

DOE/PC/89869-T7

DEVELOPMENT OF A STABLE COBALT-RUTHENIUM FISCHER-TROPSCH CATALYST

Contract DE-AC22-89PC89869

Hayim Abrevaya
UOP
25 E. Algonquin Road
Des Plaines, Illinois

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Contract Objective

The objective of this contract is to examine the relationship between catalytic properties and the function of cobalt Fischer-Tropsch catalysts and to apply this fundamental knowledge to the development of a stable cobalt-based catalyst with a low methane-plus-ethane selectivity for use in slurry reactors.

DISCLAIMER

Contract Tasks

- Task 1.0: Project Management Report
- Task 2.0: Reference Cobalt Catalyst
- Task 3.1: Modifier Role for Ruthenium
- Task 3.2: Particle Size Effects with Ruthenium
- Task 4.1: Identification of the Synergy between Cobalt and a Second Bimetallic Element, such as Ruthenium
- Task 4.2: Development of a Bimetallic Catalyst
- Task 5.0: Demonstration of Stability

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Scope of Work during the Reporting Period

The work during this reporting period involved Task 4.1. The objective of this task is to determine the synergy between cobalt and a second bimetallic component. No activity was previously reported for a bimetallic cobalt catalyst prepared on a sulfur-containing support (No. 7). Recent work focused on the preparation of a bimetallic cobalt catalyst on a support that is sulfur free. Catalyst 585R2723 was the first attempt to make a catalyst with this new support (No. 8) and bimetallic component 1 using reverse micelle technique. Intermediate-size (1 to 3 nm and 15 to 30 nm) bimetallic crystallites were observed by scanning transmission electron microscopy (STEM) examination of this catalyst, which was then evaluated under different conditions in Runs 59 to 61 to identify a suitable set of conditions for screening catalysts.

As a result of the work in Runs 59 to 61, selection was made for amount of diluent, catalyst testing temperature (210°C), pressure (21 atm), and space velocity (2.2 and 4.2 NL/hr/gCo).

Five other catalysts were later prepared and tested under the conditions identified above. Catalyst 585R2748, which is a remake of catalyst 585R2723 and, according to STEM, is indistinguishable from catalyst 585R2723 was tested in Run 62. Catalyst 585R2797 had the same formulation as catalysts 585R2723 and 585R2748 but was activated at a lower temperature. According to STEM, the crystallites in this new catalyst were still bimetallic in nature, as in the two earlier preparations, but the crystallites were smaller: mostly 2 to 4 nm. Catalyst 585R2797 was tested in Run 66. In addition to cobalt and bimetallic component 1, catalyst 585R2757 contained promoters 2 and 3. A STEM analysis indicated that the crystallites are no more than 10 nm. The crystallites were mostly cobalt and had no obvious incorporation of the bimetallic component and the promoters. Catalyst 585R2757 was tested in Run 63. Catalyst 585R2774 was a cobalt catalyst

containing the same bimetallic component 1 but a new support (No. 9). The promoters were left out of the formulation. This catalyst had crystallites of 3 to 6 nm according to STEM examination. Analysis also showed that the second bimetallic component was segregated from the cobalt and dispersed on the support. Catalyst 585R2774 was tested in Run 64. The fourth new cobalt catalyst, catalyst 585R2796, had the same bimetallic component 1 used in earlier preparations and used a new support (No. 10). The STEM analysis indicated that the crystallites are mostly 2 to 4 nm in size and apparently bimetallic. Catalyst 585R2796 was tested in Run 67.

Finally, cobalt catalyst TC 211, prepared during work done under an earlier Union Carbide contract with the DOE, was evaluated. After the merger of Union Carbide's Molecular Sieve Division with UOP, all intellectual property developed under the earlier Union Carbide contract was transferred to the joint venture. The catalyst TC 211 had promoters X9 and X11 in the formulation. A STEM analysis showed that the catalyst TC 211 consists of thin (5 to 10 nm) crystallites. Cobalt crystallites contained the X11 promoter but not the X9 promoter, which is mostly dispersed on the support. The catalyst TC 211 was tested in Run 65.

Experimental

The fixed-bed pilot plant (700A) was previously described in the technical progress report covering the period of 3/16/88 to 6/16/88 for Contract DE-AC22-87PC79812. Nine runs, 59 to 67, were conducted. Table 1 summarizes the following information for each run: catalyst identifications, nature of diluents, catalyst and diluent weights, and diluent mesh size ranges. The reduced catalysts were loaded into the reactor under nitrogen atmosphere at room temperature. Following catalyst loading, the reactor was purged with nitrogen before the introduction of hydrogen. The catalysts were reduced again under hydrogen flow for 2 hours and cooled to reaction temperature. For each

run, the temperature at which hydrogen was introduced and the maximum temperature at which the catalyst was reduced again are also summarized in Table 1. When reaction temperature was reached, the flow of gas was stopped and nitrogen gas was introduced to pressurize the reactor to reaction pressure. Once reaction pressure was reached, the flow of nitrogen was stopped, and synthesis gas was introduced. Table 1 also summarizes the following information: the catalyst inlet temperatures (2 inches before the catalyst bed), catalyst maximum temperature, inlet and outlet pressures, feed compositions, feed rates, and space velocities at different hours on-stream.

The synthesis gas contained about 6% argon, which was used as an internal standard for determining conversions and light hydrocarbon selectivities. The expressions used for calculating conversions and selectivities were previously described in the technical progress report covering the period of 9/16/88 to 12/16/88 for Contract DE-AC22-87PC79812.

Results and Discussion

The objective of Runs 59 to 61 was to evaluate the same catalyst under different testing conditions and to identify a suitable set of conditions for screening catalysts.

Run 59

A large exotherm (60°C above 250°C inlet temperature) was observed in Run 59 with catalyst 585R2723. At 310°C, a maximum CO + H₂ conversion of 77% was obtained with 72% selectivity to methane. The catalyst rapidly deactivated at this high temperature.

Run 60

To reduce the large exotherm, the weight ratio of diluent to catalyst was increased from 3:1 to 10:1 in Run 60. The initial exotherm decreased from 60°C to 17°C (at 246°C inlet temperature). During the first 4 hours of the run, the space velocity was lowered from 29 NL/hr/gCo (normal liters per hour per gram cobalt) to 15 NL/hr/gCo, and the inlet temperature was lowered from 246°C to 225°C. Under these new conditions, the catalyst maximum temperature was within 2°C of the inlet temperature. The pressure was initially 35 atm. Later in the run, the effects of lower pressures (21, 15, and 25 atm) were evaluated. Also, at 21 atm, performances at 225°C and 240°C were compared. The CO + H₂ conversions and methane selectivities obtained under various conditions are summarized in Table 2.

Table 2
Activity and Selectivity of Catalyst 585R2723 in Run 60

Hours On-Stream	T, °C	P, atm	Space Velocity, NL/hr/gCo	CO + H ₂ Conversion, %	Methane Selectivity, %
16	225	35	15	16	38
27	225	21	15	13	16
37	225	15	15	10	?
60	240	21	15	28	30
75	225	21	15	14	18

Cobalt catalysts are expected to exhibit higher methane selectivities (compared to an iron-based Fischer-Tropsch catalyst) at lower conversions. Lower methane selectivity was obtained at lower conversion when the pressure was reduced from 35 to 21 atm. The selectivity could not be measured at 15 atm because of gas chromatography (GC) problems; however, lowering the pressure certainly

had an adverse effect on the conversion level. Conversion increased, as expected, with temperature. but the methane selectivity also increased. At the end of Run 60, the 225°C and 21 atm conditions were restored and only minor deviations were observed from the data obtained early in the run. This information suggests that the performance variations with time should not influence the observations that were made.

Run 61

Catalyst 585R2723 was also evaluated at 210°C, 21 atm, and a space velocity of 26 NL/hr/gCo. In this run, α -alumina diluent was used instead of γ -alumina to achieve higher weight of diluent per unit of reactor volume (ratio of α -alumina to catalyst weight is 29). The catalyst maximum temperature was within 2°C from the catalyst inlet temperature. In this run, a CO + H₂ conversion of 8% was obtained, and selectivity to methane was 10%. Comparison of data from Run 61 with data from Runs 59 and 60 suggests that methane selectivity is reduced by lowering the temperature from 225°C to 210°C.

As a result of the work done in Runs 59 to 61, 210°C and 21 atm were identified as operating conditions that lead to relatively low methane selectivity, and these operating conditions were used in subsequent catalyst screening work. Also, α -alumina was identified as a suitable diluent medium. Starting with Run 65, 13 g of catalyst and 155 g of diluent were used for each test (the ratio of diluent to catalyst weight is 12). With these amounts of catalyst and diluent, the catalyst maximum temperatures were within 2°C from the catalyst inlet temperature.

Catalysts Screening

The objective of Runs 62 to 64, 66 and 67 was to screen a variety of different catalyst formulations and to identify a catalyst for further development work.

Run 62 and 66

Catalyst 585R2748 (cobalt and bimetallic component 1 on support No. 8) had 1 to 3 nm and 15 to 30 nm bimetallic crystallites. The results of the test in Run 62 are summarized in Table 3. The initial CO + H₂ conversion was 16% at 210°C, 21 atm, and 4.6 NL/hr/gCo. The selectivity to methane, under these conditions, was 15%. The conversion increased to 31% at 232°C. Under these conditions, the methane selectivity was 20%. These results indicate undesirably high methane selectivity with this type of catalyst. The molar rate of propylene to propane was 0.9 at 210°C and as expected increased to 1.5 at 232°C.

Table 3
Comparison of Activity and Selectivity observed in Run 62 and 66

Run No.	T, °C	P, atm	Space Velocity, NL/hr/gCo	CO + H ₂ Conversion, %	Methane Selectivity, %	Propylene: Propane
62	210	21	4.6	15	15	0.9
	232	21	4.6	31	20	1.5
66	210	21	4.8	9	16	
	230	21	4.8	20	17	

Catalyst 585R2797 had the same formulation as catalyst 585R2748 but was reduced with hydrogen gas at a lower temperature. The bimetallic crystallites were smaller in size: 2 to 4 nm. This

catalyst was less active relative to catalyst 585R2748 (Run 66 in Table 3). However, despite the lower conversion level, the catalyst with the low reduction temperature showed comparable or possibly lower methane formation. This result suggests an improvement in selectivity.

Run 63

With the additional promoters 2 and 3, catalyst 585R2757 (cobalt, bimetallic component 1, promoter 2, and promoter 3 on support No. 8) had crystallites no larger than 10 nm. This catalyst showed CO + H₂ conversion of 16% and a methane selectivity of 12% at 210°C, 21 atm, and 4.3 NL/hr/gCo (Run 63 in Table 4). Under these conditions, the ratio of propylene to propane was 1.9. The conversion decreased, and the methane selectivity increased when the pressure was lowered from 21 atm to 15 atm. Also, the ratio of propylene to propane increased as expected at the lower pressure. The conversion did not recover when the pressure was set back to 21 atm. This result suggests a permanent deactivation caused by the decrease in the pressure. However, the methane selectivity decreased when the pressure was increased back to 21 atm.

Table 4
Activity and Selectivity of Catalyst 585R2757 in Run 63

Hrs. On-Stream	T, °C	P, atm	Space Velocity NL/hr/gCo	CO + H ₂ Conversion, %	Methane Selectivity, %	Propylene: Propane
15	210	21	4.3	16	12	1.9
24	210	15	4.3	12	20	2.3
41	210	21	4.3	10	13	
85	230	21	4.3	20	15	3.2

At 230°C and 20% CO + H₂ conversion, the methane selectivity was 15% and the ratio of propylene to propane was 3.2 (Table 4). A Comparison of the results in Run 63 at 210°C and 230°C with the results obtained in Runs 62 and 66 (Table 3) indicate that promoters 2 and 3 may have slightly improved selectivity by lowering methane formation. Also, the propylene to propane molar ratio increased by the addition of promoters. However, these results need to be verified by another set of catalysts with and without promoters 2 and 3.

Run 64

Catalyst 585R2774 (Cobalt and bimetallic component 1 on support No. 9) had 3 to 6 nm cobalt crystallites. This catalyst was tested in Run 64 and did not show any observable activity. Therefore, support No. 9 appeared to be unsuitable for impregnation of cobalt.

Run 67

Catalyst 585R2796 (Cobalt and bimetallic component 1 on support No. 10) had 2 to 4 nm bimetallic crystallites with bimetallic component 1 : CO ratio of 0.07 and was tested in Run 67. During the first 30 hours, this catalyst was tested at 210°C, 21 atm, and 4.4 NL/hr/gCo. A high initial CO + H₂ conversion of 94% was obtained. The catalyst rapidly lost activity, to 20% conversion, by the end of 30 hours (Figure 1). Initially, the methane selectivity was low: about 5% (Figure 2). As the conversion decreased to 20%, the methane selectivity rapidly increased to about 25%. Ethane selectivity was much lower than methane selectivity, as is typical with cobalt catalysts. However, the ethane selectivity followed the same behavior as the methane selectivity during the first 30 hours (Figure 3).

After 30 hours, the temperature was increased from 210°C to 230°C. Following the temperature increase, the CO + H₂ conversion increased to about 58% (Figure 1). This conversion is substantially higher than the conversions obtained with cobalt catalysts prepared on supports No. 8 and No. 9. Therefore, support No. 10 appears to be a promising support for achieving high cobalt activity. An interesting aspect of this catalyst was that the conversion seemed to be quite stable at 230°C following the rapid deactivation at 210°C.

Future work will address this issue by determining whether this apparent stability improvement is a time-on-stream effect or is caused by the higher temperature operation. At 58% conversion, the methane selectivity was about 18% (Figure 2). At the same conversion level and with the same catalyst at 210°C, the methane selectivity was about 11%. Ethane selectivity followed a behavior similar to methane selectivity between hours 30 to 60 (Figure 3).

After 60 hours, the space velocity was decreased to 2.1 NL/hr/gCo. The CO + H₂ conversion increased to about 86% and then gradually decreased to about 67% conversion at 150 hours on-stream. A comparison of performances at 30 to 60 hours and beyond 60 hours suggests that high conversion enhances deactivation of the cobalt catalyst (Figure 1).

The methane selectivity showed about 2% increase during the deactivation period between 60 and 150 hours (Figure 2). A slight increase in ethane selectivity was also observed during this period (Figure 3). These results are consistent with the expected behavior of cobalt catalysts, which show increasing light-ends formation with decreasing conversion level.

Catalyst 585R2774 made only a small amount of CO₂ (less than 5% CO₂ selectivity based on the total amount of CO converted). This result is consistent with the expected behavior of cobalt

catalysts, which do not typically catalyze the subsequent water-gas shift reaction during Fischer-Tropsch synthesis. The $H_2:CO$ usage ratio was surprisingly less than 2 during the first 25 hours, although no significant amount of water-gas shift activity occurred (Figure 4). Between 25 and 30 hours, the usage ratio increased to 3. This increase is expected based on the high selectivity to methane observed during this period. Later in the run, the usage ratio was about 2. This ratio is the expected result for a Fischer-Tropsch catalyst, which does not make an excessive amount of methane and is not active in the water-gas shift reaction.

The selectivities to C_1 - C_4 paraffins and olefins and to alcohols and aldehydes in Run 67 are summarized in Figures 5 and 6.

Evaluation of TC 211 Catalyst for Reference Performance

The objective of this evaluation was to obtain baseline data on a reference catalyst for comparison with cobalt-based catalysts developed under the current program. Catalyst TC 211 was one of the best catalysts developed during work performed under Union Carbide's contract DE-AC22-84PC70028.¹ A sample of this catalyst was available to UOP and so, was selected as the reference cobalt catalyst.

Run 65

The results of the test in the Bertly reactor in Run 12570-03, obtained under contract DE-AC22-84PC70028, are summarized in Table 5. The methane selectivity was 3.5 to 4% at 45 to 46% $CO + H_2$ conversion, 239°C, 21 atm, and $H_2:CO$ feed ratio of 1:1, and 4.6 NL/hr/gCo.

In this work, the same catalyst was evaluated in the fixed-bed reactor instead of the Bertly reactor; also, a $H_2:CO$ feed ratio of 2 was used instead of 1. The implication of the higher ratio feed gas in this work is that it allows high conversion to be achieved; however, the methane selectivity becomes higher. During the first 65 hours, $210^\circ C$, 21 atm, and 4.5 NL/hr/gCo were selected for operating conditions in Run 65. The $CO + H_2$ conversion reached about 70% and then decreased to about 40% (Figure 7). Under these conditions, the methane selectivity was 7.7% (Figure 8). This methane selectivity is higher than that reported in the Union Carbide contract, this is explained by the lower $H_2:CO$ feed gas ratio used in that work. Under similar operating conditions, bimetallic cobalt catalyst 585R2796 prepared on support No. 10 showed 10 to 11% methane selectivity.

Between 65 and 350 hours on-stream, the space velocity was varied. The relation between space velocity and $CO + H_2$ conversion with TC 211 catalyst at $210^\circ C$, 21 atm, and $H_2:CO$ feed ratio of 2 is shown in Figure 9. The methane selectivities obtained at different conversion levels is shown in Figure 10. As expected, the methane selectivity increases when the conversion level decreases, particularly below 40%.

After 350 hours on-stream, the original 4.5 NL/hr/gCo space velocity was restored and was held until 638 hours on-stream. The $CO + H_2$ conversion decreased from 40% to about 18%. This decrease indicates that deactivation has occurred during the test periods at various space velocities (Figure 11).

After 638 hours, the temperature was increased from $210^\circ C$ to $230^\circ C$. The $CO + H_2$ conversion increased to 93%, then decreased to 86% by 684 hours. During this period, the methane selectivity was 8.5%, which is about 2% higher than the methane selectivity obtained at the same conversion level at $210^\circ C$ (Figures 10 and 12). At 684 hours, the temperature was increased to

250°C. The CO + H₂ conversion increased to about 96%, and the methane selectivity increased to 28%. These results show that beyond 230°C, methane selectivity increases rapidly with temperature. At 710 hours, the space velocity was increased from 4.5 to 6.3 NL/hr/gCo. At this higher space velocity, the CO + H₂ conversion decreased to 87% and the methane selectivity increased to 33%. Finally, at 720 hours, the space velocity was increased to 12 NL/hr/gCo. This increase brought the conversion down to 72% and possibly caused some further increase in methane selectivity.

The usage ratio and the selectivities to CO₂, ethane, C₁-C₄ paraffins and olefins, and C₁-C₄ alcohols and aldehydes are summarized in Figures 13-16.

Summary and Implications for Future Work

An experimental cobalt catalyst 585R2723 was tested three times in the fixed-bed reactor. The objective of the tests was to identify suitable testing conditions for screening catalyst. The α -alumina was determined to be a suitable diluent medium for controlling the catalyst bed temperature close to the inlet temperature. With 13 g of catalyst and 155 g of diluent, the catalyst maximum temperature were within 2°C from the inlet temperatures. As a result of this work, 210°C and 21 atm were shown to result in low methane selectivity and were used as initial conditions in the catalyst screening test. Ethane, which along with methane is undesirable, is typically produced with low selectivity and follows the same trend as methane. Other work reported here indicated that methane selectivity increases with increasing temperature but is not excessively high at 230°C. Consequently, the catalyst screening test should include an evaluation of the catalyst performance at 230°C. During Run 67, the increase in temperature from 210°C to 230°C was initiated at 30 hours on-stream. In this new catalyst screening test, the space velocity is decreased at 60 hours on-

stream to evaluate the performance at high conversion. This test method is going to be used in the future for screening catalysts.

All experimental cobalt catalysts tested here include bimetallic component 1. Three different supports (Nos. 8 to 10) were evaluated for impregnating cobalt. The highest activity and lowest methane selectivity were obtained with support No. 10. A cobalt catalyst with two additional promoters (Nos. 2 and 3) was prepared on support No. 8. These promoters lower the selectivity of methane. The beneficial effect of these promoters will be verified in the future by preparing promoted cobalt catalysts on the most promising support No. 10.

Cobalt catalyst TC 211 was one of the best catalysts developed during work done under Union Carbide's contract DE-AC22-84PC70028. Catalyst TC 211 contains the proprietary X9 and X11 promoters on a proprietary support. According to STEM examination, the crystallites are 5 to 10 nm and thin. The X11 promoter was incorporated in the support, but the X9 promoter was dispersed on the support. After the merger of the Molecular Sieve Division of Union Carbide with UOP, all intellectual property for contract DE-AC22-84PC70028 was transferred to the new UOP. Catalyst TC 211 was tested in this program to establish reference performance at 210°C, 230°C, and 250°C in the fixed-bed reactor.

The most promising cobalt catalyst (585R2796) prepared so far in this program contains bimetallic component 1 on support No. 10. According to STEM examination, this catalyst has 2 to 4 nm bimetallic crystallites. Catalyst 585R2796 showed high conversion and low methane selectivity at 210°C, 21 atm, 4.4 NL/hr/gCo and a 2:1 H₂:CO feed ratio. The catalyst appears unstable at 210°C and shows a substantial stability improvement following an increase in temperature to 230°C. With decrease in space velocity to 2.1 NL/hr/gCo, a CO + H₂ conversion of about 86% was

obtained with about 15% selectivity to methane. At 230°C, 21 atm, 4.5 NL/hr/gCo, and 2:1 H₂:CO feed ratio, the TC 211 catalyst showed 87% CO + H₂ conversion and 8.5% methane selectivity. These results indicate that TC 211 catalyst is more active and shows lower methane selectivity.

The current catalyst development program is using reverse micelle impregnation as a tool for preparing cobalt catalysts. This procedure allows good control of crystallite size and crystallite composition for cobalt catalysts.² Crystallite size and crystallite composition, i.e., the concentrations of promoters, are the two key catalytic properties that influence catalytic performance. Future work will aim at developing superior cobalt catalysts by optimization of these two properties. Knowledge gained during the development of TC 211 catalyst will also be applied to the development work to be conducted in this work.

References

1. Improved Catalysts for Liquid Hydrocarbon Fuels from Syngas, Final Report Contract DE-AC22-84PC70028, Union Carbide Corporation, Tarrytown Technical Center, Tarrytown, NY.
2. H. Abrevaya and W. M. Targos, The Development of a Selective Cobalt Catalyst, Contract DE-AC22-89PC89869, Indirect Liquefaction Contractors' Review Meeting, Sept. 1991.

Table 1
Run Information

Run	Plant	Catalyst		H ₂ Treatment		Diluent		Temperature		Pressure		Feed								
		Number	% CO	Wt. g	Low T, °C	Max T, °C	Nature	Mesh	Wt., g	In, °C	Max, °C	In, atm	Out, atm	% H ₂	% CO	% AR	H ₂ CO	Rate sec/min	NL/hr /gCo	Hrs
59	700A	585R2723	5.84	10	30	550	α-Al ₂ O ₃	200-325	30	250	310	43	43	62.7	31.3	6.0	2.0	111	11	1
										250 ↓ 220	310 ↓ 220	43-47								2-9
										220	220	35	37-52							9-28
										230	235	35	37							28-32
										250	256	34-47	36-49							32-35
60	700A	585R2723	5.84	4	150	550	α-Al ₂ O ₃	60-100	40	245 ↓ 225	236 234	35	35	62.7	3.13	6.0	2.0	111	29	1-4
										225	225	35	35					57	15	4-15
												21	21							15-28
												15	15							28-44
												21	21							44-54
										240	242									54-64
										225	227									64-76
										225	227	25	25							76-84
61	700	585R2723	5.84	6.5			α-Al ₂ O ₃	40-80	185.3	210	210	21	21	62.7	31.3	6.0	2.0	165	26	0-16
										222	222									16-55

Table 1 (continued)
Run Information

Run	Plant	Catalyst		H ₂ Treatment		Diluent		Temperature		Pressure		Feed									
		Number	% CO	Wt. g	Low T, °C	Max T, °C	Nature	Mesh	Wt., g	In, °C	Max, °C	In, atm	Out, atm	% H ₂	% CO	% AR	H ₂ :CO	Rate sec/min	NL/hr /gCo	Hrs	
62	700A	585R2748	6.90	13			α -Al ₂ O ₃	40-80	175	210		21	↓	61.9	31.9	6.2	1.9	68	4.6	0-40	
										232				↓	↓	↓	↓	↓	↓	↓	40-42
63	700A	585R2757	7.34	13	37	550	α -Al ₂ O ₃	40-80	160	210		21		61.0	33.0	6.0	1.8	68	4.3	0-18	
										↓		15		↓	↓	↓	↓	↓	↓	↓	18-27
										↓		21	↓	↓	↓	↓	↓	↓	↓	↓	27-43
										↓		↓		↓	↓	↓	↓	↓	↓	↓	43-80
64	700A	585R2774	1.76	8			α -Al ₂ O ₃	40-80	160	210		19		62.2	30.9	6.9	2.0	42	18	0-13	
										↓		22		↓	↓	↓	↓	137	58	13-32	
										↓		21		↓	↓	↓	↓	82	35	32-60	

TABLE 5

RESULT OF SYNGAS OPERATION

RUN NO. 12570-03
 CATALYST CO/X9/X11-TG123 80 CC 41.3 G AFTER USE: 60.2 G (-18.9 G)
 FEED H₂:CO OF 50:50 @ 400 CC/MIN OR 300 GHSV (CAT#12524-27)

RUN & SAMPLE NO.	12570-03-02	570-03-03	570-03-04	570-03-05	570-03-06
FEED H ₂ :CO:AR	50:50:0	50:50:0	50:50:0	50:50:0	50:50:0
HRS ON STREAM	21.50	45.50	69.50	93.50	118.5
PRESSURE, PSIG	300.00	300.00	298.60	300.00	293.00
TEMP. C	239.00	239.00	239.00	239.00	239.00
FEED CC/MIN	400.00	400.00	400.00	400.00	400.00
HOURS FEEDING	17.00	24.00	24.00	24.00	24.00
EFFLNT GAS LITER	180.65	306.95	304.45	310.00	324.74
ON AQUEOUS LAYER	44.58	48.61	48.29	48.09	48.12
ON OIL	6.68	38.04	39.09	37.68	42.58
MATERIAL BALANCE					
ON ATOM CARBON %	66.83	94.20	94.44	94.61	100.47
ON ATOM HYDROGEN %	75.30	91.82	92.26	91.49	97.71
ON ATOM OXYGEN %	87.28	92.54	91.52	92.84	95.24
RATIO CH ₄ /(H ₂ O-CO ₂)	0.3754	1.0627	1.1129	1.0678	1.2010
RATIO X IN CH ₄	2.2781	2.1959	2.1885	2.1921	2.1831
USAGE H ₂ /CO PRODT	3.2557	1.8648	1.8497	1.8808	1.8052
FEED H ₂ /CO FRM EFFLNT	1.1268	0.9747	0.9767	0.9671	0.9725
RESIDUAL H ₂ /CO RATIO	0.5691	0.5612	0.5607	0.5579	0.5733
RATIO CO ₂ /(H ₂ O-CO ₂)	0.0483	0.0646	0.0559	0.0563	0.0510
X SHIFT IN EFFLNT	0.0289	0.0387	0.0332	0.0333	0.0308
SPECIFIC ACTIVITY SA	1.7370	3.0447	3.1785	3.0559	3.1750
CONVERSION					
ON CO %	20.76	31.72	32.27	30.93	32.41
ON H ₂ %	59.98	60.69	51.12	60.16	60.15
ON CO-H ₂ %	41.53	46.02	44.53	45.30	46.09
PROD SELECTIVITY, WT %					
CH ₄	8.48	4.00	3.54	3.72	3.25
C ₂ HC'S	1.09	0.60	0.64	0.62	0.64
C ₃ H ₈	1.65	0.77	0.79	0.86	0.79
C ₃ H ₆ =	3.30	1.79	1.79	1.95	1.81
C ₄ H ₁₀	2.09	0.89	0.90	0.97	0.88
C ₄ H ₈ =	4.67	2.34	2.51	2.65	2.39
C ₅ H ₁₂	2.33	1.13	1.15	1.21	1.13
C ₅ H ₁₀ =	3.27	1.51	1.70	0.18	1.42
C ₆ H ₁₄	3.80	1.82	1.86	1.81	1.74
C ₆ H ₁₂ = & CYCLO'S	2.14	1.31	1.24	1.43	1.25
C ₇ - IN GAS	8.39	3.73	3.95	4.17	3.75
LIQ HC'S	58.80	80.11	79.92	80.63	80.95
TOTAL	100.00	100.00	100.00	100.00	100.00
SUB-GROUPING					
C1 -C4	21.27	10.40	10.18	10.78	9.76
C5 -420 F	35.80	21.75			
420-700 F	34.28	32.37			
700-END FT	8.64	35.49			

TABLE 5 (CONTINUED)

	78.73	89.60	89.82	89.22	90.24
CS--END PT					
ISO/NORMAL MOLE RATIO	0.1332	0.0000	0.0000	0.0000	0.0000
C4	0.0483	0.0562	0.0259	0.0000	0.0147
C5	0.3473	0.3428	0.1900	0.1323	0.2072
C6	0.0534	0.0443	0.0266	0.0261	0.0153
C6+					
PARAFFIN/OLEFIN RATIO	0.4766	0.4112	0.4178	0.4201	0.4157
C3	0.4316	0.3678	0.3474	0.3533	0.3537
C4	0.6933	0.7311	0.6384	6.4536	0.7770
C5					
SCHULZ-FLORY DISTRIBUTION					
ALPHA (EXP(SLOPE))	0.8429	0.9084			
RATIO C _n /(1-A) ⁿ⁻²	3.4366	4.7664			
ALPHA FRM CORRELATION	0.8395	0.8401			
ALPHA (EXPTL/CORR)	1.0041	1.0812			
WICH4 FRM CORRELATION	13.0182	12.8053			
WICH4 (EXPTL/CORR)	0.6512	0.3124			
LIG HC COLLECTION					
PHYS. APPEARANCE	OIL WAX	OIL WAX	OIL WAX	OIL WAX	OIL WAX
DENSITY					
N. REFRACTIVE INDEX					
SIMULT'D DISTILLATE					
10 WT % @ DEG F	343.00	381.00			
16	380.00	422.00			
30	515.00	664.00			
64	690.00	945.00			
90	753.00	1034.00			
RANGE(16-64 X)	310.00	523.00			
WT % @ 420 F	27.00	15.30			
WT % @ 700 F	85.30	53.70			

TABLE 5 (CONTINUED)

RESULT OF SYNGAS OPERATION

RUN NO. 12570-03
 CATALYST CO/XB/X11-TC123 80 CC 41.3 G AFTER USE: 60.2 G (-18.9 G)
 FEED H₂:CO OF 50:50 @ 400 CC/HR OR 300 CMHV (CAT#12524-27)

RUN & SAMPLE NO.	12570-03-07	570-03-08	570-03-09	570-03-10	570-03-11
FEED H ₂ :CO:AR	50:50:0	50:50:0	50:50:0	50:50:0	50:50:0
HRS ON STREAM	166.50	190.50	216.50	238.50	267.50
PRESSURE, PSIG	300.00	297.00	299.00	300.00	300.00
TEMP. C	238.00	260.00	259.00	259.00	259.00
FEED CC/MIN	400.00	400.00	400.00	400.00	400.00
HOURS FEEDING	48.00	24.00	24.00	24.00	24.00
EFFLNT GAS LITER	628.56	266.85	262.95	265.10	286.99
GM AQUEOUS LAYER	93.91	51.70	54.72	55.95	54.87
GM OIL	71.19	40.54	40.25	38.88	38.44
MATERIAL BALANCE					
GM ATOM CARBON X	94.37	85.17	93.53	92.64	98.89
GM ATOM HYDROGEN X	91.90	80.14	92.09	92.53	94.36
GM ATOM OXYGEN X	92.09	91.95	91.20	91.37	96.73
RATIO CH ₄ /(H ₂ O+CO ₂)	1.0902	0.7859	1.0731	1.0397	1.0614
RATIO X IN CH ₄	2.1861	2.2788	2.2565	2.2574	2.2595
USAGE H ₂ /CO FEED	1.8854	1.7754	1.8970	1.7437	1.7209
FEED H ₂ /CO FRM EFFLNT	0.9738	0.9410	0.9846	0.9987	0.9561
RESIDUAL H ₂ /CO RATIO	0.5749	0.4727	0.4744	0.4877	0.4708
RATIO CO ₂ /(H ₂ O+CO ₂)	0.0472	0.1802	0.1645	0.1314	0.1369
K SHIFT IN EFFLNT	0.0285	0.1039	0.0806	0.0738	0.0747
SPECIFIC ACTIVITY SA	2.9688	1.4817	1.8516	1.7266	1.7046
CONVERSION					
ON CO X	30.44	35.94	41.64	40.69	38.82
ON H ₂ X	58.94	67.82	71.76	71.03	69.87
ON CO+H ₂ X	44.50	51.40	56.58	55.85	54.00
PROD SELECTIVITY, WT X					
CH ₄	3.51	10.59	6.77	6.85	7.01
C ₂ HC'S	0.57	2.15	1.38	1.42	1.49
C ₃ H ₈	0.90	2.52	1.58	1.57	1.62
C ₃ H ₆	2.03	3.16	2.18	2.21	2.29
C ₄ H ₁₀	1.02	2.43	1.58	1.55	1.58
C ₄ H ₈	2.77	4.02	3.36	3.35	3.50
C ₅ H ₁₂	1.28	3.15	2.02	2.03	2.04
C ₅ H ₁₀	3.37	2.26	1.58	1.53	2.27
C ₆ H ₁₄	2.17	5.27	3.19	3.27	3.55
C ₆ H ₁₂ & CYCLO'S	1.34	3.08	1.90	2.00	2.20
C ₇ + IN GAS	4.30	8.56	5.20	5.59	5.55
LIQ HC'S	76.74	52.80	69.27	68.63	66.90
TOTAL	100.00	100.00	100.00	100.00	100.00
SUB-GROUPING					
C1-C4	10.80	24.88	16.84	16.95	17.49
C5-420 F	26.35	4.08	32.59		
420-700 F	26.24	38.88	29.37		
700-EDC FT	38.60	32.16	21.20		

TABLE 5 (CONTINUED)

CS--END FT	89.20	75.12	83.16	83.05	82.51
ISO/NORMAL MOLE RATIO					
C4	0.0000	0.0195	0.0192	0.0158	0.0000
C5	0.0000	0.0471	0.0464	0.0468	0.0000
C6	0.2073	0.3706	0.3237	0.2901	0.2358
C6+	0.0280	0.0719	0.0504	0.0499	0.0483
PARAFFIN/OLEFIN RATIO					
C1	0.4235	0.7590	0.6920	0.6786	0.6726
C4	0.3369	0.5841	0.4542	0.4460	0.4368
C5	0.3704	1.3526	1.2441	1.2920	0.8749
SCHULZ-FLORY DISTRIBUTION					
ALPHA (EXP(SLOPE))	0.8940	0.8650	0.8688		
RATIO C _n /(1-A) ⁿ⁻¹	3.1202	5.8137	3.9212		
ALPHA FRM CORRELATION	0.8390	0.8473	0.8470		
ALPHA (DOPTL/CORR)	1.0655	1.0209	1.0256		
WYCH ₄ FRM CORRELATION	12.9241	15.1865	15.0740		
WYCH ₄ (DOPTL/CORR)	0.2714	0.6975	0.4490		
LIQ HC COLLECTION					
PHYS. APPEARANCE	OIL WAX	OIL WAX	OIL WAX	OIL WAX	OIL WAX
DENSITY					
N. REFRACTIVE INDEX					
SIMULT'D DISTILLATE					
10 WT % @ DEG F	381.00	333.00	308.00		
16	421.00	373.00	348.00		
50	703.00	573.00	568.00		
84	1015.00	882.00	852.00		
90	1093.00	970.00	940.00		
RANGE(16-84 %)	594.00	509.00	504.00		
WT % @ 420 F	15.50	26.00	27.00		
WT % @ 700 F	49.70	66.50	69.40		

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TABLE 5 (CONTINUED)
RESULT OF SYNGAS OPERATION

RUN NO. 12570-03
 CATALYST CO/X9/X11-TC123 80 CC 41.3 @ AFTER USE: 60.2 C (-18.9 C)
 FEED H₂:CO OF 50:50 @ 400 CC/MIN OR 300 CHEV (CAT#12524-27)

RUN & SAMPLE NO.	12570-03-12	570-03-13	570-03-14	570-03-15	570-03-16
FEED H ₂ :CO:AR	50:50:0	50:50:0	50:50:0	60:40:0	60:40:0
ERS ON STREAM	287.30	310.50	334.50	358.50	383.50
PRESSURE, PSIG	298.00	298.00	299.50	500.00	500.00
TEMP. C	259.00	259.00	259.00	259.00	260.00
FEED CC/MIN	400.00	400.00	400.00	400.00	400.00
HOURS FEEDING	25.00	47.00	24.00	23.00	25.00
EFFLNT GAS LITER	278.76	535.54	271.90	183.63	155.70
ON AQUEOUS LAYER	58.80	111.28	54.72	67.98	83.99
ON OIL	42.59	78.40	39.89	50.23	57.13
MATERIAL BALANCE					
ON ATOM CARBON %	95.70	97.54	96.84	100.15	92.67
ON ATOM HYDROGEN %	93.45	93.97	92.57	90.94	90.48
ON ATOM OXYGEN %	92.91	94.69	93.32	92.91	90.46
RATIO CHEV/(H ₂ O-CO ₂)	1.0869	1.0886	1.1127	1.1369	1.0399
RATIO X IN CHEV	2.2493	2.2475	2.2545	2.3668	2.3164
USAGE H ₂ /CO FEED	1.7388	1.7471	1.7289	1.6292	1.7829
FEED H ₂ /CO FAN EFFLNT	0.9766	0.9634	0.9559	1.3622	2.4646
RESIDUAL H ₂ /CO RATIO	0.4571	0.4457	0.4458	0.7420	0.7318
RATIO CO ₂ /(H ₂ O-CO ₂)	0.1214	0.1170	0.1211	0.1875	0.1259
X SHIFT IN EFFLNT	0.0632	0.0591	0.0614	0.1713	0.1054
SPECIFIC ACTIVITY SA	1.8967	1.9090	1.9100	1.4236	1.3426
CONVERSION					
ON CO %	40.53	39.78	39.76	69.80	69.71
ON H ₂ %	72.17	72.14	71.91	83.60	84.87
ON CO-H ₂ %	56.16	55.66	55.47	77.80	78.72
PROD SELECTIVITY, WT %					
CH ₄	6.48	6.41	6.76	11.89	9.50
C ₂ HC'S	1.29	1.33	1.30	1.91	1.63
C ₃ ES	1.48	1.50	1.53	2.74	2.15
C ₃ H ₆	2.18	2.26	2.27	1.22	1.15
C ₄ H ₁₀	1.50	1.54	1.56	2.36	1.86
C ₄ H ₈	3.26	3.46	3.41	2.61	2.14
C ₅ H ₁₂	2.09	2.14	2.11	2.94	2.31
C ₅ H ₁₀	1.66	2.42	2.33	0.86	0.78
C ₆ H ₁₄	1.28	3.58	3.43	3.98	3.29
C ₆ H ₁₂ & CYCLO'S	2.16	2.22	2.04	1.25	1.15
C ₇ - IN GAS	5.38	5.61	5.39	4.54	4.07
LIQ HC'S	69.25	67.54	67.87	63.91	69.97
TOTAL	100.00	100.00	100.00	100.00	100.00
SUB-GROUPING					
C1 -C4	16.19	16.49	16.83	22.52	18.43
C5 -420 F				30.83	32.24
420-700 F				22.43	28.41
700-END FT				24.22	20.92

TABLE 5 (CONTINUED)

	83.81	83.51	83.17	77.48	81.57
CS--END PT					
ISO/NORMAL MOLE RATIO	0.0166	0.0140	0.0152	0.0262	0.0234
C4	0.0464	0.0454	0.0464	0.0666	0.0617
C5	0.2936	0.3661	0.3746	0.2971	0.3108
C6	0.0482	0.0470	0.0483	0.0848	0.0765
C6+					
PARAFFIN/OLEFIN RATIO	0.6457	0.6344	0.6432	2.1473	1.7838
C3	0.4427	0.4293	0.4411	0.9478	0.8397
C4	1.2204	0.8620	0.8780	3.3076	2.8613
C5					
SCHULZ-FLORY DISTRIBUTION				3.8678	0.8661
ALPHA (EXP(SLOPE))				6.8032	5.2997
RATIO C ₄ /(1-A)**2				0.8254	0.8260
ALPHA FRM CORRELATION				1.0514	1.0486
ALPHA (EXPTL/CORR)				21.7723	21.7921
W ₄ C ₄ FRM CORRELATION				0.5460	0.4360
W ₄ C ₄ (EXPTL/CORR)					
LIQ EC COLLECTION	OIL MAX	OIL MAX	OIL MAX	OIL MAX	OIL MAX
PHYS. APPEARANCE					
DENSITY					
N. REFRACTIVE INDEX				302.00	301.00
SIMULT'D DISTILLATE				346.00	346.00
10 WT % @ DEG F				596.00	546.00
16				919.00	876.00
50				1006.00	978.00
84					
90				573.00	530.00
RANGE(16-84 %)				27.00	29.50
WT % @ 420 F				62.10	70.10
WT % @ 700 F					

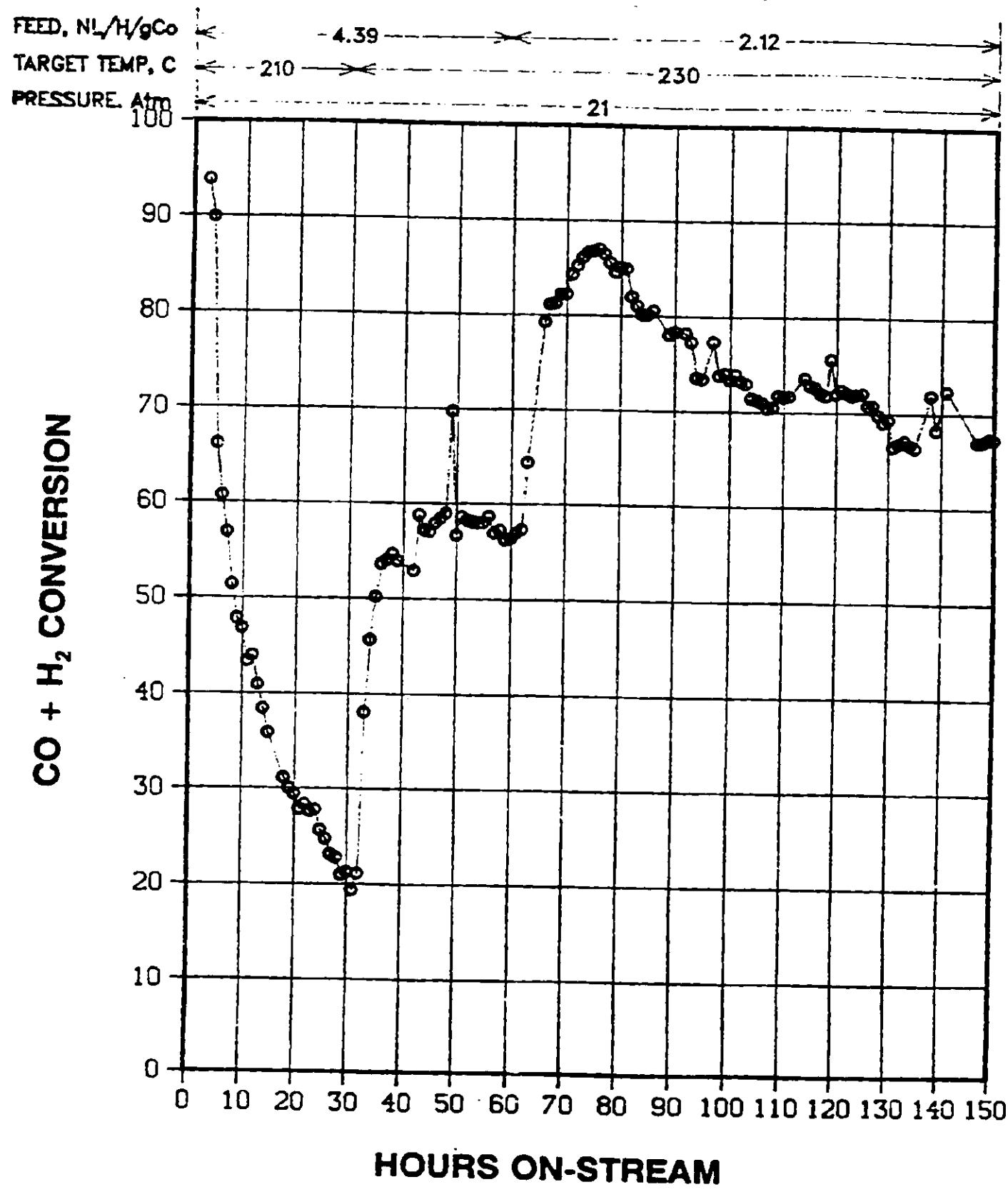
TABLE 5 (CONTINUED)
RESULT OF SYNGAS OPERATION

RUN NO. 12570-03				
CATALYST CO/X2/X11-TC123 80 CC 41.3 G AFTER USE:60.2 G (+18.9 G)				
FEED H2:CO OF 50:50 @ 600 CC/HR OR 300 GHSV (CAT#12524-27)				
RUN & SAMPLE NO.	12570-03-17	570-03-18	570-03-19	570-03-20
FEED H2:CO:AR	60:40:0	60:40:0	60:40:0	60:40:0
HRS ON STREAM	407.50	429.00	456.00	478.50
PRESSURE.PSIC	500.00	500.00	500.00	500.00
TEMP. C	260.00	260.00	260.00	260.00
FEED CC/MIN	400.00	400.00	400.00	400.00
HOURS FEEDING	24.00	21.50	26.00	22.50
EFFLNT GAS LITER	131.50	139.50	172.20	151.90
GM AQUEOUS LAYER	81.48	70.57	87.81	76.75
GM OIL	54.58	54.47	57.46	51.06
MATERIAL BALANCE				
GM ATOM CARBON X	92.82	98.49	92.28	94.38
GM ATOM HYDROGEN X	91.42	94.18	90.86	92.36
GM ATOM OXYGEN X	90.99	89.14	90.86	91.80
RATIO CH4/(H2O+CO2)	1.0326	1.1758	1.0264	1.0475
RATIO X IN CH4	2.3289	2.2925	2.3126	2.3068
USAGE H2/CO PRODT	1.7970	1.7449	1.8613	1.8559
FEED H2/CO FRM EFFLNT	1.4774	1.4343	1.4736	1.4680
RESIDUAL H2/CO RATIO	0.7444	0.7346	0.7306	0.7292
RATIO CO2/(H2O+CO2)	0.1240	0.1079	0.0968	0.0925
K SHIFT IN EFFLNT	0.1054	0.0888	0.0783	0.0743
SPECIFIC ACTIVITY SA	1.3289	1.3074	1.1577	1.1398
CONVERSION				
ON CO X	69.63	69.26	65.71	65.58
ON H2 X	84.70	84.26	83.00	82.90
ON CO+H2 X	78.62	78.10	76.01	75.88
PRODT SELECTIVITY, WT X				
CH4	10.12	8.47	9.46	9.20
C2 HC'S	1.73	1.48	1.56	1.60
C3H8	2.31	1.89	2.05	2.01
C3H6	1.17	1.98	1.28	1.25
C4H10	1.94	1.65	1.82	1.77
C4H8	2.20	2.00	2.16	2.25
C5H12	2.30	2.05	2.24	2.18
C5H10	0.75	1.26	1.38	1.66
C6H14	3.14	2.85	2.99	2.56
C6H12 & CYCLO'S	1.10	1.09	1.15	1.10
C7+ IN GAS	3.79	3.61	3.52	3.94
LIQ HC'S	69.46	71.67	70.39	70.46
TOTAL	100.00	100.00	100.00	100.00
SUB-GROUPING				
C1 -C4	19.46	17.47	18.33	18.09
C5 -420 F		29.49		29.41
420-700 F		27.23		26.78
700-END FT		25.80		25.72

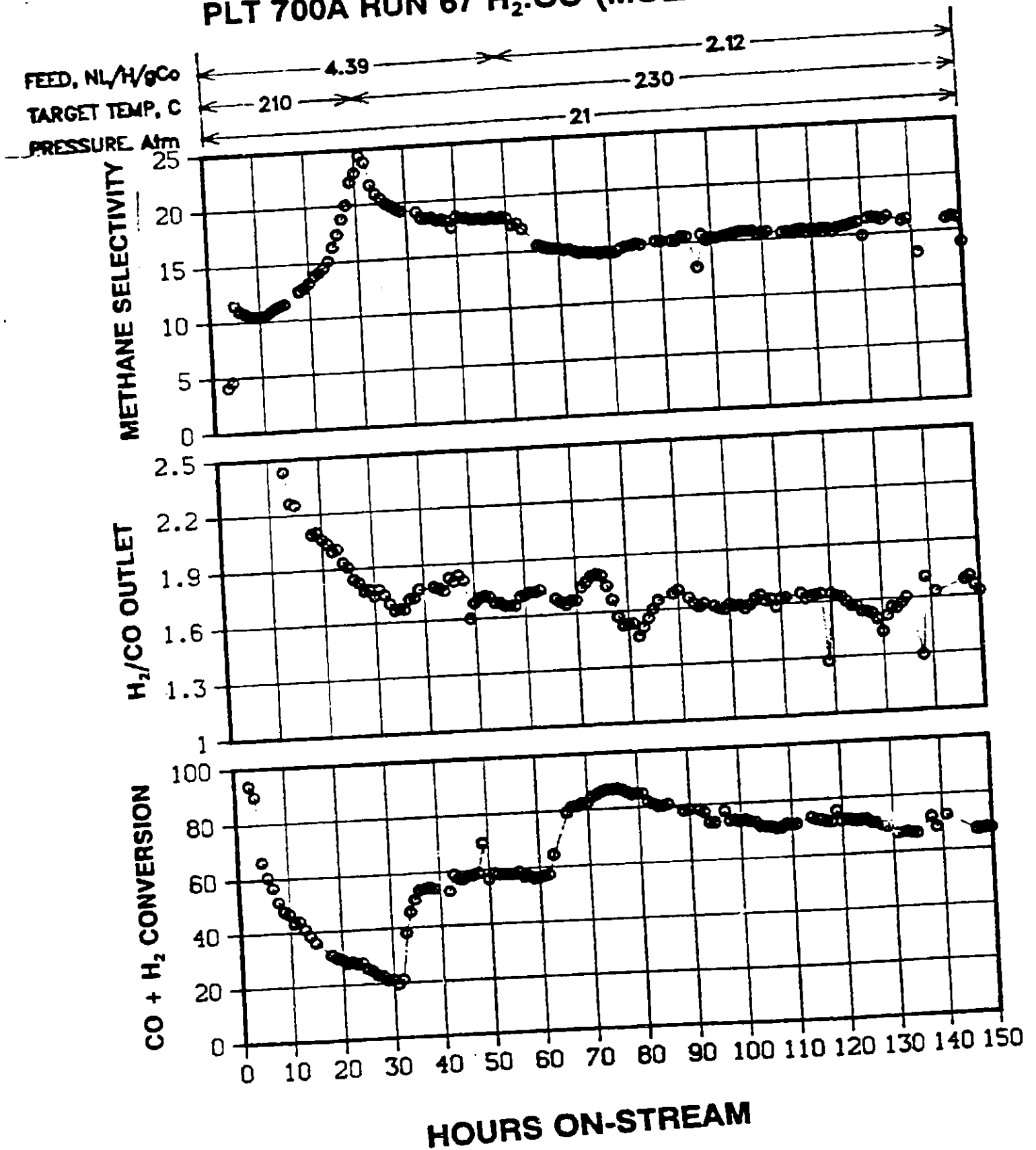
TABLE 5 (CONTINUED)

	80.54	82.53	81.67	81.91
CS--END FT				
ISO/NORMAL MOLE RATIO	0.0251	0.0246	0.0257	0.0243
C4	0.0628	0.0604	0.0568	0.0589
C5	0.2367	0.2254	0.2311	0.0809
C6	0.0787	0.0746	0.0208	0.0750
C6+				
PARAFFIN/OLEFIN RATIO	1.8763	0.9070	1.5299	1.5369
C3	0.8507	0.7959	0.8137	0.7811
C4	2.9852	1.5852	1.5786	1.2796
C5				
SCHULZ-FLORY DISTRIBUTION		0.8757		0.8772
ALPHA (EXP(SLOPE))		5.4785		6.1008
RATIO C ₄ /(1-A)**2				
ALPHA FRM CORRELATION		0.8258		0.8262
ALPHA (DOPTL/CORR)		1.0604		1.0618
WCH4 FRM CORRELATION		21.8482		21.7368
WCH4 (DOPTL/CORR)		0.3875		0.4233
LIG HC COLLECTION				
PHYS. APPEARANCE	OIL WAX	OIL WAX	OIL WAX	OIL WAX
DENSITY				
N. REFRACTIVE INDEX				
SIMULT'D DISTILLATE				
10 WT % @ DEG F		304.00		304.00
16		348.00		349.00
50		592.00		595.00
84		917.00		909.00
90		1010.00		997.00
RANGE(16-84 %)		349.00		360.00
WT % @ 420 F		28.00		29.50
WT % @ 700 F		64.00		63.50

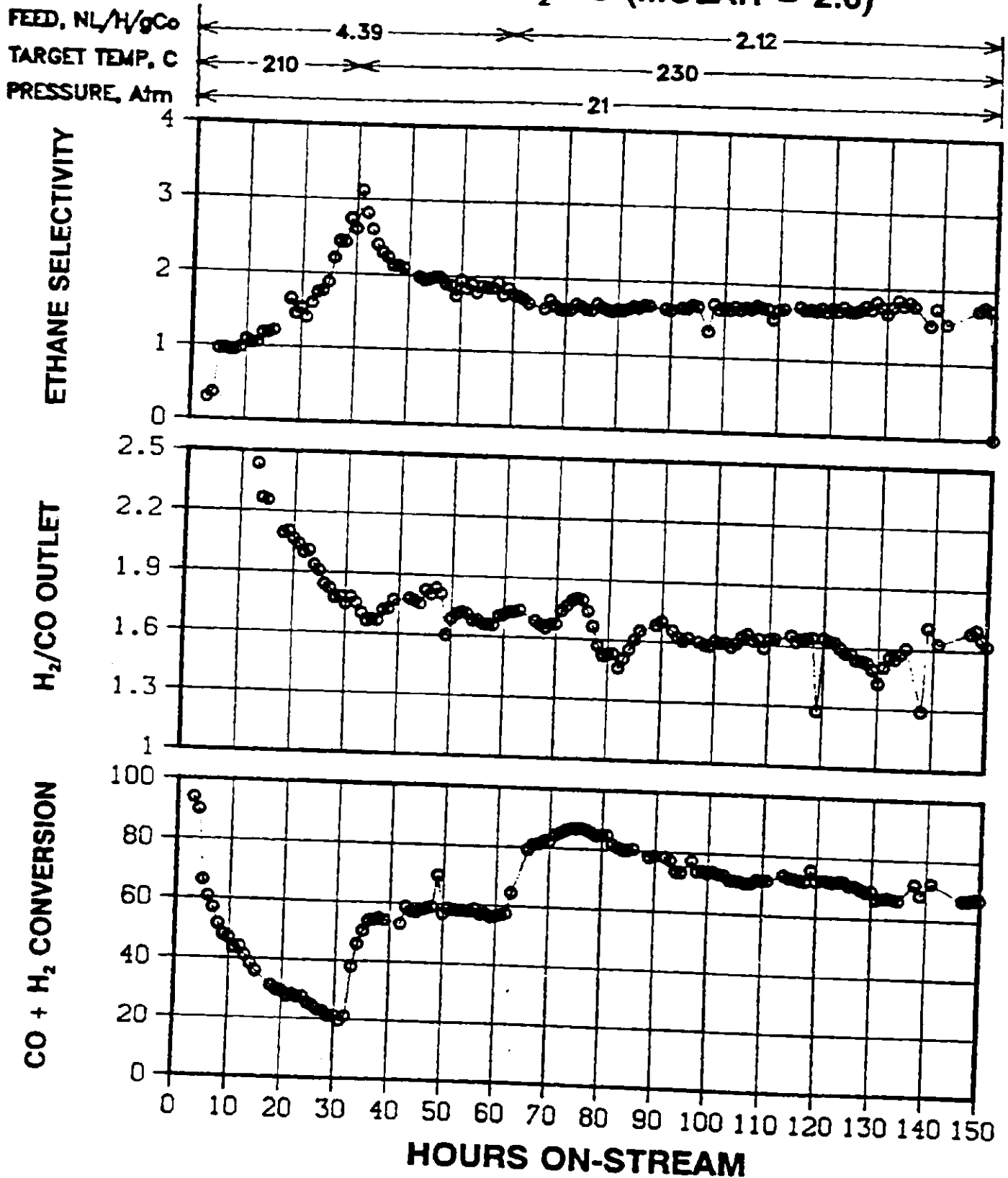
**FIGURE 1: BIMETALLIC Co CATALYST 585R2796
 PLT 700A RUN 67 H₂:CO (MOLAR) = 2.0**



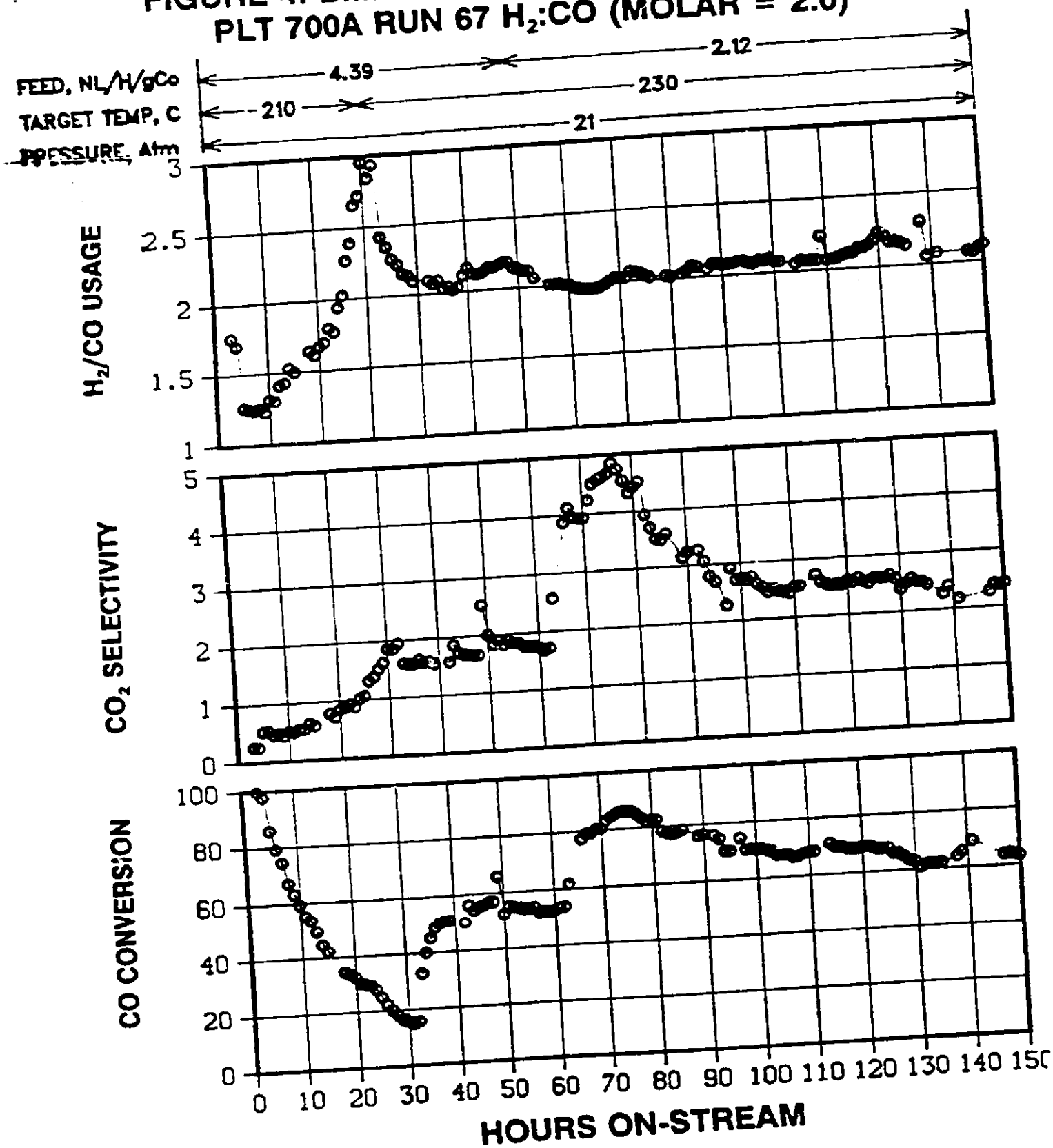
**FIGURE 2: BIMETALLIC Co CATALYST 585R2796
 PLT 700A RUN 67 H₂:CO (MOLAR = 2.0)**



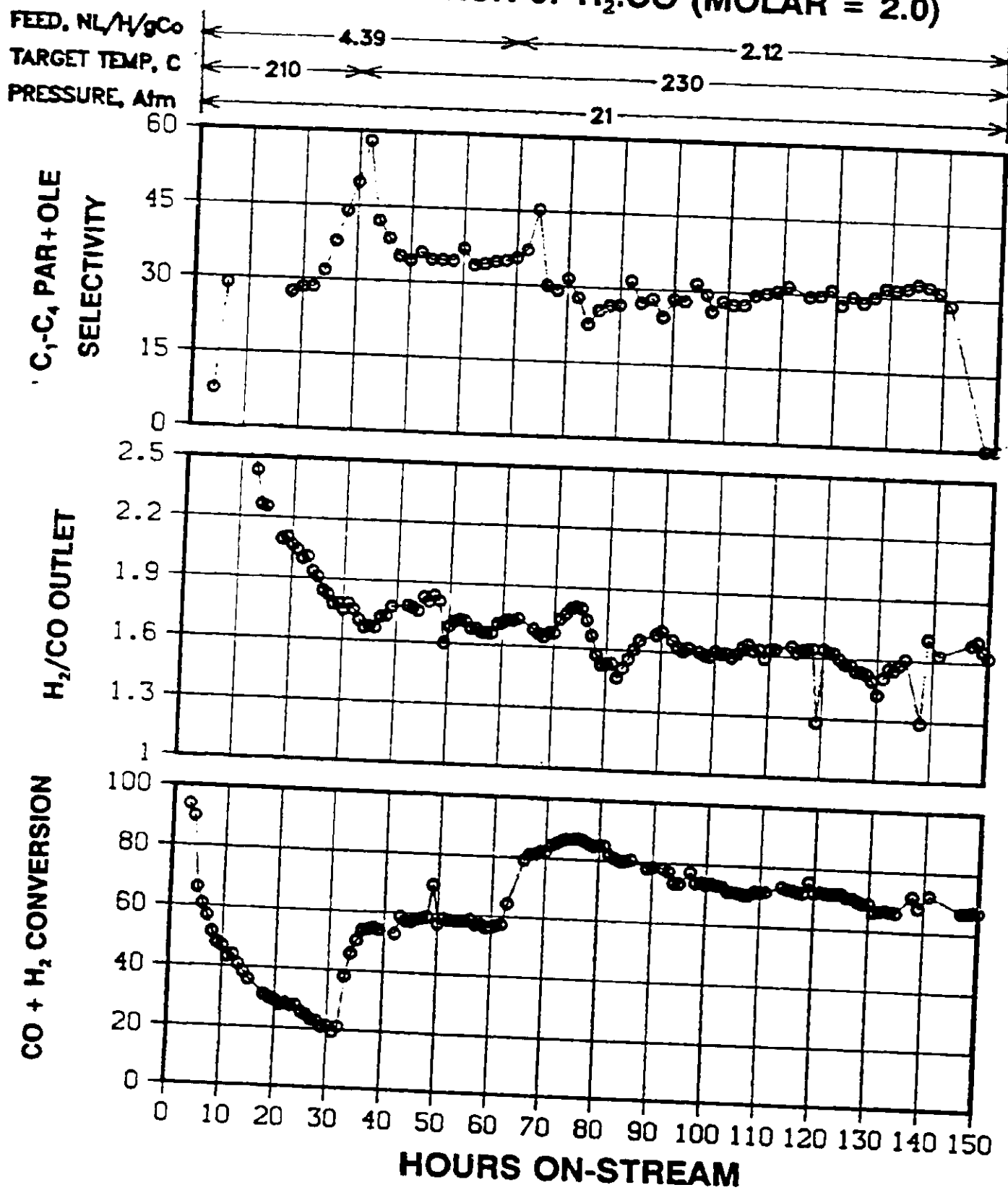
**FIGURE 3: BIMETALLIC Co CATALYST 585R2796
 PLT 700A RUN 67 H₂:CO (MOLAR = 2.0)**



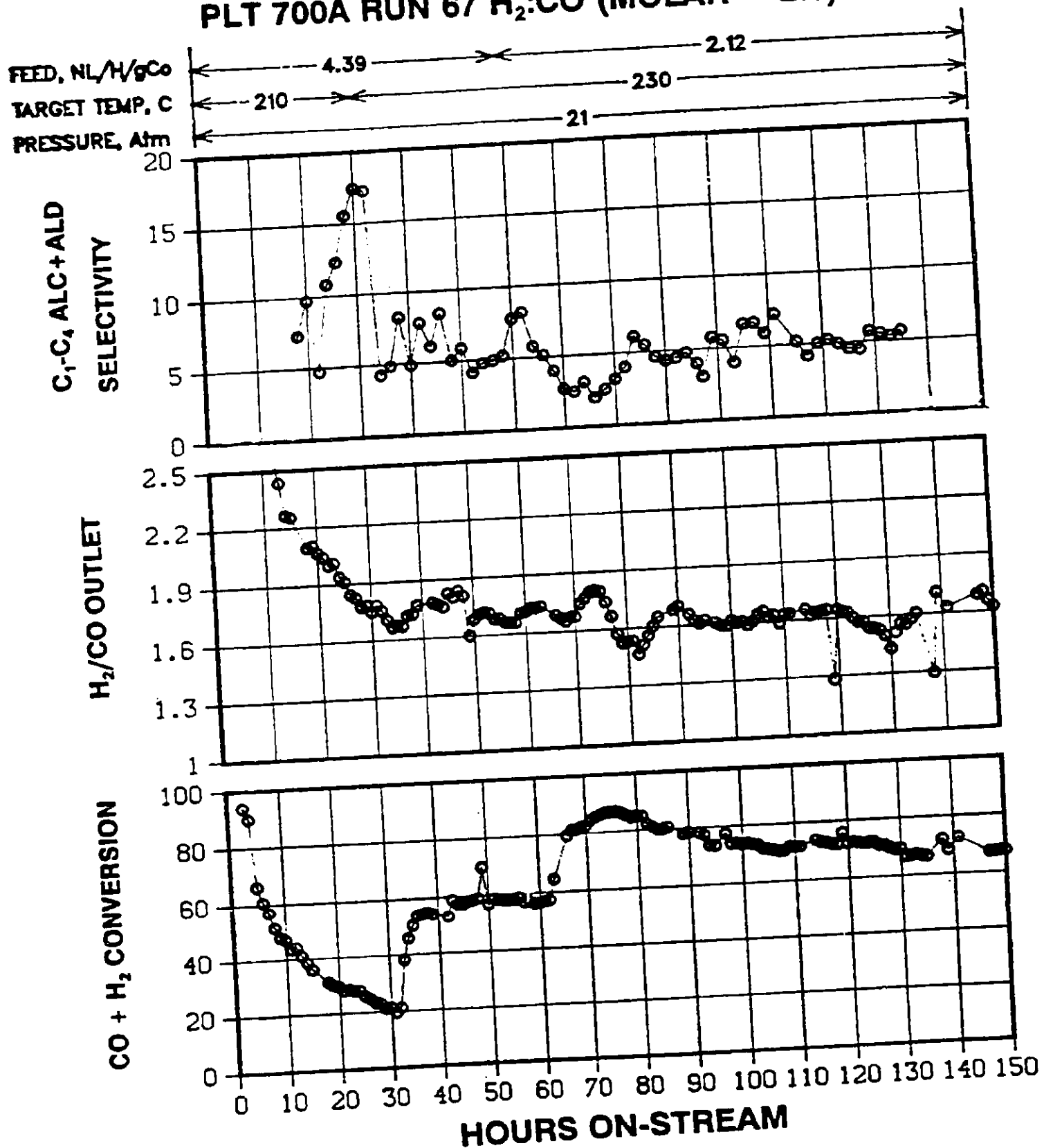
**FIGURE 4: BIMETALLIC Co CATALYST 585R2796
 PLT 700A RUN 67 H₂:CO (MOLAR = 2.0)**



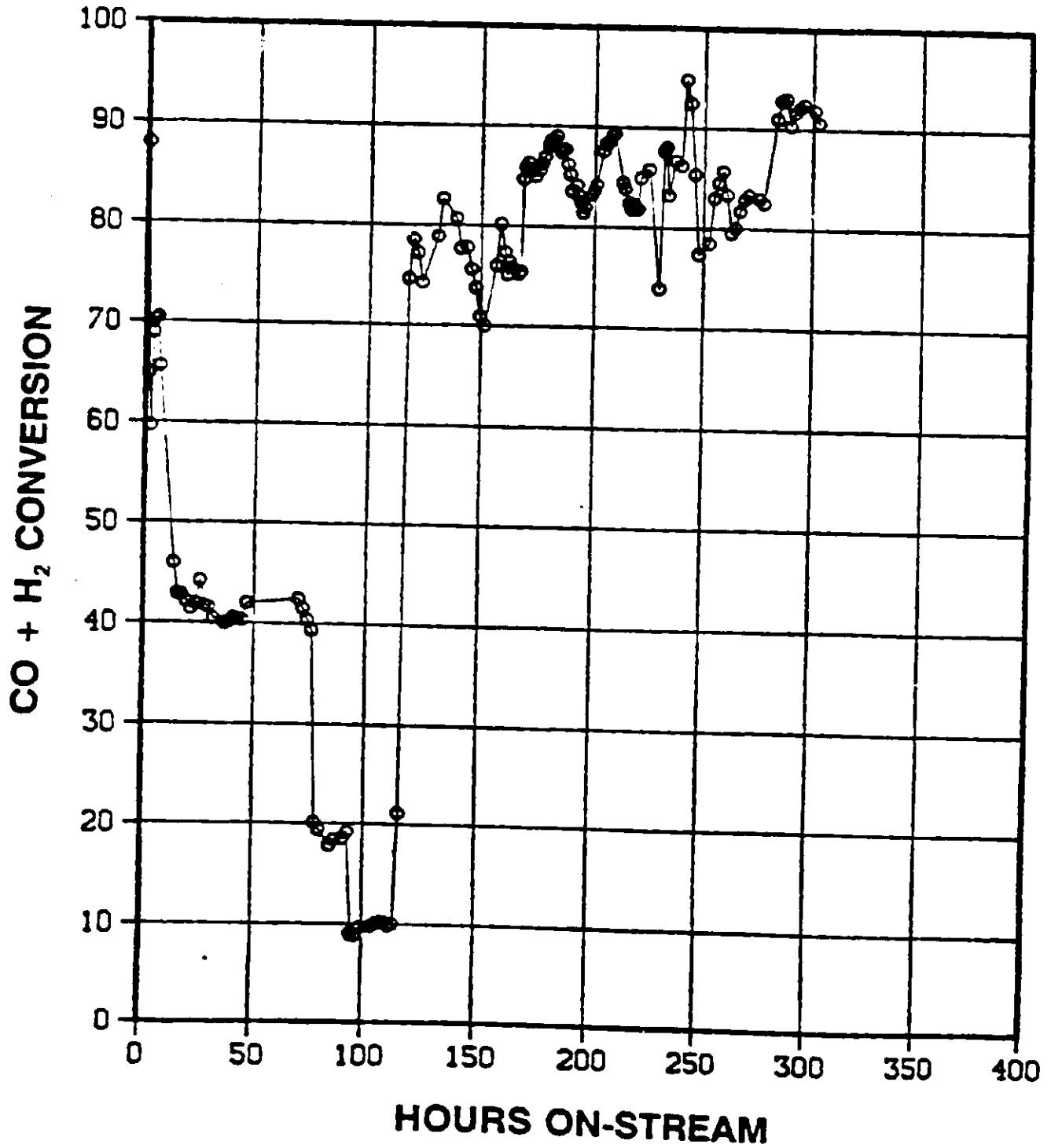
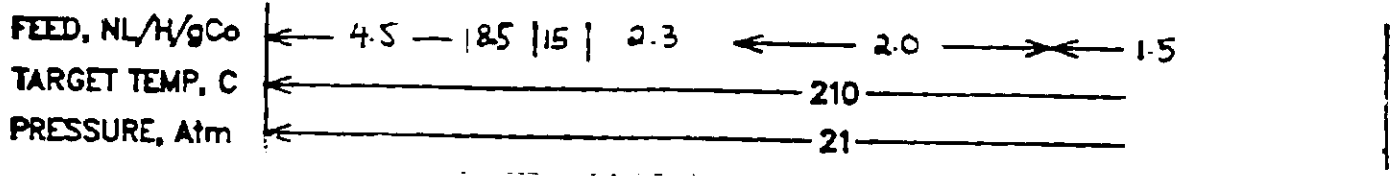
**FIGURE 5: BIMETALLIC Co CATALYST 585R2796
PLT 700A RUN 67 H₂:CO (MOLAR = 2.0)**



**FIGURE 6: BIMETALLIC Co CATALYST 585R2796
 PLT 700A RUN 67 H₂:CO (MOLAR = 2.0)**



**FIGURE 7: REFERENCE CATALYST TC 211
PLT 700A RUN 65 H₂:CO (FEED = 2.0)**



**FIGURE 8: BIMETALLIC Co CATALYST TC 211
 PLT 700A RUN 65 H₂:CO (FEED = 2.0)**

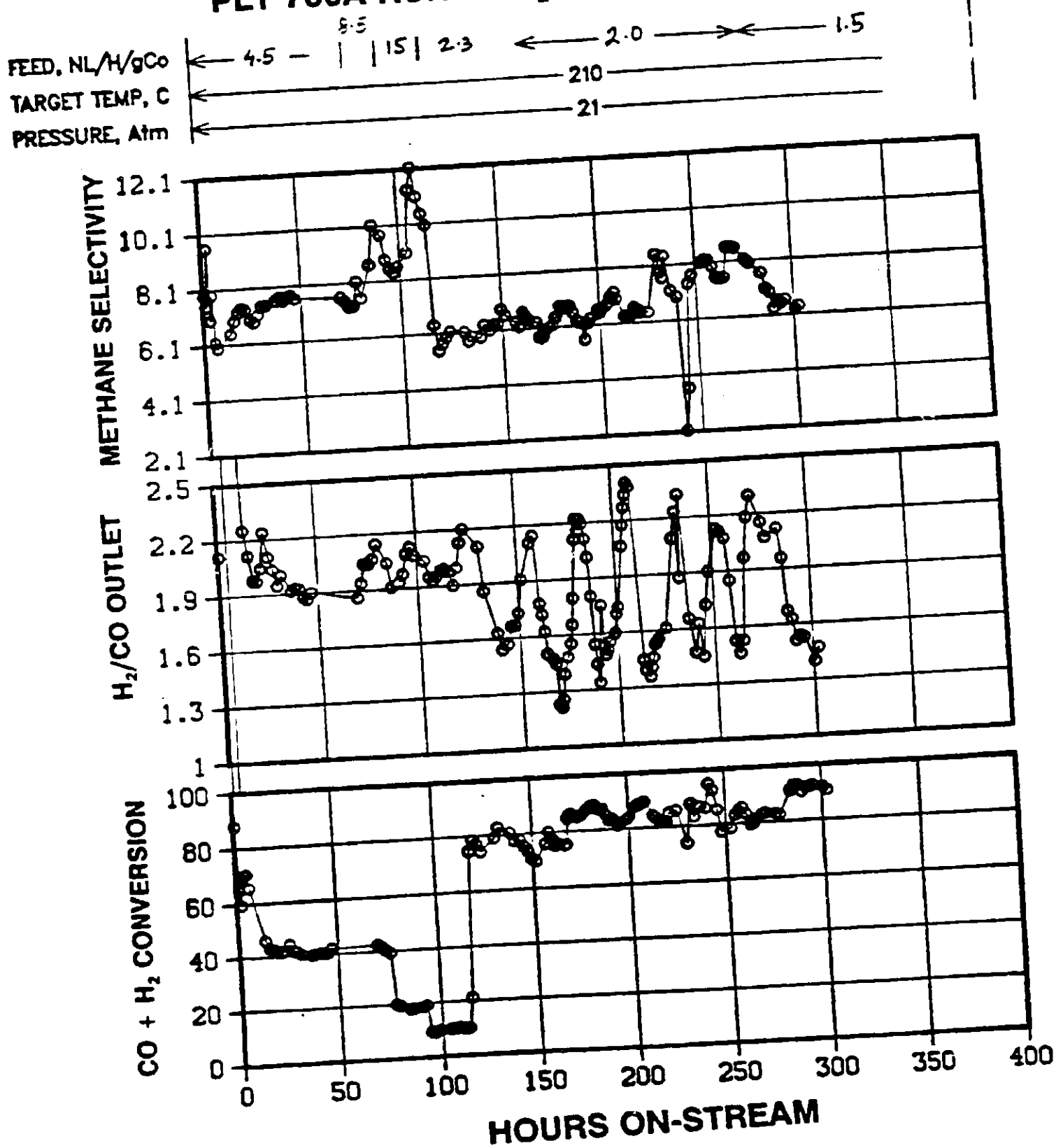
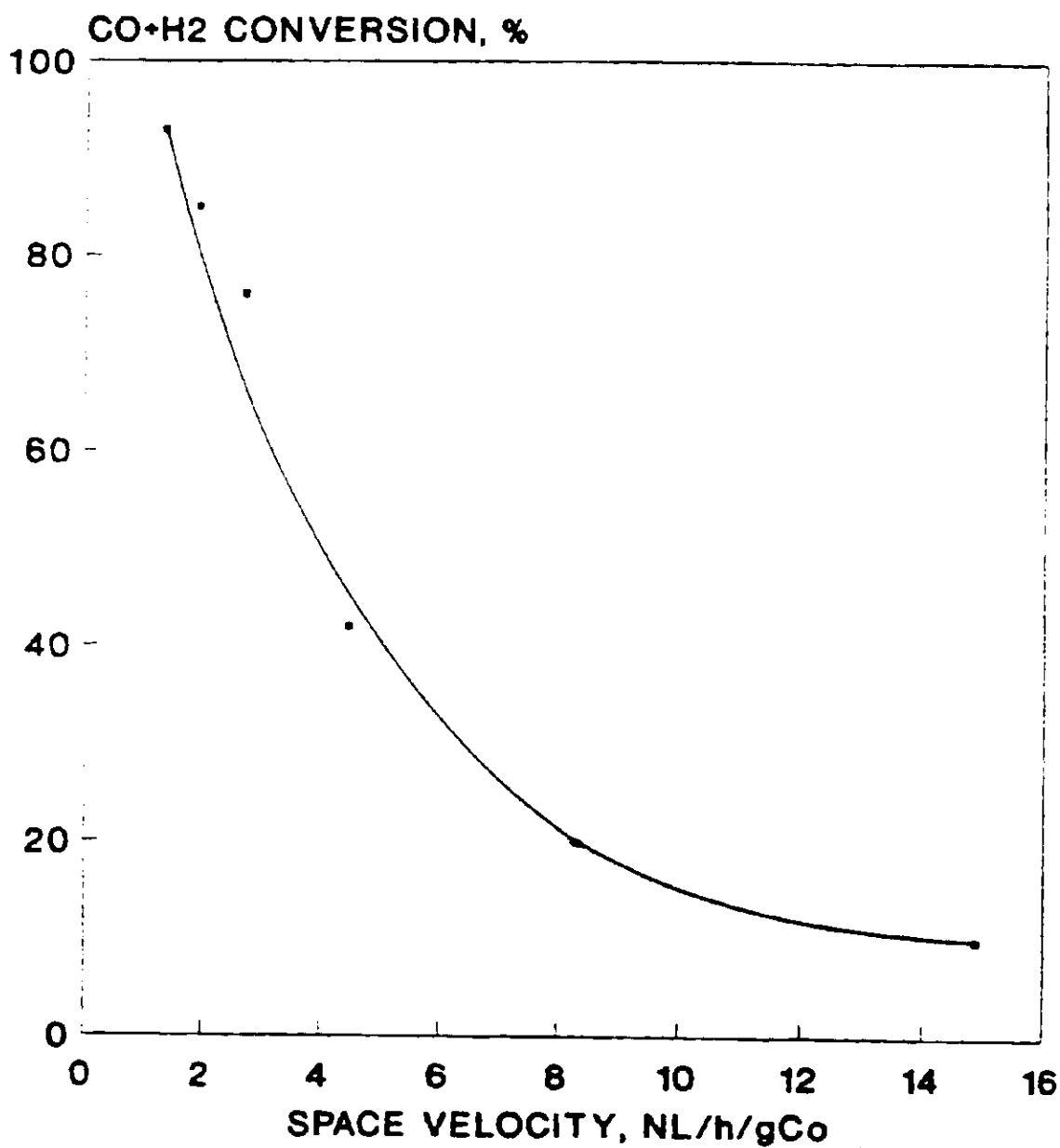
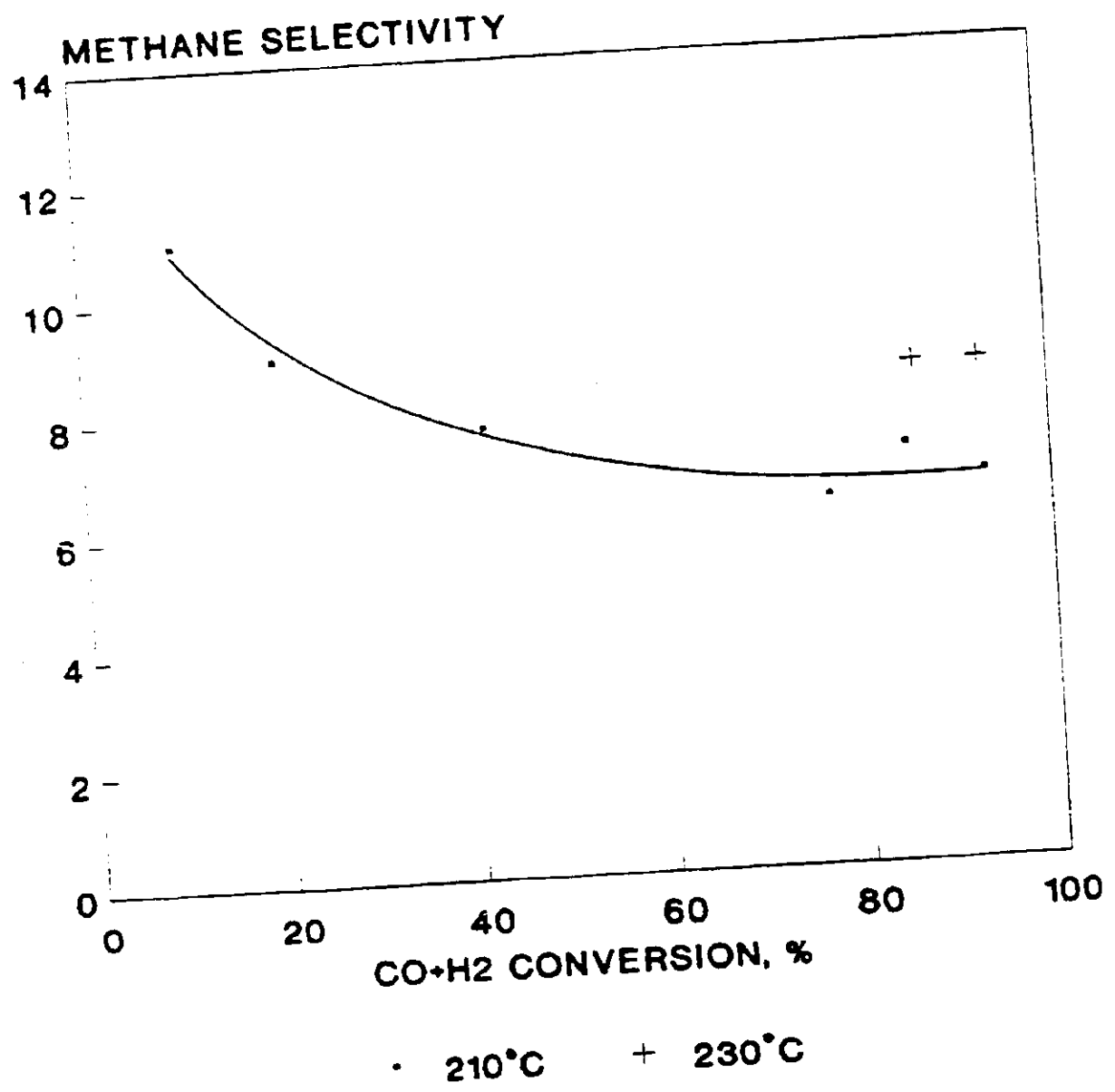


Figure 9: RELATION BETWEEN CO+H₂ CONVERSION AND SPACE VELOCITY WITH REFERENCE Co CATALYST TC 211



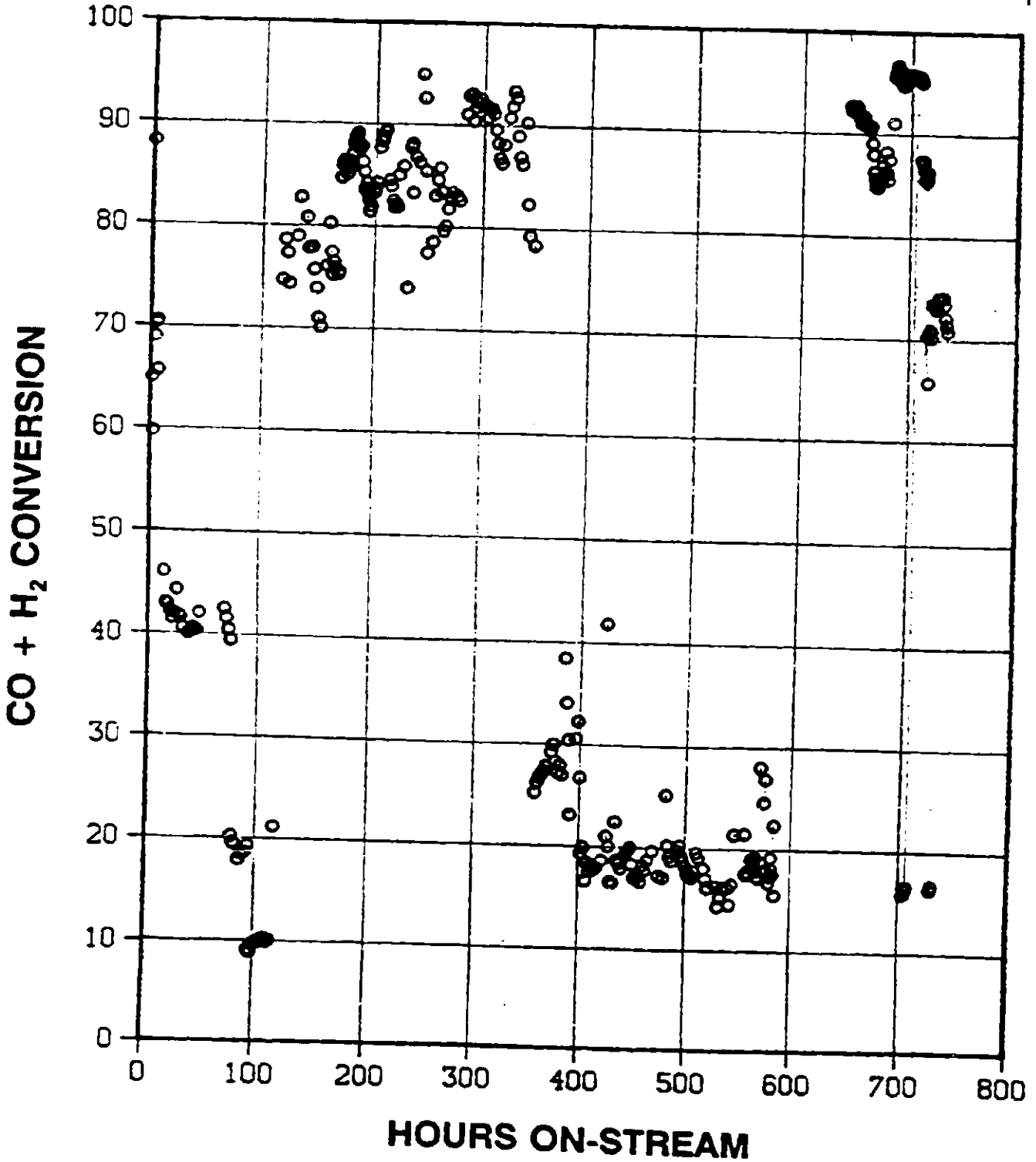
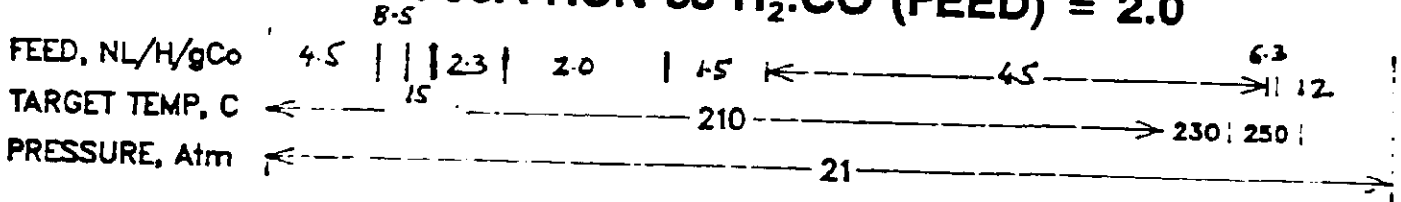
210 C, 21 Atm, 2H₂:1CO Feed

Figure 10: METHANE SELECTIVITY
AT DIFFERENT CONVERSIONS
WITH REFERENCE Co CATALYST TC 211

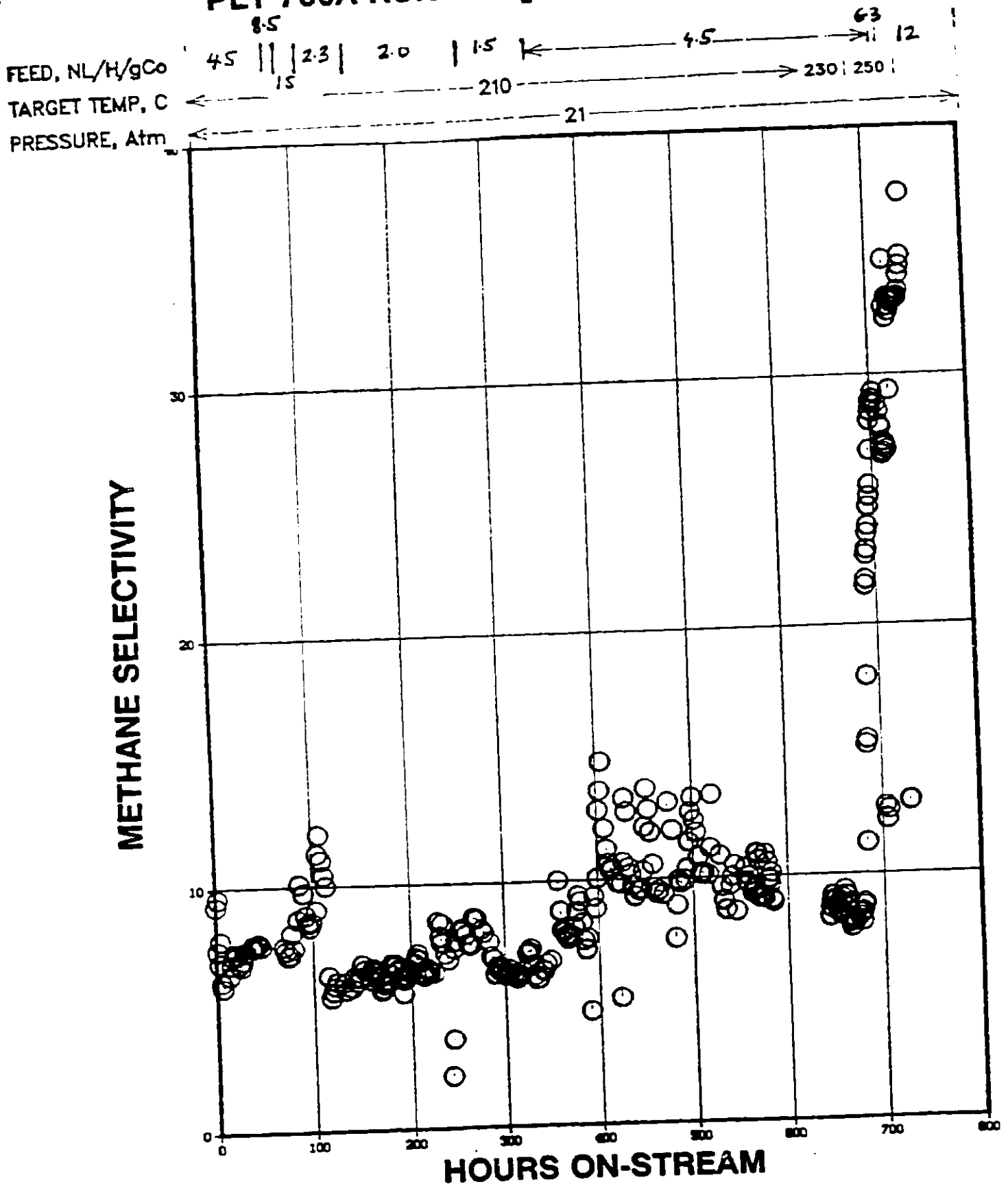


21 Atm, 2H2:1CO Feed

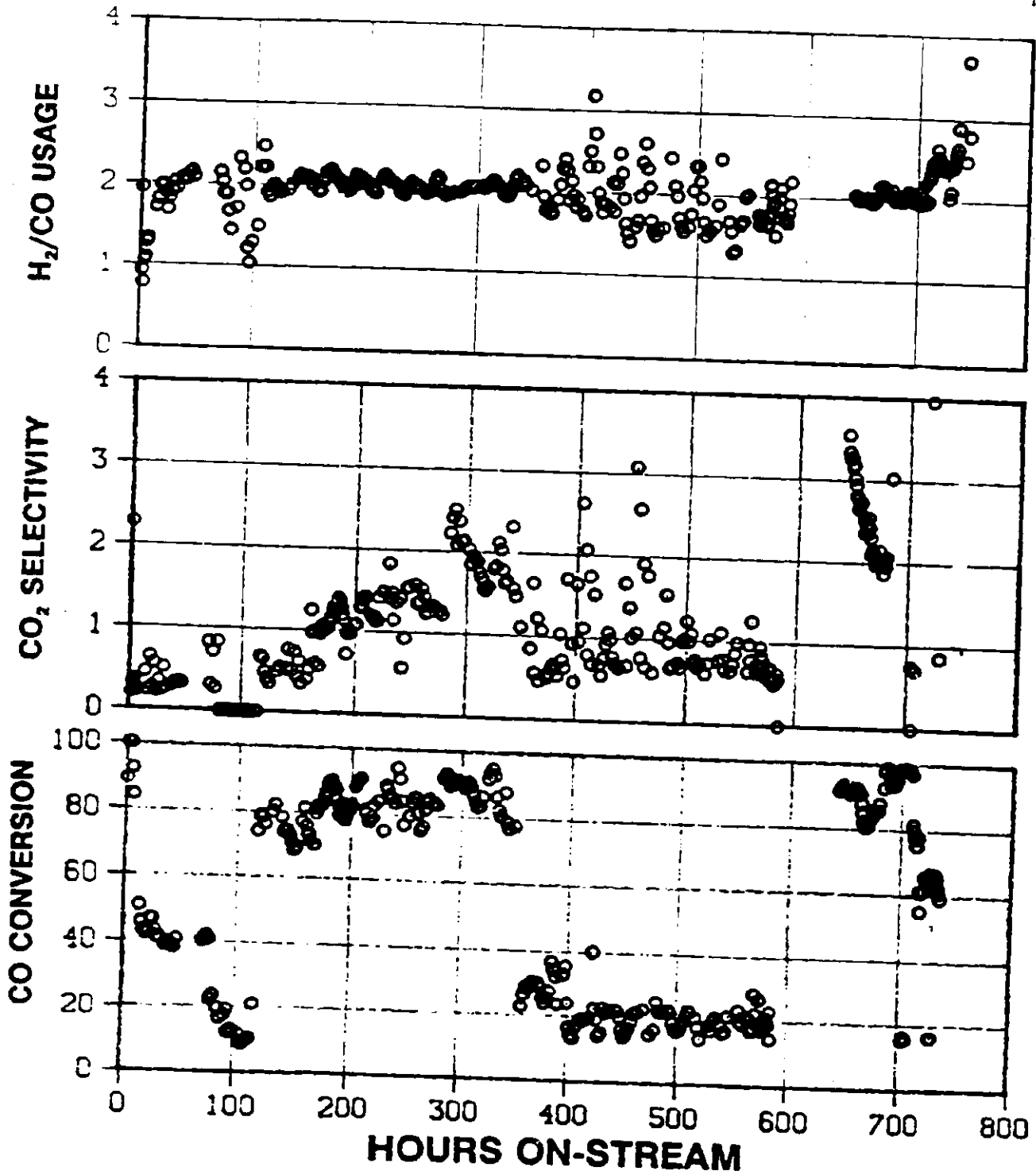
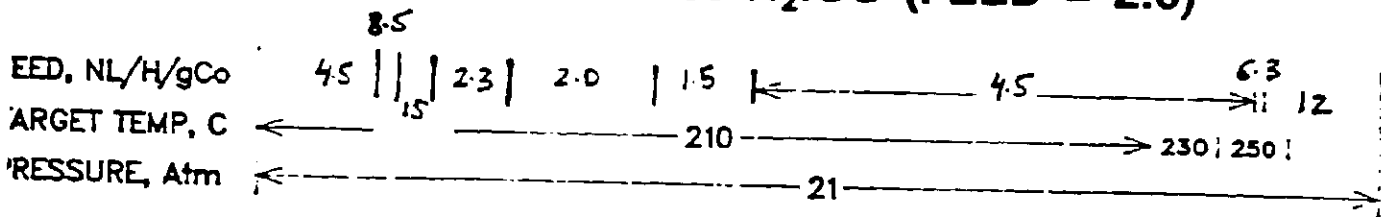
FIGURE 11: REFERENCE Co CATALYST TC 211
PLT 700A RUN 65 H₂:CO (FEED) = 2.0



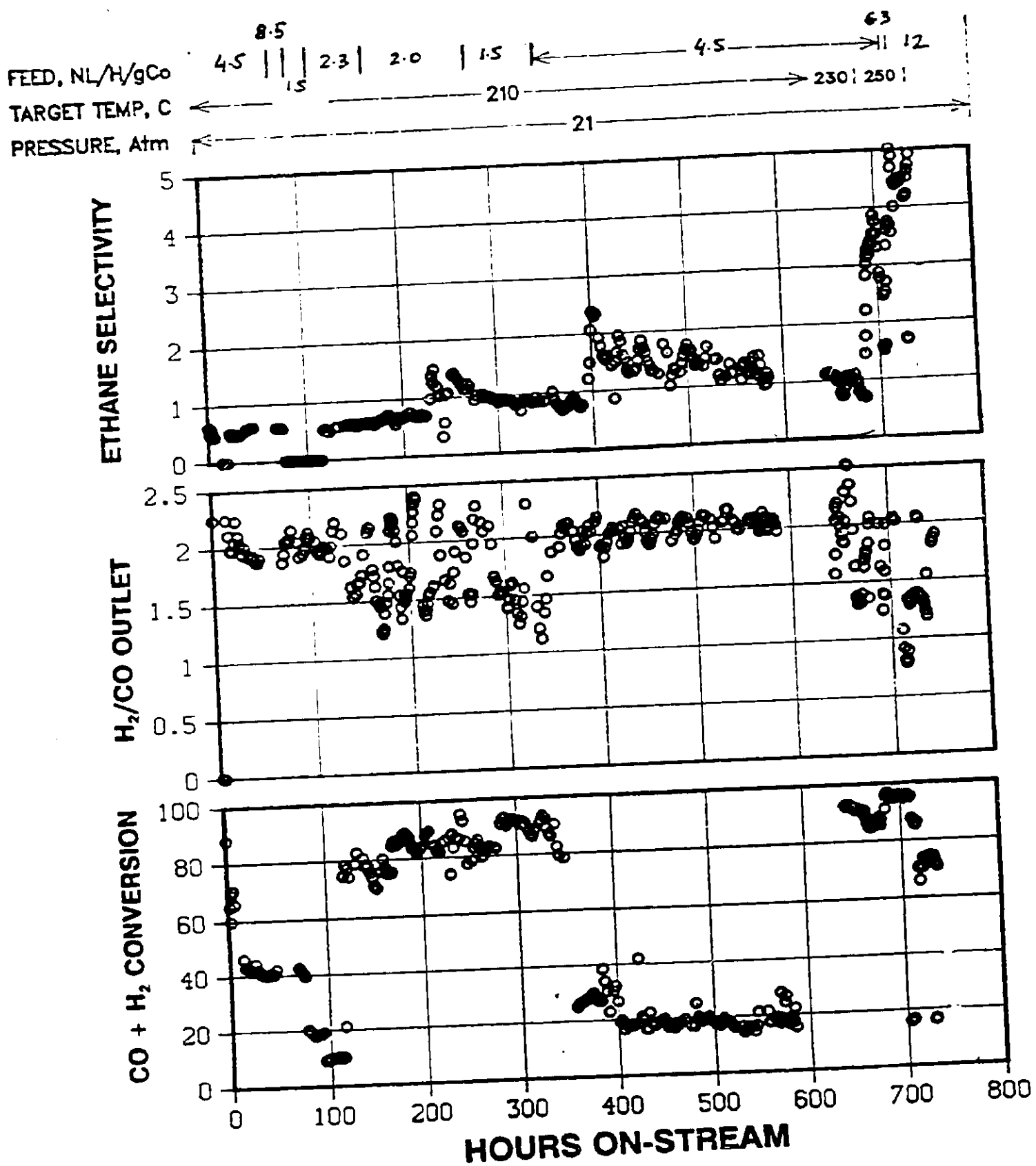
**FIGURE 12: REFERENCE Co CATALYST TC 211
PLT 700A RUN 65 H₂:CO (FEED = 2.0)**



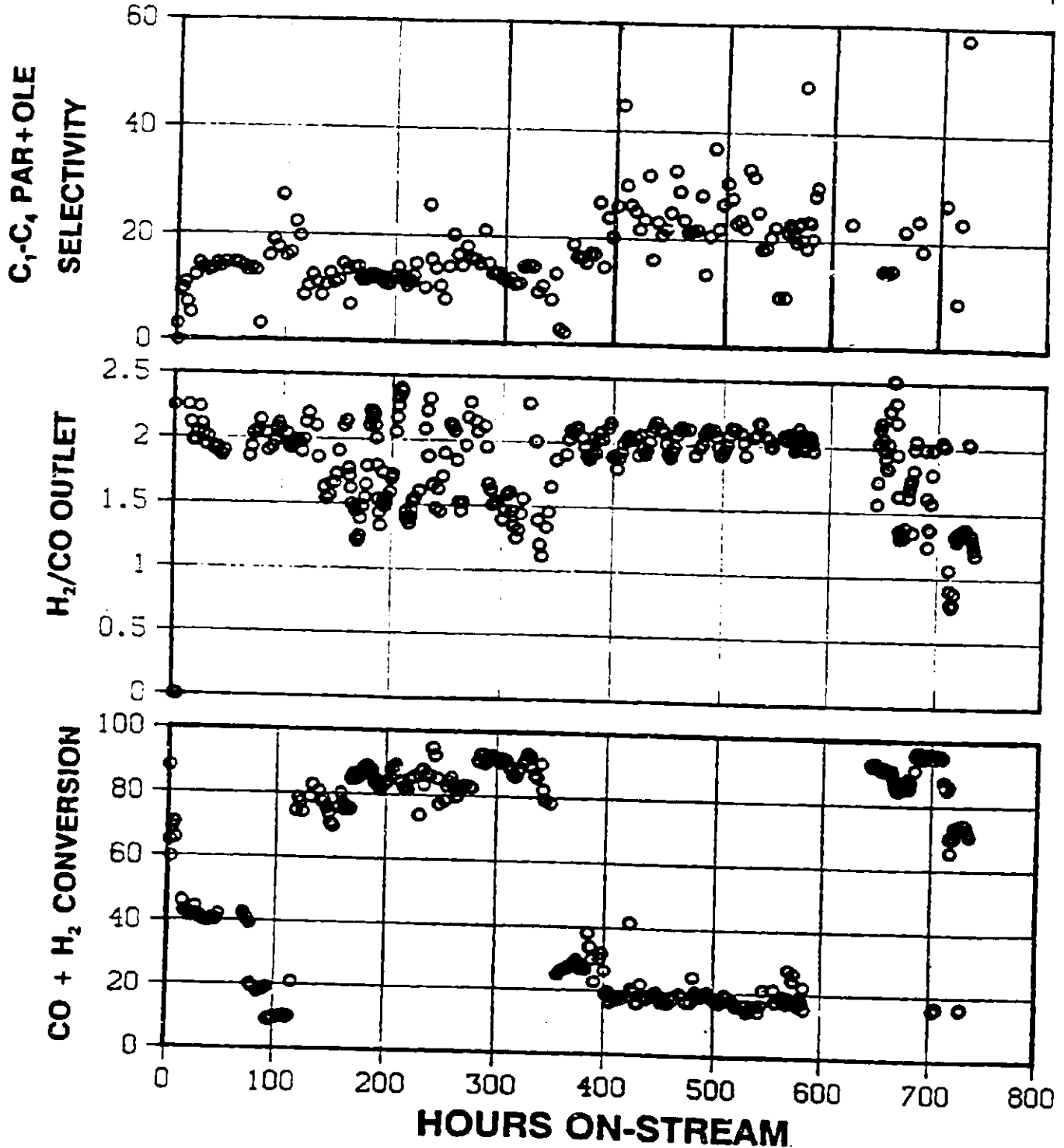
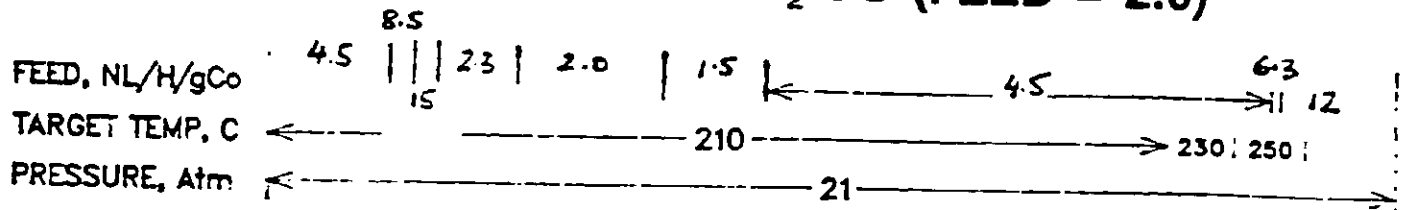
**FIGURE 13: REFERENCE Co CATALYST TC 211
PLT 700A RUN 65 H₂:CO (FEED = 2.0)**



**FIGURE 14: REFERENCE Co CATALYST TC 211
PLT 700A RUN 65 H₂:CO (FEED = 2.0)**



**FIGURE 15: REFERENCE Co CATALYST TC 211
PLT 700A RUN 65 H₂:CO (FEED = 2.0)**



**FIGURE 16: REFERENCE Co CATALYST TC 211
PLT 700A RUN 65 H₂:CO (FEED = 2.0)**

