

APPENDIX C - PROCESS ECONOMIC STUDY

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

and

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

Process Economics Study - Outline

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

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

1. Introduction

1.1. Process Design Options.

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

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

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

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

2.2. Methanol Product Purification Cost.

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

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

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

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

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

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

Process Economics Study - Outline

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

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

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

2.6. Recommendations for Further Study.

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

Part Two - Baseload Power and Methanol Coproduction

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

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

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

3.1 Gasification Plant Size Fixed

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

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

3.2 Power Plant Size Fixed

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

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

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

End of Part Two.

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LPMEOH™ Process, as an add-on to IGCC for Coproduction
Part Three - Coproduction for Intermediate Electric Load Following.

4. Intermediate Load Coproduction

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

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

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

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

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

4.2. Intermediate Load Coproduction - for Methanol Sales.

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

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

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

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

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

End of Part Three.

Process Economics Study - Outline

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

Part Four - Methanol Fuel Applications

5. Premium Methanol Fuel Applications

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

5. . Hydrogen Source for:

- Hydrogen fuel cells, being developed for transportation applications, can achieve 65% system efficiency, as compared to 45% for diesel IC engines and 32% for gasoline IC engines. Methanol is a storable, transportable liquid fuel which can be reformed under mild conditions to provide H₂. For small H₂ applications, *and at low utilization factors*, methanol reforming is a more economical source of hydrogen than : a) natural gas reforming, b) distillate (oil) reforming; and is cheaper than liquid H₂.

5.1.1. Fuel Cells for Transportation

5.1.2. Fuel Cells for Stationary Power

(See also dispersed power below).

5.1.3. Industrial Applications - Small Hydrogen Plants

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

5.2. Dispersed Power

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

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

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

End of Part Four.

Memorandum



To: Distribution Dept./Loc.:

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

Date: 31 March 1997

Subject: LPMEOH™ Process Economics - for IGCC Coproduction

Distribution:

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

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

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

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

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

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

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

Bill

APPENDIX D - DME DESIGN VERIFICATION TESTING

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PRODUCTS

30 June 1997

Mr. Robert M. Kornosky
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**Subject: Cooperative Agreement DE-FC22-92PC90543
Liquid Phase Methanol Demonstration Project
Liquid Phase Dimethyl Ether Design Verification Testing -
Recommendation**

Dear Bob:

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

Very truly yours,



Edward C. Heydorn
Program Manager
LPMEOH™ Demonstration Project

Enclosure

cc: Mr. William C. Jones - Eastman Chemical Co.
Mr. William J. O'Dowd - DOE-FETC
Mr. Edward Schmetz - DOE-FE-HQ
Dr. John Shen - DOE-FE-HQ
Mr. Barry T. Street - Eastman Chemical Co.
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LPDME Recommendation

Summary

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

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

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

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

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

LPDME Recommendation

The LPDME Process Concept: - Three Concurrent Reactions:

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

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

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

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

Status of the LPDME DVT Work

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

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

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

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

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

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

LPDME Recommendation

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

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

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

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

LPDME Recommendation

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

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



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

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

B. Laboratory R&D Results

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

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

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

LPDME Recommendation

Progress has been made in the laboratory effort. Figure 3 shows the performance for the first DME catalyst which was tested; goals from the Liquid Fuels Program are provided for reference. After further study, an improved DME catalyst (AB-05) was tested with two LPMEOH™ catalysts (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 H₂-rich syngas would create water buildup. Therefore, the basis for commercializing LPDME must come from:

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

LPDME Recommendation

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

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

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

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

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

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

LPDME Recommendation

Impact on CCT Project

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

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

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

References

1. Transportation energy consumption by fuel, 1975, 1995 and 2015: History: Energy Information Administration, *Short-Term Energy Outlook*, DOE/EIA-0202(96/4Q) (Washington, DC, October 1996), and *State Energy Data Report 1994*, DOE/EIA-0214(98). Projections: Table A2. Internet access at <http://www/eia.doe.gov/oiaf/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.

(end).

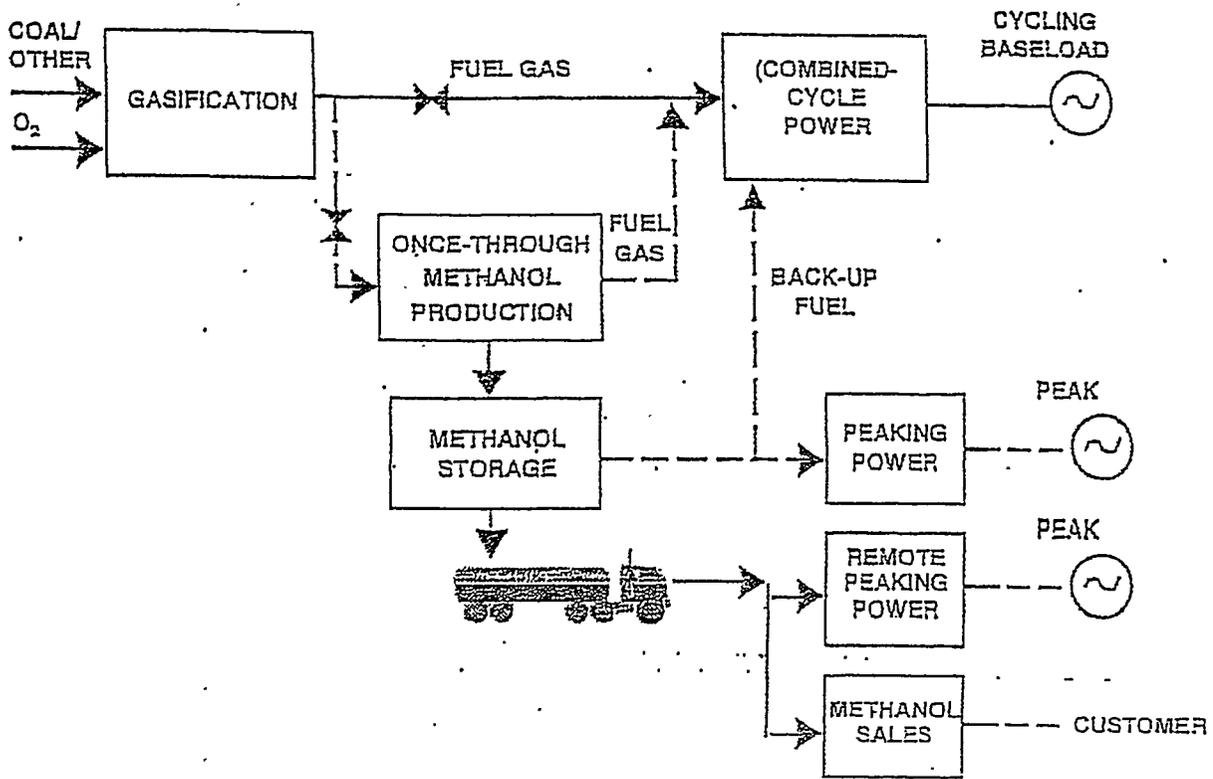
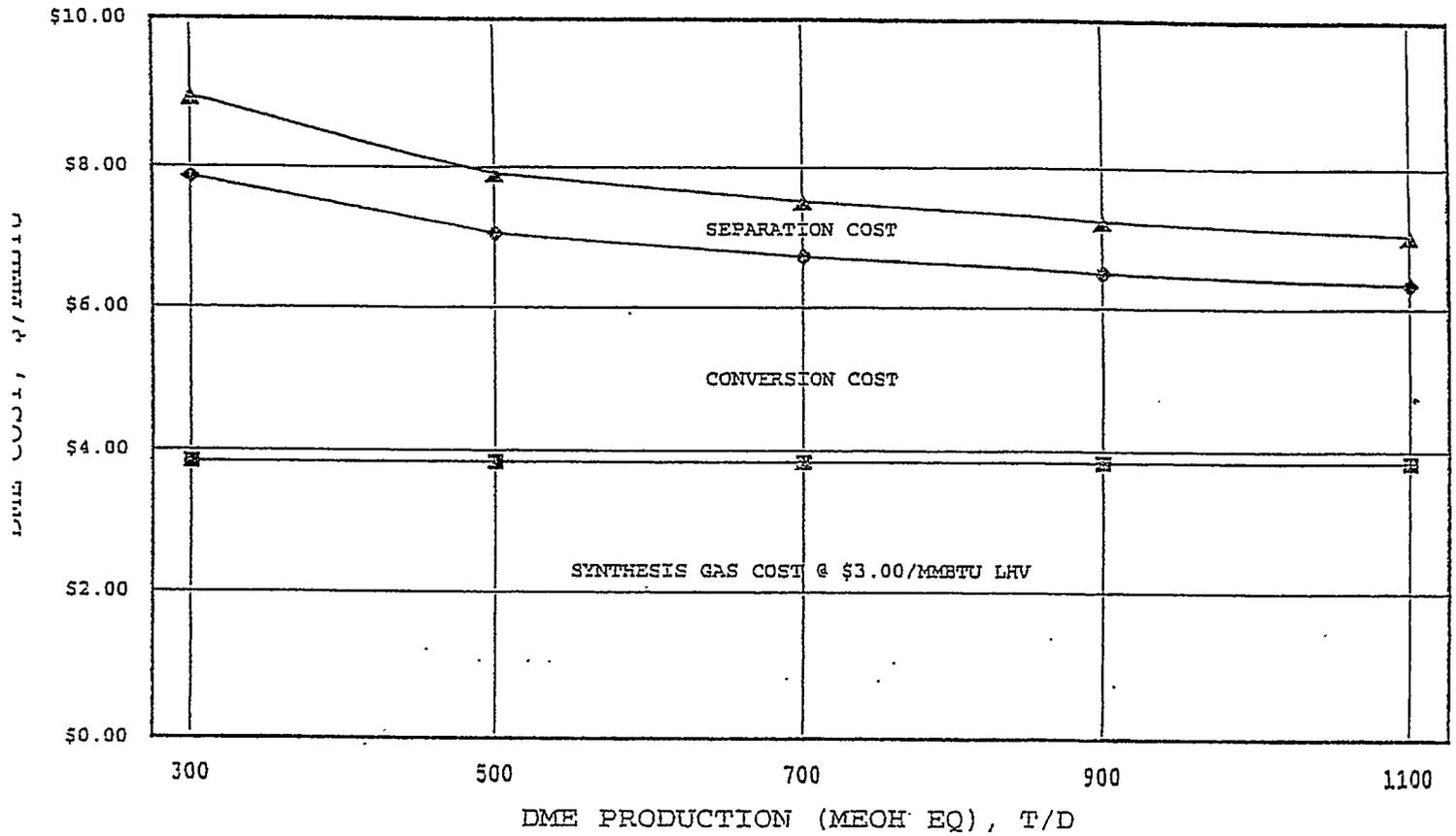


Figure 1. Once-through Methanol Coproduction with IGCC Electric Power

Figure 2

DME COST VERSUS SIZE

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



— SYNTHESIS GAS ONLY

— SYNTHESIS GAS PLUS CONVERSION

— TOTAL DME COST

GOALS

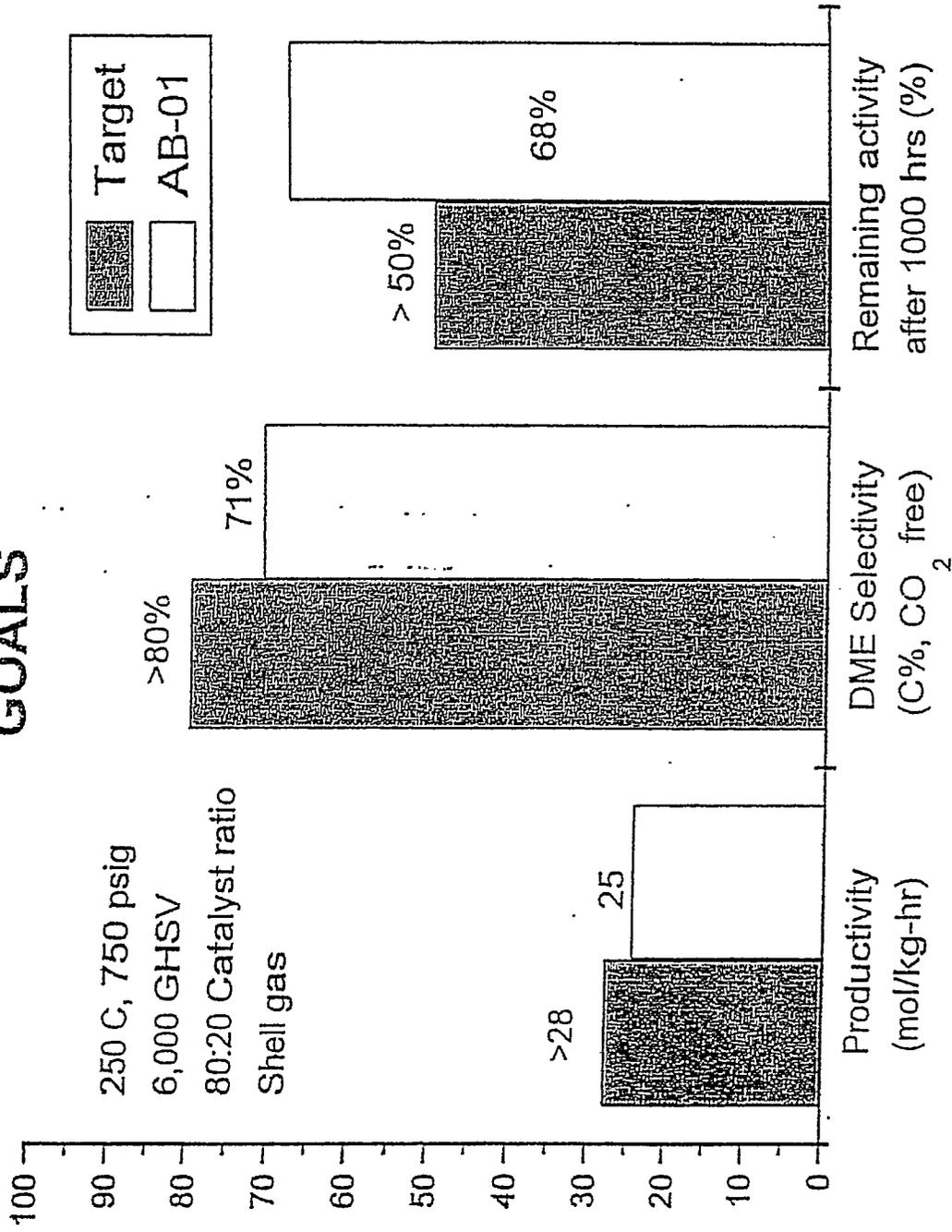
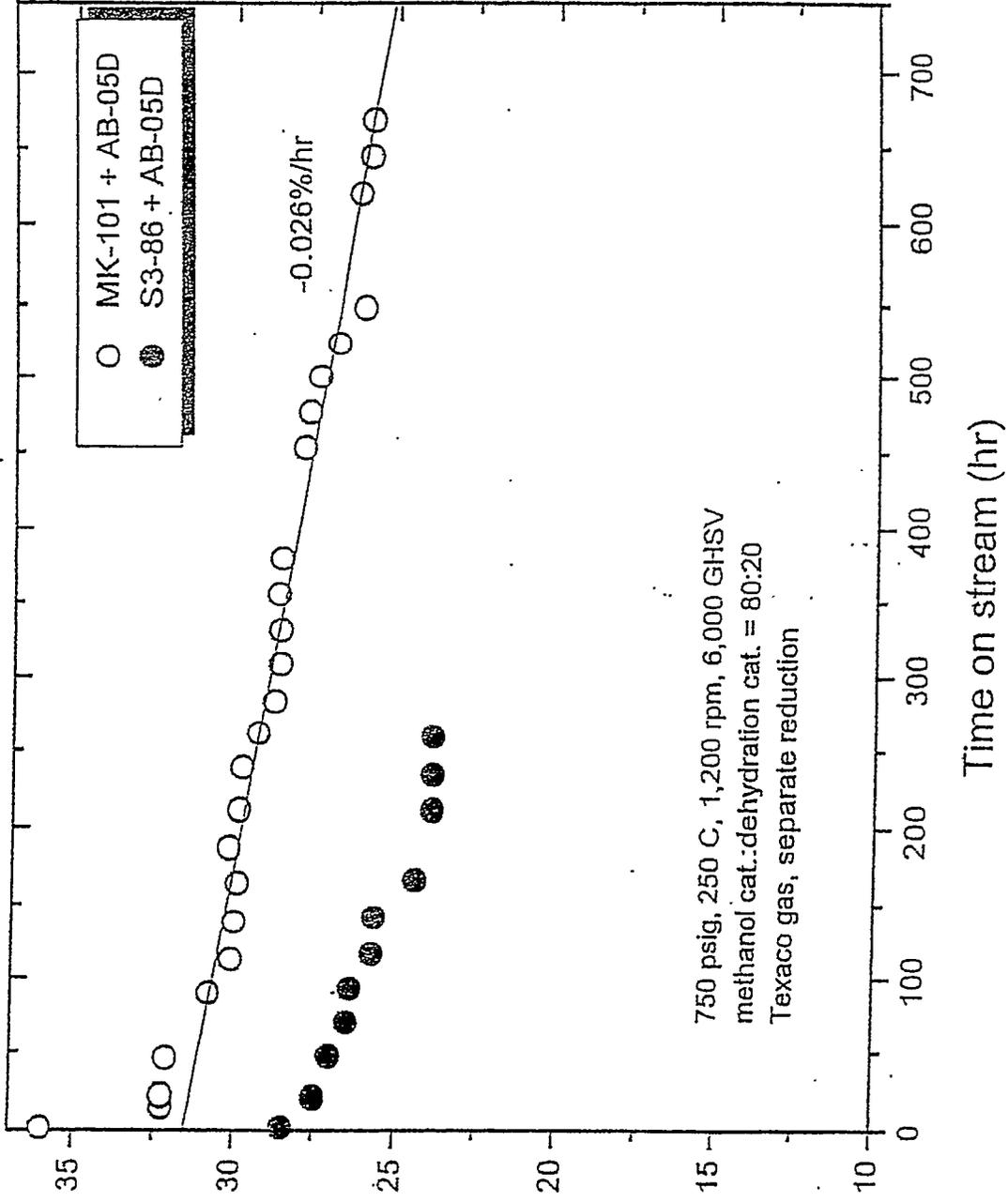


FIGURE 3

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

FIGURE 4



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

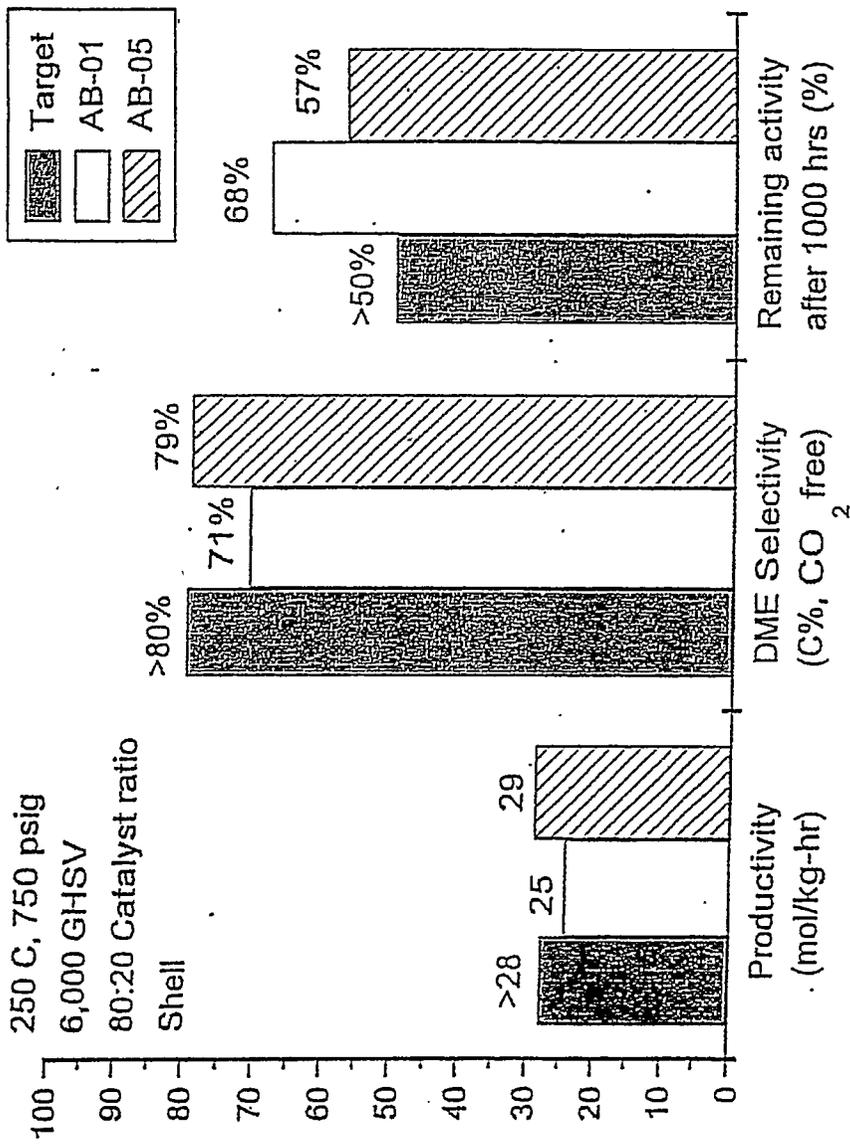


FIGURE 5

APPENDIX E - SAMPLES OF DETAILED MATERIAL BALANCE REPORTS

RUN NO:

K6-01/02

TITLE: Catalyst Addition and Aging

Balance Period:

Start Date
End Date

02Jan98 07:00:00
03Jan98 07:00:00

Time From Start of Run (days)

Start
End

13.3
14.3

Reaction Conditions

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

454
680
9601
0.77

Slurry Data

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

19,500
31.0
53.5
52.5
2249

Performance Results

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

279.8
267.2
40.7
1.14

(gas measurements)
(liquid measurements)

Energy Balance

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

21015
203
1687
24.2
49.7
1929
153

Syngas Conversion (% LHV)

79.8

Atom/Mass Balance Closure (% of reactor inlet)

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

68,296
2.50
35.73
0.119
4.58

C
H
O
N
Total Mass

99.42
99.91
98.85
102.25
99.19

Liquid Product Analysis (wt%)

1/2
19:00

1/3
7:00

1/2
19:00

1/3
7:00

CRUDE PRODUCT

Methanol
Ethanol
Water
Oil
Total

99.96
0.01
0.03
0.00
100.00

99.96
0.01
0.03
0.00
100.00

91.73
0.10
8.13
0.06
100.02

91.93
0.10
8.00
0.06
100.09

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

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

RUN NO: K3-02/08

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

Balance Period:

Start Date	08feb98 19:00:00
End Date	09feb98 07:00:00

Time From Start of Run (days)

Start	50.8
End	51.3

Reaction Conditions

Temperature (°F)	437
Pressure (psig)	710
Space Velocity (sL/kg-hr)	7216
Vg (inlet)	0.69

Slurry Data

Catalyst Weight (lb oxide)	24,800
Slurry Concentration (wt %)	33.7
Slurry Level (ft)	62.0
Gas Holdup (vol %)	54.2
Gassed Slurry Volume (ft3)	2611

Performance Results

Raw MeOH Production (ton/day)	211.7	(gas measurements)
Raw MeOH Production (ton/day)	199.7	(liquid measurements)
Syngas Utilization (SCF/lb MeOH)	44.2	
Catalyst Life (eta)	0.89	

Energy Balance

CO Conversion (total) (%)	12.5
CO Conversion to MeOH (%)	12.4
CO Conversion to H2 (%)	-0.03
Syngas Conversion (% LHV)	72.1
Syngas Usage (BTU/gallon MeOH)	68,422
Recycle Ratio	3.12
MeOH Productivity (g/mol/kg-hr)	21.15
Rxr Volumetric Productivity (ton/day-ft3)	0.076
Spatger "K"-value	4.78

Atom/Mass Balance Closure (% of reactor inlet)

C	99.64
H	100.90
O	99.70
N	100.01
Total Mass	99.78

Liquid Product Analysis (wt%)

Methanol	99.20	99.27	99.30
Ethanol	0.26	0.24	0.25
Water	0.34	0.30	0.29
Oil	0.20	0.20	0.20
Total	100.00	100.01	100.04

FUEL-Grade PRODUCT

2/8	2/9	2/9
21:26	0:13	5:42

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

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

RUN NO: K6-03/07

TITLE: Catalyst Addition and Aging

Balance Period:

Start Date
End Date

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

Time From Start of Run (days)

Start
End

77.3
78.3

Reaction Conditions

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

437
710
6295
0.67

Slurry Data

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

27,450
33.5
58.5
45.8
2462

Performance Results

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

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

Energy Balance

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

16796
195
3812
21.4
54.7
2123
144

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

81.9
67,536
3.04
20.93
0.089
5.50

Atom/Mass Balance Closure (% of reactor inlet)

C
H
O
N
Total Mass

100.23
101.04
99.81
100.95
100.26

Liquid Product Analysis (wt%)

REFINED PRODUCT

CRUDE PRODUCT

Methanol
Ethanol
Water
Oil
Total

37
19:00
99.99
0.01
0.00
0.00
100.00

3/8
7:00
99.99
0.09
11.20
0.06
100.11

3/8
7:00
88.99
0.09
10.96
0.06
100.10

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

	FRESH FEED	CO MAKEUP	H2 MAKEUP	K-01 OUTLET	REACTOR FEED	C-05 OUTLET	MAIN PURGE	DISTILL. PURGE	CRUDE PRODUCT	REFINED PRODUCT
T	87	58	59	269	283	244	90	64	221	79
P	745	867	690	738	731	702	691	6	185	138
Comp (mol %)										
H2	67.57	1.93	77.66	75.23	73.12	67.79	75.23	8.00	0.00	0.00
CO	29.22	97.05	9.12	12.82	16.91	11.58	12.82	7.00	0.00	0.00
N2	0.43	0.97	6.50	4.91	3.80	4.46	4.91	2.00	0.00	0.00
CH4	0.09	0.05	0.92	1.03	0.80	0.94	1.03	0.00	0.00	0.00
CO2	2.69	0.00	3.76	5.01	4.41	4.67	5.01	62.00	0.00	0.00
DME	0.00	0.00	0.01	0.00	0.00	0.01	0.00	4.00	0.00	0.00
MeOH	0.00	0.00	0.06	0.29	0.56	9.40	0.29	10.00	81.80	99.99
EIOH	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.06	0.01
H2O	0.00	0.00	0.00	0.00	0.00	0.51	0.00	0.00	18.14	0.01
others	0.00	0.00	1.97	0.69	0.41	0.64	0.69	7.00	0.01	0.00
TOTAL	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Mole Wt	10.857	27.501	7.770	9.161	9.647	11.293	9.161	39.217	29.523	32.040
LHV	203.4	0.0	0.0	572.1	777.5	756.0	16.0	0.7	45.6	121.0
Enthalpy	-35.31	0.00	0.00	-80.80	-114.07	-139.95	-2.47	-1.41	-19.81	-42.81
Flow	728,230	0	0	2,211,719	2,924,595	2,491,955	61,909	4,132	72,675	157,909
lbmol/hr	1,919.7	0.0	0.0	5,830.4	7,709.6	6,569.1	163.2	10.9	191.6	416.3
lb/hr	20,861	0	0	53,410	74,375	74,183	1,495	427	5,656	13,337

APPENDIX F - RESULTS OF DEMONSTRATION PLANT OPERATION

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

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

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

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

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

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

Figure 1

Catalyst Age (η)

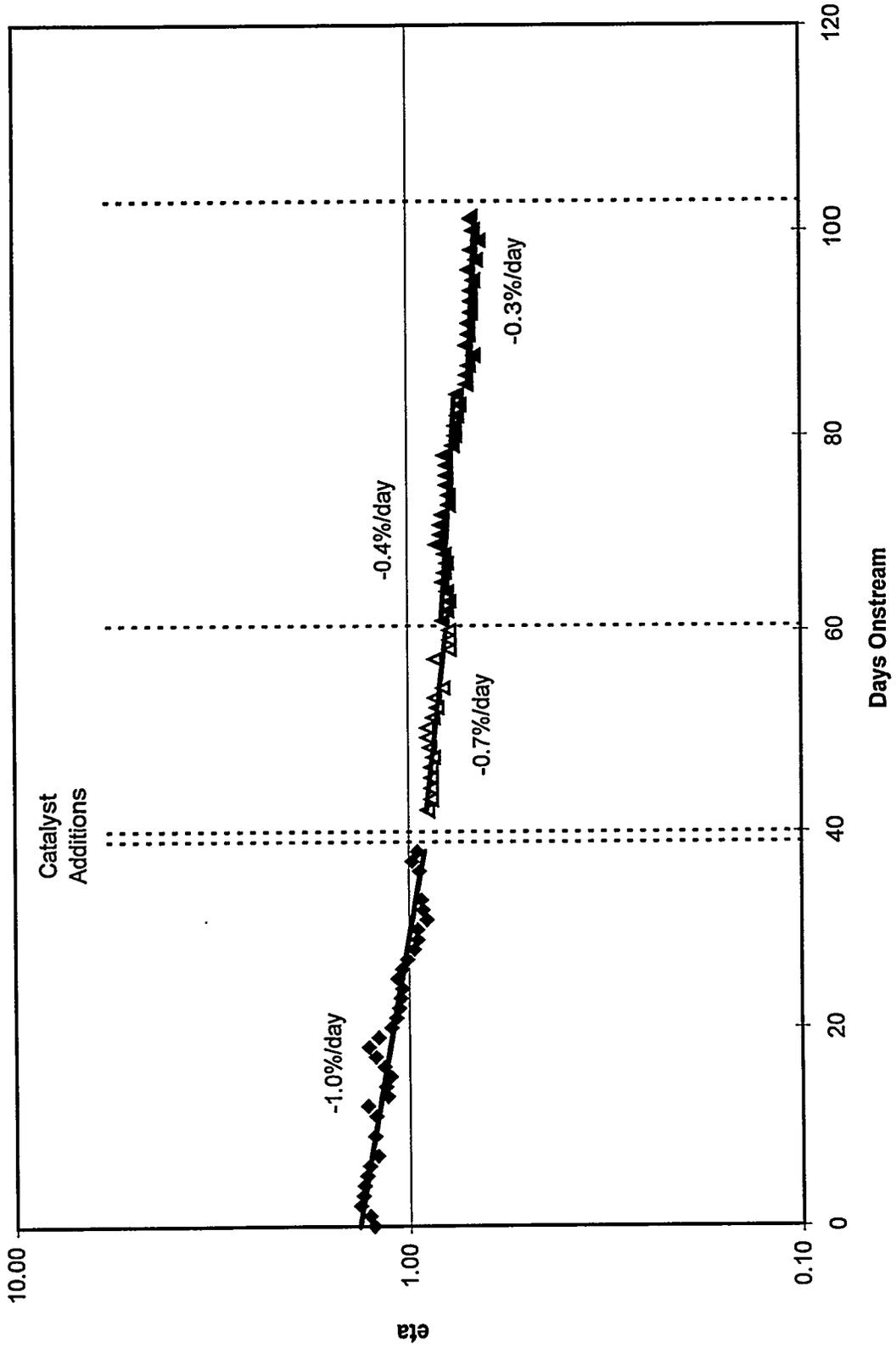


Figure 2

Sparger Resistance Coefficient (Post-December 1997 Restart)

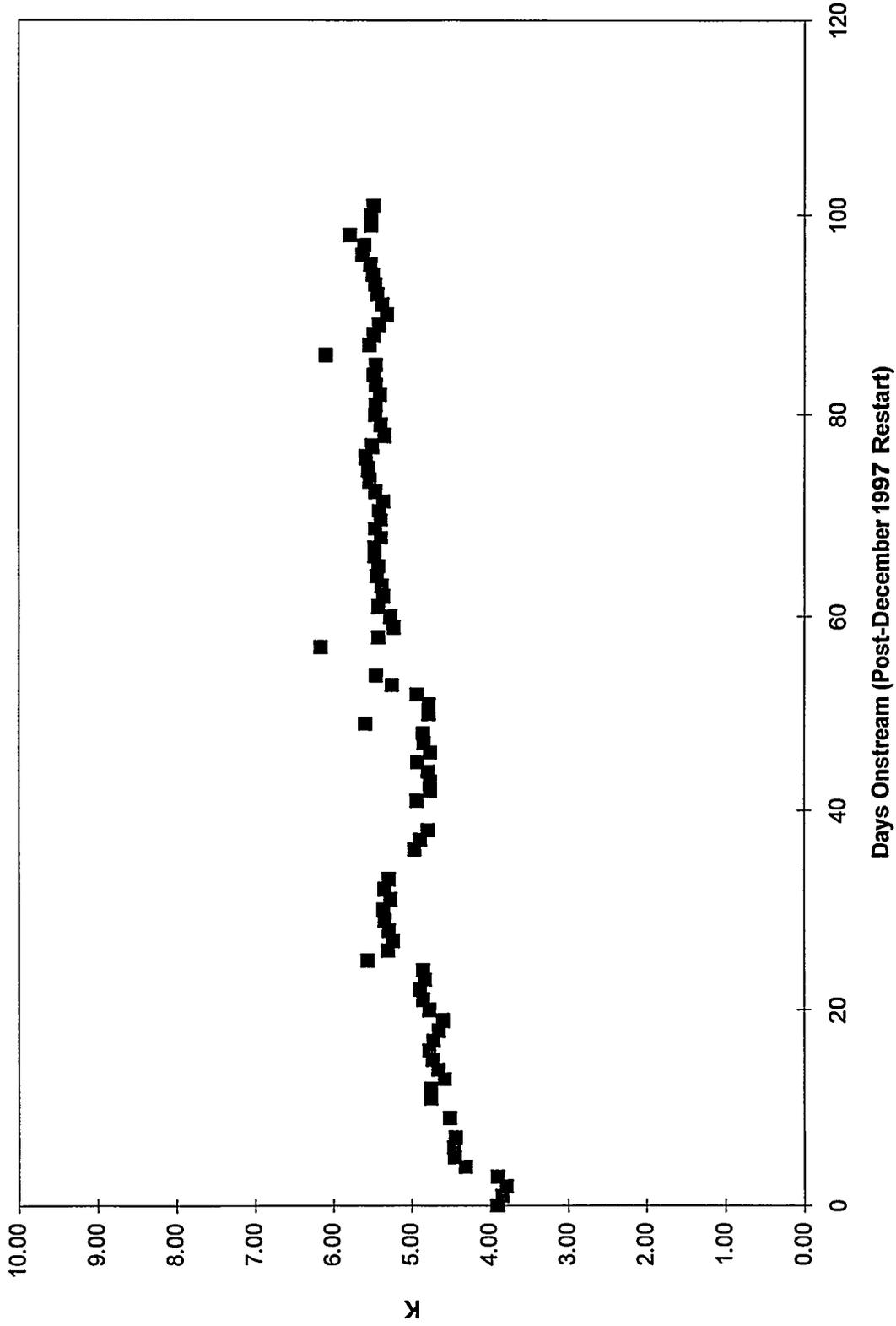
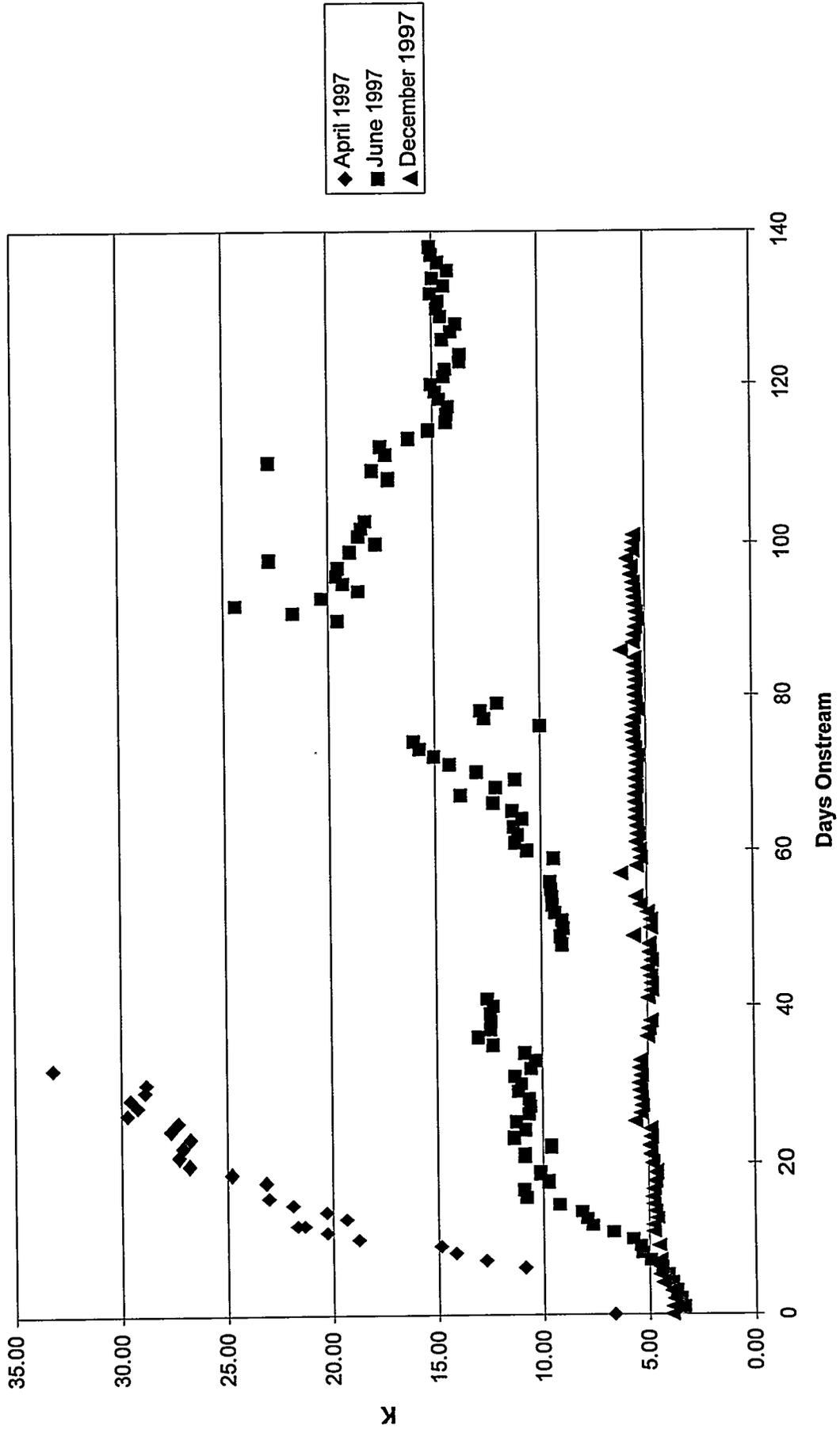


Figure 3

Sparger Resistance Coefficient (since April 1997 Startup)



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

Analysis Report

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

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

SUMMARY:

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

PROBLEM DEFINITION:

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

ANALYTICAL PROCEDURES:

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

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

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

Table 1

Concentration of Impurities (weight percent)

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

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

Table 2

Concentration of Impurities (weight percent)

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

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

Table 3

Operating Conditions for Gas Chromatographic Analysis

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

Oven Temperature Program

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

Carrier Gas Helium

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

Injector Temperature 250 °C

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

Detector Type Flame Ionization Detector

Detector Temperature	300 °C
Detector Range	1

Analysis Report



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

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

SUMMARY:

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

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

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

ANALYTICAL PROCEDURES:

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

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

Analyst:CMS

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

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

LIQUID PHASE METHANOL DEMONSTRATION - PHASE III

Task Name	Start Date	Duration (Months)	End Date	Percent Complete
Task 2.1.1 - Process Shutdown and Catalyst Aging				
Test 1 - Initial Shutdown, and Design Production Tests	4/2/97	68.00W	7/22/98	74%
Test 2 - Gassed Slurry Level	4/2/97	5.43W	5/10/97	100%
Test 3 - Reactor Feed: Toxaco-Type Syngas	4/2/97	28.28W	10/17/97	100%
Test 4 - Early Testing @ High Superficial Velocity	2/8/98	0.71W	2/13/98	100%
Test 5 - Check @ Test 1 Conditions	2/13/98	3.00W	3/6/98	0%
Test 6 - Catalyst Addition and Aging	6/10/97	3.43W	7/1/97	100%
Test 7 - Free-Drain Entrained/ Condensed Oil to Reactor	1/27/98	13.25W	4/30/98	66%
Test 8 - Operation @ Design Feed Gas Rates	4/2/97	53.00W	4/8/98	87%
Test 9 - Check for Limitation on Catalyst Slurry Concentration	5/1/98	2.00W	5/15/98	0%
Test 10 - Catalyst Addition to Reach Max Productivity	5/15/98	7.00W	7/3/98	0%
Task 2.1.2 - Process Operational Test Phase				
Test 11 - Catalyst Addition/ Withdrawal Test	7/22/98	105.00W	7/25/00	1%
Test 12 - Test 11 Conditions with No CO Make-up	7/22/98	6.00W	9/2/98	0%
Test 13 - Test 11 Conditions with No H2 Make-up	9/2/98	2.00W	9/16/98	0%
Test 14 - Test 11 Conditions with No H2 or CO Make-up	9/16/98	3.00W	10/7/98	0%
Test 15 - Repeat of Test 11 Conditions	10/7/98	2.00W	10/21/98	0%
Test 16 - Design Fresh Feed Operation Test	10/21/98	2.00W	11/4/98	0%
Test 17 - Testing @ High Superficial Velocity	11/4/98	3.00W	11/25/98	0%
Test 18 - Turndown and Ramping	11/25/98	2.00W	12/9/98	0%
Test 19 - Load-Following, Cyclone, & On/Off Tests	12/9/98	4.00W	1/6/99	0%
Test 20 - Reactor Feed: Toxaco-Type Syngas	1/6/99	7.00W	2/24/99	0%
Test 21 - Reactor Feed: Destec-Type Syngas	2/24/99	4.00W	3/24/99	0%
Test 22 - Reactor Feed: BGL-Type Syngas	3/24/99	3.00W	4/14/99	0%
Test 23 - Repeat of Test 15 Conditions	4/14/99	3.00W	5/5/99	10%
Test 24 - Reactor Feed: Nat. Gas Reforming-Type Syngas	5/5/99	3.00W	5/26/99	0%
Test 25 - Reactor Feed: Shell-Type Syngas with Steam Injection and 1:1 Recycle	5/26/99	3.00W	6/16/99	0%
Test 26 - Repeat of Test 15 Conditions	6/16/99	3.00W	7/7/99	0%
Test 27 - Repeat of Test 16 Conditions	7/7/99	3.00W	7/28/99	0%
Test 28 - Reactor Operation @ 260 deg C	7/28/99	2.00W	8/11/99	0%
Test 29 - Repeat of Test 26 Conditions	8/11/99	2.00W	8/25/99	25%
Test 30 - Reactor Inspection - Then, Continue with Alternative Catalyst	8/25/99	2.00W	9/8/99	0%
Test 31 - Plant Shutdown	9/8/99	4.00W	10/6/99	0%
Test 32 - Reactor Feed: Toxaco-Type Syngas	10/6/99	6.00W	11/17/99	0%
Test 33 - Catalyst Aging	11/17/99	3.00W	12/8/99	0%
Test 34 - Catalyst Addition/ Withdrawal to Achieve Target Slurry Concentration	12/8/99	22.00W	5/10/00	0%
Test 35 - Reactor Feed: Toxaco-Type Syngas	5/10/00	6.00W	6/21/00	0%
Task 2.1.3 - Extended Optimum Operation				
Test 36 - Stable Operation	6/21/00	5.00W	7/26/00	0%
Test 37 - Commercial Test Run	7/26/00	35.00W	3/28/01	0%
	1/31/01	27.00W	1/31/01	0%
	3/28/01	8.00W	3/28/01	0%

COPY

U.S. DEPARTMENT OF ENERGY COST MANAGEMENT REPORT

1. TITLE	2. REPORTING PERIOD		3. IDENTIFICATION NUMBER		4. ESTIMATED ACCRUED COSTS		5. COST PLAN DATE		6. START DATE		7. COMPLETION DATE		8. ELEMENT		9. REPORTING ELEMENT		10. ACCRUED COSTS		11. ESTIMATED ACCRUED COSTS		12. Total Plan Value		13. Variance						
	a. Actual	b. Plan	c. Actual	d. Plan	a. Subsequent Reporting Period	b. Balance of Fiscal Year	c. FY 1999 (1)	d. FY 2000 (2)	e. FY 2001 (3)	d. Subsequent FYs (4)	Total	Total	Total	a. Actual	b. Plan	c. Actual	d. Plan	a. Subsequent Reporting Period	b. Balance of Fiscal Year	c. FY 1999 (1)	d. FY 2000 (2)	e. FY 2001 (3)	d. Subsequent FYs (4)	Total	Total	Total	Variance		
	0	0	16,304	16,304	0	0	0	0	0	0	0	0	0	Prior to Mod 2		0	0	0	0	0	0	0	0	0	16,304	16,304	0	0	
1.1.1	0	0	1,011	1,011	0	0	0	0	0	0	0	0	0	Project Definition		0	0	0	0	0	0	0	0	0	1,011	1,011	0	0	
1.1.2	0	0	248	248	0	0	0	0	0	0	0	0	0	Permitting		0	0	0	0	0	0	0	0	0	248	248	0	0	
1.1.3	15	0	10,895	10,895	0	0	0	0	0	0	0	0	0	Design Engr.		0	0	0	0	0	0	0	0	0	10,895	10,895	0	0	
1.1.4	15	106	164	541	54	646	0	0	0	0	0	0	0	Off-site Testing		54	646	0	0	0	0	0	0	0	864	864	0	0	
1.1.5	0	4	3,001	3,018	2	27	0	0	0	0	0	0	0	Planning, Admin., & DMB Verif. Testing		2	27	0	0	0	0	0	0	0	3,030	3,030	0	0	
1.2.1	0	0	10,232	10,226	0	0	0	0	0	0	0	0	0	Procurement		0	0	0	0	0	0	0	0	0	10,232	10,226	6	6	
1.2.2	0	0	11,730	11,728	0	0	0	0	0	0	0	0	0	Construction		0	0	0	0	0	0	0	0	0	11,730	11,728	2	2	
1.2.3	0	46	585	864	46	279	0	0	0	0	0	0	0	Train. & Commissioning		46	279	0	0	0	0	0	0	0	864	864	0	0	
1.2.4	81	67	117	398	66	612	0	0	0	0	0	0	0	Off-Site Test - Proc. & Constr.		66	612	0	0	0	0	0	0	0	795	795	0	0	
1.2.5	0	2	954	966	2	22	0	0	0	0	0	0	0	Planning & Admin		2	22	0	0	0	0	0	0	0	978	978	0	0	
1.3.1	1	0	1,507	1,497	0	0	0	0	0	0	0	0	0	Startup		0	0	0	0	0	0	0	0	0	1,507	1,497	10	10	
1.3.2	0	0	0	0	0	0	0	0	0	0	0	0	0	Operations		0	0	0	0	0	0	0	0	0	0	0	0	0	
1.3.2.1	2,137	2,374	26,249	27,595	3,154	13,217	34,635	36,137	33,489	0	0	0	0	Methanol Operation		3,154	13,217	34,635	36,137	33,489	0	0	0	0	0	146,822	146,862	(40)	(40)
1.3.2.2	0	0	0	0	0	0	560	1,207	0	0	0	0	0	DMB Design, Mod., Oper.		0	0	560	1,207	0	0	0	0	0	1,767	1,767	0	0	
1.3.2.3	0	0	0	0	0	0	0	0	0	0	0	0	LPMEOH Dismantlement		0	0	0	0	0	0	0	0	0	0	0	0	0	0	
1.3.3	0	0	0	0	0	0	0	0	0	0	0	0	0	On-Site Product Use Demo		0	0	0	0	0	0	0	0	0	0	0	0	0	
1.3.4	0	2	0	12	2	22	24	1,595	45	0	0	0	0	Off-Site Product Use Demo		2	22	24	1,595	45	0	0	0	0	5	5	0	0	
1.3.5	14	3	148	85	4	34	47	49	14	0	0	0	0	Data Analysis & Reports		4	34	47	49	14	0	0	0	0	1,688	1,688	0	0	
1.3.6	35	81	903	947	81	449	1,149	652	936	0	0	0	0	Planning & Admin.		81	449	1,149	652	936	0	0	0	0	4,170	4,170	0	0	
14. TOTAL	2,297	2,685	84,069	86,335	3,365	15,308	36,415	39,645	34,956	0	0	0	0			3,365	15,308	36,415	39,645	34,956	0	0	0	0	213,700	213,700	0	0	

17. SIGNATURE OF PARTICIPANT'S AUTHORIZED FINANCIAL REPRESENTATIVE AND DATE
[Signature] 4/13/98
 DATE

16. SIGNATURE OF PARTICIPANT'S PROJECT MANAGER AND DATE
[Signature] 4/13/98
 DATE

15. DOLLARS EXPRESSED IN:
 Thousands