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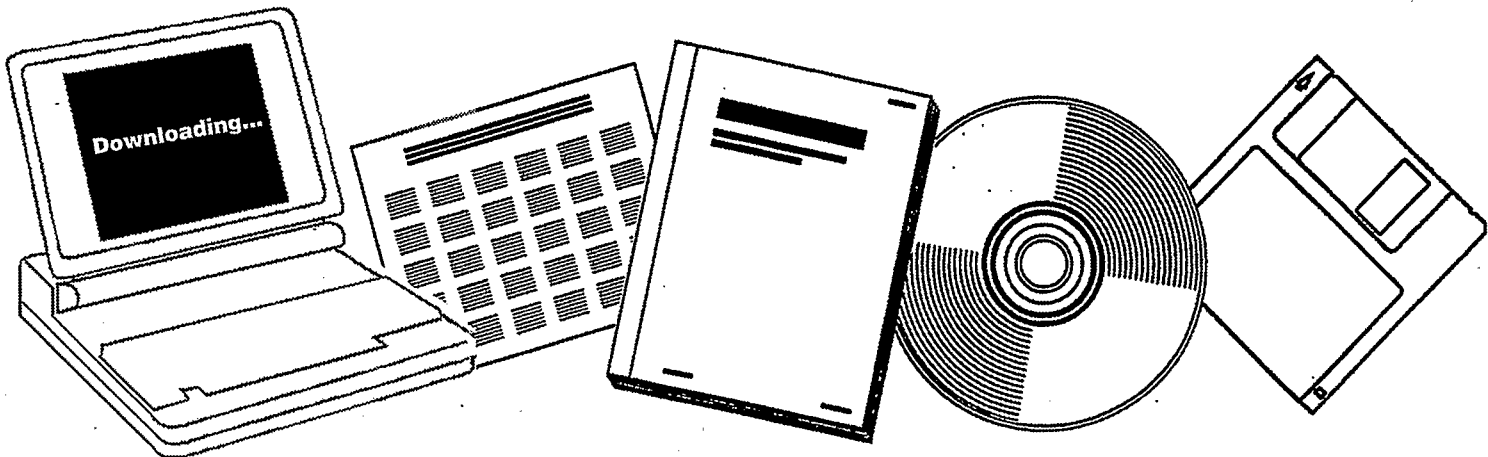
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**TRANSITION METAL-GRAPHITE CATALYSTS FOR
PRODUCTION OF LIGHT HYDROCARBONS FROM
SYNTHESIS GAS. QUARTERLY REPORT, AUGUST 1,
1977--OCTOBER 31, 1977**

TEXAS A AND M UNIV., COLLEGE STATION.
DEPT. OF CHEMISTRY

NOV 1977



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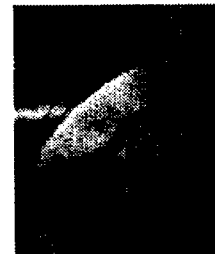
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TRANSITION METAL-GRAPHITE CATALYSTS FOR PRODUCTION
OF LIGHT HYDROCARBONS FROM SYNTHESIS GAS

Quarterly Report for the Period
August 1, 1977 - October 31, 1977

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Date Submitted - November 1977

PREPARED FOR THE UNITED STATES

ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION

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ABSTRACT

Commercial kieselguhr-supported cobalt (containing 39 wt% Co) is an effective catalyst, at 200-300°C, for the Fischer-Tropsch hydrogenation of carbon monoxide. For reaction in a batch system at a total initial pressure of 1 atm and an initial H₂/CO reactant ratio of 2/1, methane is the major carbon-containing product observed in all cases, with selectivities ranging from ~ 55% at 200°C to ~ 90% at 300°C. The increase in selectivity to methane with increasing reaction temperature occurs primarily at the expense of the C₄⁺ hydrocarbon fraction which decreases from ~ 20 mole% of the carbon-containing product at 200°C to virtually zero at 300°C. Production of C₂-C₃ hydrocarbons, on the other hand, is affected much less by variations in reaction temperature, remaining relatively constant at 5-10% of the total product over the temperature range studied.

The total initial rate of carbon monoxide conversion at 250°C is ~ 1.2 moles of CO/mole of Co/hr, with a temperature coefficient of 23.8 kcal/mole. The reaction rate decreases with increasing "time-on-stream" and stabilizes at only ~ 5% of its initial value after 15-20 total hrs of use at 250°C and 1 atm. A doubly-promoted catalyst, containing Co : ThO₂ : MgO = 31.4 : 1.9 : 3.8 wt% on kieselguhr, exhibited a much higher activity, per cobalt atom, and generated substantially less methane and more C₄⁺ hydrocarbons at 200-225°C than did the unpromoted cobalt catalyst. However, its use resulted in considerably higher levels of coke deposition and consequent loss of carbon from the gas phase.

I. OBJECTIVE AND SCOPE OF WORK

The objective of this research is the development of a novel process for the production of petrochemical feedstocks based on coal or other carbonaceous materials. Specifically, the project is to investigate the catalytic activities and selectivities of novel alkali and transition metal-graphites in producing light (C_1 - C_3) hydrocarbons from H_2/CO synthesis gas via the Fischer-Tropsch process.

II. SUMMARY OF PROGRESS TO DATE

A comparison of actual research progress to date vs. project schedule is contained in the "Project Plan and Progress Chart" shown in Fig. 1. In our first Annual Report for this project (containing material for the period August 1, 1976 - July 31, 1977), we described the completion of our preliminary studies of the catalytic behavior of an iron-graphite intercalate for the Fischer-Tropsch synthesis at 1 atm and its comparison to that of a commercial alumina-supported iron catalyst. During the most recent contract quarter, we have begun to evaluate the corresponding activity/selectivity characteristics of cobalt-graphite intercalates for this reaction. Technical details of research progress during the past quarter are described in the following Section.

III. DETAILED DESCRIPTION OF TECHNICAL PROGRESS

In order to establish a suitable basis for comparison of the catalytic behavior of cobalt-graphite intercalates for the Fischer-Tropsch process, studies during the past quarter have focused on characterizing the activity/selectivity features for this reaction of a commercial kieselguhr-supported cobalt catalyst and of a ThO_2/MgO -promoted cobalt catalyst that was prepared in our laboratory. The former was obtained from Harshaw Chemical Co. (No. CO-0127) as 3/16" kieselguhr tablets containing 39 wt% of cobalt in a reduced and stabilized form. The material had a reported BET- N_2 surface area of $115\text{ m}^2/\text{g}$ and a total pore volume of $0.44\text{ cm}^3/\text{g}$. For the present experiments, the tablets were crushed and sized to 20/40 mesh granules, evacuated overnight at 400°C , treated with 1 atm of circulating hydrogen for 8 hrs followed by evacuation for 16 hrs at the same temperature, and then stored under dry argon in a sealed container from which samples were removed as needed for experimentation.

The doubly-promoted cobalt catalyst that was employed for certain comparison experiments was synthesized in our laboratory by preparing the following three solutions: (1) 50 g of 325 mesh kieselguhr (Alfa Chemical Co. No. 89381, surface area = $30\text{ m}^2/\text{g}$) and 3 g of MgO dispersed in 250 ml of water; (2) 123 g of $Co(NO_3)_2 \cdot 6H_2O$ and 3.2 g of $Th(NO_3)_4 \cdot 4H_2O$ dissolved in 650 ml of water, and (3) 46 g of Na_2CO_3 dissolved in 250 ml of water. All solutions were heated to boiling, solutions (1) and (3) were added to

solution (2) with vigorous stirring, the product filtered, washed free of Na^+ ions with 10 liters of de-ionized water, dried for 48 hrs at 110°C , and then crushed and screened to 20/40 mesh granules. Reduction/decomposition of the $\text{CoCO}_3\text{-MgO-Th}(\text{CO}_3)_2$ -containing material was performed in situ in the catalytic reactor following each loading of a fresh sample, by heating at $5^\circ/\text{min}$ in circulating hydrogen to 400°C , maintaining for 3 hrs at the latter temperature, and then evacuating overnight at 300°C . The final material in all cases had a composition corresponding to $\text{Co} : \text{ThO}_2 : \text{MgO} = 31.4 : 1.9 : 3.8 \text{ wt}\%$ on kieselguhr.

All experiments were performed using the closed-loop recirculation reactor and analysis system that was described in our first Quarterly Report for this project (November 1976). Reaction parameters common to all experiments included a total catalyst weight of 0.10 g (except as noted), an initial H_2/CO reactant ratio of 2/1, and a total initial pressure of 750 torr. Results of individual catalyst tests are described in the following sub-Sections.

A. Cobalt/Kieselguhr (39 wt% Co)

1. Catalyst Deactivation and Stabilization

Comparative results obtained from six successive runs of 4-7 hrs each at 250°C using a single sample of the commercial cobalt/kieselguhr catalyst are contained in Table I, and time-dependent conversion data are summarized for all runs in Fig. 2. Catalyst treatment between experiments involved overnight evacuation at 250°C . It is evident that considerable deactivation of the catalyst occurs during the first 15-20 hrs of operation (Runs 1-A to 1-C), presumably due to coke deposition on the catalyst surface, but that little further decrease in activity occurs beyond this point, as shown by the virtually identical activities observed in Runs 1-D to 1-F. (The reason for the abnormally large carbon mass balances observed in Run 1-A is not clear, but may be related to the stabilization procedure employed by Harshaw for this catalyst, viz., surface saturation with carbon dioxide.) However, although catalyst activity remained apparently stabilized and reproducible during the last three runs in the series, the total initial rate of CO conversion to all products in these latter experiments was 0.068 moles of CO/mole of Co/hr, which represents only $\sim 5\%$ of the initial activity observed in Run 1-A.

The product distributions observed for each of these six experiments varied, as expected, with the extent of CO conversion. Although methane was the predominant carbon-containing product ($> 60\%$) at all levels of CO conversion, significant amounts of $\text{C}_2\text{-C}_3$ olefins, which serve as precursors to the corresponding paraffins, were only observed at low conversions ($< \sim 10\%$). At this reaction temperature, carbon dioxide constituted only $\sim 5\text{-}8\%$ of the product, irrespective of the extent of reaction. Although the overall catalyst activity varied considerably among the six experiments, the product distributions, when compared at identical conversion levels, did not differ significantly (compare, for example, the selectivities for

the 1.00, 3.00, and 6.00 hr samples of Runs 1-B, 1-C, and 1-D, respectively).

2. Catalyst Reproducibility

Reproducibility of catalyst behavior was established by performing a series of three runs at 250°C, each employing a separate and freshly-pre-treated catalyst sample. The excellent repeatability of catalytic activities observed for these experiments is displayed in Fig. 3, where the extent of CO conversion is plotted vs. reaction time for each run. Likewise, the selectivities at comparable conversion levels were virtually identical, as shown in Table II where these results are summarized. (Compare, for example, the product distributions at identical reaction times for each of the three runs.) Although these experiments were performed at only a single reaction temperature (250°C), it is deemed likely that the assumption of reproducible behavior among separate catalyst samples would be equally valid at other temperatures in the range 200-300°C, as described in the following sub-Section.

3. Effect of Reaction Temperature on Catalytic Behavior

In order to assess the effect of reaction temperature variations on both the activity and the Fischer-Tropsch selectivity of the cobalt/kieselguhr catalyst, a series of five separate experiments was performed in which the reaction temperature was varied in 25° increments over the range 200-300°C. In view of the considerably lower reaction rates at 200° and 225°C, it was desirable to compare catalytic behaviors at relatively high activity levels. Consequently, each of these runs employed a separate and freshly-treated catalyst sample, since, as demonstrated in Section A.1. above, only the overall activity, and not the product distribution, appeared to be dependent on the extent of catalyst deactivation. The results of these experiments are contained in Table III.

Apart from the expected increase in total CO conversion rate, the principal effect of increasing reaction temperature was to decrease the extent of coke deposition, as shown by the decreasing carbon imbalances with increasing reaction temperature, and to drastically increase the reaction selectivity to methane (~ 50% at 200°C to ~ 90% at 300°C) at the expense of higher hydrocarbons. The most significant reduction occurred for the C₄⁺ fraction which decreased from ~ 20 mole% of the carbon-containing product at 200°C to virtually zero at 300°C. At CO conversions in excess of a few percent, the amount of CO₂ in the product was almost unaffected by changes in reaction temperature.

The temperature dependence of the initial reaction rate is shown, for the range studied, by the pseudo-Arrhenius plot in Fig. 4. The plot is quite linear, and the slope of the "best-fit" line corresponds to a reaction temperature coefficient of 23.8 kcal/mole, which is a typical value for non-diffusion-limited Fischer-Tropsch syntheses over cobalt catalysts.

B. Doubly-Promoted Cobalt/Kieselguhr (31.4 wt% Co)

Two experiments were performed using a "classical" ThO₂/MgO-promoted cobalt/kieselguhr catalyst, in order to determine the effect of these two promoters on the observed activity and Fischer-Tropsch Selectivity behavior under our reaction conditions. The results, for runs at 200° and 225°C, are presented in Table IV, and should be compared, for reference purposes, to the corresponding runs made with the unpromoted cobalt/kieselguhr catalyst (Series 4, Run 1-A and Series 5, Run 1-A in Table III). (Note that, unlike the situation for all other experiments discussed here, the total weight of catalyst employed in Series 2, Run 1-A of Table IV was 0.01 g, and not 0.10g).

As expected, the principal effect of the two promoters on product distribution was to lower reaction selectivity to methane and to increase it for the C₄ and C₅⁺ hydrocarbon fractions. The decline in methane production, relative to the unpromoted catalyst, was more pronounced at 225° than at 200°C. Relatively little change, on the other hand, was observed in the selectivities to CO₂ and C₂-C₃ hydrocarbons. The substantially greater activity, per cobalt atom, observed in both experiments with the promoted catalyst compared to the corresponding runs made using its unpromoted counterpart may be due both to the presence of the two promoters in the former case and/or to different (and undetermined) percentages of exposure of cobalt atoms in the two catalysts. It should also be noted that, at identical conversion levels and for roughly comparable reaction rates (e.g., Series 5, Run 1-A of Table III and Series 2, Run 1-A of Table IV), the levels of coke deposition observed for the promoted cobalt catalyst were noticeably larger than those obtained with the unpromoted material, as indicated by the respective carbon mass balances for the two cases.

IV. CONCLUSIONS

Commercial kieselguhr-supported cobalt (containing 39 wt% Co) is an effective catalyst, at 200-300°C, for the Fischer-Tropsch hydrogenation of carbon monoxide. For reaction in a batch system at a total initial pressure of 1 atm and an initial H₂/CO reactant ratio of 2/1, methane is the major carbon-containing product observed in all cases, with selectivities ranging from ~ 55% at 200°C to ~ 90% at 300°C. The increase in selectivity to methane with increasing reaction temperature occurs primarily at the expense of the C₄⁺ hydrocarbon fraction which decreases from ~ 20 mole% of the carbon-containing product at 200°C to virtually zero at 300°C. Production of C₂-C₃ hydrocarbons, on the other hand, is affected much less by variations in reaction temperature, remaining relatively constant at 5-10% of the total product over the temperature range studied.

The total initial rate of carbon monoxide conversion at 250°C is ~ 1.2

moles of CO/mole of Co/hr, with a temperature coefficient of 23.8 kcal/mole. The reaction rate decreases with increasing "time-on-stream" and stabilizes at only ~ 5% of its initial value after 15-20 total hrs of use at 250°C and 1 atm. A doubly-promoted catalyst, containing Co : ThO₂ : MgO = 31.4 : 1.9 : 3.8 wt% on kieselguhr, exhibited a much higher activity, per cobalt atom, and generated substantially less methane and more C₄⁺ hydrocarbons at 200-225°C than did the unpromoted cobalt catalyst. However, its use resulted in considerably higher levels of coke deposition and consequent loss of carbon from the gas phase.

V. FUTURE WORK

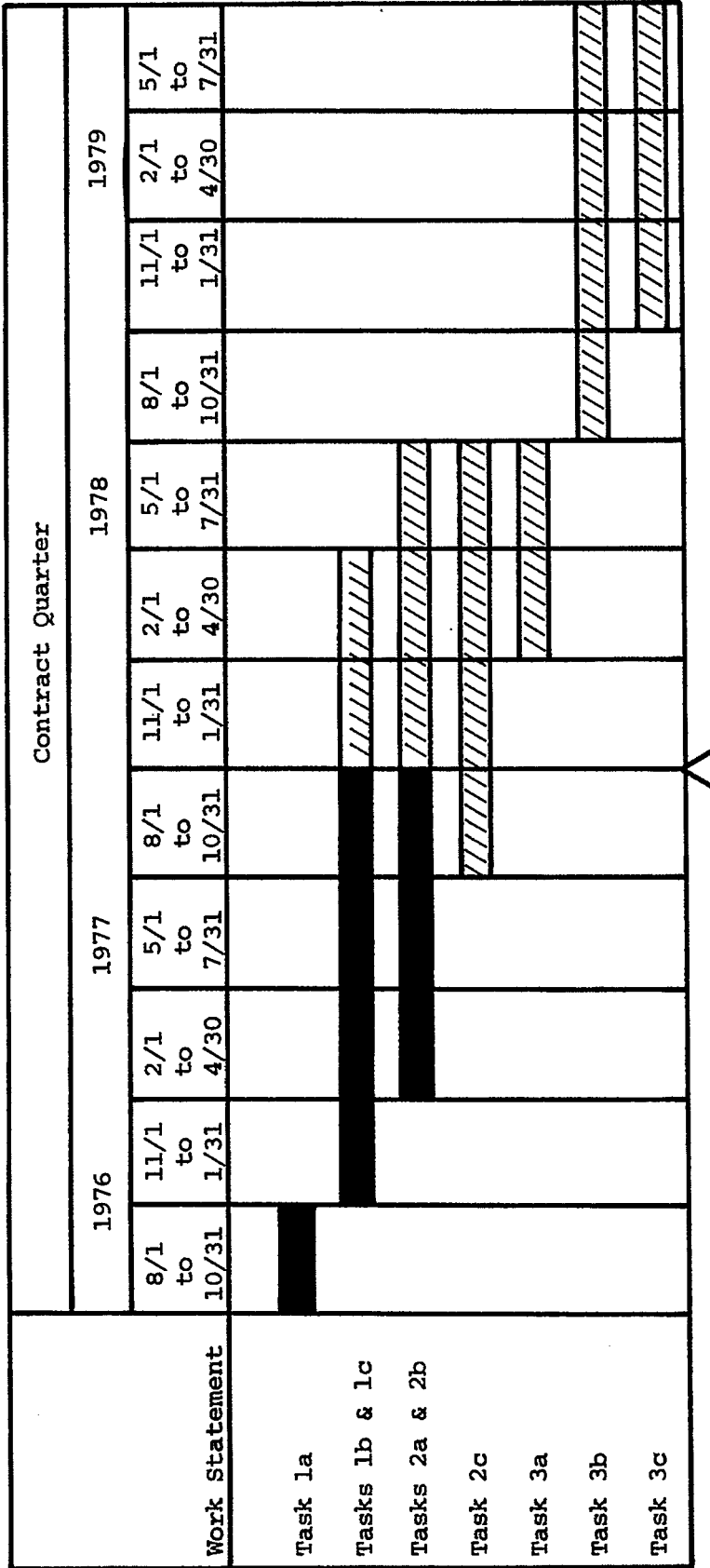
During the next quarter, we plan to begin a study of the catalytic behavior, for the Fischer-Tropsch reaction, of a commercially-available cobalt-graphite intercalate (containing 3.4 wt% Co). Reaction temperature and pretreatment conditions will be varied in an effort to optimize catalyst performance. Emphasis will be placed on identifying and characterizing significant differences in product distribution, catalyst stability, and overall activity between the graphite-based catalyst and the kieselguhr-supported cobalt catalyst described in the present report.

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- Fig. 2 % CO Conversion vs. Reaction Time for Cobalt/Kieselguhr at 250°C.
Data and other reaction parameters are given in Table I.
- Fig. 3 % CO Conversion vs. Reaction Time for Cobalt/Kieselguhr at 250°C.
Data and other reaction parameters are given in Table II.
- Fig. 4 Temperature Dependence of Initial CO Reaction Rate over Cobalt/
Kieselguhr.
Data and other reaction parameters are given in Table III.

FIGURE 1

Project Plan and Progress Chart



LEGEND



Scheduled



Completed



End of Reporting Period

FIGURE 2

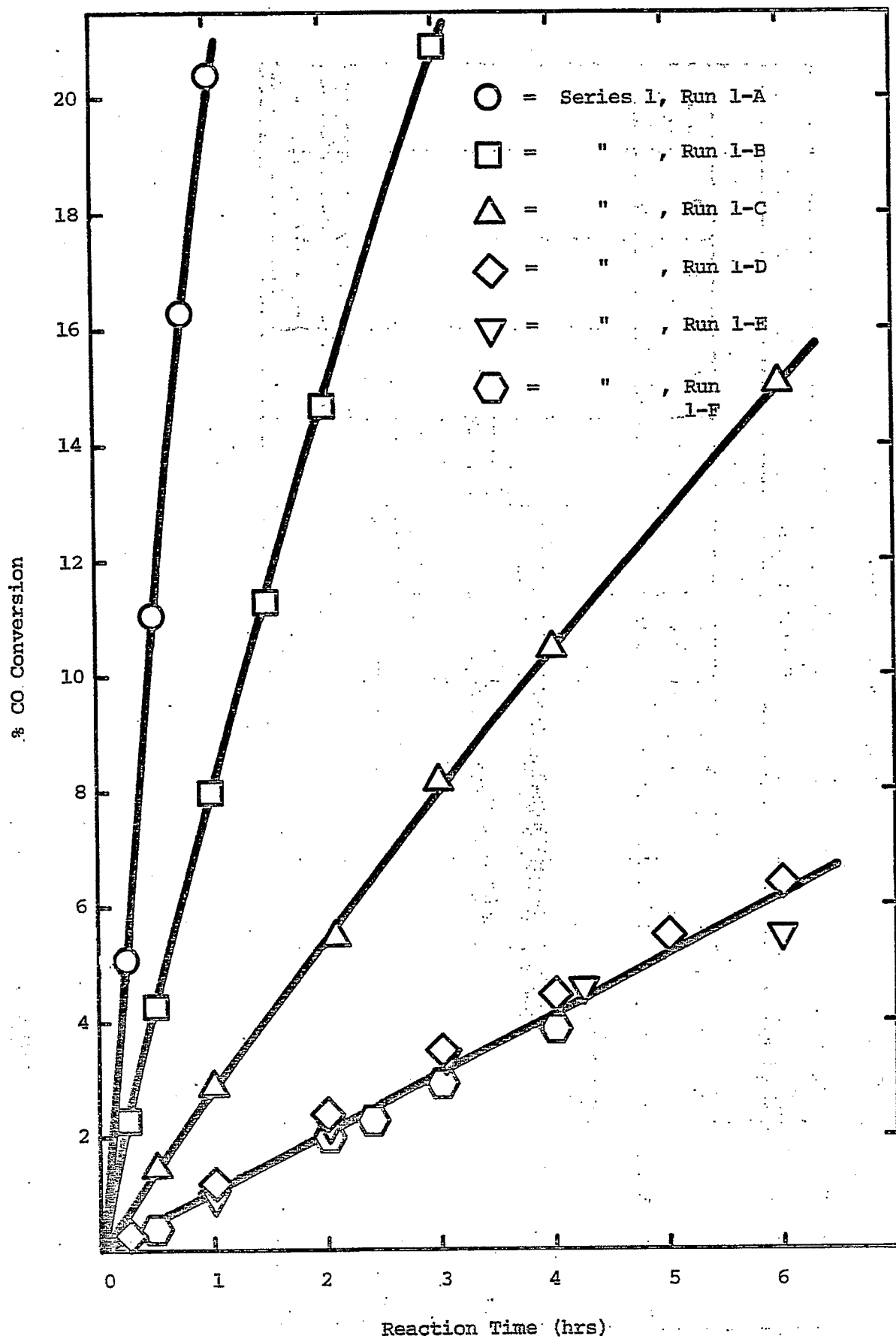


FIGURE 3

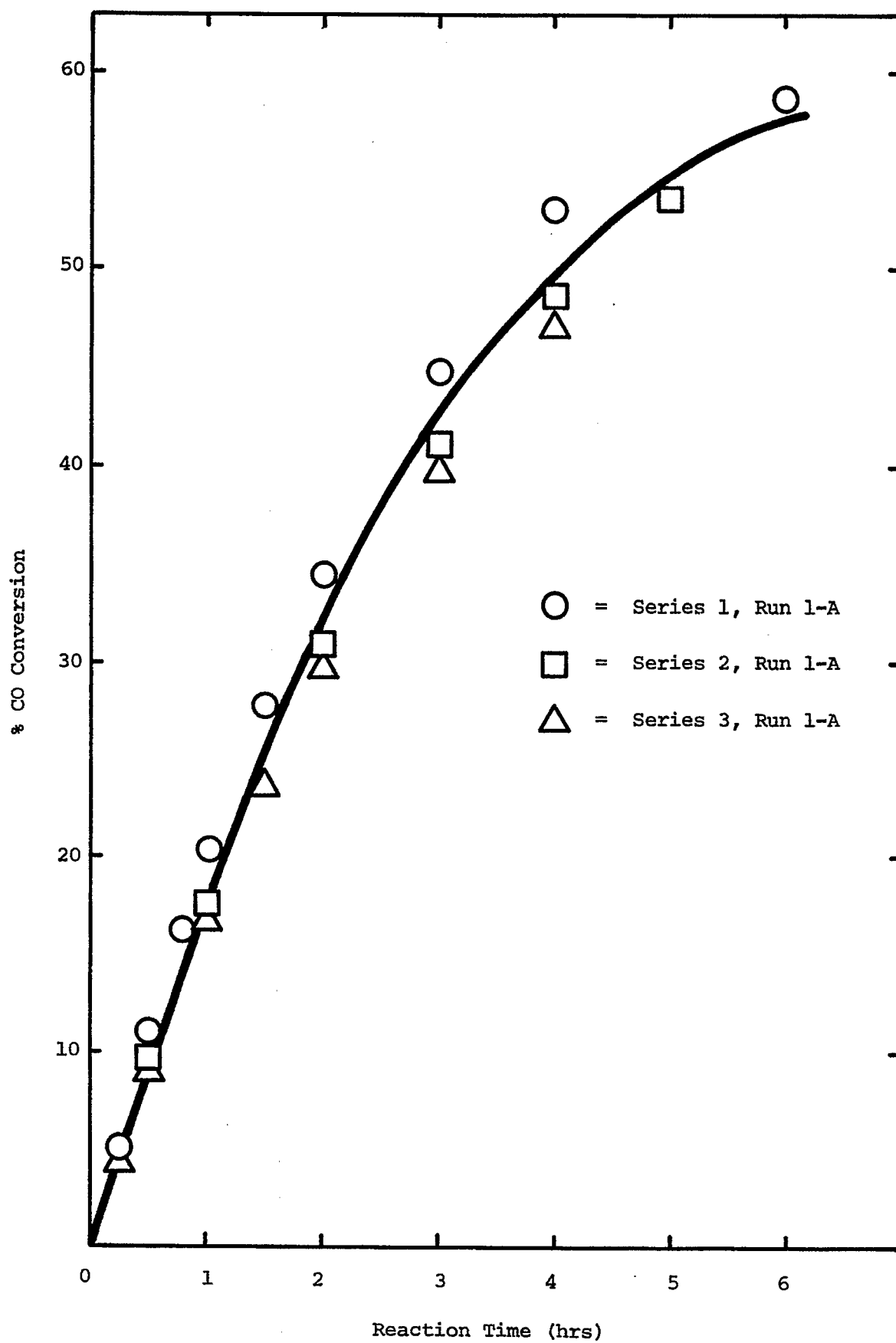


FIGURE 4

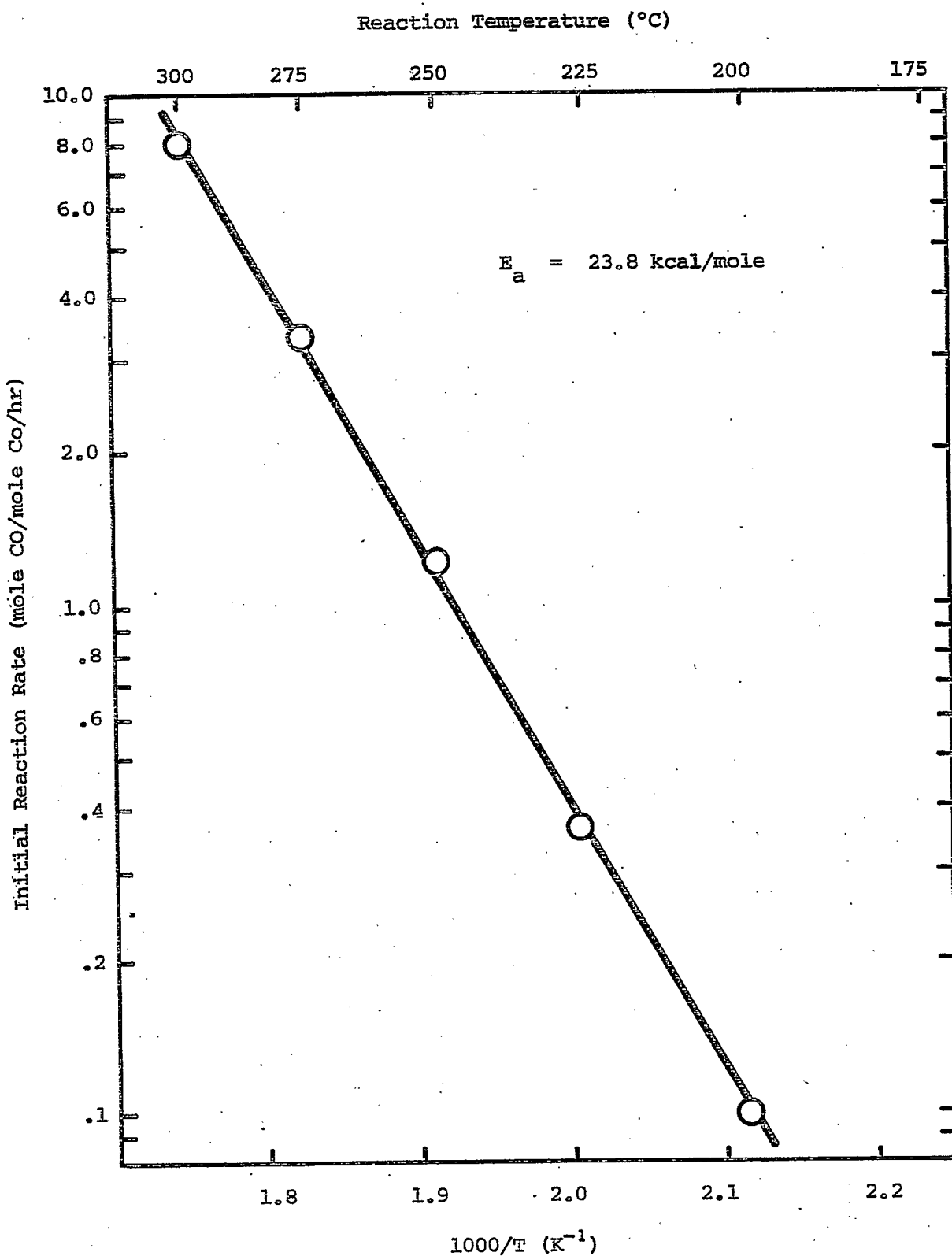


Table I

Fischer-Tropsch Reaction Results

Expt. No. - Series 1, Run 1-A

Catalyst - Cobalt on Kieselguhr (39 wt% Co); Harshaw Chemical Co. No. CO-0127; Pretreated in H₂ for 8 hrs at 400°C, then evacuated for 16 hrs at 300°C.

Weight - 0.10 g Total

Reaction Temperature = 250°C

Initial H₂/CO Ratio = 2.00

Initial Total Pressure = 750.0 Torr

Reaction Time (hrs)	Mole Percent in Gas Phase							
	CO ₂	CH ₄	C ₂ H ₄	C ₂ H ₆	C ₃ H ₆	C ₃ H ₈	C ₄ 's	C ₅ ⁺
0.25	5.2	68.8	0.8	10.4	3.3	6.0	3.9	1.6
0.50	3.4	76.5	0.3	8.5	1.5	5.2	3.0	1.6
0.78	3.1	77.9	0.2	8.4	1.0	5.4	2.6	1.3
1.02	3.0	78.5	0.2	8.4	0.8	5.6	2.5	1.0
1.50	3.0	79.2	0.1	8.3	0.5	5.5	2.3	1.1
2.00	3.0	79.9	0.1	8.1	0.4	5.4	2.1	1.0
3.00	3.2	80.7	0.1	8.0	0.2	5.2	1.9	0.8
4.00	3.5	81.2	0.1	7.8	0.2	4.9	1.7	0.7
6.00	3.8	81.1	0.0	7.7	0.2	4.9	1.7	0.7
7.00	4.6	80.7	0.1	7.6	0.2	4.7	1.6	0.6

Percent CO Conversion

Reaction Time (hrs)	Based on Product Formation	Based on Gas Phase CO Decrease	Rate of CO Conversion (μ-mole/hr)	Percent Carbon Mass Balance in Gas Phase
0.25	5.1	4.3	886.6	100.8
0.50	11.1	8.9	1035.3	102.2
0.78	16.3	12.7	796.2	103.6
1.02	20.4	15.9	734.8	104.5
1.50	27.8	20.6	667.6	107.2
2.00	34.5	27.8	578.7	106.7
3.00	44.8	37.1	444.3	107.6
4.00	53.0	45.1	354.5	107.8
6.00	58.7	50.2	124.6	108.6
7.00	65.1	57.3	274.9	107.8

CO conversions and carbon mass balances are based on the total amount of CO admitted into the reactor at time t = 0.

Table I (cont.)

Fischer-Tropsch Reaction Results

Expt. No. - Series 1, Run 1-B

Catalyst - Cobalt on Kieselguhr (39 wt% Co); Harshaw Chemical Co. No. CO-0127; Evacuated for 16 hrs at 250°C following Series 1, Run 1-A.

Weight - 0.10 g Total

Reaction Temperature = 250°C

Initial H₂/CO Ratio = 2.00

Initial Total Pressure = 750.0 Torr

<u>Reaction Time (hrs)</u>	<u>Mole Percent in Gas Phase</u>							
	<u>CO₂</u>	<u>CH₄</u>	<u>C₂H₄</u>	<u>C₂H₆</u>	<u>C₃H₆</u>	<u>C₃H₈</u>	<u>C₄'s</u>	<u>C₅⁺</u>
0.25	9.4	59.9	4.1	6.6	8.7	2.4	5.4	3.3
0.50	8.4	62.2	2.5	7.9	7.6	3.3	4.7	3.5
1.00	7.9	64.2	1.3	8.6	5.6	4.8	5.0	2.7
1.50	7.8	65.6	0.8	9.0	4.4	5.2	4.7	2.5
2.00	7.8	65.9	0.7	9.0	3.7	5.8	4.6	2.6
3.00	7.9	66.8	0.5	9.0	2.5	6.6	4.4	2.3
4.00	7.9	67.5	0.3	9.1	2.0	7.0	4.2	2.0

<u>Reaction Time (hrs)</u>	<u>Percent CO Conversion</u>			
	<u>Based on Product Formation</u>	<u>Based on Gas Phase CO Decrease</u>	<u>Rate of CO Conversion (μ-mole/hr)</u>	<u>Percent Carbon Mass Balance in Gas Phase</u>
0.25	2.3	1.1	390.6	101.2
0.50	4.3	3.1	355.3	101.2
1.00	8.0	6.2	317.3	101.7
1.50	11.3	9.4	289.9	101.9
2.00	14.7	12.7	297.9	102.1
3.00	20.9	18.1	269.4	102.8
4.00	26.3	23.7	232.8	102.6

CO conversions and carbon mass balances are based on the total amount of CO admitted into the reactor at time t = 0.

Table I (cont.)

Fischer-Tropsch Reaction Results

Expt. No. - Series 1, Run 1-C

Catalyst - Cobalt on Kieselguhr (39 wt% Co); Harshaw Chemical Co. No. CO-0127; Evacuated for 16 hrs at 250°C following Series 1, Run 1-B.

Weight - 0.10 g Total

Reaction Temperature = 250°C

Initial H₂/CO Ratio = 2.00

Initial Total Pressure = 750.0 Torr

<u>Reaction Time (hrs)</u>	<u>Mole Percent in Gas Phase</u>							
	<u>CO₂</u>	<u>CH₄</u>	<u>C₂H₄</u>	<u>C₂H₆</u>	<u>C₃H₆</u>	<u>C₃H₈</u>	<u>C₄'s</u>	<u>C₅⁺</u>
0.25	13.7	55.4	10.4	3.4	9.6	0.0	4.6	2.9
0.50	10.6	58.3	8.7	4.7	10.3	1.4	3.8	2.3
1.00	9.5	58.6	6.3	6.6	9.4	1.7	4.5	3.4
2.08	9.1	60.0	3.8	8.7	8.4	2.8	4.2	3.1
3.00	9.0	60.3	2.5	9.5	7.4	3.6	4.4	3.3
4.00	9.0	60.8	1.9	10.0	6.7	4.2	4.4	3.0
6.00	9.1	61.4	1.3	10.4	5.4	5.2	4.5	2.8

<u>Reaction Time (hrs)</u>	<u>Percent CO Conversion</u>			
	<u>Based on Product Formation</u>	<u>Based on Gas Phase CO Decrease</u>	<u>Rate of CO Conversion (μ-mole/hr)</u>	<u>Percent Carbon Mass Balance in Gas Phase</u>
0.25	0.8	0.3	131.9	100.4
0.50	1.5	1.0	123.2	100.5
1.00	2.9	2.1	125.4	100.8
2.08	5.5	4.8	104.1	100.7
3.00	8.2	6.7	126.3	101.5
4.00	10.5	9.6	101.1	100.9
6.00	15.1	14.2	98.7	100.9

CO conversions and carbon mass balances are based on the total amount of CO admitted into the reactor at time t = 0.

Table I (cont.)

Fischer-Tropsch Reaction Results

Expt. No. - Series I, Run 1-D

Catalyst - Cobalt on Kieselguhr (39 wt% Co); Harshaw Chemical Co. No. CO-0127; Evacuated for 16 hrs at 250°C following Series I, Run 1-C.

Weight - 0.10 g Total

Reaction Temperature = 250°C
 Initial H₂/CO Ratio = 2.00
 Initial Total Pressure = 750.0 Torr

<u>Reaction Time (hrs)</u>	<u>Mole Percent in Gas Phase</u>							
	<u>CO₂</u>	<u>CH₄</u>	<u>C₂H₄</u>	<u>C₂H₆</u>	<u>C₃H₆</u>	<u>C₃H₈</u>	<u>C₄'s</u>	<u>C₅⁺</u>
0.25	18.8	55.5	13.9	2.5	9.2	0.0	0.0	0.0
1.00	8.1	65.9	10.4	4.5	9.1	0.0	1.1	0.9
2.00	6.6	67.5	7.3	6.0	8.5	0.8	2.3	1.0
3.00	6.2	68.2	5.7	7.2	8.2	1.8	1.8	0.9
4.00	5.9	65.8	4.3	7.8	7.8	1.9	3.8	2.6
5.00	5.9	67.3	3.6	8.5	7.3	1.9	3.3	2.2
6.00	6.1	67.6	3.1	8.9	7.2	2.2	2.9	2.0

<u>Reaction Time (hrs)</u>	<u>Percent CO Conversion</u>			
	<u>Based on Product Formation</u>	<u>Based on Gas Phase CO Decrease</u>	<u>Rate of CO Conversion (μ-mole/hr)</u>	<u>Percent Carbon Mass Balance in Gas Phase</u>
0.25	0.3	1.1	45.6	99.2
1.00	1.2	1.9	52.0	99.3
2.00	2.4	3.7	52.4	98.7
3.00	3.5	5.9	50.6	97.7
4.00	5.3	8.2	74.7	97.1
5.00	6.3	10.0	43.1	96.3
6.00	7.2	11.6	43.2	95.6

CO conversions and carbon mass balances are based on the total amount of CO, admitted into the reactor at time t = 0.

Table I (cont.)

Fischer-Tropsch Reaction Results

Expt. No. - Series 1, Run 1-E

Catalyst - Cobalt on Kieselguhr (39 wt% Co); Harshaw Chemical Co. No. CO-0127; Evacuated for 16 hrs at 250°C following Series 1, Run 1-D.

Weight - 0.10 g Total

Reaction Temperature = 249°C

Initial H₂/CO Ratio = 2.00

Initial Total Pressure = 750.0 Torr

<u>Reaction Time (hrs)</u>	<u>Mole Percent in Gas Phase</u>							
	<u>CO₂</u>	<u>CH₄</u>	<u>C₂H₄</u>	<u>C₂H₆</u>	<u>C₃H₆</u>	<u>C₃H₈</u>	<u>C₄'s</u>	<u>C₅⁺</u>
1.00	8.4	67.7	11.5	3.4	9.1	0.0	0.0	0.0
2.00	6.2	67.6	8.4	4.6	8.3	0.0	2.7	2.2
3.03	5.8	66.7	8.5	5.6	7.8	0.6	2.8	2.3
4.25	5.8	68.8	5.6	6.7	7.1	1.2	2.6	2.2
6.00	5.7	68.5	4.8	7.1	7.5	1.4	2.9	2.0

<u>Reaction Time (hrs)</u>	<u>Percent CO Conversion</u>			
	<u>Based on Product Formation</u>	<u>Based on Gas Phase CO Decrease</u>	<u>Rate of CO Conversion (μ-mole/hr)</u>	<u>Percent Carbon Mass Balance in Gas Phase</u>
1.00	0.9	1.3	37.4	99.6
2.00	2.2	2.7	55.1	99.4
3.03	3.4	3.0	52.9	100.4
4.25	4.6	5.2	40.6	99.4
6.00	5.5	6.8	21.5	98.6

CO conversions and carbon mass balances are based on the total amount of CO admitted into the reactor at time $t = 0$.

Table I (cont.)

Fischer-Tropsch Reaction Results

Expt. No. - Series I, Run I-F

Catalyst - Cobalt on Kieselguhr (39 wt% Co); Harshaw Chemical Co. No. CO-0127; Evacuated for 16 hrs at 250°C following Series I, Run I-E.

Weight - 0.10 g Total

Reaction Temperature = 249°C

Initial H₂/CO Ratio = 2.00

Initial Total Pressure = 750.0 Torr

Reaction Time (hrs)	Mole Percent in Gas Phase							
	CO ₂	CH ₄	C ₂ H ₄	C ₂ H ₆	C ₃ H ₆	C ₃ H ₈	C ₄ 's	C ₅ ⁺
0.50	15.7	62.1	11.4	2.4	8.4	0.0	0.0	0.0
1.00	9.7	64.4	10.2	2.9	7.9	1.1	2.4	1.4
2.00	7.3	68.9	8.6	4.3	8.1	0.5	1.5	0.9
2.40	7.0	69.1	8.1	4.6	8.1	0.6	1.5	0.9
3.00	6.8	69.2	7.3	5.1	7.9	0.9	1.8	0.9
4.00	6.5	69.8	6.2	6.0	7.8	1.3	1.8	0.7

Percent CO Conversion

Reaction Time (hrs)	Based on Product Formation	Based on Gas Phase CO Decrease	Rate of CO Conversion (μ-mole/hr)	Percent Carbon Mass Balance in Gas Phase
0.50	0.4	-4.1	36.3	104.5
1.00	1.0	-1.5	50.6	102.5
2.00	2.0	-0.2	42.1	102.1
2.40	2.3	0.8	36.9	101.5
3.00	2.9	2.6	44.0	100.3
4.00	3.9	3.8	43.0	100.1

CO conversions and carbon mass balances are based on the total amount of CO admitted into the reactor at time t = 0.

Table II

Fischer-Tropsch Reaction Results

Expt. No. - Series 1, Run 1-A

Catalyst - Cobalt on Kieselguhr (39 wt% Co); Harshaw Chemical Co. No. CO-0127; Pretreated in H₂ for 8 hrs at 400°C, then evacuated for 16 hrs at 300°C.

Weight - 0.10 g Total

Reaction Temperature = 250°C

Initial H₂/CO Ratio = 2.00

Initial Total Pressure = 750.0 Torr

Reaction Time (hrs)	Mole Percent in Gas Phase							
	CO ₂	CH ₄	C ₂ H ₄	C ₂ H ₆	C ₃ H ₆	C ₃ H ₈	C ₄ 's	C ₅ ⁺
0.25	5.2	68.8	0.8	10.4	3.3	6.0	3.9	1.6
0.50	3.4	76.5	0.3	8.5	1.5	5.2	3.0	1.6
0.78	3.1	77.9	0.2	8.4	1.0	5.4	2.6	1.3
1.02	3.0	78.5	0.2	8.4	0.8	5.6	2.5	1.0
1.50	3.0	79.2	0.1	8.3	0.5	5.5	2.3	1.1
2.00	3.0	79.9	0.1	8.1	0.4	5.4	2.1	1.0
3.00	3.2	80.7	0.1	8.0	0.2	5.2	1.9	0.8
4.00	3.5	81.2	0.1	7.8	0.2	4.9	1.7	0.7
6.00	3.8	81.1	0.0	7.7	0.2	4.9	1.7	0.7
7.00	4.6	80.7	0.1	7.6	0.2	4.7	1.6	0.6

Percent CO Conversion

Reaction Time (hrs)	Based on Product Formation	Based on Gas Phase CO Decrease	Rate of CO Conversion (μ-mole/hr)	Percent Carbon Mass Balance in Gas Phase
0.25	5.1	4.3	886.6	100.8
0.50	11.1	8.9	1035.3	102.2
0.78	16.3	12.7	796.2	103.6
1.02	20.4	15.9	734.8	104.5
1.50	27.8	20.6	667.6	107.2
2.00	34.5	27.8	578.7	106.7
3.00	44.8	37.1	444.4	107.6
4.00	53.0	45.1	354.5	107.8
6.00	58.7	50.2	124.6	108.6
7.00	65.1	57.3	274.9	107.8

CO conversions and carbon mass balances are based on the total amount of CO admitted into the reactor at time t = 0.

Table II (cont.)

Fischer-Tropsch Reaction Results

Expt. No. - Series 2, Run 1-A

Catalyst - Cobalt on Kieselguhr (39 wt% Co); Harshaw Chemical Co. No. CO-0127; Pretreated in H₂ for 8 hrs at 400°C, then evacuated for 16 hrs at 300°C.

Weight - 0.10 g Total

Reaction Temperature = 250°C

Initial H₂/CO Ratio = 2.00

Initial Total Pressure = 750.0 Torr

Reaction Time (hrs)	Mole Percent in Gas Phase							
	CO ₂	CH ₄	C ₂ H ₄	C ₂ H ₆	C ₃ H ₆	C ₃ H ₈	C ₄ 's	C ₅ ⁺
0.25	5.4	73.4	0.6	8.2	3.3	4.3	3.1	1.6
0.50	4.1	75.4	0.3	8.3	1.9	5.3	3.1	1.6
1.00	3.5	77.3	0.2	8.3	0.9	5.5	2.8	1.5
2.00	3.4	78.6	0.1	8.1	0.4	5.5	2.5	1.4
3.00	3.5	79.6	0.1	8.0	0.3	5.2	2.2	1.2
4.00	3.8	79.9	0.0	7.9	0.2	5.1	2.1	1.1
5.00	4.2	80.2	0.0	7.8	0.1	4.9	2.0	0.9

Reaction Time (hrs)	Percent CO Conversion			
	Based on Product Formation	Based on Gas Phase CO Decrease	Rate of CO Conversion (μ-mole/hr)	Percent Carbon Mass Balance in Gas Phase
0.25	5.1	7.0	890.3	98.2
0.50	9.7	12.4	792.8	97.3
1.00	17.7	21.4	697.9	96.3
2.00	31.0	34.8	577.0	96.2
3.00	41.2	45.6	439.1	95.6
4.00	48.7	50.6	327.7	98.1
5.00	53.6	57.4	212.5	96.2

CO conversions and carbon mass balances are based on the total amount of CO admitted into the reactor at time t = 0.

Table II (cont.)

Fischer-Tropsch Reaction Results

Expt. No. - Series 3, Run 1-A

Catalyst - Cobalt on Kieselguhr (39 wt% Co); Harshaw Chemical Co. No. CO-0127; Pretreated in H₂ for 8 hrs at 400°C, then evacuated for 16 hrs at 300°C.

Weight - 0.10 g Total

Reaction Temperature = 250°C

Initial H₂/CO Ratio = 2.00

Initial Total Pressure = 750.0 Torr

<u>Reaction Time (hrs)</u>	<u>Mole Percent in Gas Phase</u>							
	<u>CO₂</u>	<u>CH₄</u>	<u>C₂H₄</u>	<u>C₂H₆</u>	<u>C₃H₆</u>	<u>C₃H₈</u>	<u>C₄'s</u>	<u>C₅⁺</u>
0.25	8.0	73.1	0.7	7.9	3.6	3.9	1.9	0.9
0.50	5.2	75.2	0.4	8.0	2.0	4.8	3.0	1.4
1.00	4.0	77.7	0.2	8.0	0.9	5.1	2.8	1.2
1.50	3.7	78.8	0.1	7.9	0.6	5.2	2.5	1.2
2.00	3.6	79.4	0.1	7.8	0.4	5.2	2.3	1.2
3.00	3.6	80.3	0.1	7.6	0.3	4.9	2.1	1.1
4.00	3.8	80.7	0.0	7.5	0.2	4.7	2.0	1.1

<u>Reaction Time (hrs)</u>	<u>Percent CO Conversion</u>			
	<u>Based on Product Formation</u>	<u>Based on Gas Phase CO Decrease</u>	<u>Rate of CO Conversion (μ-mole/hr)</u>	<u>Percent Carbon Mass Balance in Gas Phase</u>
0.25	4.4	4.4	761.9	100.0
0.50	9.0	12.9	797.6	96.0
1.00	16.7	21.4	677.0	95.3
1.50	23.7	29.1	602.9	94.6
2.00	29.8	34.6	535.2	95.2
3.00	39.8	43.6	435.6	96.2
4.00	47.1	51.3	317.1	95.8

CO conversions and carbon mass balances are based on the total amount of CO admitted into the reactor at time t = 0.

Table III

Fischer-Tropsch Reaction Results

Expt. No. - Series 4, Run 1-A

Catalyst - Cobalt on Kieselguhr (39 wt% Co); Harshaw Chemical Co. No. CO-0127; Pretreated in H₂ for 8 hrs at 400°C; then evacuated for 16 hrs at 300°C.

Weight - 0.10 g Total

Reaction Temperature = 200°C

Initial H₂/CO Ratio = 2.00

Initial Total Pressure = 750.0 Torr

Reaction Time (hrs)	Mole Percent in Gas Phase							
	CO ₂	CH ₄	C ₂ H ₄	C ₂ H ₆	C ₃ H ₆	C ₃ H ₈	C ₄ 's	C ₅ ⁺
0.26	87.2	0.0	5.0	0.0	1.2	0.0	2.7	3.9
0.50	42.1	43.2	4.0	4.3	1.4	0.0	2.5	2.5
1.00	20.6	47.7	1.8	5.8	9.1	3.2	5.8	6.0
2.00	11.0	52.6	0.8	5.9	6.5	4.5	8.0	10.6
3.00	8.2	54.8	0.5	6.1	5.6	5.5	8.1	11.1
4.51	6.5	55.1	0.3	6.3	3.9	7.0	9.0	11.9
5.00	6.1	55.6	0.3	6.3	3.6	7.3	9.3	11.5
6.00	5.7	56.1	0.2	6.4	3.1	7.8	9.2	11.6

Reaction Time (hrs)	Percent CO Conversion			
	Based on Product Formation	Based on Gas Phase CO Decrease	Rate of CO Conversion (μ-mole/hr)	Percent Carbon Mass Balance in Gas Phase
0.26	0.2	2.4	26.1	97.8
0.50	0.3	2.7	32.2	97.7
1.00	1.0	4.0	61.3	97.0
2.00	2.6	6.8	68.6	95.8
3.00	4.1	9.7	65.8	94.5
4.51	6.6	13.5	71.1	93.1
5.00	7.3	14.4	63.1	92.9
6.00	8.9	17.3	66.8	91.5

CO conversions and carbon mass balances are based on the total amount of CO admitted into the reactor at time t = 0.

Table III (cont.)

Fischer-Tropsch Reaction Results

Expt. No. - Series 5, Run 1-A

Catalyst - Cobalt on Kieselguhr (39 wt% Co); Harshaw Chemical Co. No. CO-0127; Pretreated in H₂ for 8 hrs at 400°C, then evacuated for 16 hrs at 300°C.

Weight - 0.10 g Total

Reaction Temperature = 226°C
Initial H₂/CO Ratio = 2.00
Initial Total Pressure = 750.0 Torr

<u>Reaction Time (hrs)</u>	<u>Mole Percent in Gas Phase</u>							
	<u>CO₂</u>	<u>CH₄</u>	<u>C₂H₄</u>	<u>C₂H₆</u>	<u>C₃H₆</u>	<u>C₃H₈</u>	<u>C₄'s</u>	<u>C₅⁺</u>
0.25	11.3	55.3	2.6	6.3	8.6	2.8	7.0	6.0
0.50	6.8	63.0	1.3	7.2	7.7	4.0	5.7	4.3
1.00	4.6	65.3	0.6	7.8	5.2	6.0	6.1	4.5
2.00	3.5	69.4	0.3	7.4	2.3	6.9	4.8	5.5
3.00	3.3	68.4	0.2	7.9	1.5	7.7	6.0	5.0
4.05	3.2	68.8	0.1	7.8	1.1	7.9	5.8	5.3
5.00	3.2	69.5	0.1	7.8	0.8	7.9	5.7	5.0

<u>Reaction Time (hrs)</u>	<u>Percent CO Conversion</u>			
	<u>Based on Product Formation</u>	<u>Based on Gas Phase CO Decrease</u>	<u>Rate of CO Conversion (μ-mole/hr)</u>	<u>Percent Carbon Mass Balance in Gas Phase</u>
0.25	1.3	2.6	224.9	98.7
0.50	2.6	5.1	229.4	97.5
1.00	5.7	9.0	266.5	96.7
2.00	11.9	17.3	270.4	94.7
3.00	17.2	24.4	229.1	92.9
4.05	22.9	30.8	234.6	92.1
5.00	27.3	36.0	201.9	91.4

CO conversions and carbon mass balances are based on the total amount of CO admitted into the reactor at time t = 0.

Table III (cont.)

Fischer-Tropsch Reaction Results

Expt. No. - Series 3, Run 1-A

Catalyst - Cobalt on Kieselguhr (39 wt% Co); Harshaw Chemical Co. No. CO-0127; Pretreated in H₂ for 8 hrs at 400°C, then evacuated for 16 hrs at 300°C.

Weight - 0.10 g Total

Reaction Temperature = 250°C

Initial H₂/CO Ratio = 2.00

Initial Total Pressure = 750.0 Torr

Reaction Time (hrs)	Mole Percent in Gas Phase							
	CO ₂	CH ₄	C ₂ H ₄	C ₂ H ₆	C ₃ H ₆	C ₃ H ₈	C ₄ 's	C ₅ ⁺
0.25	8.0	73.1	0.7	7.9	3.6	3.9	1.9	0.9
0.50	5.2	75.2	0.4	8.0	2.0	4.8	3.0	1.4
1.00	4.0	77.7	0.2	8.0	0.9	5.1	2.8	1.2
1.50	3.7	78.8	0.1	7.9	0.6	5.2	2.5	1.2
2.00	3.6	79.4	0.1	7.8	0.4	5.2	2.3	1.2
3.00	3.6	80.3	0.1	7.6	0.3	4.9	2.1	1.1
4.00	3.8	80.7	0.0	7.5	0.2	4.7	2.0	1.1

Percent CO Conversion

Reaction Time (hrs)	Based on Product Formation	Based on Gas Phase CO Decrease	Rate of CO Conversion (μ-mole/hr)	Percent Carbon Mass Balance in Gas Phase
0.25	4.4	4.4	761.9	100.0
0.50	9.0	12.9	797.6	96.0
1.00	16.7	21.4	677.0	95.3
1.50	23.7	29.1	602.9	94.6
2.00	29.8	34.6	535.2	95.2
3.00	39.8	43.6	435.6	96.2
4.00	47.1	51.3	317.1	95.8

CO conversions and carbon mass balances are based on the total amount of CO admitted into the reactor at time t = 0.

Table III (cont.)

Fischer-Tropsch Reaction Results

Expt. No. - Series 6, Run 1-A

Catalyst - Cobalt on Kieselguhr (39 wt% Co); Harshaw Chemical Co. No. CO-0127; Pretreated in H₂ for 8 hrs at 400°C, then evacuated for 16 hrs at 300°C.

Weight - 0.10 g Total

Reaction Temperature = 275°C

Initial H₂/CO Ratio = 2.00

Initial Total Pressure = 750.0 Torr

<u>Reaction Time (hrs)</u>	<u>Mole Percent in Gas Phase</u>							
	<u>CO₂</u>	<u>CH₄</u>	<u>C₂H₄</u>	<u>C₂H₆</u>	<u>C₃H₆</u>	<u>C₃H₈</u>	<u>C₄'s</u>	<u>C₅⁺</u>
0.25	3.6	87.4	0.2	5.9	0.5	1.9	0.4	0.0
0.50	3.5	88.0	0.1	5.7	0.2	1.9	0.4	0.1
0.75	3.6	88.5	0.1	5.5	0.1	1.8	0.3	0.1
1.00	3.8	88.5	0.1	5.4	0.1	1.7	0.4	0.1
1.50	4.2	88.5	0.0	5.2	0.1	1.6	0.3	0.0
2.01	4.8	87.9	0.0	5.2	0.1	1.6	0.3	0.0

<u>Reaction Time (hrs)</u>	<u>Percent CO Conversion</u>			
	<u>Based on Product Formation</u>	<u>Based on Gas Phase CO Decrease</u>	<u>Rate of CO Conversion (μ-mole/hr)</u>	<u>Percent Carbon Mass Balance in Gas Phase</u>
0.25	12.1	12.8	2108.2	99.4
0.50	22.7	23.0	1833.9	99.7
0.75	31.3	31.7	1490.0	99.6
1.00	38.3	38.8	1223.1	99.6
1.50	48.6	49.3	889.6	99.3
2.01	54.8	56.0	526.2	98.8

CO conversions and carbon mass balances are based on the total amount of CO admitted into the reactor at time t = 0.

Table III (cont.)

Fischer-Tropsch Reaction Results

Expt. No. - Series 7, Run 1-A

Catalyst - Cobalt on Kieselguhr (39 wt% Co); Harshaw Chemical Co. No. CO-0127; Pretreated in H₂ for 8 hrs at 400°C; then evacuated for 16 hrs at 300°C.

Weight - 0.10 g Total

Reaction Temperature = 300°C

Initial H₂/CO Ratio = 2.00

Initial Total Pressure = 750.0 Torr

<u>Reaction Time (hrs)</u>	<u>Mole Percent in Gas Phase</u>							
	<u>CO₂</u>	<u>CH₄</u>	<u>C₂H₄</u>	<u>C₂H₆</u>	<u>C₃H₆</u>	<u>C₃H₈</u>	<u>C₄'s</u>	<u>C₅⁺</u>
0.25	6.4	90.3	0.1	2.9	0.1	0.3	0.0	0.0
0.51	6.8	89.7	0.0	2.9	0.1	0.4	0.0	0.0
1.00	7.9	88.4	0.1	3.1	0.1	0.4	0.0	0.0
1.50	8.8	87.0	0.1	3.2	0.2	0.5	0.1	0.0

<u>Reaction Time (hrs)</u>	<u>Percent CO Conversion</u>			
	<u>Based on Product Formation</u>	<u>Based on Gas Phase CO Decrease</u>	<u>Rate of CO Conversion (μ-mole/hr)</u>	<u>Percent Carbon Mass Balance in Gas Phase</u>
0.25	26.2	28.0	4528.1	98.2
0.51	40.1	41.9	2320.4	98.2
1.00	51.9	55.0	1039.3	96.9
1.50	56.6	59.6	405.8	97.0

CO conversions and carbon mass balances are based on the total amount of CO admitted into the reactor at time t = 0.

Table IV

Fischer-Tropsch Reaction Results

Expt. No. - Series 1, Run 1-A

Catalyst - Cobalt on Kieselguhr, doubly-promoted (Co : ThO₂ : MgO : Kieselguhr = 31.4 : 1.9 : 3.8 : 63.0 wt%); Reduced in H₂ for 3 hrs at 400°C, then evacuated for 16 hrs at 300°C.

Weight - 0.10 g Total

Reaction Temperature = 200°C

Initial H₂/CO Ratio = 2.00

Initial Total Pressure = 750.0 Torr

Reaction Time (hrs)	Mole Percent in Gas Phase							
	CO ₂	CH ₄	C ₂ H ₄	C ₂ H ₆	C ₃ H ₆	C ₃ H ₈	C ₄ 's	C ₅ ⁺
0.25	4.7	47.4	0.6	6.1	6.1	5.8	12.2	17.1
0.50	3.5	48.2	0.4	6.1	5.3	6.8	12.2	17.4
1.00	2.9	49.7	0.2	6.4	3.4	8.4	12.0	16.8
1.50	2.8	49.9	0.1	6.5	2.3	9.2	12.7	16.5
2.00	2.8	50.7	0.1	6.7	1.7	9.8	12.5	15.8
2.50	2.8	51.2	0.1	6.8	1.4	10.2	12.2	15.3
3.00	2.9	51.6	0.1	6.9	1.2	10.4	12.1	14.9

Reaction Time (hrs)	Percent CO Conversion			
	Based on Product Formation	Based on Gas Phase CO Decrease	Rate of CO Conversion (μ-mole/hr)	Percent Carbon Mass Balance in Gas Phase
0.25	5.9	14.9	1022.2	91.0
0.50	10.7	28.3	848.2	82.4
1.00	18.2	40.9	650.7	77.3
1.50	24.7	52.9	565.3	71.8
2.00	29.6	61.8	431.3	67.8
2.50	33.4	69.0	328.4	64.4
3.00	36.7	74.6	283.7	62.0

CO conversions and carbon mass balances are based on the total amount of CO admitted into the reactor at time t = 0.

Table IV (cont.)

Fischer-Tropsch Reaction Results

Expt. No. - Series 2, Run 1-A

Catalyst - Cobalt on Kieselguhr, doubly-promoted (Co : ThO₂ : MgO : Kieselguhr = 31.4 : 1.9 : 3.8 : 63.0 wt%); Reduced in H₂ for 3 hrs at 400°C, then evacuated for 16 hrs at 300°C.

Weight - 0.01 g Total

Reaction Temperature = 225°C

Initial H₂/CO Ratio = 2.00

Initial Total Pressure = 750.0 Torr

Reaction Time (hrs)	Mole Percent in Gas Phase							
	CO ₂	CH ₄	C ₂ H ₄	C ₂ H ₆	C ₃ H ₆	C ₃ H ₈	C ₄ 's	C ₅ ⁺
0.25	6.4	53.2	7.1	5.5	17.4	0.0	3.5	6.8
0.50	4.9	56.2	4.5	7.1	14.1	0.0	5.5	7.7
1.00	3.7	52.3	2.0	7.0	11.4	3.1	8.7	11.7
1.50	3.4	51.8	1.5	6.9	9.9	3.9	10.3	12.3
2.00	3.4	52.8	1.2	7.3	9.0	4.0	10.2	12.1
3.00	3.3	54.3	0.8	7.5	7.9	5.1	9.8	11.4
5.51	3.2	55.6	0.5	7.7	5.9	6.5	9.8	10.8
7.00	3.2	56.6	0.4	7.8	5.1	7.1	9.7	10.1
9.00	3.3	57.2	0.4	7.9	4.2	7.4	9.5	10.0
16.38	3.5	58.7	0.2	8.0	2.9	8.3	9.1	9.2
18.00	3.5	58.8	0.2	8.0	2.7	8.4	9.2	9.1

Reaction Time (hrs)	Percent CO Conversion			
	Based on Product Formation	Based on Gas Phase CO Decrease	Rate of CO Conversion (μ-mole/hr)	Percent Carbon Mass Balance in Gas Phase
0.25	0.6	2.2	98.9	98.4
0.50	1.2	4.8	105.0	96.4
1.00	3.0	6.1	160.0	96.9
1.50	4.7	8.8	144.3	95.9
2.00	6.0	12.0	115.9	94.1
3.00	8.5	16.8	108.0	91.7
5.51	14.3	24.5	100.2	89.8
7.00	17.2	29.7	81.8	87.5
9.00	20.9	36.1	81.8	84.9
16.38	32.0	52.1	64.9	79.9
18.00	34.1	54.7	56.0	79.4

CO conversions and carbon mass balances are based on the total amount of CO admitted into the reactor at time t = 0.



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