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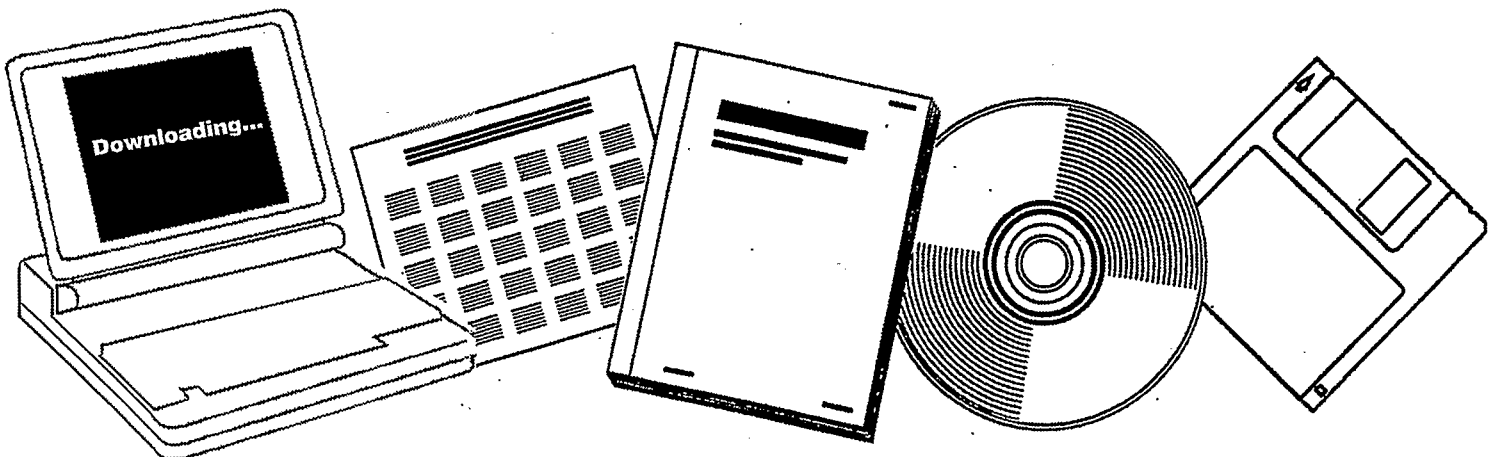
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**FISCHER-TROPSCH SLURRY PHASE PROCESS  
VARIATIONS TO UNDERSTAND WAX FORMATIONS:  
QUARTERLY REPORT FOR PERIOD OCTOBER 1,  
1987 TO DECEMBER 31, 1987**

MASSACHUSETTS INST. OF TECH., CAMBRIDGE

1987



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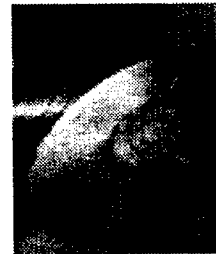
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Fischer-Tropsch Slurry Phase  
Process Variations to Understand  
Wax Formations

Quarterly Report for Period  
October 1, 1987 to December 31, 1987

Report No.: DOE/PC80015-9  
Contract No.: DE-AC22-85PC80015

DOE/PC/80015---9  
DE88 007210

by

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**MASTER**

#### ACKNOWLEDGEMENT

This work was sponsored by the Pittsburgh Energy Technology Center (PETC) of the United States Department of Energy (DOE). The author would like to express his appreciation to Mr. Swenam R. Lee at PETC, Project Manager, and Dr. John Shen at DOE-Washington, for their contributions to this project.

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Summary

Some of the effects of high syngas conversion on the secondary reactions of olefins formed by Fischer-Tropsch synthesis on a reduced fused magnetite catalyst were simulated by studies of olefins in the presence of hydrogen and low concentrations of CO, or none at all. Kinetic models were developed for the effect of CO partial pressure on hydrogenation rates at 232°C and 0.30 to 0.79 MPa.

Secondary reactions of olefins on a fused iron magnetite Fischer-Tropsch catalyst are sensitive to carbon monoxide partial pressure. In the absence of carbon monoxide, olefin hydrogenation is much more rapid than in its presence. A model for olefin hydrogenation indicates that a hydrogenated carbon monoxide species on the catalyst may be responsible for the inhibition of olefin hydrogenation, and that olefin adsorption is rate-limiting. The formation of secondary olefins appeared to follow similar trends with carbon monoxide partial pressure. Olefin incorporation was not observed in the small amount of Fischer-Tropsch products obtained in these experiments. The chain growth probability,  $\alpha$ , appeared to correlate with the  $H_2/CO$  feed ratio.

## EXPERIMENTAL

The studies were performed in either of two 1-L continuous flow stirred autoclaves, in an experimental system described previously (Huff and Satterfield, 1983, 1982). One system was modified to provide the capability for on-line mixing with two Brooks 5850 mass flow controllers. For each of two sets of experiments about 50 g of a fused magnetite catalyst (Girdler C-73) was ground to 52-92  $\mu\text{m}$ , (170-270 mesh), prereduced according to a procedure previously described (Huff and Satterfield, 1984), and suspended in about 500 ml of purified octacosane. Estimates of the effectiveness factors and variations of stirring rate indicated no significant internal or external mass transfer resistances.

Two types of studies were made. First were hydrogenation of olefins in the absence of CO (supplied in premixed cylinders of 2% olefin in prepurified hydrogen from Matheson). Table I gives our analysis of the organics in the various mixtures. Secondly were studies with the same mixtures, but with on-line addition of varying amounts of carbon monoxide to the feed as well. The olefins were ethene, propene, 1-butene, and 2-butenes (a mixture of cis and trans). These mixtures in  $\text{H}_2$  were found to be stable for several months with no observable hydrogenation or isomerization occurring in the cylinders.

All experiments were run at 232°C and at total pressures of 0.30 to 0.79. Analysis of the products was achieved with a refinery gas analyzer and a capillary column gas chromatograph equipped with a flame ionization detector (FID). While water was

formed when carbon monoxide was fed, the amounts were so low as to make accurate measurement difficult.

## RESULTS AND DISCUSSION

The experimental results are presented in two sections. The first discusses the hydrogenation of olefins in the absence of carbon monoxide, the second section, reactions in the presence of carbon monoxide.

### Carbon Monoxide Not Fed

When the catalyst, which had previously reached steady state Fischer-Tropsch synthesis activity, was subjected to a hydrogen-olefin mixture in the absence of carbon monoxide, its activity for olefin hydrogenation gradually increased with time, reaching steady-state after about 50 hours on stream. This is illustrated in Figure 1 which shows the paraffin/olefin ratio for propene as a function of time from the switch from a syngas feed to a hydrogen-propene feed. During the run the feed was changed several times between 0.5 and 1.0 L/min. At the lower feed rate (longer residence time) the paraffin/olefin ratio in the products is higher because of increased secondary hydrogenation. The catalyst had been previously started up at 263°C for 75 hours and then run for 60 hours at 232°C with 0.7 H<sub>2</sub>/CO syngas at 1.48 MPa before the olefin hydrogenation experiments.

The increase in activity may have reflected the removal of surface carbon from the catalyst in the form of methane. Methane was observed in the outlet stream of the reactor, but only about 10 percent of it was produced by cracking of the olefins, based on the formation of C<sub>2</sub> products when propene was fed and C<sub>3</sub>



products when butenes were fed. No more than 2 percent of the olefins cracked during the experiments.

Reaction of Butenes: The hydrogenation of 1-butene or 2-butene was studied over periods of about twelve hours on consecutive days. Hydrogenation products were measured no sooner than 3 hours after a change in feed or in reaction conditions, which also corresponded to at least 20 to 30 turnovers of gas in the reactor. Figure 2 shows power law correlations for the hydrogenation data. Two sets of studies with 1-butene were made before and after the study of 2-butene to test for reproducibility. The two pairs of overlapping points were checks on reproducibility after changing space velocity.

The hydrogenation rate for 2-butene gave a second-order rate expression by regression. The rate for 1-butene gave a best fit with exponent of 1.7, but a forced fit with a second-order rate equation also appeared to fit quite well. This is shown by the two lines on Figure 2. A concentration of 2-butenes an order of magnitude greater than that of 1-butene is required here for the rate of hydrogenation of the two olefins to be equal.

Some literature suggests that the rates are more closely comparable. Uchytel et al. (1981) reported that hydrogenation of the 2-butenes was only about 20 percent slower than that of 1-butene, using a sulfided Ni-W catalyst at 300°C. Cerveny and Ruzicka (1969) found that hydrogenation of trans-2-hexene was about 40 percent slower than that of 1-hexene on supported platinum catalysts at 20°C. More recently, however, Derrien (1986) in a review article describing hydroisomerization

processes estimated 1-butene hydrogenation rates to be an order of magnitude faster than the 2-butenes. In fact, in a simplified model of the process, they assumed that all butane formed came from 1-butene.

Figure 3 compares the ratio of cis to trans in the 2-butenes and the ratio of trans-2-butene to 1-butene in the exit gases with that expected at equilibrium. When 1-butene was fed, product gases approached equilibrium more closely than when the 2-butenes were fed. With 1-butene the cis/trans ratios in the 2-butene are close to the equilibrium ratio of 0.623; the trans-2-butene/1-butene ratio approaches 2.0 as compared to the equilibrium value of 4.6. With the 2-butenes feed, products remained quite far from equilibrium. This is consistent with its seeming generally lower reactivity. Interestingly, the 2-butene data tended to move away from equilibrium with higher conversions. One might hypothesize that the cis-isomer and 1-butene are more reactive than the trans isomer, which would give a low cis/trans ratio and a high trans-2-butene/1-butene ratio.

#### Carbon Monoxide Added

The experiments with the addition of carbon monoxide were performed with a second batch of catalyst which previously had been used for 800 hours in experiments involving the addition of carbon dioxide to the syngas feed. Since no irreversible effects were observed, the catalyst was deemed fit to be used for the present study.

The activity of the catalyst in the presence of some carbon monoxide was found to be stable. Inlet carbon monoxide

concentrations were varied from 0.5 to 5 percent of the feed. Low levels of carbon monoxide were used to minimize the amount of Fischer-Tropsch synthesis products and hence make it easier to determine the fate of the olefin fed to the reactor. Correlations were developed based on hydrogen-carbon monoxide feeds with no olefins present to subtract out products formed from the Fischer-Tropsch reaction. Such corrections amounted to no more than 15 percent of an individual compound. The reactions, hydrogenation, isomerization, and incorporation are discussed separately below.

#### OLEFIN HYDROGENATION

The literature provides little guidance for modeling these reactions. The study of olefin hydrogenation as such on metallic catalysts has been largely restricted to the hydrogenation of ethene and propene at relatively low temperatures, usually below 175°C. Olefin hydrogenation data were reviewed by Bond and Wells (1964). More recently they have been reviewed by Kiperman (1986). Higher temperature studies have been conducted on hydrotreating catalysts (Fott and Schneider, 1980; Uchytel, et al., 1980, 1981).

Sudheimer and Gaube (1985) studied hexene and decene hydrogenation on potassium promoted precipitated iron catalyst at 220°C. From power law fits of their data, they concluded that olefin hydrogenation was about first order in both 1-alkene and hydrogen, and roughly negative first order in carbon monoxide. Based on these observations, a general Langmuir-Hinshelwood expression was used here to fit the experimental data. This

equation has the form,

$$R_{\text{olefin hyd}} = \frac{k_{\text{hyd}} K_{\text{olefin}} P_{\text{olefin}} P_{\text{H}_2}^n}{1 + K_{\text{CO}} P_{\text{CO}} P_{\text{H}_2}^m} \quad (1)$$

Addition of an adsorption term for olefin in the denominator did not give a satisfactory correlation. The implication is that olefin adsorption was negligible relative to that of CO. In order to fit the experimental data, the above equation was linearized to the form,

$$\frac{P_{\text{olefin}}}{R_{\text{olefin}}} = \frac{P_{\text{H}_2}^{-n}}{k_{\text{hyd}} K_{\text{olefin}}} + \frac{K_{\text{CO}} P_{\text{CO}} P_{\text{H}_2}^{m-n}}{k_{\text{hyd}} K_{\text{olefin}}} \quad (2)$$

Ethane Hydrogenation: Figure 4 shows the data for ethene hydrogenation plotted as  $P_{\text{C}_2\text{H}_4}/R_{\text{C}_2\text{H}_6}$  versus  $P_{\text{CO}}$ . At the lowest carbon monoxide feed levels, essentially 100% conversion of CO occurred (the same was true for butene), and rate data scattered greatly. The catalyst activity in this region is probably greatly sensitive to slight changes in CO concentration. If the two points with unusually high rates are ignored, intercepts are closely comparable for the two different total pressures. This would imply that  $n$  is equal to zero. The dependence of the slope of the lines with respect to total pressure appears to be about second order, which implies that  $m$  is equal to 2. A plot of the left hand side versus  $P_{\text{CO}} \cdot P_{\text{H}_2}^2$  should result in a single line as indeed it does, as shown in Figure 5. The fitted equation for ethene hydrogenation is,

$$R_{\text{C}_2\text{H}_6} = \frac{3.0 \times 10^4 P_{\text{C}_2\text{H}_4}}{1 + 4600 P_{\text{CO}} P_{\text{H}_2}^2} \quad (3)$$

Pressure is expressed in MPa and reaction rate as  $\mu\text{mol}/(\text{min})(\text{g}\cdot\text{cat})$ .

As another test of Equation 3, the Fischer-Tropsch synthesis reaction was run in the absence of added  $\text{C}_2\text{H}_4$  using partial pressures of  $\text{H}_2$  and  $\text{CO}$  in the same range as when  $\text{C}_2\text{H}_4$  was added to the system. Hence all  $\text{C}_2\text{H}_4$  present was formed by in situ synthesis. Figure 6 shows a reasonable fit of equation 3 to these experiments. The predicted rates of formation of  $\text{C}_2\text{H}_6$  are about 10 to 20% lower than the actual rates. An interesting implication is that most of the ethane formed in the Fischer-Tropsch synthesis is not a primary product but rather is produced in a secondary reaction by hydrogenation of ethylene.

Butene Hydrogenation: The 1-butene hydrogenation system is complicated by the simultaneous isomerization to form 2-butenes. In the absence of carbon monoxide, the rate of hydrogenation of the 2-butenes was found here to be some two orders of magnitude slower than that of the 1-butene. Since in the experiments with carbon monoxide, the concentrations of 2-butenes were no more than about three times the concentration of 1-butene, the hydrogenation of the 2-butenes could be viewed as not contributing significantly to the formation of butane. This follows the model of Derrien (1986) and simplifies the modeling of the butene system, in that the reaction network can be modelled as two parallel reactions rather than a triangular network.

A plot similar to the ones presented above is shown in Figure 7. The results differ from Figure 4 for ethene in that

the intercepts increase approximately linearly with total pressure. This means that equation 1 is insufficient to model the butene system and an additional inhibition term is required. Since the inhibition is linear in total pressure, the new term probably includes the partial pressure of hydrogen, which is by far the major component in these experiments. A modification of equation 2 to correlate the butene hydrogenation data is given by Eq. 4:

$$\frac{P_{1\text{-butene}}}{R_{\text{butane}}} = \frac{1}{k_{\text{hyd}} K_{1\text{-butene}}} + \frac{K_{\text{CO}} P_{\text{CO}} P_{\text{H}_2}^2}{k_{\text{hyd}} K_{1\text{-butene}}} + \frac{K_{\text{H}_2} P_{\text{H}_2}}{k_{\text{hyd}} K_{1\text{-butene}}} \quad (4)$$

The correlation obtained was

$$R_{\text{butane}} = \frac{1.5 \times 10^4 P_{1\text{-butene}}}{1 + 1.3 \times 10^5 P_{\text{CO}} P_{\text{H}_2}^2 + 1.9 P_{\text{H}_2}} \quad (5)$$

Figure 8 shows a comparison of the model and experimental butane formation rates from the H<sub>2</sub>/1-butene/CO experiments. Figure 9 is a comparison of the model with experimental butane production for H<sub>2</sub>/CO experiments.

The correlations for olefin hydrogenation show a first order dependence on olefin partial pressure and what appears to be an inverse dependence on hydrogen pressure. For most Fischer-Tropsch synthesis conditions carbon monoxide partial pressures will be much higher than those studied here, whereupon the kinetic expressions for both ethene and 1-butene hydrogenation reduce to the form,

$$R_{\text{paraffin}} = \frac{k_{\text{hyd}} P_{\text{olefin}}}{P_{\text{CO}} P_{\text{H}_2}^2} \quad (6)$$

An equation of this form suggests a mechanism in which olefin adsorption is rate limiting and is inhibited by competitive adsorption with some surface intermediates. An interesting process implication is that there does not seem to be selectivity penalty for operating a Fischer-Tropsch reactor at higher pressures to increase reactor throughput.

#### Isomerization

Upon feeding 1-butene the equilibrium between cis- and trans-isomers of 2-butene was approached very closely, as shown in Figure 10. The trans-2-butene/1-butene ratio approached 2.0 at the highest 1-butene conversions. Attempts to model the isomerization with equations of the form used above failed, even when accounting for the approach to equilibrium. Figure 11 shows the rate of 2-butenes formation versus partial pressures of carbon monoxide or 1-butene. The rate of 2-butene formation increases with partial pressure of either, but the total pressure appears to have a depressing effect.

#### Olefin Incorporation Into Chains

In a recent study with this same catalyst (Hanlon and Satterfield, 1988) selected 1-olefins including ethylene and 1-butene were added to synthesis gas during the Fischer-Tropsch synthesis. Incorporation into growing chains could be detected, but was relatively minor. In effect the same kind of study was repeated here except that now the olefin concentration in the reactor was much greater than that of CO, i.e., this simulated conditions at a very high degree of Fischer-Tropsch syngas conversions. Figure 12 is a Schulz-Flory plot of carbon number

product distribution for four runs at approximately the same carbon monoxide partial pressure, without and with added olefins. There appears to be a slight increase in the concentration of the C+1 product when olefin of carbon number C is added. This is consistent with our previous studies with this catalyst, that the effect is small.

The alpha for the C<sub>3</sub> to C<sub>7</sub> fraction was 0.42, which is much lower than the 0.55 to 0.65 range we normally observe on this catalyst with feed gas H<sub>2</sub>/CO ratios from 0.5 to 1.8. This appears to be caused by the high H<sub>2</sub>/CO feed ratio. Figure 13 shows Schulz-Flory plots for data obtained at H<sub>2</sub>/CO feed ratios of approximately 10 and 50 at each of three total pressures. There is no effect of pressure as such, but the higher H<sub>2</sub>/CO ratio feed results in a lower alpha and a greater relative methane production. This suggests a lower concentration of surface carbide, a condition which would favor a "first order" process such as methanation over chain growth which might be considered a "second order" process.



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TABLE I

Analysis of Organics in 2% Olefin in H<sub>2</sub> Mixtures

| Olefin   | Mole percent<br>of organics | Minor components<br>(mole percent)                            |
|----------|-----------------------------|---|
| ethene   | 99.5                        | ethane (0.4), methane (0.1)                                   |
| propene  | 99.6                        | propane (0.4)   |
| 1-butene | 99.7                        | butane (0.1), trans-2-butene (0.1)                            |
| 2-butene | 55.6 (trans)<br>42.0 (cis)  | 1-butene (1.3), butane (0.9),<br>unidentified impurity (0.2)* |

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\* Passed through reactor unconverted.

C<sub>3</sub> PARAFFIN/OLEFIN RATIO VS TIME AFTER SWITCH FROM H<sub>2</sub>/CO TO H<sub>2</sub>/ PROPENE FEED

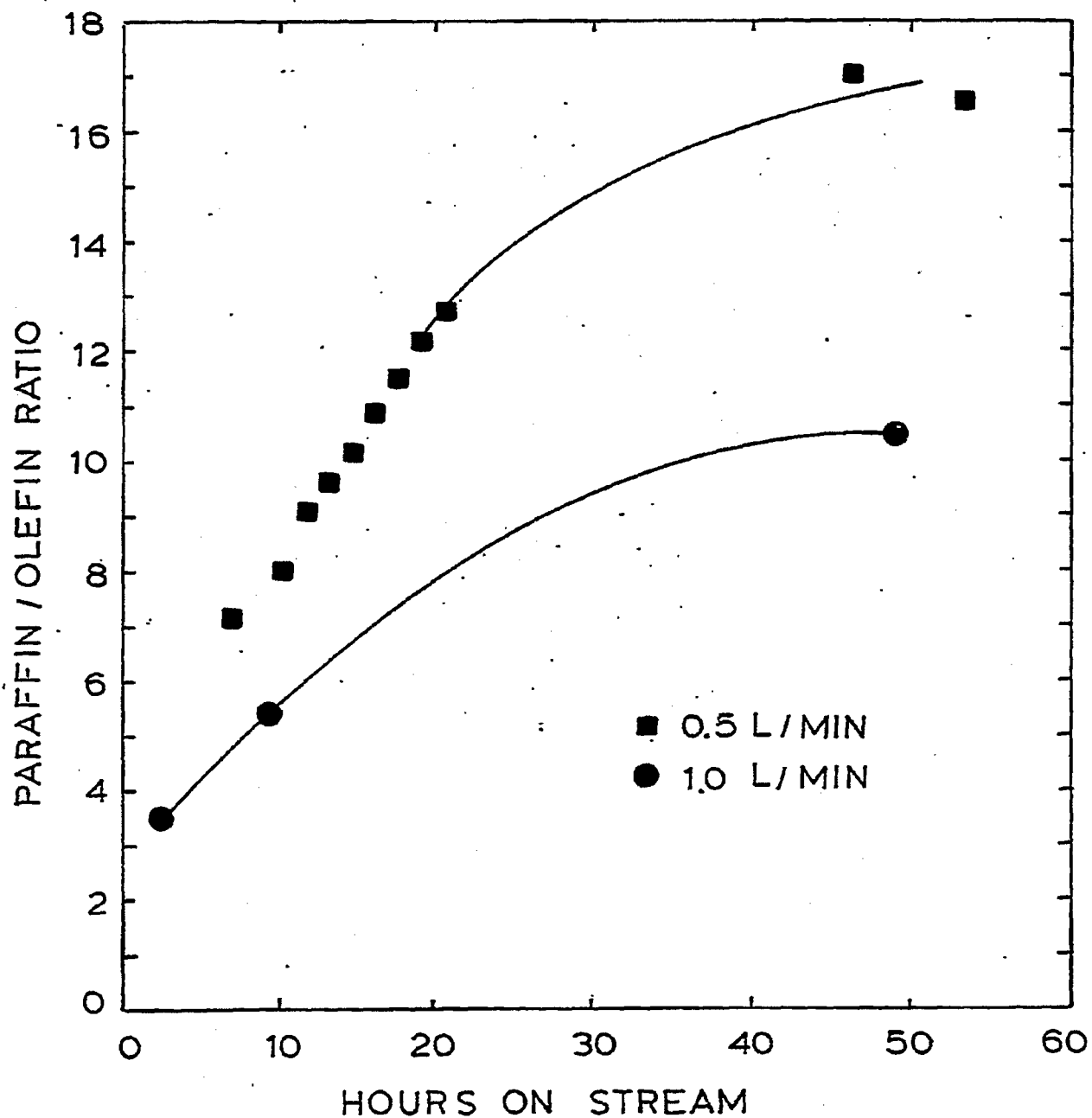


FIGURE 1

HYDROGENATION OF  
 POWER-LAW CORRELATION OF 1-BUTENE  
 AND 2-BUTENE, CO ABSENT <sup>^</sup>

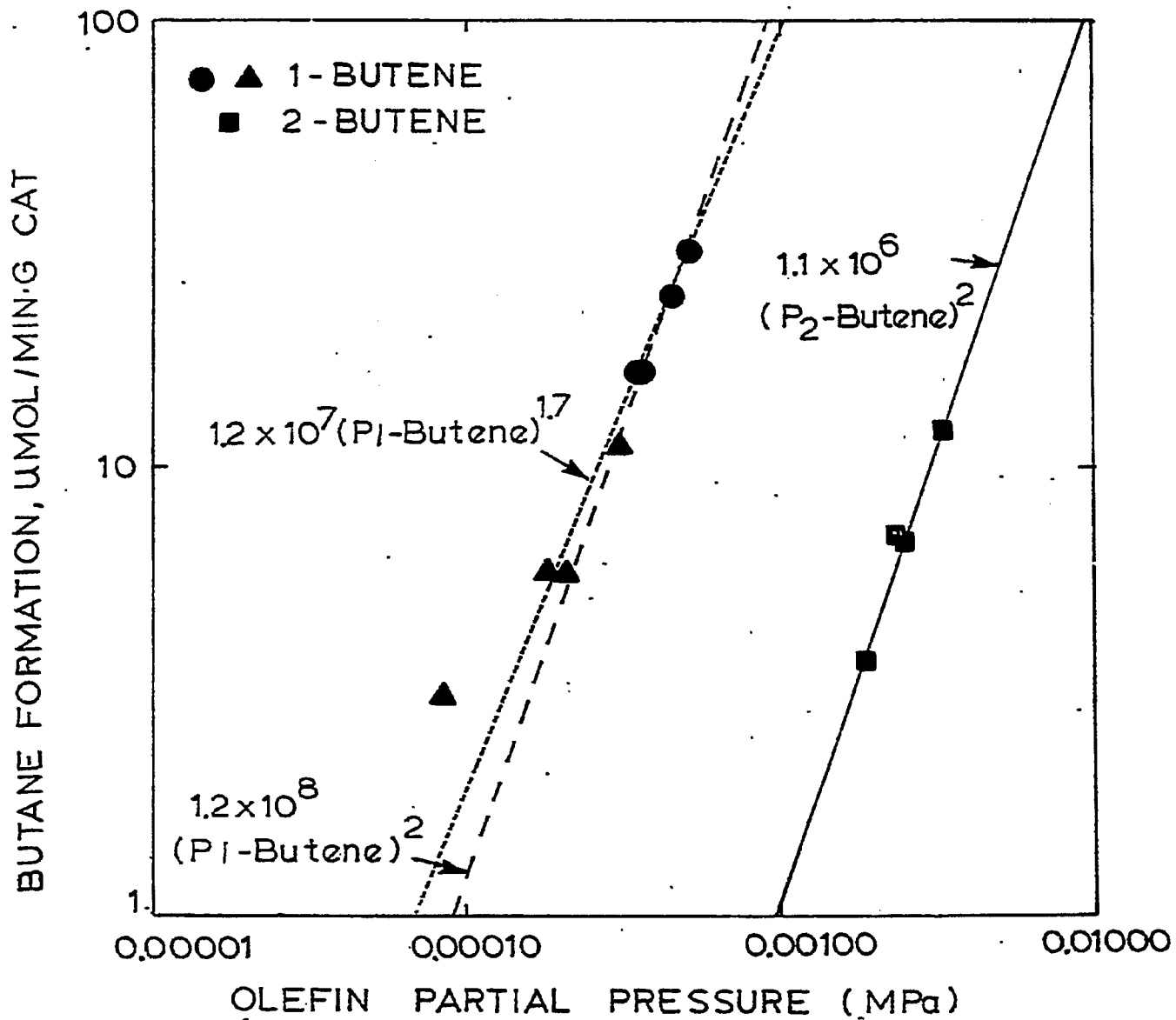


FIGURE 2

BUTENE SELECTIVITY, CO ABSENT

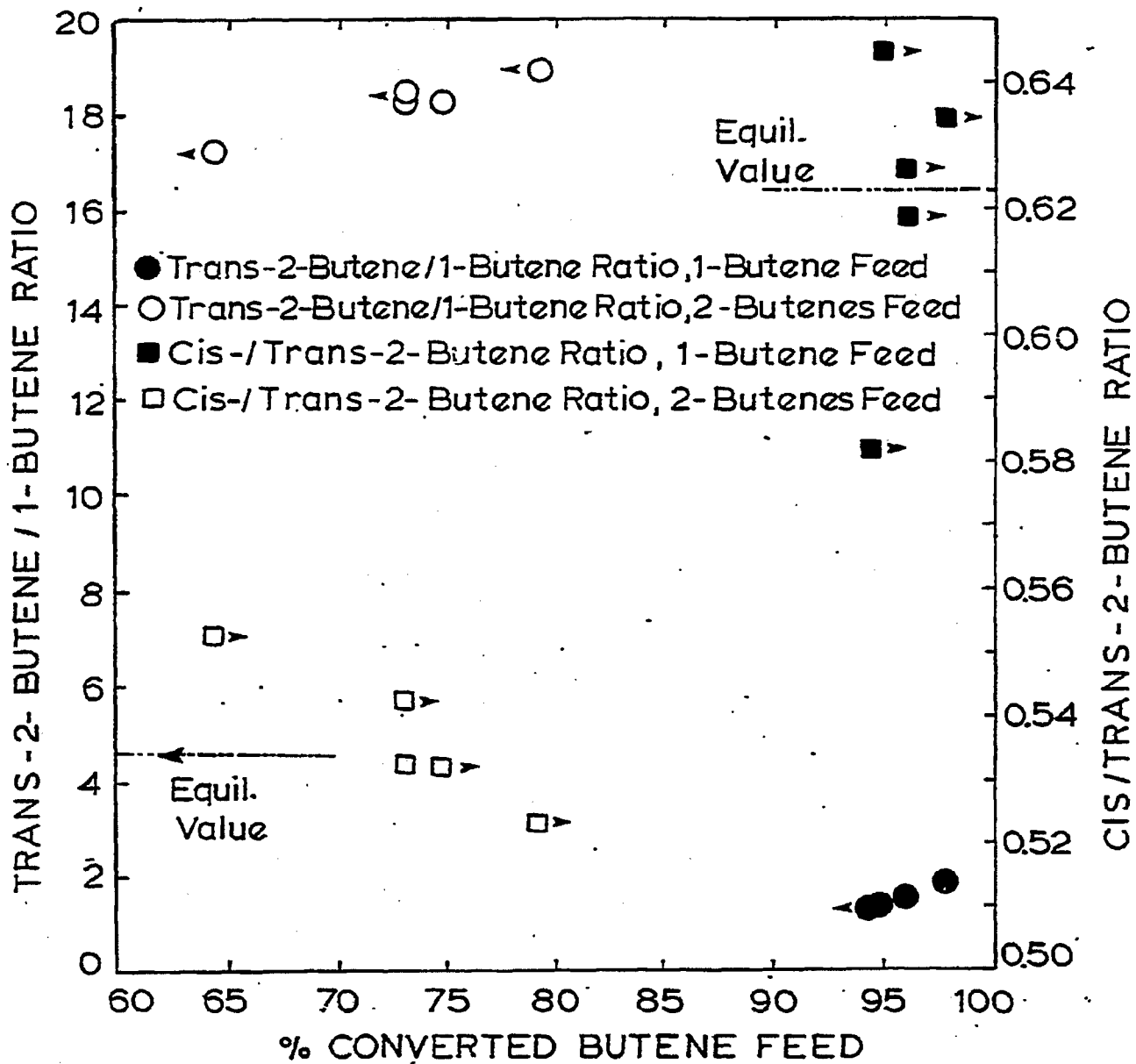


FIGURE 3

EFFECT OF CO PRESSURE ON ETHENE  
HYDROGENATION

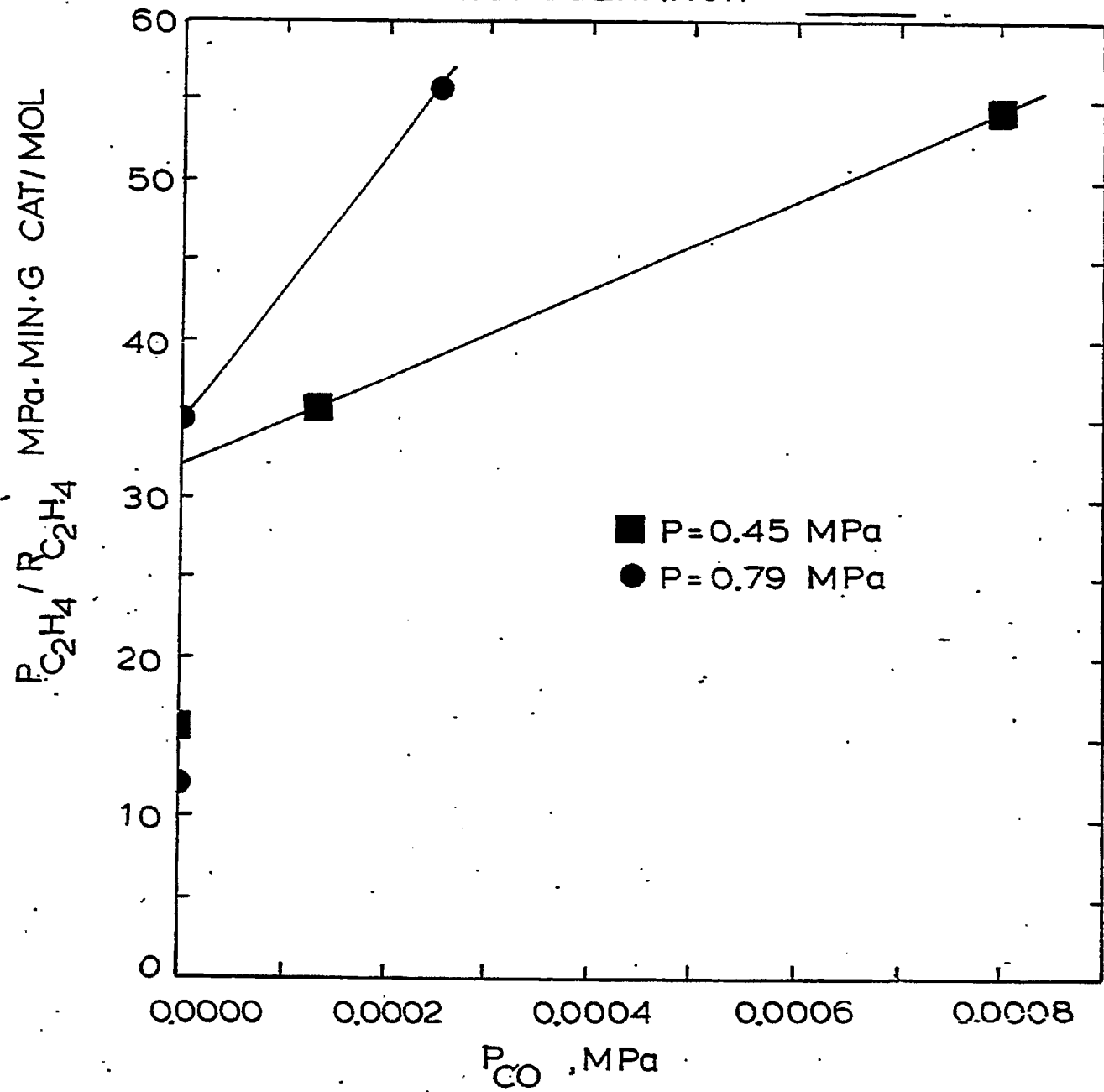


FIGURE 4

# CORRELATION OF ETHYLENE HYDROGENATION DATA

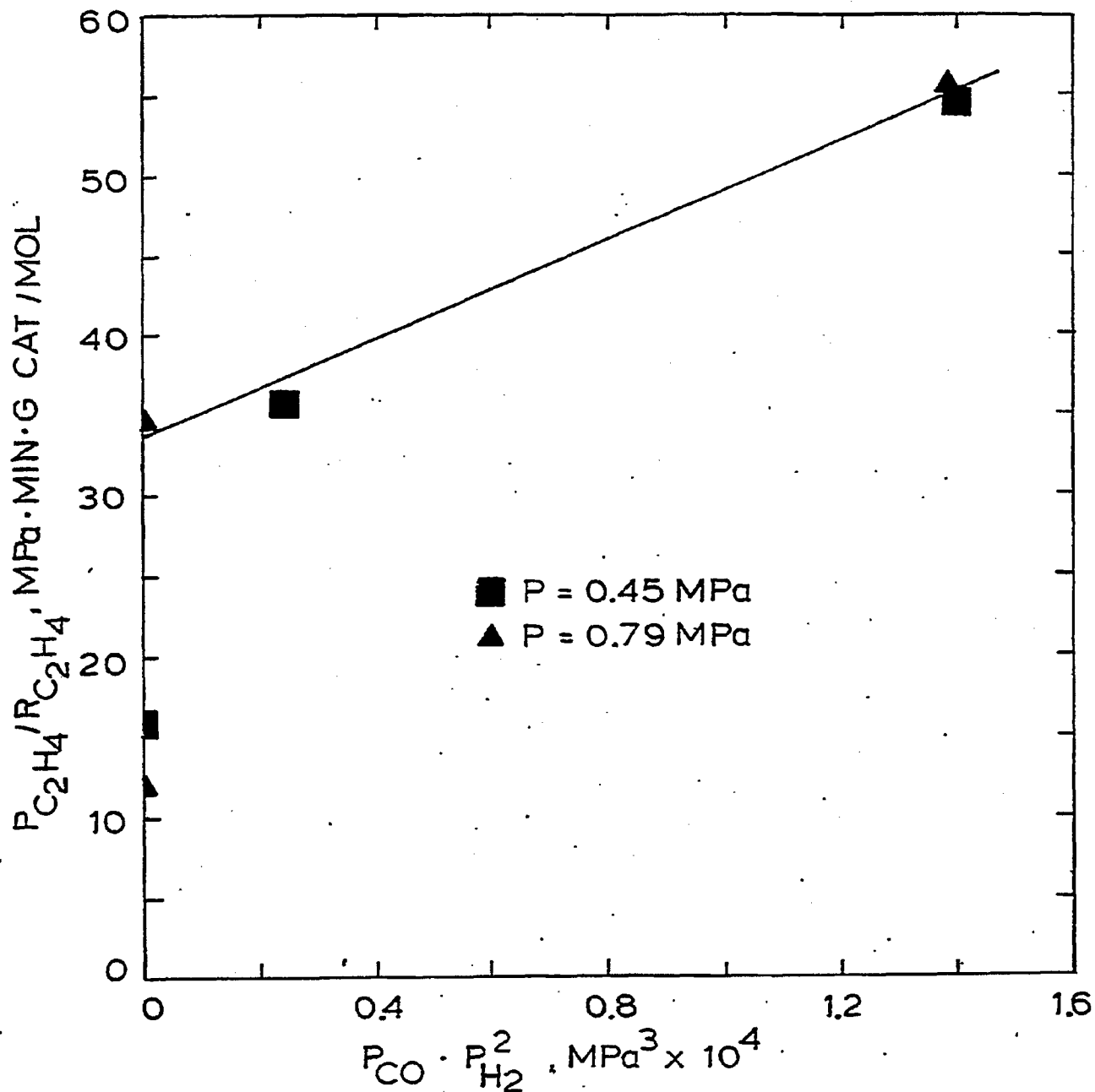


FIGURE 5

TEST OF ETHENE HYDROGENATION MODEL,  
H<sub>2</sub>/CO FEED

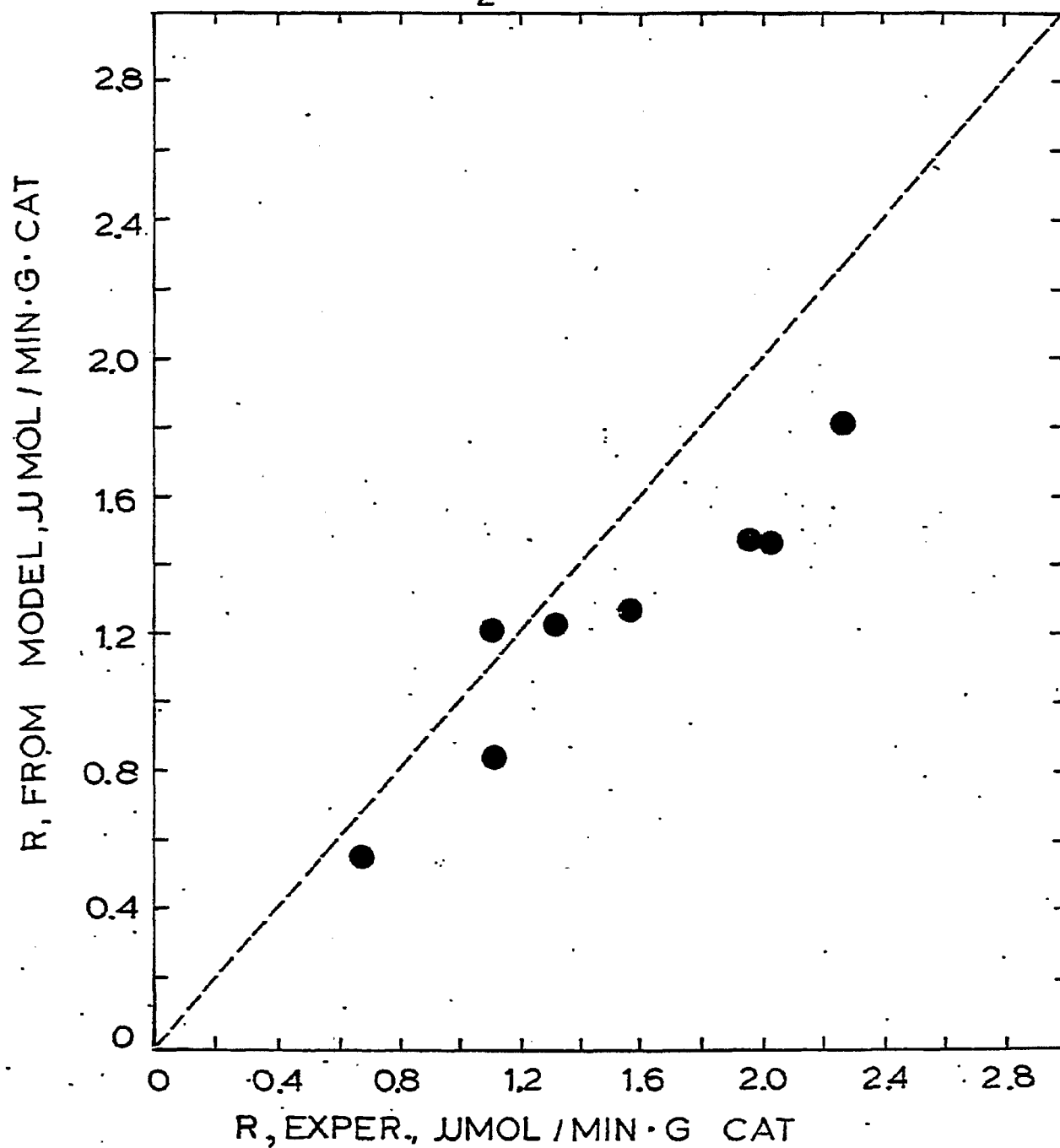


FIG. 6



# HYDROGENATION OF 1-BUTENE

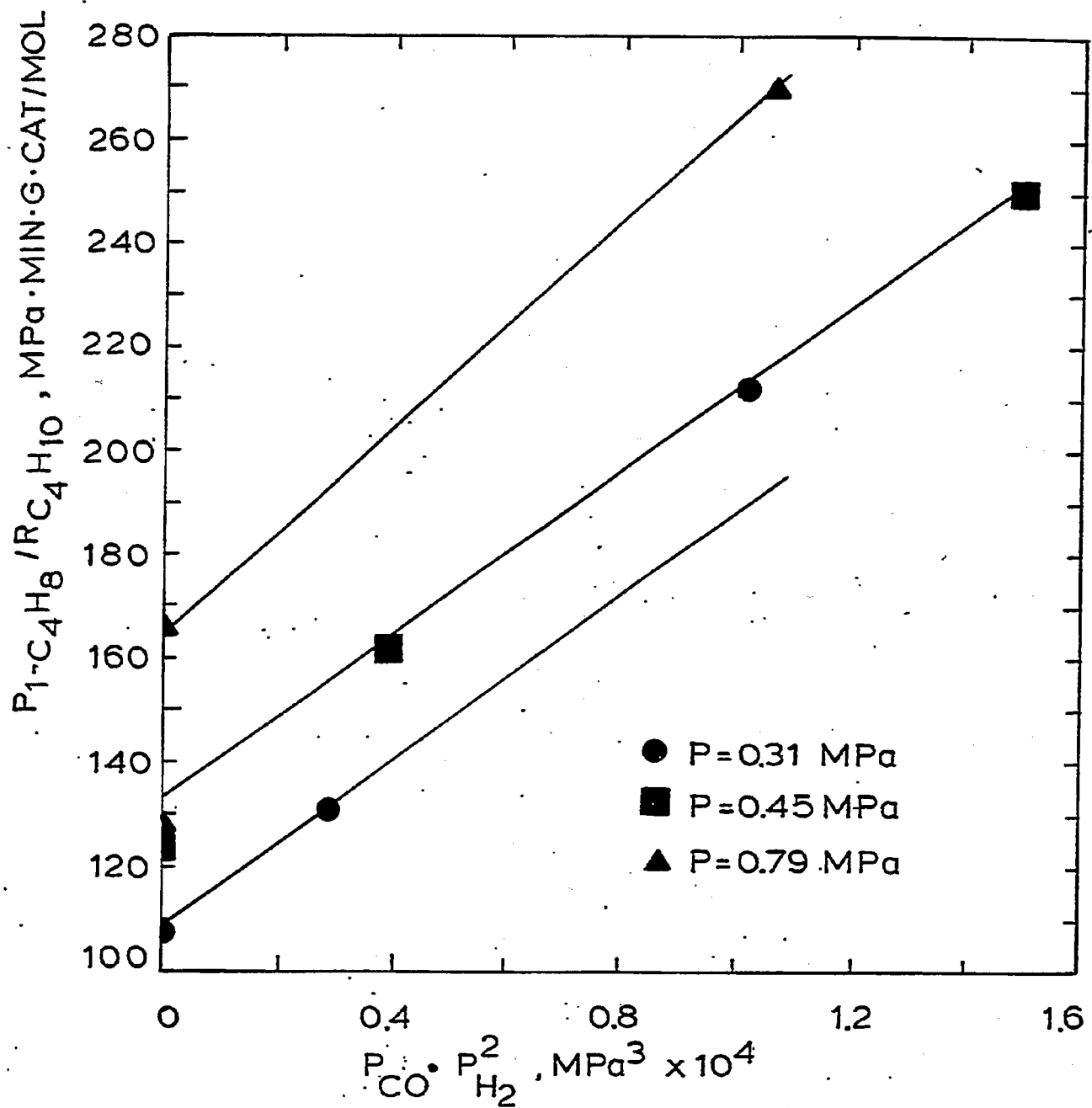


FIGURE 7

EXPERIMENTAL vs PREDICTED 1-BUTENE  
HYDROGENATION RATES, H<sub>2</sub> / 1-BUTENE /  
CO FEED

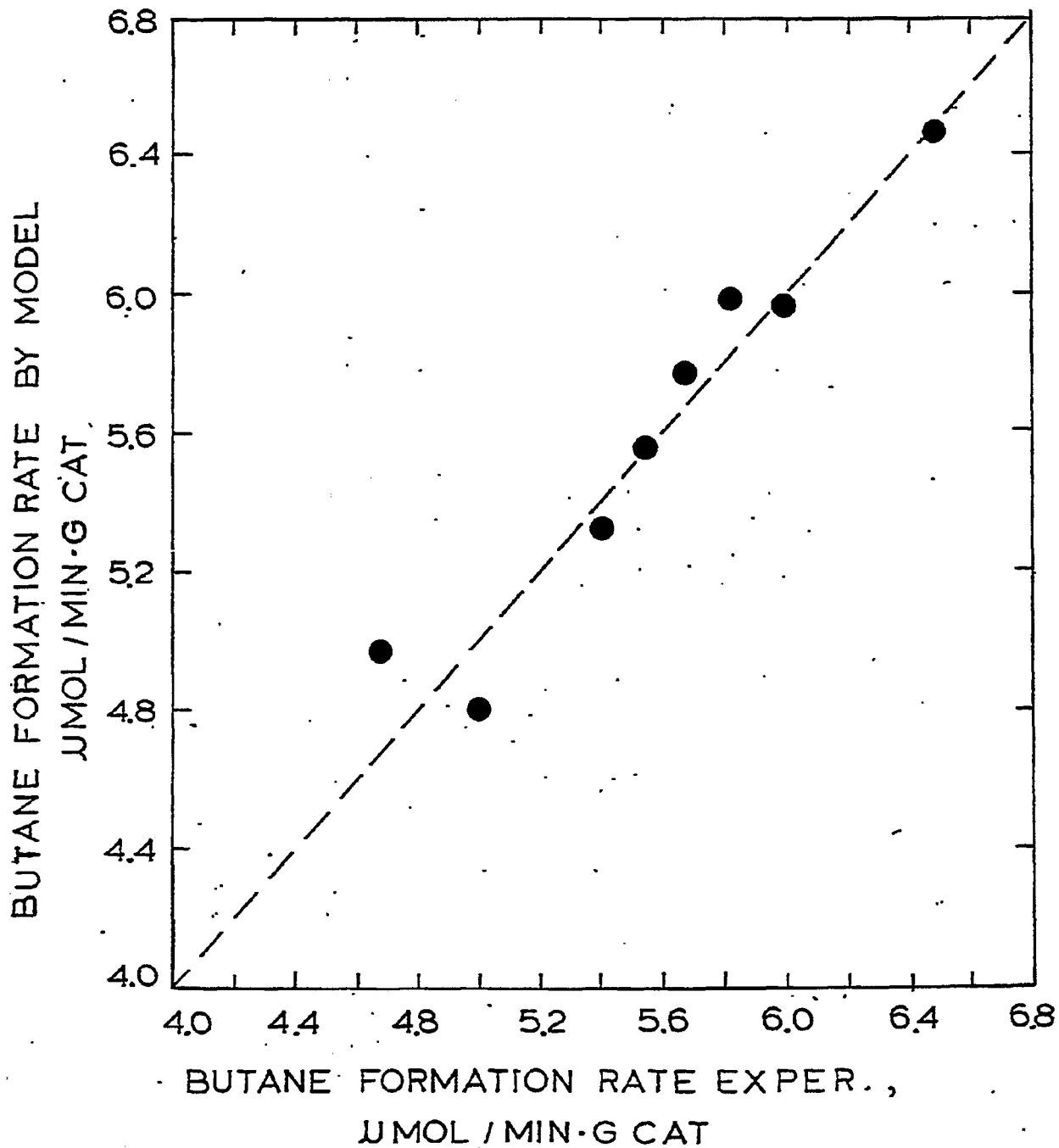


FIGURE 8

EXPERIMENTAL vs PREDICTED 1-BUTENE  
HYDROGENATION RATES., H<sub>2</sub>/CO FEED

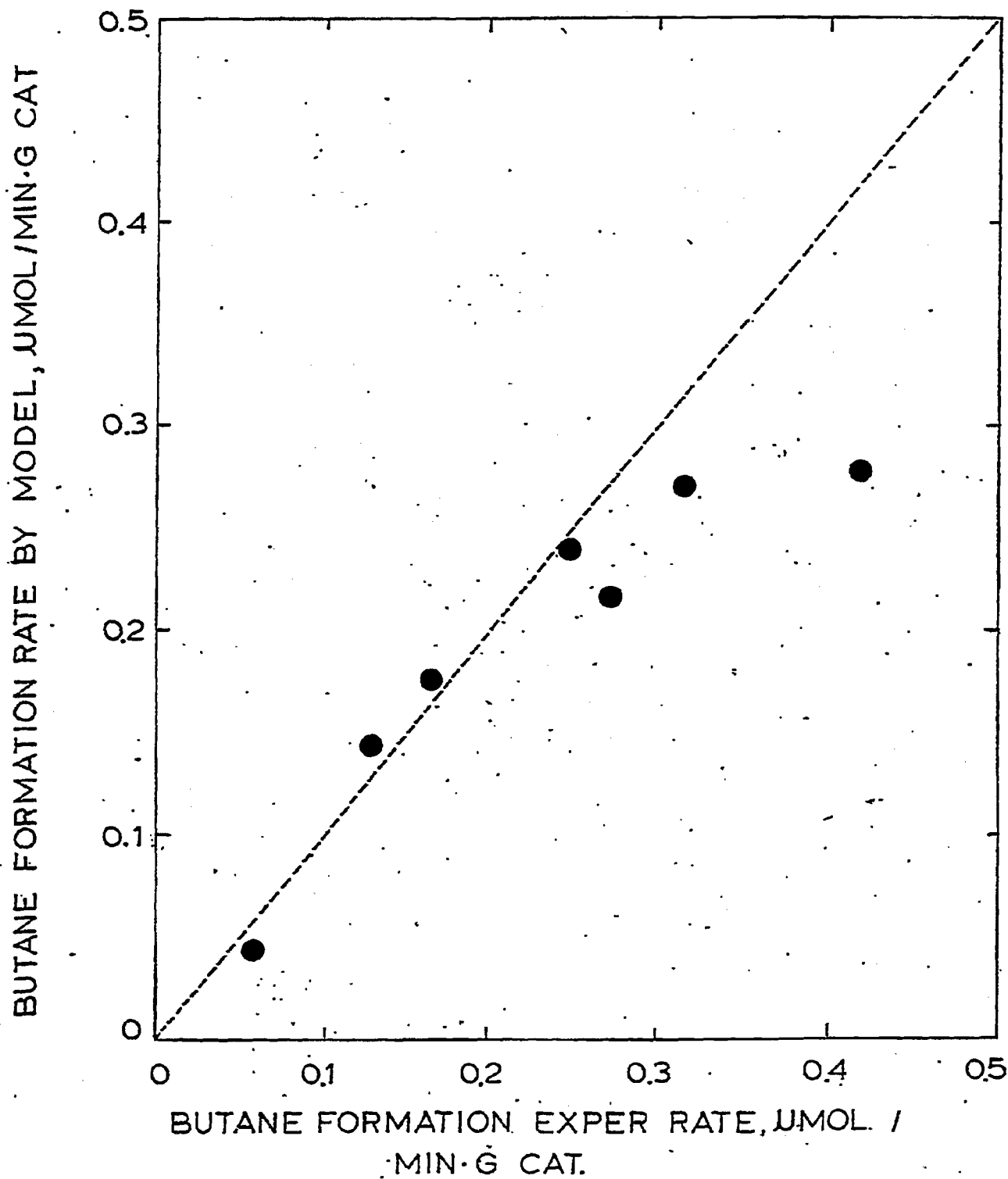


FIGURE 9

# BUTENE ISOMERIZATION AS A FUNCTION OF 1-BUTENE CONVERSION

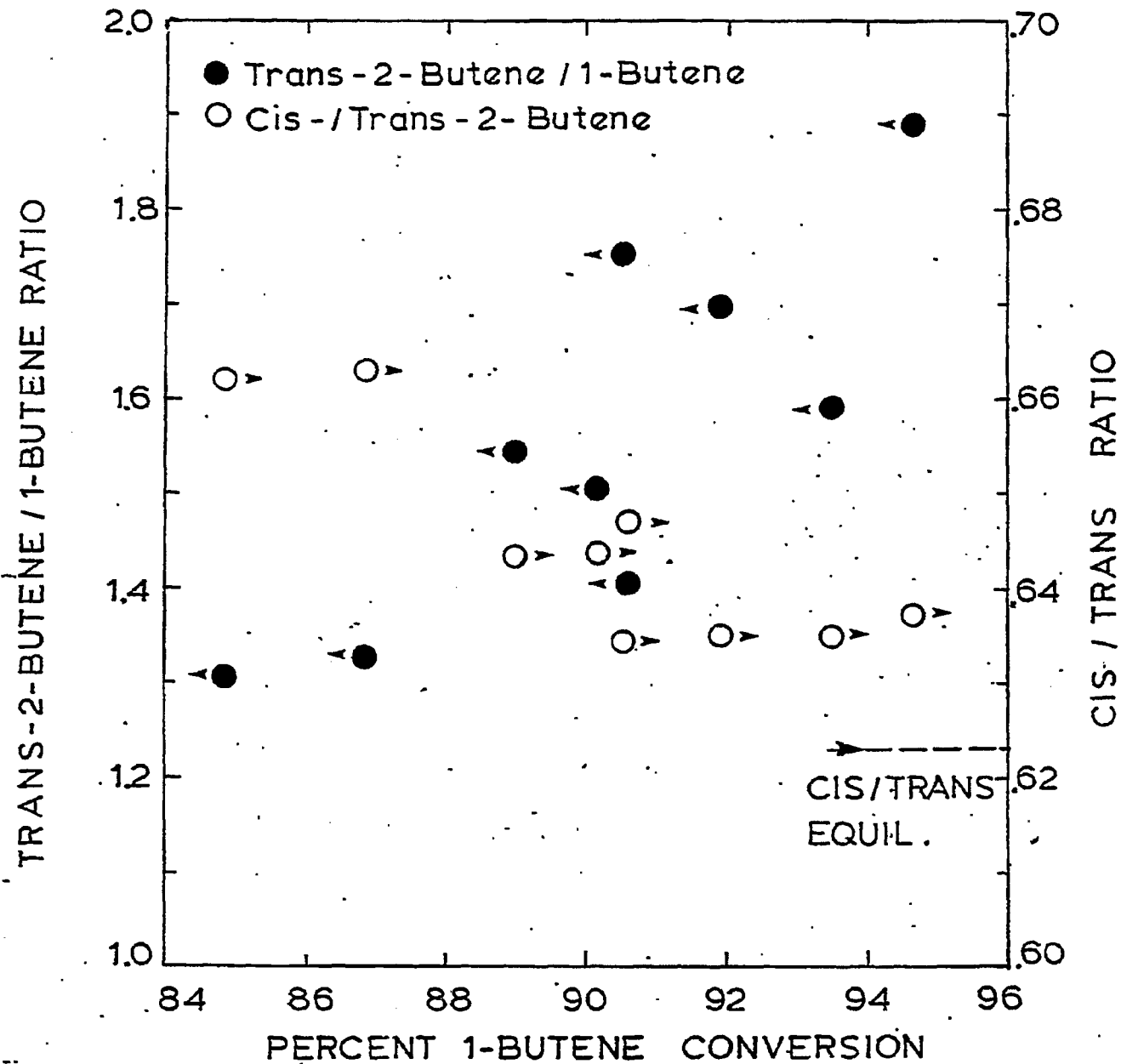
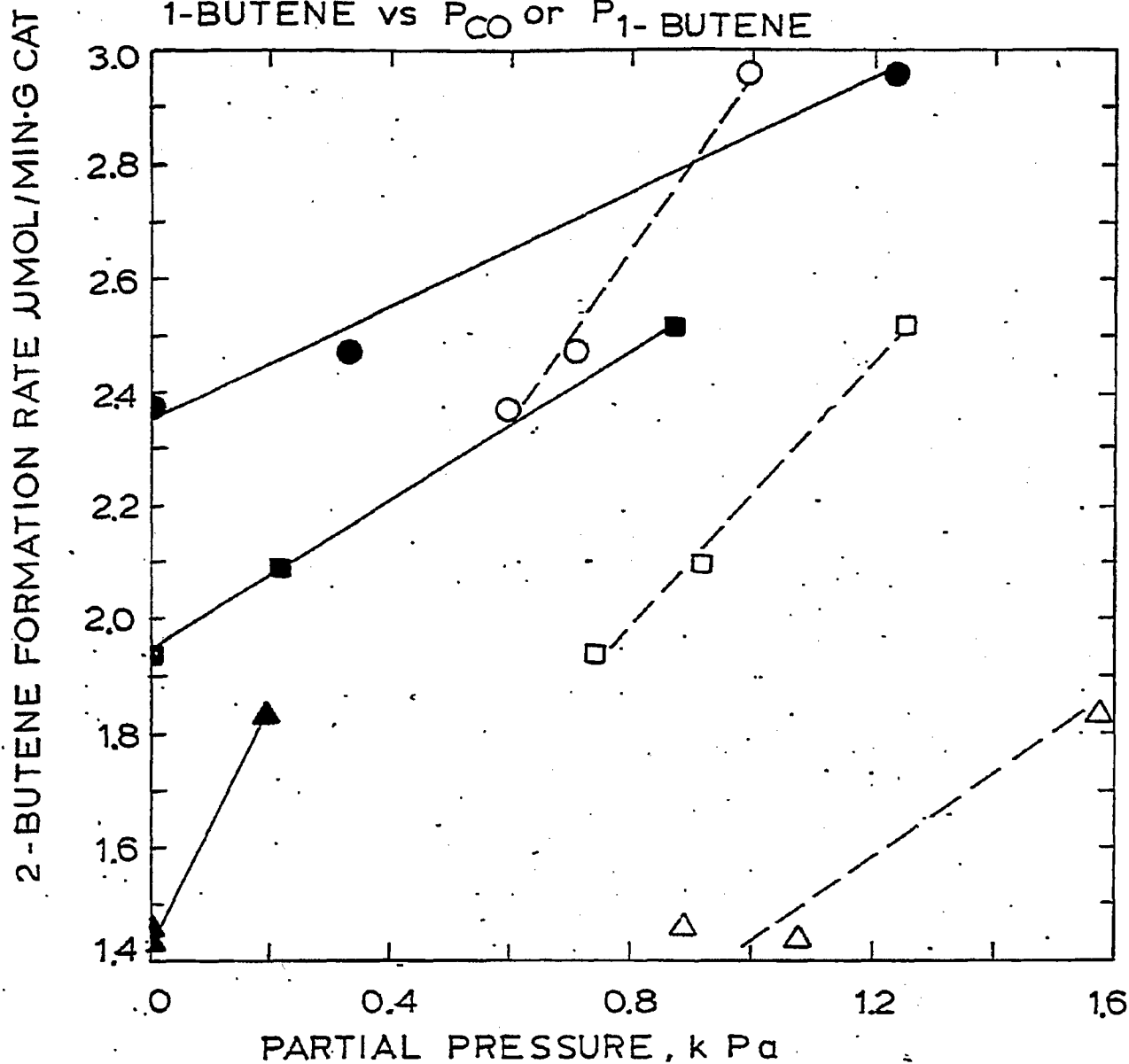


FIGURE 10

RATE OF FORMATION OF 2-BUTENE FROM  
1-BUTENE vs  $P_{CO}$  or  $P_{1-BUTENE}$



- vs pp of CO,  $P_{tot} = 0.31$  MPa
- vs pp of CO,  $P_{tot} = 0.45$  MPa
- ▲ vs pp of CO,  $P_{tot} = 0.79$  MPa
- vs pp of 1-Butene,  $P_{tot} = 0.31$  MPa
- vs pp of 1-Butene,  $P_{tot} = 0.45$  MPa
- △ vs pp of 1-Butene,  $P_{tot} = 0.79$  MPa

FIGURE 11

ADDED OLEFIN HAS NO SIGNIFICANT EFFECT ON FISCHER-TROPSCH  
CARBON NUMBER DISTRIBUTION.  $H_2/CO$  IN FEED = 5,  
TOTAL PRESSURE = 0.79 MPa.

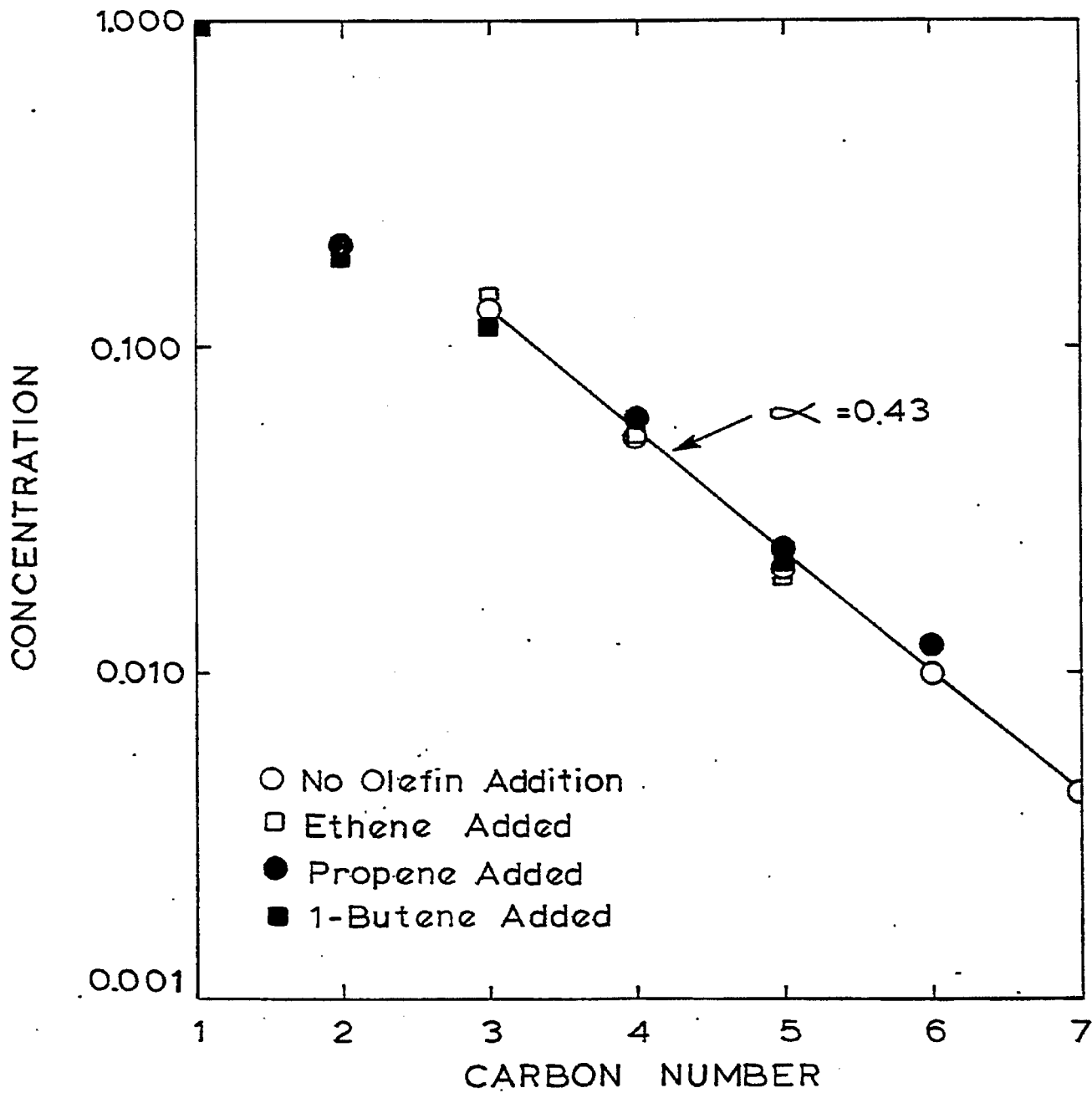


FIGURE - 12

# EFFECT OF H<sub>2</sub>/CO RATIO ON CARBON NUMBER DISTRIBUTION

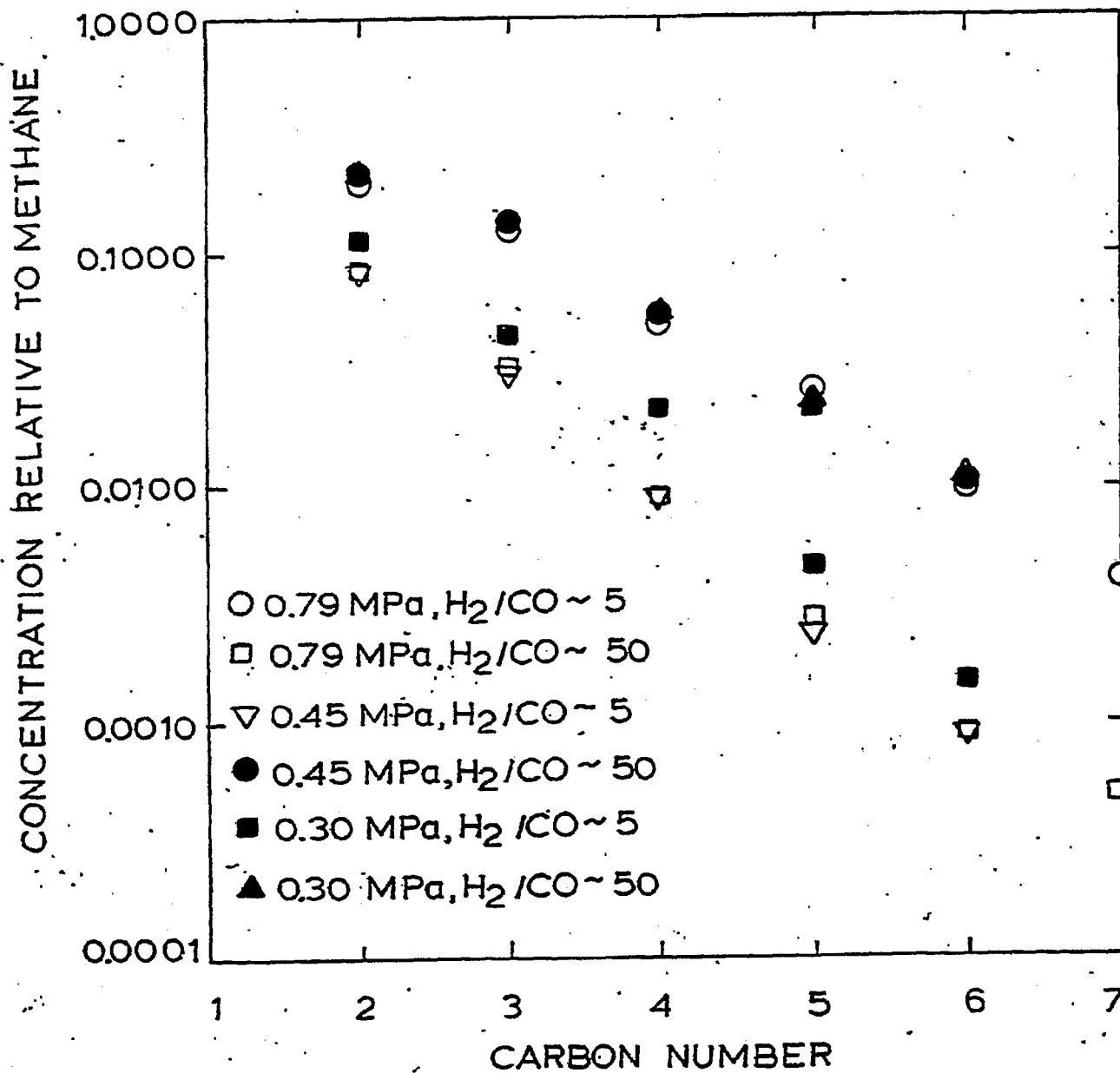


FIGURE 13

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