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SYNTHESIS OF OCTANE ENHANCERS DURING SLURRY-PHASE FISCHER-TROPSCH. QUARTERLY TECHNICAL PROGRESS REPORT NO. 7, APRIL 1, 1992--JUNE 30, 1992

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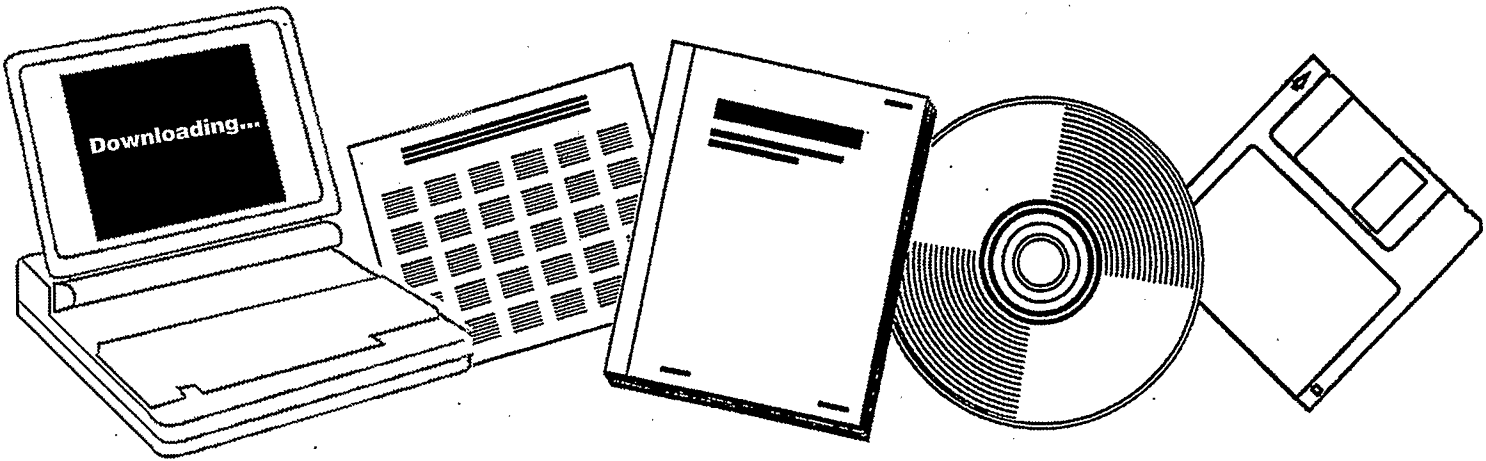
PITTSBURGH, PA

24 SEP 1992



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DE93 007556

QUARTERLY TECHNICAL PROGRESS REPORT No. 7

Covering the Period April 1, 1992 to June 30, 1992

SYNTHESIS OF OCTANE ENHANCERS DURING SLURRY-PHASE FISCHER-TROPSCH

Prepared for the U.S. Department of Energy Under
Contract No. DE-AC22-90PC90047

September 24, 1992

MASTER

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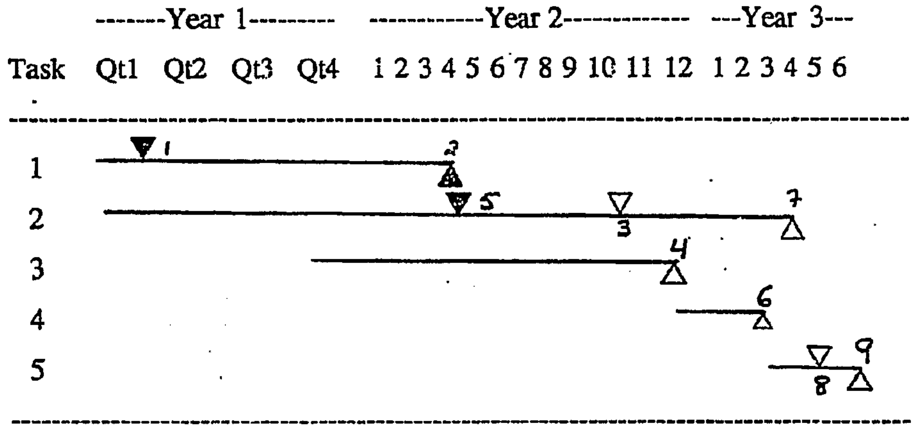
PLANNED TASK AND MILESTONE SCHEDULE



M = denotes milestone

List of Milestones

Number	Description
M1	Finalize design of slurry bubble column reactor (SBCR).
M2	Finalize construction and testing of SBCR. Topical report prepared.
M3	Completion of catalyst screenings for study of i-butylene addition during formation of alcohols. Decision made on catalysts to be studied in SBCR.
M4	Completion of slurry reaction of i-butylene addition during formation of alcohols.
M5	Completion of catalyst screenings for study of i-butylene addition to FT liquid products using acid catalysts. Decision made on catalysts to be studied in SBCR.
M6	Completion of slurry reaction of i-butylene addition to FT liquid products using acid catalysts.
M7	Completion of catalyst screenings.
M8	Completion of slurry reaction of alcohol addition during iso-olefin synthesis.
M9	Final report issued.

OBJECTIVES

The objective of this project is to investigate three possible routes to the formation of ethers, in particular methyl tert-butyl ether (MTBE), during slurry phase Fischer-Tropsch reaction. The three reaction schemes to be investigated are:

- Addition of i-butylene during the formation of methanol and/or higher alcohols directly from CO and H_2 during slurry-phase Fischer-Tropsch.
- Addition of i-butylene to FT liquid products including alcohols in a slurry-phase reactor containing an MTBE or other acid catalyst.
- Addition of methanol to slurry phase FT synthesis making iso-olefins.

WORK ACCOMPLISHED THIS QUARTER

During the seventh quarter we continued the shake down experiments for the SBCR and conducted an initial aborted run. We have also re-started experiments on Scheme 1, i.e., the addition of iso-butylene during CO hydrogenation. Using a dual bed arrangement, we have demonstrated the synthesis of MTBE from syngas and iso-butylene.

SEVENTH QUARTER'S TECHNICAL REPORT

1. OPERATION OF SLURRY BUBBLE-COLUMN REACTOR

The slurry bubble column reactor is completed and operational. The final design was described in the Sixth Quarterly Technical Report. Preliminary runs were attempted for the synthesis of MTBE from methanol and iso-butylene using one of the catalysts identified in the catalyst screening experiments, mainly the LZ210-12 zeolite. Approximately 25 g of the catalyst were dispersed in Synfluid (Chevron Chemicals) using nitrogen as an inert dispersing gas. When the catalyst was in the slurried form and with the reactor at a temperature of 100°C, i-butylene was introduced in the liquid form by pumping it using an HPLC pump from an inverted pressurized cylinder. Unfortunately, this approach did not succeed as we were not able to obtain a stable i-butylene flow and thus the run was aborted. We experienced similar problems in the fixed-reactor screenings (see Section 2.2.1) and these were resolved by utilizing a mixture of i-butylene in helium and regulating the flow with a gas mass-flow controller. We have thus modified the SBCR to operate in this manner, ordered the required gas mixture, and expect to resume the runs shortly.

2. ETHER SYNTHESIS DURING FT REACTION

2.1. INTRODUCTION

One of the proposed schemes for the direct synthesis of MTBE from syngas involves the incorporation of i-butylene during methanol synthesis from syngas. This incorporation can be envisioned to take place in one of two ways: by interception of a methanol reaction intermediate by i-butylene at a methanol synthesis site; or by secondary reaction of methanol and i-butylene on an acid site. The purpose of this portion of the work is to test each of these approaches to the synthesis of MTBE.

Because the formation of MTBE from methanol and i-butylene is thermodynamically unfavored at high temperatures, we have concentrated our work in low temperature alcohol synthesis catalysts, such as Rh or Pd-based catalysts. Some of these catalysts were promoted with either Mo (to increase the methanol yield) or alkali metals (to reduce the hydrogenation activity). The latter promotion was found necessary to reduce the undesirable hydrogenation of i-butylene to i-butane. Composite catalysts containing acid sites, suitable for secondary synthesis of MTBE, consisted of physical mixtures of the low-temperature methanol synthesis catalyst and a zeolite material.

This section of the report details some of the work in this area.

2.2. EXPERIMENTAL

2.2.1. Reaction System

Various modifications have been done to the reaction system which was originally described in the Third Technical Quarterly Report. These modifications were primarily for the purpose achieving a stable i-butylene injection flow and for improved product analysis. A schematic of the current experimental setup is shown in Figure 1. The i-butylene (IB) can be either be pumped as a liquid into the syngas mixture with a HPLC liquid pump capable of delivering liquid at a flow rate as low as 0.01 cc/min, or by using a gas mass flow controller and a pre-mixed gas mixture of i-butylene in He when very low

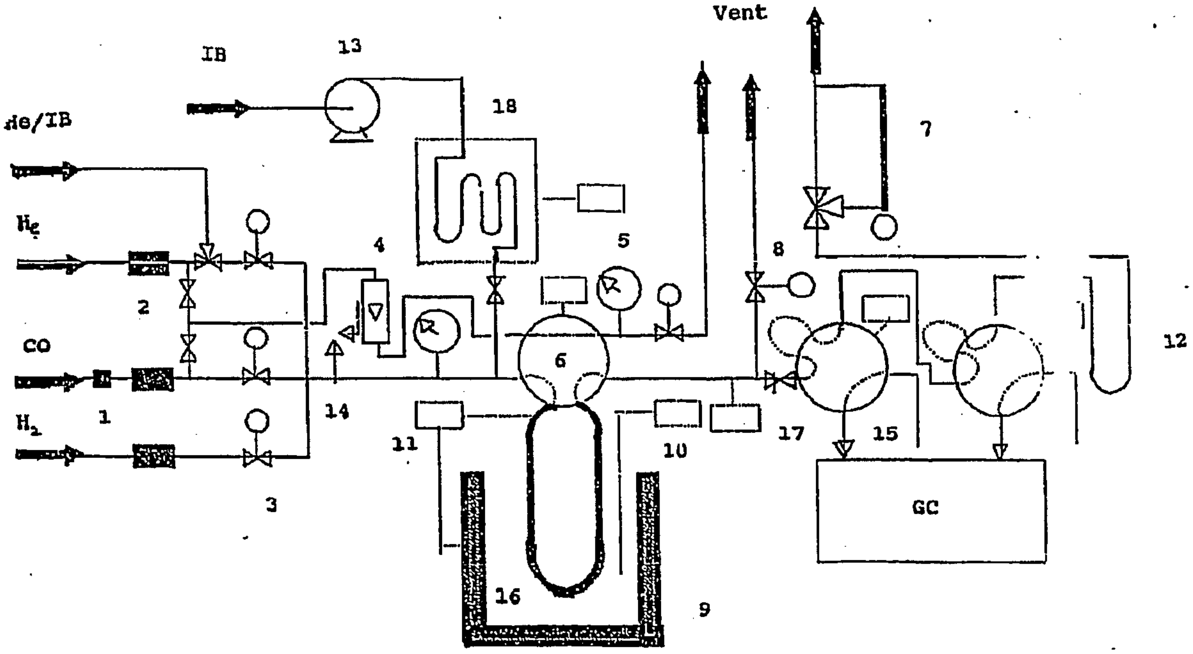


Figure 1. Schematic diagram of reactor system.

Legend:

1. Deoxo unit
2. Mol-sieve trap
3. Mass flow controller
4. Rotometer
5. Pressure gauge
6. Six-port valve
7. Bubble flow meter
8. Pressure regulator
9. Furnace
10. Temperature control
11. Thermocouple
12. Liquid trap
13. HPLC pump
14. Pressure relief
15. Six-port valve
16. Reactor
17. Leak valve
18. Heaters

IB concentrations are required. In practice, we have found the latter method to result in more stable flows and all the work reported in this report used such a gas mixture for delivering the i-butylene. All the lines in the reaction system downstream of the i-butylene injection point are heated to 175°C to prevent any reaction product and/or i-butylene condensation. The reactor can be bypassed during the stabilization period of the i-butylene flow by using a six-port valve. During the bypass period the catalyst can be kept under H_2 or inert gas (He) flow. Thermocouples installed at various sections of the reactor system are used to monitor the temperature.

The reaction was carried out by operating the reactor in a differential mode, i.e., CO conversion was kept below 5%. Prior to reaction, the catalysts were reduced in-situ. CO hydrogenation reaction was carried out under following set of conditions:

Temperature:	150 - 250°C
Pressure:	1 - 8 atm.
H_2/CO ratio:	1.0
Syngas flow rate:	30 - 100 cc/min
Catalysts weight:	0.4 - 0.5 g

Conversions and selectivities were determined either at steady-state for each temperature (typically after about three hours), or at initial conditions. For the latter the analysis was taken after the reaction had been on-stream for ten minutes. Between analyses the catalyst was taken off-line and bracketed with hydrogen.

The co-addition of i-butylene during CO hydrogenation was also studied over various FTS catalysts. For these studies, the flow of IB/He/CO/H_2 was maintained at 0.06/0.54/15/15 cc/min.

All the products were analyzed on-line using a Varian 3700 gas chromatograph equipped with a capillary GS alumina PLOT (porous layer open tubular column) column and a Porapak-Q filled packed column with two flame ionization detectors. The first column (PLOT) was used for analysis of the light hydrocarbons while the second column (Porapak-Q) was used for oxygenates separation.

2.2.2. Preparation of Catalysts

2.2.2.1. Rh-Mo/SiO_2 and Rh-Mo/Al_2O_3

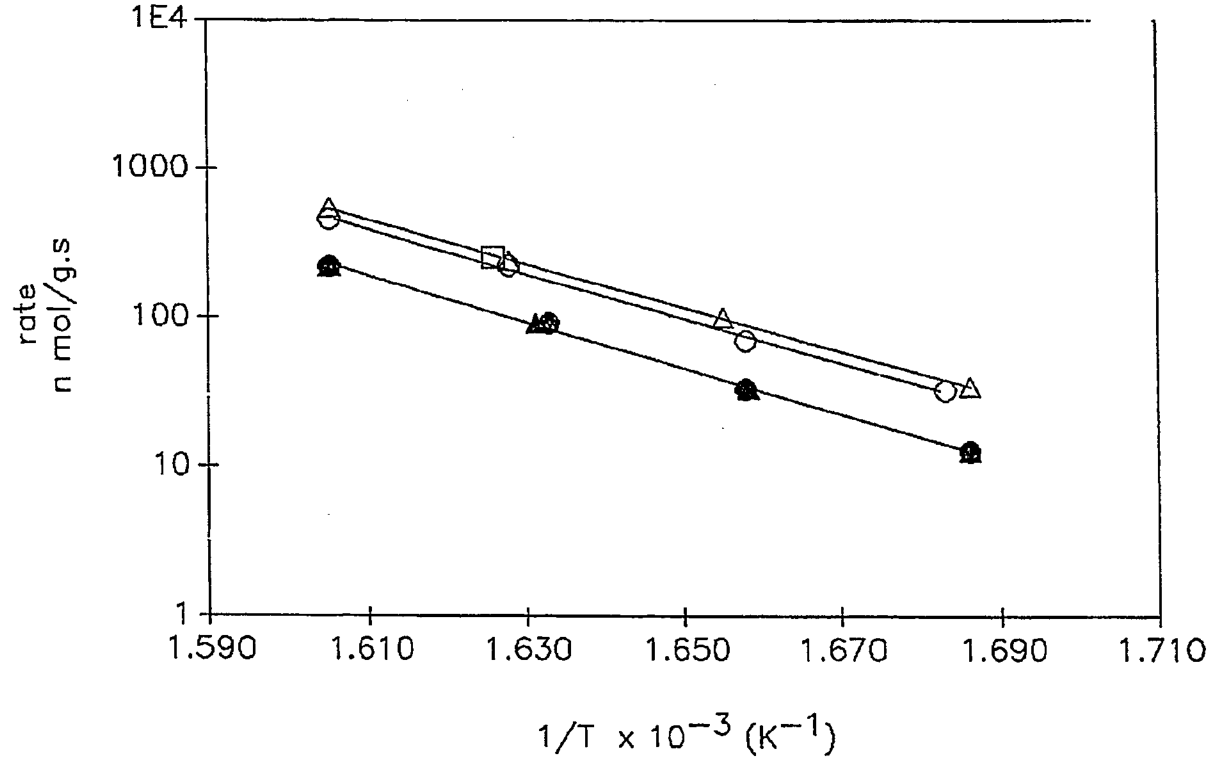
SiO_2 (Cab-O-Sil) was used as received. Molybdenum oxide promoted SiO_2 was prepared by incipient wetting of the SiO_2 support with an aqueous solution of $(NH_4)_6Mo_7O_{24}.4H_2O$ (Aldrich), drying at 110°C for 16 hours, and calcining at 450°C for 3 hours. The rhodium was then deposited by incipient wetness impregnation of an aqueous solution of $Rh(NO)_3$ and was subsequently dried in air at 110°C (16 hr). To remove nitrogenous residues from the precursor, the catalyst was calcined at 450°C (3 hr). The loading of Rh was 1.5 wt% and the Mo/Rh molar ratio was one.

The γ-Al_2O_3 used as support was prepared from Vista B alumina obtained from Vista Chemical Company. The Vista B was calcined in air at 500°C for a minimum of 5 hours. Analysis by XRD showed the characteristic γ-Al_2O_3 pattern after this treatment. The same impregnation procedure as described for Rh-Mo/SiO_2 was used for preparing this catalyst, except that $RhCl_3$ was used as the precursor for Rh.

2.2.2.2. Pd/SiO_2 and alkali promoted Pd/SiO_2

The promoted catalysts were prepared from a base, unpromoted catalyst to ensure similar Pd particle size distribution. A 5 wt% Pd/SiO_2 base catalyst was prepared by impregnating SiO_2 (Davison, grade 59) with an aqueous solution of $PdCl_2$ (Aldrich) using incipient wetness technique. Before impregnation, the SiO_2 was first ground and screened between 240 to 400 mesh screens. The impregnated catalyst was dried in an oven for 5 hours at 90°C and then calcined at 400°C in an air flow of 50 cc/min for four hours. The calcined catalyst was then reduced for 2 hours at 300°C, and 3 hours at 400°C in a 50 cc/min of H_2 flow. This procedure was used to prepare four 20 gm. batches of the base catalysts. To make sure the catalysts from all the batches are similar, ethane hydrogenolysis reaction was carried out over all batches[1,2]. One of the batches was found to be less active than other two and it was thus discarded. Arrhenius plots for ethane hydrogenolysis for all the batches of base catalysts are shown in Figure 2. The three

Figure 2. Arrhenius plot for ethane hydrogenolysis over 5% [illegible] SiO$_2$ catalysts. ●,▲: batch 1; ○: batch 2; △: batch 3; □:ba[illegible]



batches of base catalysts with similar activity were then mixed. Small portions of the base catalyst were modified by impregnating with an aqueous solution of various promoters precursors. These promoted catalysts were again reduced using the same procedure described above. Table 1 shows the various the promoted catalysts prepared by this method.

2.2.2.3. PdKY zeolite

Commercial NaY zeolite (Strem Chemicals) was exchanged three times with aqueous KNO_3 for 24 hours at 60°C. PdKY was then prepared by ion-exchanging the KY with a solution of $Pd(NH_3)_4(NO_3)_2$ (Aldrich) using the same procedure. After the last exchange, the solid was washed several times with deionized water and dried overnight at 60°C. The resulting catalyst was heated in air to 300°C for 18 hours and then reduced in flowing H_2 at 450°C for five hours.

2.2.2.4. Composite catalysts

A composite catalyst was prepared by physically mixing the Li-Pd/SiO_2 catalysts with the LZ210-12 HY zeolite reported in previous Quarterly Reports. The Pd catalyst and zeolite were mixed in a 5:1 weight ratio. Prior to reaction the catalyst was treated in-situ by heating in flowing hydrogen at 1°C/minute to 400°C.

2.2.3. Dual Bed Experiments

The reaction was also studied using a dual-bed arrangement of catalysts with the methanol synthesis function and the acid function separated. The dual bed consisted of a bed of 0.97 g of Li-Pd/SiO^2 followed by bed of 0.10 g of zeolite LZ210-12. The reaction was conducted at 180°C and 7 atm pressure. All other reaction conditions were as described in Section 2.2.1.

Table 1. List of Promoters Used for Pd/SiO_2 Catalysts

Promoter(M)	Precursor	Source	Purity	M/Pd
Li	$LiNO_3$	Aldrich	99.99%	1
Na	$NaNO_3$	"	99.995%	"
K	KNO_3	"	99.99%	"
Mg	$Mg(NO_3)_2.6H_{2O}$	"	99.95%	"
Ca	$Ca(NO_3)_2.xH_2O$	"	99.997%	"
La	$La(NO_3)_3.5H_2O$	"	99.0%	"

2.3. RESULTS AND DISCUSSION

2.3.1. CO Hydrogenation

Table 2 and Figure 3 summarize the steady-state reaction results obtained with the unpromoted Rh/Al_2O_3 at 8 atm. At temperatures between 200 and 240°C, the selectivity for oxygenates ranged between 38 and 53% and was primarily distributed among methanol, ethanol and acetaldehyde. Table 3 and Figure 4 show the results of a similar study using the $Rh\text{-}Mo/SiO_2$ catalyst. For this catalyst, the overall activity was higher by a factor of three and the selectivity for oxygenates increased somewhat. The most noticeable change is the increase in selectivity for methanol which was over 90% at temperatures below 200°C. Table 4 and Figure 5 shows the results for the same catalyst but at one atmosphere pressure. As expected, at the lower pressure both reaction rate and total oxygenate selectivity decreased. In all three cases the major hydrocarbon product formed was methane.

CO hydrogenation was also carried out over Pd based catalysts. These catalysts include Pd/SiO_2, $Li\text{-}Pd/SiO_2$, and $K\text{-}Pd/SiO_2$. K-Pd/SiO2 was found to have almost no activity at the temperature and pressure range of the study. This is probably because a K/Pd ratio (atomic) of one is too high and the promoter has completely covered the active reaction sites. Lower K/Pd ratios (0.1 or less) might be more suitable for this reaction.

Initial reaction data over Pd/SiO_2 and $Li\text{-}Pd/SiO_2$ catalysts at different temperatures are shown in Tables 5 and 6. In both the cases the main product was methanol and the main by-product was methane with only trace amounts of ethane. For both catalysts the selectivity for methanol was above 95% at all temperatures. The activity of both catalysts were comparable. Arrhenius plots for these reactions are plotted in Figures 6 and 7. The calculated activation energies are in agreement with the literature[3].

Table 2. Summary of for CO Hydrogenation Reaction over 5% Rh/Al_2O_3 at 8 atm.

Temperature, °C	200	220	240
CO Conversion, %	0.16	0.39	0.79
Rate, mol $kg^{-1}h^{-1}$	9	22	44
SELECTIVITIES			
Hydrocarbons	48.3	47.1	61.9
Oxygenates	51.7	52.9	38.1
OXYGENATES			
Methanol	54.5	40.1	26.0
Ethanol	19.8	36.3	49.6
Acetaldehyde	25.7	23.5	24.4

Figure 3

Arrhenius Plot for CO hydrogenation over 5% Rh/Al_2O_3 at 8 atm.

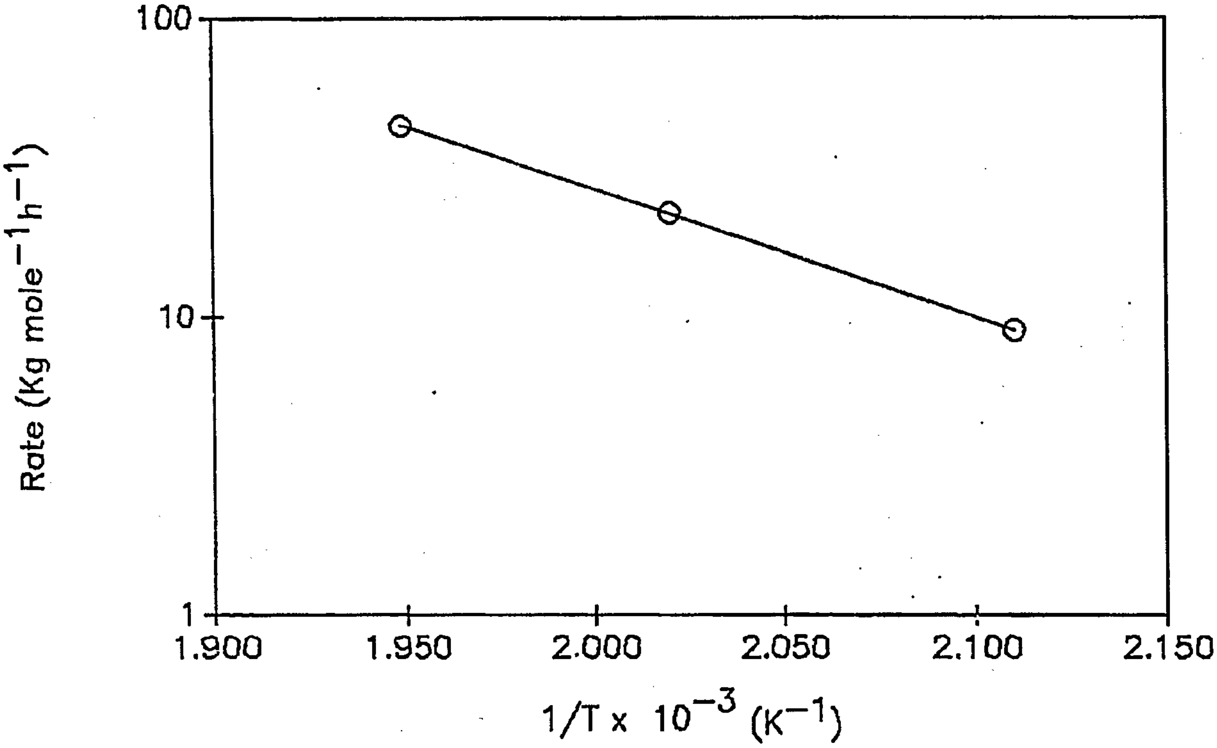


Table 3. Summary of CO Hydrogenation Reaction over 1.5% Rh-Mo/SiO_2 at 8 atm.

Temperature, °C	180	200	230	250
CO Conversion, %	0.35	0.53	2.25	2.75
Rate, mol $kg^{-1}h^{-1}$	20	29	98	150
SELECTIVITIES				
Hydrocarbons	29.0	26.1	44.1	55.0
Oxygenates	71.0	73.9	55.9	45.0
OXYGENATES				
Methanol	95.1	91.8	89.4	82.8
Ethanol	4.9	8.2	10.6	15.5
Acetaldehyde	0	0	0	1.6

Figure 4

Arrhenius Plot for CO hydrogenation over Rh–Mo/SiO_2
8 atm.

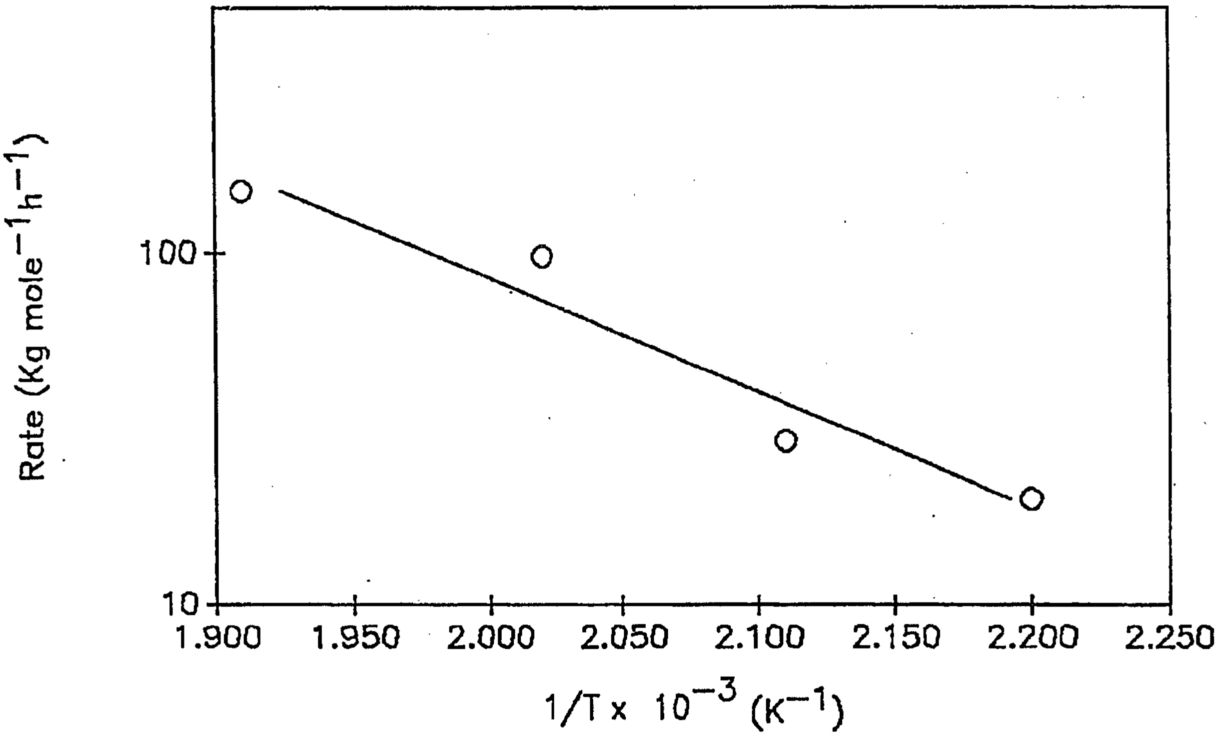


Table 4. Summary of CO Hydrogenation Reaction over 1.5% Rh-Mo/SiO_2 at 1 atm.

Temperature, °C	180	200	230	250
CO Conversion, %	0.11	0.26	0.61	0.75
Rate, mol $kg^{-1}h^{-1}$	6	15	34	42
SELECTIVITIES				
Hydrocarbons	23.2	47.6	70.6	86.9
Oxygenates	76.8	52.4	29.4	13.1
OXYGENATES				
Methanol	81.9	84.5	81.1	68.0
Ethanol	4.0	9.8	12.5	55.4
Acetaldehyde	14.2	5.6	6.4	6.6

Figure 5

Arrhenius Plot for CO hydrogenation over Rh–Mo/SiO_2
1 atm.

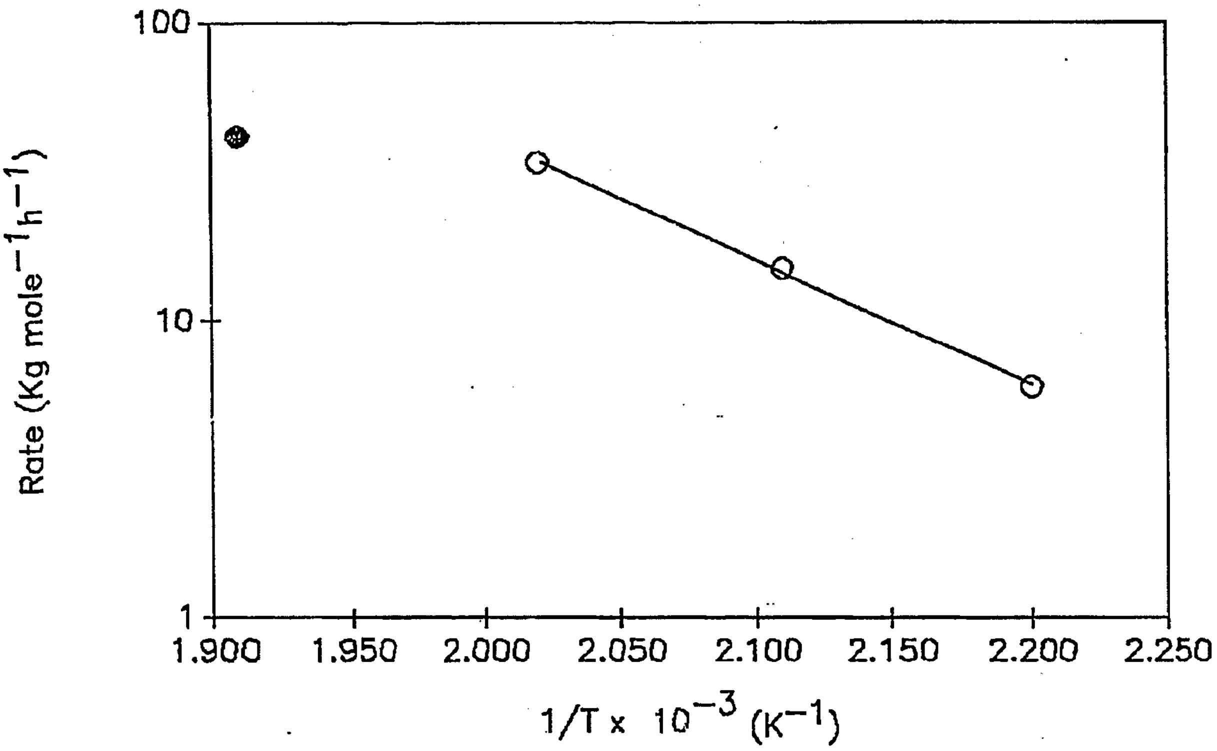


Table 5. Summary of CO Hydrogenation Reaction over 5% Pd/SiO_2 at 7 atm.

Temperature, °C	170	185	200
CO Conversion, %	0.05	0.15	0.37
Rate, mol $kg^{-1}h^{-1}$	4.7	13.6	33.8
SELECTIVITIES			
Methane	1.7	0.6	0.4
MeOH	98.3	99.4	99.6

Table 6. Summary of CO Hydrogenation Reaction over Li Promoted 5% Pd/SiO_2 at 7 atm.

Temperature, °C	155	170	185	200
CO Conversion, %	0.07	0.16	0.18	0.23
Rate, mol $kg^{-1}h^{-1}$	6.7	14.9	16.3	21.7
SELECTIVITIES				
Methane	6.0	2.8	2.4	2.0
MeOH	94.0	97.2	97.6	98.0

FIGURE 6

Arrhenius Plot for CO Hydrogenation over 5% Pd/SiO_2

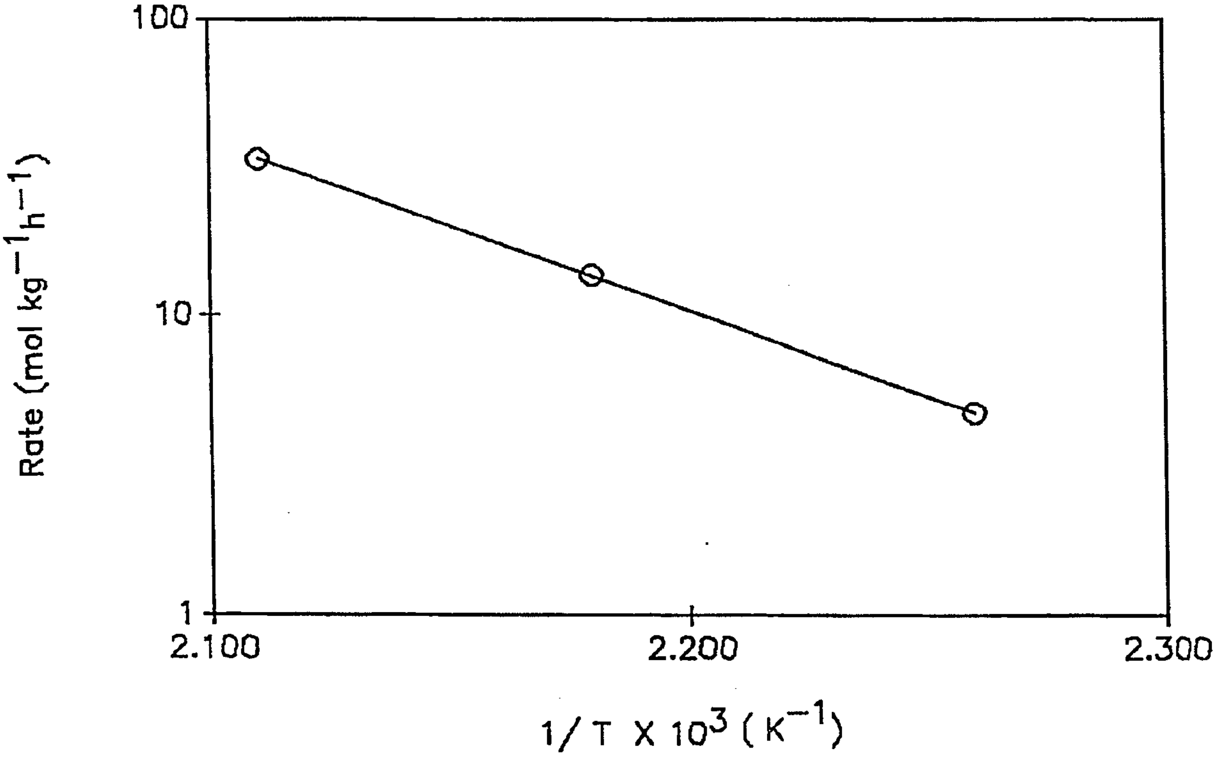
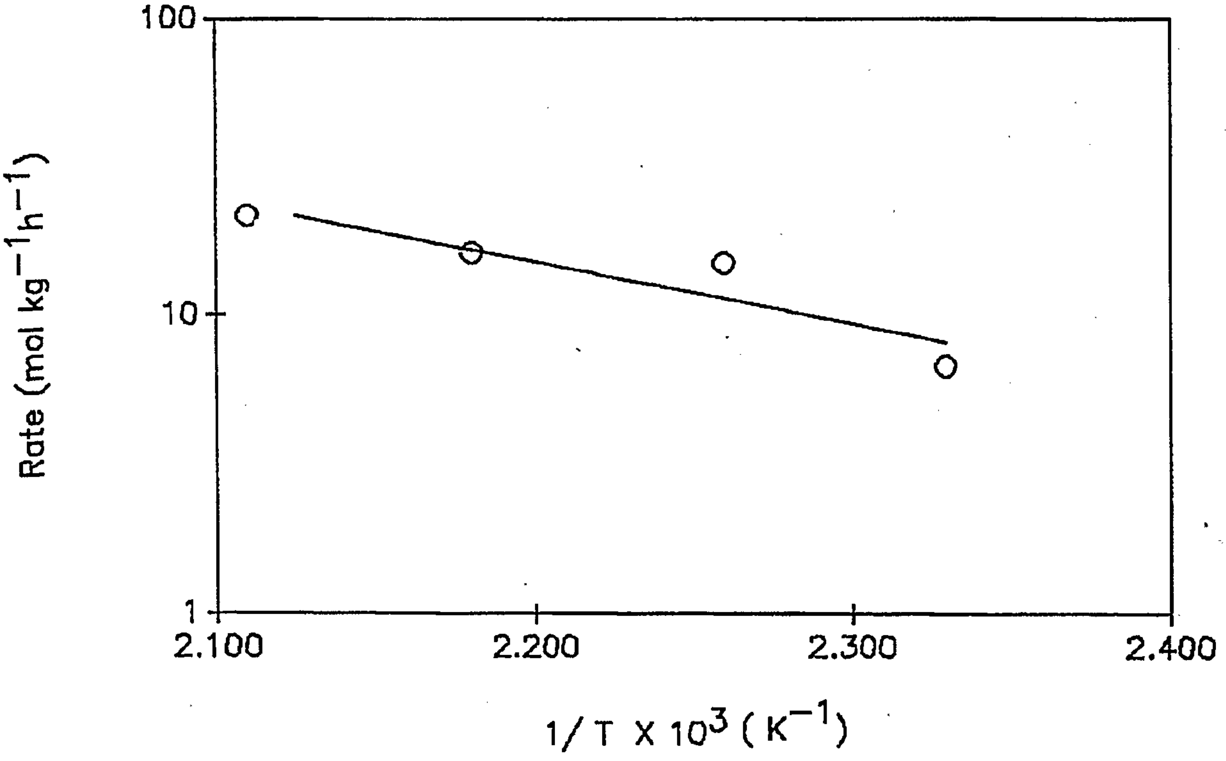


FIGURE 7

Arrhenius Plot for CO Hydrogenation over Li–Pd/SiO_2



Similar experiments were also conducted with the PdKY catalyst. It showed negligible activity in the temperature range of study and mainly produced methane at 250°C.

2.3.2. Addition of i-Butylene During CO Hydrogenation over FTS Catalysts

Addition of i-butylene during CO hydrogenation was studied over various FTS catalysts. Table 7 and Figure 8 summarizes the initial (10 min) reaction results for CO hydrogenation in the presence of i-butylene over Pd/SiO_2. As can be seen, in the presence of i-butylene there is a minor decrease in the activity for methanol synthesis. This suggest that the added i-butylene has blocked some of the active sites for methanol synthesis. The only significant product observed during the addition of i-butylene to the syngas feed over the two Pd catalysts was the hydrogenated product isobutane. No MTBE was observed during the reaction. Table 8 summarizes the results for the rate of hydrogenation of i-butylene to isobutane during CO hydrogenation over the same two catalysts. Arrhenius plot for the same data has been plotted in Figure 9. It can be seen that Li promotion significantly decreased the hydrogenation activity of the catalyst resulting in a lower rate for i-butylene hydrogenation as compared to unpromoted Pd/SiO_2 catalysts. In spite of this lowered hydrogenation activity, however, no interception of the methanol intermediate by the i-butylene, resulting in MTBE, was observed. Thus, it appears that metal sites are not capable of carrying out the etherification reaction.

2.3.3. Addition of i-Butylene During CO Hydrogenation over Composite Catalysts

Table 9 summarizes the results for this catalyst. In the presence of the zeolite the activity of Li-Pd/SiO_2 was 6 to 10 times less than in a previous run without zeolite. This might be due to condensation of methanol in the zeolite pores. Furthermore there was an increase in activity when temperature was increased from 160°C to 200°C. However, when the temperature was further increased to 220°C there was a decline in activity.

Table 7. Summary of CO Hydrogenation Reaction over 5% Pd/SiO_2 in the presence of IB at 7 atm.

Temperature, °C	155	170	185	200
CO Conversion, %	0.04	0.07	0.13	0.3
Rate, mol $kg^{-1}h^{-1}$	3.6	6.2	12.1	27.6
SELECTIVITIES				
Methane	0.1	1.1	1.0	0.5
MeOH	99.9	98.9	99.0	99.5

FIGURE 8

Arrhenius Plot for CO Hydrogenation over Pd/SiO_2 in the presence of isobutylene

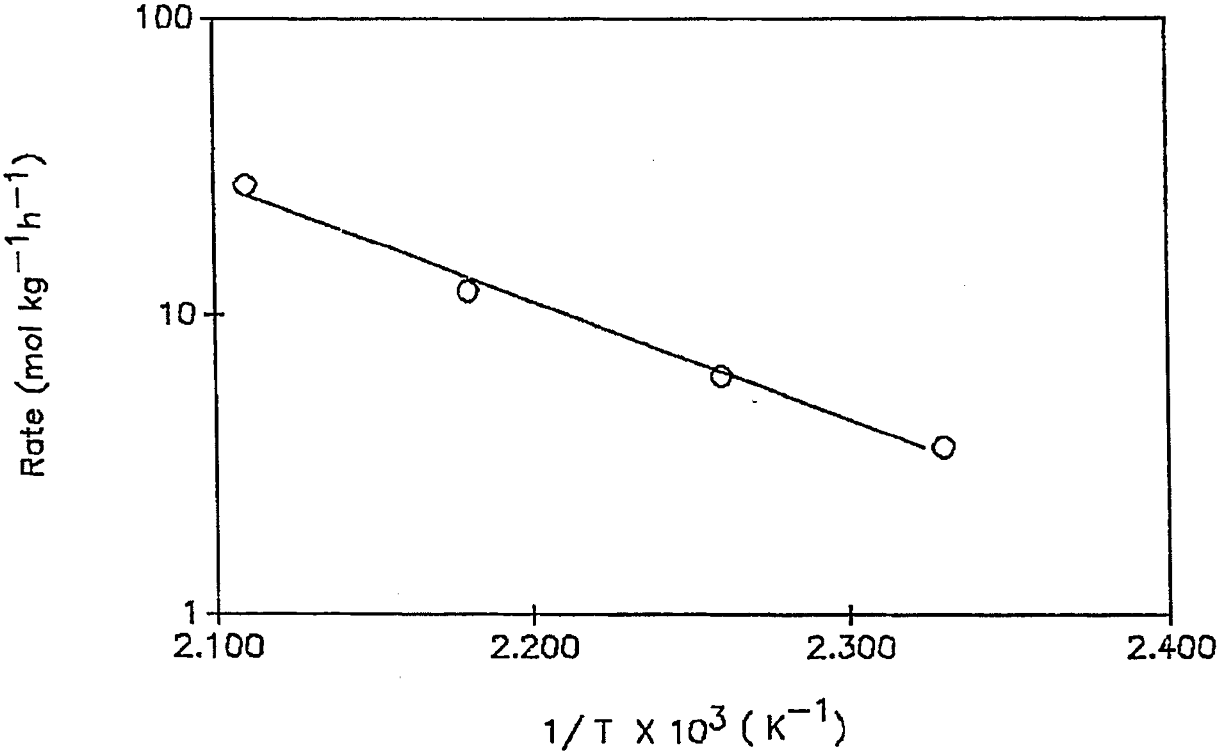


Table 8. Hydrogenation of IB in the Presence of H_2 and CO at 7 atm, H_2:CO = 1.0

Temperature, °C	Pd/SiO_2		Li-Pd/SiO_2	
	Conv. %	Rate μ mol/g.hr	Conv. %	Rate μ mol/g.hr
200	90.6	0.052	29.5	0.017
185	76.6	0.043	15.4	0.009
170	52.3	0.03	7.3	0.004
155	27.3	0.016	3.3	0.002

FIGURE 9

Arrhenius Plot for hydrogenation of isobutylene in the presence of CO and H_2

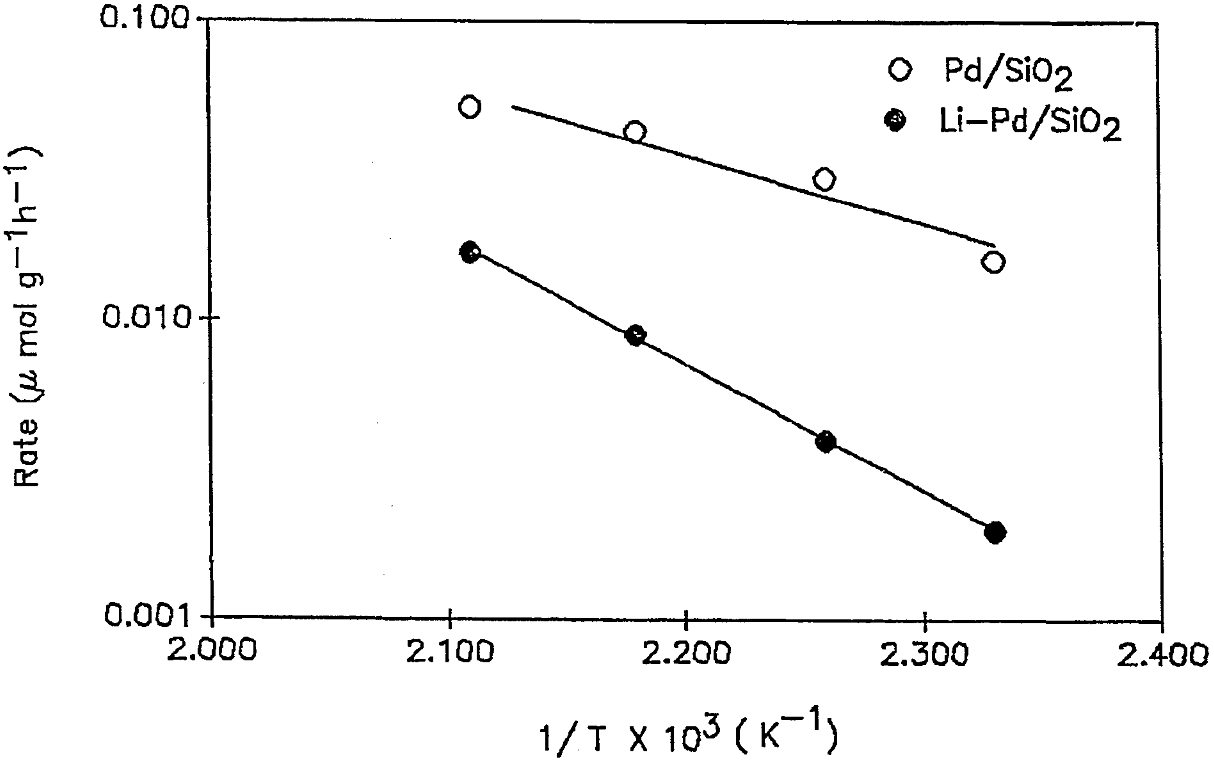


Table 9. CO Hydrogenation over Li-Pd/SiO_2 + LZ210-12# Composite Catalyst @ (Physical Mixture)

Temperature, °C	160	180	200	220
CO Conversion, %	0.003	0.016	0.049	0.033
Rate, mol $kg^{-1}h^{-1}$	0.25	1.46	4.58	3.07
SELECTIVITIES				
Methane	*	*	2.5	40.8
MeOH	99.5+	99.5+	97.5	59.2

* Traces.
# HY zeolite (Si/Al = 6.0)
@ Li-Pd/SiO_2 / LZ210-12 = 5:1

The majority of the i-butylene was converted to unidentified heavy hydrocarbons, with a small portion being hydrogenated to i-butane. The high degree of oligomerization of the i-butylene in this experiment suggests that the quantity of acid sites were too high. Further experiments are planned with the composite catalysts in which a lower concentration of the zeolite catalyst is used.

2.3.4. Addition of i-Butylene During CO Hydrogenation over a Dual-Bed Catalyst Arrangement

The results for the experiments utilizing the dual-bed arrangement are somewhat puzzling. Figure 10 shows the effect of time-on-stream on the rate of methanol formation. The reaction was started with only syngas as a reactant, i.e., in the absence of i-butylene. These data are represented by the open circles. After eight hours, the i-butylene feed was started and these data are represented by the closed circles. Through this period the apparent rate of methanol formation was found to increase almost in a linear fashion with time. Although we have no definitive explanation for this phenomenon, it is perhaps due to the initial retention of methanol in the pores of the catalyst or to a reaction induction period.

The product distributions in the absence and in the presence of i-butylene are shown in Table 11. As with the composite catalysts, the major portion of i-butylene was hydrogenated to i-butane. In this case, however, measurable amounts of MTBE were formed. The amount of MTBE observed in the product slate was also found to increase with time. Unfortunately, we were not able to achieve steady-state due a reactor malfunction. The formation of MTBE in this experiment suggests the possibility of using a bifunctional type catalyst of similar proportions to the dual-bed for the same reaction.

Table 10. Conversion of IB over Li-Pd/SiO_2 + LZ210-12[#] Composite Catalyst [@] (Physical Mixture)

Temperature, °C	160	180	200	220
Rate, μ mol $g^{-1}h^{-1}$	0.067	0.066	0.064	0.056
IB Conversion, %	100	99.91	99.05	99.32
IB Conversion to iso-butane	0.0	0.73	5.07	9.72
IB conversion to unknowns	100	99.27	94.93	90.28

\# HY zeolite (Si/Al = 6.0)

@ Li-Pd/SiO_2 / LZ210-12 = 5:1

Figure 10. Rate of methanol formation in CO hydrogenation over dual bed catalyst. O: in absence of i-butylene; ●: in presence of i-butylene. Other conditions described in text.

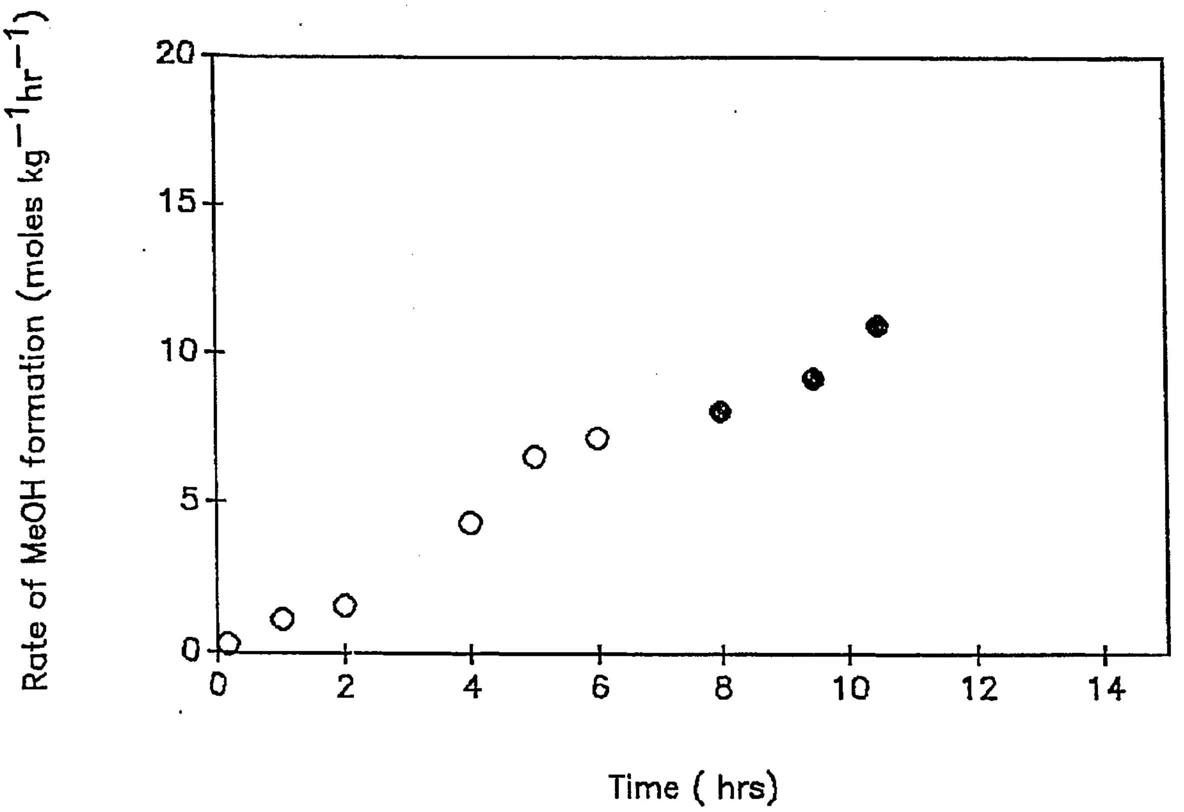


Table 11. Product Distribution for CO Hydrogenation over a Li-Pd/SiO_2/LZ210-12 (10:1) Dual-Bed
T = 180°C , P = 7 atm., H_2/CO = 1

	Before IB addition	After IB addition
Reaction Time (hrs)	8.5	10.5
C_1	10.66	0.22
C_2	3.44	0.61
C_3	1.53	0.05
Iso-Butane	-	65.79
C_4	1.34	0.58
C_5	-	1.11
C_6	-	0.41
C_7	-	0.79
C_8	-	9.52
MeOH	72.95	20.09
DME	10.08	0.7
MTBE	-	0.13

2.4. REFERENCES

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3. WORK PLANNED FOR NEXT QUARTER

Work for next quarter will concentrate in the SBCR runs for the synthesis of MTBE from i-butylene and methanol, and the continuation of studies involving the addition of i-butylene during the synthesis of alcohols.

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