 19.71 | 9.16 |
| | **lb/hr** | 1459.9 | 3708.5 | 44.4 | 5209.8 | 0.0 | 5209.8 | 5184.5 | 4861.6 | 396.2 | 16.8 | 807.8 | 304.2 |

**RUN NO: AF-R17.1u** **TITLE: LPDME with Shell Syngas : SV=6000**

**Balance Period:**

| | | |
|---|---|---|
| Start Date | 10/30/1999 | 8:00 |
| End Date | 10/31/1999 | 2:00 |

**Time From Start of Run (hr)**

| | |
|---|---|
| Start | 420.00 |
| End | 438.00 |

**Reaction Conditions**

| | |
|---|---|
| Temperature (°F) | 481.29 |
| Pressure (psig) | 751.59 |
| Space Velocity (sL/kg-hr) | 6014 |
| Vg (inlet) | 0.57 |

**Slurry Data**

| | |
|---|---|
| Catalyst Weight (lb oxide) | 941 |
| Slurry Level (Reactor Height, ft) | 43.5 |
| Slurry Conc. based on NDG (wt %) | 42.3 |
| Slurry Conc. based on DP (wt %) | 36.4 |
| Gas Holdup based on NDG (vol %) | 54.7 |
| Gas Holdup based on DP (vol %) | 42.6 |

**Performance Results**

| | |
|---|---|
| CO Conversion to H2 (%) | -0.1 |
| CO Conversion to MeOH (%) | 5.5 |
| CO Conversion to DME (%) | 14.0 |
| Methanol Production (Ton/day) | 3.57 |
| DME Production (Ton/day) | 4.33 |
| Methanol Productivity (gmole/kg-hr) | 9.87 |
| DME Productivity (gmole/kg-hr) | 8.32 |
| H2O Productivity (gmole/kg-hr) | 0.04 |
| MeOH Rate Constant (1-CSTR) | 3.58 |
| DME Rate Constant (1-CSTR) | 20.92 |

**Atomic/Mass Balance (% of reactor inlet)**

| | |
|---|---|
| C | 97.7 |
| H | 99.7 |
| O | 98.8 |
| N | 100.4 |
| Total Mass | 98.4 |

**Liquid Product Analysis (wt%)**

| | Sample# 10:25 | Sample# 22:30 |
|---|---|---|
| Methanol | 86.879 | 86.137 |
| Ethanol | 1.456 | 1.409 |
| 1-Propanol | 0.571 | 0.534 |
| iso-Propanol | 0.045 | 0.044 |
| 1-Butanol | 0.273 | 0.255 |
| 2-Butanol | 0.052 | 0.047 |
| iso-Butanol | 0.099 | 0.101 |
| 2-Methyl-1-Butanol | 0 | 0 |
| 1-Pentanol | 0.099 | 0.118 |
| 2-Methyl-1-Pentanol | 0 | 0 |
| 1-Hexanol | 0.045 | 0.146 |
| 2-Methyl-1-Isobutyrate | 0 | 0 |
| Methyl Acetate | 0.26 | 0.272 |
| Ethyl Acetate | 0 | 0 |
| Methyl Formate | 0.827 | 0.787 |
| DME | 6.683 | 7.443 |
| CO2 | 0 | 0 |
| Water | 0.263 | 0 |
| Oil | 0.2 | 0.2 |
| Total | 97.752 | 97.493 |

**RUN NO:** AF-R17.1u **TITLE:** LPDME with Shell Syngas : SV=6000

| | | FRESH MAKE-UP | RECYCLE | HP H2 MAKEUP | DRY FEED | ALCOHOL INJECT. | REACT FEED | REACT EFFL | 22.10 VAPOR | PURGE 1 PIC-201 | PURGE 2 22.11 | PURGE 3 07.20 | LIQUID PROD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| T | F | 287.8 | 115.7 | 74.2 | 272.8 | 100.0 | 264.5 | 457.0 | 81.5 | 76.1 | 73.2 | 74.4 | 80.0 |
| P | psig | 815.5 | 813.8 | 776.6 | 782.2 | 782.2 | 782.2 | 751.6 | 739.9 | 703.2 | 29.8 | 120.0 | 1.0 |
| | | | | | | | | | | 7.90 | | | |
| Comp | H2 | 34.17 | 17.46 | 100.00 | 29.92 | 0.00 | 29.92 | 15.53 | 17.23 | 17.46 | 2.84 | 1.50 | 0.00 |
| (mole%) | CO | 65.51 | 71.32 | 0.00 | 66.41 | 0.00 | 66.41 | 66.56 | 68.19 | 71.32 | 28.88 | 17.78 | 0.00 |
| | N2 | 0.32 | 6.93 | 0.00 | 0.88 | 0.00 | 0.88 | 1.12 | 1.19 | 6.93 | 0.36 | 0.22 | 0.00 |
| | CH4 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | CO2 | 0.00 | 4.29 | 0.00 | 2.80 | 0.00 | 2.80 | 7.90 | 9.06 | 4.29 | 32.44 | 42.58 | 0.00 |
| | DME | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 3.94 | 3.95 | 0.00 | 30.81 | 35.82 | 5.24 |
| | MeAc | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.12 |
| | EtAc | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | MeFm | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.46 |
| | MeOH | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.68 | 0.38 | 0.00 | 4.68 | 2.11 | 92.24 |
| | H2O | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.08 | 0.00 | 0.00 | 0.00 | 0.00 | 0.25 |
| | Etoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 | 0.00 | 0.00 | 0.00 | 0.00 | 1.06 |
| | 1-Proh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 | 0.00 | 0.00 | 0.00 | 0.00 | 0.31 |
| | iso-Proh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 |
| | IBOH | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 |
| | 1-Buoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.12 |
| | 2-Buoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | 2-Methyl 1-Buoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | 1-Peoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.04 |
| | 2-Methyl 1-Peoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | 1-hexanol | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 |
| | 2-Methyl 1-Isobutyrate | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | others | 0.00 | 0.00 | 0.00 | 0.00 | 100.00 | 0.00 | 0.12 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | TOTAL | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| Mole Wt | lb/lb mole | 19.129 | 24.159 | 2.020 | 20.682 | 58.974 | 20.682 | 26.177 | 25.707 | 24.159 | 38.194 | 40.965 | 33.293 |
| Flow | SCFH | 29532 | 59588 | 8506 | 97626 | 0 | 97626 | 76714 | 72500 | 6315 | 176 | 7517 | 3511 |
| | lb mole/hr | 76.38 | 154.12 | 22.00 | 252.50 | 0.00 | 252.50 | 198.41 | 187.51 | 16.33 | 0.45 | 19.44 | 9.08 |
| | lb/hr | 1461.1 | 3723.3 | 44.4 | 5222.2 | 0.0 | 5222.2 | 5193.8 | 4820.4 | 394.6 | 17.4 | 796.4 | 302.3 |

**RUN NO: AF-R17.1v** **TITLE: LPDME with Shell Syngas : SV=6000**

**Balance Period:**

| | | |
|---|---|---|
| Start Date | 10/31/1999 | 2:00 |
| End Date | 10/31/1999 | 20:00 |

**Time From Start of Run (hr)**

| | |
|---|---|
| Start | 438.00 |
| End | 456.00 |

**Reaction Conditions**

| | |
|---|---|
| Temperature (°F) | 481.34 |
| Pressure (psig) | 752.47 |
| Space Velocity (sL/kg-hr) | 5947 |
| Vg (inlet) | 0.56 |

**Slurry Data**

| | |
|---|---|
| Catalyst Weight (lb oxide) | 941 |
| Slurry Level (Reactor Height, ft) | 43.5 |
| Slurry Conc. based on NDG (wt %) | 42.3 |
| Slurry Conc. based on DP (wt %) | 36.5 |
| Gas Holdup based on NDG (vol %) | 54.7 |
| Gas Holdup based on DP (vol %) | 42.8 |

**Performance Results**

| | |
|---|---|
| CO Conversion to H2 (%) | -0.1 |
| CO Conversion to MeOH (%) | 5.4 |
| CO Conversion to DME (%) | 14.1 |
| Methanol Production (Ton/day) | 3.45 |
| DME Production (Ton/day) | 4.30 |
| Methanol Productivity (gmole/kg-hr) | 9.55 |
| DME Productivity (gmole/kg-hr) | 8.27 |
| H2O Productivity (gmole/kg-hr) | 0.04 |
| MeOH Rate Constant (1-CSTR) | 3.32 |
| DME Rate Constant (1-CSTR) | 20.24 |

**Atomic/Mass Balance (% of reactor inlet)**

| | |
|---|---|
| C | 100.2 |
| H | 94.5 |
| O | 100.8 |
| N | 101.8 |
| Total Mass | 100.4 |

**Liquid Product Analysis (wt%)**

| | Sample# 9:45 |
|---|---|
| Methanol | 87.33 |
| Ethanol | 1.447 |
| 1-Propanol | 0.554 |
| iso-Propanol | 0.044 |
| 1-Butanol | 0.268 |
| 2-Butanol | 0.055 |
| iso-Butanol | 0.104 |
| 2-Methyl-1-Butanol | 0 |
| 1-Pentanol | 0.118 |
| 2-Methyl-1-Pentanol | 0 |
| 1-Hexanol | 0.059 |
| 2-Methyl-1-Isobutyrate | 0 |
| Methyl Acetate | 0.267 |
| Ethyl Acetate | 0 |
| Methyl Formate | 0.836 |
| DME | 6.284 |
| CO2 | 0 |
| Water | 0.217 |
| Oil | 0.2 |
| Total | 97.783 |

**RUN NO:** **AF-R17.1v** **TITLE:** **LPDME with Shell Syngas : SV=6000**

| | | FRESH MAKE-UP | RECYCLE | HP H2 MAKEUP | DRY FEED | ALCOHOL INJECT. | REACT FEED | REACT EFFL | 22.10 VAPOR | PURGE 1 PIC-201 | PURGE 2 22.11 | PURGE 3 07.20 | LIQUID PROD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **T** | **F** | 286.2 | 112.0 | 69.2 | 272.8 | 100.0 | 264.7 | 458.1 | 77.9 | 72.3 | 66.2 | 71.4 | 80.0 |
| **P** | **psig** | 814.6 | 812.8 | 797.7 | 782.1 | 782.1 | 782.1 | 752.5 | 743.5 | 705.1 | 29.5 | 120.0 | 1.0 |
| | | | | | | | | | | 7.80 | | | |
| **Comp** | **H2** | 34.19 | 17.42 | 100.00 | 29.99 | 0.00 | 29.99 | 15.83 | 15.75 | 17.42 | 3.00 | 1.51 | 0.00 |
| **(mole%)** | **CO** | 65.49 | 77.08 | 0.00 | 66.38 | 0.00 | 66.38 | 66.48 | 70.99 | 77.08 | 26.56 | 17.99 | 0.00 |
| | **N2** | 0.32 | 1.28 | 0.00 | 0.87 | 0.00 | 0.87 | 1.11 | 1.16 | 1.28 | 9.05 | 0.22 | 0.00 |
| | **CH4** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **CO2** | 0.00 | 4.22 | 0.00 | 2.76 | 0.00 | 2.76 | 7.80 | 8.25 | 4.22 | 30.18 | 42.83 | 0.00 |
| | **DME** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 3.96 | 3.59 | 0.00 | 27.34 | 35.62 | 4.64 |
| | **MeAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.12 |
| | **EtAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **MeFm** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.47 |
| | **MeOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.56 | 0.25 | 0.00 | 3.87 | 1.84 | 92.67 |
| | **H2O** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.08 | 0.00 | 0.00 | 0.00 | 0.00 | 0.41 |
| | **Etoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 | 0.00 | 0.00 | 0.00 | 0.00 | 1.07 |
| | **1-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 | 0.00 | 0.00 | 0.00 | 0.00 | 0.31 |
| | **iso-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **IBOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 |
| | **1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.12 |
| | **2-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 |
| | **2-Methyl 1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 |
| | **2-Methyl 1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-hexanol** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **2-Methyl 1-Isobutyrate** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **others** | 0.00 | 0.00 | 0.00 | 0.00 | 100.00 | 0.00 | 0.11 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **TOTAL** | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| **Mole Wt** | **lb/lb mole** | 19.125 | 24.158 | 2.020 | 20.656 | 58.783 | 20.656 | 26.082 | 25.892 | 24.158 | 37.134 | 40.954 | 33.187 |
| **Flow** | **SCFH** | 29469 | 58571 | 8504 | 96544 | 0 | 96544 | 76092 | 72293 | 6459 | 186 | 7403 | 3692 |
| | **lb mole/hr** | 76.22 | 151.48 | 21.99 | 249.70 | 0.00 | 249.70 | 196.80 | 186.97 | 16.70 | 0.48 | 19.15 | 9.55 |
| | **lb/hr** | 1457.7 | 3659.6 | 44.4 | 5157.8 | 0.0 | 5157.8 | 5132.9 | 4841.2 | 403.6 | 17.9 | 784.1 | 316.9 |

**RUN NO: AF-R17.1w** **TITLE: LPDME with Shell Syngas : SV=6000**

**Balance Period:**

| | | |
|---|---|---|
| Start Date | 10/31/1999 | 23:00 |
| End Date | 11/1/1999 | 14:00 |

**Time From Start of Run (hr)**

| | |
|---|---|
| Start | 459.00 |
| End | 474.00 |

**Reaction Conditions**

| | |
|---|---|
| Temperature (°F) | 481.47 |
| Pressure (psig) | 752.23 |
| Space Velocity (sL/kg-hr) | 5876 |
| Vg (inlet) | 0.56 |

**Slurry Data**

| | |
|---|---|
| Catalyst Weight (lb oxide) | 941 |
| Slurry Level (Reactor Height, ft) | 43.5 |
| Slurry Conc. based on NDG (wt %) | 42.1 |
| Slurry Conc. based on DP (wt %) | 36.5 |
| Gas Holdup based on NDG (vol %) | 54.4 |
| Gas Holdup based on DP (vol %) | 42.7 |

**Performance Results**

| | |
|---|---|
| CO Conversion to H2 (%) | -0.1 |
| CO Conversion to MeOH (%) | 5.5 |
| CO Conversion to DME (%) | 14.3 |
| Methanol Production (Ton/day) | 3.41 |
| DME Production (Ton/day) | 4.29 |
| Methanol Productivity (gmole/kg-hr) | 9.42 |
| DME Productivity (gmole/kg-hr) | 8.25 |
| H2O Productivity (gmole/kg-hr) | 0.04 |
| MeOH Rate Constant (1-CSTR) | 3.23 |
| DME Rate Constant (1-CSTR) | 20.52 |

**Atomic/Mass Balance (% of reactor inlet)**

| | |
|---|---|
| C | 99.3 |
| H | 93.1 |
| O | 99.9 |
| N | 100.4 |
| Total Mass | 99.4 |

**Liquid Product Analysis (wt%)**

| | Sample# 8:00 |
|---|---|
| Methanol | 87.303 |
| Ethanol | 1.374 |
| 1-Propanol | 0.525 |
| iso-Propanol | 0.046 |
| 1-Butanol | 0.255 |
| 2-Butanol | 0.048 |
| iso-Butanol | 0.1 |
| 2-Methyl-1-Butanol | 0 |
| 1-Pentanol | 0.113 |
| 2-Methyl-1-Pentanol | 0 |
| 1-Hexanol | 0.047 |
| 2-Methyl-1-Isobutyrate | 0 |
| Methyl Acetate | 0.254 |
| Ethyl Acetate | 0 |
| Methyl Formate | 0.835 |
| DME | 6.601 |
| CO2 | 0 |
| Water | 0.225 |
| Oil | 0.2 |
| Total | 97.926 |

**RUN NO:** AF-R17.1w **TITLE:** LPDME with Shell Syngas : SV=6000

| | | FRESH MAKE-UP | RECYCLE | HP H2 MAKEUP | DRY FEED | ALCOHOL INJECT. | REACT FEED | REACT EFFL | 22.10 VAPOR | PURGE 1 PIC-201 | PURGE 2 22.11 | PURGE 3 07.20 | LIQUID PROD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| T | F | 291.1 | 117.3 | 69.2 | 280.1 | 100.0 | 272.0 | 458.1 | 85.8 | 78.4 | 64.3 | 79.6 | 80.0 |
| P | psig | 813.2 | 811.4 | 787.3 | 781.3 | 781.3 | 781.3 | 752.2 | 744.6 | 705.9 | 29.4 | 120.0 | 1.0 |
| | | | | | | | | | | 8.10 | | | |
| Comp | H2 | 33.79 | 17.57 | 100.00 | 30.28 | 0.00 | 30.28 | 16.10 | 15.87 | 17.57 | 2.90 | 1.49 | 0.00 |
| (mole%) | CO | 65.89 | 76.61 | 0.00 | 65.93 | 0.00 | 65.93 | 65.90 | 70.58 | 76.61 | 28.49 | 17.40 | 0.00 |
| | N2 | 0.32 | 1.25 | 0.00 | 0.85 | 0.00 | 0.85 | 1.08 | 1.15 | 1.25 | 0.36 | 0.21 | 0.00 |
| | CH4 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | CO2 | 0.00 | 4.57 | 0.00 | 2.95 | 0.00 | 2.95 | 8.10 | 8.51 | 4.57 | 32.66 | 42.24 | 0.00 |
| | DME | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.00 | 3.65 | 0.00 | 30.54 | 36.25 | 4.86 |
| | MeAc | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.12 |
| | EtAc | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | MeFm | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.47 |
| | MeOH | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.57 | 0.23 | 0.00 | 5.04 | 2.42 | 92.52 |
| | H2O | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.08 | 0.00 | 0.00 | 0.00 | 0.00 | 0.42 |
| | Etoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 | 0.00 | 0.00 | 0.00 | 0.00 | 1.01 |
| | 1-Proh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 | 0.00 | 0.00 | 0.00 | 0.00 | 0.30 |
| | iso-Proh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 |
| | IBOH | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 |
| | 1-Buoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.12 |
| | 2-Buoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | 2-Methyl 1-Buoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | 1-Peoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.04 |
| | 2-Methyl 1-Peoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | 1-hexanol | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | 2-Methyl 1-Isobutyrate | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | others | 0.00 | 0.00 | 0.00 | 0.00 | 100.00 | 0.00 | 0.11 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | TOTAL | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| Mole Wt | lb/lb mole | 19.228 | 24.176 | 2.020 | 20.613 | 58.863 | 20.613 | 26.065 | 25.913 | 24.176 | 38.181 | 41.004 | 33.193 |
| Flow | SCFH | 29359 | 57532 | 8494 | 95384 | 0 | 95384 | 75049 | 70615 | 6524 | 132 | 7418 | 3613 |
| | lb mole/hr | 75.93 | 148.80 | 21.97 | 246.70 | 0.00 | 246.70 | 194.10 | 182.63 | 16.87 | 0.34 | 19.19 | 9.34 |
| | lb/hr | 1460.1 | 3597.4 | 44.4 | 5085.2 | 0.0 | 5085.2 | 5059.4 | 4732.6 | 407.9 | 13.0 | 786.7 | 310.2 |

**RUN NO: AF-R17.1x** **TITLE: LPDME with Shell Syngas : SV=6000**

**Balance Period:**

| | | |
|---|---|---|
| Start Date | 11/1/1999 | 14:00 |
| End Date | 11/2/1999 | 8:00 |

**Time From Start of Run (hr)**

| | |
|---|---|
| Start | 474.00 |
| End | 492.00 |

**Reaction Conditions**

| | |
|---|---|
| Temperature (°F) | 481.43 |
| Pressure (psig) | 751.76 |
| Space Velocity (sL/kg-hr) | 5810 |
| Vg (inlet) | 0.55 |

**Slurry Data**

| | |
|---|---|
| Catalyst Weight (lb oxide) | 941 |
| Slurry Level (Reactor Height, ft) | 43.5 |
| Slurry Conc. based on NDG (wt %) | 42.1 |
| Slurry Conc. based on DP (wt %) | 36.3 |
| Gas Holdup based on NDG (vol %) | 54.4 |
| Gas Holdup based on DP (vol %) | 42.3 |

**Performance Results**

| | |
|---|---|
| CO Conversion to H2 (%) | -0.1 |
| CO Conversion to MeOH (%) | 5.6 |
| CO Conversion to DME (%) | 14.2 |
| Methanol Production (Ton/day) | 3.46 |
| DME Production (Ton/day) | 4.22 |
| Methanol Productivity (gmole/kg-hr) | 9.56 |
| DME Productivity (gmole/kg-hr) | 8.12 |
| H2O Productivity (gmole/kg-hr) | 0.04 |
| MeOH Rate Constant (1-CSTR) | 3.40 |
| DME Rate Constant (1-CSTR) | 20.04 |

**Atomic/Mass Balance (% of reactor inlet)**

| | |
|---|---|
| C | 99.9 |
| H | 93.2 |
| O | 100.5 |
| N | 100.5 |
| Total Mass | 100.0 |

**Liquid Product Analysis (wt%)**

| | Sample# 21:00 |
|---|---|
| Methanol | 87.591 |
| Ethanol | 1.392 |
| 1-Propanol | 0.529 |
| iso-Propanol | 0 |
| 1-Butanol | 0.258 |
| 2-Butanol | 0.046 |
| iso-Butanol | 0.104 |
| 2-Methyl-1-Butanol | 0 |
| 1-Pentanol | 0.109 |
| 2-Methyl-1-Pentanol | 0 |
| 1-Hexanol | 0.042 |
| 2-Methyl-1-Isobutyrate | 0 |
| Methyl Acetate | 0.249 |
| Ethyl Acetate | 0 |
| Methyl Formate | 0.798 |
| DME | 6.444 |
| CO2 | 0 |
| Water | 0.242 |
| Oil | 0.2 |
| Total | 98.004 |

**RUN NO:** AF-R17.1x **TITLE:** LPDME with Shell Syngas : SV=6000

| | | FRESH MAKE-UP | RECYCLE | HP H2 MAKEUP | DRY FEED | ALCOHOL INJECT. | REACT FEED | REACT EFFL | 22.10 VAPOR | PURGE 1 PIC-201 | PURGE 2 22.11 | PURGE 3 07.20 | LIQUID PROD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| T | F | 290.1 | 118.9 | 72.3 | 279.3 | 100.0 | 271.3 | 457.5 | 88.2 | 81.0 | 69.6 | 81.3 | 80.0 |
| P | psig | 812.8 | 810.5 | 796.3 | 781.0 | 781.0 | 781.0 | 751.8 | 743.7 | 706.2 | 29.6 | 120.0 | 1.0 |
| | | | | | | | | | | 8.04 | | | |
| Comp | H2 | 33.78 | 17.46 | 100.00 | 30.13 | 0.00 | 30.13 | 15.76 | 15.76 | 17.46 | 2.94 | 1.48 | 0.00 |
| (mole%) | CO | 65.97 | 77.10 | 0.00 | 66.31 | 0.00 | 66.31 | 66.45 | 70.72 | 77.10 | 28.89 | 17.45 | 0.00 |
| | N2 | 0.25 | 0.94 | 0.00 | 0.64 | 0.00 | 0.64 | 0.82 | 0.86 | 0.94 | 0.28 | 0.16 | 0.00 |
| | CH4 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | CO2 | 0.00 | 4.50 | 0.00 | 2.91 | 0.00 | 2.91 | 8.04 | 8.53 | 4.50 | 32.01 | 42.06 | 0.00 |
| | DME | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 3.99 | 3.76 | 0.00 | 30.99 | 36.19 | 4.74 |
| | MeAc | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.11 |
| | EtAc | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | MeFm | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.45 |
| | MeOH | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.69 | 0.36 | 0.00 | 4.88 | 2.66 | 92.65 |
| | H2O | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.08 | 0.00 | 0.00 | 0.00 | 0.00 | 0.46 |
| | Etoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.04 | 0.00 | 0.00 | 0.00 | 0.00 | 1.02 |
| | 1-Proh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 | 0.00 | 0.00 | 0.00 | 0.00 | 0.30 |
| | iso-Proh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | IBOH | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 |
| | 1-Buoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.12 |
| | 2-Buoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | 2-Methyl 1-Buoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | 1-Peoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.04 |
| | 2-Methyl 1-Peoh | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | 1-hexanol | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.01 |
| | 2-Methyl 1-Isobutyrate | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | others | 0.00 | 0.00 | 0.00 | 0.00 | 100.00 | 0.00 | 0.11 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | TOTAL | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| Mole Wt | lb/lb mole | 19.230 | 24.192 | 2.020 | 20.643 | 58.705 | 20.643 | 26.147 | 25.969 | 24.192 | 38.142 | 40.975 | 33.157 |
| Flow | SCFH | 29014 | 56800 | 8504 | 94319 | 0 | 94319 | 74082 | 70640 | 6497 | 149 | 7425 | 3249 |
| | lb mole/hr | 75.04 | 146.90 | 22.00 | 243.94 | 0.00 | 243.94 | 191.60 | 182.70 | 16.80 | 0.39 | 19.20 | 8.40 |
| | lb/hr | 1443.1 | 3554.0 | 44.4 | 5035.8 | 0.0 | 5035.8 | 5009.8 | 4744.4 | 406.5 | 14.7 | 786.8 | 278.6 |

**RUN NO: AF-R17.1y** **TITLE: LPDME with Shell Syngas : SV=6000**

**Balance Period:**

| | | |
|---|---|---|
| Start Date | 11/2/1999 | 8:00 |
| End Date | 11/2/1999 | 23:00 |

**Time From Start of Run (hr)**

| | |
|---|---|
| Start | 492.00 |
| End | 507.00 |

**Reaction Conditions**

| | |
|---|---|
| Temperature (°F) | 481.25 |
| Pressure (psig) | 751.76 |
| Space Velocity (sL/kg-hr) | 5874 |
| Vg (inlet) | 0.56 |

**Slurry Data**

| | |
|---|---|
| Catalyst Weight (lb oxide) | 941 |
| Slurry Level (Reactor Height, ft) | 43.5 |
| Slurry Conc. based on NDG (wt %) | 42.4 |
| Slurry Conc. based on DP (wt %) | 36.4 |
| Gas Holdup based on NDG (vol %) | 54.8 |
| Gas Holdup based on DP (vol %) | 42.6 |

**Performance Results**

| | |
|---|---|
| CO Conversion to H2 (%) | -0.1 |
| CO Conversion to MeOH (%) | 5.6 |
| CO Conversion to DME (%) | 13.9 |
| Methanol Production (Ton/day) | 3.51 |
| DME Production (Ton/day) | 4.20 |
| Methanol Productivity (gmole/kg-hr) | 9.71 |
| DME Productivity (gmole/kg-hr) | 8.08 |
| H2O Productivity (gmole/kg-hr) | 0.04 |
| MeOH Rate Constant (1-CSTR) | 3.38 |
| DME Rate Constant (1-CSTR) | 19.77 |

**Atomic/Mass Balance (% of reactor inlet)**

| | |
|---|---|
| C | 100.7 |
| H | 94.2 |
| O | 101.3 |
| N | 99.7 |
| Total Mass | 100.8 |

**Liquid Product Analysis (wt%)**

| | Sample# 21:00 |
|---|---|
| Methanol | 87.65 |
| Ethanol | 1.361 |
| 1-Propanol | 0.526 |
| iso-Propanol | 0.039 |
| 1-Butanol | 0.27 |
| 2-Butanol | 0.049 |
| iso-Butanol | 0.096 |
| 2-Methyl-1-Butanol | 0 |
| 1-Pentanol | 0.11 |
| 2-Methyl-1-Pentanol | 0 |
| 1-Hexanol | 0.032 |
| 2-Methyl-1-Isobutyrate | 0 |
| Methyl Acetate | 0.241 |
| Ethyl Acetate | 0 |
| Methyl Formate | 0.733 |
| DME | 6.77 |
| CO2 | 0 |
| Water | 0 |
| Oil | 0.2 |
| Total | 98.077 |

**RUN NO:** **AF-R17.1y** **TITLE:** **LPDME with Shell Syngas : SV=6000**

| | | FRESH MAKE-UP | RECYCLE | HP H2 MAKEUP | DRY FEED | ALCOHOL INJECT. | REACT FEED | REACT EFFL | 22.10 VAPOR | PURGE 1 PIC-201 | PURGE 2 22.11 | PURGE 3 07.20 | LIQUID PROD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **T** | **F** | 281.3 | 114.5 | 69.8 | 267.3 | 100.0 | 258.4 | 456.3 | 83.0 | 76.1 | 64.6 | 76.9 | 80.0 |
| **P** | **psig** | 812.7 | 810.9 | 803.3 | 780.8 | 780.8 | 780.8 | 751.8 | 744.0 | 705.4 | 29.6 | 120.0 | 1.0 |
| | | | | | | | | | | 7.92 | | | |
| **Comp** | **H2** | 33.78 | 17.53 | 100.00 | 30.06 | 0.00 | 30.06 | 15.79 | 15.88 | 17.53 | 2.98 | 1.51 | 0.00 |
| **(mole%)** | **CO** | 65.95 | 77.03 | 0.00 | 66.38 | 0.00 | 66.38 | 66.54 | 71.00 | 77.03 | 29.64 | 17.74 | 0.00 |
| | **N2** | 0.27 | 1.00 | 0.00 | 0.69 | 0.00 | 0.69 | 0.87 | 0.90 | 1.00 | 0.30 | 0.17 | 0.00 |
| | **CH4** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **CO2** | 0.00 | 4.44 | 0.00 | 2.88 | 0.00 | 2.88 | 7.92 | 8.35 | 4.44 | 32.85 | 42.28 | 0.00 |
| | **DME** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 3.91 | 3.60 | 0.00 | 29.94 | 36.05 | 4.99 |
| | **MeAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.11 |
| | **EtAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **MeFm** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.41 |
| | **MeOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.70 | 0.26 | 0.00 | 4.30 | 2.26 | 92.90 |
| | **H2O** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.08 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **Etoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 | 0.00 | 0.00 | 0.00 | 0.00 | 1.00 |
| | **1-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 | 0.00 | 0.00 | 0.00 | 0.00 | 0.30 |
| | **iso-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **IBOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.04 |
| | **1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.12 |
| | **2-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **2-Methyl 1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.04 |
| | **2-Methyl 1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-hexanol** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.01 |
| | **2-Methyl 1-Isobutyrate** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **others** | 0.00 | 0.00 | 0.00 | 0.00 | 100.00 | 0.00 | 0.11 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **TOTAL** | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| **Mole Wt** | **lb/lb mole** | 19.230 | 24.163 | 2.020 | 20.659 | 58.765 | 20.659 | 26.106 | 25.877 | 24.163 | 38.056 | 40.963 | 33.247 |
| **Flow** | **SCFH** | 29030 | 57830 | 8496 | 95356 | 0 | 95356 | 75092 | 72087 | 6548 | 134 | 7253 | 3491 |
| | **lb mole/hr** | 75.08 | 149.57 | 21.97 | 246.62 | 0.00 | 246.62 | 194.21 | 186.44 | 16.93 | 0.35 | 18.76 | 9.03 |
| | **lb/hr** | 1443.8 | 3614.0 | 44.4 | 5095.0 | 0.0 | 5095.0 | 5070.2 | 4824.6 | 409.2 | 13.1 | 768.4 | 300.2 |

**RUN NO: AF-R17.1z** **TITLE: LPDME with Shell Syngas : SV=6000**

**Balance Period:**

| | | |
|---|---|---|
| Start Date | 11/2/1999 | 23:00 |
| End Date | 11/3/1999 | 19:00 |

**Time From Start of Run (hr)**

| | |
|---|---|
| Start | 507.00 |
| End | 527.00 |

**Reaction Conditions**

| | |
|---|---|
| Temperature (°F) | 481.35 |
| Pressure (psig) | 751.49 |
| Space Velocity (sL/kg-hr) | 5911 |
| Vg (inlet) | 0.56 |

**Slurry Data**

| | |
|---|---|
| Catalyst Weight (lb oxide) | 941 |
| Slurry Level (Reactor Height, ft) | 43.5 |
| Slurry Conc. based on NDG (wt %) | 42.5 |
| Slurry Conc. based on DP (wt %) | 36.6 |
| Gas Holdup based on NDG (vol %) | 55.0 |
| Gas Holdup based on DP (vol %) | 42.9 |

**Performance Results**

| | |
|---|---|
| CO Conversion to H2 (%) | -0.1 |
| CO Conversion to MeOH (%) | 5.6 |
| CO Conversion to DME (%) | 13.7 |
| Methanol Production (Ton/day) | 3.52 |
| DME Production (Ton/day) | 4.17 |
| Methanol Productivity (gmole/kg-hr) | 9.72 |
| DME Productivity (gmole/kg-hr) | 8.01 |
| H2O Productivity (gmole/kg-hr) | 0.04 |
| MeOH Rate Constant (1-CSTR) | 3.24 |
| DME Rate Constant (1-CSTR) | 19.65 |

**Atomic/Mass Balance (% of reactor inlet)**

| | |
|---|---|
| C | 101.5 |
| H | 95.5 |
| O | 102.1 |
| N | 100.3 |
| Total Mass | 101.6 |

**Liquid Product Analysis (wt%)**

| | Sample# 8:50 |
|---|---|
| Methanol | 88.111 |
| Ethanol | 1.379 |
| 1-Propanol | 0.53 |
| iso-Propanol | 0.038 |
| 1-Butanol | 0.273 |
| 2-Butanol | 0.05 |
| iso-Butanol | 0.093 |
| 2-Methyl-1-Butanol | 0 |
| 1-Pentanol | 0.115 |
| 2-Methyl-1-Pentanol | 0 |
| 1-Hexanol | 0.03 |
| 2-Methyl-1-Isobutyrate | 0 |
| Methyl Acetate | 0.242 |
| Ethyl Acetate | 0 |
| Methyl Formate | 0.902 |
| DME | 6.001 |
| CO2 | 0 |
| Water | 0 |
| Oil | 0.2 |
| Total | 97.964 |

**RUN NO:** **AF-R17.1z** **TITLE:** **LPDME with Shell Syngas : SV=6000**

| | | FRESH MAKE-UP | RECYCLE | HP H2 MAKEUP | DRY FEED | ALCOHOL INJECT. | REACT FEED | REACT EFFL | 22.10 VAPOR | PURGE 1 PIC-201 | PURGE 2 22.11 | PURGE 3 07.20 | LIQUID PROD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **T** | **F** | 282.5 | 111.7 | 63.2 | 269.8 | 100.0 | 261.8 | 458.2 | 79.6 | 72.3 | 58.1 | 73.8 | 80.0 |
| **P** | **psig** | 812.0 | 811.2 | 792.3 | 780.4 | 780.4 | 780.4 | 751.5 | 744.3 | 704.2 | 29.4 | 120.0 | 1.0 |
| | | | | | | | | | | 7.84 | | | |
| **Comp** | **H2** | 34.07 | 17.58 | 100.00 | 30.07 | 0.00 | 30.07 | 16.05 | 15.92 | 17.58 | 3.04 | 1.51 | 0.00 |
| **(mole%)** | **CO** | 65.66 | 76.92 | 0.00 | 66.31 | 0.00 | 66.31 | 66.47 | 70.93 | 76.92 | 30.31 | 17.77 | 0.00 |
| | **N2** | 0.27 | 1.01 | 0.00 | 0.70 | 0.00 | 0.70 | 0.88 | 0.92 | 1.01 | 0.31 | 0.17 | 0.00 |
| | **CH4** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **CO2** | 0.00 | 4.49 | 0.00 | 2.93 | 0.00 | 2.93 | 7.84 | 8.35 | 4.49 | 33.38 | 42.42 | 0.00 |
| | **DME** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 3.84 | 3.58 | 0.00 | 29.07 | 36.12 | 4.42 |
| | **MeAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.11 |
| | **EtAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **MeFm** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.51 |
| | **MeOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.66 | 0.30 | 0.00 | 3.90 | 2.01 | 93.35 |
| | **H2O** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.08 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **Etoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 | 0.00 | 0.00 | 0.00 | 0.00 | 1.02 |
| | **1-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 | 0.00 | 0.00 | 0.00 | 0.00 | 0.30 |
| | **iso-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **IBOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.04 |
| | **1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.12 |
| | **2-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **2-Methyl 1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.04 |
| | **2-Methyl 1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-hexanol** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.01 |
| | **2-Methyl 1-Isobutyrate** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **others** | 0.00 | 0.00 | 0.00 | 0.00 | 100.00 | 0.00 | 0.11 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **TOTAL** | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| **Mole Wt** | **lb/lb mole** | 19.156 | 24.160 | 2.020 | 20.664 | 58.764 | 20.664 | 26.012 | 25.867 | 24.160 | 37.950 | 40.987 | 33.198 |
| **Flow** | **SCFH** | 29085 | 58374 | 8500 | 95959 | 0 | 95959 | 75907 | 73190 | 6520 | 21 | 7083 | 3658 |
| | **lb mole/hr** | 75.23 | 150.97 | 21.99 | 248.18 | 0.00 | 248.18 | 196.32 | 189.29 | 16.86 | 0.05 | 18.32 | 9.46 |
| | **lb/hr** | 1441.0 | 3647.6 | 44.4 | 5128.4 | 0.0 | 5128.4 | 5106.7 | 4896.5 | 407.4 | 2.0 | 750.9 | 314.1 |

**RUN NO: AF-R17.1aa** **TITLE: LPDME with Shell Syngas : SV=6000**

**Balance Period:**

| | | |
|---|---|---|
| Start Date | 11/4/1999 | 0:00 |
| End Date | 11/4/1999 | 11:00 |

**Time From Start of Run (hr)**

| | |
|---|---|
| Start | 532.00 |
| End | 543.00 |

**Reaction Conditions**

| | |
|---|---|
| Temperature (°F) | 481.46 |
| Pressure (psig) | 750.04 |
| Space Velocity (sL/kg-hr) | 5819 |
| Vg (inlet) | 0.55 |

**Slurry Data**

| | |
|---|---|
| Catalyst Weight (lb oxide) | 941 |
| Slurry Level (Reactor Height, ft) | 43.5 |
| Slurry Conc. based on NDG (wt %) | 42.5 |
| Slurry Conc. based on DP (wt %) | 36.3 |
| Gas Holdup based on NDG (vol %) | 55.0 |
| Gas Holdup based on DP (vol %) | 42.3 |

**Performance Results**

| | |
|---|---|
| CO Conversion to H2 (%) | -0.1 |
| CO Conversion to MeOH (%) | 5.5 |
| CO Conversion to DME (%) | 14.1 |
| Methanol Production (Ton/day) | 3.42 |
| DME Production (Ton/day) | 4.18 |
| Methanol Productivity (gmole/kg-hr) | 9.46 |
| DME Productivity (gmole/kg-hr) | 8.03 |
| H2O Productivity (gmole/kg-hr) | 0.04 |
| MeOH Rate Constant (1-CSTR) | 3.24 |
| DME Rate Constant (1-CSTR) | 19.88 |

**Atomic/Mass Balance (% of reactor inlet)**

| | |
|---|---|
| C | 105.5 |
| H | 98.9 |
| O | 106.2 |
| N | 100.3 |
| Total Mass | 105.6 |

**Liquid Product Analysis (wt%)**

| | Sample# 0:00 | Sample# 4:30 |
|---|---|---|
| Methanol | 88.132 | 87.369 |
| Ethanol | 1.348 | 1.309 |
| 1-Propanol | 0.511 | 0.503 |
| iso-Propanol | 0.035 | 0.204 |
| 1-Butanol | 0.25 | 0.083 |
| 2-Butanol | 0.05 | 0 |
| iso-Butanol | 0.091 | 0.208 |
| 2-Methyl-1-Butanol | 0 | 0 |
| 1-Pentanol | 0.108 | 0.106 |
| 2-Methyl-1-Pentanol | 0 | 0 |
| 1-Hexanol | 0.032 | 0 |
| 2-Methyl-1-Isobutyrate | 0 | 0 |
| Methyl Acetate | 0.239 | 0 |
| Ethyl Acetate | 0 | 0 |
| Methyl Formate | 0.839 | 0.755 |
| DME | 6.119 | 7.471 |
| CO2 | 0 | 0 |
| Water | 0.236 | 0 |
| Oil | 0.2 | 0.2 |
| Total | 98.19 | 98.208 |

**RUN NO:** **AF-R17.1aa** **TITLE:** **LPDME with Shell Syngas : SV=6000**

| | | FRESH MAKE-UP | RECYCLE | HP H2 MAKEUP | DRY FEED | ALCOHOL INJECT. | REACT FEED | REACT EFFL | 22.10 VAPOR | PURGE 1 PIC-201 | PURGE 2 22.11 | PURGE 3 07.20 | LIQUID PROD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **T** | **F** | 291.6 | 117.2 | 68.0 | 281.1 | 100.0 | 273.5 | 459.0 | 85.4 | 78.0 | 63.7 | 78.9 | 80.0 |
| **P** | **psig** | 811.7 | 809.8 | 784.6 | 780.5 | 780.5 | 780.5 | 750.0 | 743.2 | 704.4 | 29.6 | 120.0 | 1.0 |
| | | | | | | | | | | 7.94 | | | |
| **Comp** | **H2** | 33.69 | 17.66 | 100.00 | 30.20 | 0.00 | 30.20 | 16.06 | 16.06 | 17.66 | 2.89 | 1.52 | 0.00 |
| **(mole%)** | **CO** | 65.96 | 76.59 | 0.00 | 66.03 | 0.00 | 66.03 | 66.09 | 70.49 | 76.59 | 28.25 | 17.57 | 0.00 |
| | **N2** | 0.35 | 1.27 | 0.00 | 0.87 | 0.00 | 0.87 | 1.10 | 1.10 | 1.27 | 0.66 | 0.21 | 0.00 |
| | **CH4** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **CO2** | 0.00 | 4.49 | 0.00 | 2.90 | 0.00 | 2.90 | 7.94 | 8.43 | 4.49 | 31.12 | 42.10 | 0.00 |
| | **DME** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 3.93 | 3.62 | 0.00 | 29.08 | 36.22 | 4.99 |
| | **MeAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 |
| | **EtAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **MeFm** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.45 |
| | **MeOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.62 | 0.30 | 0.00 | 8.00 | 2.39 | 92.73 |
| | **H2O** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.08 | 0.00 | 0.00 | 0.00 | 0.00 | 0.22 |
| | **Etoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 | 0.00 | 0.00 | 0.00 | 0.00 | 0.98 |
| | **1-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 | 0.00 | 0.00 | 0.00 | 0.00 | 0.29 |
| | **iso-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.07 |
| | **IBOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.07 |
| | **1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.08 |
| | **2-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.01 |
| | **2-Methyl 1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.04 |
| | **2-Methyl 1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-hexanol** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.01 |
| | **2-Methyl 1-Isobutyrate** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **others** | 0.00 | 0.00 | 0.00 | 0.00 | 100.00 | 0.00 | 0.10 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **TOTAL** | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| **Mole Wt** | **lb/lb mole** | 19.254 | 24.139 | 2.020 | 20.626 | 58.163 | 20.626 | 26.037 | 25.849 | 24.139 | 37.796 | 40.965 | 33.190 |
| **Flow** | **SCFH** | 28958 | 57000 | 8501 | 94460 | 0 | 94460 | 74455 | 74856 | 6620 | 11 | 7176 | 3673 |
| | **lb mole/hr** | 74.90 | 147.42 | 21.99 | 244.31 | 0.00 | 244.31 | 192.57 | 193.60 | 17.12 | 0.03 | 18.56 | 9.50 |
| | **lb/hr** | 1442.1 | 3558.6 | 44.4 | 5039.0 | 0.0 | 5039.0 | 5013.8 | 5004.5 | 413.3 | 1.1 | 760.3 | 315.3 |

**RUN NO: AF-R17.1bb** **TITLE: LPDME with Shell Syngas : SV=6000**

**Balance Period:**

| | | |
|---|---|---|
| Start Date | 11/4/1999 | 22:00 |
| End Date | 11/5/1999 | 16:00 |

**Time From Start of Run (hr)**

| | |
|---|---|
| Start | 554.00 |
| End | 572.00 |

**Reaction Conditions**

| | |
|---|---|
| Temperature (°F) | 481.44 |
| Pressure (psig) | 749.08 |
| Space Velocity (sL/kg-hr) | 5819 |
| Vg (inlet) | 0.55 |

**Slurry Data**

| | |
|---|---|
| Catalyst Weight (lb oxide) | 941 |
| Slurry Level (Reactor Height, ft) | 43.5 |
| Slurry Conc. based on NDG (wt %) | 42.2 |
| Slurry Conc. based on DP (wt %) | 35.9 |
| Gas Holdup based on NDG (vol %) | 54.6 |
| Gas Holdup based on DP (vol %) | 41.5 |

**Performance Results**

| | |
|---|---|
| CO Conversion to H2 (%) | -0.1 |
| CO Conversion to MeOH (%) | 5.7 |
| CO Conversion to DME (%) | 14.0 |
| Methanol Production (Ton/day) | 3.55 |
| DME Production (Ton/day) | 4.16 |
| Methanol Productivity (gmole/kg-hr) | 9.80 |
| DME Productivity (gmole/kg-hr) | 8.00 |
| H2O Productivity (gmole/kg-hr) | 0.04 |
| MeOH Rate Constant (1-CSTR) | 3.40 |
| DME Rate Constant (1-CSTR) | 19.87 |

**Atomic/Mass Balance (% of reactor inlet)**

| | |
|---|---|
| C | 101.0 |
| H | 95.6 |
| O | 101.5 |
| N | 100.2 |
| Total Mass | 101.1 |

**Liquid Product Analysis (wt%)**

| | Sample# 4:30 | Sample# 13:30 |
|---|---|---|
| Methanol | 88.087 | 88.711 |
| Ethanol | 1.312 | 1.317 |
| 1-Propanol | 0.49 | 0.497 |
| iso-Propanol | 0.034 | 0.036 |
| 1-Butanol | 0.243 | 0.246 |
| 2-Butanol | 0.045 | 0.05 |
| iso-Butanol | 0.089 | 0.088 |
| 2-Methyl-1-Butanol | 0 | 0 |
| 1-Pentanol | 0.109 | 0.108 |
| 2-Methyl-1-Pentanol | 0 | 0 |
| 1-Hexanol | 0.042 | 0.044 |
| 2-Methyl-1-Isobutyrate | 0 | 0 |
| Methyl Acetate | 0.254 | 0.238 |
| Ethyl Acetate | 0 | 0 |
| Methyl Formate | 0.859 | 0.818 |
| DME | 6.261 | 5.71 |
| CO2 | 0 | 0 |
| Water | 0.228 | 0.229 |
| Oil | 0.2 | 0.2 |
| Total | 98.253 | 98.292 |

**RUN NO:** **AF-R17.1bb** **TITLE:** **LPDME with Shell Syngas : SV=6000**

| | | FRESH MAKE-UP | RECYCLE | HP H2 MAKEUP | DRY FEED | ALCOHOL INJECT. | REACT FEED | REACT EFFL | 22.10 VAPOR | PURGE 1 PIC-201 | PURGE 2 22.11 | PURGE 3 07.20 | LIQUID PROD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **T** | **F** | 297.0 | 123.1 | 78.1 | 285.1 | 100.0 | 277.6 | 458.3 | 91.2 | 84.3 | 72.2 | 84.5 | 80.0 |
| **P** | **psig** | 812.7 | 809.7 | 800.8 | 780.5 | 780.5 | 780.5 | 749.1 | 740.3 | 703.3 | 29.6 | 120.0 | 1.0 |
| | | | | | | | | | | 8.14 | | | |
| **Comp** | **H2** | 33.70 | 17.80 | 100.00 | 30.33 | 0.00 | 30.33 | 16.01 | 16.15 | 17.80 | 3.02 | 1.50 | 0.00 |
| **(mole%)** | **CO** | 66.01 | 76.40 | 0.00 | 65.89 | 0.00 | 65.89 | 65.96 | 70.25 | 76.40 | 28.60 | 17.13 | 0.00 |
| | **N2** | 0.29 | 1.05 | 0.00 | 0.71 | 0.00 | 0.71 | 0.91 | 0.95 | 1.05 | 0.35 | 0.17 | 0.00 |
| | **CH4** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **CO2** | 0.00 | 4.75 | 0.00 | 3.06 | 0.00 | 3.06 | 8.14 | 8.59 | 4.75 | 31.30 | 41.62 | 0.00 |
| | **DME** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 3.92 | 3.72 | 0.00 | 30.30 | 36.69 | 4.38 |
| | **MeAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.11 |
| | **EtAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **MeFm** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.47 |
| | **MeOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.80 | 0.35 | 0.00 | 6.43 | 2.88 | 93.10 |
| | **H2O** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.08 | 0.00 | 0.00 | 0.00 | 0.00 | 0.43 |
| | **Etoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.05 | 0.00 | 0.00 | 0.00 | 0.00 | 0.96 |
| | **1-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 | 0.00 | 0.00 | 0.00 | 0.00 | 0.28 |
| | **iso-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **IBOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.04 |
| | **1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.11 |
| | **2-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **2-Methyl 1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.04 |
| | **2-Methyl 1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-hexanol** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.01 |
| | **2-Methyl 1-Isobutyrate** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **others** | 0.00 | 0.00 | 0.00 | 0.00 | 100.00 | 0.00 | 0.11 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **TOTAL** | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| **Mole Wt** | **lb/lb mole** | 19.252 | 24.143 | 2.020 | 20.616 | 58.963 | 20.616 | 26.088 | 25.869 | 24.143 | 37.946 | 40.999 | 33.102 |
| **Flow** | **SCFH** | 29008 | 56959 | 8498 | 94465 | 0 | 94465 | 74291 | 71517 | 6671 | 10 | 7183 | 3562 |
| | **lb mole/hr** | 75.03 | 147.32 | 21.98 | 244.32 | 0.00 | 244.32 | 192.14 | 184.97 | 17.25 | 0.03 | 18.58 | 9.21 |
| | **lb/hr** | 1444.4 | 3556.7 | 44.4 | 5036.9 | 0.0 | 5036.9 | 5012.7 | 4784.9 | 416.6 | 1.0 | 761.7 | 305.0 |

**RUN NO: AF-R17.1cc** **TITLE: LPDME with Shell Syngas : SV=6000**

**Balance Period:**

| | | |
|---|---|---|
| Start Date | 11/5/1999 | 16:00 |
| End Date | 11/6/1999 | 10:00 |

**Time From Start of Run (hr)**

| | |
|---|---|
| Start | 572.00 |
| End | 590.00 |

**Reaction Conditions**

| | |
|---|---|
| Temperature (°F) | 481.41 |
| Pressure (psig) | 749.71 |
| Space Velocity (sL/kg-hr) | 5861 |
| Vg (inlet) | 0.56 |

**Slurry Data**

| | |
|---|---|
| Catalyst Weight (lb oxide) | 941 |
| Slurry Level (Reactor Height, ft) | 43.5 |
| Slurry Conc. based on NDG (wt %) | 42.5 |
| Slurry Conc. based on DP (wt %) | 36.1 |
| Gas Holdup based on NDG (vol %) | 55.0 |
| Gas Holdup based on DP (vol %) | 41.8 |

**Performance Results**

| | |
|---|---|
| CO Conversion to H2 (%) | -0.1 |
| CO Conversion to MeOH (%) | 5.7 |
| CO Conversion to DME (%) | 13.7 |
| Methanol Production (Ton/day) | 3.59 |
| DME Production (Ton/day) | 4.11 |
| Methanol Productivity (gmole/kg-hr) | 9.92 |
| DME Productivity (gmole/kg-hr) | 7.89 |
| H2O Productivity (gmole/kg-hr) | 0.04 |
| MeOH Rate Constant (1-CSTR) | 3.39 |
| DME Rate Constant (1-CSTR) | 19.50 |

**Atomic/Mass Balance (% of reactor inlet)**

| | |
|---|---|
| C | 100.5 |
| H | 94.8 |
| O | 101.1 |
| N | 100.4 |
| Total Mass | 100.7 |

**Liquid Product Analysis (wt%)**

| | Sample# 1:30 |
|---|---|
| Methanol | 88.605 |
| Ethanol | 1.326 |
| 1-Propanol | 0.496 |
| iso-Propanol | 0.035 |
| 1-Butanol | 0.255 |
| 2-Butanol | 0.043 |
| iso-Butanol | 0.089 |
| 2-Methyl-1-Butanol | 0 |
| 1-Pentanol | 0.105 |
| 2-Methyl-1-Pentanol | 0 |
| 1-Hexanol | 0.04 |
| 2-Methyl-1-Isobutyrate | 0 |
| Methyl Acetate | 0.236 |
| Ethyl Acetate | 0 |
| Methyl Formate | 0.809 |
| DME | 5.834 |
| CO2 | 0 |
| Water | 0.24 |
| Oil | 0.2 |
| Total | 98.313 |

**RUN NO:** **AF-R17.1cc** **TITLE:** **LPDME with Shell Syngas : SV=6000**

| | | FRESH MAKE-UP | RECYCLE | HP H2 MAKEUP | DRY FEED | ALCOHOL INJECT. | REACT FEED | REACT EFFL | 22.10 VAPOR | PURGE 1 PIC-201 | PURGE 2 22.11 | PURGE 3 07.20 | LIQUID PROD |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **T** | **F** | 296.8 | 122.1 | 72.8 | 285.2 | 100.0 | 277.6 | 459.2 | 91.0 | 83.3 | 70.5 | 84.2 | 80.0 |
| **P** | **psig** | 813.5 | 811.2 | 786.4 | 781.5 | 781.5 | 781.5 | 749.7 | 740.0 | 702.9 | 29.5 | 120.0 | 1.0 |
| | | | | | | | | | | 8.08 | | | |
| **Comp** | **H2** | 33.57 | 17.74 | 100.00 | 30.16 | 0.00 | 30.16 | 15.96 | 16.15 | 17.74 | 3.11 | 1.40 | 0.00 |
| **(mole%)** | **CO** | 66.16 | 76.51 | 0.00 | 66.08 | 0.00 | 66.08 | 66.24 | 70.45 | 76.51 | 29.46 | 17.20 | 0.00 |
| | **N2** | 0.27 | 0.97 | 0.00 | 0.66 | 0.00 | 0.66 | 0.84 | 0.88 | 0.97 | 0.30 | 0.15 | 0.00 |
| | **CH4** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **CO2** | 0.00 | 4.78 | 0.00 | 3.10 | 0.00 | 3.10 | 8.08 | 8.54 | 4.78 | 31.81 | 41.57 | 0.00 |
| | **DME** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 3.83 | 3.56 | 0.00 | 29.84 | 36.78 | 4.27 |
| | **MeAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.11 |
| | **EtAc** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **MeFm** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.45 |
| | **MeOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 4.81 | 0.42 | 0.00 | 5.48 | 2.89 | 93.20 |
| | **H2O** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.08 | 0.00 | 0.00 | 0.00 | 0.00 | 0.45 |
| | **Etoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.04 | 0.00 | 0.00 | 0.00 | 0.00 | 0.97 |
| | **1-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 | 0.00 | 0.00 | 0.00 | 0.00 | 0.28 |
| | **iso-Proh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **IBOH** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.04 |
| | **1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.12 |
| | **2-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **2-Methyl 1-Buoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.04 |
| | **2-Methyl 1-Peoh** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **1-hexanol** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.01 |
| | **2-Methyl 1-Isobutyrate** | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| | **others** | 0.00 | 0.00 | 0.00 | 0.00 | 100.00 | 0.00 | 0.11 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 |
| | **TOTAL** | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| **Mole Wt** | **lb/lb mole** | 19.286 | 24.163 | 2.020 | 20.667 | 58.853 | 20.667 | 26.076 | 25.835 | 24.163 | 37.885 | 41.035 | 33.077 |
| **Flow** | **SCFH** | 28925 | 57728 | 8493 | 95147 | 0 | 95147 | 75053 | 71987 | 6713 | 132 | 7104 | 3460 |
| | **lb mole/hr** | 74.81 | 149.30 | 21.97 | 246.08 | 0.00 | 246.08 | 194.11 | 186.18 | 17.36 | 0.34 | 18.37 | 8.95 |
| | **lb/hr** | 1442.8 | 3607.7 | 44.4 | 5085.8 | 0.0 | 5085.8 | 5061.6 | 4810.1 | 419.5 | 12.9 | 754.0 | 296.0 |